

EUROPEAN DRUG MASTER FILE
FOR
BETADEX SULFOBUTYL ETHER SODIUM
(NON-STERILE, EXCIPIENT)

(CTD, APPLICANTS PART)

Module 3: Quality

Name of EDMF holder: Watson International Ltd. P. R. China

Name of Excipient: Betadex Sulfobutyl Ether Sodium

Version No.:

Watson/Betadex Sulfobutyl Ether Sodium/AP/01/2016.02.19

February, 2016

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3.2 BODY OF DATA

3.2.S EXCIPIENT

3.2.S.1 General Information

3.2.S.1.1 Nomenclature

INN: Betadex Sulfobutyl Ether Sodium

U.S. Pharmacopeia Name: Betadex Sulfobutyl Ether Sodium

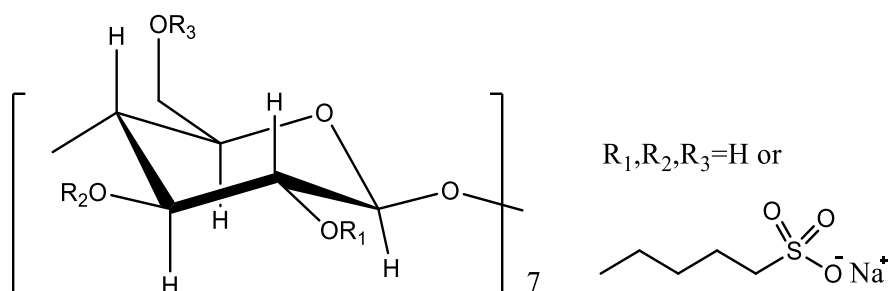
System chemical name: (4- sulfoacid-1- butyl ether) – annular heptamer D-glucopyranose

Laboratory name: SBE-β-CD

CAS registry No.: 182410-00-0

3.2.S.1.2 Structure

Structure formula:



Molecular formula: $\text{C}_{42}\text{H}_{70-n}\text{O}_{35} \cdot (\text{C}_4\text{H}_8\text{SO}_3\text{Na})_n$ (n is the average degree of substitution, n=6.2~6.9)

Molecular weight: $1134.98 + 158.15n$

3.2.S.1.3 General Properties

The product is white or off-white powder, very soluble in water; slightly soluble in methanol; practically insoluble in ethanol or dichloromethane.

<i>Item</i>	<i>Character</i>
Character	The product is white or slightly gray powder without foreign matters
pH	pH of water solution of this product at $25^\circ\text{C} \pm 0.5^\circ\text{C}$ is 4.0~6.8
Crystal form	Our product is amorphous powder.

3.2.S.2 Manufacture**3.2.S.2.1 Manufacturer****Manufacturer**

Name: Watson International Ltd	
Address: No.688 Middle Maoshatang Road, Kunshan City, Jiangsu Province, China	
Postcode: 215332	Website: www.watson-int.com
Tel.: +86-512-81867260/70/80	City: Kunshan
Fax: +86-512-81867260/70/80-3	Province: Jiangsu
Email: contact@watson-int.com	Country: People's Republic of China

DMF holder

Name: Watson International Ltd	
Address: No.688 Middle Maoshatang Road, Kunshan City, Jiangsu Province, China	
Postcode: 215332	Postcode: 215332
Tel.: +86-512-81867260/70/80	Tel.: +86-512-81867260/70/80
Fax: +86-512-81867260/70/80-3	Fax: +86-512-81867260/70/80-3
Email: contact@watson-int.com	Email: contact@watson-int.com

Manufacturing site

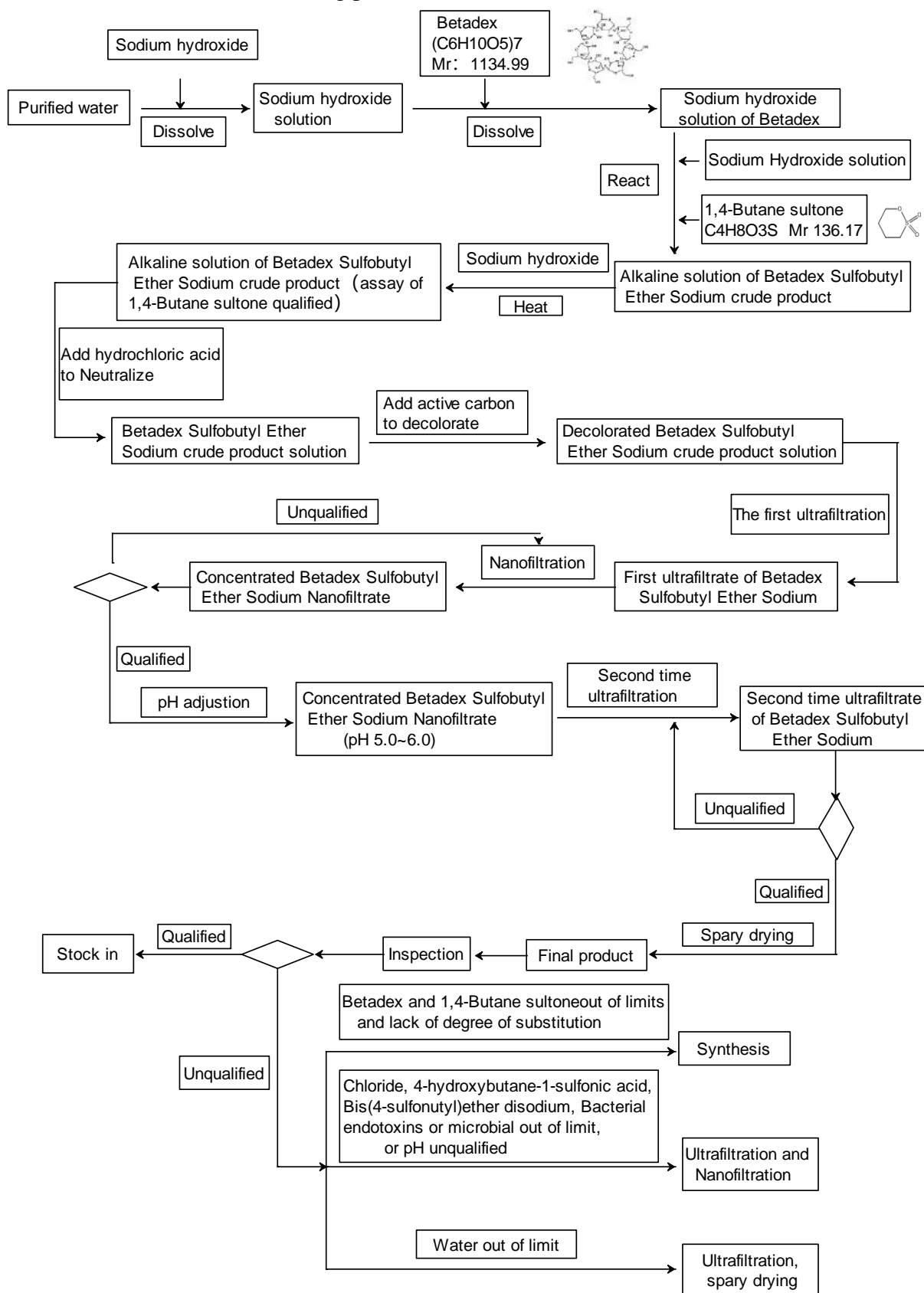
Name: Watson International Ltd	
Address: No.688 Middle Maoshatang Road, Kunshan City, Jiangsu Province, China	
Postcode: 215332	Postcode: 215332
Tel.: +86-512-81867260/70/80	Tel.: +86-512-81867260/70/80
Fax: +86-512-81867260/70/80-3	Fax: +86-512-81867260/70/80-3
Email: contact@watson-int.com	Email: contact@watson-int.com

3.2.S.2.2 Description of manufacturing process and process controls

1. Brief description of manufacturing process and process controls

Betadex is the starting material, and it is translated to crude product liquid at alkaline condition through one step of hydroxyalkylation reaction with 1,4-Butane sultone; and the crude product liquid is then handled through first time ultrafiltration, nanofiltration and adjustment of pH value, and the Betadex Sulfobutyl Ether Sodium concentrated nanofiltration is obtained, then the concentration is ultrafiltrated for another time and spray dried, and the Betadex Sulfobutyl Ether Sodium final product is achieved.

2. Flow chart of the manufacturing process



3. Detailed description of manufacturing process and process controls

It is reported in the restricted part of the EDMF.

3.2.S.2.3 Control of Materials

List of material used in the manufacturing process

<i>Serials NO.</i>	<i>Materials name</i>	<i>Molecular formula</i>	<i>Category and process used</i>	<i>Grade</i>	<i>Standard</i>
1	Betadex	$(C_6H_{10}O_5)_7$	1 st step	Medicinal grade	In-house standard
2	1,4-Butane sultone	$C_4H_8O_3S$	1 st step	Industrial grade	In-house standard
3	Purified water	H_2O	1 st , 2 nd , 3 rd , 4 th and 5 th step	/	In-house standard
4	Sodium hydroxide	$NaOH$	1 st and 4 th step	Food grade	In-house standard
5	Hydrochloric acid	HCl	1 st and 4 th step	Food grade	In-house standard
6	Active carbon	/	1 st step	Food grade	In-house standard

Specifications, analysis procedures and CoAs are reported in the restricted part of the EDMF.

3.2.S.2.4 Controls of Critical Steps and Intermediates

It is reported in the restricted part of the EDMF.

3.2.S.3 Characterization

3.2.S.3.1 Elucidation of Structure and Other Characteristics

In order to confirm the structure of Betadex Sulfobutyl Ether Sodium manufactured by Watson International Ltd elemental analysis, UV spectrophotometry, infrared spectrophotometry, nuclear magnetic resonance spectroscopy, X-ray diffraction and thermal analysis are performed. The information of sample and reference standard is listed in the following table.

<i>Name</i>	<i>Origin</i>	<i>Batch No.</i>
Betadex Sulfobutyl Ether Sodium RS	Cydex Pharmaceuticals Company	NC-04A-140128
Betadex Sulfobutyl Ether Sodium sample	Watson International Ltd	20130923

1. Elemental analysis

- Performed by: Shanghai Analysis and Test Center
- Instrument: Elementar Vario ELIII elemental analyzer

Results: The data obtained can be seen below.

Data of elemental analysis

Element name	Betadex Sulfobutyl Ether Sodium sample			Betadex Sulfobutyl Ether Sodium RS		
	1	2	Average	1	2	Average
C	34.64	34.97	34.80	34.67	34.79	34.73
H	5.00	4.96	4.98	4.98	4.92	4.95

Conclusion: the content of carbon and hydrogen of Betadex Sulfobutyl Ether Sodium sample is consistent with the values of Betadex Sulfobutyl Ether Sodium RS.

N₂: SFW149047

共 2 页 第 2 页

[illegible]

表 1-1 磺丁基醚倍他环糊精钠盐样品及对照品的元素分析结果

2. Infrared spectroscopy (IR) analysis

- Instrument: Bruker Vertex-70 FT-IR
- Test condition: KBr film
- Performed by: Shanghai Analysis and Test Center

Results: The test results can be seen below,

Test result of IR

Absorption peak of sample (cm^{-1})	Absorption peak of RS (cm^{-1})	Vibration type	Attribution group
3430	3432	$\nu_{\text{O-H}}$	-OH
2937	2937	ν_{CH_2}	-CH ₂ -
1459	1459	δ_{CH_2}	-CH ₂ -

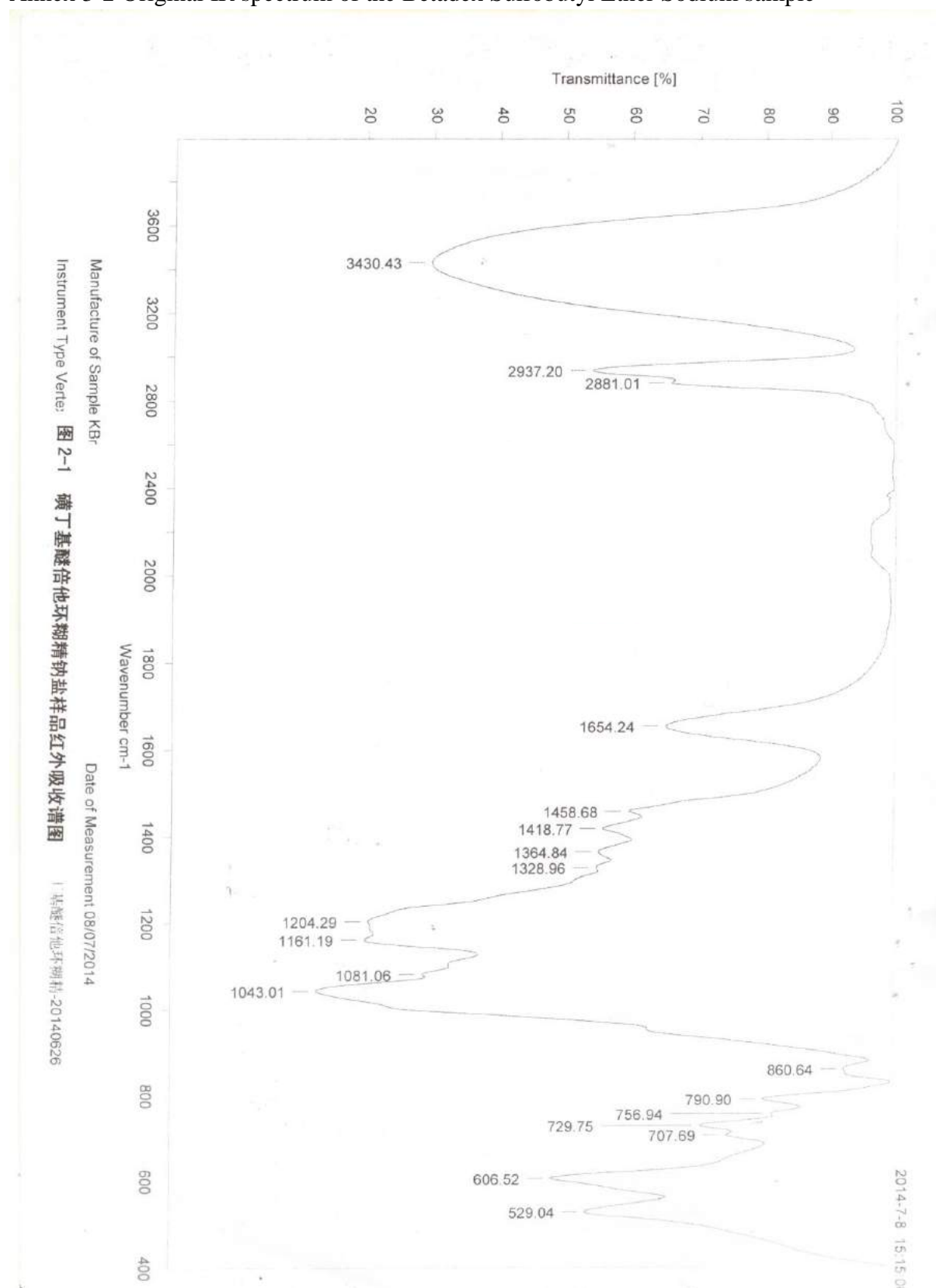
Analysis:

1. Stretching vibration absorption at the location 3430cm^{-1} indicates Hydroxyl groups exist in the sample structure.
2. Stretching vibration absorption of CH in methylene at the location 2937cm^{-1} and bending vibration of CH in methylene 1459cm^{-1} indicate Methylenes exist in the sample structure.

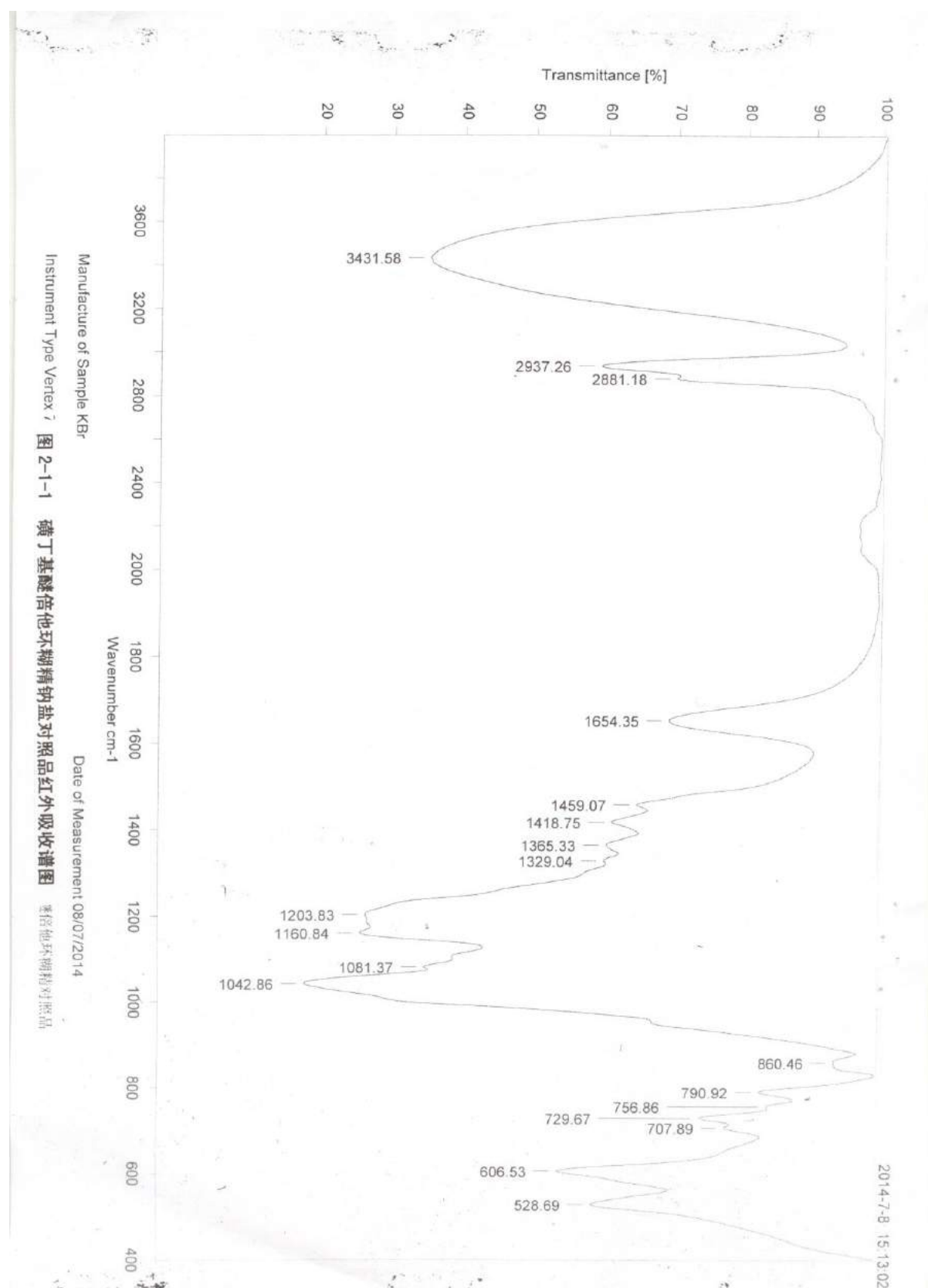
Conclusion:

The IR spectrum of the Betadex Sulfobutyl Ether Sodium is consistent with that of the Betadex Sulfobutyl Ether Sodium RS.

Annex 3-2 Original IR spectrum of the Betadex Sulfobutyl Ether Sodium sample



Annex 3-3 Original IR spectrum of the Betadex Sulfobutyl Ether Sodium RS



3. UV spectrophotometry

- Instrument: SHIMADZU UV-2550 ultraviolet-visible spectrophotometer
- Test condition: dissolved in water and test the absorbance at 190~400nm.
- Performed by: Shanghai Analysis and Test Center

Results: The test results can be seen below,

Test results of UV spectrophotometry

Solvent	Test sample	$\lambda_{\max}(\text{nm})$	A	E (L·mol ⁻¹ ·cm ⁻¹)	Remarks
Water	Betadex Sulfobutyl Ether Sodium sample	198	0.032	/	/
	Betadex Sulfobutyl Ether Sodium RS	198	0.015	/	/

Conclusion:

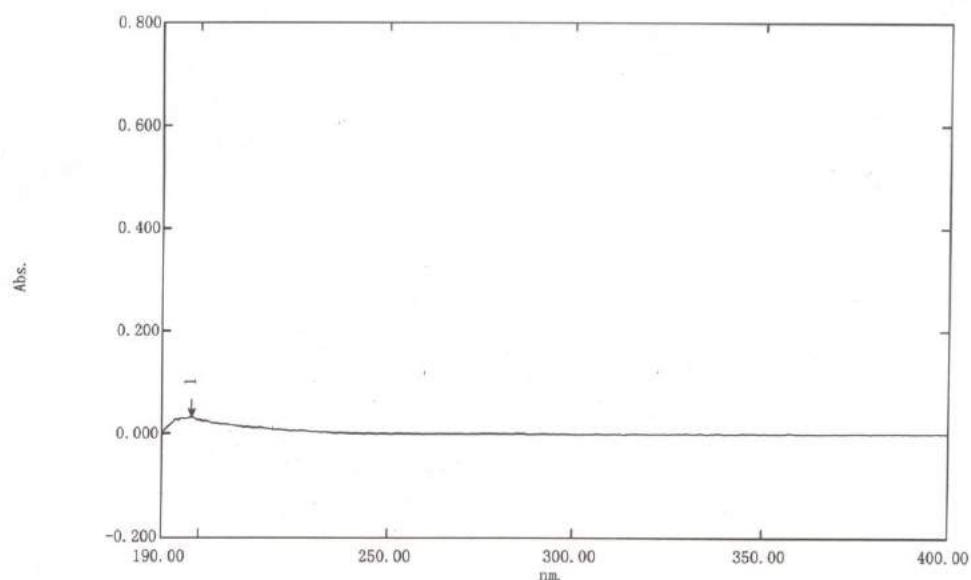
The λ_{\max} and the absorbance of Betadex Sulfobutyl Ether Sodium sample are consistent with that of the RS.

Annex 3-4 Original spectrum of Betadex Sulfobutyl Ether Sodium sample

光谱峰值检测报告

2014-07-15 10:36:30

数据集: Storage 103056 - RawData - E:\磺丁基醚倍他环糊精钠盐\样-20140626-H2O.spc



测定属性
波长范围 (nm.): 190.00 到 400.00
扫描速度: 高速
采样间隔: 0.2
自动采样间隔: 激活
扫描模式: 单一的

No.	P/V	波长 (nm)	吸收值
1		197.80	0.032

试样准备属性

重量:
体积:
稀释:
光程长:
附加信息:

仪器属性

仪器类型: UV-2500PC Series
测定方式: 吸收值
狭缝宽: 2.0 nm
光源转换波长: 360.0 nm
S/R 转换: 标准

附件属性

附件: 无

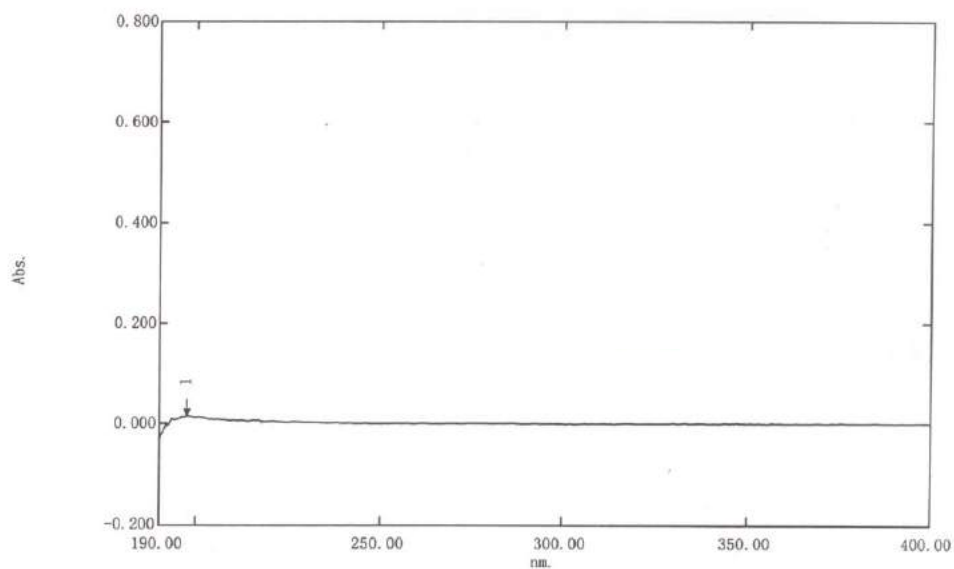
图 3-1 磺丁基醚倍他环糊精钠盐样品在水中的紫外图谱

Annex 3-5 Original spectrum of Betadex Sulfobutyl Ether Sodium RS

光谱峰值检测报告

2014-07-15 10:39:17

数据集: Storage 103828 - RawData - E:\磺丁基醚倍他环糊精钠盐\对-H2O.spc



测定属性
波长范围 (nm.): 190.00 到 400.00
扫描速度: 高速
采样间隔: 0.2
自动采样间隔: 激活
扫描模式: 单一的

No.	P/V	波长 (nm)	吸收值
1		197.60	0.015

试样准备属性
重量:
体积:
稀释:
光程长:
附加信息:

仪器属性
仪器类型: UV-2500PC Series
测定方式: 吸收值
狭缝宽: 2.0 nm
光源转换波长: 360.0 nm
S/R 转换: 标准

附件属性
附件: 无

图 3-1-1 磺丁基醚倍他环糊精钠盐对照品在水中的紫外图谱

4. Nuclear magnetic resonance spectroscopy (NMR) analysis

- Instrument: Varian INOVA-600 NMR analyzer
- Solvent: D₂O
- Performed by: Shanghai Analysis and Test Center

¹H-NMR

Results: The test results of the ¹H-NMR can be seen below,

Signal of proton	Test sample	P ₁	P ₂ P ₃ P ₄ P ₅ P ₆ H ₁	H ₄	H ₂ H ₃
Chemical shift (ppm)	Sample	5.101~4.944	3.871~3.390	2.811~2.802	1.666~1.623
	RS	5.104~4.964	3.891~3.366	~2.826	1.677~1.655

Remark: P is glucose, Q is sulfobutyl ether.

Analysis:

1. δ1.666~1.623 is the signal of proton of methylene on the second and third location of sulfobutyl ether. δ2.811~2.802 is the signal of proton of methylene that connects sulfo groups in sulfobutyl ether.
2. δ5.101~4.944 is the representative chemical shift of proton on carbon that connect two oxygen, and δ3.871~3.390 is the other carbon that only connect with one oxygen.

¹³C -NMR

Results: The test results of the ¹³C -NMR can be seen below,

Signal of proton	Test sample	P ₁	P ₂ P ₃ P ₄ P ₅ P ₆ H ₁	H ₄	H ₂	H ₃
Chemical shift (ppm)	Sample	101.486~98.976	80.973~60.215	50.702~50.580	28.197~27.434	20.881~20.515
	RS	101.540~100.296	81.026~60.245	50.724~50.610	28.220~27.464	20.911~20.560

Remark: P is glucose, Q is sulfobutyl ether.

Analysis:

1. δ20.881~20.515 and δ28.197~27.434 is respectively the signal of carbons on the third and the second location in sulfobutyl ether.
2. δ50.702~50.580 is the signal of carbon of methylene that connects sulfo groups.
3. δ101.486~98.976 is the con-bond of carbon and the two oxygen next to the carbon, and δ80.973~60.215 is the con-bond of carbon and the one oxygen next to the carbon.

Conclusion:

The affiliation of the spectrum peak in ¹H-NMR and ¹³C-NMR of the sample is reasonable

and consistent with the RS.

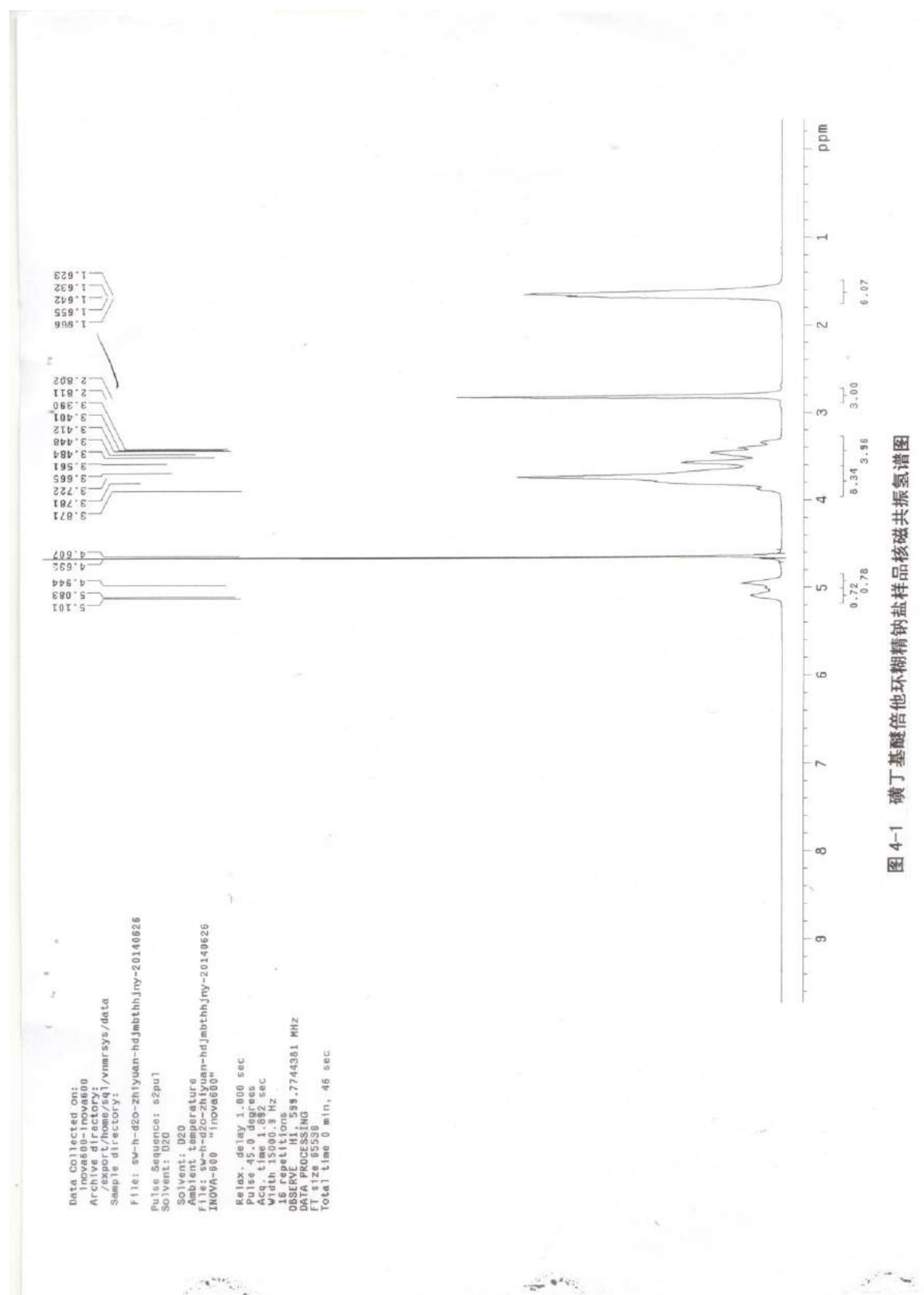
Annex 3-6 Original ^1H -NMR spectrum of Betadex Sulfobutyl Ether Sodium sample

图 4-1 磺丁基醚倍他环糊精钠盐样品核磁共振氢谱图

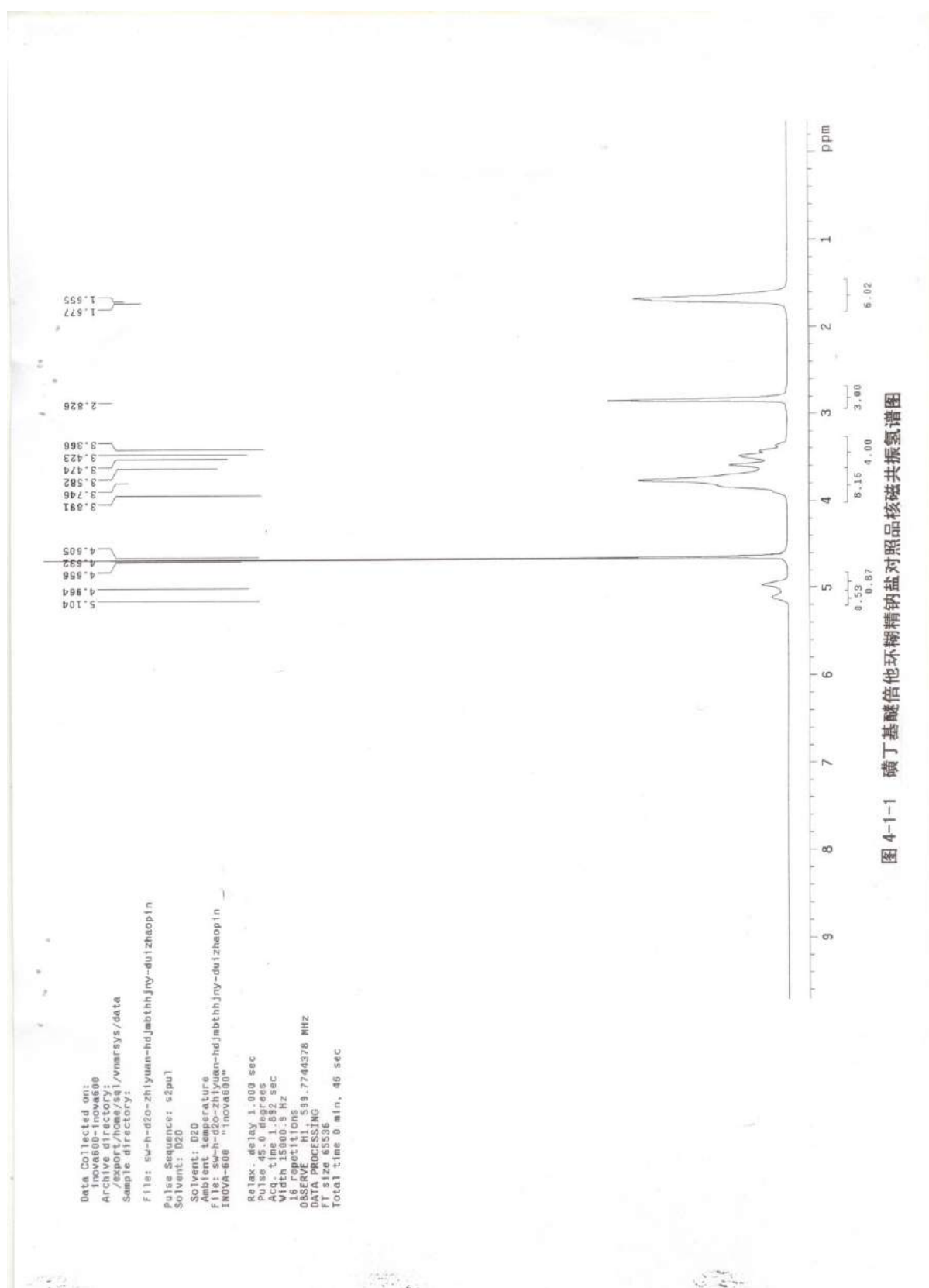
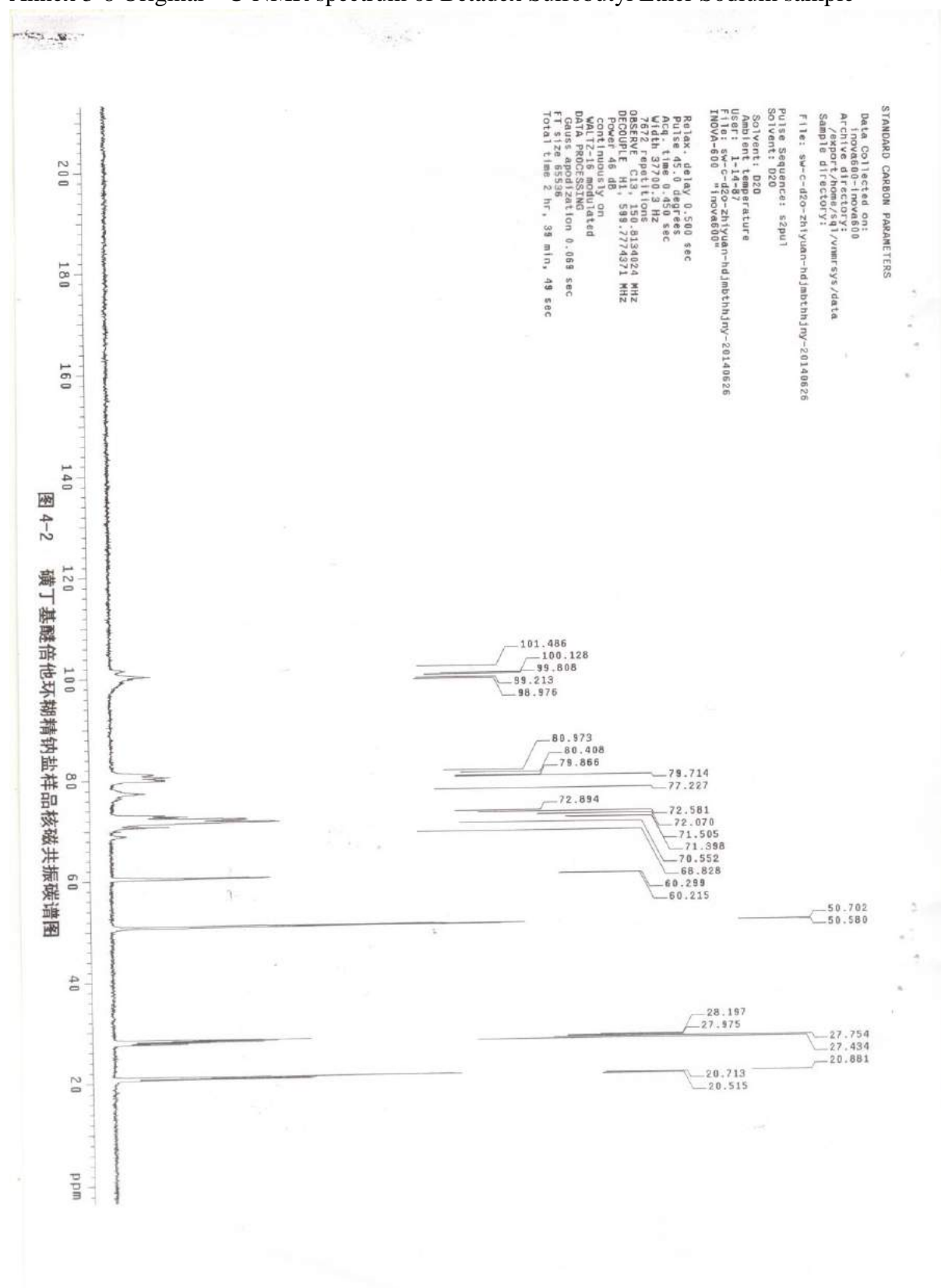
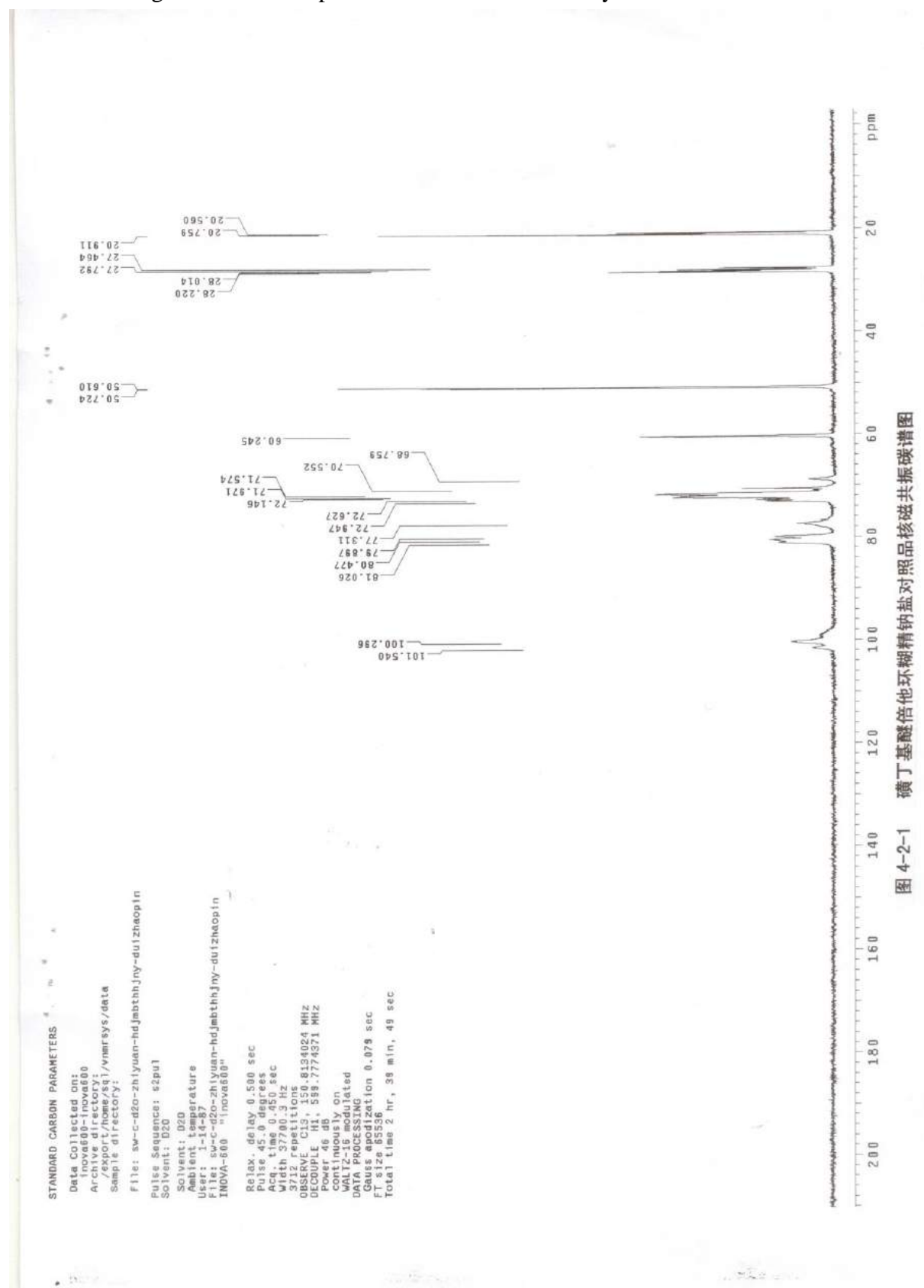
Annex 3-7 Original ^1H -NMR spectrum of Betadex Sulfobutyl Ether Sodium RS

图 4-1-1 磺丁基醚倍他环糊精钠盐对照品核磁共振氢谱图

Annex 3-8 Original ^{13}C -NMR spectrum of Betadex Sulfobutyl Ether Sodium sample

Annex 3-9 Original ^{13}C -NMR spectrum of Betadex Sulfobutyl Ether Sodium RS

5. Thermal analysis

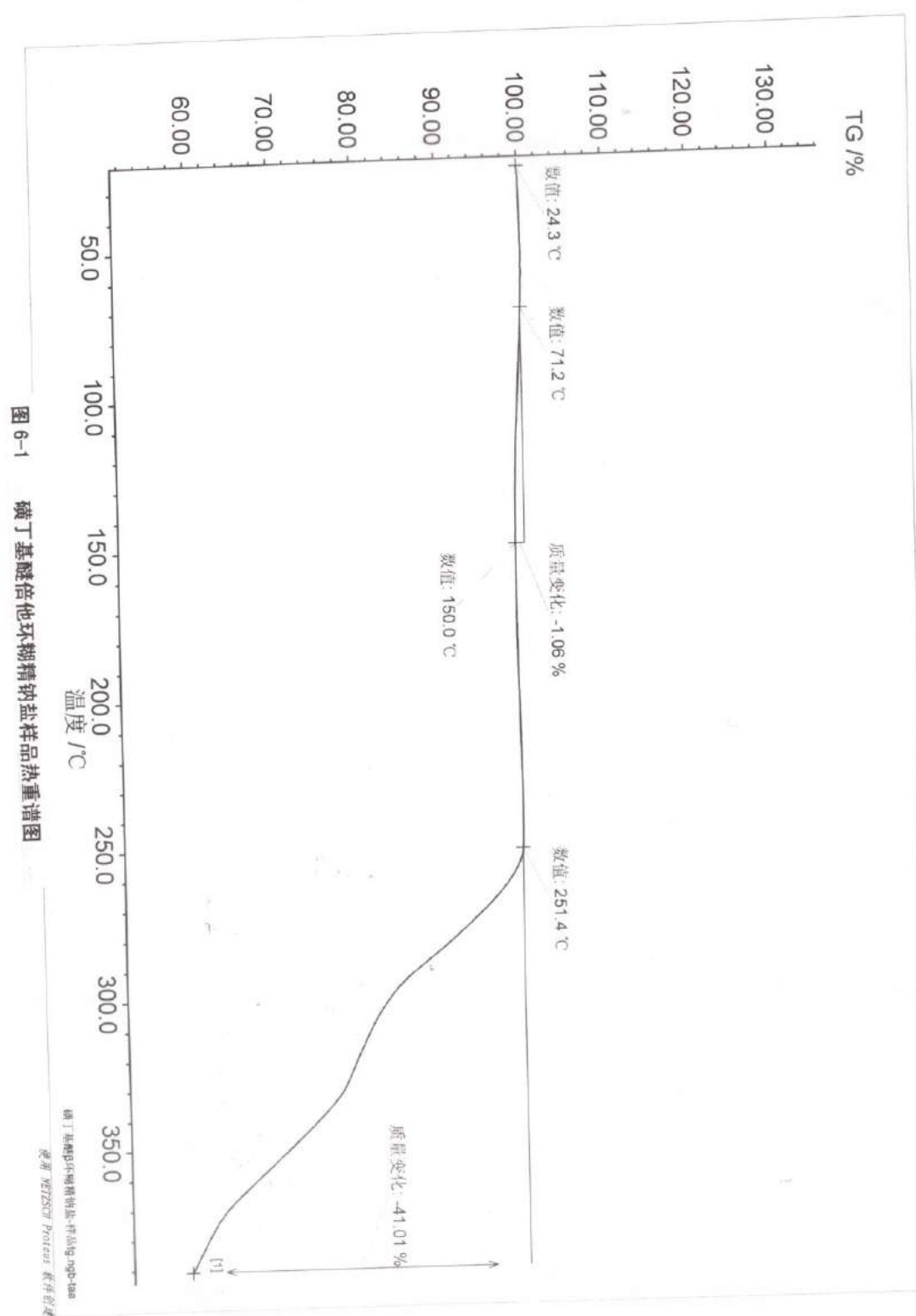
- Instrument: NETZSCH STA 449 F3 simultaneous thermal analyzer
- Testing condition: heating rate 10°C/min, testing temperature range 23~400°C, purge gas helium, flow rate 40ml/min.
- Performed by: Shanghai Analysis and Test Center

Analysis:

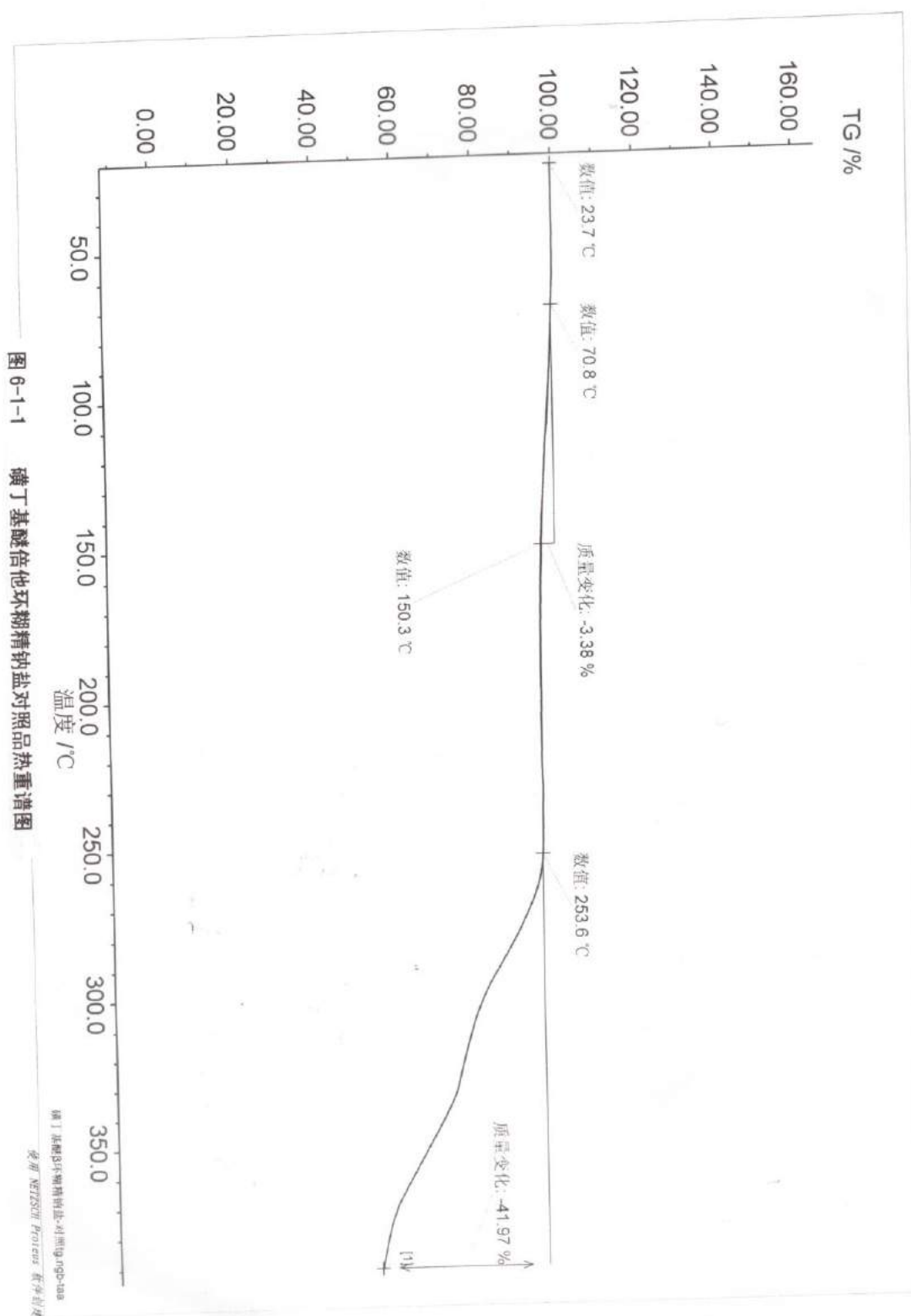
The TG spectrum of the Betadex Sulfobutyl Ether Sodium sample, basically consistent with that of RS, indicates that the sample starts resolving at 251°C.

The DSC spectrum of the Betadex Sulfobutyl Ether Sodium sample, basically consistent with that of RS, indicates the endothermic peaks at 271°C and 355°C are the resolving endothermic peaks.

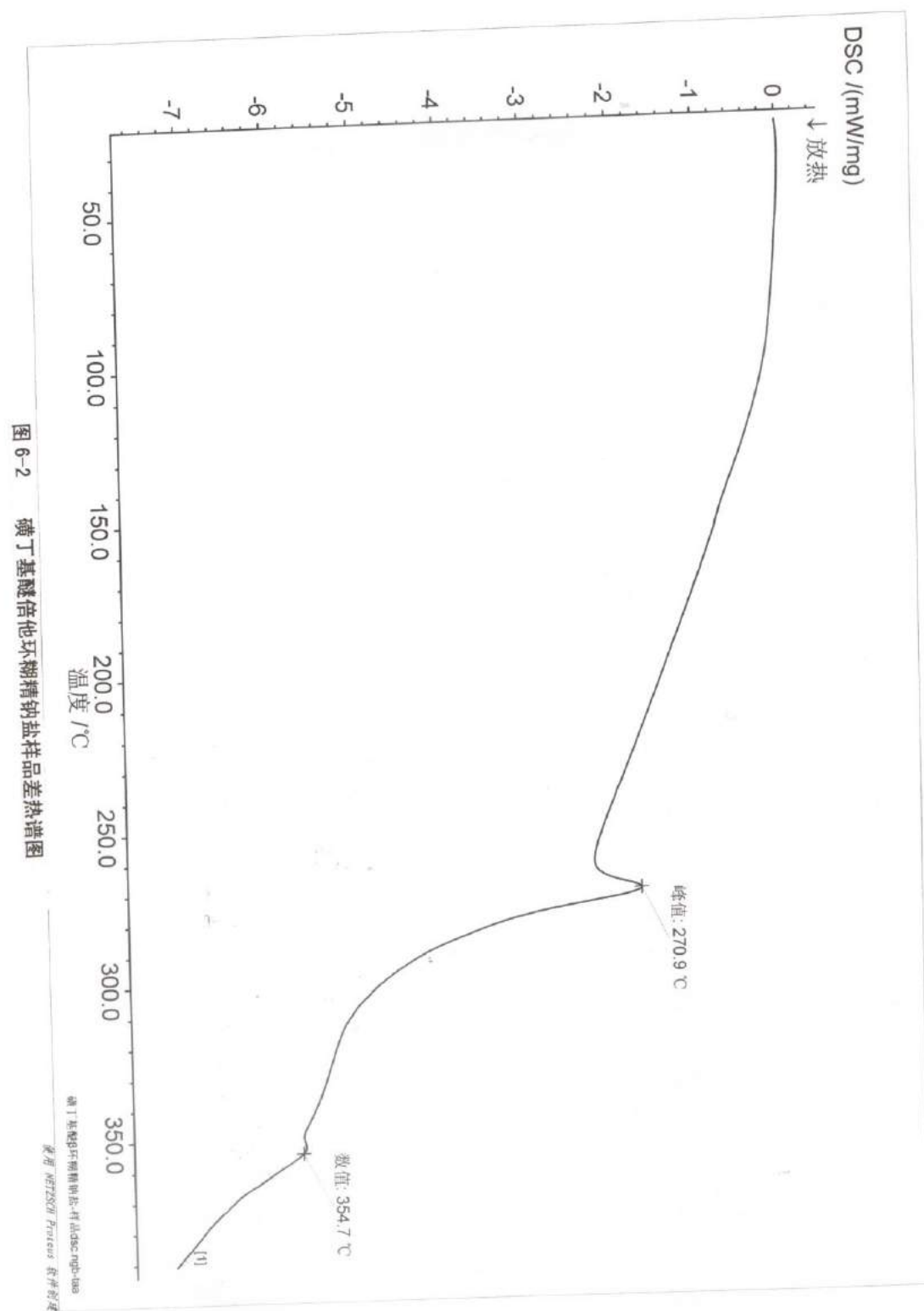
Annex 3-10 Original TG spectrum of Betadex Sulfobutyl Ether Sodium sample



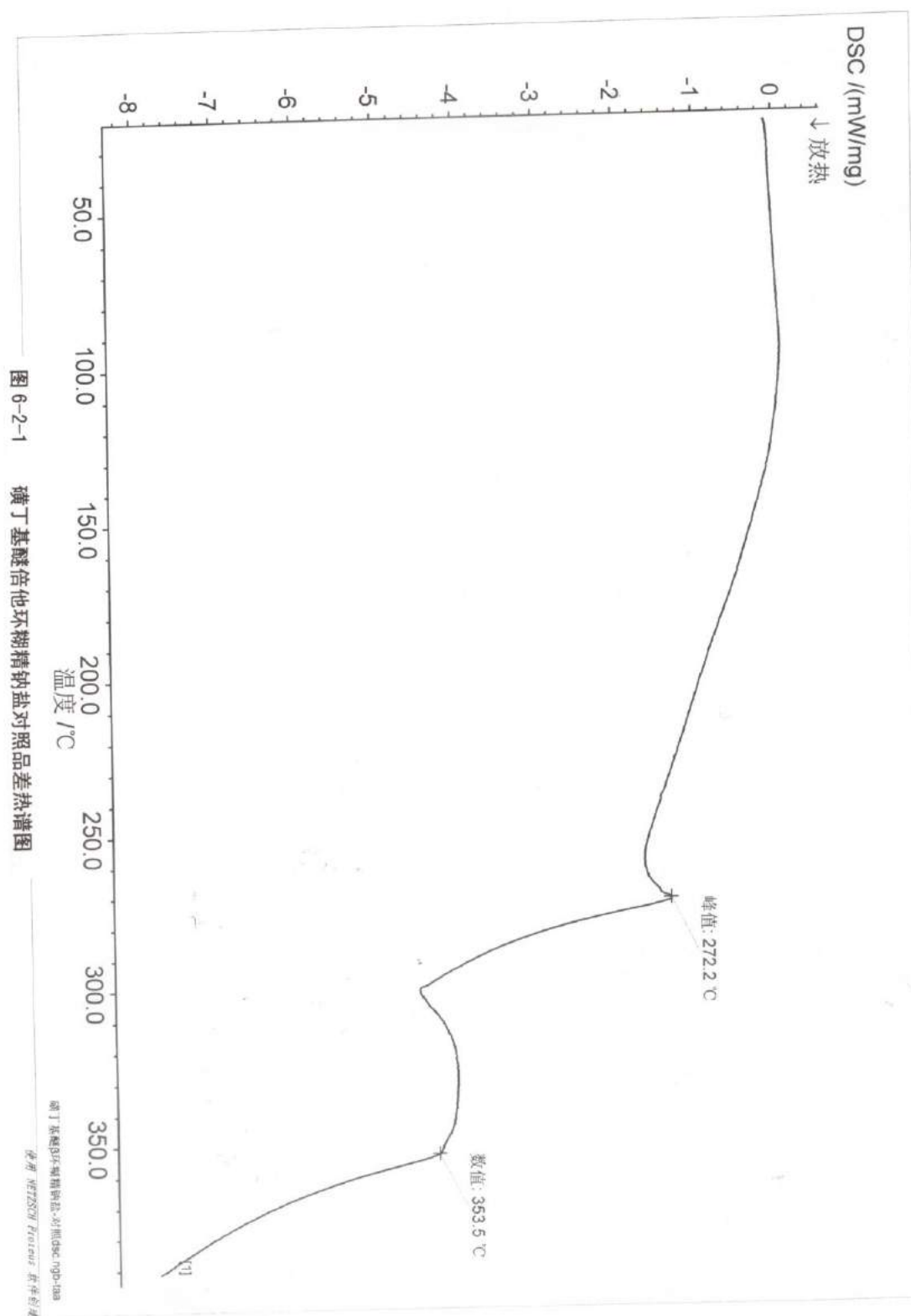
Annex 3-11 Original TG spectrum of Betadex Sulfobutyl Ether Sodium RS



Annex 3-12 Original DSC spectrum of Betadex Sulfobutyl Ether Sodium sample



Annex 3-13 Original DSC spectrum of Betadex Sulfobutyl Ether Sodium RS



6. X-ray diffraction

- Instrument: Holland PANalytical EMPYREAN X-ray diffractometer
- Testing condition: Cu K_{α} ray, 40kV, 40mA
- Performed by: Shanghai Analysis and Test Center

Analysis:

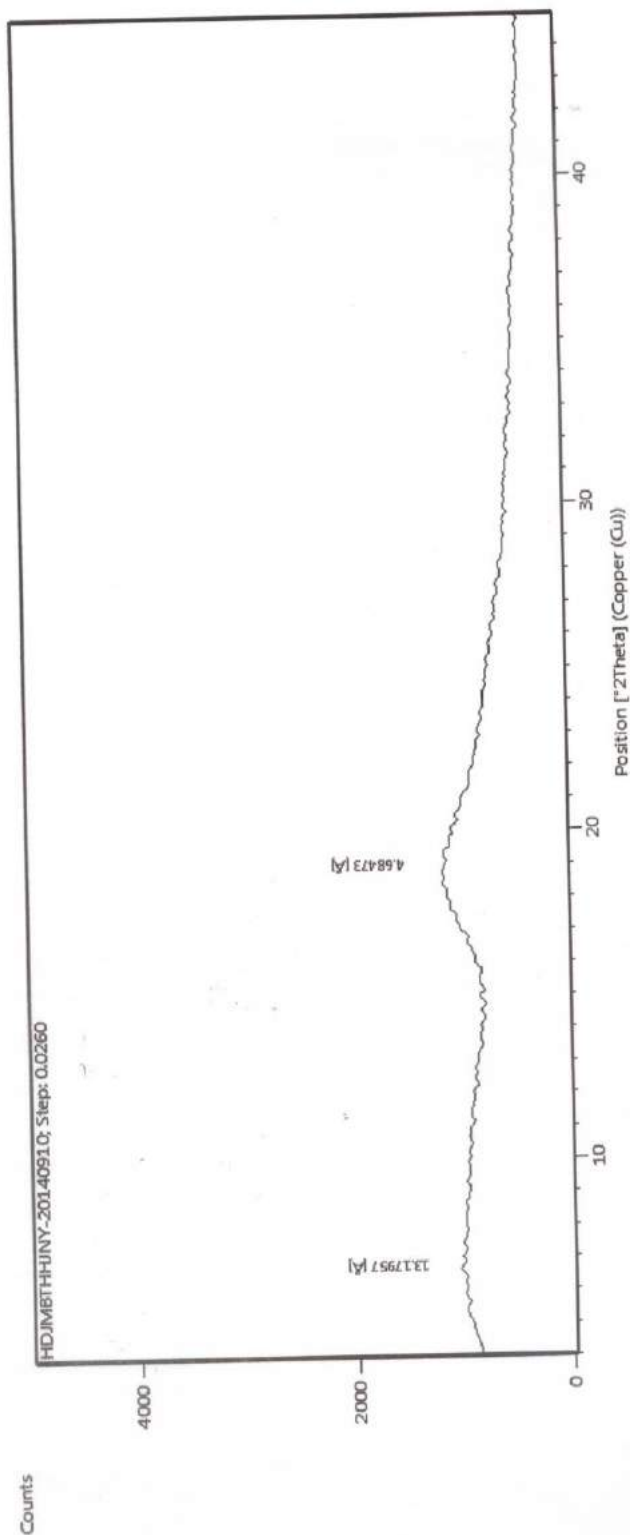
The spectrums obtained show that the sample of Betadex Sulfobutyl Ether Sodium is basically amorphous form.

Annex 3-14 Original X-ray spectrum of Betadex Sulfobutyl Ether Sodium sample 20140910

Shandong Analysis & Test Center

File: HDJMBTHHJNY-20140910

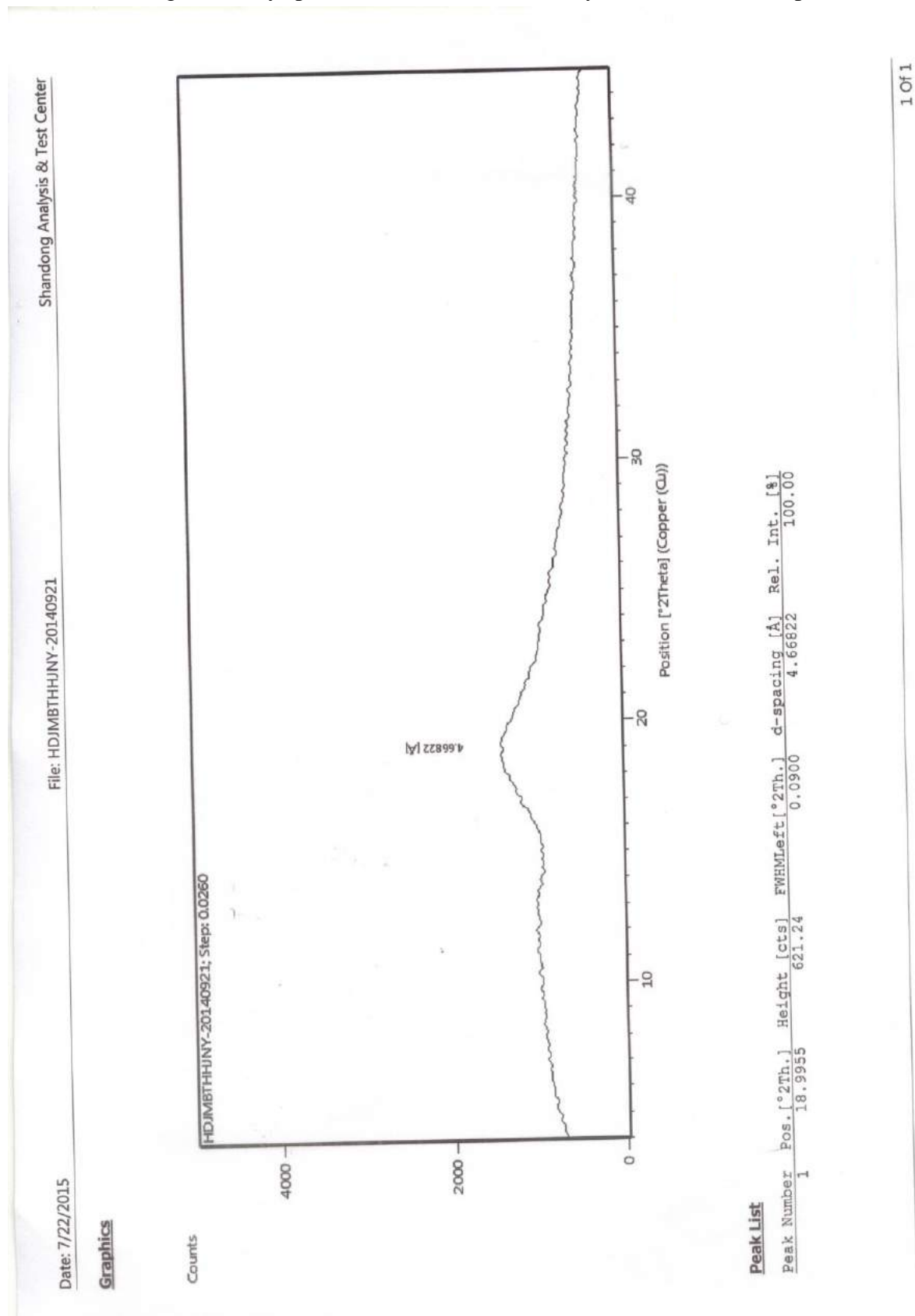
Date: 7/22/2015

Graphics**Peak List**

Peak Number	Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing [Å]	Rel. Int. [%]
1	6.7013	231.46	0.0900	13.17957	51.47
2	18.9280	449.74	0.0900	4.68473	100.00

1 Of 1

Annex 3-15 Original X-ray spectrum of Betadex Sulfobutyl Ether Sodium sample 20140921

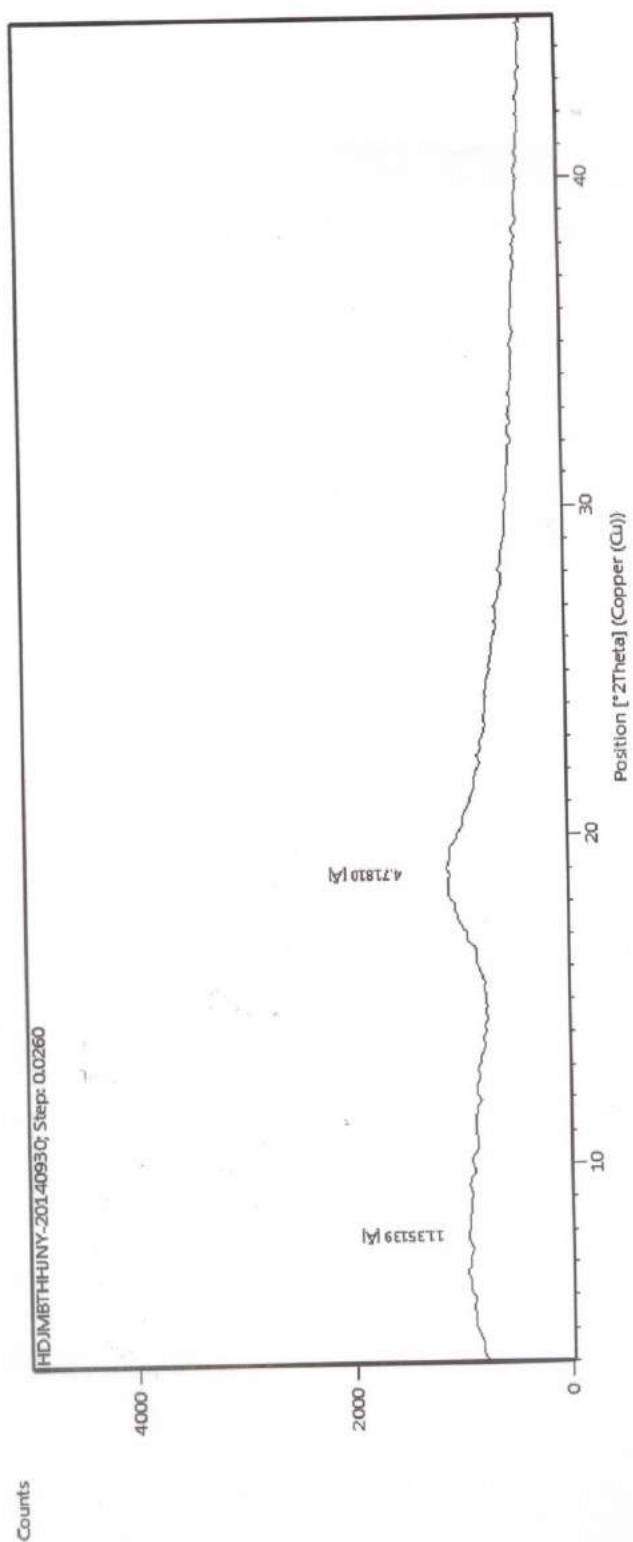


Annex 3-16 Original X-ray spectrum of Betadex Sulfobutyl Ether Sodium sample 20140930

Shandong Analysis & Test Center

File: HDJMBTHHUNY-20140930

Date: 7/22/2015

Graphics**Peak List**

Peak Number	Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing [Å]	Rel. Int. [%]
1	7.7821	175.21	0.0900	11.35139	34.02
2	18.7929	514.99	0.0900	4.71810	100.00

1 Of 1

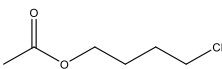

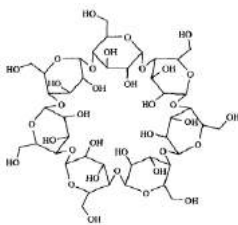
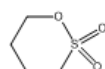
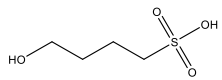
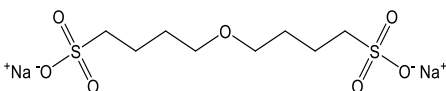
Comprehensive interpretation

1. δ 1.666~1.623 is the signal of proton of methylene on the second and third location of sulfobutyl ether. δ 2.811~2.802 is the signal of proton of methylene that connects sulfo groups in sulfobutyl ether. δ 20.881~20.515 is the signal of carbon on the third location in sulfobutyl ether. δ 28.197~27.434 is the signal of carbons on the second location in sulfobutyl ether. δ 50.702~50.580 is the signal of carbon of methylene that connects sulfo groups.
2. The absorbance at 3430cm^{-1} in IR proves the existence of hydroxyl.
3. Absorbance at 2937cm^{-1} in IR indicates the presence of methyl and methylene.
4. Signals at δ 5.101~4.944 and δ 3.871~3.390 in ^1H -NMR can be the evidence of the existence of ether bone.
5. Signal at δ 5.101~4.944 in ^1H -NMR is the representative chemical shift of proton on carbon that connect two oxygen.
6. Signal at δ 80.973~60.215 in ^{13}C -NMR and δ 3.871~3.390 in ^1H -NMR can indicate that other carbons connect to only one oxygen.

In conclusion, the spectrums and data of the sample and RS for confirmation are basically unanimous, and the results conform to the chemical architectural feature, so the product for confirmation is Betadex Sulfobutyl Ether Sodium.

3.2.S.3.2 Impurities

List of organic impurities

Number	Name	Structural formula	Origin
1	Reducing sugar	/	Impurities in starting material
2	Enzyme	/	
3	Neoprene acetate		
4	Acetylchloride		
5	Sulfonating agent	/	
6	Betadex		Starting material
7	1,4-butane sultone		
8	4-hydroxybutane-1-sulfonic acid		By-product/impurity in start material
9	Bis(4-sulfobutyl) ether disodium		By-product

1. Reducing sugar

In the process of Betadex, un-cyclized carbohydrate, mainly reducing sugar, can be produced. The un-cyclized materials can react with 1,4-butane sultone in the present process. But monose or biose will be significantly less than the final product even though they involved in the synthesis because of their small molecular mass, and can be nanofiltrated. So we control it according to ChP2015 and will not exercise single control on final product.

2. Enzyme

Enzyme is residual impurity from enzymatic manufacture. Enzymatic is famous for efficiency, so the actual inventory is very small and it can be removed by the later process. And the ultrafiltration and nanofiltration in our manufacture process can remove enzyme too, our final product has no adverse effect proved by irritability test with voriconazole. So it is not necessary to control enzyme in the final product.

3. Neoprene acetate

It may exist with an eye to the process of 1,4-butane sultone, but the supplier declares that, according to process and actual inspection, there is no Neoprene acetate in 1,4-butane sultone

4. Acetylchloride

Refer to 3. Neoprene acetate

5. Sulfonating agent

Refer to 3. Neoprene acetate

6. Betadex

It is the starting material of the final product and may be remained, we control it according to USP37 with a limit of 0.1%.

7. 1,4-butane sultone

It is the starting material of the final product and may be remained, we control it according to in-house standard, based on USP37, with a limit of 0.1%.

8. 4-hydroxybutane-1-sulfonic acid

It may exist in the raw material of 1,4-butane sultone, and it is a by-product of Batedex sulfobutyl ether sodium, we control it according to USP37 with a limit of 0.09%.

9. Bis(4-sulfobutyl) ether disodium

It is a by-product and very likely to exist in the final product, what is more it has potential genotoxic, so we control it according to USP37 with a limit of 0.05%.

List of inorganic impurities

Number	Name	Structural formula	Origin
1	Sodium chloride	Na-Cl	By-product
2	Heavy metal	/	From raw materials
3	Active carbon	C	Material

1. Sodium chloride

It is a by-product and controlled according to USP37 with a limit of 0.2%.

2. Heavy metal



Heavy metal comes from raw materials and is controlled according to USP37 with a limit of 5ppm.

3. Active carbon

Active carbon is used before treatment of crude product liquid, and there are ultrafiltration, nanodfiltration, second time ultrafiltraion and spray drying after the use of active carbon. The usage of active carbon is only 1~2% of the weight of final product, so the effect from active carbon is very small, so special control is not necessary.

List of solvents

Number	Name	Structural formula	Origin
--------	------	--------------------	--------

1	Cyclohexane		Impurities from start materials
2	Tetrahydrofuran		
3	Water	H-O-H	Process

1. Cyclohexane

It is a material used in the synthesis of Betadex and should be restricted as a type two solvent, its limit in the specification of Betadex in ChP2015 is 0.388%. The mass rate of Betadex in the process of the final product is 50%, so Cyclohexane in final product is only 0.19%, less than the standard limit, even if it is 0.388% in Betadex. Cyclohexane will not take part in any reaction in the manufacturing process, and the ultrafiltration, nanofiltration and spray drying process can help clearing it, so the assay of Cyclohexane in final product should be lower. So we control it according to ChP2015 and will not exercise single control on final product.

2. Tetrahydrofuran

Tetrahydrofuran is a raw material in the manufacture of 1,4-butane sultone and should be restricted as a type two solvent with a limit of 0.072%. It is inter-miscible with water and hard to produce stable clathrate compound with Betadex. Formula mass of this solvent is very small so it can be removed by nanofiltration. And its boiling point is 65°C, so it can be removed by spray drying too. So the theoretical residue of tetrahydrofuran in the final product should be far lower than 0.072% and special control is not necessary.

3. Water

Water is from the manufacturing process and controlled according to the current USP with a limit of 10.0%.

Inspection result of impurities in three batches

Items	Acceptance criteria	Inspection result		
		20140910	20140921	20140930
Betadex	0.1%	<0.009%	<0.009%	<0.009%
1,4-butane sultone	0.5µg/g	0.3	0.2	0.2
Sodium chloride	0.2%	0.067	0.067	0.067
4-hydroxybutane-1-sulfonic acid	0.09%	0.023	0.024	0.024
Bis(4-sulfobutyl) ether disodium	0.05%	0.031	0.032	0.029
Heavy metal	5ppm	<5ppm	<5ppm	<5ppm
Water	10.0%	4.8%	4.7%	4.1%

3.2.S.4 Control of excipient

3.2.S.4.1 Specification

Items	Acceptance criteria	
Character	White or off-white amorphous or crystalline powder. Very soluble in water, slightly soluble in methanol, practically insoluble in ethanol and dichloromethane.	
Identification	(1) In assay inspection, retention time of main peak in sample solution is corresponding to that in reference solution, (2) IR spectrum of sample solution should be accordance with that of reference standard, (3) Solution of this substance shows positive reaction of Sodium,	
Clarity degree and color of solution	It should be qualified	
Betadex	NMT 0.1 %	
Average degree of substitution	6.2-6.9	
1,4-butane sultone	NMT 0.5µg/g	
Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium	Sodium chloride NMT0.2% ; 4-hydroxybutane-1-sulfonic acid NMT0.09% ; Bis(4-sulfobutyl) ether disodium NMT0.05 %	
pH	4.0~6.8	
Water	NMT 10.0%	
Heavy metal	NMT 5ppm	
Arsenic salt	NMT 0.0002%	
Sulfate	NMT 0.02%	
Bacterial endotoxic	NMT 0.02EU/mg	
Microbial limit	TAMC	≤100cfu/g
	TYMC	≤50cfu/g
	E.coli	Should not be detected
Assay (calculated in anhydrous substance)	95.0%~105.0%	

3.2.S.4.2 Analytical Procedures

1. Character:

White or off-white amorphous or crystalline powder. Very soluble in water, slightly soluble in methanol, practically insoluble in ethanol and dichloromethane.

2. Identifications:

- (1) In assay inspection, retention time of main peak in sample solution is corresponding to that in reference solution,
- (2) IR spectrum of sample solution should be accordance with that of reference standard,
- (3) Solution of this substance shows positive reaction of Sodium.

3. Clarity degree and color of solution

Dissolve 3g of the sample in water to prepare a solution with a concentration of 0.3g/ml, it should be clear and colorless. If it shows any color, it should not be denser than yellow No. 2 colorimetric liquid (ChP2015)

4. Betadex

Chromatographic condition,

Guard column: 4.0mm×5cm anion exchange column packed with L61;

Analysis column: 4.0mm×25cm anion exchange column packed with L61;

Temperature: 50°C;

Mobil phase A: 25mmol/L sodium hydroxide;

Mobil phase B: 250mmol/L sodium hydroxide and 1mol/L potassium nitrate;

Gradient elution chart

Time (min)	A (%)	B (%)
0	100	0
4	100	0
5	0	100
10	0	100
11	100	0
30	100	0

Flow rate: 1.0ml/min;

Detector: electrochemical detector controlled as the chart below,

Time (s)	Voltage (V)
0.00	0.10
0.30	Start integration
0.50	0.10
0.50	Stop integration
0.51	0.60
0.59	0.60
0.60	-0.60
0.65	-0.60

Injection volume: 20μl

Preparation,

Standard solution: 2 μg/mL of USP Betadex RS

Sample solution: 2 mg/mL of Betadex Sulfobutyl Ether Sodium

System suitability

RSD of standard solution: NMT 5.0%

Analysis,

Perform injections respectively with standard solution and sample solution with injection volumes of 20µl, record chromatograms and calculate the content of Betadex as the formula below,

$$\text{Result} = \frac{r_U \times C_S}{r_S \times C_U} \times F \times 100$$

In which,

r_U = peak response for betadex from the sample solution

r_S = peak response for betdex from the standard solution

C_S = concentration of USP Betadex RS in the standard solution (µg/mL)

C_U = concentration of Betadex Sulfobutyl Ether Sodium in the sample solution (mg/mL)

F = conversion factor (10³mg/µg)

Acceptance criteria,

NMT 0.1%

5. Average degree of substitution

Electrophoretic system

Mode: High-performance CE

Column: 50-µm × 50-cm uncoated elastic quartz capillary tube

Column temperature: 25°C

Run buffer solution: 30 mM benzoic acid and adjusted to pH7.5 by addition of 100 mM tris(hydroxymethyl) aminomethane buffer.

Detecting wavelength: 200nm indirect detecting.

Applied voltage: 0.00 to +30.00 kV in 10 min, then at 30 kV for a further 20min.

Injection size: under a pressure of 0.5 psi for 10 s

Capillary rinsing procedure: A new capillary requires rinsing before its first use. Rinse the new capillary with 1 M sodium hydroxide for 1 h, followed by a 2-h water rinse. Perform pre-analysis rinses on a daily basis before each analysis, rinse the capillary with 0.1 N sodium hydroxide for 30 min, with water for NLT 2 h, and with Run buffer solution for NLT 1 h. Perform pre-injection rinses between two different injections as follows. Rinse the capillary with 0.1 N sodium hydroxide for 5 min, and with Run buffer solution for 15 min.

System suitability

Sample: Standard solution

See the table below for the approximate relative migration times for betadex sulfobutyl ether sodium peaks I–X (betadex sulfobutyl ether sodium peaks I, II, III, ..., X, contains betadex molecule with 1, 2, 3, ..., 10 sulfobutyl substituent(s), respectively). The relative migration times are for informational purposes only to aid in peak identification. NLT 0.9, between betadex sulfobutyl ether sodium peak IX and betadex sulfobutyl ether sodium peak X.

Betadex sulfobutyl ether sodium I-X	Relative migration time
I	0.58
II	0.63
III	0.69
IV	0.77
V	0.83
VI	0.91
VII	1.00
VIII	1.10
IX	1.20
X	1.30

Preparation,

Standard solution: 10 mg/mL of USP Betadex Sulfobutyl Ether Sodium RS

Sample solution: 10 mg/mL of Betadex Sulfobutyl Ether Sodium

Analysis,

Perform injections with the solutions obtained and record the electropherograms, calculate the corrected peak area, A_i , for each peak,

$$\text{Corrected Peak Area } A_i = \frac{\text{Peak Area} \times \text{Effective Capillary Length (cm)}}{\text{Migration Time}}$$

Normalize the corrected peak areas by presenting each as a percentage of the total corrected substitution envelope area,

$$\text{Normalized Area, } NA_i = \frac{A_i}{\sum_{i=1}^n A_i} \times 100$$

In which, n=highest level of substitution.

Determine the average degree of substitution as the formula below,

$$\text{Average degree of substitution} = \frac{\sum_{i=1}^n (\text{Level of Substitution for Peak} \times NA_i)}{100}$$

Acceptance criteria:

6.2–6.9 for average degree of substitution.

For each of betadex sulfobutyl ether sodium peaks I–X, see limit range (% peak area) in the chart below,

Betadex Sulfobutyl Ether Sodium Peaks I-X	Limit Range (% Peak Area)
I	0-0.3
II	0-0.9
III	0.5-5.0

IV	2.0-10.0
V	10.0-20.0
VI	15.0-25.0
VII	20.0-30.0
VIII	10.0-25.0
IX	2.0-12.0
X	0-4.0

6. 1,4-butane sultone

Chromatographic condition,

Mold: GC

Column: capillary column with methylpolysilicone as stationary liquid (or similar polarity).

Temperature program

Initial temperature (°C)	Temperature ramp (°C/min)	Final temperature (°C)	Hold time at final temperature (min)
70	0	70	1
70	15	200	8

Detector: Flame ionization, 270°C;

Injector temperature: 200°C;

Carrier gas: nitrogen;

Flow rate: 3ml/min;

Injection size: 1.0µl

Injection type: Splitless injection for 0.5 min, then split at 50 mL/min.

Preparation,

Internal standard solution: 0.25 µg/mL of diethyl sulfone,

Standard stock solution A: 0.5 µg/mL of 1,4-butane sultone,

Standard stock solution B: 1.0 µg/mL of 1,4-butane sultone,

Standard stock solution C: 2.0 µg/mL of 1,4-butane sultone,

Sample stock solution: 250 mg/mL of Betadex Sulfobutyl Ether Sodium in the Internal standard solution.

Prepare solution as the chart below in 10ml glass test tubes with stopper, mix on a vortex mixer each test tube for 30 s, and allow it stand for at least 5 min or until complete separation of the phase, the sublayer as sample solutions.

Name of solutions	Name and volume of solution added, ml	Name and volume of solution added, ml	Methylene Chloride Added, ml
Blank solution	Internal standard solution, 4.0	Water, 1.0	1.0
Sample solution A	Sample stock solution, 4.0	Standard stock solution A, 1.0	1.0

Sample solution B	Sample stock solution, 4.0	Standard stock solution B, 1.0	1.0
Sample solution C	Sample stock solution, 4.0	Standard stock solution C , 1.0	1.0
Sample solution D	Sample stock solution, 4.0	Water, 1.0	1.0

System suitability

Sample: Sample solution B

The relative retention times for diethyl sulfone and 1,4-butane sultone are 0.7 and 1.0, respectively.

Relative standard deviation: NMT 10.0%

Analysis,

Perform injections with blank solution and sample solution A~D respectively with injections volumes of 11µl, record chromatograms.

Correct the ratio of peak responses of the 1,4-butane sultone to diethyl sulfone in Sample solution A, B, C, or D by subtracting the ratio of peak responses of the 1,4-butane sultone to ethyl sulfone in the Blank solution. Plot the corrected ratio of peak response of 1,4-butane sultone to peak response of diethyl sulfone in Sample solution A, B, C or D, versus the added quantity, in µg, of 1,4-butane sultone. Regression equation can be seen below,

$$Y=aX+b..... (A)$$

In which,

Y = the corrected ratio of peak response of 1,4-butane sultone to peak response of diethyl sulfone in Sample solutions;

X = the added quantity, in µg, of 1,4-butane sultone.

Calculate the content of 1,4-butane sultone in the portion of Betadex Sulfobutyl Ether Sodium taken:

$$Result = A / (V_{Ext} \times C_U \times F)$$

In which,

A =the X value in formula A when Y=0,

V_{Ext}=volume of the sample stock solution used in the extraction step, 4.0ml,

C_U=concentration of Betadex Sulfobutyl Ether Sodium in the sample stock solution, mg/ml,

F=conversion factor, 10⁻³g/mg

Acceptance criteria:

NMT 0.5 ppm

7. Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium

Chromatographic condition,

Column: [NOTE—At the end of each run, clean the column using Column wash solution A at a flow rate of 1 mL/min for 35 min then using Column wash solution B at the same flow rate for 35 min.]

Guard: 4.0-mm × 5.0-cm anion-exchange; packing L61

Analytical: 4.0-mm × 25-cm anion-exchange; packing L61

Column temperature: 30°C

Solution A: 5 mM sodium hydroxide, degas in a closed vessel for 15 min

Solution B: 25 mM sodium hydroxide, degas in a closed vessel for 15 min

Gradient elute chart,

Time (min)	Solution A (%)	Solution B (%)
0	100	0
4	100	0
10	70	30
24	70	30
25	100	0
40	100	0

Flow rate: 1.0ml/min,

Detector: Conductivity with a range of 30 µS and a current of 100mA,

System suitability requirements,

Solution: sample solution,

Theoretical plates number of 4-hydroxybutane-1-sulfonic acid NLT3000, retention time of 4-hydroxybutane-1-sulfonic ion, chloridion and Bis(4-sulfobutyl) ether ion are respectively 1.0, 1.4 and 8.6, resolution between chloridion and 4-hydroxybutane-1-sulfonic ion or Bis(4-sulfobutyl) ether ion NLT 2.0. RSD NMT 10.0%

Preparation,

Sample solution: 4 mg/mL of Betadex Sulfobutyl Ether Sodium.

Standard solution: prepare a solution having known concentrations of 8 µg/mL of USP Sodium Chloride RS, 4 µg/mL of 4-hydroxybutane-1-sulfonic acid, and 4 µg/mL of bis(4-sulfobutyl) ether disodium.

Analysis,

Perform injections with the solutions obtained, record chromatograms and calculate the assay of the three substance according to the formula below,

$$\text{Result} = (r_U / r_S) \times (C_S / C_U) \times F \times 100$$

In which,

r_U = peak response for sodium chloride, 4-hydroxybutane-1-sulfonic acid, or bis(sulfobutyl) ether disodium from the Sample solution

r_S = peak response for sodium chloride, 4-hydroxybutane-1-sulfonic acid, or bis(sulfobutyl) ether disodium from the Standard solution

C_S = concentration of sodium chloride, 4-hydroxybutane-1-sulfonic acid, or bis(sulfobutyl) ether disodium in the Standard solution ($\mu\text{g/mL}$)

C_U = concentration of Betadex Sulfobutyl Ether Sodium in the Sample solution (mg/mL)

F = conversion factor ($10^3 \text{ mg}/\mu\text{g}$)

Acceptance criteria

Sodium chloride: NMT 0.2%

4-Hydroxybutane-1-sulfonic acid: NMT 0.09%

Bis(sulfobutyl) ether disodium: NMT 0.05%

8. pH

4.0~6.8, determined on a solution containing 0.3g of the sample per ml, according to general chapter 0631 in ChP2015.

9. Water

Water content should be NMT 10.0% determined according to general chapter 0832 method one 1 in ChP2015.

10. Heavy metal

Transfer 2.0g of the specimen, accurately weighed, to a platinum crucible, moistened by 4ml of sulfuric acid, heated slowly to absolutely charred, added 2ml of nitrate acid and 5 drops of sulfuric acid, heated until no nitric oxide is produced, ignited to absolutely ashed, cooled down. The content of heavy metal should be NMT 5ppm determined according to general chapter 0821 method two in ChP2015.

11. Arsenic salt

Add 10ml of sulfuric acid upon 1.0g of the sample, heat slowly to absolutely charred, dropwise add hydrogen peroxide until reaction completed, namely no foam produced, continue to heat and add 30% hydrogen peroxide solution to colorless, cool down, add 10ml of water, evaporate until dense smoke occurred to remove hydrogen peroxide, add 5ml of hydrochloric acid and defined amount of water to make the total volume 28ml. The content of arsenic should be NMT 0.0002% determined according to general chapter 0822 method one in ChP2015.

12. Sulfate

Determined according to general chapter 0802 in ChP2015 on 0.5g of the sample, the sample solution should not be denser than that prepared of 1.0ml of standard potassium sulfate solution, 0.02%.

13. Bacterial endotoxin

NMT 0.02EU determined according to general chapter 1143 in ChP2015.

14. Microbial limit

Determined according to general chapter 1106 and 1107 in ChP2015, TAMC \leq 100cfu/g, TYMC \leq 50cfu/g, E.coli Should not be detected

15. Assay

Chromatographic condition,

Column: exclusion chromatographic column, 7.8mm \times 30cm,

Temperature: 35°C

Mobile phase: 0.1 M potassium nitrate in a mixture of acetonitrile and water (11:89)

Flow rate: 0.6ml/min

Detector: Refractive index

Detector temperature: 35°C

Injection size: 20 μ l

System suitability requirement,

Number of theoretical plates of the main peak NLT1500, RSD NLT2.0%.

Preparation,

Sample solution: 12 mg/mL of Betadex Sulfobutyl Ether Sodium in Mobile phase

Standard solution: 12 mg/mL of USP Betadex Sulfobutyl Ether Sodium RS (dried at 120°C for 3 hours)in Mobile phase

Analysis,

Perform injection with the solution s obtained, record chromatograms and calculate the percentage of Betadex Sulfobutyl Ether Sodium in the portion of Betadex Sulfobutyl Ether Sodium taken by external standard method.

Acceptance criteria,

95.0%–105.0% on the anhydrous basis

3.2.S.4.3 Validation of Analytical Procedures

Validation of analysis procedure for Betadex**1. Equipments, reagents and solvents**

Equipments	Name	Type	Manufacturer
	HPIC	ICS-5000 ⁺	Dionex
	Balance	BSM220.4	Shanghai zhuojing electronics technology Ltd.
Reagents and solvents	Name	Grade	Manufacturer
	Water	Purified water	Wahaha
	50% sodium hydroxide	/	Beijing Beilingwei science and technology Ltd.
	Potassium nitrate	Primary substance	Tianjin Kemiou chemical reagent Ltd.

2. Analysis procedure

Refer to 3.2.S.4.2 Betadex.

3. Validation items and summary

<i>Item</i>	<i>Validation result</i>
Specificity	(1) The blank has no interference on the detection of Betadex either before or after destroyed; (2) The resolution between Betadex peak and other adjacent peaks are more than 1.5.
System suitability	RSD of Betadex peak areas is 0.2%, less than 2.0%.
LOD and LOQ	LOD of Betadex is 0.17μg /ml equals to 0.009% of the sample; LOQ is 0.52μg /ml equals to 0.026% of the sample, and RSD of peak areas from five injections is 3.8% which is less than 5.0%.
Linearity and range	Linearity formula is $y = 0.3361x + 0.0636$, ($r=0.9998$; $n=6$), with concentration of Betadex in the range of 0.026~0.16% of sample solution.
Accuracy	Mean recovery of Betadex is 101.1% with a RSD of 1.4%.
Repeatability and intermediate precision	RSD of repeatability and intermediate precision is 1.0%.
Solution stability	Reference solution is stable in 23 hours and sample solution is stable in 25 hours.
Robustness	Little changes of flow rate, column temperature and concentration of gradient elution solution A have no influence on the analysis results.

4. Content of validation

4.1 Specificity

Preparation,

Reference solution: dissolve defined amount of the reference standard (dried at 105°C for 2 hours) in water to prepare a solution with concentration of 2µg/ml.

Sample solution: accurately weigh 50mg of the sample to 25ml volumetric flask, add water to dissolve and dilute to the mark, mix well.

Blank: water.

Perform injections with 20µl of each of the solutions above and record the chromatograms. Retention time of Betadex peak is 2.080min, resolution between Betadex and adjacent peak is 15.36, the blank has no interference on the detection of Betadex.

Representative chromatograms refer to Annex 3-1-1~Annex 3-1-3.

4.2 System suitability

Preparation,

System suitability solution: accurately weigh 20.9mg of Betadex (dried at 105°C for 2 hours), transfer it into 100ml volumetric flask, add water to dissolve and dilute to the mark, the system suitability stock solution is obtained. Dilute 1ml of the stock solution to 100ml, the system suitability solution is obtained. Perform six injections with 20µl respectively. The results can be seen below,

No.	1	2	3	4	5	6	Mean	RSD%
Peak area of Betadex	0.7613	0.7601	0.7590	0.7564	0.7581	0.7684	0.7589	0.2

Representative chromatograms refer to Annex 3-1-4~Annex 3-1-5.

4.3 LOD and LOQ

Stepwise dilute the *System suitability solution* and perform injections. It is LOQ when S/N of Betadex peak is 10:1, perform five injections with the LOQ solution and calculate RSD of five peak areas. It is LOD when S/N of Betadex peak is 3:1.

Results of LOD

	LOD(µg/ml)	LOQ(µg/ml)
Betadex	0.17	0.52

Results of LOQ

No.	1	2	3	4	5	Mean	RSD%
Betadex	0.2109	0.2244	0.2260	0.2249	0.2351	0.2243	3.8

The result indicates that the RSD of peak areas obtained from five continuous injections is 3.8% which is less than 5.0%, relevant requirement is met.

Representative chromatograms refer to Annex 3-1-6~Annex 3-1-7.

4.4 Linearity and range

Preparation,

Linearity stock solution: dissolve 2.5ml of System suitability stock solution to 50ml with water, mix well.

Linearity solutions,

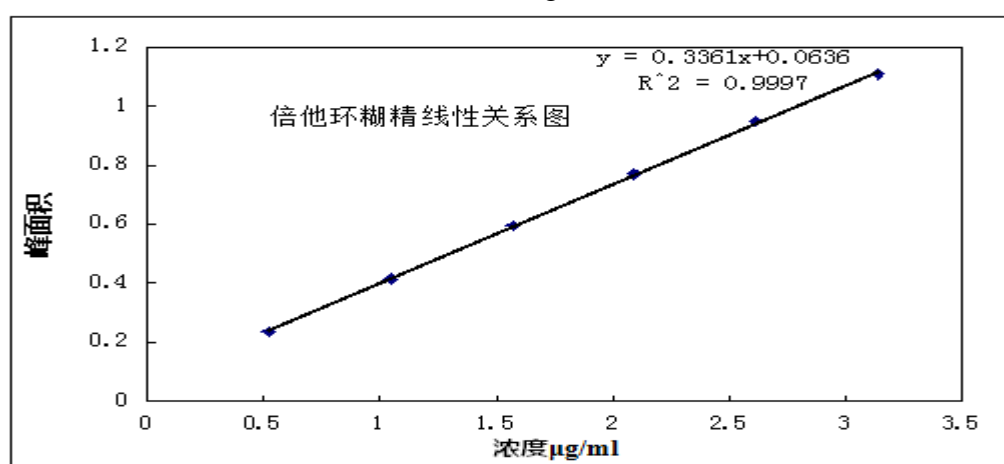
Linearity solution 1: LOQ solution.

Linearity solution 2~5: respectively dissolve 1ml, 1.5ml, 2.0ml, 2.5ml and 3.0ml of the *Linearity stock solution* to 10ml.

Perform injections respectively with the solution obtained with injection volumes of 20 μ l, record chromatograms, and draw diagram with concentrations of Betadex as abscissa and peak areas as ordinate, the results can be seen below,

No.	1	2	3	4	5	6
Concentration of Betadex (mg/ml)	0.522	1.045	1.5675	2.09	2.6125	3.135
Peaks areas	0.2351	0.4136	0.5943	0.7693	0.9489	1.1058
Regression equation	$y = 0.3361x + 0.0636$, $n=6$					
r	0.9998					

Linear diagram



The result indicates that linear equation is $y = 0.3661x + 0.0636$ ($n=6$, $r=0.9998$) when, but not limited, the concentration of Betadex is in the range of 0.522~3.135 μ g/ml, linear relation is fine.

Representative chromatograms refer to Annex 3-1-8~Annex 3-1-10.

4.5 Repeatability and intermediate precision

Preparation,

Reference solution: dissolve defined amount of the reference standard (dried at 105 $^{\circ}$ C for 2 hours) in water to prepare a solution with concentration of 2.10 μ g/ml.

Sample solution for repeability: accurately weigh 20mg of the sample to 10ml volumetric flask, add *Reference solution* to dissolve and dilute to the mark, mix well, prepare five

duplicates.

Sample solution for intermediate precision: accurately weigh 20mg of the sample to 10ml volumetric flask, add *Reference solution* to dissolve and dilute to the mark, mix well, prepare three duplicates.

Perform injections respectively with the solution obtained with injection volumes of 20 μ l, record chromatograms, and calculate the assay of Betadex which can be seen below.

No.	Repeatability	Intermediate precision
1	0.0530	0.0529
2	0.0526	0.0522
3	0.0528	0.0513
4	0.0530	0.0517
5	0.0529	/
6	0.0527	/
Average	0.0525	
RSD%	1.0	

RSD of assay from all injections is 1.0% which indicates that the precision of the method is fine.

Representative chromatograms refer to Annex 3-1-11~Annex 3-1-15.

4.6 Accuracy

The assay of Betadex in our final product is 0.0% which is obtained from sample inspection.

Preparation,

Reference stock solution: dissolve 21.7mg of the reference standard (dried at 105°C for 2 hours) in water and dilute to 100ml.

Reference solution: dissolve 2.5ml of the *Reference stock solution* to 50ml, mix well.

80% sample solution: transfer 20mg of the sample, accurately weighed, to a 10ml volumetric flask, add 1.6ml of *Reference stock solution*, dissolve in water and dilute to the volume, mix well, prepare two duplicates.

100% sample solution: transfer 20mg of the sample, accurately weighed, to a 10ml volumetric flask, add 2.0ml of *Reference stock solution*, dissolve in water and dilute to the volume, mix well, prepare two duplicates.

120% sample solution: transfer 20mg of the sample, accurately weighed, to a 10ml volumetric flask, add 2.4ml of *Reference stock solution*, dissolve in water and dilute to the volume, mix well, prepare two duplicates.

Perform injections respectively with the solution obtained with a injection volume of 20 μ l, record chromatograms, and calculate the recovery.

No.	Level	Peak area	Inventory (μ g)	Weight of sample (mg)	Assay calculated (μ g)	Recovery (%)	Mean recovery (%)	RSD%
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Betadex	1	80%	0.4045	17.36	20.5	17.41	100.3	101.1	1.4
	2		0.4095		21.2	17.36	101.5		
	3		0.4079		21.0	17.56	101.1		
	4	100%	0.5191	21.7	20.6	22.35	102.9		
	5		0.5038		20.1	21.69	99.9		
	6		0.4994		20.4	21.50	99.1		
	7	120%	0.6226	26.04	20.7	26.81	102.9		
	8		0.6181		20.9	26.61	102.2		
	9		0.6058		20.0	26.08	100.1		

The result indicates that the average recovery is 101.1% with a RSD of 1.4%, both are in required ranges, the method is accurate to control Betadex in the final product.

Representative chromatograms refer to Annex 3-1-16~Annex 3-1-19.

4.7 Solution stability

Reference solution: prepared refer to **Repeatability and intermediate precision**.

Perform injections with 20μl of the solution obtained respectively at 0h, 4h, 6h, 8h, 18h, 23h, record chromatograms and calculate the RSD of peak areas from different timing.

Sample solution: accurately weigh 50mg of the sample and transfer it to 25ml volumetric flask, add water to dissolve and dilute to the mark, mix well.

Perform injections with 20μl of the solution obtained respectively at 0h, 2h, 4h, 6h, 8h, 25h, record chromatograms and calculate the RSD of Betadex assays from different timing.

Result of reference solution

Time (h)		0	4	6	8	18	23	RSD (%)
Betadex	Peak area	0.6983	0.6783	0.7036	0.7004	0.6937	0.6937	1.3
	Deviation with that of 0h(%)	/	1.5	0.4	0.1	0.3	0.3	

Result of sample solution

Time (h)		1	2	4	6	8	25	RSD (%)
Betadex	Peak area	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	/
	Deviation with that of 0h(%)	/	/	/	/	/	/	

The result indicates that RSD of peak areas of Betadex after the reference solution has been placed in room temperature for 23 hours is 1.3%; RSD of Betadex assay after the sample solution has been placed in room temperature for 25 hours is 0.0%; both of them are less than 2%, and the two solutions are stable for respectively 23h and 25h.

Representative chromatograms refer to Annex 3-1-20~Annex 3-1-23.

4.8 Robustness

Preparation,

Reference solution: refer to the preparation in Solution stability.

Sample solution: refer to the preparation in Solution stability.

Perform injections with the solutions obtained in different chromatographic conditions as below,

Condition 1: normal condition;

Condition 2: flow rate, 0.9ml/min;

Condition 3: flow rate, 1.1ml/min;

Condition 4: temperature of column, 48°C;

Condition 5: temperature of column, 52°C;

Condition 6: concentration of solution A, 24.5mmol/L sodium hydroxide;

Condition 7: concentration of solution A, 25.5mmol/L sodium hydroxide.

Results from different chromatographic conditions

No.	Chromatographic condition	Betadex (%)
1	Normal condition	Not detected (<0.009%)
2	Flow rate, 0.9ml/min	Not detected (<0.009%)
3	Flow rate, 1.1ml/min	Not detected (<0.009%)
4	Temperature of column, 48°C	Not detected (<0.009%)
5	Temperature of column, 52°C	Not detected (<0.009%)
6	Concentration of solution A, 24.5mmol/L sodium hydroxide	Not detected (<0.009%)
7	Concentration of solution A, 25.5mmol/L sodium hydroxide	Not detected (<0.009%)

Representative chromatograms refer to Annex 3-1-24~Annex 3-1-35.

5. Conclusion of validation

After validation we can see, all the items validated meet relevant requirements, the analysis method has enough specificity and sensitivity to control Betadex in the final product.

Validation of analysis procedure for average degree of substitution**1. Equipments, reagents and solvents**

Equipments	Name	Type	Manufacturer
	Capillary electrophoresis apparatus	P-ACE	Beckman
	Balance	BSM220.4	Shanghai zhuojing electronics technology Ltd.
Reagents and solvents	Name	Grade	Manufacturer
	Water	Purified water	Wahaha
	Benzoic acid	Primary substance	Tianjin Kemiou chemical reagent Ltd.
	Tris(hydroxymethyl)amino methane	Analytically grade	Tianjin Kemiou chemical reagent Ltd.

2. Analysis procedure

Refer to 3.2.S.4.2 Average degree of substitution.

3. Validation items and summary

<i>Item</i>	<i>Validation result</i>
Specificity	(1) The blank has no interference on the detection; (2) The resolution between peak IX and peak X. is 0.9 and the peak shape and base line are fine.
System suitability	The resolution between peak IX and peak X. is 0.9 and the peak shape and base line are fine; migration times of peaks, with peak VII as reference, do not deviate more than ± 0.13 from reference values.
Instrument precision	With peak VII as reference, RSDs of the relative migration times of all the peaks NMT 1.8%, RSD of average degree of substitution is 0.064%.
Concentration range	RSD of average degree of substitution is 0.09% when concentration is in the range of 8~12mg/ml.
Robustness	Little changes of column temperature and pH of running operation buffer solution have no influence on the analysis results.
Repeatability	Repeatability of this method is fine because of RSDs of the relative migration times of all the peaks NMT 2.5%, RSDs of relative percentage of peak areas all NMT 3.9%, RSD of average degree of substitution is 0.20%.
Solution stability	Sample solution is stable in 24 hours because of resolution between peak IX and peak X always more than 0.9; RSD of average degree of substitution is 0.099%.
Identification of peak substitution	The one substitution substance identifies the I place.

4. Content of validation

4.1 Specificity

Reference solution: Prepare reference solution of 10.24mg/ml with Betadex Sulfobutyl Ether Sodium reference standard.

Blank: water

Perform electrophoresis as 3.2.S.4.2 and record electrophoretograms.

Result,

The blank has no interference on the detection. The resolution between peak IX and peak X is 0.9 and the peak shape and base line are fine.

Representative chromatograms refer to Annex 3-2-1~Annex 3-2-2.

4.2 System suitability

Perform six injections with the reference solution in **Specificity** and record electrophoretograms. Results of **System suitability** can be seen below,

Resolution between peak IX and peak X

1	2	3	4	5	6
1.11	1.34	1.18	1.24	1.21	1.38

Migration times of peaks

Name	1	2	3	4	5	6	Reference value	Deviation
Peak I	0.66	0.69	0.67	0.66	0.66	0.66	0.58	0.11
Peak II	0.73	0.73	0.73	0.73	0.73	0.73	0.63	0.1
Peak III	0.79	0.79	0.79	0.79	0.79	0.79	0.69	0.1
Peak IV	0.85	0.85	0.85	0.85	0.86	0.86	0.77	0.09
Peak V	0.90	0.90	0.90	0.90	0.90	0.90	0.83	0.07
Peak VI	0.95	0.95	0.95	0.95	0.95	0.95	0.91	0.04
Peak VII	1.0	1.0	1.0	1.0	1.0	1.0	1.00	0
Peak VIII	1.06	1.06	1.06	1.06	1.06	1.06	1.10	0.04
Peak IX	1.13	1.12	1.12	1.12	1.12	1.12	1.20	0.08
Peak X	1.17	1.18	1.17	1.17	1.17	1.17	1.30	0.13

Conclusion,

The resolution between peak IX and peak X are all more than 0.9, and the peak shape and base line are fine; migration times of peaks, with peak VII as reference, do not deviate more than $\pm 0.13\%$ from reference values.

Representative chromatograms refer to Annex 3-2-3~Annex 3-2-4.

4.3 Instrument precision

Refer to the electrophoretograms, result of Instrument precision can be seen below

Migration times of peaks

Name	1	2	3	4	5	6	RSD%
Peak I	0.66	0.69	0.67	0.66	0.66	0.66	1.8
Peak II	0.73	0.73	0.73	0.73	0.73	0.73	0
Peak III	0.79	0.79	0.79	0.79	0.79	0.79	0
Peak IV	0.85	0.85	0.85	0.85	0.86	0.86	0.6
Peak V	0.90	0.90	0.90	0.90	0.90	0.90	0
Peak VI	0.95	0.95	0.95	0.95	0.95	0.95	0
Peak VII	1.0	1.0	1.0	1.0	1.0	1.0	0
Peak VIII	1.06	1.06	1.06	1.06	1.06	1.06	0
Peak IX	1.13	1.12	1.12	1.12	1.12	1.12	0.36
Peak X	1.17	1.18	1.17	1.17	1.17	1.17	0.35

Average degree of substitution

Name	1	2	3	4	5	6	RSD%
Average degree of substitution	6.42	6.42	6.42	6.42	6.41	6.42	0.064%

Conclusion,

With peak VII as reference, RSDs of the relative migration times of all the peaks NMT 1.8%, RSD of average degree of substitution is 0.064%. instrument precision is fine.

4.4 Concentration range

Prepare solutions that contain 8.14mg, 10.35mg and 12.16mg per ml of Betadex Sulfobutyl Ether Sodium with the final product, and perform injections with the solutions obtained, record electrophoretograms and calculate degree of substitution,

Name	8.14mg/ml	10.35mg/ml	12.16mg/ml	RSD%
Average degree of substitution	6.41	6.41	6.42	0.09%

Conclusion,

RSD of the three substitutions is 0.09%, so changes in the range of 8.14~12.16mg/ml will have no effect on the analysis.

Representative chromatograms refer to Annex 3-2-5~Annex 3-2-6.

4.5 Robustness

Prepare solution as required, perform injections at the conditions below,

1. Normal condition;
2. Column temperature 23°C;
3. Column temperature 27°C;
4. pH of buffer solution 7.7;
5. pH of buffer solution 7.3.

Result of robustness

No.	Electrophoresis conditions	Average degree of substitution
1	Normal condition	6.42

2	Column temperature 23°C	6.42
3	Column temperature 27°C	6.40
4	pH of buffer solution 7.7	6.42
5	pH of buffer solution 7.3	6.41
Average		6.41
RSD(%)		0.14%

Conclusion,

RSD% is 0.14%, so slight changes on the two conditions above have no significant effect on the inspection result.

Representative chromatograms refer to Annex 3-2-7~Annex 3-2-11.

4.6 Repeatability

Prepare five solutions that contain about 10mg per ml of Betadex Sulfobutyl Ether Sodium with the final product, perform injections and record electrophoretograms, result of this item can be seen below,

Migration times of peaks

Name	1	2	3	4	5	RSD%
Peak I	0.64	0.66	0.67	0.68	0.68	2.5
Peak II	0.72	0.72	0.73	0.73	0.73	0.75
Peak III	0.78	0.79	0.79	0.79	0.80	0.90
Peak IV	0.85	0.85	0.85	0.86	0.86	0.52
Peak V	0.90	0.90	0.90	0.90	0.90	0
Peak VI	0.95	0.95	0.95	0.95	0.95	0
Peak VII	1.0	1.0	1.0	1.0	1.0	0
Peak VIII	1.06	1.06	1.06	1.06	1.06	0
Peak IX	1.13	1.13	1.12	1.12	1.12	0.40
Peak X	1.18	1.17	1.18	1.17	1.17	0.47

Average degree of substitution

Name	1	2	3	4	5	RSD%
Average degree of substitution	6.40	6.42	6.39	6.39	6.39	0.20%

Conclusion,

The results indicate that the RSDs of all relative Migration times of peaks are NMT2.5%, RSD of average degree of substitution is 0.20%, repeatability of this method is fine.

Representative chromatograms refer to Annex 3-2-12~Annex 3-2-13.

4.7 Solution stability

Preparation,

Transfer about 250mg of the sample into 25ml volumetric flask, add water to dissolve and dilute to the mark, perform injections to the electrophoresis respectively at 0, 2, 4, 6, 8, 10, 12

and 24 hour after the preparation and record electrophoregrams.

Resolution between peak IX and peak X

Name	0	2	4	6	8	10	12	24
Resolution	1.21	1.30	1.34	1.19	1.20	1.33	1.12	1.44

Average degree of substitution

Name	0	2	4	6	8	10	12	24	RSD%
Average degree of substitution	6.46	6.47	6.46	6.47	6.47	6.47	6.48	6.47	0.099%

Conclusion,

Resolution between peak IX and peak X always more than 0.9; RSD of average degree of substitution is 0.099%. The sample solution is stable for 24 hours.

Representative chromatograms refer to Annex 3-2-14~Annex 3-2-16.

4.8 Identification of peak substitution

Preparation,

Prepare sample solutions with the substances below,

Home made one substitution Betadex Sulfobutyl Ether Sodium, Betadex, 4-hydroxybutane-1-sulfonic acid, Betadex and 4-hydroxybutane-1-sulfonic acid added one substitution Betadex Sulfobutyl Ether Sodium, batch 20140910 Betadex Sulfobutyl Ether Sodium, one substitution Betadex Sulfobutyl Ether Sodium added 20140910 sample.

Perform injections with the solutions obtained and results can be seen below,

Name		1	2	3	4	5	6
One substitution	Retention time	7.667	/	/	7.70	7.579	7.704
	Peak area	6626	/	/	7192	225	6517
Betadex	Retention time	6.971	6.971	/	7.013	/	7.008
	Peak area	17930	21542	/	45240	/	19117
4-hydroxybutane-1-sulfonic acid	Retention time	9.750	/	9.738	9.917	/	/
	Peak area	1381	/	3389	66782	/	/

We can see that there are three peaks in injection 1, and two peaks of the above are obvious bigger in injection 4, so two impurities can be excluded; in injection 6, peak of one substitution Betadex Sulfobutyl Ether Sodium is obvious bigger than that in injections 5, so the place of one substitution Betadex Sulfobutyl Ether Sodium can be identified.

Representative chromatograms refer to Annex 3-2-17~Annex 3-2-22.

5. Conclusion of validation

Via validation, all the items meet relevant requirement, so this method is suitable for the

control of average degree of substitution.

Validation of analysis procedure for 1,4-butane sultone**1. Equipments, reagents and solvents**

Equipments	Name	Type	Manufacturer
	GC	7890B	Agilent
	Balance	XS205	Mettler
Reagents and solvents	Name	Grade	Manufacturer
	Dichloromethane	GC	Aladdin
	Dichloromethane	GC	ACS
	Ultrapure water	HPLC	Milli-Q

2. Analysis procedure

Refer to 3.2.S.4.2 1,4-butane sultone.

3. Validation items and summary

<i>Item</i>	<i>Validation result</i>
Specificity	The blank has no interference on the peaks of diethyl sulfone or 1,4-butane sultone.
System suitability	The RSD of peak area ratio of 1,4-butane sultoneform and diethyl from six injections is 6.7%.
LOD and LOQ	LOD of 1,4-butane sultone is 0.10.1µg/g while LOQ is 0.3µg/g
Linearity and range	Linear equation is $Y=1.13X-0.039$ ($r=0.9999$; $n=8$) while concentration is 0.1~3.0µg/ml which equals to 20%~600% of the limit concentration.
Precision	RSD of repeatability is 2.2% ($n=6$), RSD of repeatability and intermediate precision is 2.7% ($n=9$).
Accuracy	Average recovery is 101.1% and RSD is 4.1%.
Solution stability	Sample solution is stable in 21 hours at room temperature.
Robustness	Resolution between main peak and adjacent peak can meet requirement and RSD of assays of 1,4-butane sultone is 12.1% when chromatographic conditions changed slightly.

4. Content of validation**4.1 Specificity**

Prepare sample solutions with limit concentrations of diethyl sulfone or 1,4-butane sultone, perform injections as required, the blank has no interference on the peaks of diethyl sulfone or 1,4-butane sultone.

Representative chromatograms refer to Annex 3-3-1~Annex 3-3-2.

4.2 System suitability

Prepare reference solution with limit concentration of 1,4-butane sultone and perform six injections as required, retention time of diethyl sulfone and 1,4-butane sultone are

respectively 0.7, 1.0min, calculate RSD of peak area ratio of 1,4-butane sultone and diethyl sulfone, result is as below,

No.	1	2	3	4	5	6	Average	RSD%
Ratio	0.64	0.63	0.62	0.71	0.70	0.72	0.70	6.7

Representative chromatograms refer to Annex 3-3-3~Annex 3-3-4.

4.3 LOD and LOQ

Dilute the reference solution in System suitability until S/N is 10:1, that is the LOQ, perform six injections with LOQ solution and calculate peak area ratio of 1,4-butane sultone and diethyl sulfone; continue diluting the reference solution until S/N is 3:1, that is the LOD solution. Results can be seen below,

	LOD($\mu\text{g/g}$)	LOQ($\mu\text{g/g}$)
1,4-butane sultone	0.1	0.3

Result of six LOQ solution

No.	1	2	3	4	5	6	Average	RSD%
Ratio	0.36	0.37	0.40	0.41	0.43	0.50	0.4	12.1

Representative chromatograms refer to Annex 3-3-5~Annex 3-3-7.

4.4 Linearity and range

Internal standard solution: 0.25 $\mu\text{g/mL}$ of diethyl sulfone,

Reference solution: weigh defined amount of 1,4-butane sultone reference standard and prepare reference solutions with concentrations of 3.0 $\mu\text{g/mL}$, 2.0 $\mu\text{g/mL}$, 1.5 $\mu\text{g/mL}$, 1.0 $\mu\text{g/mL}$, 0.8 $\mu\text{g/mL}$, 0.5 $\mu\text{g/mL}$, 0.3 $\mu\text{g/mL}$ and 0.1 $\mu\text{g/mL}$,

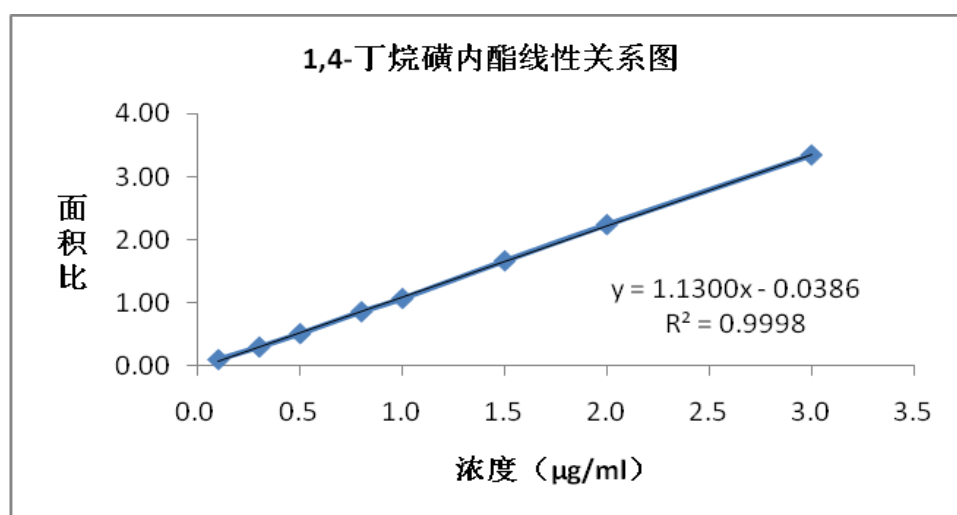
Blank solution: transfer 4.0ml of internal standard solution into a 10ml glass test tube with stopper, add 1.0ml of water and 1.0 ml of dichloromethane,

Linearity solutions: transfer 4.0ml of internal standard solution into each 10ml glass test tubes with stopper, add 1.0ml of relevant reference solution and 1.0 ml of dichloromethane,

Preparation of samples: mix blank solution and linearity solutions on a vortex mixer each test tube for 30 s, and allow them stand for at least 5 min or until complete separation of the phase, the sublayer as sample solutions. Perform injections with solutions obtained and record chromatograms. Respectively calculate peak area ratios of 1,4-butane sultone and diethyl sulfone. Draw regression curve with concentrations as x axis and peak area ratios of 1,4-butane sultone and diethyl sulfone, taken that ratio in blank solution out, as y axis.

No.	1	2	3	4	5	6	7	8
Concentration ($\mu\text{g/mL}$)	0.1	0.3	0.5	0.8	1.0	1.5	2.0	3.0
Peak area ratio	0.10	0.30	0.51	0.86	1.07	1.66	2.24	3.34
Regression equation	$Y=1.13X-0.039, n=8$							
r	0.9999							

Regression curve



Conclusion,

Linear equation is $Y=1.13X-0.039$ ($r=0.9999$; $n=8$) while concentration is $0.1\sim 3.0\mu\text{g/ml}$ which equals to 20%~600% of the limit concentration.

Representative chromatograms refer to Annex 3-3-8~Annex 3-3-9.

4.5 Repeatability

Prepare blank solution, sample solution A, B, C and six sample solution D, perform injections with the solutions above and calculate assays of 1,4-butanedisulfone in sample as required, result can be seen below,

	1	2	3	4	5	6	Average	RSD%
1,4-butanedisulfone($\mu\text{g/g}$)	0.29	0.29	0.28	0.27	0.29	0.29	0.3	2.2

Conclusion,

From the RSD we can see that the repeatability of this method is fine.

Representative chromatograms refer to Annex 3-3-10~Annex 3-3-12.

4.6 Accuracy

Preparation,

Prepare blank solution and sample solution A, B, C as repeatability, and recovery solutions as below,

Name of solutions	Name and volume of solution added, ml	Name and volume of solution added, ml	Methylene Chloride Added, ml
Recovery solution-LOQ concentration	Sample stock solution, 4.0	$0.3\mu\text{g/ml}$ linearity solution, 1.0	1.0
Recovery solution-100% concentration	Sample stock solution, 4.0	$0.5\mu\text{g/ml}$ linearity solution, 1.0	1.0
Recovery solution-200%	Sample stock solution, 4.0	$1.0\mu\text{g/ml}$ linearity solution, 1.0	1.0

concentration			
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Perform injections with the solutions above, calculate the assay of 1,4-butane sultone through linearity regression equation in repeatability and recoveries in different concentrations.

No.	Percentage that equals to limit concentration	Inventory (µg/g)	Quantity detected (µg/g)	Recovery (%)	Mean recovery (%)	RSD (%)
1	20%, LOQ	0.30	0.29	97.4	101.1	4.1
2		0.30	0.29	96.8		
3		0.30	0.30	101.1		
4	100%	0.50	0.50	99.9		
5		0.50	0.53	106.8		
6		0.50	0.48	95.5		
7	200%	1.00	1.02	102.1		
8		1.00	1.06	106.5		
9		1.00	1.04	103.9		

Conclusion,

Recovery values, mean recovery and RSD all meet requirement, accuracy of this method is fine.

Representative chromatograms refer to Annex 3-3-13~Annex 3-3-15.

4.7 Intermediate precision

Proceeded by another analyst on another day as repeatability, prepare blank solution, sample solution A, B, C and three sample solution D, perform injections and calculate assay of 1,4-butane sultone.

No.	Result of repeatability	Result of intermediate precision
	Assay (µg/g)	Assay (µg/g)
1	0.29	0.28
2	0.29	0.30
3	0.28	0.29
4	0.27	/
5	0.29	/
6	0.29	/
Mean value(µg/g)	0.3	
RSD%	2.7	

Conclusion,

Assays of 1,4- butane sultone and RSD of them all meet relevant requirement, precision of this method is fine.

Representative chromatograms refer to Annex 3-3-16~Annex 3-3-19.

4.8 Solution stability

Prepare blank solution and sample solution A, B, C, D as repeatability, store them at room temperature and perform injections with the solutions obtained at 0h, 2h, 4h, 12h, 16h and 21h after prepared, record chromatograms and calculate the deviations of assay 1,4- butane sultone with 0h.

Result of solution stability

Time (h)	0	2	4	12	16	21
1,4- butane sultone (µg/g)	0.31	0.29	0.28	0.28	0.30	0.30
Deviation with 0h (%)	/	3.3	4.0	4.5	0.5	0.7

Conclusion,

The biggest deviations of assay 1,4- butane sultone with 0h is 4.5% which can be accepted, so the solutions is stable in 23hours.

Representative chromatograms refer to Annex 3-3-20~Annex 3-3-25.

4.9 Robustness

Prepare solutions as repeatability and perform injections at chromatographic conditions below,

Condition 1, normal condition;

Condition 2, flow rate 2.8ml/min;

Condition 3, flow rate 3.2ml/min;

Condition 4, heating rate 13°C/min;

Condition 5, heating rate 17°C/min.

Prepare solutions as below and perform injections at normal condition,

Prepare condition 1, mixed on a vortex mixer for 25 s, and allow it stand for 4 min;

Prepare condition 2, mixed on a vortex mixer for 35 s, and allow it stand for 6 min.

Result of robustness

No.	Prepare and chromatographic conditions	Assay of 1,4- butane sultone (µg/g)
1	Normal condition	0.28
2	Mixed on a vortex mixer for 25 s, and allow it stand for 4 min	0.31
3	Mixed on a vortex mixer for 35 s, and allow it stand for 6 min	0.27
4	Flow rate 2.8ml/min	0.30
5	Flow rate 3.2ml/min	0.36
6	Heating rate 13°C/min	0.29
7	Heating rate 17°C/min	0.24
Mean assay (µg/g)		0.3

RSD (%)	12.1
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Representative chromatograms refer to Annex 3-3-26~Annex 3-3-35.

5. Validation conclusion

Via validation, all the items meet relevant requirement, so this method is suitable for the control of 1,4- butane sultone.

Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium

1. Equipments, reagents and solvents

Equipments	Name	Type	Manufacturer
	HPIC	ICS-5000 ⁺	Dionex
	Balance	BSM220.4	Shanghai zhuojing electronics technology Co.,Ltd.
Reagents and solvents	Name	Grade	Manufacturer
	Water	Purified water	Wahaha
	Sodium hydroxide	50wt.% solution	Beijing Bailingwei science and technology Co.,Ltd.
	Sodium citrate	Chromatographically pure	Tianjin Kemiou chemical reagent Co.,Ltd.

2. Analysis procedure

Refer to 3.2.S.4.2 Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium.

3. Validation items and summary

<i>Item</i>	<i>Validation result</i>
Specificity	The blank has no interference on the inspection, .resolution between object and adjacent peaks are more than 2.0.
System suitability	Mean peak area and RSD% of Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium are respectively 1.665, 0.15%; 0.0833, 1.15%; 0.179, 0.25%.
LOD	LOD of Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium are respectively 0.016µg/ml, 0.017µg/ml and 0.02µg/ml; respectively equal to 0.0004%, 0.0005% and 0.0005% of the principal component concentration.
LOQ	LOQ of Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium are respectively 0.032µg/ml, 0.035µg/ml and 0.04µg/ml; respectively equal to 0.0008%, 0.001% and 0.001% of the principal component concentration.
Linearity and range	<p>① 4-hydroxybutane-1-sulfonic acid, linear equation is $Y=0.0313X+0.0086$($r=0.9998$) while concentration is 0.035~5.30µg/ml which equals to 0.001%~0.13% of the principal component concentration;</p> <p>② Sodium chloride, linear equation is $Y=0.1832X-0.0217$ ($r=0.9997$) while concentration is 0.032~12.21µg/ml which equals to 0.001%~0.31% of the principal component concentration;</p> <p>③ Bis(4-sulfobutyl) ether disodium, linear equation is $Y=0.0443X+0.0002$ ($r=0.9998$) while concentration is 0.04~6.21µg/ml which equals to 0.001%~0.16% of the principal component concentration;</p>

Accuracy	Average recovery of 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium are respectively 99.47%, 98.60% and 98.64%; while relevant RSD% are respectively 1.4%, 1.8% and 1.4%.
Intermediate precision	RSD of intermediate precision for 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium are respectively 1.8%, 1.8% and 1.5%.
Solution stability	Sample solution and reference solution are stable in 26 hours at room temperature.
Robustness	Resolution between main peak and adjacent peak can meet requirement when chromatographic conditions changed slightly.

4. Content of validation

4.1 Specificity

Preparation,

Blank solution: water;

Positioning solution: dissolve defined amount of the three reference standards respectively in water;

Reference solution: dissolve defined amount of the reference standard to prepare a solution with 3.74µg/ml of 4-hydroxybutane-1-sulfonic acid, 8.28µg/ml of Sodium chloride and 4.18µg/ml of Bis(4-sulfobutyl) ether disodium.

Sample solution: dissolve 102.1mg of the specimen in water and dilute to 25ml.

Analyses,

Perform injections with the solutions obtained with injection sizes of 20µl, record chromatograms.

Conclusion,

Retention times of 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium are respectively 2.360min, 3.360min, 18.637min. in the chromatogram obtained from sample solution resolution between the three peaks are 42.32 and 6.15, all meet requirement.

Representative chromatograms refer to Annex 3-4-1~Annex 3-4-6.

4.2 System suitability

Reference solution: with 3.98µg/ml of 4-hydroxybutane-1-sulfonic acid, 8.02µg/ml of Sodium chloride and 4.00µg/ml of Bis(4-sulfobutyl) ether disodium.

Perform six injections with the solution obtained with injection sizes of 20µl, result of this item can be seen below,

No.	1	2	3	4	5	6	Mean	RSD%
4-hydroxybutane-1-sulfonic acid	0.084	0.084	0.083	0.083	0.083	0.083	0.0833	1.15
Sodium chloride	1.663	1.664	1.665	1.665	1.662	1.669	1.665	0.15

Bis(4-sulfobutyl) ether disodium	0.179	0.179	0.180	0.179	0.179	0.179	0.179	0.25
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Conclusion,

Theoretical plates number and resolutions all meet requirement referring to the chromatograms, RSD of the three peaks from six injections are respectively 1.15%, 0.15% and 0.25%. So the system suitability of this method is fine.

Representative chromatograms refer to Annex 3-4-7~Annex 3-4-8.

4.3 LOD and LOQ

Dilute the positioning solutions of 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium respectively until S/Ns are 10:1, those are the LOQs of the three substances, respectively perform five injections with the LOQ solutions and calculate each RSD; continue diluting the positioning solutions until S/Ns are 3:1, those are LOD of the three substances.

Result of LOD and LOQ for the three substance

	LOQ (μg/ml)	LOD (μg/ml)
4-hydroxybutane-1-sulfonic acid	0.035	0.017
Sodium chloride	0.032	0.016
Bis(4-sulfobutyl) ether disodium	0.040	0.020

Result of LOQ

No.	1	2	3	4	5	Mean	RSD%
4-hydroxybutane-1-sulfonic acid	0.0011	0.0011	0.0011	0.0011	0.0012	0.011	4.5
Sodium chloride	0.0082	0.0084	0.0082	0.0083	0.0082	0.0082	1.2
Bis(4-sulfobutyl) ether disodium	0.0044	0.0043	0.0041	0.0042	0.0045	0.0043	3.2

Conclusion,

LOD of Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium are respectively 0.016μg/ml, 0.017μg/ml and 0.02μg/ml; respectively equal to 0.0004%, 0.0005% and 0.0005% of the principal component concentration. All of the LODs are low enough.

LOQ of Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium are respectively 0.032μg/ml, 0.035μg/ml and 0.04μg/ml; respectively equal to 0.0008%, 0.001% and 0.001% of the principal component concentration, and the RSDs for each style of peak are 1.2%, 4.5% and 3.2%.

Representative chromatograms refer to Annex 3-4-9~Annex 3-4-14.

4.4 Linearity and range

Preparation,

Linearity stock solution: respectively with 20.2μg/ml of 4-hydroxybutane-1-sulfonic acid, 40.7μg/ml of Sodium chloride and 20.7μg/ml of Bis(4-sulfobutyl) ether disodium.

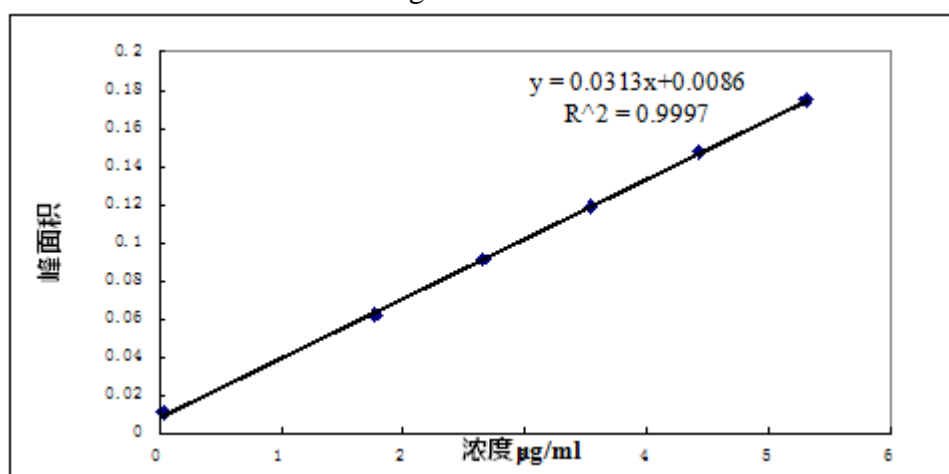
Linearity solutions: transfer 1ml, 1.5ml, 2ml, 2.5ml and 3ml of the stock solution into separate 10ml of volumetric flasks and add water to mark.

Analyses,

Perform injections with the solutions obtained, draw curve with concentrations as axis and relevant peak areas as y axis.

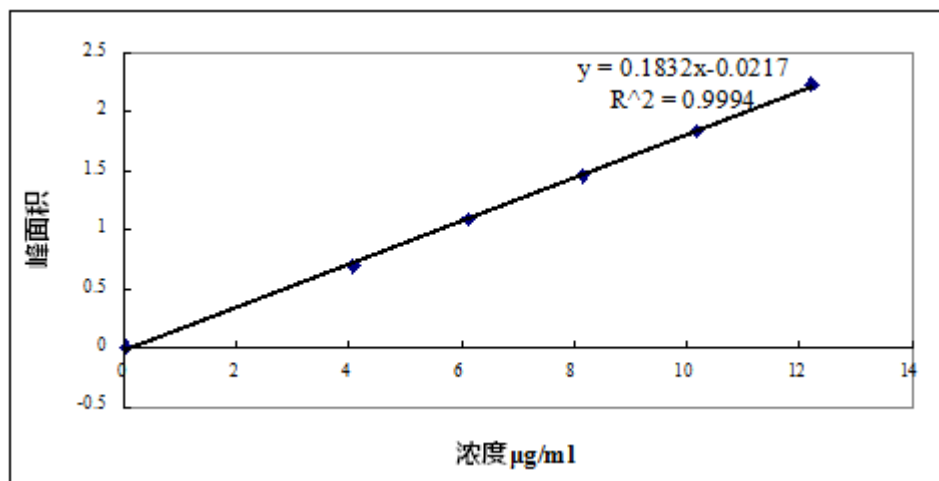
Regression equation for 4-hydroxybutane-1-sulfonic acid is $Y=0.0313X+0.0086$ ($r=0.9998$) at the range of 0.035~5.30 $\mu\text{g/ml}$.

Regression curve



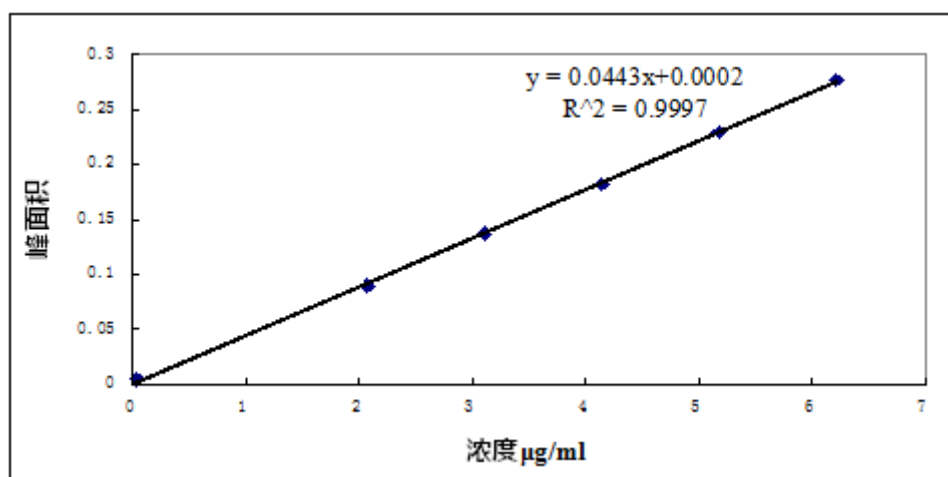
Regression equation for Sodium chloride is $Y=0.1832X-0.0217$ ($r=0.9997$) at the range of 0.032~12.21 $\mu\text{g/ml}$.

Regression curve



Regression equation for Bis(4-sulfobutyl) ether disodium is $Y=0.0443X+0.0002$ ($r=0.9998$) at the range of 0.04~6.21 $\mu\text{g/ml}$.

Regression curve



Conclusion,

All the three r values are more than 0.999, linearity of the three substances are fine.

Representative chromatograms refer to Annex 3-4-15~Annex 3-4-17.

4.5 Accuracy

From the batch analyses we can see that the assay of 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium are respectively 0.023%, 0.067% and 0.031%.

Preparation,

Mix reference stock solution: respectively with 20.8 $\mu\text{g/ml}$ of 4-hydroxybutane-1-sulfonic acid, 40.3 $\mu\text{g/ml}$ of Sodium chloride and 20.4 $\mu\text{g/ml}$ of Bis(4-sulfobutyl) ether disodium.

Reference solution: dilute the mix reference stock solution to limit concentration.

80% recovery solution: transfer 0.1g of sample 20140910 into 25ml volumetric flask, add 2.5 ml of the mix reference stock solution, add water to the mark and mix well, prepare two duplicates.

100% recovery solution: transfer 0.1g of sample 20140910 into 25ml volumetric flask, add 3.5 ml of the mix reference stock solution, add water to the mark and mix well, prepare two duplicates.

120% recovery solution: transfer 0.1g of sample 20140910 into 25ml volumetric flask, add 4.5 ml of the mix reference stock solution, add water to the mark and mix well, prepare two duplicates.

Analyses,

Perform injections with the solutions obtained and calculate recoveries.

Result of accuracy for 4-hydroxybutane-1-sulfonic acid

	Level	Peak area	Inventor ry (μg)	Sample weight(mg)	Assay in sample (μg)	Amount detecte d(μg)	Recover y (%)	Mean recovery (%)	RS D%
1	80%	0.0748	45.5	96.1	19.34	63.32	96.65	99.47	1.4

2		0.0774	45.5	102.2	20.57	65.52	98.78		
3		0.0782	45.5	99.5	20.23	66.20	101.5		
4	100%	0.0990	63.7	99.0	19.93	83.80	100.3		
5		0.0996	63.7	100.1	20.15	84.31	100.7		
6		0.0989	63.7	102.3	20.59	83.72	99.10		
7	120%	0.1197	81.9	98.5	19.83	101.33	99.51		
8		0.1208	81.9	105	21.14	102.26	99.05		
9		0.1224	81.9	109.1	21.96	103.61	99.70		

Result of accuracy for Sodium chloride

	Level	Peak area	Inventor y (µg)	Sample weght(mg)	Assay in sample (µg)	Amount detecte d(µg)	Recove ry (%)	Mean recovery (%)	RS D%
1	80%	1.1794	100.75	96.1	64.38	162.21	97.09	98.60	1.8
2		1.2368	100.75	102.2	68.47	170.10	100.9		
3		1.2266	100.75	99.5	66.67	168.70	101.3		
4	100%	1.4828	141.05	99	66.33	203.93	97.56		
5		1.4939	141.05	100.1	67.07	205.46	98.12		
6		1.4981	141.05	102.3	68.54	206.04	97.48		
7	120%	1.8027	181.35	98.5	65.99	247.93	100.3		
8		1.806	181.35	105	70.35	248.39	98.17		
9		1.8034	181.35	109.1	73.10	248.03	96.46		

Result of accuracy for Bis(4-sulfobutyl) ether disodium

	Level	Peak area	Inventor y (µg)	Sample weght(mg)	Assay in sample (µg)	Amount detecte d(µg)	Recove ry (%)	Mean recovery (%)	RS D%
1	80%	0.1613	51	96.1	29.79	80.18	98.80	98.64	1.4
2		0.1642	51	102.2	31.68	81.62	97.92		
3		0.1625	51	99.5	30.85	80.77	97.90		
4	100%	0.201	71.4	99	30.69	99.91	96.95		
5		0.2018	71.4	100.1	31.03	100.31	97.03		
6		0.2097	71.4	102.3	31.71	104.24	101.6		
7	120%	0.2445	91.8	98.5	30.54	121.54	99.13		
8		0.2486	91.8	105	32.55	123.57	99.15		

9		0.2515	91.8	109.1	33.82	125.01	99.34		
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Conclusion,

Recoveries for the three substances and RSDs of relevant recoveries are all in required range.

Accuracy of the method is fine.

Representative chromatograms refer to Annex 3-4-18~Annex 3-4-21.

4.6 Intermediate precision

Proceed the below operations by two analysts on two different day.

Prepare reference solution and sample solutions, and perform injections with the solutions obtained as required.

Result of intermediate precision for 4-hydroxybutane-1-sulfonic acid

No.	Intermediate precision(1)	Intermediate precision(2)
1	0.023	0.023
2	0.023	0.023
3	0.023	0.023
4	0.023	0.022
5	0.023	0.022
6	0.023	0.023
Average	0.023	
RSD%	1.8	

Result of intermediate precision for Sodium chloride

No.	Intermediate precision(1)	Intermediate precision(2)
1	0.065	0.065
2	0.065	0.064
3	0.063	0.062
4	0.062	0.063
5	0.063	0.063
6	0.062	0.062
Average	0.063	
RSD%	1.8	

Result of intermediate precision for Bis(4-sulfobutyl) ether disodium

No.	Intermediate precision(1)	Intermediate precision(2)
1	0.031	0.031
2	0.031	0.031

3	0.031	0.030
4	0.030	0.031
5	0.031	0.030
6	0.030	0.030
Average	0.031	
RSD%	1.5	

Conclusion,

All the assays of the three substances and RSD of relevant assays are in the required range, the intermediate precision of this method is fine.

Representative chromatograms refer to Annex 3-4-22~Annex 3-4-25.

4.7 Repeatability

Prepare reference solution and six sample solutions and perform injections as required, calculate the assay of 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium and relevant RSD.

No.	1	2	3	4	5	6	Average	RSD%
4-hydroxybutane-1-sulfonic acid	0.023	0.024	0.024	0.024	0.024	0.024	0.024	0.6
Sodium chloride	0.061	0.062	0.062	0.062	0.062	0.062	0.062	0.3
Bis(4-sulfobutyl) ether disodium	0.031	0.030	0.030	0.031	0.031	0.031	0.031	1.0

Conclusion,

All the assays of the three substance are in the required range and RSDs of relevant assays are low enough, repeatability of this method is fine.

Representative chromatograms refer to Annex 3-4-26~Annex 3-4-27.

4.8 Robustness

Prepare reference solution and sample solution as required, perform injections at the conditions below,

Condition 1: normal condition;

Condition 2: flow rate 0.9ml/min;

Condition 3: flow rate 1.1ml/min;

Condition 4: column temperature 28°C;

Condition 5: column temperature 32°C;

Condition 6: solution A 4.5mmol/L sodium hydroxide;

Condition 7: solution A 5.5mmol/L sodium hydroxide;

Result of robustness for 4-hydroxybutane-1-sulfonic acid

No.	Conditions	Assay of 4-hydroxybutane-1-sulfonic acid
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		(%)
1	Normal condition	0.0228
2	Flow rate 0.9ml/min	0.0228
3	Flow rate 1.1ml/min	0.0227
4	Column temperature 28°C	0.0227
5	Column temperature 32°C	0.0228
6	Solution A 4.5mmol/L sodium hydroxide	0.0230
7	Solution A 4.5mmol/L sodium hydroxide	0.0228
Average (%)		0.0228
RSD (%)		0.5

Result of robustness for Sodium chloride

No.	Conditions	Assay of Sodium chloride (%)
1	Normal condition	0.0671
2	Flow rate 0.9ml/min	0.0674
3	Flow rate 1.1ml/min	0.0670
4	Column temperature 28°C	0.0670
5	Column temperature 32°C	0.0673
6	Solution A 4.5mmol/L sodium hydroxide	0.0670
7	Solution A 4.5mmol/L sodium hydroxide	0.0672
Average (%)		0.0671
RSD (%)		0.2

Result of robustness for Bis(4-sulfobutyl) ether disodium

No.	Conditions	Assay of Bis(4-sulfobutyl) ether disodium (%)
1	Normal condition	0.0318
2	Flow rate 0.9ml/min	0.0321
3	Flow rate 1.1ml/min	0.0319
4	Column temperature 28°C	0.0321
5	Column temperature 32°C	0.0321
6	Solution A 4.5mmol/L sodium hydroxide	0.0318
7	Solution A 4.5mmol/L sodium hydroxide	0.0319
Average (%)		0.0319
RSD (%)		0.4

Conclusion,

We can see that slight changes on the chromatographic conditions will not significantly effect the detections of 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium, robustness of this method is fine.

Representative chromatograms refer to Annex 3-4-28~Annex 3-4-41.

4.9 Solution stability

Prepare reference solution and sample solution as intermediate precision, perform injections with the solutions obtained at different times, record chromatograms and count the change of the three substances.

Result of reference solution

Time/h		0	2	8	10	12	24	26	RSD(%)
4-hydroxybutane-1-sulfonic acid	Peak area	0.1080	0.1083	0.1084	0.1093	0.1107	0.1084	0.1098	0.9
	Deviation with 0h (%)	/	0.14	0.18	0.60	1.23	0.18	0.83	
Sodium chloride	Peak area	1.4306	1.4198	1.4541	1.4386	1.4449	1.4380	1.4310	0.77
	Deviation with 0h (%)	/	0.38	0.81	0.28	0.50	0.26	0.01	
Bis(4-sulfobutyl) ether disodium	Peak area	0.1841	0.1899	0.1866	0.1850	0.1898	0.1898	0.1842	1.47
	Deviation with 0h (%)	/	1.55	0.54	0.24	1.52	1.52	0.027	

Result of sample solution

Time/h		0	1	5	8	10	12	24	26	RSD(%)
4-hydroxybutane-1-sulfonic acid	Peak area	0.0301	0.0304	0.0300	0.0301	0.0304	0.0303	0.0302	0.0303	0.50
	Deviation with 0h (%)	/	0.49	0.16	0	0.50	0.33	0.17	0.33	
Sodium chloride	Peak area	0.4414	0.4419	0.4343	0.4466	0.4335	0.4446	0.4412	0.4458	1.11
	Deviation with 0h (%)	/	0.06	0.87	0.59	0.90	0.36	0.02	0.50	
Bis(4-sulfobutyl) ether disodium	Peak area	0.0557	0.0535	0.0539	0.0542	0.0533	0.0549	0.0534	0.0538	1.54
	Deviation with 0h (%)	/	2.01	1.64	1.27	2.20	0.72	2.1	1.74	

Conclusion,

Peak areas of 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether

disodium from both reference solution and sample solution have no obvious changes, RSD of relevant peak areas and deviations are all less than 2.0%, reference solution and sample solution are stable for 26hours.

Representative chromatograms refer to Annex 3-4-42~Annex 3-4-49.

5. Validation conclusion

After validation we can see, all the items validated meet relevant requirements, the analysis method has enough specificity and sensitivity to control 4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfobutyl) ether disodium in the final product.

Validation of analysis procedure for Assay**1. Equipments, reagents and solvents**

Equipments	Name	Type	Manufacturer
	HPLC	Agilent 1260	Agilent
	Differential detector	Agilent 1260-RID	Agilent
	Balance	XS205	Mettler
Reagents and solvents	Name	Grade	Manufacturer
	Potassium nitrate	ACS	Aladdin
	Acetonitrile	HPLC	ACS
	Ultrapure water	HPLC	Milli-Q

2. Analysis procedure

Refer to 3.2.S.4.2 Assay.

3. Validation items and summary

<i>Item</i>	<i>Validation result</i>
Specificity	The blank has no interference on the detection of main substance.
System suitability	RSD of main peak areas is 0.3%.
LOQ	LOQ of Betadex Sulfobutyl Ether Sodium is 0.04mg /ml.
Linearity and range	Linearity formula is $Y=260001.8X+47206.0$ ($r=0.9998$; $n=5$), with concentration of Betadex Sulfobutyl Ether Sodium in the range of 9.0~13.5mg/ml.
Precision	RSD of repeatability is 1.1% ($n=6$); RSD of intermediate precision is 0.8% ($n=9$).
Solution stability	Reference solution and sample solution are stable in 24 hours.
Robustness	Little changes of chromatographic condition have no influence on the analysis results, main peak can still separate with the adjacent peak and RSD of analysis result is 0.6%..

4. Content of validation**4.1 Specificity**

Preparation,

Reference solution: weigh defined amount of Betadex Sulfobutyl Ether Sodium reference standard dried at 120°C for 3hours and prepare solution of 12mg/ml, mix well.

Blank: mobile phase.

Analyses,

Perform injections with the solutions obtained as required, and the chromatograms indicate that blank has no interference on the detection of main peak.

Representative chromatograms refer to Annex 3-5-1~Annex 3-5-2.

4.2 System suitability

Perform six injections with the reference solution in specificity and calculate RSD of the peak areas.

No.	1	2	3	4	5	6	Average	RSD%
Peak area	3.22×10^6	3.23×10^6	3.23×10^6	3.24×10^6	3.22×10^6	3.23×10^6	3.23×10^6	0.3

Conclusion,

RSD of the peak areas is 0.3%, it is low enough to indicate that the system suitability is fine.

Representative chromatograms refer to Annex 3-5-3~Annex 3-5-4.

4.3 LOQ

Dilute the reference solution above and perform injection with it until S/N is 10:1, the concentration of that solution, 0.04mg/ml, is LOQ and perform six injections with it, calculate RSD of the six peak areas.

No.	1	2	3	4	5	6	Average	RSD%
Peak area	13747.0	11444.3	14247.2	12767.4	14718.7	12179.6	13184.0	9.7

Conclusion,

LOQ of Betadex Sulfobutyl Ether Sodium is 0.04mg/ml, and RSD of peak areas from six injections of LOQ solution is 9.7%.

Representative chromatograms refer to Annex 3-5-5~Annex 3-5-6.

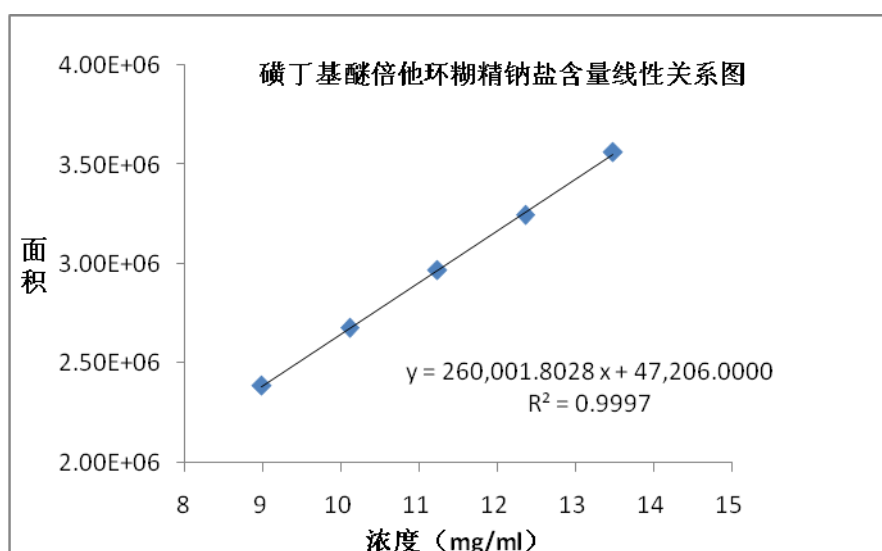
4.4 Linearity and range

Preparation,

Accurately weigh defined amount of Betadex Sulfobutyl Ether Sodium reference standard, diluted with mobile phase to prepare linearity solutions with concentrations of 9.0mg/ml, 10.1mg/ml, 11.2mg/ml, 12.4mg/ml and 13.5mg/ml, perform injections with the solutions obtained and draw linearity regression curve with concentrations as x axis and peak areas as y axis.

Linearity regression equation is $Y=260001.8X+47206.0$ ($r=0.9998$; $n=5$), in the range of 9.0~13.5mg/ml, which is equal to 75%~112% of the concentration of main substance.

Linearity regression curve



Representative chromatograms refer to Annex 3-5-7~Annex 3-5-9.

4.5 Repeatability

Preparation,

Reference solution: weigh defined amount of Betadex Sulfobutyl Ether Sodium reference standard dried at 120°C for 3hours and prepare solution of 12mg/ml, mix well.

Sample solution: prepare sample solution with concentration of 12mg/ml with the specimen, mix well, prepare five duplicates.

Analyses,

Perform injections with the solutions obtained, calculate assays of the six sample solutions and RSD of the six assays.

Result of repeatability

Assay of Betadex Sulfobutyl Ether Sodium(%)							Average	RSD%
No.	1	2	3	4	5	6		
Weight of sample(mg)	120.1	120	120	120.1	120	119.8	/	/
Assay(%)	100.3	100.2	102.4	101.9	102.4	102.4	101.6	1.1

Conclusion,

All the six assays are in the required limit and RSD of them is 1.1% which is low enough to indicate the repeatability of this method is fine.

Representative chromatograms refer to Annex 3-5-10~Annex 3-5-12.

4.6 Intermediate precision

Preparation,

Reference solution: refer to the preparation of reference solution in repeatability.

Sample solution: refer to the preparation of sample solution in repeatability, prepare two duplicates.

Analyses,

Perform injections with the solutions obtained, calculate assays of the sample solutions and RSD of the assays obtained from this item and repeatability.

Result of intermediate precision

No.	Result of repeatability	Result of intermediate precision	
	Assay (%)	Weight of sample(mg)	Assay (%)
1	100.3	120.1	101.3
2	100.2	120.2	101.4
3	102.4	120.1	101.2
4	101.9	/	/
5	102.4	/	/
6	102.4	/	/
Average (%)	101.5		
RSD(%)	0.8		

Conclusion,

All the assays are in the required limit and RSD of them is 0.8% which is low enough to indicate the precision of this method is fine.

Representative chromatograms refer to Annex 3-5-13~Annex 3-5-18.

4.7 Solution suitability

Preparation,

Reference solution: refer to the preparation of reference solution in repeatability.

Sample solution: namely the No. 6 sample solution in repeatability.

Analyses,

Reference solution should be kept in normal place, one portion of sample solution should be kept in dark place and another portion in normal place, perform injections with the solutions at suitable time, record chromatograms, calculate the peak area deviations with 0h in reference solution, and assay deviations with 0h in sample solutions.

Result of reference solution

Time (h)	Peak area	Deviation with 0h (%)
0	2935110	/
1	2946750	0.20
2	2953560	0.31
4	2943040	0.13
6	2955820	0.35

8	2959180	0.41
10	2959360	0.41
12	2959930	0.42
24	2973380	0.65

Result of sample solution

Storage condition	Time (h)	0	1	2	5	8	11	14	26
Dark place	Assay (%)	102.2	102.2	102.1	100.5	100.7	100.6	100.4	102.0
	Deviation with 0h (%)	/	0.01	0.06	0.85	0.74	0.79	0.88	0.09
Normal place	Assay (%)	101.9	101.8	101.7	100.0	100.0	100.7	100.5	102.0
	Deviation with 0h (%)	/	0.04	0.08	0.96	0.93	0.62	0.70	0.03

Representative chromatograms refer to Annex 3-5-19~Annex 3-5-29.

4.8 Robustness

Prepare reference solution and sample solution as required and perform injections at chromatographic conditions as below,

Condition 1, normal condition;

Condition 2, flow rate 0.5ml/min;

Condition 3, flow rate 0.7ml/min;

Condition 4, column temperature, 33°C;

Condition 5, column temperature, 37°C;

Condition 6, 0.1 M potassium nitrate in a mixture of acetonitrile and water (13:87);

Condition 7, 0.1 M potassium nitrate in a mixture of acetonitrile and water (9:91).

Result of robustness

No.	Chromatographic condition	Assay (%)
1	Normal condition	101.6
2	Flow rate 0.5ml/min	102.3
3	Flow rate 0.7ml/min	102.1
4	Column temperature, 33°C	102.6
5	Column temperature, 37°C	102.3
6	Acetonitrile : water (87:13)	100.9
7	Acetonitrile : water (91:9)	101.5
Average (%)		101.9
RSD (%)		0.6

Conclusion,

Assays from different conditions are all in required range and RSD of them is 0.6%, so robustness of this method is fine.

Representative chromatograms refer to Annex 3-5-30~Annex 3-5-41.

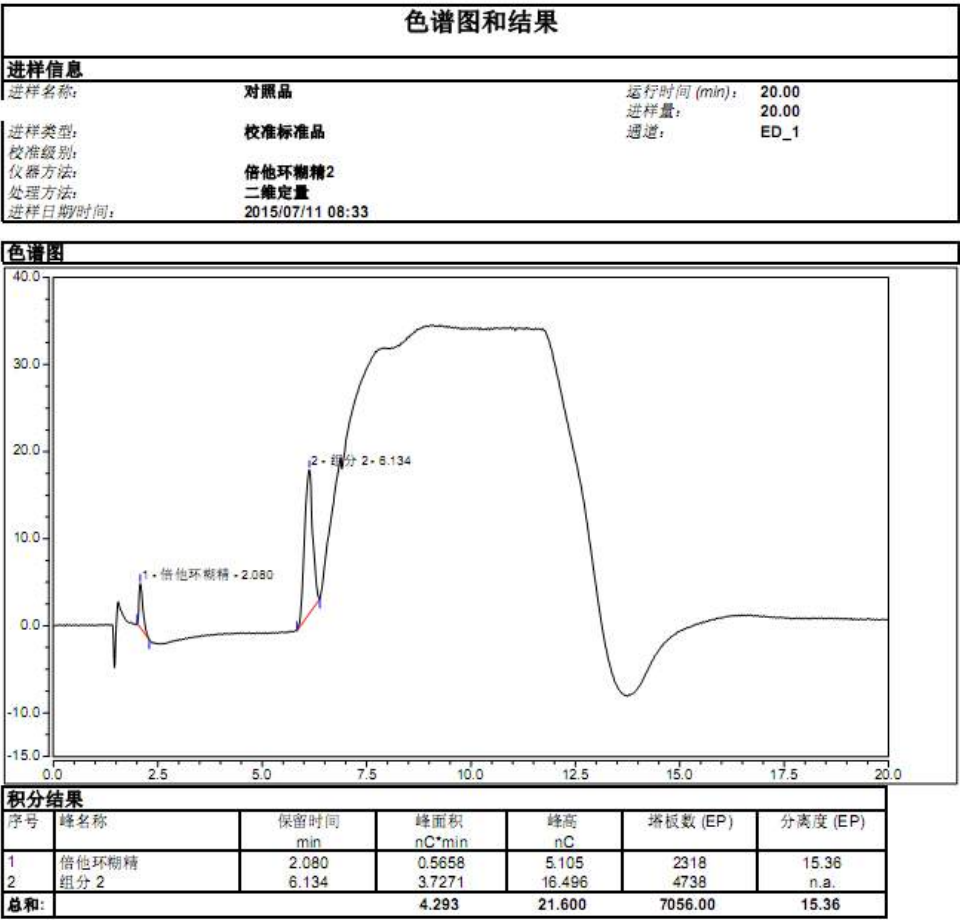
5. Validation conclusion

After validation we can see, all the items validated meet relevant requirements, the analysis method has enough specificity and sensitivity to control the assay of the final product.

Annex 3-1-1 Validation of analysis procedure for Betadex-Specificity-Reference solution

仪器:CS-5000+ 序列:专属性

页码 1/3



附图10. 4. 8 -14HPLC中倍他环糊精的测定方法验证图（专属性-对照品溶液）

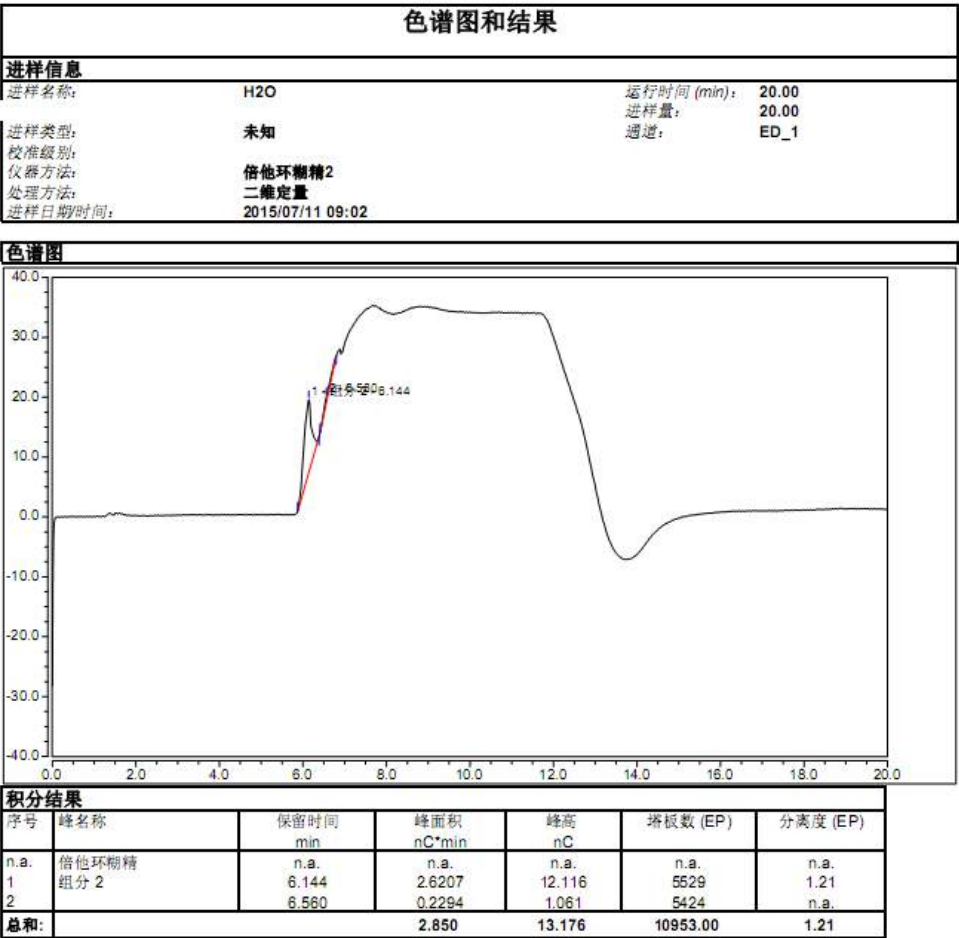
Default积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-2 Validation of analysis procedure for Betadex-Specificity-Blank

仪器:CS-5000+ 序列:专属性

页码:2/3

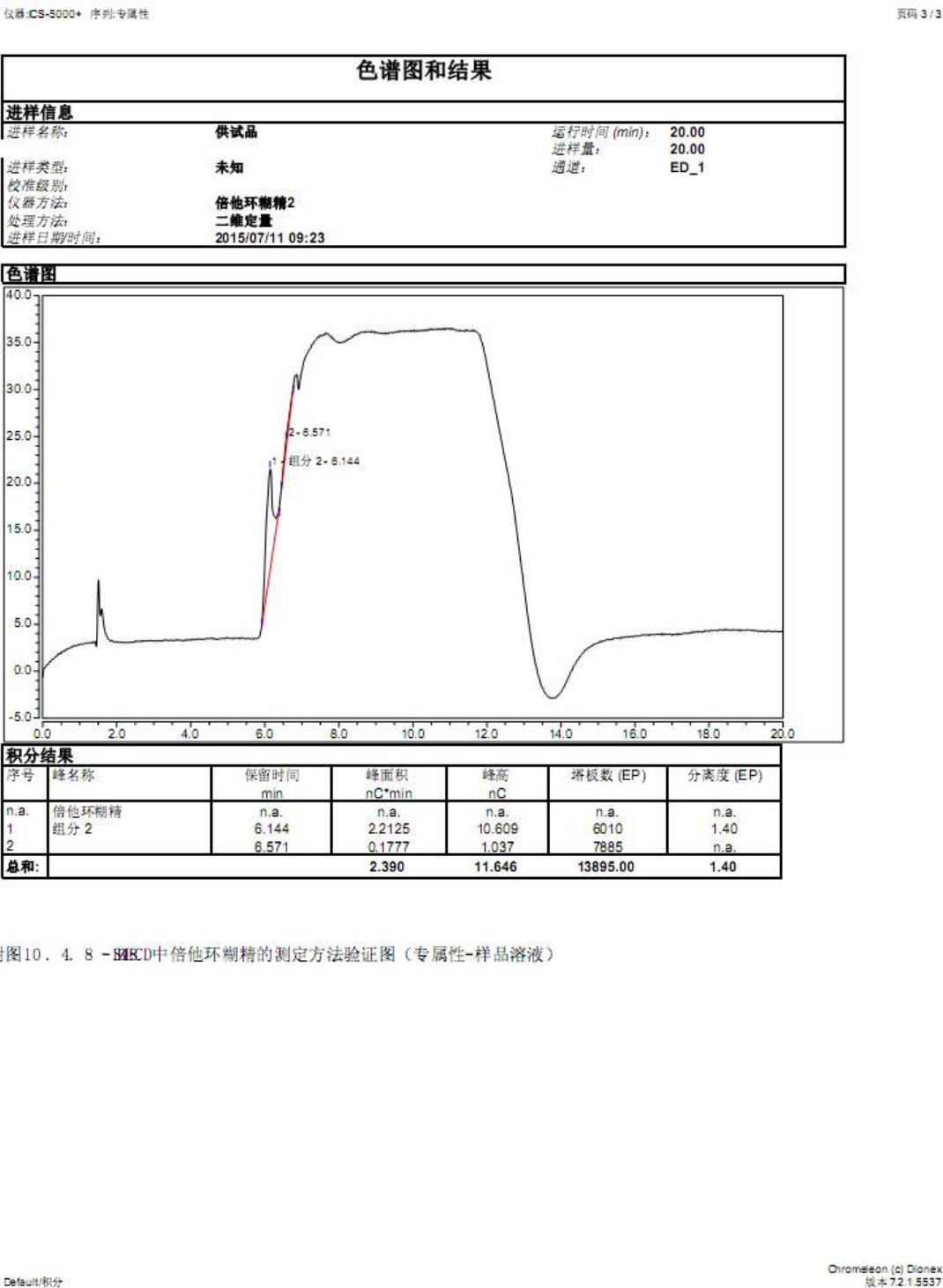


附图10. 4. 8 -146BECD中倍他环糊精的测定方法验证图（专属性-空白溶剂）

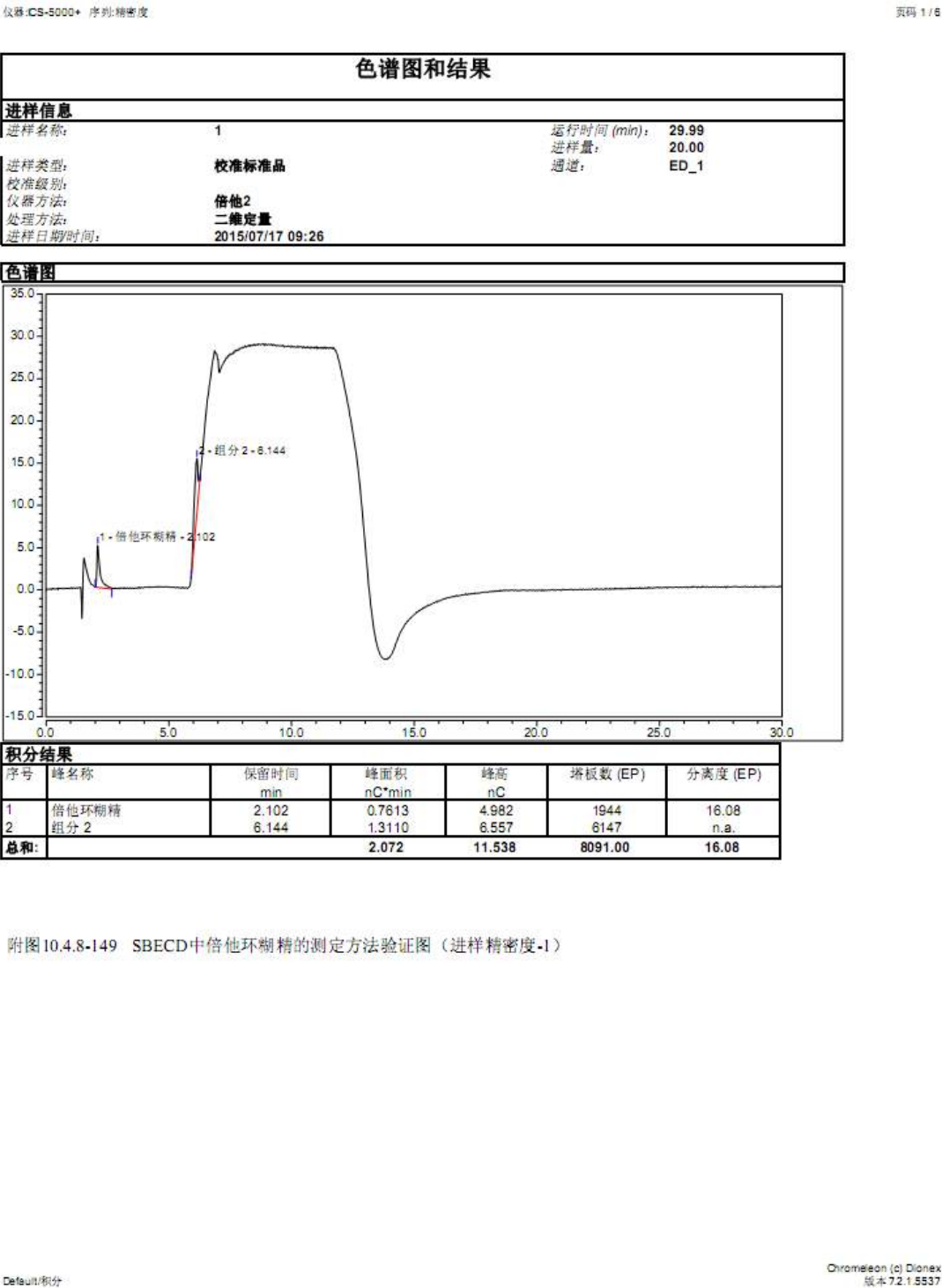
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-3 Validation of analysis procedure for Betadex-Specificity-Sample solution



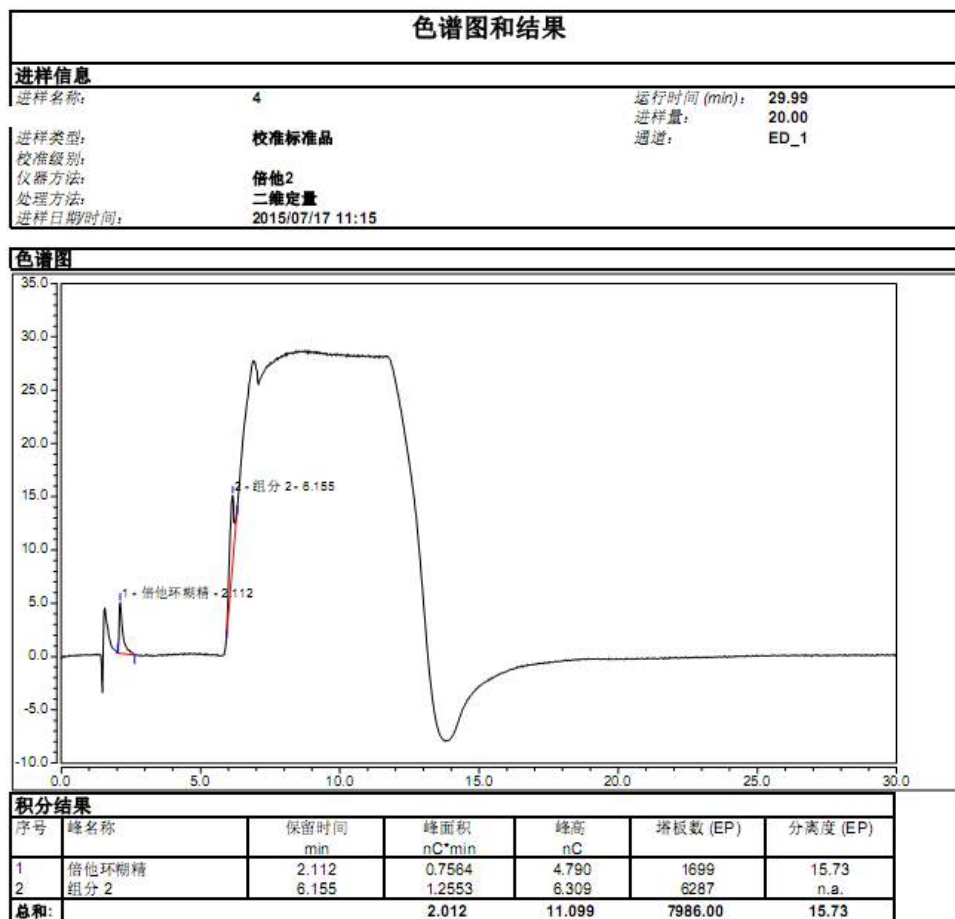
Annex 3-1-4 Validation of analysis procedure for Betadex-System suitability-Reference solution 1



Annex 3-1-5 Validation of analysis procedure for Betadex-System suitability-Reference solution 4

仪器:CS-5000+ 序列:精密度

页码 4 / 6



附图10.4.8-152 SBECD中倍他环糊精的测定方法验证图(进样精密度-4)

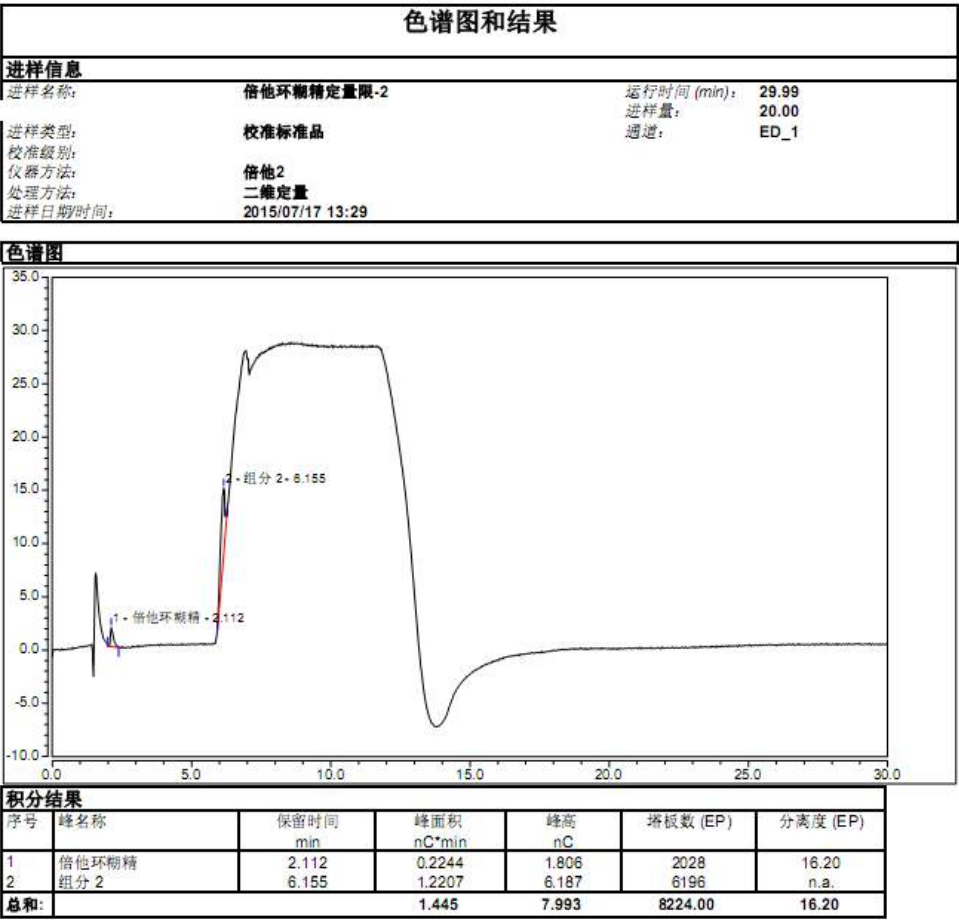
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-6 Validation of analysis procedure for Betadex-LOD and LOQ-LOQ 2

仪器:ICS-5000+ 序列:定量限, 检测限, 线性

页码 2 / 11

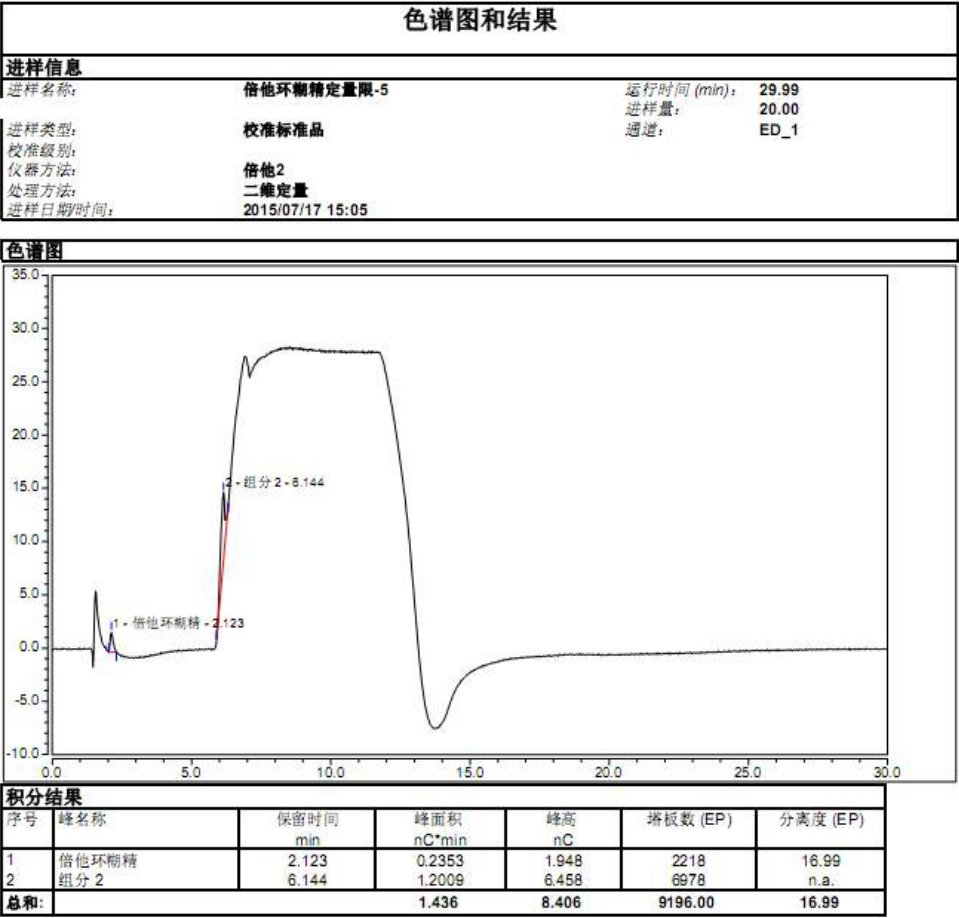


附图10.4.8-157 SBECD中倍他环糊精的测定方法验证图（定量限-2）

Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-7 Validation of analysis procedure for Betadex-LOD and LOQ-LOQ 5

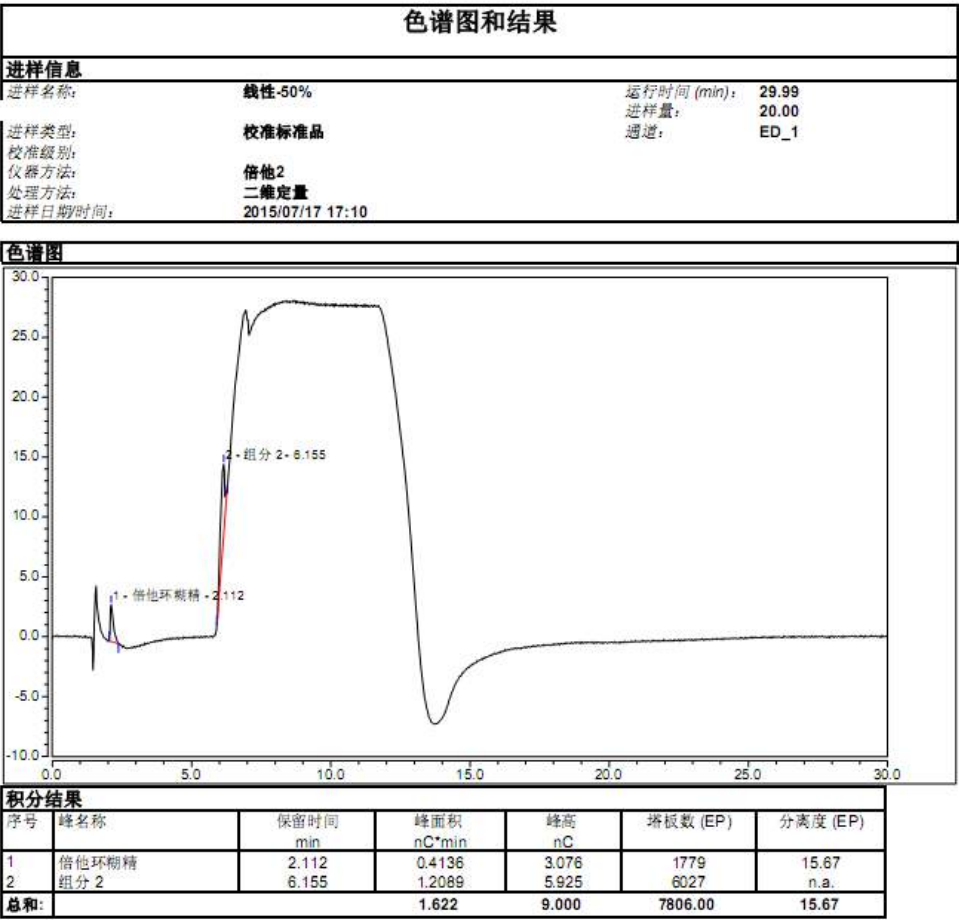


附图10.4.8-160 SBECD中倍他环糊精的测定方法验证图（定量限·5）

Annex 3-1-8 Validation of analysis procedure for Betadex-Linearity and range-Linearity 2

仪器:ICS-5000+ 序列:定量限, 检测限, 线性

页码 7 / 11



附图10.4.8-161 SBECD中倍他环糊精的测定方法验证图 (线性-50%)

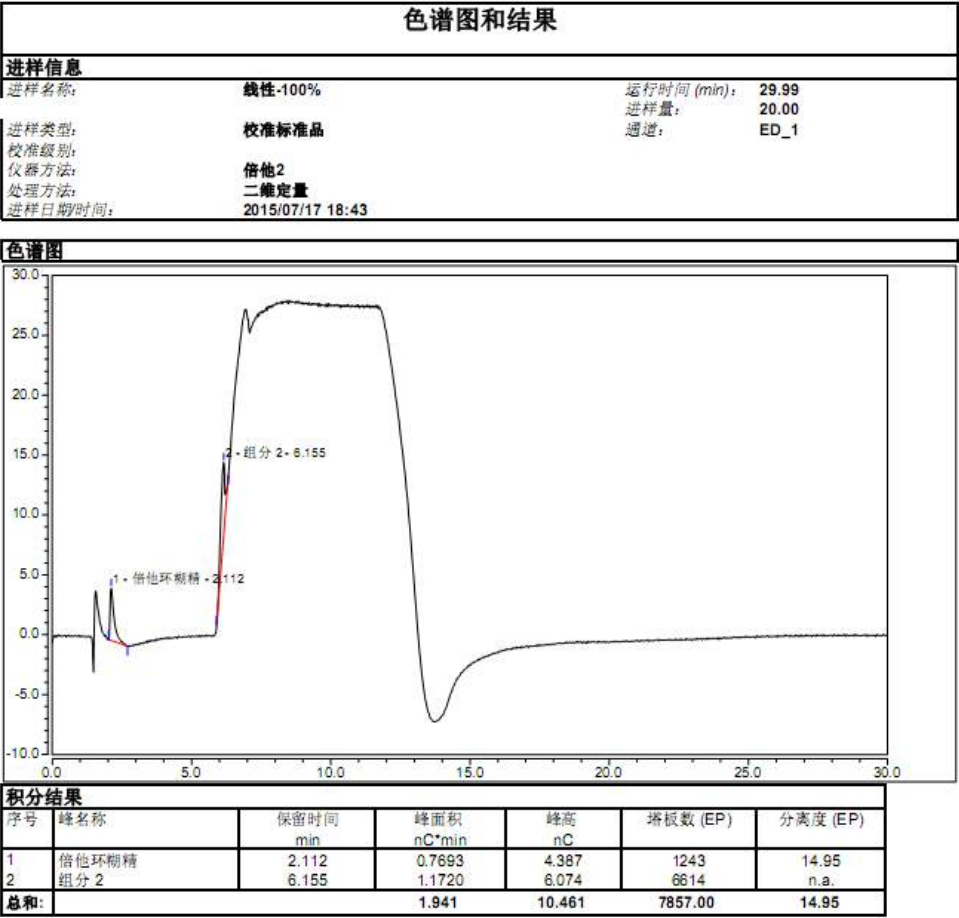
Default积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-9 Validation of analysis procedure for Betadex-Linearity and range-Linearity 4

仪器:ICS-5000+ 序列:定量限, 检测限, 线性

页码 9 / 11

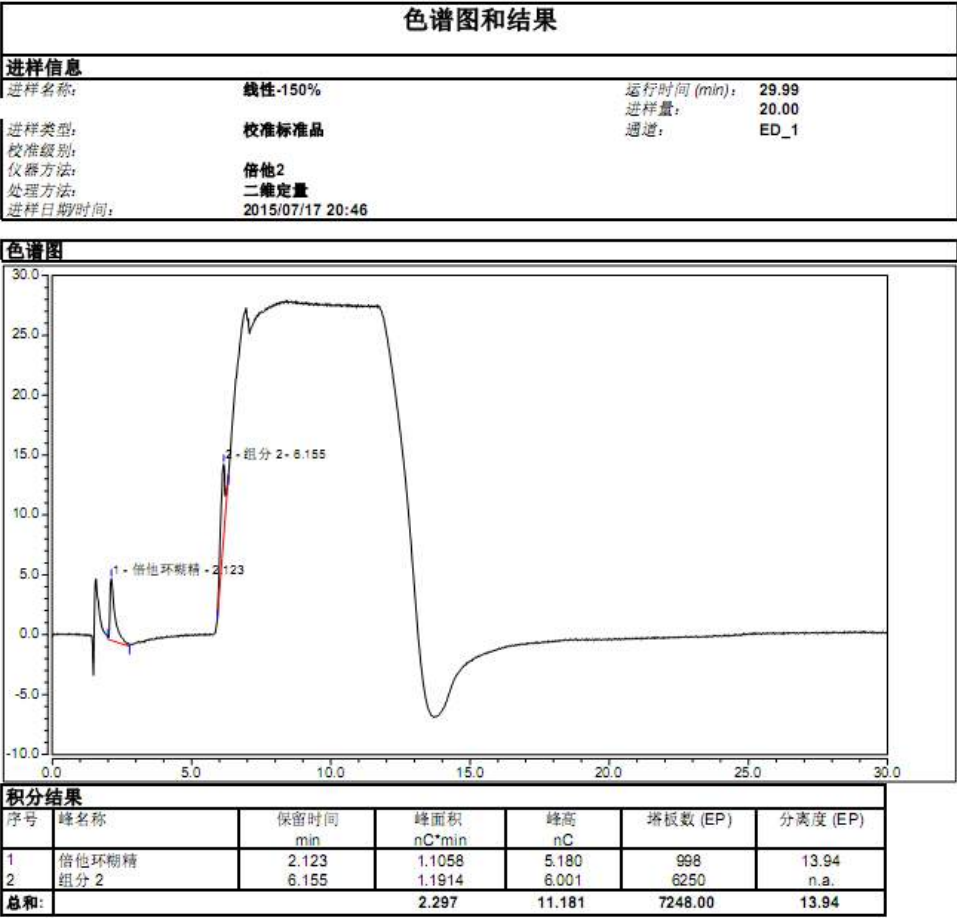


附图10.4.8-163 SBECD中倍他环糊精的测定方法验证图（线性-100%）

Default积分

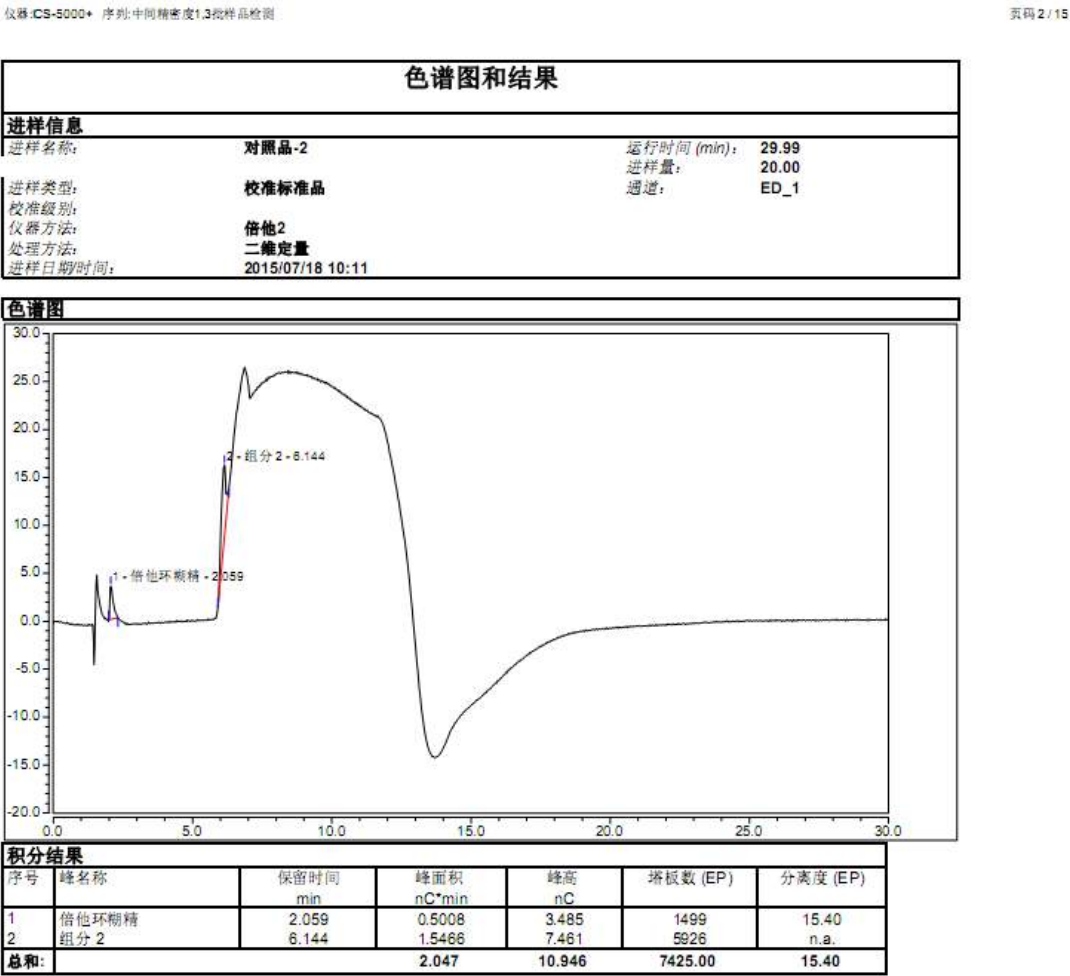
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-10 Validation of analysis procedure for Betadex-Linearity and range-Linearity 6



附图10.4.8-165 SBECD中倍他环糊精的测定方法验证图（线性-150%）

Annex 3-1-11 Validation of analysis procedure for Betadex-Repeatability and intermediate precision-Reference solution1-2

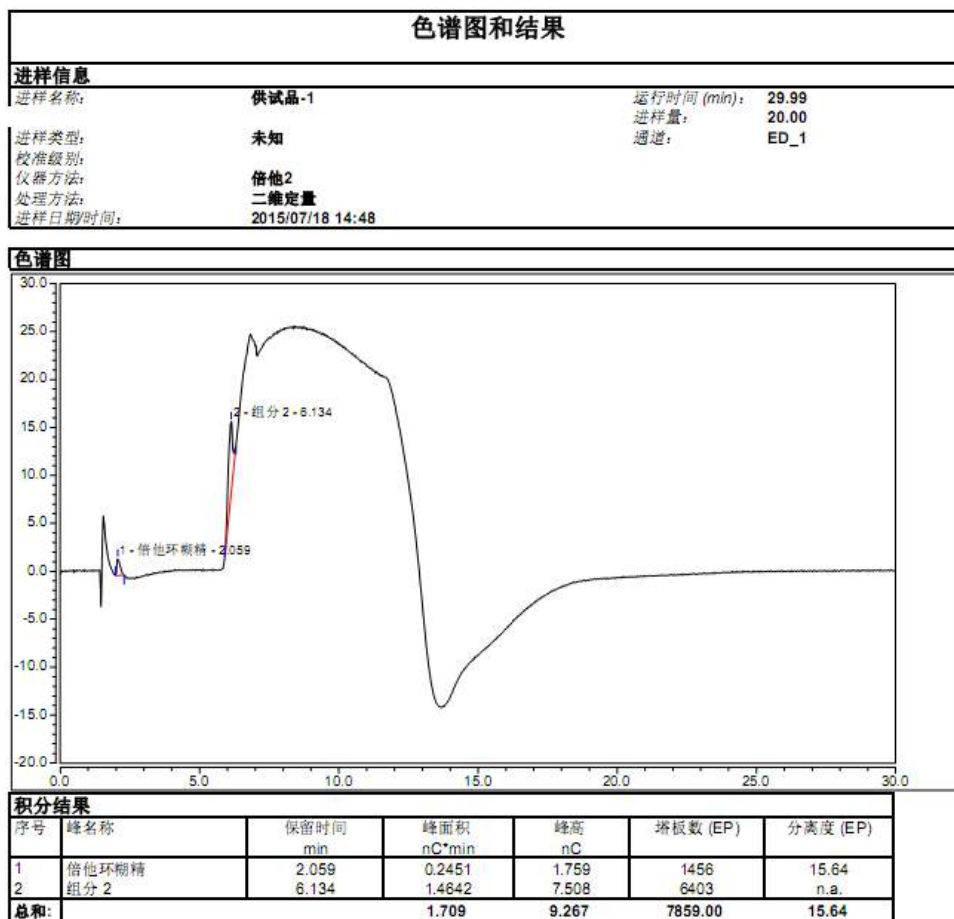


附图10.4.8-167 SBECD中倍他环糊精的测定方法验证图（准确度，3批样品检测，中间精密度1-对照-2）

Annex 3-1-12 Validation of analysis procedure for Betadex-Repeatability and intermediate precision-Repeatability sample solution 1

仪器:CS-5000+ 序列:中间精密度1.3批样品检测

页码 10 / 15

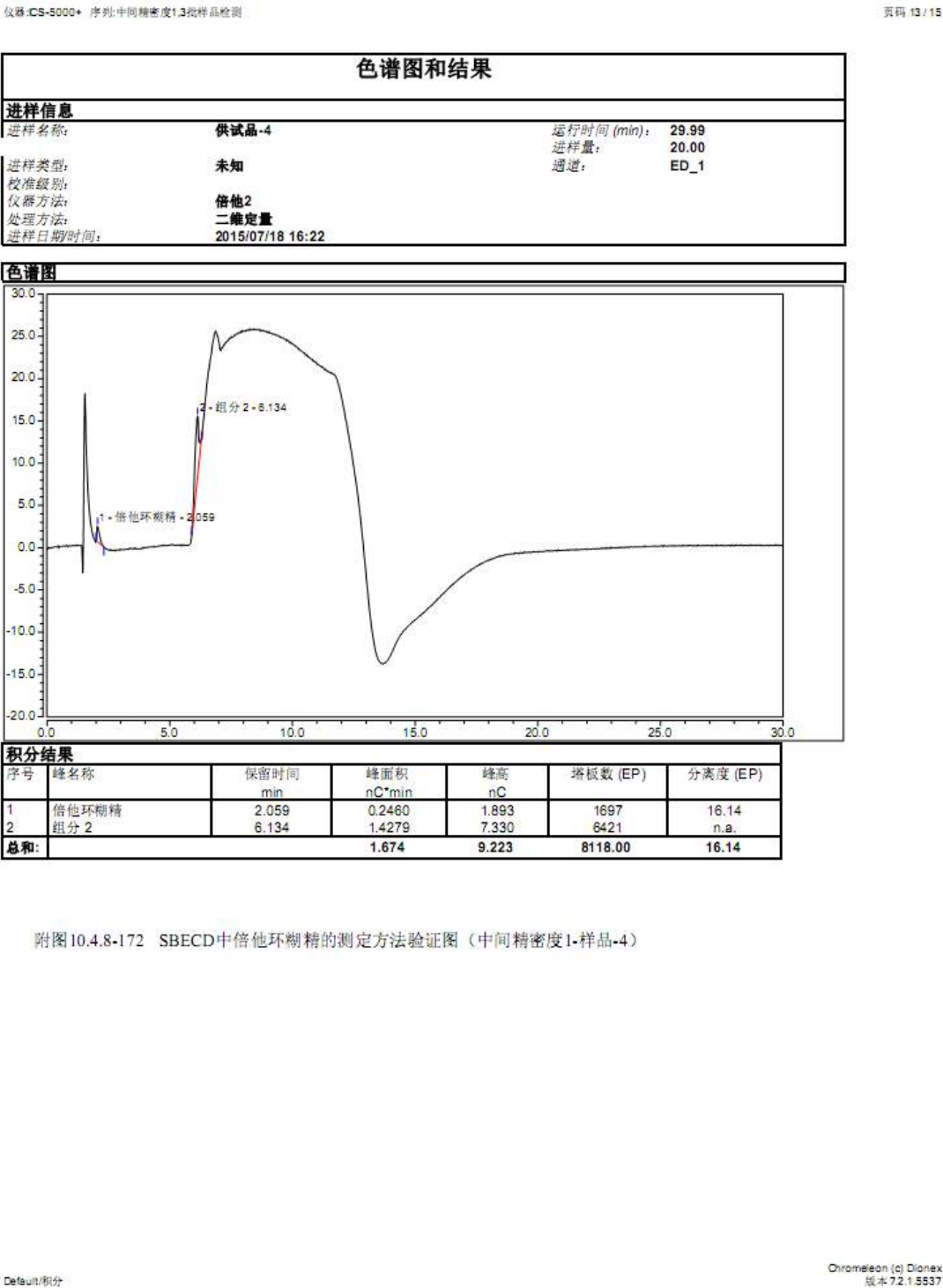


附图10.4.8-169 SBECD中倍他环糊精的测定方法验证图 (中间精密度1-样品-1)

Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-13 Validation of analysis procedure for Betadex-Repeatability and intermediate precision-Repeatability sample solution 4



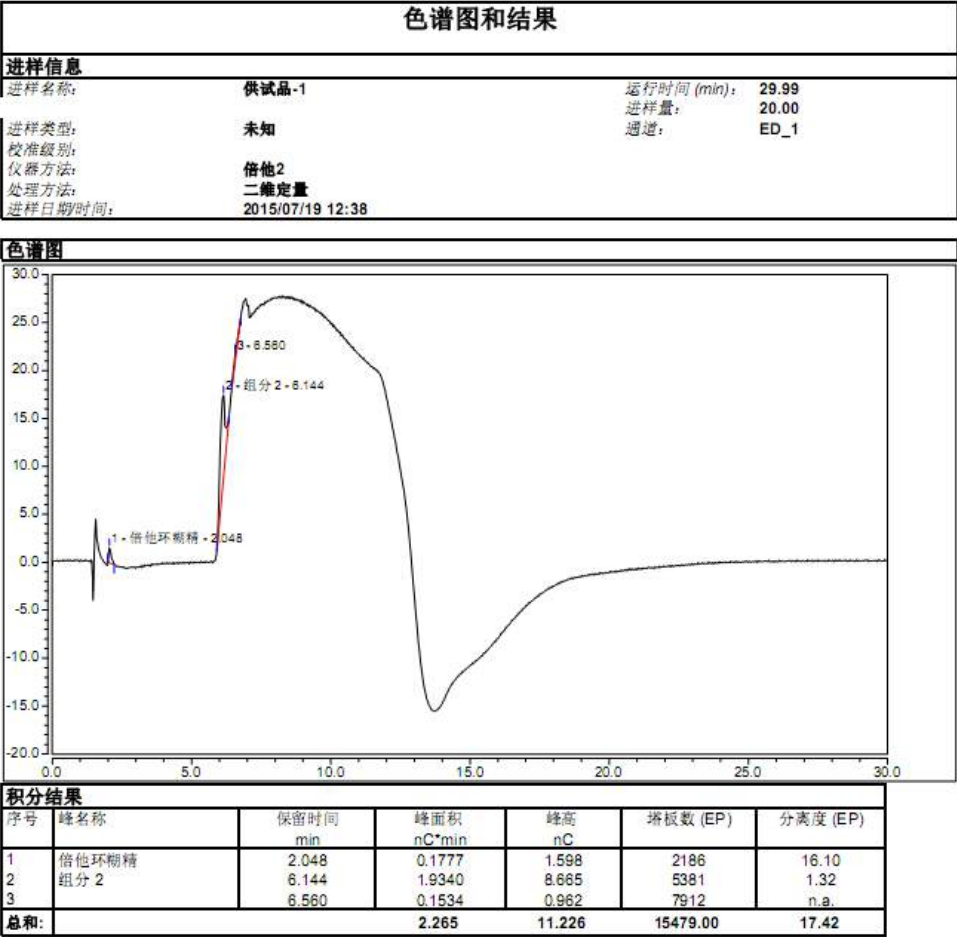
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-14 Validation of analysis procedure for Betadex-Repeatability and intermediate precision- Intermediate precision sample solution 1

仪器:CS-5000+ 序列:中间精密度2

页码 1 / 4

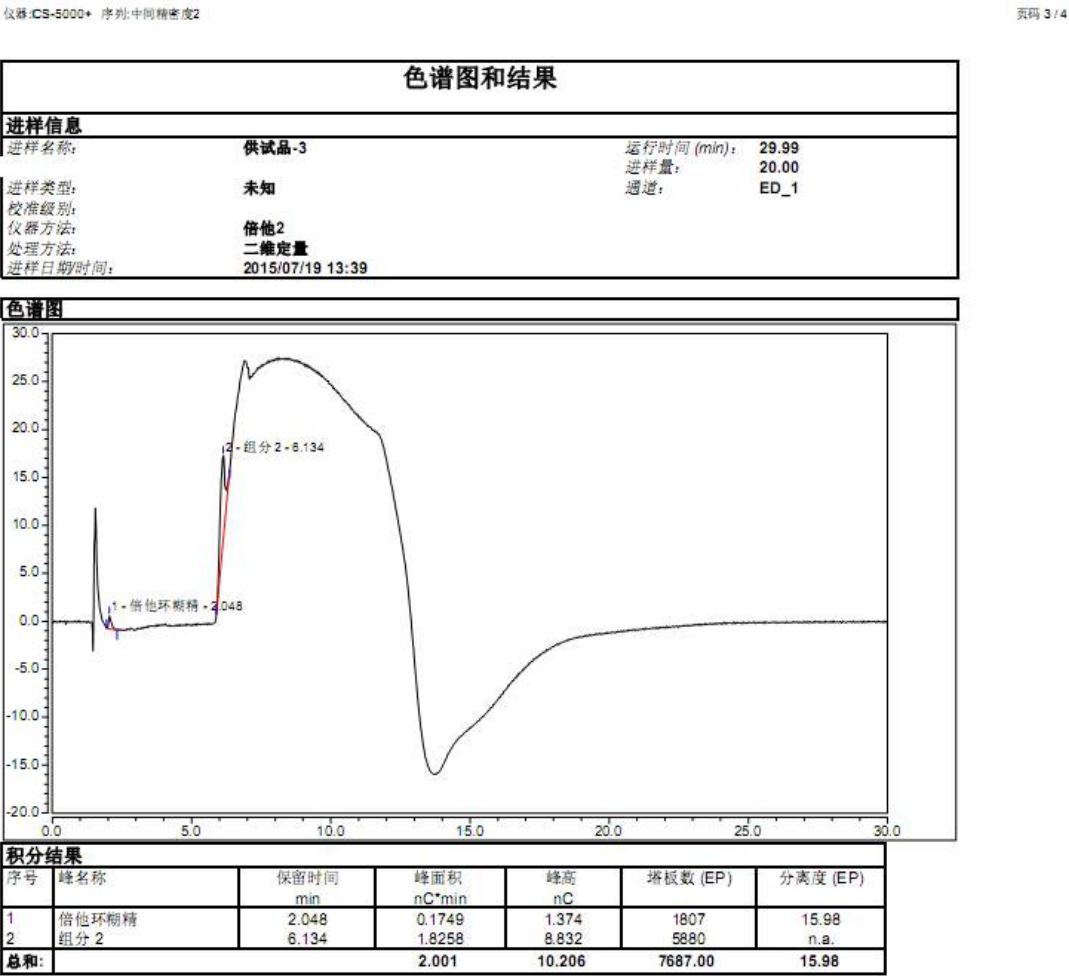


附图10. 4. 8 -HPLC中倍他环糊精的测定方法验证图（中间精密度2 -样品-1）

Default积分

Chromleon (c) Dionex
版本 7.2.1.5537

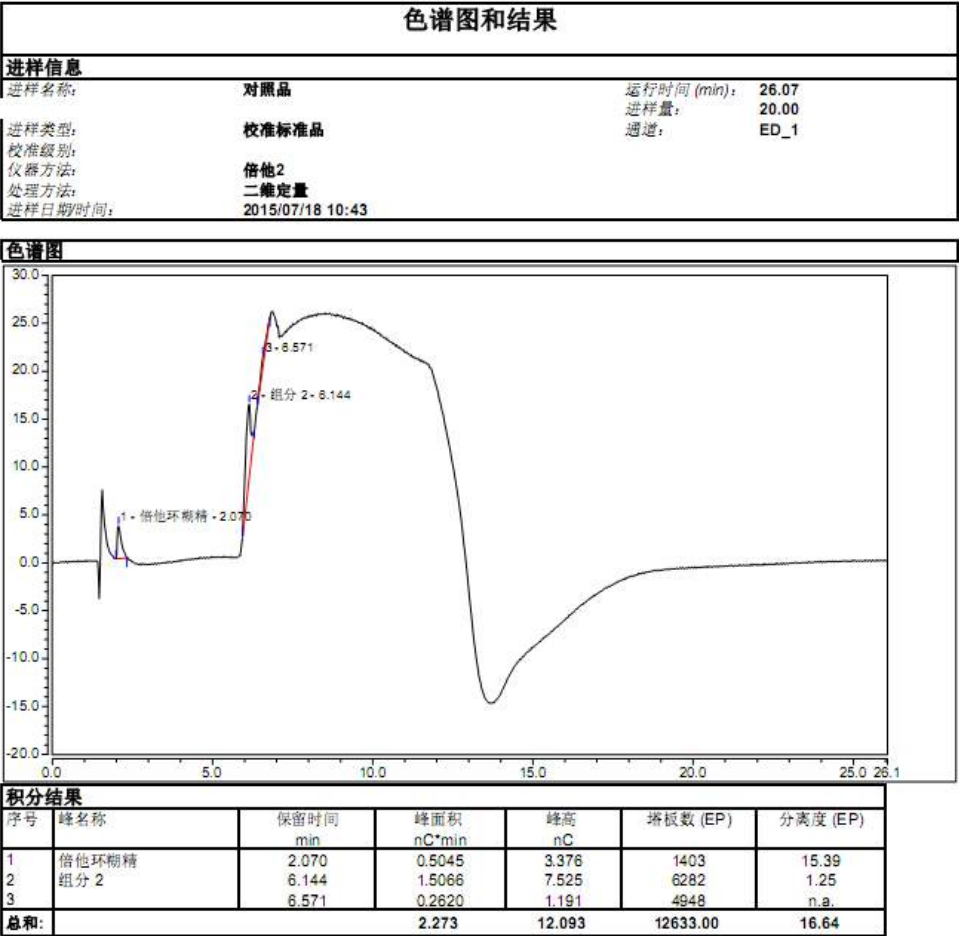
Annex 3-1-15 Validation of analysis procedure for Betadex-Repeatability and intermediate precision- Intermediate precision sample solution 3



Annex 3-1-16 Validation of analysis procedure for Betadex-Accuracy-Reference solution2-1

仪器:CS-5000+ 序列:中间精密度1.3批样品检测

页码 3 / 15



附图10.4.8-168 SBECD中倍他环糊精的测定方法验证图（中间精密度2-对照-1）

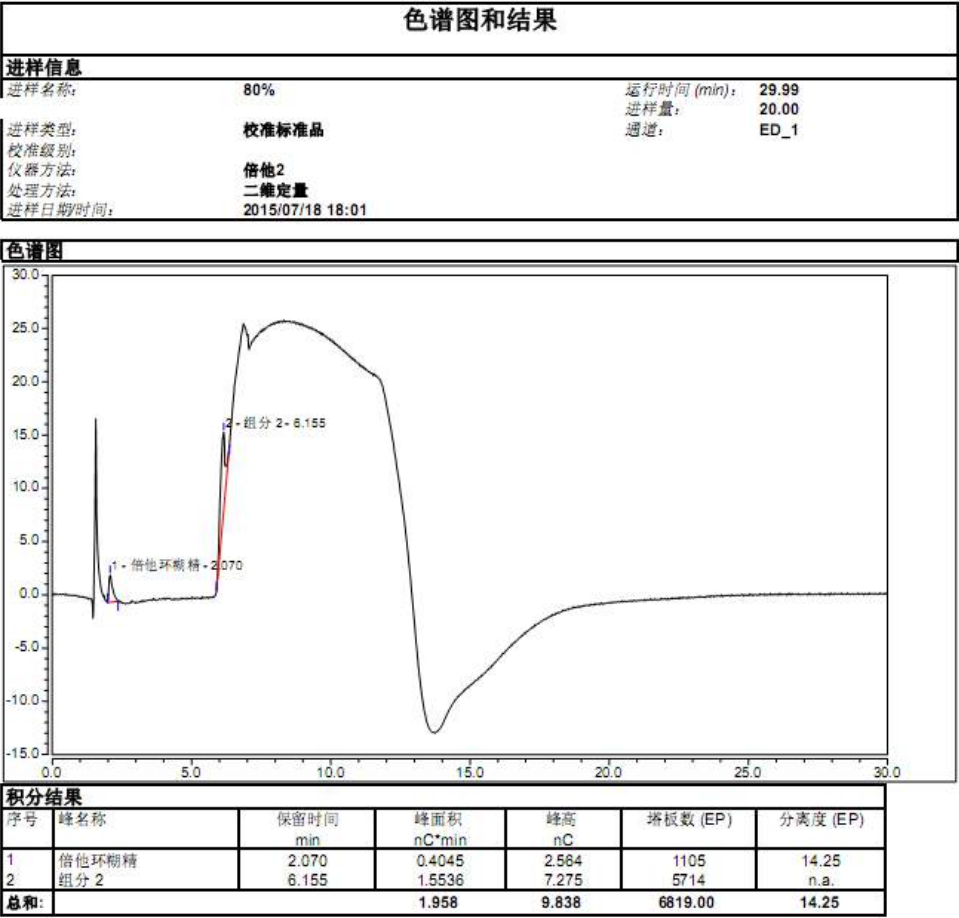
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-17 Validation of analysis procedure for Betadex-Accuracy-80%-1

仪器:CS-5000+ 序列:准确度

页码 1/9



附图10.4.8-179 SBECD中倍他环糊精的测定方法验证图（准确度-80%）

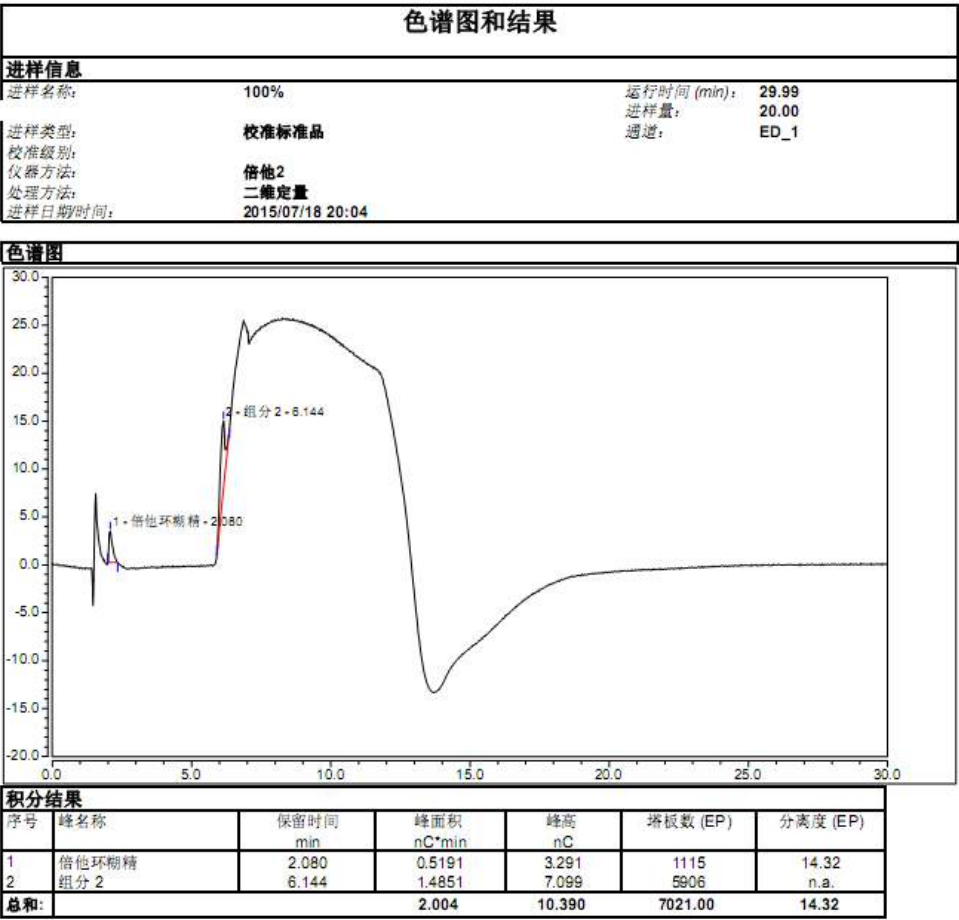
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-18 Validation of analysis procedure for Betadex-Accuracy-100%-1

仪器:CS-5000+ 序列:准确度

页码 4 / 9



附图10.4.8-182 SBECD中倍他环糊精的测定方法验证图 (准确度-100%)

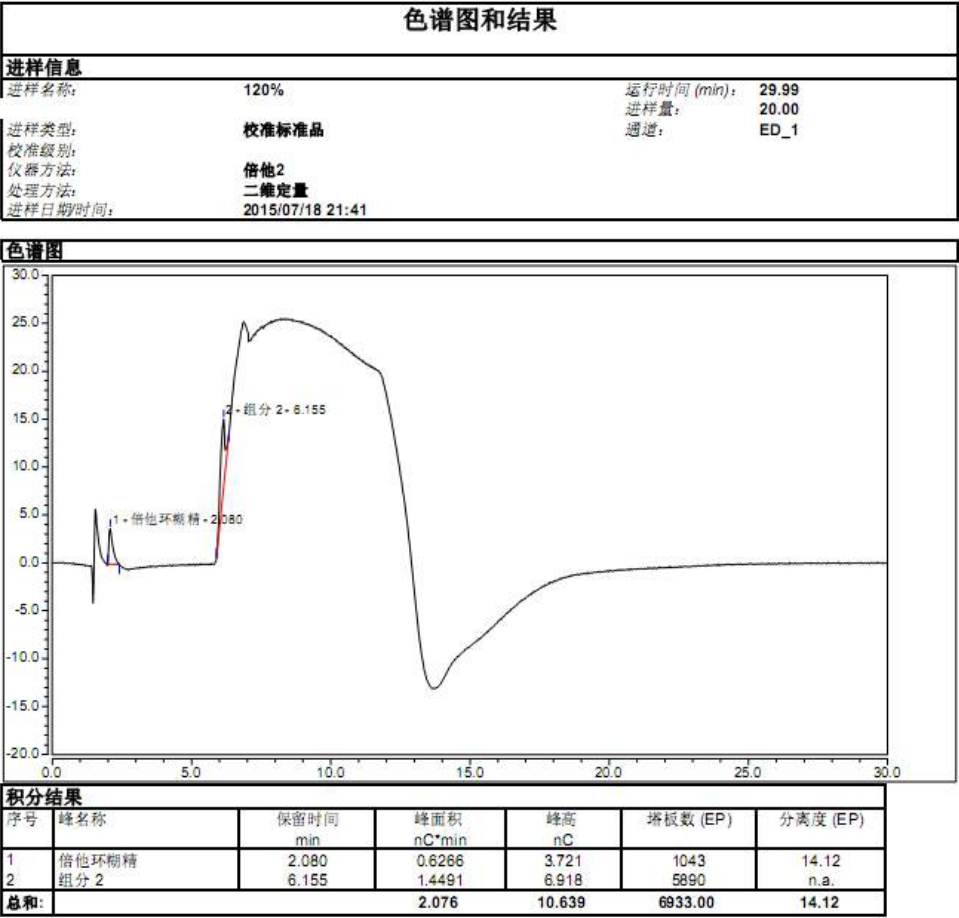
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-19 Validation of analysis procedure for Betadex-Accuracy-120%-1

仪器:CS-5000+ 序列:准确度

页码 7/9



附图10.4.8.185 SBECD中倍他环糊精的测定方法验证图 (准确度-120%)

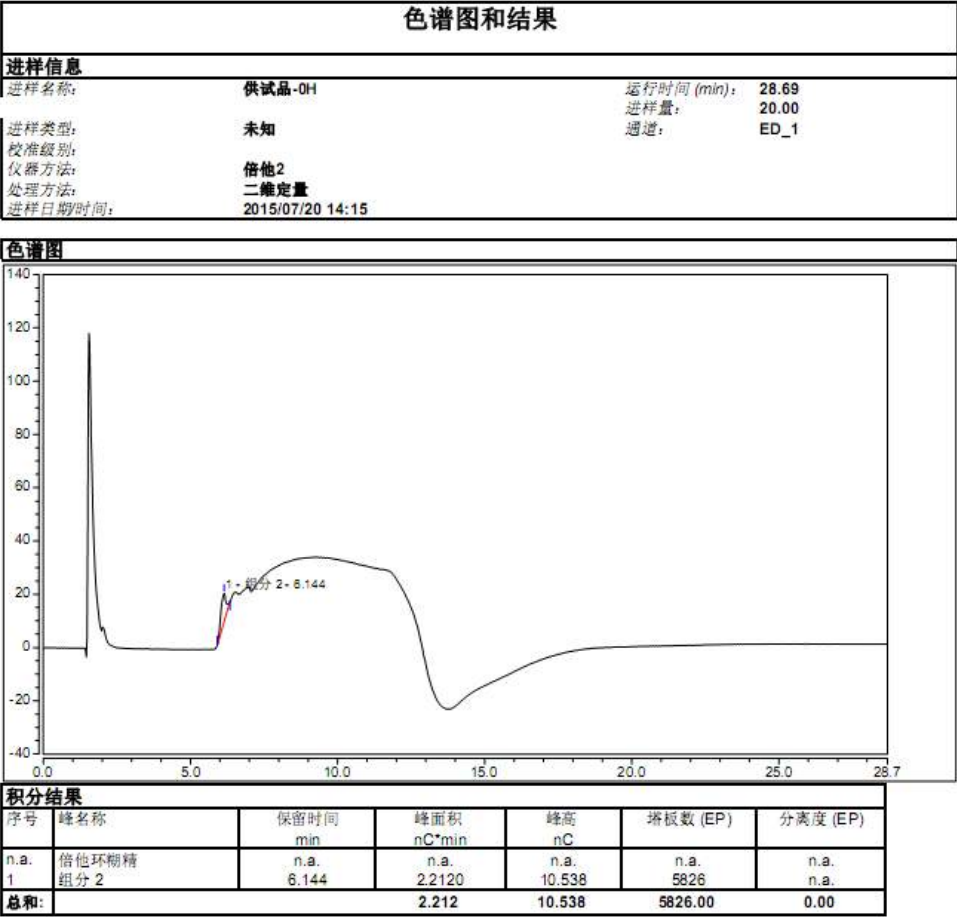
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-20 Validation of analysis procedure for Betadex-Solution stability-Sample solution
0h

仪器:ICS-5000+ 序列:溶液稳定性

页码 1 / 12

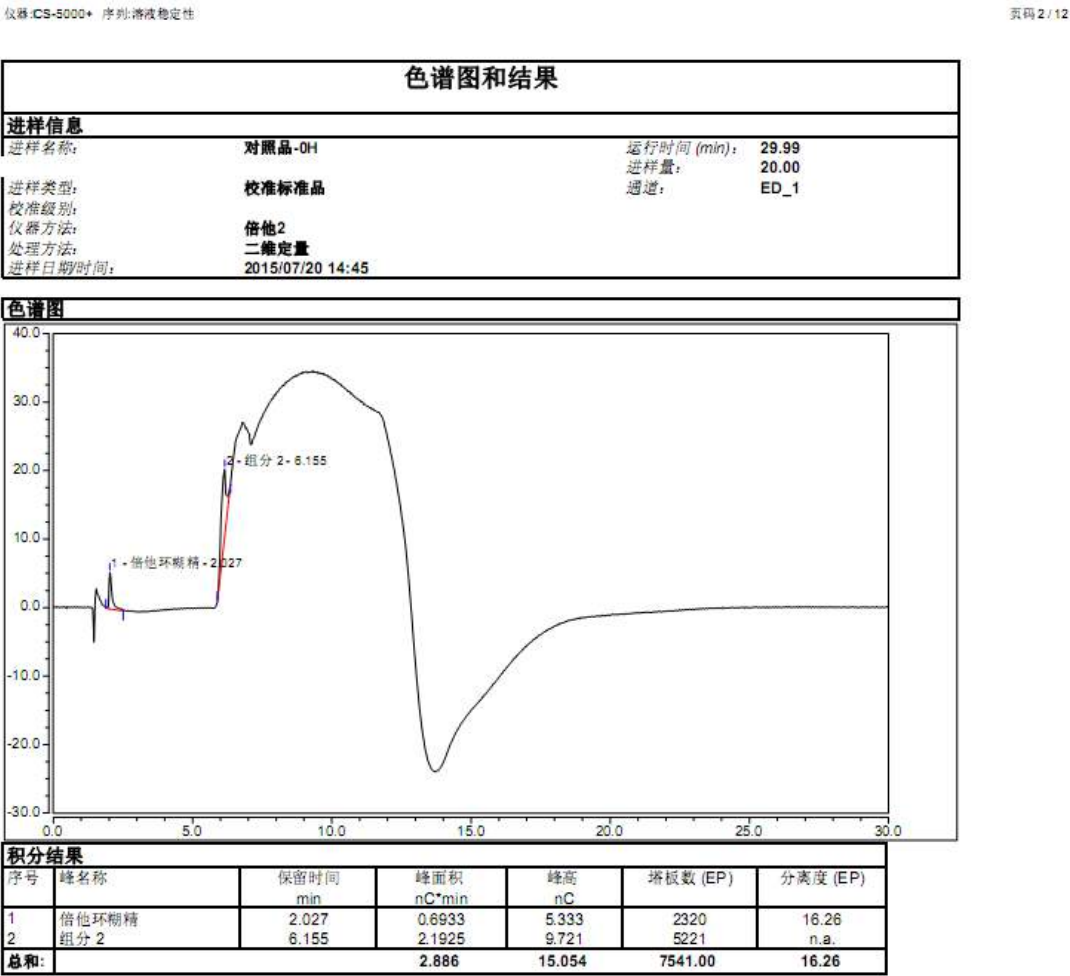


附图10.4.8-194 SBECD中倍他环糊精的测定方法验证图（样品溶液稳定性-0H）

Default:积分

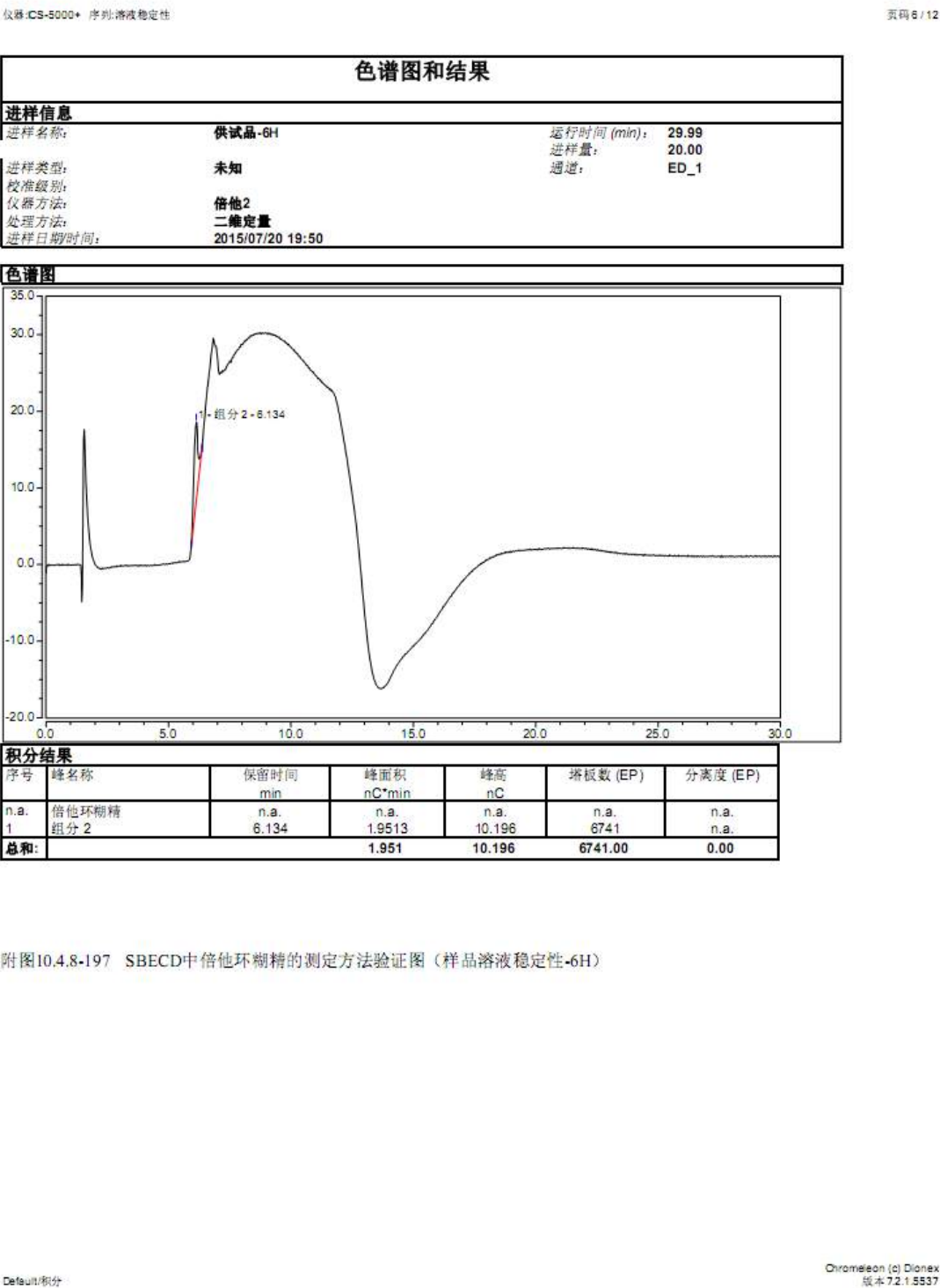
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-21 Validation of analysis procedure for Betadex-Solution stability-Reference solution 0h



附图10.4.8-188 SBECD中倍他环糊精的测定方法验证图（对照品溶液稳定性-0H）

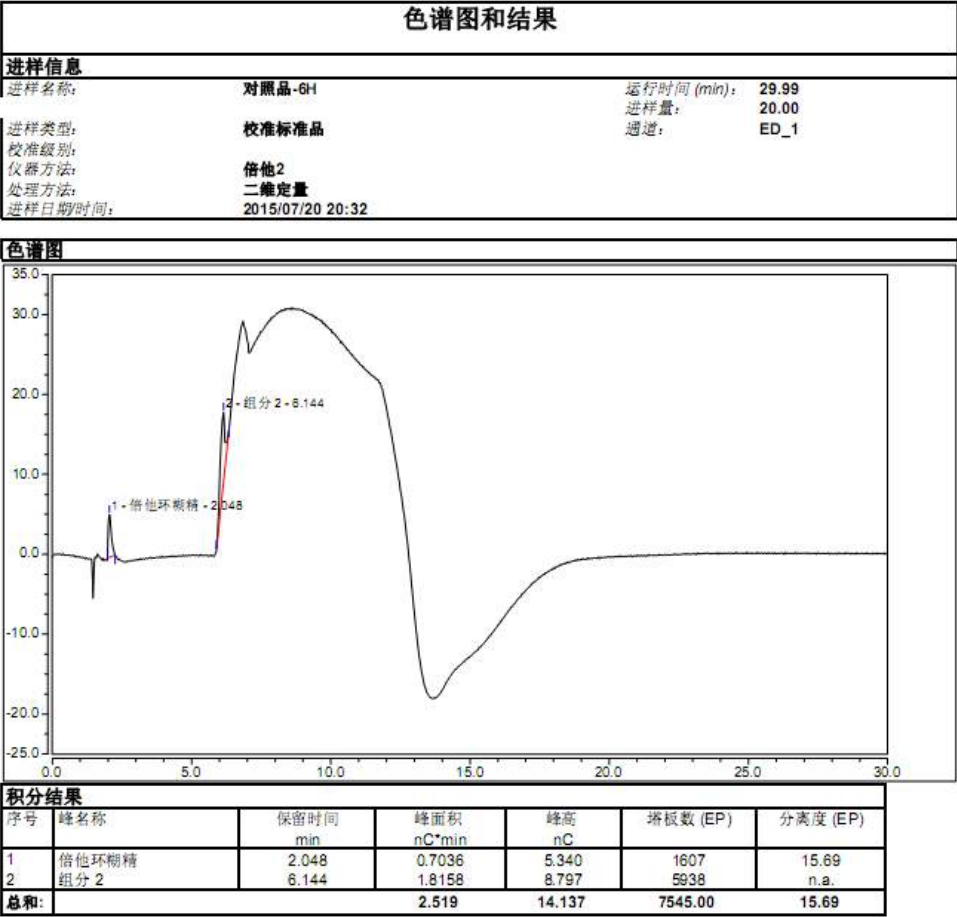
Annex 3-1-22 Validation of analysis procedure for Betadex-Solution stability-Sample solution 6h



Annex 3-1-23 Validation of analysis procedure for Betadex-Solution stability-Reference solution 6h

仪器:ICS-5000+ 序列:溶液稳定性

页码 7 / 12



附图10.4.8-190 SBECD中倍他环糊精的测定方法验证图（对照品溶液稳定性-6H）

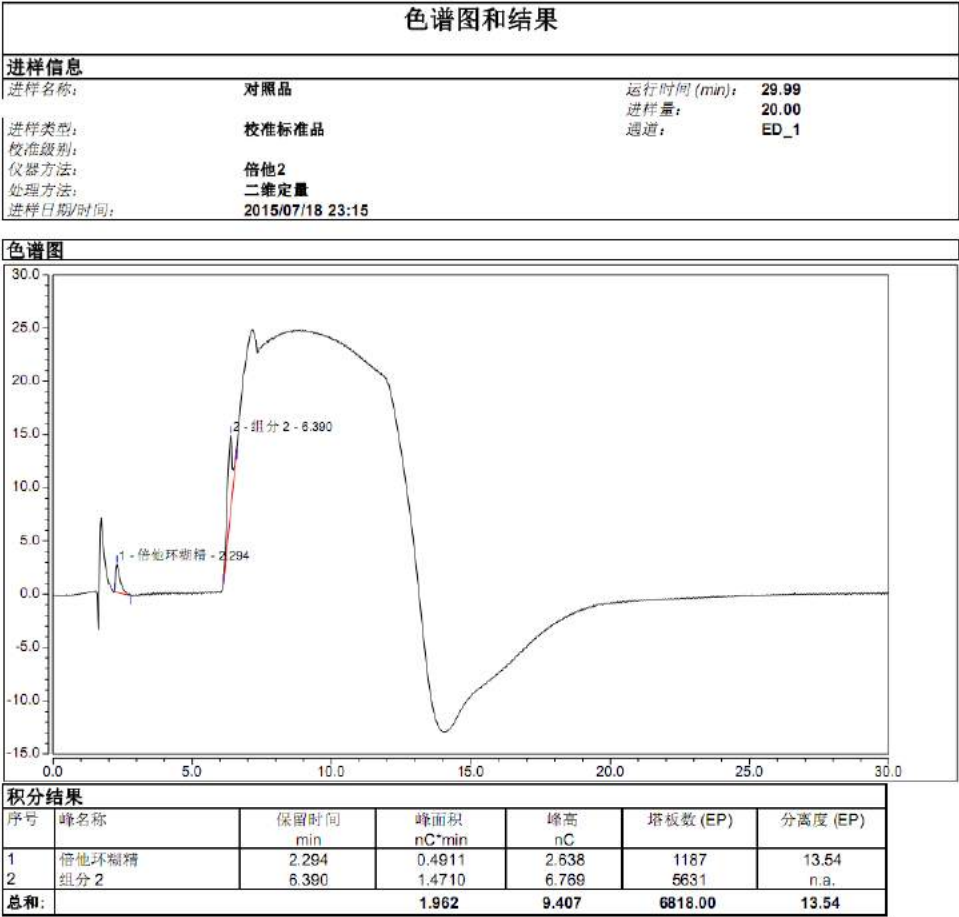
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-24 Validation of analysis procedure for Betadex-Robustness-Condition
2-Reference solution

仪器:ICS-5000+ 序列:0.9ml

页码 1/2

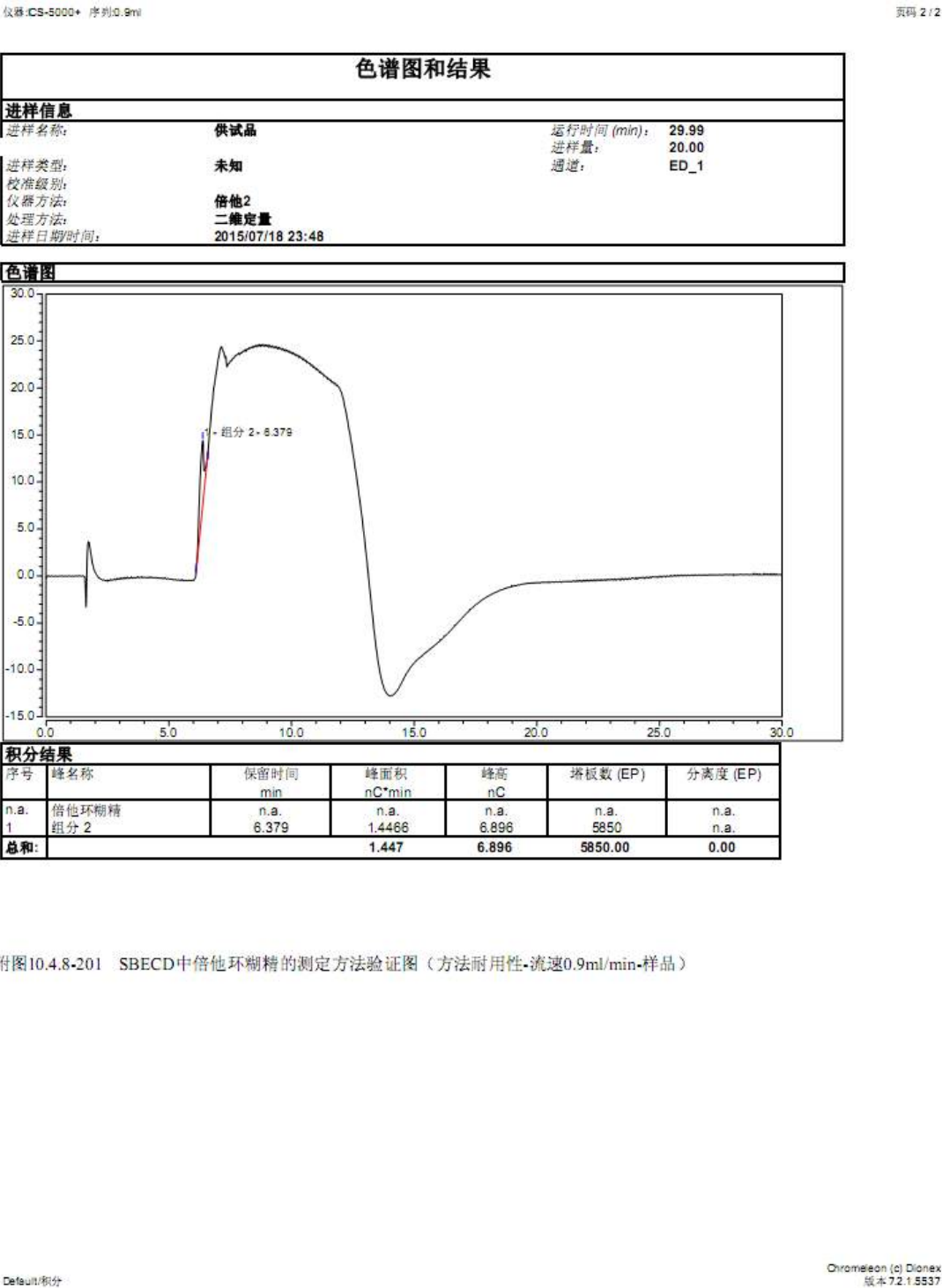


附图10.4.8-200 SBECD中倍他环糊精的测定方法验证图（方法耐用性-流速0.9ml/min-对照）

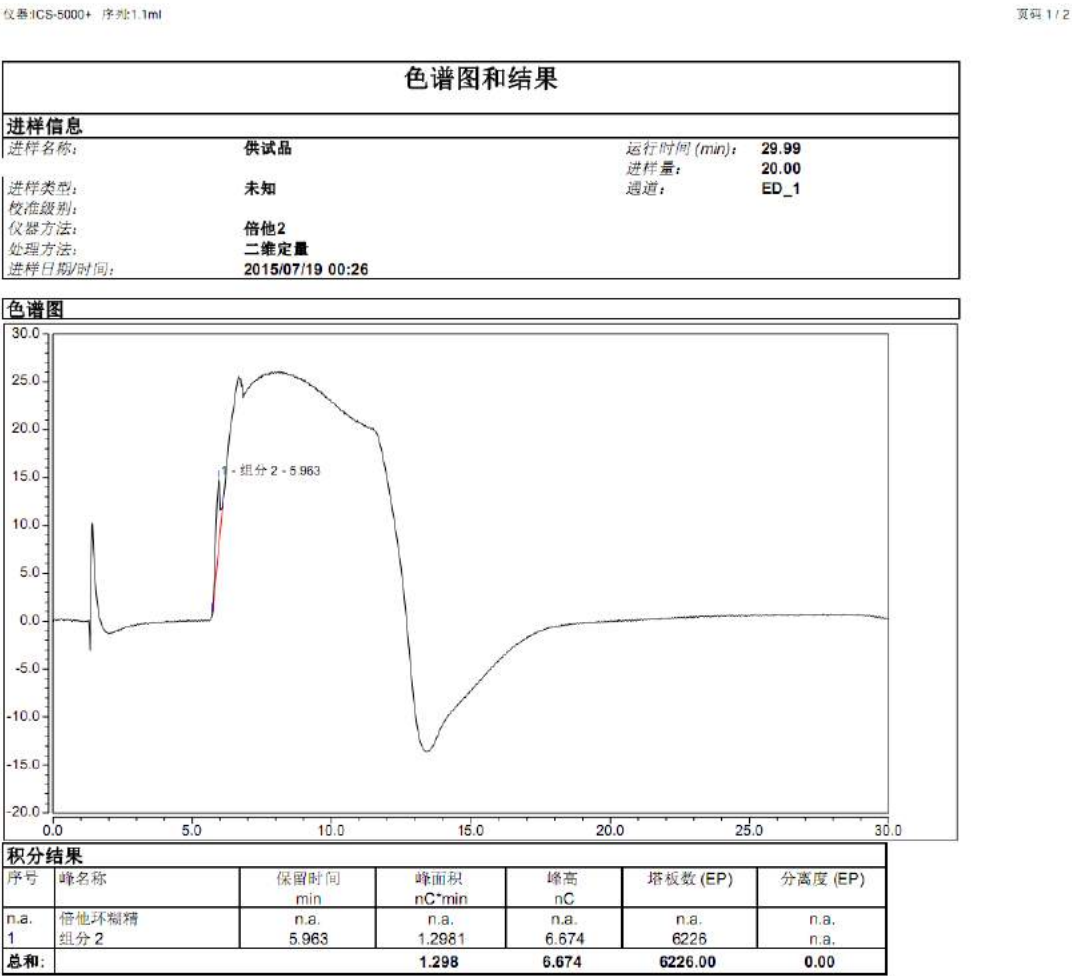
Default部分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-25 Validation of analysis procedure for Betadex-Robustness-Condition 2-Sample solution

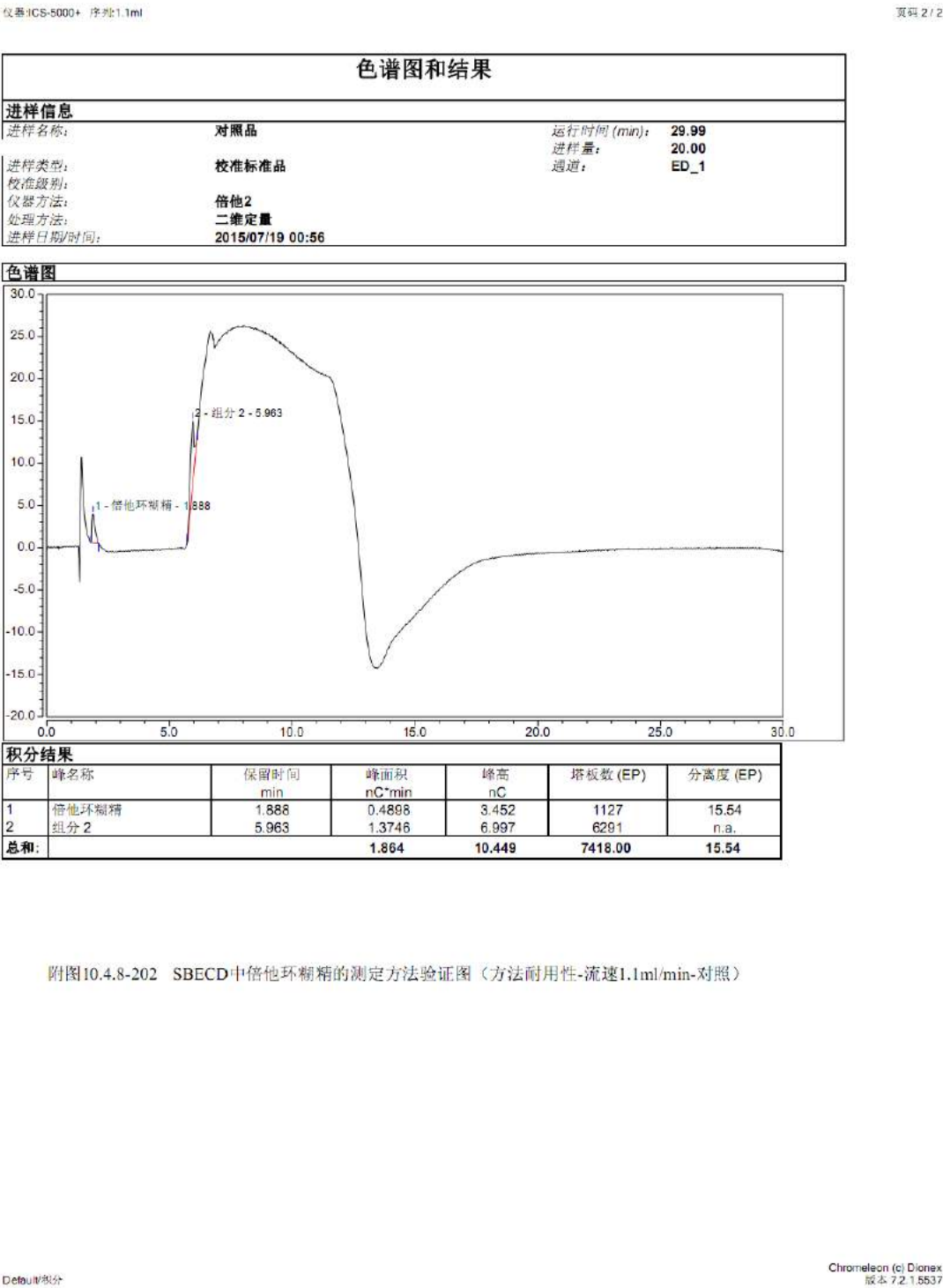


Annex 3-1-26 Validation of analysis procedure for Betadex-Robustness-Condition
3-Reference solution



附图10.4.8-203 SBECD中倍他环糊精的测定方法验证图（方法耐用性-流速1.1ml/min-样品）

Annex 3-1-27 Validation of analysis procedure for Betadex-Robustness-Condition 3-Sample solution



Annex 3-1-28 Validation of analysis procedure for Betadex-Robustness-Condition

4-Reference solution

仪器:ICS-5000+ 序列:48℃

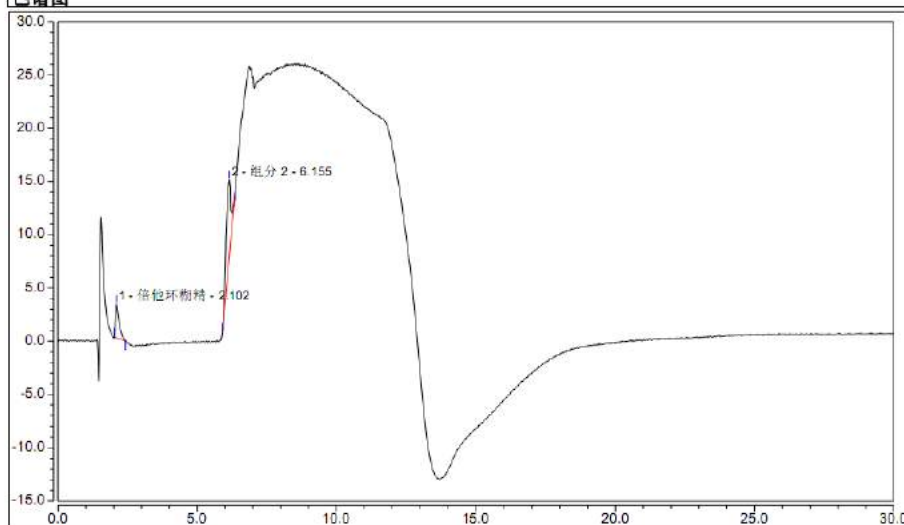
頁碼 1 / 2

色谱图和结果

进样信息	
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进样名称:	对照品	运行时间 (min):	29.99
进样类型:	校准标准品	进样量:	20.00
校准级别:		通道:	ED_1
仪器方法:	倍他2		
处理方法:	二维定量		
进样日期/时间:	2015/07/19 01:27		

色谱图



积分结果

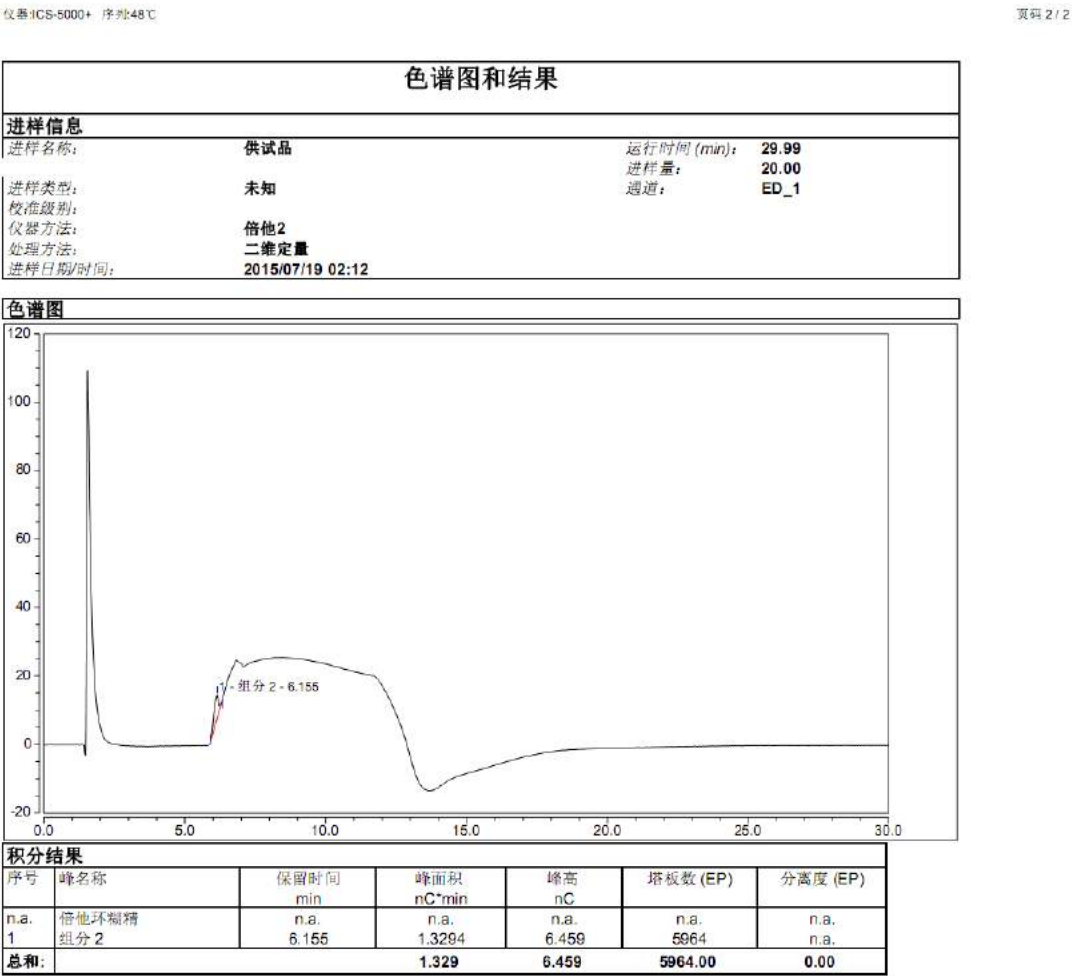
序号	峰名称	保留时间 min	峰面积 nC*min	峰高 nC	塔板数 (EP)	分离度 (EP)
1	倍他环糊精	2.102	0.4902	3.142	1212	14.50
2	组分 2	6.155	1.4814	7.039	5955	n.a.
总和:			1.972	10.182	7167.00	14.50

附图10.4.8-204 SBECD中倍他环糊精的测定方法验证图(方法耐用性-柱温48℃-对照)

Default 积分

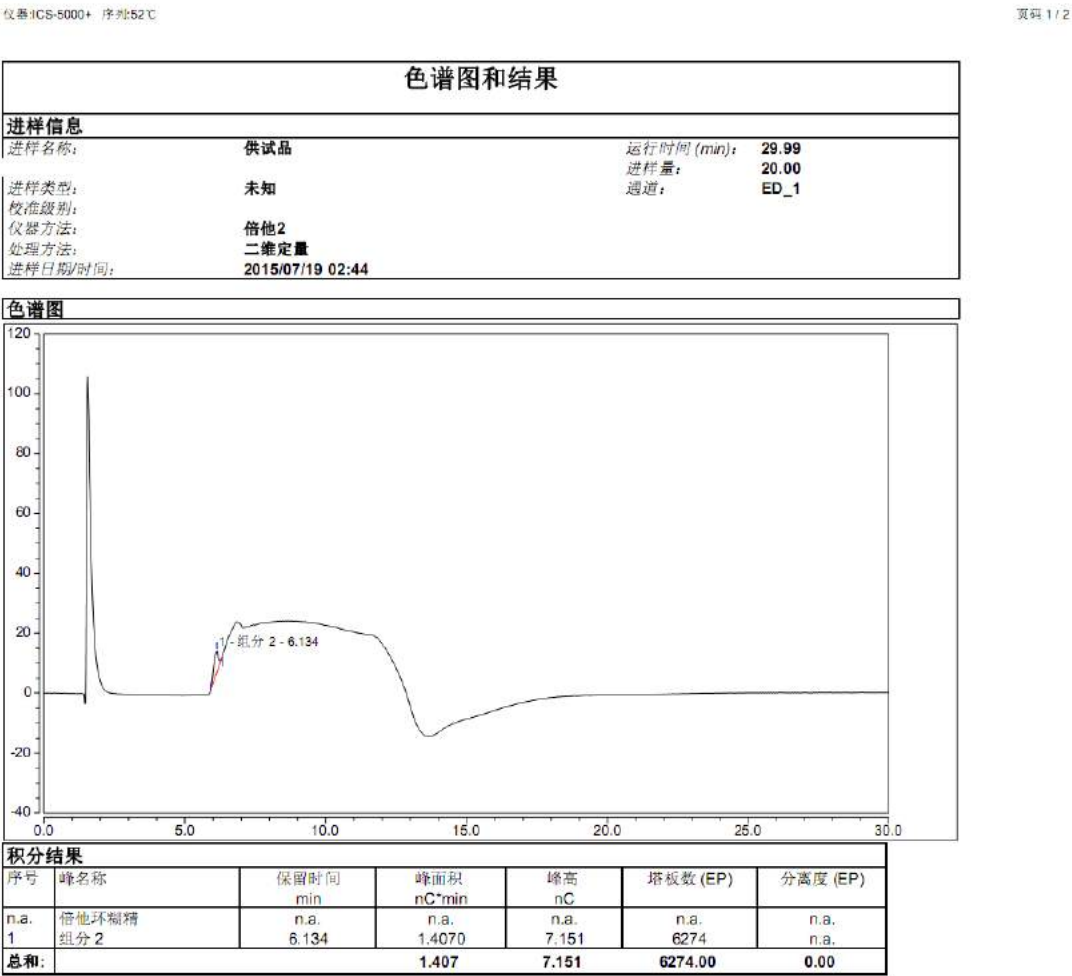
Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-29 Validation of analysis procedure for Betadex-Robustness-Condition 4-Sample solution



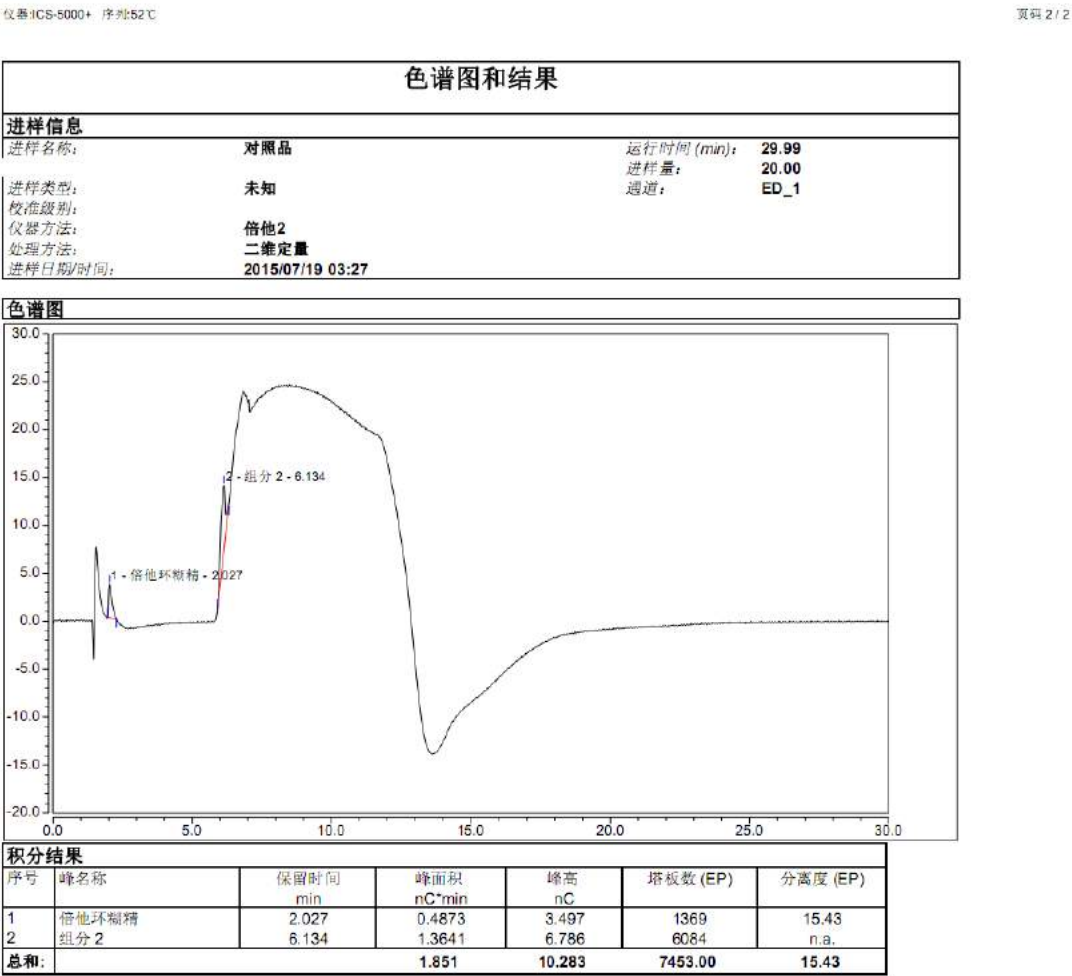
附图10.4.8-205 SBECD中倍他环糊精的测定方法验证图（方法耐用性-柱温48℃-样品）

Annex 3-1-30 Validation of analysis procedure for Betadex-Robustness-Condition
5-Reference solution



附图10.4.8-207 SBECD中倍他环糊精的测定方法验证图（方法耐用性-柱温52℃-对照）

Annex 3-1-31 Validation of analysis procedure for Betadex-Robustness-Condition 5-Sample solution

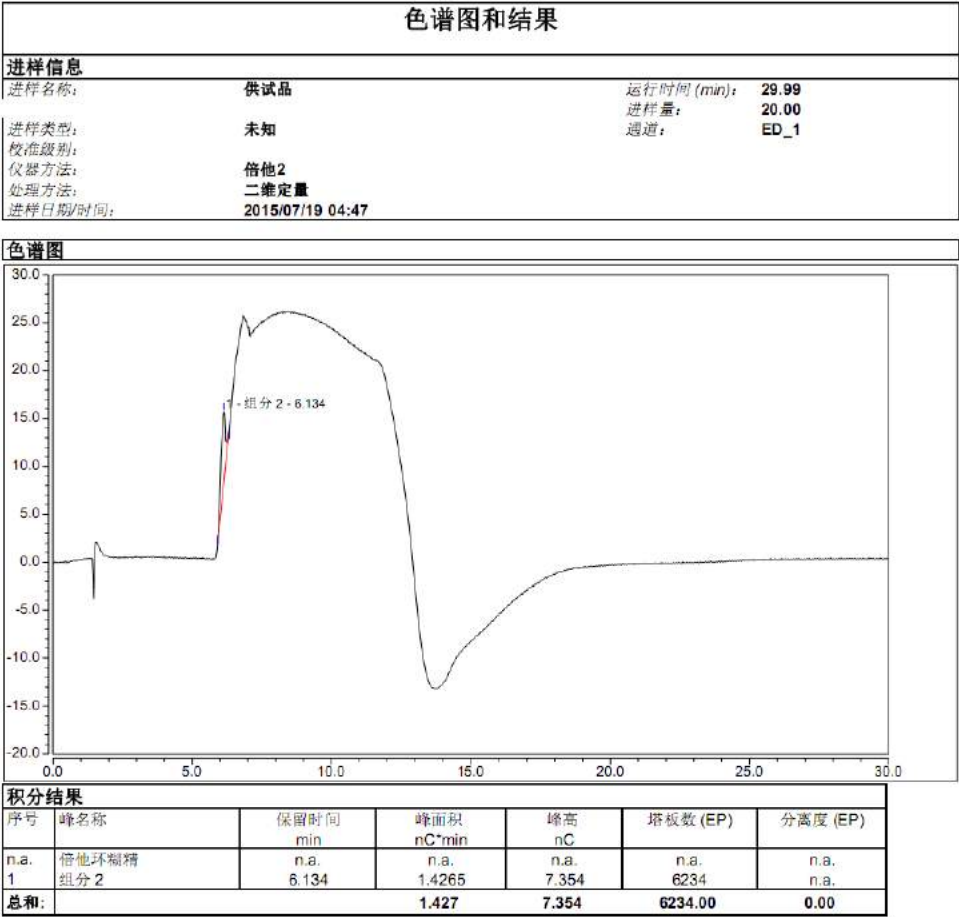


附图10.4.8-206 SBECD中倍他环糊精的测定方法验证图（方法耐用性-柱温52℃-对照）

Annex 3-1-32 Validation of analysis procedure for Betadex-Robustness-Condition
6-Reference solution

仪器:ICS-5000+ 序列:谱剂A24.5mmol

页码 1/2

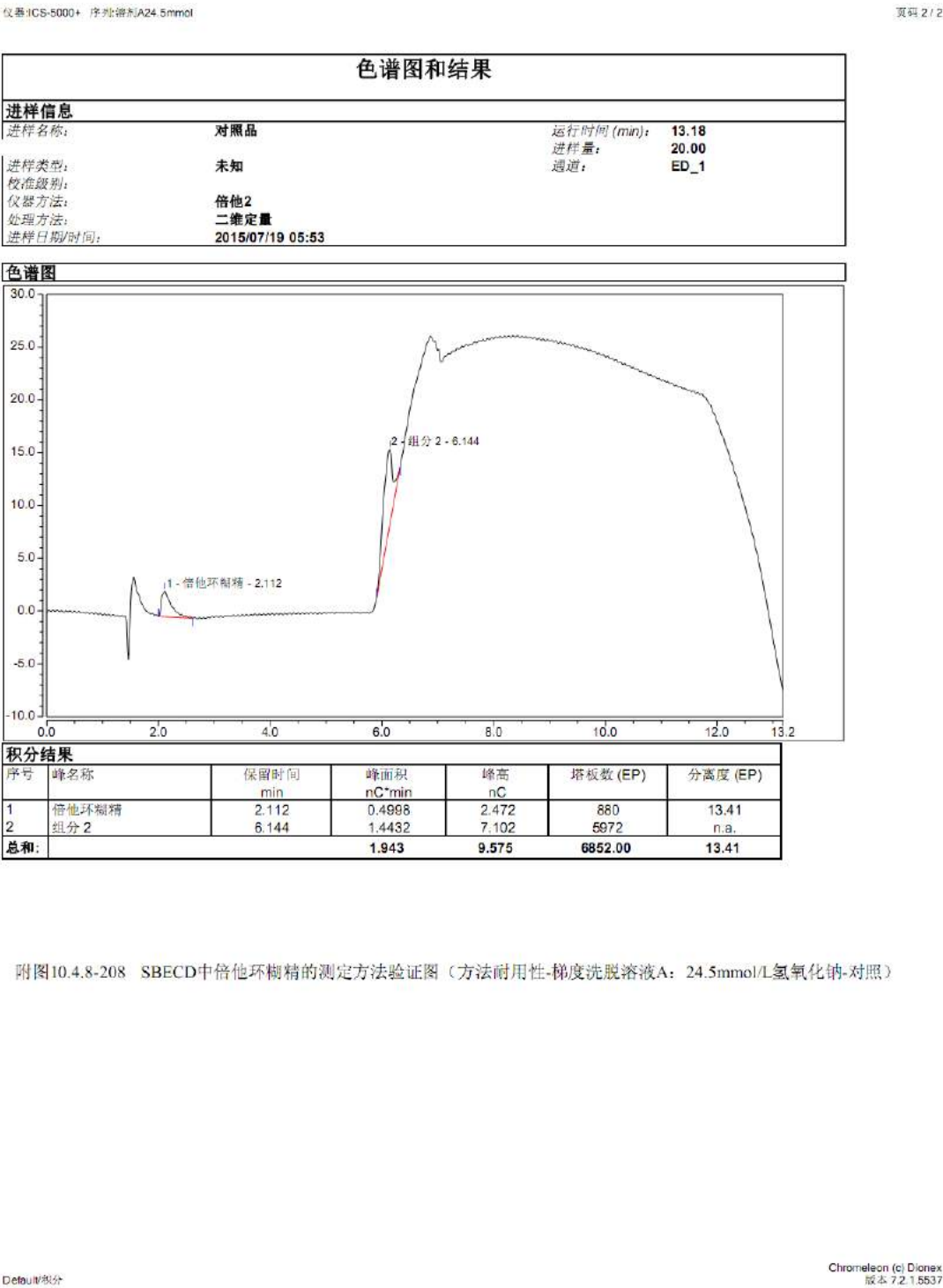


附图10.4.8-209 SBECD中倍他环糊精的测定方法验证图（方法耐用性-梯度洗脱溶液A：24.5mmol/L氢氧化钠-样品）

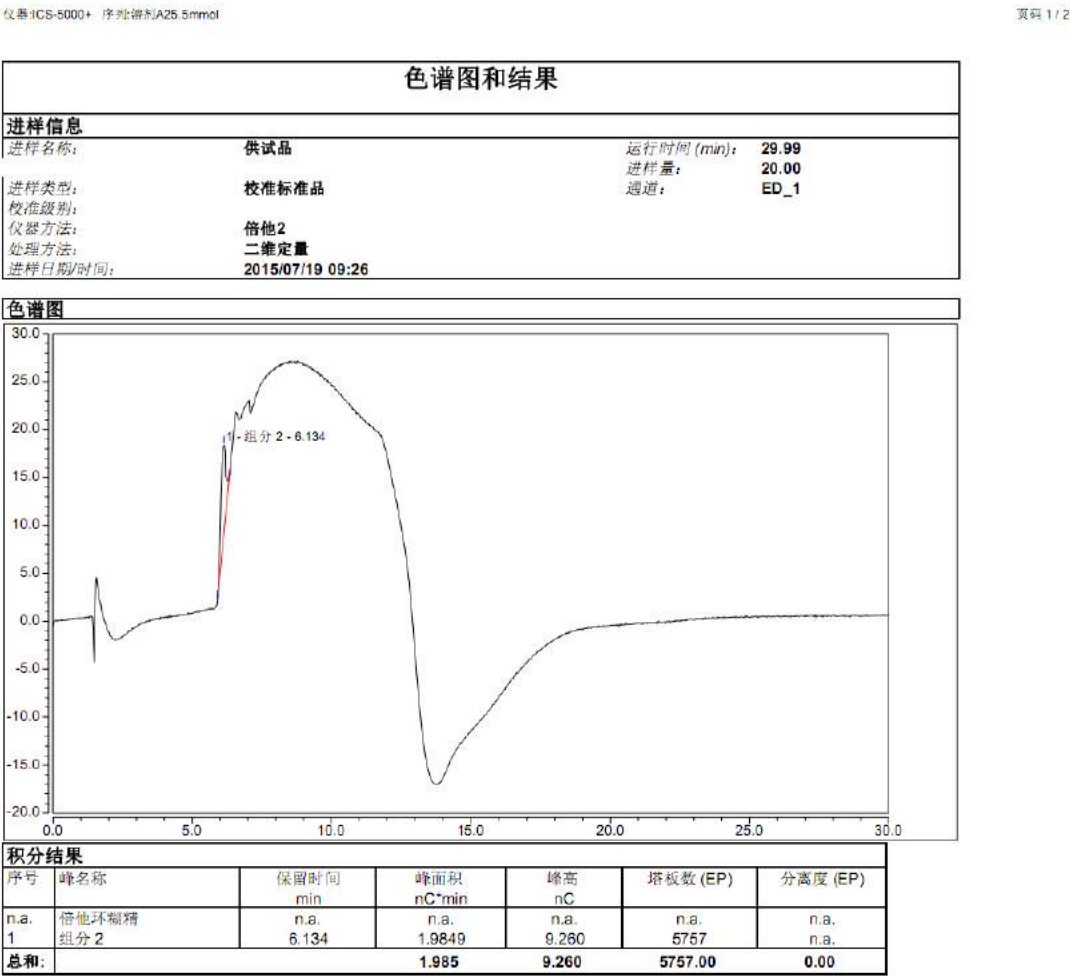
Default部分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-1-33 Validation of analysis procedure for Betadex-Robustness-Condition 6-Sample solution

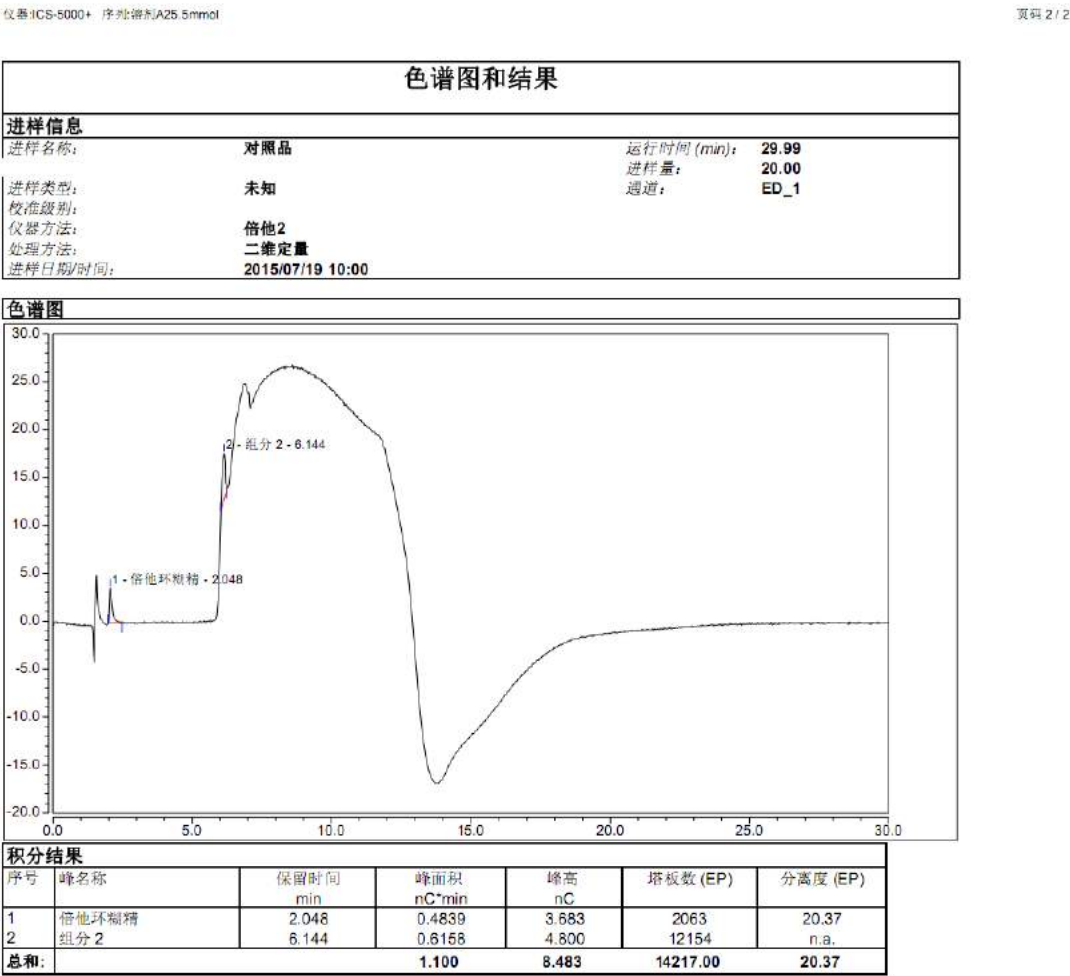


Annex 3-1-34 Validation of analysis procedure for Betadex-Robustness-Condition
7-Reference solution



附图10. 4. 8 -SEC中倍他环糊精的测定方法验证图（方法耐用性-梯度洗脱溶液A 25.5mmol/L氢氧化钠-柱

Annex 3-1-35 Validation of analysis procedure for Betadex-Robustness-Condition 7-Sample solution



附图10. 4. 8 -SEED中倍他环糊精的测定方法验证图（方法耐用性-梯度洗脱溶液A 25.5mmol/L氢氧化钠-X）

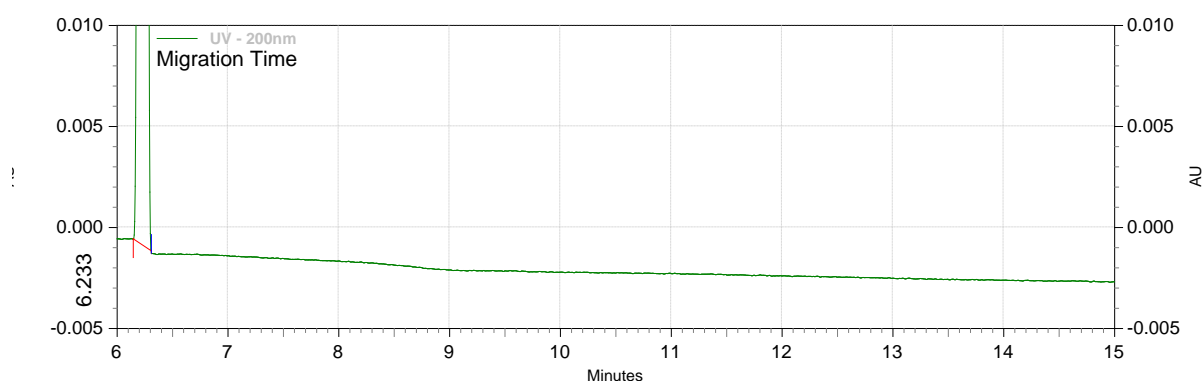
Annex 3-2-1 Validation of analysis procedure for Average degree of substitution-Specificity-Blank

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\specificity experiment\150620 blank experiment.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/20/2015 3:18:07 PM

Printed: 7/2/2015 1:32:21 PM



UV - 200nm

Results

Name	Migration Time	Area	Theoretical plates (USP)	Resolution (USP)
	6.233	323723		

Totals		323723		
--------	--	--------	--	--

附图 10.4.9-14 SBECD 中平均取代度的测定方法验证图（专属性-空白溶剂）

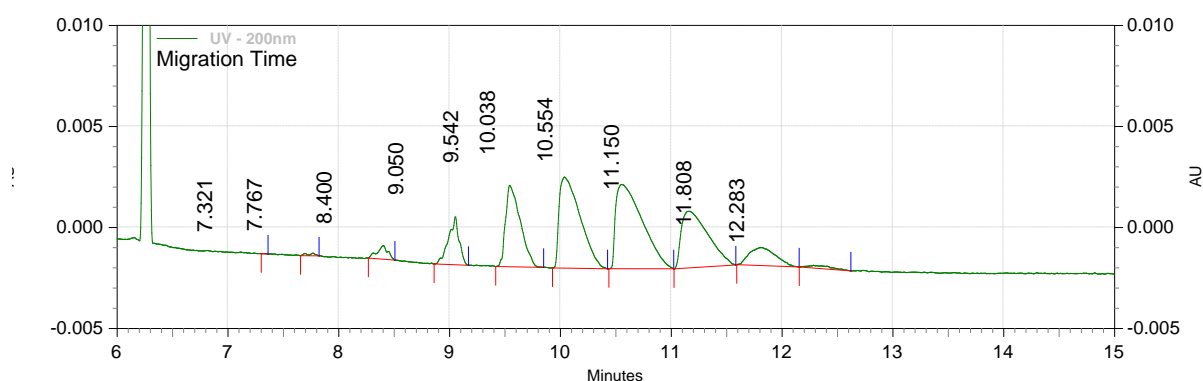
Annex 3-2-2 Validation of analysis procedure for Average degree of substitution-Specificity-Reference solution

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\specificity experiment\150620 reference substance.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/20/2015 1:53:11 PM

Printed: 7/2/2015 1:33:17 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Theoretical plates (USP)	Resolution (USP)
1	0.694	0.025	226233	0.00
2	0.736	0.351	46968	4.35
3	0.796	2.182	55197	4.42
4	0.857	7.100	43712	4.11
5	0.904	16.012	21474	2.27
6	0.951	24.275	12565	1.60
7	1.000	27.010	8462	1.26
8	1.056	17.220	9167	1.29
9	1.119	5.122	10547	1.42
10	1.164	0.701	16832	1.13

Totals		100.000		
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附图 10.4.9-15 SBECD 中平均取代度的测定方法验证图（专属性-对照品）

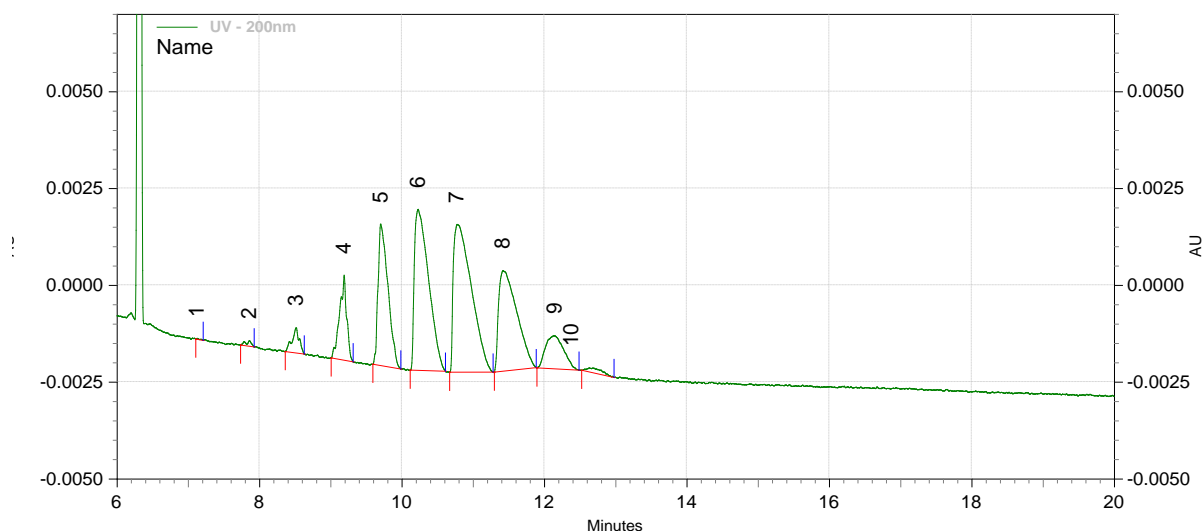
Annex 3-2-3 Validation of analysis procedure for Average degree of substitution-System suitability-Reference solution1

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\system suitability test and the test instrument accuracy\150620 reference substance001.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/20/2015 5:57:32 PM

Printed: 7/2/2015 2:19:01 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.66	0.043	0.00
2	0.73	0.417	10.15
3	0.79	2.291	5.56
4	0.85	7.076	4.17
5	0.90	15.635	2.26
6	0.95	24.291	1.60
7	1.00	26.851	1.26
8	1.06	17.371	1.32
9	1.13	5.376	1.50
10	1.17	0.649	1.11

Totals		100.000	
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附图 10. 4. 9-16 SBECD 中平均取代度的测定方法验证图（系统适应性-1）

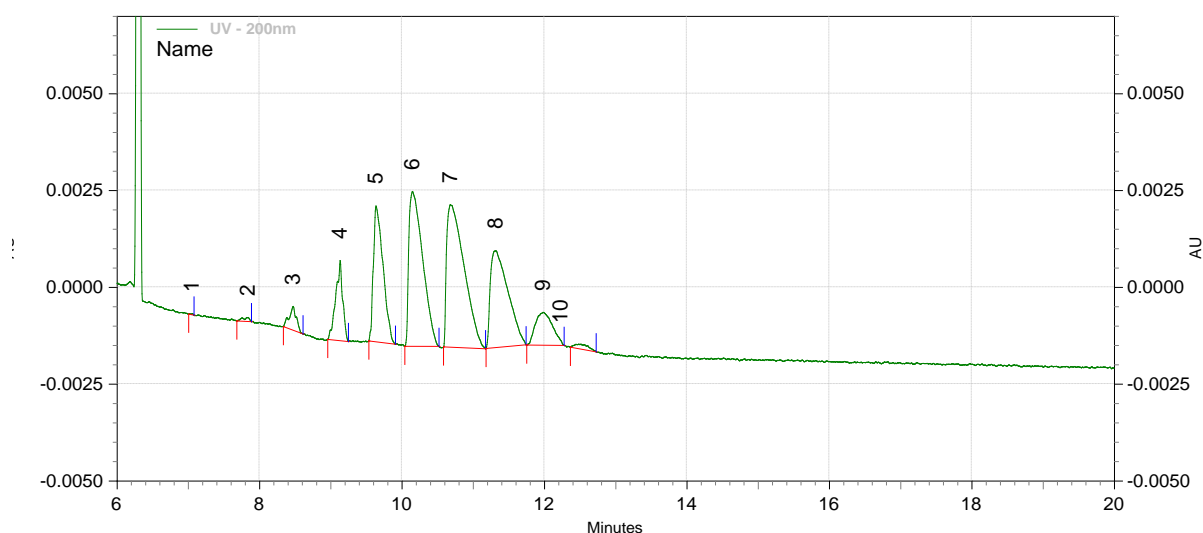
Annex 3-2-4 Validation of analysis procedure for Average degree of substitution-System suitability-Reference solution4

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\system suitability test and the test instrument accuracy\150620 reference substance004.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/20/2015 8:04:55 PM

Printed: 7/2/2015 2:30:09 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.66	0.042	0.00
2	0.73	0.411	10.10
3	0.79	2.317	4.93
4	0.85	7.012	4.11
5	0.90	15.642	2.30
6	0.95	24.297	1.63
7	1.00	26.853	1.27
8	1.06	17.365	1.34
9	1.12	5.431	1.42
10	1.17	0.631	1.24

Totals		100.000	
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附图 10. 4. 9-19 SBECD 中平均取代度的测定方法验证图（系统适应性-4）

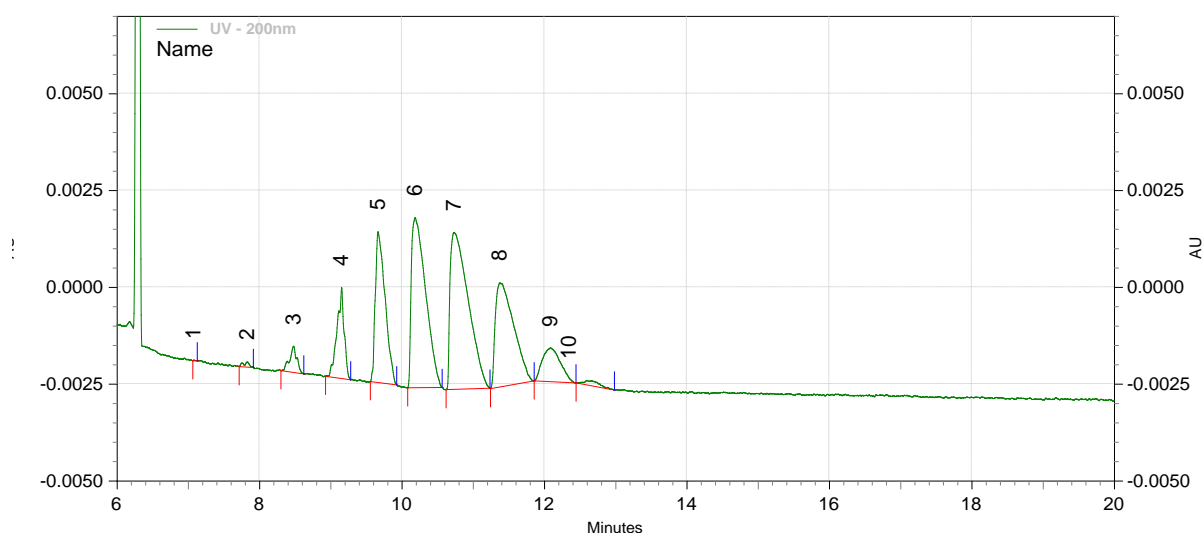
Annex 3-2-5 Validation of analysis procedure for Average degree of substitution-Concentration range- Concentration 1

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\The sample concentration range test\150621 test samples 8mg.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/21/2015 10:38:18 AM

Printed: 7/2/2015 5:02:40 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.66	0.014	0.00
2	0.73	0.396	0.00
3	0.79	2.290	5.40
4	0.85	7.245	4.15
5	0.90	15.616	2.27
6	0.95	24.406	1.60
7	1.00	27.077	1.26
8	1.06	17.149	1.30
9	1.13	5.179	1.45
10	1.17	0.628	1.23

Totals		100.000	
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附图 10. 4. 9-22 SBECD 中平均取代度的测定方法验证图（样品浓度范围-8mg）

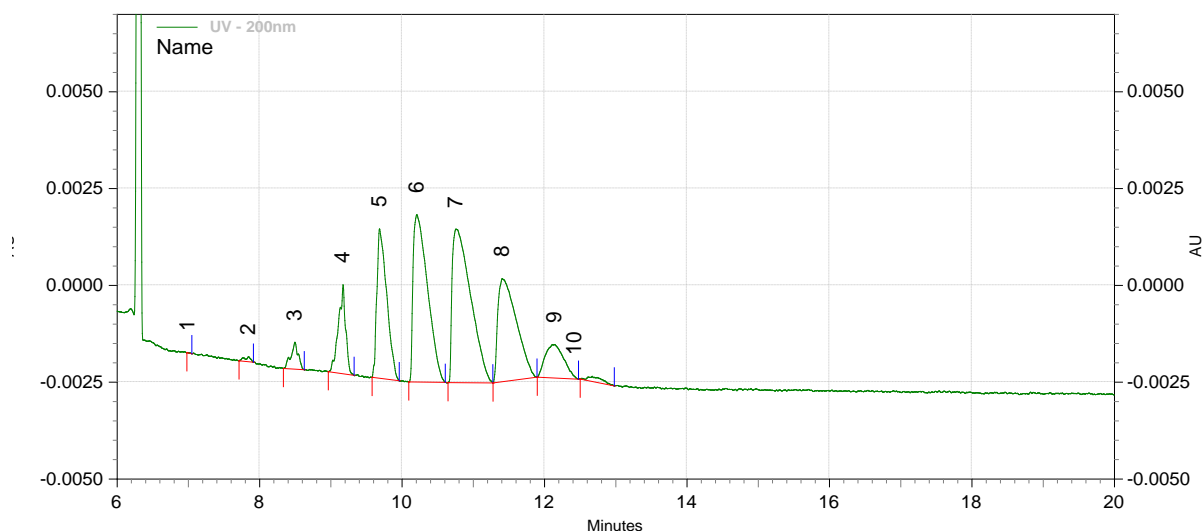
Annex 3-2-6 Validation of analysis procedure for Average degree of substitution-Concentration range- Concentration 3

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\The sample concentration range test\150621 test samples 12mg.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/21/2015 12:06:42 PM

Printed: 7/2/2015 5:04:24 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.65	0.013	0.00
2	0.73	0.398	0.00
3	0.79	2.359	3.90
4	0.85	7.192	4.02
5	0.90	15.620	2.25
6	0.95	24.285	1.59
7	1.00	27.018	1.24
8	1.06	17.204	1.30
9	1.13	5.282	1.47
10	1.18	0.628	1.22

Totals		100.000	
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附图 10. 4. 9-24 SBECD 中平均取代度的测定方法验证图（样品浓度范围-12mg）

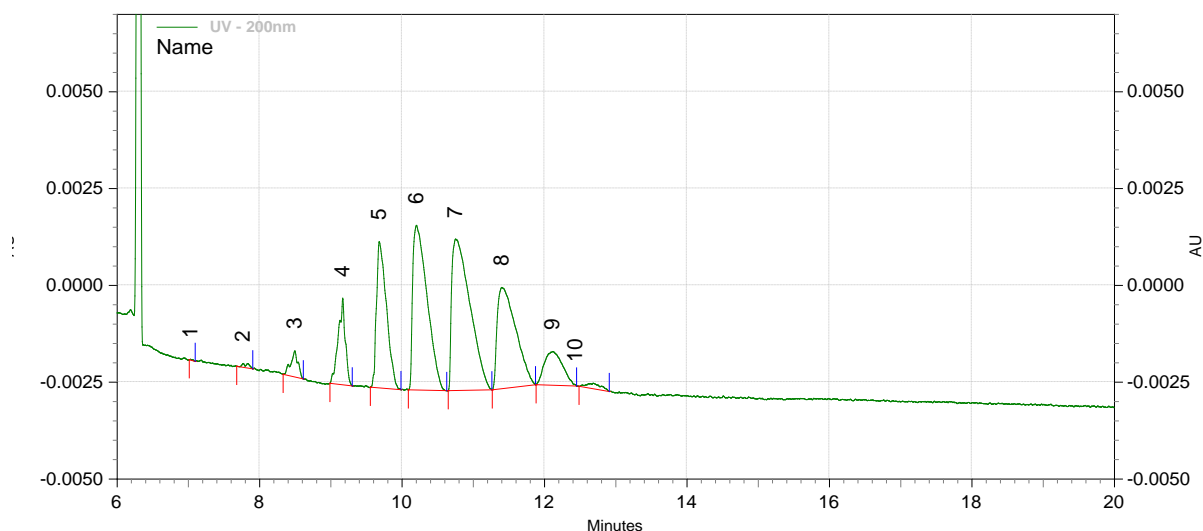
Annex 3-2-7 Validation of analysis procedure for Average degree of substitution-Robustness-Normal condition

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\Durability test\150621 test samples

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/21/2015 12:54:17 PM

Printed: 7/2/2015 5:09:00 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.65	0.038	0.00
2	0.72	0.367	6.30
3	0.79	2.321	4.62
4	0.85	7.008	4.11
5	0.90	15.762	2.29
6	0.95	24.270	1.60
7	1.00	26.921	1.25
8	1.06	17.284	1.31
9	1.13	5.369	1.44
10	1.18	0.660	0.00

Totals		100.000	
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附图 10.4.9-25 SBECD 中平均取代度的测定方法验证图（耐用性-正常条件）

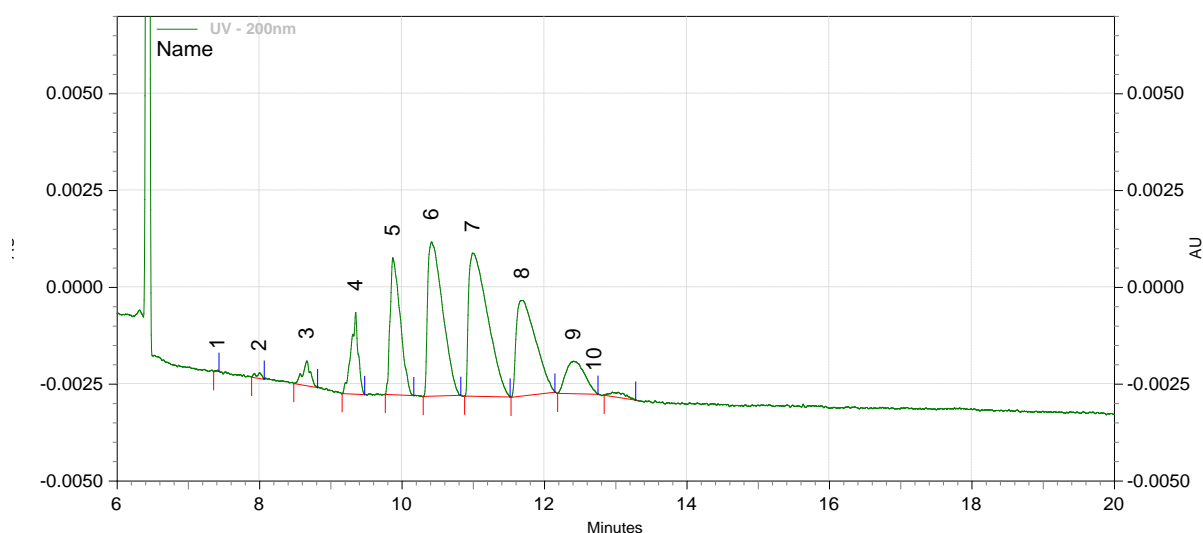
Annex 3-2-8 Validation of analysis procedure for Average degree of substitution-Robustness-Column temperature 23°C

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\Durability test\150621 test samples T 23.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/21/2015 3:43:35 PM

Printed: 7/2/2015 5:16:12 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.67	0.046	0.00
2	0.73	0.439	0.00
3	0.79	2.431	5.31
4	0.85	6.886	4.19
5	0.90	15.321	2.27
6	0.95	24.320	1.61
7	1.00	27.094	1.28
8	1.06	17.373	1.34
9	1.13	5.443	1.40
10	1.18	0.647	1.24

Totals		100.000	
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附图 10.4.9-28 SBECD 中平均取代度的测定方法验证图（耐用性-柱温 23°C）

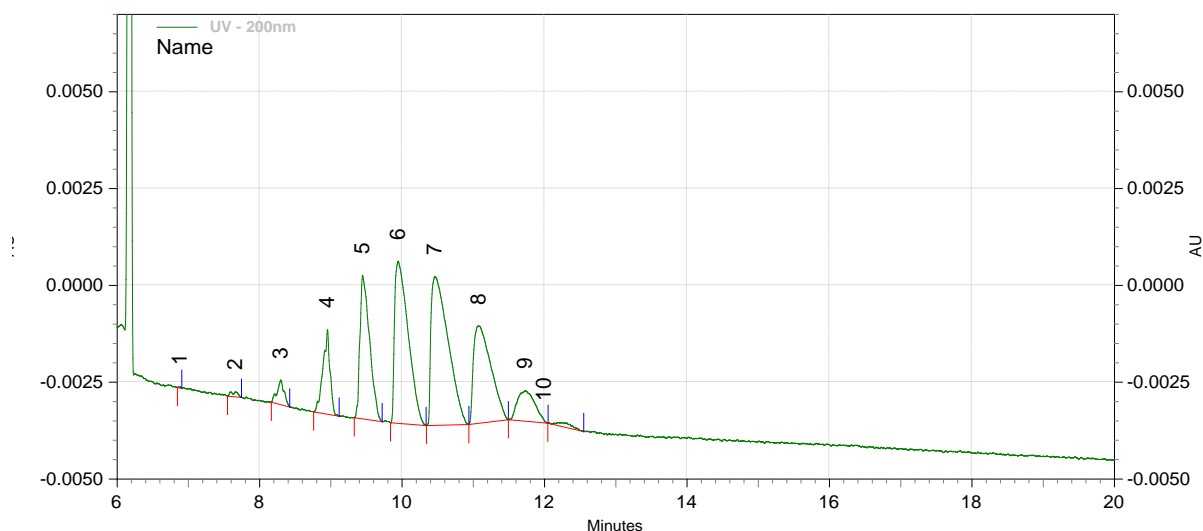
Annex 3-2-9 Validation of analysis procedure for Average degree of substitution-Robustness-Column temperature 27°C

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\Durability test\150621 test samples T 27.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/21/2015 5:14:25 PM

Printed: 7/2/2015 5:16:48 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.66	0.039	0.00
2	0.73	0.493	6.86
3	0.79	2.272	4.01
4	0.86	7.357	3.90
5	0.90	16.114	2.20
6	0.95	24.431	1.61
7	1.00	26.793	1.24
8	1.06	16.896	1.32
9	1.12	5.018	1.41
10	1.17	0.586	1.24

Totals		100.000	
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附图 10.4.9-29 SBECD 中平均取代度的测定方法验证图（耐用性-柱温 27°C）

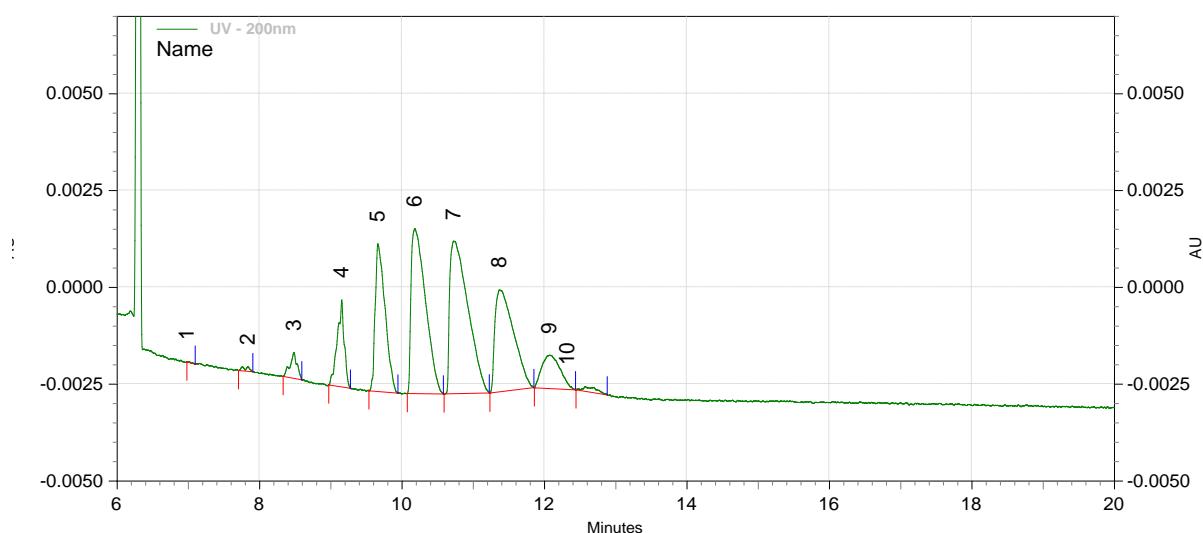
Annex 3-2-10 Validation of analysis procedure for Average degree of substitution-Robustness-pH of buffer solution 7.7

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\Durability test\150621 test samples PH7.7.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/21/2015 1:43:20 PM

Printed: 7/2/2015 5:15:29 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.65	0.038	0.00
2	0.73	0.380	0.00
3	0.79	2.251	4.22
4	0.85	6.895	4.10
5	0.90	15.764	2.22
6	0.95	24.345	1.58
7	1.00	27.010	1.25
8	1.06	17.372	1.29
9	1.13	5.343	1.41
10	1.17	0.600	1.17

Totals		100.000	
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附图 10. 4. 9-27 SBECD 中平均取代度的测定方法验证图（耐用性-pH7. 7）

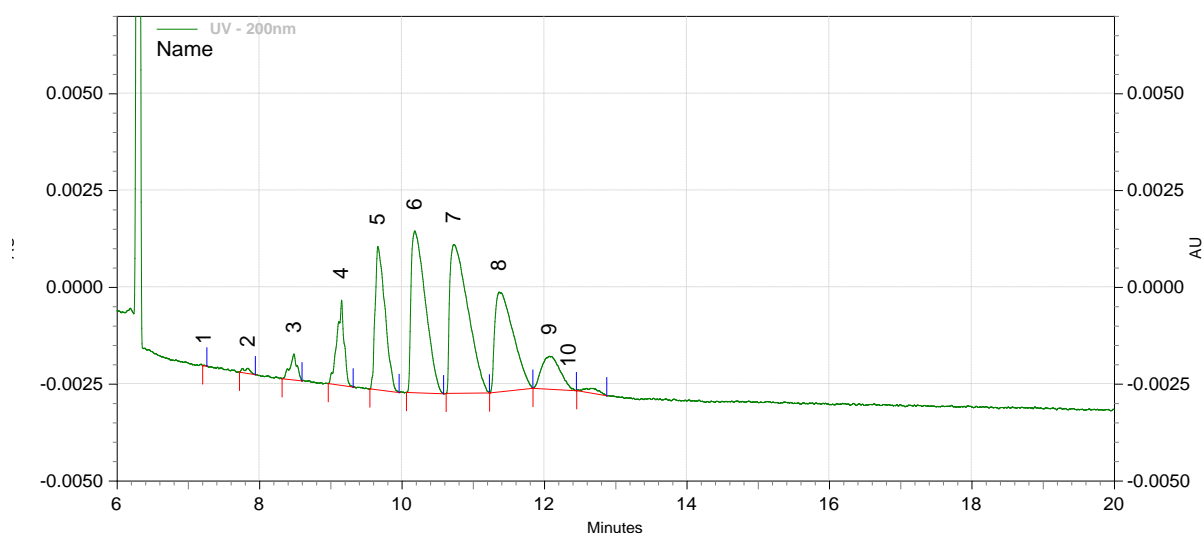
Annex 3-2-11 Validation of analysis procedure for Average degree of substitution-Robustness-pH of buffer solution 7.3

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\Durability test\150621 test samples PH7.3.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/21/2015 2:31:21 PM

Printed: 7/2/2015 5:14:36 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.67	0.022	0.00
2	0.73	0.478	5.30
3	0.79	2.316	4.00
4	0.85	7.097	4.05
5	0.90	15.632	2.26
6	0.95	24.288	1.60
7	1.00	26.816	1.25
8	1.06	17.300	1.28
9	1.13	5.437	1.42
10	1.17	0.615	0.00

Totals		100.000	
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附图 10. 4. 9-26 SBECD 中平均取代度的测定方法验证图（耐用性-pH7.3）

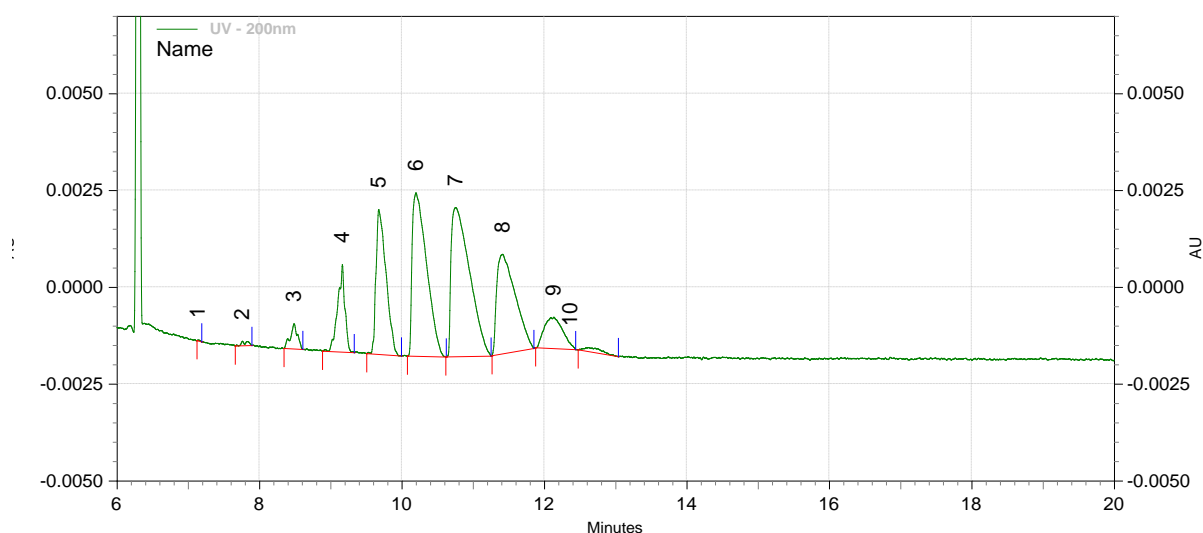
Annex 3-2-12 Validation of analysis procedure for Average degree of substitution-Repeatability- Solution 2

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\method repeatability test\150622 test samples002.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/22/2015 9:41:20 AM

Printed: 7/2/2015 3:16:44 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.66	0.042	0.00
2	0.72	0.398	8.55
3	0.79	2.230	6.51
4	0.85	7.270	4.20
5	0.90	15.802	2.22
6	0.95	24.412	1.58
7	1.00	27.030	1.26
8	1.06	17.070	1.30
9	1.13	5.079	1.42
10	1.17	0.668	1.02

Totals		100.000	
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附图 10. 4. 9-31 SBECD 中平均取代度的测定方法验证图（方法重复性-2）

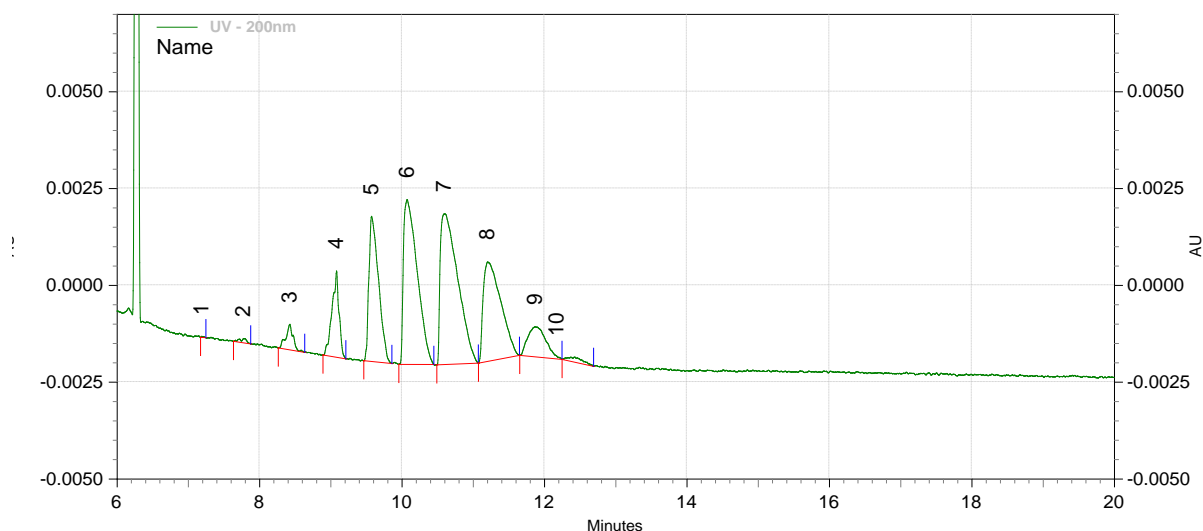
Annex 3-2-13 Validation of analysis procedure for Average degree of substitution-Repeatability- Solution 5

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\method repeatability test\150622 test samples005.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/22/2015 12:46:11 PM

Printed: 7/2/2015 3:22:47 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.68	0.040	0.00
2	0.73	0.415	7.47
3	0.80	2.370	5.04
4	0.86	7.218	4.04
5	0.90	15.870	2.20
6	0.95	24.396	1.59
7	1.00	27.043	1.25
8	1.06	16.954	1.28
9	1.12	5.023	1.40
10	1.17	0.670	0.00

Totals		100.000	
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附图 10. 4. 9-34 SBECD 中平均取代度的测定方法验证图（方法重复性-5）

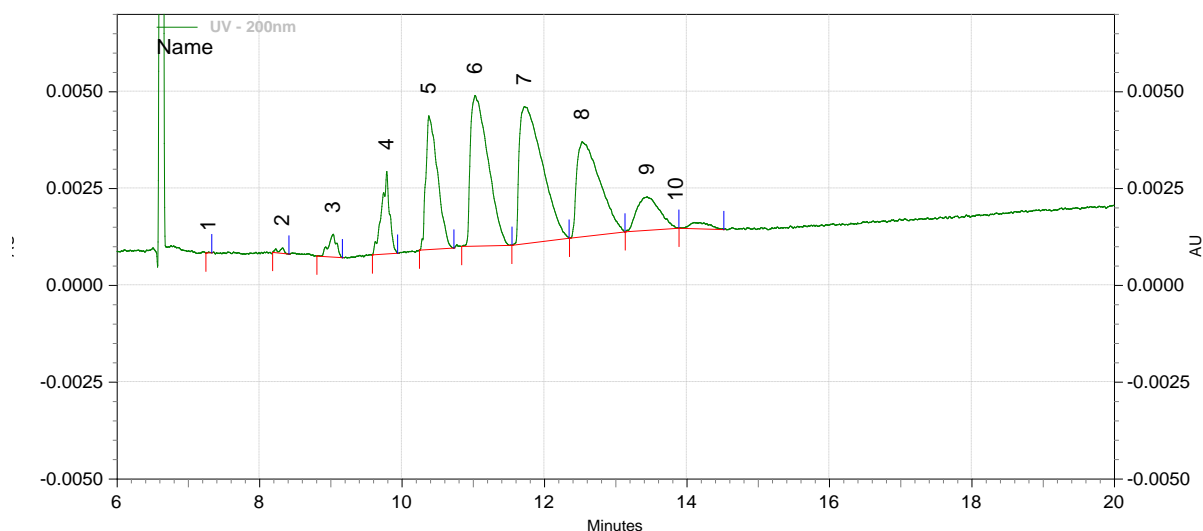
Annex 3-2-14 Validation of analysis procedure for Average degree of substitution-Solution suitability- Sample solution 0h

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\solution stability test\150625 test samples 0h 001.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/25/2015 11:45:30 AM

Printed: 7/2/2015 4:03:07 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.62	0.026	0.00
2	0.71	0.372	14.04
3	0.77	2.096	5.26
4	0.83	6.776	4.14
5	0.89	15.199	2.27
6	0.94	24.268	1.63
7	1.00	27.049	1.27
8	1.07	17.590	1.31
9	1.15	5.721	1.49
10	1.20	0.903	1.21

Totals		100.000	
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附图 10.4.9-35 SBEDC 中平均取代度的测定方法验证图（溶液稳定性-0h）

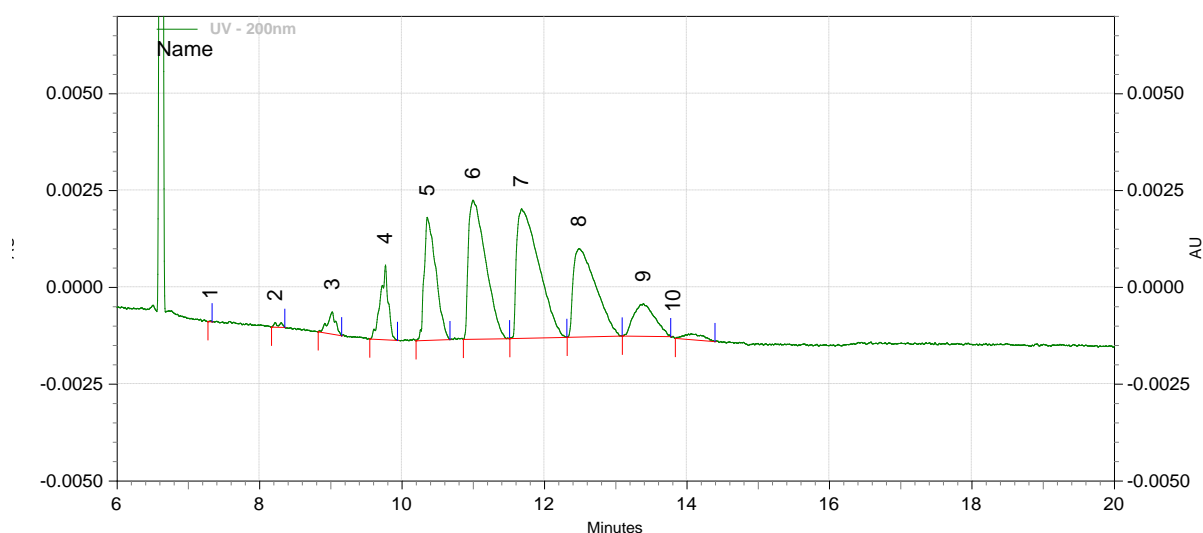
Annex 3-2-15 Validation of analysis procedure for Average degree of substitution-Solution suitability- Sample solution 8h

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\solution stability test\150625 test samples 8h 005.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/25/2015 9:11:35 PM

Printed: 7/2/2015 4:09:52 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.63	0.026	0.00
2	0.70	0.379	8.55
3	0.77	2.176	4.89
4	0.84	6.620	4.20
5	0.89	15.215	2.27
6	0.94	24.148	1.65
7	1.00	27.112	1.28
8	1.07	17.674	1.33
9	1.15	5.720	1.49
10	1.20	0.930	1.20

Totals		100.000	
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附图 10.4.9-39 SBECD 中平均取代度的测定方法验证图（溶液稳定性-8h）

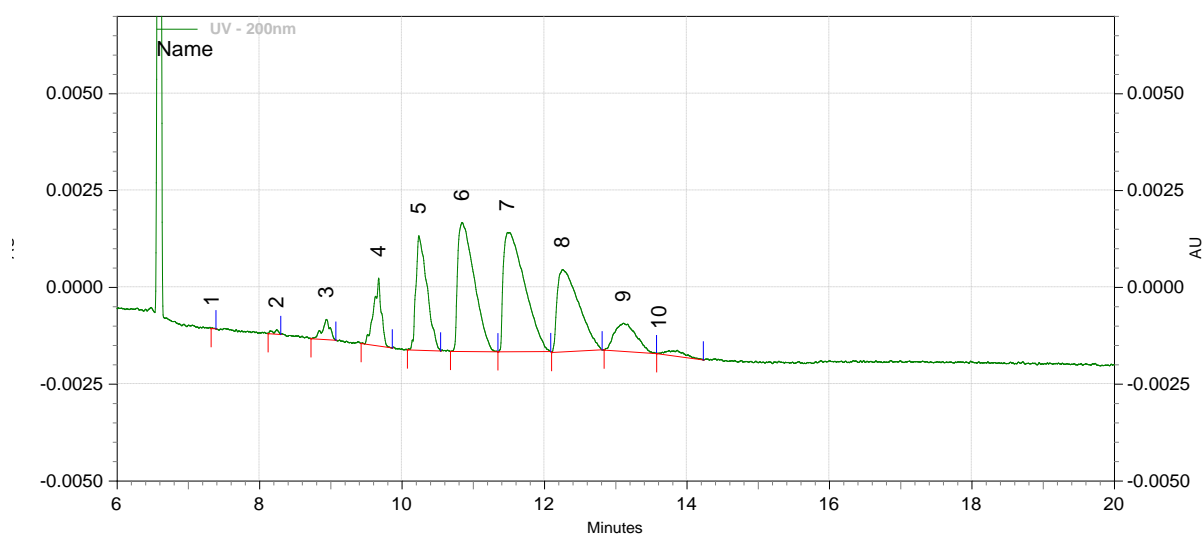
Annex 3-2-16 Validation of analysis procedure for Average degree of substitution-Solution suitability- Sample solution 24h

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\solution stability test\150625 test samples 24h 008.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/26/2015 1:13:31 PM

Printed: 7/2/2015 4:17:36 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.64	0.029	0.00
2	0.72	0.362	0.00
3	0.78	2.203	5.71
4	0.84	6.552	4.29
5	0.89	15.070	2.28
6	0.94	24.137	1.64
7	1.00	27.148	1.26
8	1.07	17.837	1.33
9	1.14	5.745	1.46
10	1.21	0.917	1.44

Totals		100.000	
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附图 10. 4. 9-42 SBECD 中平均取代度的测定方法验证图（溶液稳定性-24h）

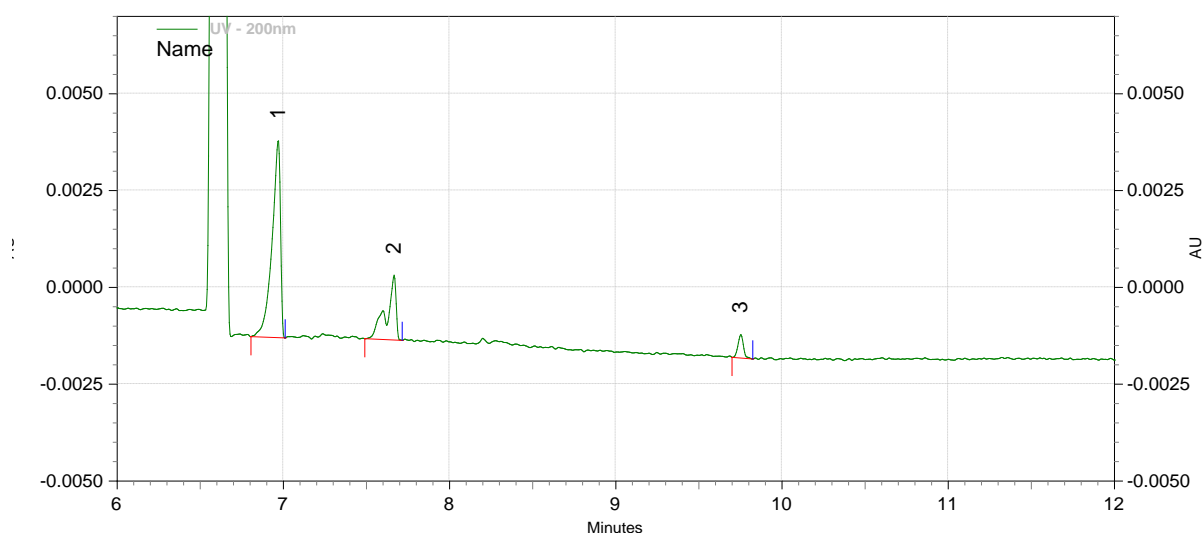
Annex 3-2-17 Validation of analysis procedure for Average degree of substitution- Identification of peak substitution - Sample solution 1

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\peak test to identify the degree of substitution\SBECD-1----20150724

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 7/24/2015 11:28:58 AM

Printed: 7/24/2015 5:29:11 PM



UV - 200nm Results

Name	Migration Time	Area	Resolution (USP)
1	6.971	17930	0.00
2	7.667	6626	8.26
3	9.750	1381	30.47
Totals		25937	

附图 10. 4. 9-46 SBECD 中平均取代度的测定方法验证图（色谱峰取代度的指认试验-SBECD-1）

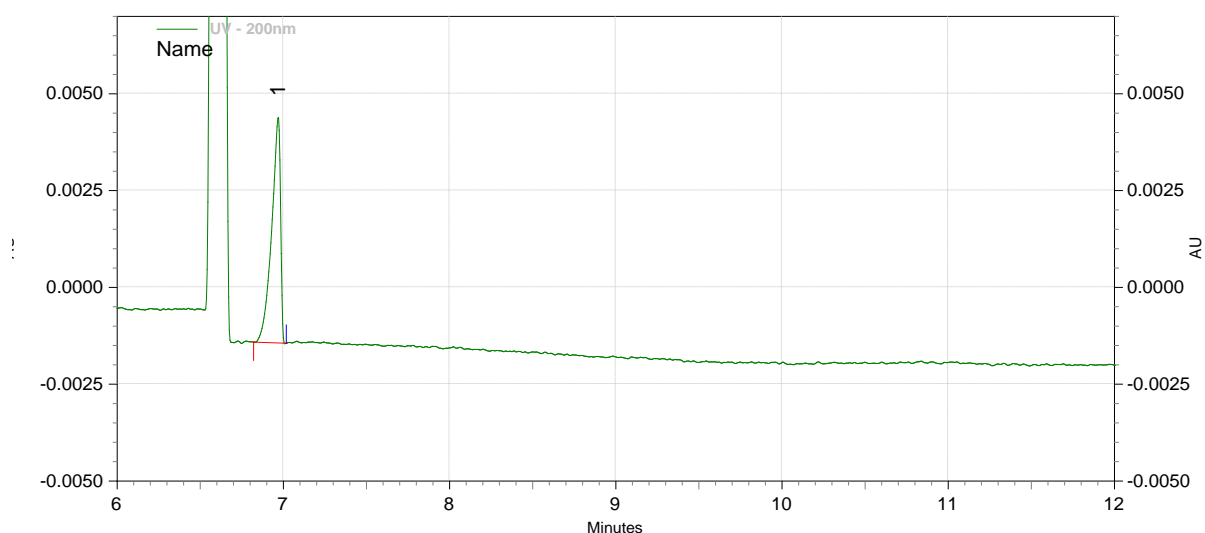
Annex 3-2-18 Validation of analysis procedure for Average degree of substitution- Identification of peak substitution - Sample solution 2

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\peak test to identify the degree of substitution\Betacyclodextrin----20150724

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 7/24/2015 1:34:55 PM

Printed: 7/24/2015 5:31:34 PM



UV - 200nm Results

Name	Migration Time	Area	Resolution (USP)
1	6.971	21542	0.00
Totals			
		21542	

附图 10. 4. 9-48 SBECD 中平均取代度的测定方法验证图（色谱峰取代度的指认试验-Betacyclodextrin）

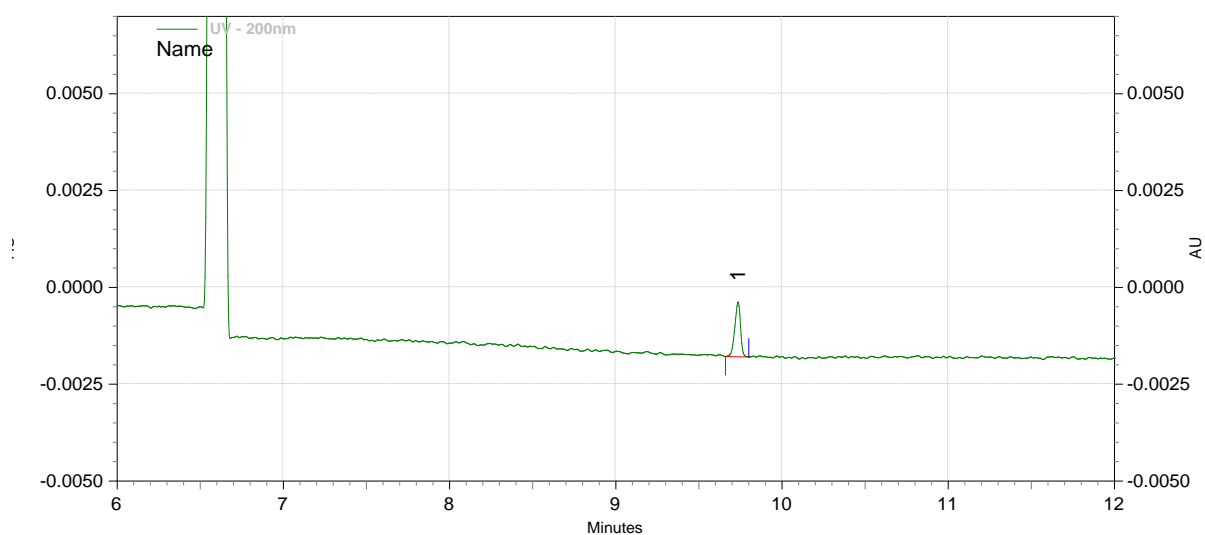
Annex 3-2-19 Validation of analysis procedure for Average degree of substitution- Identification of peak substitution - Sample solution 3

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\peak test to identify the degree of substitution\4-Hydroxybutanesulfonate sodiun salt ----20150724

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 7/24/2015 12:53:39 PM

Printed: 7/24/2015 5:32:47 PM



UV - 200nm Results

Name	Migration Time	Area	Resolution (USP)
1	9.738	3389	0.00
Totals			
		3389	

附图 10. 4. 9-47 SBECD 中平均取代度的测定方法验证图（色谱峰取代度的指认试验-4-Hydroxybutanesulfonate sodiun salt）

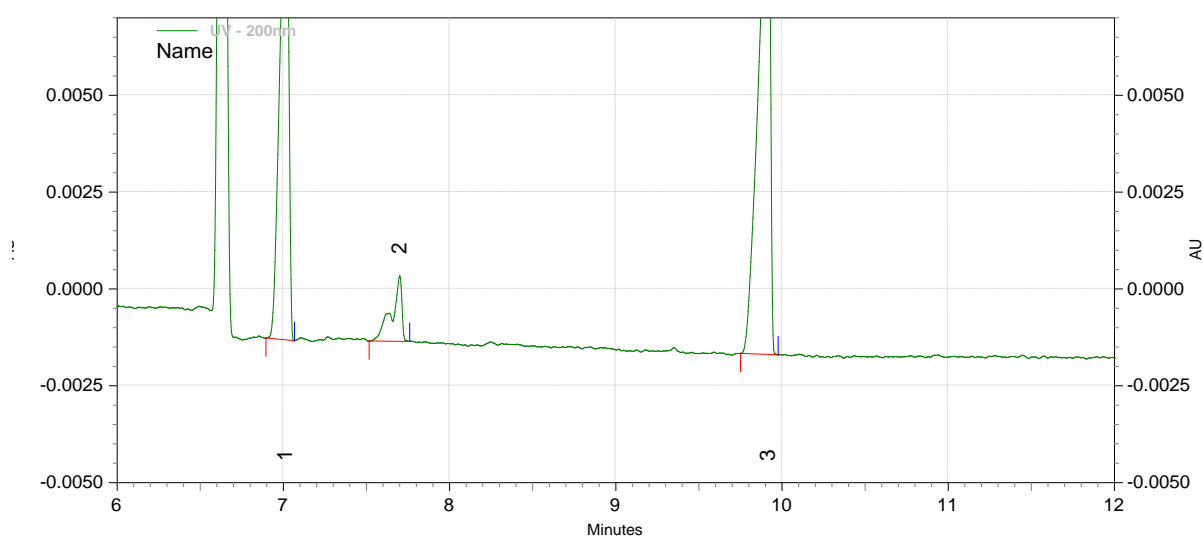
Annex 3-2-20 Validation of analysis procedure for Average degree of substitution- Identification of peak substitution - Sample solution 4

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\peak test to identify the degree of substitution\SBECD-1+4-Hydroxybutanesulfonate sodium salt +Betacyclodextrin----20150724

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 7/24/2015 12:11:17 PM

Printed: 7/24/2015 5:35:05 PM



UV - 200nm Results

Name	Migration Time	Area	Resolution (USP)
1	7.013	45240	0.00
2	7.700	7192	7.55
3	9.917	66782	21.39

Totals		119214	
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附图 10. 4. 9-49 SBECD 中平均取代度的测定方法验证图（色谱峰取代度的指认试验-SBECD-1+4-Hydroxybutanesulfonate sodium salt +Betacyclodextrin）

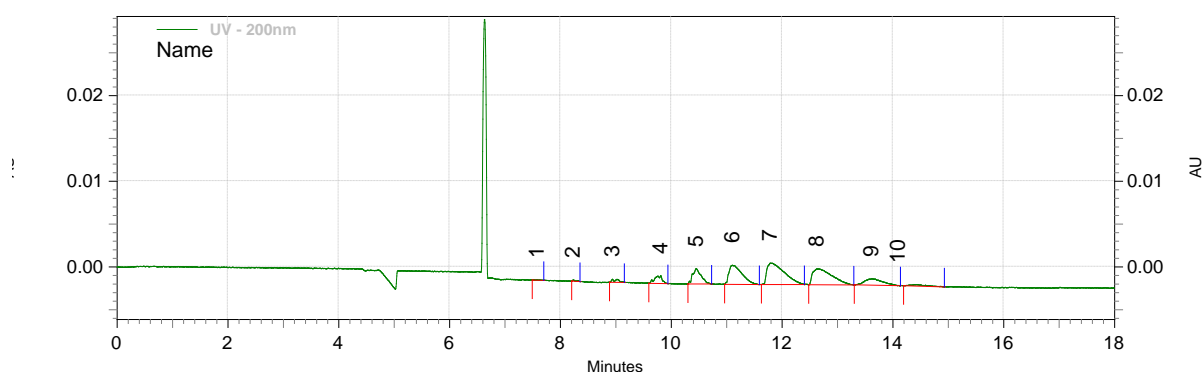
Annex 3-2-21 Validation of analysis procedure for Average degree of substitution- Identification of peak substitution - Sample solution5

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\peak test to identify the degree of substitution\SBECD----20150724

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 7/24/2015 2:20:52 PM

Printed: 7/24/2015 4:31:14 PM



UV - 200nm

Results

Name	Migration Time	Area	Theoretical plates (USP)	Resolution (USP)
1	7.579	225	144150	0.00
2	8.233	524	304071	9.37
3	8.938	2716	33131	5.50
4	9.813	8505	36468	4.35
5	10.450	18909	23787	2.68
6	11.104	36612	9871	1.82
7	11.788	54601	5976	1.29
8	12.629	45316	5660	1.31
9	13.613	17041	7615	1.52
10	14.417	3301	14542	1.46

Totals		187750		
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附图 10. 4. 9-50 SBECD 中平均取代度的测定方法验证图（色谱峰取代度的指认试验-SBECD）

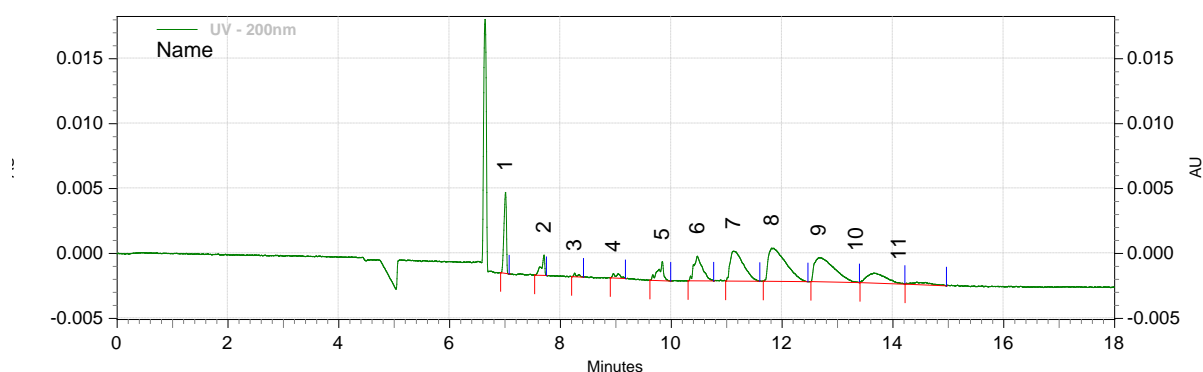
Annex 3-2-22 Validation of analysis procedure for Average degree of substitution- Identification of peak substitution - Sample solution 6

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\peak test to identify the degree of substitution\SBECD+SBECD-1----20150724

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 7/24/2015 3:03:09 PM

Printed: 7/24/2015 4:32:29 PM



UV - 200nm

Results

Name	Migration Time	Area	Theoretical plates (USP)	Resolution (USP)
1	7.008	19117	108643	0.00
2	7.704	6517	164650	8.64
3	8.254	1217	227158	7.57
4	8.954	2659	32946	5.25
5	9.838	9965	38455	4.44
6	10.471	20408	22200	2.63
7	11.125	38532	9610	1.78
8	11.817	56026	6107	1.31
9	12.663	46536	5541	1.32
10	13.654	18792	6653	1.47
11	14.433	4461	0	0.00

Totals		224230		
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附图 10.4.9-51 SBECD 中平均取代度的测定方法验证图（色谱峰取代度的指认试验-SBECD+SBECD-1）

Annex 3-3-1 Validation of analysis procedure for 1,4- butane sultone-Specificity-Blank

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited54.D

Sample Name: KB-1

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Acq. Operator   : SYSTEM                      Seq. Line :   33
Acq. Instrument : GC-03                      Location  :   106
Injection Date  : 11/20/2014 4:52:12 AM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 10:08:25 AM by SYSTEM
                 (modified after loading)
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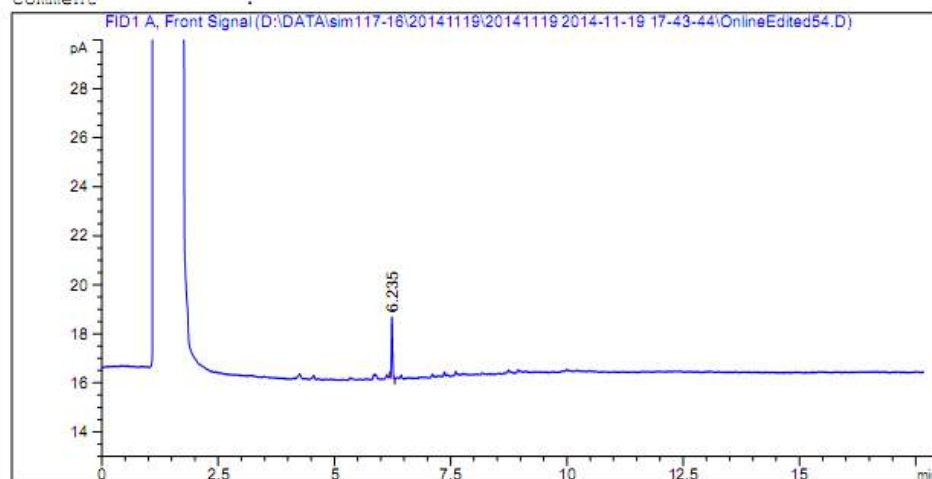
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Column(s)

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Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM              Manufacturer: Agilent
Diameter          : 250.00 µm                Length      : 30.0 m
Film thickness     : 0.25 µm                 Void time   : 0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====

```



Area Percent Report with Performance and Noise

```

=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise		
from	to	(6*SD)	(PtoP)	(ASTM)	Wander	Drift
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000	0.500	6.875e-2	7.151e-2	-	-	5.644

附图10.4.10-31 SBECD中1,4-丁烷磺内酯的测定方法验证图(空白)

GC-03 11/20/2014 10:08:44 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited54.D
Sample Name: KB-1

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.235	6.11	4.40633	2.47564	0.84	0.0272	290627	-	36.0

*** End of Report ***

附图10.4.10-31 SBECD中1,4-丁烷磺内酯的测定方法验证图（空白）

GC-03 11/20/2014 10:08:44 AM SYSTEM

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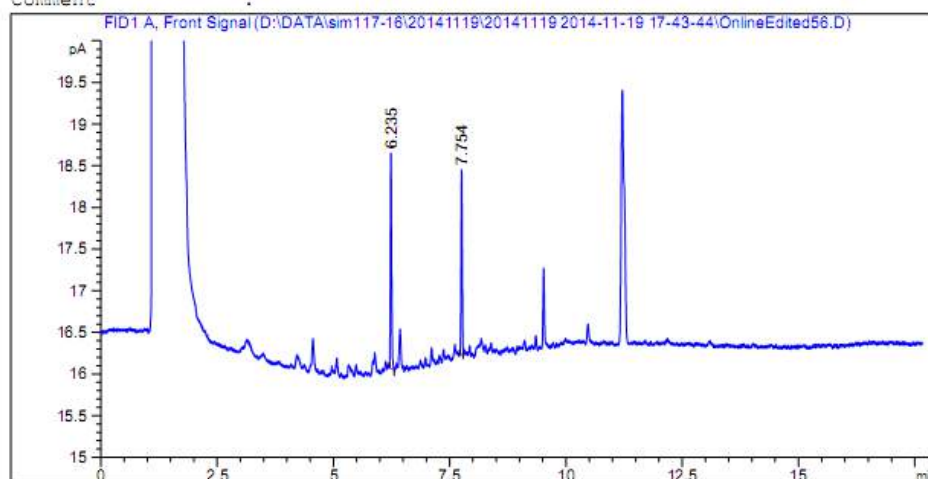
Annex 3-3-2 Validation of analysis procedure for 1,4- butane sultone-Specificity-Sample solution A

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited56.D
Sample Name: Sample-A

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   35
Acq. Instrument : GC-03                      Location  :  108
Injection Date  : 11/20/2014 5:33:56 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44
                  \DB-200-20141114.M (Sequence Method)
Last changed    : 3/31/2015 11:31:45 AM by SYSTEM
                  (modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm               Length      :  30.0 m
Film thickness     : 0.25 µm                Void time   :  0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select ivity
6.235	6.11	4.68519	2.58718	0.77	0.0278	279043	-	-
7.754	7.84	3.74479	2.21933	0.93	0.0269	458819	32.63	1.28

附图10.4.10-8*** SEC中1,4-丁烷磺内酯的测定方法验证图（专属性）

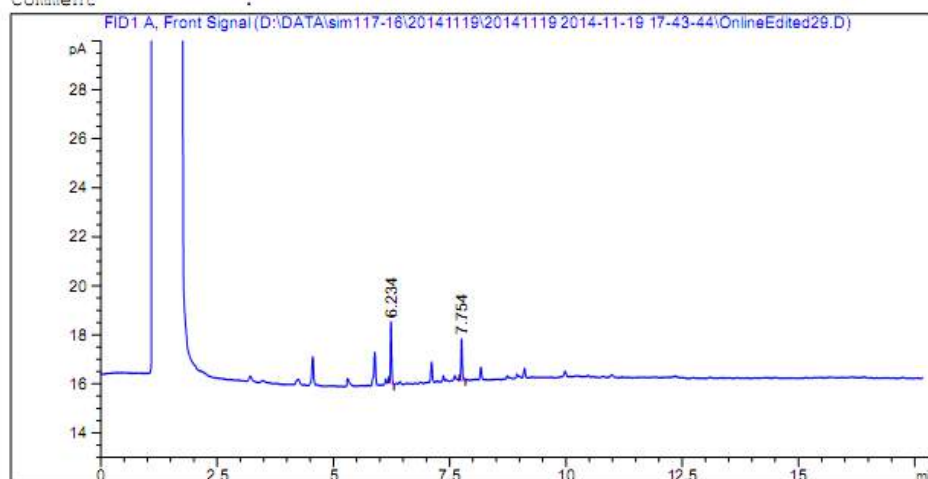
Annex 3-3-3 Validation of analysis procedure for 1,4- butane sultone-System suitability-Solution 2

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited29.D
Sample Name: 0.5ppm

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   25
Acq. Instrument : GC-03                      Location  :   104
Injection Date  : 11/20/2014 2:05:14 AM      Inj       :    2
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 9:58:50 AM by SYSTEM
                (modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm              Length      :   30.0 m
Film thickness     : 0.25 µm               Void time   :   0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range	Noise	Noise	Noise	Wander	Drift
from to	(6*SD)	(PtoP)	(ASTM)		
[min] [min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000 0.500	6.637e-2	5.804e-2	-	-	8.389

附图10.4.10-10 SBECD中1,4-丁烷磺内酯的测定方法验证图（进样精密度-2）

GC-03 11/20/2014 9:59:52 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited29.D
Sample Name: 0.5ppm

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.234	6.11	4.53335	2.50631	0.67	0.0275	284633	-	37.8
7.754	7.84	3.22857	1.69269	0.89	0.0275	440469	32.49	25.5

=====
*** End of Report ***

附图10.4.10-10 SBECD中1,4-丁烷磺内酯的测定方法验证图（进样精密度-2）

GC-03 11/20/2014 9:59:52 AM SYSTEM

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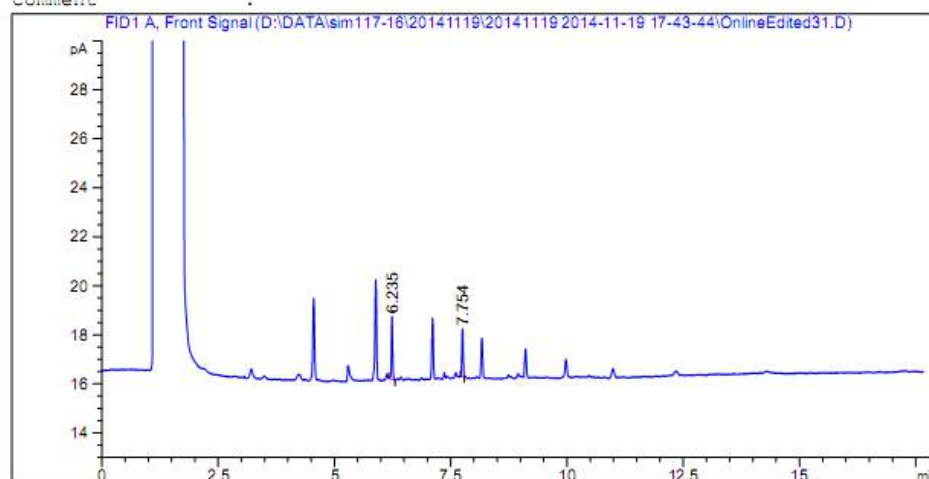
Annex 3-3-4 Validation of analysis procedure for 1,4- butane sultone-System suitability-Solution 4

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited31.D
Sample Name: 0.5ppm

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   27
Acq. Instrument : GC-03                      Location  :   104
Injection Date  : 11/20/2014 2:46:57 AM      Inj       :    4
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 10:03:08 AM by SYSTEM
(modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm               Length      :   30.0 m
Film thickness     : 0.25 µm                Void time   :   0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range	Noise	Noise	Noise	Wander	Drift
from to	(6*SD)	(PtoP)	(ASTM)		
[min] [min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000 0.500	7.376e-2	7.469e-2	-	-	4.393

附图10.4.10-12 SBECD中1,4-丁烷磺内酯的测定方法验证图(进样精密度-4)

GC-03 11/20/2014 10:03:41 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited31.D
Sample Name: 0.5ppm

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.235	6.11	4.76867	2.55736	0.87	0.0278	279118	-	34.7
7.754	7.84	3.76960	1.95453	1.28	0.0303	363278	30.73	26.5

=====
*** End of Report ***

附图10.4.10-12 SBECD中1,4-丁烷磺内酯的测定方法验证图（进样精密度-4）

GC-03 11/20/2014 10:03:41 AM SYSTEM

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Annex 3-3-5 Validation of analysis procedure for 1,4- butane sultone-LOD and LOQ-LOD

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\102F0202.D

Sample Name: 0.1ppm

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :    5
Acq. Instrument : GC-03                      Location  :   102
Injection Date  : 11/19/2014 7:08:40 PM      Inj       :    2
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 6:40:01 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 8:23:03 AM by SYSTEM
                 (modified after loading)
=====

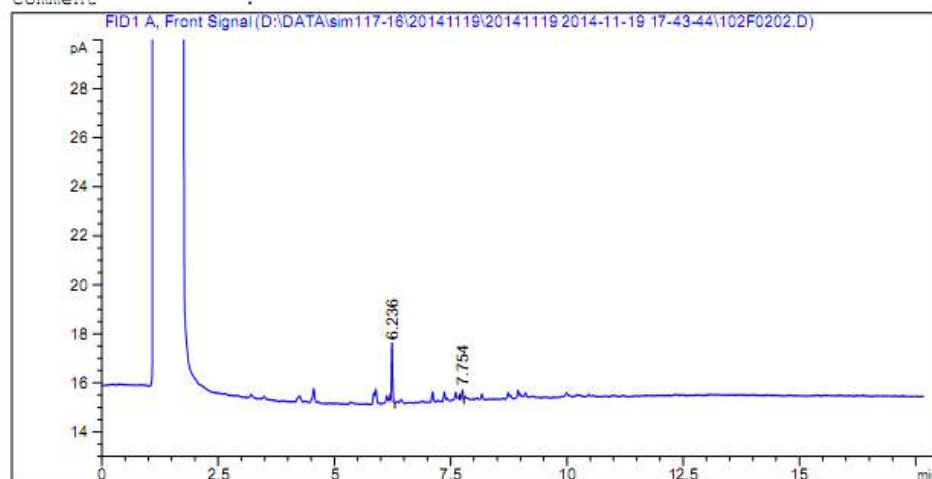
```

Column(s)

```

=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM              Manufacturer: Agilent
Diameter          : 250.00 µm                Length       :   30.0 m
Film thickness     : 0.25 µm                 Void time    :   0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====

```



Area Percent Report with Performance and Noise

```

=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise	Wander	Drift
from	to	(6*SD)	(PtoP)	(ASTM)		
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000	0.500	7.290e-2	7.019e-2	-	-	4.920

附图10.4.10-15 SBECD中1,4-丁烷磺内酯的测定方法验证图(检测限)

GC-03 11/20/2014 8:25:53 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\102F0202.D
Sample Name: 0.1ppm

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.236	6.11	4.24085	2.36321	0.86	0.0275	284861	-	32.4
7.754	7.84	7.53307e-1	3.63827e-1	1.53	0.0317	332109	30.15	5.0

=====
*** End of Report ***

附图10.4.10-15 SBECD中1,4-丁烷磺内酯的测定方法验证图（检测限）

GC-03 11/20/2014 8:25:53 AM SYSTEM

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Annex 3-3-6 Validation of analysis procedure for 1,4- butane sultone-LOD and LOQ-LOQ3

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited23.D

Sample Name: 0.3ppm

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :   18
Acq. Instrument : GC-03                      Location  :   103
Injection Date  : 11/19/2014 11:39:26 PM      Inj       :    3
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 8:59:44 AM by SYSTEM
                 (modified after loading)
=====

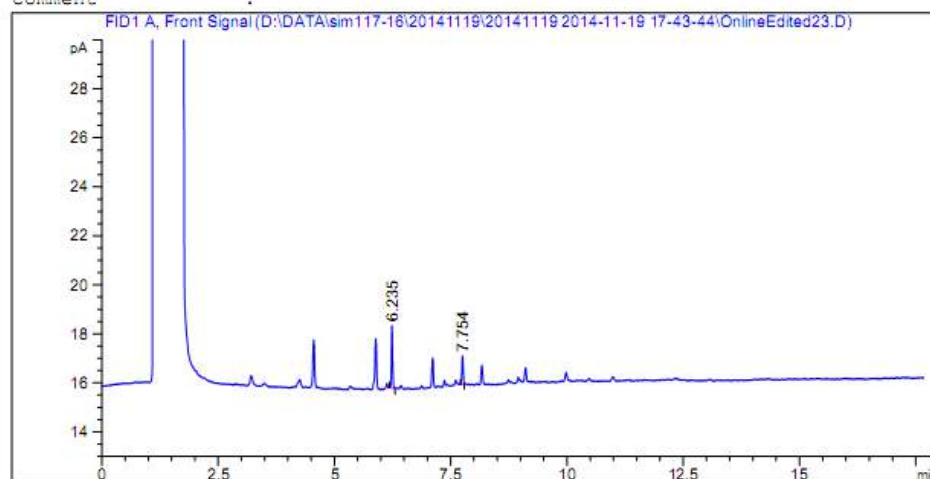
```

Column(s)

```

=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM              Manufacturer: Agilent
Diameter          : 250.00 µm                Length      : 30.0 m
Film thickness    : 0.25 µm                  Void time   : 0.877 min
# Injections      : 0
Maximum Temperature: 300.0 °C
Comment          :
=====

```



Area Percent Report with Performance and Noise

```

=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise		
from	to	(6*SD)	(PtoP)	(ASTM)	Wander	Drift
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000	0.500	5.720e-2	6.020e-2	-	-	15.099

附图10.4.10-18 SBECD中1,4-丁烷磺内酯的测定方法验证图(定量限-3)

GC-03 11/20/2014 9:00:18 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited23.D
Sample Name: 0.3ppm

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.235	6.11	4.58282	2.54326	0.82	0.0275	284786	-	44.5
7.754	7.84	2.21670	1.15732	1.13	0.0303	363278	30.89	20.2

=====
*** End of Report ***

附图10.4.10-18 SBECD中1,4-丁烷磺内酯的测定方法验证图（定量限-3）

GC-03 11/20/2014 9:00:18 AM SYSTEM

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Annex 3-3-7 Validation of analysis procedure for 1,4- butane sultone-LOD and LOQ-LOQ6

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited26.D

Sample Name: 0.3ppm

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :   21
Acq. Instrument : GC-03                      Location  :   103
Injection Date  : 11/20/2014 12:41:57 AM      Inj       :    6
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 9:14:50 AM by SYSTEM
                 (modified after loading)
=====

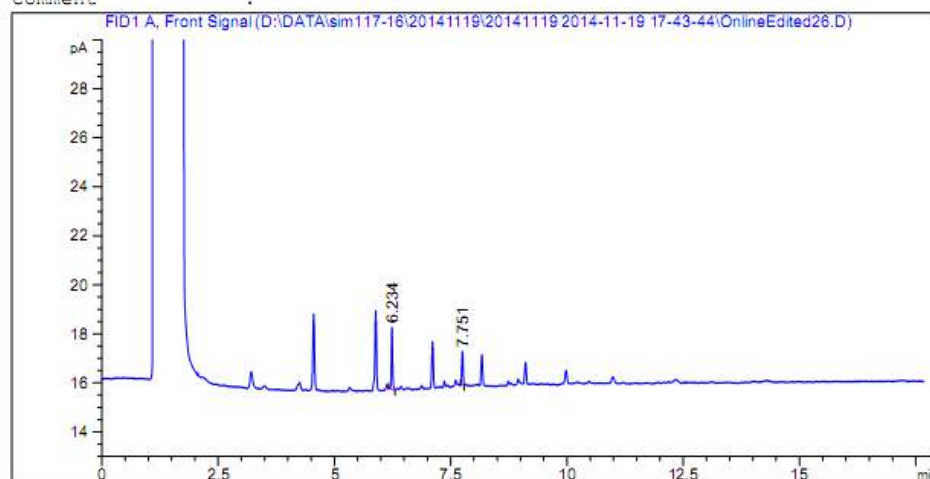
```

Column(s)

```

=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM              Manufacturer: Agilent
Diameter          : 250.00 µm                Length      : 30.0 m
Film thickness     : 0.25 µm                 Void time   : 0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====

```



Area Percent Report with Performance and Noise

```

=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise	Wander	Drift
from	to	(6*SD)	(PtoP)	(ASTM)		
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000	0.500	7.112e-2	6.237e-2	-	-	5.372

附图10.4.10-21 SBECD中1,4-丁烷磺内酯的测定方法验证图(定量限-6)

GC-03 11/20/2014 9:15:07 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited26.D
Sample Name: 0.3ppm

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.234	6.11	4.70873	2.53399	0.80	0.0278	279043	-	35.6
7.751	7.84	2.72512	1.37545	1.01	0.0322	320550	29.70	19.3

=====
*** End of Report ***

附图10.4.10-21 SBECD中1,4-丁烷磺内酯的测定方法验证图（定量限-6）

GC-03 11/20/2014 9:15:07 AM SYSTEM

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Annex 3-3-8 Validation of analysis procedure for 1,4- butane sultone-Linearity-Solution 2

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\103F0308.D

Sample Name: 0.3ppm

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :    7
Acq. Instrument : GC-03                      Location  :   103
Injection Date  : 11/19/2014 7:50:19 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 7:28:07 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 8:27:25 AM by SYSTEM
                 (modified after loading)
=====

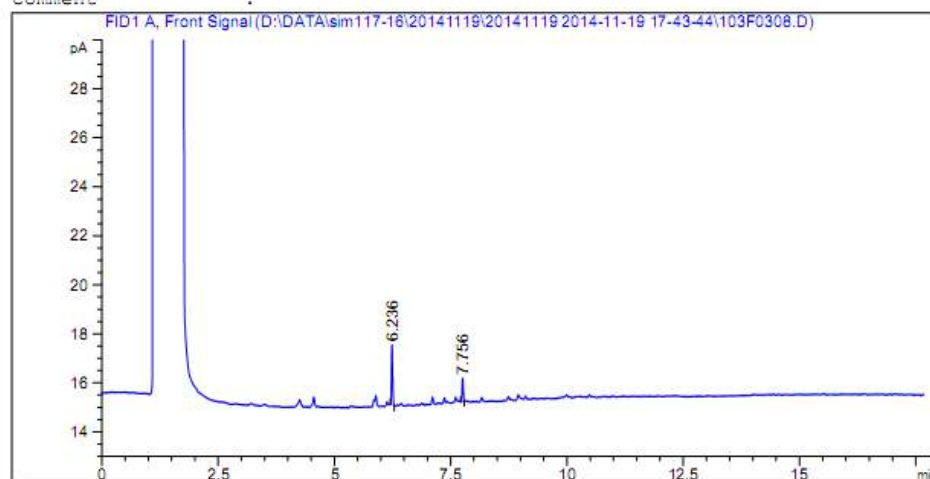
```

Column(s)

```

=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM              Manufacturer: Agilent
Diameter          : 250.00 µm                Length      : 30.0 m
Film thickness     : 0.25 µm                 Void time   : 0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====

```



Area Percent Report with Performance and Noise

```

=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise	Wander	Drift
from	to	(6*SD)	(PtoP)	(ASTM)		
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000	0.500	6.339e-2	6.754e-2	-	-	3.263

附图10.4.10-23 SBECD中1,4-丁烷磺内酯的测定方法验证图(线性-2)

GC-03 11/20/2014 8:27:35 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\103F0308.D
Sample Name: 0.3ppm

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Signal ution /Noise
6.236	6.11	4.39201	2.45224	0.93	0.0281	273765	-	38.7
7.756	7.84	1.65115	9.48320e-1	1.11	0.0273	447411	32.26	15.0

=====
*** End of Report ***

附图10.4.10-23 SBECD中1,4-丁烷磺内酯的测定方法验证图（线性-2）

GC-03 11/20/2014 8:27:35 AM SYSTEM

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Annex 3-3-9 Validation of analysis procedure for 1,4- butane sultone-Linearity-Solution 7

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\108F0801.D

Sample Name: 2.0ppm

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :   13
Acq. Instrument : GC-03                      Location  :   108
Injection Date  : 11/19/2014 9:55:20 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 8:29:06 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 8:41:17 AM by SYSTEM
                  (modified after loading)
=====

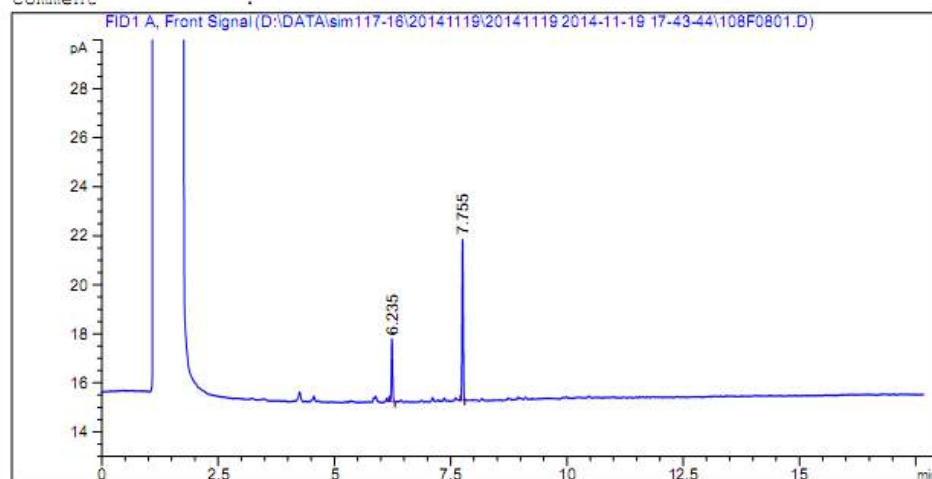
```

Column(s)

```

=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM              Manufacturer: Agilent
Diameter          : 250.00 µm                Length       : 30.0 m
Film thickness     : 0.25 µm                 Void time    : 0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====

```



Area Percent Report with Performance and Noise

```

=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise		
from	to	(6*SD)	(PtoP)	(ASTM)	Wander	Drift
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000	0.500	5.829e-2	5.160e-2	-	-	5.928

附图10. 4. 10-28 SBECD中1, 4-丁烷磺内酯的测定方法验证图 (线性-7)

GC-03 11/20/2014 8:42:35 AM SYSTEM

Page 1 of 2

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\108F0801.D
Sample Name: 2.0ppm

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Signal ution /Noise
6.235	6.11	4.68080	2.56432	0.78	0.0278	279117	-	44.0
7.755	7.84	10.84597	6.56986	0.99	0.0261	488677	33.16	112.7

=====
*** End of Report ***

附图10.4.10-28 SBECD中1,4-丁烷磺内酯的测定方法验证图（线性-7）

GC-03 11/20/2014 8:42:35 AM SYSTEM

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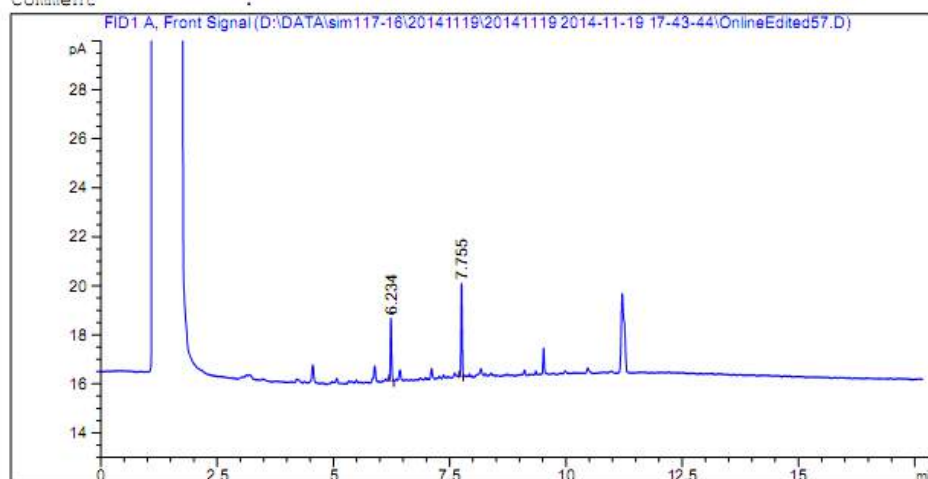
Annex 3-3-10 Validation of analysis procedure for 1,4- butane sultone-Repeatability-Sample solution B

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited57.D
Sample Name: Sample-B

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   36
Acq. Instrument : GC-03                      Location  :   109
Injection Date  : 11/20/2014 5:54:50 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 10:08:25 AM by SYSTEM
                (modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm              Length      :   30.0 m
Film thickness     : 0.25 µm               Void time   :   0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise			
from	to	(6*SD)	(PtoP)	(ASTM)	Wander	Drift	
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]	
0.000	0.500	6.254e-2	5.468e-2	-	-	3.928	

附图10.4.10-33 SBECD中1,4-丁烷磺内酯的测定方法验证图（样品溶液B）

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited57.D
Sample Name: Sample-B

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.234	6.11	4.68072	2.55092	0.71	0.0283	268208	-	40.8
7.755	7.84	6.35204	3.78187	0.97	0.0264	478341	32.65	60.5

=====
*** End of Report ***

附图10. 4. 10-33 SBECD中1, 4-丁烷磺内酯的测定方法验证图 (样品溶液B)

GC-03 11/20/2014 10:09:41 AM SYSTEM

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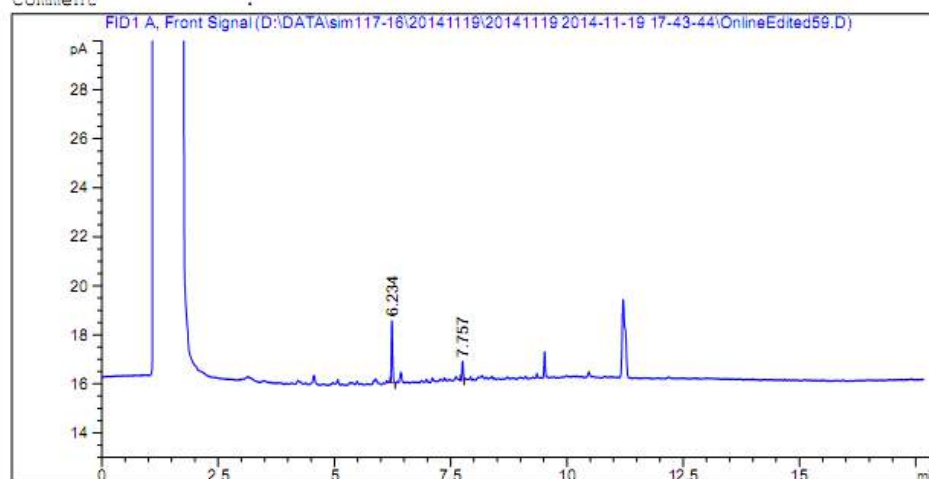
Annex 3-3-11 Validation of analysis procedure for 1,4- butane sultone-Repeatability-Sample solution D-1

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited59.D
Sample Name: Sample-D1

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   38
Acq. Instrument : GC-03                      Location  :   111
Injection Date  : 11/20/2014 6:36:36 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 10:08:25 AM by SYSTEM
(modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM      Manufacturer: Agilent
Diameter          : 250.00 µm       Length :    30.0 m
Film thickness     : 0.25 µm        Void time : 0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range		Noise	Noise	Noise	Wander	Drift
from	to	(6*SD)	(PtoP)	(ASTM)		
[min]	[min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000	0.500	5.893e-2	6.369e-2	-	-	5.725

附图10.4.10-35 SBECD中1,4-丁烷磺内酯的测定方法验证图(重复性-样品溶液D-1)

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited59.D
Sample Name: Sample-D1

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.234	6.11	4.70539	2.52659	0.73	0.0289	257991	-	42.9
7.757	7.85	1.34134	7.58394e-1	1.31	0.0283	415206	31.26	12.9

=====
*** End of Report ***

附图10.4.10-35 SBECD中1,4-丁烷磺内酯的测定方法验证图（重复性-样品溶液D-1）

GC-03 11/20/2014 10:10:01 AM SYSTEM

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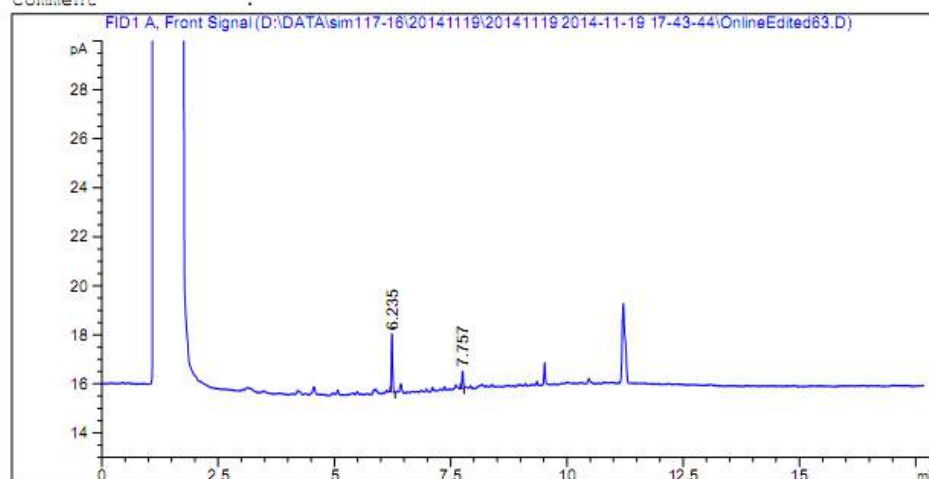
Annex 3-3-12 Validation of analysis procedure for 1,4- butane sultone-Repeatability-Sample solution D-5

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited63.D
 Sample Name: Sample-D5

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   42
Acq. Instrument : GC-03                      Location  :   115
Injection Date  : 11/20/2014 8:00:08 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/20/2014 10:08:25 AM by SYSTEM
                  (modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm               Length      :   30.0 m
Film thickness     : 0.25 µm                Void time   :   0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range	Noise	Noise	Noise	Wander	Drift
from to	(6*SD)	(PtoP)	(ASTM)		
[min] [min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000 0.500	6.995e-2	7.933e-2	-	-	2.280

附图10. 4. 10-39 SBECD中1,4-丁烷磺内酯的测定方法验证图(重复性-样品溶液D-5)

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited63.D
Sample Name: Sample-D5

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Signal ution /Noise
6.235	6.11	4.46052	2.38608	0.81	0.0294	248414	-	34.1
7.757	7.85	1.28087	7.09336e-1	1.34	0.0281	423560	31.10	10.1

=====
*** End of Report ***

附图10.4.10-39 SBECD中1,4-丁烷磺内酯的测定方法验证图（重复性-样品溶液D-5）

GC-03 11/20/2014 10:36:52 AM SYSTEM

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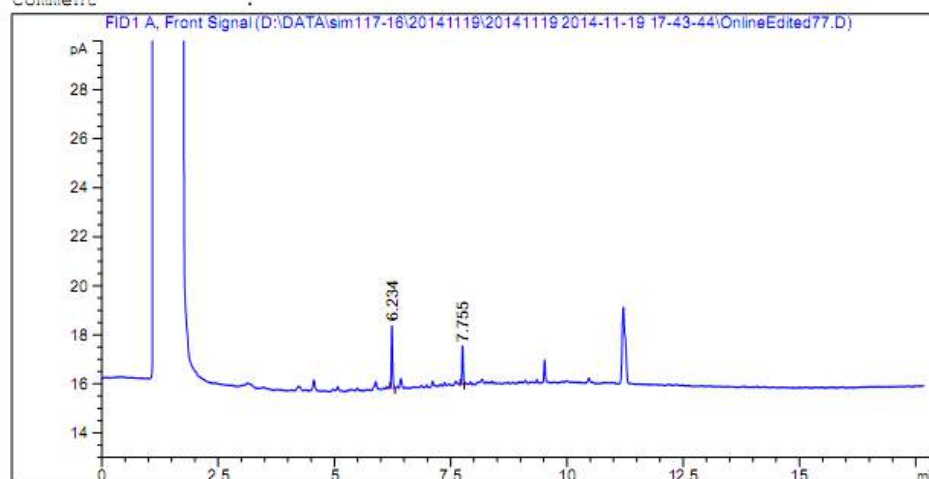
Annex 3-3-13 Validation of analysis procedure for 1,4- butane sultone-Accuracy-LOQ concentration 2

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited77.D
 Sample Name: ZQD-0.3-2

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   45
Acq. Instrument : GC-03                      Location  :   118
Injection Date  : 11/20/2014 9:02:39 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/20/2014 10:08:25 AM by SYSTEM
                  (modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm              Length      :   30.0 m
Film thickness     : 0.25 µm               Void time   :   0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range	Noise	Noise	Noise	Wander	Drift
from to	(6*SD)	(PtoP)	(ASTM)		
[min] [min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000 0.500	6.557e-2	8.720e-2	-	-	4.374

附图10.4.10-42 SBECD中1,4-丁烷磺内酯的测定方法验证图 (准确度-定量限浓度-2)

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited77.D
Sample Name: ZQD-0.3-2

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.234	6.11	4.62972	2.54116	0.74	0.0278	279043	-	38.8
7.755	7.84	2.74823	1.56083	1.03	0.0275	440563	32.32	23.8

=====
*** End of Report ***

附图10. 4. 10-42 SBECD中1, 4-丁烷磺内酯的测定方法验证图 (准确度-定量限浓度-2)

GC-03 11/20/2014 10:42:26 AM SYSTEM

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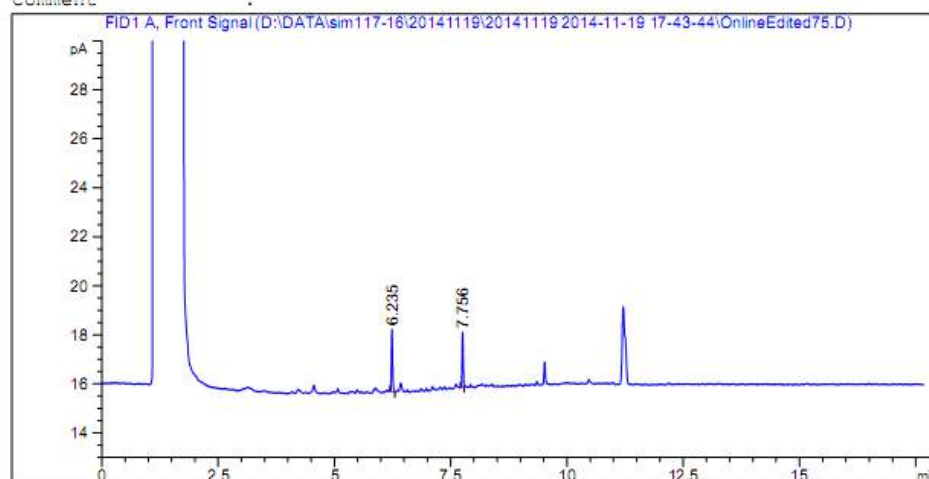
Annex 3-3-14 Validation of analysis procedure for 1,4- butane sultone-Accuracy-100% limit concentration 1

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited75.D
 Sample Name: ZQD-0.5-1

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   47
Acq. Instrument : GC-03                      Location  :   120
Injection Date  : 11/20/2014 9:44:15 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/19/2014 10:57:20 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/20/2014 10:08:25 AM by SYSTEM
                  (modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm              Length      :   30.0 m
Film thickness     : 0.25 µm               Void time   :   0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range	Noise	Noise	Noise			
from to	(6*SD)	(PtoP)	(ASTM)	Wander	Drift	
[min] [min]	[pA]	[pA]	[pA]	[pA]	[pA/h]	
0.000 0.500	6.541e-2	6.383e-2	-	-	1.270	

附图10.4.10-44 SBECD中1,4-丁烷磺内酯的测定方法验证图（准确度-限度浓度-1）

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited75.D
Sample Name: ZQD-0.5-1

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.235	6.11	4.69673	2.53900	0.74	0.0283	268279	-	38.8
7.756	7.84	3.79619	2.25124	1.14	0.0267	468628	32.51	34.4

=====
*** End of Report ***

附图10.4.10-44 SBECD中1,4-丁烷磺内酯的测定方法验证图(准确度-限度浓度-1)

GC-03 11/20/2014 10:44:39 AM SYSTEM

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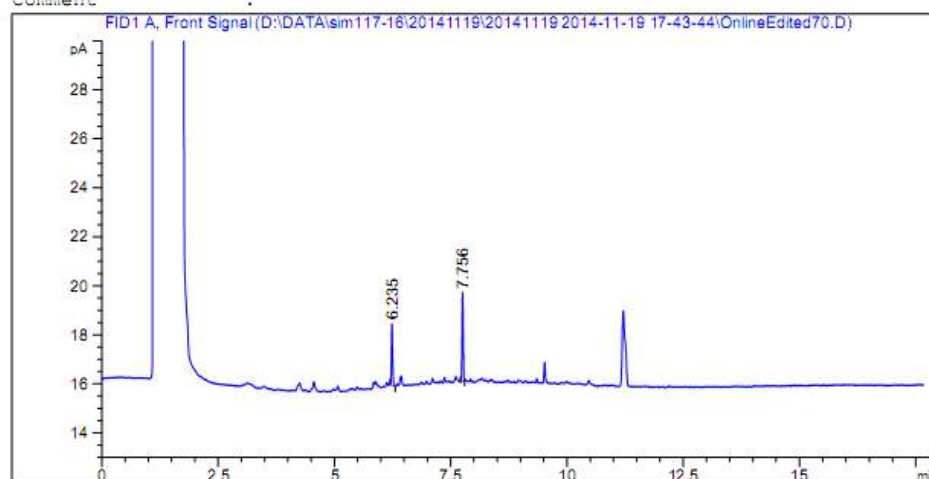
Annex 3-3-15 Validation of analysis procedure for 1,4- butane sultone-Accuracy-200% limit concentration 3

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited70.D
 Sample Name: ZQD-1.0-3

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   52
Acq. Instrument : GC-03                      Location  :   125
Injection Date  : 11/20/2014 11:28:15 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 10:55:30 AM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/20/2014 11:55:49 AM by SYSTEM
(modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM      Manufacturer: Agilent
Diameter          : 250.00 µm       Length :    30.0 m
Film thickness     : 0.25 µm        Void time :   0.877 min
# Injections       :    0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance and Noise

```
=====
Multiplier       :    1.0000
Dilution         :    1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Noise determination:

Time range	Noise	Noise	Noise	Wander	Drift
from to	(6*SD)	(PtoP)	(ASTM)		
[min] [min]	[pA]	[pA]	[pA]	[pA]	[pA/h]
0.000 0.500	7.081e-2	8.541e-2	-	-	4.784

附图10.4.10-49 SBECD中1,4-丁烷磺内酯的测定方法验证图(准确度-2倍限度浓度-3)

GC-03 11/20/2014 11:56:06 AM SYSTEM

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Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited70.D
Sample Name: ZQD-1.0-3

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Signal /Noise
6.235	6.11	4.58366	2.51025	0.76	0.0275	284785	-	35.4
7.756	7.84	6.24665	3.62406	1.13	0.0269	459016	32.83	51.2

=====
*** End of Report ***

附图10.4.10-49 SBECD中1,4-丁烷磺内酯的测定方法验证图(准确度-2倍限度浓度-3)

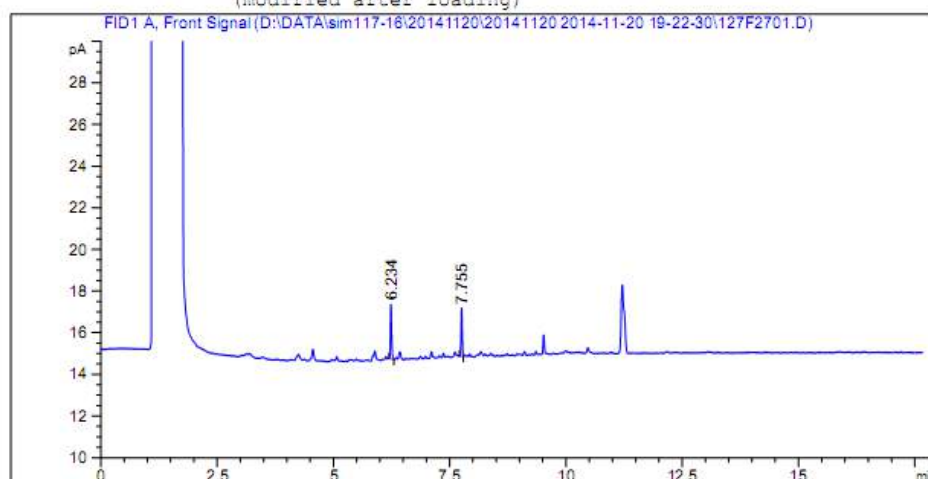
GC-03 11/20/2014 11:56:06 AM SYSTEM

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Annex 3-3-16 Validation of analysis procedure for 1,4- butane sultone-Intermediate precision-Sample solution A

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\127F2701.D
Sample Name: ZJJMD-A

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   27
Acq. Instrument : GC-03                      Location  :   127
Injection Date  : 11/21/2014 4:26:12 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:54:21 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.234	BBA	0.0267	4.79530	2.63291	54.98854
2	7.755	BB	0.0228	3.92524	2.31218	45.01146

Totals : 8.72054 4.94509

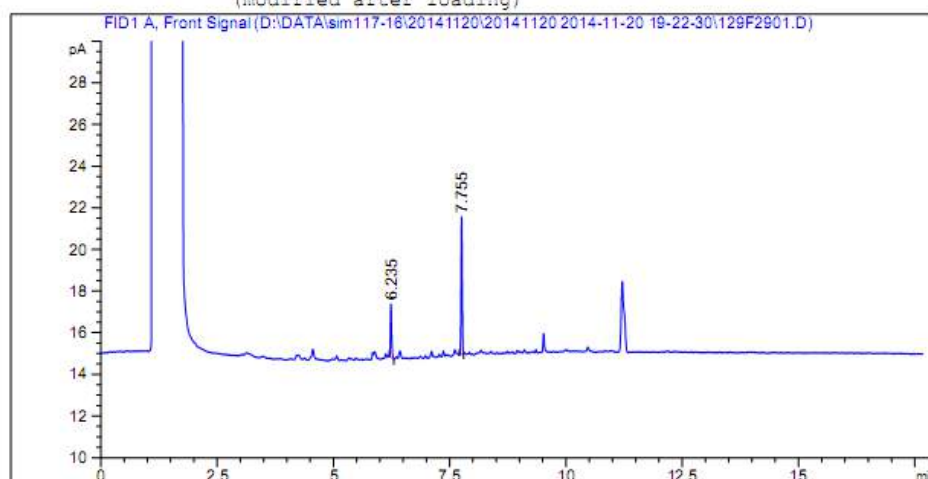
```
=====
*** End of Report ***
=====
```

附图10.4.10-51 SBECD中1,4-丁烷磺内酯的测定方法验证图（中间精密度-样品溶液A）

Annex 3-3-17 Validation of analysis procedure for 1,4- butane sultone-Intermediate precision-Sample solution C

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\129F2901.D
Sample Name: 2JJMD-C

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   29
Acq. Instrument : GC-03                      Location  :   129
Injection Date  : 11/21/2014 5:07:51 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:56:27 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.235	BBA	0.0268	4.62049	2.55887	29.53213
2	7.755	BBA	0.0262	11.02515	6.61198	70.46787

Totals : 15.64563 9.17085

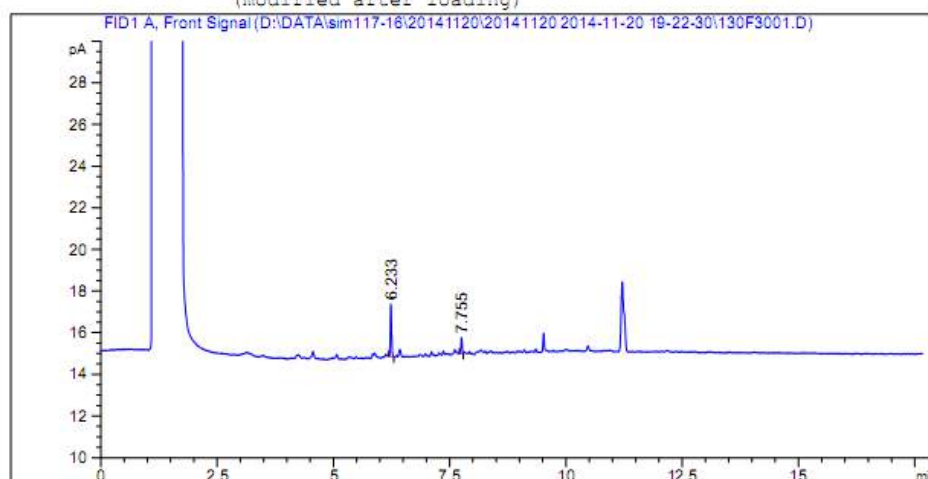
```
=====
*** End of Report ***
=====
```

附图10.4.10-53 SBECD中1,4-丁烷磺内酯的测定方法验证图（中间精密度-样品溶液C）

Annex 3-3-18 Validation of analysis procedure for 1,4- butane sultone-Intermediate precision-Sample solution D-1

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\130F3001.D
Sample Name: 2JJMD-D1

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   30
Acq. Instrument : GC-03                      Location  :   130
Injection Date  : 11/21/2014 5:28:41 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:56:27 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.233	BBA	0.0266	4.66921	2.52454	77.06495
2	7.755	BB	0.0219	1.38959	7.85402e-1	22.93505

Totals : 6.05879 3.30994

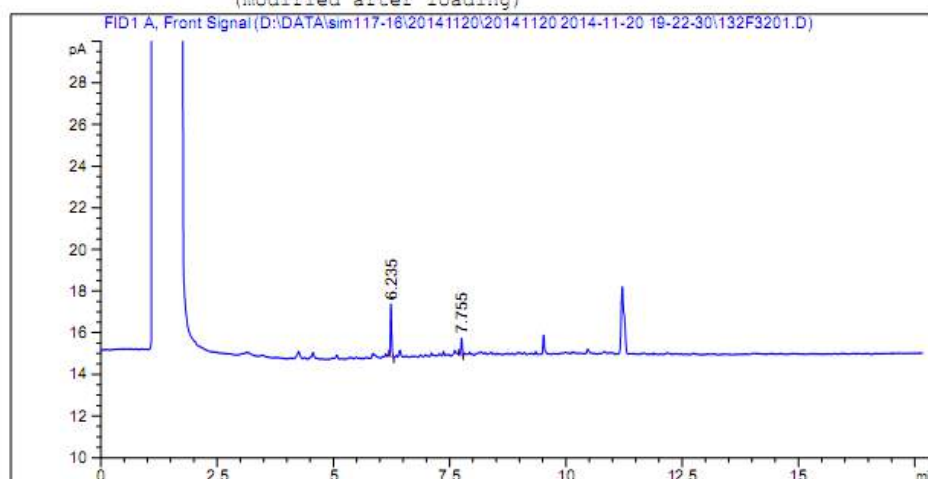
```
=====
*** End of Report ***
=====
```

附图10.4.10-54 SBECD中1,4-丁烷磺内酯的测定方法验证图（中间精密度-样品溶液D-1）

Annex 3-3-19 Validation of analysis procedure for 1,4- butane sultone-Intermediate precision-Sample solution D-3

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\132F3201.D
Sample Name: 2JJMD-D3

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   32
Acq. Instrument : GC-03                      Location  :   132
Injection Date  : 11/21/2014 6:10:25 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:56:27 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.235	BBA	0.0282	4.63208	2.51678	76.57463
2	7.755	BBA	0.0228	1.41702	7.96335e-1	23.42537

Totals : 6.04910 3.31312

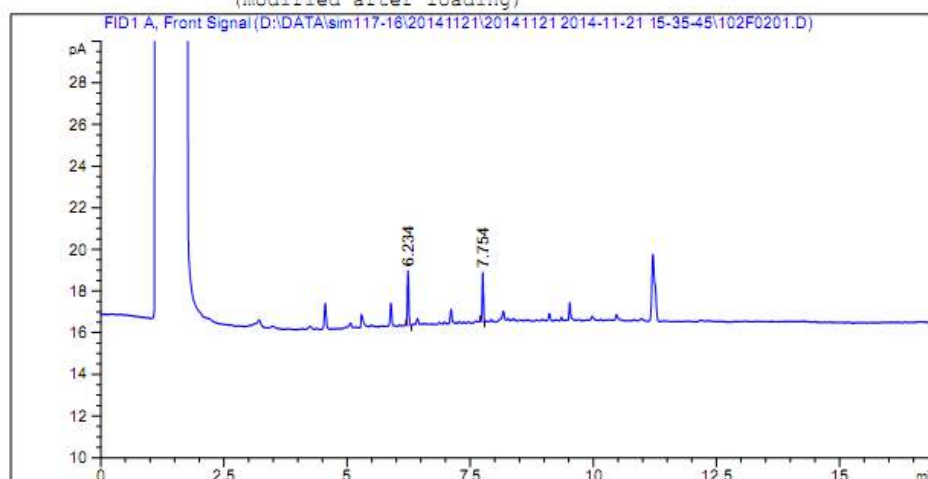
```
=====
*** End of Report ***
=====
```

附图10.4.10-56 SBECD中1,4-丁烷磺内酯的测定方法验证图（中间精密度-样品溶液D-3）

Annex 3-3-20 Validation of analysis procedure for 1,4- butane sultone-Solution stability-Sample solutionA 0h

Data File D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\102F0201.D
Sample Name: A-0H

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :    2
Acq. Instrument : GC-03                      Location  :   102
Injection Date  : 11/21/2014 3:58:32 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M
Last changed    : 11/21/2014 3:35:45 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/22/2014 1:14:02 PM by SYSTEM
                  (modified after loading)
=====
```



Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.234	BB	0.0276	4.86061	2.62592	54.64562
2	7.754	BBA	0.0249	4.03418	2.36255	45.35438

Totals : 8.89479 4.98847

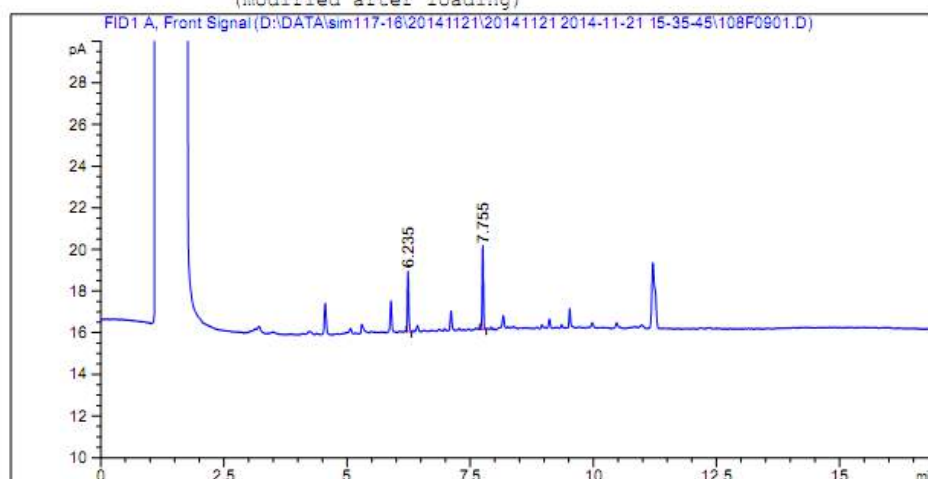
*** End of Report ***

附图10.4.10-58 SBECD中1,4-丁烷磺内酯的测定方法验证图(溶液稳定性0时-样品溶液A)

Annex 3-3-21 Validation of analysis procedure for 1,4- butane sultone-Solution stability-Sample solutionB 2h

Data File D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\108F0901.D
Sample Name: B-2H

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :    9
Acq. Instrument : GC-03                      Location  :   108
Injection Date  : 11/21/2014 6:23:55 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M
Last changed    : 11/21/2014 6:00:15 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/22/2014 1:14:02 PM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.235	BB	0.0295	5.25150	2.84426	42.80778
2	7.755	BBA	0.0286	7.01613	3.96937	57.19222

Totals : 12.26762 6.81364

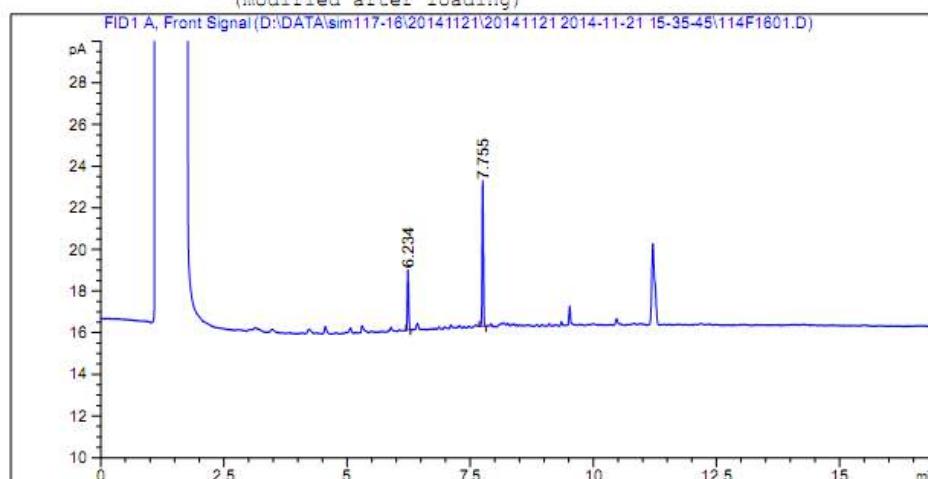
```
=====
*** End of Report ***
=====
```

附图10.4.10-64 SBECD中1,4-丁烷磺内酯的测定方法验证图（溶液稳定性2小时-样品溶液B）

Annex 3-3-22 Validation of analysis procedure for 1,4- butane sultone-Solution stability-Sample solutionC 4h

Data File D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\114F1601.D
Sample Name: C-4H

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   16
Acq. Instrument : GC-03                      Location  :   114
Injection Date  : 11/21/2014 8:49:14 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M
Last changed    : 11/21/2014 6:00:15 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/22/2014 1:14:02 PM by SYSTEM
                  (modified after loading)
=====
```



Area Percent Report

```
=====
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.234	BBA	0.0294	5.10564	2.78038	30.10683
2	7.755	BBA	0.0281	11.85278	6.87480	69.89317

Totals : 16.95843 9.65517

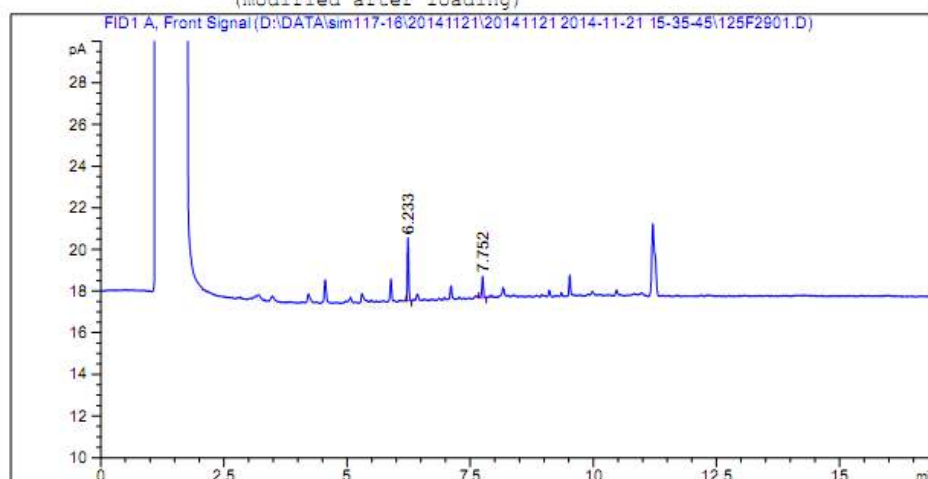
*** End of Report ***

附图10.4.10-70 SBECD中1,4-丁烷磺内酯的测定方法验证图（溶液稳定性4小时-样品溶液C）

Annex 3-3-23 Validation of analysis procedure for 1,4- butane sultone-Solution stability-Sample solutionD 12h

Data File D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\125F2901.D
Sample Name: D-12H

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   41
Acq. Instrument : GC-03                      Location  :   125
Injection Date  : 11/22/2014 5:12:52 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M
Last changed    : 11/21/2014 8:46:14 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/22/2014 11:39:30 AM by SYSTEM
                  (modified after loading)
=====
```



Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.233	BV R	0.0238	5.57198	3.04546	72.02875
2	7.752	VV R	0.0287	2.16379	1.03188	27.97125

Totals : 7.73577 4.07734

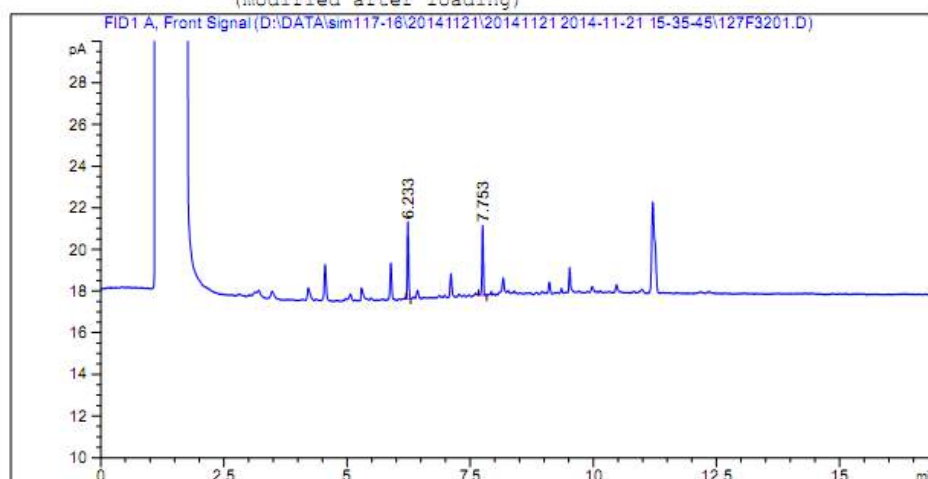
*** End of Report ***

附图10.4.10-76 SBECD中1,4-丁烷磺内酯的测定方法验证图（溶液稳定性12小时-样品溶液D）

Annex 3-3-24 Validation of analysis procedure for 1,4- butane sultone-Solution stability-Sample solutionA 16h

Data File D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\127F3201.D
Sample Name: A-16H

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   50
Acq. Instrument : GC-03                      Location  :   127
Injection Date  : 11/22/2014 8:11:43 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M
Last changed    : 11/21/2014 8:46:14 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/22/2014 1:02:06 PM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.233	BBA	0.0291	6.59022	3.64294	51.27977
2	7.753	BB	0.0301	6.26128	3.30227	48.72023

Totals : 12.85150 6.94521

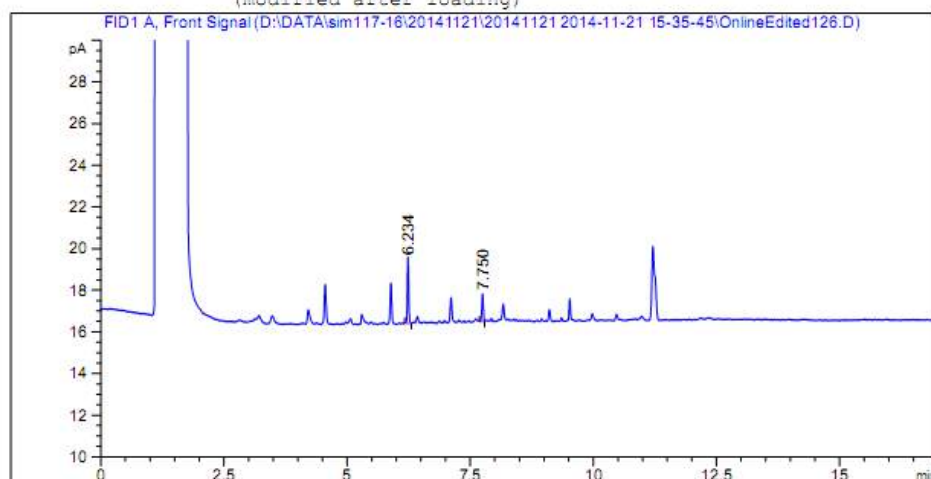
```
=====
*** End of Report ***
=====
```

附图10.4.10-78 SBECD中1,4-丁烷磺内酯的测定方法验证图（溶液稳定性16小时-样品溶液A）

Annex 3-3-25 Validation of analysis procedure for 1,4- butane sultone-Solution stability-Sample solutionD 21h

Data File D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\OnlineEdited126.D
Sample Name: D-21H

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   66
Acq. Instrument : GC-03                      Location  :   135
Injection Date  : 11/22/2014 1:36:00 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M
Last changed    : 11/22/2014 1:49:55 PM by SYSTEM
                  (modified after loading)
Analysis Method : D:\DATA\sim117-16\20141121\20141121 2014-11-21 15-35-45\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/22/2014 2:22:38 PM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.234	BB	0.0303	5.88229	3.07740	68.23972
2	7.750	BB	0.0343	2.73775	1.31331	31.76028

Totals : 8.62004 4.39071

```
=====
*** End of Report ***
=====
```

附图10.4.10-86 SBECD中1,4-丁烷磺内酯的测定方法验证图（溶液稳定性21小时-样品溶液D）

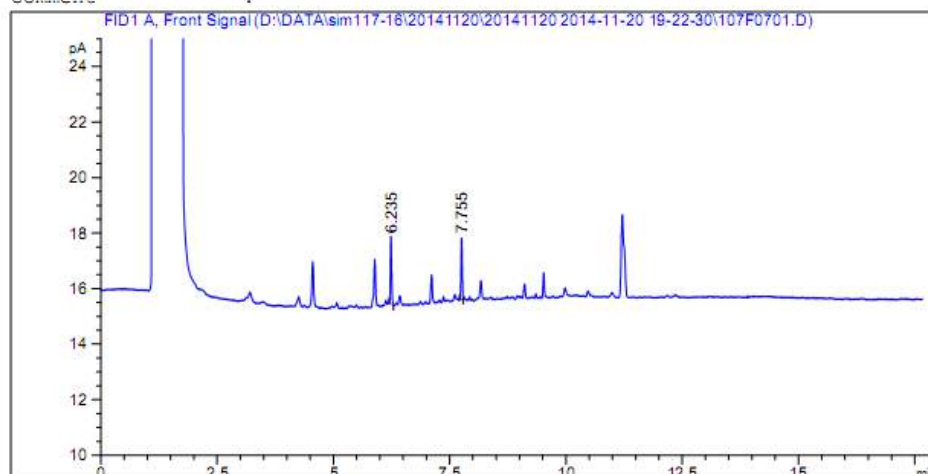
Annex 3-3-26 Validation of analysis procedure for 1,4- butane sultone-Robustness-Prepare condition 1-Sample solution A

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\107F0701.D
Sample Name: NYX-25S-4MIN-A

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :    7
Acq. Instrument : GC-03                      Location  :   107
Injection Date  : 11/20/2014 9:29:28 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114.M (Sequence Method)
Last changed    : 3/31/2015 11:43:02 AM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM      Manufacturer: Agilent
Diameter          : 250.00 µm       Length : 30.0 m
Film thickness     : 0.25 µm        Void time : 0.877 min
# Injections      : 0
Maximum Temperature: 300.0 °C
Comment          :
=====
```



Area Percent Report with Performance

```
=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select
							ution	ivity
6.235	6.11	4.54066	2.43841	0.84	0.0289	258060	-	-
7.755	7.84	3.88639	2.21854	1.03	0.0275	440468	31.65	1.28

*** End of Report ***

附图10.4.10-88 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-蜗旋25秒后静置4分钟-样品溶液A)

GC-03 3/31/2015 11:43:46 AM SYSTEM

Page 1 of 1

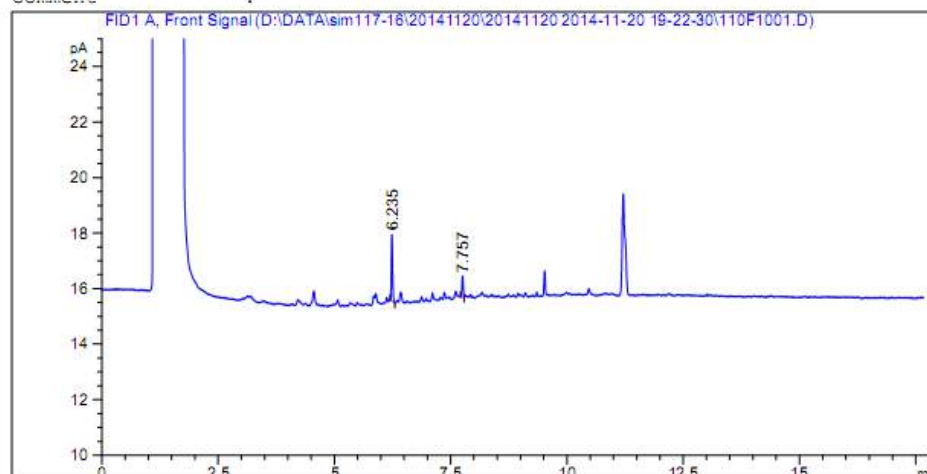
Annex 3-3-27 Validation of analysis procedure for 1,4- butane sultone-Robustness-Prepare condition 1-Sample solution D

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\110F1001.D
Sample Name: NYX-25S-4MIN-D

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   10
Acq. Instrument : GC-03                      Location  :   110
Injection Date  : 11/20/2014 10:32:11 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114.M (Sequence Method)
Last changed    : 3/31/2015 11:43:02 AM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM      Manufacturer: Agilent
Diameter          : 250.00 µm       Length      : 30.0 m
Film thickness    : 0.25 µm         Void time   : 0.877 min
# Injections      : 0
Maximum Temperature: 300.0 °C
Comment          :
=====
```



Area Percent Report with Performance

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol ution	Select ivity
6.235	6.11	4.44260	2.39823	0.77	0.0286	263025	-	-
7.757	7.85	1.38549	7.42244e-1	1.35	0.0292	391820	30.95	1.28

*** End of Report ***

附图10.4.10-91 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-蜗旋25秒后静置4分钟-样品溶液D)
GC-03 3/31/2015 11:44:14 AM SYSTEM Page 1 of 1

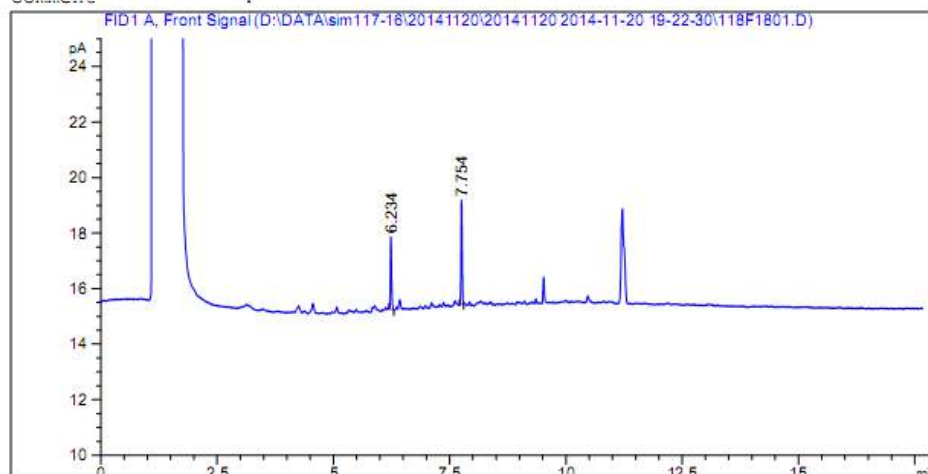
Annex 3-3-28 Validation of analysis procedure for 1,4- butane sultone-Robustness-Prepare condition 2-Sample solution B

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\118F1801.D
Sample Name: NYX-35S-6MIN-B

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   18
Acq. Instrument : GC-03                      Location  :   118
Injection Date  : 11/21/2014 1:19:04 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114.M (Sequence Method)
Last changed    : 3/31/2015 11:43:02 AM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm               Length      : 30.0 m
Film thickness     : 0.25 µm                Void time   : 0.877 min
# Injections      : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select
							ution	ivity
6.234	6.11	4.73411	2.62328	0.70	0.0278	278969	-	-
7.754	7.84	6.29132	3.76916	0.85	0.0264	478238	32.97	1.28

*** End of Report ***

附图10.4.10-94 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-蜗旋35秒后静置6分钟-样品溶液B)

GC-03 3/31/2015 11:54:02 AM SYSTEM

Page 1 of 1

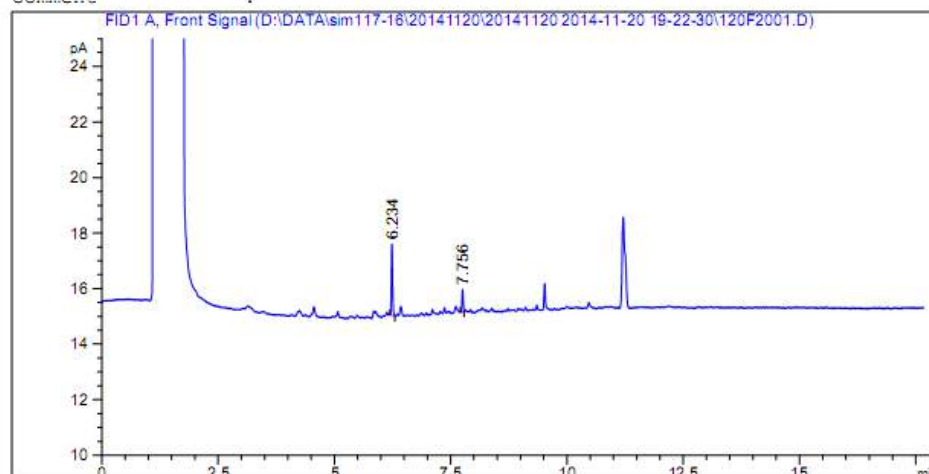
Annex 3-3-29 Validation of analysis procedure for 1,4- butane sultone-Robustness-Prepare condition 2-Sample solution D

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\120F2001.D
Sample Name: NYX-35S-6MIN-D

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   20
Acq. Instrument : GC-03                      Location  :   120
Injection Date  : 11/21/2014 2:00:42 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114.M (Sequence Method)
Last changed    : 3/31/2015 11:43:02 AM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm              Length      : 30.0 m
Film thickness    : 0.25 µm                Void time   : 0.877 min
# Injections      : 0
Maximum Temperature: 300.0 °C
Comment          :
=====
```



Area Percent Report with Performance

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select
							ution	ivity
6.234	6.11	4.70005	2.56707	0.68	0.0281	273545	-	-
7.756	7.84	1.41005	8.11814e-1	1.31	0.0272	449697	32.36	1.28

*** End of Report ***

附图10.4.10-96 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-蜗旋35秒后静置6分钟-样品溶液D)
GC-03 3/31/2015 11:54:29 AM SYSTEM Page 1 of 1

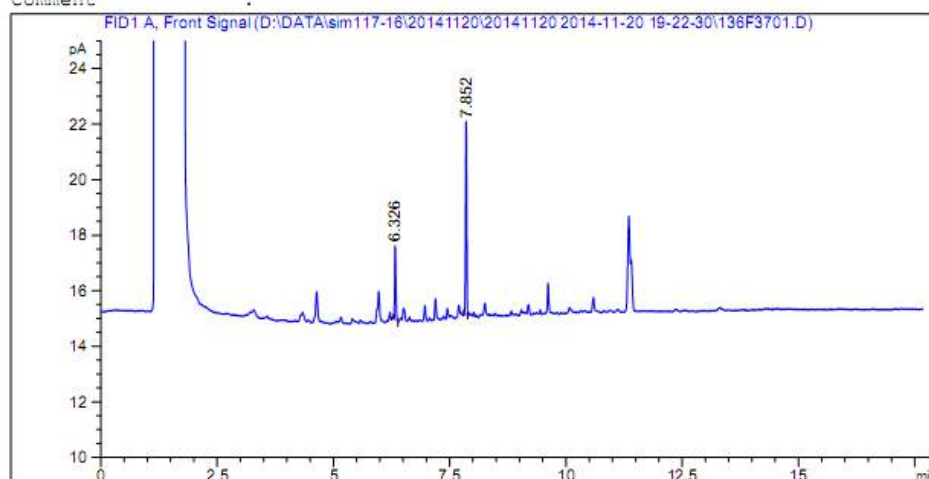
Annex 3-3-30 Validation of analysis procedure for 1,4- butane sultone-Robustness-Condition 1-Sample solution C

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\136F3701.D
Sample Name: C-2.8ml/min

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   38
Acq. Instrument : GC-03                      Location  :   136
Injection Date  : 11/21/2014 8:13:01 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114-2.8
                                           .M
Last changed    : 11/20/2014 7:22:31 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114-2.8.M (Sequence Method)
Last changed    : 3/31/2015 1:35:38 PM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm               Length      :   30.0 m
Film thickness     : 0.25 µm                Void time   :   0.916 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
=====
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select ivity
6.326	5.91	4.75239	2.65428	0.65	0.0278	287310	-	-
7.852	7.57	11.51784	6.94391	1.05	0.0261	500937	33.28	1.28

*** End of Report ***

附图10.4.10-100 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-氮气流速2.8ml/min-样品溶
GC-03 3/31/2015 1:36:20 PM SYSTEM Page 1 of 1

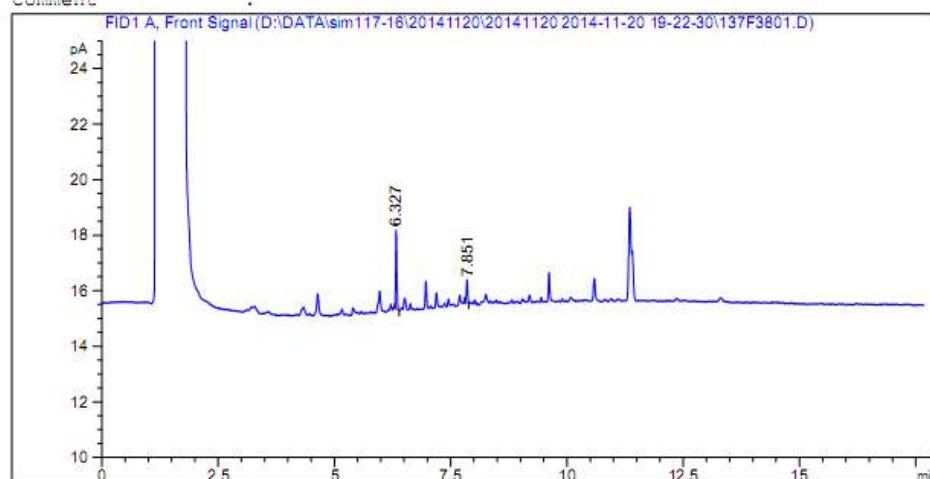
Annex 3-3-31 Validation of analysis procedure for 1,4- butane sultone-Robustness-Condition 1-Sample solution D

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\137F3801.D
Sample Name: D-2.8ml/min

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   39
Acq. Instrument : GC-03                      Location  :   137
Injection Date  : 11/21/2014 8:33:55 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114-2.8
                                           .M
Last changed    : 11/20/2014 7:22:31 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114-2.8.M (Sequence Method)
Last changed    : 3/31/2015 1:35:38 PM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm              Length      :   30.0 m
Film thickness     : 0.25 µm               Void time   :   0.916 min
# Injections       :    0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
=====
Multiplier       :    1.0000
Dilution         :    1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select
							ution	ivity
6.327	5.91	4.82689	2.85619	0.74	0.0258	332276	-	-
7.851	7.57	1.59339	8.36964e-1	1.29	0.0292	401391	32.57	1.28

*** End of Report ***

附图10.4.10-101 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-氮气流速2.8ml/min-样品溶
GC-03 3/31/2015 1:36:32 PM SYSTEM Page 1 of 1

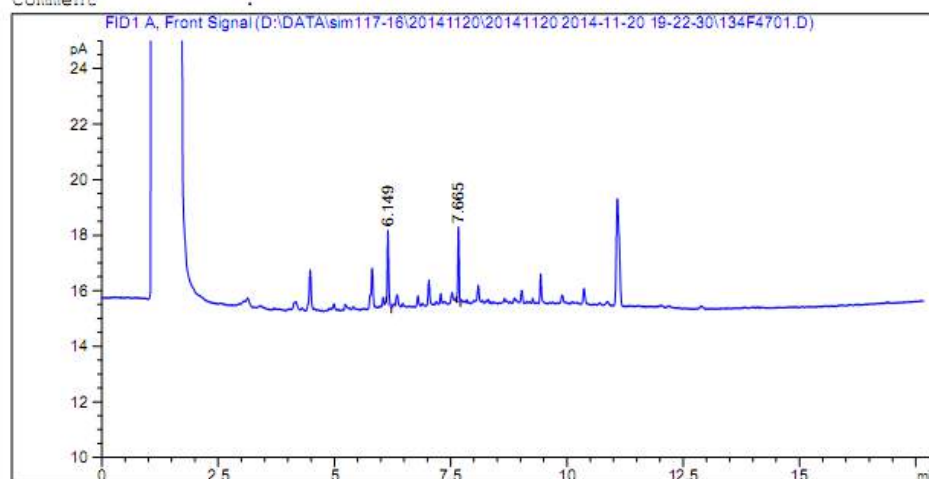
Annex 3-3-32 Validation of analysis procedure for 1,4- butane sultone-Robustness-Condition 2-Sample solution A

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\134F4701.D
Sample Name: A-3.2ml/min

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   42
Acq. Instrument : GC-03                      Location  :   134
Injection Date  : 11/21/2014 9:35:21 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114-3.2
                                           .M
Last changed    : 11/20/2014 7:22:32 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114-3.2.M (Sequence Method)
Last changed    : 3/31/2015 1:44:19 PM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm               Length      :   30.0 m
Film thickness     : 0.25 µm                Void time   :   0.842 min
# Injections       :    0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
=====
Multiplier      :    1.0000
Dilution        :    1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select ivity
6.149	6.30	5.06868	2.64004	0.71	0.0292	246179	-	-
7.665	8.10	4.69454	2.69551	0.94	0.0281	413520	31.14	1.29

*** End of Report ***

附图10.4.10-103 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-氮气流速3.2ml/min-样品溶
GC-03 3/31/2015 1:47:08 PM SYSTEM Page 1 of 1

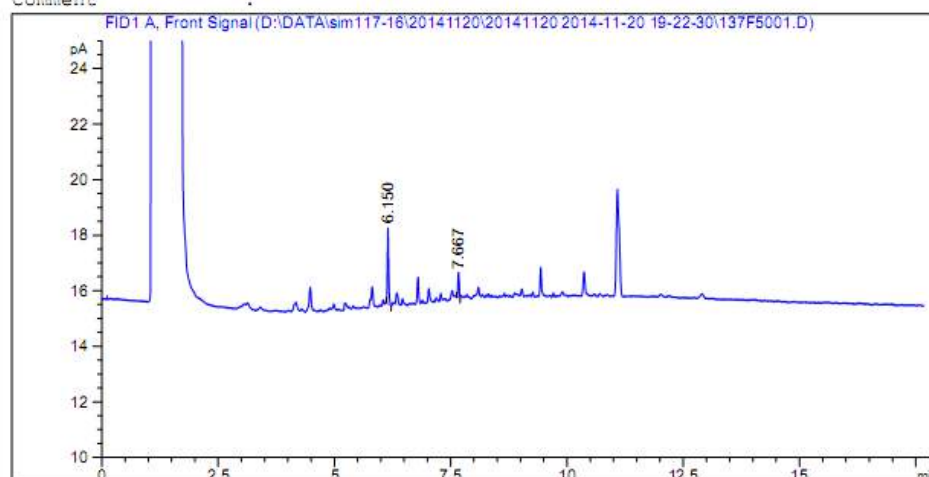
Annex 3-3-33 Validation of analysis procedure for 1,4- butane sultone-Robustness-Condition
2-Sample solution D

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\137F5001.D
Sample Name: D-3.2ml/min

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   45
Acq. Instrument : GC-03                      Location  :   137
Injection Date  : 11/21/2014 10:38:10 AM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114-3.2
                                           .M
Last changed    : 11/20/2014 7:22:32 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
                  \DB-200-20141114-3.2.M (Sequence Method)
Last changed    : 3/31/2015 1:44:19 PM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM             Manufacturer: Agilent
Diameter          : 250.00 µm               Length      :   30.0 m
Film thickness     : 0.25 µm                Void time   :   0.842 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
=====
Multiplier       : 1.0000
Dilution         : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select
							ution	ivity
6.150	6.30	5.03116	2.72416	0.81	0.0278	271560	-	-
7.667	8.10	1.68727	8.98666e-1	1.17	0.0297	368605	31.00	1.29

*** End of Report ***

附图10.4.10-106 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-氮气流速3.2ml/min-样品溶
GC-03 3/31/2015 1:47:35 PM SYSTEM Page 1 of 1

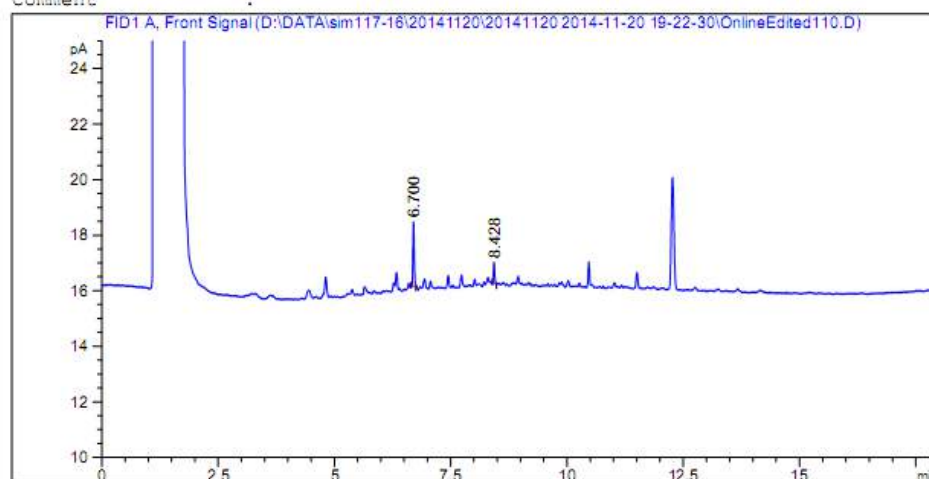
Annex 3-3-34 Validation of analysis procedure for 1,4- butane sultone-Robustness-Condition 3-Sample solution D

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\OnlineEdited110.D
Sample Name: NYX-13C/min-D

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   51
Acq. Instrument : GC-03                      Location  :   142
Injection Date  : 11/21/2014 12:49:05 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114-13.
M
Last changed    : 11/20/2014 7:22:33 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
\DB-200-20141114-13.M (Sequence Method)
Last changed    : 3/31/2015 1:45:12 PM by SYSTEM
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM      Manufacturer: Agilent
Diameter          : 250.00 µm       Length      :   30.0 m
Film thickness     : 0.25 µm        Void time   :   0.877 min
# Injections       :    0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
=====
Multiplier      :    1.0000
Dilution        :    1.0000
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A, Front Signal

RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select ivity
6.700	6.64	4.54732	2.33407	0.98	0.0303	271209	-	-
8.428	8.61	1.55239	7.82992e-1	0.81	0.0308	413873	33.23	1.30

*** End of Report ***

附图10.4.10-111 SBECD中1,4-丁烷磺内酯的测定方法验证图(耐用性-升温速率13°C/min-样品溶液
GC-03 3/31/2015 1:48:24 PM SYSTEM Page 1 of 1

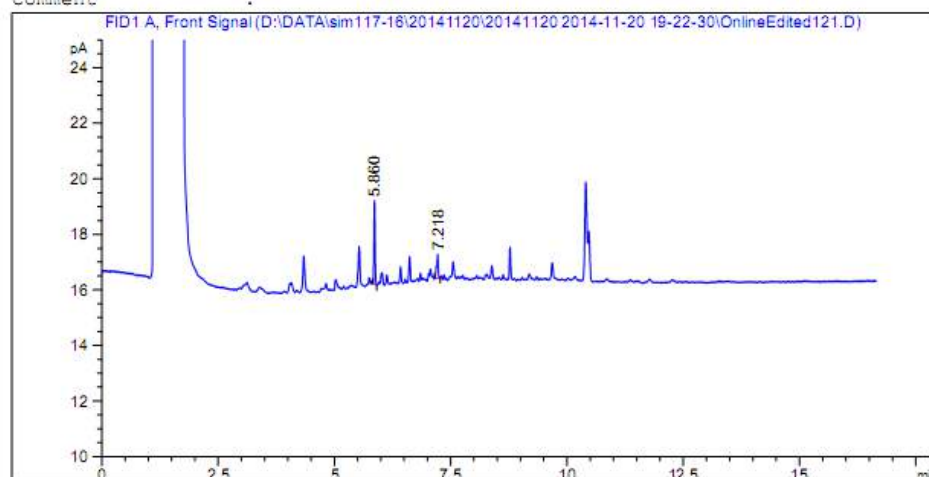
Annex 3-3-35 Validation of analysis procedure for 1,4- butane sultone-Robustness-Condition 4-Sample solution D

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\OnlineEdited121.D
Sample Name: NYX-17C/min-D

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   57
Acq. Instrument : GC-03                      Location  :   142
Injection Date  : 11/21/2014 2:49:41 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114-17.
M
Last changed    : 11/21/2014 2:02:39 PM by SYSTEM
Analysis Method : E:\GC-BACKUP\20150227\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30
\DB-200-20141114-17.M (Sequence Method)
Last changed    : 3/31/2015 1:48:45 PM by SYSTEM
(modified after loading)
=====
```

Column(s)

```
=====
Column Description : DB-200
Inventory#         : autoID-1
Model#            : 122-2032LTM      Manufacturer: Agilent
Diameter          : 250.00 µm      Length       : 30.0 m
Film thickness     : 0.25 µm       Void time    : 0.877 min
# Injections       : 0
Maximum Temperature: 300.0 °C
Comment           :
=====
```



Area Percent Report with Performance

```
Multiplier      : 1.0000
Dilution        : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

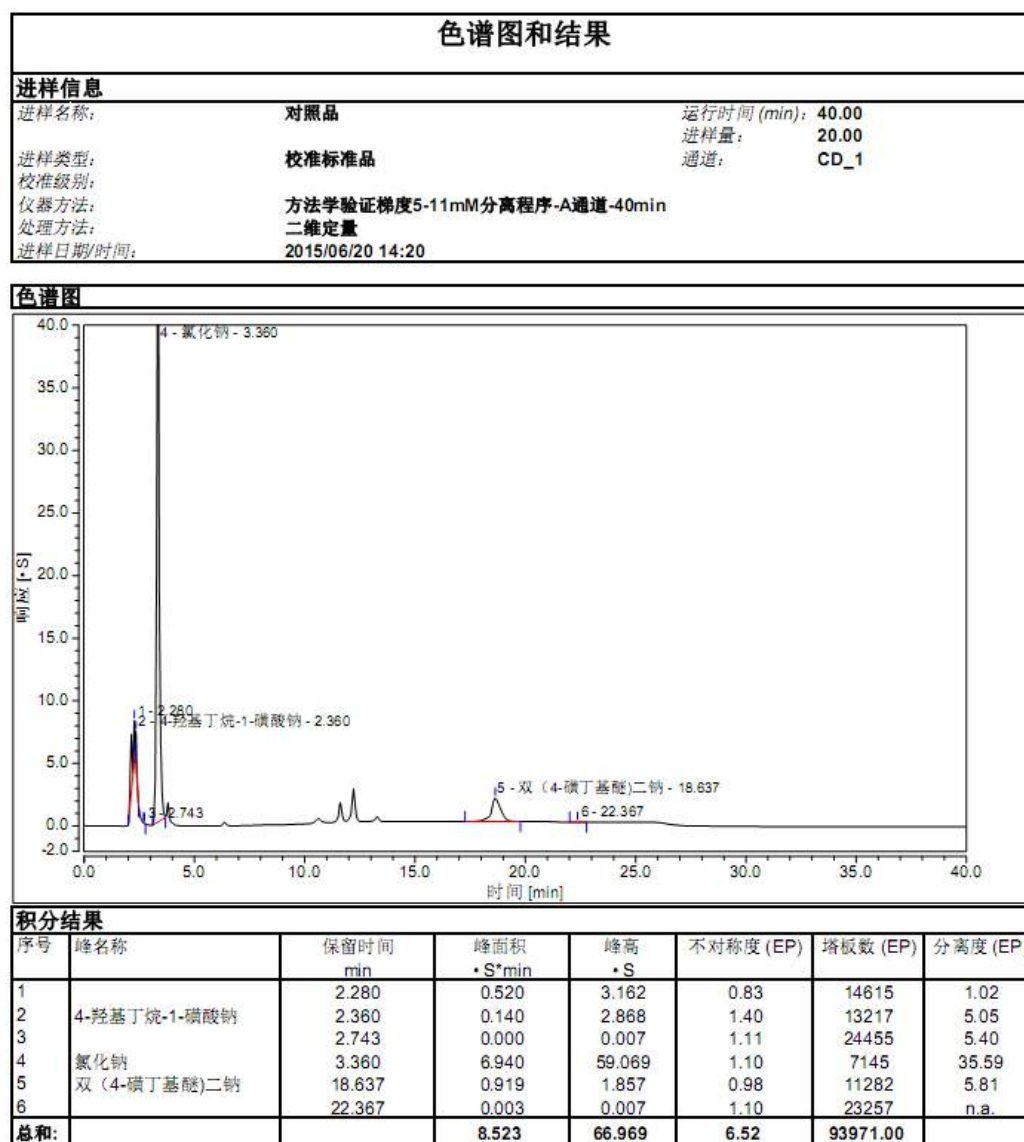
RetTime [min]	k'	Area [pA*s]	Height [pA]	Symm.	Width [min]	Plates	Resol	Select
							ution	ivity
5.860	5.68	4.93833	3.01460	0.88	0.0258	285066	-	-
7.218	7.23	2.11325	8.56847e-1	1.89	0.0292	339321	29.02	1.27

附图10.4.10-116 SBEC中1,4-丁烷磺内酯的测定方法验证图(耐用性-升温速率17°C/min-样品溶液)
GC-03 3/31/2015 1:49:20 PM SYSTEM Page 1 of 2

Annex 3-4-1 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium -Specificity-Reference solution

仪器:ICS-5000+ 序列:专属性

页码 1 / 7



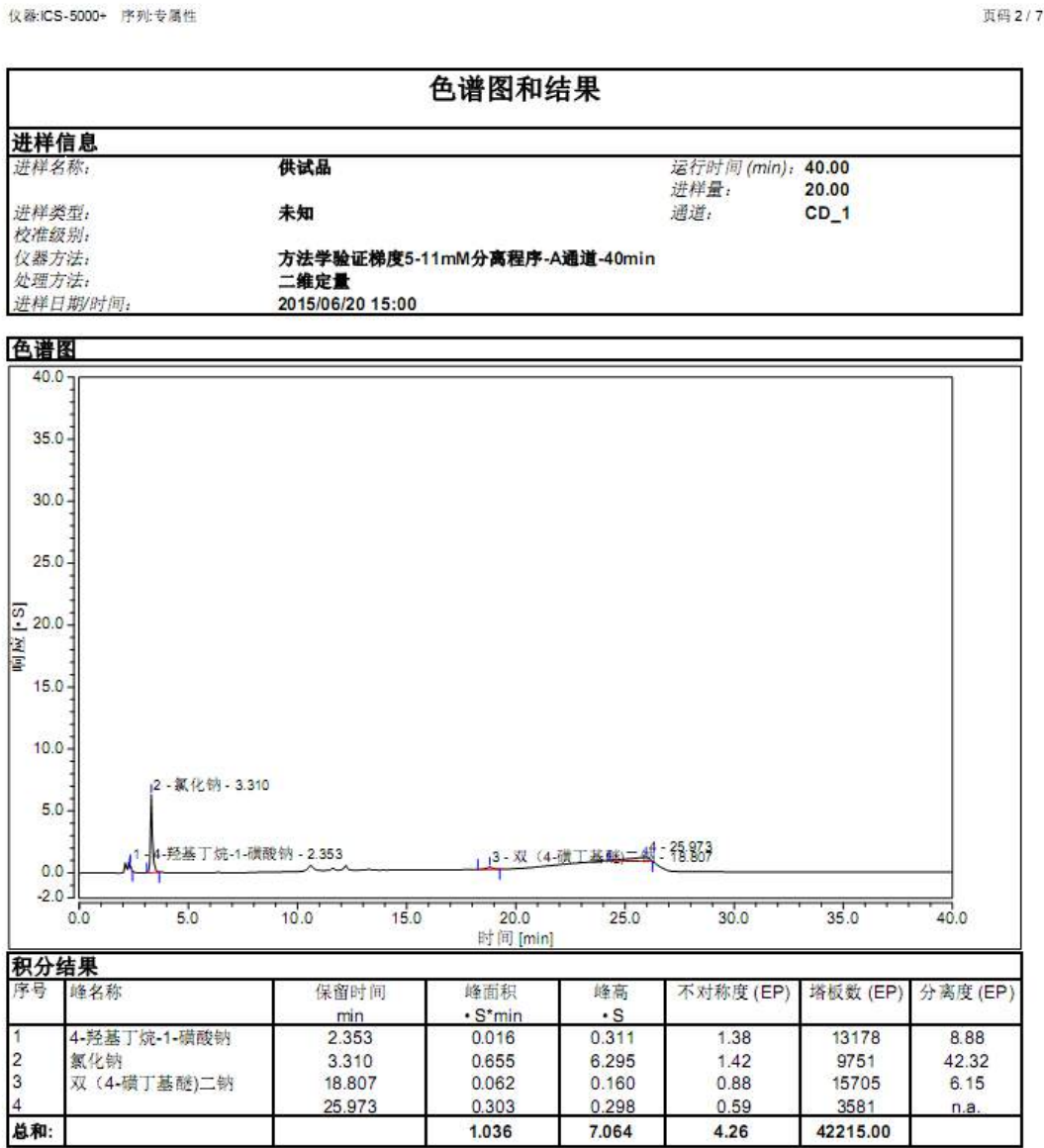
附图10.4.11-169

SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(专属性-对照品)

Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-2 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium -Specificity-Sample solution

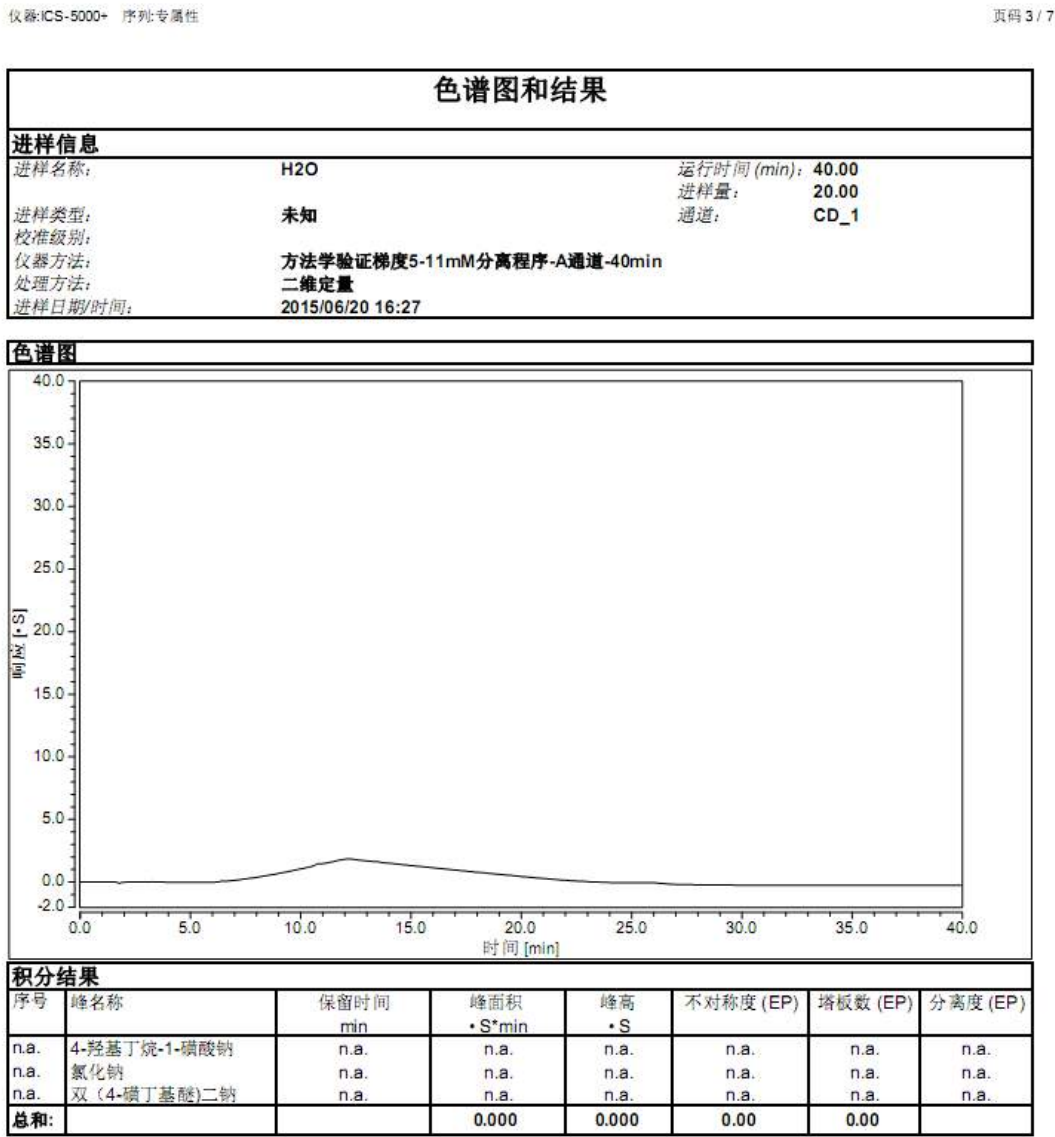


附图10.4.11-170 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(专属性-供试品)

Default/积分

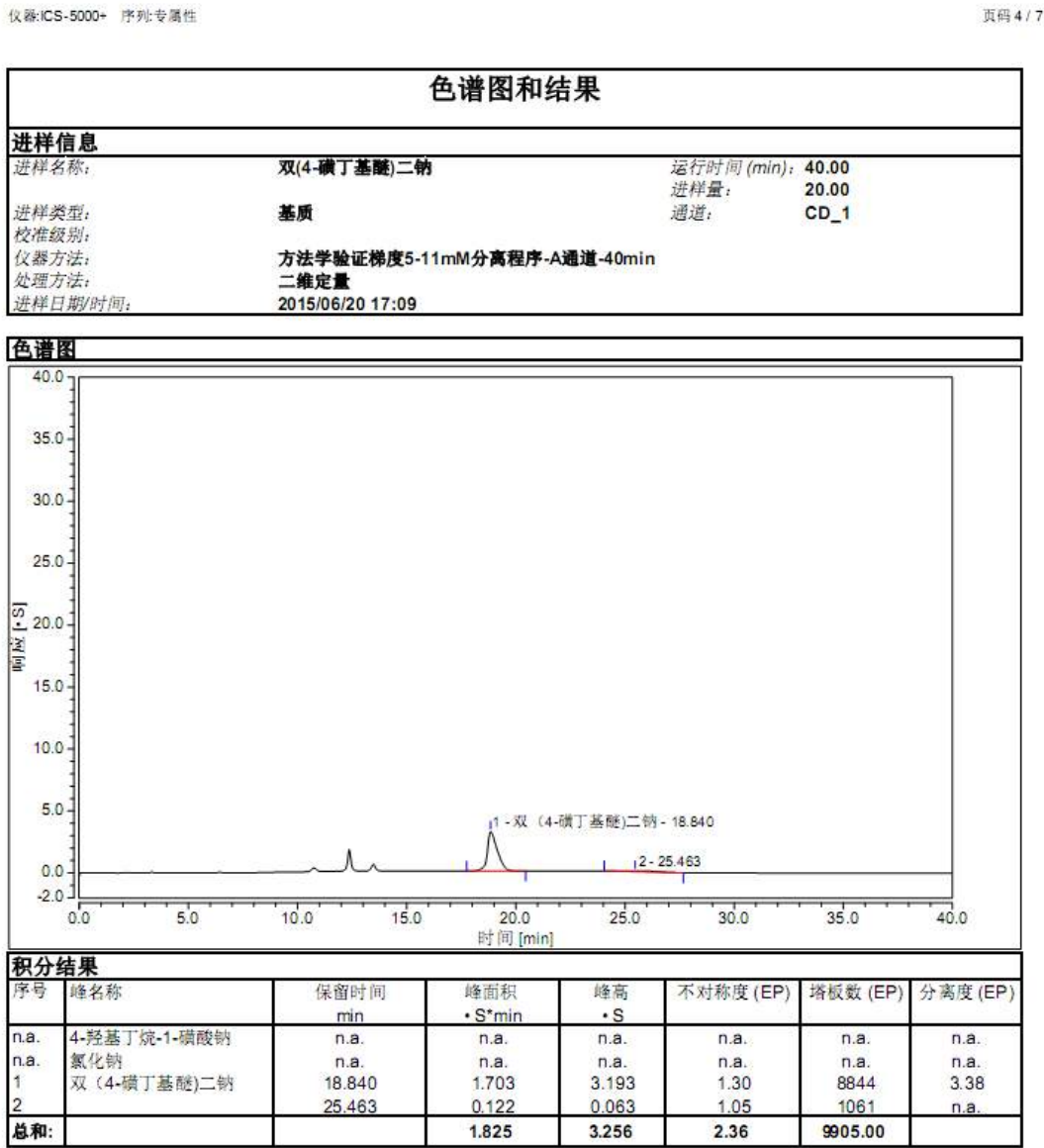
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-3 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium -Specificity-Blank solution



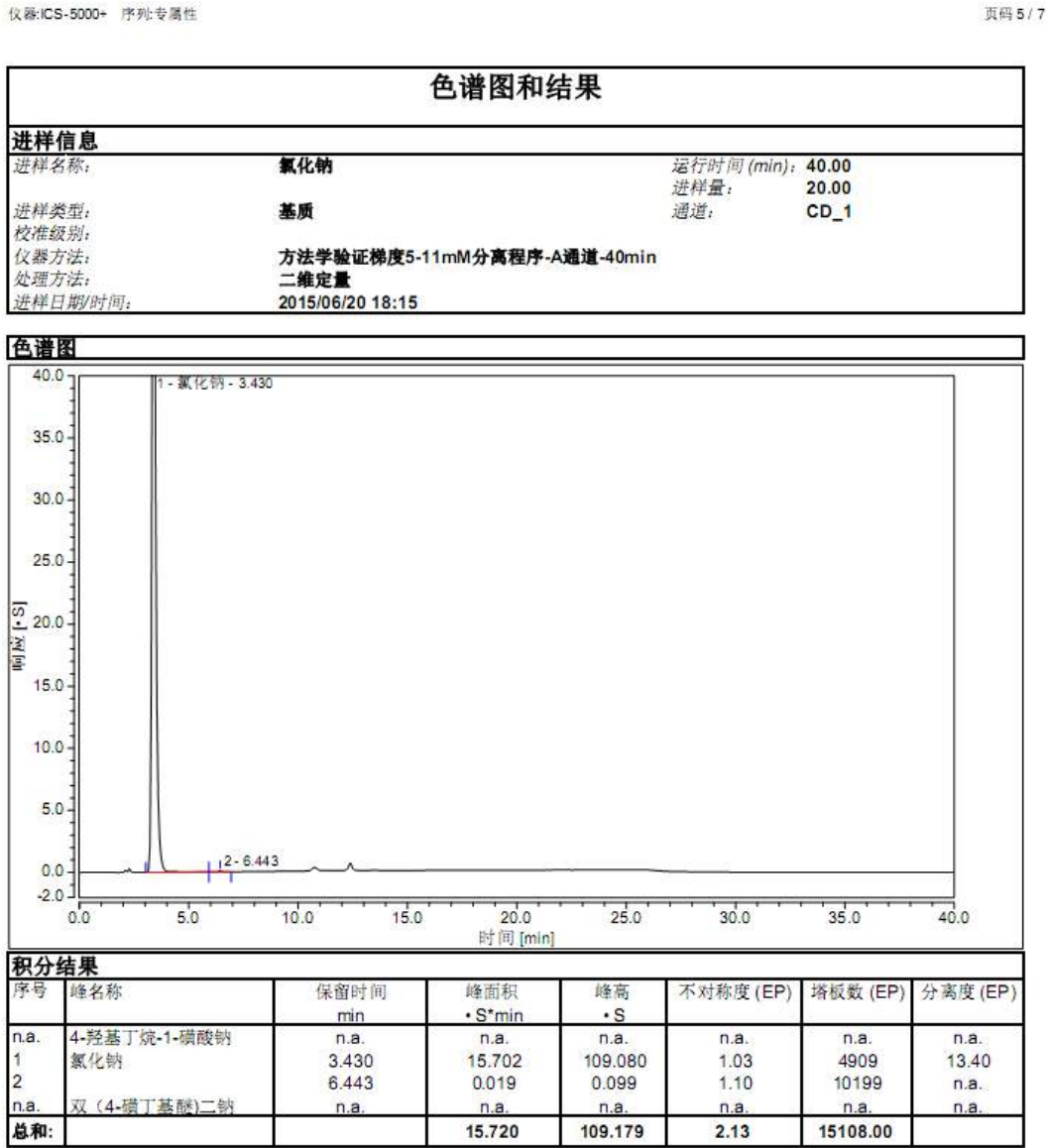
附图10.4.11-168 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双 (4-磺丁基) 醚二钠的测定方法验证图 (专属性-空白溶剂)

Annex 3-4-4 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Specificity-Bis(4-sulfobutyl) ether disodium



附图10.4.11-173 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双 (4-磺丁基) 醚二钠的测定方法验证图 (专属性-双 (4-磺丁基) 醚二钠溶剂)

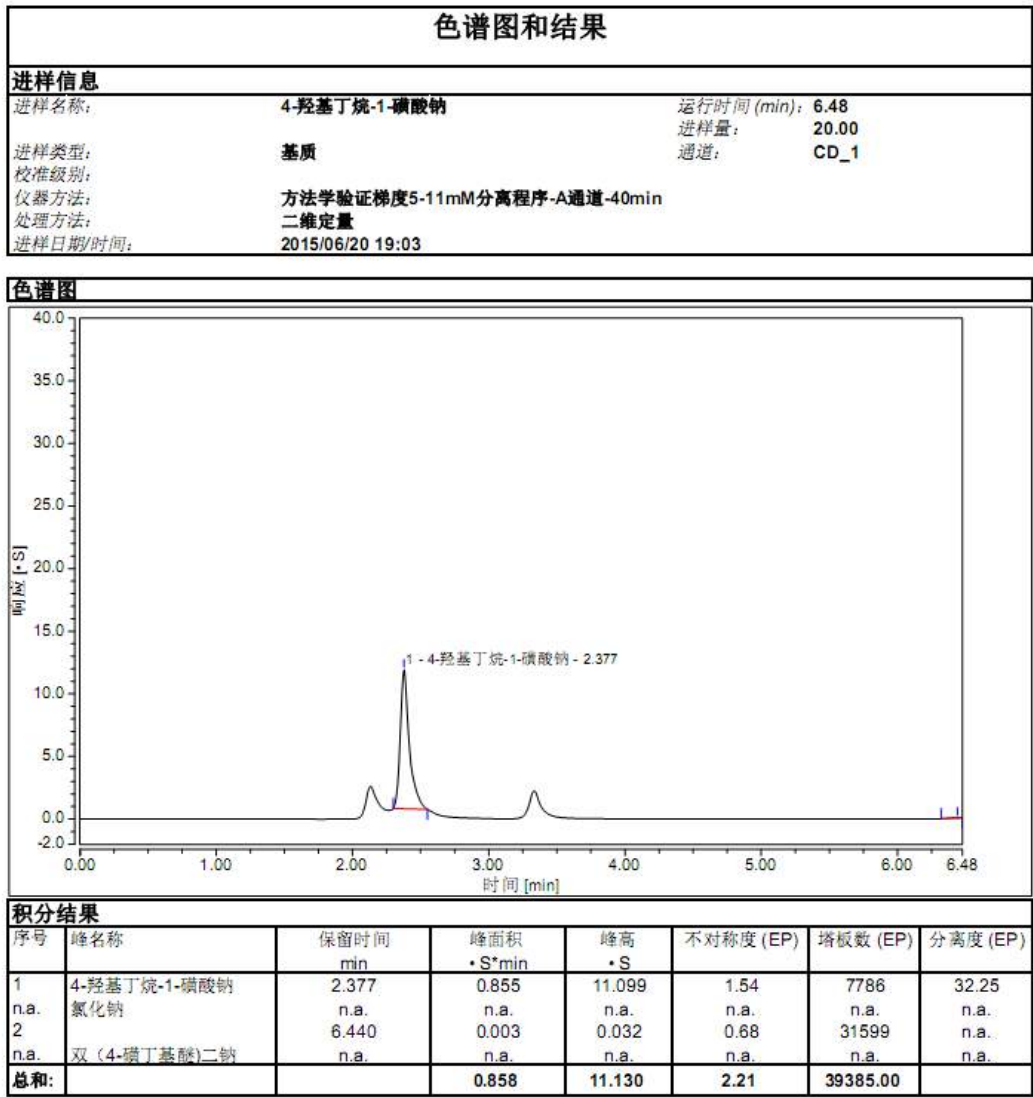
Annex 3-4-5 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Specificity- Sodium chloride



附图10.4.11-172 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(专属性-氯化钠溶剂)

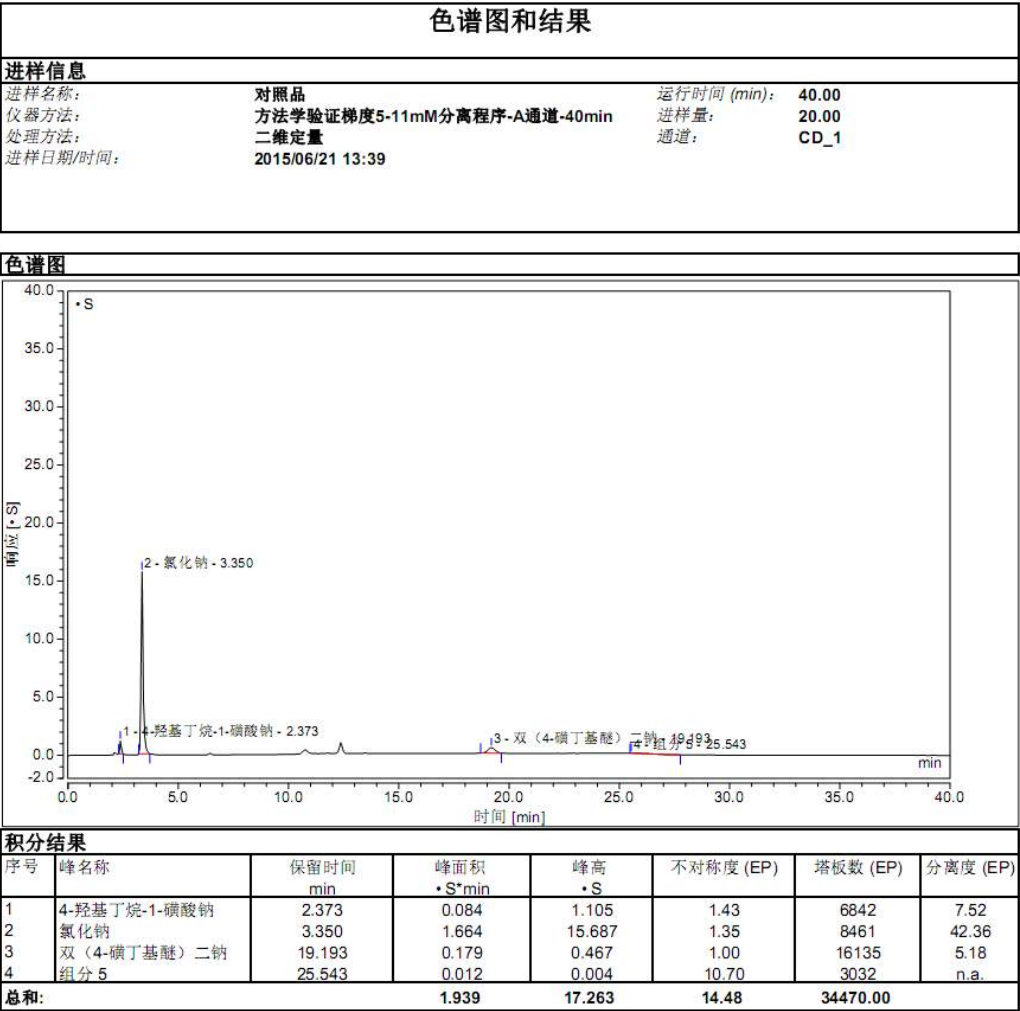
Annex 3-4-6 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Specificity-4-hydroxybutane-1-sulfonic acid

仪器:ICS-5000+ 序列:专属性 页码 6 / 7



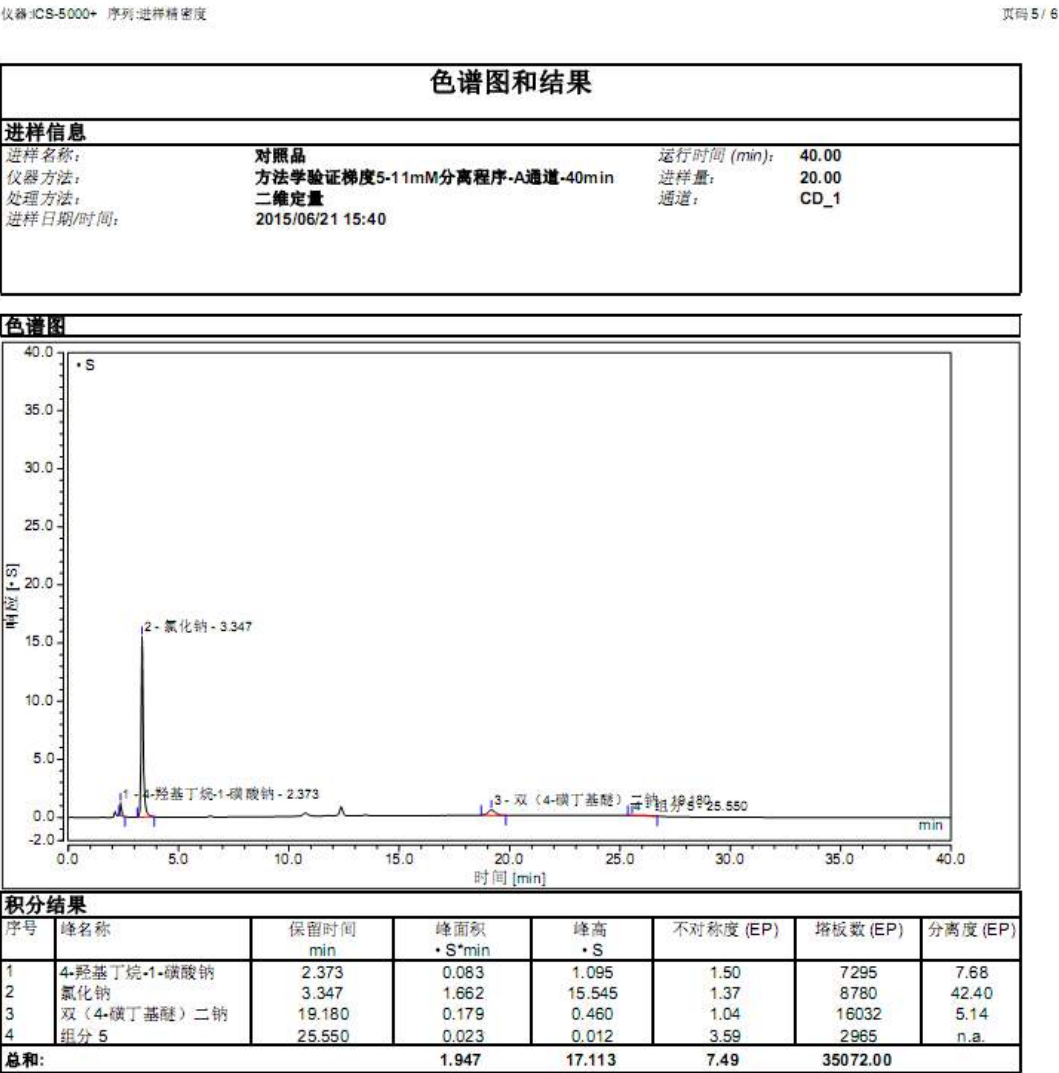
附图10.4.11-171 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双 (4-磺丁基) 醚二钠的测定方法验证图 (专属性-4-羟基丁烷-1-磺酸溶剂)

Annex 3-4-7 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-System suitability-Solution 2



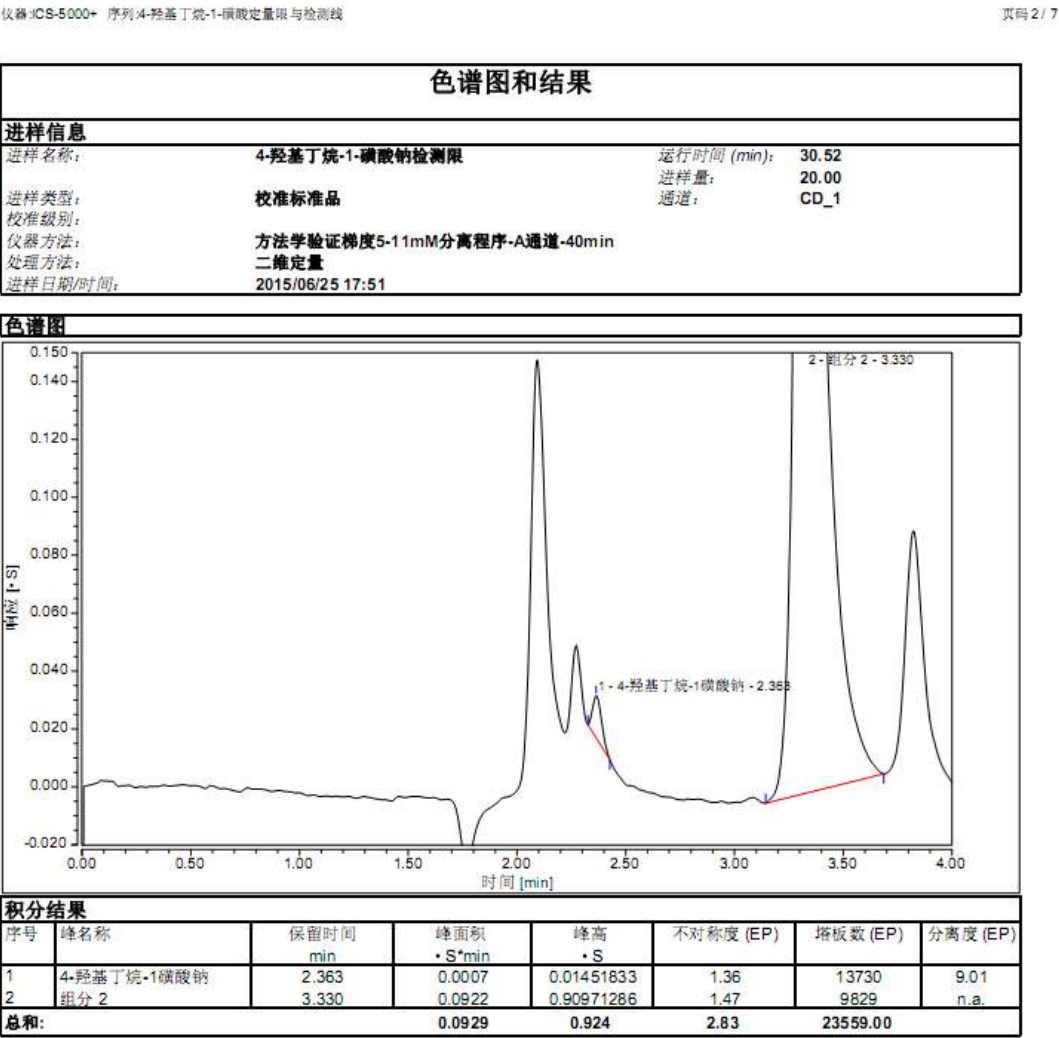
附图10.4.11-175 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（进样精密度-2）

Annex 3-4-8 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-System suitability-Solution 5



附图10.4.11-178 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（进样精密度-5）

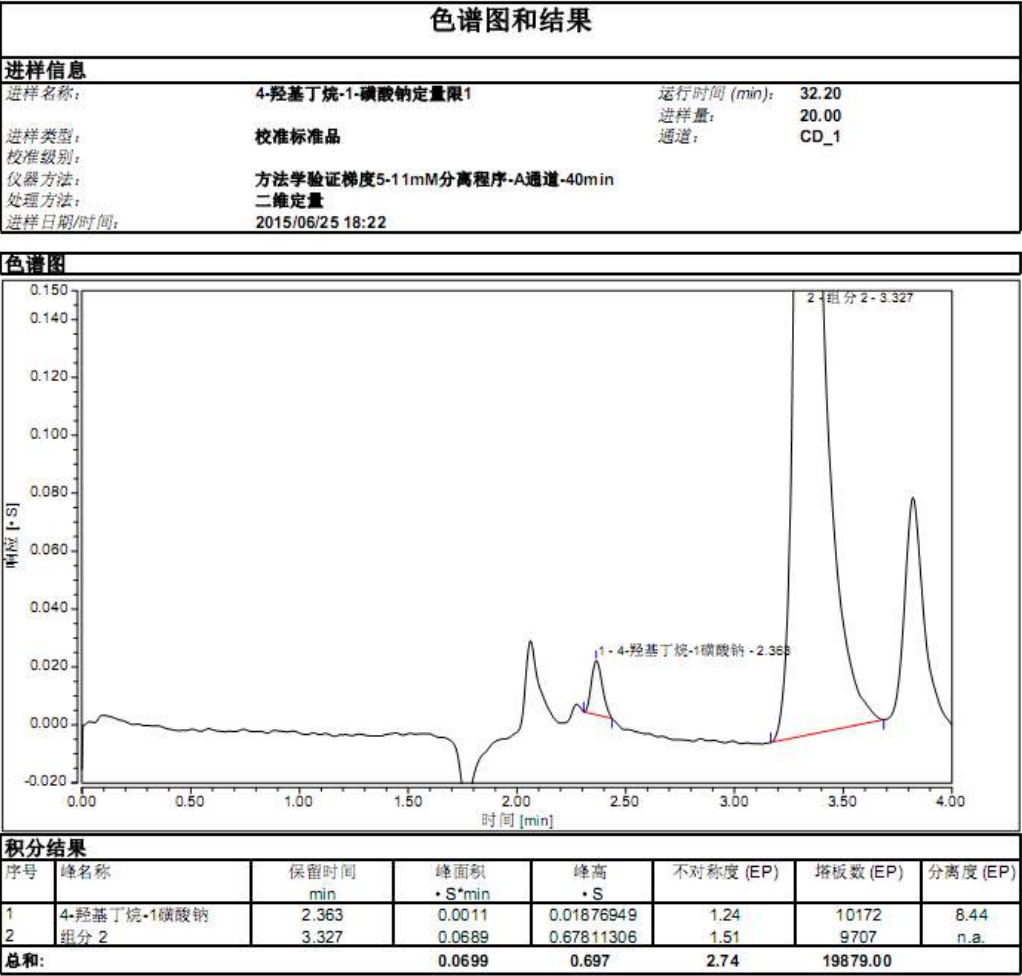
Annex 3-4-9 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-LOD and LOQ-LOD of 4-hydroxybutane-1-sulfonic acid



附图10.4.11-181 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（4-羟基丁烷-1-磺酸检测限Jcx-1）

Annex 3-4-10 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-LOD and LOQ-LOQ of 4-hydroxybutane-1-sulfonic acid

仪器:ICS-5000+ 序列:4-羟基丁烷-1-磺酸定量限与检测线 页码 3 / 7



附图10.4.11-182 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（4-羟基丁烷-1-磺酸定量限-1，线性）

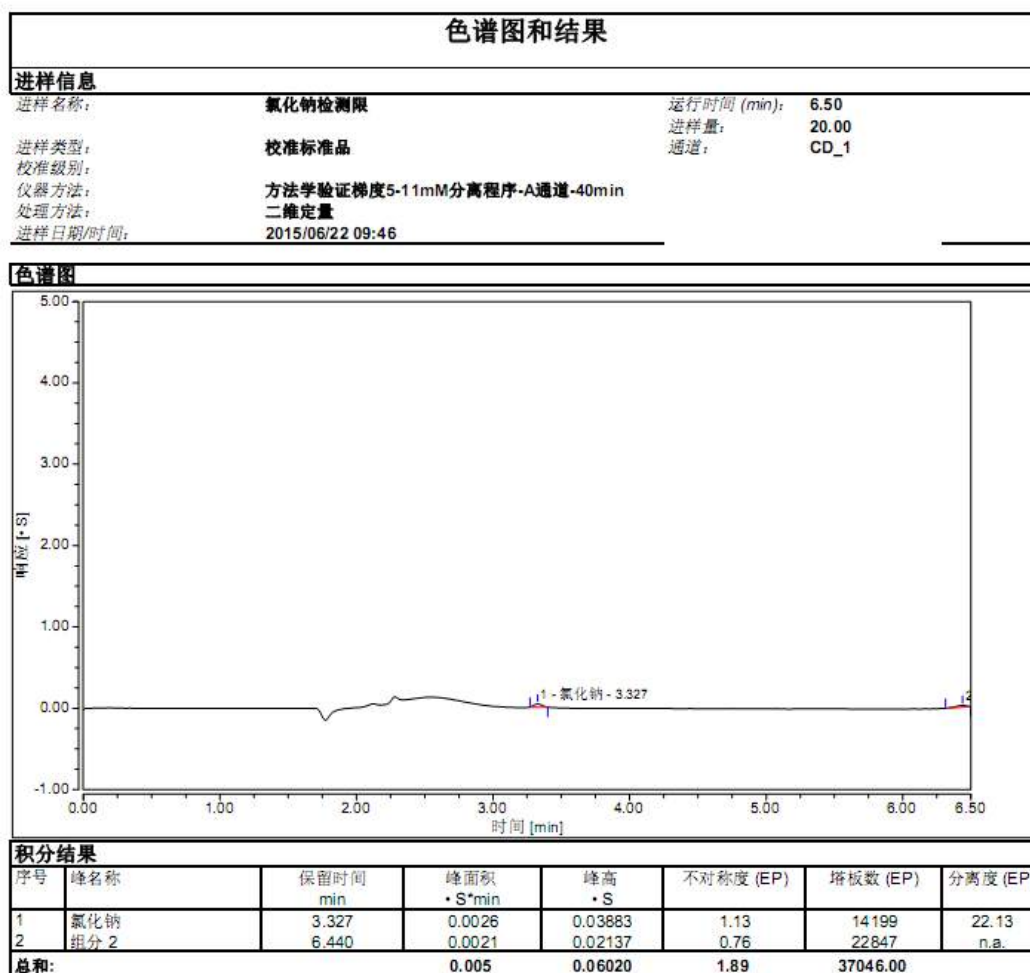
Default/积分

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版本 7.2.1.5537

Annex 3-4-11 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-LOD and LOQ-LOD of Sodium chloride

仪器:ICS-5000+ 序列:氯化钠检测限

页码 2 / 7



附图10.4.11-187 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(氯化钠检测限)

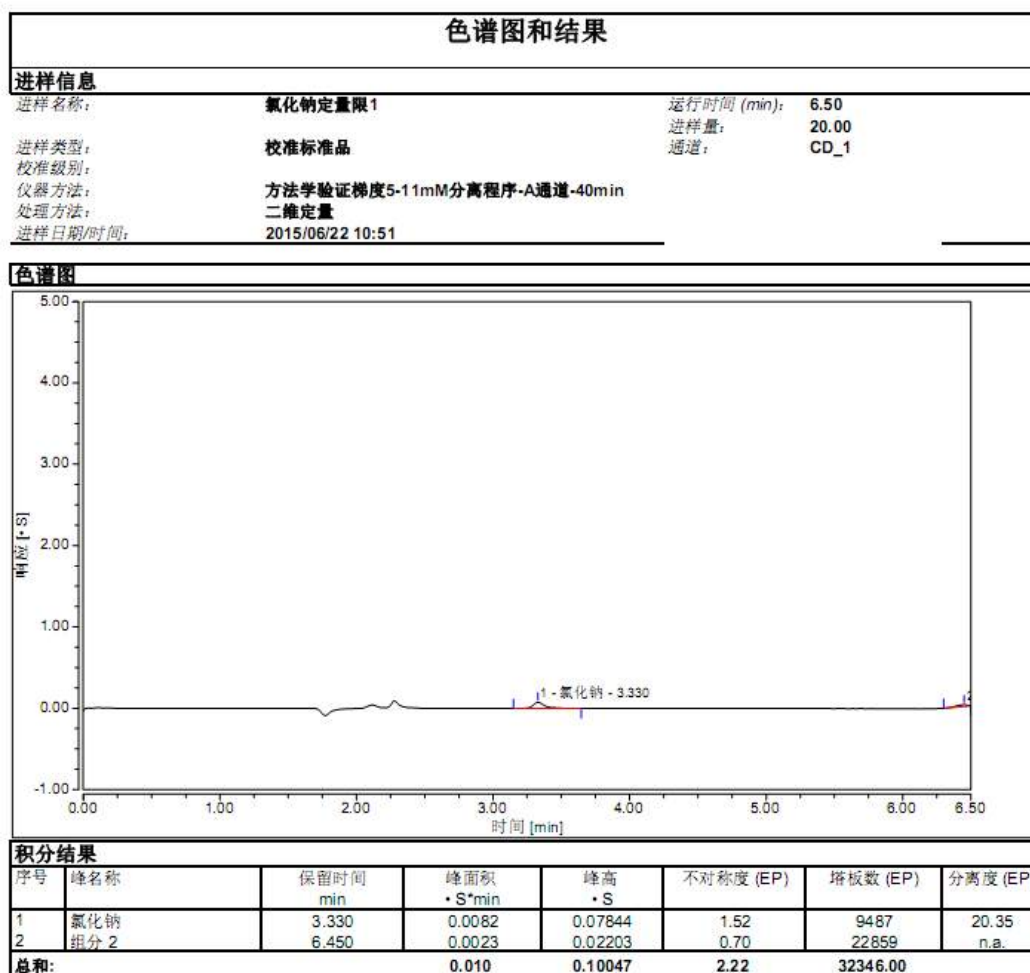
Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-12 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-LOD and LOQ-LOQ of Sodium chloride

仪器:ICS-5000+ 序列:氯化钠定量限

页码 3 / 7



附图10.4.11-188 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(氯化钠定量限-1, 线性)

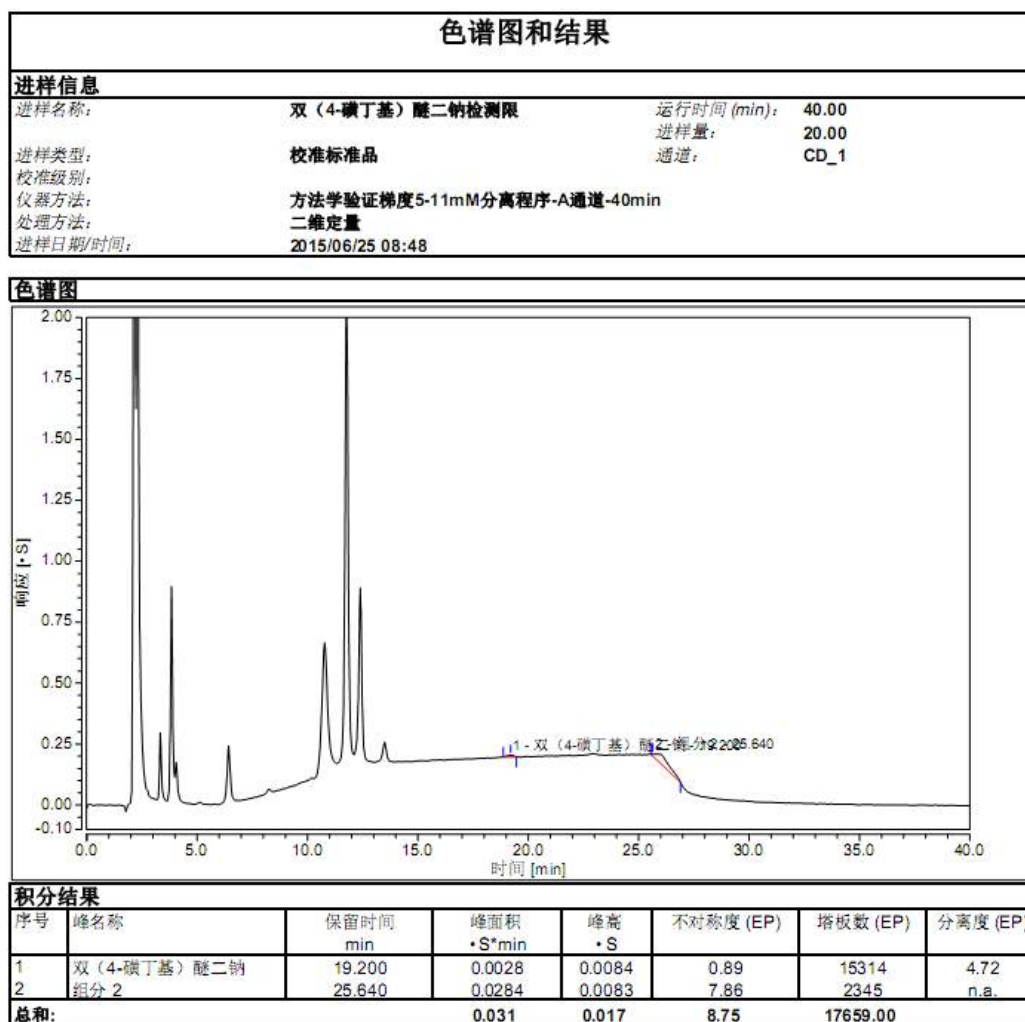
Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-13 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-LOD and LOQ-LOD of Bis(4-sulfobutyl) ether disodium

仪器: ICS-5000+ 序列: 双(4-磺丁基)醚二钠

页码 6 / 6



附图10.4.11-193 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(双(4-磺丁基)醚二钠检测限)

Default/积分

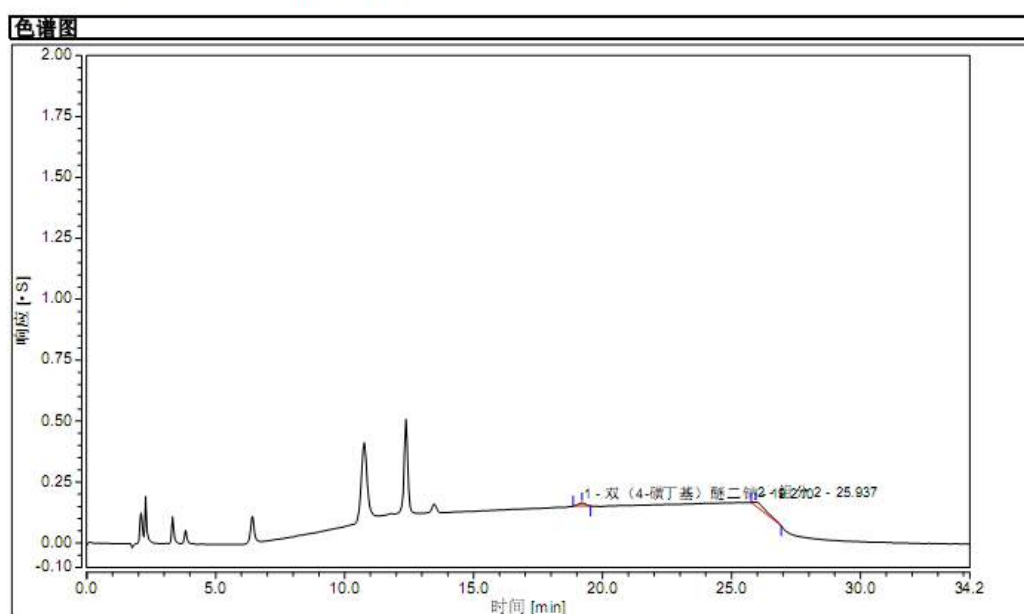
Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-14 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-LOD and LOQ-LOQ of Bis(4-sulfobutyl) ether disodium

仪器: ICS-5000+ 序列: 双(4-磺丁基)醚二钠

页码 5/6

色谱图和结果			
进样信息			
进样名称:	双(4-磺丁基)醚二钠定量限5	运行时间 (min):	34.24
进样类型:	校准标准品	进样量:	20.00
校准级别:		通道:	CD_1
仪器方法:	方法学验证梯度5-11mM分离程序-A通道-40min		
处理方法:	二维定量		
进样日期/时间:	2015/06/24 17:40		



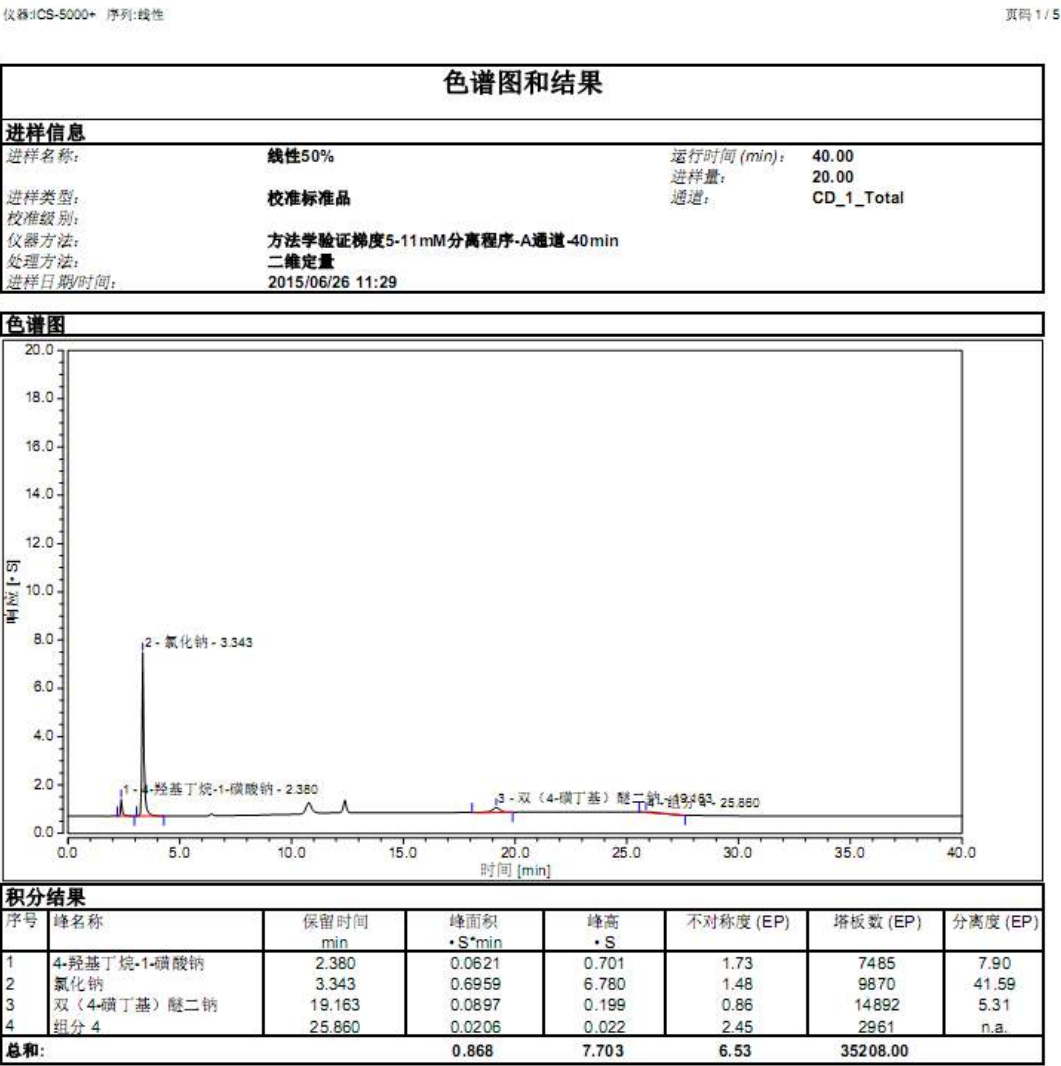
积分结果							
序号	峰名称	保留时间 min	峰面积 •S*min	峰高 •S	不对称度 (EP)	塔板数 (EP)	分离度 (EP)
1	双(4-磺丁基)醚二钠	19.210	0.0045	0.0135	0.94	19117	7.86
2	组分 2	25.937	0.0123	0.0153	3.49	7991	n.a.
总和:			0.017	0.029	4.43	27108.00	

附图10.4.11-198 SBEC中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(双(4-磺丁基)醚二钠定量限-5)

Default/积分

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Annex 3-4-15 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Linearity-50%

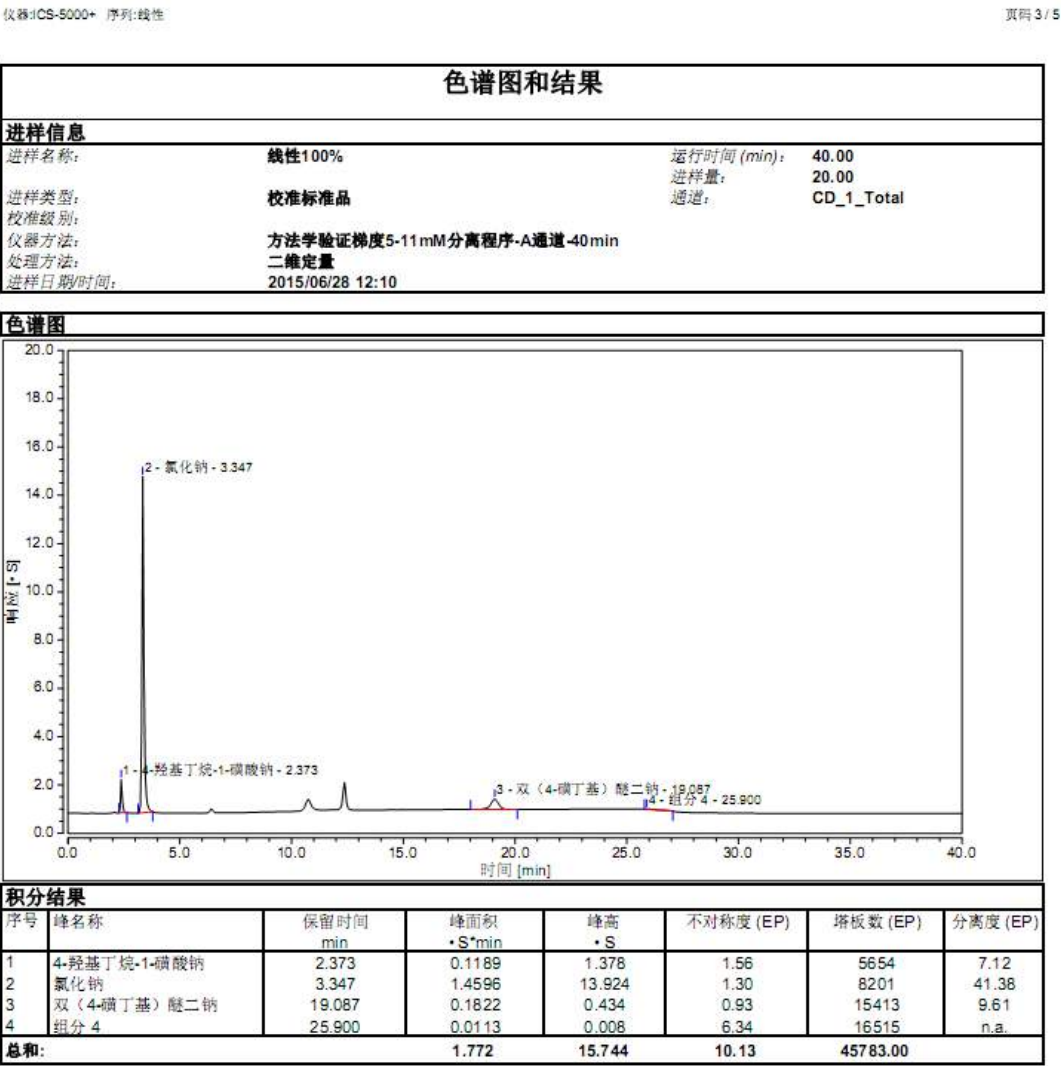


附图 I0.4.11-199 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(线性-50%)

Default积分

Chromaleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-16 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Linearity-100%

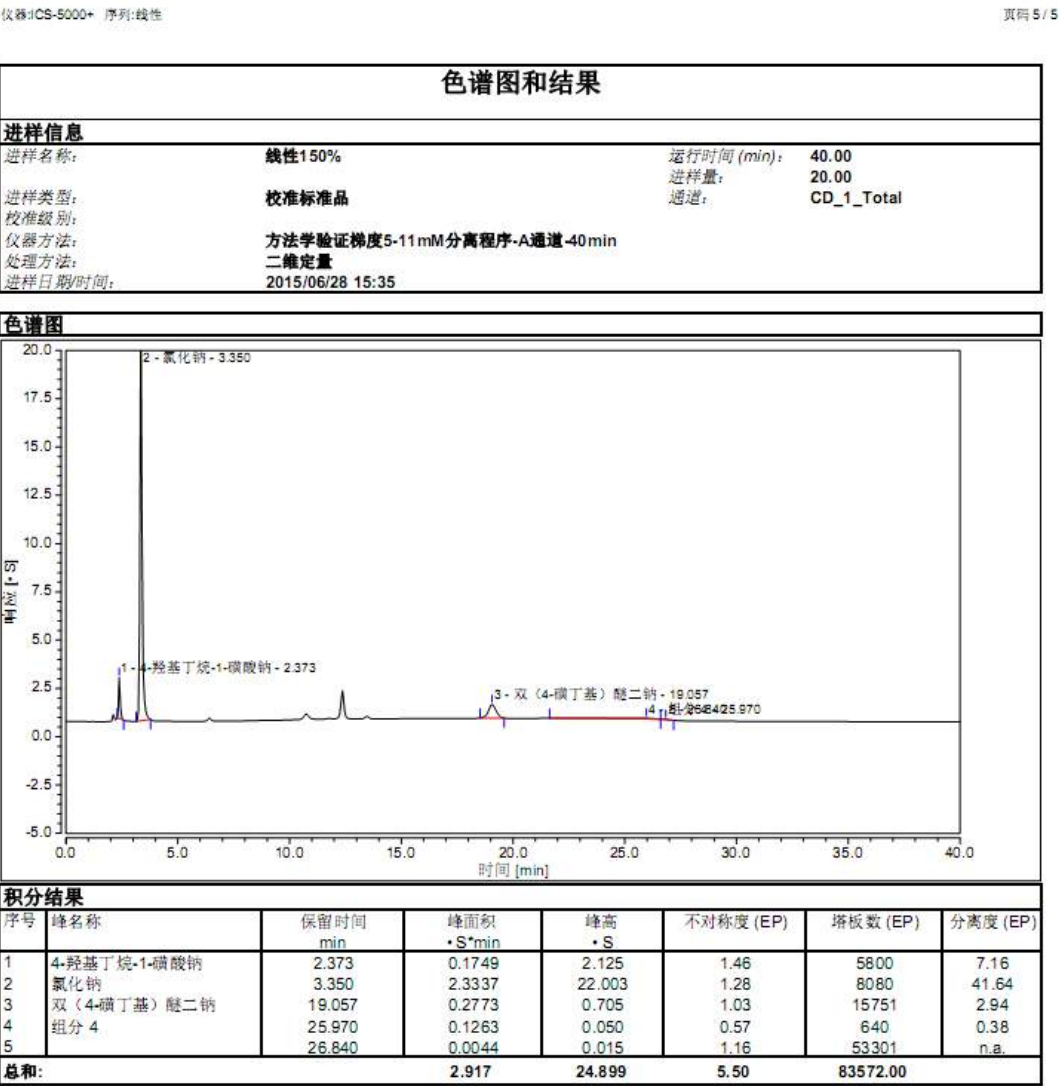


附图10.4.11-201 S BECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(线性-100%)

Default积分

Chromaleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-17 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Linearity-150%

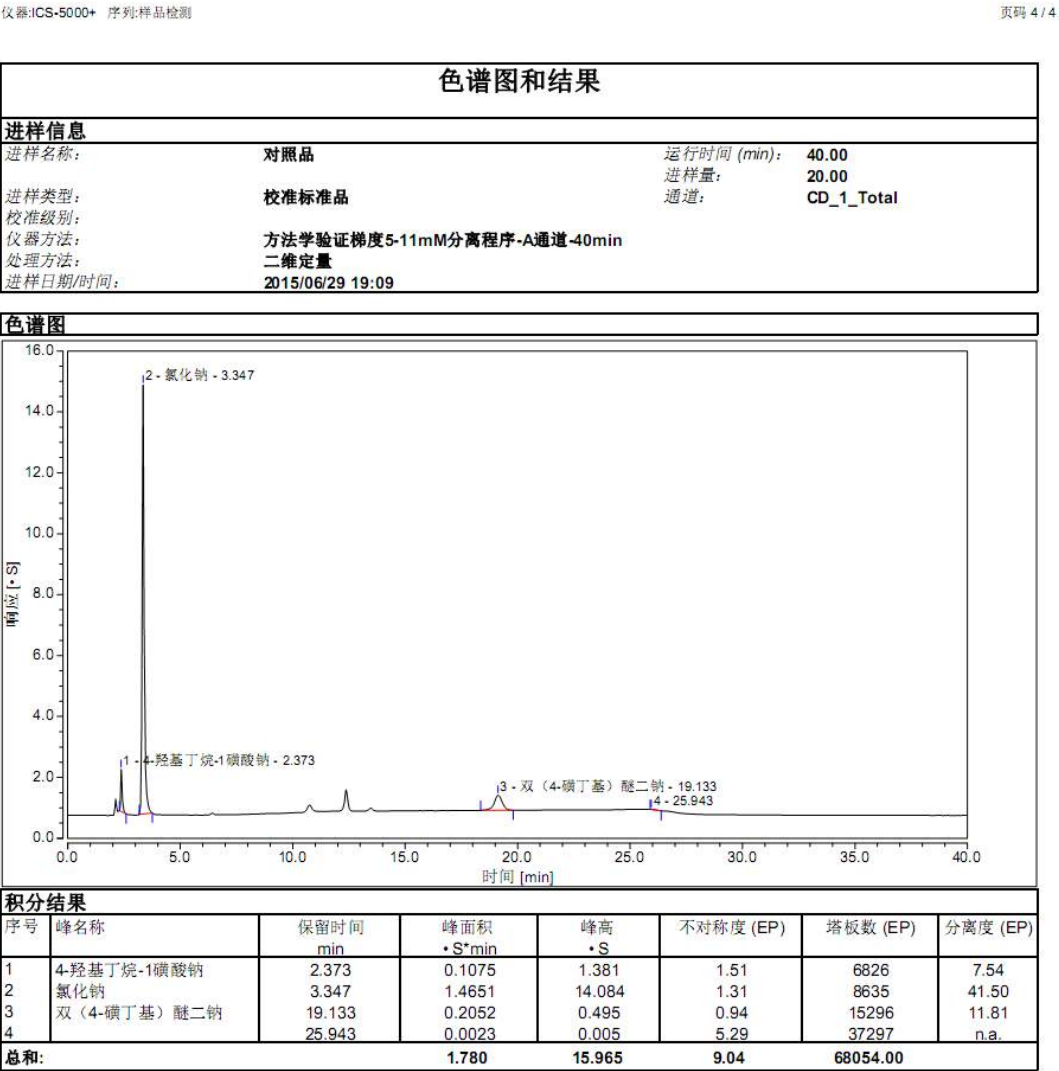


附图10.4.11-203 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(线性:150%)

Default积分

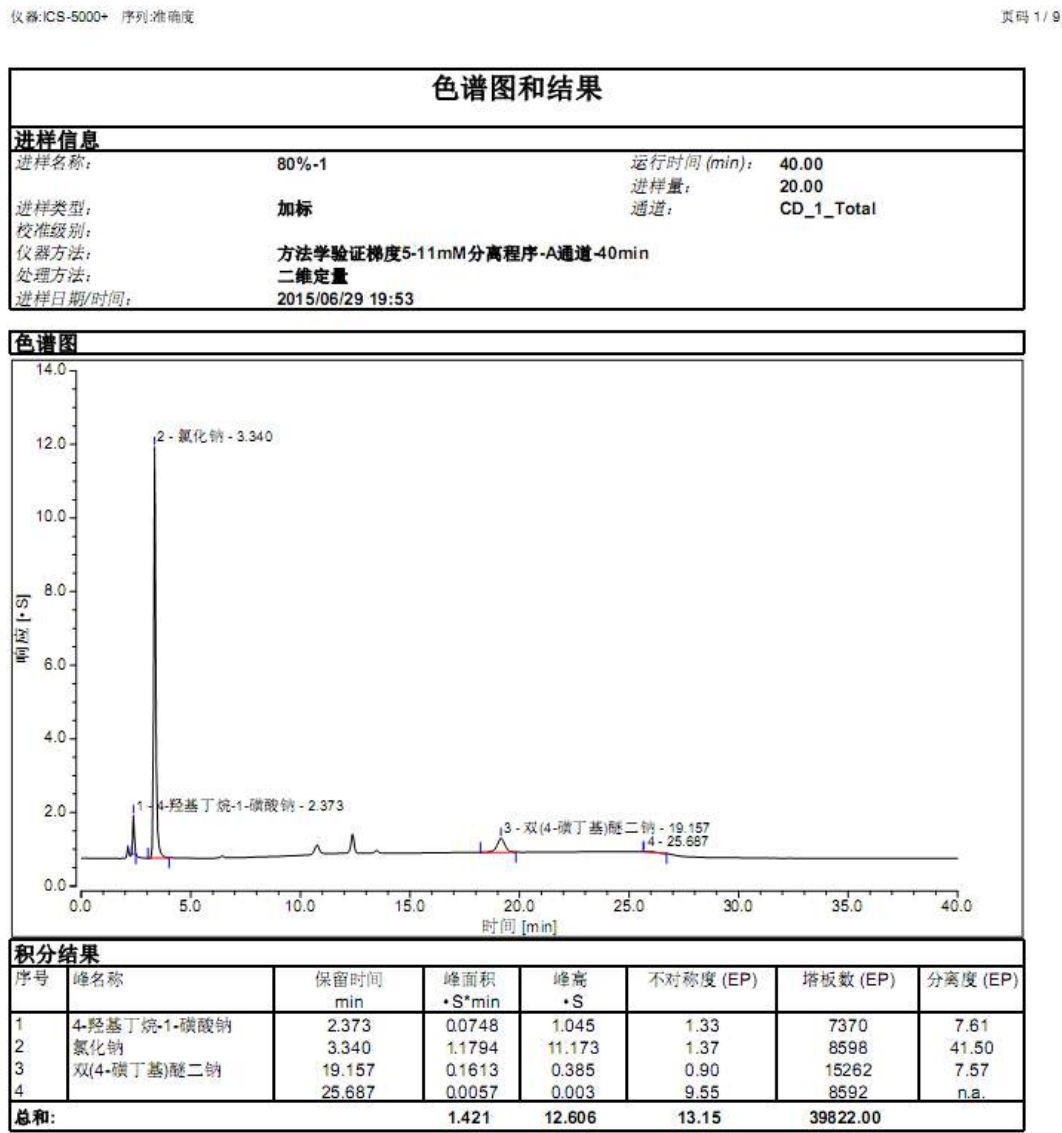
Chromaleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-18 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Accuracy-Reference solution



附图10.4.11-263 SBECD-0时4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定图(对照品)

Annex 3-4-19 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Accuracy-Sample solution 80%-1

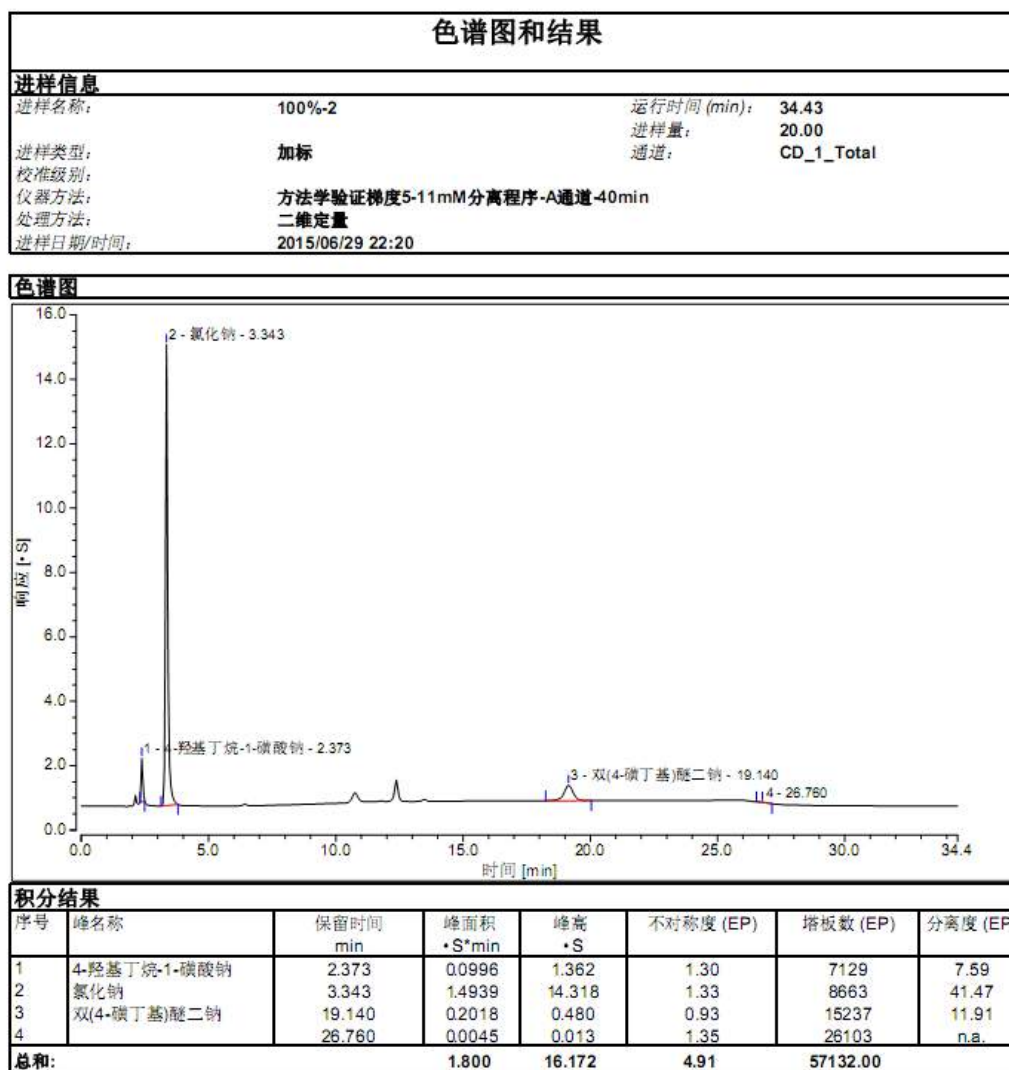


附图10. 4. 11-310CD中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（准确度-8 0 %-1

Annex 3-4-20 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Accuracy- Sample solution 100%-2

仪器: ICS-5000+ 序列: 准确度

页码 5/ 9

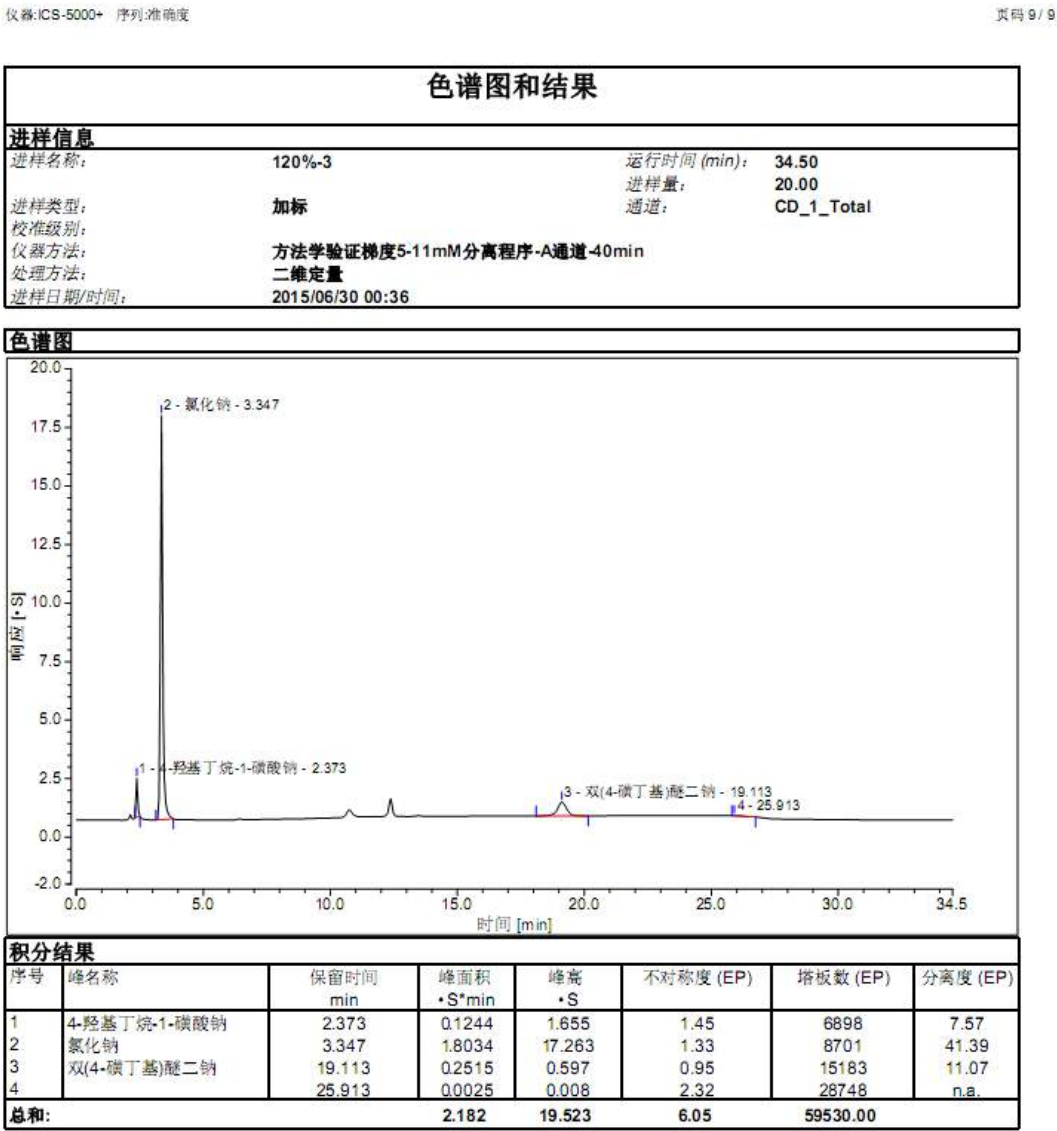


附图10. 4. 11-930(8)中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(准确度-100%-2)

Default/积分

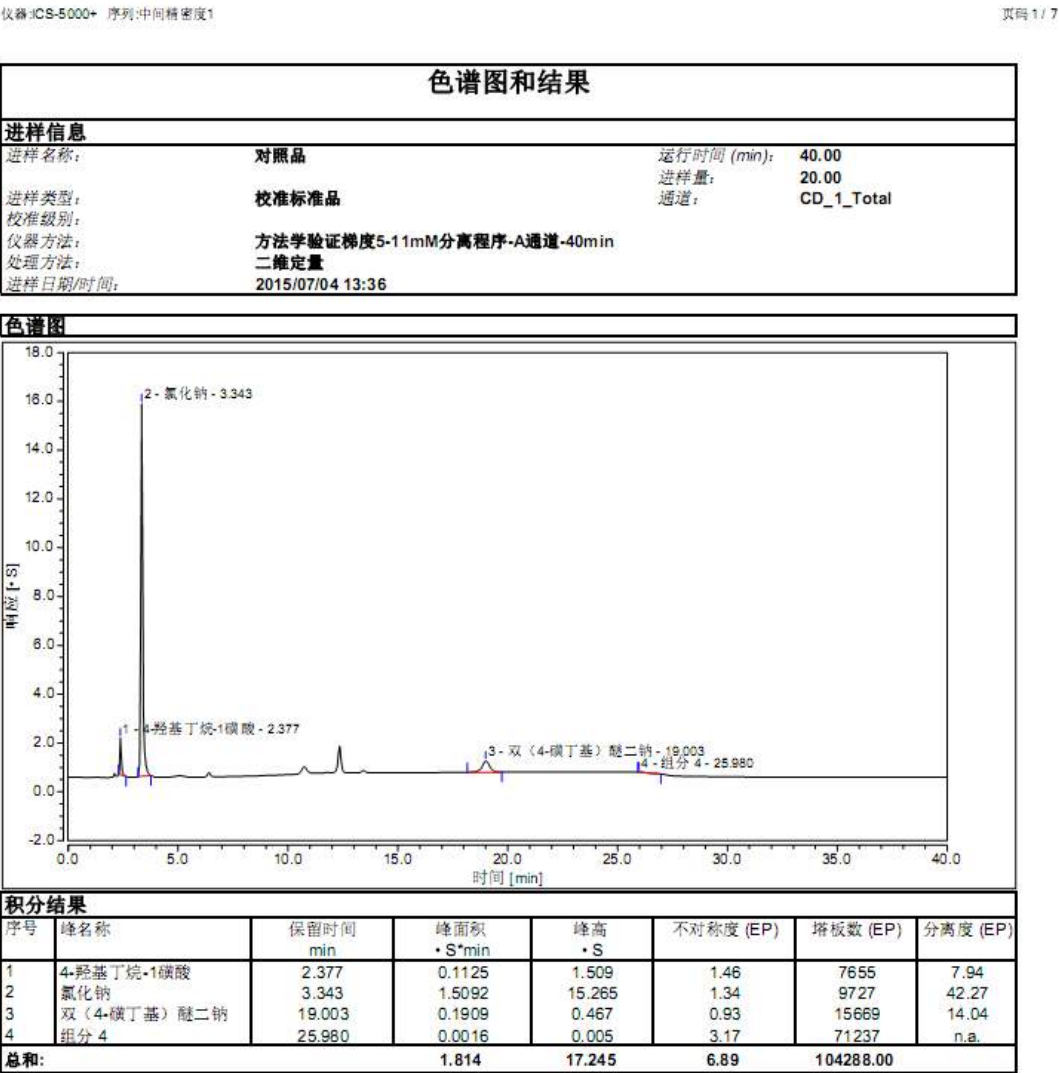
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-21 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Accuracy- Sample solution 120%-3



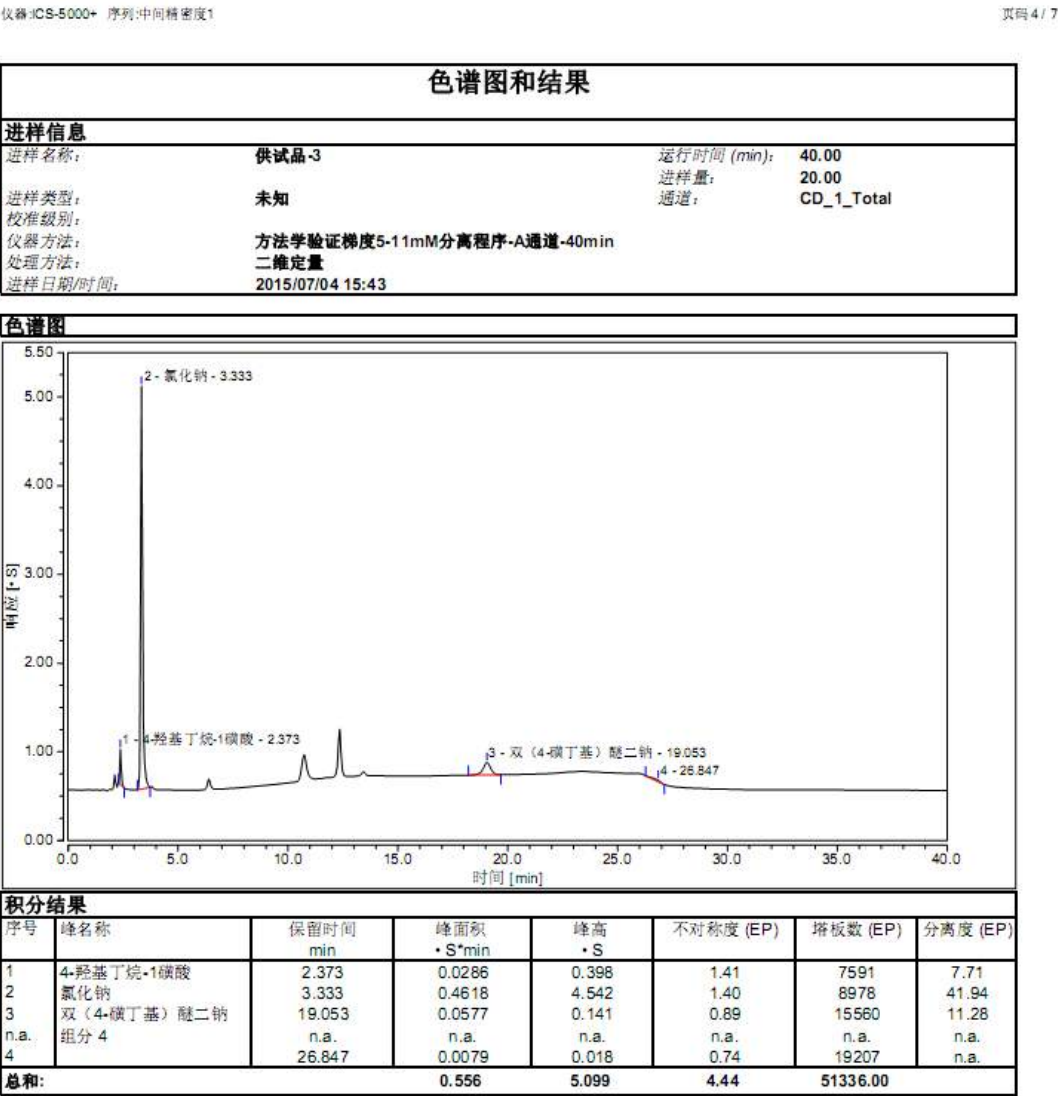
附图10. 4. 11-3BE2D中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（准确度-120%-3

Annex 3-4-22 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Intermediate precision
1- Reference solution



附图10. 4. 11-3113D中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(中间精密度1-对照品)

Annex 3-4-23 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Intermediate precision 1- Sample solution -3

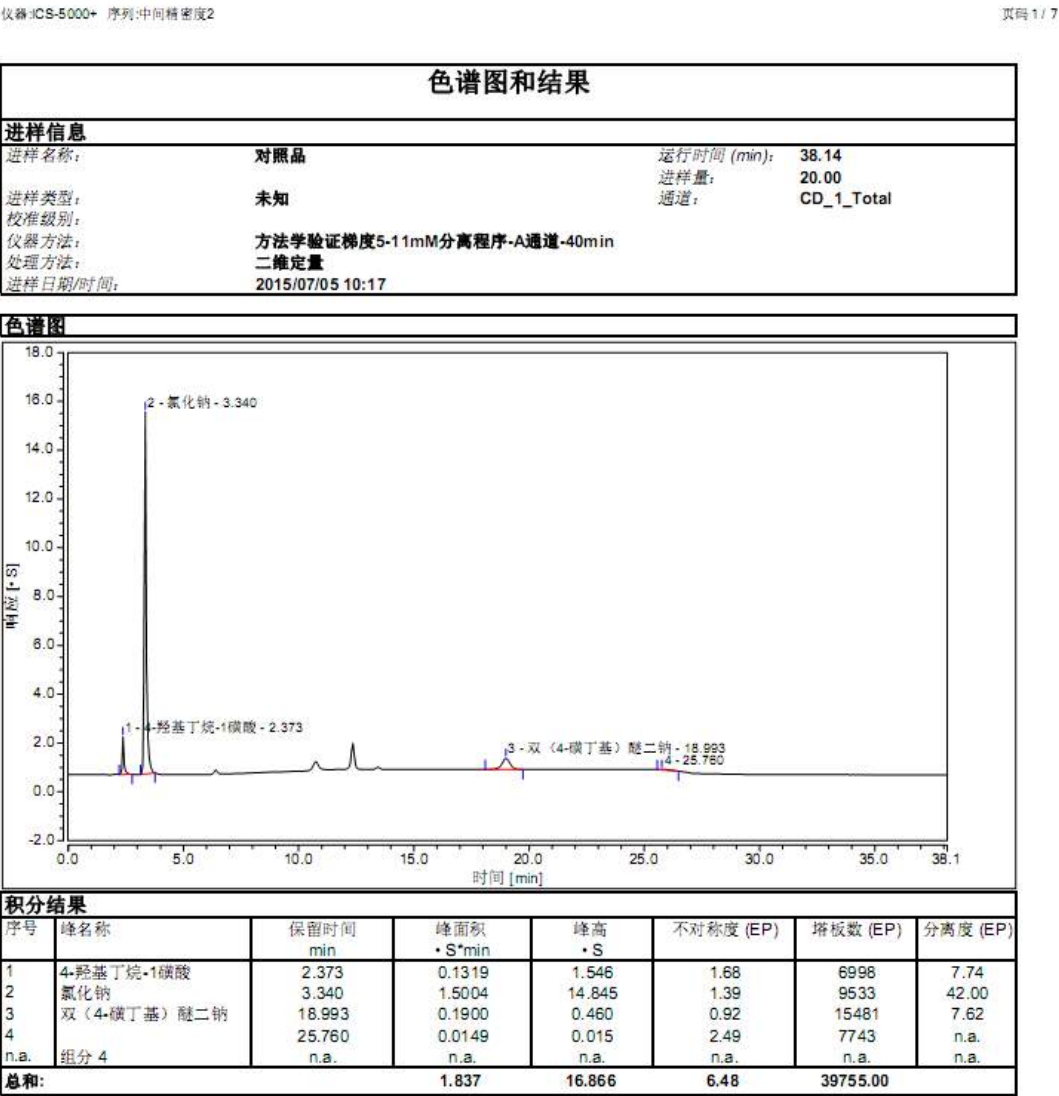


附图10. 4. 11-33E6D中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(中间精密度1-供试品-3)

1/积分

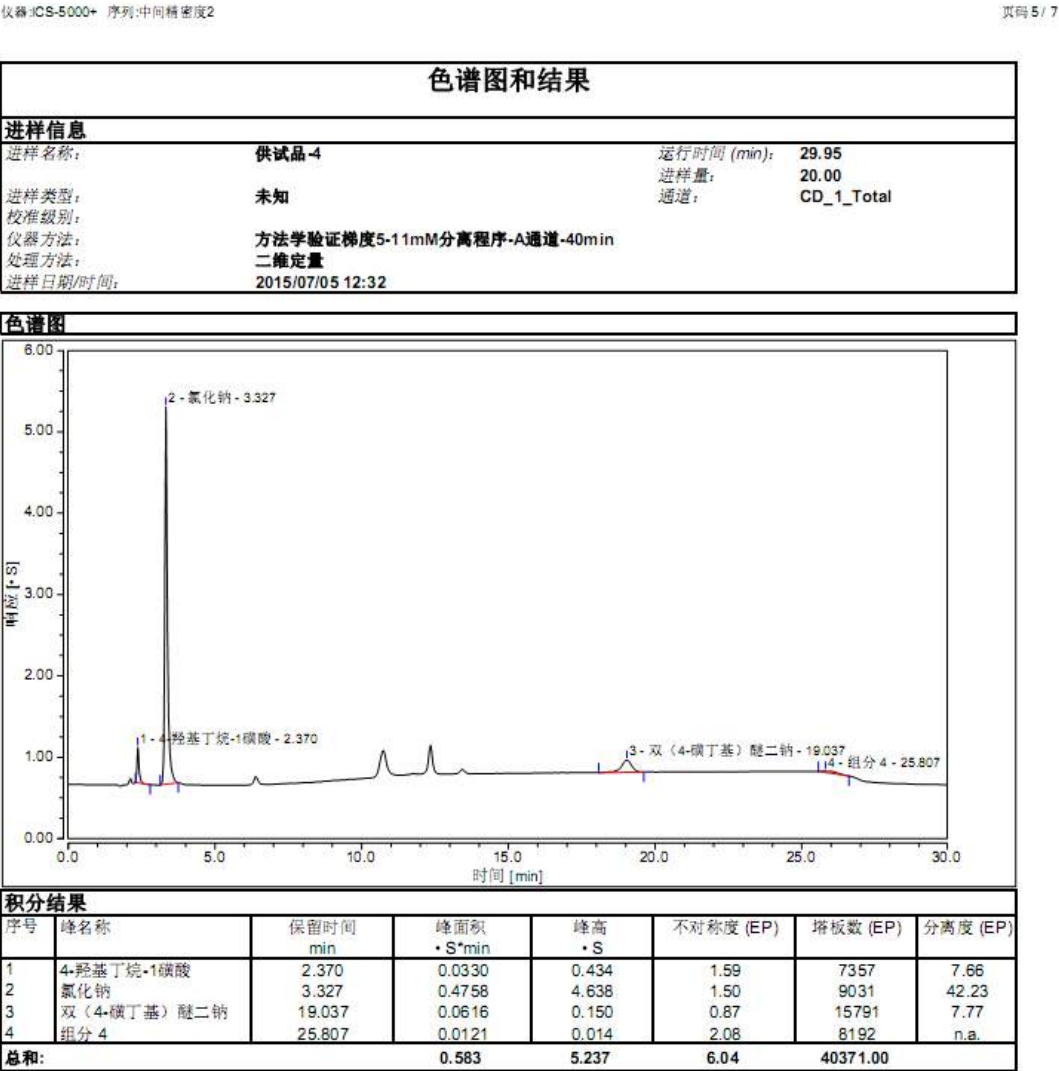
Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-24 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Intermediate precision
2- Reference solution



附图10.4.11-220 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(中间精密度2-对照品)

Annex 3-4-25 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Intermediate precision 2- Sample solution -4

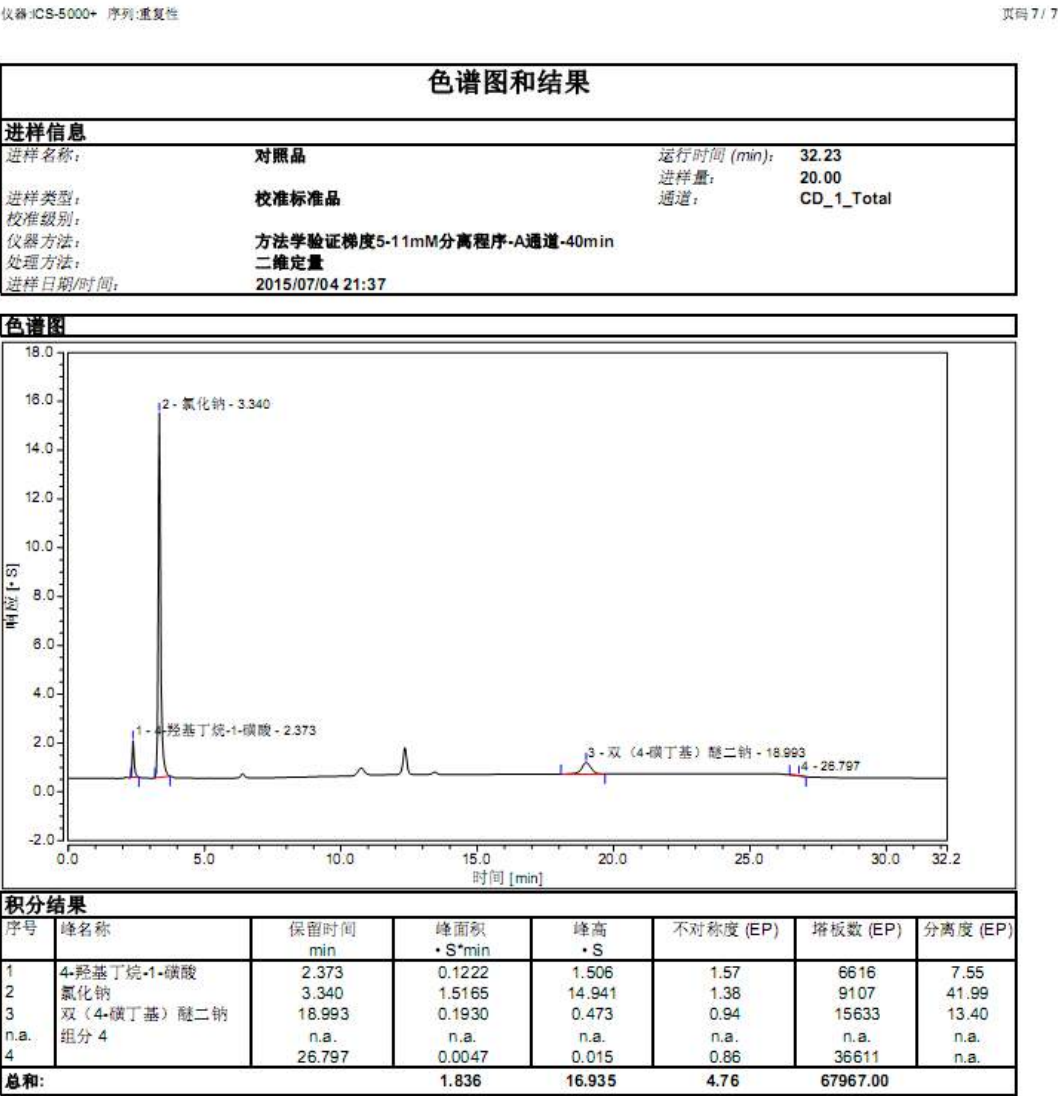


附图10.4.11-224 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(中间精密度2-供试品-4)

1/积分

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版本 7.2.1.5537

Annex 3-4-26 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Repeatability-Reference solution

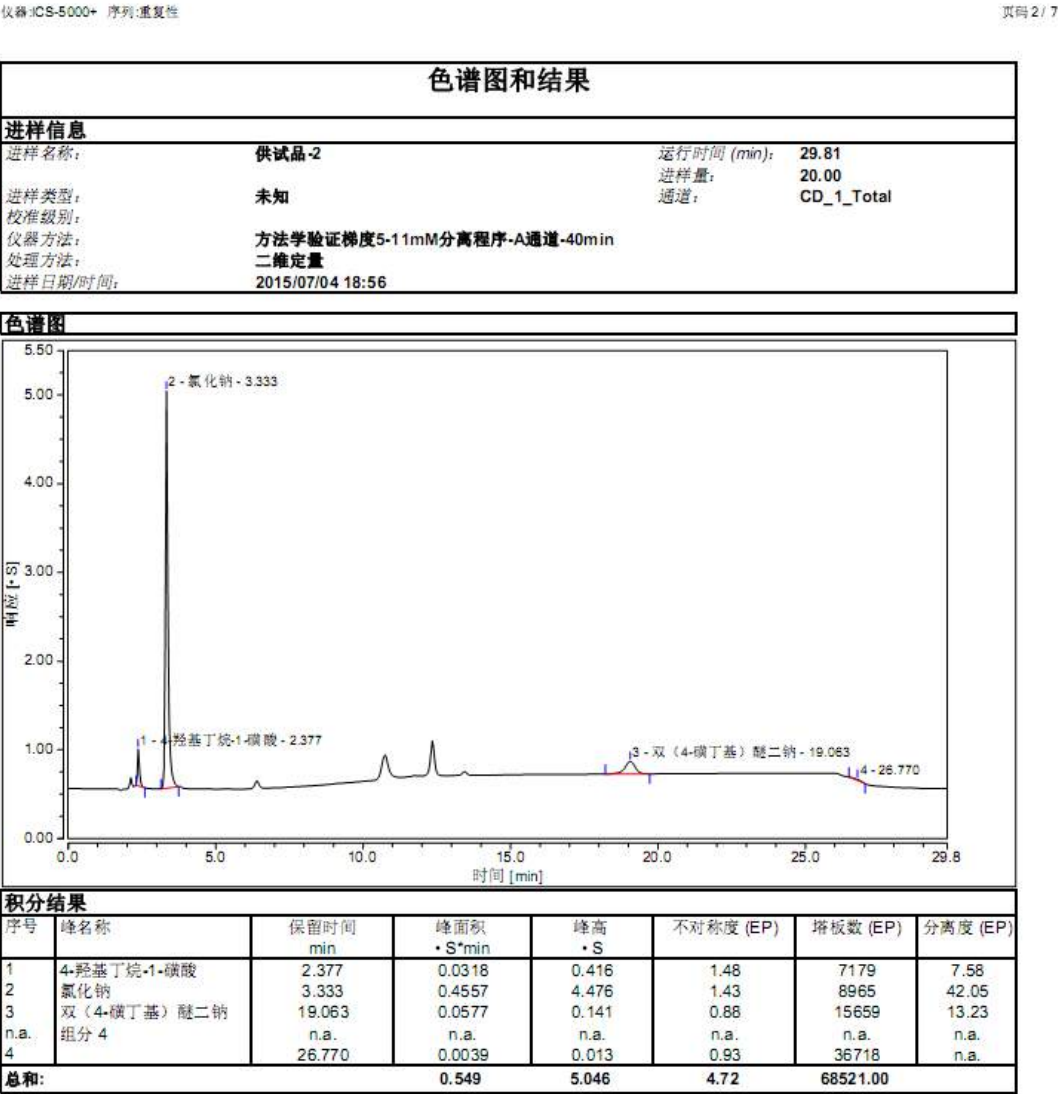


附图10.4.11-227 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(重复性-对照品)

1/积分

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版本 7.2.1.5537

Annex 3-4-27 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Repearability-Sample solution-2

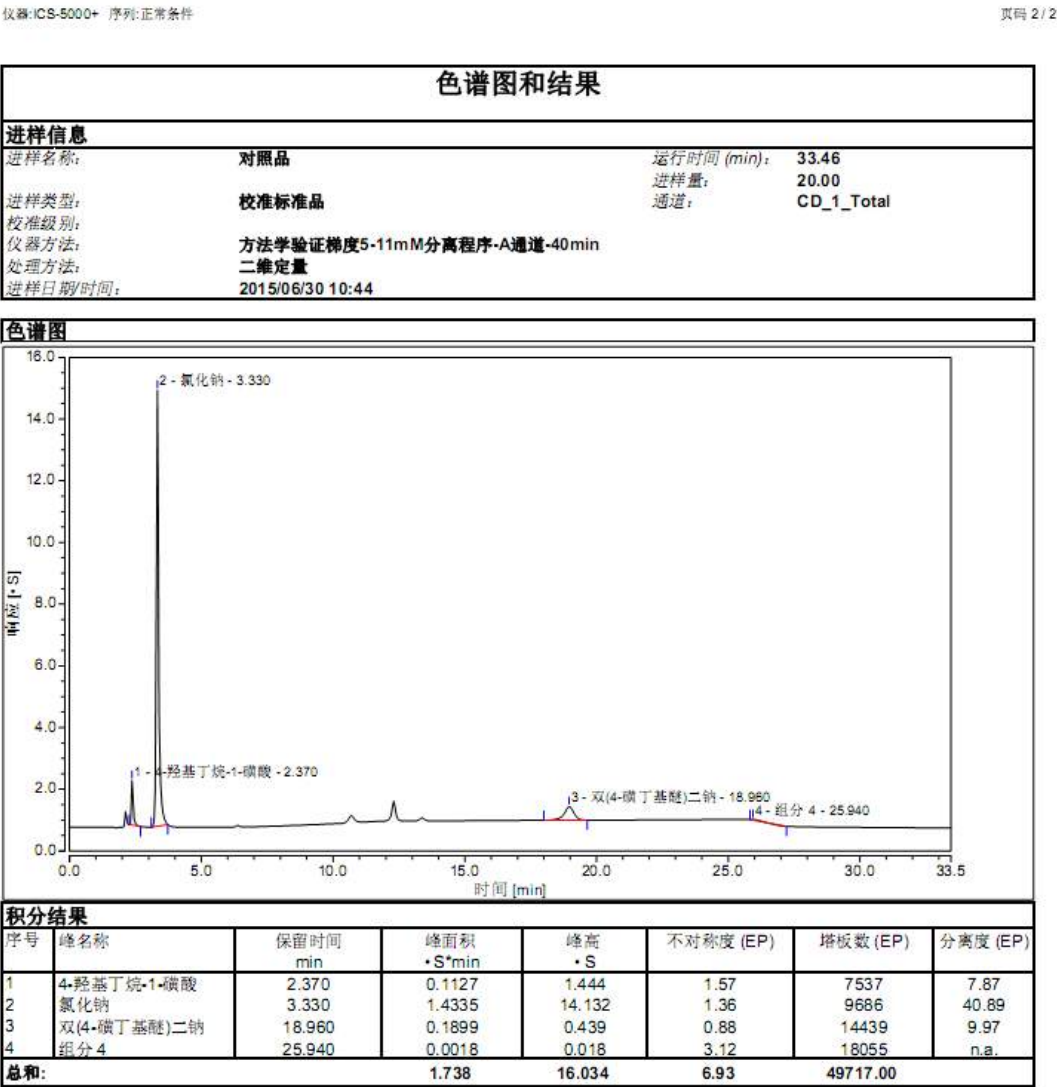


附图10.4.11-229 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(重复性-供试品-2)

1/积分

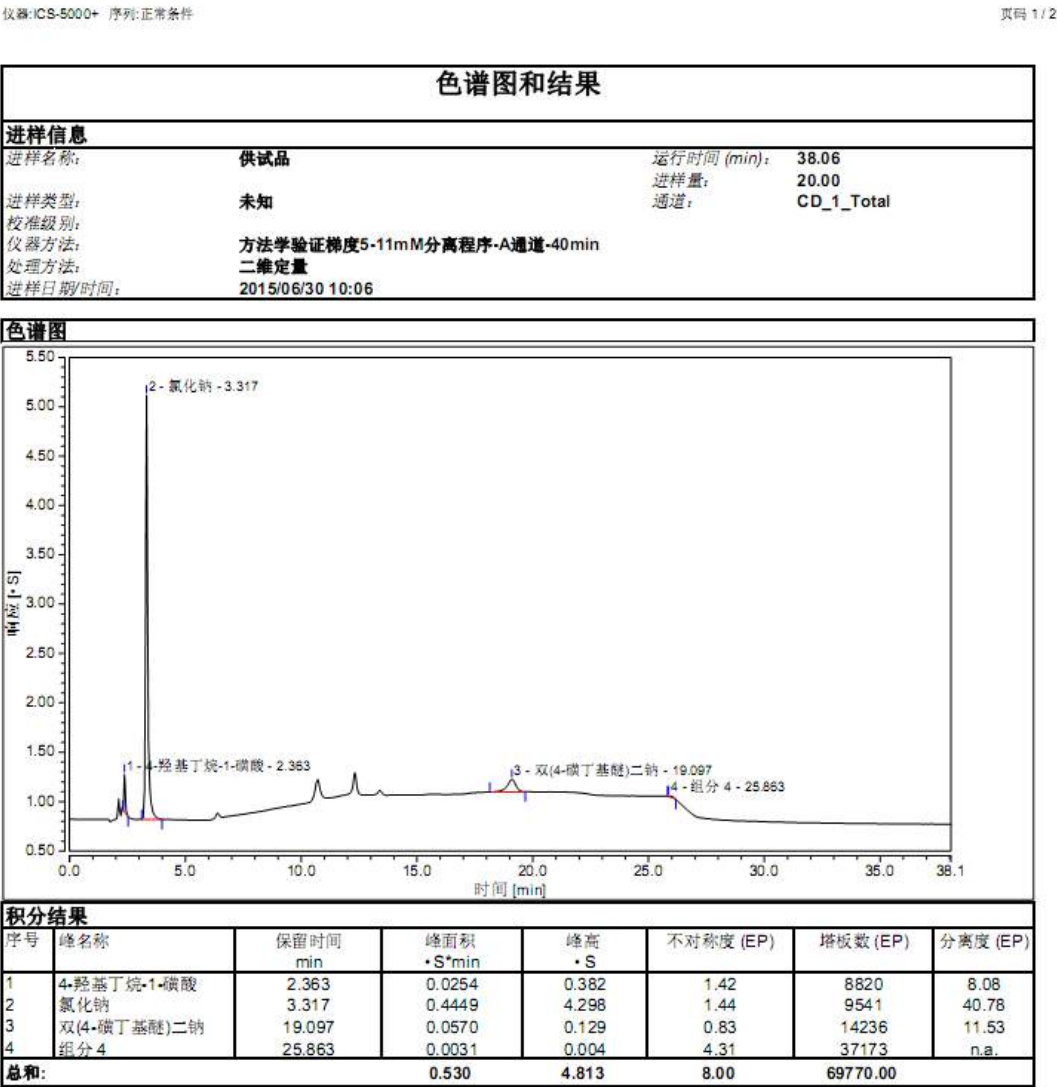
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-28 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Normal condition-Reference solution



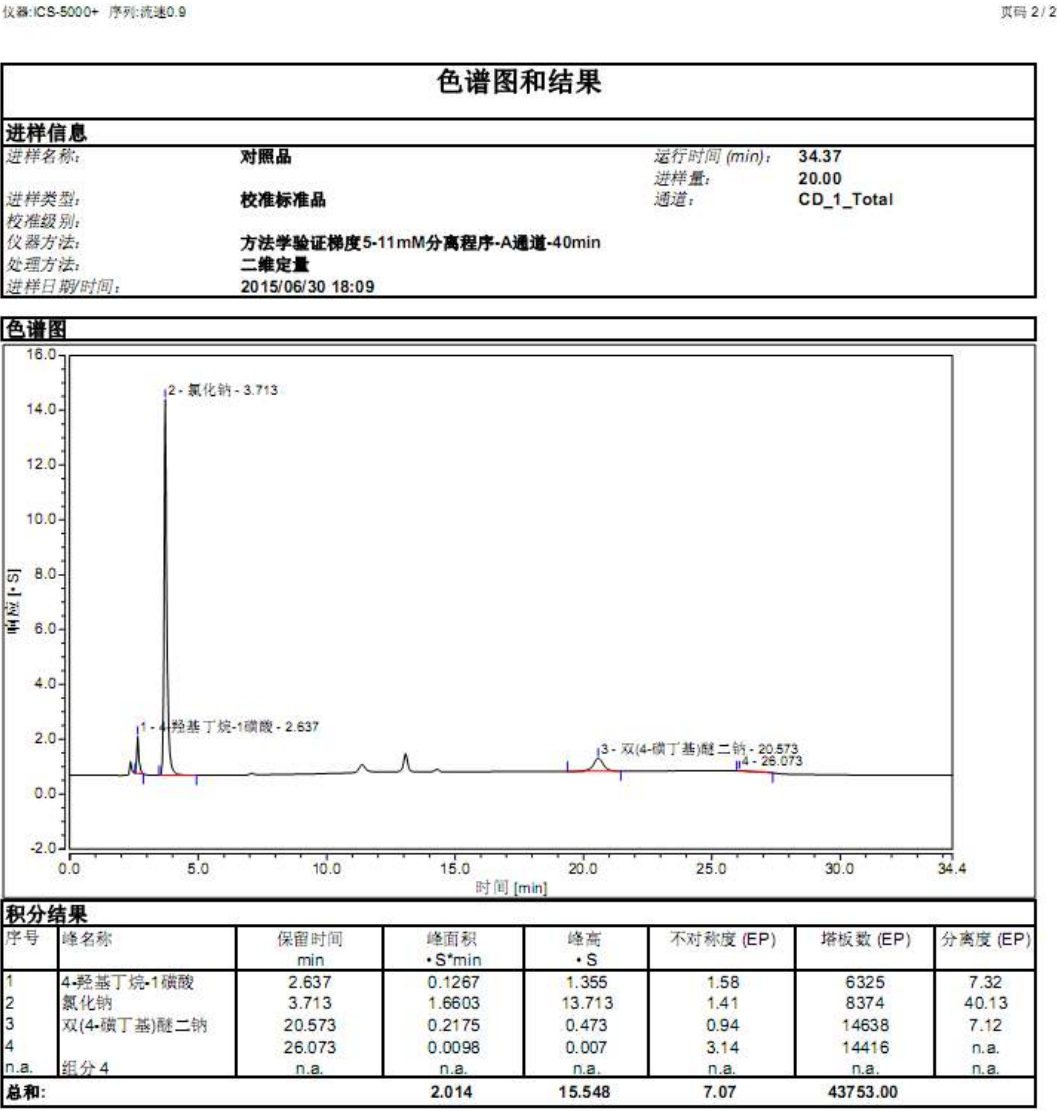
附图10.4.11-261 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（耐用性-正常条件-对照品）

Annex 3-4-29 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Normal condition-Sample solution



附图10.4.11-262 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（耐用性-正常条件-供试品）

Annex 3-4-30 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 2-Reference solution



附图10. 4. 11-83100中4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定方法验证图（耐用性-0.9 ml/min）

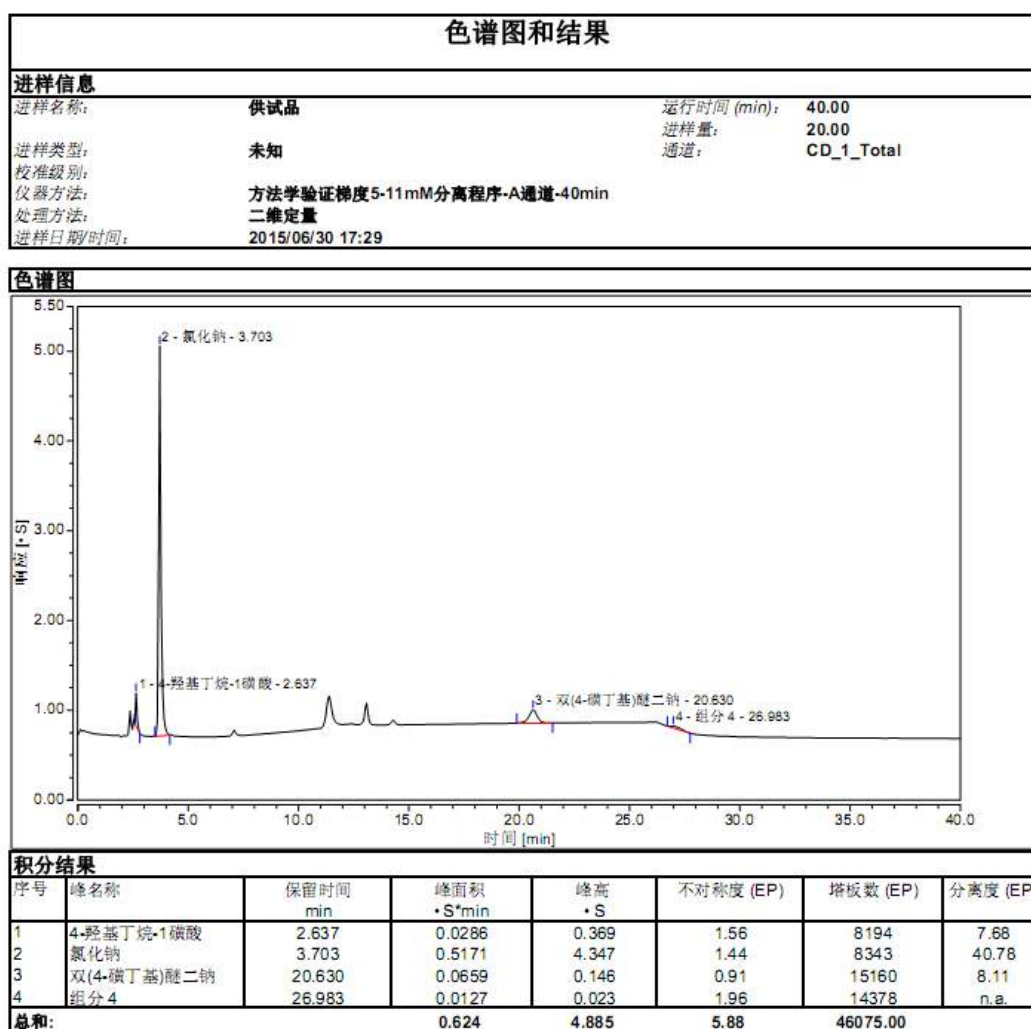
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-31 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 2-Sample solution

仪器:ICS-5000+ 序列:流速0.9

页码 1/2

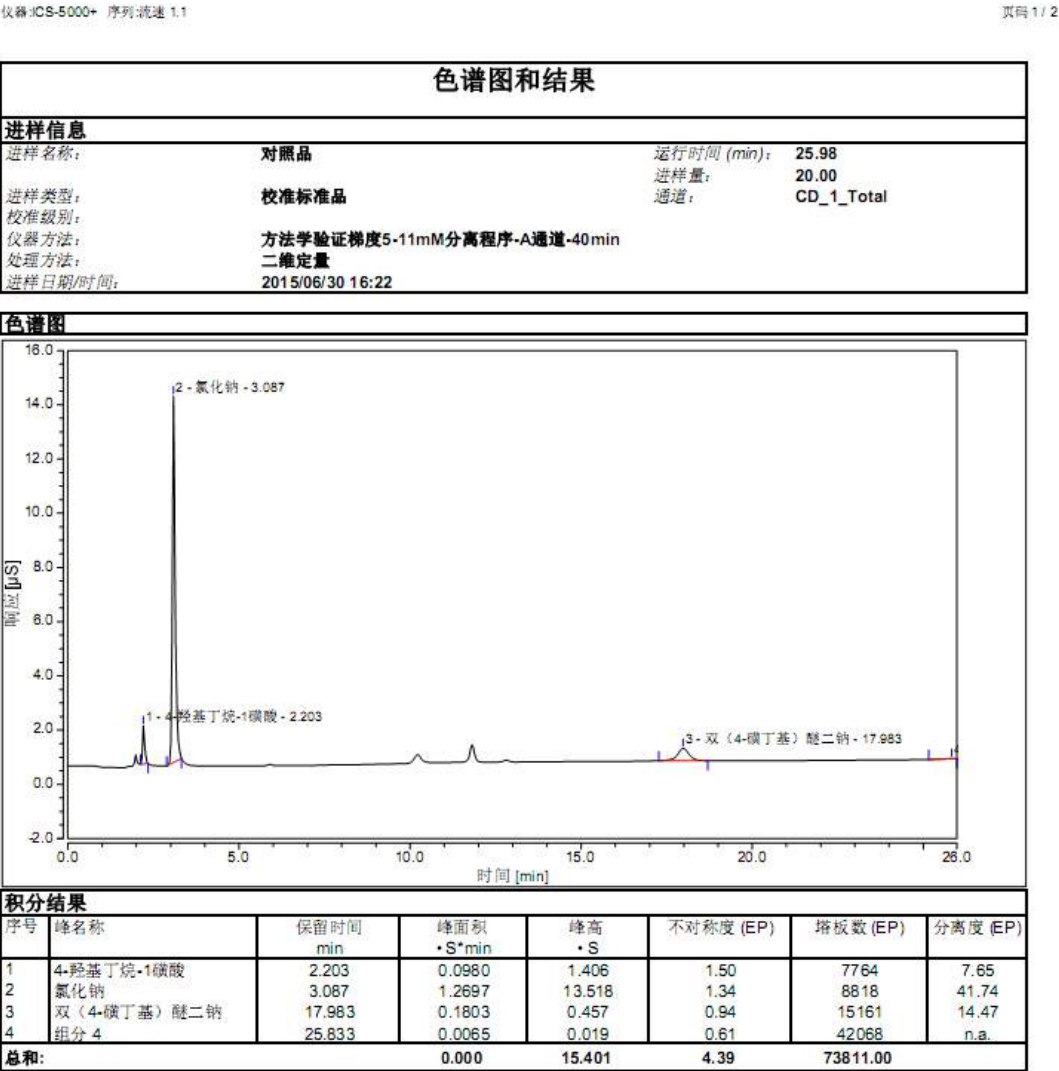


附图10.4.11-250 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-0.9ml/min-供试品)

Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-32 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 3-Reference solution

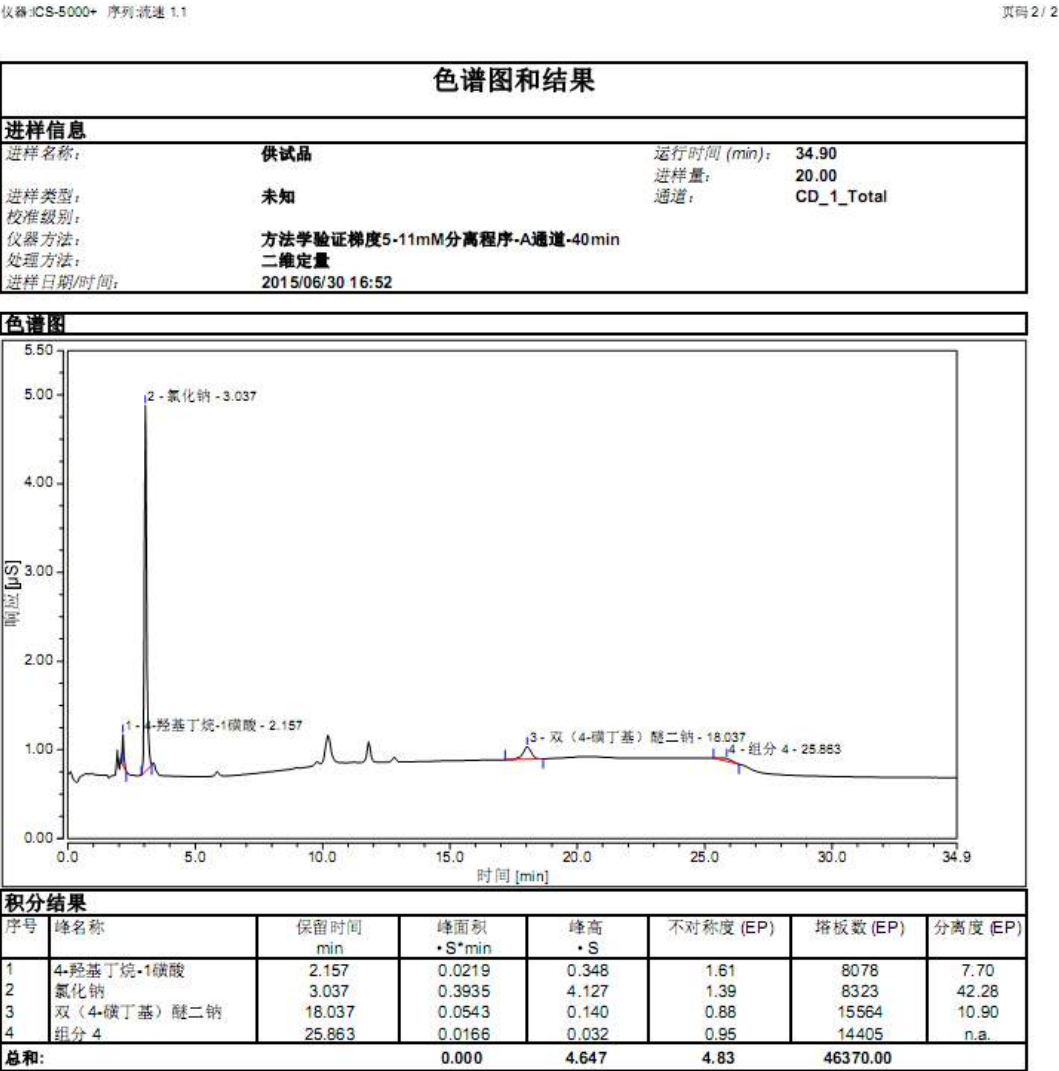


附图10. 4. 11-9350D中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-1.1ml/min)

Default/积分

Chromeleon(c) Dionex
版本 7.2.1.5537

Annex 3-4-33 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 3-Sample solution



附图10. 4. 11-8352D中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-1. 1ml/min -

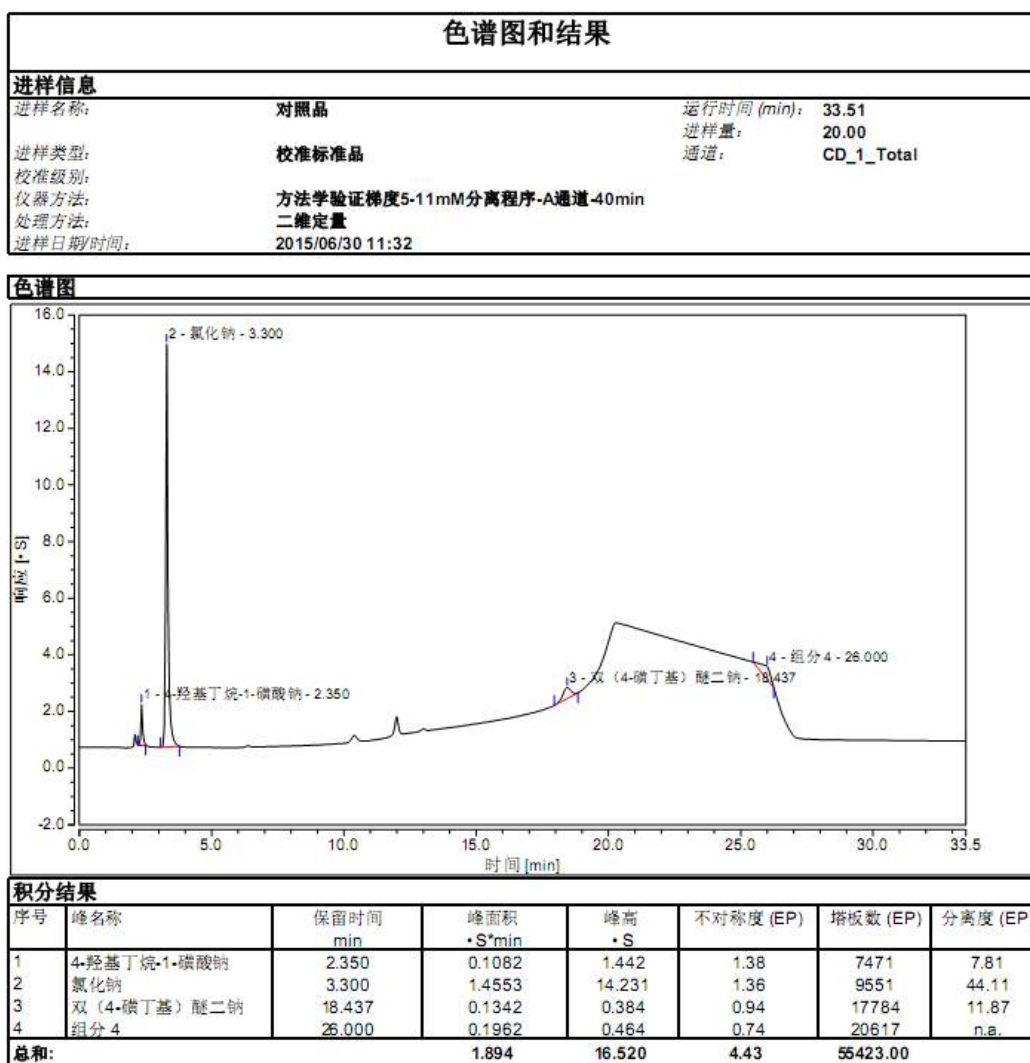
Default/积分

Chromeleon(c) Dionex
版本 7.2.1.5537

Annex 3-4-34 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 4-Reference solution

仪器:ICS-5000+ 序列:柱温28

页码 1/2



附图10.4.11-253 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性:柱温28度-对照品)

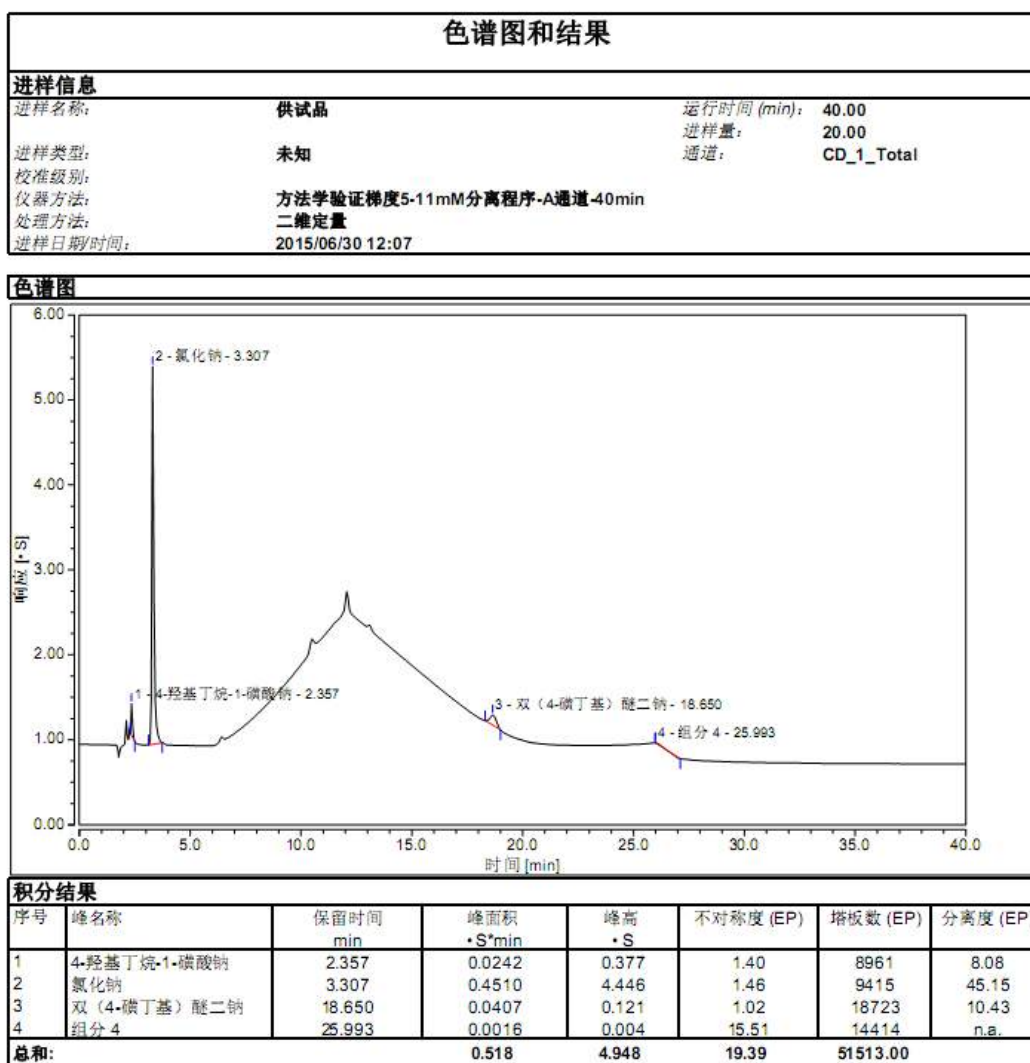
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-35 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 4-Sample solution

仪器:ICS-5000+ 序列:柱温28

页码 2 / 2



附图 I0.4.11-254 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-柱温28度-供试品)

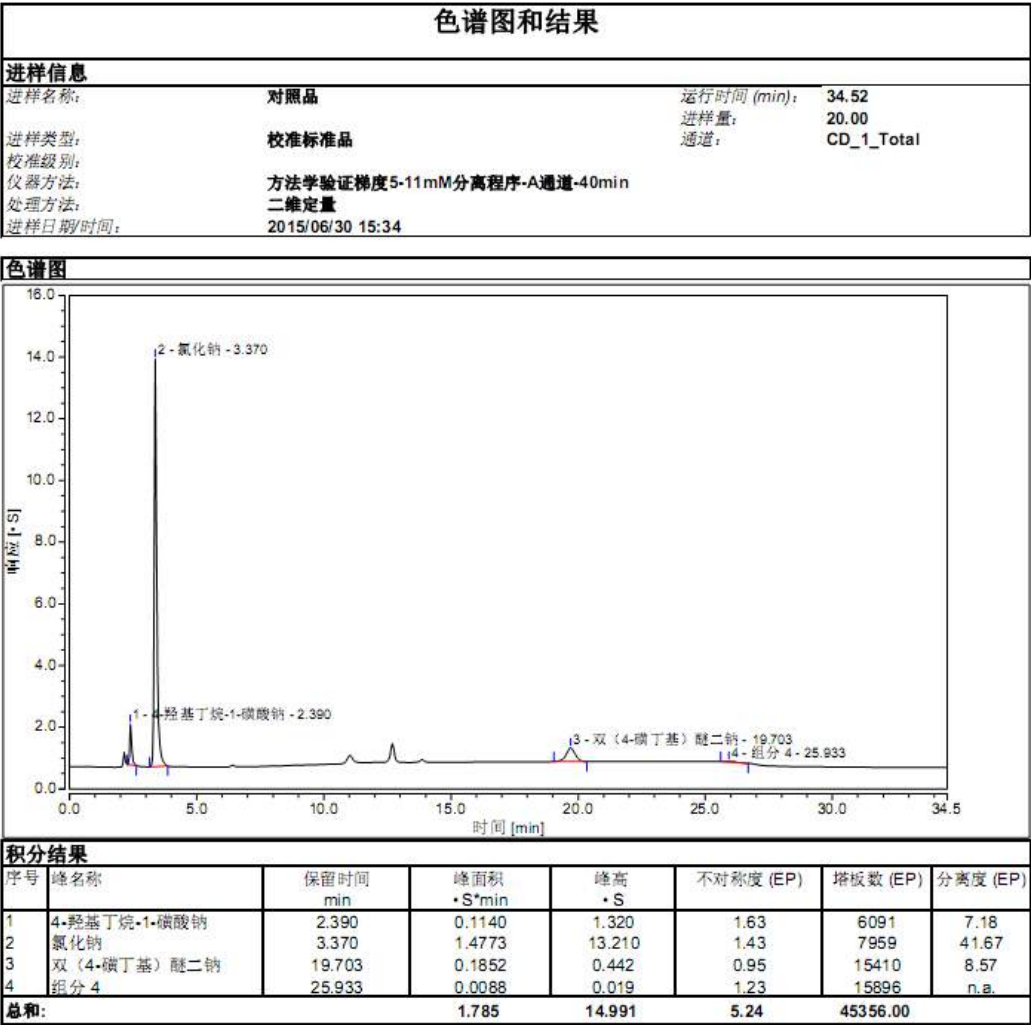
Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-36 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 5-Reference solution

仪器:ICS-5000+ 序列:柱温32

页码 2/2



附图10. 4. 11-3165D中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-柱温32度-对照品)

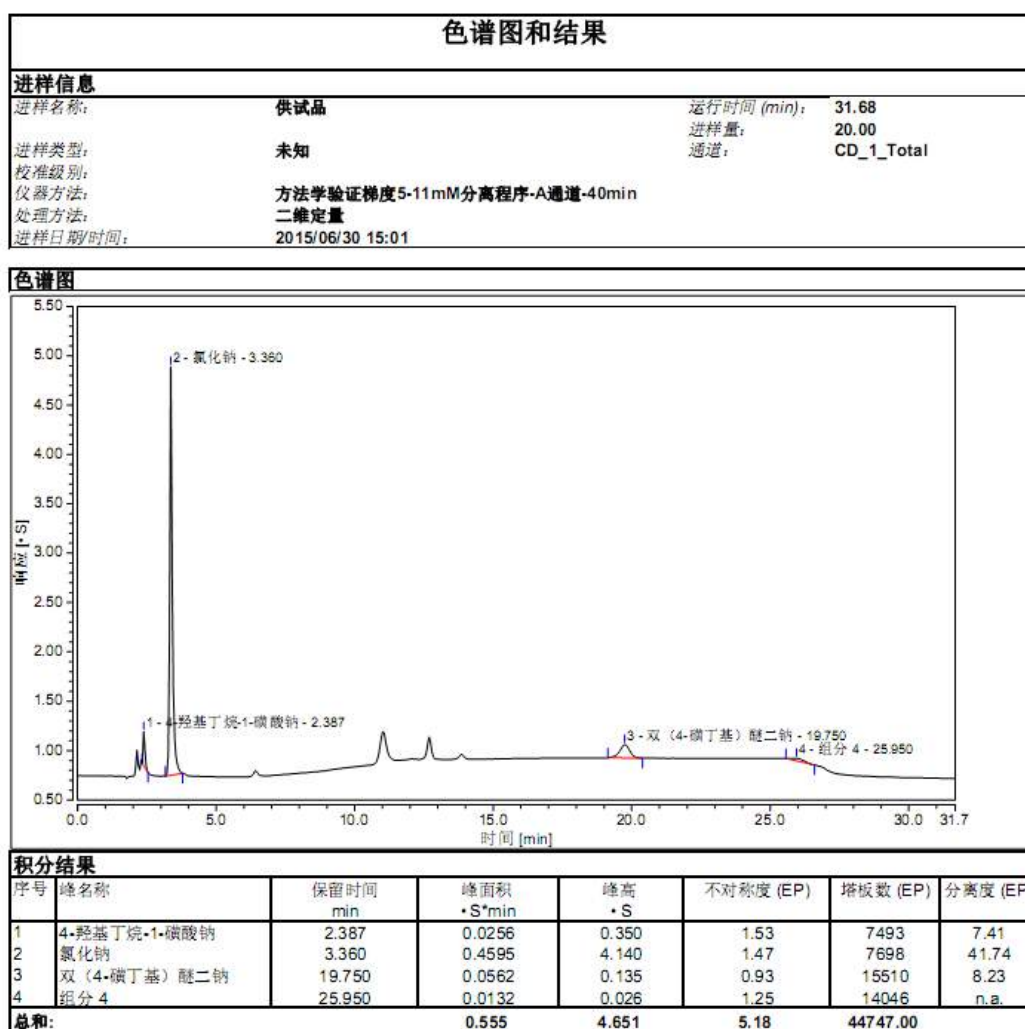
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-37 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 5-Sample solution

仪器: ICS-5000+ 序列: 柱温32

页码 1 / 2

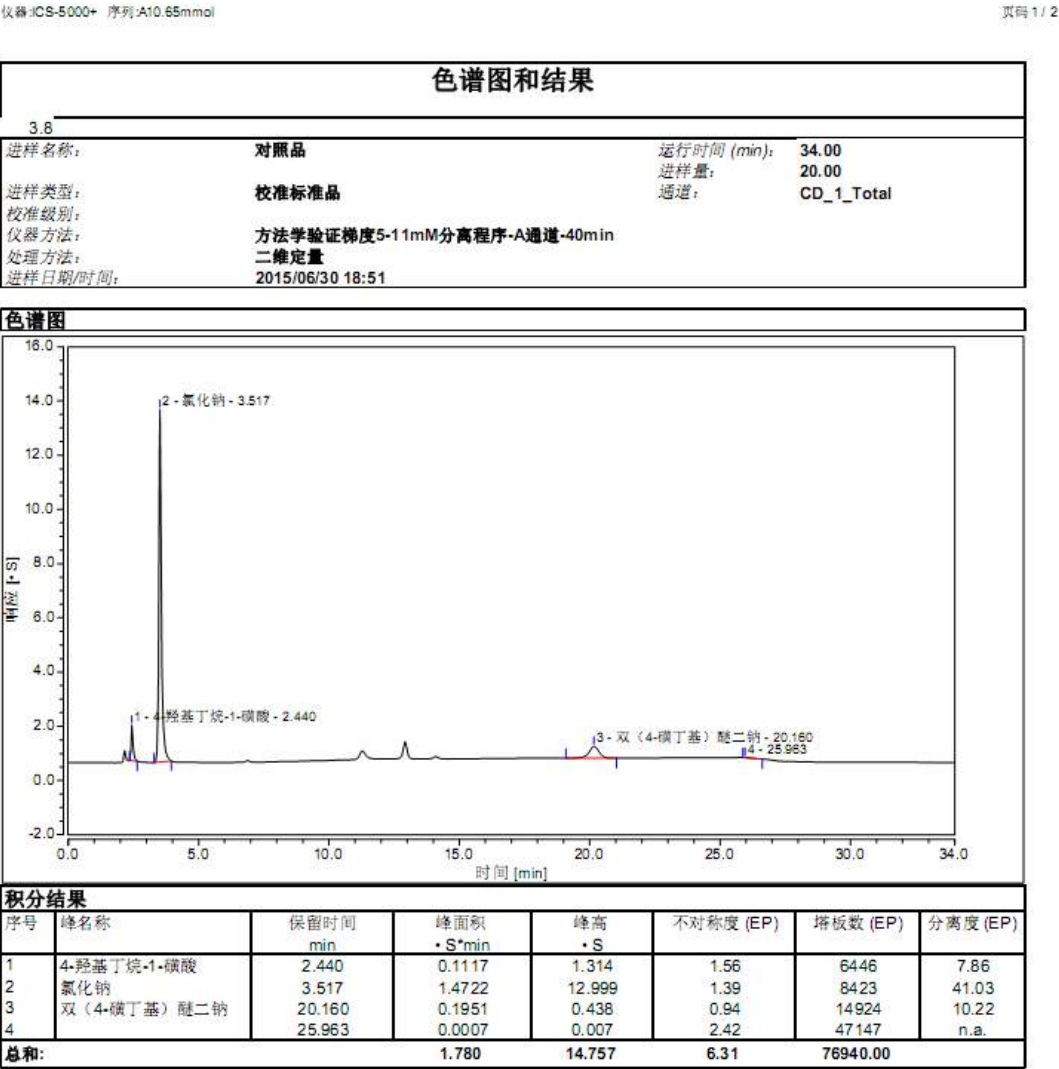


附图10. 4. 11-3356D中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-柱温32度-供试品)

Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-38 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 6-Reference solution

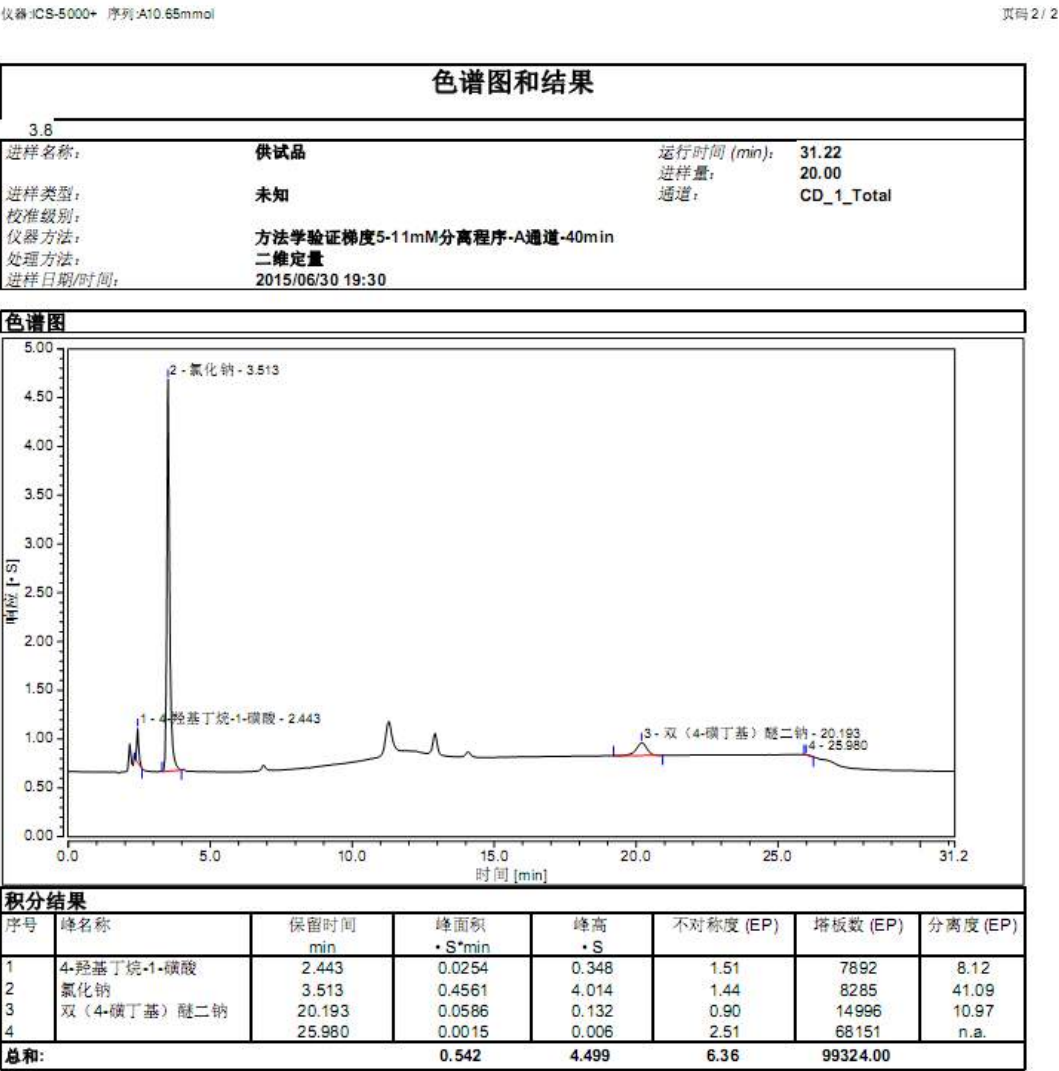


附图10. 4. 11-8367D中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-梯度脱洗A-对照品)

Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-39 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 6-Sample solution

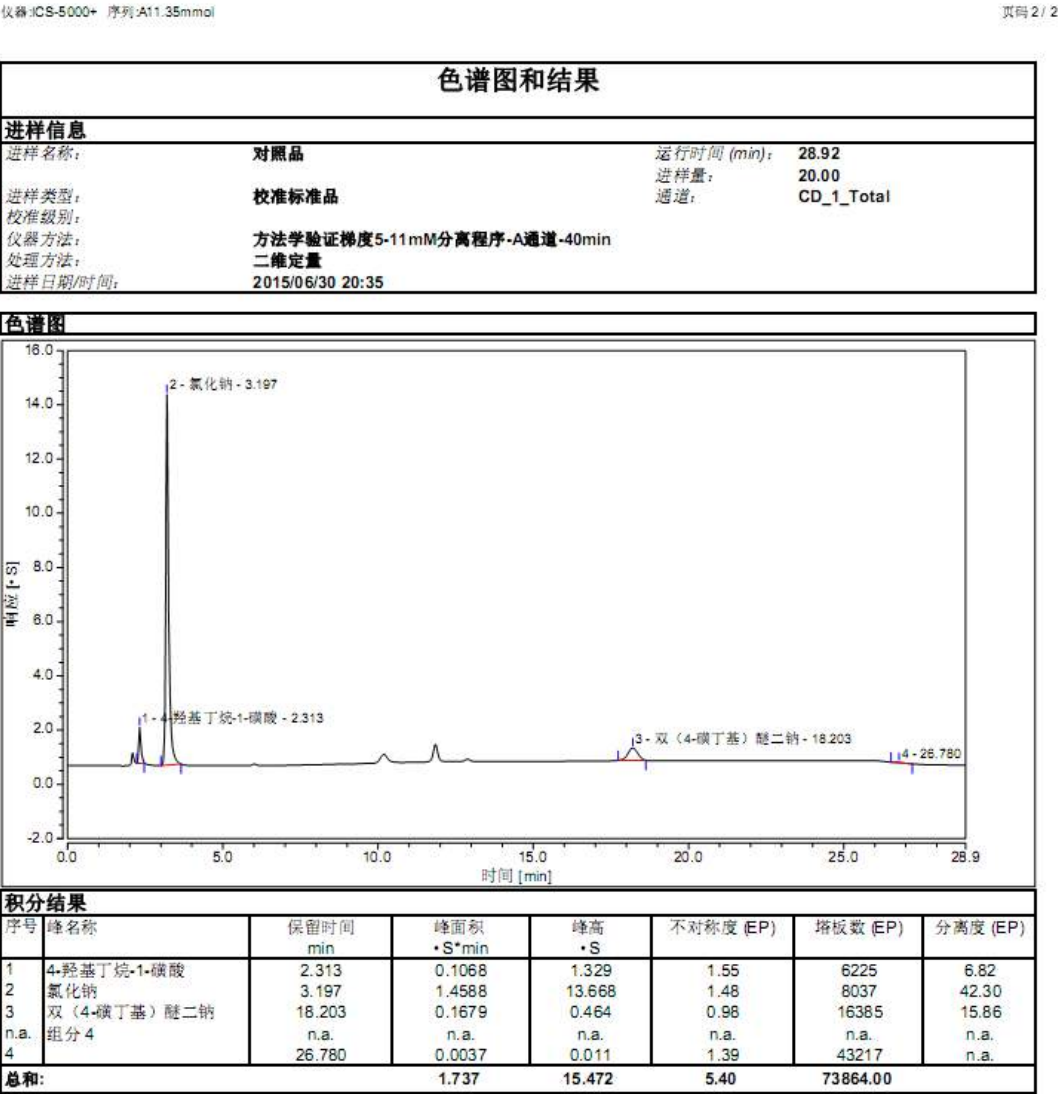


附图10. 4. 11-315SD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-梯度脱洗A-供试品)

Default/积分

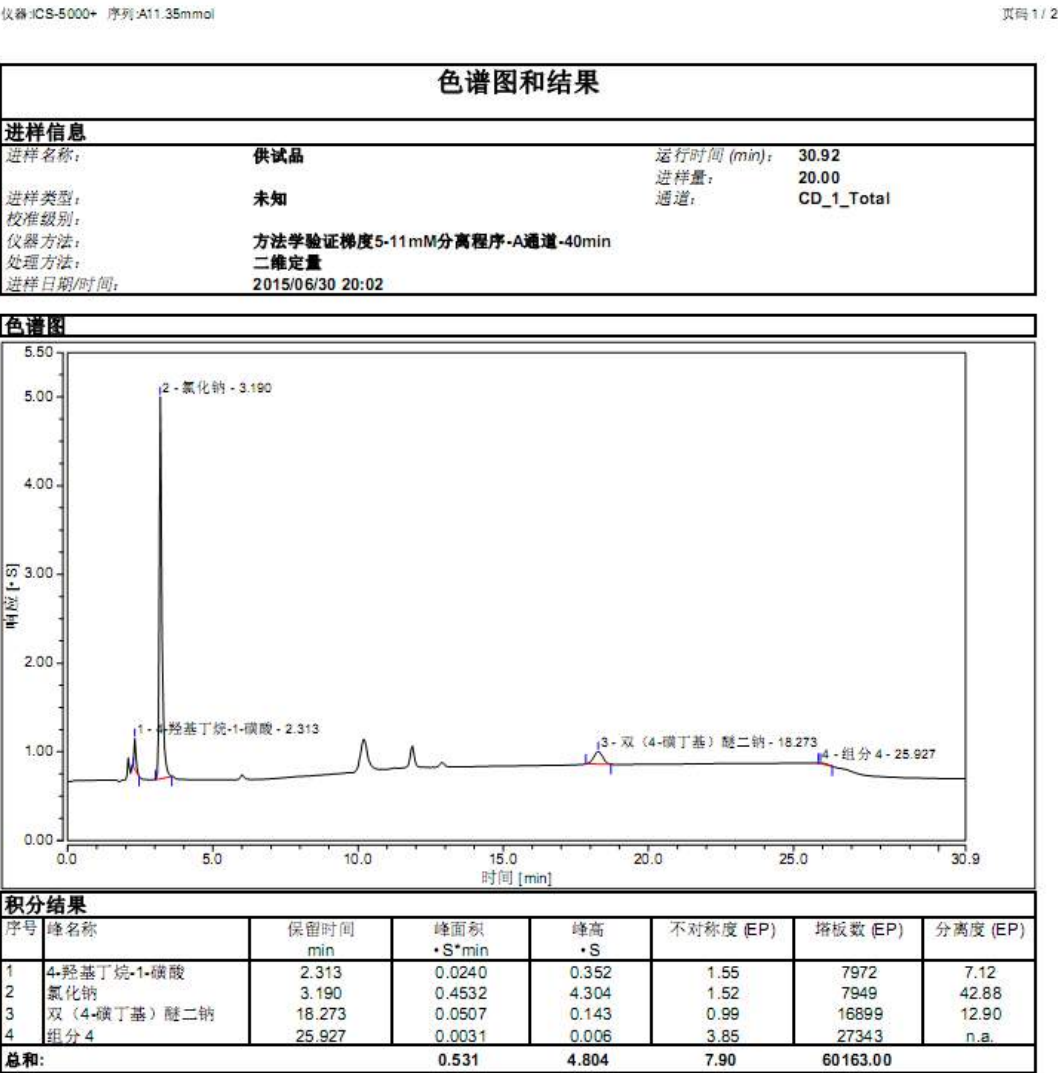
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-40 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 7-Reference solution



附图10. 4. 11-83560中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-梯度脱洗B-对照品)

Annex 3-4-41 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Robustness-Condition 7-Sample solution



附图10， 4. 11-9360)中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(耐用性-梯度脱洗B-供试品)

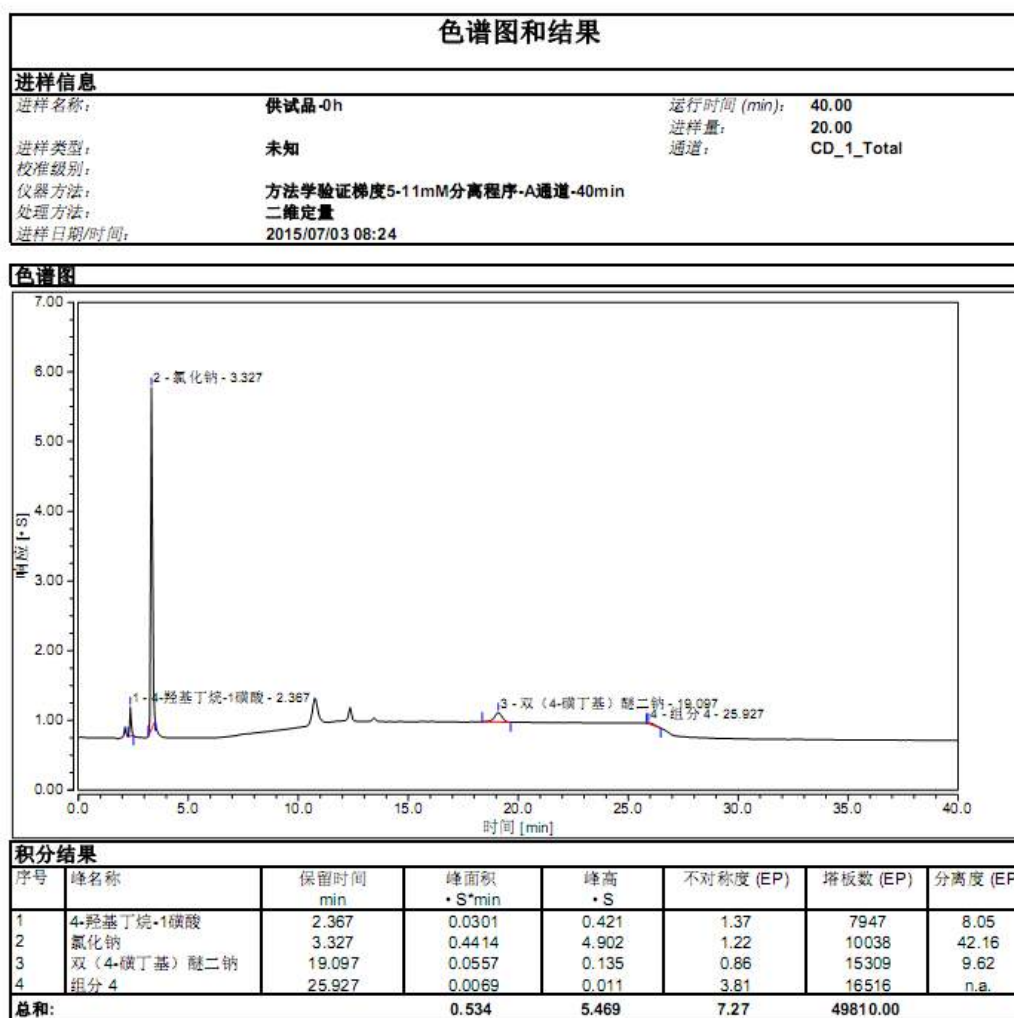
Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-42 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Reference solution 0h

仪器:ICS-5000+ 序列:溶液稳定性

页码 1 / 15

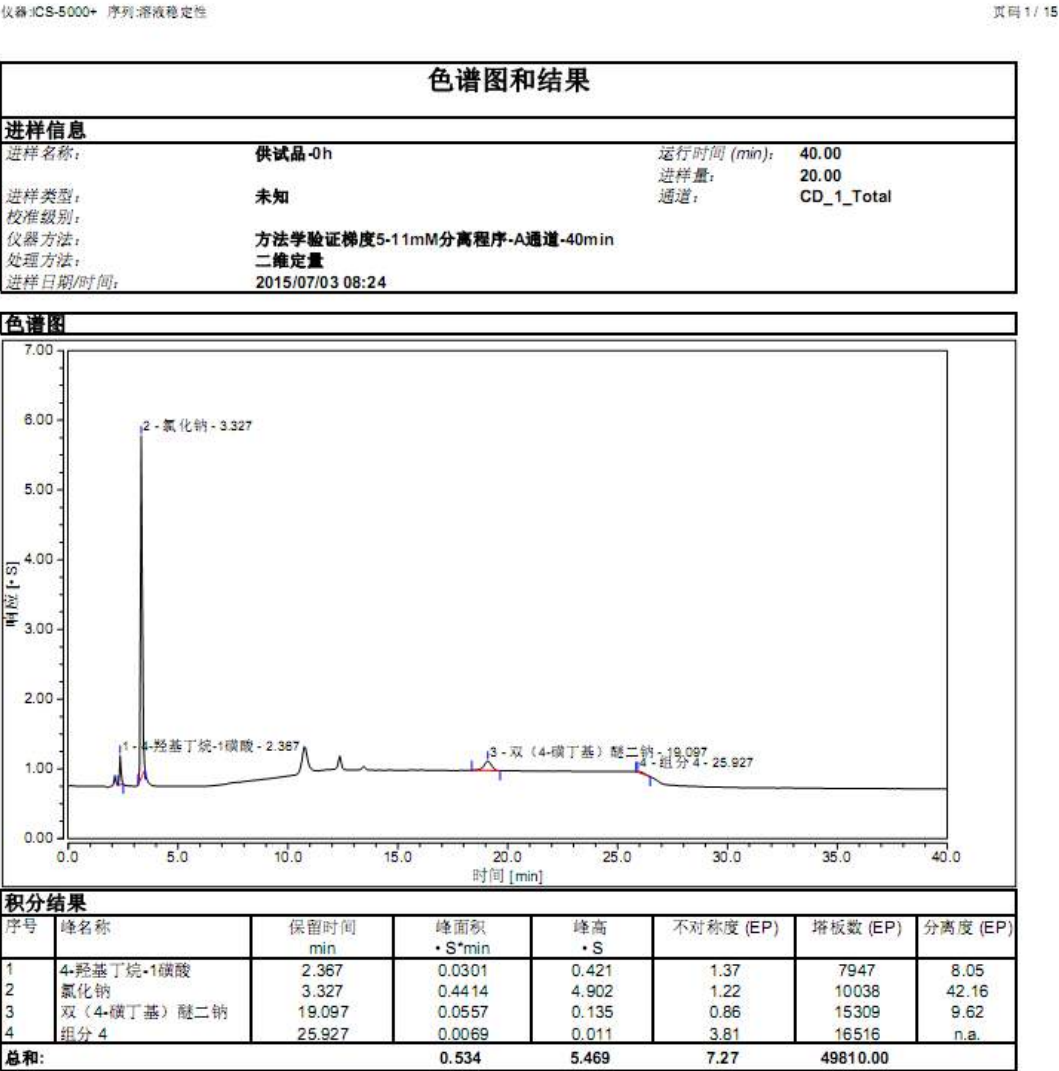


附图10.4.11-241 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(供试品溶液稳定性-0h)

1/积分

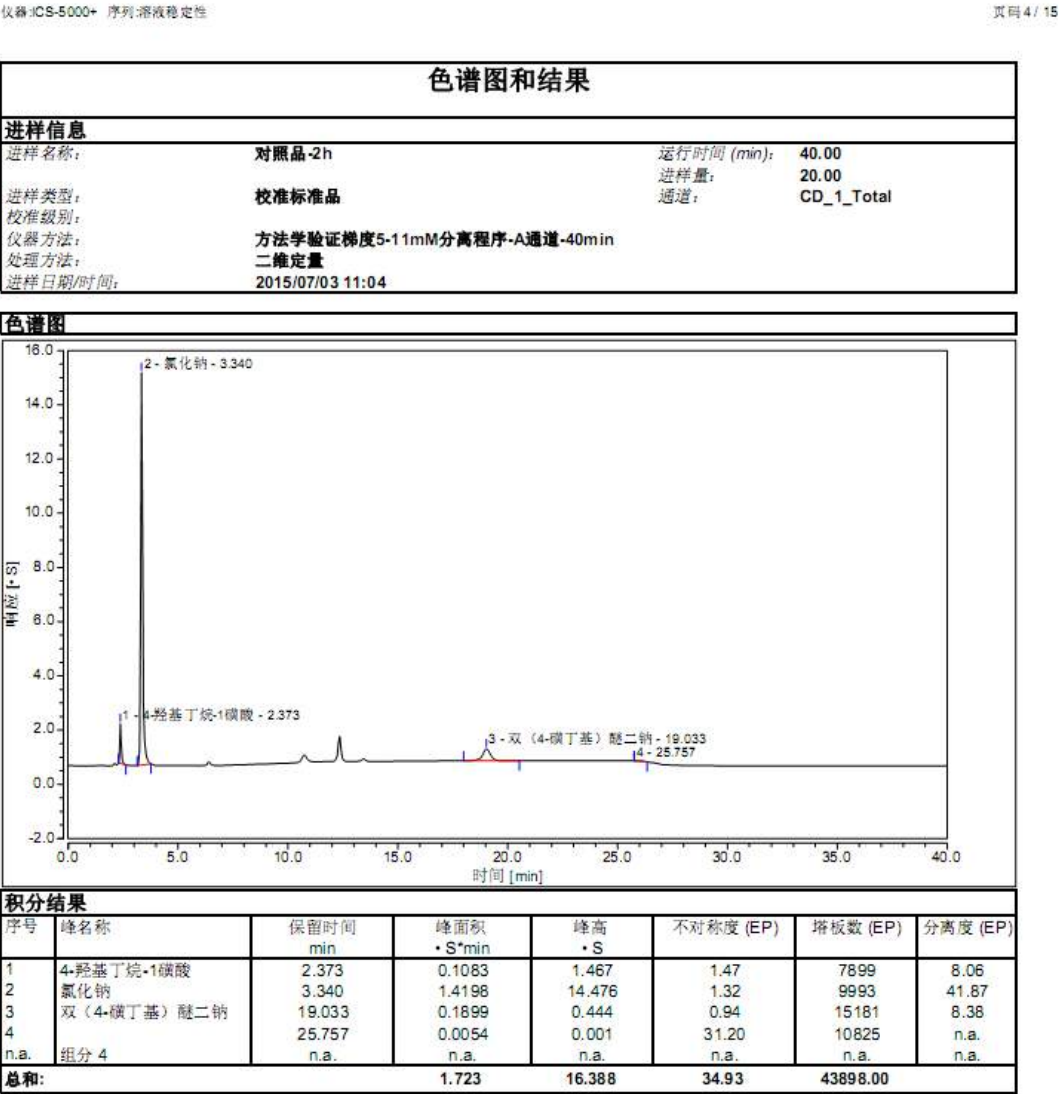
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-43 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Sample solution 0h



附图10.4.11-241 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(供试品溶液稳定性-0h)

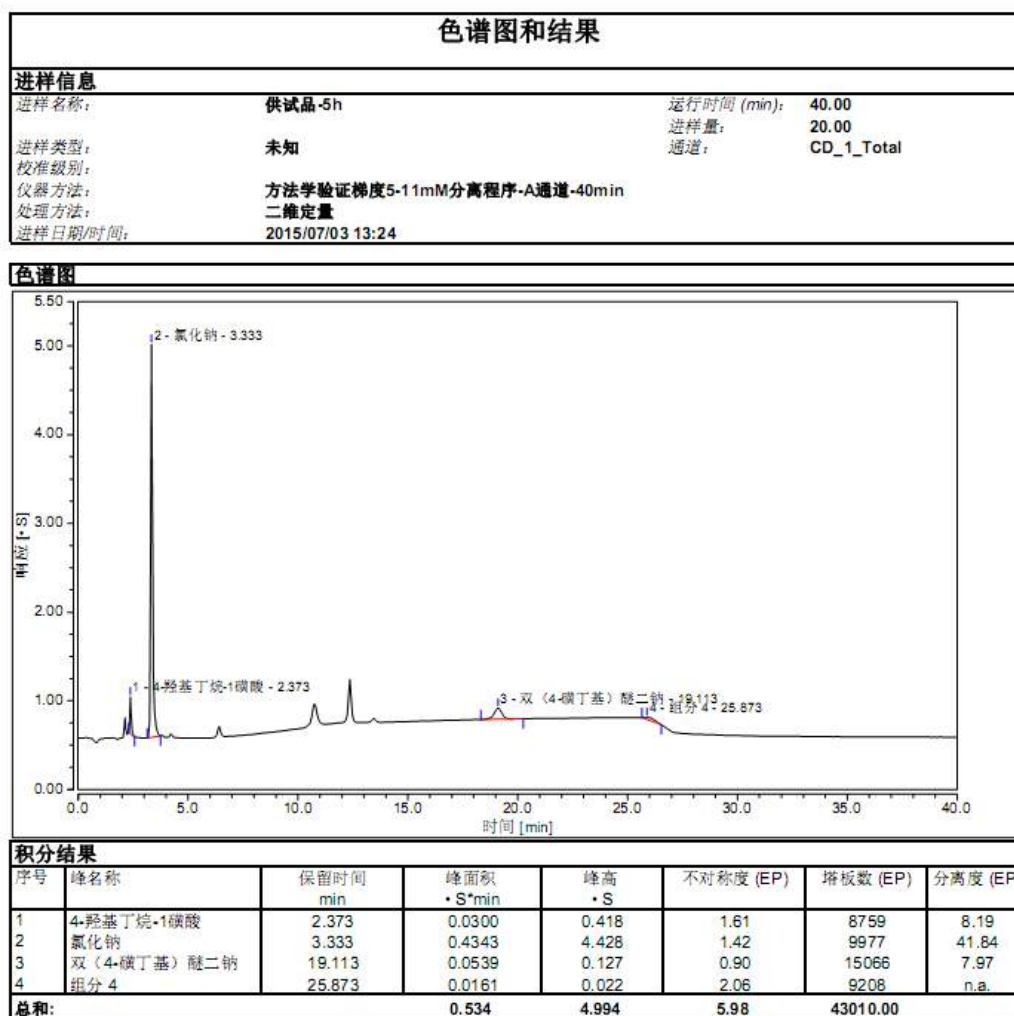
Annex 3-4-44 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Reference solution 2h



Annex 3-4-45 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Sample solution 5h

仪器:ICS-5000+ 序列:溶液稳定性

页码 5 / 15

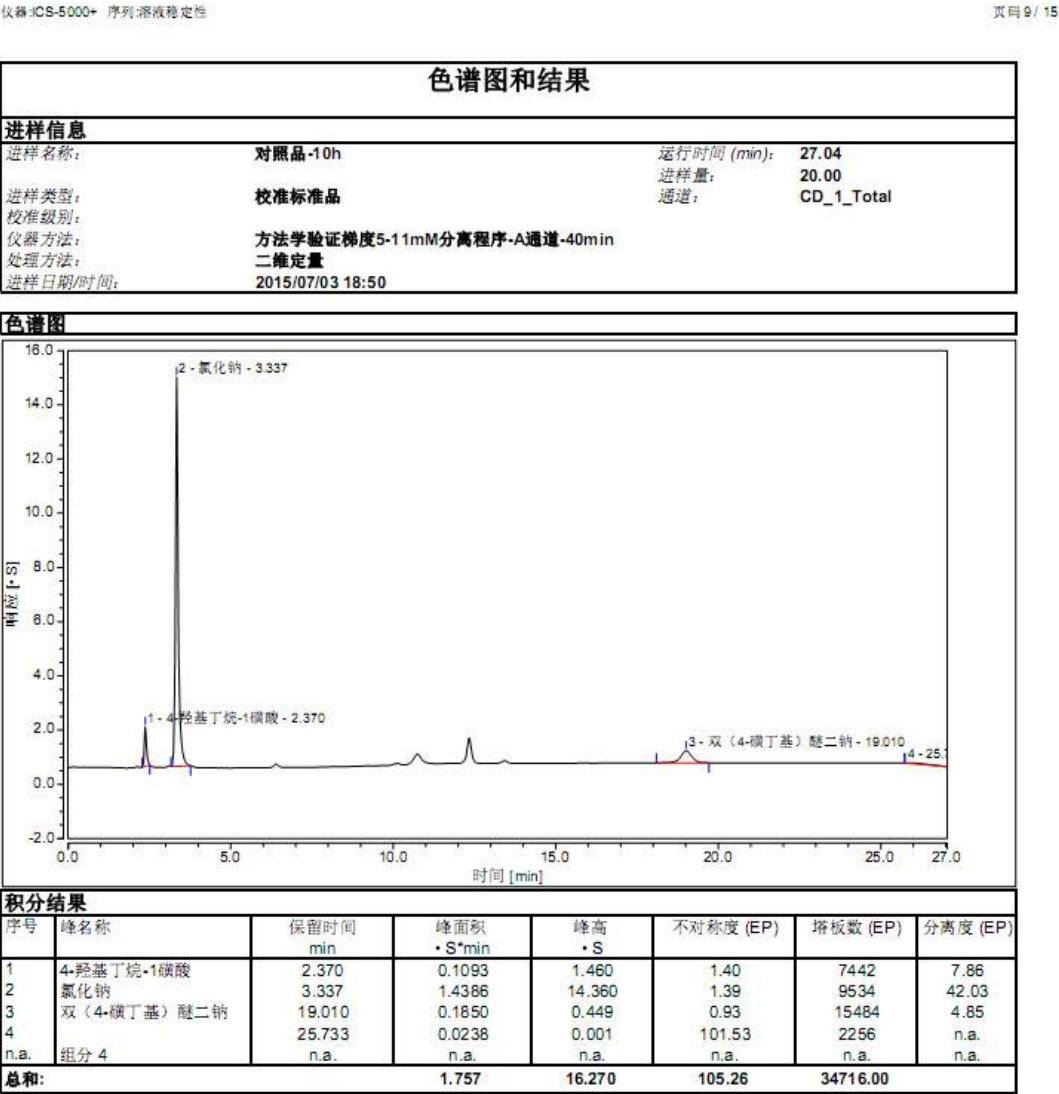


附图10.4.11-243 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(供试品溶液稳定性-5H)

1/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-46 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Reference solution 10h



附图10.4.11-237 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(对照品溶液稳定性-10H)

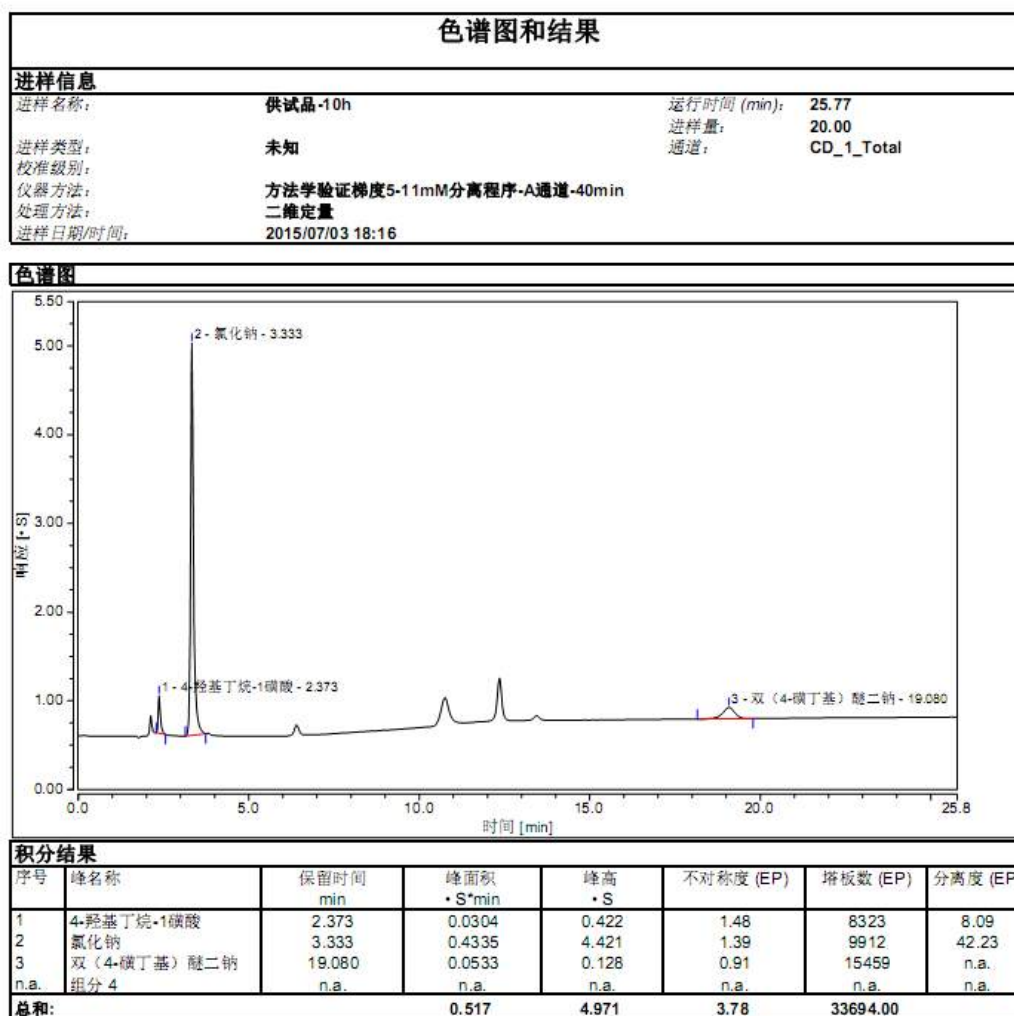
1/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-47 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Sample solution 10h

仪器:ICS-5000+ 序列:溶液稳定性

页码 8 / 15



附图 10.4.11-245 SBECD 中 4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图 (供试品溶液稳定性-10H)

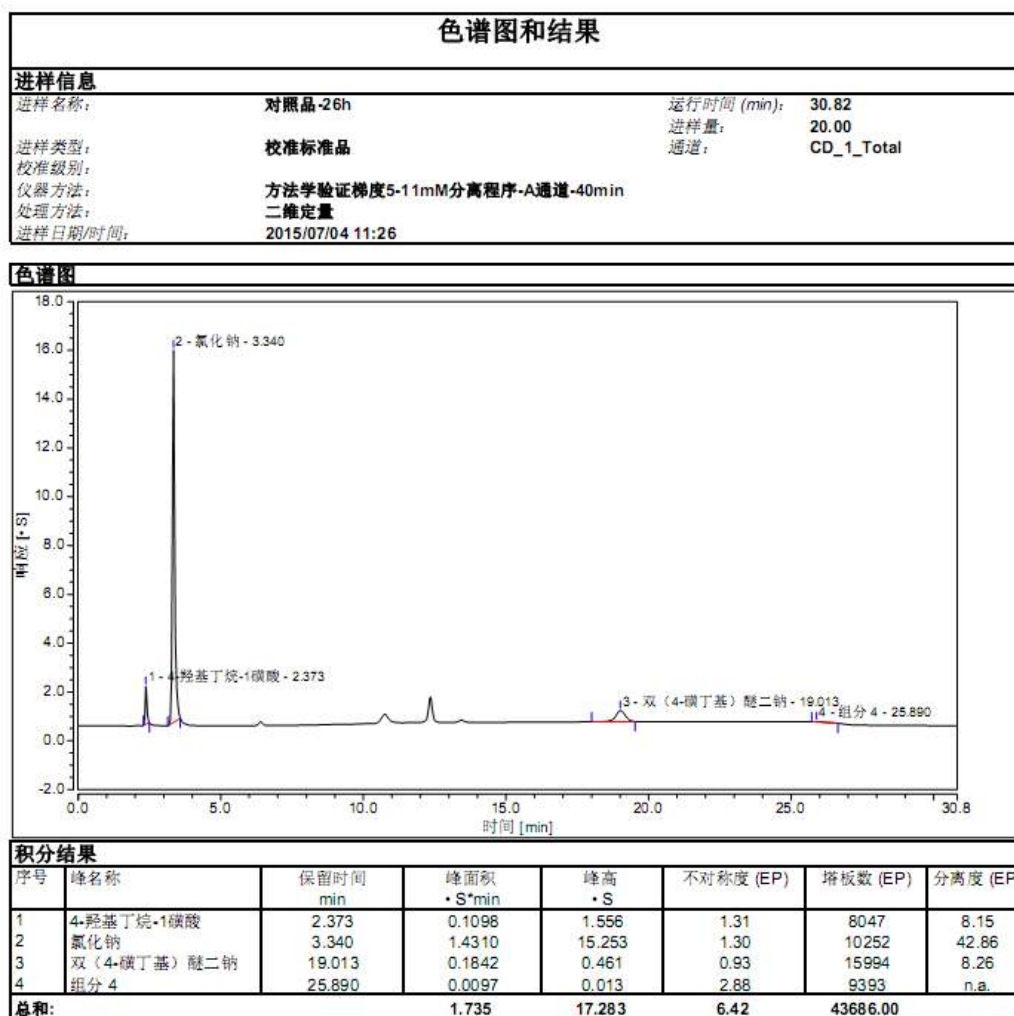
1/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-48 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Reference solution 26h

仪器:ICS-5000+ 序列:溶液稳定性

页码 15 / 15

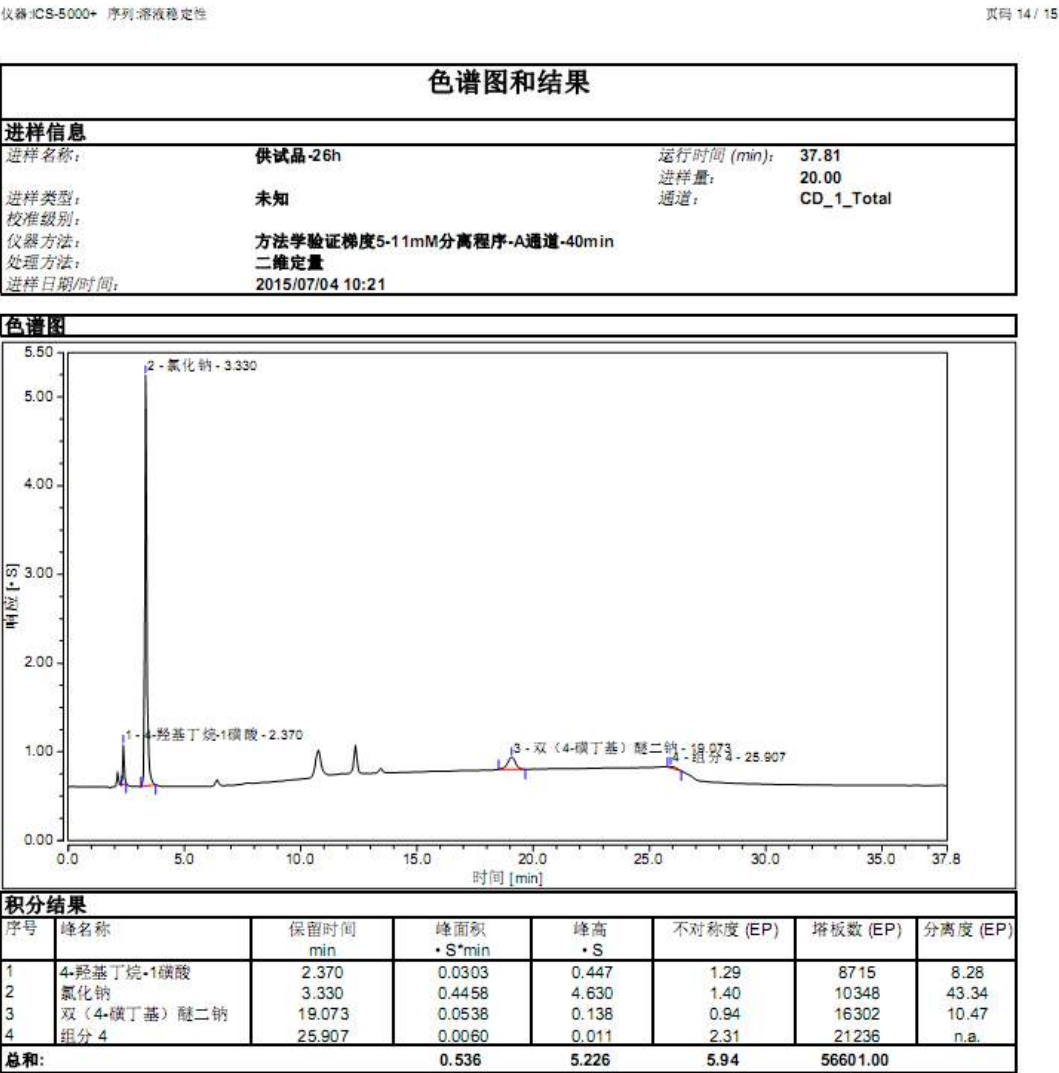


附图10.4.11-240 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(对照品溶液稳定性-26H)

1/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

Annex 3-4-49 Validation of analysis procedure for Sodium chloride, 4-hydroxybutane-1-sulfonic acid and Bis(4-sulfobutyl) ether disodium-Solution stability-Sample solution 26h



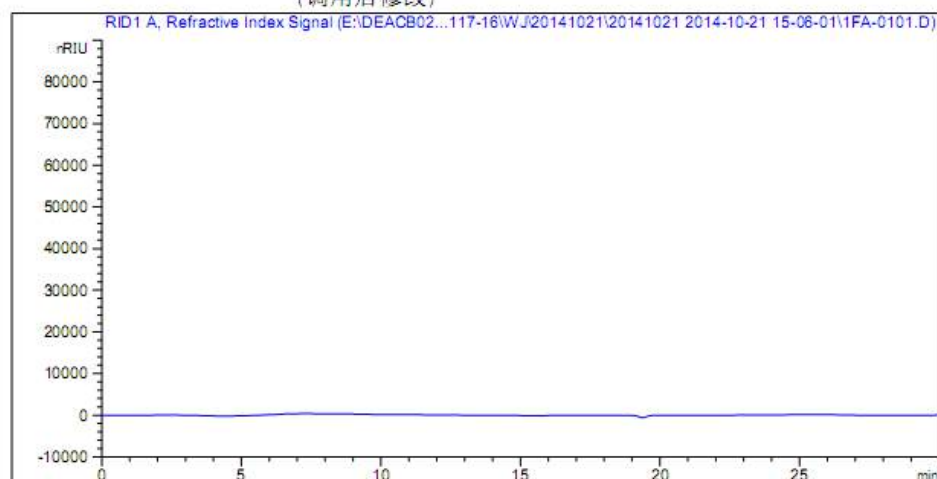
附图10.4.11-248 SBECD中4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定方法验证图(供试品溶液稳定性-26H)

Annex 3-5-1 Validation of analysis procedure for Assay-Specificity-Blank

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FA-0101.D

样品名称: KB

```
=====
操作者       : Weijing                      序列行   :    1
仪器         : 1260-2                      位置     : Pl-F-01
进样日期     : 2014/10/21 15:06:34          进样次数  :    1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/21 15:06:01 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:32:22 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

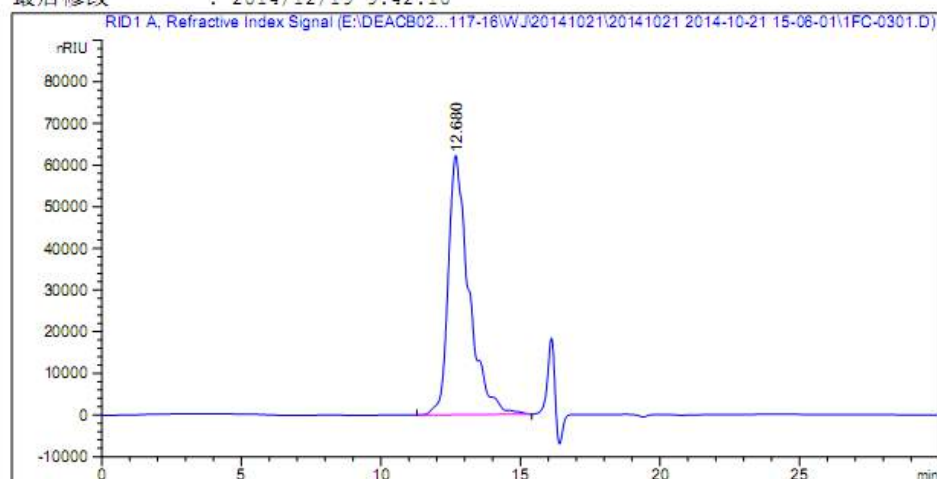
附图10.5-4 SBECD含量测定方法验证图(专属性-空白溶剂)

Annex 3-5-2 Validation of analysis procedure for Assay-Specificity-Reference solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FC-0301.D

样品名称: DZ1

```
=====
操作者       : Weijing                      序列行   :    3
仪器         : 1260-2                      位置     : P1-F-03
进样日期     : 2014/10/21 16:07:43          进样次数  :    1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/12/19 9:42:10
=====
```



面积百分比报告 (包含性能计算)

```
=====
乘积因子       : 1.0000
稀释因子       : 1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.680	-	3.21922e6	6.22476e4	0.54	0.6927	1856	-	-

*** 报告结束 ***

附图10.5-5 SBECD含量测定方法验证图 (专属性\重复性对照-1\进样精密度-1)

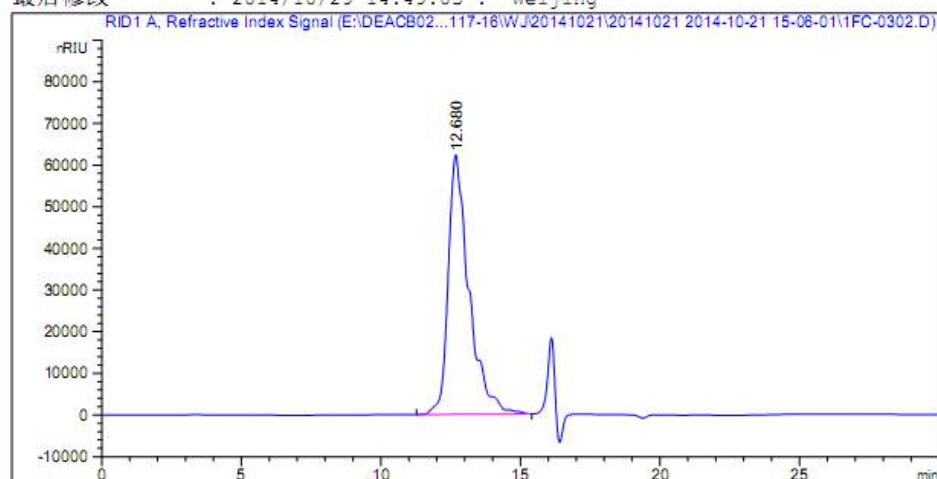
Annex 3-5-3 Validation of analysis procedure for Assay-System suitability-Solution 2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FC-0302.D

样品名称: DZ1

```
=====
操作者       : Weijing                      序列行   :    3
仪器         : 1260-2                      位置     : P1-F-03
进样日期     : 2014/10/21 16:38:17          进样次数  :    2
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:49:03 : Weijing
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.680	BB	0.7167	3.22867e6	6.23238e4	100.0000

总量 : 3.22867e6 6.23238e4

*** 报告结束 ***

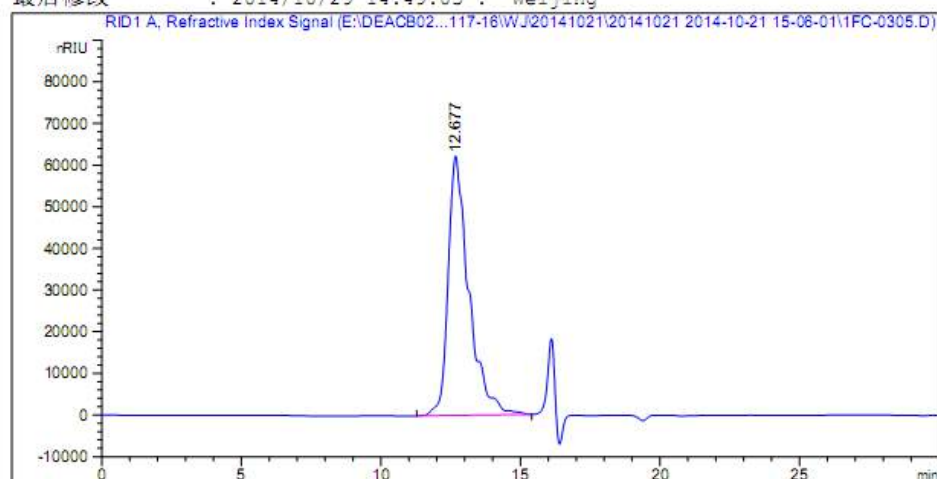
附图10.5-6 SBECD含量测定方法验证图 (重复性对照-2\进样精密度-2)

Annex 3-5-4 Validation of analysis procedure for Assay-System suitability-Solution 5

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FC-0305.D

样品名称: DZ1

```
=====
操作者       : Weijing                      序列行   :    3
仪器         : 1260-2                      位置     : P1-F-03
进样日期     : 2014/10/21 18:10:00          进样次数  :    5
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:49:03 : Weijing
=====
```



面积百分比报告

```
=====
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.677	BB	0.7156	3.21717e6	6.22180e4	100.0000

总量 : 3.21717e6 6.22180e4

*** 报告结束 ***

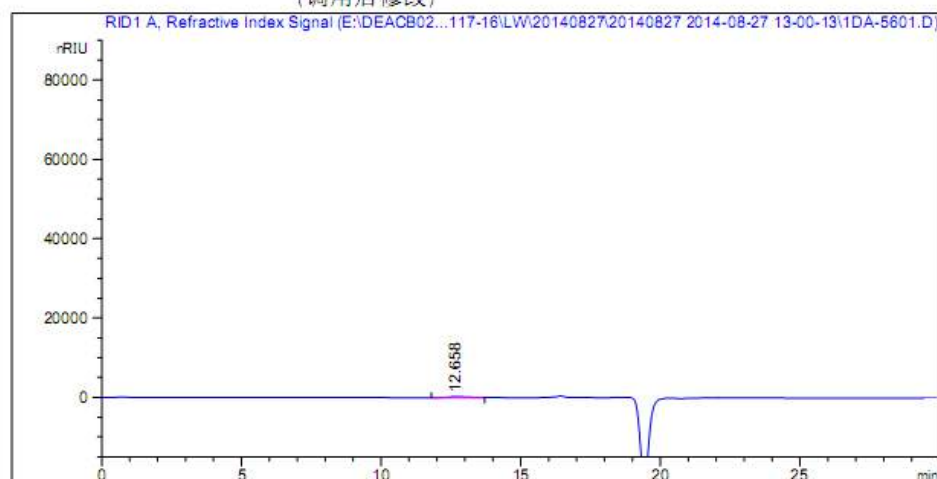
附图10.5-9 SBECD含量测定方法验证图 (重复性对照-5\进样精密度-5)

Annex 3-5-5 Validation of analysis procedure for Assay-LOQ-LOQ1

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\1DA-5601.D
 样品名称: DLX

```

=====
操作者       : linping                      序列行   : 56
仪器        : 1260-2                      位置     : Pl-D-01
进样日期    : 2014/8/29 3:19:02          进样次数  : 1
                                           进样量   : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
              SBECD-0.6.M
最后修改    : 2014/8/28 16:09:34 : linping
分析方法    : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/2/8 20:26:35 : Weijing
              (调用后修改)
=====
  
```



=====
 面积百分比报告 (包含性能和噪声计算)
 =====

```

乘积因子       : 1.0000
稀释因子       : 1.0000
内标中不使用乘积因子和稀释因子
  
```

信号 1: RID1 A, Refractive Index Signal

噪声确定:

时间范围		噪声	噪声	噪声	波动	漂移
开始	结束	(6*SD)	(PtoP)	(ASTM)		
[min]	[min]	[nRIU]	[nRIU]	[nRIU]	[nRIU]	[nRIU/h]
14.000	15.000	26.1986	13.4910	-	-	-1966.481

保留时间	k'	峰面积	峰高	对称	峰宽	塔板数	分离度	信号
[min]		[nRIU*s]	[nRIU]	因子	[min]			噪声
12.658	-	1.37470e4	301.91891	0.57	0.8064	1365	-	11.5

*** 报告结束 ***

附图10.5-11 SBECD含量测定方法验证图 (定量限-1)

Annex 3-5-6 Validation of analysis procedure for Assay-LOQ-LOQ4

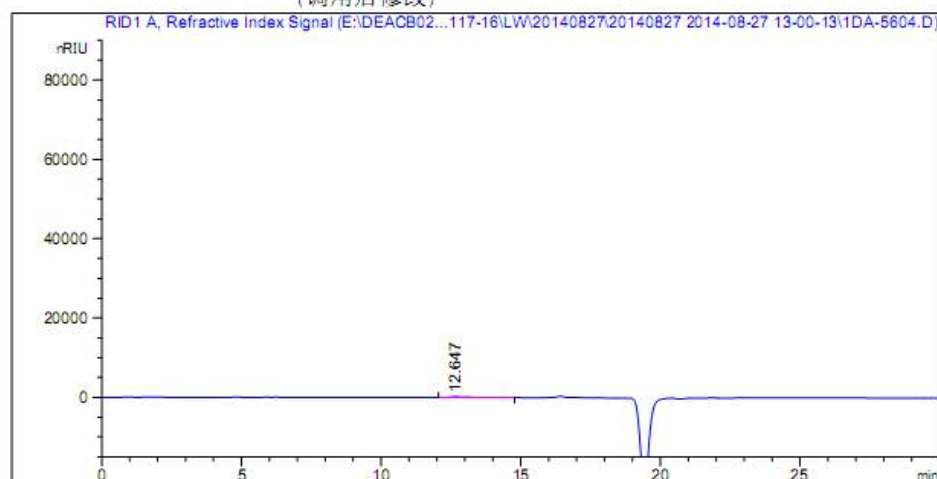
数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\1DA-5604.D

样品名称: DLX

```

=====
操作者       : linping                      序列行 : 56
仪器         : 1260-2                      位置   : Pl-D-01
进样日期     : 2014/8/29 4:50:44          进样次数 : 4
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M
最后修改     : 2014/8/28 16:09:34 : linping
分析方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M (序列方法)
最后修改     : 2015/2/8 20:21:52 : Weijing
              (调用后修改)
=====

```



面积百分比报告 (包含性能和噪声计算)

```

=====
乘积因子       : 1.0000
稀释因子       : 1.0000
内标中不使用乘积因子和稀释因子
=====

```

信号 1: RID1 A, Refractive Index Signal

噪声确定:

时间范围		噪声	噪声	噪声	波动	漂移
开始	结束	(6*SD)	(PtoP)	(ASTM)		
[min]	[min]	[nRIU]	[nRIU]	[nRIU]	[nRIU]	[nRIU/h]
14.000	15.000	20.2222	11.3580	-	-	-2515.106

保留时间	k'	峰面积	峰高	对称	峰宽	塔板数	分离度	信号
[min]		[nRIU*s]	[nRIU]	因子	[min]			噪声
12.647	-	1.27674e4	253.87097	0.39	0.6192	2310	-	12.6

*** 报告结束 ***

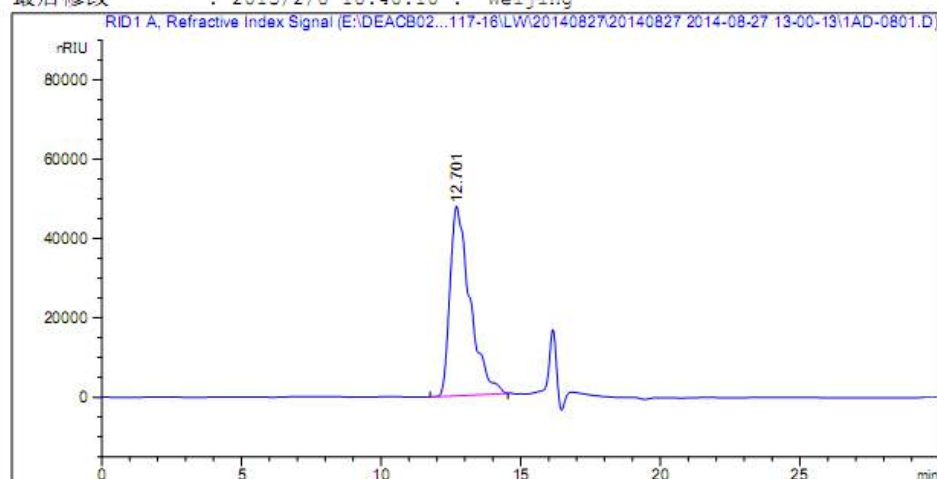
附图10.5-14 SBECD含量测定方法验证图 (定量限-4)

Annex 3-5-7 Validation of analysis procedure for Assay-Linearity-Solution 1

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\1AD-0801.D

样品名称: XX-1

```
=====
操作者       : linping                      序列行 :    8
仪器         : 1260-2                      位置   : Pl-A-04
进样日期     : 2014/8/27 16:44:43          进样次数 :    1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
               SBECD-0.6.M
最后修改     : 2014/8/27 14:34:01 : linping
分析方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2015/2/8 18:40:10 : Weijing
=====
```



面积百分比报告

```
=====
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.701	BB	0.6844	2.38654e6	4.77645e4	100.0000

总量 : 2.38654e6 4.77645e4

*** 报告结束 ***

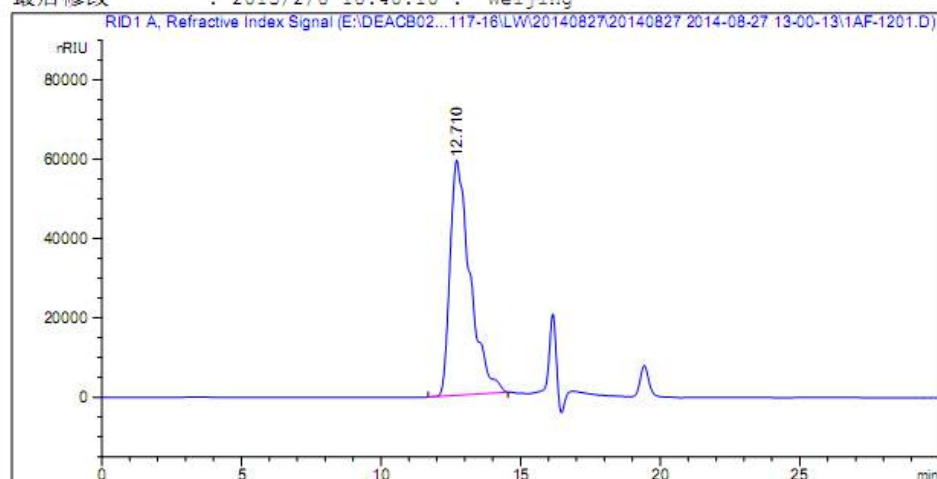
附图10.5-17 SBECD含量测定方法验证图 (线性-1)

Annex 3-5-8 Validation of analysis procedure for Assay-Linearity-Solution 3

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\1AF-1201.D

样品名称: XX-3

```
=====
操作者       : linping                      序列行   : 12
仪器         : 1260-2                      位置     : Pl-A-06
进样日期     : 2014/8/27 18:47:00          进样次数  : 1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
               SBECD-0.6.M
最后修改     : 2014/8/27 14:34:01 : linping
分析方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2015/2/8 18:40:10 : Weijing
=====
```



面积百分比报告

```
=====
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.710	BB	0.6854	2.96691e6	5.92807e4	100.0000

总量 : 2.96691e6 5.92807e4

*** 报告结束 ***

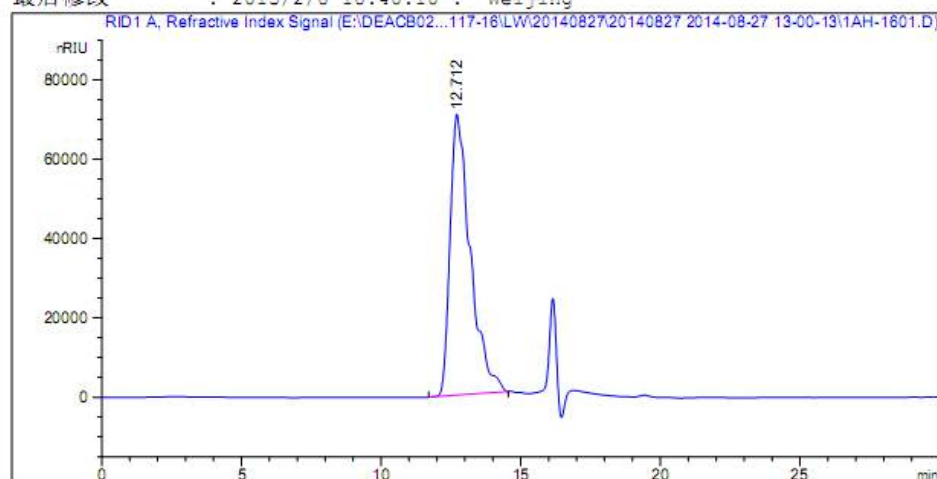
附图10.5-19 SBECD含量测定方法验证图（线性-3）

Annex 3-5-9 Validation of analysis procedure for Assay-Linearity-Solution 5

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\1AH-1601.D

样品名称: XX-5

```
=====
操作者       : linping                      序列行 : 16
仪器         : 1260-2                      位置   : Pl-A-08
进样日期     : 2014/8/27 20:49:16          进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
               SBECD-0.6.M
最后修改     : 2014/8/27 14:34:01 : linping
分析方法     : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2015/2/8 18:40:10 : Weijing
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.712	BB	0.6885	3.56310e6	7.08166e4	100.0000

总量 : 3.56310e6 7.08166e4

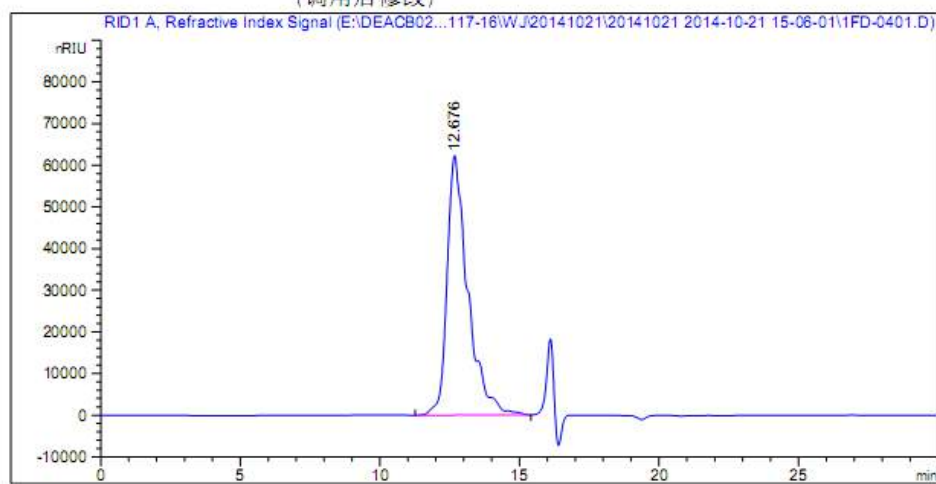
*** 报告结束 ***

附图10.5-21 SBECD含量测定方法验证图 (线性-5)

Annex 3-5-10 Validation of analysis procedure for Assay-Precision-Repeatability reference solution 2-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FD-0401.D
样品名称: D22

```
=====
操作者       : Weijing                      序列行 : 4
仪器         : 1260-2                      位置   : P1-F-04
进样日期     : 2014/10/21 19:11:09          进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:32:22 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.676	BB	0.7169	3.22571e6	6.22435e4	100.0000

总量 : 3.22571e6 6.22435e4

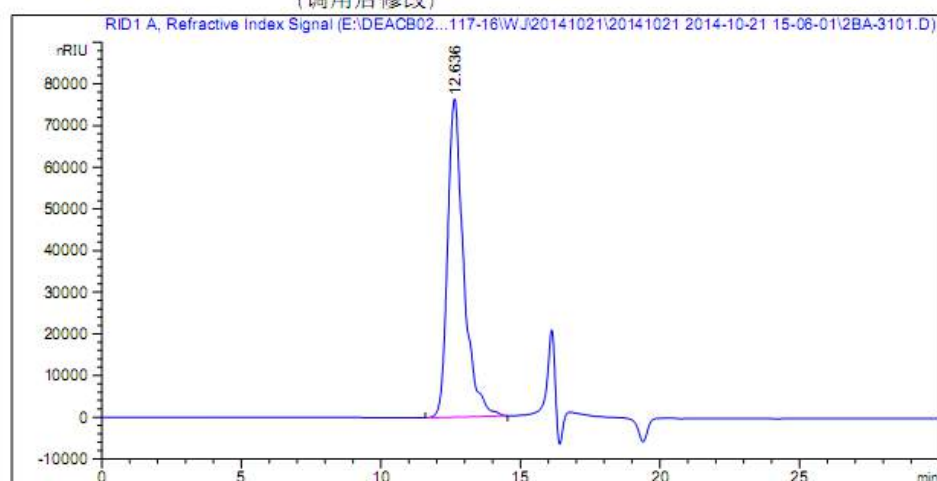
=====
*** 报告结束 ***

附图10.5-35 SBECD含量测定方法验证图 (重复性-对照2-1)

Annex 3-5-11 Validation of analysis procedure for Assay-Precision-Repeatability sample solution 1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2BA-3101.D
样品名称: cfx-1

```
=====
操作者       : Weijing                      序列行 : 31
仪器         : 1260-2                      位置   : P2-B-01
进样日期     : 2014/10/22 16:04:29         进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/22 14:56:33 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:38:18 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.636	BB	0.5901	3.06498e6	7.62164e4	100.0000

总量 : 3.06498e6 7.62164e4

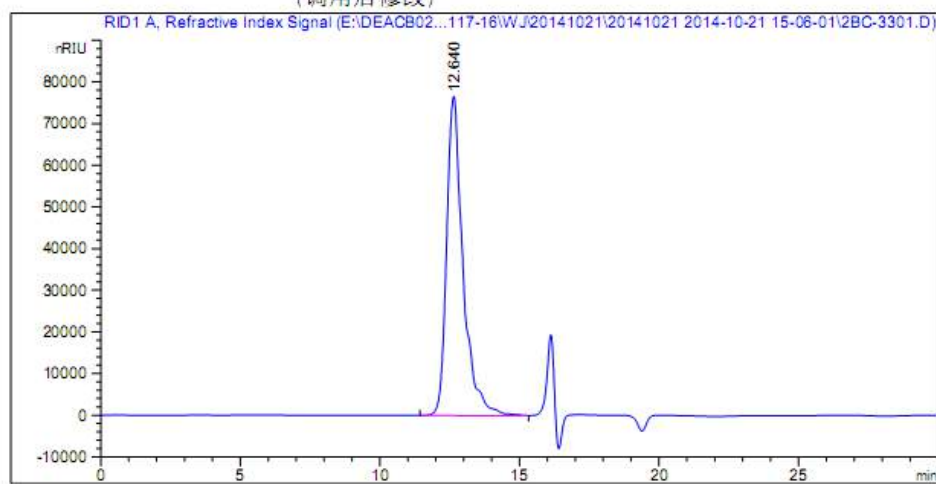
=====
*** 报告结束 ***

附图10.5-38 SBECD含量测定方法验证图 (重复性-样品1)

Annex 3-5-12 Validation of analysis procedure for Assay-Precision-Repeatability sample solution 3

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2BC-3301.D
样品名称: cfx-3

```
=====
操作者       : Weijing                      序列行 : 33
仪器         : 1260-2                      位置   : P2-B-03
进样日期     : 2014/10/22 17:05:37         进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/22 16:50:55 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:38:18 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.640	BBA	0.5878	3.12625e6	7.67112e4	100.0000

总量 : 3.12625e6 7.67112e4

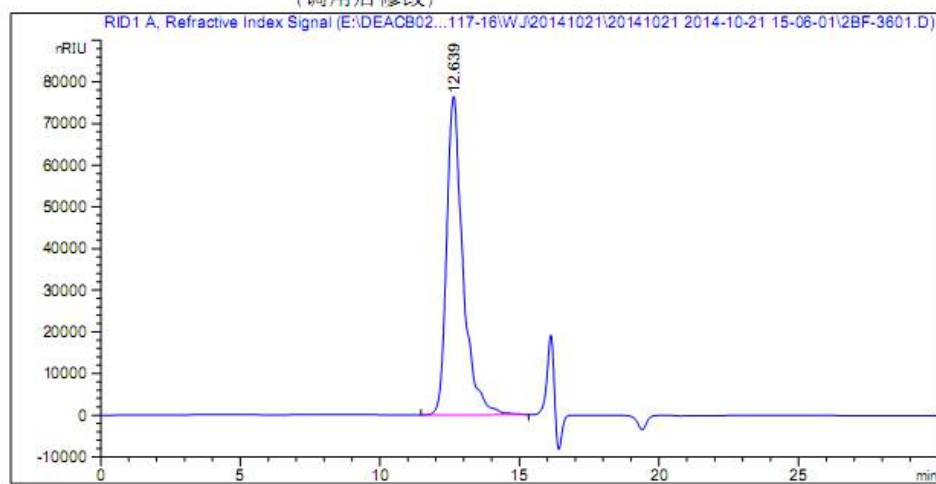
=====
*** 报告结束 ***

附图10.5-40 SBECD含量测定方法验证图 (重复性-样品3)

Annex 3-5-13 Validation of analysis procedure for Assay-Precision-Repeatability sample solution 6

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2BF-3601.D
样品名称: cfx-6

```
=====
操作者       : Weijing                      序列行 : 36
仪器         : 1260-2                      位置   : P2-B-06
进样日期     : 2014/10/22 18:37:22         进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/22 16:50:55 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:38:18 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.639	BBA	0.5896	3.12111e6	7.62954e4	100.0000

总量 : 3.12111e6 7.62954e4

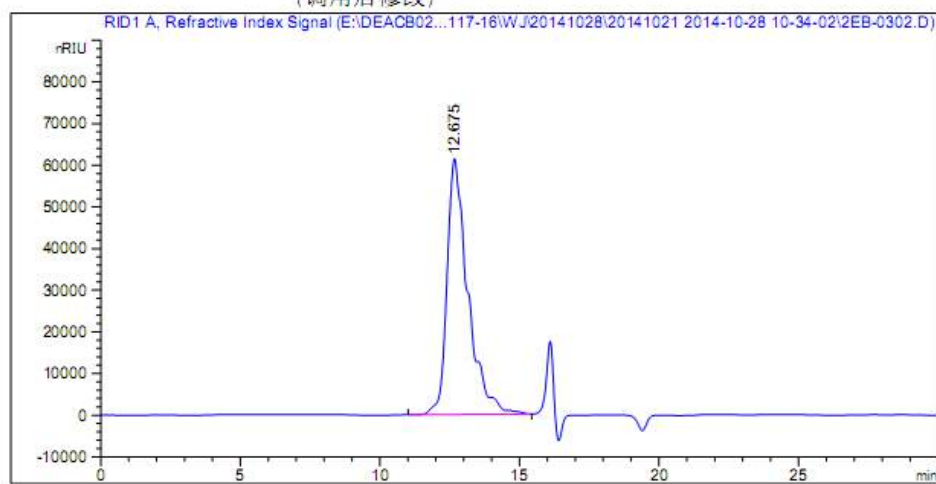
=====
*** 报告结束 ***

附图10.5-43 SBECD含量测定方法验证图 (重复性-样品6)

Annex 3-5-14 Validation of analysis procedure for Assay-Precision-Intermediate precision reference solution 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\2EB-0302.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   :    3
仪器         : 1260-2                      位置     : P2-E-02
进样日期     : 2014/10/28 12:06:20         进样次数  :    2
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/28 10:34:02 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:57:28 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.675	BB	0.7186	3.18973e6	6.13848e4	100.0000

总量 : 3.18973e6 6.13848e4

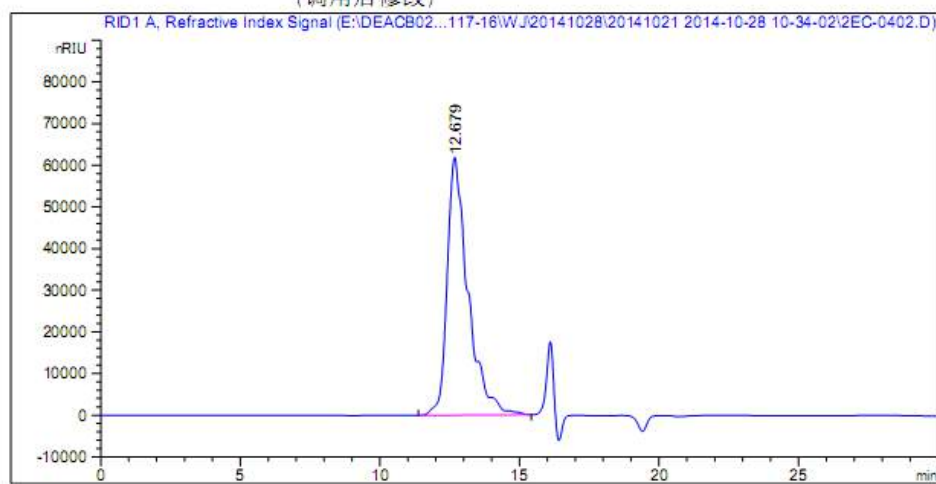
=====
*** 报告结束 ***

附图10.5-24 SBECD含量测定方法验证图 (中间精密度-对照1-2)

Annex 3-5-15 Validation of analysis procedure for Assay-Precision-Intermediate precision reference solution 2-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\2EC-0402.D
样品名称: DZ-2

```
=====
操作者       : Weijing                      序列行 : 4
仪器         : 1260-2                      位置   : P2-E-03
进样日期     : 2014/10/28 13:38:05         进样次数 : 2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/28 12:46:32 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:57:28 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.679	BB	0.7197	3.22252e6	6.18953e4	100.0000

总量 : 3.22252e6 6.18953e4

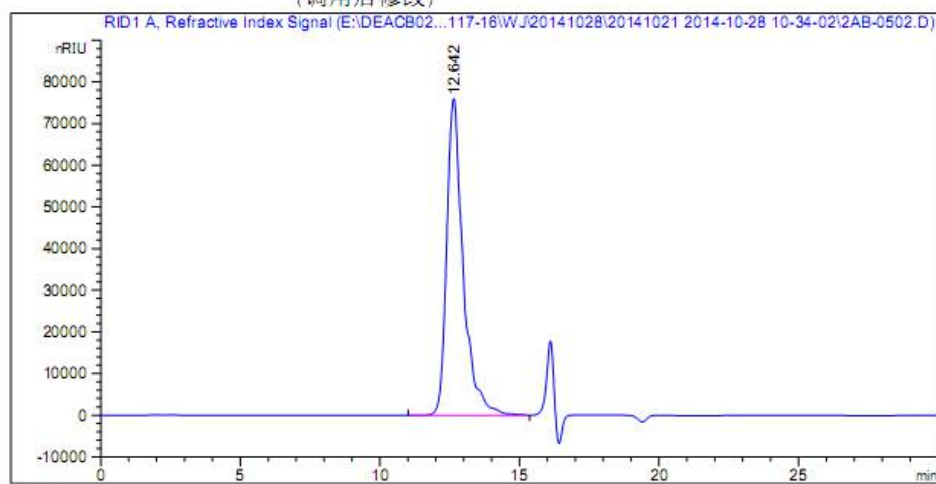
=====
*** 报告结束 ***

附图10.5-27 SBECD含量测定方法验证图 (中间精密度-对照2-2)

Annex 3-5-16 Validation of analysis procedure for Assay-Precision-Intermediate precision sample solution 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\2AB-0502.D
样品名称: 中间精密度-1

```
=====
操作者       : Weijing                      序列行 : 5
仪器         : 1260-2                      位置   : P2-A-02
进样日期     : 2014/10/28 15:09:48         进样次数 : 2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/28 12:46:32 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:57:28 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.642	BB	0.5904	3.11839e6	7.60968e4	100.0000

总量 : 3.11839e6 7.60968e4

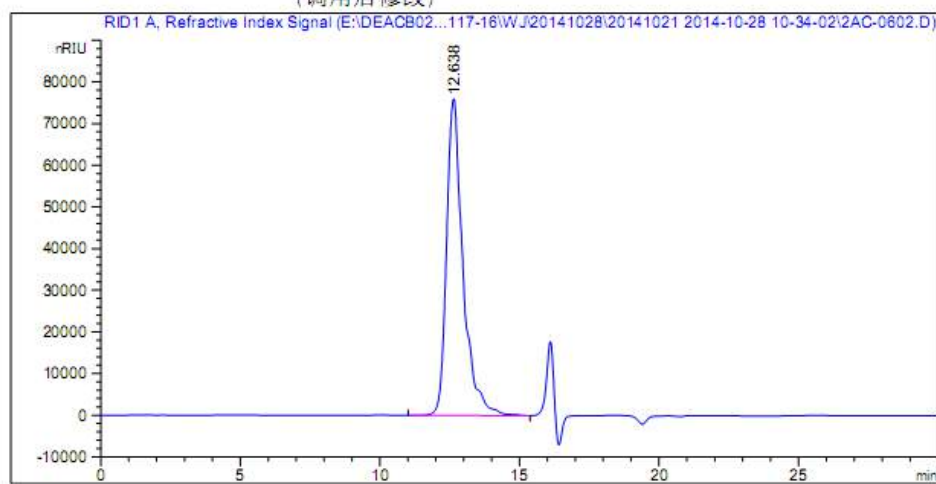
=====
*** 报告结束 ***

附图10.5-30 SBECD含量测定方法验证图 (中间精密度-样品1-2)

Annex 3-5-17 Validation of analysis procedure for Assay-Precision-Intermediate precision sample solution 2-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\2AC-0602.D
样品名称: 中间精密度-2

```
=====
操作者       : Weijing                      序列行 :    6
仪器         : 1260-2                      位置   : P2-A-03
进样日期     : 2014/10/28 16:10:58         进样次数:    2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/28 12:46:32 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:57:28 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.638	BB	0.5911	3.11311e6	7.58540e4	100.0000

总量 : 3.11311e6 7.58540e4

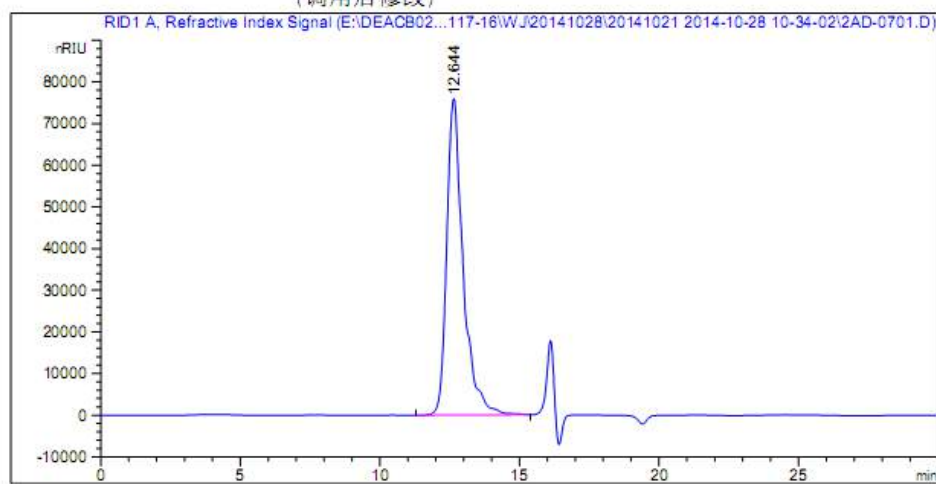
=====
*** 报告结束 ***

附图10.5-32 SBECD含量测定方法验证图 (中间精密度-样品2-2)

Annex 3-5-18 Validation of analysis procedure for Assay-Precision-Intermediate precision sample solution 3-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\2AD-0701.D
样品名称: 中间精密度-3

```
=====
操作者       : Weijing                      序列行 :    7
仪器         : 1260-2                      位置   : P2-A-04
进样日期     : 2014/10/28 16:41:32          进样次数:    1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/28 12:46:32 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141028\20141021 2014-10-28 10-34-02\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:57:28 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.644	BB	0.5890	3.10643e6	7.60321e4	100.0000

总量 : 3.10643e6 7.60321e4

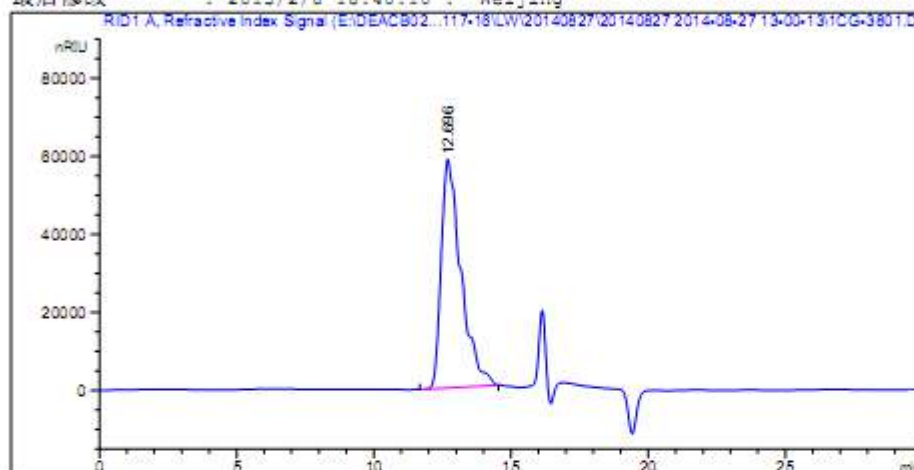
=====
*** 报告结束 ***

附图10.5-33 SBECD含量测定方法验证图 (中间精密度-样品3-1)

Annex 3-5-19 Validation of analysis procedure for Assay-Solution stability-Reference solution 0h

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\ICG-3801.D
样品名称: DZ-1

操作者 : linping 序列行 : 38
仪器 : 1260-2 位置 : P1-C-07
进样日期 : 2014/8/28 11:35:43 进样次数 : 1
进样量 : 20.000 µl
采集方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M
最后修改 : 2014/8/27 14:34:01 : linping
分析方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M (序列方法)
最后修改 : 2015/2/8 18:40:10 : Weijing

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面积百分比报告

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.696	BB	0.6857	2.93511e6	5.86182e4	100.0000

总量 : 2.93511e6 5.86182e4

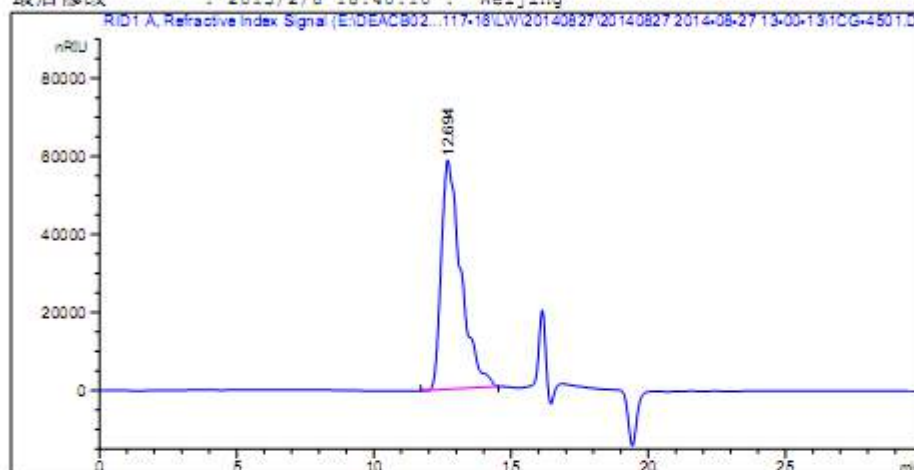
*** 报告结束 ***

附图10.5-44 SBECD含量测定方法验证图 (对照溶液稳定性-0h)

Annex 3-5-20 Validation of analysis procedure for Assay-Solution stability-Reference solution 4h

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\ICG-4501.D
样品名称: DZ-1-4h

操作者 : linping 序列行 : 45
仪器 : 1260-2 位置 : P1-C-07
进样日期 : 2014/8/28 15:40:38 进样次数 : 1
进样量 : 20.000 µl
采集方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M
最后修改 : 2014/8/27 14:34:01 : linping
分析方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M (序列方法)
最后修改 : 2015/2/8 18:40:10 : Weijing

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面积百分比报告

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.694	BB	0.6863	2.94304e6	5.87129e4	100.0000

总量 : 2.94304e6 5.87129e4

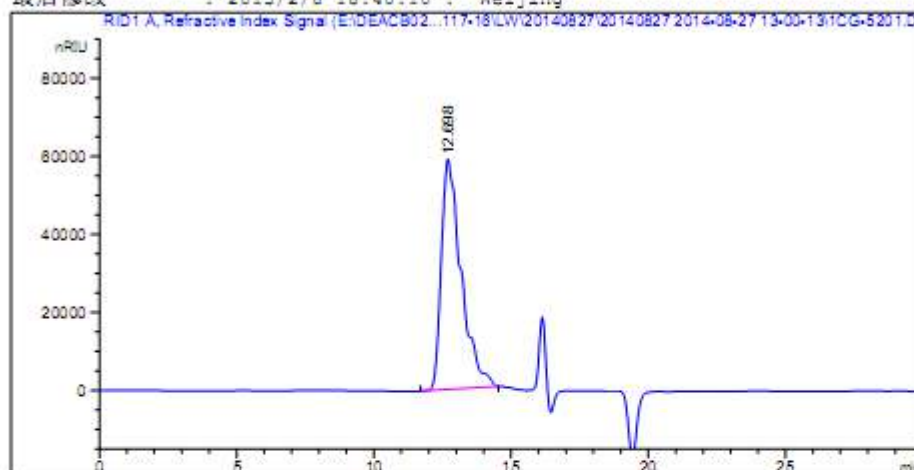
*** 报告结束 ***

附图10.5-47 SBECD含量测定方法验证图 (对照溶液稳定性-4H)

Annex 3-5-21 Validation of analysis procedure for Assay-Solution stability-Reference solution 10h

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\ICG-5201.D
样品名称: DZ-1-10h

操作者 : linping 序列行 : 52
仪器 : 1260-2 位置 : P1-C-07
进样日期 : 2014/8/28 21:44:09 进样次数 : 1
进样量 : 20.000 µl
采集方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M
最后修改 : 2014/8/28 16:09:34 : linping
分析方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M (序列方法)
最后修改 : 2015/2/8 18:40:10 : Weijing

-----
面积百分比报告

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.698	BB	0.6841	2.95936e6	5.90470e4	100.0000

总量 : 2.95936e6 5.90470e4

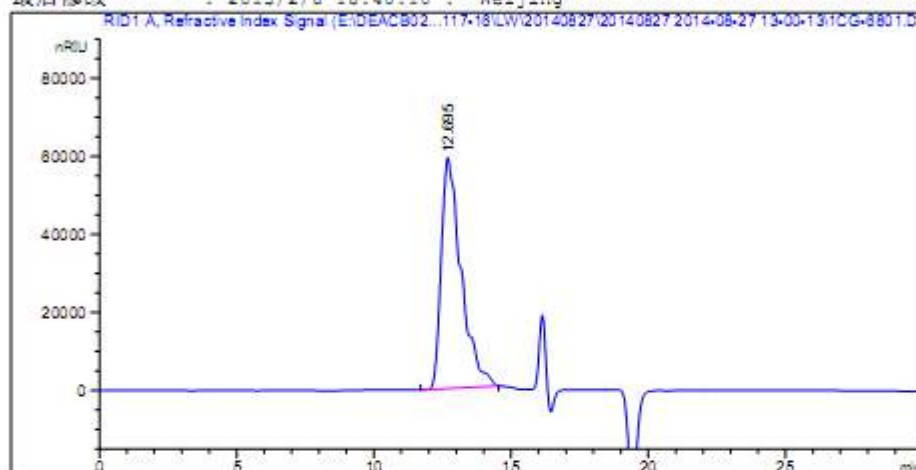
*** 报告结束 ***

附图10.5-50 SBECD含量测定方法验证图 (对照溶液稳定性-10H)

Annex 3-5-22 Validation of analysis procedure for Assay-Solution stability-Reference solution 24h

数据文件: E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\ICG-6801.D
样品名称: DZ-1-24h

操作者 : linping 序列行 : 68
仪器 : 1260-2 位置 : P1-C-07
进样日期 : 2014/8/29 11:53:30 进样次数 : 1
进样量 : 20.000 µl
采集方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M
最后修改 : 2014/8/29 9:25:21 : linping
分析方法 : E:\DEACB02694\SIM117-16\LW\20140827\20140827 2014-08-27 13-00-13\117-16-
SBECD-0.6.M (序列方法)
最后修改 : 2015/2/8 18:40:10 : Weijing

-----
面积百分比报告

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.695	BB	0.6878	2.97338e6	5.91738e4	100.0000

总量 : 2.97338e6 5.91738e4

*** 报告结束 ***

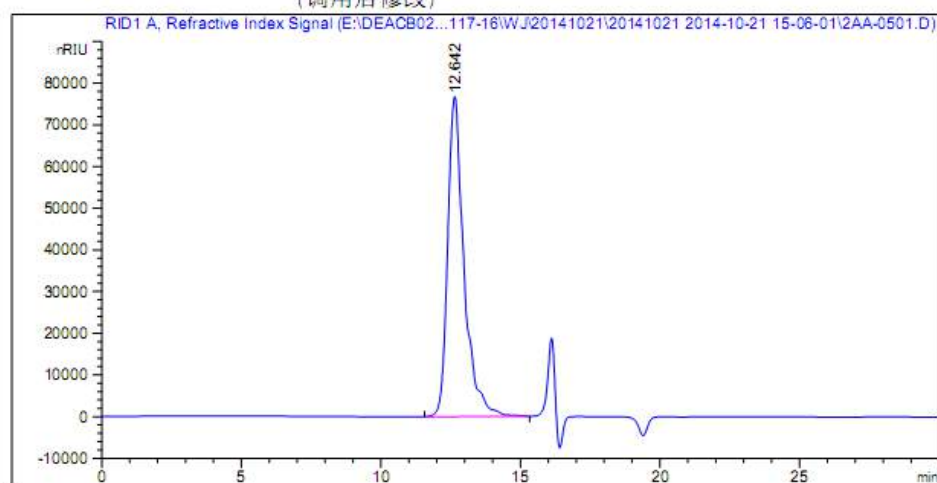
附图10.5-52 SBECD含量测定方法验证图 (对照溶液稳定性-24h)

Annex 3-5-23 Validation of analysis procedure for Assay-Solution stability-Sample solution
0h

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2AA-0501.D
样品名称: WDX-0h

=====

操作者	: Weijing	序列行	: 5
仪器	: 1260-2	位置	: P2-A-01
进样日期	: 2014/10/21 20:42:52	进样次数	: 1
		进样量	: 20.000 µl
采集方法	: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-SBECD-0.6.M		
最后修改	: 2014/10/21 16:02:21 : Weijing		
分析方法	: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-SBECD-0.6.M (序列方法)		
最后修改	: 2014/10/29 14:35:33 : Weijing (调用后修改)		



面积百分比报告

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.642	BBA	0.5868	3.12367e6	7.67995e4	100.0000

总量 : 3.12367e6 7.67995e4

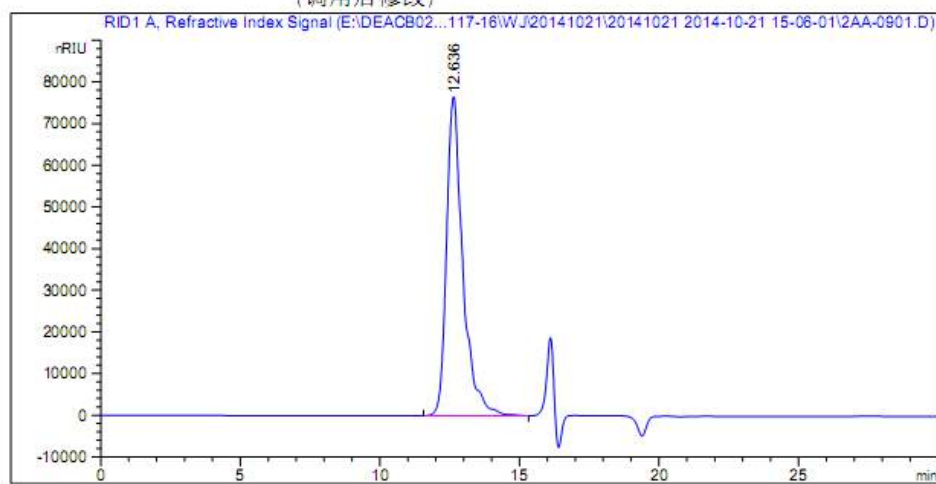
*** 报告结束 ***

附图10. 5-53 SBECD含量测定方法验证图 (样品溶液稳定性-避光0h)

Annex 3-5-24 Validation of analysis procedure for Assay-Solution stability-Sample solution stored in darkness for 2h

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2AA-0901.D
样品名称: WDX-2h

```
=====
操作者       : Weijing                      序列行   :    9
仪器         : 1260-2                      位置     : P2-A-01
进样日期     : 2014/10/21 22:45:09          进样次数  :    1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:35:33 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.636	BBA	0.5972	3.11997e6	7.64351e4	100.0000

总量 : 3.11997e6 7.64351e4

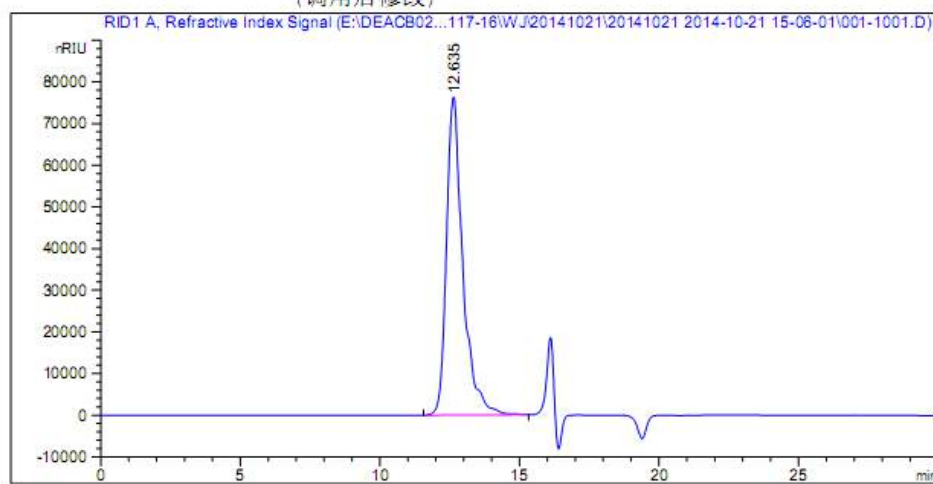
=====
*** 报告结束 ***

附图10.5-57 SBECD含量测定方法验证图 (样品溶液稳定性-避光2H)

Annex 3-5-25 Validation of analysis procedure for Assay-Solution stability-Sample solution stored in normal place for 2h

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\001-1001.D
样品名称: WDX-light2h

```
=====
操作者       : Weijing                      序列行 : 10
仪器         : 1260-2                      位置   : 样品瓶 1
进样日期     : 2014/10/21 23:15:42         进样次数 : 1
                                           进样量 : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:35:33 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.635	BBA	0.5972	3.10921e6	7.61738e4	100.0000

总量 : 3.10921e6 7.61738e4

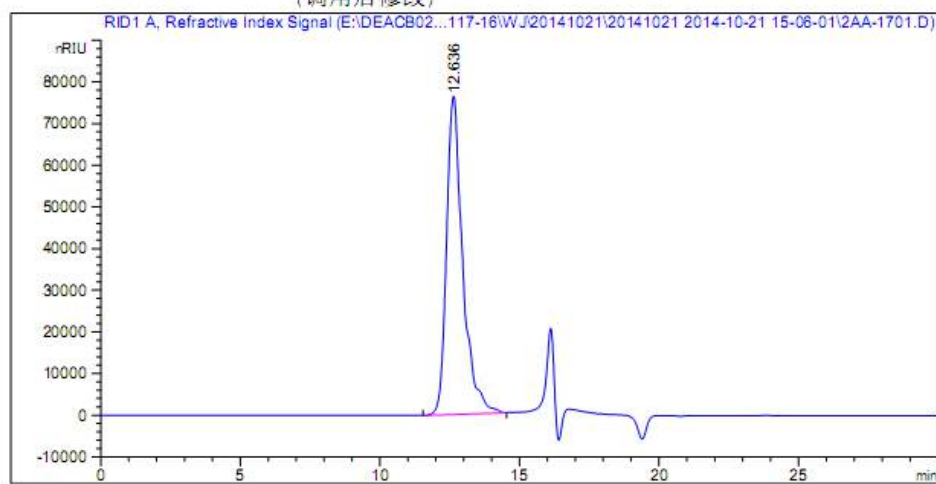
=====
*** 报告结束 ***

附图10.5-58 SBECD含量测定方法验证图 (样品溶液稳定性-不避光2H)

Annex 3-5-26 Validation of analysis procedure for Assay-Solution stability-Sample solution stored in darkness for 8h

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2AA-1701.D
样品名称: WDX-8h

```
=====
操作者       : Weijing                      序列行 : 17
仪器         : 1260-2                      位置   : P2-A-01
进样日期     : 2014/10/22 4:51:58          进样次数: 1
                                           进样量 : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:38:18 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.636	BB	0.5920	3.07771e6	7.62331e4	100.0000

总量 : 3.07771e6 7.62331e4

=====
*** 报告结束 ***

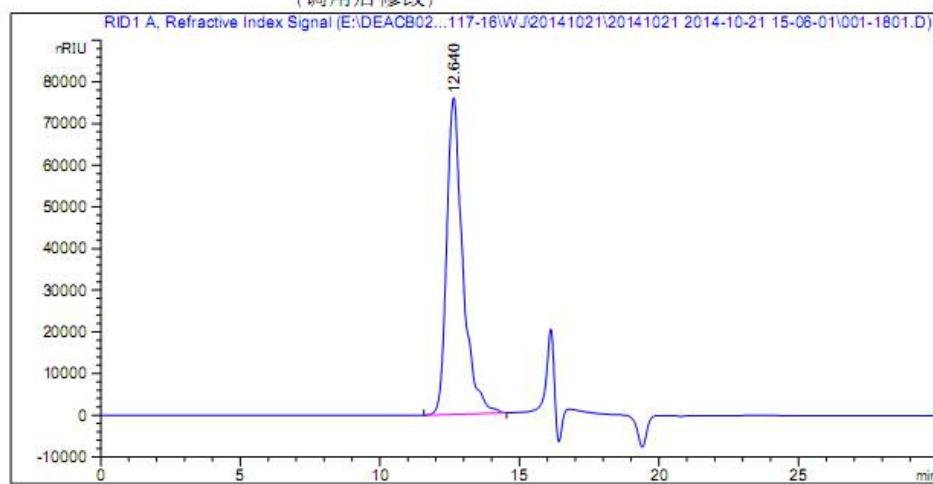
附图10.5-61 SBECD含量测定方法验证图 (样品溶液稳定性-避光8H)

Annex 3-5-27 Validation of analysis procedure for Assay-Solution stability-Sample solution stored in normal place for 8h

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\001-1801.D
样品名称: WDX-light8h

=====

操作者	: Weijing	序列行	: 18
仪器	: 1260-2	位置	: 样品瓶 1
进样日期	: 2014/10/22 5:22:31	进样次数	: 1
		进样量	: 20.000 µl
采集方法	: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-SBECB-0.6.M		
最后修改	: 2014/10/21 16:02:21 : Weijing		
分析方法	: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-SBECB-0.6.M (序列方法)		
最后修改	: 2014/10/29 14:38:18 : Weijing (调用后修改)		



面积百分比报告

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.640	BB	0.5810	3.05707e6	7.60922e4	100.0000

总量 : 3.05707e6 7.60922e4

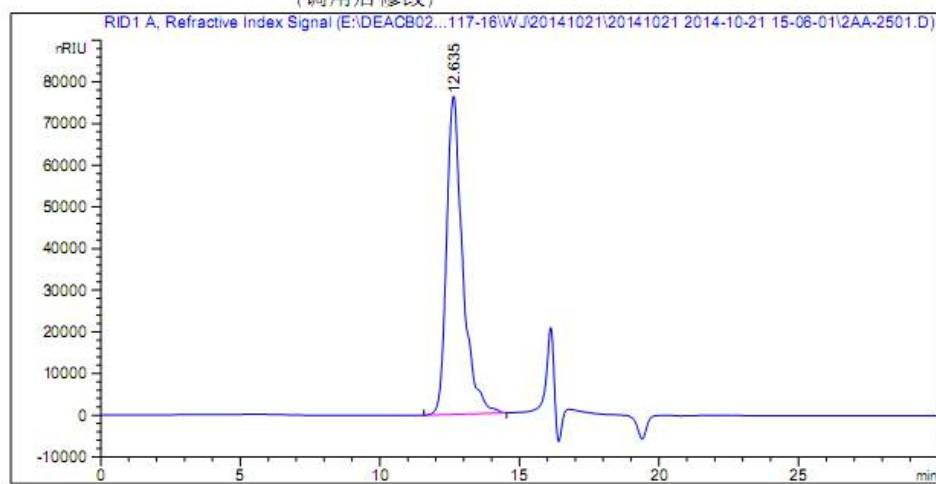
*** 报告结束 ***

附图10.5-62 SBECB含量测定方法验证图 (样品溶液稳定性-不避光8H)

Annex 3-5-28 Validation of analysis procedure for Assay-Solution stability-Sample solution stored in darkness for 14h

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2AA-2501.D
样品名称: WDX-14h

```
=====
操作者       : Weijing                      序列行 : 25
仪器         : 1260-2                      位置   : P2-A-01
进样日期     : 2014/10/22 10:58:45         进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/22 10:34:48 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:38:18 : Weijing
                                           (调用后修改)
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.635	BB	0.5908	3.06887e6	7.62103e4	100.0000

总量 : 3.06887e6 7.62103e4

=====
*** 报告结束 ***

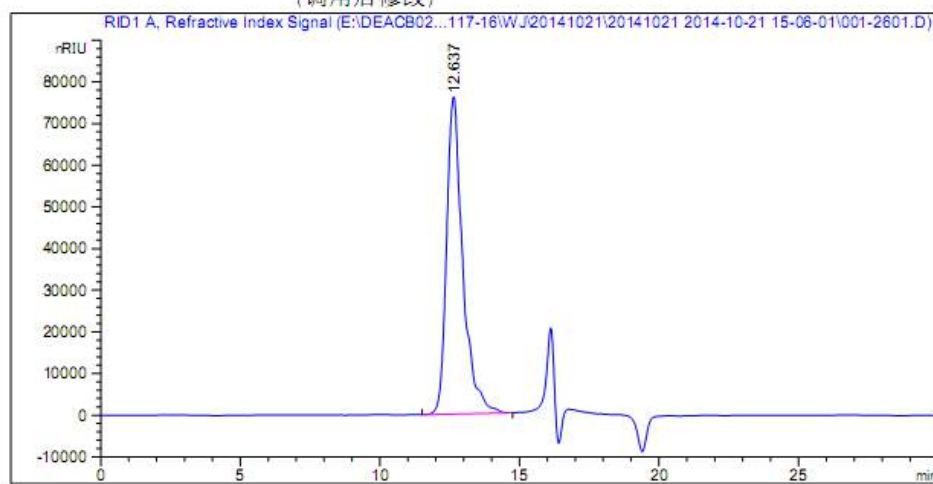
附图10.5-65 SBECD含量测定方法验证图 (样品溶液稳定性-避光14h)

Annex 3-5-29 Validation of analysis procedure for Assay-Solution stability-Sample solution stored in normal place for 14h

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\001-2601.D
样品名称: WDX-light14h

=====

操作者	: Weijing	序列行	: 26
仪器	: 1260-2	位置	: 样品瓶 1
进样日期	: 2014/10/22 11:29:18	进样次数	: 1
		进样量	: 20.000 µl
采集方法	: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-SBECD-0.6.M		
最后修改	: 2014/10/22 10:34:48 : Weijing		
分析方法	: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-SBECD-0.6.M (序列方法)		
最后修改	: 2014/10/29 14:38:18 : Weijing (调用后修改)		



=====

面积百分比报告

=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.637	BB	0.5839	3.07140e6	7.59813e4	100.0000

总量 : 3.07140e6 7.59813e4

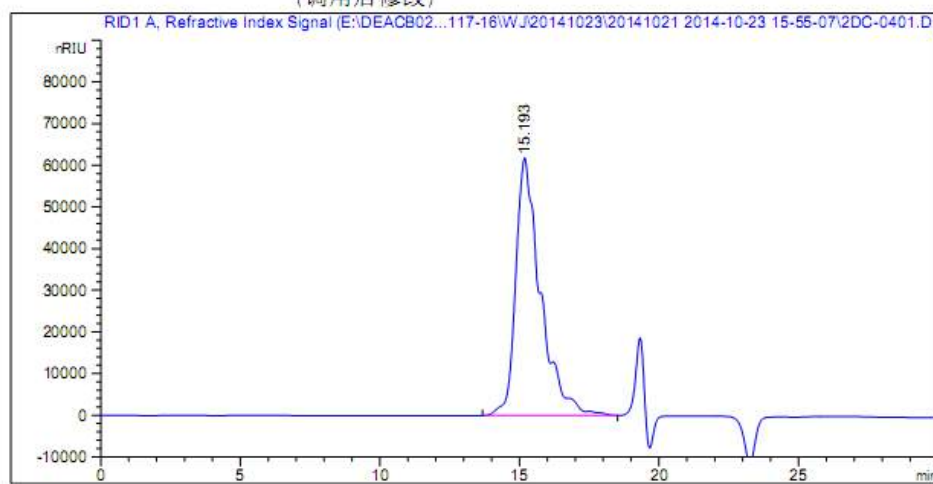
*** 报告结束 ***

附图10.5-66 SBECD含量测定方法验证图 (样品溶液稳定性-不避光14h)

Annex 3-5-30 Validation of analysis procedure for Assay-Robustness-Condition 2- Reference solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DC-0401.D
样品名称: DZ-2

```
=====
操作者       : Weijing                      序列行   :    4
仪器         : 1260-2                      位置     : P2-D-03
进样日期     : 2014/10/23 17:58:01          进样次数  :    1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-0.5.M
最后修改     : 2014/10/23 15:55:07 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-0.5.M (序列方法)
最后修改     : 2015/3/31 14:26:32 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
15.193	-	3.81587e6	6.17390e4	0.53	0.8029	1982	-	-

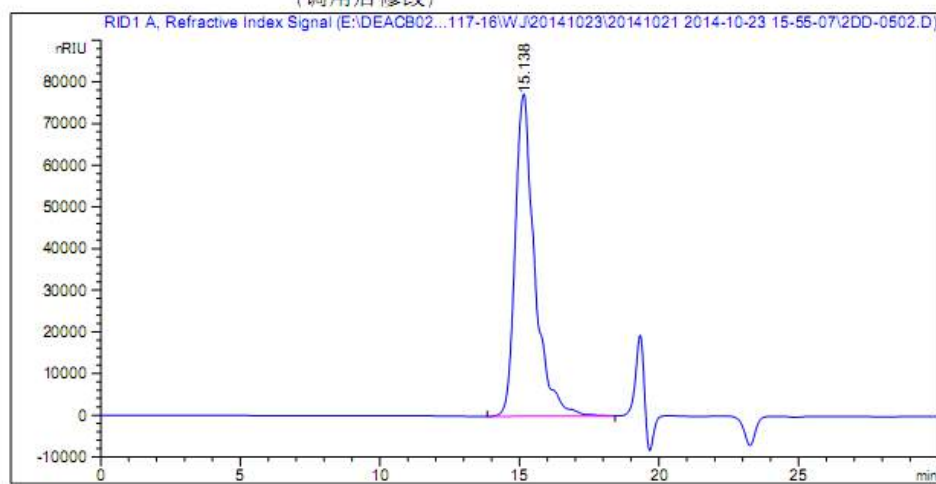
=====
*** 报告结束 ***

附图10.5-72 SBECD含量测定方法验证图 (方法耐用性-流速0.5ml/min-对照)

Annex 3-5-31 Validation of analysis procedure for Assay- Robustness-Condition 2- Sample solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DD-0502.D
样品名称: NYX-0.5ml/min-1

```
=====
操作者       : Weijing                      序列行   :    5
仪器         : 1260-2                      位置     : P2-D-04
进样日期     : 2014/10/23 19:29:44         进样次数  :    2
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-0.5.M
最后修改     : 2014/10/23 15:55:07 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-0.5.M (序列方法)
最后修改     : 2015/3/31 14:26:32 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
15.138	-	3.76548e6	7.66902e4	0.64	0.6981	2611	-	-

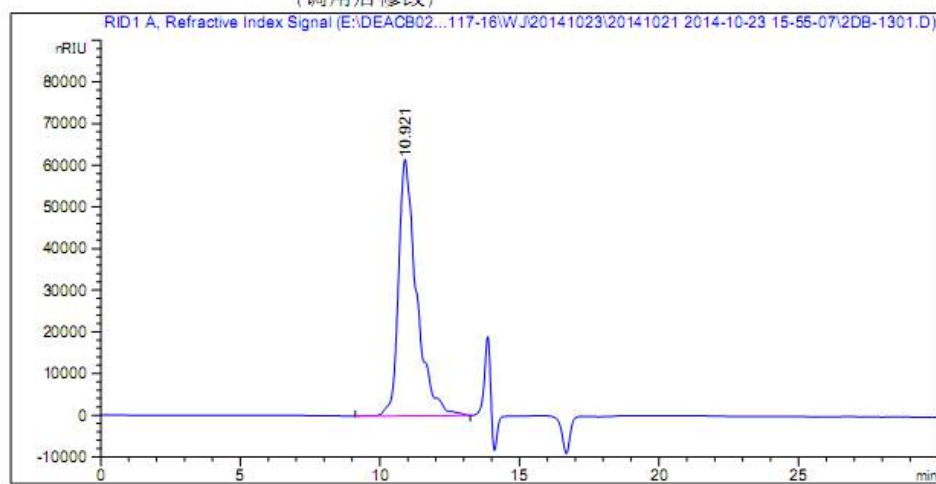
=====
*** 报告结束 ***
=====

附图10.5-75 SBECD含量测定方法验证图 (方法耐用性-流速0.5ml/min-样品)

Annex 3-5-32 Validation of analysis procedure for Assay-Robustness-Condition 3- Reference solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DB-1301.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行 : 13
仪器         : 1260-2                      位置   : P2-D-02
进样日期     : 2014/10/24 0:33:09          进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-0.7.M
最后修改     : 2014/10/23 15:55:07 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-0.7.M (序列方法)
最后修改     : 2015/3/31 14:28:13 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
10.921	-	2.77394e6	6.12365e4	0.58	0.6088	1776	-	-

=====
*** 报告结束 ***

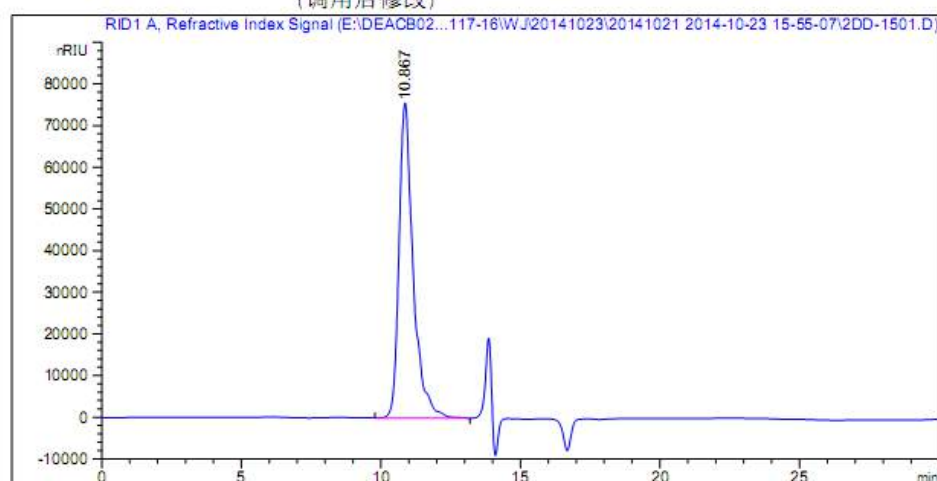
附图10.5-79 SBECD含量测定方法验证图 (方法耐用性-流速0.7ml/min-对照)

Annex 3-5-33 Validation of analysis procedure for Assay- Robustness-Condition 3- Sample solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DD-1501.D
样品名称: NYX-0.7ml/min-1

=====

操作者	: Weijing	序列行	: 15
仪器	: 1260-2	位置	: P2-D-04
进样日期	: 2014/10/24 2:35:27	进样次数	: 1
		进样量	: 20.000 µl
采集方法	: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-SBECB-0.7.M		
最后修改	: 2014/10/23 15:55:07 : Weijing		
分析方法	: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-SBECB-0.7.M (序列方法)		
最后修改	: 2015/3/31 14:28:13 : linping (调用后修改)		



=====

面积百分比报告 (包含性能计算)

=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
10.867	-	2.67422e6	7.53073e4	0.67	0.5040	2574	-	-

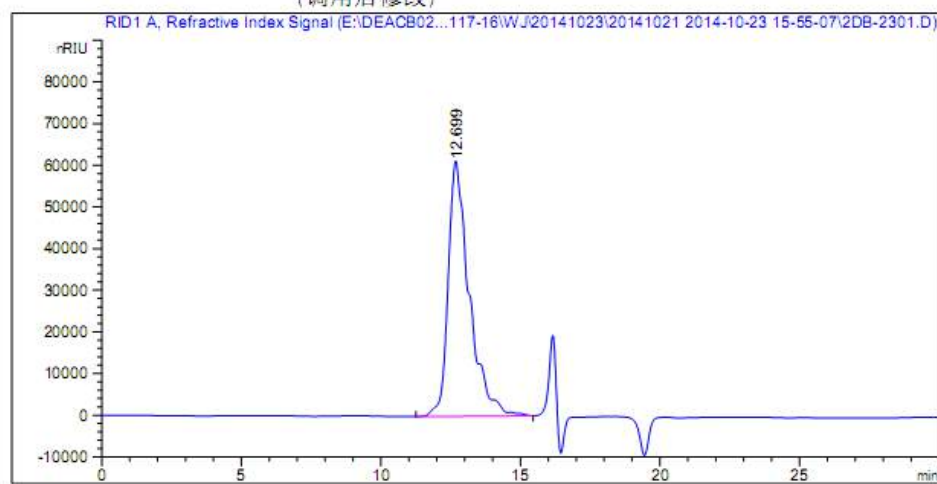
*** 报告结束 ***

附图10.5-83 SBECB含量测定方法验证图 (方法耐用性-流速0.7ml/min-样品)

Annex 3-5-34 Validation of analysis procedure for Assay-Robustness-Condition 4- Reference solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DB-2301.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行 : 23
仪器         : 1260-2                      位置   : P2-D-02
进样日期     : 2014/10/24 8:10:06          进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-33.M
最后修改     : 2014/10/23 15:55:07 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-33.M (序列方法)
最后修改     : 2015/3/31 14:29:30 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.699	-	3.22249e6	6.09808e4	0.56	0.6932	1853	-	-

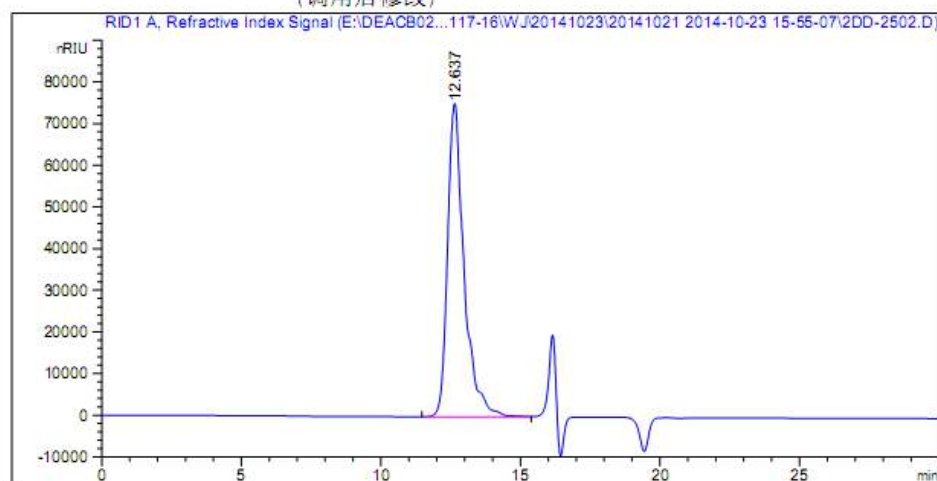
=====
*** 报告结束 ***

附图10.5-88 SBECD含量测定方法验证图 (方法耐用性-柱温33℃-对照1-1)

Annex 3-5-35 Validation of analysis procedure for Assay- Robustness-Condition 4- Sample solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DD-2502.D
样品名称: NYX-33du-1

```
=====
操作者       : Weijing                      序列行   : 25
仪器         : 1260-2                      位置     : P2-D-04
进样日期     : 2014/10/24 10:42:58          进样次数  : 2
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-33.M
最后修改     : 2014/10/24 9:10:40 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-33.M (序列方法)
最后修改     : 2015/3/31 14:29:30 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.637	-	3.10154e6	7.50856e4	0.65	0.5874	2567	-	-

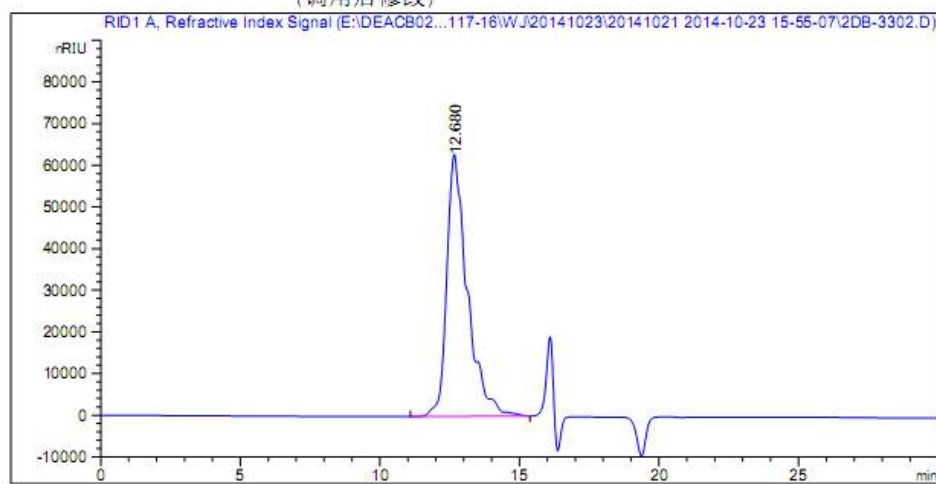
=====
*** 报告结束 ***

附图10.5-93 SBECD含量测定方法验证图 (方法耐用性-柱温33℃-样品1-2)

Annex 3-5-36 Validation of analysis procedure for Assay-Robustness-Condition 5- Reference solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DB-3302.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行 : 33
仪器         : 1260-2                      位置   : P2-D-02
进样日期     : 2014/10/24 16:17:46         进样次数 : 2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-37.M
最后修改     : 2014/10/23 15:55:07 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-37.M (序列方法)
最后修改     : 2015/3/31 14:30:17 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.680	-	3.23292e6	6.20108e4	0.57	0.6786	1930	-	-

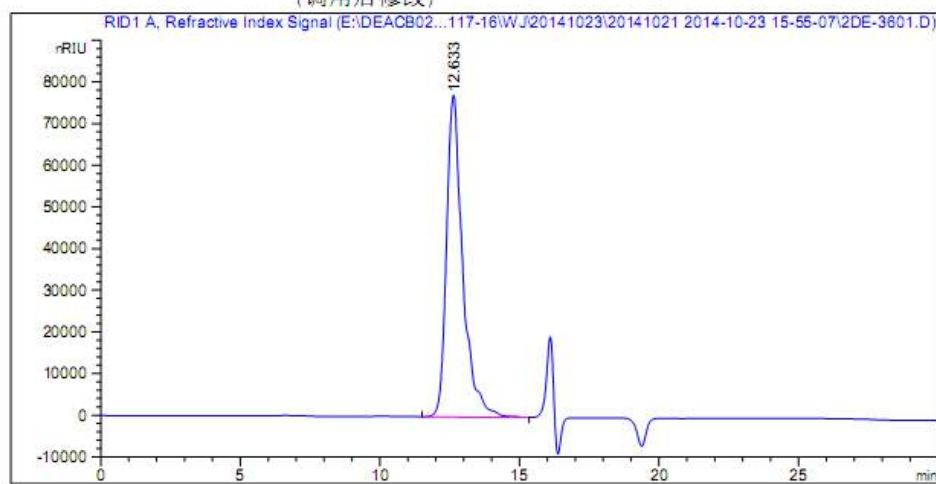
=====
*** 报告结束 ***

附图10.5-98 SBECD含量测定方法验证图 (方法耐用性-柱温37℃-对照1-2)

Annex 3-5-37 Validation of analysis procedure for Assay- Robustness-Condition 5- Sample solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\2DE-3601.D
样品名称: NYX-37du-2

```
=====
操作者       : Weijing                      序列行 : 36
仪器         : 1260-2                      位置   : P2-D-05
进样日期     : 2014/10/24 18:50:37          进样次数 : 1
                                           进样量 : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-37.M
最后修改     : 2014/10/23 15:55:07 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141023\20141021 2014-10-23 15-55-07\117-16-
                                           SBECD-37.M (序列方法)
最后修改     : 2015/3/31 14:30:17 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.633	-	3.12132e6	7.70862e4	0.66	0.5761	2668	-	-

=====
*** 报告结束 ***

附图10.5-103 SBECD含量测定方法验证图 (方法耐用性-柱温37℃-样品2-1)

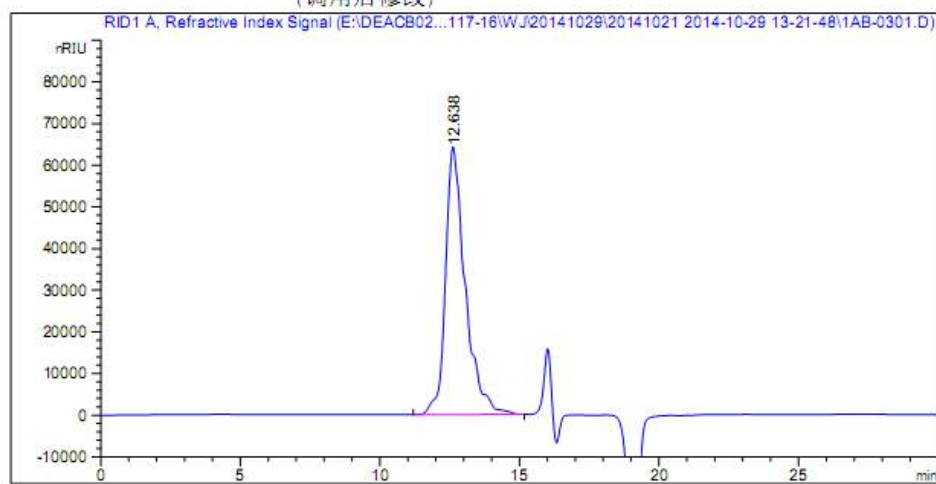
1260-2 2015/3/31 14:30:50 linping

页 1/1

Annex 3-5-38 Validation of analysis procedure for Assay-Robustness-Condition 6- Reference solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 13-21-48\1AB-0301.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   :    3
仪器         : 1260-2                      位置     : Pl-A-02
进样日期     : 2014/10/29 14:23:31          进样次数  :    1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 13-21-48\117-16-
                SBECD-0.6.M
最后修改     : 2014/10/29 13:21:48 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 13-21-48\117-16-
                SBECD-0.6.M (序列方法)
最后修改     : 2015/3/31 14:19:47 : linping
                (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.638	-	3.15155e6	6.37832e4	0.62	0.7032	1783	-	-

=====
*** 报告结束 ***

附图10.5-106 SBECD含量测定方法验证图 (方法耐用性-水相: 有机相=87: 13-对照1-1)

1260-2 2015/3/31 14:19:51 linping

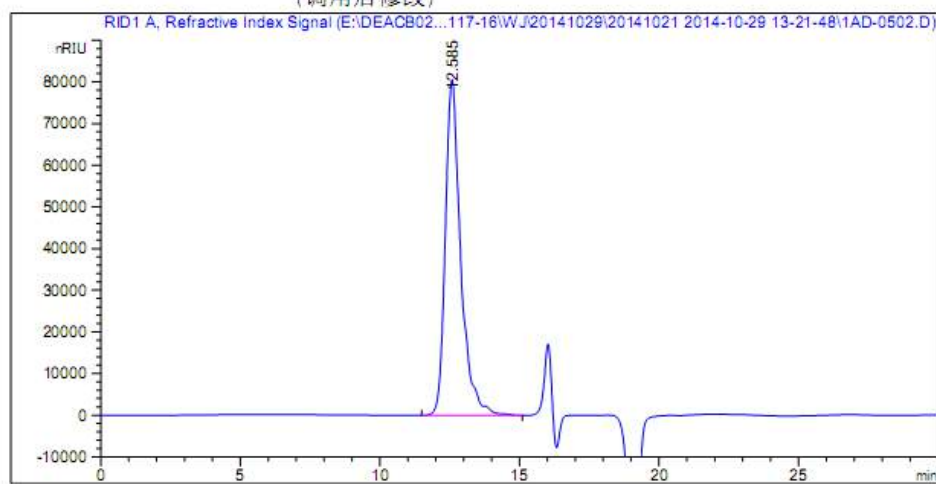
页 1/1

Annex 3-5-39 Validation of analysis procedure for Assay- Robustness-Condition 6- Sample solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 13-21-48\1AD-0502.D
样品名称: NYX-870:130-1

=====

操作者	: Weijing	序列行	: 5
仪器	: 1260-2	位置	: Pl-A-04
进样日期	: 2014/10/29 16:56:23	进样次数	: 2
		进样量	: 20.000 µl
采集方法	: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 13-21-48\117-16-SBEC-0.6.M		
最后修改	: 2014/10/29 15:42:55 : Weijing		
分析方法	: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 13-21-48\117-16-SBEC-0.6.M (序列方法)		
最后修改	: 2015/3/31 14:19:47 : linping (调用后修改)		



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面积百分比报告 (包含性能计算)

=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.585	-	3.09355e6	8.03252e4	0.70	0.5505	2895	-	-

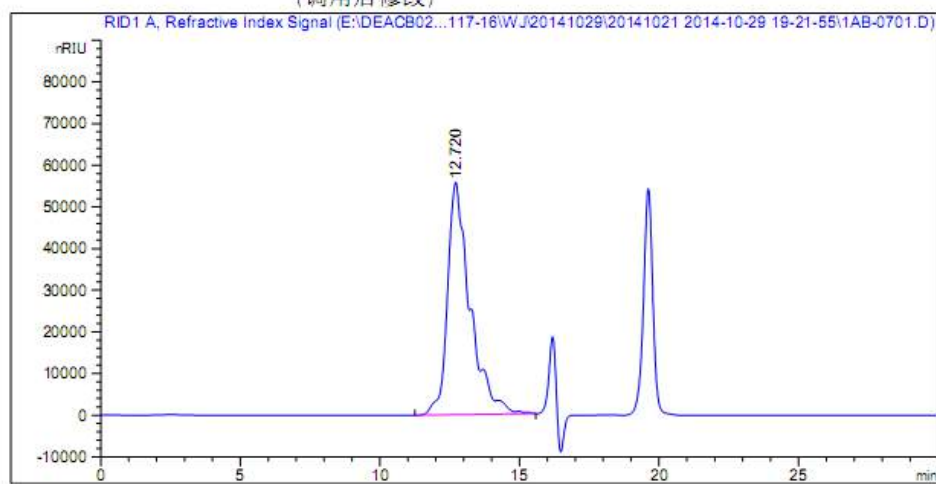
*** 报告结束 ***

附图10.5-111 SBEC含量测定方法验证图 (方法耐用性-水相: 有机相=87: 13-样品1-2)

Annex 3-5-40 Validation of analysis procedure for Assay-Robustness-Condition 7- Reference solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 19-21-55\1AB-0701.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   :    7
仪器         : 1260-2                      位置     : Pl-A-02
进样日期     : 2014/10/30 0:28:14          进样次数  :    1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 19-21-55\117-16-
                                           SBECD-0.6.M
最后修改     : 2014/10/29 19:21:55 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 19-21-55\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改     : 2015/3/31 14:22:09 : linping
                                           (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.720	-	3.13325e6	5.55963e4	0.53	0.7262	1699	-	-

=====
*** 报告结束 ***

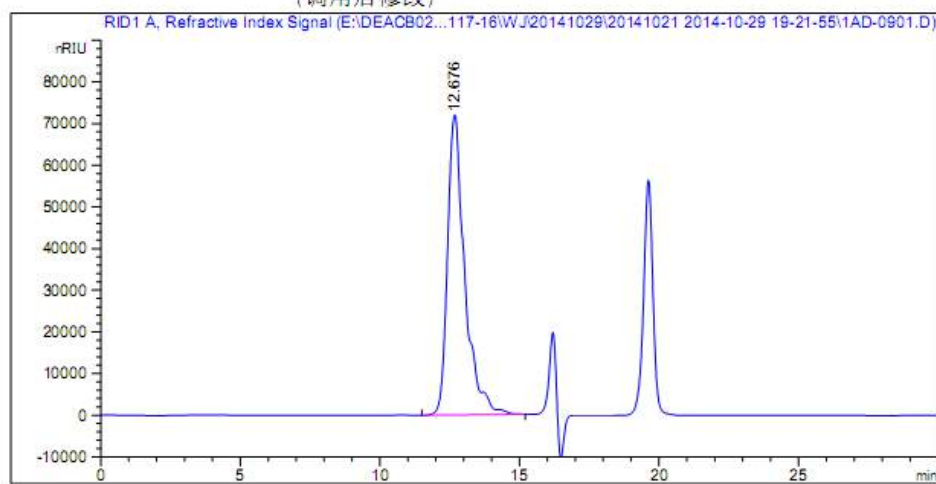
附图10.5-115 SBECD含量测定方法验证图 (方法耐用性-水相: 有机相=91: 9-对照1-1)

Annex 3-5-41 Validation of analysis procedure for Assay- Robustness-Condition 7- Sample solution

数据文件: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 19-21-55\1AD-0901.D
样品名称: NYX-910:90-1

=====

操作者	: Weijing	序列行	: 9
仪器	: 1260-2	位置	: Pl-A-04
进样日期	: 2014/10/30 2:30:31	进样次数	: 1
		进样量	: 20.000 µl
采集方法	: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 19-21-55\117-16-SBECd-0.6.M		
最后修改	: 2014/10/29 19:21:55 : Weijing		
分析方法	: E:\DEACB02694\SIM117-16\WJ\20141029\20141021 2014-10-29 19-21-55\117-16-SBECd-0.6.M (序列方法)		
最后修改	: 2015/3/31 14:22:09 : linping (调用后修改)		



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面积百分比报告 (包含性能计算)

=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.676	-	3.08900e6	7.19999e4	0.61	0.6216	2308	-	-

*** 报告结束 ***

附图10.5-119 SBECd含量测定方法验证图 (方法耐用性-水相: 有机相=91: 9-样品1-1)

1260-2 2015/3/31 14:22:41 linping

页 1/1

3.2.S.4.4 Batch Analyses

We inspected three batches of final product and all the detected items qualified, batch analyses reports can be seen below, and representative chromatograms refer to Annex 3-BA-1~Annex 3-BA-34.

Analysis report for 20140910

山东滨州智源生物科技有限公司
磺丁基醚倍他环糊精钠盐检验报告

CAS No.:182410-00-0

编号: SBE20140910

批号	20140910	包装规格	2kg/袋
生产日期	2014-09-10	数量	126.8kg
取样日期	2014-09-24	有效期至	2016-09-09
检验依据	磺丁基醚倍他环糊精钠盐申报生产用质量标准		
检验项目	指标	检验结果	项目结论
性状	本品为白色或类白色粉末。水中极易溶解；在甲醇中微溶；在乙醇或二氯甲烷中几乎不溶。	本品为白色或类白色粉末。水中极易溶解；在甲醇中微溶；在乙醇或二氯甲烷中几乎不溶。	符合规定
鉴别	主峰保留时间与对照品主峰保留时间一致	主峰保留时间与对照品主峰保留时间一致	符合规定
	红外光吸收图谱与对照品图谱一致	红外光吸收图谱与对照品图谱一致	符合规定
	水溶液显示钠盐的鉴别反应	水溶液显示钠盐的鉴别反应	符合规定
酸度	4.0~6.8	6.3	符合规定
溶液的澄清度与颜色	澄清无色；如显色，与黄色2号标准比色液比较，不得更深	澄清无色	符合规定
倍他环糊精	不得过 0.1%	小于 0.009%	符合规定
平均取代度	平均取代度 6.2-6.9	6.5	符合规定
1,4-丁烷磺内酯	不得过 0.5ppm	0.3ppm	符合规定
氯化钠	不得过 0.1%	0.067%	符合规定
4-羟基丁烷-1-磺酸	不得过 0.09%	0.023%	符合规定
双(4-磺丁基)醚二钠	不得过 0.05%	0.031%	符合规定
水分	不得过 10.0%	4.8%	符合规定
重金属	不得过百万分之五	小于百万分之五	符合规定
砷盐	不得过 0.0002%	小于 0.0002%	符合规定
硫酸盐	不得过 0.02%	小于 0.02%	符合规定
含量	95%-105%	101.8%	符合规定
细胞内毒素	不得过 0.02Eu/mg	小于 0.02Eu/mg	符合规定
微生物限度	细菌数不得过 100cfu/g	细菌数小于 10cfu/g	符合规定
	霉菌、酵母菌不得过 50cfu/g	霉菌、酵母菌小于 10cfu/g	
	大肠埃希菌不得检出	大肠埃希菌未检出	
结论：本品按磺丁基醚倍他环糊精钠盐申报生产用质量标准测定，结果符合规定			
检验员：	葛斯楠, 梁行彪	复核人：吴岐, 李俊	报告日期：2015-07-29

Watson International Ltd
Inspection report for Betadex Sulfobutyl Ether Sodium

CAS No.: 182410-00-0

Number: SEB20140910

Batch No.	20140910	Packing size	2kg/bag
Manufacturing time	2014.9.10	Batch size	126.8kg
Sampling time	2014-9.24	Period of validity	2016.09.09
Inspecting standard	Specification for manufacturing application of Betadex Sulfobutyl Ether Sodium		
Item	Acceptance criteria	Inspection result	Item conclusion
Character	White or off-white powder, very soluble in water; slightly soluble in methanol; practically insoluble in ethanol or methylene dichloride.	White or off-white powder, very soluble in water; slightly soluble in methanol; practically insoluble in ethanol or methylene dichloride.	Qualified
Identification	The retention time of sample is the same to that of reference standard	The retention time of sample is the same to that of reference standard	Qualified
	IR spectrum is corresponding to that of the reference standard	IR spectrum is corresponding to that of the reference standard	Qualified
	Positive reaction of sodium	Positive reaction of sodium	Qualified
pH value	4.0~6.8	6.3	Qualified
Clarity degree of solution	30% w/v solution is clear and without foreign matter	Clear and colorless	Qualified
Betadex	≤0.1%	<0.009%	Qualified
Average degree of substitution	6.2~6.9	6.5	Qualified
1,4-butane sultone	≤0.5ppm	0.3ppm	Qualified
Sodium chloride	≤0.2%	0.067%	Qualified
4-hydroxybutane-1-sulfonic acid	≤0.09%	0.023%	Qualified
Bis(4-sulfonutyl)ether disodium	≤0.05%	0.031%	Qualified
Water	≤10%	4.8%	Qualified
Heavy metal	≤5ppm	<5ppm	Qualified
Arsenic salt	≤0.0002%	<0.0002%	
Sulfate	≤0.02%	<0.02%	
Assay	95%-105%	101.8%	Qualified
Bacterial endotoxin	≤0.02EU/mg	<0.02EU/MG	Qualified
Microbial items	TAMC≤100cfu/g	<10cfu/g	Qualified
	TYMC≤50cfu/g	<10cfu/g	
	Escherichia coli. should not be detected	Not detected	
Conclusion: the product inspected meets the requirement of specification for manufacturing application of Betadex Sulfobutyl Ether Sodium			
Analyst: Sijuan Nie, Ningning Liang, Jing Wei		Reviewer: Xiao Wu, Yuanyuan Li	Reporting date: 2015-07-29

Analysis report for 20140921

山东滨州智源生物科技有限公司

磺丁基醚倍他环糊精钠盐检验报告

CAS No.:182410-00-0

编号: SBE20140921

批号	20140921	包装规格	2kg/袋
生产日期	2014-09-21	数量	128.4kg
取样日期	2014-10-04	有效期至	2016-09-20
检验依据	磺丁基醚倍他环糊精钠盐申报生产用质量标准		
检验项目	指标	检验结果	项目结论
性状	本品为白色或类白色粉末。水中极易溶解；在甲醇中微溶；在乙醇或二氯甲烷中几乎不溶。	本品为白色或类白色粉末。水中极易溶解；在甲醇中微溶；在乙醇或二氯甲烷中几乎不溶。	符合规定
鉴别	主峰保留时间与对照品主峰保留时间一致	主峰保留时间与对照品主峰保留时间一致	符合规定
	红外光吸收图谱与对照品图谱一致	红外光吸收图谱与对照品图谱一致	符合规定
	水溶液显示钠盐的鉴别反应	水溶液显示钠盐的鉴别反应	符合规定
酸度	4.0~6.8	6.2	符合规定
溶液的澄清度与颜色	澄清无色；如显色，与黄色2号标准比色液比较，不得更深	澄清无色	符合规定
倍他环糊精	不得过 0.1%	小于 0.009%	符合规定
平均取代度	平均取代度 6.2-6.9	6.6	符合规定
1,4-丁烷磺内酯	不得过 0.5ppm	0.2ppm	符合规定
氯化钠	不得过 0.1%	0.067%	符合规定
4-羟基丁烷磺酸	不得过 0.09%	0.024%	符合规定
双(4-磺丁基)醚二钠	不得过 0.05%	0.032%	符合规定
水分	不得过 10.0%	4.7%	符合规定
重金属	不得过百万分之五	小于百万分之五	符合规定
砷盐	不得过 0.0002%	小于 0.0002%	符合规定
硫酸盐	不得过 0.02%	小于 0.02%	符合规定
含量	95%-105%	101.3%	符合规定
细胞内毒素	不得过 0.02Eu/mg	小于 0.02Eu/mg	符合规定
微生物限度	细菌数不得过 100cfu/g	细菌数小于 10cfu/g	符合规定
	霉菌、酵母菌不得过 50cfu/g	霉菌、酵母菌数小于 10cfu/g	
	大肠埃希菌不得检出	大肠埃希菌未检出	
结论：本品按磺丁基醚倍他环糊精钠盐申报生产用质量标准测定，结果符合规定			
检验员：夏斯明		复核人：吴明 李德俊	报告日期：2015-07-29

Watson International Ltd
Inspection report for Betadex Sulfobutyl Ether Sodium

CAS No.: 182410-00-0

Number: SEB20140921

Batch No.	20140921	Packing size	2kg/bag
Manufacturing time	2014.9.21	Batch size	128.4kg
Sampling time	2014-10.04	Period of validity	2016.09.20
Inspecting standard	Specification for manufacturing application of Betadex Sulfobutyl Ether Sodium		
Item	Acceptance criteria	Inspection result	Item conclusion
Character	White or off-white powder, very soluble in water; slightly soluble in methanol; practically insoluble in ethanol or methylene dichloride.	White or off-white powder, very soluble in water; slightly soluble in methanol; practically insoluble in ethanol or methylene dichloride.	Qualified
Identification	The retention time of sample is the same to that of reference standard	The retention time of sample is the same to that of reference standard	Qualified
	IR spectrum is corresponding to that of the reference standard	IR spectrum is corresponding to that of the reference standard	Qualified
	Positive reaction of sodium	Positive reaction of sodium	Qualified
pH value	4.0~6.8	6.2	Qualified
Clarity degree of solution	30% w/v solution is clear and without foreign matter	Clear and colorless	Qualified
Betadex	≤0.1%	<0.009%	Qualified
Average degree of substitution	6.2~6.9	6.6	Qualified
1,4-butane sultone	≤0.5ppm	0.2ppm	Qualified
Sodium chloride	≤0.2%	0.067%	Qualified
4-hydroxybutane-1-sulfonic acid	≤0.09%	0.024%	Qualified
Bis(4-sulfonutyl)ether disodium	≤0.05%	0.032%	Qualified
Water	≤10%	4.7%	Qualified
Heavy metal	≤5ppm	<5ppm	Qualified
Arsenic salt	≤0.0002%	<0.0002%	
Sulfate	≤0.02%	<0.02%	
Assay	95%-105%	101.3%	Qualified
Bacterial endotoxin	≤0.02EU/mg	<0.02EU/MG	Qualified
Microbial items	TAMC≤100cfu/g	<10cfu/g	Qualified
	TYMC≤50cfu/g	<10cfu/g	
	Escherichia coli. should not be detected	Not detected	
Conclusion: the product inspected meets the requirement of specification for manufacturing application of Betadex Sulfobutyl Ether Sodium			
Analyst: Sijuan Nie, Ningning Liang, Jing Wei		Reviewer: Xiao Wu, Yuanyuan Li	Reporting date: 2015-07-29

Analysis report for 20140930

山东滨州智源生物科技有限公司

磺丁基醚倍他环糊精钠盐检验报告

CAS No.:182410-00-0

编号: SBE20140930

CAS No. 182410-00-0

编号: SBE20140930

批号	20140930	包装规格	2kg/袋
生产日期	2014-09-30	数量	129.0kg
取样日期	2014-10-13	有效期至	2016-09-29
检验依据	磺丁基醚倍他环糊精钠盐申报生产用质量标准		
检验项目	指标	检验结果	项目结论
性状	本品为白色或类白色粉末。水中极易溶解；在甲醇中微溶；在乙醇或二氯甲烷中几乎不溶。	本品为白色或类白色粉末。水中极易溶解；在甲醇中微溶；在乙醇或二氯甲烷中几乎不溶	符合规定
鉴别	主峰保留时间与对照品主峰保留时间一致	主峰保留时间与对照品主峰保留时间一致	符合规定
	红外光吸收图谱与对照品图谱一致	红外光吸收图谱与对照品图谱一致	符合规定
	水溶液显示钠盐的鉴别反应	水溶液显示钠盐的鉴别反应	符合规定
酸度	4.0~6.8	6.2	符合规定
溶液的澄清度与颜色	澄清无色；如显色，与黄色2号标准比色液比较，不得更深	澄清无色	符合规定
倍他环糊精	不得过 0.1%	小于 0.009%	符合规定
平均取代度	平均取代度 6.2-6.9	6.5	符合规定
1,4-丁烷磺内酯	不得过 0.5ppm	0.2ppm	符合规定
氯化钠	不得过 0.1%	0.067%	符合规定
4-羟基丁烷-1-磺酸	不得过 0.09%	0.024%	符合规定
双(4-磺丁基)醚二钠	不得过 0.05%	0.029%	符合规定
水分	不得过 10.0%	4.1%	符合规定
重金属	不得过百万分之五	小于百万分之五	符合规定
砷盐	不得过 0.0002%	小于 0.0002%	符合规定
硫酸盐	不得过 0.02%	小于 0.02%	符合规定
含量	95%-105%	100.8%	符合规定
细胞内毒素	不得过 0.02Eu/mg	小于 0.02Eu/mg	符合规定
微生物限度	细菌数不得过 100cfu/g	细菌数小于 10cfu/g	符合规定
	霉菌、酵母菌不得过 50cfu/g	霉菌、酵母菌小于 10cfu/g	
	大肠埃希菌不得检出	大肠埃希菌未检出	
结论：本品按磺丁基醚倍他环糊精钠盐申报生产用质量标准测定，结果符合规定			
检验员：葛斯娟		复核人：吴晓 李海洁	报告日期：2015-07-29

Watson International Ltd
Inspection report for Betadex Sulfobutyl Ether Sodium

CAS No.: 182410-00-0

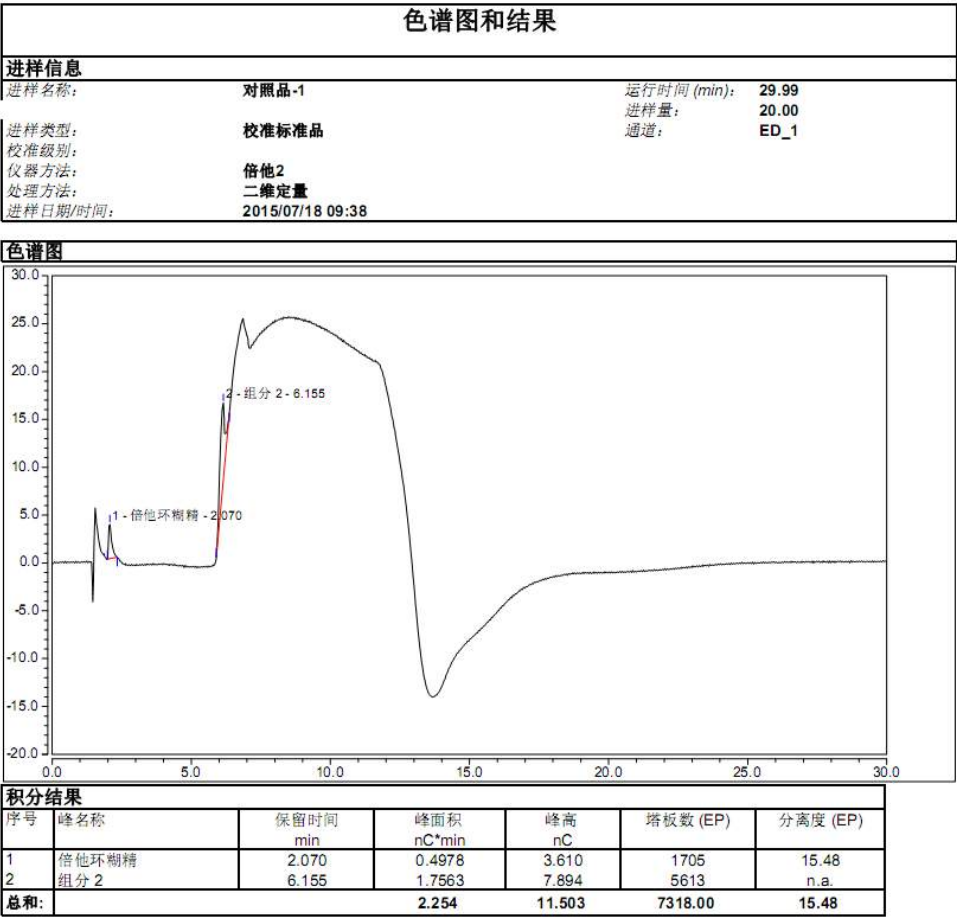
Number: SEB20140930

Batch No.	20140930	Packing size	2kg/bag
Manufacturing time	2014.9.30	Batch size	129.0kg
Sampling time	2014-10.13	Period of validity	2016.09.29
Inspecting standard	Specification for manufacturing application of Betadex Sulfobutyl Ether Sodium		
Item	Acceptance criteria	Inspection result	Item conclusion
Character	White or off-white powder, very soluble in water; slightly soluble in methanol; practically insoluble in ethanol or methylene dichloride.	White or off-white powder, very soluble in water; slightly soluble in methanol; practically insoluble in ethanol or methylene dichloride.	Qualified
Identification	The retention time of sample is the same to that of reference standard	The retention time of sample is the same to that of reference standard	Qualified
	IR spectrum is corresponding to that of the reference standard	IR spectrum is corresponding to that of the reference standard	Qualified
	Positive reaction of sodium	Positive reaction of sodium	Qualified
pH value	4.0~6.8	6.2	Qualified
Clarity degree of solution	30% w/v solution is clear and without foreign matter	Clear and colorless	Qualified
Betadex	≤0.1%	<0.009%	Qualified
Average degree of substitution	6.2~6.9	6.5	Qualified
1,4-butane sultone	≤0.5ppm	0.2ppm	Qualified
Sodium chloride	≤0.2%	0.067%	Qualified
4-hydroxybutane-1-sulfonic acid	≤0.09%	0.024%	Qualified
Bis(4-sulfonutyl)ether disodium	≤0.05%	0.029%	Qualified
Water	≤10%	4.1%	Qualified
Heavy metal	≤5ppm	<5ppm	Qualified
Arsenic salt	≤0.0002%	<0.0002%	
Sulfate	≤0.02%	<0.02%	
Assay	95%-105%	100.8%	Qualified
Bacterial endotoxin	≤0.02EU/mg	<0.02EU/MG	Qualified
Microbial items	TAMC≤100cfu/g	<10cfu/g	Qualified
	TYMC≤50cfu/g	<10cfu/g	
	Escherichia coli. should not be detected	Not detected	
Conclusion: the product inspected meets the requirement of specification for manufacturing application of Betadex Sulfobutyl Ether Sodium			
Analyst: Sijuan Nie, Ningning Liang, Jing Wei		Reviewer: Xiao Wu, Yuanyuan Li	Reporting date: 2015-07-29

Annex 3-BA-1 Batch analyses-Betadex-Reference solution

仪器:ICS-5000+ 序列:中间精密度1,3批样品检测

页码 1 / 15



附图10.4.8-166 SBECD中倍他环糊精的测定方法验证图 (准确度, 3批样品检测, 中间精密度1-对照-1)

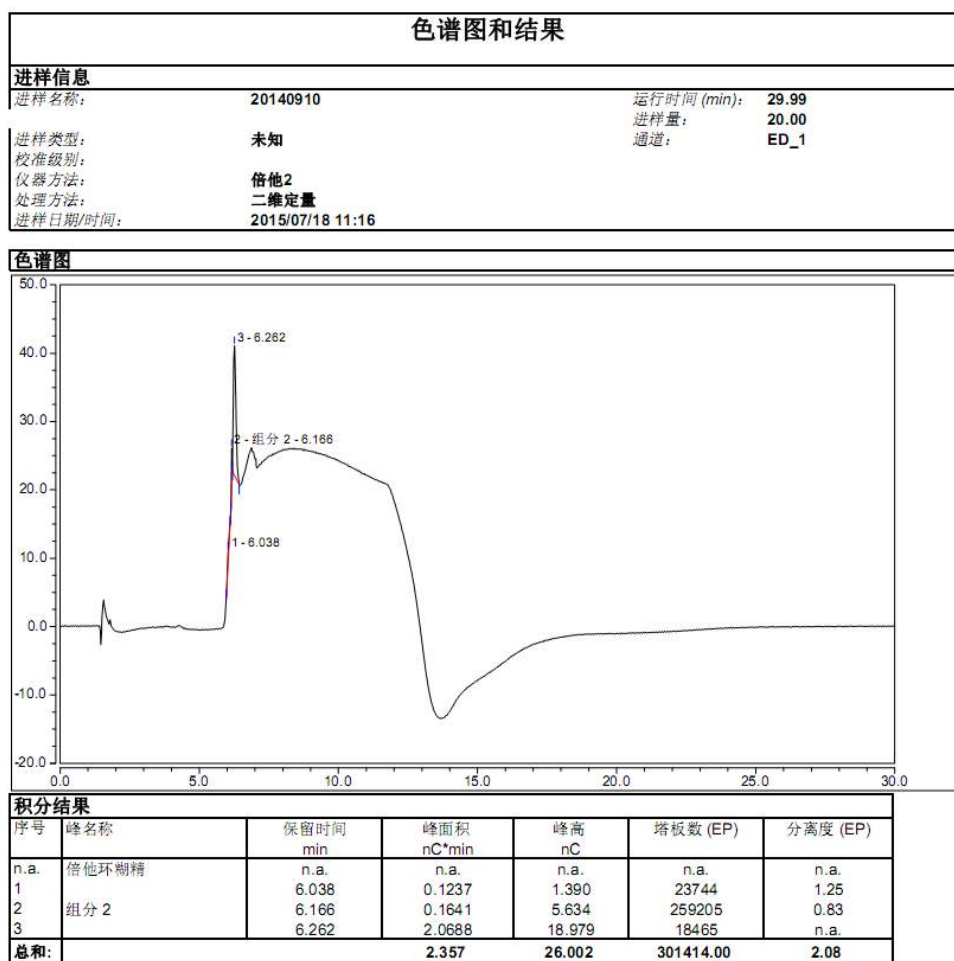
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-BA-2 Batch analyses-Betadex-Sample solution 20140910

仪器:ICS-5000+ 序列:中间精密度1,3批样品检测

页码 4 / 15



附图10.4.8-212 SBEC-D-0时倍他环糊精的测定图 (20140910-1)

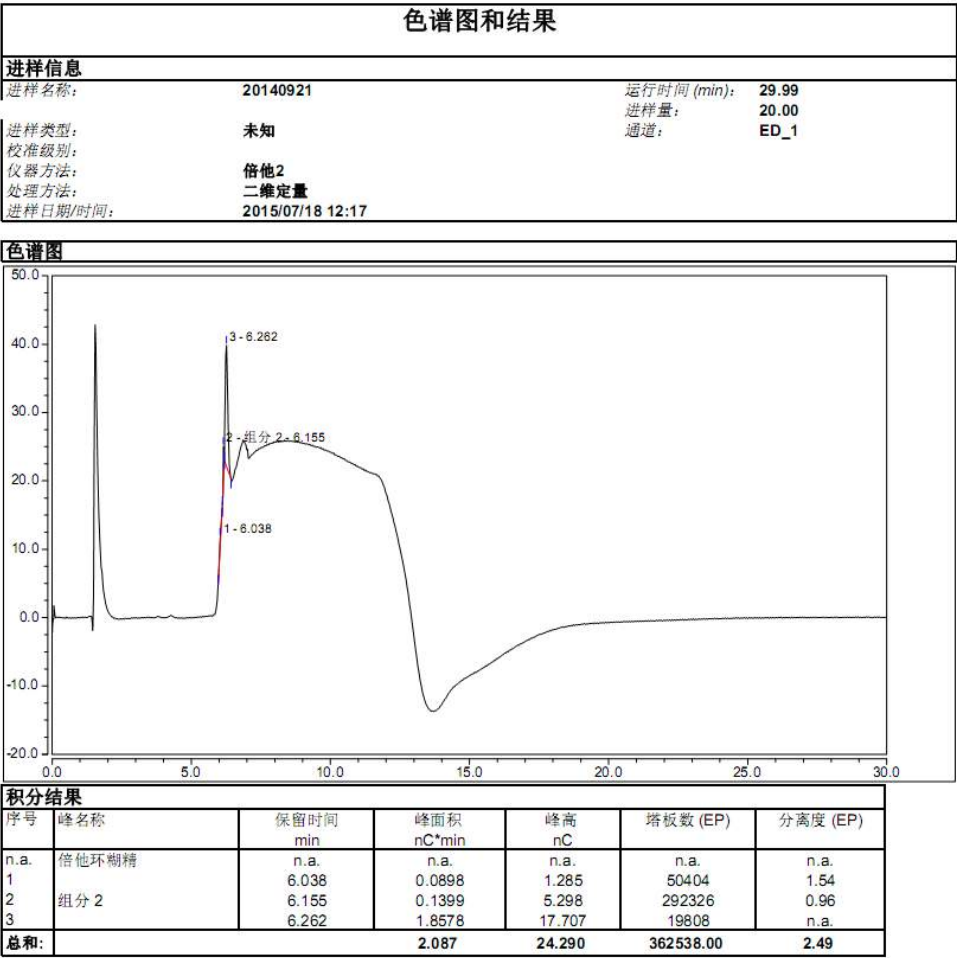
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-BA-3 Batch analyses-Betadex-Sample solution 20140921

仪器:ICS-5000+ 序列:中间精密度1,3批样品检测

页码 6 / 15



附图10. 4. 8 -SEC-0 时倍他环糊精的测定图 (20140921-

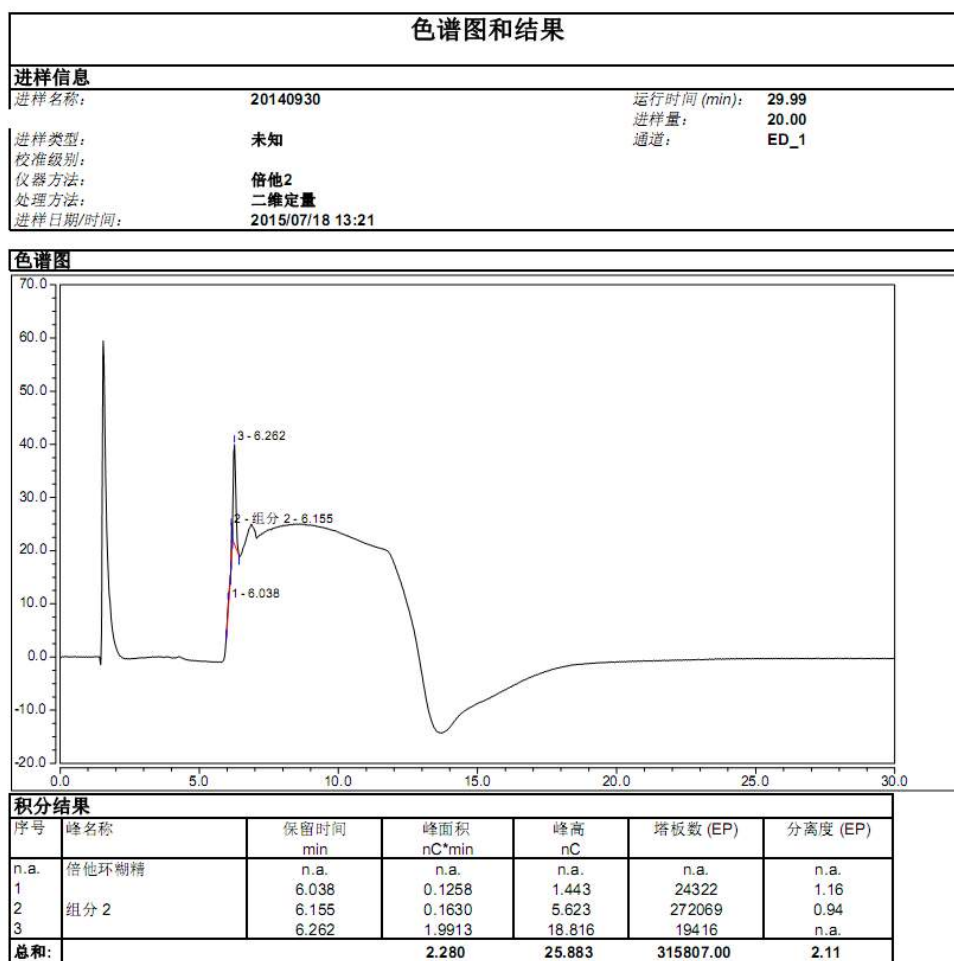
Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-BA-4 Batch analyses-Betadex-Sample solution 20140930

仪器:ICS-5000+ 序列:中间精密度1,3批样品检测

页码 8 / 15



附图10. 4. 8 - SEC-0 时倍他环糊精的测定图 (20140930 -

Default/积分

Chromeleon (c) Dionex
版本 7.2.1.5537

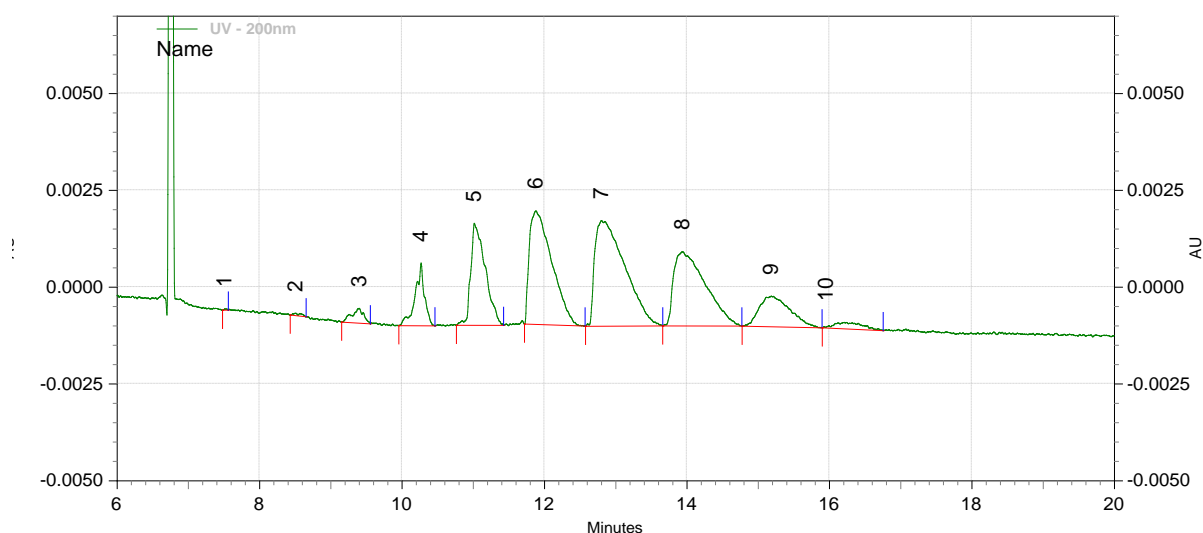
Annex 3-BA-5 Batch analyses-Average degree of substitution-Sample solution 20140910

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\test samples\150629 test sample 20140910 001.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/29/2015 11:27:47 AM

Printed: 7/3/2015 3:36:10 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.59	0.052	0.00
2	0.66	0.266	10.78
3	0.73	2.029	3.91
4	0.80	6.167	3.09
5	0.86	14.690	2.43
6	0.93	23.389	1.74
7	1.00	26.962	1.31
8	1.09	18.401	1.38
9	1.19	6.789	1.49
10	1.27	1.255	1.32

Totals		100.000	
--------	--	---------	--

附图 10.4.9-43 SBECD 中平均取代度的测定方法验证图（样品检测-20140910）

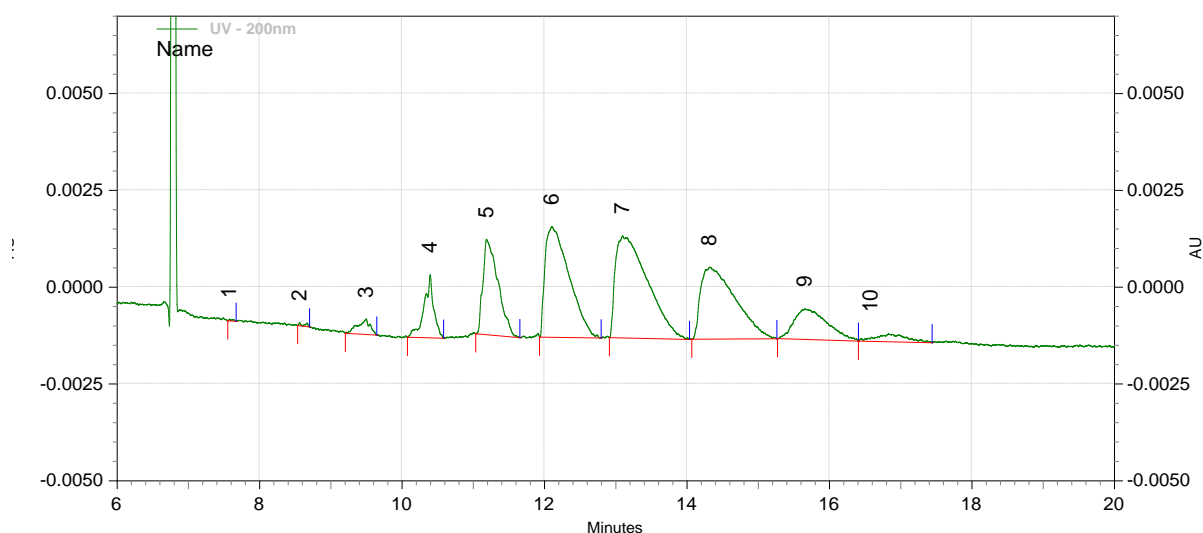
Annex 3-BA-6 Batch analyses-Average degree of substitution-Sample solution 20140921

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\test samples\150629 test sample 20140921 001.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/29/2015 12:16:18 PM

Printed: 7/3/2015 3:38:05 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.58	0.091	0.00
2	0.65	0.192	11.85
3	0.73	2.118	4.82
4	0.79	6.244	3.18
5	0.85	13.775	2.41
6	0.92	23.570	1.73
7	1.00	26.874	1.32
8	1.09	18.573	1.36
9	1.20	7.015	1.46
10	1.29	1.548	1.38

Totals		100.000	
--------	--	---------	--

附图 10.4.9-44 SBECD 中平均取代度的测定方法验证图（样品检测-20140921）

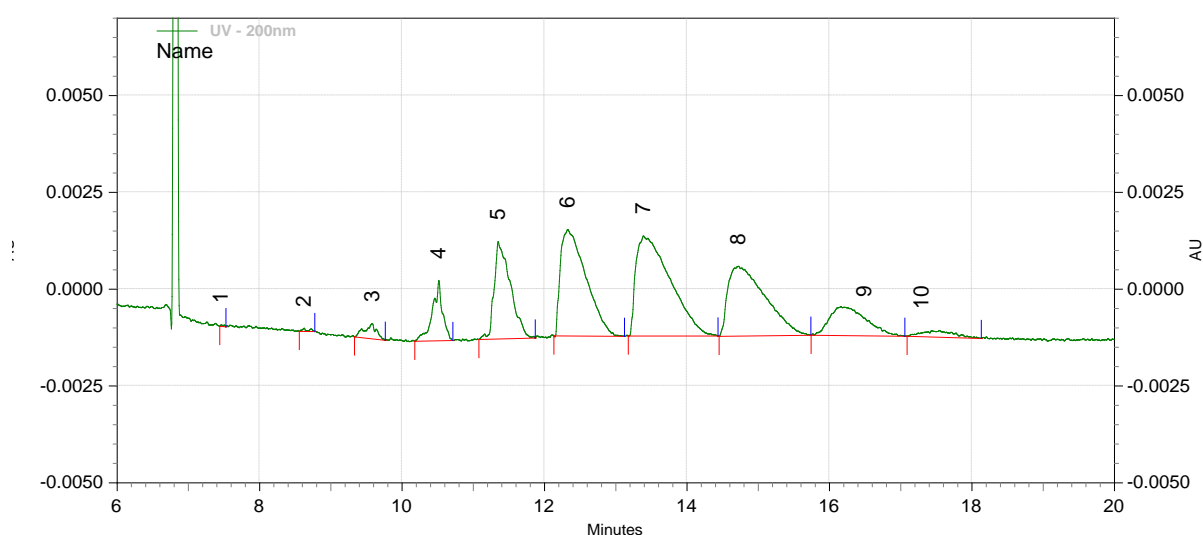
Annex 3-BA-7 Batch analyses-Average degree of substitution-Sample solution 20140930

Data File: C:\32Karat\BZZY\Average degree of substitution\Conditions fumble\test samples\150629 test sample 20140930 001.dat

Method: C:\32Karat\Projects\Default\Method\UV OQ1.met

Acquired: 6/29/2015 1:26:28 PM

Printed: 7/3/2015 3:39:06 PM



UV - 200nm Results

Name	Relative MT	Corrected Area Percent	Resolution (USP)
1	0.56	0.075	0.00
2	0.64	0.223	12.83
3	0.72	1.979	4.76
4	0.79	6.063	3.48
5	0.85	14.686	2.49
6	0.92	23.217	1.74
7	1.00	27.060	1.32
8	1.10	18.637	1.38
9	1.23	6.791	1.76
10	1.31	1.269	0.00

Totals		100.000	
--------	--	---------	--

附图 10.4.9-45 SBECD 中平均取代度的测定方法验证图（样品检测-20140930）

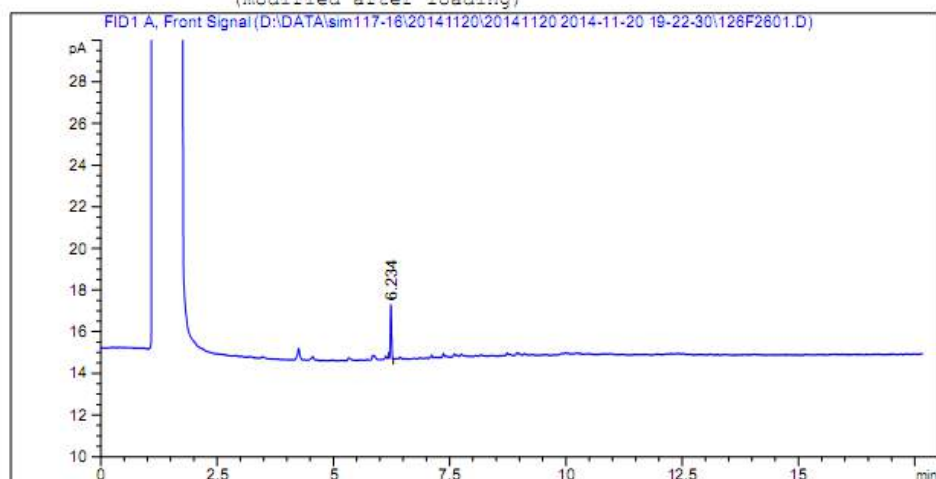
Annex 3-BA-8 Batch analyses-1,4-butane sultone-Blank 20140910

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\126F2601.D

Sample Name: ZJJMD-KB

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   26
Acq. Instrument : GC-03                      Location  :   126
Injection Date  : 11/21/2014 4:05:16 AM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:54:21 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.234	BBA	0.0240	4.57852	2.58031	1.000e2

```
Totals :                      4.57852    2.58031
```

```
=====
*** End of Report ***
=====
```

附图10.4.10-50 SBECD中1,4-丁烷磺内酯的测定方法验证图(中间精密度-空白)

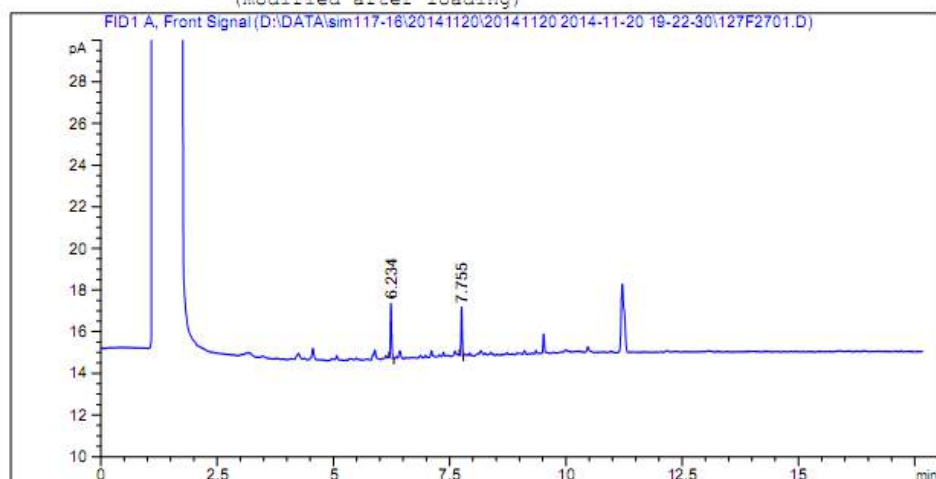
Annex 3-BA-9 Batch analyses-1,4-butane sultone- Sample solution A 20140910

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\127F2701.D

Sample Name: ZJJMD-A

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   27
Acq. Instrument : GC-03                      Location  :   127
Injection Date  : 11/21/2014 4:26:12 AM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:54:21 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.234	BBA	0.0267	4.79530	2.63291	54.98854
2	7.755	BB	0.0228	3.92524	2.31218	45.01146

```
Totals :                      8.72054    4.94509
```

```
=====
*** End of Report ***
=====
```

附图10.4.10-51 SBECD中1,4-丁烷磺内酯的测定方法验证图（中间精密度-样品溶液A）

Annex 3-BA-10 Batch analyses-1,4-butane sultone-Sample solution B 20140910

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\128F2801.D

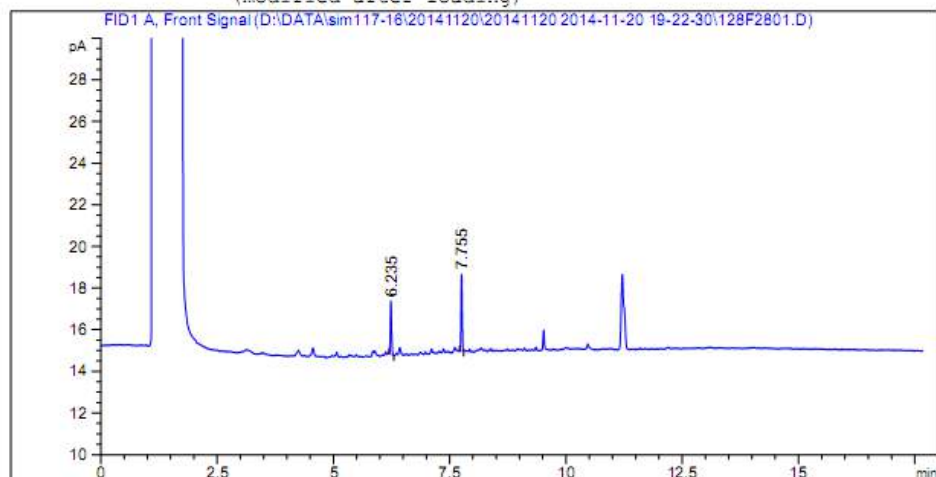
Sample Name: ZJJMD-B

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :   28
Acq. Instrument : GC-03                      Location  :   128
Injection Date  : 11/21/2014 4:47:04 AM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/21/2014 10:54:21 AM by SYSTEM
                  (modified after loading)
=====

```



```

=====
                          Area Percent Report
=====

```

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.235	BBA	0.0272	4.63521	2.55667	42.57175
2	7.755	BB	0.0235	6.25278	3.69484	57.42825

```
Totals :                      10.88799    6.25151
```

```

=====
*** End of Report ***
=====

```

附图10.4.10-52 SBECD中1,4-丁烷磺内酯的测定方法验证图(中间精密度-样品溶液B)

Annex 3-BA-11 Batch analyses-1,4-butane sultone-Sample solution C 20140910

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\129F2901.D

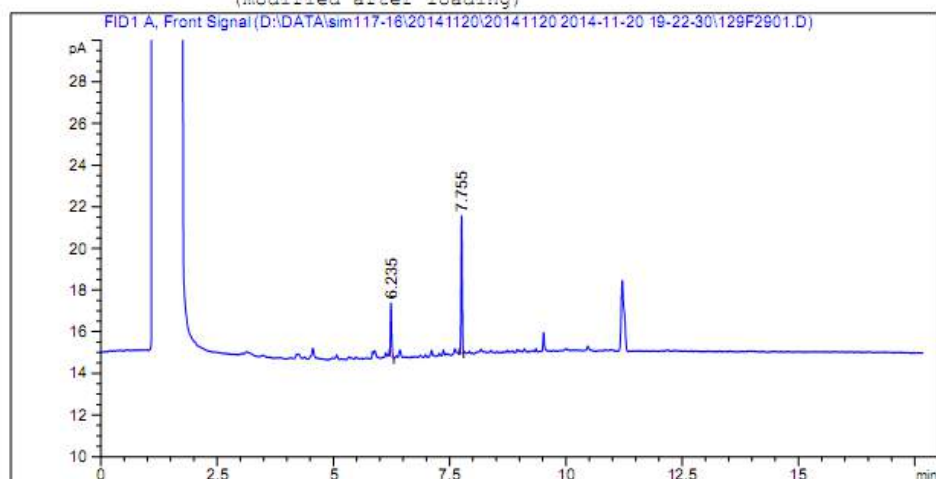
Sample Name: ZJJMD-C

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :   29
Acq. Instrument : GC-03                      Location  :   129
Injection Date  : 11/21/2014 5:07:51 AM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
Sequence Method)
Last changed    : 11/21/2014 10:56:27 AM by SYSTEM
                  (modified after loading)
=====

```



```

=====
                          Area Percent Report
=====

```

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.235	BBA	0.0268	4.62049	2.55887	29.53213
2	7.755	BBA	0.0262	11.02515	6.61198	70.46787

```
Totals :                      15.64563    9.17085
```

```

=====
*** End of Report ***
=====

```

附图10.4.10-53 SBECD中1,4-丁烷磺内酯的测定方法验证图(中间精密度-样品溶液C)

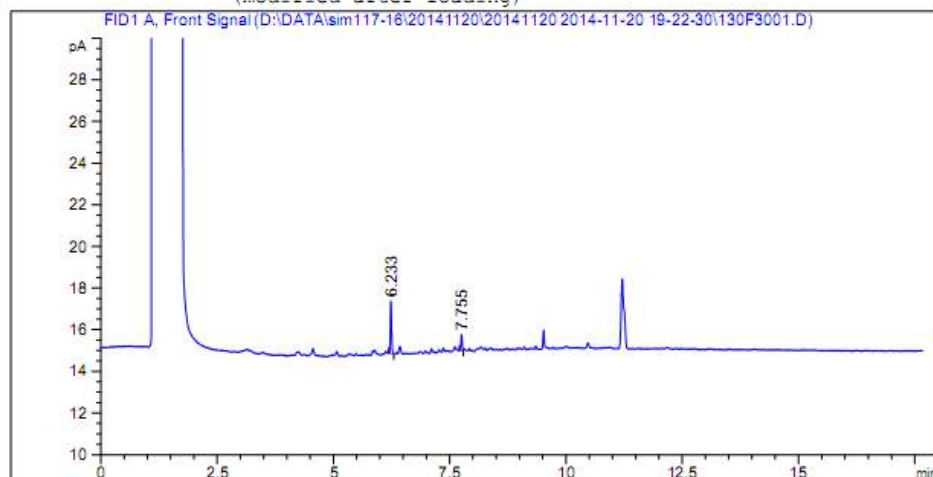
Annex 3-BA-12 Batch analyses-1,4-butane sultone-Sample solution D 20140910

Data File D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\130F3001.D

Sample Name: ZJJMD-D1

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   30
Acq. Instrument : GC-03                      Location  :   130
Injection Date  : 11/21/2014 5:28:41 AM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M
Last changed    : 11/20/2014 7:22:30 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141120\20141120 2014-11-20 19-22-30\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:56:27 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.233	BBA	0.0266	4.66921	2.52454	77.06495
2	7.755	BB	0.0219	1.38959	7.85402e-1	22.93505

```
Totals :                      6.05879    3.30994
```

```
=====
*** End of Report ***
=====
```

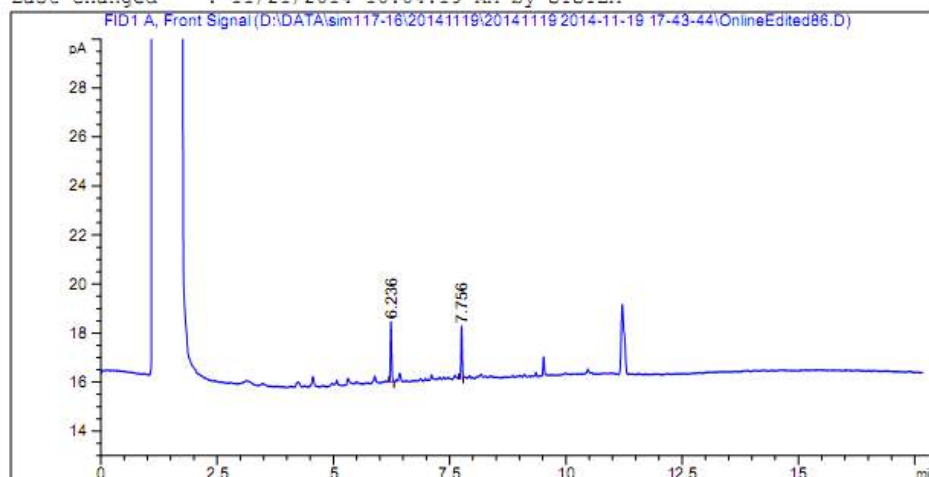
附图10.4.10-54 SBECD中1,4-丁烷磺内酯的测定方法验证图(中间精密度-样品溶液D-1)

Annex 3-BA-13 Batch analyses-1,4-butane sultone- Sample solution A 20140921

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited86.D

Sample Name: 20140921-A

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   59
Acq. Instrument : GC-03                      Location  :   130
Injection Date  : 11/20/2014 1:54:36 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.236	BBA	0.0304	4.61429	2.40317	55.39256
2	7.756	BBA	0.0288	3.71587	2.08490	44.60744

```
Totals :                      8.33016    4.48807
```

```
=====
*** End of Report ***
=====
```

附图10.4.10-122 SBECD-0时1,4-丁烷磺内酯的测定图 (20140921-A)

GC-03 11/21/2014 10:05:59 AM SYSTEM

Page 1 of 1

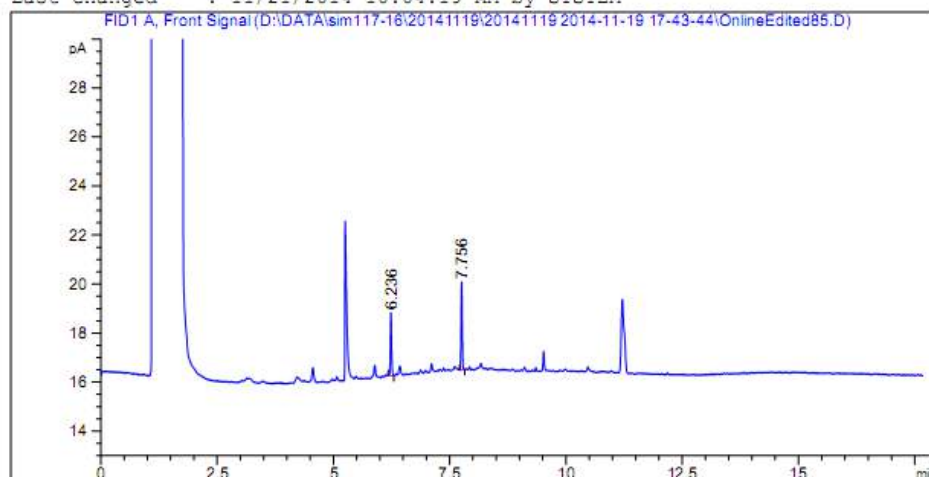
Annex 3-BA-14 Batch analyses-1,4-butane sultone-Sample solution B 20140921

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited85.D

Sample Name: 20140921-B

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   60
Acq. Instrument : GC-03                      Location  :   131
Injection Date  : 11/20/2014 2:15:34 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.236	BB	0.0295	4.61383	2.50919	42.54204
2	7.756	BBA	0.0285	6.23152	3.55494	57.45796

```
Totals :                      10.84536    6.06412
```

```
=====
*** End of Report ***
```

附图10.4.10-123 SBECD-0时1,4-丁烷磺内酯的测定图(20140921-B)

GC-03 11/21/2014 10:06:10 AM SYSTEM

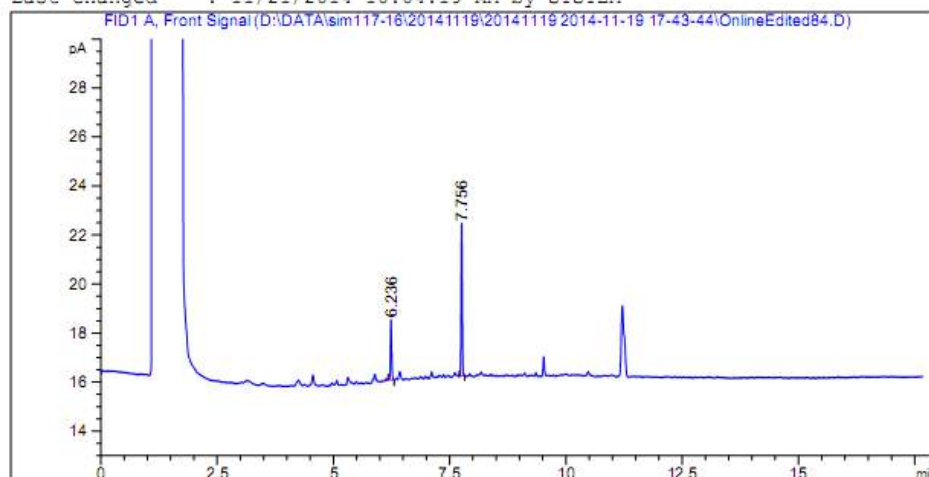
Page 1 of 1

Annex 3-BA-15 Batch analyses-1,4-butane sultone-Sample solution C 20140921

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited84.D
Sample Name: 20140921-C

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   61
Acq. Instrument : GC-03                      Location  :   132
Injection Date  : 11/20/2014 2:36:32 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.236	BBA	0.0277	4.47386	2.40790	30.07210
2	7.756	BBA	0.0258	10.40326	6.14102	69.92790

Totals : 14.87712 8.54892

```
=====
*** End of Report ***
=====
```

附图10.4.10-124 SBECD-0时1,4-丁烷磺内酯的测定图 (20140921-C)

GC-03 11/21/2014 10:06:20 AM SYSTEM

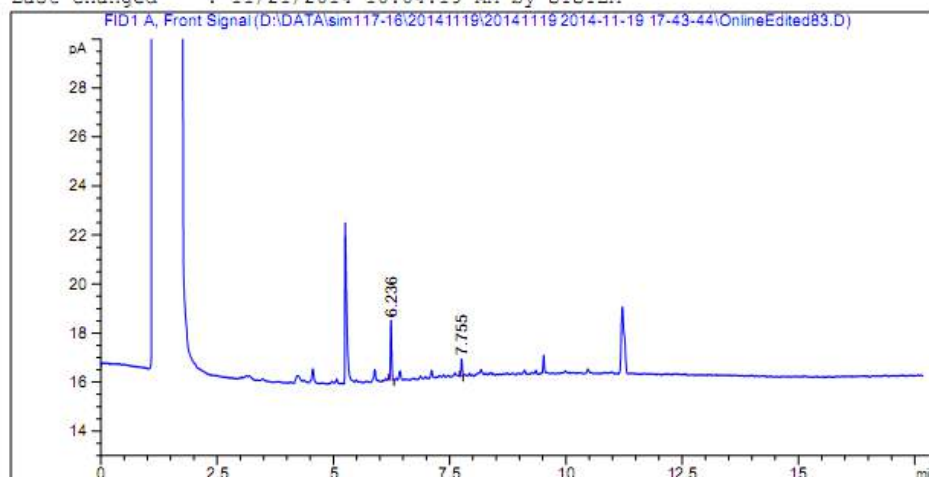
Page 1 of 1

Annex 3-BA-16 Batch analyses-1,4-butane sultone-Sample solution D 20140921

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited83.D
Sample Name: 20140921-D

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   62
Acq. Instrument : GC-03                      Location  :   133
Injection Date  : 11/20/2014 2:57:27 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.236	BBA	0.0302	4.56057	2.39834	77.77888
2	7.755	BBA	0.0300	1.30294	6.89622e-1	22.22112

Totals : 5.86350 3.08796

```
=====
*** End of Report ***
```

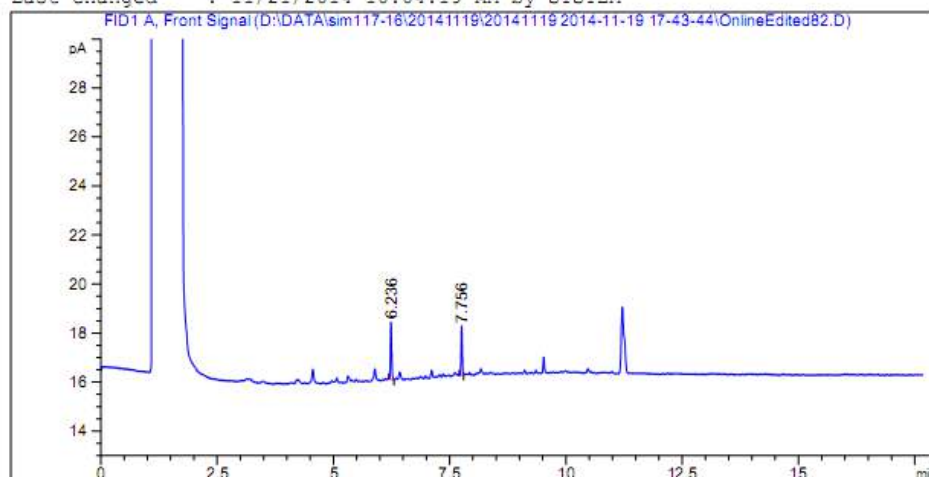
附图10.4.10-125 SBECD-0时1,4-丁烷磺内酯的测定图 (20140921-D)

Annex 3-BA-18 Batch analyses-1,4-butane sultone- Sample solution A 20140930

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited82.D
Sample Name: 20140930-A

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   63
Acq. Instrument : GC-03                      Location  :   134
Injection Date  : 11/20/2014 3:18:30 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.236	BBA	0.0302	4.41692	2.32511	55.97286
2	7.756	BBA	0.0266	3.47426	1.96952	44.02714

Totals : 7.89119 4.29462

```
=====
*** End of Report ***
=====
```

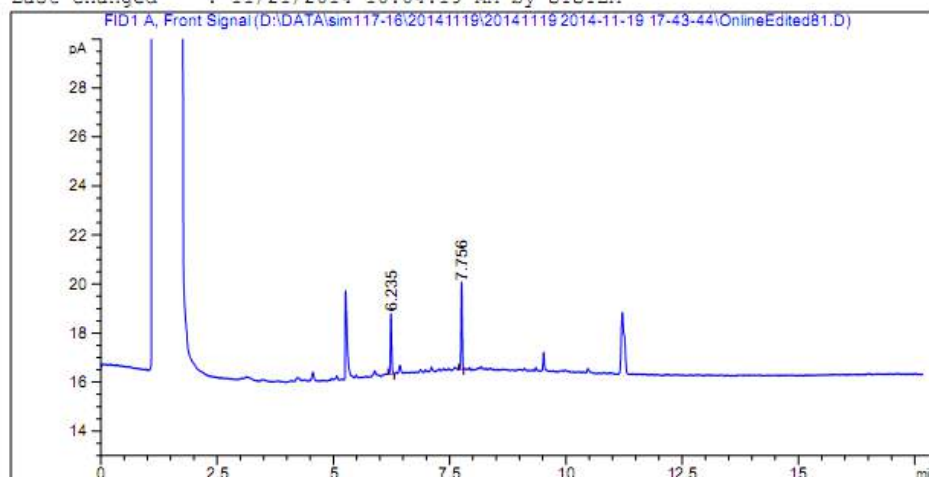
附图10.4.10-126 SBECD-0时1,4-丁烷磺内酯的测定图 (20140930-A)

Annex 3-BA-19 Batch analyses-1,4-butane sultone-Sample solution B 20140930

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited81.D

Sample Name: 20140930-B

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   64
Acq. Instrument : GC-03                      Location  :   135
Injection Date  : 11/20/2014 3:39:27 PM      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.235	BBA	0.0307	4.72893	2.42833	43.69918
2	7.756	BBA	0.0284	6.09262	3.48711	56.30082

```
Totals :                      10.82154    5.91544
```

```
=====
*** End of Report ***
```

附图10.4.10-127 SBECD-0时1,4-丁烷磺内酯的测定图(20140930-B)

GC-03 11/21/2014 10:06:58 AM SYSTEM

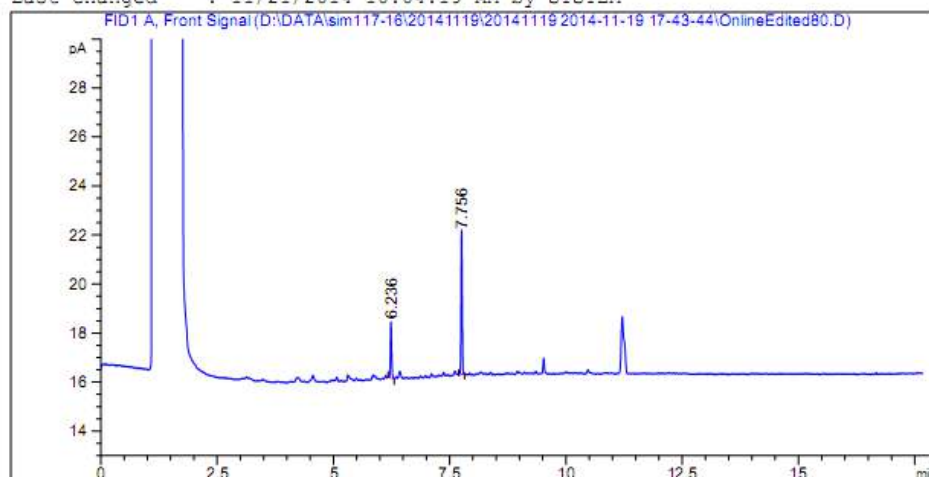
Page 1 of 1

Annex 3-BA-20 Batch analyses-1,4-butane sultone-Sample solution C 20140930

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited80.D
Sample Name: 20140930-C

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   65
Acq. Instrument : GC-03                      Location  :   136
Injection Date  : 11/20/2014 4:00:26 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.236	BBA	0.0306	4.33579	2.23340	30.22819
2	7.756	BBA	0.0282	10.00773	5.78966	69.77181

Totals : 14.34352 8.02305

```
=====
*** End of Report ***
=====
```

附图10.4.10-128 SBECD-0时1,4-丁烷磺内酯的测定图 (20140930-C)

GC-03 11/21/2014 10:07:18 AM SYSTEM

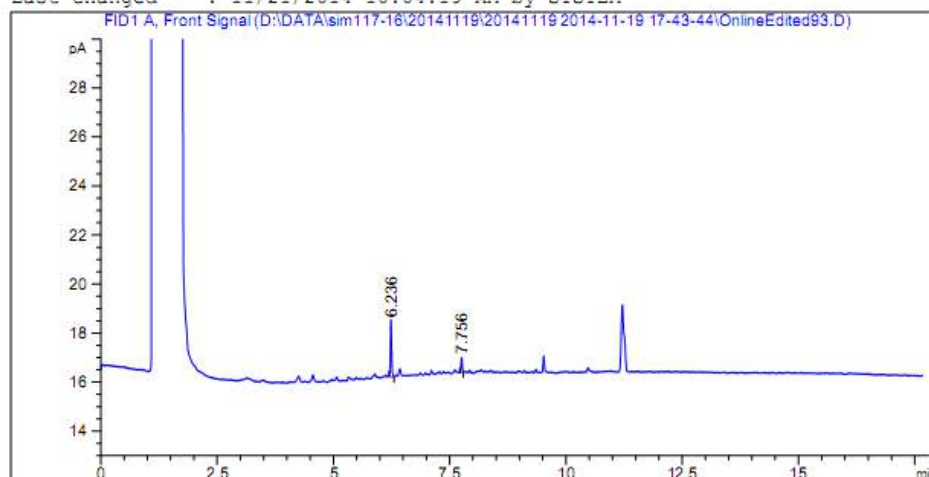
Page 1 of 1

Annex 3-BA-21 Batch analyses-1,4-butane sultone-Sample solution D 20140930

Data File D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\OnlineEdited93.D
 Sample Name: 20140930-D

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :   66
Acq. Instrument : GC-03                      Location  :   137
Injection Date  : 11/20/2014 4:21:37 PM      Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M
Last changed    : 11/20/2014 12:01:54 PM by SYSTEM
Analysis Method : D:\DATA\sim117-16\20141119\20141119 2014-11-19 17-43-44\DB-200-20141114.M (
                  Sequence Method)
Last changed    : 11/21/2014 10:04:19 AM by SYSTEM
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.236	BB	0.0305	4.39244	2.27862	80.04723
2	7.756	BBA	0.0295	1.09487	5.93753e-1	19.95277

```
Totals :                      5.48730    2.87238
```

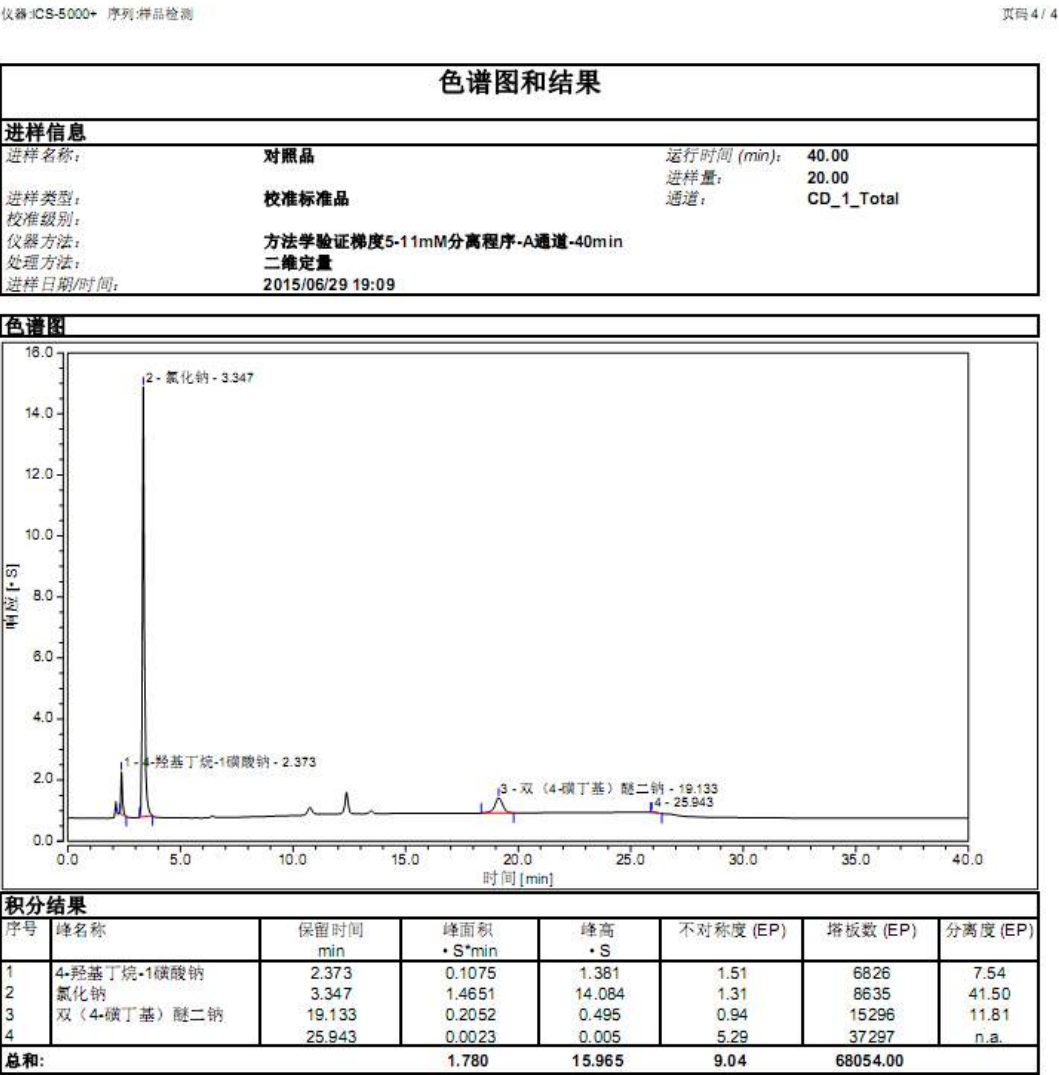
```
=====
*** End of Report ***
```

附图10.4.10-129 SBECD-0时1,4-丁烷磺内酯的测定图 (20140930-D)

GC-03 11/21/2014 10:07:30 AM SYSTEM

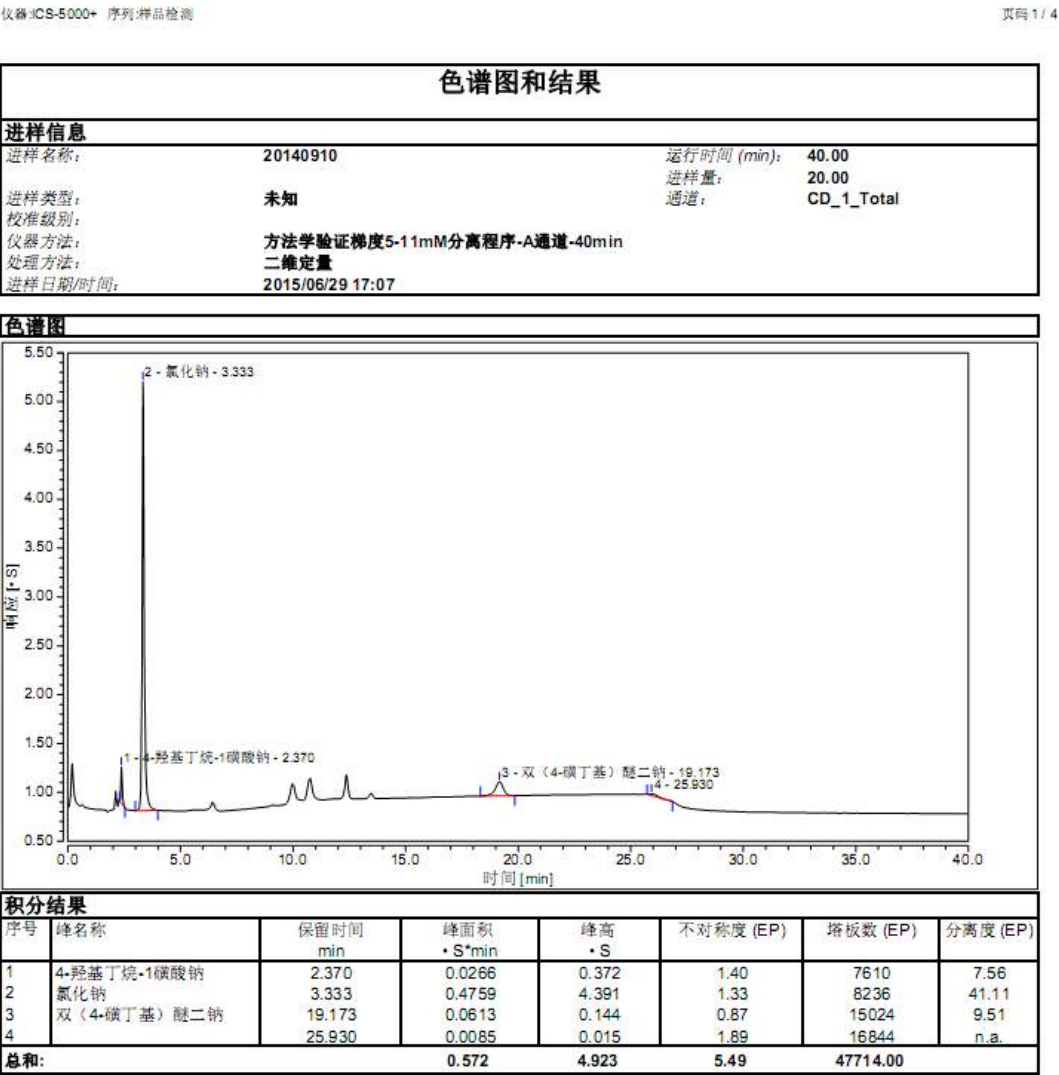
Page 1 of 1

Annex 3-BA-22 Batch analyses-4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfonutyl)ether disodium –Reference solution



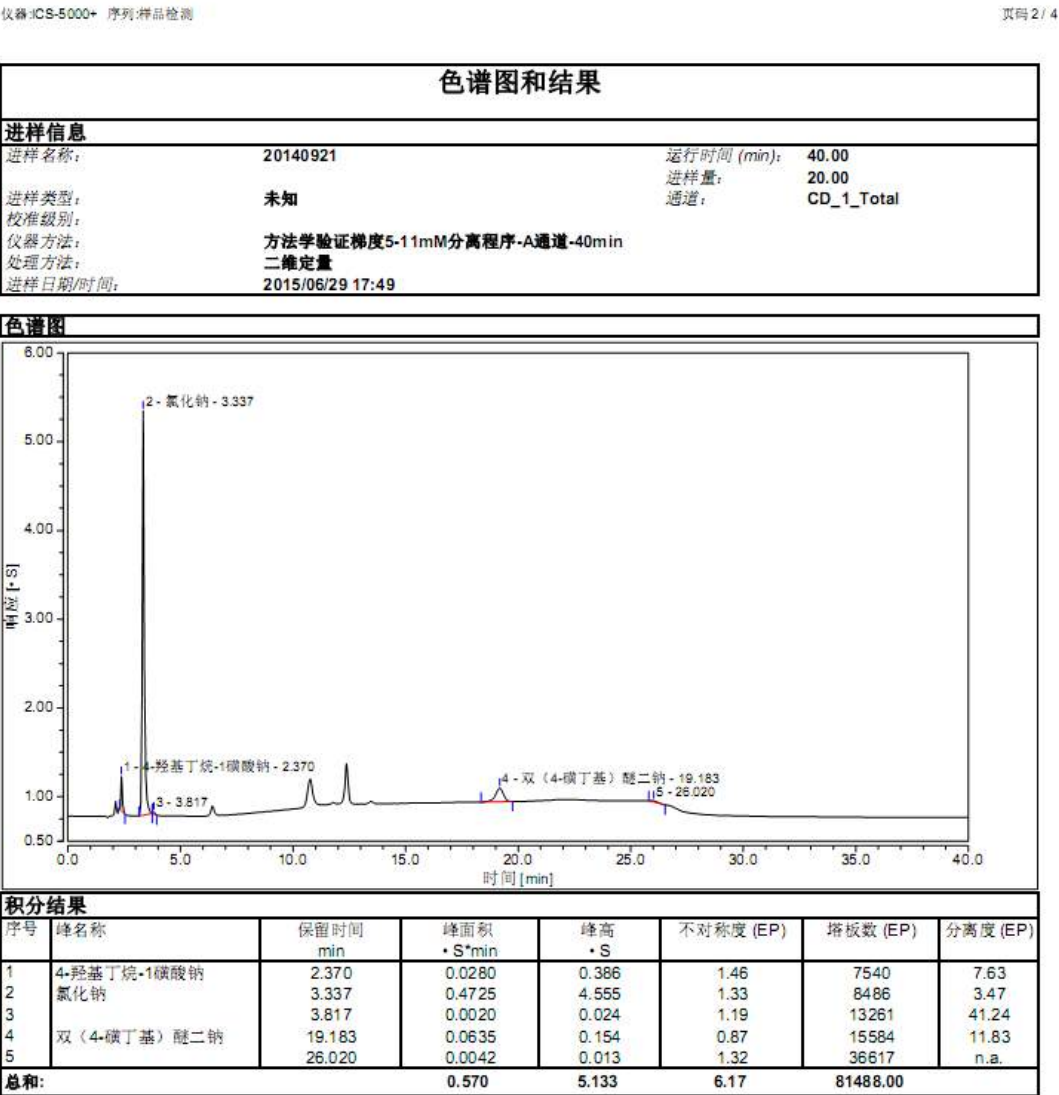
附图 10.4.11-263 SBECD-0时4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定图（对照品）

Annex 3-BA-23 Batch analyses-4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfonutyl)ether disodium –Sample solution 20140910



附图10.4.11-264 SBECD-0时4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定图(20140910)

Annex 3-BA-24 Batch analyses-4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfonutyl)ether disodium –Sample solution 20140921

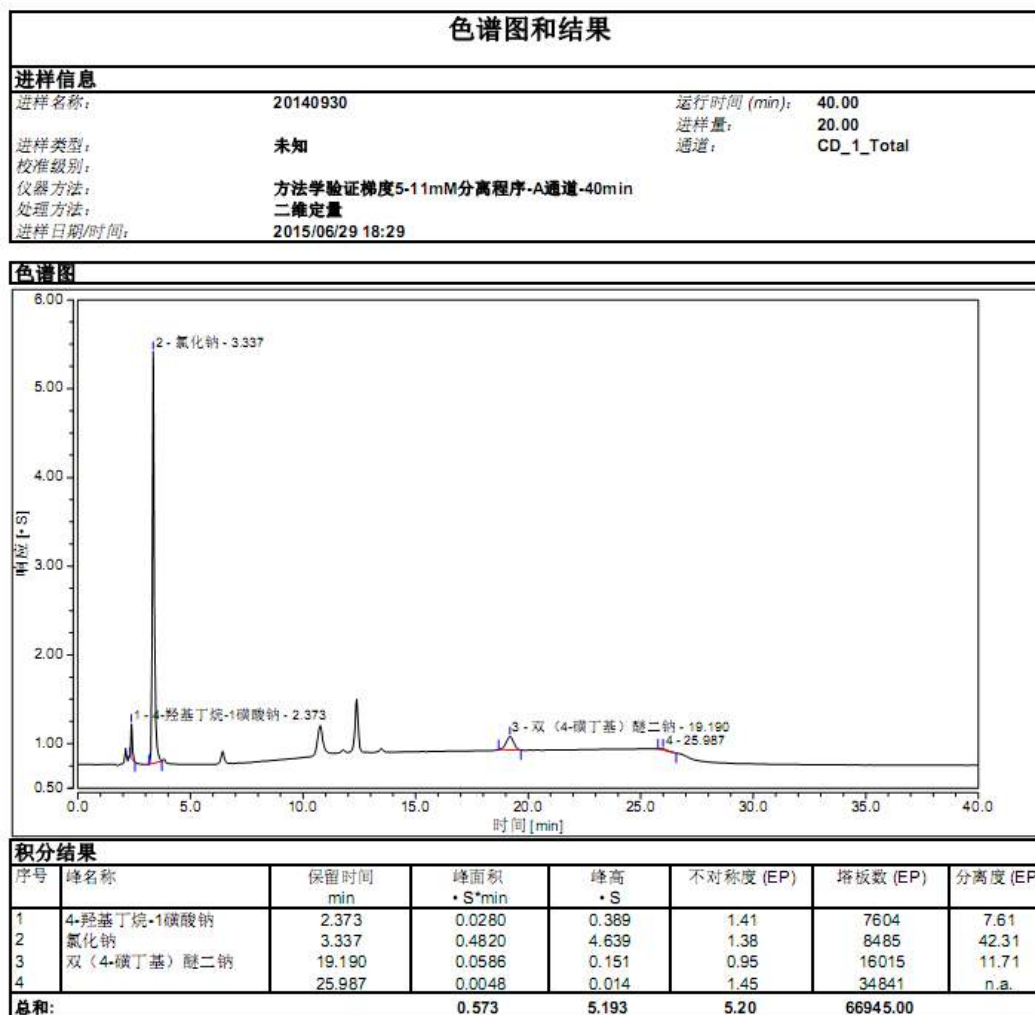


附图10.4.11-265 SBECD-0时4-羟基丁烷-1-磺酸、氯化钠和双（4-磺丁基）醚二钠的测定图（20140921）

Annex 3-BA-25 Batch analyses-4-hydroxybutane-1-sulfonic acid, Sodium chloride and Bis(4-sulfonutyl)ether disodium –Sample solution 20140930

仪器:ICS-5000+ 序列:样品检测

页码 3 / 4



附图10.4.11-266

SBECD-0时4-羟基丁烷-1-磺酸、氯化钠和双(4-磺丁基)醚二钠的测定图(20140930)

Default/积分

Chromleon (c) Dionex
版本 7.2.1.5537

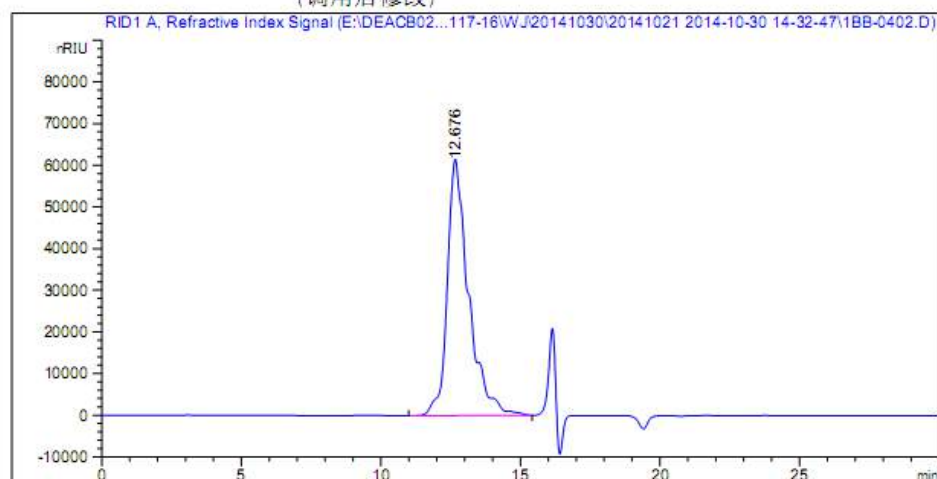
Annex 3-BA-26 Batch analyses-Assay-Reference solution 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BB-0402.D

样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   :    4
仪器         : 1260-2                      位置     : Pl-B-02
进样日期     : 2014/10/30 16:35:39          进样次数  :    2
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/30 14:32:47 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.676	BB	0.7990	3.25964e6	6.07277e4	100.0000

```
总量 :                3.25964e6  6.07277e4
```

```
=====
                        *** 报告结束 ***
=====
```

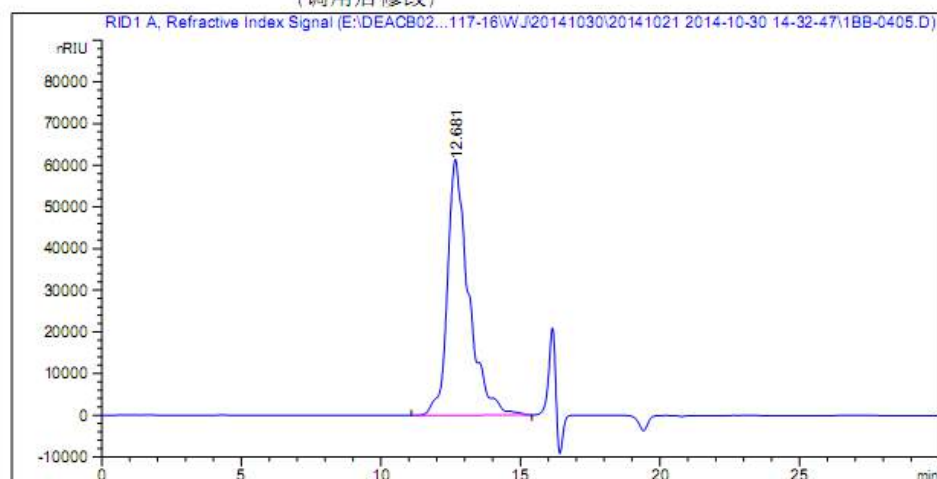
附图10.5-125 SBECD-0时含量测定图 (对照1-2)

Annex 3-BA-27 Batch analyses-Assay-Reference solution 1-5

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BB-0405.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   :    4
仪器         : 1260-2                      位置     : Pl-B-02
进样日期     : 2014/10/30 18:07:23          进样次数  :    5
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序          :    信号
乘积因子       :    1.0000
稀释因子       :    1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.681	BB	0.7965	3.24815e6	6.07628e4	100.0000

总量 : 3.24815e6 6.07628e4

*** 报告结束 ***

附图10.5-128 SBECD-0时含量测定图 (对照1-5)

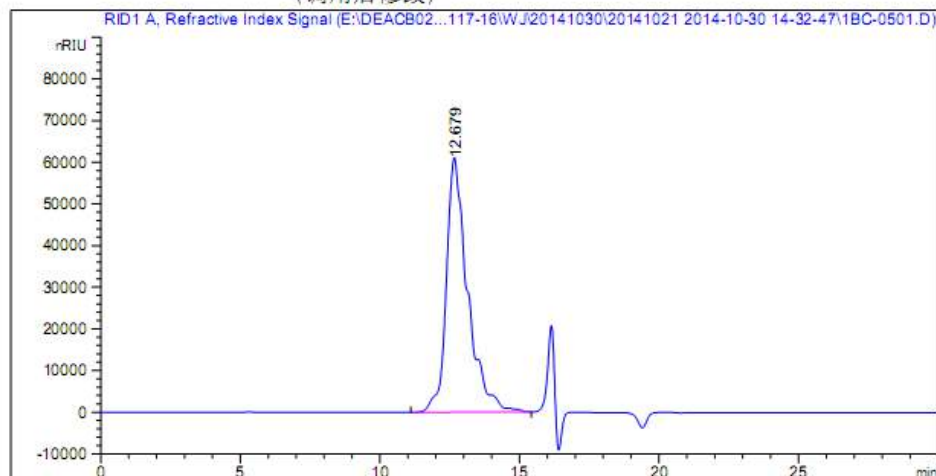
Annex 3-BA-28 Batch analyses-Assay-Reference solution 2-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BC-0501.D

样品名称: DZ-2

```
=====
操作者       : Weijing                      序列行   :    5
仪器         : 1260-2                      位置     : Pl-B-03
进样日期     : 2014/10/30 19:08:32          进样次数  :    1
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
              (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.679	BB	0.7975	3.23515e6	6.04142e4	100.0000

总量 : 3.23515e6 6.04142e4

*** 报告结束 ***

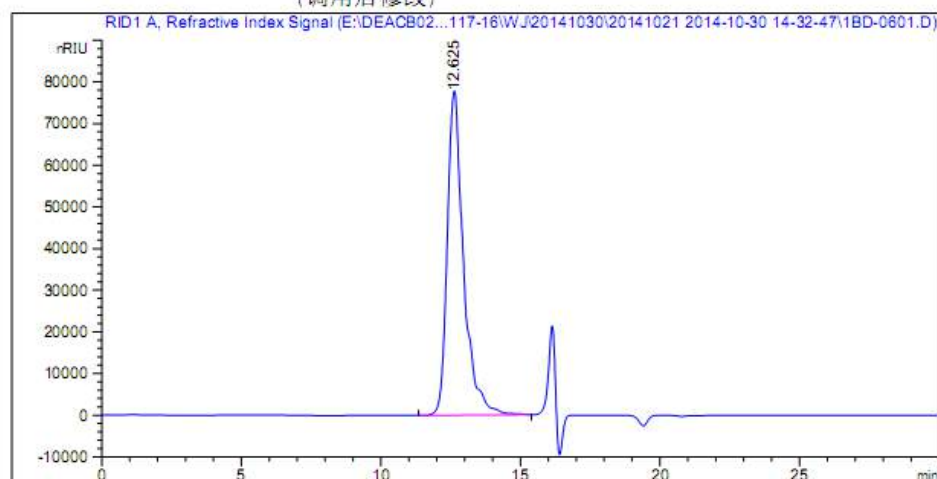
附图10.5-130 SBECD-0时含量测定图 (对照2-1)

Annex 3-BA-29 Batch analyses-Assay-Sample solution 20140910-1-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BD-0601.D
样品名称: 20140910-1

```
=====
操作者       : Weijing                      序列行   :    6
仪器         : 1260-2                      位置     : Pl-B-04
进样日期     : 2014/10/30 20:40:19          进样次数  :    1
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
              (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序          :    信号
乘积因子      :    1.0000
稀释因子      :    1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.625	BB	0.6130	3.16230e6	7.77143e4	100.0000

总量 : 3.16230e6 7.77143e4

=====
*** 报告结束 ***

附图10.5-133 SBECD-0时含量测定图 (20140910-1-1)

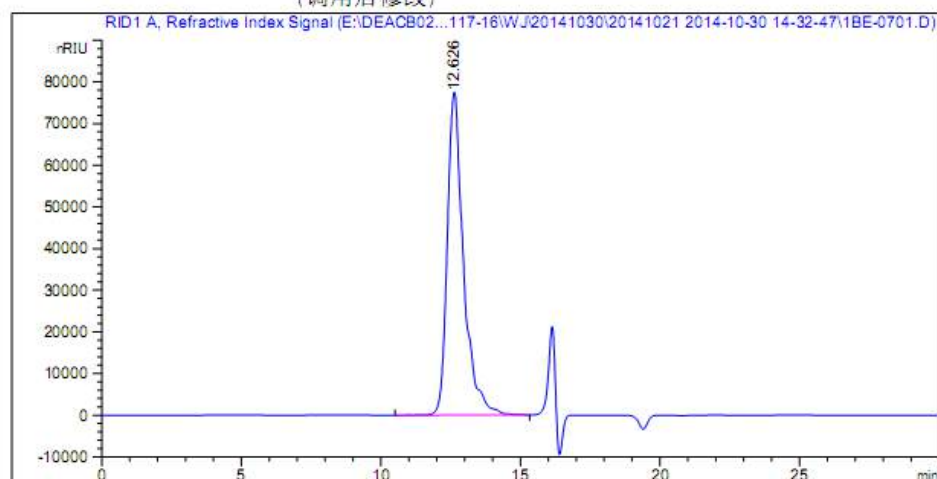
Annex 3-BA-30 Batch analyses-Assay-Sample solution 20140910-2-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BE-0701.D

样品名称: 20140910-2

```
=====
操作者       : Weijing                      序列行 :    7
仪器         : 1260-2                      位置   : P1-B-05
进样日期     : 2014/10/30 21:41:28          进样次数:    1
                                           进样量  : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
              (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.626	BB	0.6114	3.13863e6	7.73953e4	100.0000

总量 : 3.13863e6 7.73953e4

*** 报告结束 ***

附图10.5-135 SBECD-0时含量测定图 (20140910-2-1)

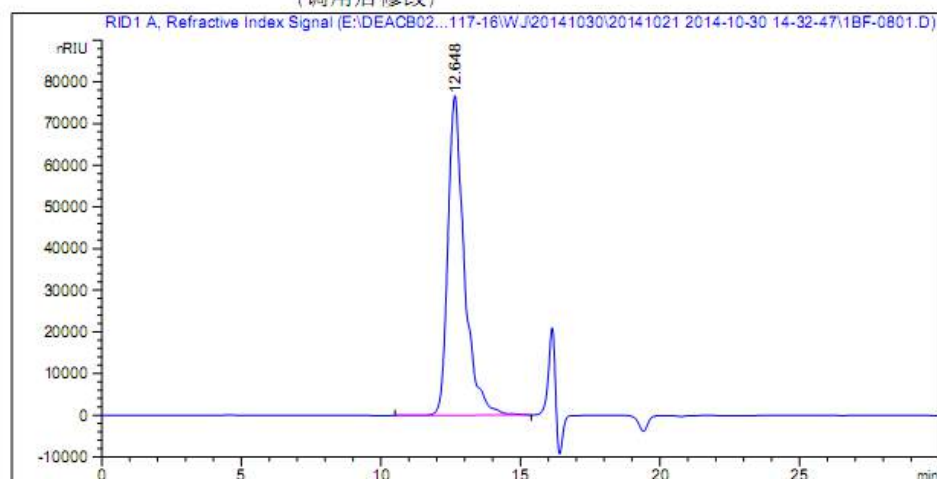
Annex 3-BA-31 Batch analyses-Assay-Sample solution 20140921-1-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BF-0801.D

样品名称: 20140921-1

```
=====
操作者       : Weijing                      序列行 :    8
仪器         : 1260-2                      位置   : Pl-B-06
进样日期     : 2014/10/30 22:42:37          进样次数:    1
                                           进样量  : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
              (调用后修改)
=====
```



面积百分比报告

```
=====
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.648	BB	0.6366	3.15041e6	7.64043e4	100.0000

总量 : 3.15041e6 7.64043e4

*** 报告结束 ***

附图10.5-137 SBECD-0时含量测定图 (20140921-1-1)

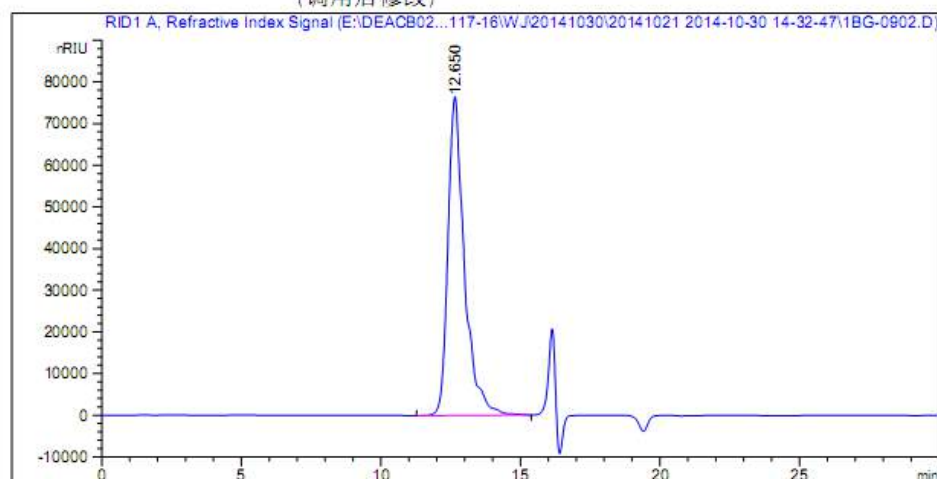
Annex 3-BA-32 Batch analyses-Assay-Sample solution 20140921-2-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BG-0902.D

样品名称: 20140921-2

```
=====
操作者       : Weijing                      序列行   :    9
仪器         : 1260-2                      位置     : Pl-B-07
进样日期     : 2014/10/31 0:14:23          进样次数  :    2
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
               (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.650	BB	0.6382	3.15041e6	7.61544e4	100.0000

总量 : 3.15041e6 7.61544e4

*** 报告结束 ***

附图10.5-140

SBECD-0时含量测定图 (20140921-2-2)

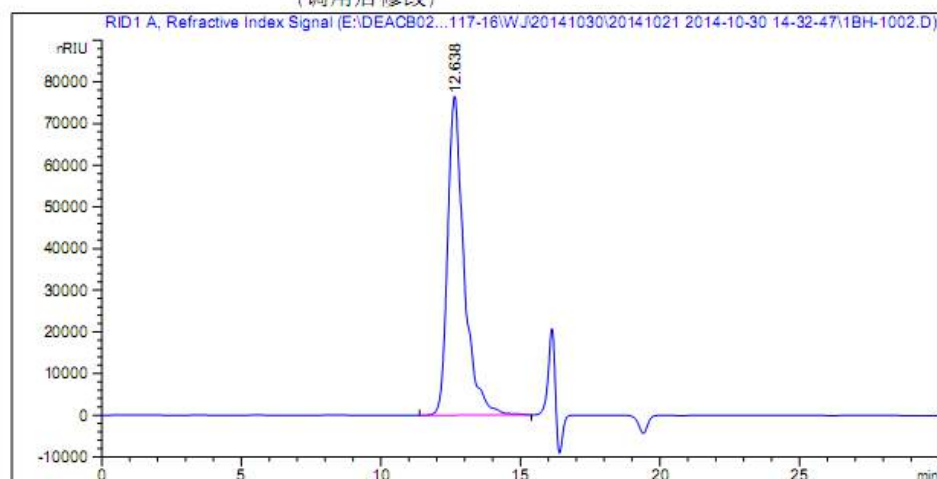
Annex 3-BA-33 Batch analyses-Assay-Sample solution 20140930-1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BH-1002.D

样品名称: 20140930-1

```
=====
操作者       : Weijing                      序列行 : 10
仪器         : 1260-2                      位置   : Pl-B-08
进样日期     : 2014/10/31 1:15:33          进样次数 : 2
                                           进样量  : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
               (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.638	BB	0.6360	3.14463e6	7.63528e4	100.0000

总量 : 3.14463e6 7.63528e4

*** 报告结束 ***

附图10.5-142

SBECD-0时含量测定图 (20140930-1-2)

1260-2 2014/10/31 10:25:00 Weijing

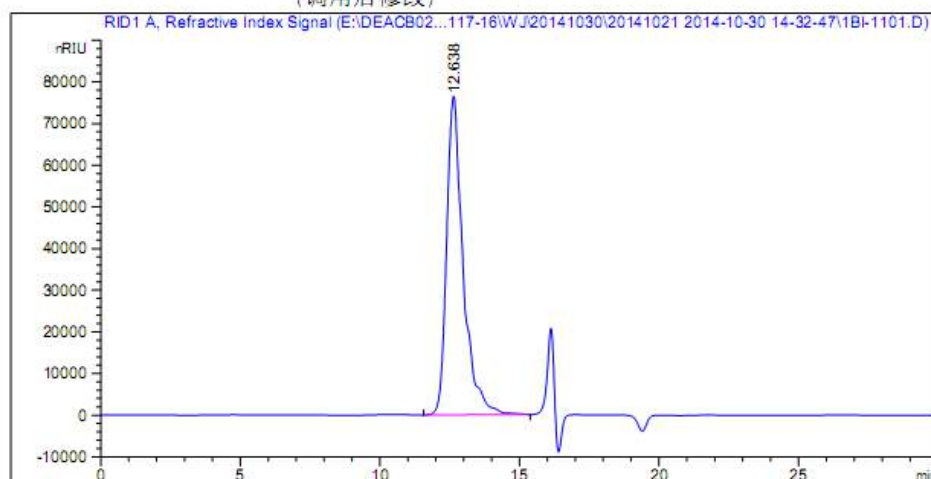
页 1/1

Annex 3-BA-34 Batch analyses-Assay-Sample solution 20140930-2-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\1BI-1101.D
样品名称: 20140930-2

```
=====
操作者       : Weijing                      序列行 : 11
仪器         : 1260-2                      位置   : PI-B-09
进样日期     : 2014/10/31 1:46:07          进样次数 : 1
                                           进样量  : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/30 17:32:54 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141030\20141021 2014-10-30 14-32-47\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/31 10:21:04 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.638	BB	0.6368	3.14730e6	7.62955e4	100.0000

总量 : 3.14730e6 7.62955e4

*** 报告结束 ***

附图10.5-143 SBECD-0时含量测定图 (20140930-2-1)

3.2.S.4.5 Justification of Specification

We added arsenic salt and sulfate on the basis of USP37 to control the quality of Betadex Sulfobutyl Ether Sodium better.

The analysis procedures for Betadex, Sodium chloride, 4-hydroxybutane-1-sulfonic acid, Bis(4-sulfonutyl)ether disodium and Average degree of substitution are corresponding to USP37; that for 1,4-butane sultone is slightly changed about heating program on the basis of USP37; that for Assay is slightly changed about mobile phase and flow rate on the basis of USP37; other items are controlled by relevant ChP method and USP limit.

All the analysis procedures are validated and qualified, so this specification is suitable to control the quality of Betadex Sulfobutyl Ether Sodium.

3.2.S.5 Reference Standards

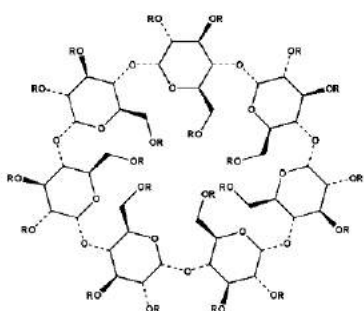
<i>Reference standard</i>	<i>Grade</i>	<i>Origin</i>	<i>Batch No.</i>
Betadex Sulfobutyl Ether Sodium	Primary standard	USP	F0K026
Betadex Sulfobutyl Ether Sodium	Innovated medicine	Cydex Pharmaceuticals Company	NC-04A-140128
Betadex Sulfobutyl Ether Sodium	Work standard	Watson International Ltd	130923S
Betadex	Primary standard	National Institutes for Food and Drug Control	100317-201203
1,4-butane sultone	Primary standard	TRC	1-AJC-182-1
Diethyl sulfone	Primary standard	TRC	4-HBN-60-1
4-hydroxybutane-1-sulfonic acid	Primary standard	Shanghai Hekang biotechnology	C8108-HK20140303Y1
Bis(4-sulfonutyl)ether disodium	Primary standard	TRC	10-DPP-102-2
Sodium chloride	Primary standard	National Institutes for Food and Drug Control	100376-201202

CoA for Betadex Sulfobutyl Ether Sodium RS from USP

U.S. Pharmacopeia
The Standard of Quality™

USP Certificate

Betadex Sulfobutyl Ether Sodium LOT F0K026

**Molecular Formula**
 $C_{42}H_{70-n}O_{35} \cdot (C_4H_8SO_3Na)_n$ **Molecular Weight**
2163 (when $n = 6.5$)**CAS Number**
182410-00-0**LABEL TEXT**

Lot No.: F0K026

**USP REFERENCE STANDARD****BETADEX SULFOBUTYL ETHER SODIUM 500 mg**

Warning! May cause an allergic skin reaction

Do not dry. For quantitative applications, determine the water content immediately at the time of use and use a value of 0.990 mg of betadex sulfobutyl ether sodium per mg of material on the analytical basis.

Keep container tightly closed. Protect from light.

This material is hygroscopic.

USP 12651 Turnbross Place, Rockville, MD, 1-201-991-9338

COTR 1055518

Intentionally over-labeled for GHS compliance

For use with specified USP compendia, this material must be tested and found to meet all requirements prior to use in manufacturing.

Contaminated work clothes must not be allowed out of the workplace. Wear protective gloves. If on skin, wash with plenty of water. If skin irritation or rash occurs, get medical advice/attention. Wash contaminated clothing before reuse. Dispose of contents/container in accordance with local/regional/national/international regulations.

Jeri L. Joth

QA Director



TYPICAL ELECTROPHEROGRAM

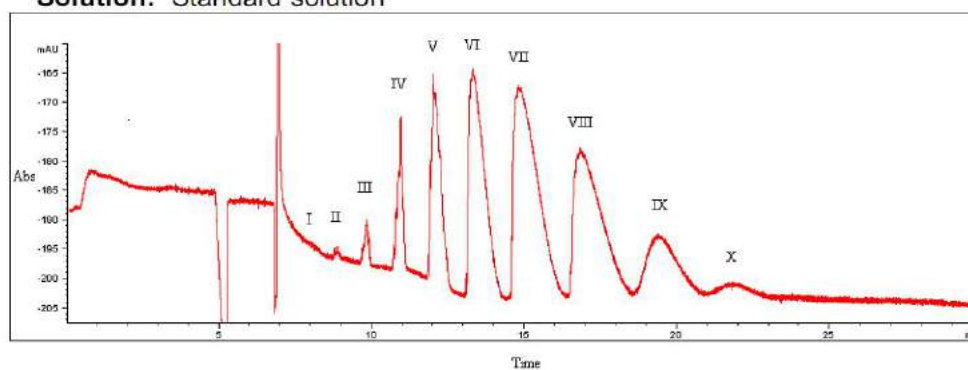
USP Betadex Sulfobutyl Ether Sodium RS

Lot: F0K026 (Cat. 1065550)

USP Monograph: Betadex Sulfobutyl Ether Sodium, *PF 37(2)*

Test: Average Degree of Substitution

Solution: Standard solution



This electropherogram is supplied for information only, unless otherwise specified in an applicable monograph or general chapter.

Calculation Value

Unless otherwise stated on the Reference Standard label, a value of 100.0% should be used in the compendial applications for which the use of this Reference Standard is intended. Please refer to the specific Reference Standard label for further information.

Expiration

Current lots are identified in the current USP Catalog. In some cases, the previous lot may still be considered valid for use. If so, it is identified in the column marked "Previous Lot/Valid Use Date."

It is the responsibility of each user to determine that this lot is current or valid when used. For the most up-to-date information, please refer to the USP Store at www.usp.org.

Instructions for Use

Follow the instructions on the label of the USP Reference Standard and in the appropriate USP documentary standard(s).

Non-Monograph Use

The suitability of this Reference Standard for use in non-compendial applications is solely the responsibility of the user.

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CoA for Betadex Sulfobutyl Ether Sodium innovated medicine from
Cydex Pharmaceuticals Company



CAPTISOL® - Research Grade
(β -Cyclodextrin Sulfobutyl Ethers, Sodium Salts)

Certificate of Analysis (rev. 0)

Batch Number: NC-04A-140128

Test	Specification	Result
Appearance	White to off-white solid essentially free from foreign matter	Pass
Identification (IR)	Spectrum is consistent with the SBECD standard	Pass
Average Degree of Substitution (CE)	6.0 - 7.1	6.6
β -Cyclodextrin	Maximum 0.5%	< 0.05%
Water (by KF)	Maximum 15.0%	5.6%*
Assay (anhydrous basis)	Minimum 95%	99.4%

These results were obtained at the time of testing. The values may change depending upon material handling and exposure to atmospheric conditions.

Date of Manufacture: January 2014
Manufactured by: Hovione FarmaCiencia SA,
Sete Casas,
Loures, Portugal 2674-506

References: 17CX01.HQ00128

Released By: 
Vincent Antle, PhD
Sr. Director of Technical Operations
& Quality Assurance

Date: 07 Oct 14

STORAGE: Store at ambient temperature in sealed containers. Protect from moisture.

CAPTISOL®-Research Grade is not for human use.

CyDex Pharmaceuticals, Inc • 2029 Becker Drive, Suite 217 • Lawrence, KS 66047 • P: 913.402.3514 • www.captisol.com

Using direction for Betadex RS

国家药品标准物质使用说明书

倍他环糊精

【类别】 化学对照品

【批号】 100317-201203

【分子式】 $(C_8H_{10}O_5)_7$

【分子量】 1134.99

【CAS 号】 7585-39-9

【用途】 本品为倍他环糊精 (Betacyclodextrin)，供含量测定用。

供 HPLC 法测定，按 $(C_8H_{10}O_5)_7$ 计，本品含量为 100.0%

【包装及装量】 棕色瓶，约 100mg/支

【保存条件】 遮光，密闭保存

【注意事项】 105℃干燥 2 小时后使用，建议 RH 30%以下使用



中国食品药品检定研究院

National Institutes for Food and Drug Control

地址:北京市东城区天坛西里 2 号 电话:01067095219 传真:01067028278 网址:www.nifdc.org.cn

Translation of the direction above

The national drug standard substance operating manual

Betadex

[Type] chemical reference standard

[Batch number] 100317-201203

[Molecular formula] $(C_6H_{10}O_5)_7$

[Molecular weight] 1134.99

[CAS number] 7585-39-9

[Usage] this substance is Betacyclodextrin, and can be used for assay determination by HPLC, the assay is 100.0% expressed in $(C_6H_{10}O_5)_7$

[Package and capacity] brown bottle, about 100mg/bottle

[Storage condition] shade and sealed preservation

[Attention] used after dried for 2h at 105℃, and RH < 30% is advised

National Institutes for Food and Drug Control

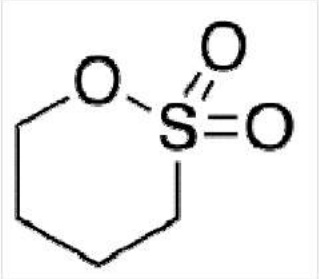

Address: No. 2 in Tiantan xi li, Dongcheng Distric, Beijing city

Telephone: 01067095219 Website: www.nifdc.org.cn

CoA for 1,4-butane sultone from the supplier

W

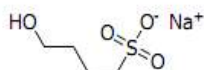
页码, 1/1(W)

CERTIFICATE OF ANALYSIS			
2 Brisbane Road, North York, ON, M3J2J8, CANADA Tel: (416) 665-9696, Fax: (416) 665-4439 Email: orders@trc-canada.com Website: www.trc-canada.com			
1. Identification			
CAS Number:	1633-83-6	Cat. Number:	B689945
Product:	1,4-Butane Sultone	Synonym:	1,2-Oxathiane 2,2-Dioxide; 4-Hydroxy-1-butanefulfonic Acid δ -Sultone; 1,4-Butane Sultone; Butane Sultone; NSC 71999; δ -Butane Sultone; δ -Valerosultone
Structure:			
		Molecular Formula:	C ₄ H ₈ O ₃ S
		Molecular weight:	136.17
		Source of Product:	
2. Analytical Information			
Lot Number:	1-AJC-182-1	Boiling Point:	N/A
Melting Point:	N/A	Atmosphere:	Air
Appearance:	Clear Colourless Oil	Solubility:	Chloroform, Methanol
Method for Determining Identity:	1H NMR (CDCl ₃) Spectroscopic and Mass Spectrometric Analysis		
Purity:	97%	Stability:	Not Determined
Additional Info.:	TLC Conditions: SiO ₂ ; Ethyl Acetate : Hexane = 3 : 2; Visualized with KMnO ₄ ; Single spot, R _f =0.65. 1H NMR and Mass spectra conform to structure.		
			
Philip Chan, Head of Quality Assurance		QC Test Date:	Retest Date:
		8/30/2012 (MM/DD/YYYY)	8/30/2015 (MM/DD/YYYY)

CoA for 4-hydroxybutane-1-sulfonic acid from the supplier

Youchemicals Limited

DATE	March 3, 2014
PRODUCT NAME	Sodium 4-Hydroxybutane-1-Sulphonate
FORMULA	C ₄ H ₉ NaO ₄ S
CAS RN	31465-25-5
Catalog No.	C8108
FORMULA WEIGHT	176.16663
BATCH NUMBER	C8108-HK20140303Y1



CERTIFICATE OF ANALYSIS

Item	Inspection Standard	Results
Appearance	White solid	Conform
NMR Spectrum	Consistent with structure	Conform
Assay by NMR	95%	≥95%
Conclusion: This product by inspection accords with the standard Corporation		

QA: Yishu Du

QC: Chaoqing He

CoA for Bis(4-sulfonutyl)ether disodium from the supplier



Bringing you products for innovative research.

CERTIFICATE OF ANALYSIS2 Brisbane Road, North York, ON, M3J 2J8 Canada Tel: (416) 665-9696 Fax: (416) 665-4439
E-mail: orders@trc-canada.com Website: www.trc-canada.com**1. Identification**CAS Number:

183278-30-0

Catalogue Number:

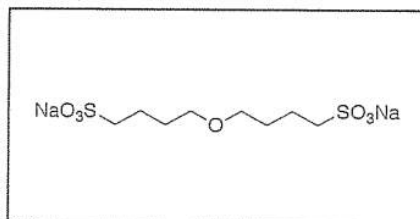
B588850

Product:

Bis(4-sulfobutyl)ether Disodium

Synonyms:

4,4'-Oxybis-1-butanefulfonic Acid Disodium Salt; WAS-18;

Structure:Molecular Formula: $C_8H_{16}Na_2O_7S_2$ Molecular Weight:

334.32

Source of Product:

Synthetic

2. Analytical InformationLot Number:

10-DPP-102-2

Melting Point:

>240°C (dec.)

Boiling Point:

N/A

Atmosphere:

Inert Gas

Appearance of Product:

White Solid

Solubility

Water

Method for Determining Identity: ^1H NMR (D_2O) and MSStability

Hygroscopic

Purity:

95%

Long Term Storage Condition:

Hygroscopic, Refrigerator, Under Inert Atmosphere

Additional Information: ^1H NMR and MS conform to structure.

Sodium Content: 14.80%

Elemental Analysis: (Found) %C: 26.65, %H: 4.40; (Calculated) %C: 28.74, %H: 4.82

Phillip Chan, Head of Quality AssuranceQC Test Date
January 17, 2013Retest Date
January 17, 2016

CoA for Diethyl sulfone from the supplier



Bringing you products for innovative research.

Certificate of Analysis

2 Brisbane Road, Toronto, ON. M3J 2J8 Canada Tel: (416) 665-9696 Fax: (416) 665-4439
E-mail: orders@trc-canada.com Website: www.trc-canada.com

1. Identification

CAS Number:

597-35-3

Catalogue Number:

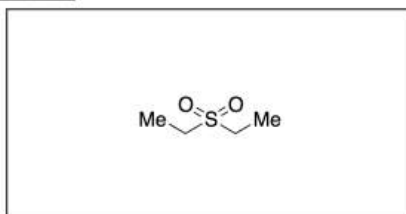
D445010

Product:

Diethyl Sulfone

Synonyms:

1,1'-Sulfonylbisethane; Ethyl Sulfone

Structure:**Molecular Formula:**C₄H₁₀O₂S**Molecular Weight:**

122.19

Source of Product:

N/A

2. Analytical Information

Lot Number:

4-HBN-60-1

Melting Point:

69 - 71 °C (dec.)

Boiling Point:

N/A

Atmosphere:

Air

Appearance of Product:

White Solid

Solubility

Chloroform, Ethyl Acetate

Method for Determining Identity:¹H NMR (CDCl₃), ¹³C NMR (CDCl₃), and MS**Stability**

Not Determined

Purity:


98%

Long Term Storage Condition:

Room Temperature

Additional Information:¹H NMR, ¹³C NMR, and MS conform to structure.

Elemental Analysis: (Found) %C: 38.38, %H: 8.40; (Calculated) %C: 39.38, %H: 8.25


Philip Chan, Head of Quality Assurance**QC Test Date**
June 30, 2014**Retest Date**
June 28, 2017

3.2.S.6 Container Closure System

1. Description of the container closure system

We chose pharmaceutical low density polyethylene bag, which is commonly used as inner package for drug, as the package material that direct contact the pharmaceutical excipients. Betadex Sulfobutyl Ether Sodium produced by Watson International Ltd packaged in two-layer pharmaceutical low density polyethylene bag have undergone six months of accelerated test and nine months of long term test, all the items are basically the same to the initial status, the above can indicate that the commercial package of Betadex Sulfobutyl Ether Sodium can keep the product from influence during manufacture, transportation and storage. The outer package of this product is fibreboard drum and physical destroy can be prevented.

2. Description of packaging materials

2.1 Inner packaging material-plastic bags

2.1.1 Description of the supplier

Name: Shandong Kaibo pharmaceutical package material Co.,Ltd.	
Address: Guanwang industrial zone at west section of Yanggan road, Anqiu city, Shandong province	
Postcode: 262100	Website: http://www.sdkaibo.net/
Telephone: +86-536-4338888	City: Anqiu City
Email: info@kxfood.be	Province: Shandong Province

Registration certificate of the supplier

【副本】

国家食品药品监督管理总局
药品包装用材料和容器注册证
(I 类)

注册证号: 国药包字20140206

根据《中华人民共和国药品管理法》和《药品包装
用材料和容器管理办法》(暂行)的规定, 兹批准下述企业
的下述药品包装用材料和容器注册。

品种名称: 药用低密度聚乙烯膜、袋

规格: _____

仅供使用本证药品备案用

企业名称: _____

地 址: 山东省安丘市安丘路西段(安丘工业园)

备 注: 1. 本证有效期至 2019年 3 月 25 日

1. 产品标准: YBB00072005 药用低密度聚乙烯膜、袋
2. 原注册证(国药包字20080017)注销

国家食品药品监督管理总局

2014年8月26日

No. 1302204

Translation of the certificate above

China Food and Drug Administration
Registration certificate for pharmaceutical package material and container
(Grade one)

Registration number: 20140206

According to the regulation in <The Drug Administration Law of the PRC> and <Administrative Measures for Pharmaceutical Package Material and Container>, the registration of the pharmaceutical package material and container produced by the company below is approved.

Name of material: pharmaceutical low density polyethylene membrane and bag

Specification: ---

Name of company: Shandong Kaibo pharmaceutical package material Co.,Ltd.

Address: Guanwang industrial zone at west section of Yanggan road, Anqiu city, Shandong province

Remark: expiry date of the certificate is 2019.03.25

1. Product standard: YBB00072005 pharmaceutical low density polyethylene membrane and bag
2. The quondam certificate 20080017 is cancelled

China Food and Drug Administration
2014.03.26
No.1302204

2.1.2 Internal specification, analysis procedure and CoA

Specification

<i>Item</i>	<i>Acceptance criteria</i>
Size	Internal bags 420*360*0.15; outer bags 400*400*900*0.15
Material	The surface should be without hole, foreign matter, odor, adhesion, uneven coating, separate in composition layer, significant damage, bubble, wrinkle or dirt.
Printing quality	The polythene plastic should be non-poisonous and odorless
Pressure resistant properties	Fracture or leak are forbidden
Drop performance	Fracture is forbidden
Airtightness	It should be airtight after heat seal.
Heavy metal	NMT1ppm
Readily oxidizable substance	NMT1.5ml
Nonvolatile matter	Difference between residue in water and the blank should not be more than 30.0mg
	Difference between residue in 65% ethanol and the blank should not be more than 30.0mg
	Difference between residue in normal hexane and the blank should not be more than 30.0mg
Microbial limit	Bacterial count $\leq 1000\text{cfu}/100\text{cm}^2$
	Mould and yeast count $\leq 100\text{cfu}/100\text{cm}^2$
	E.coli should not be detected

Analysis procedure

Size

Internal bags 420*360*0.15; outer bags 400*400*900*0.15.

Texture

There should not appear hole, foreign matter, odor, adhesion, separation between composite layer, significant damage, bubble, wrinkle, dirt etc.

Printing quality

The material is polyvinyl plastics and should be non-poisonous and odorless.

Pressure resistant properties

Take five bags, fill in water to half of its volume and heat seal (refer to the heat seal process in the manufacturing technology). Put the sample between two plates, the plates should be horizontal, non-deformation and smooth at the surface that contact bags, the area of plates should be larger than the testing bags. Add weights according to relevant requirement and hold for 1 minute, burthen is the total weight of upper plate and weights, visually detected there should not be fracture and leakage.

Drop performance

Take five bags, fill in water to half of its volume and heat seal (refer to the heat seal process in the manufacturing technology).

Drop the bag from a height of 0.5m to the ground. The test ground should be flat and hard, visually detected there should not be fracture and leakage.

Airtightness

The inner package should not leak after sealed.

Dissolved matter test

Cut 600 cm² of the sample into tablets that 3cm in length and 0.3cm in width, transfer them into conical flask with cover, prepare two duplicates. Respectively add 200ml of water (70±2°C), 65% ethanol (70±2°C), normal hexane (58±2°C) into separate flask, let them to stand for 2hours, add the solvent used to the original volume when it is cooled down to room temperature. Use the same bath of water, 65% ethanol and normal hexane as blanks.

Heavy metal

Add 2ml of pH 3.5 acetate buffer solution to 20ml of the water immersion liquid, the heavy metal content should not exceed 1ppm.

Readily oxidizable substance

Add 20ml of 0.002mol/L potassium permanganate titrating solution and 1ml of diluted sulfuric acid to 20ml of the water immersion solution, boil for 3minutes and cool down immediately, add 0.1g of potassium iodine, keep it in darkness for 5minutes, titrate with 0.01mol/L sodium thiosulfate titrating solution, add 0.25ml of starch indicating solution when the ending point is approaching, and continue titrating to colorless. Titrate the blank in the same way, the difference between the volumes consumed by sample and blank should not be more than 1.5ml.

Nonvolatile matter

Transfer 100ml of each of the immersion liquid above and relevant blank into separate evaporating dishes that have been dried to constant weight, evaporate to dryness on water bath and dry at 105°C for 2 hours, accurately weigh them after cooled down, the differences between sample solutions and relevant blanks should not exceed 30.0mg.

Microbial limit

Place a metal board with a hole of area 25cm², previously disinfected, on the inner surface of a bag as sample area. Wipe the sample area five times with a sterile swab soaked with 0.9% sterile sodium chloride solution, and then repeat the procedure with another sterile swab subjected to the same treatment. Respectively wipe sample areas at different bags for 10 times (total area: 100 cm²). After wiping, snip the swabs and put them in a conical flask (or big test tube) containing 30ml of 0.9% sterile sodium chloride solution. When all swabs are put in the flask, immediately shake the flask for 1 min and the test solution is obtained.

Use the test solution to perform the microbial limit test according to the general chapter 1106 and 1107 in ChP2015. TAMC is not more than 1000cfu/100 cm². TYMC is not more than 100 cfu/100 cm². Escherichia Coli should not be detected.

Watson International Ltd

Certificate of analysis

Purchase unit: Watson International Ltd

Inspecting standard: YBB00072005

Name		Pharmaceutical low density polyethene bag	Batch number	140722
Specification		420*360*0.15	Inspecting date	2014.07.23
Sample status		In good condition	Test amount	6
Inspecting item		According to standard	Test result	Conclusion
Appearance		The surface should be smooth, color should be uniform, hole, foreign matter or orodor are forbidden, heat seal part should be flat and without dashed.	Qualified	Qualified
Barrier properties	Water vapor transmission	NMT15g/(m ² *24h)	7.6g/(m ² *24h)	Qualified
	Oxygen transmission	NMT4000cm ³ (m ² .24h.0.1mpa)	2234cm ³ (m ² .24h.0.1mpa)	Qualified
Physical properties	Tensile strength	The mean tensile strength of lengthways and widthways should not be less than 10mpa	26mpa	Qualified
	Fracture elongate rate	The mean tensile strength of lengthways and widthways should not be less than 200%	298%	Qualified
	Sealing strength	The mean value should not be less than 7.0N/15mm	12N/15mm	Qualified
Residue on ignition		NMT0.1%	0.02%	Qualified
Heavy metal		NMT1ppm	< 1ppm	Qualified
Readily oxidizable substance		NMT1.5ml	0.22ml	Qualified
Nonvolatile matter	Water	The difference with blank should not be more than 30.0mg	5.4mg	Qualified
	Ethanol	The difference with blank should not be more than 30.0mg	6.2mg	Qualified
	Normal hexane	The difference with blank should not be more than 30.0mg	14.9mg	Qualified
Microbial limit	TAMC	NMT1000cfu/100cm ²	< 10cfu/100cm ²	Qualified
	TYMC	NMT100cfu/100cm ²	< 10cfu/100cm ²	Qualified
	E.coli	Should not be detected	Not detected	Qualified

Conclusion: the sample meets the requirement of YBB00072005.

Inspector: Xuechun Wang

Reviewer: Yujie Wang

Principal: Aiqin Zhu

STP-JS-01-014-00

Watson International Ltd
CoA for drug package bag

Number: ZY140722

Batch No.	140722	Packing size	420mm*360mm*0.15mm
Sampling time	2014.08.12	Batch size	7
Sampling site	Inspection waiting area	/	/
Inspection content			
Item	Acceptance criteria	Inspection result	Conclusion
Size	According to contract	Qualified	Qualified
Material	The surface should be without hole, foreign matter, odor, adhesion, uneven coating, separate in composition layer, significant damage, bubble, wrinkle or dirt.	Qualified	Qualified
Printing quality	The polythene plastic should be non-poisonous and odorless	Qualified	Qualified
Pressure resistant properties	Fracture or leak are forbidden	Qualified	Qualified
Drop performance	Fracture is forbidden	Qualified	Qualified
Airtightness	It should be airtight after heat seal.	Qualified	Qualified
Heavy metal	NMT1ppm	<1ppm	Qualified
Readily oxidizable substance	NMT1.5ml	0.25ml	Qualified
Nonvolatile matter	Difference between residue in water and the blank should not be more than 30.0mg	5.45mg	Qualified
	Difference between residue in 65%ethanol and the blank should not be more than 30.0mg	6.50mg	Qualified
	Difference between residue in normal hexane and the blank should not be more than 30.0mg	15.35mg	Qualified
Microbial limit	TAMC \leq 1000cfu/100cm ²	420cfu/100cm ²	Qualified
	TYMC \leq 100cfu/100cm ²	60cfu/100cm ²	Qualified
	E.coli should not be detected	Not detected	Qualified
Conclusion	All the items meet the requirement of YBB00132002.		
Inspector: Ningning Liang		Reviewer: Yuanyuan Li	Reporting date: 2014-08-17

2.2 Outer packaging materials-fibreboard drum

2.2.2 In-house specification, analysis procedure and CoA

Specification

Item	Acceptance criteria			
Size	Item	Inside diameter/mm	Inside height/mm	Outside height/mm
	Test result	370	440	460
Barrel	It should be round, complete and smooth, without out-of-round, denting, deflection, damage, wrinkle or tackless			
Barrel hoop	Without paper tongue			
Round roll rim	Firm and flat, metal hoops should not be burnthrough or cold joint, clear corrosion or chap can not be accepted. Zinc-plated hoops should be bright and without fall off.			
Obturator	It should be firmly connected and can be flexibly unlocked, can be well sealed when close the barrel and the bung, the zinc plate should bright and without fall off.			
Print	The image should be clear and uniform, obvious dirt can not be accepted.			

Analysis procedure

Appearance

Inspected visually in natural light.

Barrel: it should be round, complete and smooth, without out-of-round, denting, deflection, damage, wrinkle or tackless.

Round roll rim: no paper tongue.

Barrel hoop: firm and flat, metal hoops should not be burnthrough or cold joint, clear corrosion or chap can not be accepted. Zinc-plated hoops should be bright and without fall off.

Obturator: it should be firmly connected and can be flexibly unlocked, can be well sealed when close the barrel and the bung, the zinc plate should bright and without fall off.

Print: the image should be clear and uniform, obvious dirt can not be accepted.

Size inspection

Detected by common use measure with a precision of 1mm.

Item	Limit deviation
Inside diameter	370±2
Inside height	440±4
Outside height	460±6

STP-JS-02-020-00

Watson International Ltd
CoA for fibreboard drum

Number: ZY130408

Batch No.	130408				Packing size	370*460
Amount	200				Test amount	10
Inspection content						
Item	Acceptance criteria				Inspection result	Conclusion
Size	Item	Inside diameter/mm	Inside height/mm	Outside height/mm	Qualified	Qualified
	Test result	370	440	460		
Barrel	It should be round, complete and smooth, without out-of-round, denting, deflection, damage, wrinkle or tackless				Qualified	Qualified
Barrel hoop	Without paper tongue				Qualified	Qualified
Round roll rim	Firm and flat, metal hoops should not be burnthrough or cold joint, clear corrosion or chap can not be accepted. Zinc-plated hoops should be bright and without fall off.				Qualified	Qualified
Obturator	It should be firmly connected and can be flexibly unlocked, can be well sealed when close the barrel and the bung, the zinc plate should bright and without fall off.				Qualified	Qualified
Print	The image should be clear and uniform, obvious dirt can not be accepted.				< 1ppm	Qualified
Conclusion	All the items meet the requirement of in-house standard.					
Inspector: Ningning Liang			Reviewer: Yuanyuan Li		Reporting date: 2013-05-23	

3.2.S.7 Stability

3.2.S.7.1 Stability Summary Conclusion

Stability research, including stress testing, accelerated testing and long-term testing, has been conducted on the final product of Betadex Sulfobutyl Ether Sodium by Watson International Ltd. The tests are designed according to ICHQ1 Guideline.

Information of sample

Test type	Stress testing	Accelerated testing	Long-term testing
Batch number	20140910	20140910	20140910
		20140921	20140921
		20140930	20140930

Testing conditions,

1. Stress testing

1.1 Photolysis testing

Place a quantity of the package-removed test specimen at a place with the overall illumination of 5000lx, sample at the 5th and the 10th day and examine, and compare the results with that of batch inspection.

1.2 High temperature testing

Place a quantity of the package-removed test specimen at 60°C, sample at the 5th and the 10th day respectively and examine, and compare the results with that of batch inspection.

1.3 High humidity testing

Place a quantity of the package-removed test specimen at 25±1°C in a desiccator, at whose bottom place ammonium chloride saturated solution, for 24 hours, the moisture-absorption weight gain is 17.53%. So we place another quantity of the package-removed specimen at 25°C/RH 75%, sample at the 5th and the 10th day and examine, and compare the results with that of batch inspection.

2. Accelerated testing

Package: simulated commercial package

Testing condition: 40±2°C/75%±5% RH

Sampling frequency: 1st, 2nd, 3rd and 6th month

3. Long-term testing

Package: simulated commercial package

Testing condition: 25°C±2°C/60%±5% RH

Sampling frequency: 3rd, 6th, 9th, 12th, 18th, 24th and 36th month

Analysis procedure,

Refers to 3.2.S.4.2

Testing items,

Character, pH value, clarity degree and color of solution, water, assay, Betadex, microbial limit and bacterial endotoxin (0th, 6th, 12th, 24th and 36th month).

Conclusion

The results show that after 10days of stress testing, except the significantly changes of water because of the strong hygroscopicity of the product, other items have no change.

Upon the submission of DMF, accelerated testing and nine months of long term testing have been completed, and all the items have no significant change, so the commercial package of Betadex Sulfobutyl Ether Sodium is stable enough and safe enough to keep the product from influence of external environment during manufacture, transportation and storage.

3.2.S.7.2 Post approval Stability Protocol and Stability Commitment**Long-term testing protocol****1. Testing sample**

Batch No. 20140910, 20140921, 20140930

2. Package: simulated commercial package

3. Testing items: Character, pH value, clarity degree and color of solution, water, assay, Betadex, microbial limit and bacterial endotoxin (0th, 6th, 12th, 24th and 36th month).

4. Analytical procedures: Refer to 3.2.S.4.2 Analytical procedures**5. Testing condition:**

Long-term testing: 25°C±2°C/60%±5% RH;

6. Testing frequency:

Long-term testing: sample at the end of 12th, 18th, 24th and 36th month

Commitment

Upon the submitting of the DMF file stress testing and accelerated testing studies have been finished. Therefore, we, *Watson International Ltd* commit that we will perform the long term stability test according to the post-approval protocol described above and that we will supply the stability data when available.

3.2.S.7.3 Stability Data

Stress testing

Representative chromatograms refer to Annex 3-S-1~Annex 3-S-15.

Batch No.: 20140910

Item	Acceptance criteria	0 (day)	Photolysis testing		High temperature testing		High humidity testing	
			5	10	5	10	5	10
Character	White or off-white powder	Off-white powder	Off-white powder	Off-white powder	Off-white powder	Off-white powder	Gelatinous solid	Gelatinous solid
pH value	4.0~6.8	6.3	6.2	6.3	5.8	5.6	/	/
Clarity degree and color of solution	It should be clear and colorless, and should not be denser than No.2 yellow standard color solution	Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless	/	/
Water	≤10.0%	4.8%	9.3%	8.5%	4.3%	3.6%	/	/
Assay	95.0% ~ 105.0% on anhydrous basis	101.8%	102.5%	101.2%	102.5%	102.1%	/	/
Betadex (HPLC ^{Remark})	≤0.1%	<0.05%	<0.05%	<0.05%	<0.05%	<0.05%	<0.05%	<0.05%

Accelerated testing

Representative chromatograms refer to Annex 3-S-16~Annex 3-S-52, Annex 3-S-56~60, Annex 3-S-64~67, Annex 3-S-78~81 and Annex 3-S-85~87.

Batch No.: 20140910

Item	Acceptance criteria		Time (month)				
			0	1	2	3	6
Character	White or off-white powder		Off-white powder	Off-white powder	Off-white powder	Off-white powder	Off-white powder
pH value	4.0~6.8		6.3	6.4	6.5	6.4	6.1
Clarity degree and color of solution	It should be clear and colorless, and should not be denser than No.2 yellow standard color solution		Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless
Water	≤10.0%		4.8%	5.1%	4.7%	4.7%	5.2%
Assay	95.0%~105.0% on anhydrous basis		101.8%	101.9%	102.6%	101.3%	102.4%
Betadex	≤ 0.1%	HPLC ^{Remark}	<0.05%	<0.05%	<0.05%	<0.05%	<0.05%
		HPIC	/	/	/	<0.009%	<0.009%

Accelerated testing

Batch No.: 20140921

Item	Acceptance criteria		Time (month)				
			0	1	2	3	6
Character	White or off-white powder		Off-white powder	Off-white powder	Off-white powder	Off-white powder	Off-white powder
pH value	4.0~6.8		6.2	6.3	6.4	6.3	5.9
Clarity degree and color of solution	It should be clear and colorless, and should not be denser than No.2 yellow standard color solution		Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless
Water	≤10.0%		4.7%	5.4%	4.8%	5.0%	5.0%
Assay	95.0%~105.0% on anhydrous basis		101.3%	101.9%	102.2%	101.1%	102.6%
Betadex	≤ 0.1%	HPLC ^{Remark}	<0.05%	<0.05%	<0.05%	<0.05%	<0.05%
		HPIC	/	/	/	<0.009%	<0.009%

Accelerated testing

Batch No.: 20140930

Item	Acceptance criteria		Time (month)				
			0	1	2	3	6
Character	White or off-white powder		Off-white powder	Off-white powder	Off-white powder	Off-white powder	Off-white powder
pH value	4.0~6.8		6.2	6.3	6.4	6.4	6.0
Clarity degree and color of solution	It should be clear and colorless, and should not be denser than No.2 yellow standard color solution		Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless
Water	≤10.0%		4.1%	5.1%	4.8%	4.9%	5.1%
Assay	95.0%~105.0% on anhydrous basis		100.8%	101.7%	102.3%	101.4%	102.6%
Betadex	≤0.1%	HPLC ^{Remark}	<0.05%	<0.05%	<0.05%	<0.05%	<0.05%
		HPIC	/	/	/	<0.009%	<0.009%

Long-term testing

Representative chromatograms refer to Annex 3-S-53~55, Annex 3-S-61~63, Annex 3-S-68~70, Annex 3-S-82~84 and Annex 3-S-88~103.

Batch No.: 20140910

Item	Acceptance criteria		Time (month)			
			0	3	6	9
Character	White or off-white powder		Off-white powder	Off-white powder	Off-white powder	Off-white powder
pH value	4.0~6.8		6.3	6.2	6.2	6.4
Clarity degree and color of solution	It should be clear and colorless, and should not be denser than No.2 yellow standard color solution		Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless
Water	≤10.0%		4.8%	4.8%	5.2%	5.6%
Assay	95.0%~105.0% on anhydrous basis		101.8%	101.3%	101.7%	101.0%
Betadex	≤0.1%	HPLC ^{Remark}	<0.05%	<0.05%	<0.05%	<0.05%
		HPIC	<0.009%	<0.009%	<0.009%	<0.009%
Microbial limit	TAMC NMT100cfu/g		<10cfu/g	/	<10cfu/g	/
	TYMC NMT50cfu/g		<10cfu/g	/	<10cfu/g	/
	E.Coli should not be detected		Not detected	/	Not detected	/
Bacterial endotoxin	NMT0.02Eu/mg		<0.02Eu/mg	/	<0.02Eu/mg	/

Long-term testing

Batch No.: 20140921

Item	Acceptance criteria		Time (month)			
			0	3	6	9
Character	White or off-white powder		Off-white powder	Off-white powder	Off-white powder	Off-white powder
pH value	4.0~6.8		6.2	6.3	6.3	6.4
Clarity degree and color of solution	It should be clear and colorless, and should not be denser than No.2 yellow standard color solution		Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless
Water	$\leq 10.0\%$		4.7%	4.7%	5.5%	5.6%
Assay	95.0% ~ 105.0% on anhydrous basis		101.3%	101.1%	102.9%	102.2%
Betadex	$\leq 0.1\%$	HPLC ^{Remark}	<0.05%	<0.05%	<0.05%	<0.05%
		HPIC	<0.009%	<0.009%	<0.009%	<0.009%
Microbial limit	TAMC NMT100cfu/g		<10cfu/g	/	<10cfu/g	/
	TYMC NMT50cfu/g		<10cfu/g	/	<10cfu/g	/
	E.Coli should not be detected		Not detected	/	Not detected	/
Bacterial endotoxin	NMT0.02Eu/mg		<0.02Eu/mg	/	<0.02Eu/mg	/

Long-term testing

Batch No.: 20140930

Item	Acceptance criteria	Time (month)			
		0	3	6	9
Character	White or off-white powder	Off-white powder	Off-white powder	Off-white powder	Off-white powder
pH value	4.0~6.8	6.2	6.3	6.1	6.4
Clarity degree and color of solution	It should be clear and colorless, and should not be denser than No.2 yellow standard color solution	Clear and colorless	Clear and colorless	Clear and colorless	Clear and colorless
Water	≤10.0%	4.1%	4.9%	5.1%	5.6%
Assay	95.0%~105.0% on anhydrous basis	100.8%	101.2%	102.7%	100.8%
Betadex	≤0.1%	HPLC ^{Remark}	<0.05%	<0.05%	<0.05%
		HPIC	<0.009%	<0.009%	<0.009%
Microbial limit	TAMC NMT100cfu/g	<10cfu/g	/	<10cfu/g	/
	TYMC NMT50cfu/g	<10cfu/g	/	<10cfu/g	/
	E.Coli should not be detected	Not detected	/	Not detected	/
Bacterial endotoxin	NMT0.02Eu/mg	<0.02Eu/mg	/	<0.02Eu/mg	/

Remark,

HPLC is the method used to control Betadex in the final product at the earlier stage, but because HPIC is the method in the latest specification in USP and, it has a lower LOD (0.009% of the concentration of sample) than that of HPLC (0.05% of the concentration of sample), we choose HPIC to control Betadex at later period.

In the study of stability, stress testing, 1th, 2th, 3th and 6th of accelerated testing, 3th and 6th of long-term testing are all inspected by HPLC and qualified. At July, 2015, we detected the samples of 3th and 6th of accelerated testing, 3th and 6th of long-term testing by HPIC and all the samples qualified. The samples of follow-up stability study are inspected by HPIC.

HPLC procedures is as below,

Mode: HPLC

Column: packed with octadecyl silicane boned silica gel (Waters Atlantis T3, 4.6×150mm, 3.0μm)

Mobile phase: methanol-water (10-90)

Flow rate: 1.0ml/min

Column temperature: 35℃

Detector: differential refraction detector

Detector temperature: 40°C

Injection size: 10µl

Preparation,

Sample solution: accurately transfer 200mg of the sample into 10ml volumetric flask, add water to dissolve and dilute to the mark, mix well.

Reference solution: to a 100ml volumetric flask add 20mg of reference standard which is accurately weighed, add water to dissolve and dilute to the mark and mix well, accurately pipet 10.0ml of the solution above in to a 100ml volumetric flask, add water to dissolve and dilute to the mark, mix well.

Analyses,

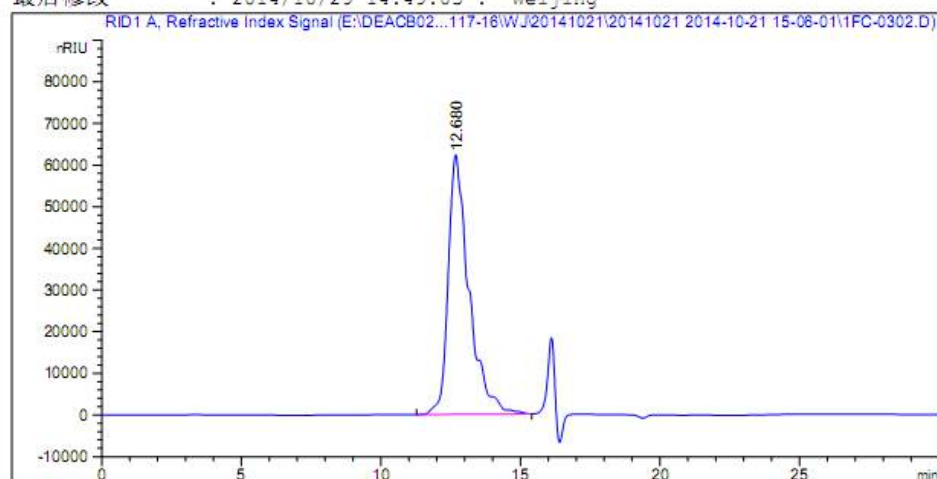
Perform injections with the solutions obtained and record chromatograms, calculate the assay of Betadex by external standard method.

Annex 3-S-1 Stress testing-Assay-5 days-Reference solution 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FC-0302.D
样品名称: DZ1

```
=====
操作者       : Weijing                      序列行   :    3
仪器         : 1260-2                      位置     : P1-F-03
进样日期     : 2014/10/21 16:38:17          进样次数  :    2
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:49:03 : Weijing
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.680	BB	0.7167	3.22867e6	6.23238e4	100.0000

总量 : 3.22867e6 6.23238e4

=====
*** 报告结束 ***

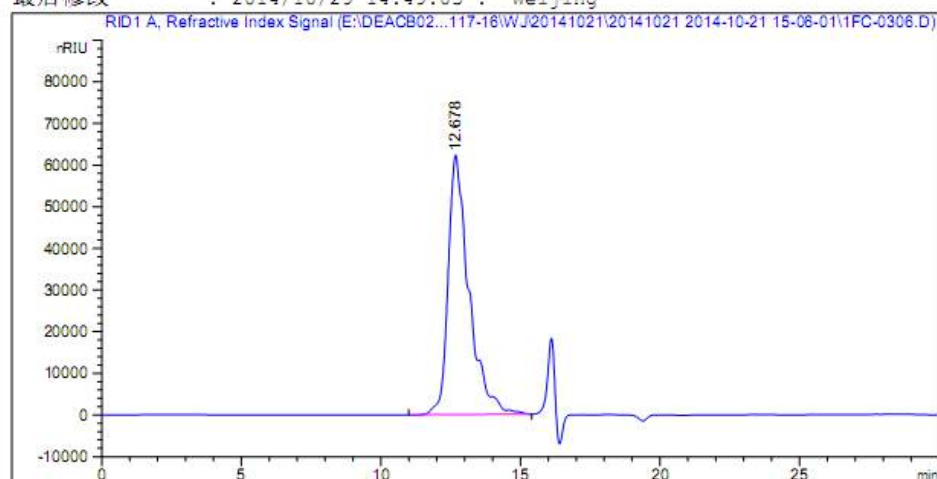
附图14-3-43 SBECD影响因素5天含量测定图 (对照1-2)

Annex 3-S-2 Stress testing-Assay-5 days-Reference solution 1-6

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FC-0306.D
样品名称: DZ1

```
=====
操作者       : Weijing                      序列行   :    3
仪器         : 1260-2                      位置     : P1-F-03
进样日期     : 2014/10/21 18:40:34          进样次数  :    6
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:49:03 : Weijing
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.678	BB	0.7172	3.22775e6	6.22509e4	100.0000

总量 : 3.22775e6 6.22509e4

=====
*** 报告结束 ***

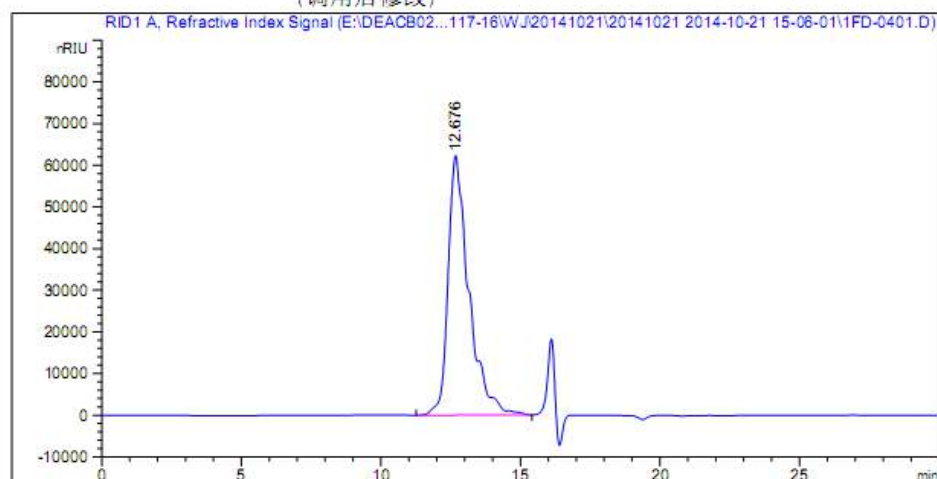
附图14-3-47 SBECD影响因素5天含量测定图 (对照1-6)

Annex 3-S-3 Stress testing-Assay-5 days-Reference solution 2-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\1FD-0401.D
样品名称: DZ2

```
=====
操作者       : Weijing                      序列行   :    4
仪器         : 1260-2                      位置     : Pl-F-04
进样日期     : 2014/10/21 19:11:09          进样次数  :    1
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/21 16:02:21 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:32:22 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序          :    信号
乘积因子      :    1.0000
稀释因子      :    1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.676	BB	0.7169	3.22571e6	6.22435e4	100.0000

总量 : 3.22571e6 6.22435e4

*** 报告结束 ***

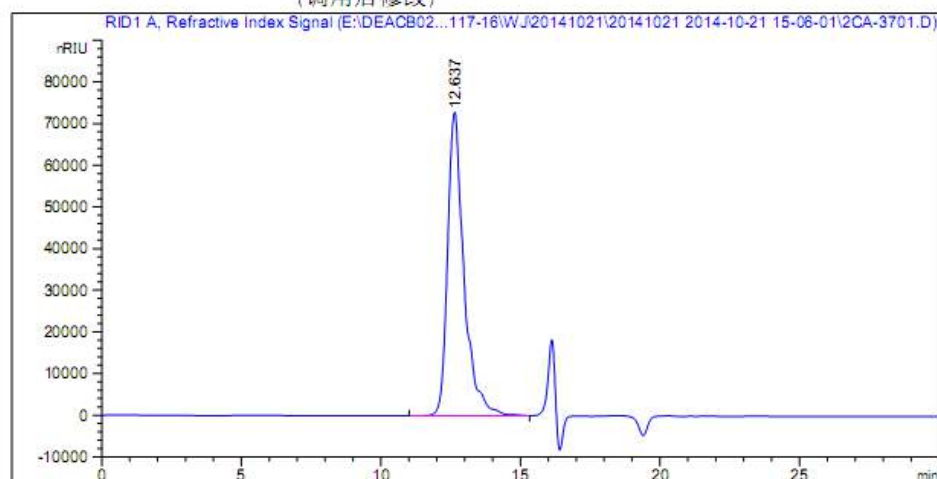
附图14-3-48 SBECD影响因素5天含量测定图 (对照2-1)

Annex 3-S-4 Stress testing-Assay-5 days- Photolysis testing 1-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2CA-3701.D
样品名称: 20140910光照5天-1

```
=====
操作者       : Weijing                      序列行 : 37
仪器         : 1260-2                      位置   : P2-C-01
进样日期     : 2014/10/22 19:07:58          进样次数 : 1
                                           进样量  : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/22 16:50:55 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:38:18 : Weijing
               (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.637	BBA	0.5896	2.97395e6	7.26999e4	100.0000

总量 : 2.97395e6 7.26999e4

*** 报告结束 ***

附图14-3-51 SBEC影响因素5天含量测定图 (20140910-光照-1-1)

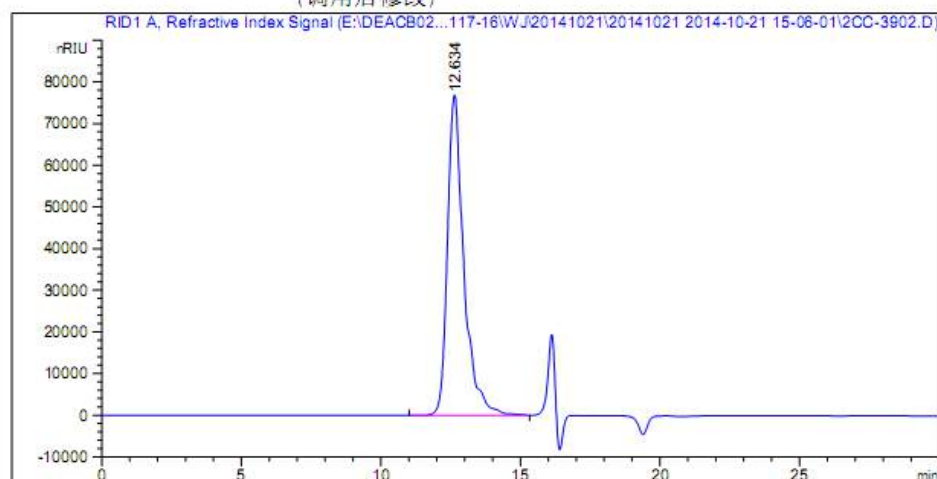
Annex 3-S-5 Stress testing-Assay-5 days- High temperature testing 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\2CC-3902.D

样品名称: 20140910-60度5天-1

```
=====
操作者       : Weijing                      序列行   : 39
仪器         : 1260-2                      位置     : P2-C-03
进样日期     : 2014/10/22 21:40:50          进样次数  : 2
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/22 16:50:55 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141021\20141021 2014-10-21 15-06-01\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 14:38:18 : Weijing
              (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.634	BBA	0.5986	3.13979e6	7.66905e4	100.0000

总量 : 3.13979e6 7.66905e4

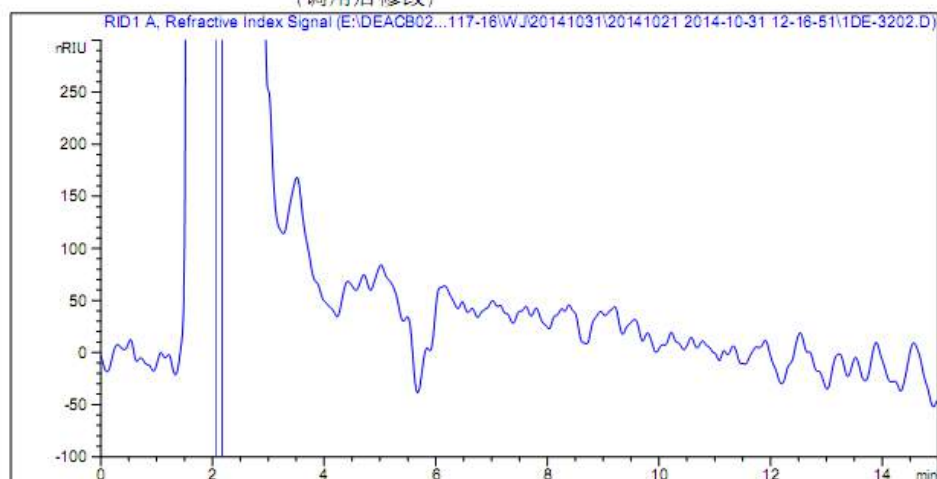
*** 报告结束 ***

附图14-3-56 SBECD影响因素5天含量测定图 (20140910-高温-1-2)

Annex 3-S-6 Stress testing-Betadex-5 days- Reference solution 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\1DE-3202.D
样品名称: 20140910-光照5天-1

```
=====
操作者       : Weijing                      序列行 : 32
仪器         : 1260-2                      位置   : P1-D-05
进样日期     : 2014/11/1 4:01:30           进样次数 : 2
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
最后修改     : 2014/10/31 15:56:20 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
               (序列方法)
最后修改     : 2015/3/31 18:24:39 : linping
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
                        *** 报告结束 ***
=====
```

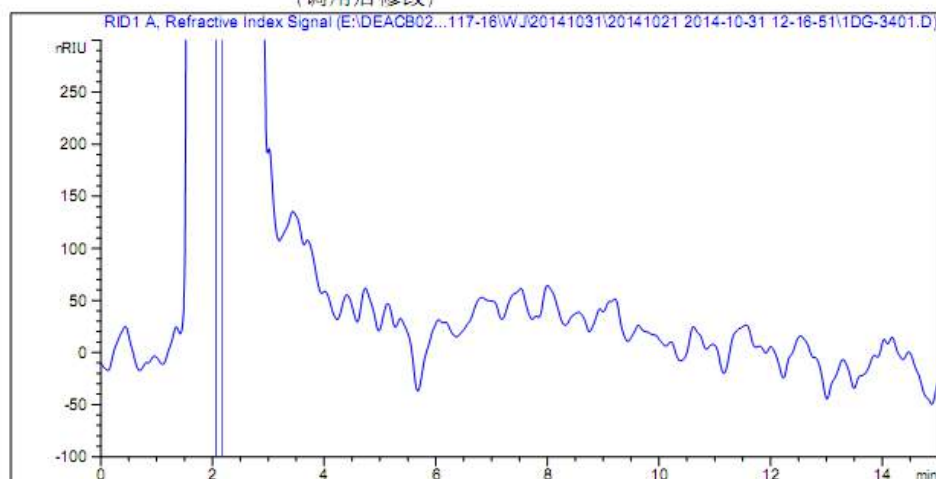
附图14-3-60 SBED影响因素5天倍他环糊精测定图 (20140910-光照-1-2)

Annex 3-S-7 Stress testing-Betadex-5 days- High temperature testing 1-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\1DG-3401.D

样品名称: 20140910-60度5天-1

```
=====
操作者       : Weijing                      序列行 : 34
仪器         : 1260-2                      位置   : Pl-D-07
进样日期     : 2014/11/1 5:18:53           进样次数 : 1
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
最后修改     : 2014/10/31 15:56:20 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
               (序列方法)
最后修改     : 2015/3/31 18:24:39 : linping
               (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

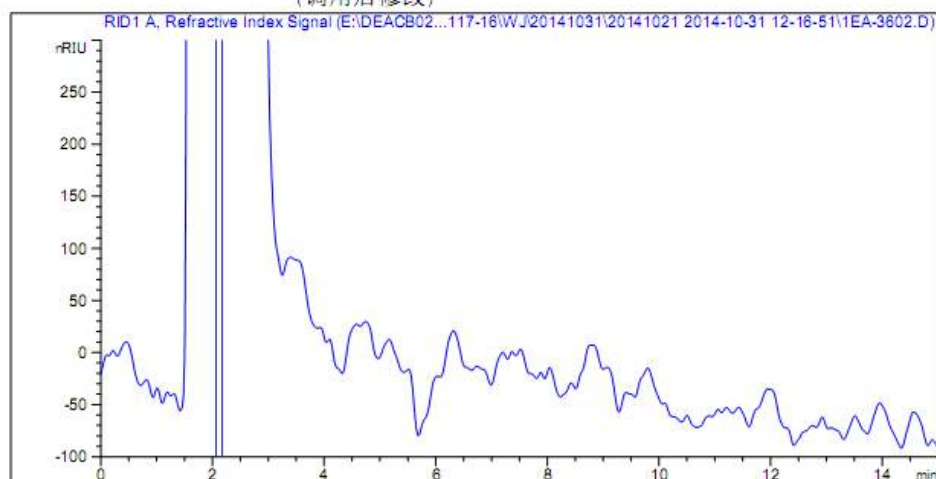
附图14-3-63 SBED影响因素5天倍他环糊精测定图 (20140910-高温-1-1)

Annex 3-S-8 Stress testing-Betadex-5 days- High humidity testing 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\1EA-3602.D

样品名称: 20140910-75%5天-1

```
=====
操作者       : Weijing                      序列行 : 36
仪器         : 1260-2                      位置   : Pl-E-01
进样日期     : 2014/11/1 7:07:13           进样次数 : 2
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
最后修改     : 2014/10/31 15:56:20 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
               (序列方法)
最后修改     : 2015/3/31 18:24:39 : linping
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
                        *** 报告结束 ***
=====
```

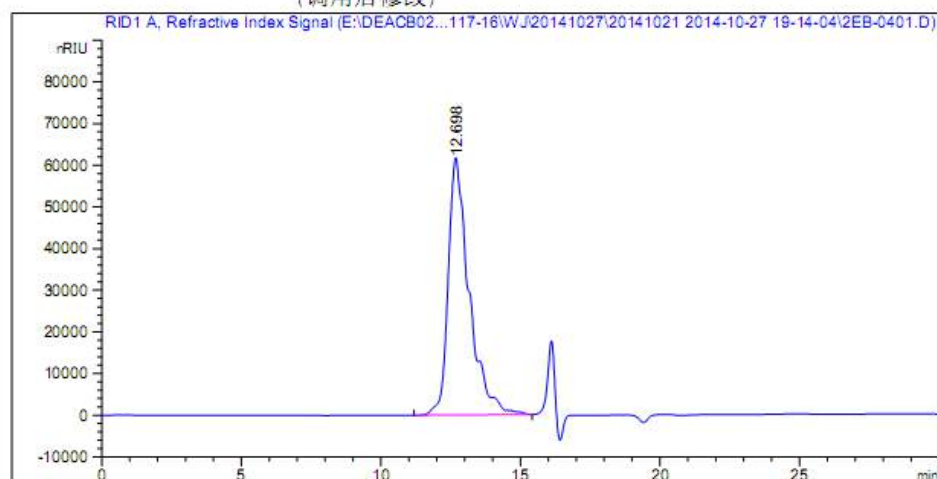
附图14-3-68 SBED影响因素5天倍他环糊精测定图(20140910-高湿-1-2)

Annex 3-S-9 Stress testing-Assay-10 days- Reference solution 1-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\2EB-0401.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   :    4
仪器         : 1260-2                      位置     : P2-E-02
进样日期     : 2014/10/27 22:18:08          进样次数  :    1
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/27 19:14:04 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/12/19 9:42:27
               (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.698	-	3.21574e6	6.14276e4	0.56	0.6938	1850	-	-

=====
*** 报告结束 ***

附图14-3-72 SBECD影响因素10天含量测定图 (对照1-1)

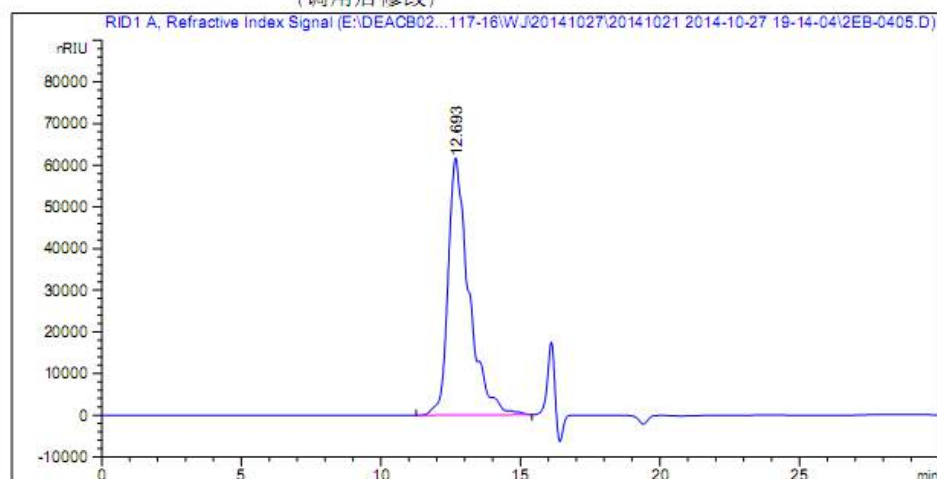
Annex 3-S-10 Stress testing-Assay-10 days- Reference solution 1-5

数据文件: E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\2EB-0405.D

样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   :    4
仪器         : 1260-2                      位置     : P2-E-02
进样日期     : 2014/10/28 0:20:26          进样次数  :    5
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/27 19:14:04 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:35:25 : Weijing
              (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.693	BB	0.7849	3.21472e6	6.12762e4	100.0000

总量 : 3.21472e6 6.12762e4

*** 报告结束 ***

附图14-3-76 SBECD影响因素10天含量测定图 (对照1-5)

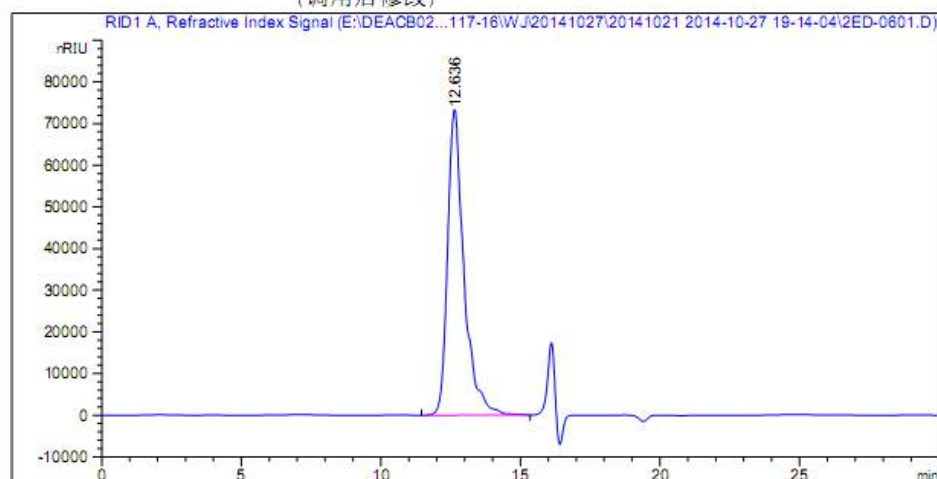
Annex 3-S-11 Stress testing-Assay-10 days- Photolysis testing 1-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\2ED-0601.D

样品名称: 20140910光照10天-1

```
=====
操作者       : Weijing                      序列行 :    6
仪器         : 1260-2                      位置   : P2-E-04
进样日期     : 2014/10/28 2:53:18          进样次数:    1
                                           进样量  : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
               SBECD-0.6.M
最后修改     : 2014/10/27 19:14:04 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:35:25 : Weijing
               (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.636	BB	0.6163	2.99805e6	7.31684e4	100.0000

总量 : 2.99805e6 7.31684e4

*** 报告结束 ***

附图14-3-81 SBECD影响因素10天含量测定图 (20140910-光照-1-1)

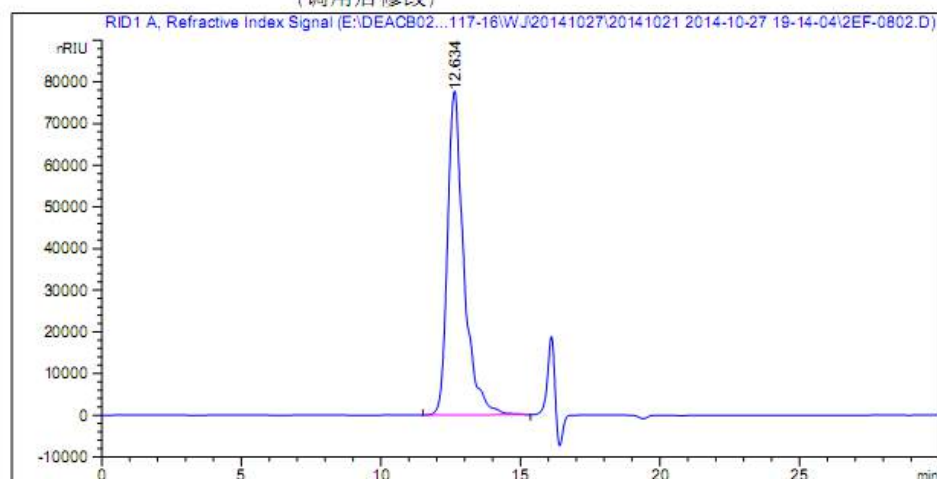
Annex 3-S-12 Stress testing-Assay-10 days- High temperature testing 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\2EF-0802.D

样品名称: 20140910-60度10天-1

```
=====
操作者       : Weijing                      序列行   :    8
仪器         : 1260-2                      位置     : P2-E-06
进样日期     : 2014/10/28 5:26:10          进样次数  :    2
                                           进样量   : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
              SBECD-0.6.M
最后修改     : 2014/10/27 19:14:04 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141027\20141021 2014-10-27 19-14-04\117-16-
              SBECD-0.6.M (序列方法)
最后修改     : 2014/10/29 15:35:25 : Weijing
              (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.634	BB	0.6163	3.18045e6	7.76084e4	100.0000

总量 : 3.18045e6 7.76084e4

*** 报告结束 ***

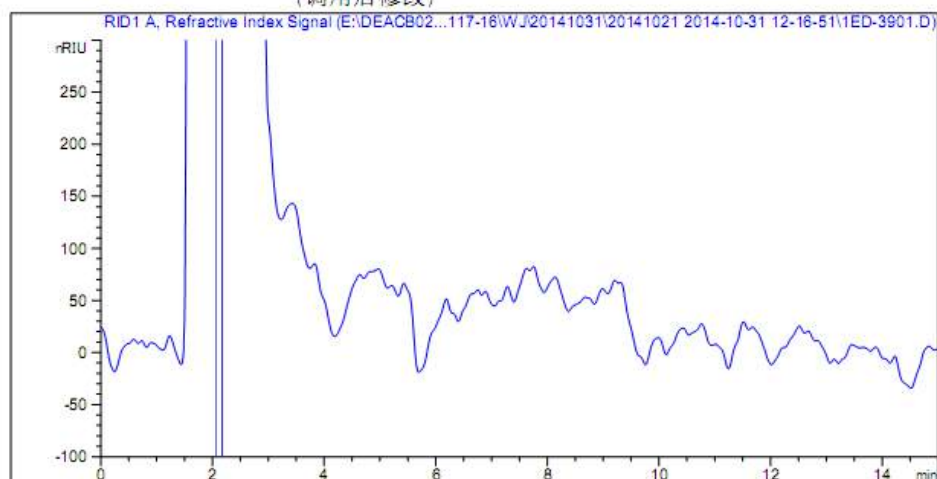
附图14-3-86 SBECD影响因素10天含量测定图 (20140910-高温-1-2)

Annex 3-S-13 Stress testing-Betadex-10 days- Photolysis testing 2-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\1ED-3901.D

样品名称: 20140910-光照10天-2

```
=====
操作者       : Weijing                      序列行 : 39
仪器         : 1260-2                      位置   : Pl-E-04
进样日期     : 2014/11/1 9:11:02           进样次数 : 1
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
最后修改     : 2014/10/31 15:56:20 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
               (序列方法)
最后修改     : 2015/3/31 18:24:39 : linping
               (调用后修改)
=====
```



面积百分比报告

```
=====
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

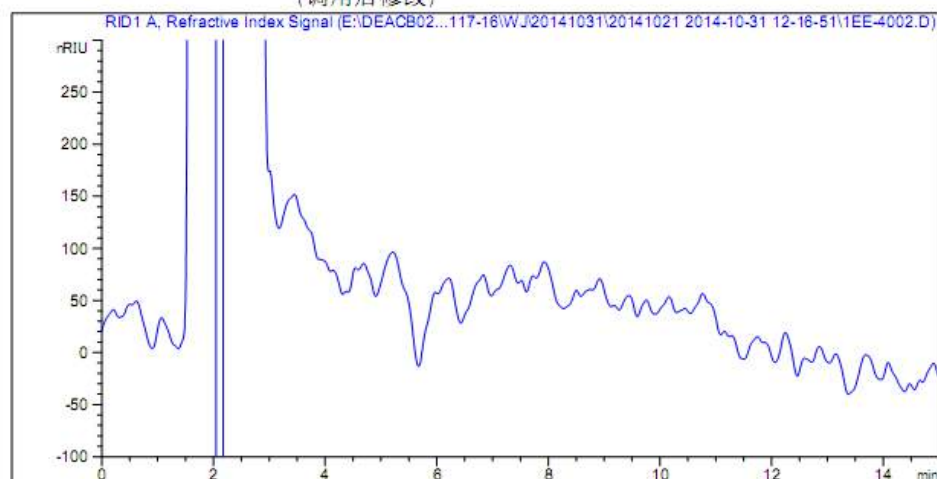
附图14-3-91 SBECD影响因素10天倍他环糊精测定图 (20140910-光照-2-1)

Annex 3-S-14 Stress testing-Betadex-10 days- High temperature 1-2

数据文件: E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\1EE-4002.D

样品名称: 20140910-60度10天-1

```
=====
操作者       : Weijing                      序列行 : 40
仪器         : 1260-2                      位置   : P1-E-05
进样日期     : 2014/11/1 10:12:55          进样次数 : 2
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
最后修改     : 2014/10/31 15:56:20 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
               (序列方法)
最后修改     : 2015/3/31 18:24:39 : linping
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
                        *** 报告结束 ***
=====
```

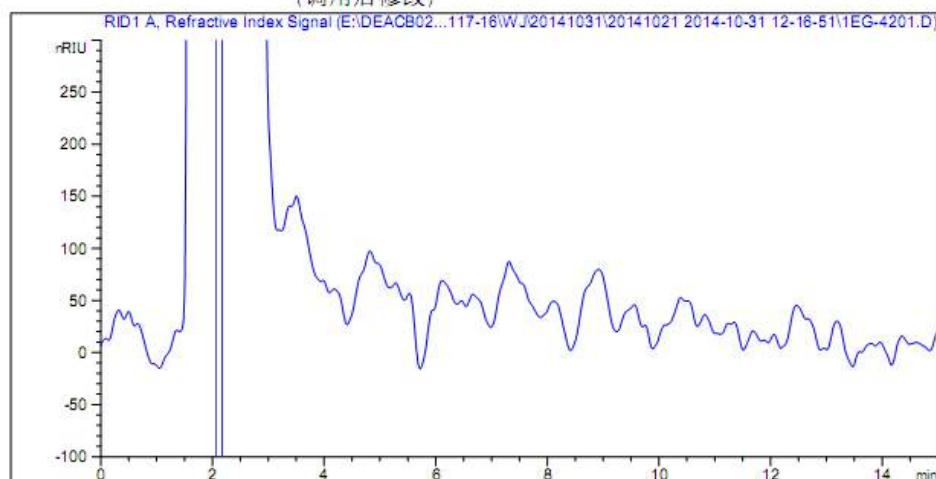
附图14-3-94 SBECD影响因素10天倍他环糊精测定图 (20140910-高温-1-2)

Annex 3-S-15 Stress testing-Betadex-10 days- High humidity 1-1

数据文件: E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\1EG-4201.D

样品名称: 20140910-75%10天-1

```
=====
操作者       : Weijing                      序列行 : 42
仪器         : 1260-2                      位置   : Pl-E-07
进样日期     : 2014/11/1 11:30:18          进样次数 : 1
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
最后修改     : 2014/10/31 15:56:20 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\WJ\20141031\20141021 2014-10-31 12-16-51\B CD T3.M
               (序列方法)
最后修改     : 2015/3/31 18:24:39 : linping
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

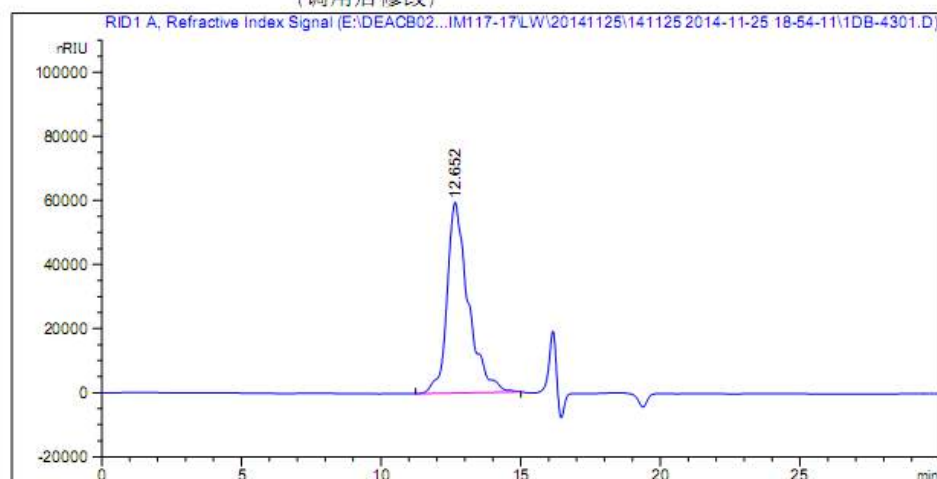
```
=====
                        *** 报告结束 ***
=====
```

附图14-3-97 SBECD影响因素10天倍他环糊精测定图 (20140910-高湿-1-1)

Annex 3- S-16 Accelerated testing-Assay-1month-Reference solution1-1

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1DB-4301.D
样品名称: D21

```
=====
操作者       : Weijing                      序列行   : 43
仪器         : 1260-2                      位置     : Pl-D-02
进样日期     : 2014/11/26 22:26:05         进样次数  : 1
                                           进样量   : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2015/3/31 11:36:28 : linping
               (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.652	-	3.14343e6	5.95157e4	0.59	0.6999	1810	-	-

=====
*** 报告结束 ***

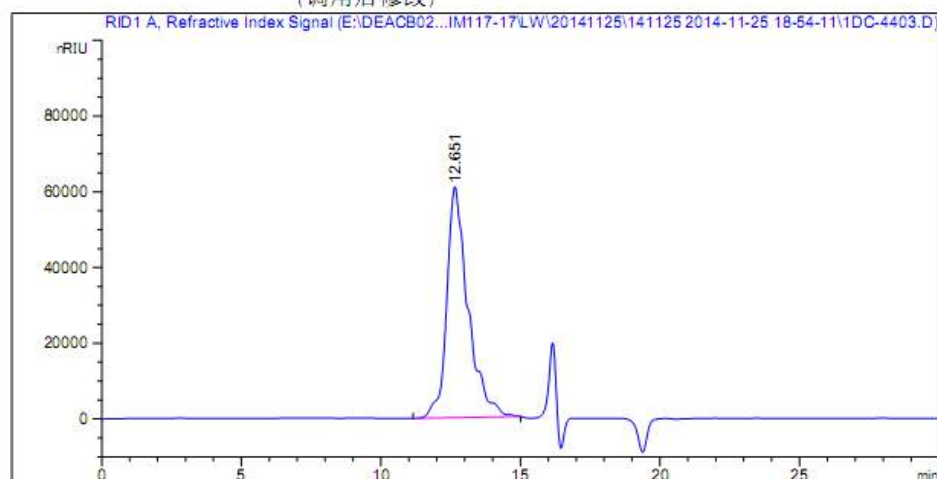
附图14-4-2 SBECD加速1月含量测定图 (对照1-1)

Annex 3- S-17 Accelerated testing-Assay-1month-Reference solution2-3

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1DC-4403.D
样品名称: DZ2

```
=====
操作者       : Weijing                      序列行 : 44
仪器         : 1260-2                      位置   : Pl-D-03
进样日期     : 2014/11/27 0:59:05          进样次数 : 3
                                           进样量  : 20.000 µl

采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/11/27 15:49:26 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.651	BBA	0.7298	3.22902e6	6.10070e4	100.0000

总量 : 3.22902e6 6.10070e4

```
=====
*** 报告结束 ***
=====
```

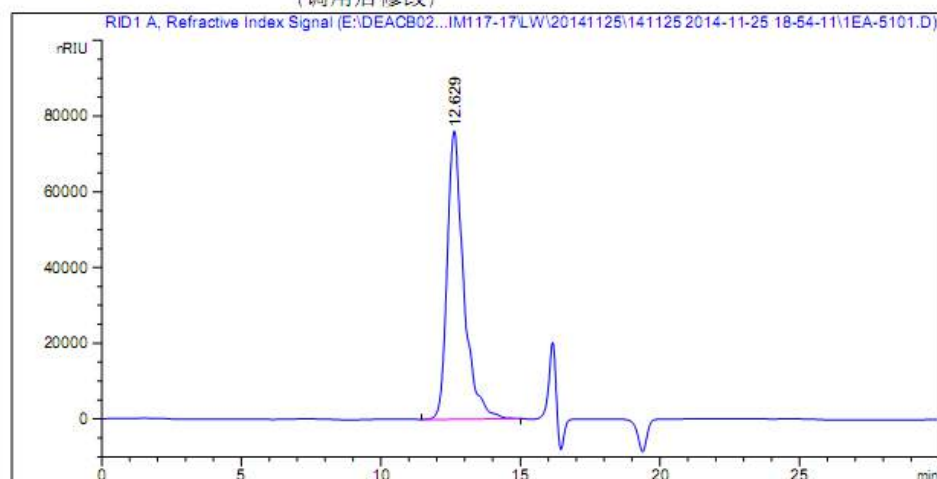
附图14-4-7 SBECD加速1月含量测定图 (对照2-3)

Annex 3- S-18 Accelerated testing-Assay-1month-20140910-1-1

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1EA-5101.D

样品名称: 20140910-40C-75%RH-1M-1

```
=====
操作者       : Weijing                      序列行 : 51
仪器         : 1260-2                      位置   : Pl-E-01
进样日期     : 2014/11/27 7:36:51          进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/11/27 15:49:26 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.629	BBA	0.5922	3.13195e6	7.61503e4	100.0000

```
总量 :                3.13195e6  7.61503e4
```

```
=====
*** 报告结束 ***
=====
```

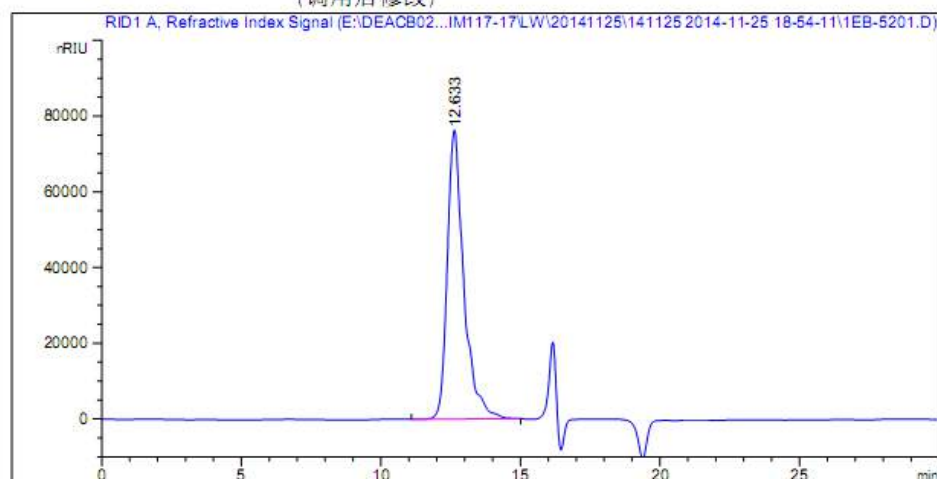
附图14-4-8 SBECD加速1月含量测定图 (20140910-1-1)

Annex 3- S-19 Accelerated testing-Assay-1month-20140910-2-1

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1EB-5201.D

样品名称: 20140910-40C-75%RH-1M-2

```
=====
操作者       : Weijing                      序列行 : 52
仪器         : 1260-2                      位置   : Pl-E-02
进样日期     : 2014/11/27 8:38:02          进样次数 : 1
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/11/27 15:49:26 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.633	BBA	0.5887	3.14516e6	7.63366e4	100.0000

总量 : 3.14516e6 7.63366e4

```
=====
*** 报告结束 ***
=====
```

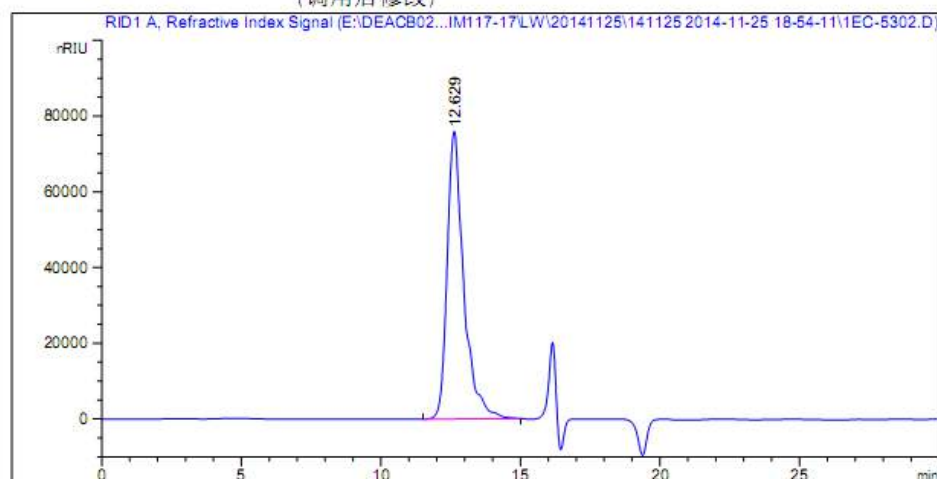
附图14-4-10 SBECD加速1月含量测定图 (20140910-2-1)

Annex 3- S-20 Accelerated testing-Assay-1month-20140921-1-2

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1EC-5302.D

样品名称: 20140921-40C-75%RH-1M-1

```
=====
操作者       : Weijing                      序列行 : 53
仪器         : 1260-2                      位置   : Pl-E-03
进样日期     : 2014/11/27 10:09:49          进样次数 : 2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/11/27 15:49:26 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.629	BBA	0.5941	3.13829e6	7.60034e4	100.0000

总量 : 3.13829e6 7.60034e4

```
=====
*** 报告结束 ***
=====
```

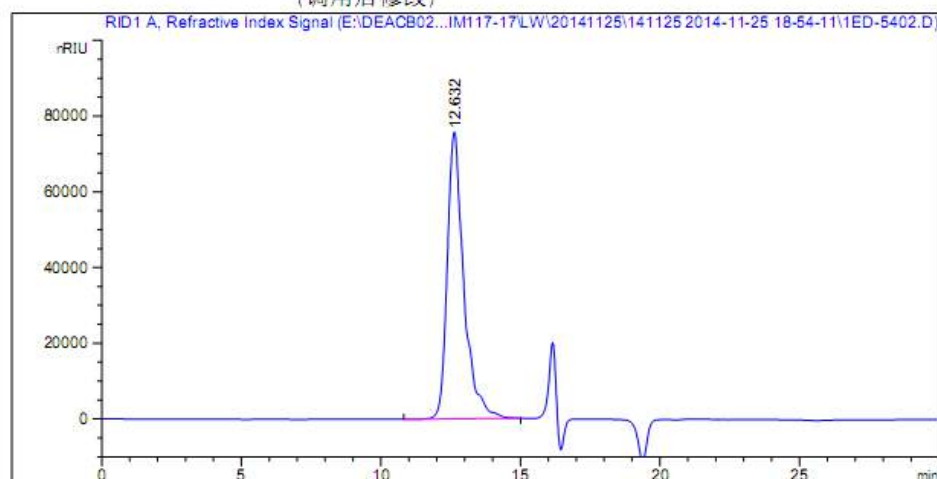
附图14-4-13 SBECD加速1月含量测定图 (20140921-1-2)

Annex 3- S-21 Accelerated testing-Assay-1month-20140921-2-2

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1ED-5402.D

样品名称: 20140921-40C-75%RH-1M-2

```
=====
操作者       : Weijing                      序列行 : 54
仪器         : 1260-2                      位置   : Pl-E-04
进样日期     : 2014/11/27 11:11:01         进样次数 : 2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/11/27 15:49:26 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.632	BBA	0.5925	3.11737e6	7.57368e4	100.0000

```
总量 :                3.11737e6  7.57368e4
```

```
=====
*** 报告结束 ***
=====
```

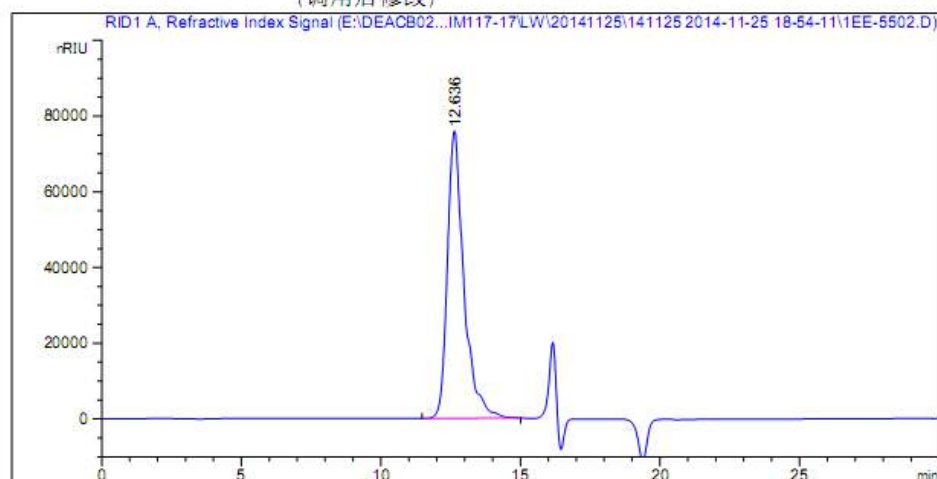
附图14-4-15 SBECD加速1月含量测定图 (20140921-2-2)

Annex 3- S-22 Accelerated testing-Assay-1month-20140930-1-2

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1EE-5502.D

样品名称: 20140930-40C-75%RH-1M-1

```
=====
操作者       : Weijing                      序列行 : 55
仪器         : 1260-2                      位置   : Pl-E-05
进样日期     : 2014/11/27 12:12:13         进样次数 : 2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/11/27 15:49:26 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.636	BBA	0.5877	3.12066e6	7.58893e4	100.0000

```
总量 :                3.12066e6  7.58893e4
```

```
=====
*** 报告结束 ***
=====
```

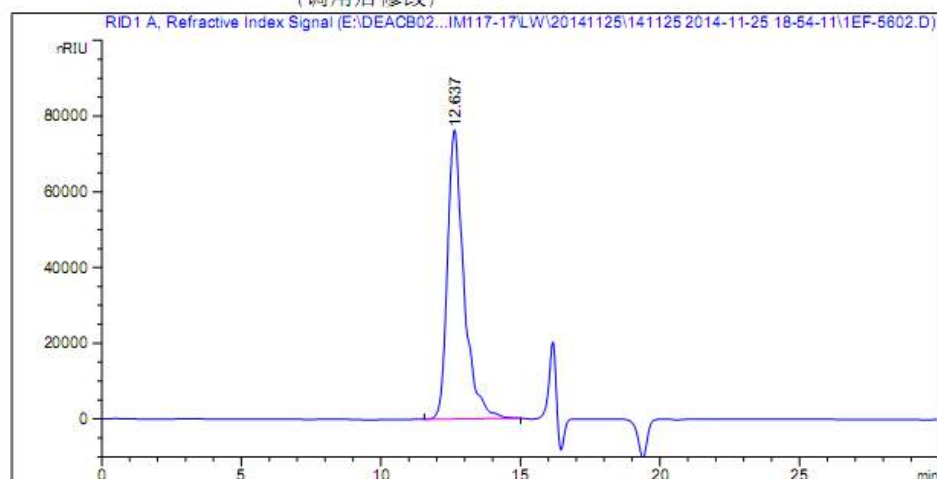
附图14-4-17 SBECD加速1月含量测定图 (20140930-1-2)

Annex 3- S-23 Accelerated testing-Assay-1month-20140930-2-2

数据文件: E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\1EF-5602.D

样品名称: 20140921-40C-75%RH-1M-2

```
=====
操作者       : Weijing                      序列行 : 56
仪器         : 1260-2                      位置   : Pl-E-06
进样日期     : 2014/11/27 13:13:25          进样次数 : 2
                                           进样量  : 20.000 µl
采集方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M
最后修改     : 2014/11/25 20:02:42 : Weijing
分析方法     : E:\DEACB02694\SIM117-17\LW\20141125\141125 2014-11-25 18-54-11\117-16-
               SBECD-0.6.M (序列方法)
最后修改     : 2014/11/27 15:49:26 : Weijing
               (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.637	BBA	0.5870	3.13366e6	7.63268e4	100.0000

```
总量 :                3.13366e6  7.63268e4
```

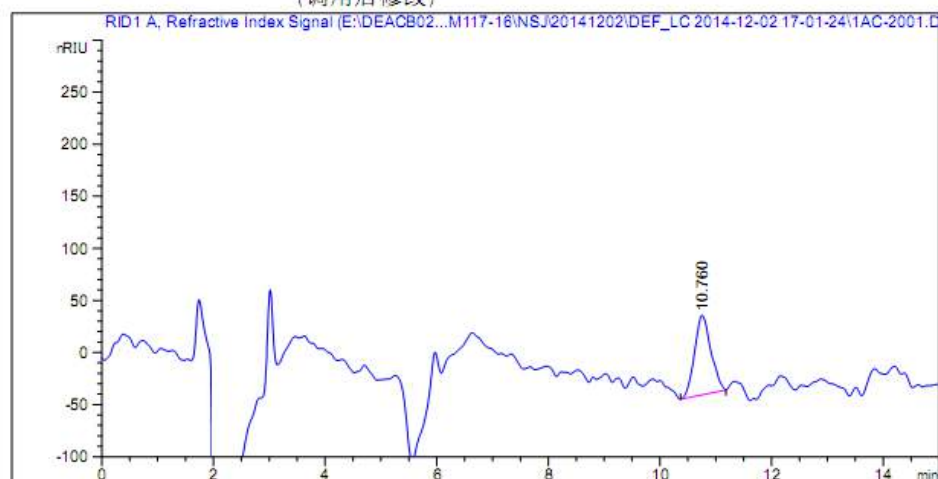
```
=====
*** 报告结束 ***
=====
```

附图14-4-19 SBECD加速1月含量测定图 (20140930-2-2)

Annex 3- S-24 Accelerated testing-Betadex-1month-Reference solution 1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1AC-2001.D
样品名称: DZ-1

```
=====
操作者       : Weijing                      序列行   : 20
仪器         : 1260-2                      位置     : Pl-A-03
进样日期     : 2014/12/2 22:11:39          进样次数  : 1
                                           进样量   : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2015/3/31 18:34:27 : linping
                                           (调用后修改)
=====
```



```
=====
                        面积百分比报告 (包含性能计算)
=====
```

```
乘积因子       : 1.0000
稀释因子       : 1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
10.760	-	1551.72498	76.15935	0.88	0.3096	6688	-	-

```
=====
*** 报告结束 ***
=====
```

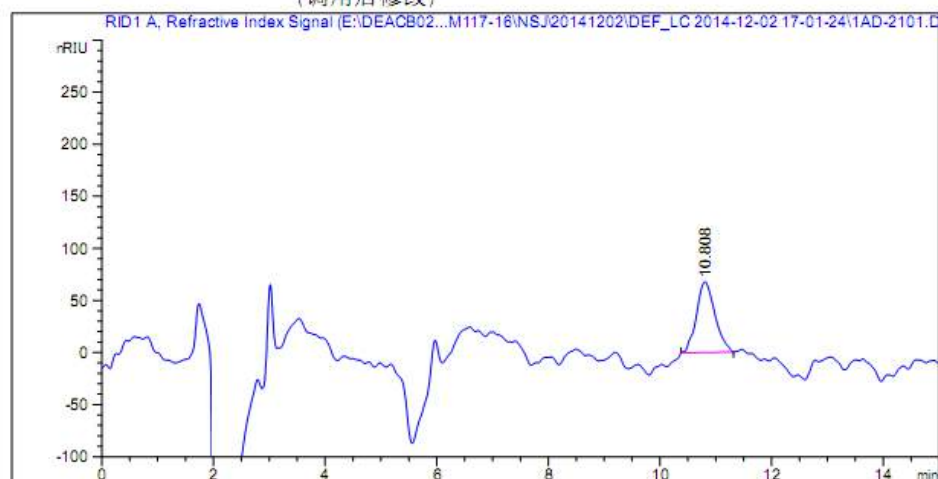
附图14-4-21 SBECD加速1月倍他环糊精测定图 (对照1-1)

Annex 3- S-25 Accelerated testing-Betadex-1month-Reference solution 2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1AD-2101.D

样品名称: DZ-2

```
=====
操作者       : Weijing                      序列行 : 21
仪器         : 1260-2                      位置   : Pl-A-04
进样日期     : 2014/12/2 23:29:13          进样次数 : 1
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2014/12/18 11:00:01
                                           (调用后修改)
=====
```



面积百分比报告

```
=====
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	10.808	BBA	0.3558	1564.79285	67.77654	100.0000

总量 : 1564.79285 67.77654

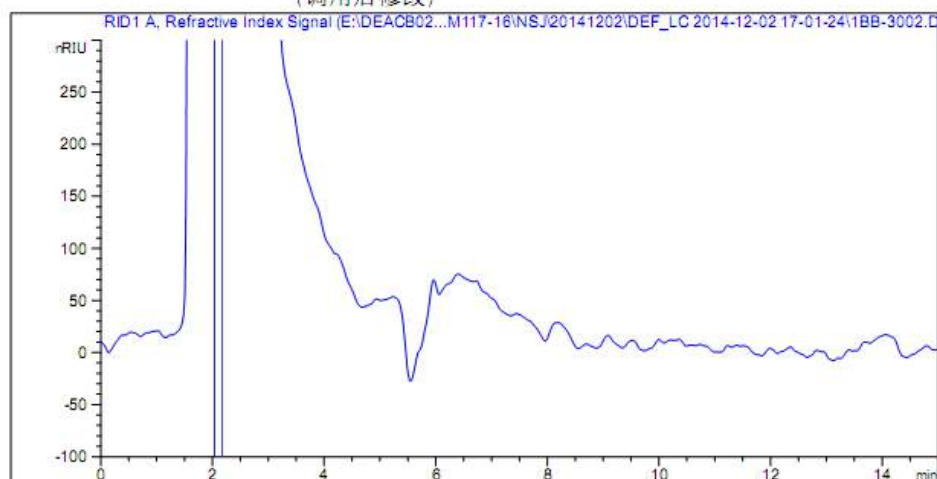
*** 报告结束 ***

附图14-4-24 SBECD加速1月倍他环糊精测定图 (对照2-1)

Annex 3- S-26 Accelerated testing-Betadex-1month-20140910-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1BB-3002.D
样品名称: 20140910-40C-1M-1

```
=====
操作者       : Weijing                      序列行   : 30
仪器         : 1260-2                      位置     : Pl-B-02
进样日期     : 2014/12/3 5:41:19           进样次数  : 2
                                           进样量   : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2014/12/18 11:00:01
                                           (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

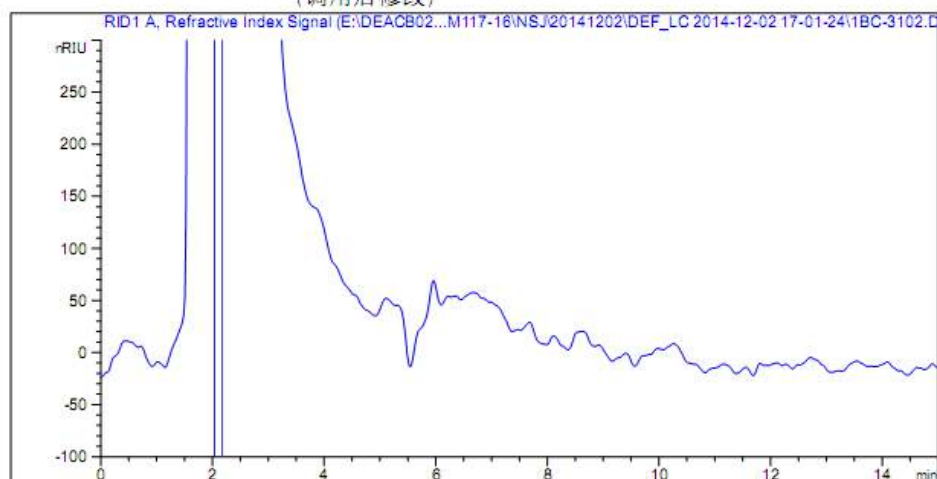
```
=====
                        *** 报告结束 ***
=====
```

附图14-4-29 SBECD加速1月倍他环糊精测定图 (20140910-1-2)

Annex 3- S-27 Accelerated testing-Betadex-1month-20140910-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1BC-3102.D
样品名称: 20140910-40C-1M-2

```
=====
操作者       : Weijing                      序列行   : 31
仪器         : 1260-2                      位置     : Pl-B-03
进样日期     : 2014/12/3 6:12:18           进样次数  : 2
                                           进样量   : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2014/12/18 11:00:01
                                           (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

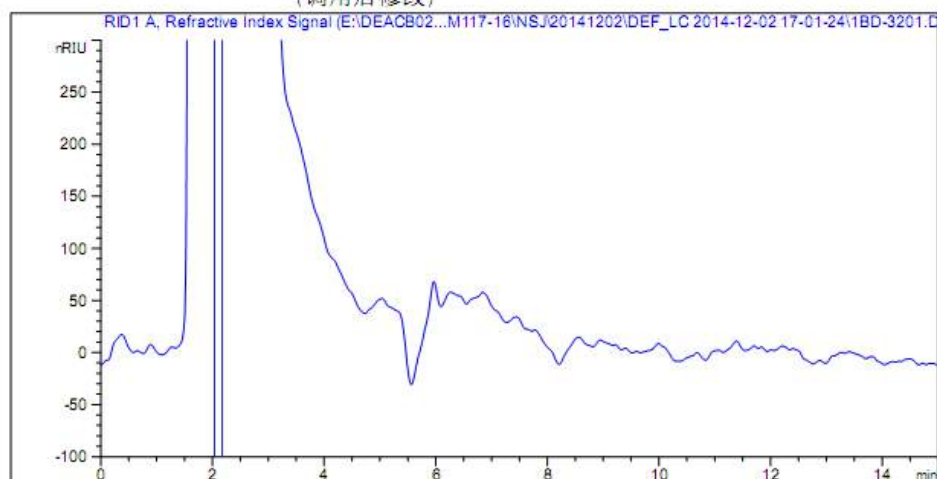
```
=====
                        *** 报告结束 ***
=====
```

附图14-4-31 SBECD加速1月倍他环糊精测定图 (20140910-2-2)

Annex 3- S-28 Accelerated testing-Betadex-1month-20140921-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1BD-3201.D
样品名称: 20140921-40C-1M-1

```
=====
操作者       : Weijing                      序列行 : 32
仪器         : 1260-2                      位置   : Pl-B-04
进样日期     : 2014/12/3 6:27:48           进样次数 : 1
                                           进样量 : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2014/12/18 11:00:01
                                           (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

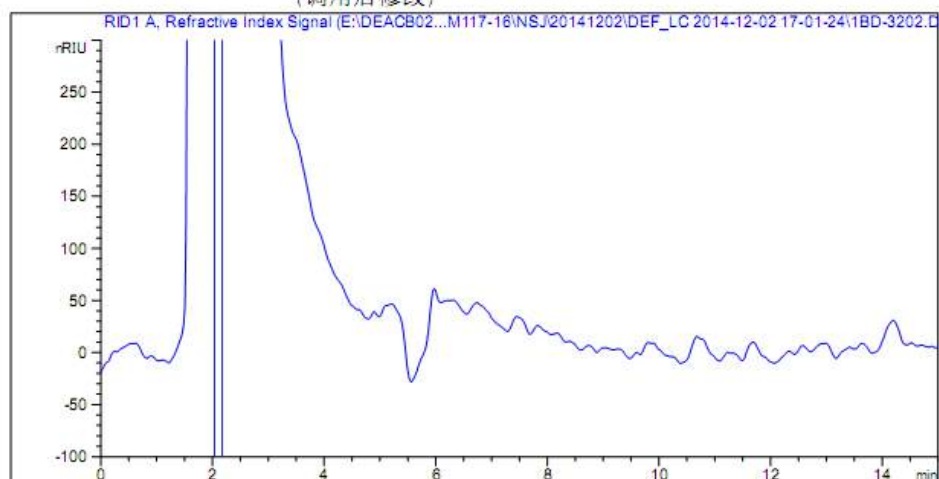
```
=====
                        *** 报告结束 ***
=====
```

附图14-4-32 SBECD加速1月倍他环糊精测定图 (20140921-1-1)

Annex 3- S-29 Accelerated testing-Betadex-1month-20140921-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1BD-3202.D
样品名称: 20140921-40C-1M-1

```
=====
操作者       : Weijing                      序列行   : 32
仪器         : 1260-2                      位置     : Pl-B-04
进样日期     : 2014/12/3 6:43:18           进样次数  : 2
                                           进样量   : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2014/12/18 11:00:01
                                           (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

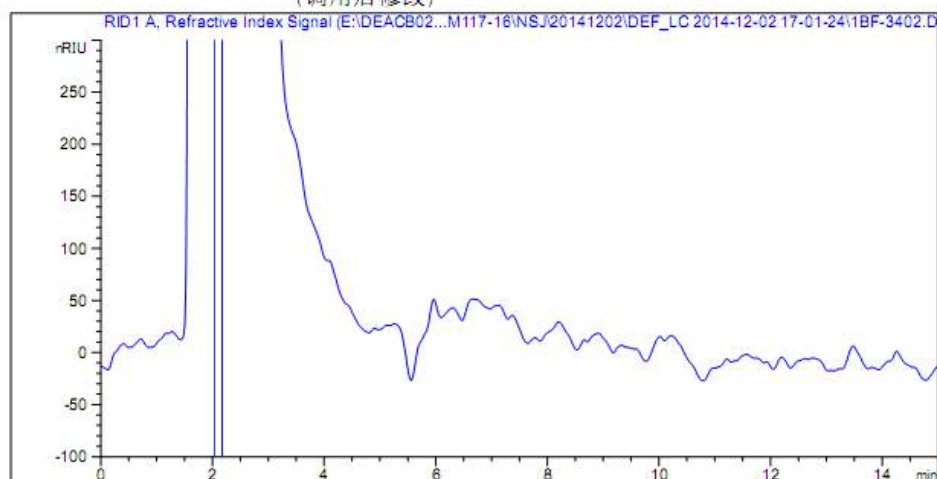
```
=====
                        *** 报告结束 ***
=====
```

附图14-4-33 SBECD加速1月倍他环糊精测定图 (20140921-1-2)

Annex 3- S-30 Accelerated testing-Betadex-1month-20140930-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1BF-3402.D
样品名称: 20140930-40C-1M-1

```
=====
操作者       : Weijing                      序列行   : 34
仪器         : 1260-2                      位置     : Pl-B-06
进样日期     : 2014/12/3 7:45:20           进样次数  : 2
                                           进样量   : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2014/12/18 11:00:01
                                           (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

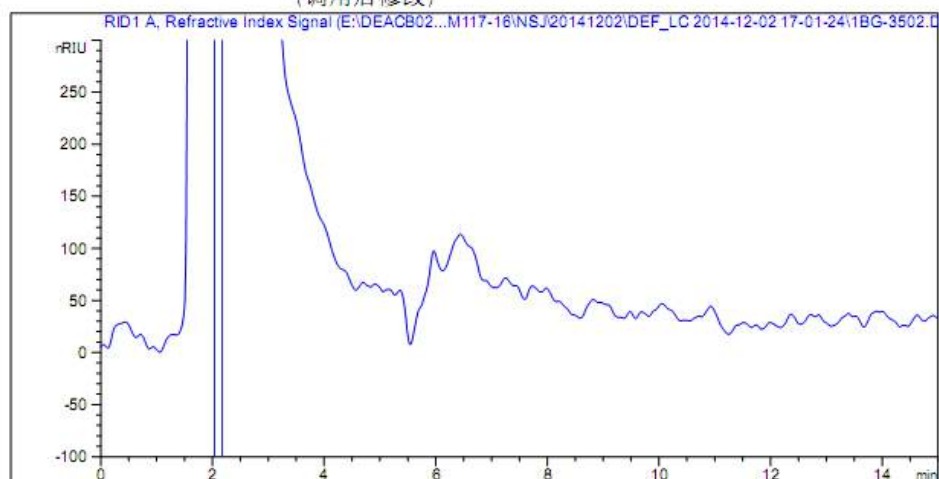
```
=====
                        *** 报告结束 ***
=====
```

附图14-4-37 SBECD加速1月倍他环糊精测定图 (20140930-1-2)

Annex 3- S-31 Accelerated testing-Betadex-1month-20140930-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\1BG-3502.D
样品名称: 20140930-40C-1M-2

```
=====
操作者       : Weijing                      序列行 : 35
仪器         : 1260-2                      位置   : Pl-B-07
进样日期     : 2014/12/3 8:16:21           进样次数 : 2
                                           进样量  : 10.000 µl
采集方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M
最后修改     : 2014/12/2 17:01:24 : Weijing
分析方法     : E:\DEACB02694\SIM117-16\NSJ\20141202\DEF_LC 2014-12-02 17-01-24\B CD T3.M (
                                           序列方法)
最后修改     : 2014/12/18 11:00:01
                                           (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序           :      信号
乘积因子       :      1.0000
稀释因子       :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

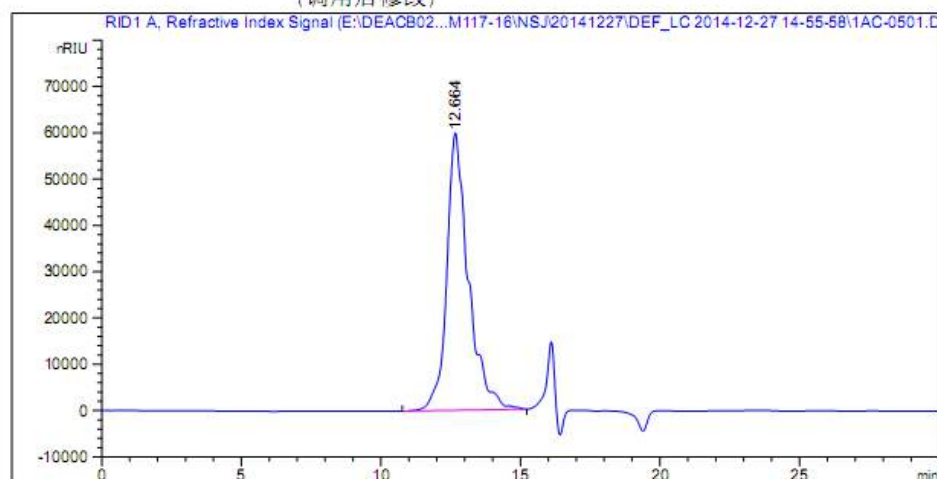
```
=====
                        *** 报告结束 ***
=====
```

附图14-4-39 SBECD加速1月倍他环糊精测定图 (20140930-2-2)

Annex 3- S-32 Accelerated testing-Assay-2month-Reference solution 1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1AC-0501.D
样品名称: DZ-1

```
=====
操作者      :                               序列行 :    5
仪器        : 1260-2                       位置   : P1-A-03
进样日期    : 2014/12/27 16:58:54          进样次数:    1
                                      进样量  : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M
最后修改    : 2014/12/27 14:55:58
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/3/31 11:42:29 : linping
              (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.664	-	3.22689e6	5.98883e4	0.63	0.7009	1809	-	-

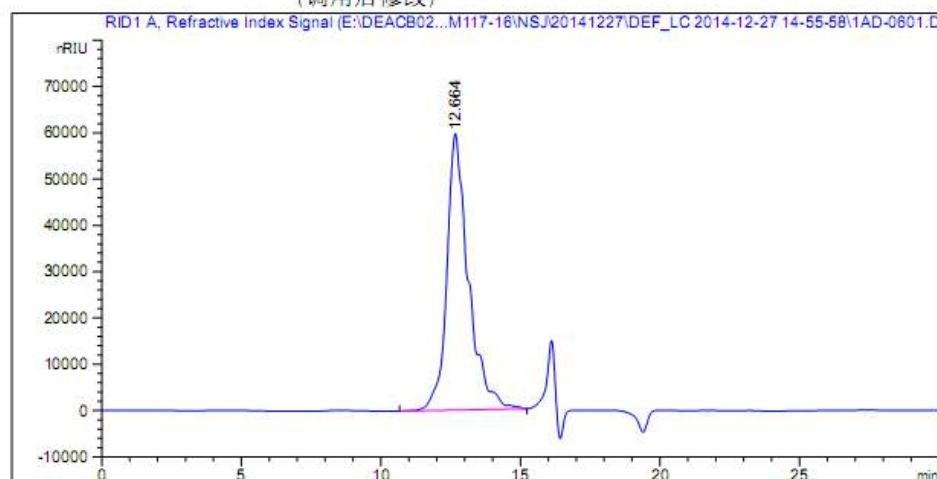
=====
*** 报告结束 ***

附图14-4-41 SBECD加速2月含量测定图 (对照1-1)

Annex 3- S-33 Accelerated testing-Assay-2month-Refence solution 2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1AD-0601.D
样品名称: DZ-2

```
=====
操作者      :                               序列行 :    6
仪器        : 1260-2                       位置   : Pl-A-04
进样日期    : 2014/12/27 19:31:54          进样次数 :    1
                                      进样量  : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M
最后修改    : 2014/12/27 17:23:33 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2014/12/29 9:32:12 : Weijing
              (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子   :      1.0000
稀释因子   :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.664	BB	0.7397	3.20607e6	5.96181e4	100.0000

总量 : 3.20607e6 5.96181e4

*** 报告结束 ***

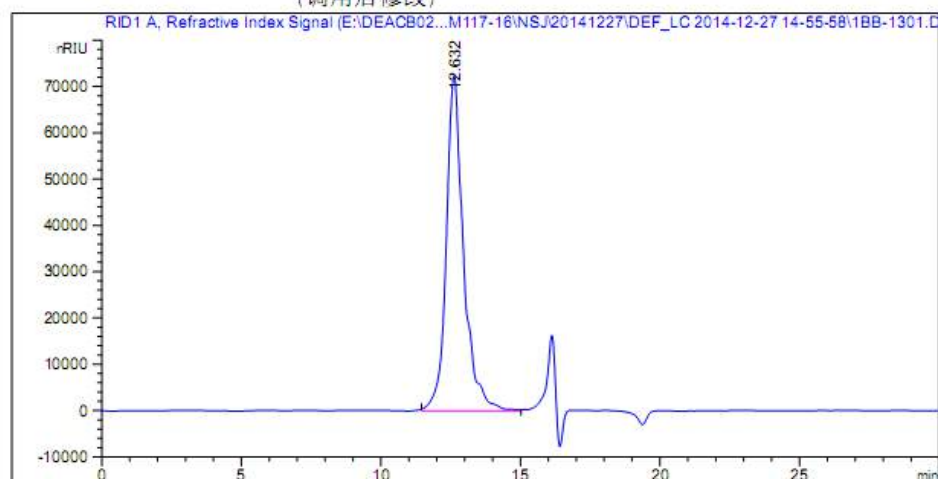
附图14-4-44 SBECD加速2月含量测定图 (对照2-1)

Annex 3- S-34 Accelerated testing-Assay-2month-20140910-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1BB-1301.D
样品名称: 20140910-40C-2M-1

```
=====
操作者      :                               序列行 :   13
仪器        : 1260-2                       位置    : PI-B-02
进样日期    : 2014/12/28 3:10:56           进样次数 :    1
                                           进样量   : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M
最后修改    : 2014/12/27 17:23:33 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/1/5 11:34:07 : Weijing
              (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子  :      1.0000
稀释因子  :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.632	BBA	0.6828	3.13228e6	7.16503e4	100.0000

总量 : 3.13228e6 7.16503e4

*** 报告结束 ***

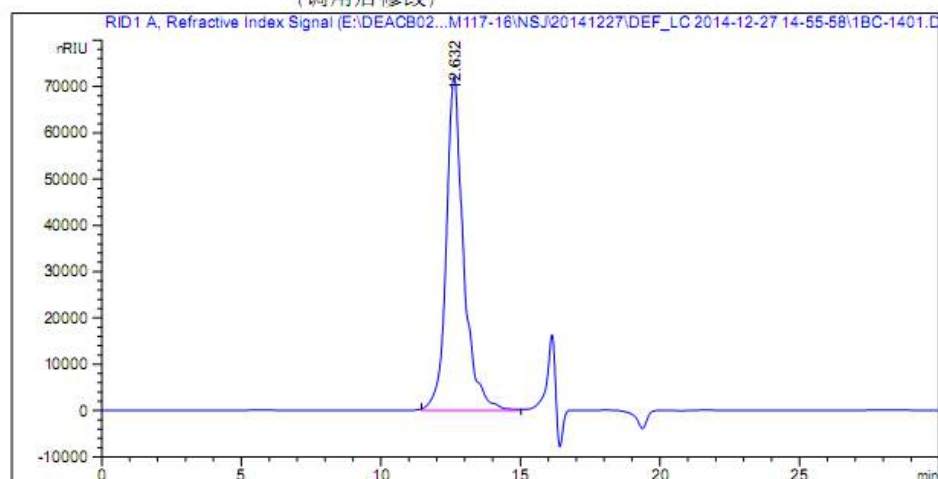
附图14-4-47 SBECD加速2月含量测定图 (20140910-1-1)

Annex 3- S-35 Accelerated testing-Assay-2month-20140910-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1BC-1401.D
样品名称: 20140910-40C-2M-2

```
=====
操作者      :                               序列行 :   14
仪器        : 1260-2                        位置   : PI-B-03
进样日期    : 2014/12/28 4:12:08            进样次数:    1
                                           进样量  : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M
最后修改    : 2014/12/27 17:23:33 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/1/5 11:34:07 : Weijing
              (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子  :      1.0000
稀释因子  :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.632	BBA	0.6821	3.11661e6	7.13979e4	100.0000

总量 : 3.11661e6 7.13979e4

*** 报告结束 ***

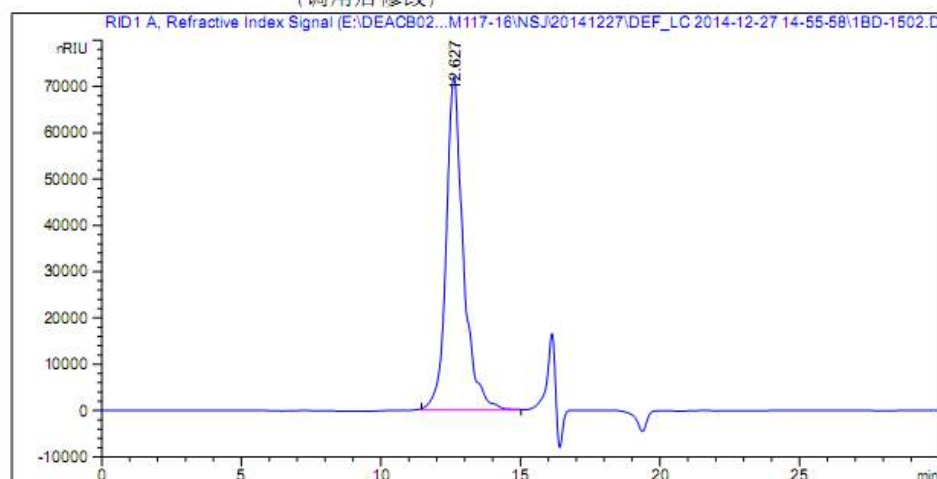
附图14-4-49 SBECD加速2月含量测定图 (20140910-2-1)

Annex 3- S-36 Accelerated testing-Assay-2month-20140921-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1BD-1502.D
样品名称: 20140921-40C-2M-1

```
=====
操作者      :                               序列行 :   15
仪器        : 1260-2                        位置   : Pl-B-04
进样日期    : 2014/12/28 5:43:57            进样次数 :    2
                                           进样量   : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M
最后修改    : 2014/12/27 17:23:33 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/1/5 11:34:07 : Weijing
              (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子  :      1.0000
稀释因子  :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.627	BBA	0.6799	3.10088e6	7.13396e4	100.0000

总量 : 3.10088e6 7.13396e4

*** 报告结束 ***

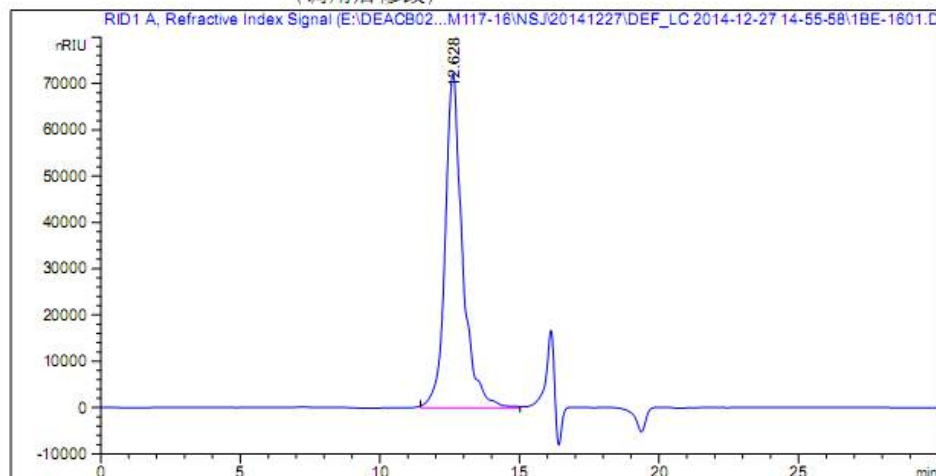
附图14-4-52 SBECD加速2月含量测定图 (20140921-1-2)

Annex 3- S-37 Accelerated testing-Assay-2month-20140921-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1BE-1601.D
样品名称: 20140921-40C-2M-2

=====

操作者	:		序列行	:	16
仪器	:	1260-2	位置	:	Pl-B-05
进样日期	:	2014/12/28 6:14:32	进样次数	:	1
			进样量	:	20.000 µl
采集方法	:	E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-SBECN-0.6.M			
最后修改	:	2014/12/27 17:23:33 : Weijing			
分析方法	:	E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-SBECN-0.6.M (序列方法)			
最后修改	:	2015/1/5 11:34:07 : Weijing			
		(调用后修改)			



=====

面积百分比报告

=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.628	BBA	0.6828	3.12594e6	7.15027e4	100.0000

总量 : 3.12594e6 7.15027e4

*** 报告结束 ***

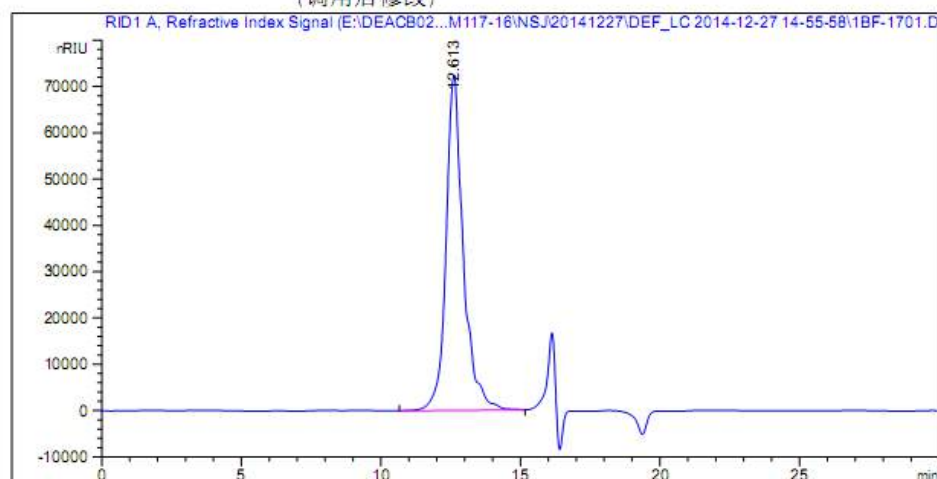
附图14-4-53 SBECN加速2月含量测定图 (20140921-2-1)

Annex 3- S-38 Accelerated testing-Assay-2month-20140930-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1BF-1701.D
样品名称: 20140930-40C-2M-1

=====

操作者	:		序列行	:	17
仪器	:	1260-2	位置	:	Pl-B-06
进样日期	:	2014/12/28 7:15:44	进样次数	:	1
			进样量	:	20.000 µl
采集方法	:	E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-SBECd-0.6.M			
最后修改	:	2014/12/27 17:23:33 : Weijing			
分析方法	:	E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-SBECd-0.6.M (序列方法)			
最后修改	:	2014/12/29 9:32:12 : Weijing (调用后修改)			



=====

面积百分比报告

=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.613	BB	0.6148	3.12258e6	7.25011e4	100.0000

总量 : 3.12258e6 7.25011e4

*** 报告结束 ***

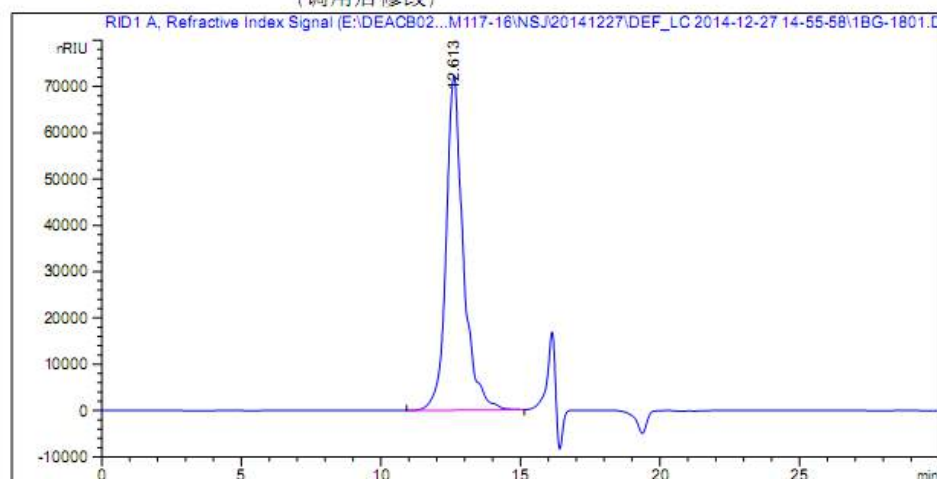
附图14-4-55 SBECd加速2月含量测定图 (20140930-1-1)

Annex 3- S-39 Accelerated testing-Assay-2month-20140930-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\1BG-1801.D
样品名称: 20140930-40C-2M-2

```
=====
操作者      :                               序列行 :   18
仪器        : 1260-2                       位置   : Pl-B-07
进样日期    : 2014/12/28 8:16:56           进样次数:    1
                                           进样量  : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M
最后修改    : 2014/12/27 17:23:33 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20141227\DEF_LC 2014-12-27 14-55-58\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2014/12/29 9:32:12 : Weijing
              (调用后修改)
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子   :      1.0000
稀释因子   :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.613	BB	0.6136	3.10543e6	7.22789e4	100.0000

总量 : 3.10543e6 7.22789e4

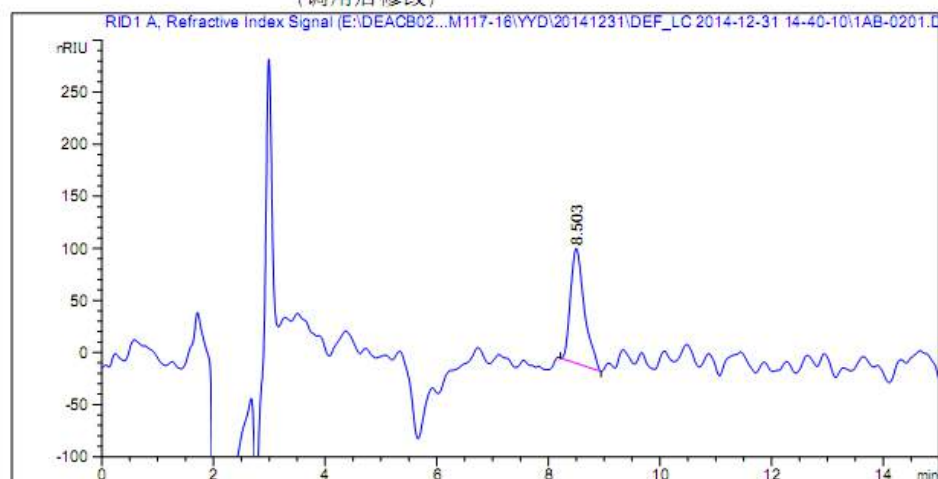
*** 报告结束 ***

附图14-4-57 SBECD加速2月含量测定图 (20140930-2-1)

Annex 3- S-40 Accelerated testing-Betadex-2month-Refence solution 1-1

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1AB-0201.D
 样品名称: DZ-1

```
=====
操作者      :                               序列行 :    2
仪器        : 1260-2                       位置   : Pl-A-02
进样日期    : 2014/12/31 16:44:35          进样次数:    1
                                      进样量  : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2014/12/31 14:40:10
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
              序列方法)
最后修改    : 2015/3/31 18:36:55 : linping
              (调用后修改)
=====
```



```
=====
                        面积百分比报告 (包含性能计算)
=====
```

```
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
8.503	-	1908.28259	109.63652	0.68	0.2520	6297	-	-

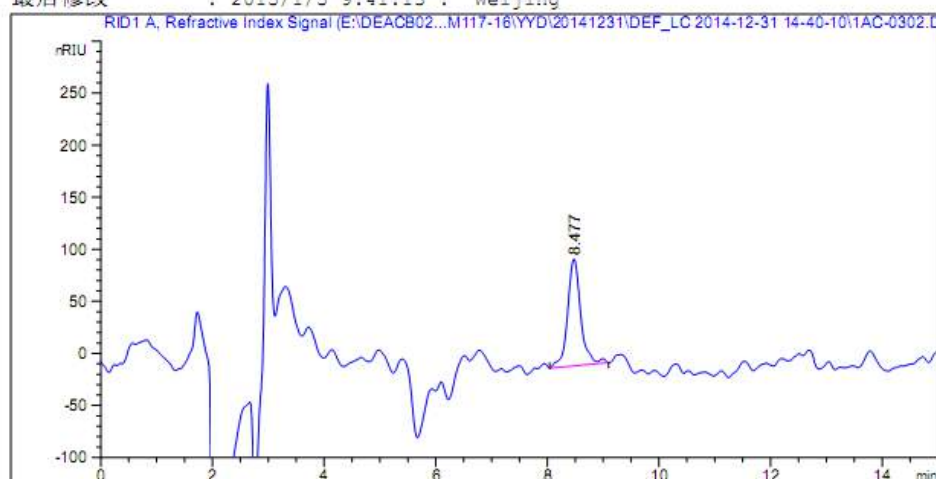
```
=====
*** 报告结束 ***
=====
```

附图14-4-60 SBECD加速2月倍他环糊精测定图 (对照1-1)

Annex 3- S-41 Accelerated testing-Betadex-2month- Refence solution 2-1

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1AC-0302.D
样品名称: DZ-2

```
=====
操作者      :                               序列行 :    3
仪器        : 1260-2                        位置   : P1-A-03
进样日期    : 2014/12/31 18:17:37          进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2014/12/31 14:40:10
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
                                           序列方法)
最后修改    : 2015/1/5 9:41:13 : Weijing
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	8.477	BB	0.2523	1679.14185	102.13190	100.0000

总量 : 1679.14185 102.13190

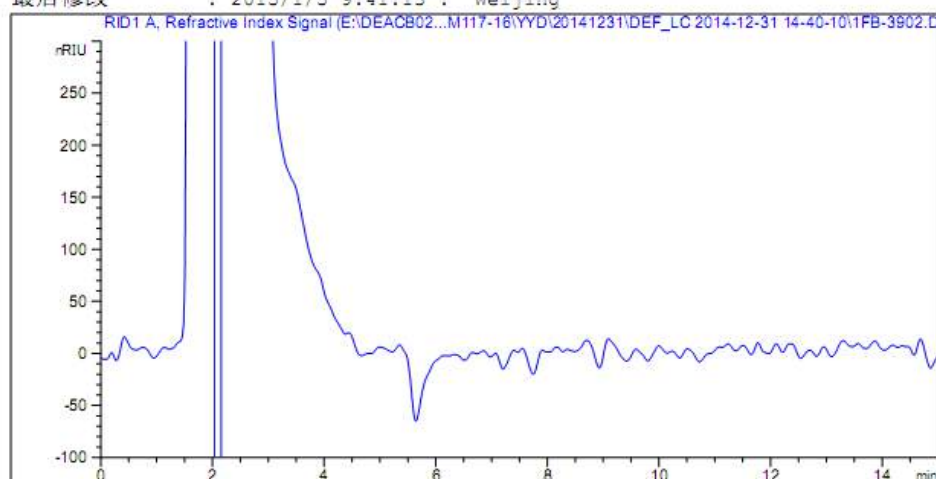
=====
*** 报告结束 ***

附图14-4-65 SBECD加速2月倍他环糊精测定图 (对照2-1)

Annex 3- S-42 Accelerated testing-Betadex-2month-20140910-1-2

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1FB-3902.D
样品名称: 40C-SBEC20140910-1

```
=====
操作者      :                               序列行 :   39
仪器        : 1260-2                       位置    : Pl-F-02
进样日期    : 2015/1/1 13:08:51             进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2015/1/1 9:37:30 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
                                           序列方法)
最后修改    : 2015/1/5 9:41:13 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

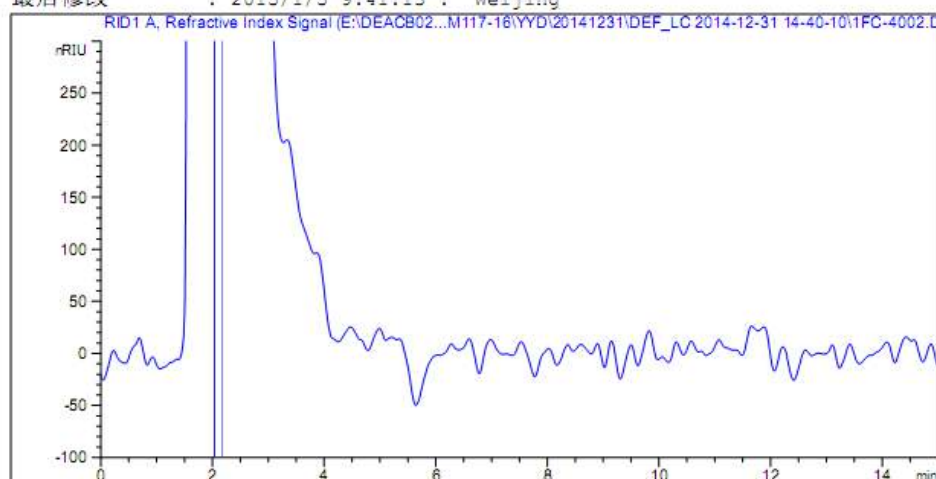
```
=====
*** 报告结束 ***
=====
```

附图14-4-69 SBEC2加速2月倍他环糊精测定图 (20140910-1-2)

Annex 3- S-43 Accelerated testing-Betadex-2month-20140910-2-2

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1FC-4002.D
样品名称: 40C-SBEC20140910-2

```
=====
操作者      :                               序列行 :   40
仪器        : 1260-2                       位置   : Pl-F-03
进样日期    : 2015/1/1 13:39:50             进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2015/1/1 9:37:30 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
                                           序列方法)
最后修改    : 2015/1/5 9:41:13 : Weijing
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

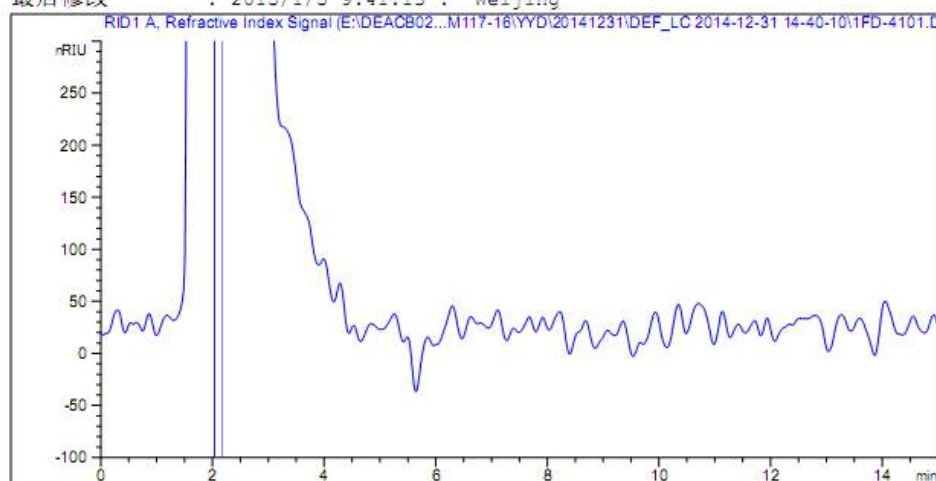
=====
*** 报告结束 ***

附图14-4-71 SBEC2加速2月倍他环糊精测定图 (20140910-2-2)

Annex 3- S-44 Accelerated testing-Betadex-2month-20140921-1-1

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1FD-4101.D
样品名称: 40C-SBEC20140921-1

```
=====
操作者      :                               序列行 :   41
仪器        : 1260-2                       位置   : Pl-F-04
进样日期    : 2015/1/1 13:55:19             进样次数 :    1
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2015/1/1 9:37:30 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
                                           序列方法)
最后修改    : 2015/1/5 9:41:13 : Weijing
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

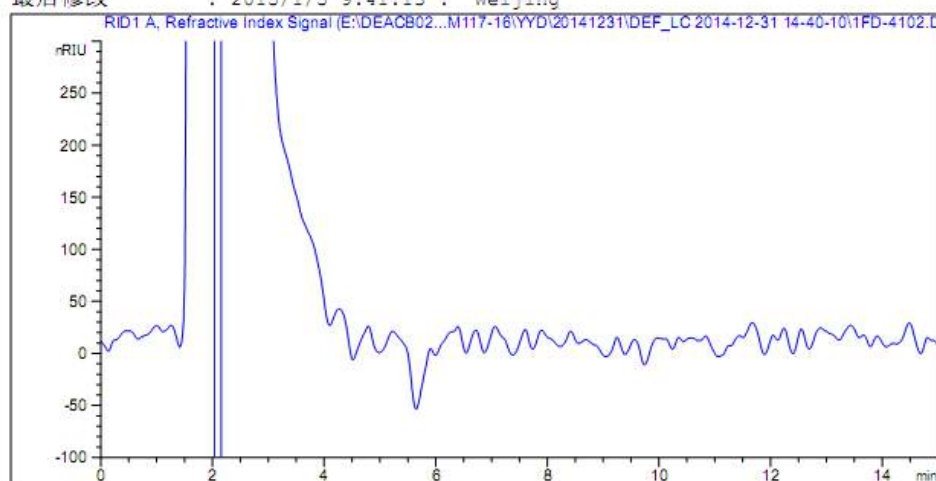
=====
*** 报告结束 ***

附图14-4-72 SBEC2加速2月倍他环糊精测定图 (20140921-1-1)

Annex 3- S-45 Accelerated testing-Betadex-2month-20140921-1-2

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1FD-4102.D
样品名称: 40C-SBEC20140921-1

```
=====
操作者      :                               序列行 :   41
仪器        : 1260-2                       位置   : Pl-F-04
进样日期    : 2015/1/1 14:10:48             进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2015/1/1 9:37:30 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
                                           序列方法)
最后修改    : 2015/1/5 9:41:13 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

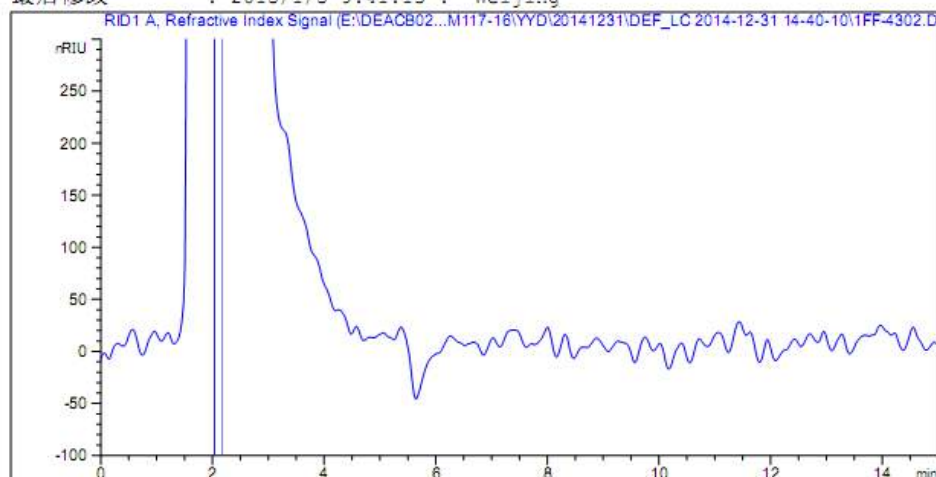
```
=====
*** 报告结束 ***
=====
```

附图14-4-73 SBEC2加速2月倍他环糊精测定图 (20140921-1-2)

Annex 3- S-46 Accelerated testing-Betadex-2month-20140930-1-2

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1FF-4302.D
样品名称: 40C-SBEC20140930-1

```
=====
操作者      :                               序列行 :   43
仪器        : 1260-2                       位置    : Pl-F-06
进样日期    : 2015/1/1 15:12:43             进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2015/1/1 9:37:30 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
                                           序列方法)
最后修改    : 2015/1/5 9:41:13 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

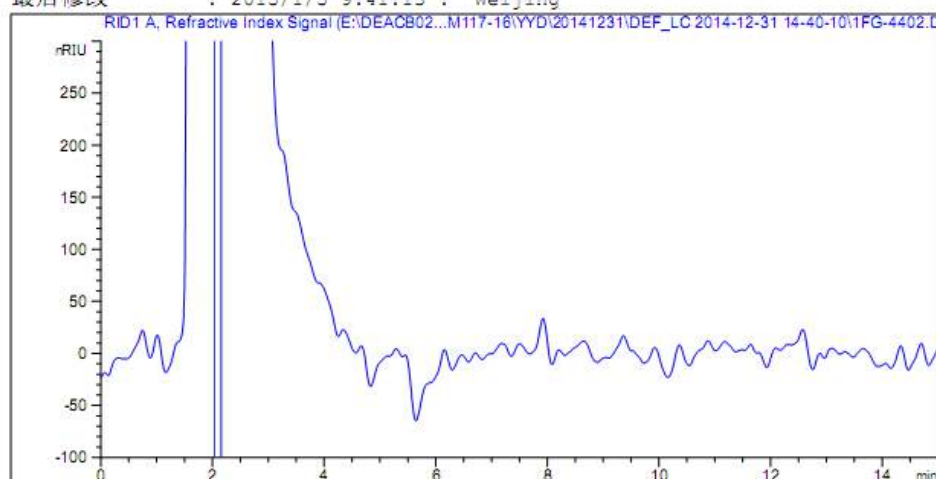
```
=====
*** 报告结束 ***
=====
```

附图14-4-77 SBEC2加速2月倍他环糊精测定图 (20140930-1-2)

Annex 3- S-47 Accelerated testing-Betadex-2month-20140930-2-2

数据文件: E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\1FG-4402.D
样品名称: 40C-SBEC20140930-2

```
=====
操作者      :                               序列行 :   44
仪器        : 1260-2                       位置   : Pl-F-07
进样日期    : 2015/1/1 15:43:41             进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M
最后修改    : 2015/1/1 9:37:30 : Weijing
分析方法    : E:\DEACB02694\SIM117-16\YYD\20141231\DEF_LC 2014-12-31 14-40-10\B CD T3.M (
                                           序列方法)
最后修改    : 2015/1/5 9:41:13 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

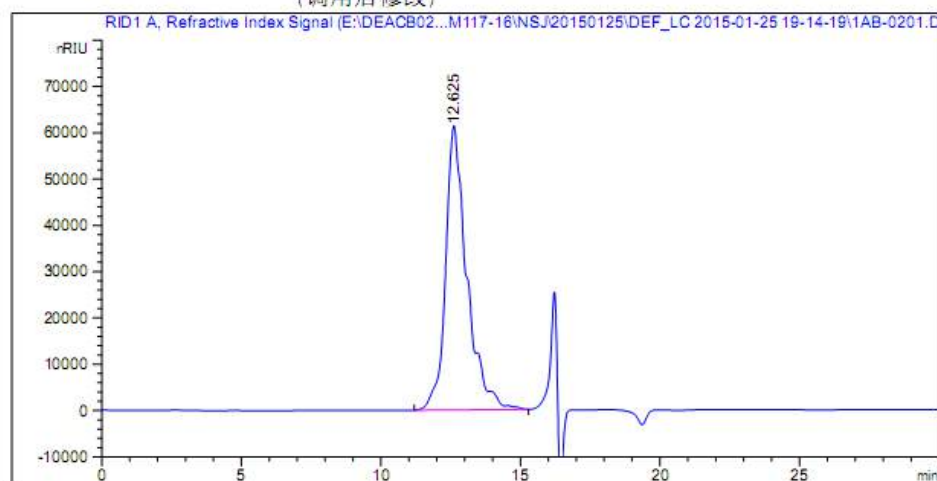
```
=====
*** 报告结束 ***
=====
```

附图14-4-79 SBEC2加速2月倍他环糊精测定图 (20140930-2-2)

Annex 3-S-48 Accelerated testing and long-term testing-Assay-3month-Reference solution
1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1AB-0201.D
样品名称: DZ-1

```
=====
操作者      :                               序列行 :    2
仪器        : 1260-2                       位置   : Pl-A-02
进样日期    : 2015/1/25 20:46:41          进样次数 :    1
                                      进样量   : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M
最后修改    : 2015/1/25 19:14:19
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/3/31 11:44:47 : linping
              (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.625	-	3.24732e6	6.09291e4	0.63	0.6900	1851	-	-

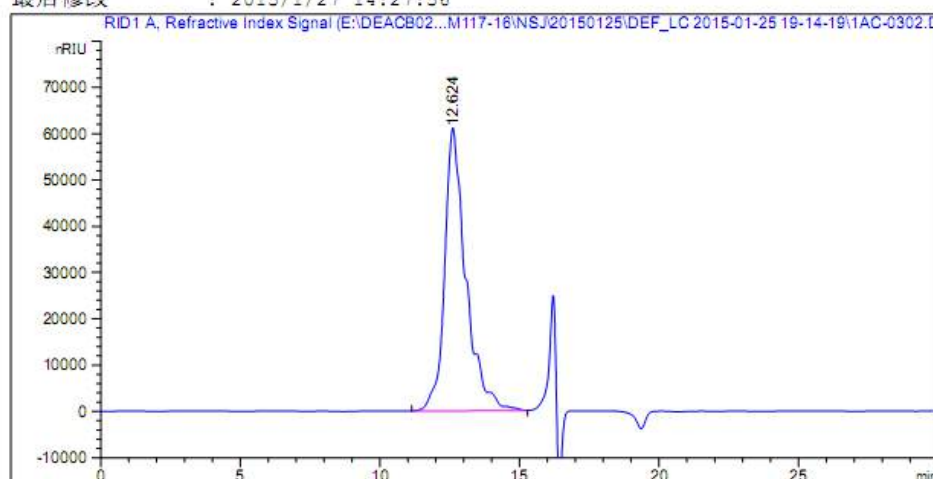
=====
*** 报告结束 ***

附图14-4-81 SBECD加速3月含量测定图 (加速3月、长期3月对照1-1)

Annex 3-S-49 Accelerated testing and long-term testing-Assay-3month-Reference solution
2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1AC-0302.D
样品名称: DZ-2

```
=====
操作者      :                               序列行 :    3
仪器        : 1260-2                         位置   : P1-A-03
进样日期    : 2015/1/25 23:50:14             进样次数 :    2
                                           进样量   : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
                                           SBECD-0.6.M
最后修改    : 2015/1/25 19:14:19
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
                                           SBECD-0.6.M (序列方法)
最后修改    : 2015/1/27 14:27:56
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.624	BBA	0.7956	3.23758e6	6.06500e4	100.0000

总量 : 3.23758e6 6.06500e4

=====
*** 报告结束 ***

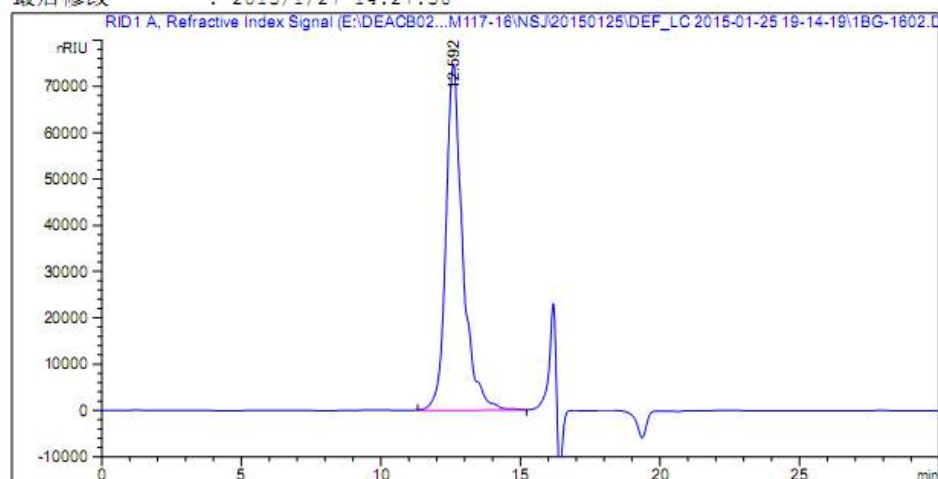
附图14-4-87 SBECD加速3月含量测定图（加速3月、长期3月对照2-2）

Annex 3-S-50 Accelerated testing -Assay-3month-20140910-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1BG-1602.D
样品名称: 20140910-40C-3M-1

```
=====
操作者      :                               序列行 :   16
仪器        :   1260-2                      位置   : P1-B-07
进样日期    :   2015/1/26 13:36:13          进样次数 :    2
                                           进样量   : 20.000 µl

采集方法    :   E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
                SBECD-0.6.M
最后修改    :   2015/1/25 19:14:19
分析方法    :   E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
                SBECD-0.6.M (序列方法)
最后修改    :   2015/1/27 14:27:56
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.592	BBA	0.6478	3.13804e6	7.43721e4	100.0000

总量 : 3.13804e6 7.43721e4

=====
*** 报告结束 ***

附图14-4-90 SBECD加速3月含量测定图 (20140910-1-2)

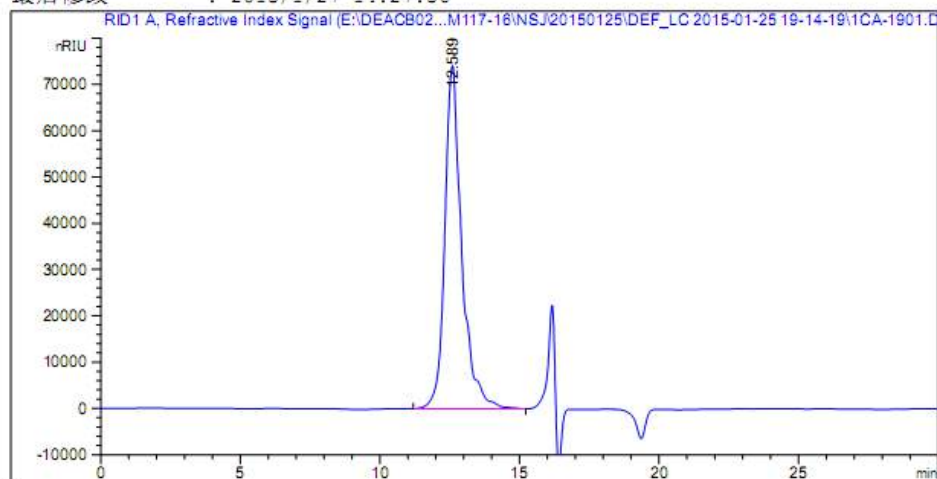
Annex 3-S-51 Accelerated testing-Assay-3month-20140921-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1CA-1901.D
样品名称: 20140921-40C-3M-2

=====

操作者	:		序列行	:	19
仪器	:	1260-2	位置	:	Pl-C-01
进样日期	:	2015/1/26 16:09:13	进样次数	:	1
			进样量	:	20.000 µl
采集方法	:	E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-SBEC-0.6.M			
最后修改	:	2015/1/26 14:01:34			
分析方法	:	E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-SBEC-0.6.M (序列方法)			
最后修改	:	2015/1/27 14:27:56			

=====



=====

面积百分比报告

=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.589	BBA	0.6514	3.13686e6	7.38059e4	100.0000

总量 : 3.13686e6 7.38059e4

*** 报告结束 ***

=====

附图14-4-95 SBEC-0.6M加速3月含量测定图 (20140921-2-1)

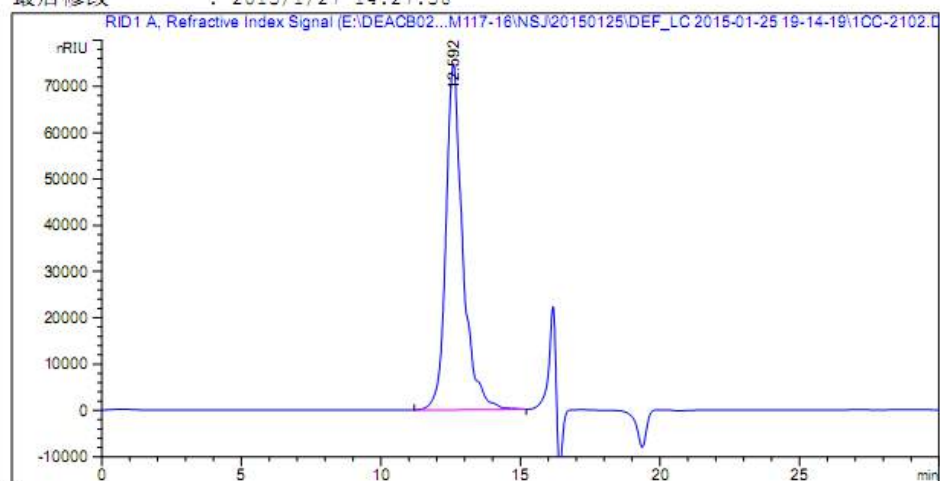
Annex 3-S-52 Accelerated testing-Assay-3month-20140930-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1CC-2102.D
样品名称: 20140930-40C-3M-2

=====

操作者	:		序列行	:	21
仪器	:	1260-2	位置	:	Pl-C-03
进样日期	:	2015/1/26 18:42:11	进样次数	:	2
			进样量	:	20.000 µl
采集方法	:	E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-SBEC-0.6.M			
最后修改	:	2015/1/26 14:01:34			
分析方法	:	E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-SBEC-0.6.M (序列方法)			
最后修改	:	2015/1/27 14:27:56			

=====



=====

面积百分比报告

=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.592	BBA	0.6496	3.14692e6	7.43147e4	100.0000

总量 : 3.14692e6 7.43147e4

*** 报告结束 ***

附图14-4-100

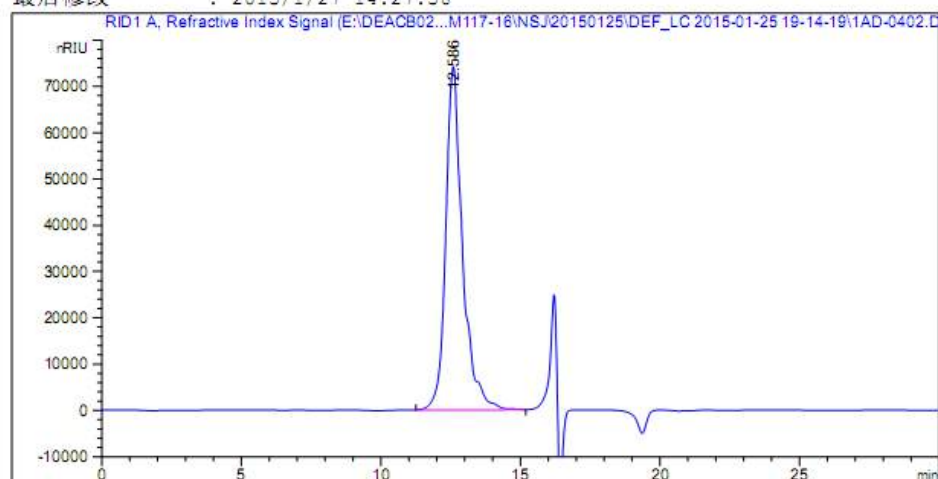
SBEC加速3月含量测定图 (20140930-2-2)

Annex 3-S-53 Long-term testing-Assay-3month-20140910-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1AD-0402.D
样品名称: 20140910-25C-3M-1

```
=====
操作者      :                               序列行 :    4
仪器        : 1260-2                        位置   : P1-A-04
进样日期    : 2015/1/26 1:21:59             进样次数:    2
                                           进样量  : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M
最后修改    : 2015/1/25 19:14:19
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/1/27 14:27:56
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.586	BBA	0.6472	3.11251e6	7.38596e4	100.0000

总量 : 3.11251e6 7.38596e4

=====
*** 报告结束 ***

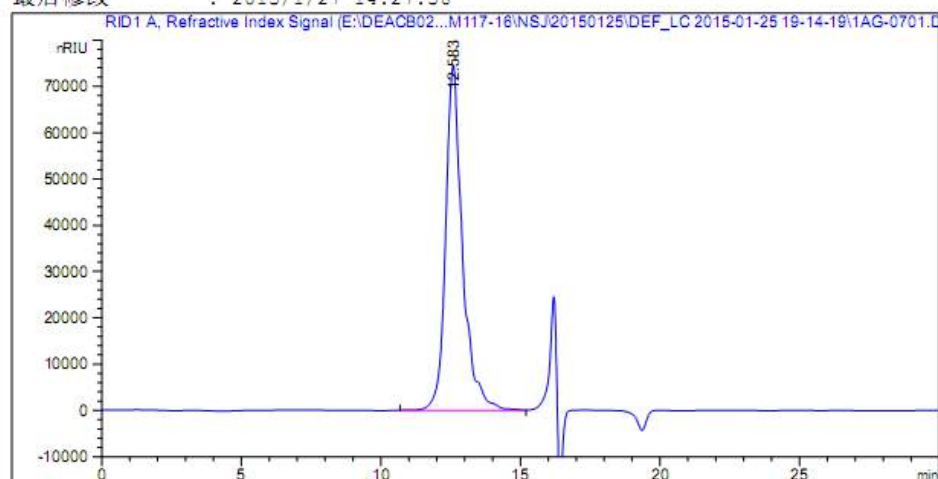
附图14-5-2 SBECD长期3月含量测定图 (20140910-1-2)

Annex 3-S-54 Long-term testing-Assay-3month-20140921-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1AG-0701.D
样品名称: 20140921-25C-3M-2

```
=====
操作者      :                               序列行 :    7
仪器        : 1260-2                        位置   : P1-A-07
进样日期    : 2015/1/26 3:54:56             进样次数:    1
                                           进样量  : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M
最后修改    : 2015/1/25 19:14:19
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/1/27 14:27:56
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.583	BBA	0.6490	3.13704e6	7.41652e4	100.0000

总量 : 3.13704e6 7.41653e4

=====
*** 报告结束 ***

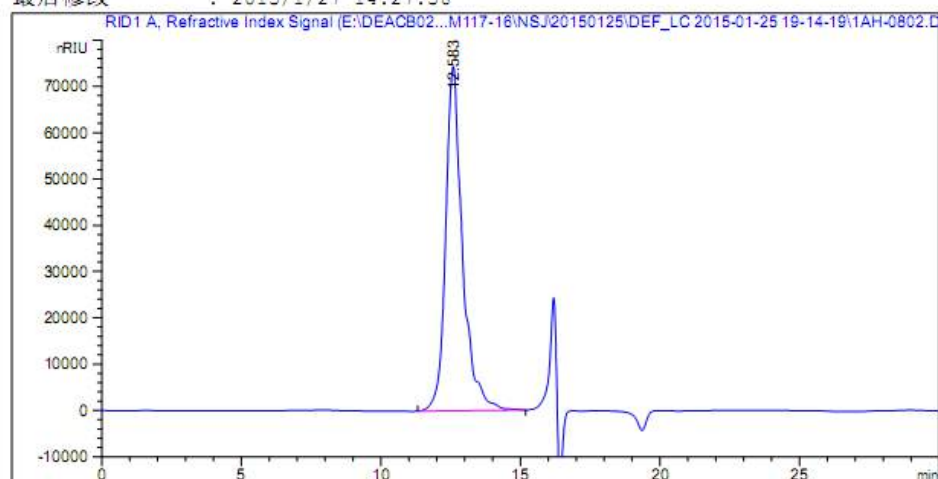
附图14-5-7 SBECD长期3月含量测定图 (20140921-2-1)

Annex 3-S-55 Long-term testing-Assay-3month-20140930-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\1AH-0802.D
样品名称: 20140930-25C-3M-1

```
=====
操作者      :                               序列行 :    8
仪器        : 1260-2                       位置   : P1-A-08
进样日期    : 2015/1/26 5:26:42             进样次数:    2
                                           进样量  : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M
最后修改    : 2015/1/25 19:14:19
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150125\DEF_LC 2015-01-25 19-14-19\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/1/27 14:27:56
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.583	BBA	0.6487	3.13235e6	7.40970e4	100.0000

总量 : 3.13235e6 7.40970e4

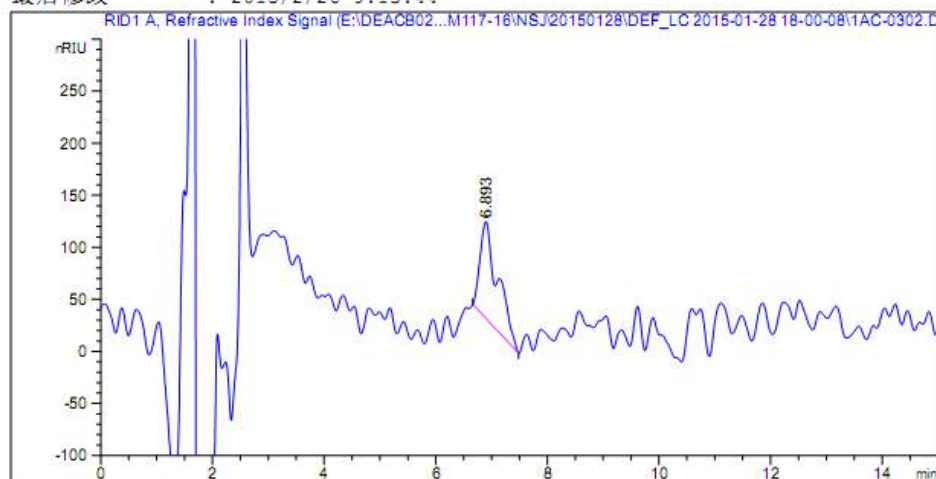
=====
*** 报告结束 ***

附图14-5-10 SBECD长期3月含量测定图 (20140930-1-2)

Annex 3-S-56 Accelerated testing and long-term testing-Betadex-3month-Reference solution
1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1AC-0302.D
样品名称: DZ-1

```
=====
操作者      :                               序列行 :    3
仪器        : 1260-2                       位置   : P1-A-03
进样日期    : 2015/1/28 19:49:07          进样次数 :    2
                                      进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:13:44
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	6.893	BBA	0.3208	2054.27026	92.05956	100.0000

总量 : 2054.27026 92.05956

=====
*** 报告结束 ***

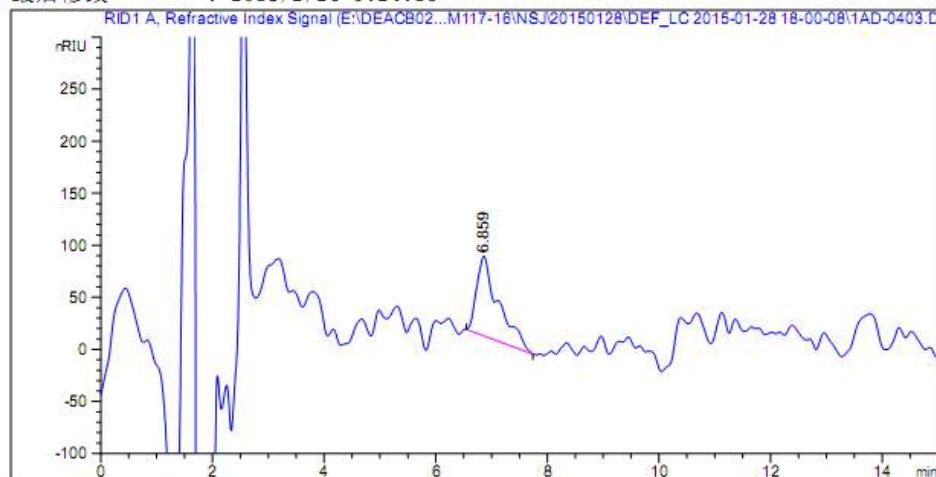
附图14-4-103

SBECD加速3月倍他环糊精测定图 (加速3月、长期3月对照1-2)

Annex 3-S-57 Accelerated testing and long-term testing-Betadex-3month-Reference solution
2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1AD-0403.D
样品名称: DZ-2

```
=====
操作者      :                               序列行 :    4
仪器        : 1260-2                         位置   : P1-A-04
进样日期    : 2015/1/28 21:37:41             进样次数 :    3
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:24:18
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	6.859	BBA	0.3956	2079.81030	76.33781	100.0000

总量 : 2079.81030 76.33781

=====
*** 报告结束 ***

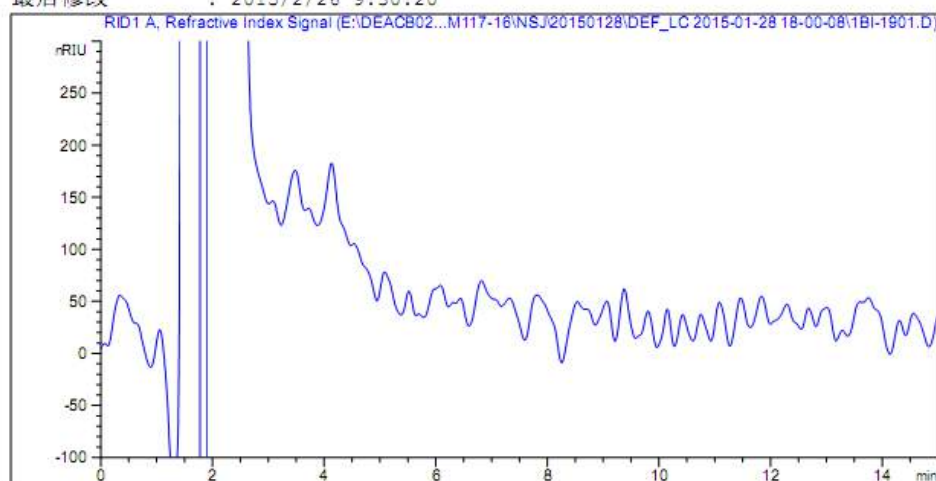
附图14-4-106

SBECD加速3月倍他环糊精测定图 (加速3月、长期3月对照2-2)

Annex 3-S-58 Accelerated testing-Betadex-3month-20140910-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1BI-1901.D
样品名称: 20140910-40C-3M-2

```
=====
操作者      :                               序列行 :   19
仪器        : 1260-2                       位置    : P1-B-09
进样日期    : 2015/1/29 5:53:53             进样次数 :    1
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:30:20
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

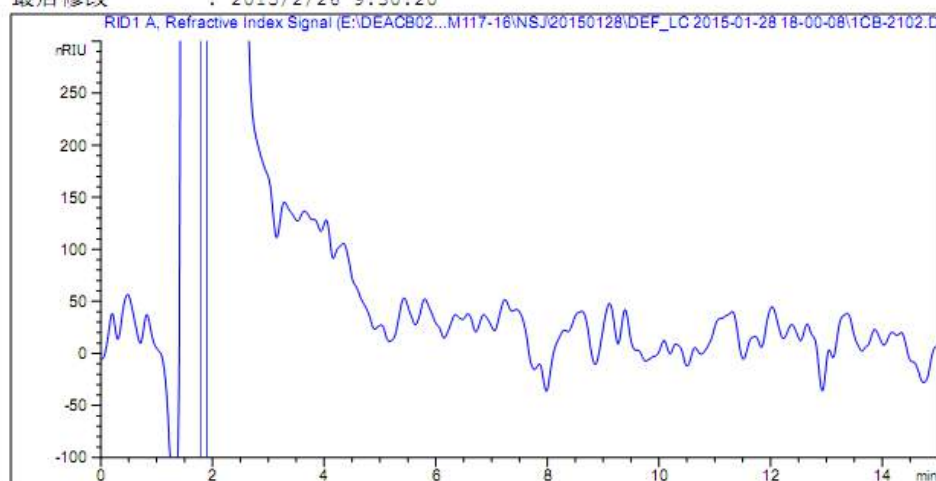
附图14-4-111

SBECD加速3月倍他环糊精测定图 (20140910-2-1)

Annex 3-S-59 Accelerated testing-Betadex-3month-20140921-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1CB-2102.D
样品名称: 20140921-40C-3M-2

```
=====
操作者      :                               序列行 :   21
仪器        : 1260-2                       位置    : P1-C-02
进样日期    : 2015/1/29 7:11:20             进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:30:20
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

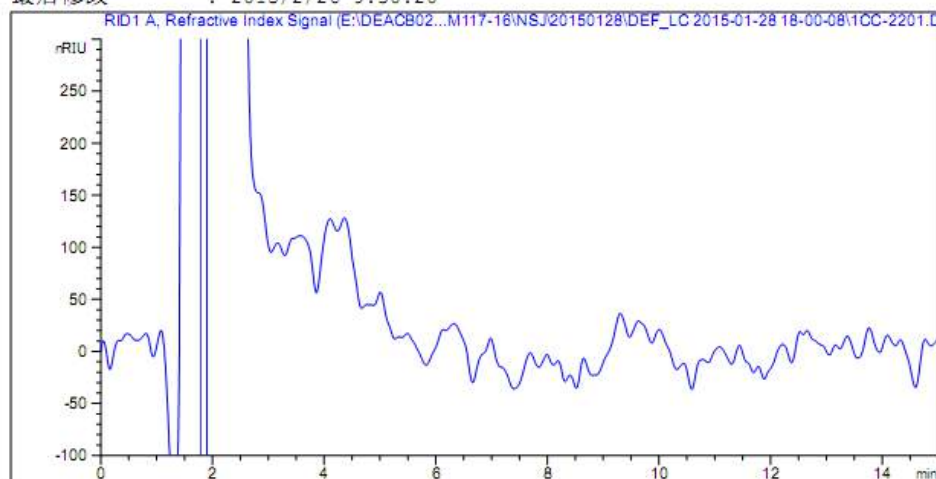
附图14-4-116

SBECD加速3月倍他环糊精测定图 (20140921-2-2)

Annex 3-S-60 Accelerated testing-Betadex-3month-20140930-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1CC-2201.D
样品名称: 20140930-40C-3M-1

```
=====
操作者      :                               序列行 :   22
仪器        : 1260-2                       位置   : P1-C-03
进样日期    : 2015/1/29 7:26:49             进样次数 :    1
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:30:20
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

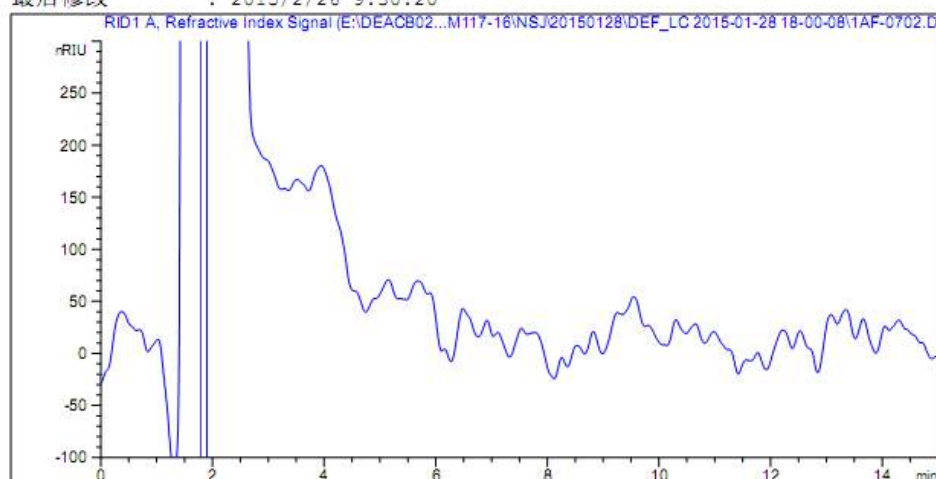
附图14-4-117

SBECD加速3月倍他环糊精测定图 (20140930-1-1)

Annex 3-S-61 Long-term testing-Betadex-3month-20140910-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1AF-0702.D
样品名称: 20140910-25C-3M-2

```
=====
操作者      :                               序列行 :    7
仪器        : 1260-2                        位置   : P1-A-06
进样日期    : 2015/1/28 23:57:13           进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:30:20
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

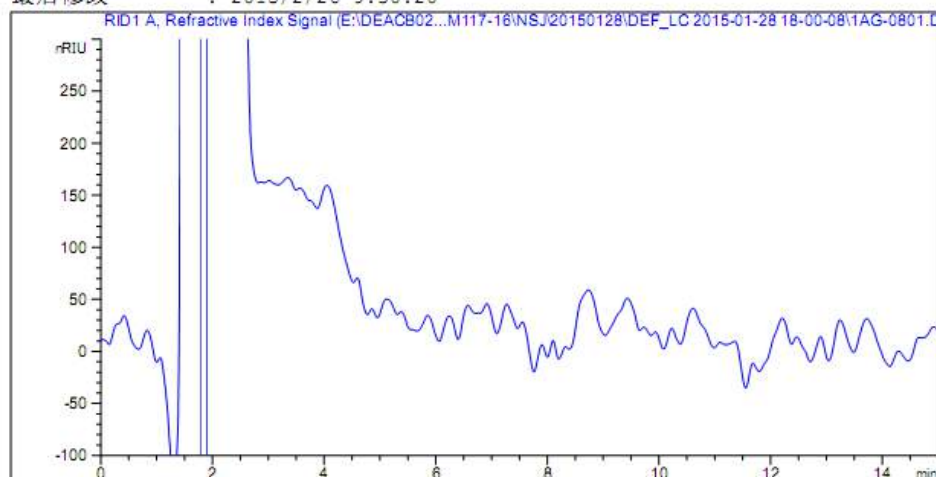
```
=====
*** 报告结束 ***
=====
```

附图14-5-16 SBECD长期3月倍他环糊精测定图 (20140910-2-2)

Annex 3-S-62 Long-term testing-Betadex-3month-20140921-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1AG-0801.D
样品名称: 20140921-25C-3M-1

```
=====
操作者      :                               序列行 :    8
仪器        : 1260-2                       位置   : P1-A-07
进样日期    : 2015/1/29 0:12:44             进样次数:    1
                                           进样量  : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:30:20
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

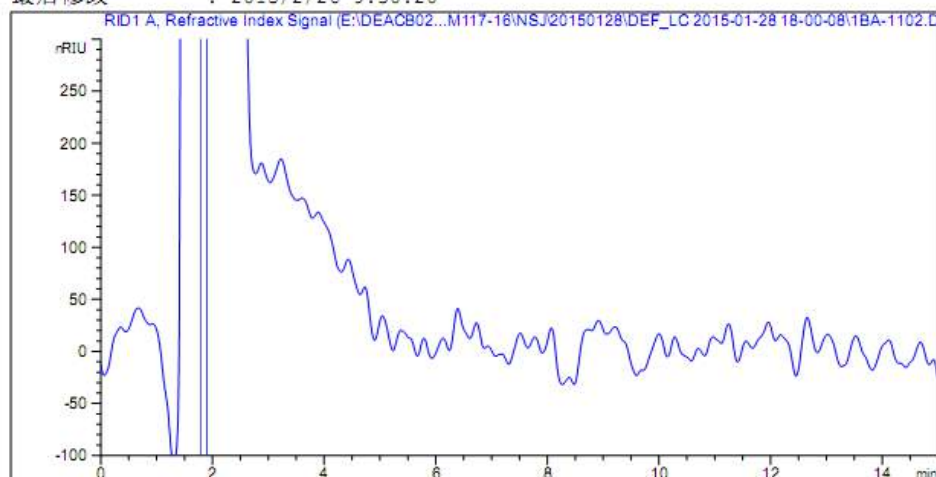
```
=====
*** 报告结束 ***
=====
```

附图14-5-17 SBECD长期3月倍他环糊精测定图 (20140921-1-1)

Annex 3-S-63 Long-term testing-Betadex-3month-20140930-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\1BA-1102.D
样品名称: 20140930-25C-3M-2

```
=====
操作者      :                               序列行 :   11
仪器        : 1260-2                       位置   : P1-B-01
进样日期    : 2015/1/29 2:01:14             进样次数 :    2
                                           进样量  : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M
最后修改    : 2015/1/28 18:00:08
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150128\DEF_LC 2015-01-28 18-00-08\B CD T3.M (
              序列方法)
最后修改    : 2015/2/26 9:30:20
=====
```



```
=====
                        面积百分比报告
=====
```

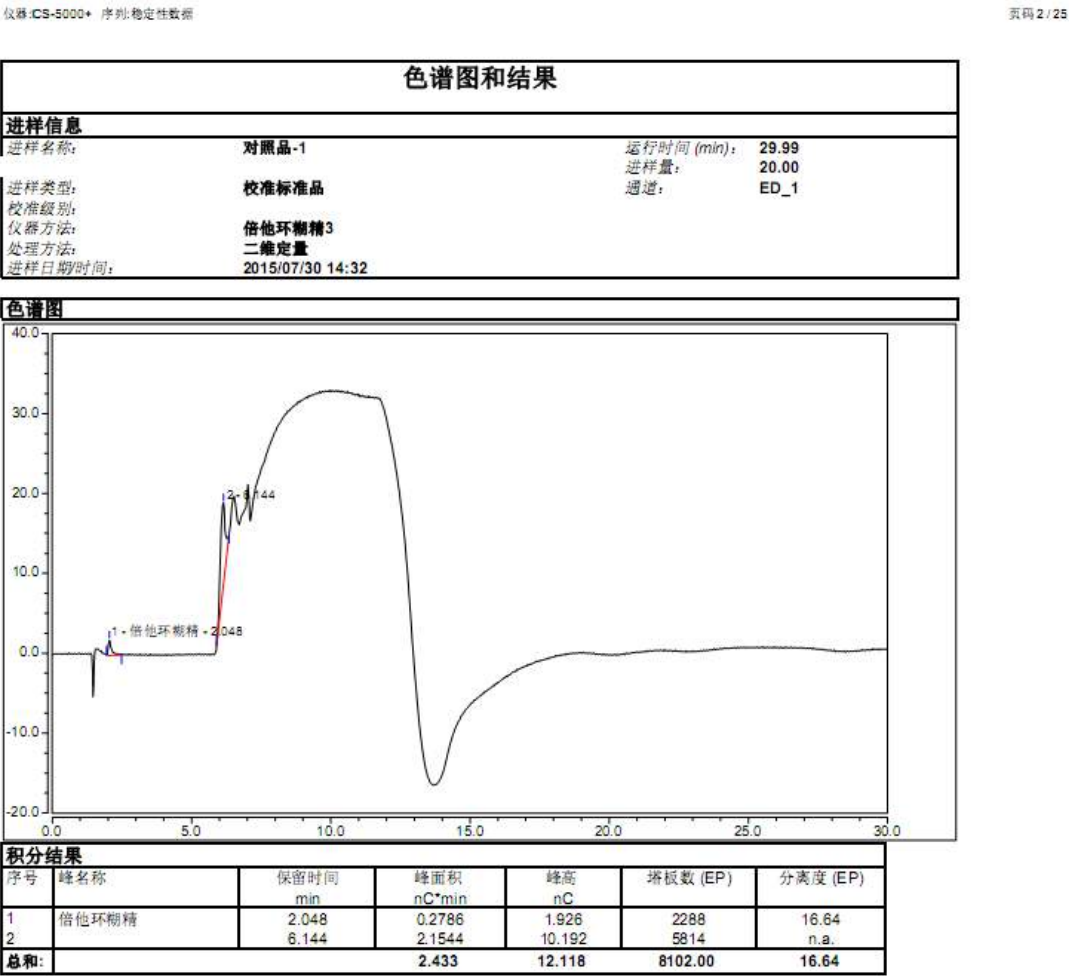
```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

附图14-5-24 SBECD长期3月倍他环糊精测定图 (20140930-2-2)

Annex 3-S-64 Accelerated testing and long-term testing-Betadex (HPIC) -3month-Reference solution -1

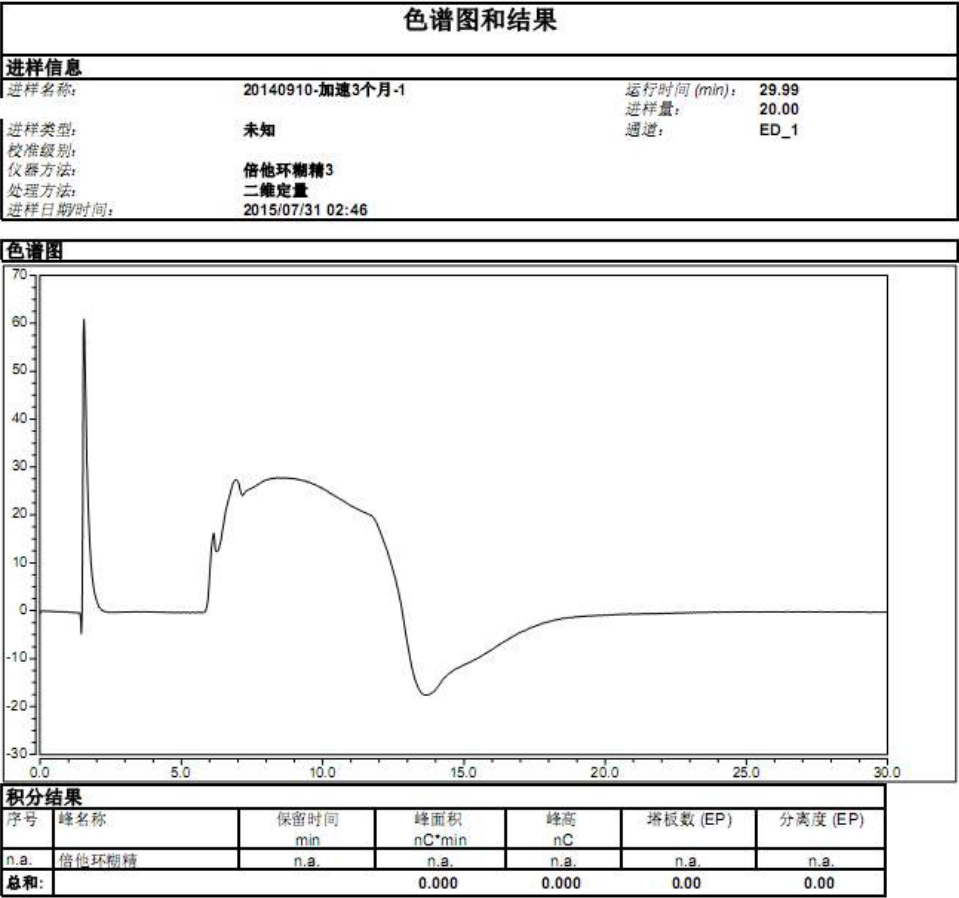


附图14-4-16.1 SBECD加速倍他环糊精的测定图 (对照品-1)

Annex 3-S-65 Accelerated testing -Betadex (HPIC) -3month-20140910-1

仪器:CS-5000+ 序列:稳定性数据

页码:16 / 25



附图14-4-16.3 SRECD加速3月倍他环糊精的测定图 (20140910-1)

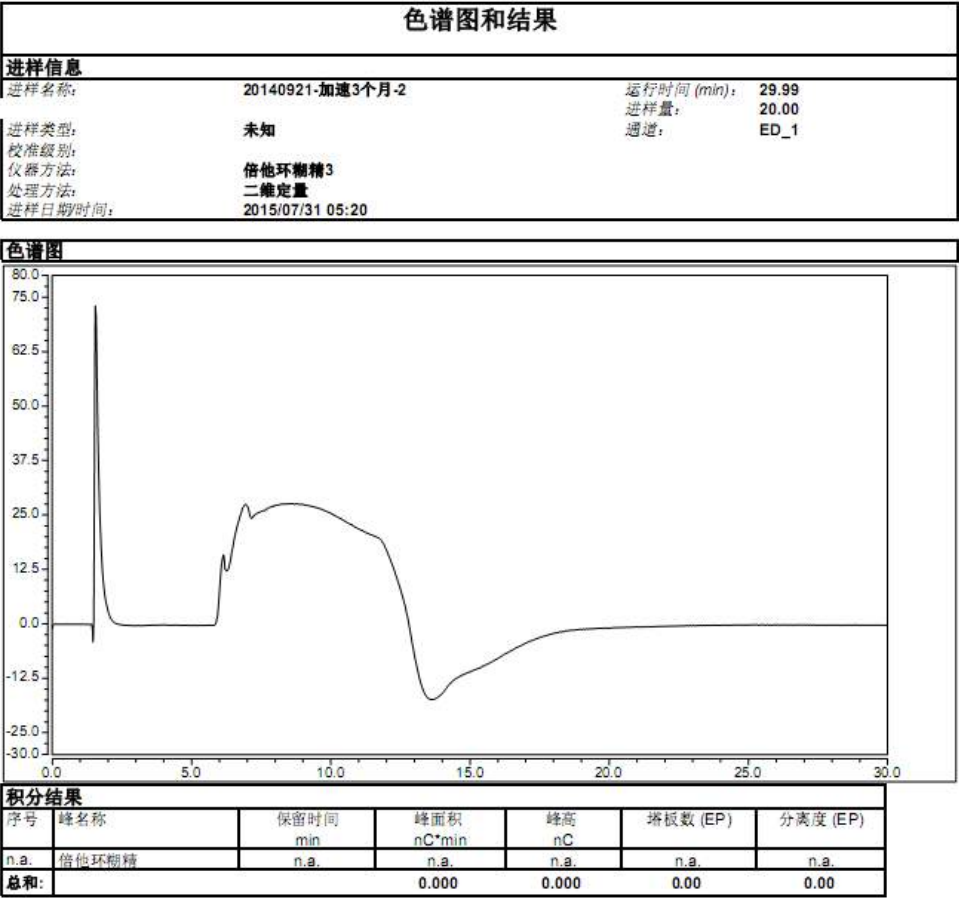
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-66 Accelerated testing-Betadex (HPIC) -3month-20140921-2

仪器:CS-5000+ 序列:稳定性数据

页码 21 / 25



附图14-4-16 6 SECD加速3月倍他环糊精的测定图 (20140921-2)

Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-67 Accelerated testing-Betadex (HPIC) -3month-20140930-1

仪器:CS-5000+ 序列:稳定性数据

页码:24/25

色谱图和结果

进样信息

进样名称: 20140930-加速3个月-1

运行时间 (min): 29.99

进样量: 20.00

通道: ED_1

进样类型:

未知

校准级别:

倍他环糊精3

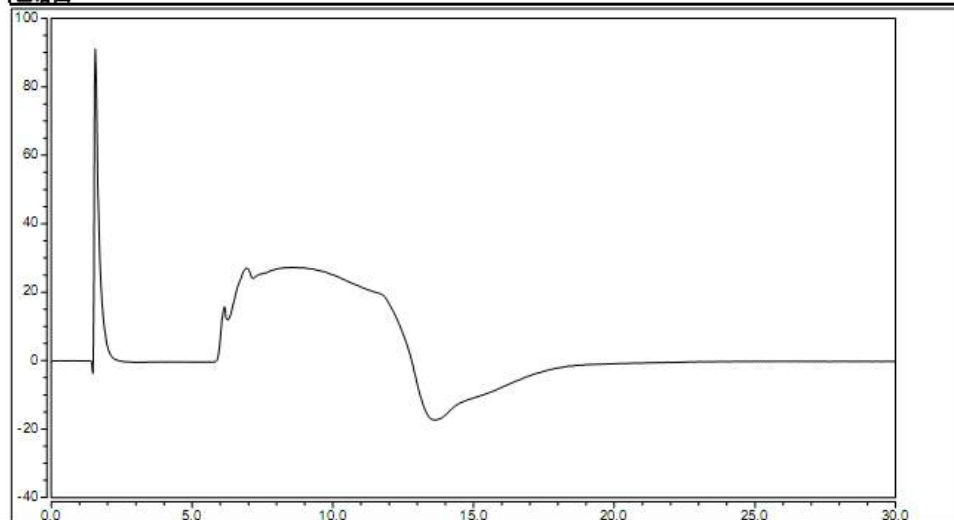
仪器方法:

二维定量

处理方法:

2015/07/31 06:54

色谱图



积分结果

序号	峰名称	保留时间 min	峰面积 nC*min	峰高 nC	塔板数 (EP)	分离度 (EP)
n.a.	倍他环糊精	n.a.	n.a.	n.a.	n.a.	n.a.
总和:			0.000	0.000	0.00	0.00

附图14-4-16.7 SEC加速3月倍他环糊精的测定图 (20140930-1)

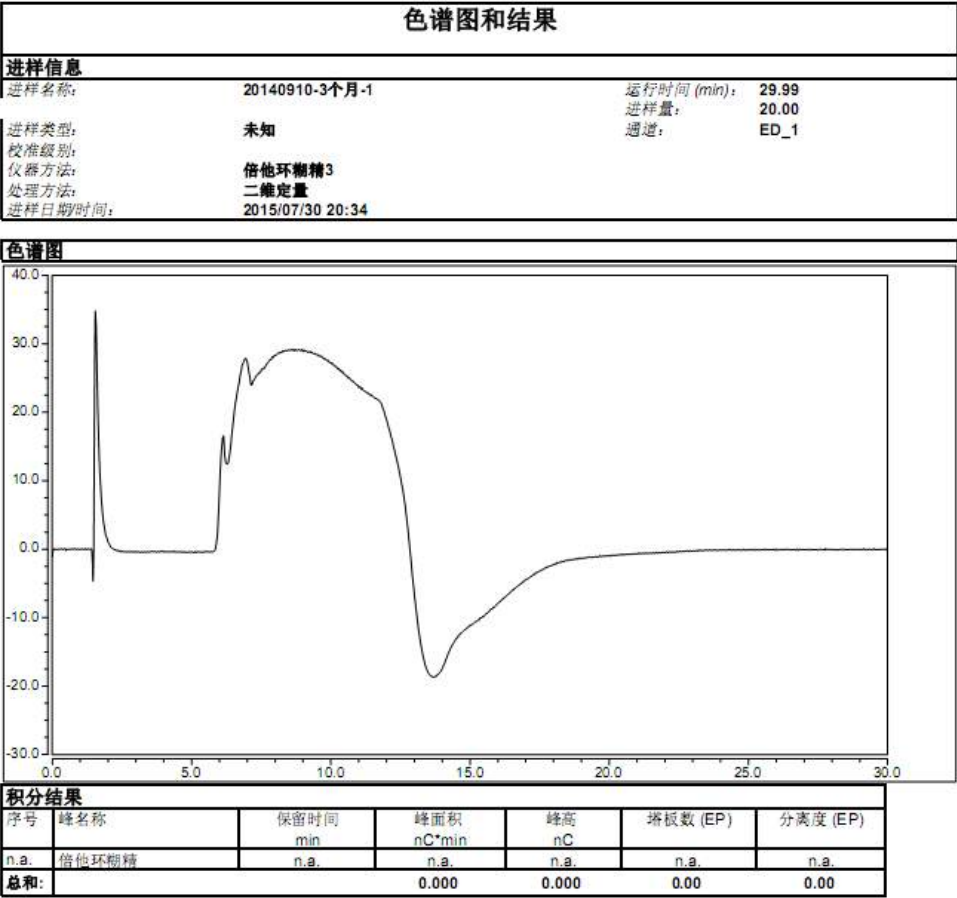
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-68 Long-term testing-Betadex (HPIC) -3month-20140910-1

仪器:CS-5000+ 序列:稳定性数据

页码 4 / 25



附图14-5-49 SECD长期3月倍他环糊精的测定图 (20140910-1)

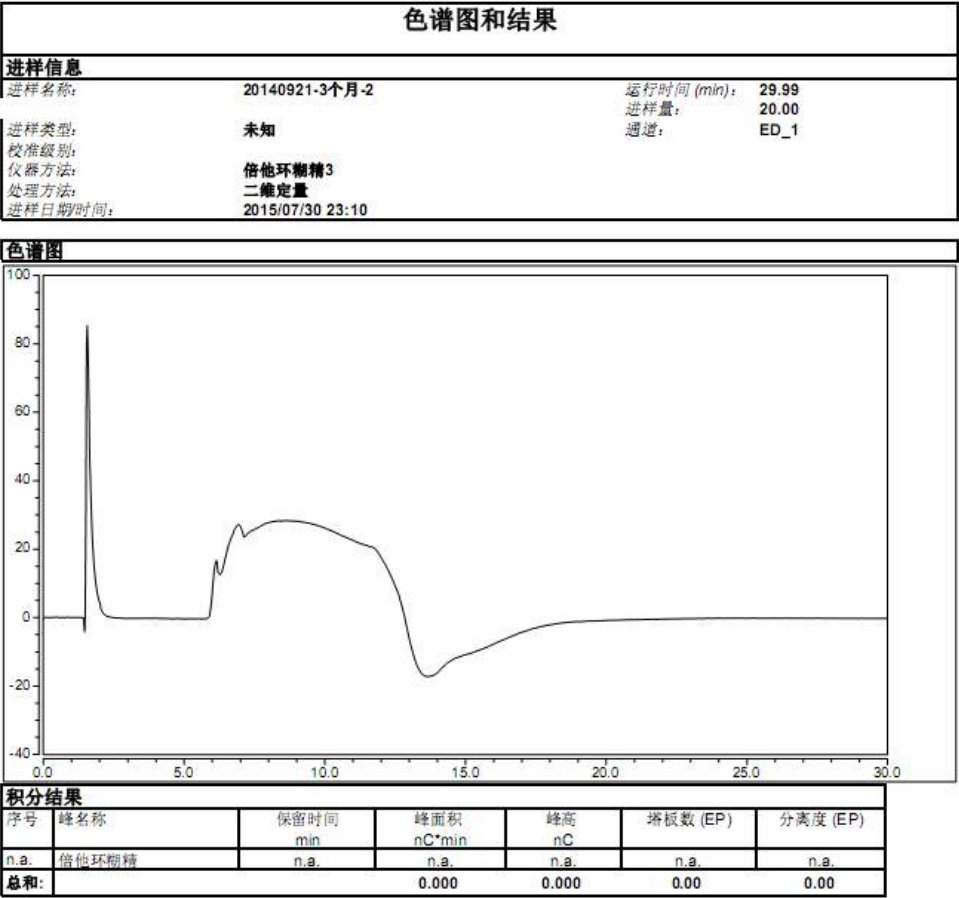
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-69 Long-term testing-Betadex (HPIC) -3month-20140921-2

仪器:CS-5000+ 序列:稳定性数据

页码 9 / 25



附图14-5-52 SBECD长期3月倍他环糊精的测定图 (20140921-2)

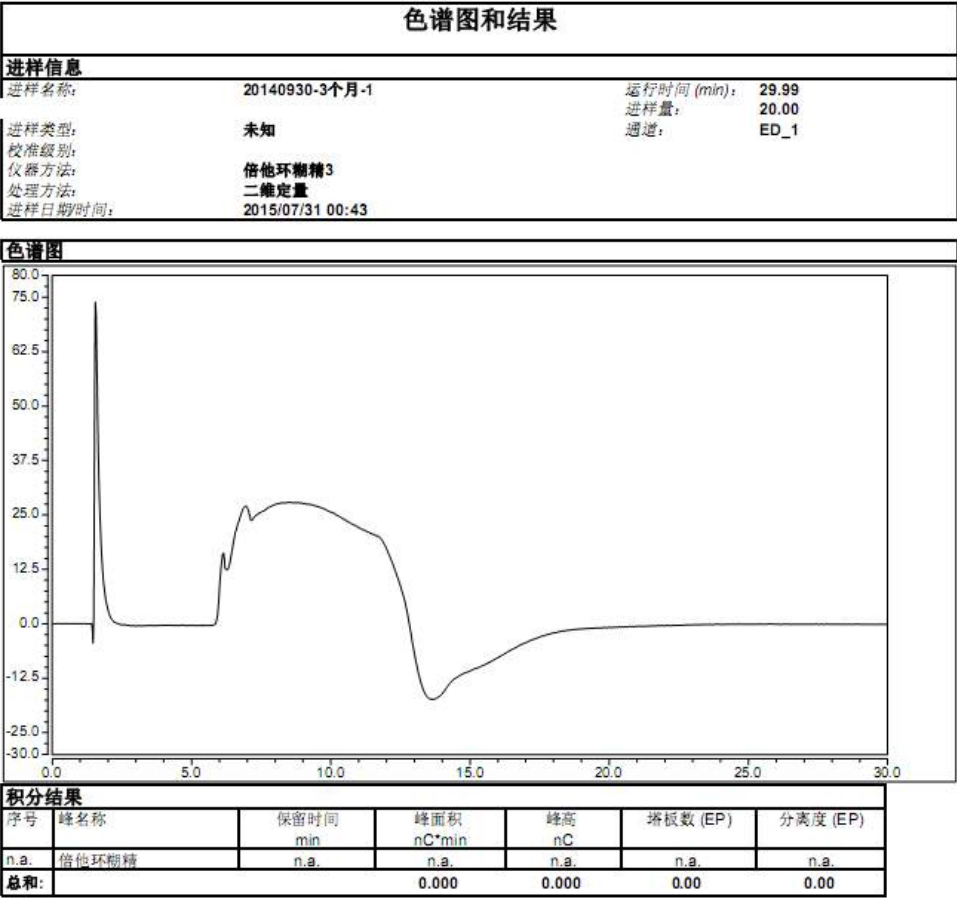
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-70 Long-term testing-Betadex (HPIC) -3month-20140930-1

仪器:CS-5000+ 序列:稳定性数据

页码:12/25



附图14-5-53 SEC长期3月倍他环糊精的测定图 (20140930-1)

Default:积分

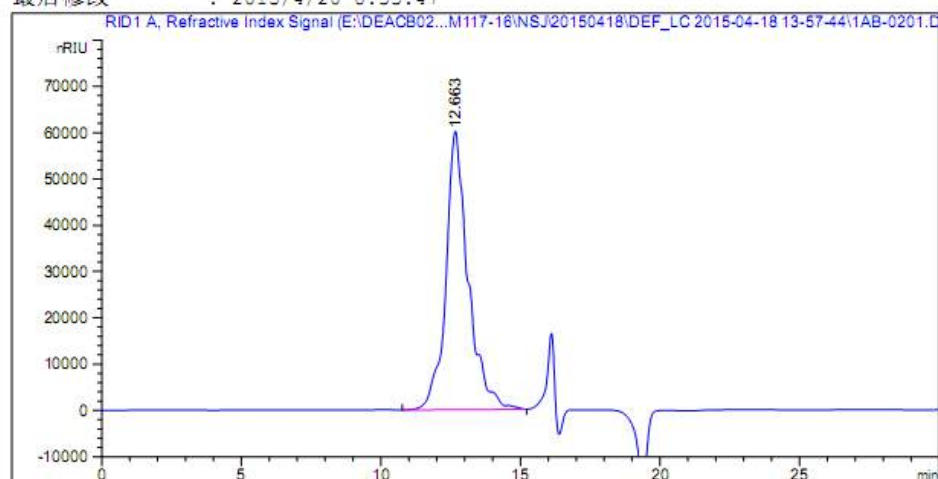
Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-71 Accelerated and long-term testing-Assay-6month-Reference solution 1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\1AB-0201.D

样品名称: DZ-1

```
=====
操作者      :                               序列行 :    2
仪器        : 1260-2                        位置   : P1-A-02
进样日期    : 2015/4/18 14:59:30            进样次数:    1
                                           进样量  : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M
最后修改    : 2015/4/18 13:57:44
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/4/20 8:33:47
=====
```



```
=====
                        面积百分比报告 (包含性能计算)
=====
```

```
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
12.663	-	3.33569e6	6.00715e4	0.71	0.7107	1761	-	-

```
=====
*** 报告结束 ***
=====
```

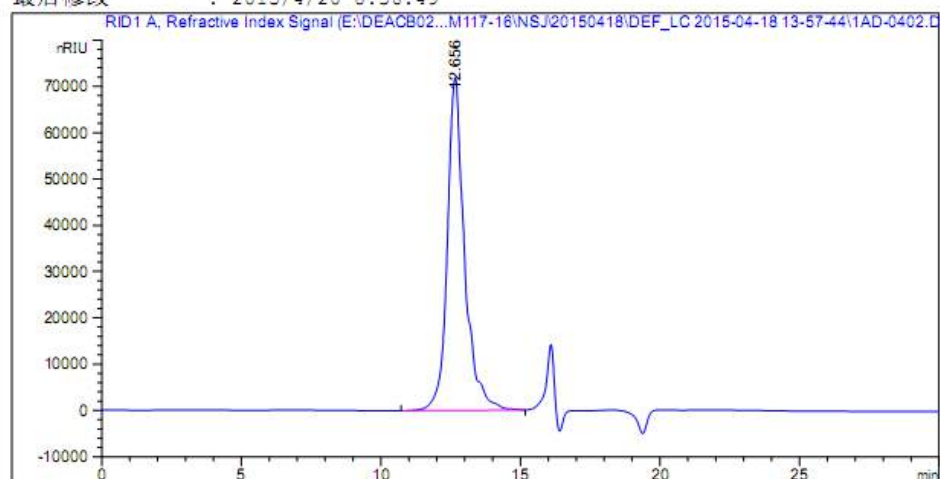
附图14-4-122

SBECD加速6月含量测定图 (加速6月、长期6月对照1-1)

Annex 3-S-72 Accelerated testing-Assay-6month-20140910-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\1AD-0402.D
样品名称: 20140910-40C-6M-1

```
=====
操作者      :                               序列行 :    4
仪器        : 1260-2                       位置   : P1-A-04
进样日期    : 2015/4/18 18:33:42           进样次数:    2
                                           进样量  : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M
最后修改    : 2015/4/18 16:22:37
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/4/20 8:36:49
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.656	BB	0.6237	3.09431e6	7.18115e4	100.0000

总量 : 3.09431e6 7.18115e4

=====
*** 报告结束 ***

附图14-4-129

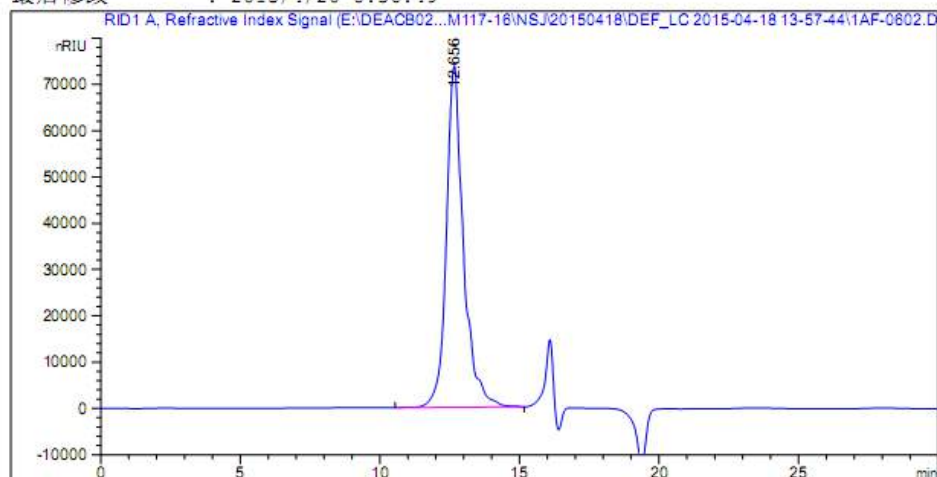
SBECD加速6月含量测定图 (20140910-1-2)

Annex 3-S-73 Accelerated testing-Assay-6month-20140921-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\1AF-0602.D
样品名称: 20140921-40C-6M-1

=====

操作者	:		序列行	:	6
仪器	:	1260-2	位置	:	P1-A-06
进样日期	:	2015/4/18 20:36:04	进样次数	:	2
			进样量	:	20.000 µl
采集方法	:	E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-SBEC-0.6.M			
最后修改	:	2015/4/18 16:22:37			
分析方法	:	E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-SBEC-0.6.M (序列方法)			
最后修改	:	2015/4/20 8:36:49			



=====

面积百分比报告

=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.656	BB	0.6205	3.15243e6	7.36222e4	100.0000

总量 : 3.15243e6 7.36223e4

*** 报告结束 ***

附图14-4-133

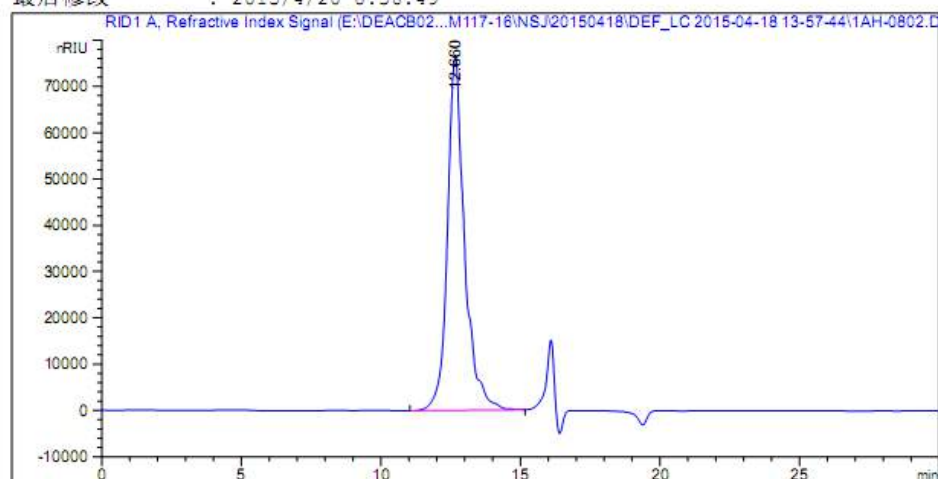
SBEC加速6月含量测定图 (20140921-1-2)

Annex 3-S-74 Accelerated testing-Assay-6month-20140930-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\1AH-0802.D
样品名称: 20140930-40C-6M-1

```
=====
操作者      :                               序列行 :    8
仪器        : 1260-2                        位置   : P1-A-08
进样日期    : 2015/4/18 22:38:28            进样次数:    2
                                           进样量  : 20.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M
最后修改    : 2015/4/18 16:22:37
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/4/20 8:36:49
=====
```



=====
面积百分比报告
=====

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.660	BB	0.6215	3.27488e6	7.63327e4	100.0000

总量 : 3.27488e6 7.63327e4

=====
*** 报告结束 ***

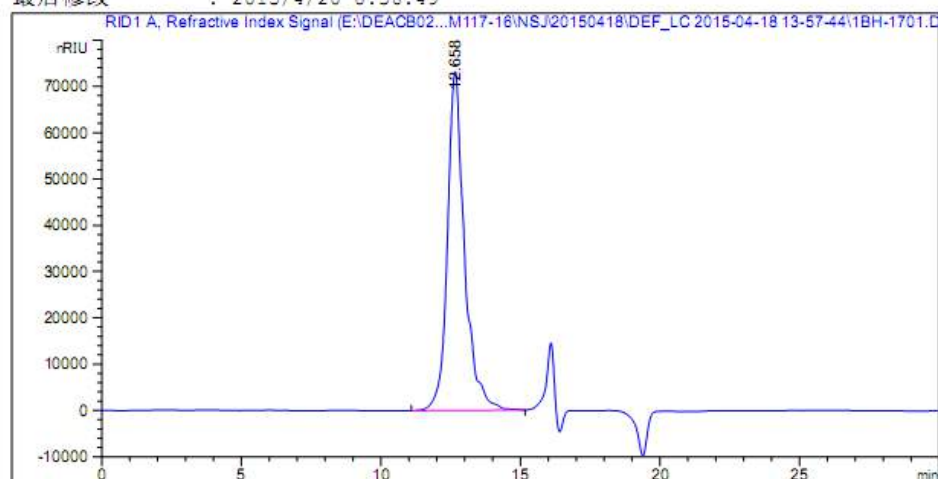
附图14-4-137

SBECD加速6月含量测定图 (20140930-1-2)

Annex 3-S-75 Long-term testing-Assay-6month-20140910-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\1BH-1701.D
样品名称: 20140910-25C-6M-2

```
=====
操作者      :                               序列行 :   17
仪器        : 1260-2                       位置   : P1-B-08
进样日期    : 2015/4/19 7:18:40           进样次数 :    1
                                           进样量  : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M
最后修改    : 2015/4/18 16:22:37
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/4/20 8:36:49
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.658	BB	0.6188	3.11768e6	7.30608e4	100.0000

总量 : 3.11768e6 7.30608e4

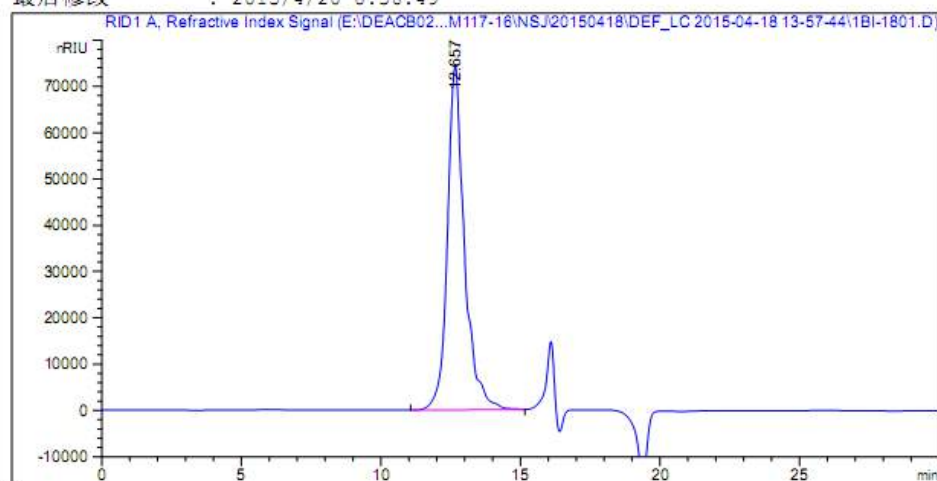
```
=====
*** 报告结束 ***
=====
```

附图14-5-27 SBECD长期6月含量测定图 (20140910-2-1)

Annex 3-S-76 Long-term testing-Assay-6month-20140921-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\1BI-1801.D
样品名称: 20140921-25C-6M-1

```
=====
操作者      :                               序列行 :   18
仪器        : 1260-2                       位置   : P1-B-09
进样日期    : 2015/4/19 8:19:50             进样次数:    1
                                           进样量  : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M
最后修改    : 2015/4/18 16:22:37
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/4/20 8:36:49
=====
```



=====
面积百分比报告
=====

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.657	BB	0.6203	3.17272e6	7.41232e4	100.0000

总量 : 3.17272e6 7.41232e4

=====
*** 报告结束 ***

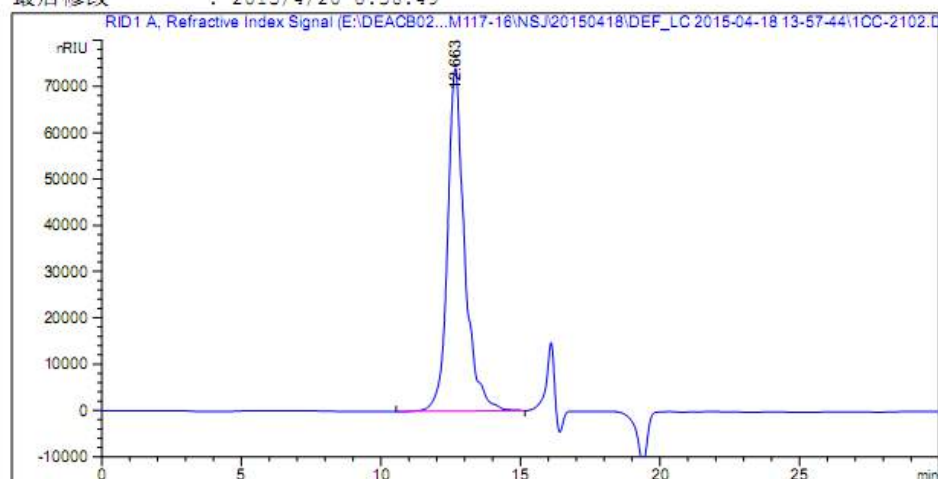
附图14-5-29 SBECD长期6月含量测定图 (20140921-1-1)

Annex 3-S-77 Long-term testing-Assay-6month-20140930-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\1CC-2102.D

样品名称: 20140930-25C-6M-2

```
=====
操作者      :                               序列行 :   21
仪器        : 1260-2                       位置   : PI-C-03
进样日期    : 2015/4/19 11:53:59           进样次数:    2
                                           进样量  : 20.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M
最后修改    : 2015/4/18 16:22:37
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150418\DEF_LC 2015-04-18 13-57-44\117-16-
              SBECD-0.6.M (序列方法)
最后修改    : 2015/4/20 8:36:49
=====
```



面积百分比报告

```
=====
排序      :      信号
乘积因子  :      1.0000
稀释因子  :      1.0000
内标中不使用乘积因子和稀释因子
=====
```

信号 1: RID1 A, Refractive Index Signal

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [nRIU*s]	峰高 [nRIU]	峰面积 %
1	12.663	BB	0.6205	3.16496e6	7.39134e4	100.0000

总量 : 3.16496e6 7.39134e4

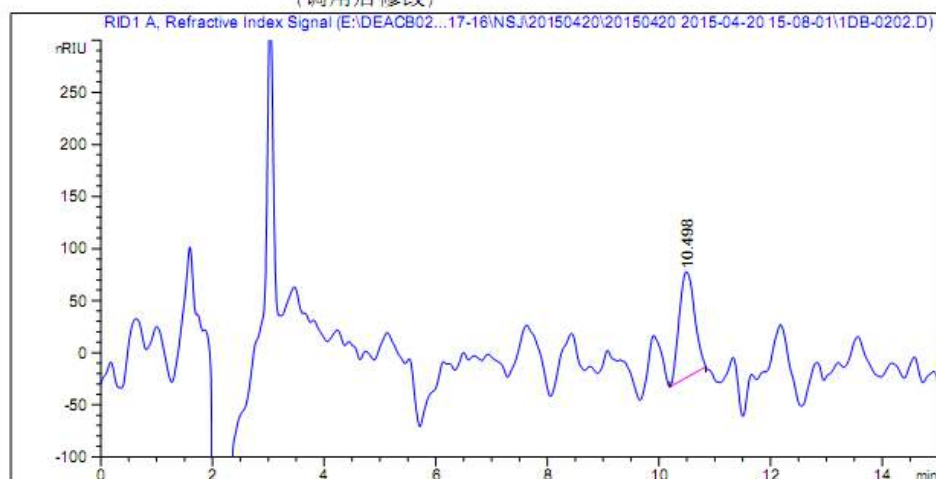
*** 报告结束 ***

附图14-5-36 SBECD长期6月含量测定图 (20140930-2-2)

Annex 3-S-78 Accelerated testing and long-term testing-Betadex-6month-Reference solution
1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\1DB-0202.D
样品名称: DZ-1

```
=====
操作者      :                               序列行 :    2
仪器        : 1260-2                         位置   : P1-D-02
进样日期    : 2015/4/20 16:10:33             进样次数 :    2
                                           进样量   : 10.000 µl
采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
最后修改    : 2015/4/20 15:49:29
              (调用后修改)
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
              (序列方法)
最后修改    : 2015/4/21 15:34:03 : Weijing
              (调用后修改)
=====
```



=====
面积百分比报告 (包含性能计算)
=====

乘积因子 : 1.0000
稀释因子 : 1.0000
内标中不使用乘积因子和稀释因子

信号 1: RID1 A, Refractive Index Signal

保留时间 [min]	k'	峰面积 [nRIU*s]	峰高 [nRIU]	对称 因子	峰宽 [min]	塔板数	分离度	选择性
10.498	-	1943.91736	101.20856	0.95	0.3096	6361	-	-

=====
*** 报告结束 ***

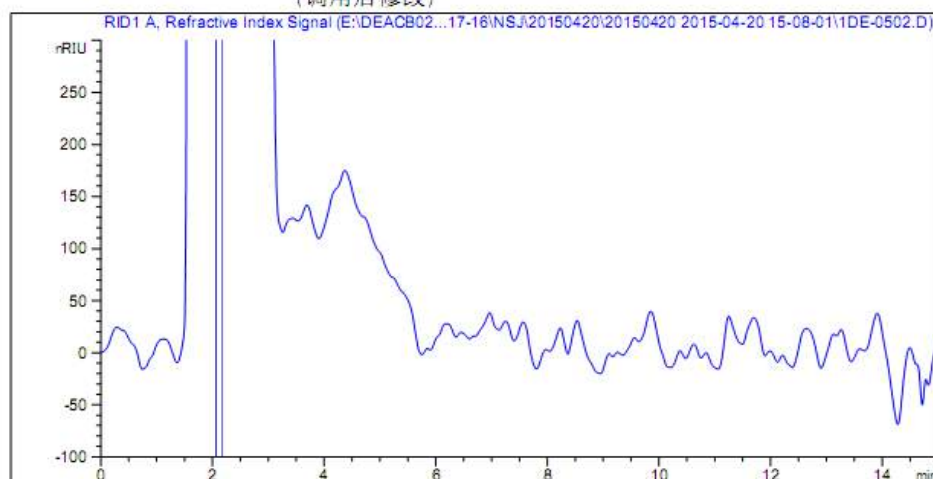
附图14-4-142 SBECD加速6月倍他环糊精测定图 (加速6月、长期6月对照1-2)

Annex 3-S-79 Accelerated testing-Betadex-6month-20140910-1-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\1DE-0502.D
样品名称: 20141910-40C-6M-1

```
=====
操作者      :                               序列行 :    5
仪器        : 1260-2                        位置   : Pl-D-05
进样日期    : 2015/4/20 19:32:05           进样次数 :    2
                                           进样量   : 10.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
最后修改    : 2015/4/20 19:06:44
              (调用后修改)
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
              (序列方法)
最后修改    : 2015/4/21 15:03:46 : Weijing
              (调用后修改)
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
*** 报告结束 ***
=====
```

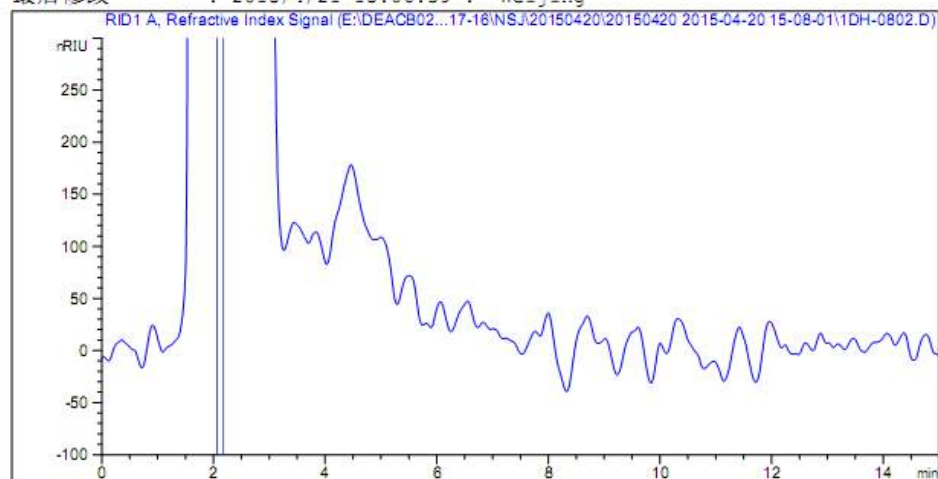
附图14-4-149 SBECD加速6月倍他环糊精测定图 (20140910-1-2)

Annex 3-S-80 Accelerated testing-Betadex-6month-20140921-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\1DH-0802.D
样品名称: 20140921-40C-6M-2

```
=====
操作者      :                               序列行 :    8
仪器        : 1260-2                        位置   : P1-D-08
进样日期    : 2015/4/20 21:51:36            进样次数 :    2
                                           进样量   : 10.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
最后修改    : 2015/4/20 19:44:57
              (调用后修改)
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
              (序列方法)
最后修改    : 2015/4/21 15:06:39 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
                        *** 报告结束 ***
=====
```

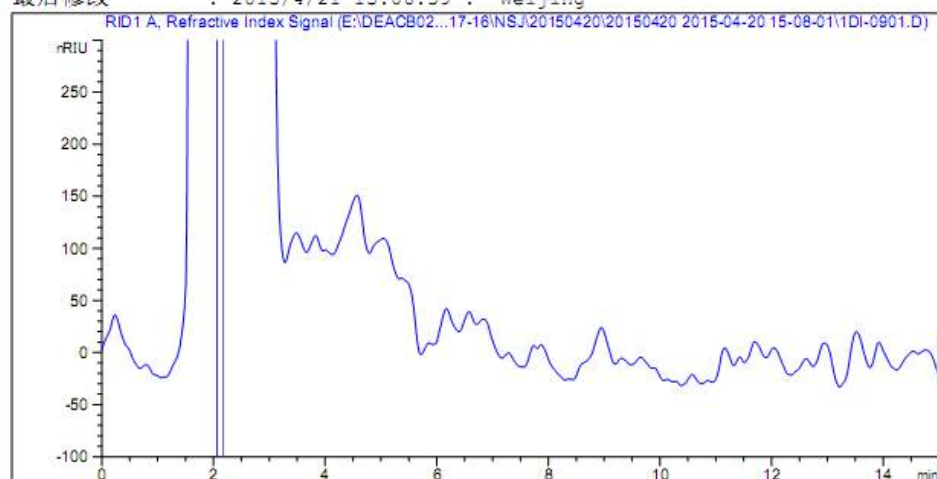
附图14-4-154 SBECD加速6月倍他环糊精测定图 (20140921-2-1)

Annex 3-S-81 Accelerated testing-Betadex-6month-20140930-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\1DI-0901.D
样品名称: 20140930-40C-6M-1

```
=====
操作者      :                               序列行 :    9
仪器        : 1260-2                        位置   : Pl-D-09
进样日期    : 2015/4/20 22:22:38            进样次数 :    1
                                           进样量   : 10.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
最后修改    : 2015/4/20 19:44:57
              (调用后修改)
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
              (序列方法)
最后修改    : 2015/4/21 15:06:39 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
                        *** 报告结束 ***
=====
```

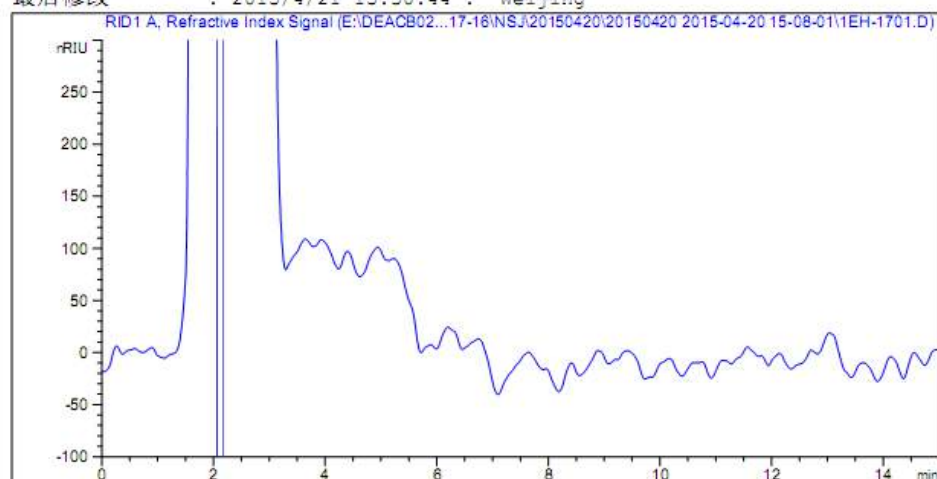
附图14-4-156 SBECD加速6月倍他环糊精测定图 (20140930-1-1)

Annex 3-S-82 Long-term testing-Betadex-6month-20140910-1-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\1EH-1701.D
样品名称: 20140910-25C-6M-1

```
=====
操作者      :                               序列行 :   17
仪器        : 1260-2                       位置    : Pl-E-08
进样日期    : 2015/4/21 4:34:21             进样次数 :    1
                                           进样量   : 10.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
最后修改    : 2015/4/20 19:44:57
              (调用后修改)
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
              (序列方法)
最后修改    : 2015/4/21 15:30:44 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
                        *** 报告结束 ***
=====
```

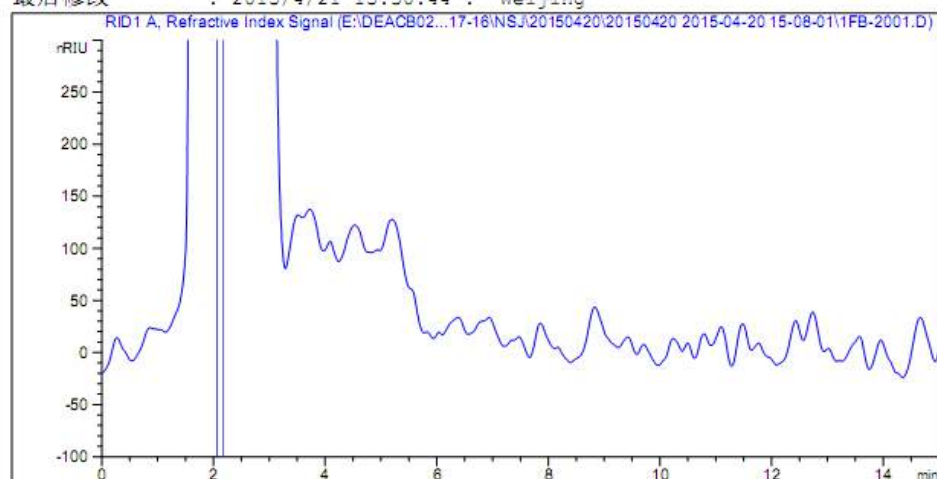
附图14-5-37 SBECD长期6月倍他环糊精测定图 (20140910-1-1)

Annex 3-S-83 Long-term testing-Betadex-6month-20140921-2-1

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\1FB-2001.D
样品名称: 20140921-25C-6M-2

```
=====
操作者      :                               序列行 :   20
仪器        : 1260-2                       位置    : Pl-F-02
进样日期    : 2015/4/21 6:53:48             进样次数 :    1
                                           进样量   : 10.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
最后修改    : 2015/4/20 19:44:57
              (调用后修改)
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
              (序列方法)
最后修改    : 2015/4/21 15:30:44 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序          :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

```
=====
                        *** 报告结束 ***
=====
```

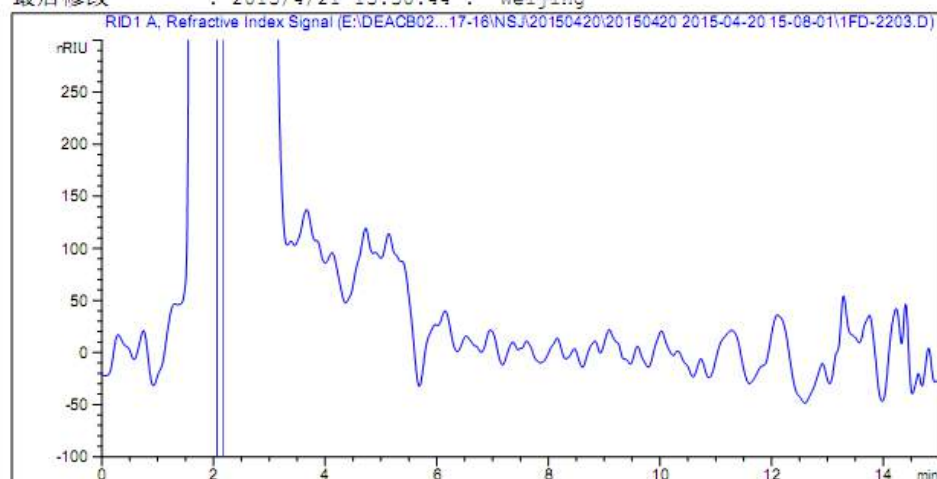
附图14-5-43 SBECD长期6月倍他环糊精测定图 (20140921-2-1)

Annex 3-S-84 Long-term testing-Betadex-6month-20140930-2-2

数据文件: E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\1FD-2203.D
样品名称: 20140930-25C-6M-2

```
=====
操作者      :                               序列行 :   22
仪器        :   1260-2                      位置   : Pl-F-04
进样日期    :   2015/4/21 8:57:41           进样次数 :    3
                                           进样量   : 10.000 µl

采集方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
最后修改    :   2015/4/21 8:36:29
              (调用后修改)
分析方法    : E:\DEACB02694\SIM117-16\NSJ\20150420\20150420 2015-04-20 15-08-01\B CD T3.M
              (序列方法)
最后修改    :   2015/4/21 15:30:44 : Weijing
=====
```



```
=====
                        面积百分比报告
=====
```

```
排序      :      信号
乘积因子      :      1.0000
稀释因子      :      1.0000
内标中不使用乘积因子和稀释因子
```

未发现峰

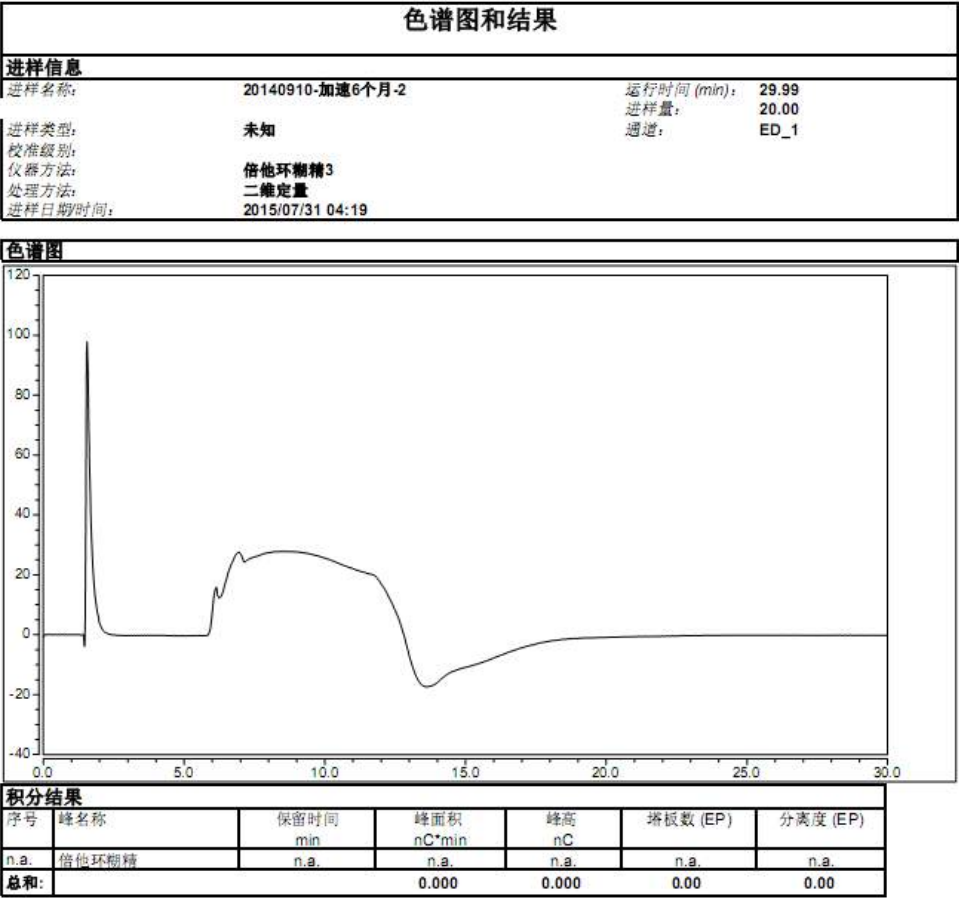
```
=====
                        *** 报告结束 ***
=====
```

附图14-5-48 SBECD长期6月倍他环糊精测定图 (20140930-2-2)

Annex 3-S-85 Accelerated testing-Betadex-6month-20140910-2

仪器:CS-5000+ 序列:稳定性数据

页码:19 / 25



附图14-4-16.9 SRECD加速6月倍他环糊精的测定图 (20140910-2)

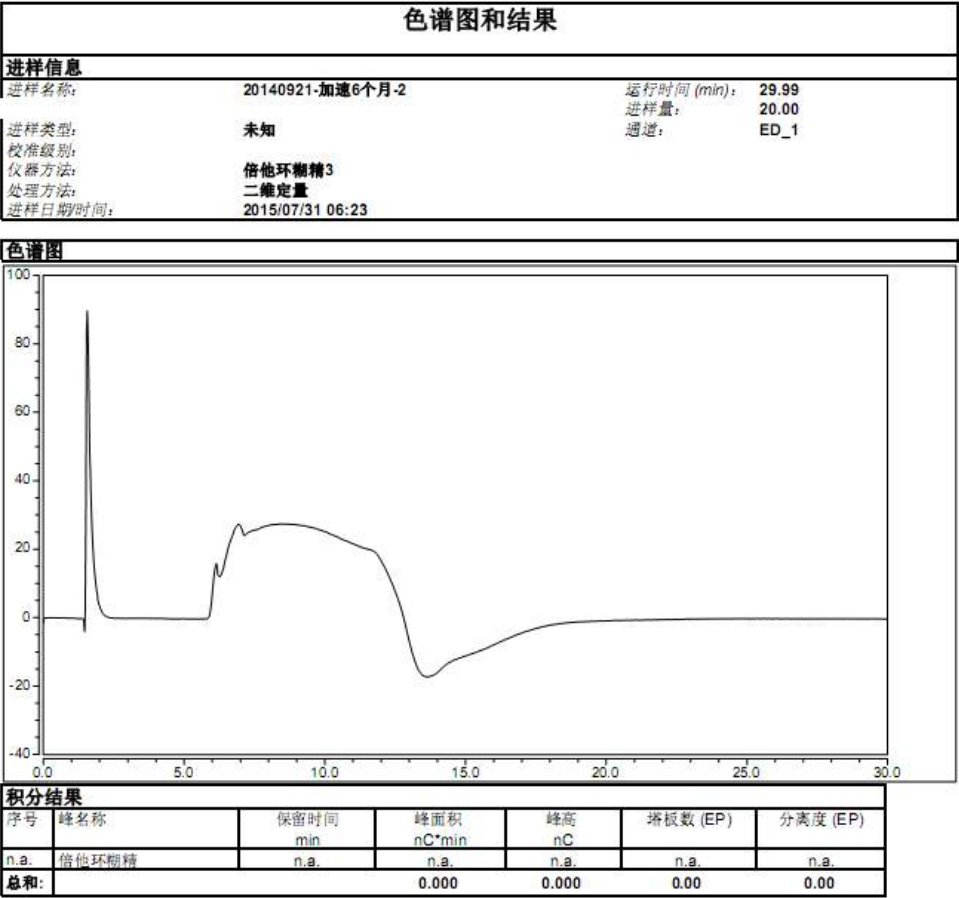
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-86 Accelerated testing-Betadex-6month-20140921-2

仪器:CS-5000+ 序列:稳定性数据

页码 23 / 25



附图14-4-17.1 SECD加速6 月倍他环糊精的测定图 (2 0 14 0 9 2 1-2)

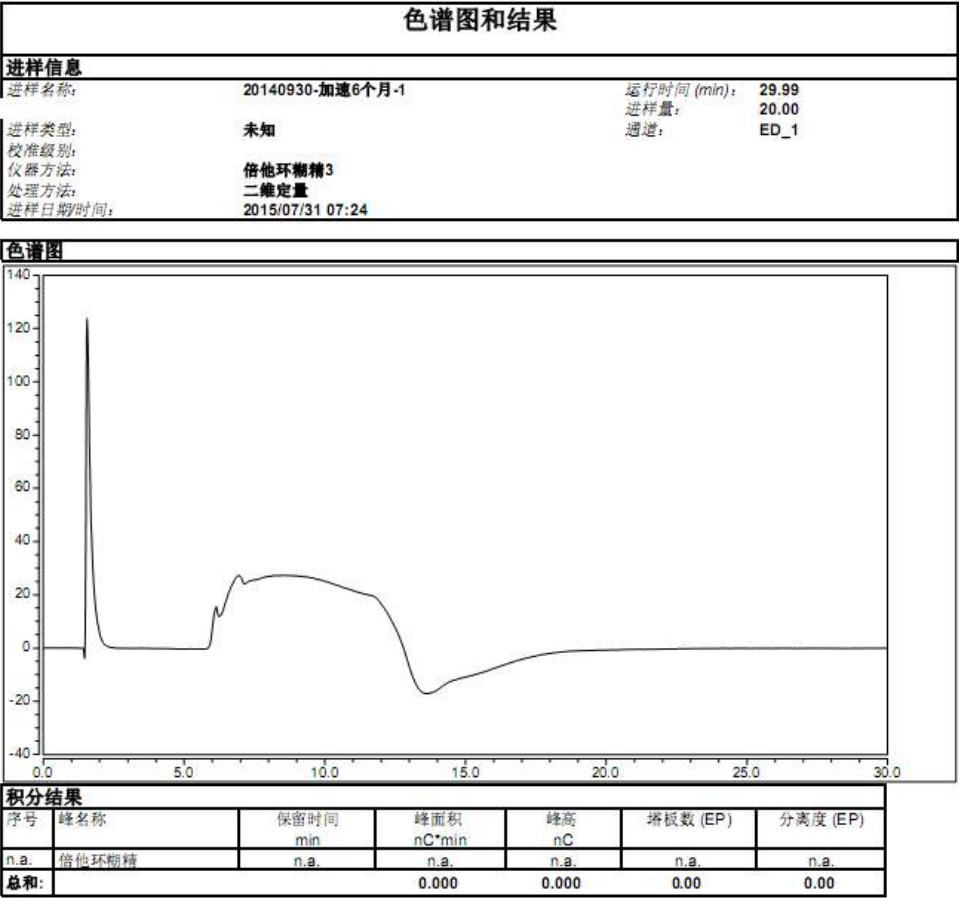
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-87 Accelerated testing-Betadex-6month-20140930-1

仪器:CS-5000+ 序列:稳定性数据

页码 25 / 25



附图14-4-172 SIECD加速6 月倍他环糊精的测定图 (2 0 14 0 9 3 0 -

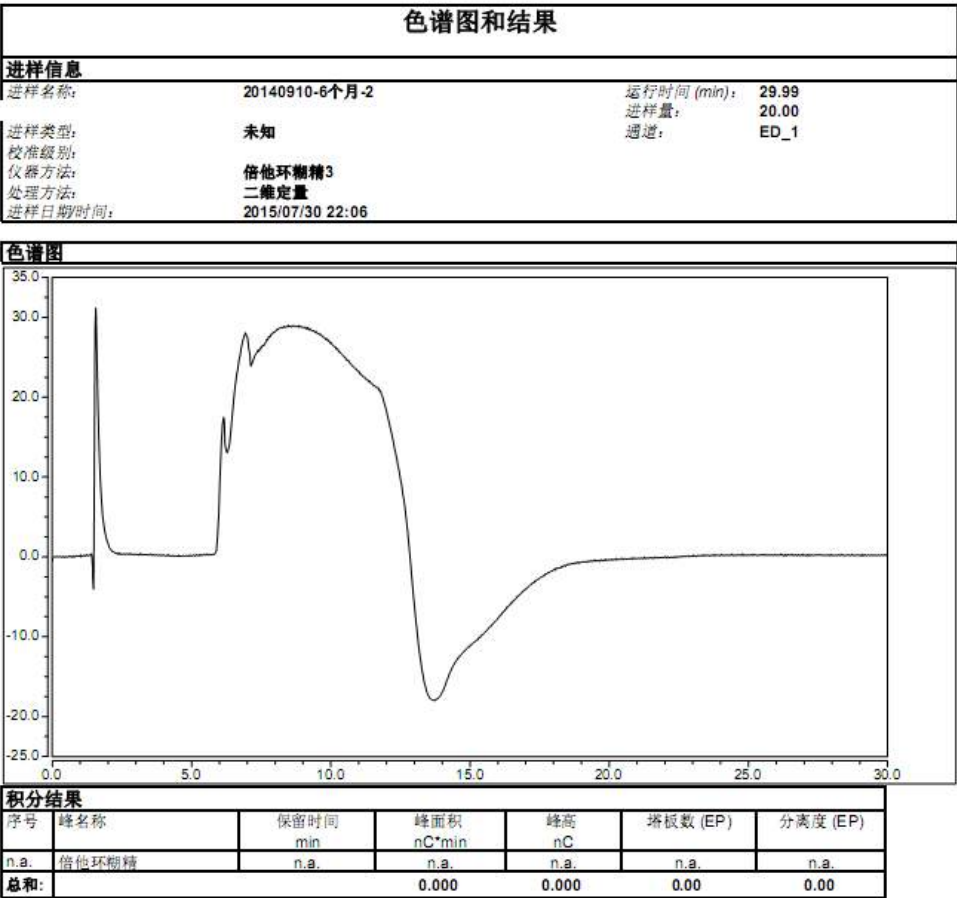
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-88 Long-term testing-Betadex-6month-20140910-2

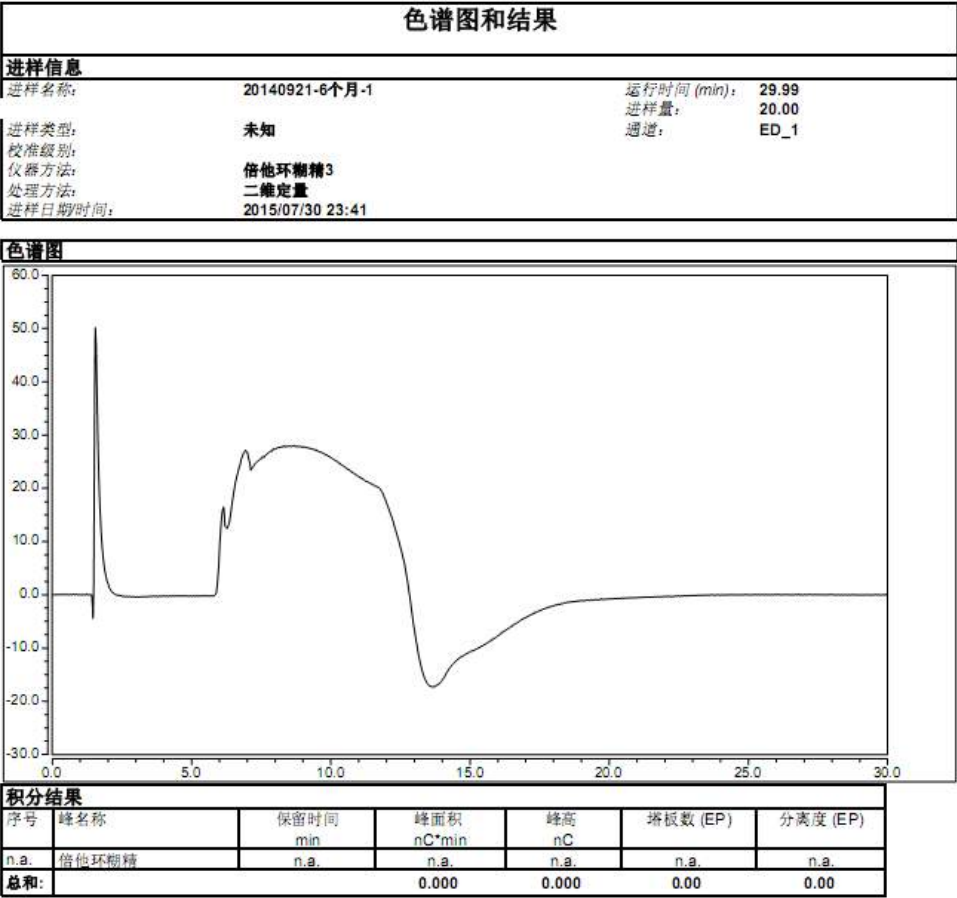
仪器:ICS-5000+ 序列:稳定性数据

页码 7 / 25



附图14-5-56 S3ECD长期6月倍他环糊精的测定图 (20140910-2)

Annex 3-S-89 Long-term testing-Betadex-6month-20140921-1

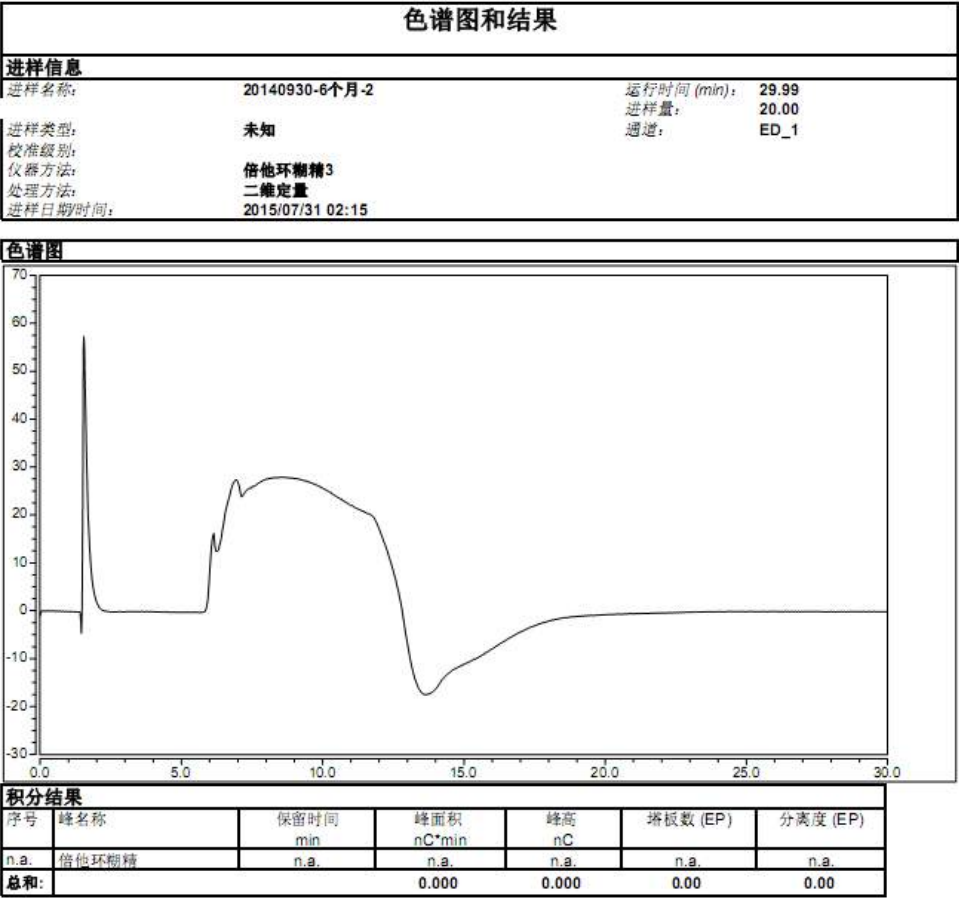


附图14-5-57 SBECD长期6月倍他环糊精的测定图 (20140921-1)

Annex 3-S-90 Long-term testing-Betadex-6month-20140930-2

仪器:CS-5000+ 序列:稳定性数据

页码:15 / 25



附图14-5-60 SBECD长期6月倍他环糊精的测定图 (20140930-2)

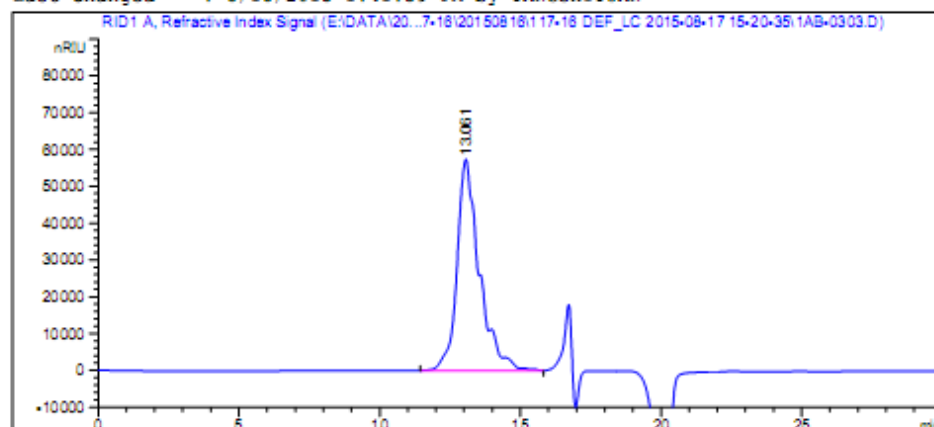
Default:积分

Chromleon (c) Dionex
版本 7.2.1.5537

Annex 3-S-91 Long-term testing-Assay-9month-Reference solution 1-3

Data File E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-17 15-20-35\1AB-0303.D
Sample Name: DZ1

```
-----  
Acq. Operator   : TXL                               Seq. Line :    3  
Acq. Instrument : Instrument 1                       Location  : P1-A-02  
Injection Date  : 8/17/2015 5:24:22 PM              Inj       :    3  
                                                    Inj Volume : 20.0 µl  
Acq. Method     : E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-17 15-20-35\117-16-HL.M  
Last changed    : 8/16/2015 4:07:42 PM by TXL  
Analysis Method : E:\DATA\201508\117-16\METHOD\117-16-HL.M  
Last changed    : 8/18/2015 1:41:39 PM by YANGSHIYUAN  
-----
```



Area Percent Report

```
Sorted By      :      Signal  
Multiplier:    :      1.0000  
Dilution:      :      1.0000  
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: RID1 A, Refractive Index Signal

Peak #	RetTime [min]	Type	Width [min]	Area [nRIU*s]	Height [nRIU]	Area %
1	13.061	BV	0.7592	3.18158e6	5.73820e4	100.0000

Totals : 3.18158e6 5.73820e4

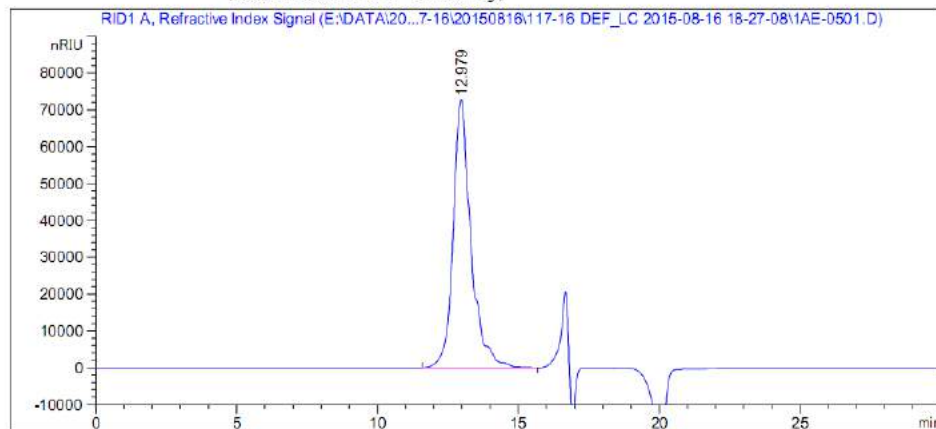
*** End of Report ***

附图14-5-6.6 SECD长期9月含量测定图(对照1-3)

Annex 3-S-92 Long-term testing-Assay-9month-20140910-2-1

Data File E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-16 18-27-08\1AE-0501.D
Sample Name: 20140910-25C-2

```
=====
Acq. Operator   : TXL                               Seq. Line :    5
Acq. Instrument : Instrument 1                       Location  : P1-A-05
Injection Date  : 8/17/2015 2:09:11 AM              Inj       :    1
                                                    Inj Volume: 20.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-16 18-27-08\117-16-HL.M
Last changed    : 8/16/2015 4:07:42 PM by TXL
Analysis Method : E:\DATA\201508\117-16\METHOD\117-16-HL.M
Last changed    : 8/18/2015 2:02:14 PM by YANGSHIYUAN
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: RID1 A, Refractive Index Signal

Peak #	RetTime [min]	Type	Width [min]	Area [nRIU*s]	Height [nRIU]	Area %
1	12.979	BV	0.6368	3.21135e6	7.26359e4	100.0000

Totals : 3.21135e6 7.26359e4

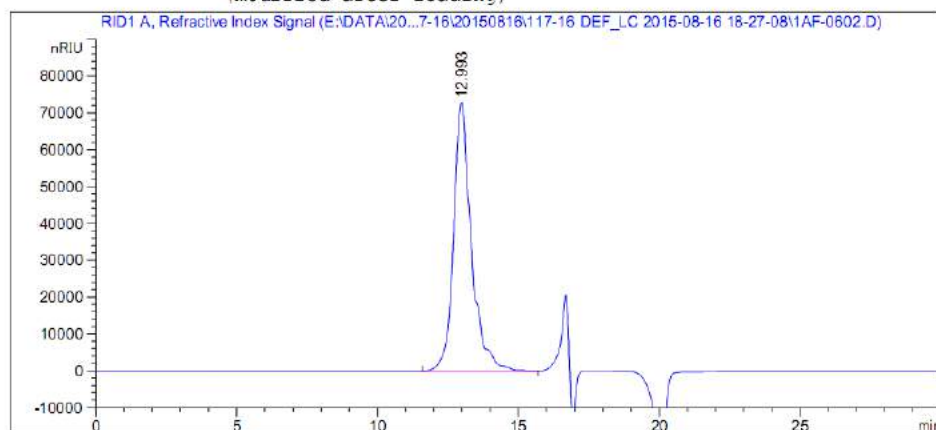
```
=====
*** End of Report ***
=====
```

附图14-5-72 SECD长期9月倍他环糊精测定图 (20140910-2-1)

Annex 3-S-93 Long-term testing-Assay-9month-20140921-1-2

Data File E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-16 18-27-08\1AF-0602.D
Sample Name: 20140921-25C-1

```
=====
Acq. Operator   : TXL                      Seq. Line :    6
Acq. Instrument : Instrument 1              Location  : P1-A-06
Injection Date  : 8/17/2015 3:41:26 AM      Inj       :    2
                                           Inj Volume: 20.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-16 18-27-08\117-16-HL.M
Last changed    : 8/16/2015 4:07:42 PM by TXL
Analysis Method : E:\DATA\201508\117-16\METHOD\117-16-HL.M
Last changed    : 8/18/2015 2:02:14 PM by YANGSHIYUAN
                  (modified after loading)
=====
```



=====
Area Percent Report
=====

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: RID1 A, Refractive Index Signal

Peak #	RetTime [min]	Type	Width [min]	Area [nRIU*s]	Height [nRIU]	Area %
1	12.993	BV	0.6289	3.23389e6	7.30373e4	100.0000

Totals : 3.23389e6 7.30373e4

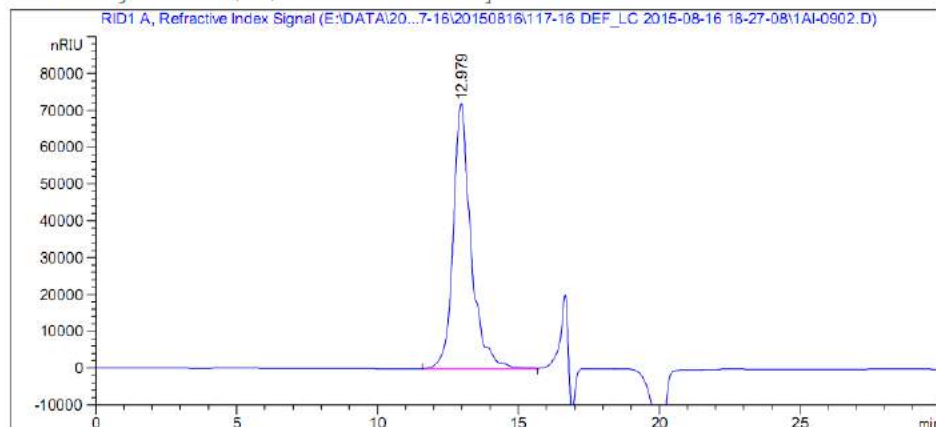
=====
*** End of Report ***

附图14-5-75 SBECD长期9月倍他环糊精测定图 (20140921-1-2)

Annex 3-S-94 Long-term testing-Assay-9month-20140930-2-2

Data File E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-16 18-27-08\1AI-0902.D
Sample Name: 20140930-25C-2

```
=====
Acq. Operator   : TXL                      Seq. Line :    9
Acq. Instrument : Instrument 1              Location  : P1-A-09
Injection Date  : 8/17/2015 6:46:04 AM      Inj       :    2
                                           Inj Volume: 20.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150816\117-16 DEF_LC 2015-08-16 18-27-08\117-16-HL.M
Last changed    : 8/16/2015 4:07:42 PM by TXL
Analysis Method : E:\DATA\201508\117-16\METHOD\117-16-HL.M
Last changed    : 8/18/2015 1:41:39 PM by YANGSHIYUAN
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: RID1 A, Refractive Index Signal

Peak #	RetTime [min]	Type	Width [min]	Area [nRIU*s]	Height [nRIU]	Area %
1	12.979	BV	0.6395	3.19216e6	7.18153e4	100.0000

Totals : 3.19216e6 7.18153e4

```
=====
*** End of Report ***
=====
```

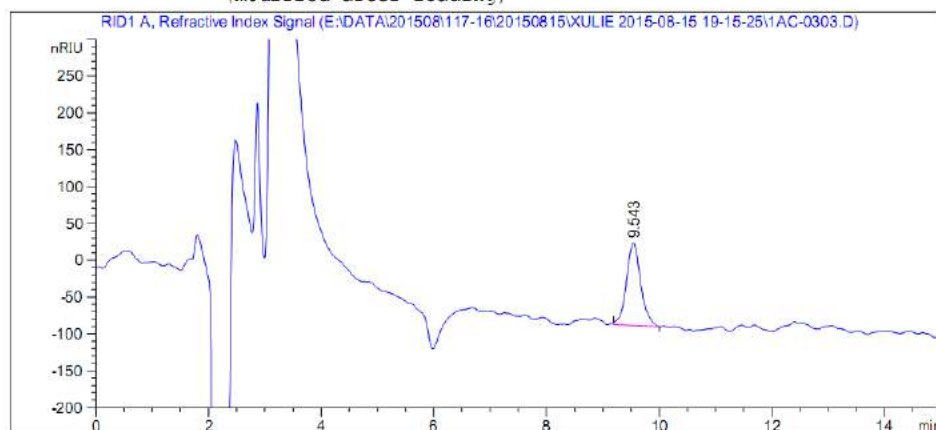
附图14-5-8 1 SECD长期9月倍他环糊精测定图 (20140930-2-)

Annex 3-S-95 Long-term testing-Betadex-9month-Reference solution 1-3

Data File E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\1AC-0303.D

Sample Name: DZ1

```
=====
Acq. Operator   : TXL                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : P1-A-03
Injection Date  : 8/15/2015 10:54:55 PM     Inj       :    3
                                           Inj Volume: 10.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG.M
Last changed    : 8/15/2015 5:11:07 PM by TXL
Analysis Method : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG-JF.M
Last changed    : 8/16/2015 11:37:35 AM by TXL
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: RID1 A, Refractive Index Signal

Peak #	RetTime [min]	Type	Width [min]	Area [nRIU*s]	Height [nRIU]	Area %
1	9.543	BBA	0.2758	1980.65063	111.78770	100.0000

```
Totals :                      1980.65063  111.78770
```

```
=====
*** End of Report ***
=====
```

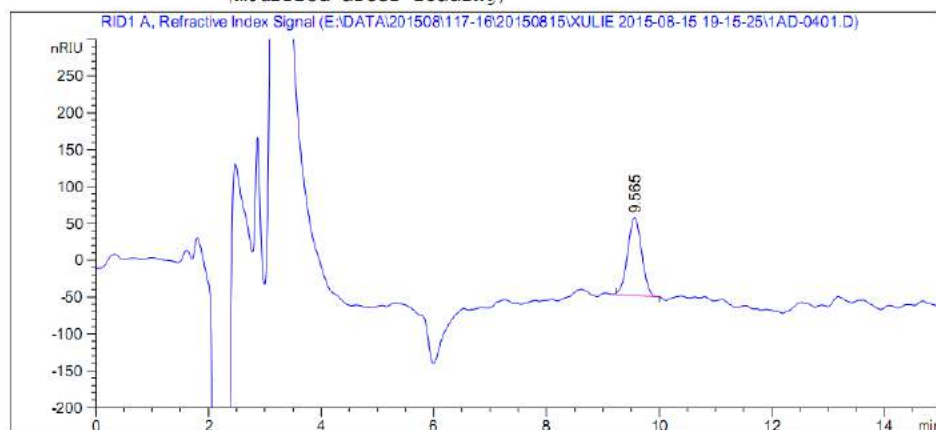
附图14-5-8 8 SECD长期9月倍他环糊精测定图(对照1-3)

Annex 3-S-96 Long-term testing-Betadex-9month-Reference solution 2-1

Data File E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\1AD-0401.D

Sample Name: DZ2

```
=====
Acq. Operator   : TXL                      Seq. Line :    4
Acq. Instrument : Instrument 1              Location  : P1-A-04
Injection Date  : 8/15/2015 11:41:51 PM    Inj       :    1
                                           Inj Volume: 10.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG.M
Last changed    : 8/15/2015 5:11:07 PM by TXL
Analysis Method : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG-JF.M
Last changed    : 8/16/2015 11:37:35 AM by TXL
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: RID1 A, Refractive Index Signal

Peak #	RetTime [min]	Type	Width [min]	Area [nRIU*s]	Height [nRIU]	Area %
1	9.565	BBA	0.2800	1827.79773	105.44226	100.0000

```
Totals :                      1827.79773  105.44226
```

```
=====
*** End of Report ***
=====
```

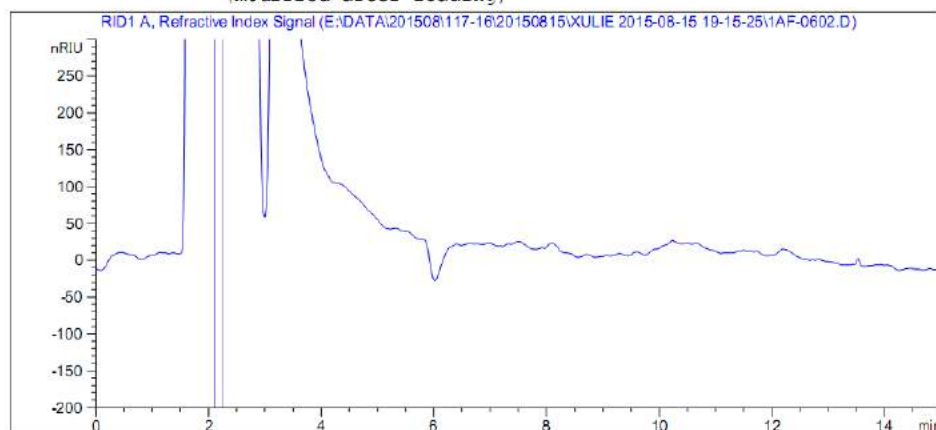
附图14-5-91 SECD长期9月倍他环糊精测定图(对照2-1)

Annex 3-S-97 Long-term testing-Betadex-9month-20140910-1-2

Data File E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\1AF-0602.D

Sample Name: 20140910-25C-9M-1

```
=====
Acq. Operator   : TXL                      Seq. Line :    6
Acq. Instrument : Instrument 1              Location  : P1-A-06
Injection Date  : 8/16/2015 1:47:02 AM      Inj       :    2
                                           Inj Volume: 10.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG.M
Last changed    : 8/15/2015 5:11:07 PM by TXL
Analysis Method : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG-JF.M
Last changed    : 8/16/2015 11:37:35 AM by TXL
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

No peaks found

```
=====
*** End of Report ***
=====
```

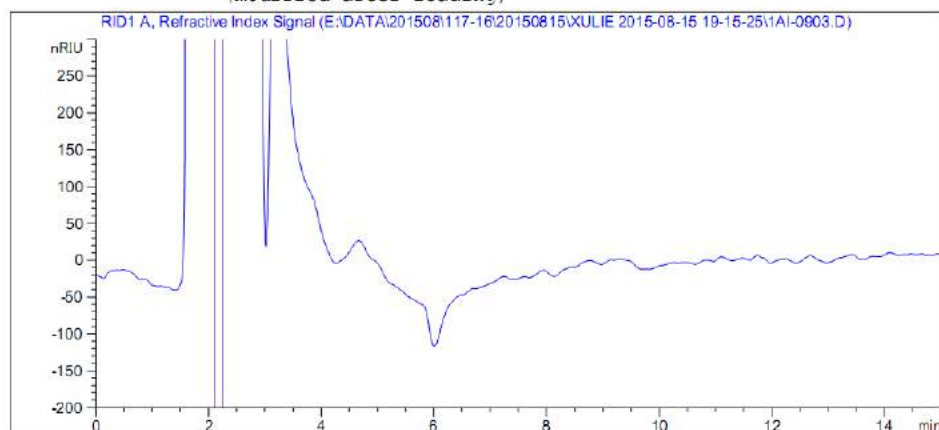
附图14-5-97 SECD长期9月倍他环糊精测定图 (20140910-1-2)

Annex 3-S-98 Long-term testing-Betadex-9month-20140921-2-3

Data File E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\1AI-0903.D

Sample Name: 20140921-25C-9M-2

```
=====
Acq. Operator   : TXL                      Seq. Line :    9
Acq. Instrument : Instrument 1              Location  : P1-A-09
Injection Date  : 8/16/2015 4:23:34 AM      Inj       :    3
                                           Inj Volume: 10.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG.M
Last changed    : 8/15/2015 5:11:07 PM by TXL
Analysis Method : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG-JF.M
Last changed    : 8/16/2015 11:37:35 AM by TXL
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

No peaks found

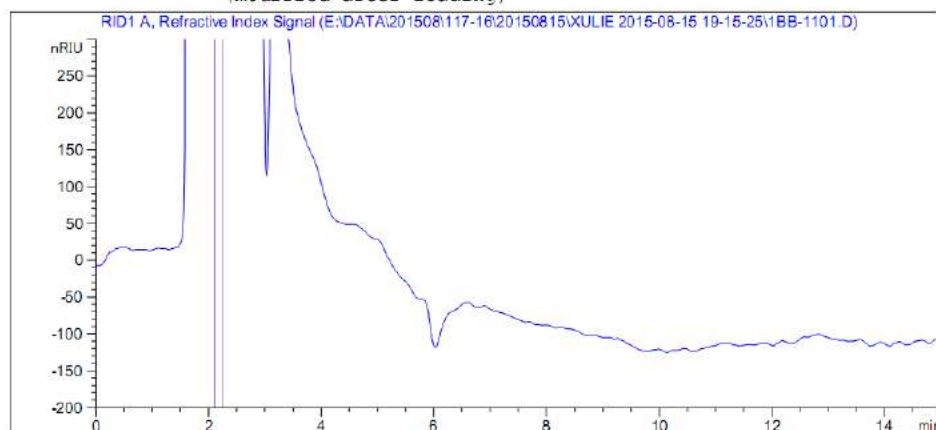
```
=====
*** End of Report ***
=====
```

附图14-5-107 SECD长期9月倍他环糊精测定图(20140921-2-)

Annex 3-S-99 Long-term testing-Betadex-9month-20140930-2-1

Data File E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\1BB-1101.D
Sample Name: 20140930-25C-9M-2

```
=====
Acq. Operator   : TXL                      Seq. Line :   11
Acq. Instrument : Instrument 1             Location  : P1-B-02
Injection Date  : 8/16/2015 5:26:08 AM     Inj       :    1
                                           Inj Volume: 10.0 µl
Acq. Method     : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG.M
Last changed    : 8/15/2015 5:11:07 PM by TXL
Analysis Method : E:\DATA\201508\117-16\20150815\XULIE 2015-08-15 19-15-25\117-16-YG-JF.M
Last changed    : 8/16/2015 11:37:35 AM by TXL
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

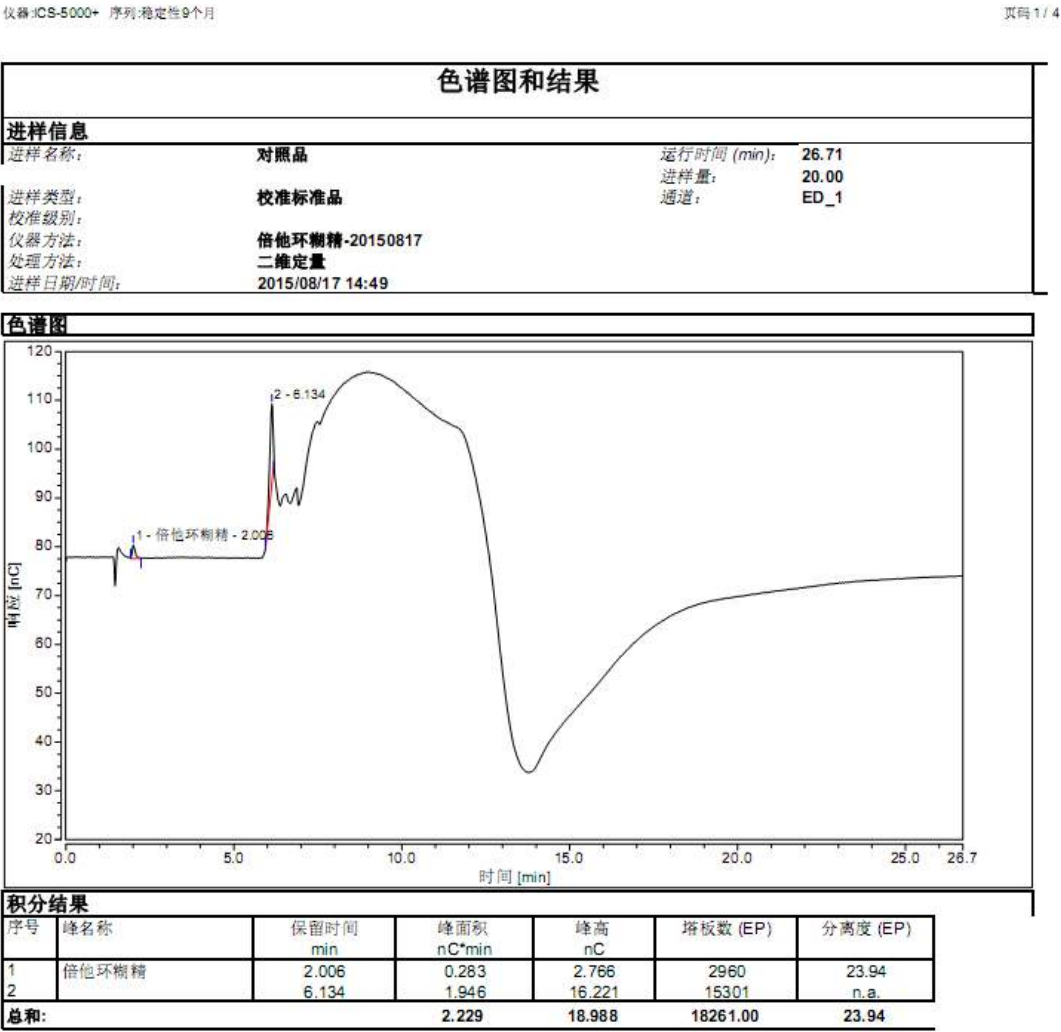
```
Sorted By      :      Signal
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

No peaks found

```
=====
*** End of Report ***
=====
```

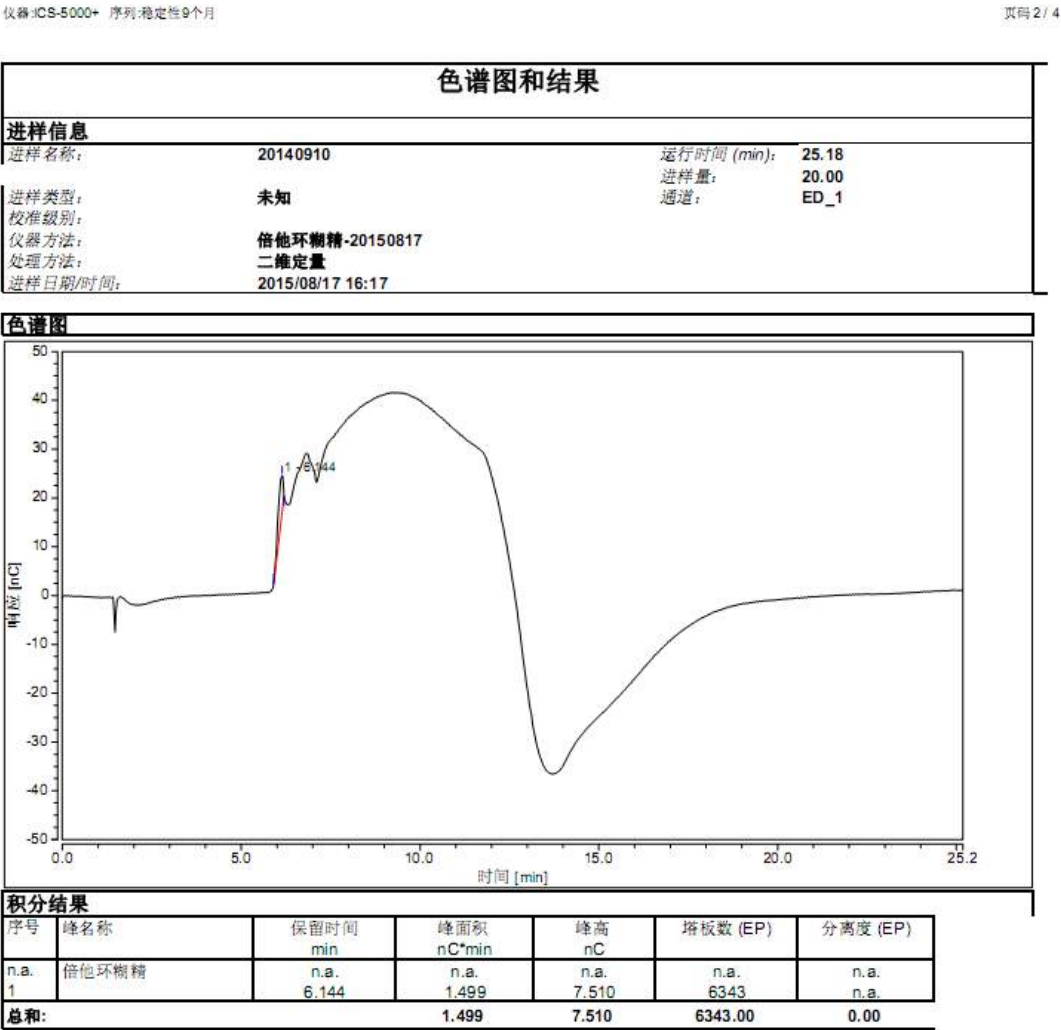
附图14-5-111 SECD长期9月倍他环糊精测定图(20140930-2-)

Annex 3-S-100 Long-term testing-Betadex (HPIC) -9month-Reference solution



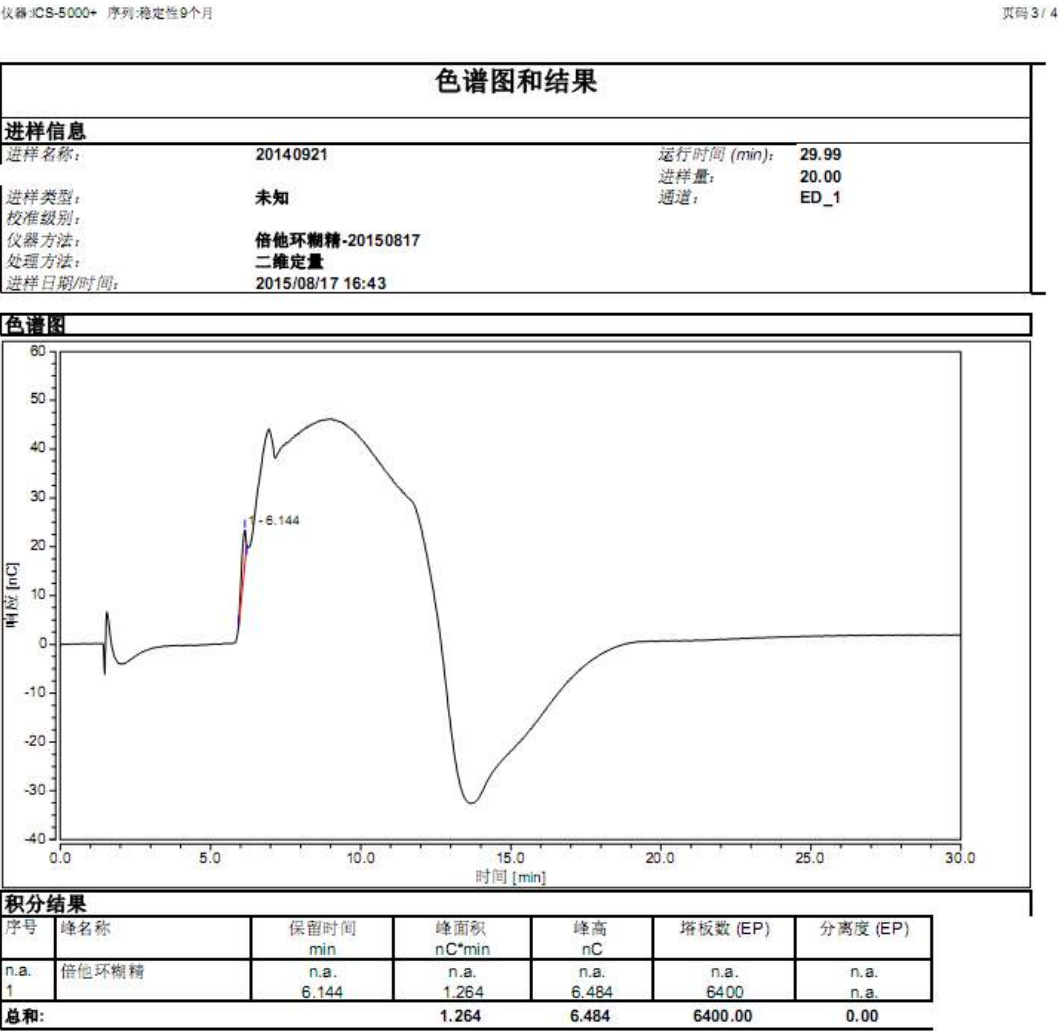
附图14-5-114 SBECD长期9月倍他环糊精测定图（对照）

Annex 3-S-101 Long-term testing-Betadex (HPIC) -9month-20140910



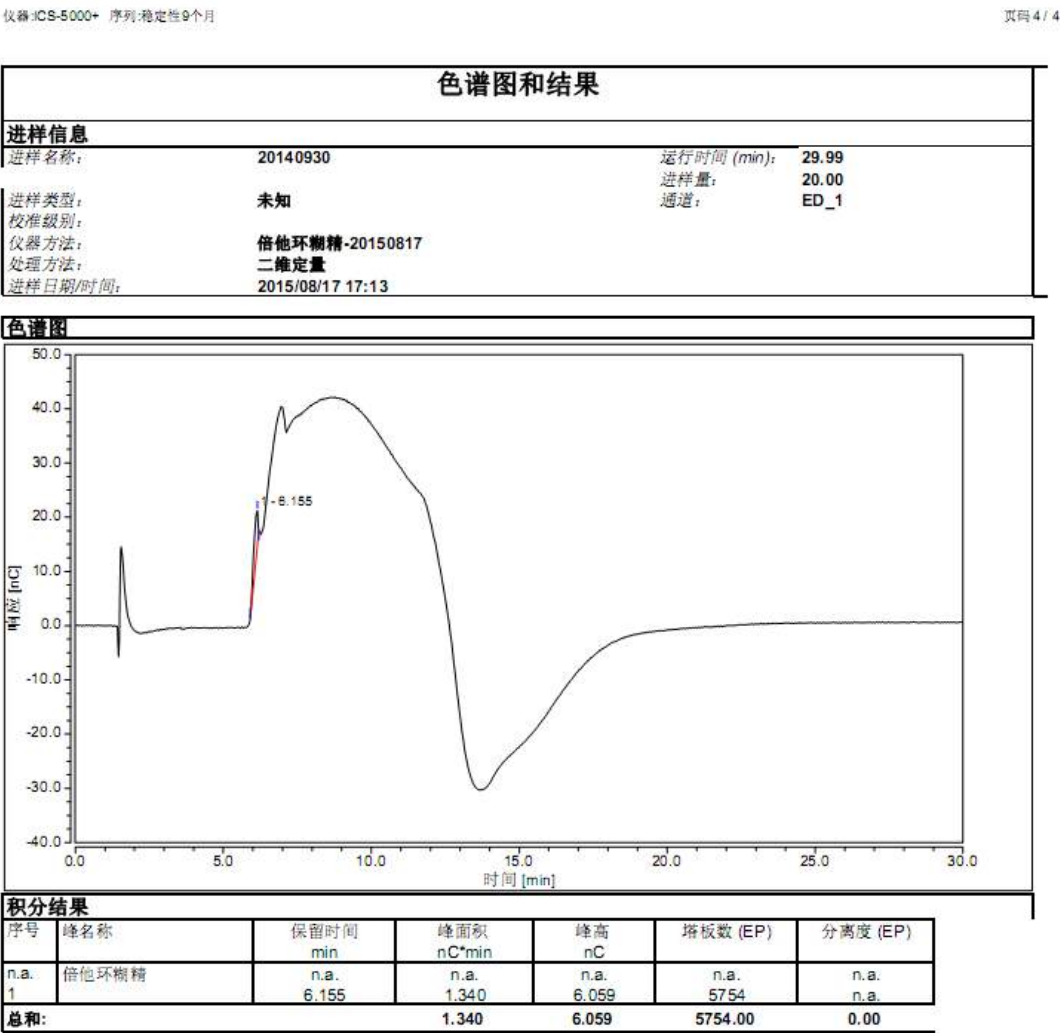
附图14-5-115 SSEC长期9月倍他环糊精测定图 (20140910)

Annex 3-S-102 Long-term testing-Betadex (HPIC) -9month-20140921



附图14-5-116 SBECD长期9月倍他环糊精测定图 (20140921)

Annex 3-S-103 Long-term testing-Betadex (HPIC) -9month-20140930



附图14-5-117 SSEC长期9月倍他环糊精测定图 (20140930)