

## 1. Determination of 1,6-Hexanediol using GsBP-5 column

1,6-Hexanediol was used for production and development of 1,6-Hexanediol Polyester, photo-curing agent UV-curable coatings, PU elastomer polycarbonate, pharmaceutical intermediates and food additives, etc. It could approve mechanical strength and advance properties of anti-hydrolysis, heat-resistance and chemical resistance. To determine 1,6-Hexanediol, GsBP-5 column with low column bleed, inert and neutral surface, is recommended. Table1-1 and figure1-1 showed the instrumentation condition and acquired Chromatogram using GsBP-5 column, respectively.

Column: GSBP-5/30m*0.32mm*0.25um	Oven: 100°C(2min)-20°C/min-260°C (10min)
Carrier: N2, pressure 0.06MPa	INJ: 250°C DET: FID, 260°C
Flow ratio: 60: 1	

Table 1-1. Instrumentation condition of 1,6-Hexanediol determination

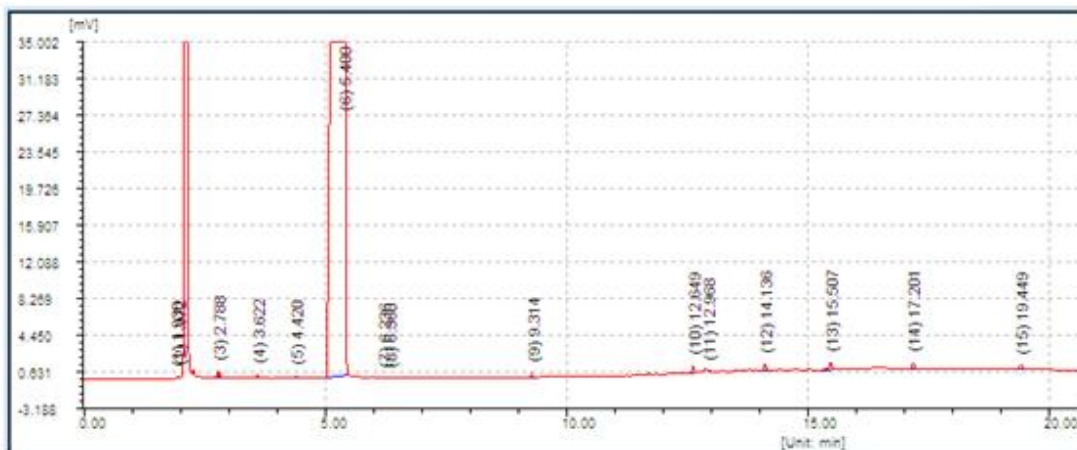
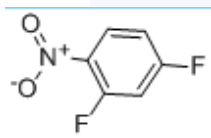


Figure 1-1. Chromatogram of 1,6-Hexanediol determination

## 2. Separation of 2,4- Difluoronitrobenzene isomers

2,4- Difluoronitrobenzene, one of Pharmaceutical Intermediates, was determined by GC with optimal conditions (recorded in table 2-1). As the typical Chromatogram and analysis result (figure 2-1) showed, the separation of isomers could be achieved on GsBP-1 column. 2,4- Difluoronitrobenzene has a synonym of 2,4-Difluoro-1-nitrobenzene, the molecular formula is C<sub>6</sub>H<sub>3</sub>F<sub>2</sub>NO<sub>2</sub>.



Structure is shown as follows

2,4- Difluoronitrobenzene is colorless or clear yellow liquid. The melting point is 9-10°C and boiling point is 203-204°C. It is used as intermediate in synthesis of Pesticides, drugs and liquid crystal materials.

Column: GsBP-1 /30m*0.32mm*1.0um	Oven: 80°C (5min) -10°C/min-180°C (5min)
Carrier: N <sub>2</sub> , flow1.5ml/min	Inj: 250°C Det: FID, 260°C
Split ratio: 50: 1	Injection: 0.1ul

Table 2-1. Instrumentation condition of 2,4- Difluoronitrobenzene isomers separation

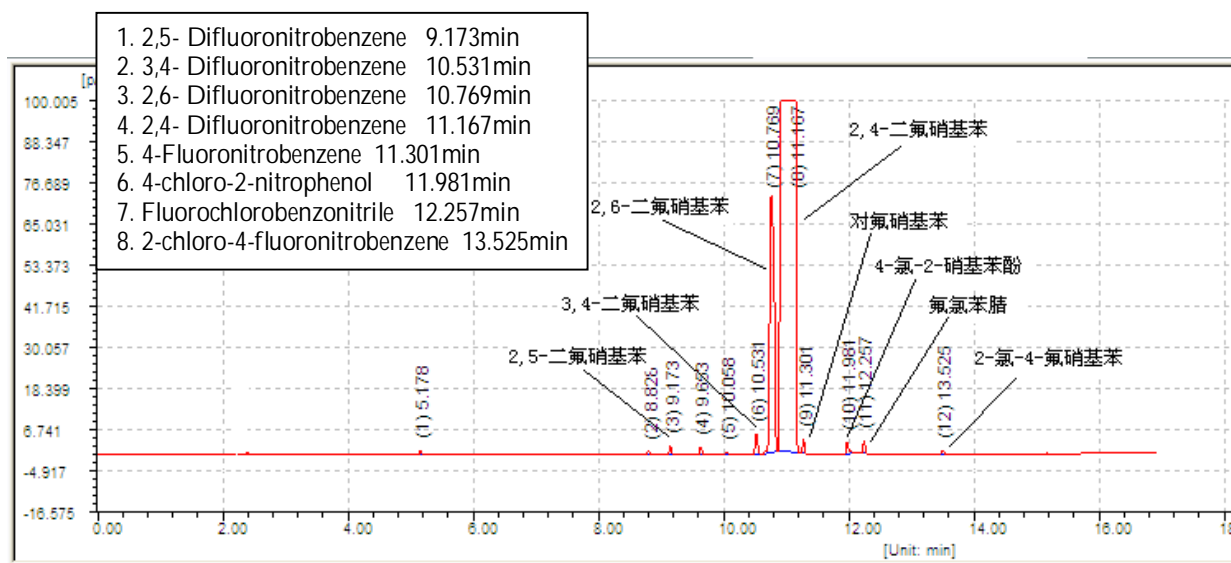


Figure 2-1. Typical Chromatogram and analysis result of separation

### 3. Determination of 2,4-Difluoro-3,5-dichloronitrobenzene

2,4-Difluoro-3,5-dichloronitrobenzene, one of Pharmaceutical intermediates, is determined by GC using GsBP-1 column. With the following recommended condition (shown in table 3-1), the chromatogram is obtained in figure 3-1.

Column: GsBP-1 /30m*0.32mm*1.0um	Oven: 130°C (5min) -10°C/min-250°C (10min)
Carrier: N2, flow 1.5ml/min	Inj: 250°C Det: FID, 260°C
Flow ratio: 50: 1	Injection: 0.1ul Makeup: 40ml/min

Table 3-1. testing condition of 2,4-Difluoro-3,5-dichloronitrobenzene

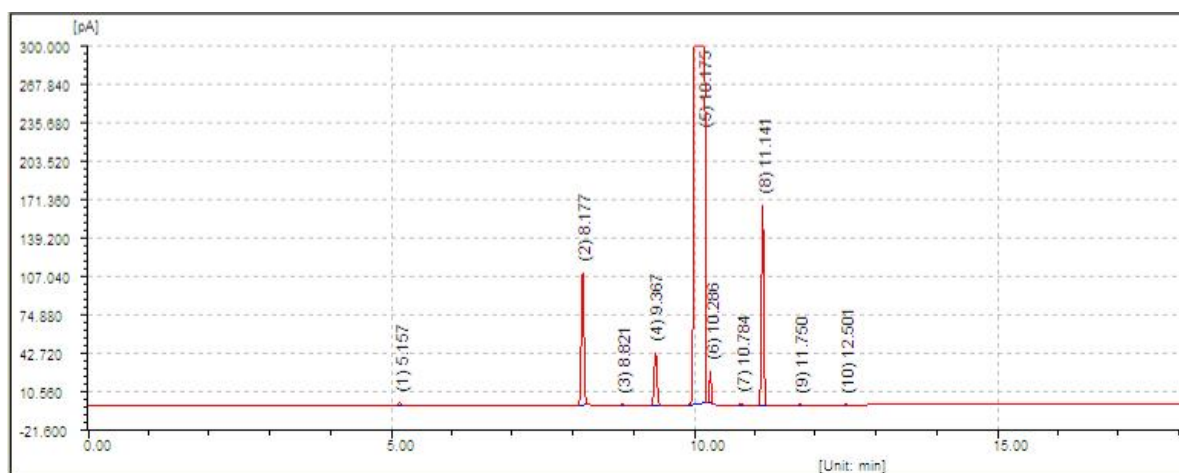
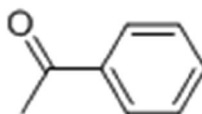


Figure 3-1. chromatogram of 2,4-Difluoro-3,5-dichloronitrobenzene determination

#### 4. Separation of Acetophenone

Acetophenone is the organic compound with the formula  $C_6H_5C(O)CH_3$ . It is the simplest aromatic ketone. This colourless viscous, low melting point, low volatility oily liquid with fruit flavor is soluble in most organic solvents. It is a precursor to useful resins and fragrances. Acetophenone is a raw material for the synthesis of some pharmaceuticals, examples include dextropropoxyphene and phenylpropanolamine. The Structure is shown as follows:



Acetophenone sample is tested by GC using GsBP-Wax column using proper condition (table 4-1). The chromatogram and peak identification is recorded in figure 4-1.

Oven : 100°C ( 2min ) -10°C/min-200°C (10min)	Inj: 250°C Det: FID, 260°C
Column: GsBP-WAX/30m*0.32mm*0.5um	Injection: 0.2ul
Carrier: N2, Pressure 0.06MPa	Quantitative Method: Area normalization
Split flow: 50ml/min	

Table 4-1 testing condition of acetophenone sample.

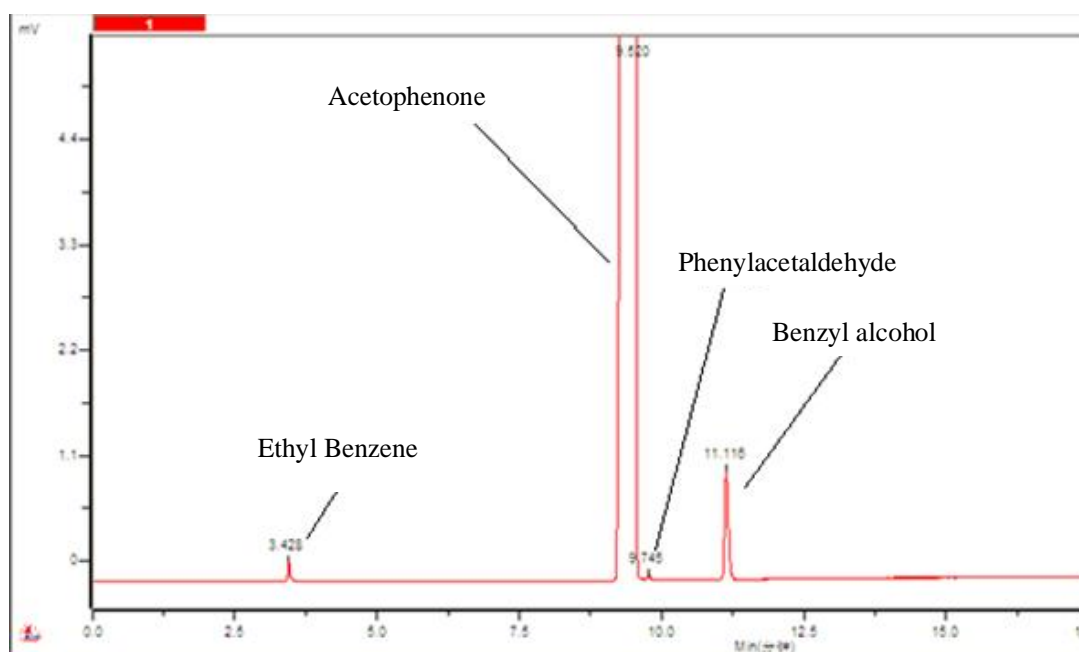


Figure 4-1 chromatogram of acetophenone sample separation.

## 5. Determination of Acrylamide with internal standard method

Acrylamide water solution is determined by GC using GsBP-5 column. Because water has no response in FID detector, 40% Acrylamide water solution and internal standard were used for qualification. The internal standard could be separated from water and soluble in water. The use of N-Methylpyrrolidone (NMP) is recommended. The reagents used in the experiment are acrylamide standard (with measured concentration) and N-Methylpyrrolidone(AR) as the internal standard. The instrumentation condition is shown in table 5-1 and result is recorded in figure 5-1.

Column: GSBP-5 30m*0.32mm*0.25um	Oven: 210°C
Injector: 250°C	Injection: 1.0ul
Detector: FID, 260°C	Carrier: N2, pressure 0.04MPa
	Split ratio: 80: 1

Table5-1. The instrumentation condition of Acrylamide water solution

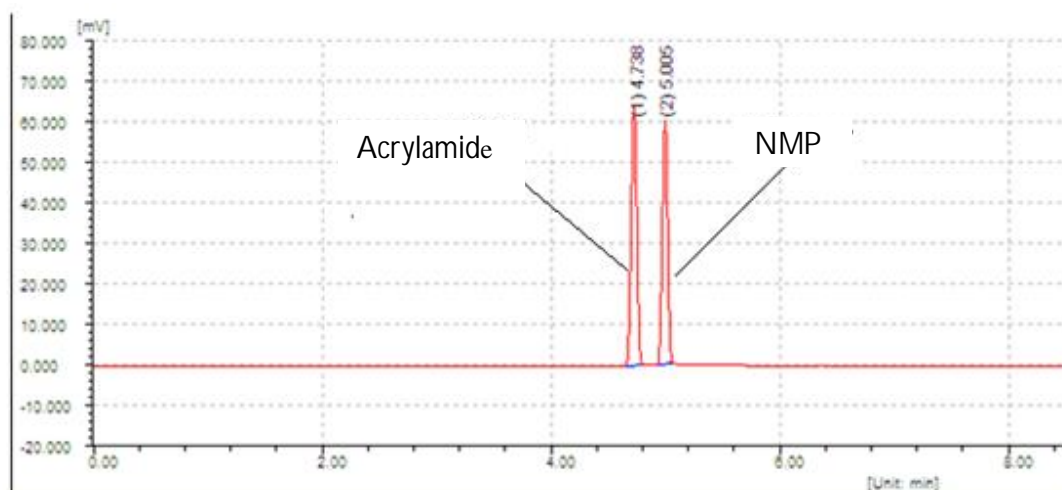


Figure 5-1. The chromatogram of acrylamide solution separation

## 6. Separation of Alkaline Solvents

The alkaline solvents are widely used in pharmaceutical application. In following condition, alkaline solvents are separated from each other using GsBP-Carbowax column. The chromatogram and peak identification are recorded in figure6-1.

Column: 120°C (5min) -10°C/min-180°C (10min)	Injection: 0.2ul
INJ: 250°C DET: FID, 220°C	Quantitative method: Area normalization
Column: GsBP-CARBOWAX20M/30m*0.32mm*0.25um	
Carrier: N2, constant flow 1.0ml/min	
flow ratio: 100: 1	

Table6-1. instrumentation condition

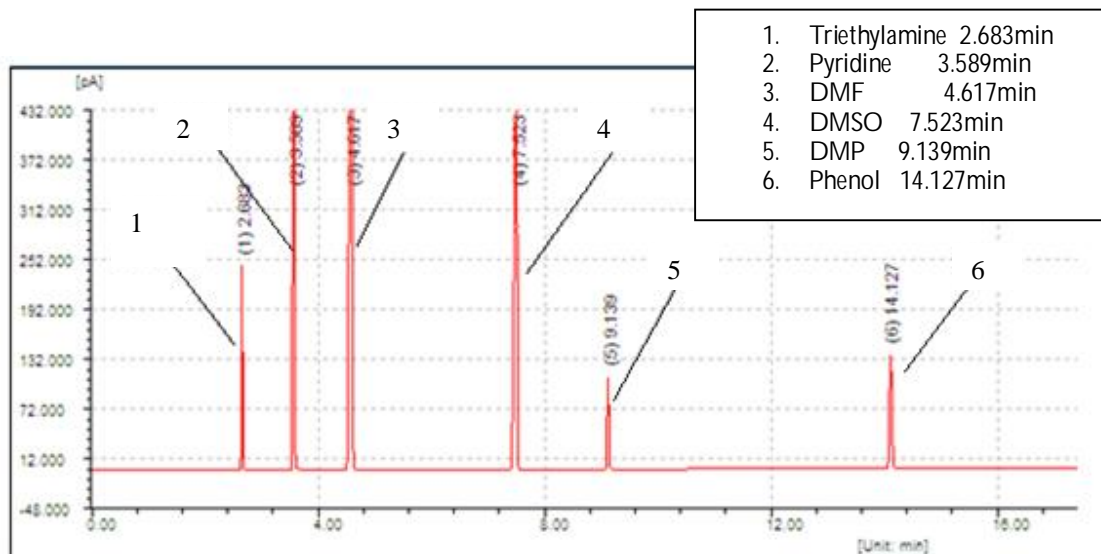


Figure 6-1. the chromatogram of Alkaline Solvents separation

## 7. Test Report of Benzoyl Chloride

The separation of Benzoyl Chloride is achieved on GSBP-5 column using proper condition in table7-1. The result is shown in figure7-1.

Column: GSBP-5/30m*0.32mm*0.25um	Oven: 100°C (6min) -20°C/min-250°C (10min)
Carrier: N2 flow 0.8ml/min	Inj: 150°C Det: FID, 260°C
Split ratio: 80: 1	Injection: 0.2ul
	Quantitative Method: Area normalization

Table 7-1. Instrumentation condition of Benzoyl Chloride Separation.

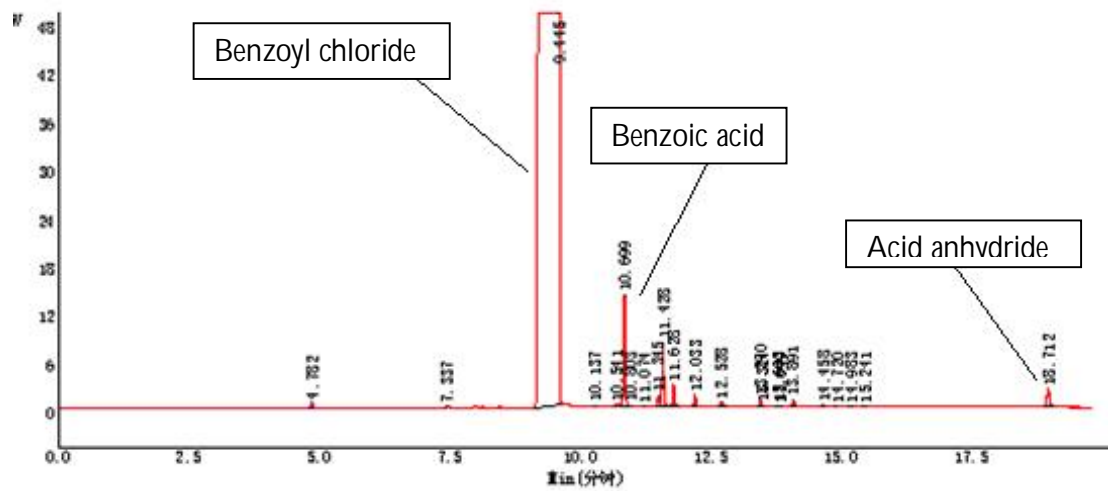


Figure 7-1. Chromatogram of Benzoyl Chloride Separation

## 8. Chromatographic Analysis of BTEX

BTEX is the abbreviation of a group of volatile organic compounds (VOCs) consisting of benzene, toluene, ethylbenzene, and o-,p- and m-Xylenes. The presence of BTEX in the environment is caused mainly by the industrial use as solvents and in the production of organic chemicals such as drugs and pesticides. The separation of BTEX could be achieved by GC using GsBP-Inowax column. The instrumentation condition which is used in the separation is shown as follows in table 8-1. The corresponding result is in figure 8-1.

Oven: 80°C	Carrier: N <sub>2</sub> , flow 0.8ml/min
Inj: 200°C Det: FID,220°C	Split ratio: 80: 1
Column: GsBP-INOWAX /30m*0.32mm*0.5um	Injection: 0.2ul

Table8-1. Instrumentation condition of BTEX analysis.

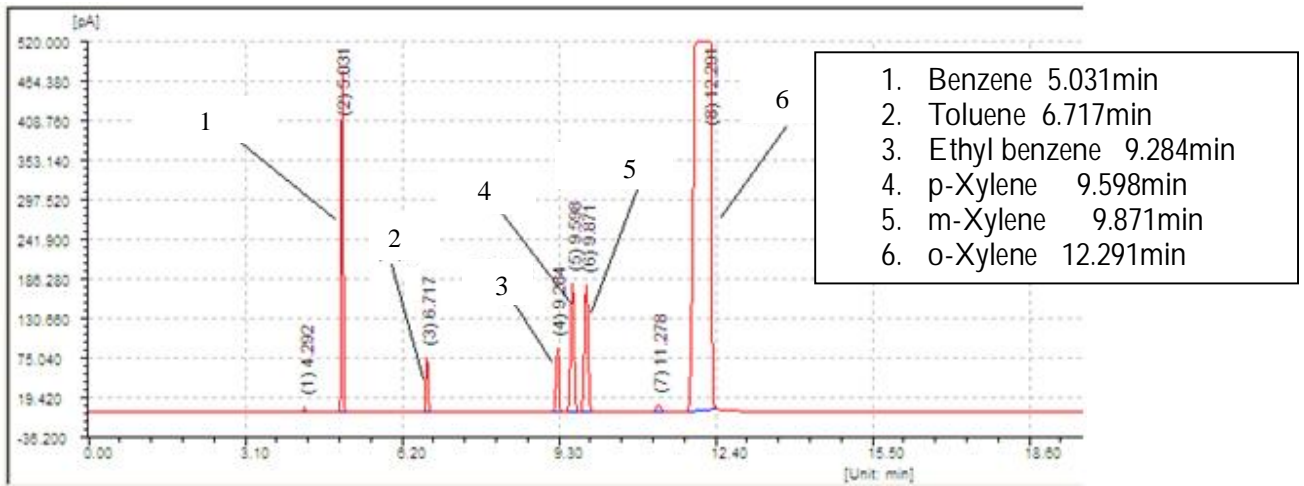


Figure 8-1. Typical Chromatogram and analytical result of BTEX

### 9. Determination of Butyl acrylate

Butyl acrylate has the abbreviation BA (C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>). Molecular weight is 128.17 and structure is H<sub>2</sub>C=CHCOOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>. It is not soluble in water but mixable with ethanol and ether, belonging to acryloyl compounds. It is determined by GC using GsBP-5 column with the optimal condition shown in table9-1. The result is recorded in Figure9-1.

Column: GSBP-5 /30m*0.32mm*0.25um	Oven: 80°C (9min) -20°C/min-250°C
Carrier: N <sub>2</sub> , pressure 0.04MPa	Inj: 250°C Det: FID, 260°C
Split ratio: 100: 1	Injection: 0.2ul
	Quantitative Method: Area normalization

Table 9-1. Instrumentation condition of Butyl acrylate determination.



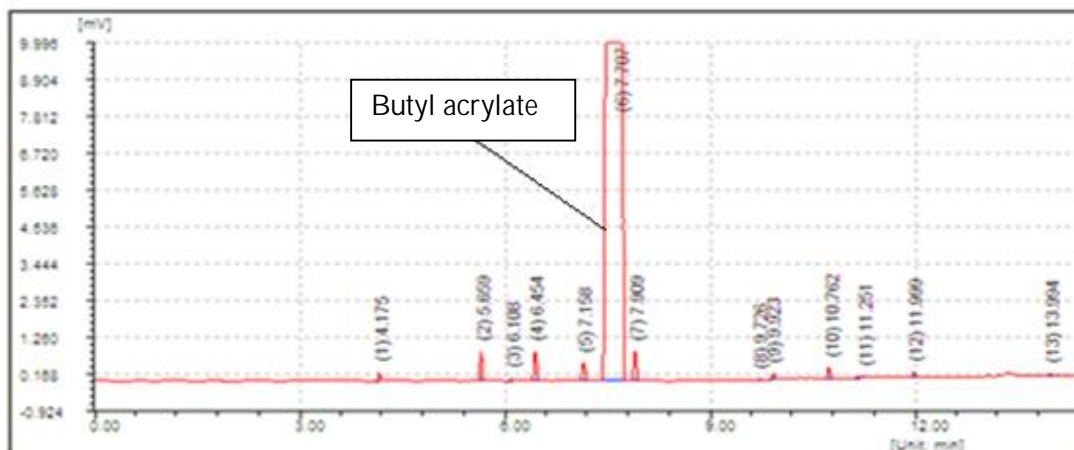


Figure9-1. Typical Chromatogram of butyl acrylate analysis.

## 10. Analysis of Chloropropanone

Chloroacetone is a chemical compound with the formula  $\text{CH}_3\text{COCH}_2\text{Cl}$ . At STP it is a colourless liquid with a pungent odour. On exposure to light, it turns to a dark yellow-amber colour. It was used as a tear gas in World War I. It is analyzed by GC using GsBP-1701 column with proper condition (in table10-1) and result is shown in figure 10-1.

Column: GsBP-1701/30m*0.32mm*0.50-1.0um	Oven: 100°C
Carrier: N2, pressure 0.06MPa	INJ: 250°C DET: FID,230°C
Split ratio: 100ml/min	Injection;0.2UL
	Quantitative Method: Area normalization

Table 10-1. Instrumentation condition for Chloropropanone analysis.

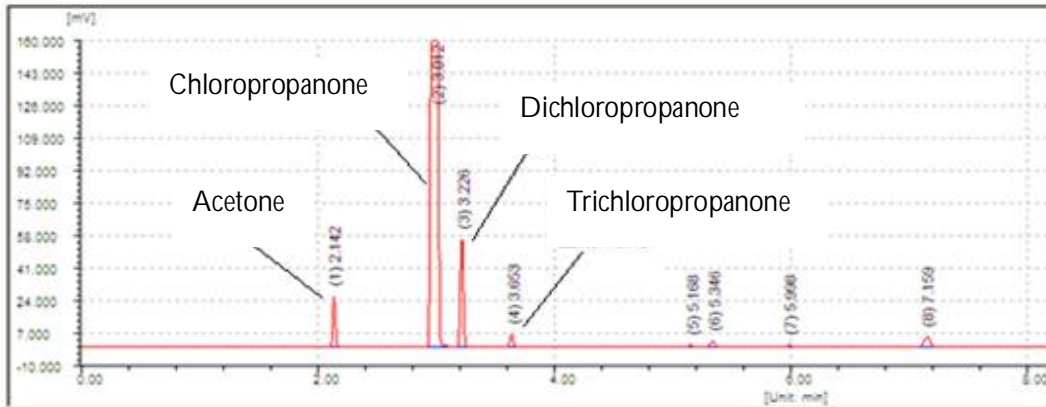


Figure10-1. Chromatogram of Chloropropanone analysis.

## 11. Determination of DiethanolaMine

Diethanolamine, often abbreviated as DEA or DEOA, is an organic compound with the formula  $\text{HN}(\text{CH}_2\text{CH}_2\text{OH})_2$ . Pure diethanolamine is a white solid at room temperature, but its tendency to absorb water and to supercool mean it is often encountered as a colorless, viscous liquid. Diethanolamine is polyfunctional, being a secondary amine and a diol. Like other organic amines, diethanolamine acts as a weak base. Reflecting the hydrophilic character of the alcohol groups, DEA is soluble in water. Amides prepared from DEA are often also hydrophilic. Diethanolamine is determined by GC using GSBP-1701. The instrumentation condition is shown in table 11-1 and the result is recorded in figure11-1.

Column: GSBP-1701/30m*0.32mm*1.0um	Oven : 120°C ( 2min ) -25°C/min-250°C ( 10min )
Carrier: N2, pressure0.08MPa	Inj: 280°C Det: 270°C
Split ratio: 40ml/min	Solvent: ethanol injection: 1.0ul
	Quantitative Method: Area normalization

Table 11-1. Instrumentation condition of DiethanolaMine determination.

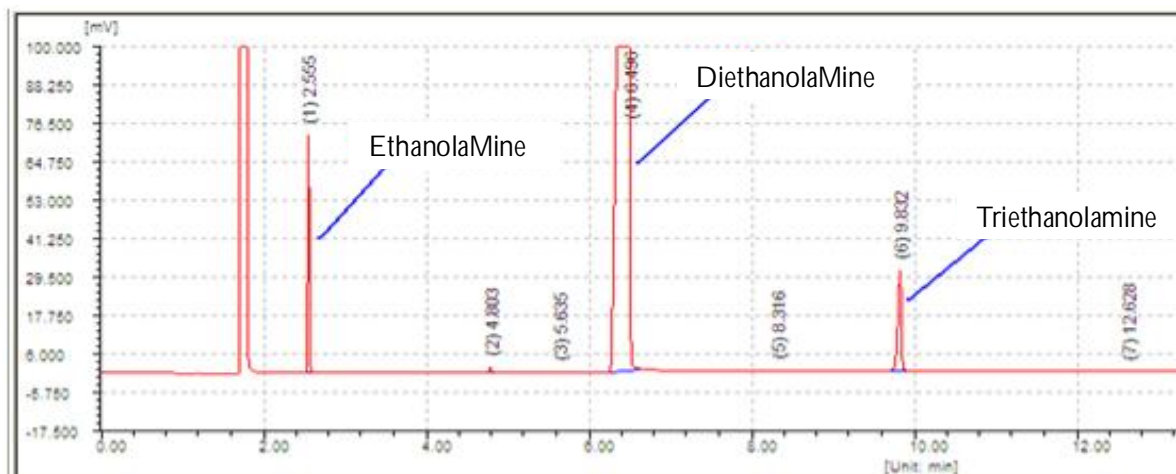


Figure11-1. Chromatogram for DiethanolaMine analysis.

## 12. Determination of lambda cyhaltrin acid

lambda cyhaltrin acid has a chemical name Z-(1R,S)-cis-2,2-dimethyl-3-(2,2-chloro-3,3,3-trifluoro-1-propenyl)Cyclopropanecarboxylic acid. The synonym is lambda cyhaltrin acid. Molecular formula is C<sub>9</sub>H<sub>10</sub>ClF<sub>3</sub>O<sub>2</sub>. The Structure is shown as follows:



It is soluble in benzene, chloroform, ethyl acetate, etc, easily soluble in alkaline solution but not soluble in water. boiling point is 107-109°C. It is the intermediate in the synthesis of Cyhalothrin, Bifenthrin, Tefluthrin and other Pyrethroid insecticides. As the customer's requirement, lambda cyhaltrin acid isomer separation is achieved by GC. Weighted 0.2g sample and added 2ml chloroform for complete dissolution. Absorbed the solution with 10ul syringe for analysis. (notice: after trace injection, syringe should be rinsed with acetone and purged dry using rubber suction bulb for further use in case of cross-contamination). The following instrumentation condition (in table12-1) is recommended. The result is shown in figure 12-1.

Column: GsBP-FFAP/30m*0.32mm*0.25um	Carrier: N2, flow 1.5ml/min
Split ratio: 80: 1	Inj: 280°C Det: FID,250°C
Oven : 100°C ( 1min ) 15°C/min-180°C (5.6min) -20°C/min-230°C (10min)	Quantitative Method: Area normalization

Table 12-1. Instrumentation condition of isomers separation.

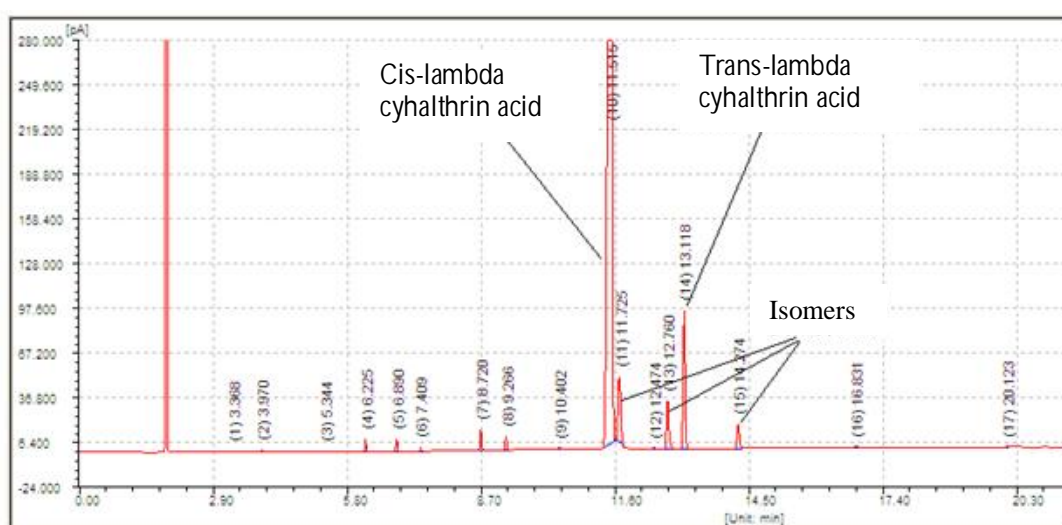


Figure12-1. Typical Chromatogram of lambda cyhalthrin acid isomers separation.

Conclusion : we utilized non-polar column (SE-30,SE-54), mid-polar column (GsBP-1701), polar column(GsBP-Inowax, FFAP) to obtain optimal separation condition of isomers. Among them, FFAP columns could help achieve complete baseline separation as well as have improved peak shapes, which determined that FFAP column is recommended for the separation of lambda cyhalthrin acid isomers.

### 13. Analysis of N- Methylpyrrolidone (NMP)

NMP is an organic compound consisting of a 5-membered lactam. It is a colorless liquid, although impure samples can appear yellow. It is miscible with water and with most common organic solvents. It also belongs to the class of dipolar aprotic solvents such as dimethylformamide and dimethyl sulfoxide. It is used in the petrochemical and pharmaceutical industries as a solvent, exploiting its nonvolatility and ability to dissolve diverse materials. It is analyzed by GC using GsBP-1701 with recommended

condition in table 13-1 and the result is recorded in figure 13-1.

Column:DB-1701/30m*0.32mm*1.0um	Oven: 100°C (1min) -10°C/min-200°C (10min)
Carrier: N2 flow 1.0ml/min	Inj: 250°C Det: FID,230°C
Flow ratio: 50: 1	Injection: 0.2ul

Table 13-1 recommended condition of N- Methylpyrrolidone separation

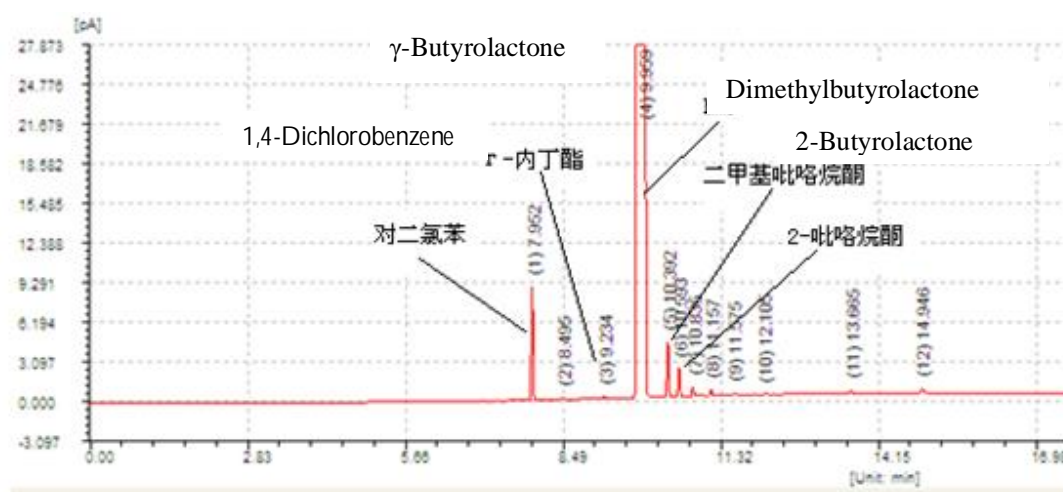


Figure 13-1. Chromatogram of N- Methylpyrrolidone separation

#### 14. O-phenylenediamine Analysis Report

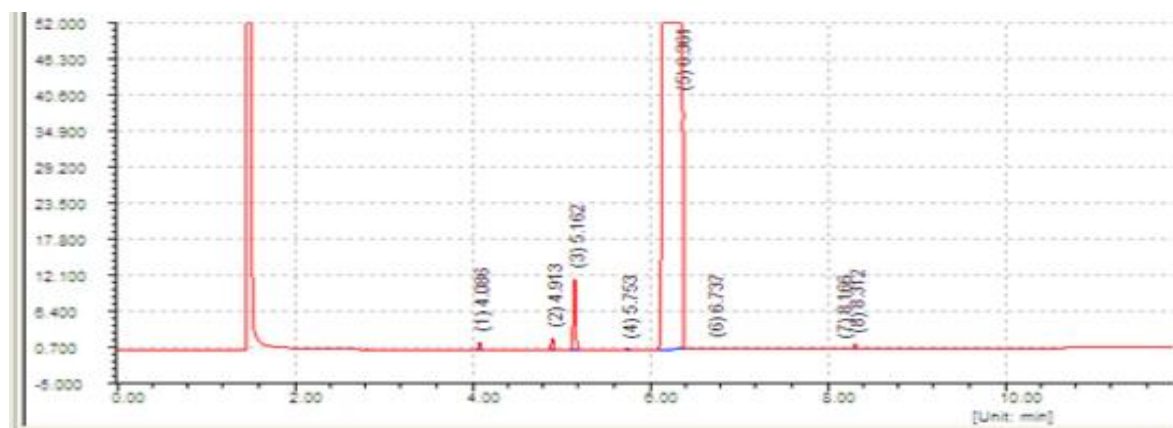
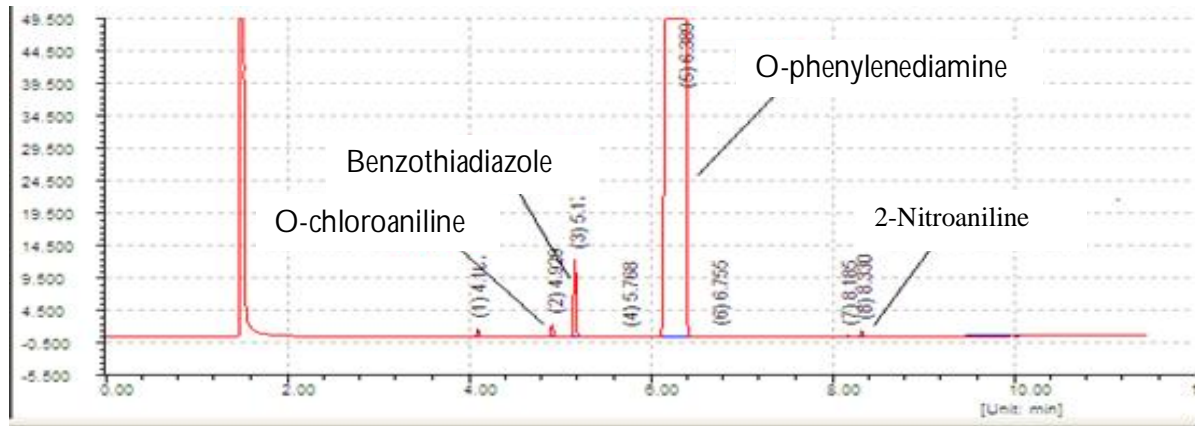
o-Phenylenediamine is an organic compound with the formula  $C_6H_4(NH_2)_2$ . This aromatic diamine is an important precursor to many heterocyclic compounds. Condensation of substituted o-phenylenediamine with various diketones is used in the preparation of a variety of pharmaceuticals. o-Phenylenediamine sample is tested by GC with proper condition in table 14-1.

Oven : (Normalization)100 °C (2min)20 °C /min-230°C (5min)	Carrier: N2 constant flow 1.2ml/min
(Internal standard method)160°C	Split ratio: 40: 1
Inj: 250°C Det: FID, 260°C	Injection: 1.0ul
Column: GSBP-50/30m*0.32mm*0.25um	Solvent: ethanol or chloroform

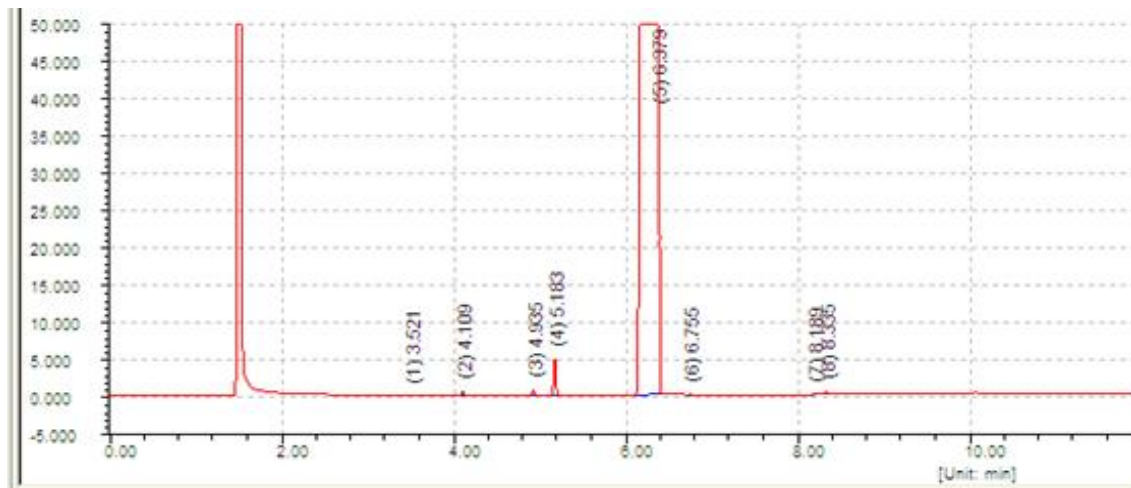
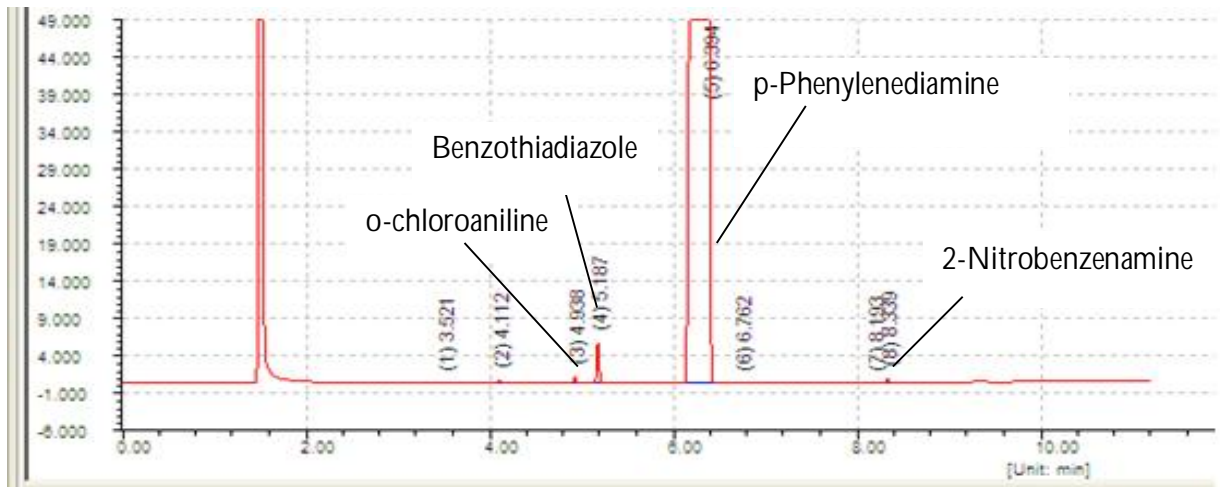
Table 14-1. Instrumentation condition of O-phenylenediamine analysis.

## Trace analysis using normalization method

### 1. Quantitative and Repeatability of sample 2# by GC with two injections



### 2. Quantitative and Repeatability of sample 1# by GC with two injections

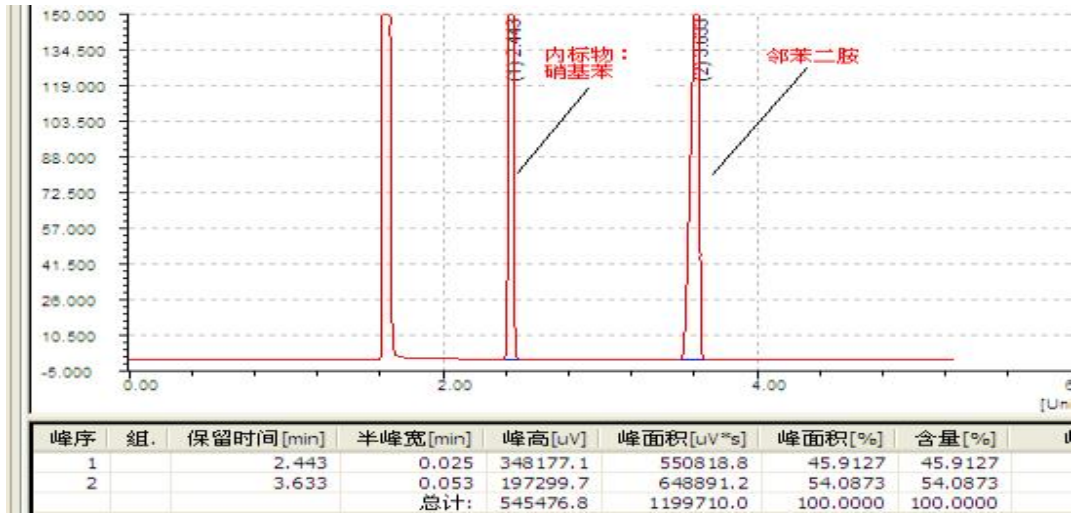


## 二. Product analysis using internal standard method (Correction factor f)

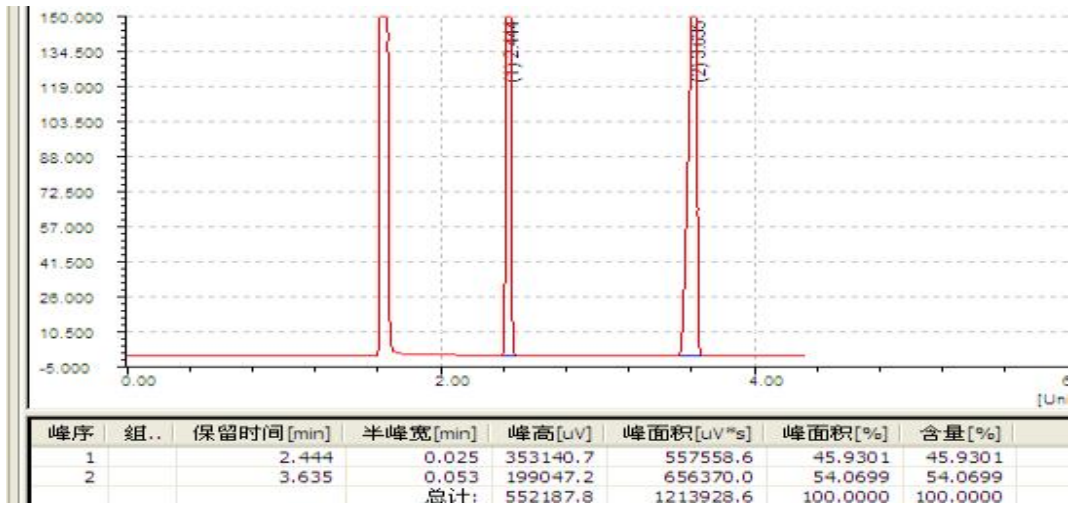
Repeatability measurement (7 consecutive injections)

1.  $f=0.8489$

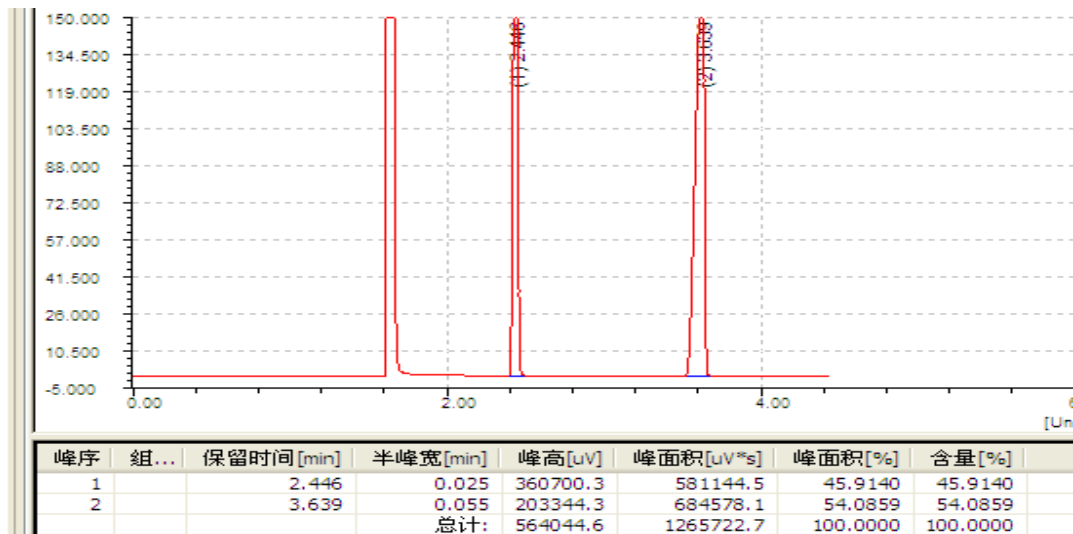




2.  $f=0.8494$

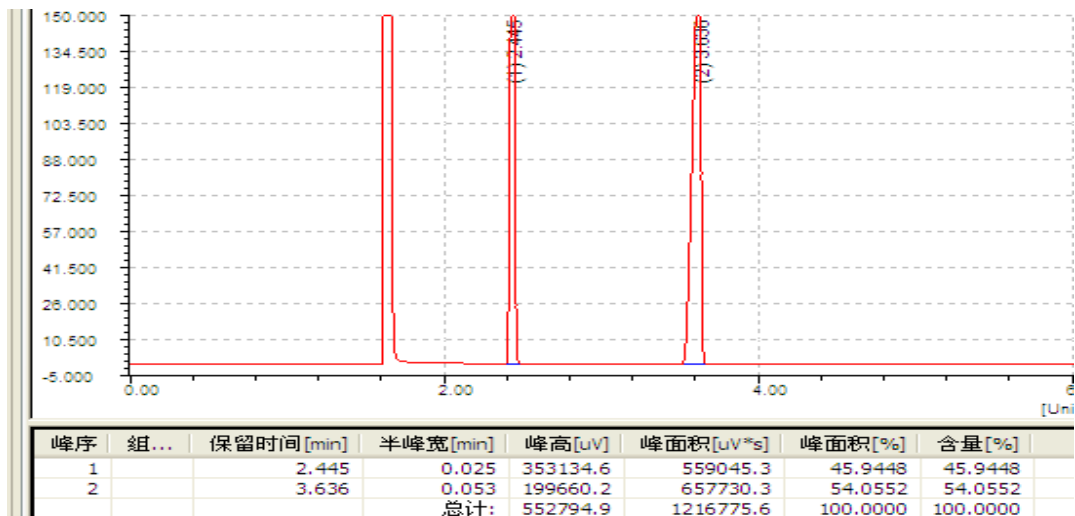


3.  $f=0.8489$

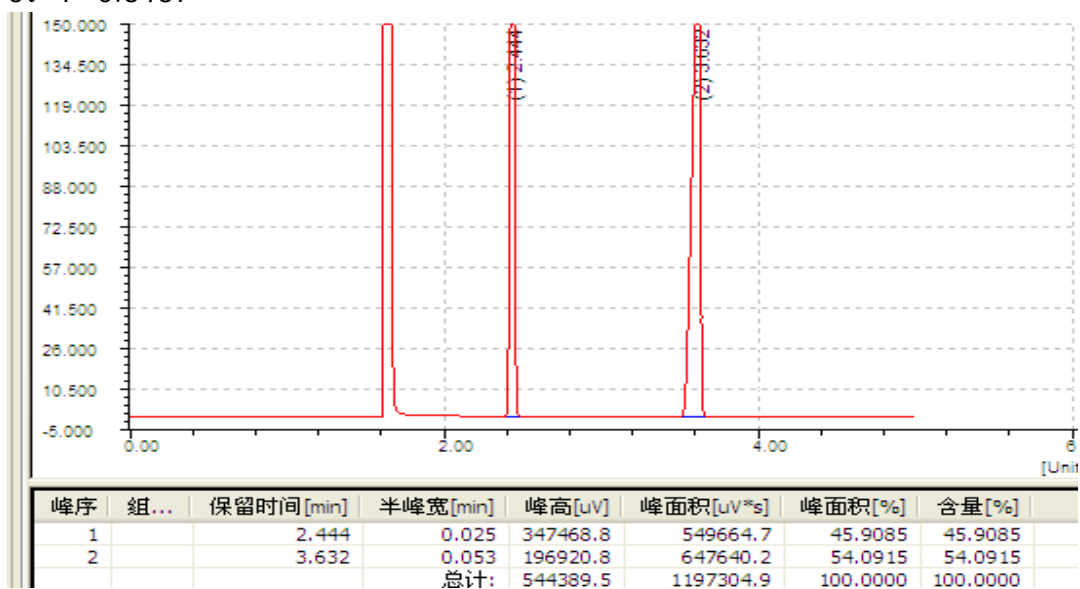




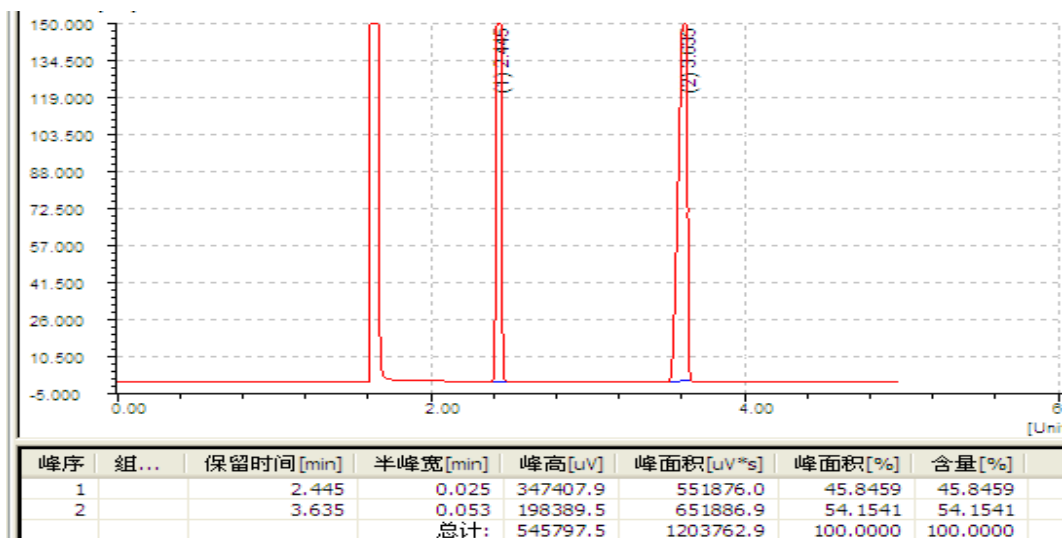
4.  $f=0.8500$



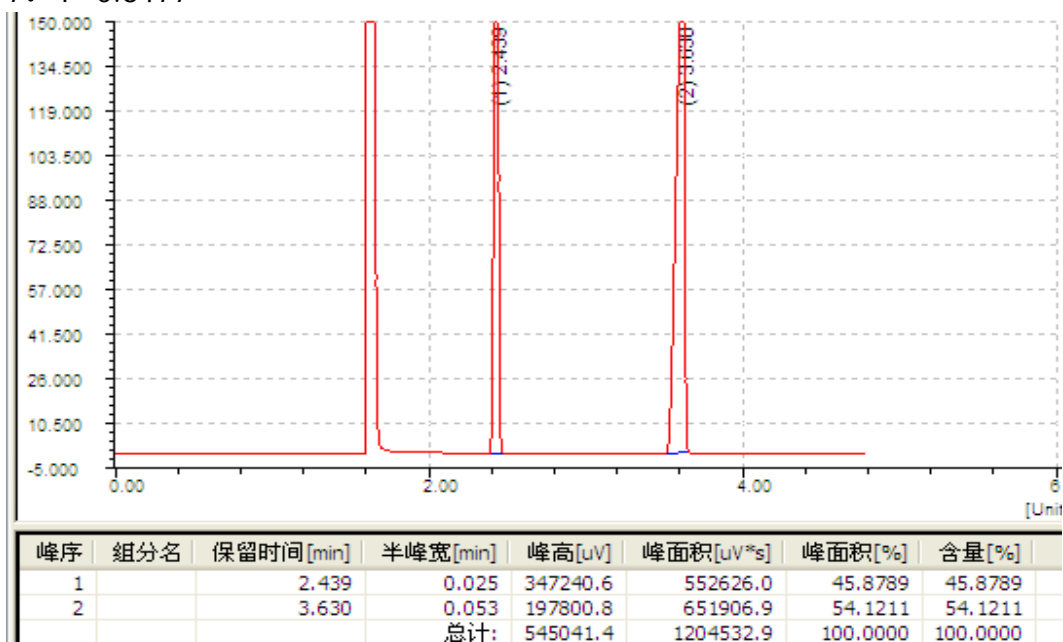
5.  $f=0.8487$



6.  $f=0.8466$



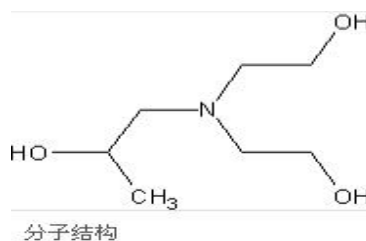
### 7. $f=0.8477$



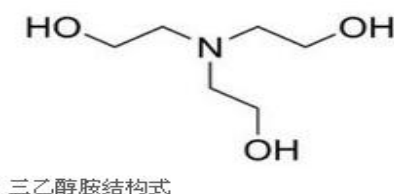
Conclusion: With 7 consecutive injections, according to acquired peak areas of internal standard and samples, calculate the correction factor  $f$  value: 0.8489, 0.8494, 0.8489, 0.8500, 0.8487, 0.8466, 0.8477. (Peak area of internal standard is divided by peak area of samples, quality is ignored.) Relative error is 0.34%, Repeatability is very good, in full compliance with the request of national standards ( $f < 1\%$ )

### 15. Separation of Diethanolisopropanolamine and Tris(2-Hydroxyethyl)Amine

Diethanolisopropanolamine is used as the cement grinding aid for the replacement of the raw materials in grinding aid Tris(2-Hydroxyethyl)Amine and Triisopropanolamine. Diethanolisopropanolamine is a new environmentally friendly raw material, have a good function as the grinding aid and have good compatibility with Tris(2-Hydroxyethyl) Amine, Triisopropanolamine and other amines, ethanols and esters. Diethanolisopropanolamine molecular formula is  $C_7H_{17}NO_3$ , molecular weight is 163.21, boiling point is  $145\text{ }^{\circ}\text{C}/0.6\text{ mm Hg(lit.)}$ . The structure is:



Tris(2-Hydroxyethyl)Amine molecular formula is  $C_6H_{15}NO_3$ , molecular weight is 149.1882, boiling point ( $^{\circ}\text{C}, 101.3\text{kPa}$ ) is 360.



As the customer required, the separation of Diethanolisopropanolamine and Tris(2-Hydroxyethyl) Amine and quantitative analysis by internal standard method are achieved. The recommended condition is shown in table 15-1.

Column: GsBP-INOWAX/ 30m*0.32mm*0.50um	Sampling: autosampler Injection: 1.0ul
Carrier: N2 pressure 0.14MPa	Solvent: ethanol
Split flow: 30ml/min	Quantitative Method: internal standard method
Oven: 240 $^{\circ}\text{C}$	
INJ: 280 $^{\circ}\text{C}$ DET: FID, 250 $^{\circ}\text{C}$	

Table 15-1. The instrumentation condition of separation on GsBP-Inowax.

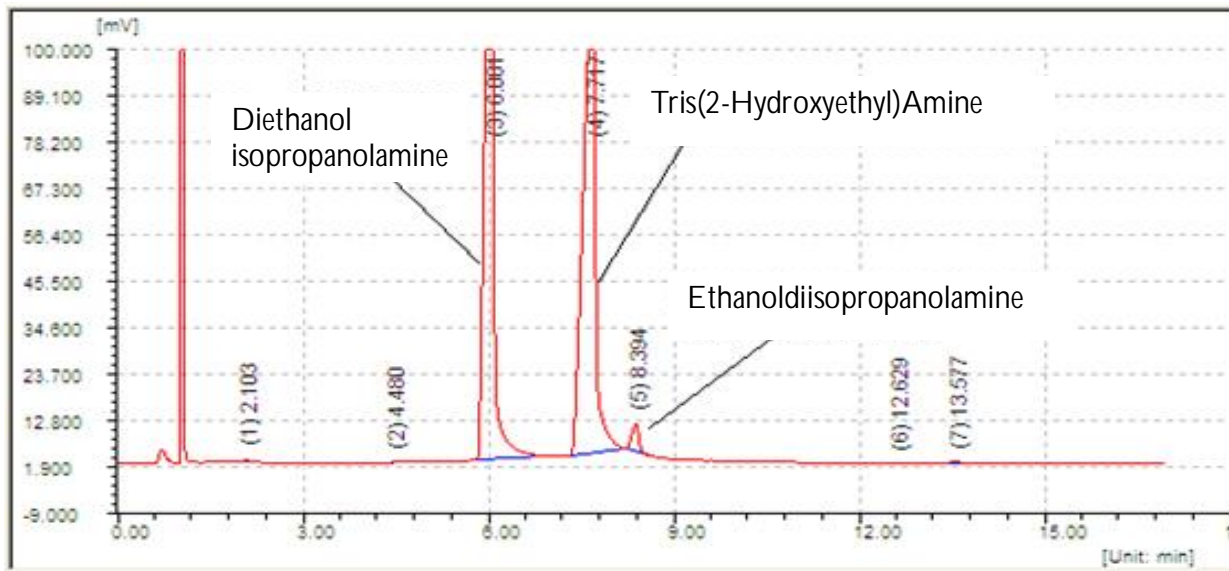


Figure 15-1. Typical Chromatogram of Mix sample

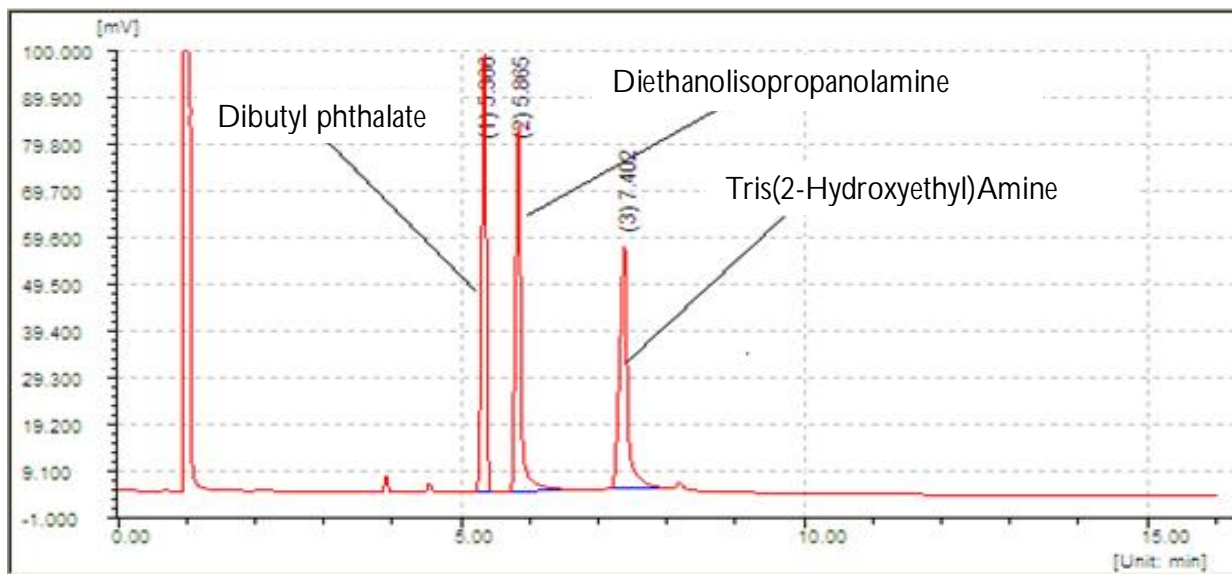
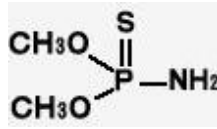


Figure 15-2. Typical Chromatogram of Internal standard Dibutyl phthalate

## 16. Determination of Spermine

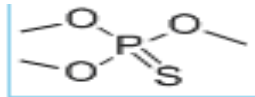
Spermine has the chemical name O, O- dimethyl aminophosphorothioate, O,O- Dimethyl phosphoramidothioate and DMPAT. Molecular formula is C<sub>2</sub>H<sub>8</sub>O<sub>2</sub>SP. Structure is



Spermene is transparent or light white liquid; the product is mainly used as the intermediate in production of acephate and methamidophos.

Main impurity is O, O, O- mercapto trimethyl phosphate, the synonym is Thio trimethyl phosphate. Molecular formula is C<sub>3</sub>H<sub>9</sub>O<sub>3</sub>PS and molecular weight is 156.1405; boiling point is 169°C at 760 mmHg.

The structure is



As Customer required, we explore the optimal separation condition on OV-17 and -624 columns. The concentration of the main impurity O, O, O- mercapto trimethyl phosphate is less than <5% and the repeatability test is achieved.

### I. Spermene analysis using condition 1

Oven: 100°C (2min) -10°C/min-250°C Inj: 270°C Det: FID,280°C Column: GSBP-50+MS/30m*0.32mm*0.25um Carrier: N <sub>2</sub> , constant flow 1.5ml/min	Injection: 1.0ul solvent; Dichloromethane Quantitative Method: Area normalization
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Table 16-1. condition 1 of Spermene determination

Spermene is analyzed using GsBP-50+MS column. Before sampling, the sample was dissolved in methylene chloride after dilution with a certain proportion and was injected using autosampler. Chromatogram was recorded in Figure 16-1.

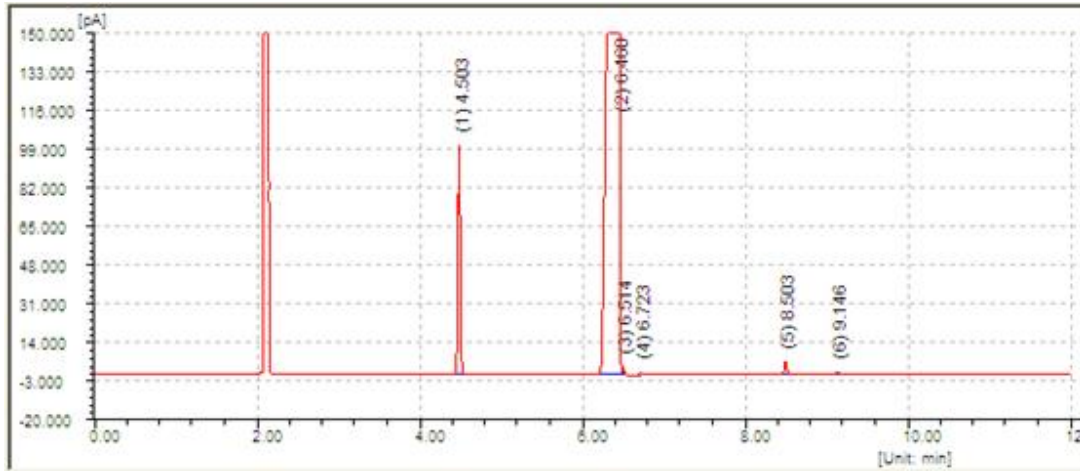


Figure16-1. Typical Chromatogram of Spermine determination.

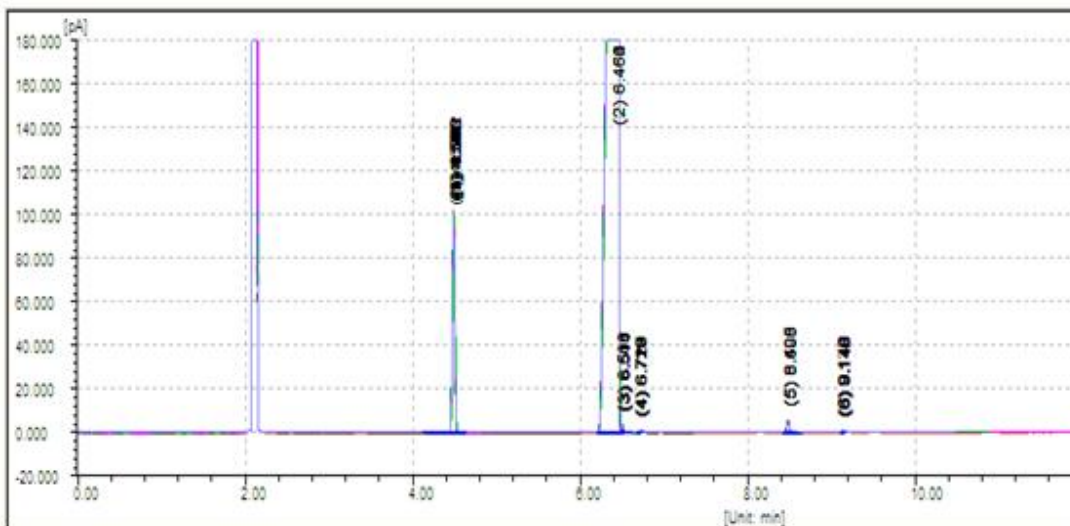


Figure 16-2. Multiplet chromatogram of repeatability test with six consecutive injections

## II. Spermine analysis using condition 2

Oven: 100°C (1min) -15°C/min-200°C (8min)	Injection: 1.0ul solvent; Dichloromethane
Inj: 270°C Det: 250°C	Quantitative Method: Area normalization
Column: GSBP-624/30m*0.32mm*1.8um	
Carrier: N2, flow1.5ml/min	

Table 16-2. condition 2 of Spermine determination

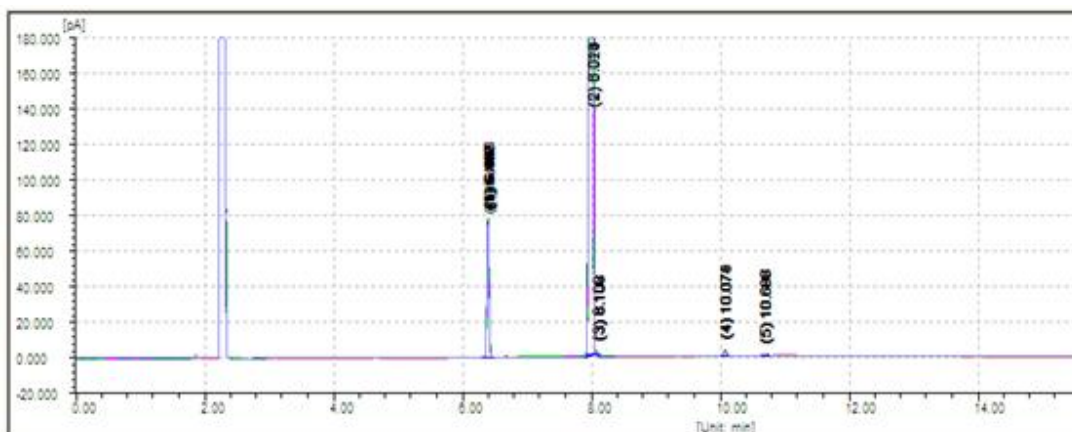
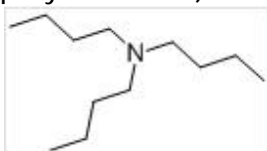


Figure 16-3. Multiplet chromatogram of repeatability test with six consecutive injections

Conclusion: We used GC-9720 + autosampler to analyze Spermine sample on GSBP-50+MS and GSBP-624 columns, the testing results are consistent with what customer required and the area RSD is less than 2% with six consecutive injections. With area normalization quantitative method, the Spermine content RSD is less than 0.04% and main impurity (O, O, O- mercapto trimethyl phosphate) content RSD is less than 0.7%.

## 17. Determination of Tributylamine

Tributylamine (TBA) is an organic compound with the molecular formula  $C_{12}H_{27}N$ . It is a colorless to yellow, hygroscopic liquid with an amine-like odor which is very poorly soluble in water. Tributylamine has a wide range of applications. It is an intermediate in the manufacture of other chemical compounds, including quaternary ammonium compounds, pharmaceuticals and dyes. It is also used as a solvent and as a catalyst (proton acceptor) in organic syntheses and polymerization (including polyurethanes). The structure is:



The sample is analyzed by GC on GsBP-1. The condition is shown in table 17-1 and the result is in figure 17-1.

Column: GsBP-1 /30m*0.32mm*1.0um Carrier: N2 flow1.5ml/min Split ratio: 50:1	Oven: 130°C (5min) -10°C/min-250°C (10min) Inj: 250°C Det: FID, 260°C Injection: 0.2ul
--	--

Table 17-1. instrumentation condition of Tributylamine determination

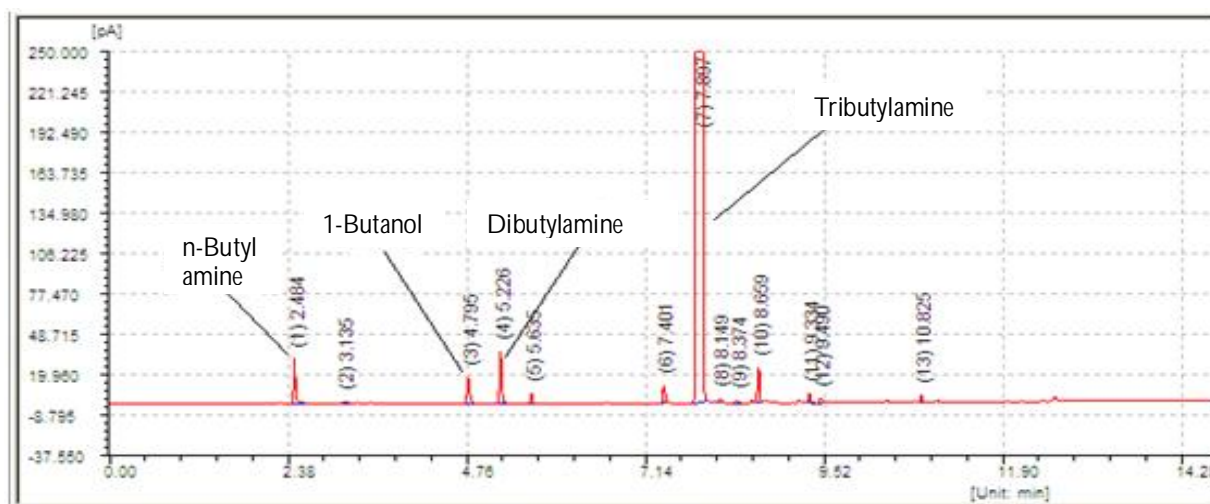


Figure 17-1. Chromatogram of Tributylamine determination

## 18. Determination of Triethylamine

Triethylamine is the chemical compound with the formula  $N(\text{CH}_2\text{CH}_3)_3$ , commonly abbreviated  $\text{Et}_3\text{N}$ . It is also abbreviated TEA, yet this abbreviation must be used carefully to avoid confusion with triethanolamine or tetraethylammonium, for which TEA is also a common abbreviation. It is a colourless volatile liquid with a strong fishy odor reminiscent of ammonia and is also the smell of the hawthorn plant. Like diisopropylethylamine (Hünig's base), triethylamine is commonly encountered in organic synthesis. Triethylamine is mainly used in the production of quaternary ammonium compounds for textile auxiliaries and quaternary ammonium salts of dyes. It is also a catalyst and acid neutralizer for condensation reactions and is useful as an intermediate for manufacturing medicines, pesticides and other chemicals. The Triethylamine sample is analyzed by GC on GsBP-1 column. The recommended condition is shown in Table 18-1 and the result is recorded in Figure 18-1.



Column: GsBP-1 /30m*0.32mm*1.0um	Split ratio: 60: 1
Injector: 250°C	Oven: 100°C (2min) -10°C/min-200°C (5min)
Detector: 260°C, FID	Injection: 0.2ul
Carrier: N2, flow 0.8ml/min	

Table 18-1. Instrumentation condition of Triethylamine determination

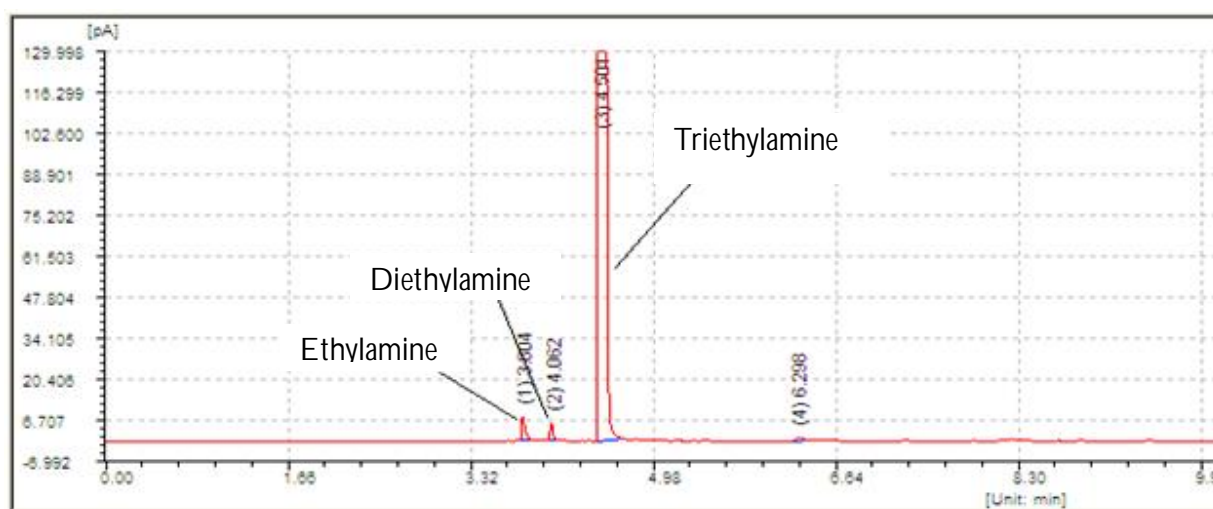
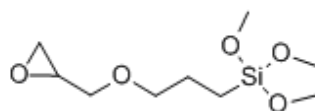


Figure 18-1. Chromatogram of Triethylamine determination

## 19. Analysis of $\gamma$ -(2,3- epoxypropoxy)propyltrimethoxysilane

$\gamma$ -(2,3- epoxypropoxy)propyltrimethoxysilane has the synonym of 3-Glycidoxy propyl trimethoxy silane and Silane coupling agent KH-560. Molecular formula is  $C_9H_{20}O_5Si$  and molecular weight is 236.34. The structure is



It is colorless and transparent liquid, soluble in most organic solvents. It easily hydrolyzes and easily form Polysiloxane via condensation reaction with high temperature, light and the existing of peroxide. Boiling point is 120°C at 2 mm

Hg(lit.). It is used as the Silane coupling agent and serves to improve the adhesion as the adhesive additive of Epoxy, polyurethane, phenol aldehyde and other resins. The sample is directly injected and chromatogram is recorded in Figure 19-1.

Column: GSBP-5/30m*0.32mm*0.25um	Oven: 100°C (2min) -10°C/min-250°C (10min)
Carrier: N2 pressure 0.06MPa	Inj: 260°C Det: FID,260°C
Split Flow: 80ml/min	Injection: 0.2ul
	Quantitative Method: Area normalization

Table 19-1. Instrumentation condition of  $\gamma$ -(2,3- epoxypropoxy)propyltrimethoxysilane analysis

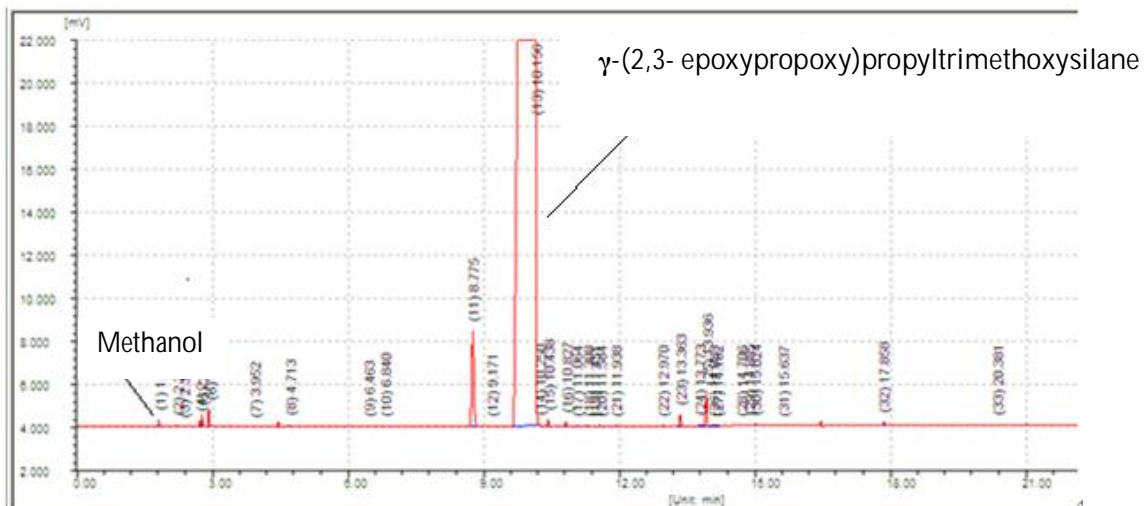


Figure 19-1. Typical Chromatogram of  $\gamma$ -(2,3- epoxypropoxy)propyltrimethoxysilane analysis

## 20. Analysis of Bing Pian (Borneolum Syntheticum)

As one of highly prized and very rare native Chinese herbs, Bing Pian (Borneol Flakes) has been extensively studied and used by generations of physicians in ancient China. Modern pharmacological study showed that it has multiple direct therapeutic effects. Very often it is classified into the species of inducing resuscitation traditionally, which makes it very popular in adjuvant therapy for a variety of conditions due to the excellent transdermal delivery ability. This crystalline particle, metaphorically speaking, is more like a needle than a piece of thread like other medicinal herbs. So to speak, this agent can deliver the medicine directly to the affected area while enhancing the therapeutic effect. For reason given above, it ever was, and is, and shall be, playing an important role in treatments of severe diseases.

Column: GSBP-INNOWAX/30m*0.32mm*0.50um	Oven : 100°C ( 2min ) -15°C/min-
Carrier: N2 pressure 0.08MPa	200°C ( 10min )
Split ratio: 80: 1	Injection: 0.6μL
Inj: 250°C Det: FID, 2400°C	Solvent: Chloroform
Quantitative method: Area normalization method	

Table20-1. instrumentation condition of Borneolum Syntheticum analysis

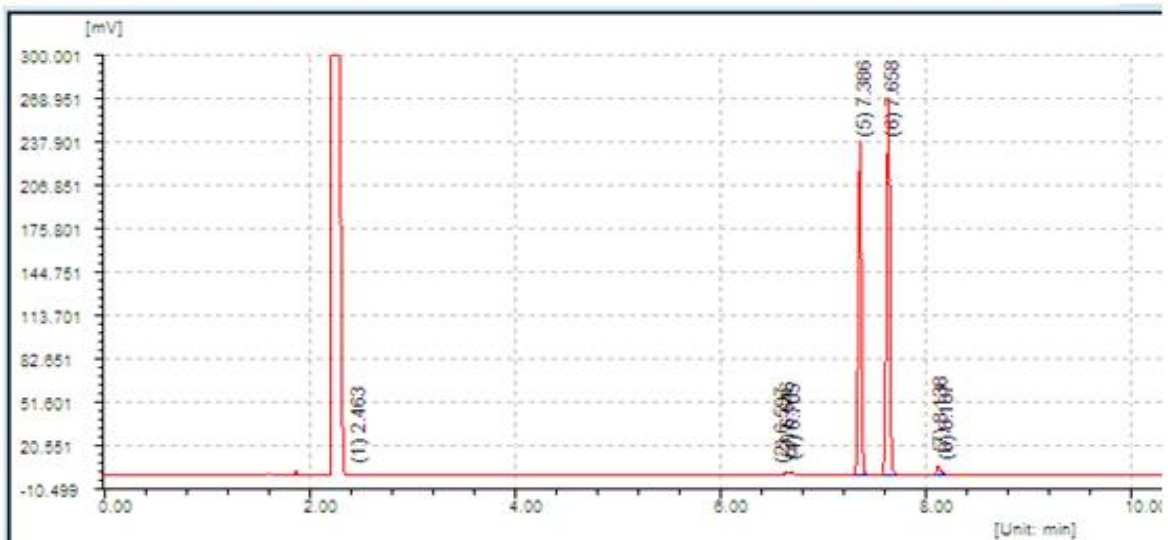


Figure 20-1. Synthetic Borneolum Syntheticum Analysis

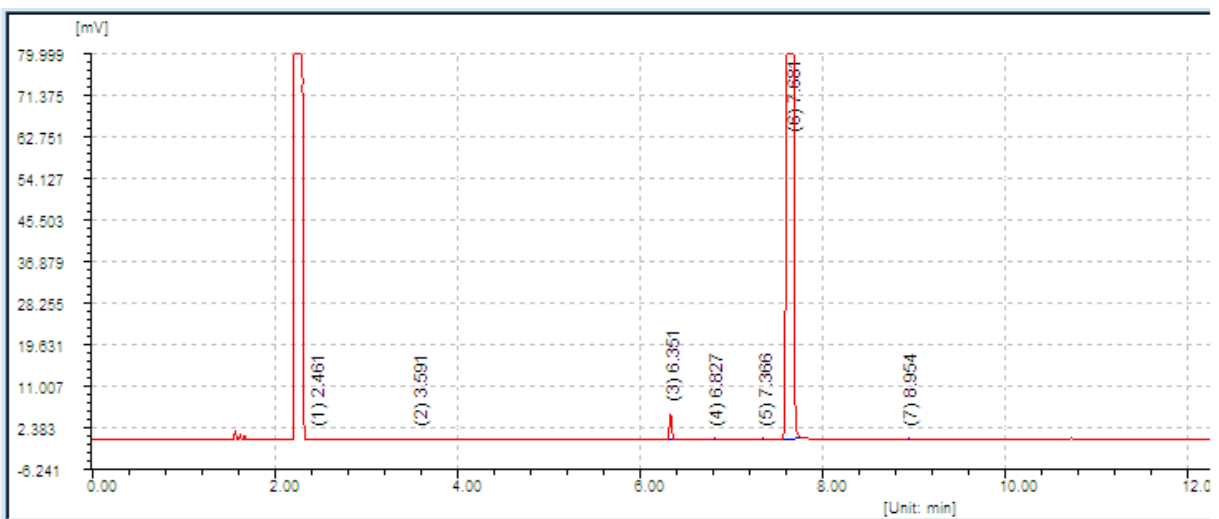
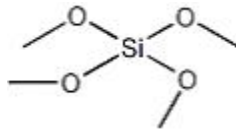


Figure 20-1. Natural Borneolum Syntheticum Analysis

## 21. Determination of Tetramethoxysilane

Tetramethoxysilane has the molecular formula of  $C_4H_{12}O_4Si$ . The molecular weight is 152.22. It is used in the sol-gel synthesis of chromium-doped silicates<sup>1</sup> and in the formation of hexagonal mesoporous silica layers. The structure is:



It is colorless liquid with the special odor and easily deliquescent. It is not soluble in water but could be soluble in most organic solvent. The boiling point is 121 °C. It is used as the optical glass processing agent in insulating material of the electronic industry or the binder in synthesis of Silicone. Tetramethoxysilane sample is tested by GC on GsBP-5 column with the proper condition in table 21-1 and the result is recorded in figure 12-1.

Oven: GSBP-5/30m*0.32mm*0.25um	Oven: 100°C (2min) -10°C/min-250°C (10min)
Carrier: N2, pressure0.06MPa	Inj: 260°C Det: FID, 260°C
Split ratio: 80ml/min	Injection: 0.2ul
	Quantitative method: Area normalization method

Table 12-1. the instrumentation condition of Tetramethoxysilane determination

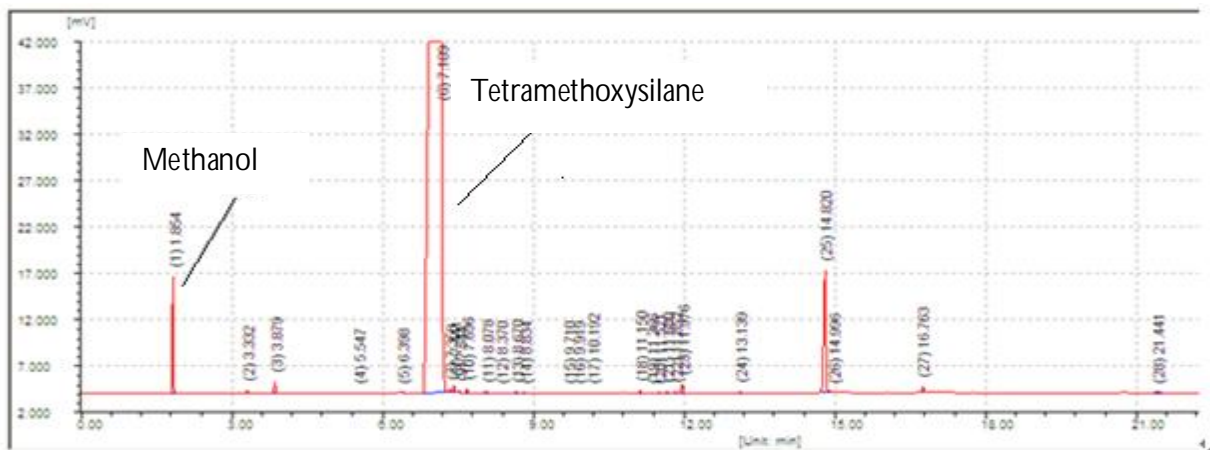


Figure 12-1. Chromatogram of Tetramethoxysilane determination

## 22. Phthalates Analysis

Phthalates are mainly used as plasticizers (substances added to plastics to increase their flexibility, transparency, durability, and longevity). Phthalates are manufactured by reacting phthalic anhydride with alcohol(s) that range from methanol and ethanol (C1/C2) up to tridecyl alcohol (C13), either as a straight chain or with some branching. They are divided into two distinct groups, with very different applications, toxicological properties, and classification, based on the number of carbon atoms in their alcohol chain. They are used primarily to soften polyvinyl chloride (PVC). Phthalates are used in a wide range of common products, and are easily released into the environment. Although there is no covalent bond between the phthalates and plastics, they are physically bound into the plastic as a result of the heating process used to make PVC articles. They can be removed only by exposure to severe heat or using strong solvents. However, people are exposed to phthalates, and most Americans tested by the Centers for Disease Control and Prevention have metabolites of multiple phthalates in their urine. Phthalate exposure may be through direct use or by indirect means through leaching and general environmental contamination. Diet is believed to be the main source of di(2-ethylhexyl) phthalate (DEHP) and other phthalates in the general population. Fatty foods such as milk, butter, and meats are a major source. In studies of rodents exposed to certain phthalates, high doses have been shown to change hormone levels and cause birth defects. The sample is analyzed by GC on GsBP-5 column. The condition used is shown in Table 22-1 and the chromatogram is recorded in Figure 22-1.

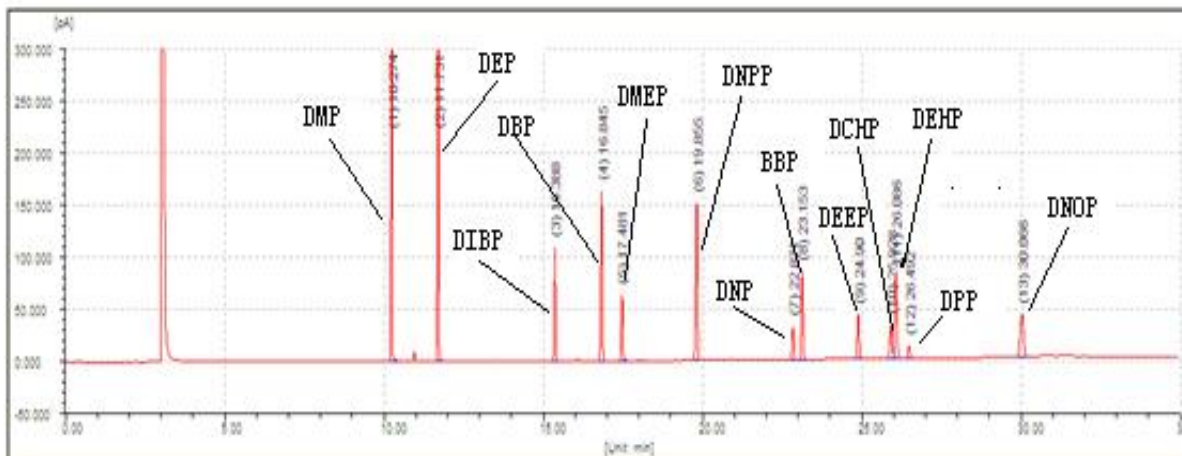


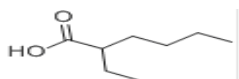
Figure 22-1. The chromatogram of Phthalates Analysis

Oven: GSBP-5/30m*0.32mm*0.25um	Oven: 80°C(1min)-10°C/min-260°C (15min )
Carrier: N2, flow 1.5ml/min	Inj: 280°C Det: FID, 280°C

Table 22-1. The instrumentation condition of Phthalates Analysis

### 23. Determination of Isooctyl acid

Isooctyl acid has a chemical name of 2-ethylhexanoic acid. The molecular formula is  $C_8H_{16}O_2$ . The molecular weight is 144.204. The structure is



It is colorless liquid with odor, soluble in hot water and ethyl ether and slightly soluble in ethanol. The boiling point is 228°C. It is used in organic synthesis in chemical and pharmaceutical industry. The Isooctyl acid sample is tested by GC using GsBP-FFAP column. The instrumentation condition is shown in table 23-1 and result is in figure 23-1.

Column: GsBP-FFAP/30m*0.32mm*0.50um	Oven : 100 °C ( 1min ) -15 °C /min-240 °C (5min)
Carrier: N2 pressure 0.1MPa	Inj: 260°C Det: FID,250°C
Split ratio: 100ml/min	Injection: 0.2ul
	Quantitative method: Area normalization method

Table 23-1. the Instrumentation condition of Isooctyl acid analysis

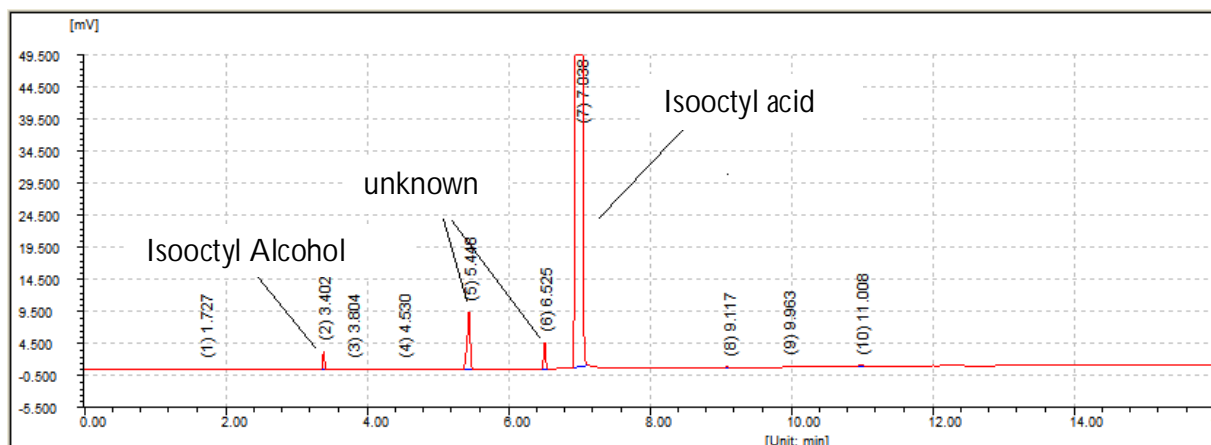
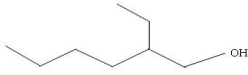


Figure 23-1. The chromatogram of Isooctyl acid analysis

## 24. Determination of Isooctyl alcohol

Isooctyl alcohol has a chemical name of 2-ethyl-1-hexanol. The molecular formula is  $C_8H_{18}O$ . The molecular weight is 130.23.

The structure is 

Isooctyl alcohol is clear colorless flammable liquid with special odor and mixable with most organic solvent. The boiling point is 185-189 °C. It is used to produce plasticizing agent, defoaming agent, dispersing agent, dressing agent and petroleum additive. Before testing, the isooctyl alcohol sample is directly injected or diluted with solvent. Then it is analyzed by GC using GsBP-FFAP column with the proper condition in table 24-1. The result is recorded in Figure 24-1.

Column: GsBP-FFAP/30m*0.32mm*0.50um	Oven : 100 °C ( 1min ) -15 °C /min-240 °C (5min)
Carrier: N2, pressure 0.1MPa	Inj: 260°C Det: FID, 250°C
Split flow: 100ml/min	Injection: 0.2ul
Quantitative method : Area normalization method	

Table 24-1. The instrumentation condition of Isooctyl alcohol determination



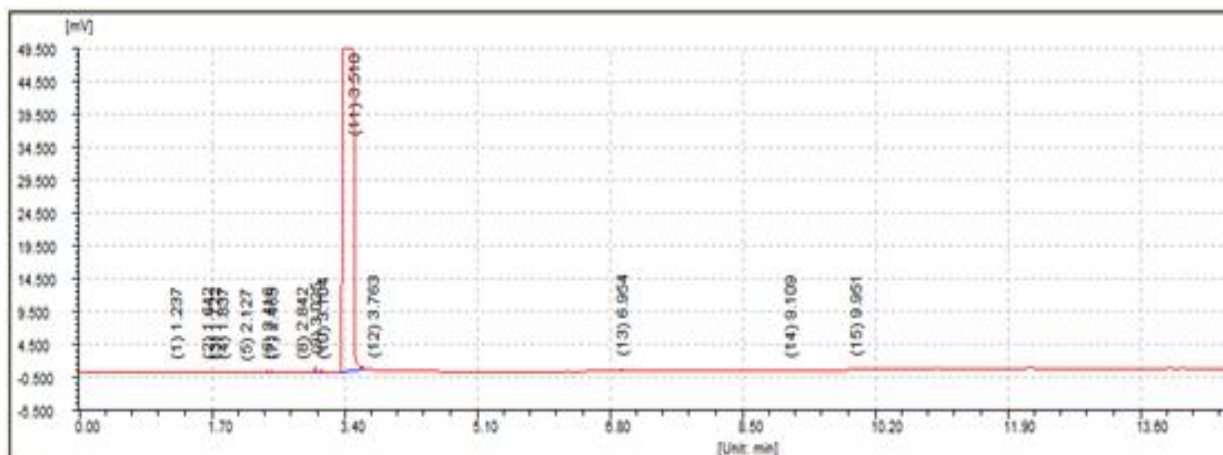


Figure 24-1. Chromatogram of Isooctyl alcohol Determination

## 25. Selection of phenoxyvinylether Separation Condition

Column: GsBP-50+MS /30m*0.32mm* 0.25um	Oven : 80 °C ( 2min ) -20 °C /min-250 °C (20min)
Carrier: N2 Flow 2.0ml/min	Inj: 260°C Det: FID,260°C
Split ratio: 40: 1	Injection: 0.2ul
	Quantitative method : Area normalization method

Table 25-1. the instrumentation condition of phenoxyvinylether separation

# 1\* phenyl ether

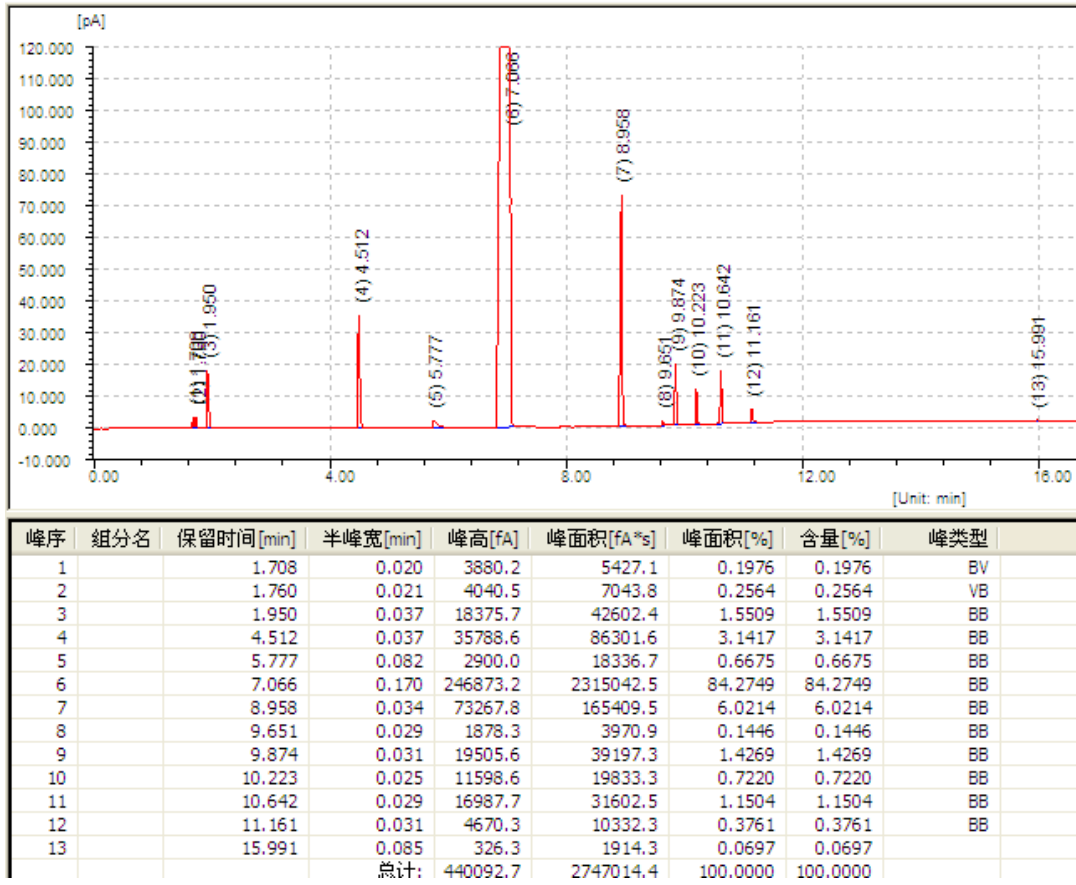


Figure 25-1. The chromatogram of 1\*phenyl ether.

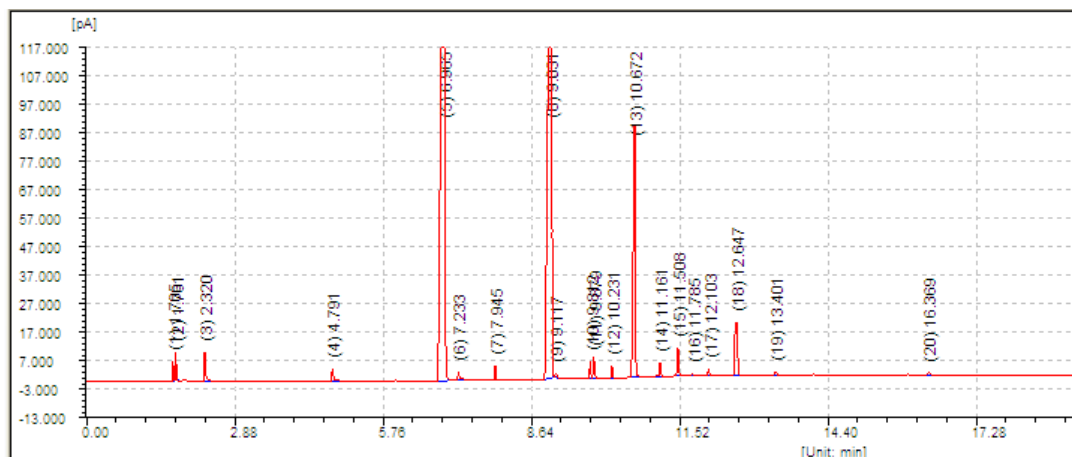


Figure 25-2. The chromatogram of 2\*phenyl ether.

Conclusion: we selected columns with different polarity to figure out the optimal separation conditions for comparison. The baseline separation of different composition in phenyl ether sample could be achieved on GsBP-50+MS columns. With the condition shown as above, benzene is eluted at 2.09min and vinyl benzene is eluted at 3.5min.

## 26. Analysis of Decamethylene diamine and Sebaconitrile

Decamethylene diamine sample and Sebaconitrile sample are tested by GC using GsBP-5 column. The instrumentation condition used is shown in table 26-1. The results are recorded in Figure 26-1 and Figure 26-2, respectively.

Column: GSBP-5/30m*0.32mm*0.25um	Injection: 0.6ul
Carrier: N2, flow 0.8ml/min	Solvent: ethanol
Split ratio: 60: 1	Quantitative method: Area normalization method
Oven: 100°C (2min) -10°C/min-250°C (10min)	
Inj: 260°C Det: FID,260°C	

Table 26-1. The instrumentation condition of decamethylene diamine analysis

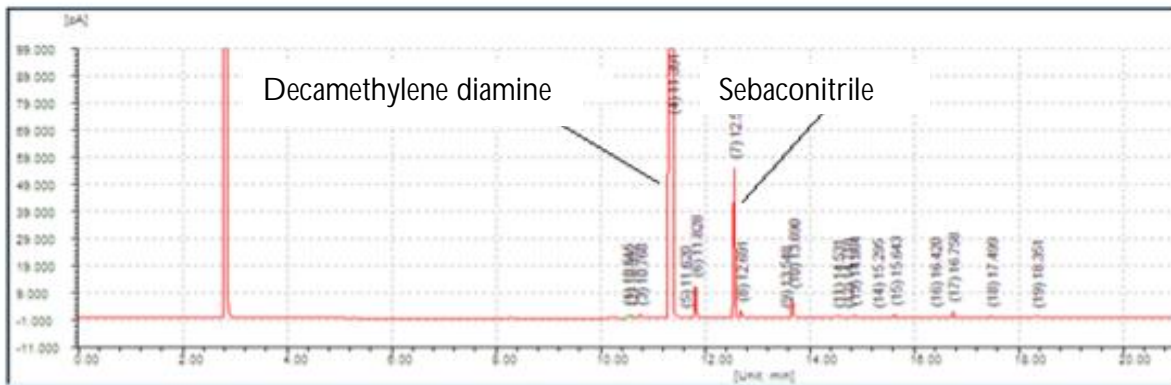


Figure 26-1. The chromatogram of decamethylene diamine analysis

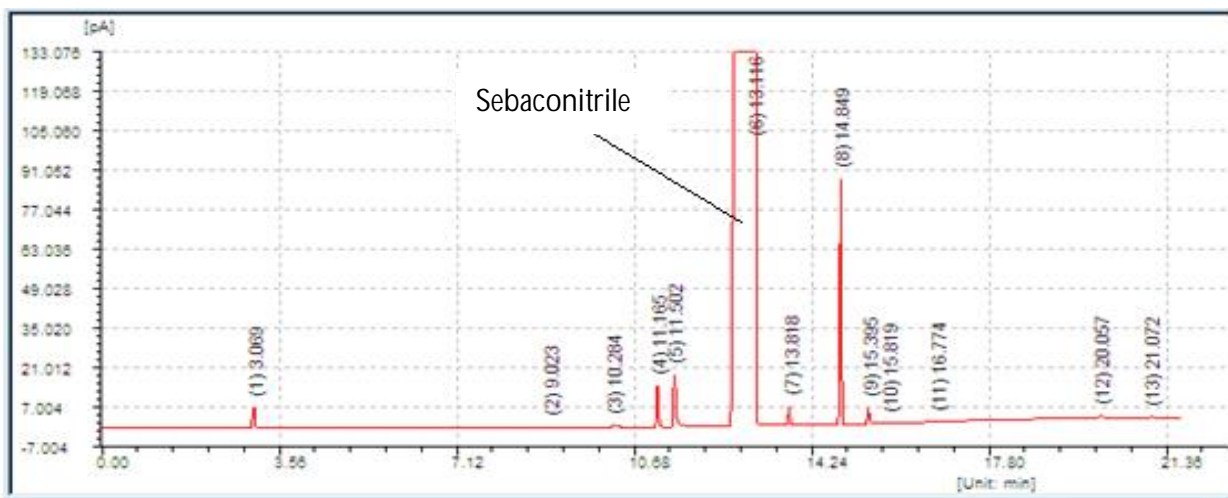


Figure 26-2. The chromatogram of sebaconitrile analysis

## 27. Determination of diallyl phthalate ( DAP )

DAP is soluble in ethanol, acetone, ethyl ether and other organic solvents, slightly soluble in mineral oil, ethylene glycol and glycerol, but not soluble in water. The sample is tested by GC on GsBP-5 column with proper condition in table 27-1. The result is recorded in Figure 27-1.

Column: GSBP-5 /30m*0.32mm*0.25um	Oven: 150°C (2min) -20°C/min-250°C (10min)
Carrier: N2, pressure 0.06MPa	Inj: 250°C Det: FID, 260°C
Split ratio: 80: 1	Injection: 0.2ul

Table 27-1. the instrumentation condition of DAP analysis

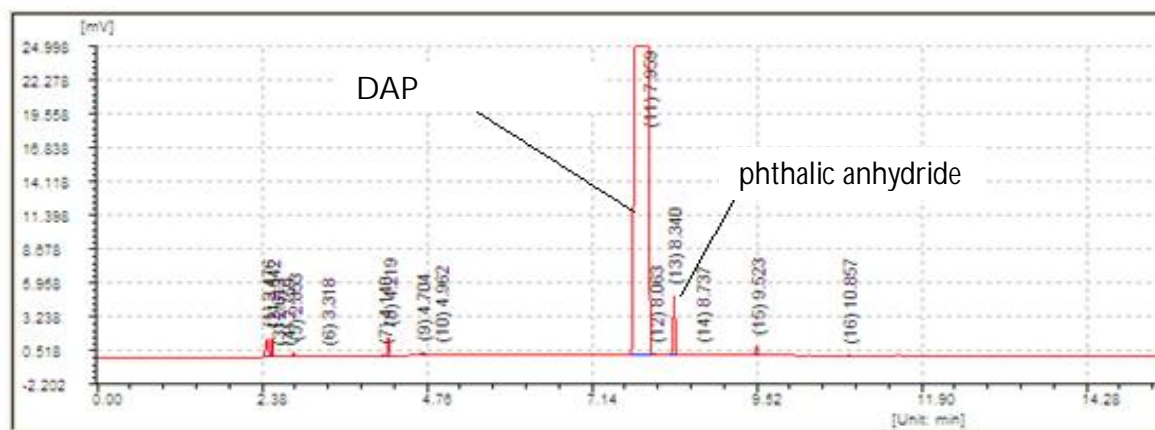


Figure 27-1. the chromatogram of DAP analysis

## 28. Analysis of higher saturated fatty alcohols

Fatty alcohols (or long-chain alcohols) are usually high-molecular-weight, straight-chain primary alcohols, but can also range from as few as 4-6 carbons to as many as 22-26, derived from natural fats and oils. The precise chain length varies with the source. Some commercially important fatty alcohols are lauryl, stearyl, and oleyl alcohols. They are colourless oily liquids (for smaller carbon numbers) or waxy solids, although impure samples may appear yellow. They are mainly used in the production of detergents and surfactants. They are components also of cosmetics, foods, and as industrial solvents. Due to their amphipathic nature, fatty alcohols behave as nonionic surfactants. They find use as emulsifiers, emollients and thickeners in cosmetics and food industry. About 50% of fatty alcohols used commercially are of natural origin, the remainder being synthetic. The sample containing different higher saturated fatty alcohols is analyzed by GC on GsBP-5 columns with proper condition shown in table 28-1. The result is recorded in Figure 28-1.

Column: GSBP-5 /30m*0.32mm*0.5um	Oven: 220°C
Carrier: N2, pressure 80KPa	Inj: 240°C Det: FID, 260°C
Split ratio: 80: 1	Injection: 0.2ul

Table 28-1. the instrumentation condition of higher saturated fatty alcohols analysis

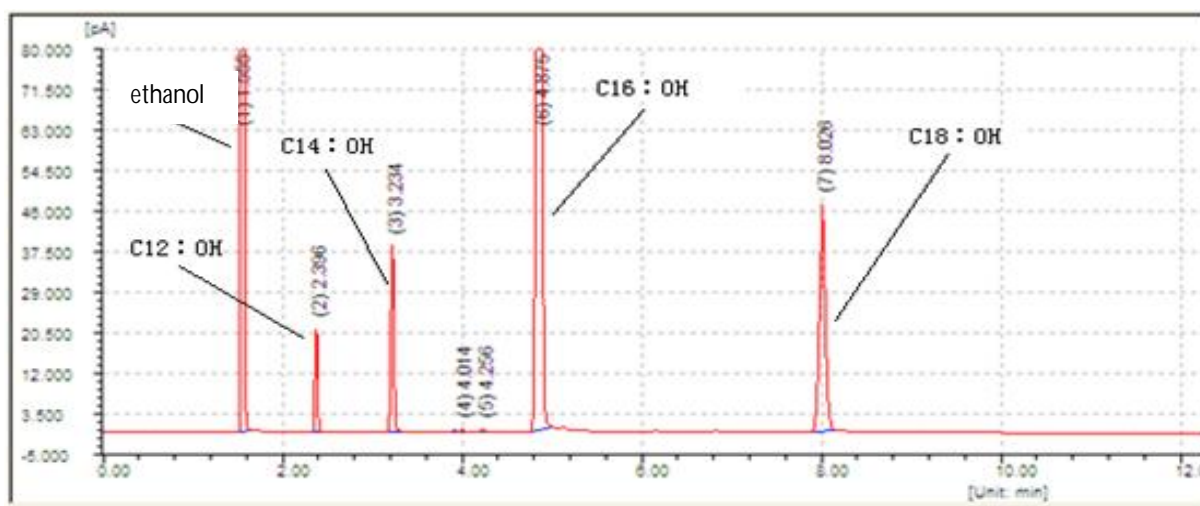


Figure 28-1. The chromatograph of higher saturated fatty alcohols analysis

## 29. Determination of 2-Nitrochlorobenzene

2-Nitrochlorobenzene is an organic compound with the formula  $C_6H_4ClNO_2$ . It is a yellow crystalline solid that is important as a precursor to other compounds due to the two reactive sites present on the molecule. 2-Nitrochlorobenzene is not valuable in itself but is a precursor to other useful compounds. The compound is particularly useful because both of its reactive sites can be utilized to create further compounds that are mutually ortho. Its derivative 2-chloroaniline is a precursor to 3,3'-dichlorobenzidine, itself a precursor to many dyes and pesticides. The sample is tested by GC on GsBP-1 column with proper condition in table 29-1. The chromatogram is recorded in figure 29-1.

Column: GsBP-1 /30m*0.32mm*1.0um	Oven: 100°C (2min) -10°C/min-250°C (5min)
Carrier: N2, flow 1.5ml/min	Inj: 250°C Det: FID, 260°C
Split ratio: 50: 1	Injection: 0.2ul

Table 29-1. The instrumentation condition of 2-Nitrochlorobenzene analysis

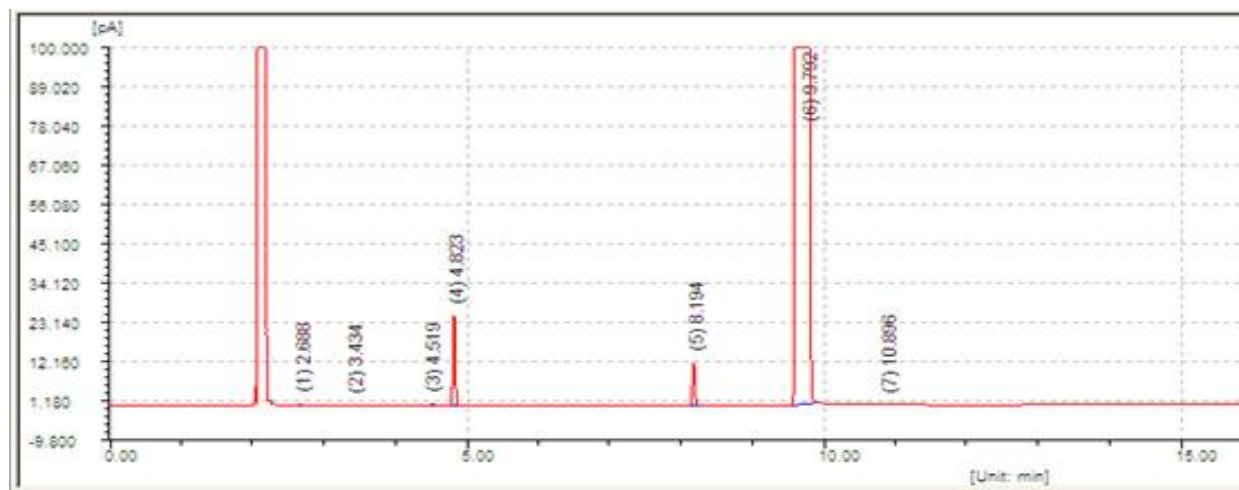
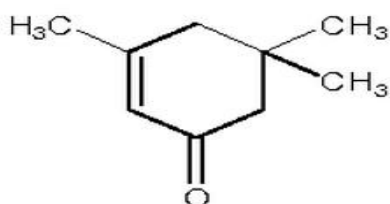


Figure 29-1. The chromatogram of 2-Nitrochlorobenzene analysis

### 30. Analysis of isophorone

Isophorone is an  $\alpha,\beta$ -unsaturated cyclic ketone, a colorless to yellowish liquid with a characteristic peppermint-like smell. It is used as a solvent and as an intermediate in organic synthesis. Isophorone also occurs naturally in cranberries. Isophorone is used as a solvent in some printing inks, paints, lacquers, adhesives, copolymers, coatings, finishings and pesticides. It is also used as a chemical intermediate and as an ingredient in wood preservatives and floor sealants. The structure is



The sample is tested by GC on GsBP-FFAP with the following condition:

Column:GsBP-FFAP/30m×0.32mm×0.50um

Oven: 80°C(2min)-15°C/min-160°C (5min)

Gasification chamber temperature : 250°C detector: 230°C

Mode: constant pressure 60KPa

Split ratio: 80: 1 Solvent: Dichloromethane

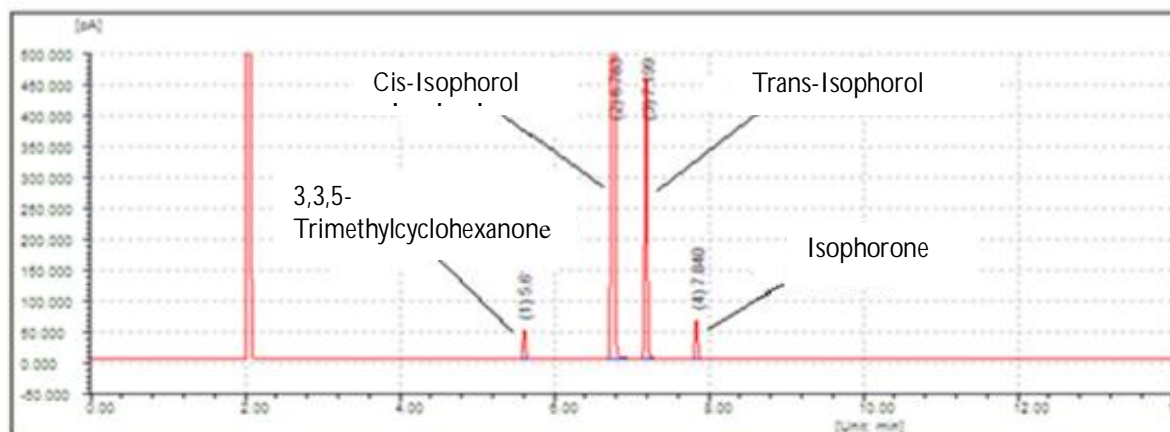


Figure 30-1. chromatogram of Isophorone analysis.

### 31. Determination of Neopentyl glycol (NPG)

NPG has a chemical name of 2,2-dimethyl-1,3-Propanediol, the abbreviation is Neo or NPG. The molecular formula is C<sub>5</sub>H<sub>12</sub>O<sub>2</sub> and molecular weight is 104.15.

The structure is



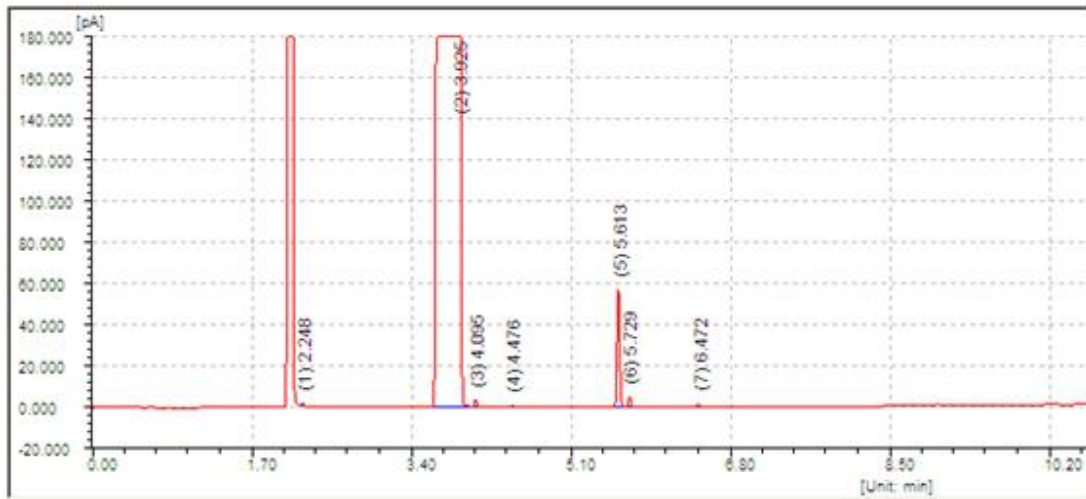
NPG is white crystalline solid, odorless and hygroscopic. The autoignition temperature is 399 °C. The sublimation point is 210 °C. It is soluble in water, lower alcohols, lower ketones, ethers and aromatic compounds, etc. The melting point is 124 ~ 130 °C and the boiling point is 210 °C. It is mainly used in the synthetic production process of chemical fibers, paints and lubricants. As the customer required, we need to explore the optimal separation conditions and achieve repeatability test. Before the experiment, the sample is diluted with ethanol or methanol with a certain proportion and tested by GC using the autosampler. The chromatogram is recorded in Figure 31-1.



Column:GSBP-50+MS/ 30m*0.32mm*0.25um Carrier:N2, flow 1.5ml/min Split ratio: 30:1	Oven: 100°C (2min) -15°C/min-250°C Inj: 280°C Det: FID, 280°C Injection: 1.0ul solvent: methanol Quantitative method: Area normalization method
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Table 31-1. The instrumentation condition of NPG determination using GsBP-50+MS

### 1#NPG



### 2#NPG

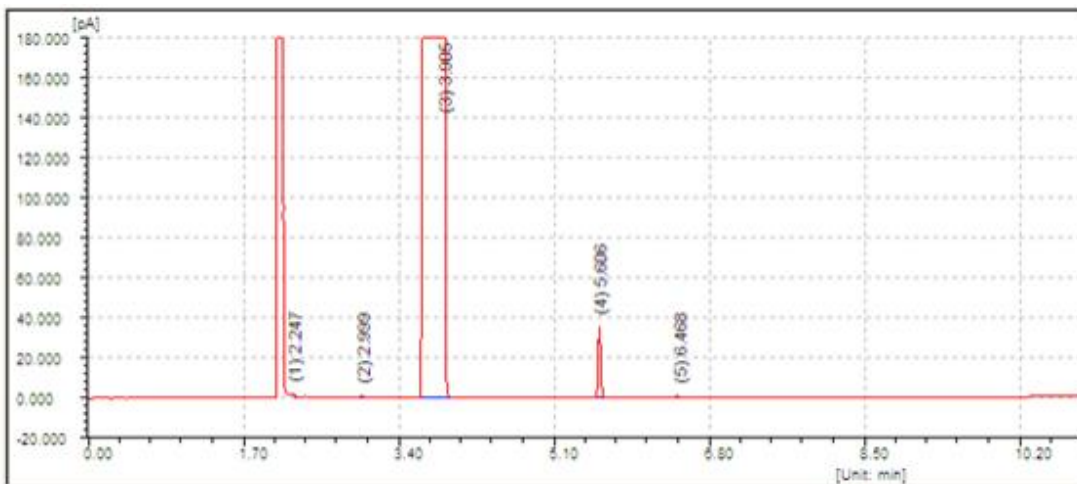


Figure 31-1. the chromatogram of 1#NPG and 2#NPG analysis.

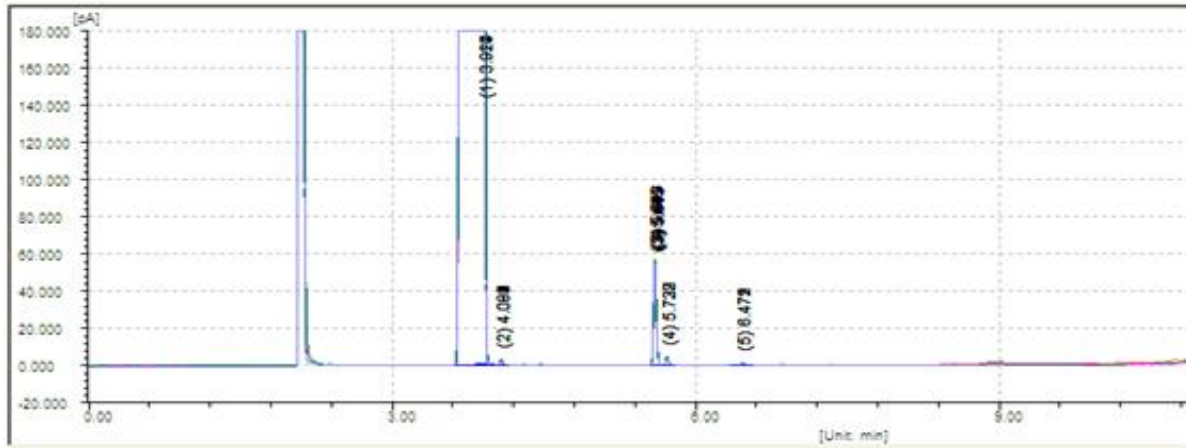


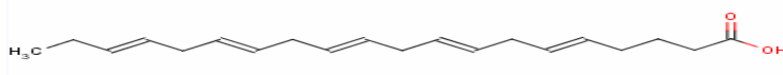
Figure 31-1. The chromatogram of 1#NPG sample in six consecutive injection repeatability test

## 32. Fish oil analysis

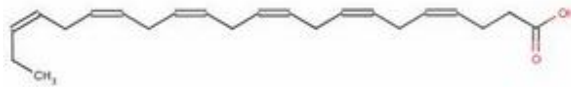
Fish oil Fish oil is oil derived from the tissues of oily fish. Fish oils contain the omega-3 fatty acids eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), precursors of certain eicosanoids that are known to reduce inflammation in the body, and have other health benefits. The fish used as sources do not actually produce omega-3 fatty acids, but instead accumulate them by consuming either microalgae or prey fish that have accumulated omega-3 fatty acids, together with a high quantity of antioxidants such as iodide and selenium, from microalgae, where these antioxidants are able to protect the fragile polyunsaturated lipids from peroxidation. Fatty predatory fish like sharks, swordfish, tilefish, and albacore tuna may be high in omega-3 fatty acids, but due to their position at the top of the food chain, these species may also accumulate toxic substances through biomagnification. For this reason, the United States Environmental Protection Agency recommends limiting consumption (especially for women of childbearing age) of certain (predatory) fish species (e.g. albacore tuna, shark, king mackerel, tilefish and swordfish) due to high levels of toxic

contaminants such as mercury, dioxin, PCBs and chlordanes.

The structure of EPA is shown as follows (molecular formula: C<sub>20</sub>H<sub>30</sub>O<sub>2</sub>, the boiling point is 468.9°C).



The structure of DHA is shown as follows (molecular formula: C<sub>22</sub>H<sub>32</sub>O<sub>2</sub>, the boiling point is 447°C).



As the customer required, we used area normalization method to calculate the content of EPA and DHA. Before testing, 1g fish oil sample is weighted in 25ml beaker and dissolved in 10ml Hexane and 5ml 10% KOH-methanol. The solution is mixed by 1min ultrasonication and rinsed with 5ml distilled water. Let the solution stand and absorb the upper organic layer to analyze.

Column: GsBP-INOWAX/30m*0.32mm*0.50um GsBP-FFAP/30m*0.32mm*0.25um Carrier: N <sub>2</sub> , flow 1.0ml/min Split ratio: 70:1	Oven : 170°C ( 1min ) 3°C/min-240°C ( 30min ) Inj: 260°C Det: FID, 260°C Injection: 0.2ul
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Table 32-1. the instrumentation condition of fish oil analysis.

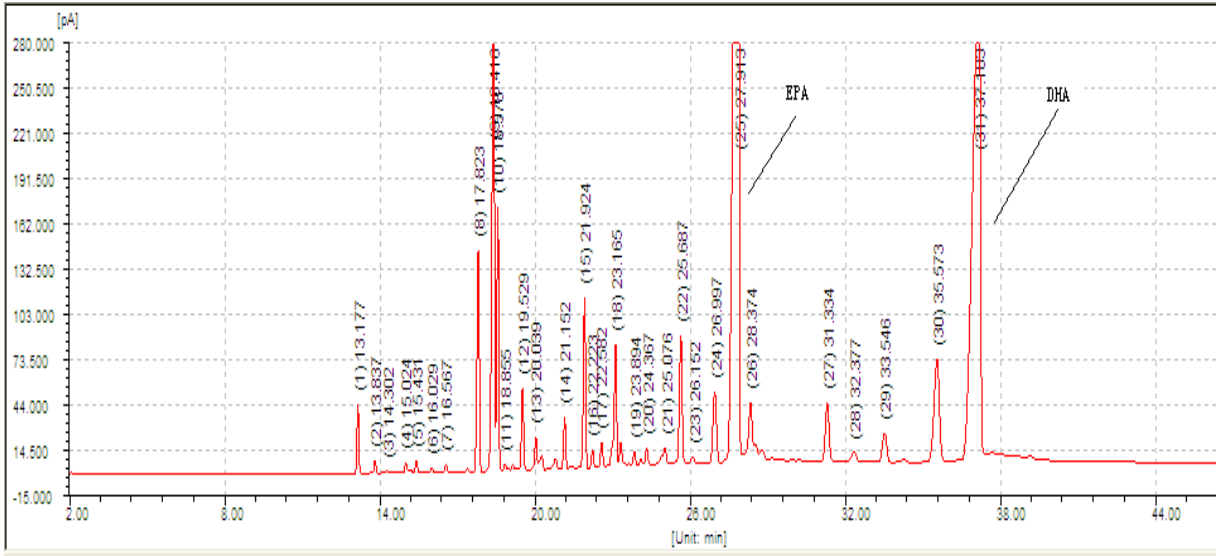


Figure 32-1. the chromatogram of fish oil analysis using GsBP-INOWAX/30m\*0.32mm\*0.50um

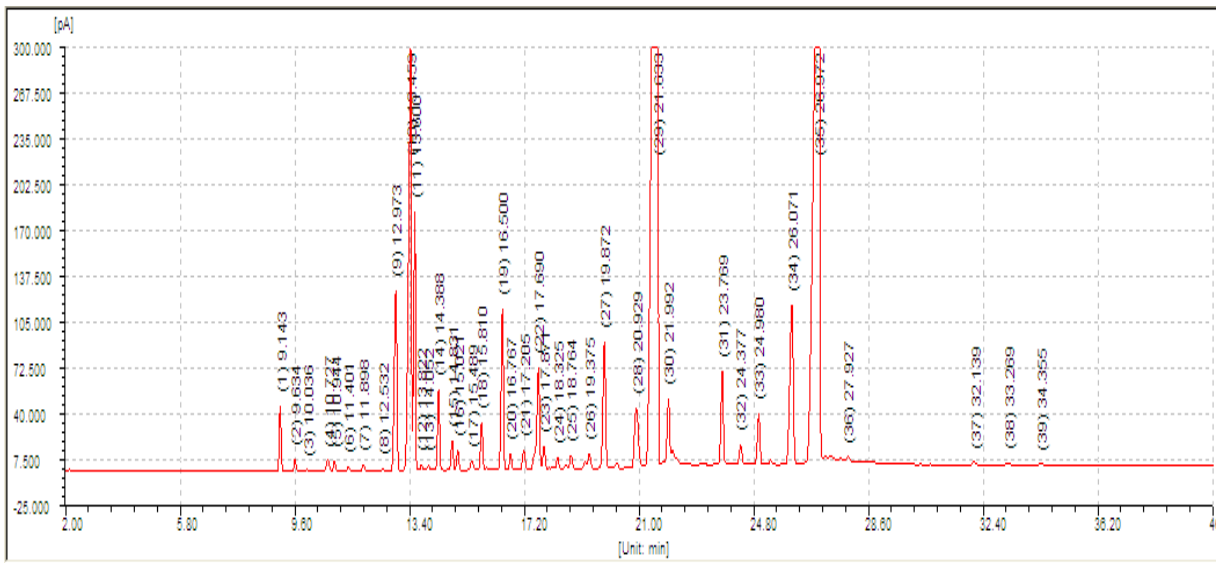


Figure 32-1. the chromatogram of fish oil analysis using GsBP-FFAP/30m\*0.32mm\*0.25um

1. Determination of 1,6-Hexanediol using 0532-3002
2. Separation of 2,4- Difluoronitrobenzene isomers using 0132-3010
3. Determination of 2,4-Difluoro-3,5-dichloronitrobenzene using 0132-3010
4. Separation of Acetophenone using 2032-3005
5. Determination of Acrylamide with internal standard method using 0532-3002
6. Separation of Alkaline Solvents using 2232-3002
7. Test Report of Benzoyl Chloride using 0532-3002
8. Chromatographic Analysis of BTEX using 2032-3005
9. Determination of Butyl acrylate using 0532-3002
10. Analysis of Chloropropanone using 6132-3005/6132-3010
11. Determination of Diethanolamine using 6132-3010
12. Determination of lambda cyhaltrin acid using 2132-3002
13. Analysis of N- Methylpyrrolidone (NMP) using 6132-3010
14. O-phenylenediamine Analysis Report using 5032-3002
15. Separation of Diethanolisopropanolamine and Tris(2-Hydroxyethyl)Amine on 2032-3005
16. Determination of Spermine using 5032-3002
17. Determination of Tributylamine using 0132-3010
18. Determination of Triethylamine using 0132-3010
19. Analysis of  $\gamma$ -(2,3- epoxypropoxy)propyltrimethoxysilane using 0532-3002
20. Analysis of Bing Pian (Borneolum Syntheticum) using 2032-3005
21. Determination of Tetramethoxysilane using 0532-3002
22. Phthalates Analysis using 0532-3002
23. Determination of Isooctyl acid using 2132-3005
24. Determination of Isooctyl alcohol using 2132-3005
25. Selection of phenoxyvinylether Separation Condition using 5032-3002
26. Analysis of Decamethylene diamine and Sebaconitrile using 0532-3002
27. Determination of diallyl phthalate ( DAP )using 0532-3002
28. Analysis of higher saturated fatty alcohols using 0532-3005
29. Determination of 2-Nitrochlorobenzene using 0132-3010
31. Determination of Neopentyl glycol (NPG) using 5032-3002
32. Fish oil analysis using 2032-3005 and 2132-3002