CYDEX-B

FOR OPTICAL AND POSITIONAL ISOMERS

PRODUCT DATA

For the Analysis of Optical and Positional Isomers by Capillary GC

INTRODUCTION

Application areas:

- Pharmaceuticals
- Natural Products
- Flavours
- Biological

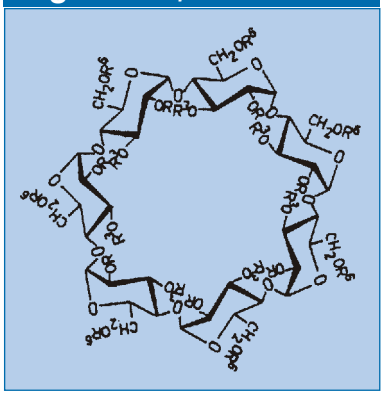
β-Cyclodextrin is an important advance in capillary gas chromatography for the separation of chiral optically active and positional isomers.

β-Cyclodextrin affects separation by selectively retaining compounds in the cavity of the structure (see **figure 1**). Compounds which exhibit greater retention in the cavity will elute later.

Cydex-B phase consists of β-Cyclodextrin dissolved in a moderately polar stationary phase. The phase is not cross linked, therefore washing of Cydex-B columns is not recommended.

Cydex-B columns combine the inertness and strength of fused silica columns with the unique properties of β-cyclodextrin enabling the separation of a wide variety of chiral and positional isomers without the need for compound derivatisation.

Figure 1 - β-CYCLODEXTRIN



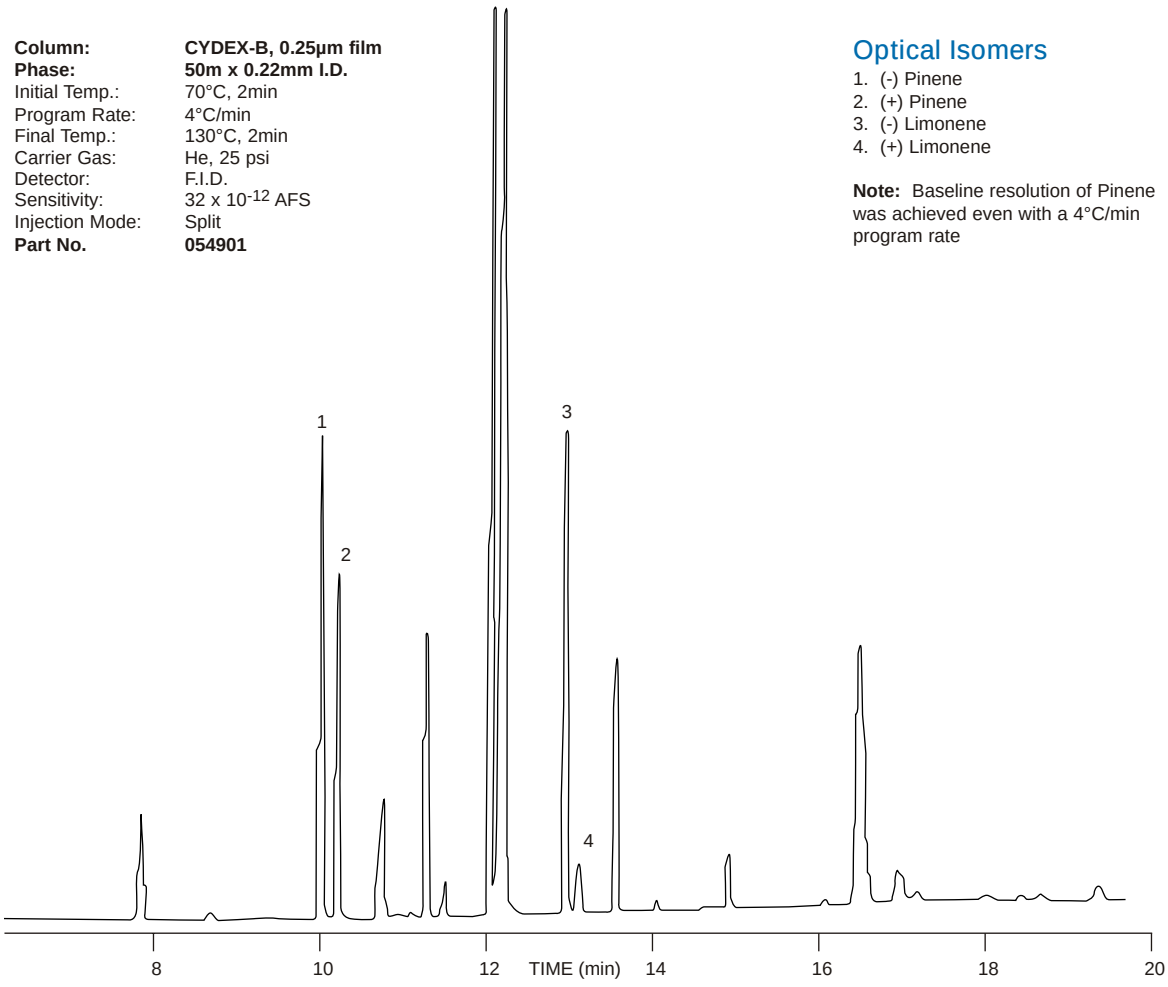
PINE OIL (PINUS SILVESTRIS)

Column:	**CYDEX-B, 0.25µm film**
Phase:	**50m x 0.22mm I.D.**
Initial Temp.:	70°C, 2min
Program Rate:	4°C/min
Final Temp.:	130°C, 2min
Carrier Gas:	He, 25 psi
Detector:	F.I.D.
Sensitivity:	32 x 10^{-12} AFS
Injection Mode:	Split
Part No.	**054901**

Optical Isomers

1. (-) Pinene
2. (+) Pinene
3. (-) Limonene
4. (+) Limonene

Note: Baseline resolution of Pinene was achieved even with a 4°C/min program rate



APPLICATION AREAS FOR CYDEX-B

The sensitivity of capillary gas chromatography using Cydex-B, enables the determination of enantiomeric purity to better than 0.1% ideal for a wide range of applications.

Application areas include:

- **Pharmaceuticals**

Enantiomeric purity of pharmaceutical precursors, intermediates or final products. This is important when products must be composed of only one enantiomeric from where the other may be inactive or toxic.

- **Natural Products**

GC chromatography using Cydex-B can be used to determine the absolute configuration of chiral natural products. Purity analysis of natural products for the existance of enantiomer or the ratio of enantiomers can be used to determine if an expensive natural product is adulterated or even replaced with a less expensive synthetic equivalent.For example see **figures 2a, 2b and 2c.**

- **Flavors**

Determination of the absolute configuration of flavor constituents.

- **Biological**

Determination of enantiospecificity of enzymic or microbiological transformations.

Figure 2a - LAVENDER OIL LINALOOL STANDARD

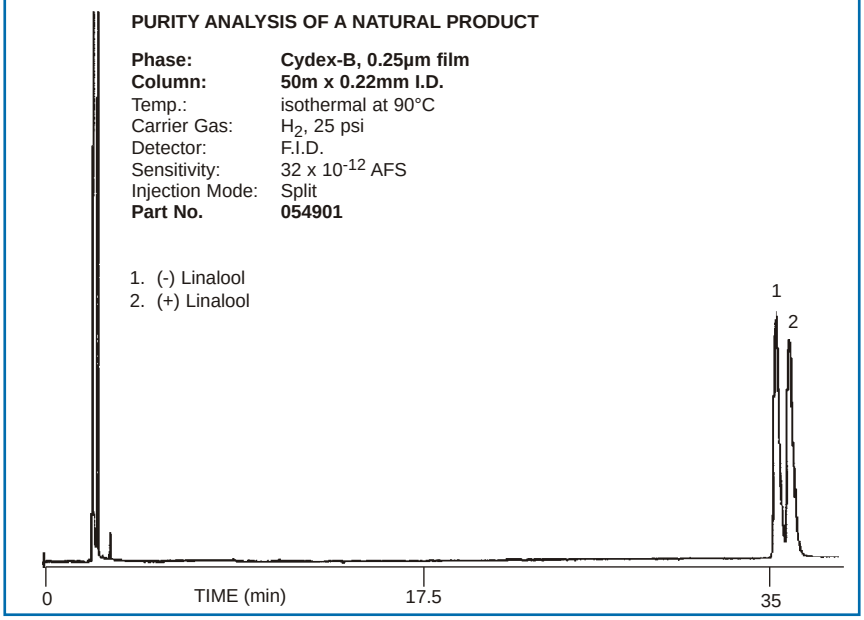


Figure 2b - UNADULTERATED (Clean) LAVENDER OIL

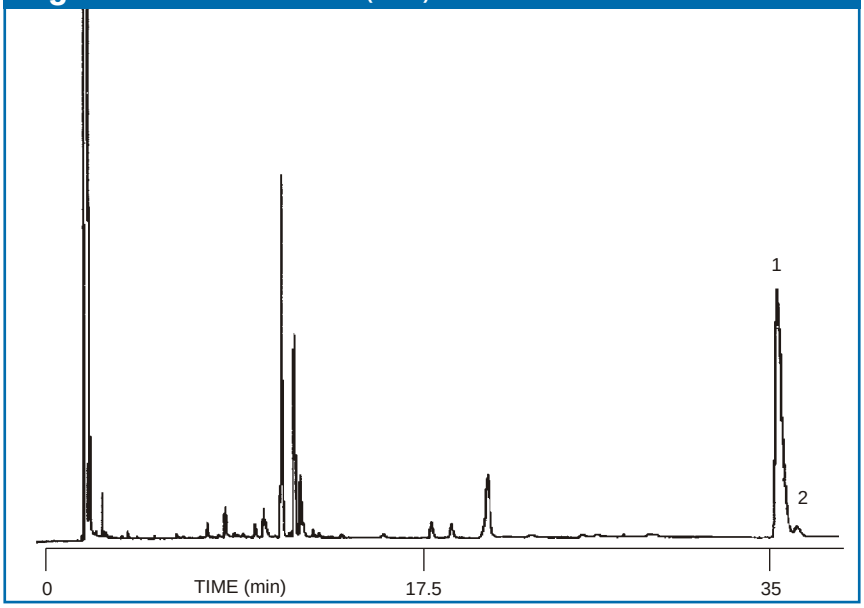
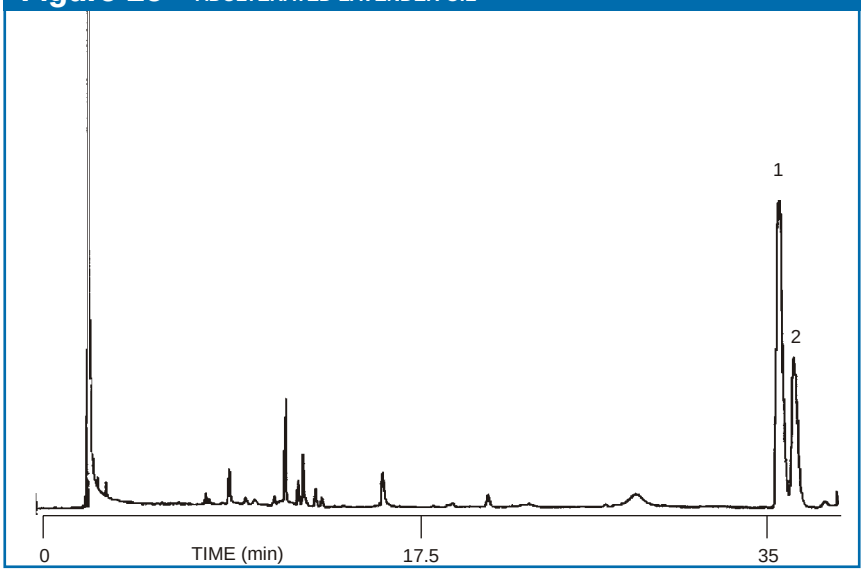


Figure 2c - ADULTERATED LAVENDER OIL



α-IONONE IN RASPBERRY PUREE

The chromatogram of a raspberry puree is extremely complex containing many hundred components. In this case α-Ionone was the component of interest which has an odor reminiscent of cedar wood.

In order to identify the respective R, S optical isomers of α-Ionone multidimensional chromatography was utilized. The isolated fraction containing the α-Ionone can then be analyzed using a Cydex-B capillary column

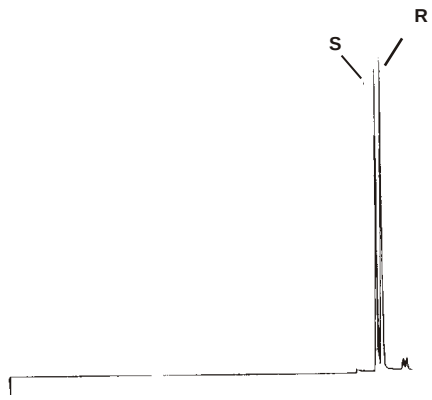
ACKNOWLEDGMENT

SGE is grateful to M. P. Debrill and Mme. C. Schippa of Sanofi Bio-Industries, Grasse, France for allowing the use of the chromatograms shown in this application.

Phase:	**Cydex-B**
Column:	**50m x 0.25mm I.D.**
Initial Temp.:	30°C
Rate:	20°C
Final Temp.:	140°C, 20 min
Carrier Gas:	Helium
Detector:	F.I.D.
Injection Mode:	Concentrated Headspace
Part No:	**054901**

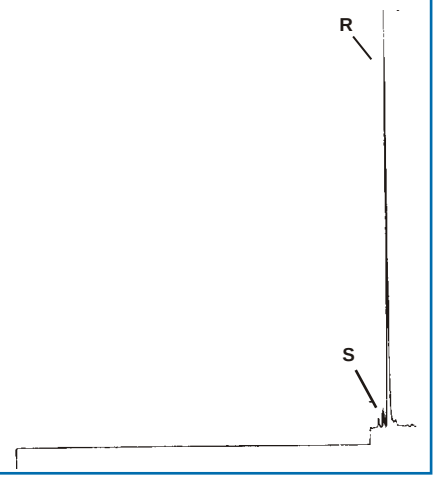
α-IRONE STANDARD

R. 50.1%
S. 49.9%



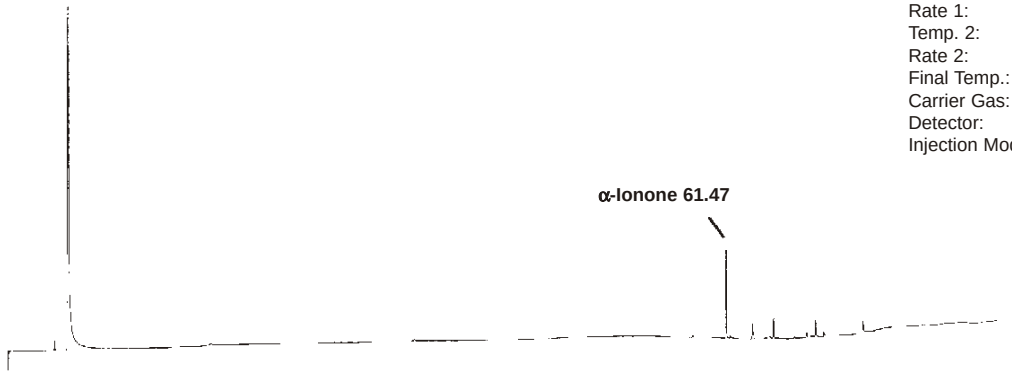
α-IRONE FROM RASPBERRY EXTRACT

R. 97.5%
S. 4.3%

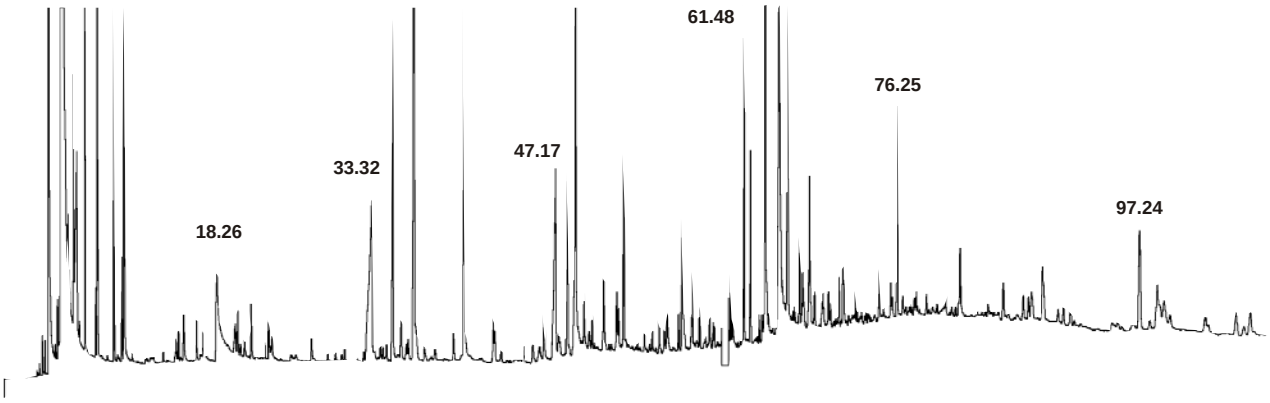


a-IRONE STANDARD

Phase:	**BP1, 0.5µl**
Column:	**50m x 0.32mm I.D.**
Initial Temp.:	40°C
Rate 1:	2°C/min
Temp. 2:	130°C
Rate 2:	4°C
Final Temp.:	250°C
Carrier Gas:	Helium
Detector:	F.I.D.
Injection Mode:	Splitless



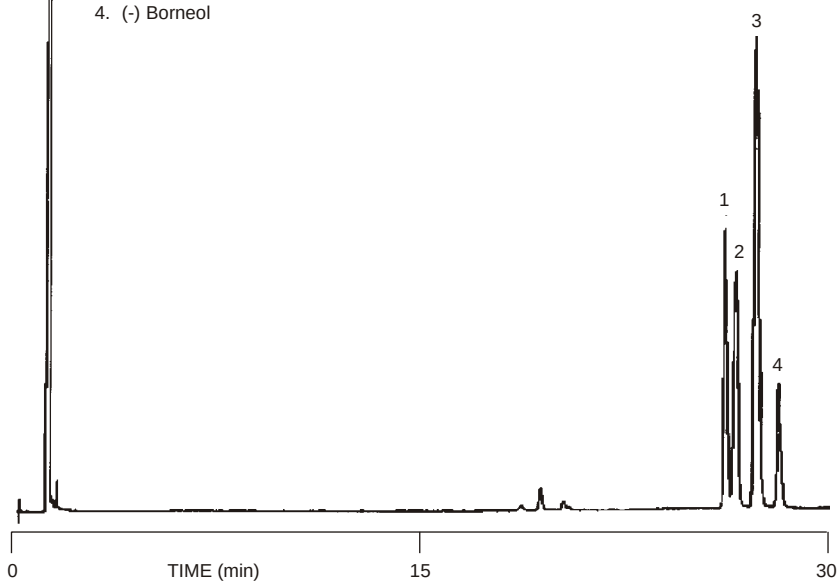
RASPBERRY EXTRACT



BORNEOLS

Phase: **Cydex-B, 0.25µm film**
Column: **25m x 0.22mm I.D.**
Initial Temp.: 90°C, 1min
Rate: 2°C/min
Final Temp.: 130°C
Carrier Gas: H_2, 12 psi
Detector: F.I.D.
Sensitivity: 32 x 10^{-12} AFS
Injection Mode: Split
Part No. 054900

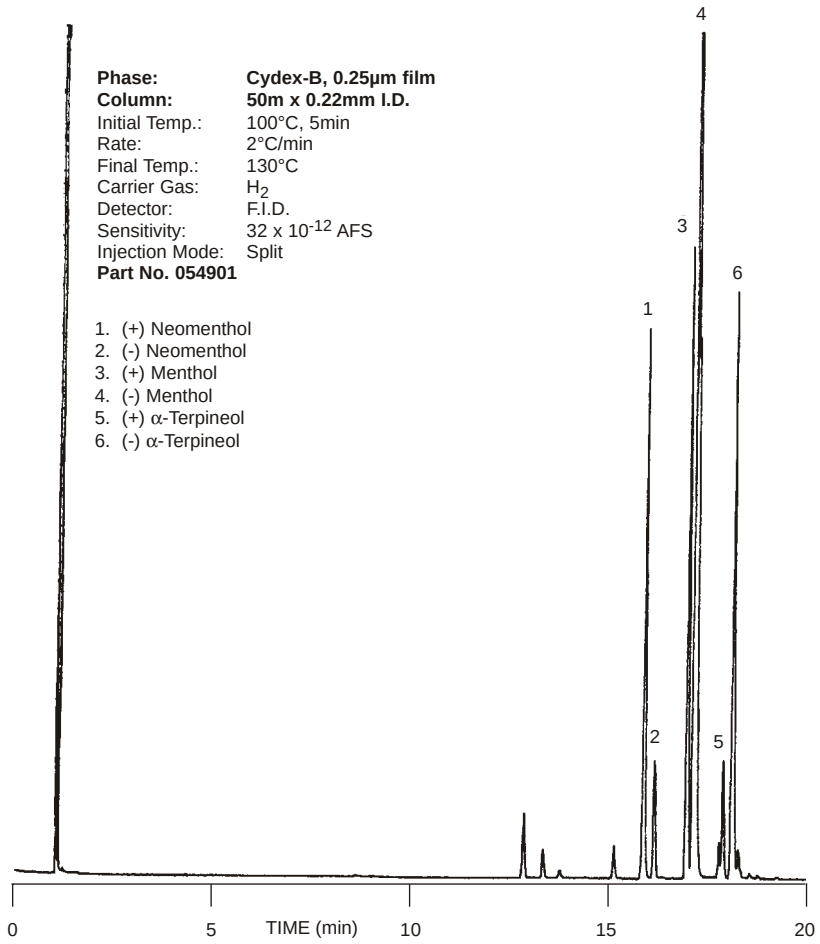
1. (+) Iso-borneol
2. (-) Iso-borneol
3. (+) Borneol
4. (-) Borneol



MENTHOL OIL

Phase: **Cydex-B, 0.25µm film**
Column: **50m x 0.22mm I.D.**
Initial Temp.: 100°C, 5min
Rate: 2°C/min
Final Temp.: 130°C
Carrier Gas: H_2
Detector: F.I.D.
Sensitivity: 32 x 10^{-12} AFS
Injection Mode: Split
Part No. 054901

1. (+) Neomenthol
2. (-) Neomenthol
3. (+) Menthol
4. (-) Menthol
5. (+) α-Terpineol
6. (-) α-Terpineol



TESTING OF CYDEX-B

As with all SGE columns, each Cydex-B column is individually tested with a specific test mix designed to examine the column's efficiency in resolving chiral and positional isomers. The test mix also probes the level of column inertness. This tough test procedure ensures that every Cydex-B column is of the highest quality, reproducibility and inertness.

SPECIFICATIONS

Cydex-B phase is not fully bonded and crosslinked, therefore washing of Cydex-B columns is not recommended. Samples injected onto the Cydex-B columns should be relatively clean or a retention gap should be used.

Minimum Operating Temp.=	30°C
Maximum Continuous Temp.=	220°C
Maximum Cycling Temp.=	240°C

OPERATIONAL HINTS

To obtain optimum efficiency with Cydex-B columns, carrier gas velocities higher than those normally set for other stationary phases should be used.

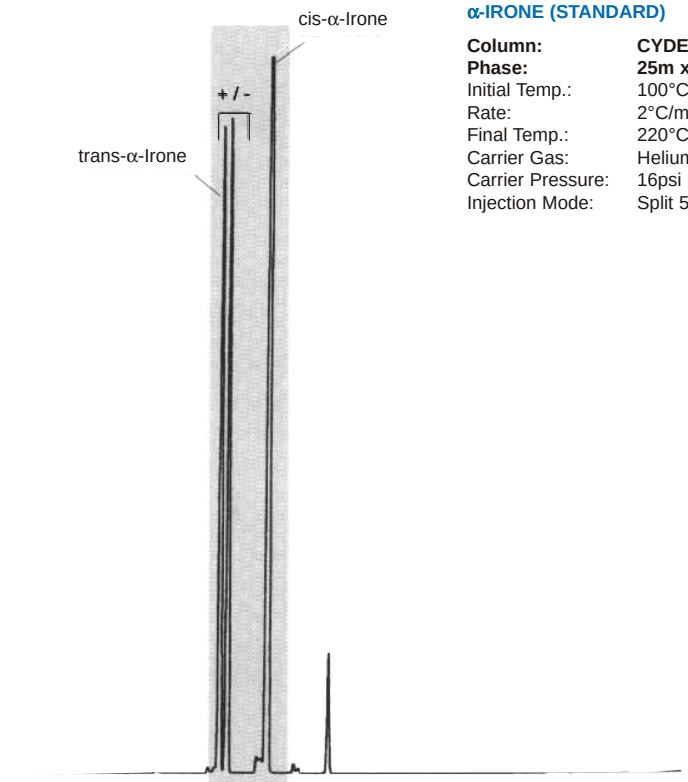
- Hydrogen 35-45 cm/sec
- Helium 30-35 cm/sec

α-IRONE IN ORRIS CONCENTRATE

Orris is widely used in the perfume and cosmetic industry. It contains irone as one of the principle odorous components but also has very high levels of mystric acid present. Irone is also the main perfume ingredient of violets. Irone exists in a cis and trans form, both having + and - enantiomers.

Combining multidimensional chromatography with the unique separation characteristics of the Cydex-B column, confirmation of cis α-irone enantiomers can be performed easily, reproducibly and without causing damage to the analytical columns.

α-IRONE (STANDARD)

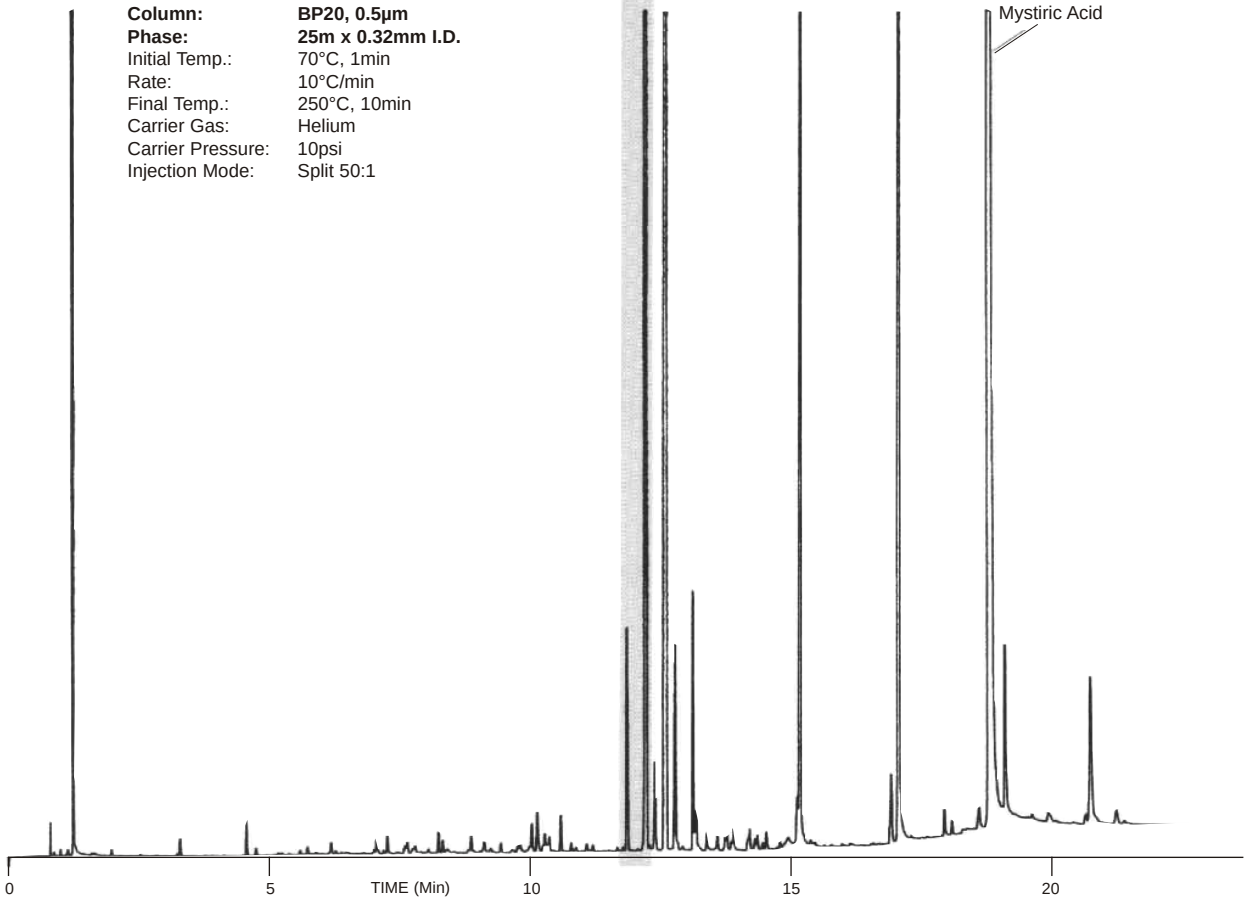


α-IRONE (STANDARD)

Column:	**CYDEX-B, 0.25µm**
Phase:	**25m x 0.22mm I.D.**
Initial Temp.:	100°C, 1min
Rate:	2°C/min
Final Temp.:	220°C
Carrier Gas:	Helium
Carrier Pressure:	16psi
Injection Mode:	Split 50:1

ORRIS CONCENTRATE

Column:	**BP20, 0.5µm**
Phase:	**25m x 0.32mm I.D.**
Initial Temp.:	70°C, 1min
Rate:	10°C/min
Final Temp.:	250°C, 10min
Carrier Gas:	Helium
Carrier Pressure:	10psi
Injection Mode:	Split 50:1



CYDEX-B

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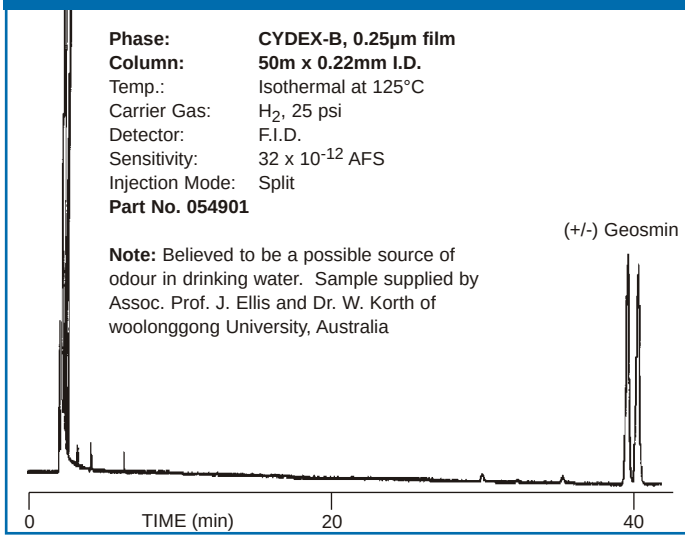
Further Cydex-B applications

GEOSMIN

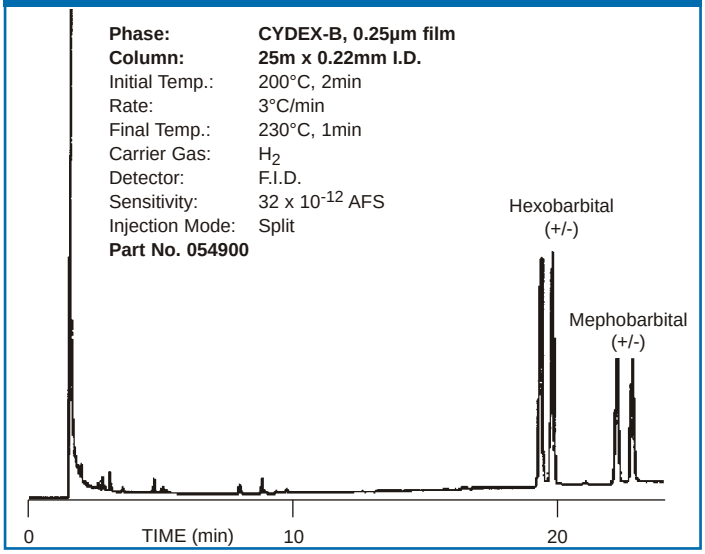
Phase: **CYDEX-B, 0.25µm film**
Column: **50m x 0.22mm I.D.**
Temp.: Isothermal at 125°C
Carrier Gas: H_2, 25 psi
Detector: F.I.D.
Sensitivity: 32 x 10^{-12} AFS
Injection Mode: Split
Part No. 054901

Note: Believed to be a possible source of odour in drinking water. Sample supplied by Assoc. Prof. J. Ellis and Dr. W. Korth of woolonggong University, Australia



UNDERIVATISED BARBITURATES

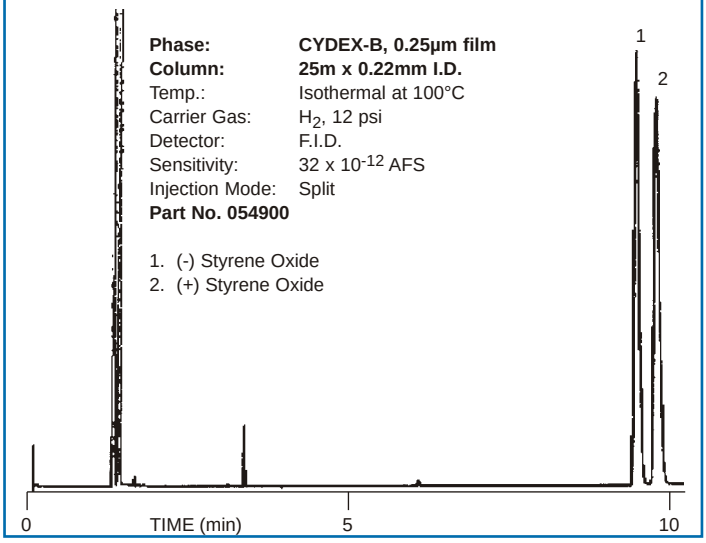
Phase: **CYDEX-B, 0.25µm film**
Column: **25m x 0.22mm I.D.**
Initial Temp.: 200°C, 2min
Rate: 3°C/min
Final Temp.: 230°C, 1min
Carrier Gas: H_2
Detector: F.I.D.
Sensitivity: 32 x 10^{-12} AFS
Injection Mode: Split
Part No. 054900



STYRENE OXIDE

Phase: **CYDEX-B, 0.25µm film**
Column: **25m x 0.22mm I.D.**
Temp.: Isothermal at 100°C
Carrier Gas: H_2, 12 psi
Detector: F.I.D.
Sensitivity: 32 x 10^{-12} AFS
Injection Mode: Split
Part No. 054900

1. (-) Styrene Oxide
2. (+) Styrene Oxide



ORDERING INFORMATION PHASE TYPE CYDEX-B

I.D. (mm)	FILM (µm)	25 METRE		50 METRE	
		Part No.	Description	Part No.	Description
0.22	0.25	054900	25QC2/CYDEX(B)-0.25	054901	50QC2/CYDEX(B)-0.25
0.32	0.25	054902	25QC3/CYDEX(B)-0.25	054903	50QC3/CYDEX(B)-0.25

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SGE International Pty. Ltd.
Tel: +61 (0) 3 9837 4200
Toll Free:1800 800 167
Fax: +61 (0) 3 9874 5672
email: support@sge.com

SGE Europe Ltd. (UK)
Tel: +44 (0) 1908 568 844
Fax: +44 (0) 1908 566 790
email: uk@sge.com

SGE Incorporated (USA)
Tel: +1 512 837 7190
Toll Free: 800 945 6154
Fax: +1 512 836 9159
email: usa@sge.com

SGE (France) Sarl
Tel: +33 (0) 1 6929 8090
Fax: +33 (0) 1 6929 0925
email: france@sge.com

SGE (Italia) Srl.
Tel: +39 06 4429 0206
Fax: +39 06 4429 0724
email: sge.italia@tin.it

SGE Deutschland GmbH
Tel: +49 (0) 6151 860486
Fax: +49 (0) 6151 860489
email: germany@sge.com

SGE China Service Centre
Tel: +86 (10) 6588 8666
Fax: +86 (10) 6588 6577
email: sge@bj.col.com.cn

SGE Japan Inc.
Tel: +81 (45) 222 2885
Fax: +81 (45) 222 2887
email: japan@sge.com

SGE India
Tel: +91 (022) 471 5896
Fax: +91 (022) 471 6592
email: sgeindia@bom4.vsnl.net.in

www.sge.com