

Cover Sheet for

**ENVIRONMENTAL CHEMISTRY METHOD**

***Pesticide Name:*** Spiroxamine

***MRID # :*** 450904-20

***Matrix:*** Soil

***Analysis:*** GC/MS

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450904-20

109145

**Study Title**

**Enforcement and Confirmatory Method for Determination of KWG 4168 in  
Drinking Water and Surface Water by GC/MS.**

**Data Requirement**

**EPA Ref.: 166-1, Prospective Ground-Water Monitoring (Supplemental)**

**Author**

**Herbert Sommer**

**Completion Date**

**May 26, 1999**

**Performing Laboratory**

**Bayer AG  
Institute for Metabolism Research & Residue Analysis, PF-E/MR  
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**Submitting Laboratory**

**Bayer Corporation  
Agriculture Division  
Research and Development Department  
P.O. Box 4913  
Kansas City, Missouri 64120-0013**

**Performing Laboratory Study Numbers**

**P 684 97022**

**Bayer Report Number**


**109145**

**Statement of Confidentiality**

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA 10 (d)(1)(A),(B) or (C).

Company: Bayer Corporation  
Agriculture Division  
Research and Development Department  
Environmental Research Section

Company Agent:

  
R. L. Graney, Ph.D.

Date: 7-21-99

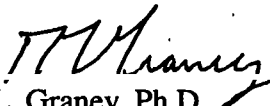
Vice-President, Environmental Research

These data are the property of the Agriculture Division of Bayer Corporation, and as such, are considered to be confidential for all purposes other than compliance with FIFRA 10. Submission of these data in compliance with FIFRA does not constitute a waiver of any rights to confidentiality which may exist under any other statute or in any other country.

**Good Laboratory Practice Certification**


The study described in this document was conducted under the requirements of the Principles of Good Laboratory Practice (GLP) [Chemikaliengesetz, attachment 1, dated July 25, 1994; and OECD Principles of GLP, dated November 26, 1997 [C(97) 186/Final]. A Good Laboratory Practice Statement and a Quality Assurance Statement are presented on pages 4 and 6, respectively, of this report.

Submitted By: Bayer Corporation  
Agriculture Division  
Research and Development Department  
Environmental Research Section

  
R. L. Graney, Ph.D.  
Vice-President, Environmental Research

Date: 7-21-99

Sponsor Representative:

  
V. E. Clay, Ph.D.  
Director, Environmental Fate

Date: 7-19-99

Study Authors


See Page 4  
Dr. H. Sommer  
Bayer AG

Date:

**Certificate of Availability of Raw Data**

It is hereby certified that the registrant has access to the original report. A permanent record of the original report is filed at Bayer AG, Crop Protection Research, Institute for Environmental Biology, Leverkusen, Germany. A copy of the final report will be retained at Bayer Corp., Hawthorn Road, Kansas City, Missouri.

Company Agent:

  
P. A. Toll  
Supervisor, Quality Assurance Unit

Date: 7/20/99

**Quality Assurance Statement**

**Study Title:** Enforcement and Confirmatory Method for Determination of KWG 4168 in Drinking Water and Surface Water by GC/MS.

**Performing Laboratory Study Number:** P 684 97022

Audits of this study were conducted under the requirements of the Principles of Good Laboratory Practice (GLP) [Chemikaliengesetz, attachment 1, dated July 25, 1994; and OECD Principles of GLP, dated November 26, 1997 [C(97) 186/Final]. The Quality Assurance Statement from the performing laboratory is presented on page 6 of this report.

**Company Agent:**



**Date:**

7/20/99

P. A. Toll  
Supervisor, Quality Assurance Unit

**Certification of Authenticity**

With the exception of the addition of a Bayer Corporation Report Number, subsequent pages of this report are presented as received from the performing laboratory.

Sponsor: Bayer Corporation  
Agriculture Division  
Environmental Research Section

Approved By:

*R. L. Graney*  
R. L. Graney, Ph.D.  
Vice-President, Environmental Research

Date: 7-21-99

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Director, Environmental Fate

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109145

Bayer AG  
Crop Protection Development  
Institute for Metabolism Research  
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D-51368 Leverkusen

Monheim, May 26, 1999  
Dr. H. Sommer / KL  
MR-151/99  
Method 00574

**Enforcement and confirmatory method for determination of KWG 4168 in  
drinking water and surface water by GC/MS.**

**Author**

Herbert Sommer

**Study Completion Date**

May 26, 1999

**Testing Facility**

Bayer AG  
PF-E/MR  
D-51368 Leverkusen

**Study No.**

P 684 97022



00574 / MO-99-006221

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**Statement of Compliance****Study Number:** P 684 97022**Test Substance:** KWG 4168**Study Director:** Dr. H. Sommer**Testing Facility:** Bayer AG  
PF-E/MR  
D-51368 Leverkusen**Title of the Study:** Enforcement and confirmatory method for determination of KWG 4168 in drinking water and surface water by GC/MS.

I hereby certify that the above mentioned study was performed in compliance with the Principles of Good Laboratory Practice [Chemikaliengesetz, attachment 1, dated July 25, 1994 (1); OECD Principles of Good Laboratory Practice (GLP), dated November 26, 1997 [C(97) 186/Final]] (2).

The test facility was inspected and certified as working in compliance with the Principles of Good Laboratory Practice by the competent authority (AktENZEICHEN IV C 4 - 31.11.62.03, March 4, 1999)

  
(Dr. H. Sommer)

05/26/99  
(Date)

**Certification of Authenticity**

The result of following trial is presented in this report:

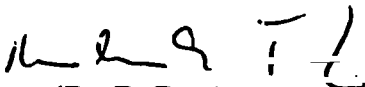
P 684 97022

**Signatures**

  
(Dr. H. Sommer)

Study Director and  
Head of Analytical Laboratory

05/26/99  
(Date)

  
(Dr. R. Fritz)

Manager  
Testing Facility  
(PF-E/MR)

May 28, 1999  
(Date)

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Referat GLP

Quality Assurance Statement

Report No.: MR-151/99

Study No.: P 684 97022

Title of report: Enforcement and Confirmatory Method for Determination of KWG 4168 in Drinking Water and Surface Water by GC/MS

The conduct of this study has been periodically inspected and this report has been audited by the Quality Assurance Unit. The dates of inspection are given below.

Date of Protocol Inspection:

3/19/1999

Date of Report to Management:

no report

Date of Study Inspection:

3/22/1999

Date of Report to Management:

3/24/1999

Date of Final Report Audit:

4/28/1999

Date of Report to Management:

4/28/1999

5/26/1999

5/26/1999

The results reported in this study have been checked on the basis of our current SOPs and accurately reflect the raw data.

*Ebel*

*May 27, 1999*

Quality Assurance Unit, PF-BPA/QM-GLP

Date:

**Summary**

This method describes the determination of KWG 4168 in drinking water and surface water. Water samples are analysed by means of GLC with MSD detection. The limit of quantification is 0.1 µg/L.

The linearity of the detector was checked for standard solutions of KWG 4168 in the concentration range from 10 µg/L to 1000 µg/L (corresponding to concentrations in water samples of 0.1 µg/L to 10 µg/L). The correlation coefficient for KWG 4168 was 0.99991.

During repeatability testing the relative standard deviation for peak areas ranged from 0.06 to 0.07. The relative standard deviation for the retention time was <0.001.

The mean recovery for KWG 4168 was 93% in drinking water with a relative standard deviation of 5% and 98% in surface water with a relative standard deviation of 8%.

## 1. Introduction

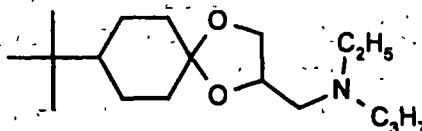
This method was developed for determination of KWG 4168 in drinking water samples and surface water samples.

### 1.1: Chemical and Physical Properties of KWG 4168

Trivial name : KWG 4168

Chemical designation : 8-(1,1-dimethylethyl)-N-ethyl-N-propyl-1,4-dioxaspiro[4.5]decane-2-methanamine

Structural formula



CAS-No. : 118134-30-8

Empirical formula :  $C_{18}H_{35}NO_2$

Molecular weight : 297.5 g/mole

## 2. Principle of the Method

The method was developed in accordance to the multi residue methods of the Deutsche Institut für Normung (DIN) (3) and of the European Committee for Standardization (CEN) (4) under special consideration of the gas chromatographic standard methods F6 (5) and F14 (6).

KWG 4168 is concentrated from water samples by solid phase extraction (4-6). After drying of the cartridges KWG 4168 is eluted, concentrated to dryness and reconstituted. Identification and quantitative determination is done by gas chromatography using mass selective detection (5, 6) (see Figure 18).

### 3. Test System

For method validation drinking water from Monheim and surface water from the river Rhine sampled in Leverkusen-Hitdorf was used. Analytical data for the surface water are listed in Table 1.

Table 1. Analytical data for the surface water from river Rhine, sampled in Leverkusen-Hitdorf

Test	Result
Total organic carbon (TOC)	19 mg/L
Dissolved organic carbon (DOC)	2 mg/L
Conductivity at 25 °C	349 µS/cm
pH	7.7
Water hardness	6.9 °dH
Deposited substance	1.5 mL/L
Dry residue after filtration	363 mg/L

### 4. Instruments

Gas chromatograph	Shimadzu GC 17 A with autosampler AOC-17 / AOC-1400 and mass selective detector QP5000 Shimadzu D-47269 Duisburg
Turbo-Vap LV Evaporator	Zymark GmbH D-6270 Idstein/Taunus
SPE-Station	Vac Elut SPS 24 Varian GmbH D-63289 Darmstadt

Alternatively comparable instruments of other manufacturers can be used.

Volumetric flasks, pipettes and other common laboratory equipment.

## 5. Reagents

Milli-Q-water	: deionised and purified by a milli-Q-unit, Millipore Co.
Methanol	: Promochem, D-46469 Wesel
N-butyl-acetate	: Riedel de Haen, D-30926 Seelze
Ammonia solution (25%)	: Riedel de Haen, D-30926 Seelze
C <sub>18</sub> -cartridges	: Bakerbond spe. Octadecyl (C <sub>18</sub> ) (40 µm), 1 g, J.T. Baker, Phillipsburg NJ 08865 USA, Art. 7020-07
Reference substance	: KWG 4168, batch M00298, purity 98.3%, determined by GLC, identity ensured by <sup>1</sup> H-NMR-spectrum expiry date July 2002

For method validation a certified reference substance of batch no. M00298 (KWG 4168, purity 98.3 %) was used. With the reference substance a primary stock solution of approx. 1000 mg/L was prepared in acetone. From this stock solution standard solutions were prepared by dilution with a mixture of 50% n-butyl-acetate, 49.5% methanol and 0.5% ammonia solution (25%) (v:v:v).

## 6. Safety Measures

The German guidelines for laboratories issued by the Trade Co-operative Association (e.g. Bulletin M006) or comparable guidelines in other countries must be considered when working according to this method.

The following solvents and pesticides classified as toxic and/or less toxic according to the Hazardous Substances Regulations are used.

Methanol	: toxic and easily flammable
Ammonia	: toxic
Acetic acid -n-butylester	: toxic and easily flammable
KWG 4168	: Xn harmful, R21/22

This classification is based on the German guidelines and has to be adapted to the respective national guidelines in case the method is used outside Germany.



## 7. Performance of Analyses

### 7.1. Sample Preparation

For concentration the water samples are adjusted to pH=3 with o-phosphoric acid. This should be done just before the concentration process starts because KWG 4168 is not stable in an acidic solution ( $t_{1/2}$  at pH 4 >250 h,  $t_{1/2}$  at pH 2.1 < 16-h). Nevertheless the acidification is necessary to increase the solubility of KWG 4168 in water and to avoid adsorption to the glass walls of the equipment.

The C<sub>18</sub>-cartridges are washed with 20 mL of methanol and then conditioned with 20 mL of milli-Q-water. After the conditioning step volumes of 100 mL of the water samples are sucked through the cartridges with a flow rate of approx. 1 drop per second. After this the cartridges are dried by sucking of ambient air through the cartridges for one hour. To prevent pollution of the cartridges during the drying process activated carbon cartridges are placed on top of the C<sub>18</sub>-cartridges. The suction pressure is approx. 20 mbar. After the drying procedure the cartridges are eluted with 10 mL of methanol / ammonia solution (25%) (990:10, v:v) and evaporated to dryness. The residue is dissolved in 1 mL (0.1 µg/L samples) or 10 mL (1.0 µg/L samples) of a mixture of 50% n-butyl-acetate, 49.5% methanol and 0.5% ammonia solution (25%) (v:v:v). From each solution a volume of 1 µL is directly injected into the GC/MSD.

### 7.2. Standard Preparation

A volume of 100 mL of surface water is adjusted to pH=3 with o-phosphoric acid (blank samples) and extracted in the same manner as described for the water samples. The residue of five blank samples is transferred into a 5-mL volumetric flask using a volume of 1 mL for each blank sample of a mixture of 50% n-butyl-acetate, 49.5% methanol and 0.5% ammonia solution (25%) (v:v:v). To this solution a defined volume of the standard stock solution KWG 4168 is added and the flask is filled to the mark with a mixture of 50% n-butyl-acetate, 49.5% methanol and 0.5% ammonia solution (25%) (v:v:v) to reach a concentration similar to the concentrated samples.

### 7.3. Evaluation

Evaluation is performed using a laboratory data system by comparing the peak areas of the samples to the peak areas of the external standard solutions. The concentration of the samples can be calculated according to the given formula:

$$C = \frac{A \times C_s}{A_s \times F}$$

- A = Peak area of sample solution [area counts]  
 A<sub>s</sub> = Peak area of standard solution [area counts]  
 C = Concentration of KWG 4168 in the sample [µg/L]  
 C<sub>s</sub> = Concentration of KWG 4168 in the standard solution [µg/L]  
 F = Concentration factor

### 7.4. Chromatographic Conditions A

- Column : OV-17, length 15 m; 0.25 mm i.d.; 0.25 µm film thickness  
 Macherey & Nagel, D-52313 Düren  
 Carrier gas : Helium, delivery pressure 0.25 bar  
 Injection volume : 1 µL  
 Injector temp. : 240 °C  
 Interface temp. : 240 °C  
 Column temp. : 90 °C, 2 min, 15 °C/min to 240 °C, 240 °C 1 min  
 Detector : KWG 4168 : m/z 100  
 Retention time : KWG 4168 Isomer A approx. 9.9 min  
 KWG 4168 Isomer B approx. 10.3 min

### 7.5. Chromatographic Conditions B

- Column : Ultra 1, length 12 m; 0.2 mm i.d.; 0.33 µm film thickness  
 Hewlett Packard, D-61352 Bad Homburg  
 Carrier gas : Helium, delivery pressure 0.25 bar  
 Injection volume : 1 µL  
 Injector temp. : 240 °C  
 Interface temp. : 240 °C  
 Column temp. : 90 °C, 2 min, 15 °C/min to 240 °C, 240 °C 1 min  
 Detector : KWG 4168 : m/z 100  
 Retention time : KWG 4168 Isomer A approx. 10.5 min  
 KWG 4168 Isomer B approx. 10.9 min

## 8. Repeatability

Standard solutions of about 10 µg/L and 1000 µg/L KWG 4168 were injected 10 times into the gas chromatograph. The peak areas and retention-times for KWG 4168 were determined and are listed in Table 2.

Table 2 Repeatability of peak areas and retention times

Standard concentration	n	Peak area		Retention time			
		Average [area counts]	RSD*	Isomer A		Isomer B	
				Average [min]	RSD*	Average [min]	RSD*
10.02 µg/L	10	5194	0.07	10.44	< 0.001	10.82	< 0.001
1001.7 µg/L	10	626719	0.06	10.42	< 0.001	10.80	< 0.001

RSD\* = Relative Standard Deviation

Representative chromatograms are given in Figure 1 to 16.

## 9. Linearity

The linearity of the detector has been tested in a range from 10 µg/L to 1000 µg/L for KWG 4168. The resulting curve is shown in Figure 16. The correlation coefficient for KWG 4168 was 0.99991.

## 10. Control Samples

In the control samples KWG 4168 was not detected (see Figures 2, 5, 9 and 12).

## 11. Confirmatory Chromatographic Methods

For confirmation of positive detects of KWG 4168 in drinking water samples or surface water samples different confirmatory techniques can be used.

### 11.1. GC/MS Detection

For validation of this method the highly specific mass selective detection has been used. Due to the mass spectroscopic properties of KWG 4168 only the ion  $m/z = 100$  has been detected. Though not 3 required ions have been detected, in none of the control samples of drinking water or surface water KWG 4168 could be detected.

### 11.2. Pattern of the A- and B Isomers

KWG 4168 can only be considered as positively detected, if signals for both isomers A and B are detected. In none of the samples a change in the isomeric pattern could be observed.

### 11.3. Different Stationary Phase of Different Selectivity

For confirmation of positive detects of KWG 4168 a second GC column with different polarity was used.

### 12. Determination of the Recovery Rates

For method validation water samples were fortified with KWG 4168.

24 samples have been analysed, each sample was injected two times into the GC/MS instrument.

Fortification levels of drinking water and surface water, recoveries and standard deviations are presented in Table 3 and Table 4.

Table 3 Recoveries for drinking water

Fortification level [µg/L]	Recoveries for column OV 17 [%]	Mean value [%]	RSD [%]	Recoveries for column Ultra 1 [%]	Mean value [%]	RSD [%]
0.1	96	96	6.3	90	91	4.1
	88			86		
	95			94		
	97			89		
	105			95		
1	95	91	2.9	94	92	2.5
	89			89		
	89			92		
	90			92		
	93			95		

Table 4 Recoveries for surface water

Fortification level [µg/L]	Recoveries for column OV 17 [%]	Mean value [%]	RSD [%]	Recoveries for column Ultra 1 [%]	Mean value [%]	RSD [%]
0.1	109 107 101 91 97	101	7.3	109 90 95 100 99	99	7.1
1	99 90 107 92 108	99	8.4	103 84 93 87 93	92	7.9

The mean recovery for KWG 4168 was 93% in drinking water with a relative standard deviation of 5% and 98% in surface water with a relative standard deviation of 8%.

### 13. Limit of Quantification

The limit of quantification for determination of KWG 4168 in drinking water or surface water is 0.1 µg/L.

**14. Figures**

- Figure 1: Chromatogram matrix standard solution KWG 4168 10 µg/L  
(Chromatographic conditions A)
- Figure 2: Chromatogram blank sample surface water, 100-fold concentrated  
(Chromatographic conditions A)
- Figure 3: Chromatogram surface water sample spiked with KWG 4168 0.1 µg/L  
100-fold concentrated (Chromatographic conditions A)
- Figure 4: Chromatogram surface water sample spiked with KWG 4168 1.0 µg/L  
10-fold concentrated (Chromatographic conditions A)
- Figure 5: Chromatogram blank sample drinking water, 100-fold concentrated  
(Chromatographic conditions A)
- Figure 6: Chromatogram drinking water sample spiked with KWG 4168 0.1 µg/L  
100-fold concentrated (Chromatographic conditions A)
- Figure 7: Chromatogram drinking water sample spiked with KWG 4168 1.0 µg/L  
10-fold concentrated (Chromatographic conditions A)
- Figure 8: Chromatogram matrix standard solution KWG 4168 10 µg/L  
(Chromatographic conditions B)
- Figure 9: Chromatogram blank sample surface water, 100-fold concentrated  
(Chromatographic conditions B)
- Figure 10: Chromatogram surface water sample spiked with KWG 4168 0.1 µg/L  
100-fold concentrated (Chromatographic conditions B)
- Figure 11: Chromatogram surface water sample spiked with KWG 4168 1.0 µg/L  
10-fold concentrated (Chromatographic conditions B)
- Figure 12: Chromatogram blank sample drinking water, 100-fold concentrated  
(Chromatographic conditions B)
- Figure 13: Chromatogram drinking water sample spiked with KWG 4168 0.1 µg/L  
100-fold concentrated (Chromatographic conditions B)
- Figure 14: Chromatogram drinking water sample spiked with KWG 4168 1.0 µg/L  
10-fold concentrated (Chromatographic conditions B)
- Figure 15: Linearity of KWG 4168 from 10 µg/L to 1000 µg/L
- Figure 16: Linearity of KWG 4168 from 10 µg/L to 1000 µg/L (Diagram)
- Figure 17: Mass spectrum of KWG 4168 (EI, 70 eV)
- Figure 18: Flow Diagram of Analysis Procedure

**15. Organisation****Sponsor:****PF-E/REG  
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D-51368 Leverkusen****Manager of Testing Facility: Dr. R. Fritz****Institute for Metabolism Research and Residue Analysis  
Bayer AG, PF-E/MR  
D-51368 Leverkusen****15.1. Archiving**

All raw data pertaining to this study and the original report are stored in the central GLP archive PF-F, Bayer AG, D-51368 Leverkusen, for at least as long as required by GLP-principles.

Test and reference substances are stored in the archives of PF-E/FT-EA, Bayer AG, D-51368 Leverkusen, as long as their quality still guarantees an evaluation.

**15.2. Time Schedule**

**Approval of the Study by  
the Study Director: March 19, 1999**

**Start of Experimental Phase: March 18, 1999**

**End of Experimental Phase: May 20, 1999**

**16. References**

1. Chemikaliengesetz ("ChemG"), Attachment 1 dated, July 25, 1994
2. OECD-Principles of Good Laboratory Practice (GLP), dated November 26, 1997 [C (97) 186/(Final)]
3. Deutsche Einheitsverfahren zur Wasser-, Abwasser- und Schlamm-Untersuchung, physikalische, chemische, biologische und bakteriologische Verfahren, Fachgruppe Wasserchemie i.d. GDCh mit dem Normenausschuß Wasserwesen (NAW) im Deutschen Institut für Normung e. V. (DIN) (Edit.), Band V, VCH Weinheim, Beuth Verlag, Berlin 1996, Gruppe F: Gemeinsam erfaßbare Stoffgruppen
4. Water quality – Determination of selected plant treatment agents – Method using high performance liquid chromatography with UV detection after solid-liquid extraction (F12) (EN ISO 11369); European standard, CEN European Committee for Standardization, Brussels, August 1997
5. German standard methods for the examination of water, waste water and sludge – Jointly determinable substances (group F) – Part 6: Determination of selected organic nitrogen and phosphorous compounds by gas chromatography after solid-liquid-extraction (F6), Normenausschuß Wasserwesen (NAW) im Deutschen Institut für Normung e. V., DIN V 38407-6, April 1995
6. German standard methods for the examination of water, waste water and sludge – Jointly determinable substances (group F) – Part 14: Determination of phenoxyalkyl carbonic acids by gas chromatography and mass spectrometric detection after solid-liquid-extraction and derivatization (F14), Normenausschuß Wasserwesen (NAW) im Deutschen Institut für Normung e. V., DIN V 38407-14, October 1994



Figure 1 : Chromatogram matrix standard solution KWG 4168 10 µg/L  
(Chromatographic conditions A)



Figure 2 : Chromatogram blank sample surface water, 100-fold concentrated  
(Chromatographic conditions A)

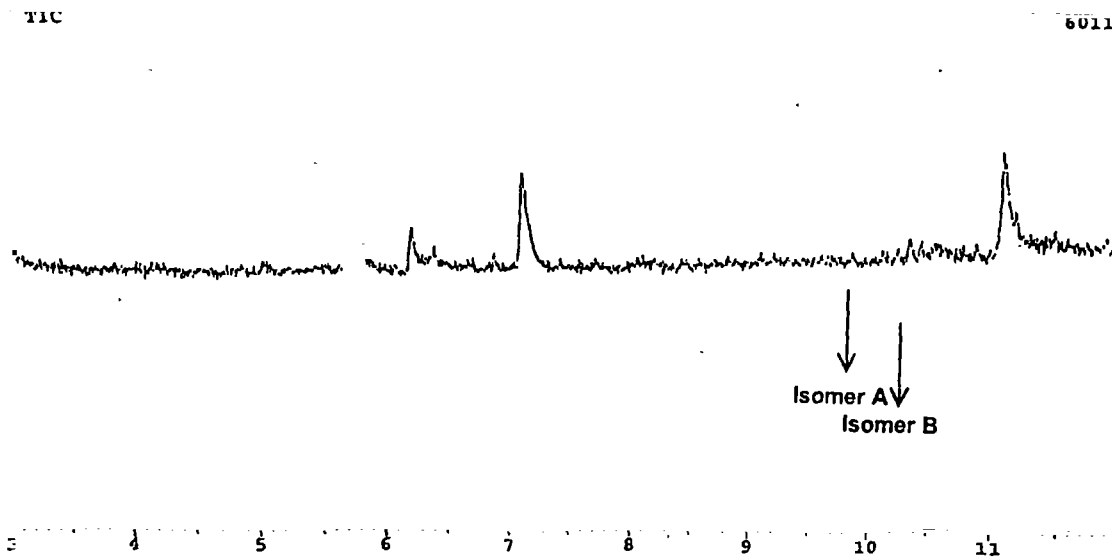


Figure 3 : Chromatogram surface water sample spiked with KWG 4168 0.1 µg/L  
100-fold concentrated (Chromatographic conditions A)

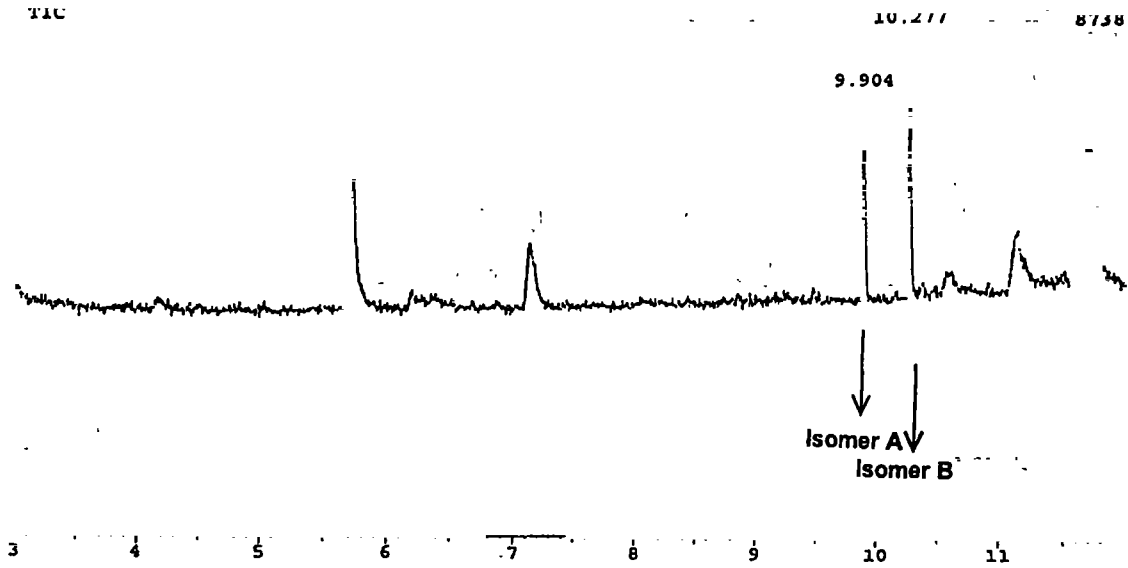


Figure 4 : Chromatogram surface water sample spiked with KWG 4168 1.0 µg/L  
10-fold concentrated (Chromatographic conditions A)

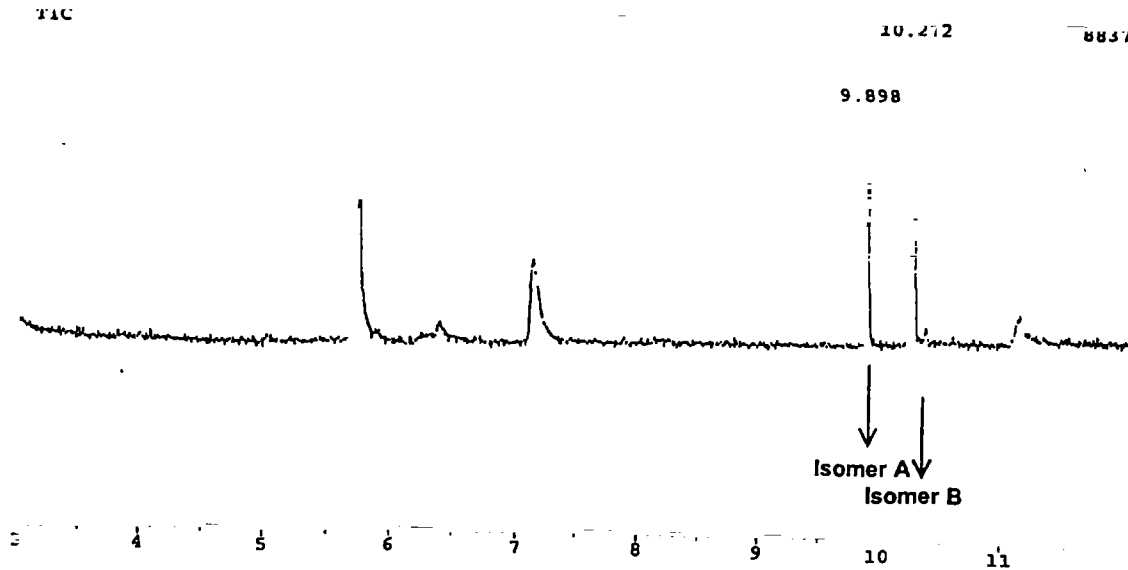


Figure 5 : Chromatogram blank sample drinking water, 100-fold concentrated (Chromatographic conditions A)

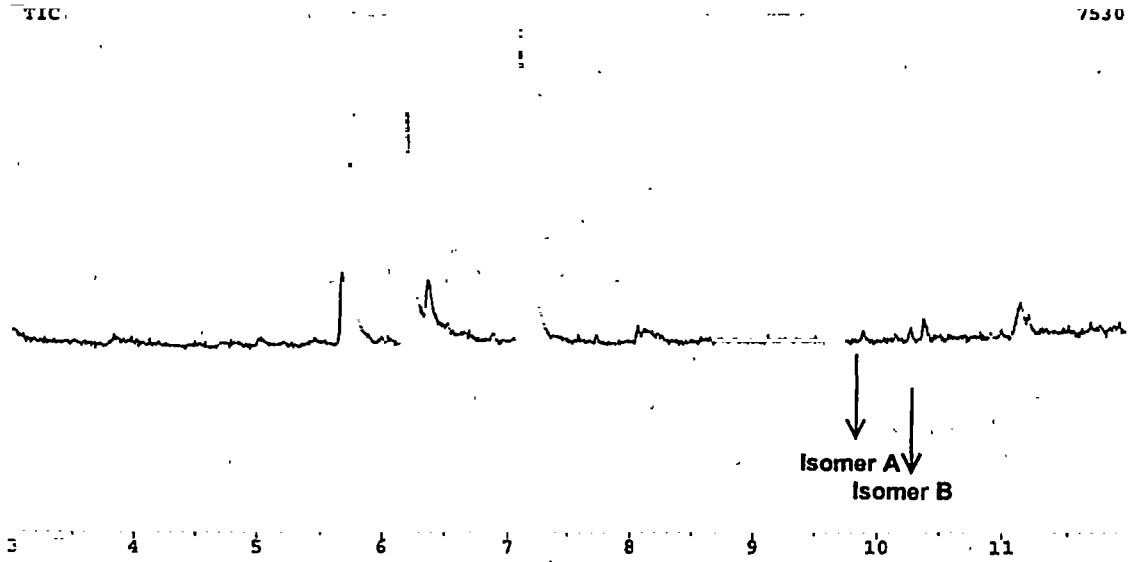


Figure 6 : Chromatogram drinking water sample spiked with KWG 4168 0.1 µg/L 100-fold concentrated (Chromatographic conditions A)

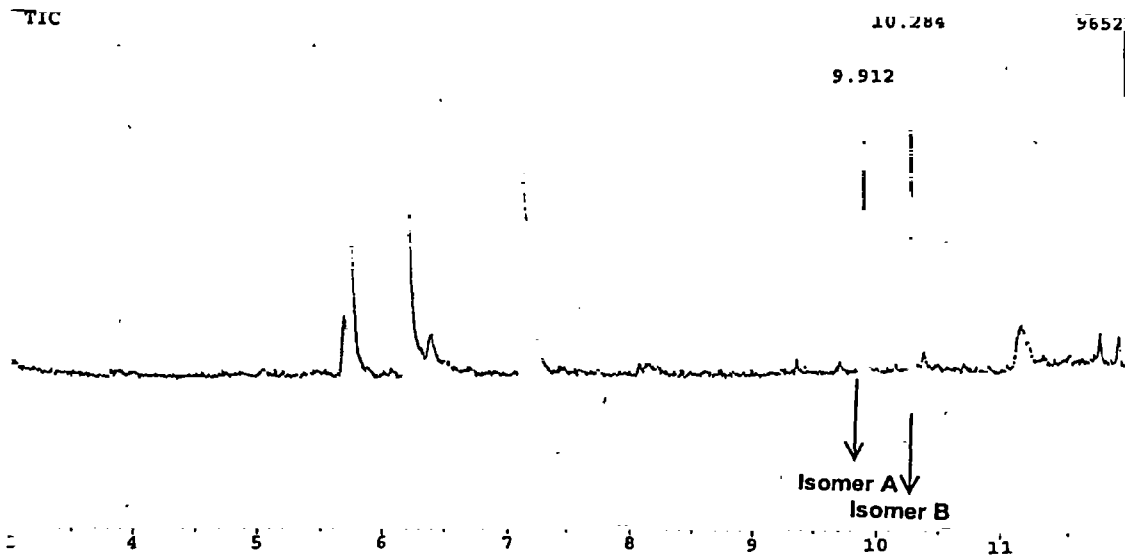


Figure 7 : Chromatogram drinking water sample spiked with KWG 4168 1.0 µg/L  
10-fold concentrated (Chromatographic conditions A)

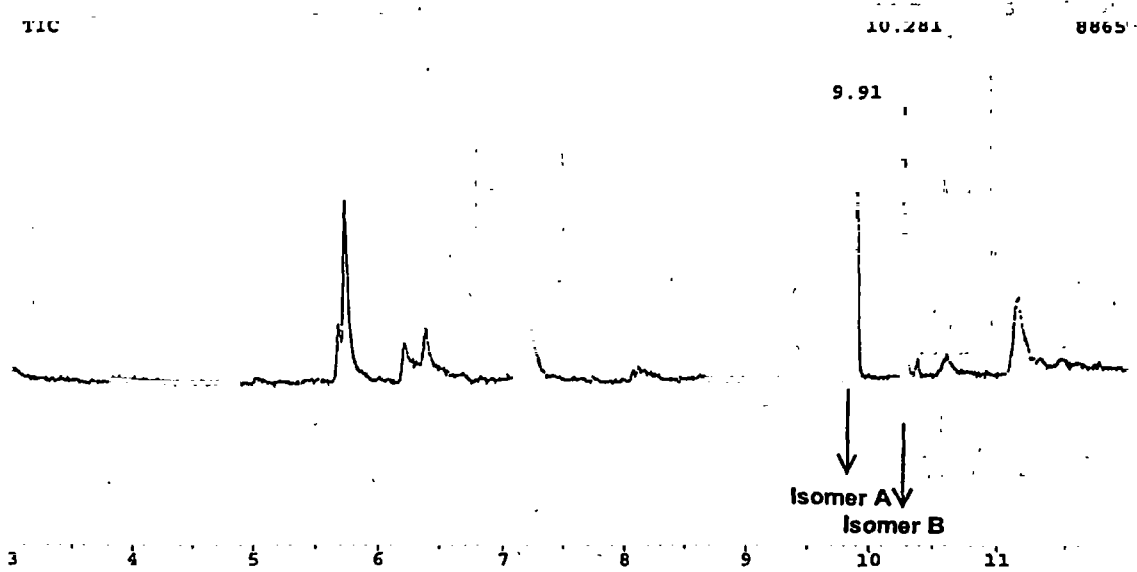


Figure 8 : Chromatogram matrix standard solution KWG 4168 10 µg/L  
(Chromatographic conditions B)

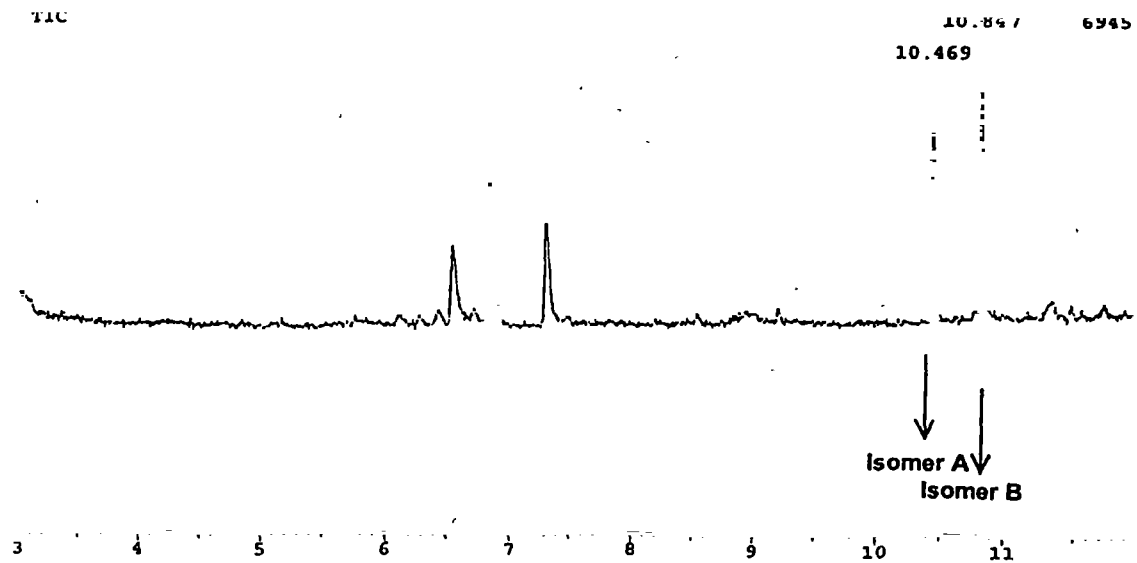


Figure 9 : Chromatogram blank sample surface water, 100-fold concentrated (Chromatographic conditions B)

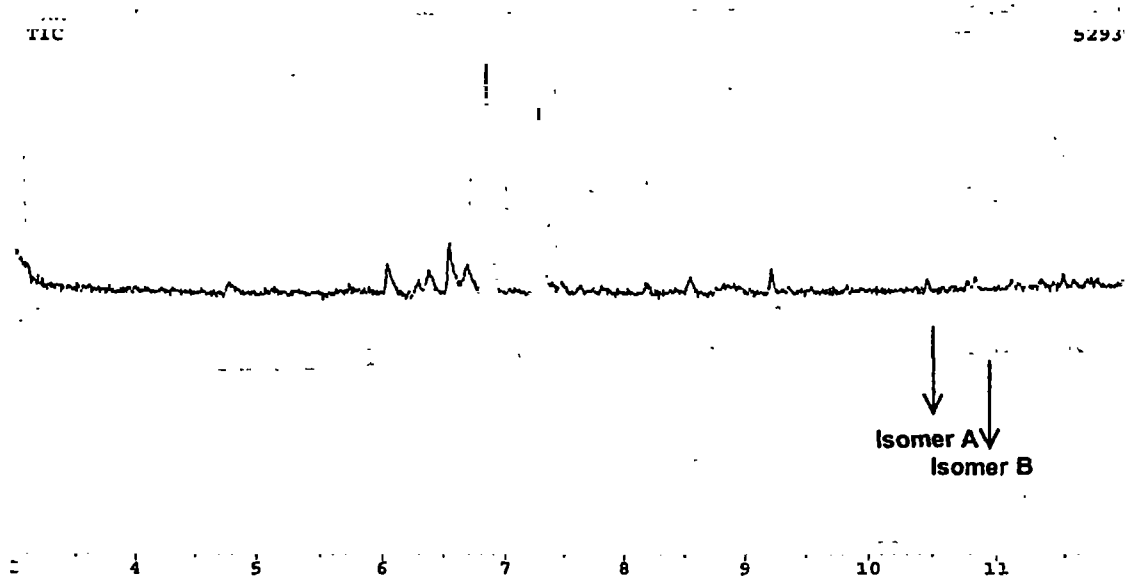


Figure 10 : Chromatogram surface water sample spiked with KWG 4168 0.1 µg/L 100-fold concentrated (Chromatographic conditions B)

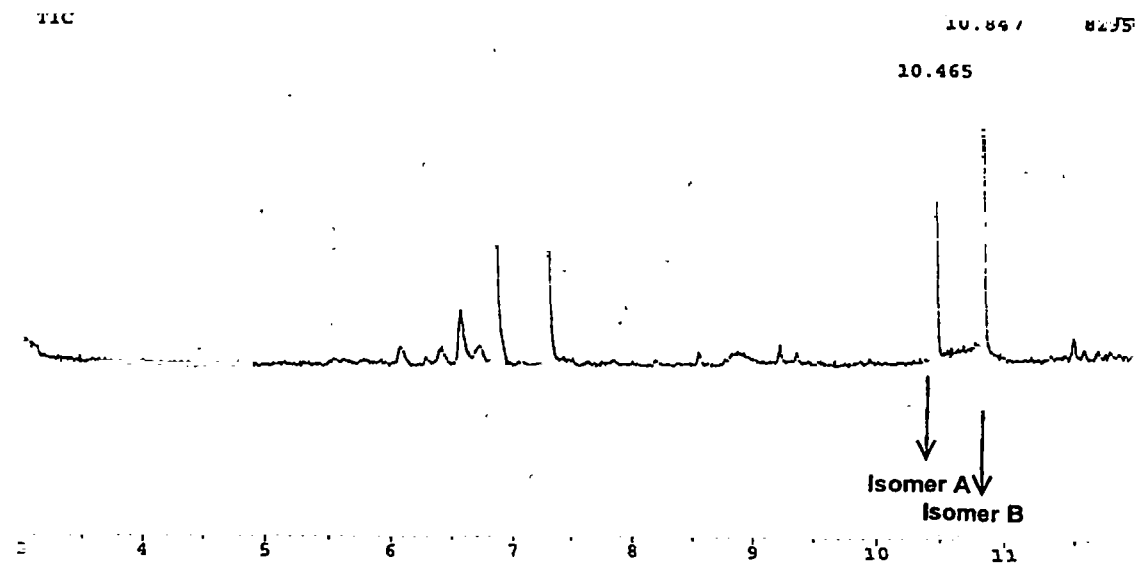


Figure 11 : Chromatogram surface water sample spiked with KWG 4168 1.0 µg/L 10-fold concentrated (Chromatographic conditions B)

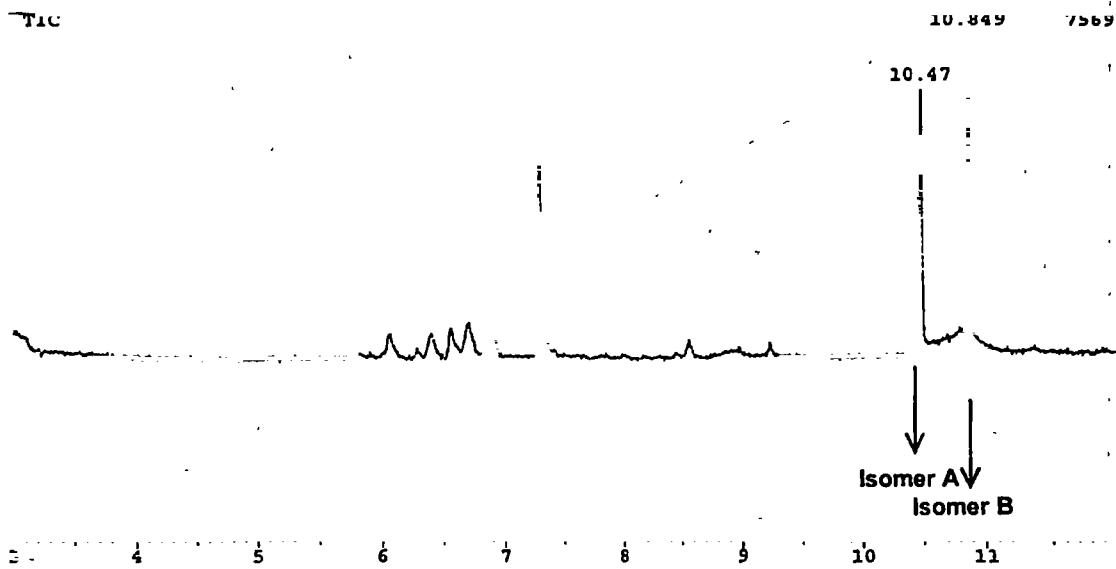


Figure 12 : Chromatogram blank sample drinking water, 100-fold concentrated (Chromatographic conditions B)

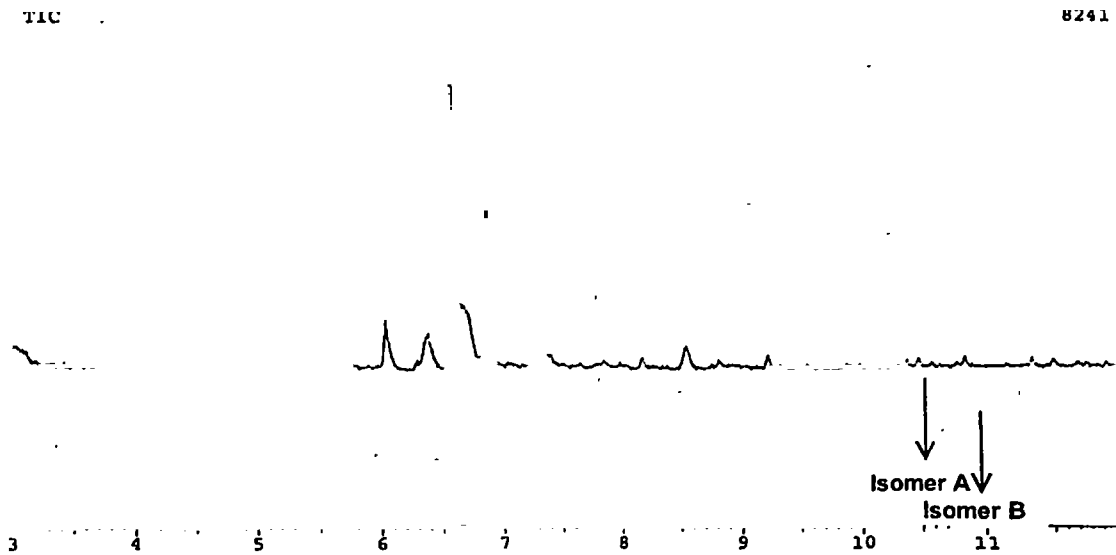


Figure 13 : Chromatogram drinking water sample spiked with KWG 4168 0.1 µg/L  
100-fold concentrated (Chromatographic conditions B)

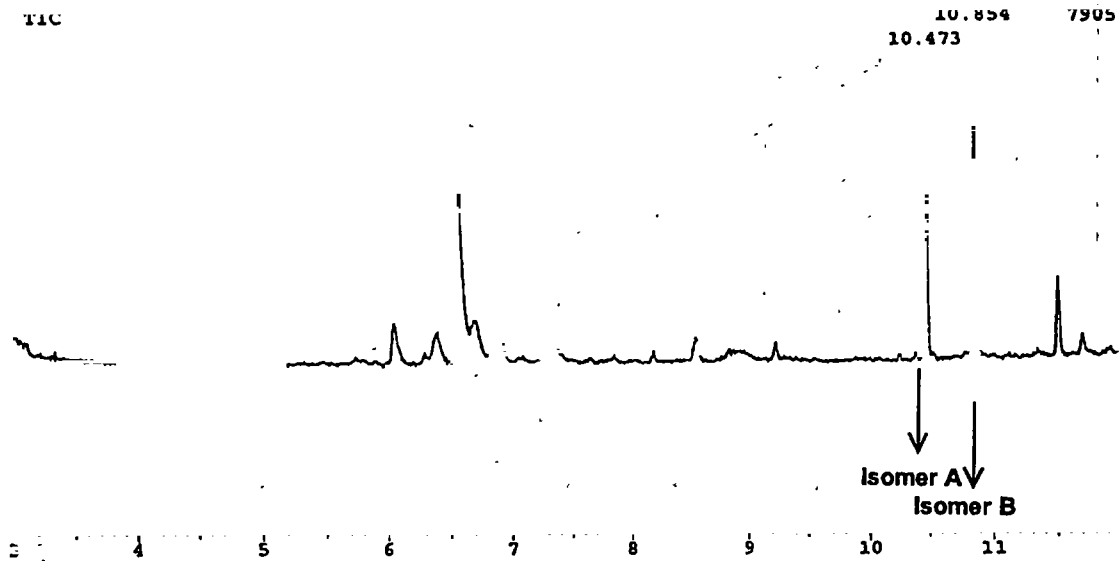


Figure 14 : Chromatogram drinking water sample spiked with KWG 4168 1.0 µg/L  
10-fold concentrated (Chromatographic conditions B)

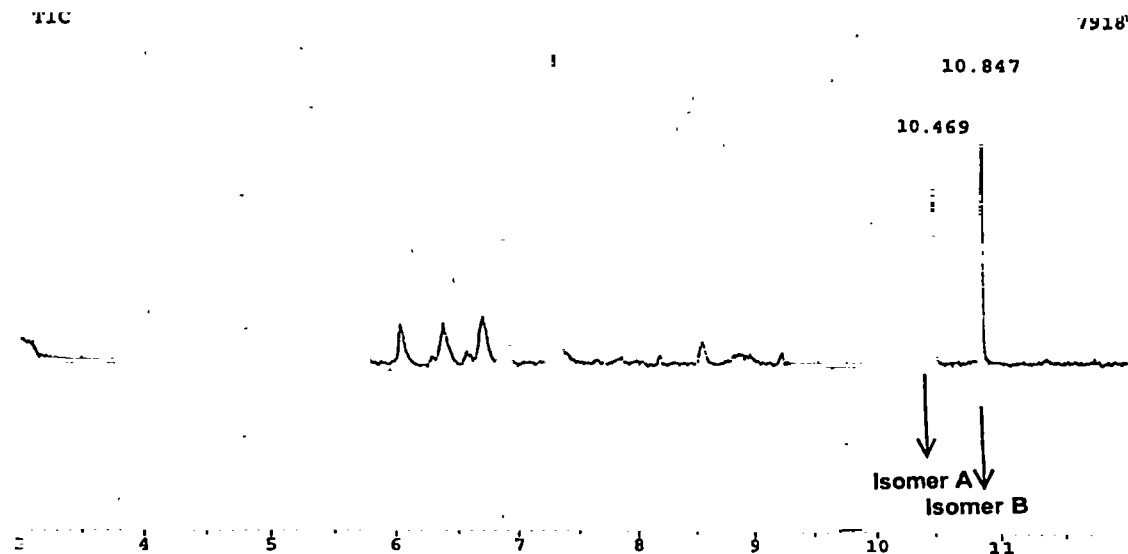


Figure 15 : Linearity of KWG 4168 from 10 µg/L to 1000 µg/L

Conc. [ µg/L ]	1. Injection Isomer A [area counts]	1. Injection Isomer B [area counts]	Mean 1. Inj. Isomer A + B [area counts]
10.02	5407	6039	5723
20.03	11271	12021	11646
50.08	27989	30796	29393
100.17	55568	62400	58984
500.84	291669	337872	314771
1001.7	593865	703317	648591

Conc. [ µg/L ]	2. Injection Isomer A [area counts]	2. Injection Isomer B [area counts]	Mean 2. Inj. Isomer A + B [area counts]	Mean Inj. 1 + 2 [area counts]	Regression [area counts]
10.02	5419	5789	5604	5664	4884
20.03	10828	12237	11533	11589	11340
50.08	27888	30735	29312	29352	30723
100.17	57338	64416	60877	59931	63031
500.84	313024	367902	340463	327617	321464
1001.7	581748	688311	635030	641810	644521

Constant (a)	645.003	-1579.2	Point of intersection (b)
Std. deviation	4.2463	1951.51	Std. deviation for b
Certainty (r <sup>2</sup> )	0.9998	3786.80	Std. deviation for y
F-Statistic	23073.16	4	Degrees of freedom
Quadr. regression	3.31E+11	5.74E+07	sum of square residuals

|correlation coefficient = 0.99991|



Figure 16 : Linearity of KWG 4168 from 10 µg/L to 1000 µg/L (Diagram)

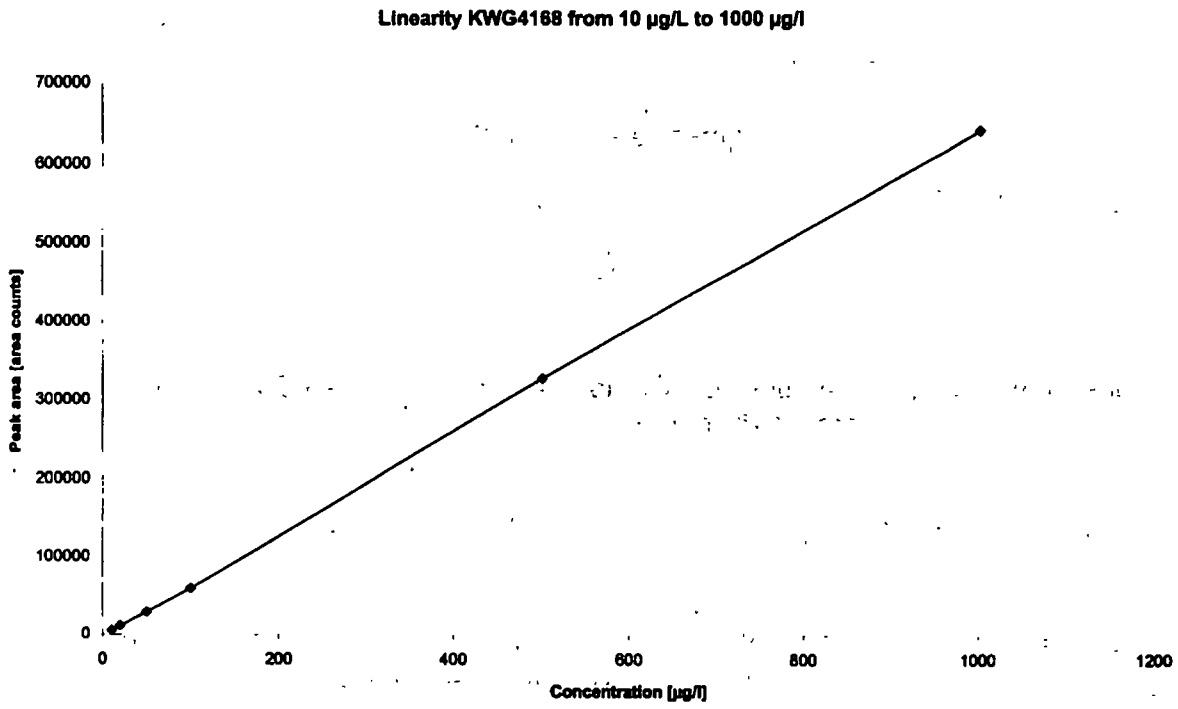


Figure 17 : Mass spectrum of KWG 4168 (EI, 70 eV)

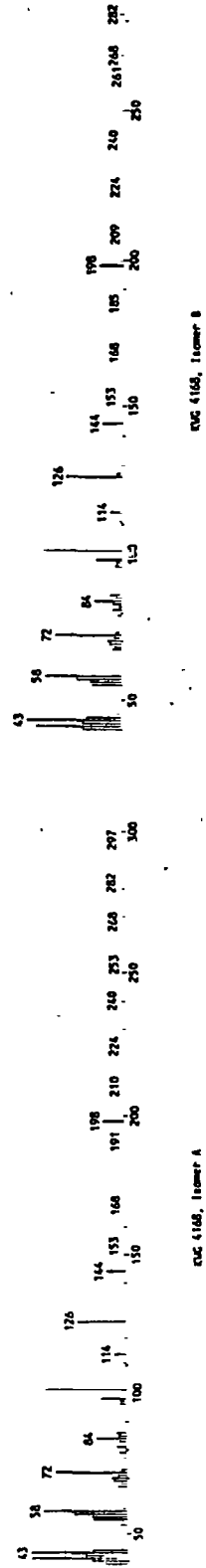


Figure 2 Mass Spectrum of KWG 4168, Isomer B

Figure 1 Mass Spectrum of KWG 4168, Isomer A

Figure 18 : Flow Diagram of Analysis Procedure

