Cover Sheet for

Environmental Chemistry Method

Pesticide Name: Iodosulfuron + Metsulfuron methyl

MRID#: 451085-22

Matrix: Water

Analysis: LC/MS/MS

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C006395

Title

Enforcement Method of Iodosulfuron-methyl-sodium and its metabolite Metsulfuron-methyl in Surface Water by HPLC incl. Validation

Extension of the enforcement method EM F 01/98 - 0 for Iodosulfuron-methyl-sodium in Drinking Water to its metabolite Metsulfuron-methyl incl. Validation

451087-22

Iodosulfuron-methyl-sodium (AE F115008)
Metsulfuron-methyl (AE F075736)

Guideline Reference

EPA OPPTS 860.1340
OECD IIA 4.5.5

Author

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Completed On

08 Feb 2000

Test Facility

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Entwicklung
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Federal Republic of Germany

Study Identification

CR 99/029

VOL. 66 of 229

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

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Date: March 20, 2000

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

The validation of this analytical method was conducted in compliance with the *Principles of Good Laboratory Practice* as adopted by the Council of OECD on 12 May 1981 [C(81)30 (Final)] and implemented at the national level.

Study Director **Dr. A. Wrede** _____ 08.02.2000
signature date (d/m/y)

Victor A Dorr

Victor A. Dorr
Representing Aventis CropScience USA LP
(Formerly AgrEvo USA Company)
Sponsor/Submitter

March 20, 2000
Date

Quality Assurance Statement

Title: **Enforcement Method of Iodosulfuron-methyl-sodium and its metabolite Metsulfuron-methyl in Surface Water by HPLC incl. Validation**

Extension of the enforcement method EM F 01/98 - 0 for Iodosulfuron-methyl-sodium in Drinking Water to its metabolite Metsulfuron-methyl incl. Validation

Iodosulfuron-methyl-sodium (AE F115008)
Metsulfuron-methyl (AE F075736)

Study: CR99/029

This study was periodically inspected and properly signed records of these inspections were submitted to testing facility management and the study director as shown below. This report has been audited by the Quality Assurance unit. The reported results accurately reflect the original data of the study.

Inspection		Phase of Study	Reported
18.11.1999		study plan	18.11.1999
22.11.1999		study conduct	22.11.1999
24.11.1999		study conduct	24.11.1999
17.1.2000		study conduct	17.1.2000
4.2.2000	7.2.2000	draft report	7.2.2000
8.2.2000		final report	8.2.2000

08. Februar 2000

Neuss

GLP Quality Assurance

APPROVALS PAGE

Study Director and Author	**Dr. A. Wrede**	signature	08.02.2000 date (d/m/y)
Head of Test Facility	**Dr. M. Uihlein**	signature	09.02.2000 date (d/m/y)
Audited by Quality Assurance Unit	08. Februar 2000; Uk		

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SUMMARY

Enforcement Method of Iodosulfuron-methyl-sodium and its metabolite Metsulfuron-methyl in Surface Water by HPLC incl. Validation

Extension of the enforcement method EM F 01/98 - 0 for Iodosulfuron-methyl-sodium in Drinking Water to its metabolite Metsulfuron-methyl incl. Validation

Iodosulfuron-methyl-sodium (AE F115008)
Metsulfuron-methyl (AE F075736)

Relevant residue
Iodosulfuron-methyl-sodium (AE F115008)
Metsulfuron-methyl (AE F075736)

Test commodity
Drinking water
Surface water

Principle of the method
Metsulfuron-methyl in drinking water:

The water sample is adjusted to pH 2.5 with phosphoric acid (2 N) and sucked through an C18-cartridge (conditioned with 5 mL methanol and 5 mL water). AE F075736 is eluated with 5 mL methanol. Metsulfuron-methyl in the final solution in acetonitrile/water (1/1, v/v) is determined by HPLC/UV.

Metsulfuron-methyl and iodosulfuron-methyl-sodium in surface water:

The water sample is adjusted to pH 2.5 with phosphoric acid (2 N) and filtered through a glass microfibre filter and a cellulose nitrate filter (0.45 µm). The sample is sucked through a NH2 / C18-cartridge (conditioned with 5 mL methanol and 5 mL water). AE F115008 and AE F075736 are eluated with 15 mL methanol/water (60:40, v/v). After reducing to dryness, the residue is dissolved in 20 mL toluene and sucked through a Silicagel-cartridge (conditioned with 5 mL toluene). AE F115008 and AE F075736 are eluted with 30 mL toluene/methanol (95:5, v/v). Iodosulfuron-methyl-sodium and metsulfuron-methyl in the final solution in acetonitrile/water (1/1, v/v) are determined by HPLC/UV.

Calibration
A curve of the form $y = a + bx + cx^2$ is applicable over the tested range of 0.1 to 2.0 µg metsulfuron-methyl/mL and 0.1 to 2.0 µg iodosulfuron-methyl-sodium/mL.

Recovery efficiency, relative standard deviation (RSD)
Recovery experiments were conducted at 0.1 µg/L and 1.0 µg/L for AE F075736 in **drinking water** and at 0.1 µg/L and 1.0 µg/L for AE F075736 and AE F115008 in **surface water**.

The mean recoveries for AE F075736 in **drinking water** were found at 79 and 92 %, with an RSD of up to 9 %.

In **surface water** the mean recoveries for AE F075736 were found at 85 and 105 %, with an RSD of up to 10 %, the confirmation method gave mean recovery values of 92 and 106 %, with an RSD of up to 18 %.

In **surface water** the mean recoveries for AE F115008 were found at 92 and 103 %, with an RSD of up to 9 %, the confirmation method gave mean recovery values of 85 and 96 %, with an RSD of up to 20 %.

The results are summarized in the table below.

Limit of quantification
The limit of quantification (LOQ) for metsulfuron-methyl in drinking water was established and validated at 0.1 µg/L.
The limit of quantification (LOQ) for iodosulfuron-methyl-sodium and metsulfuron-methyl in surface water was established and validated at 0.1 µg/L.

Specificity
Control samples of drinking water were analysed for AE F075736 and control samples of surface water were analysed for AE F115008 and AE F075736. In none of the samples apparent residues were determined.

The specificity of the method was demonstrated by a confirmatory technique using a HPLC column with a different stationary phase.

Summary table of recoveries:

Analyte	Matrix	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
AE F075736	Drinking water (Vittel)	0.10	82, 75, 87, 83, 70	79	9	5
		1.0	96, 91, 87, 93, 93	92	4	5
	Surface water	0.10	90, 78, 91, 92, 74	85	10	5
		1.0	98, 102, 105, 109, 109	105	5	5
	Surface water Confirmation method	0.10	74, 110, 92, 106, 108, 90, 67	92	18	7
		1.0	102, 106, 106, 108, 109	106	3	5
AE F115008	Surface water	0.10	108, 92, 94, 106, 113	103	9	5
		1.0	89, 90, 90, 94, 95	92	3	5
	Surface water Confirmation method	0.10	75, 78, 74, 114, 98, 73	85	20	6
		1.0	92, 97, 97, 97, 98	96	2	5

a) RSD: relative standard deviation RSD = SD / Mean recovery • 100 %

Analytical method flow sheet

Metsulfuron-methyl in drinking water

Extraction AE F075736 and C18-cartridge clean-up

1000 mL water
is adjust to pH 2.5 with phosphoric acid (2 N)
and sucked through an Chromabond 500 mg C18-cartridge
(conditioned with 5 mL methanol and 5 mL water)
with a flow rate of ca. 10 – 20 mL/min

↓

Wash used glassware with 200 mL Millipore water
and suck the washing water through the cartridge.

↓

Suck the C18-cartridge to dryness within ca. 5 min.

↓

Eluate AE F075736 with 5 mL methanol
into a test tube

↓

Reduce the eluate to dryness using a vacuum rotary evaporator
(bath temperature ca. 50 °C).

↓

HPLC

Dissolve the residue in 1.0 mL water/acetonitile (1/1, v/v) for quantification
with UV/HPLC

Iodosulfuron-methyl-sodium and metsulfuron-methyl in surface water

Extraction AE F115008 and AE F075736 and NH2 / C18-cartridge clean-up	1000 mL water is adjust to pH 2.5 with phosphoric acid (2 N), filtered through a glass microfibre filter and a cellulose nitrate filter (0.45 µm) the sample is sucked through a Chromabond 500 mg NH2 / 500 mg C18-cartridge (conditioned with 5 mL methanol and 5 mL water) with a flow rate of ca. 10 – 20 mL/min
	Wash used glassware with 200 mL Millipore water and suck the washing water through the cartridge.
	Suck the NH2 / C18-cartridge to dryness within ca. 5 min.
	Wash the NH2 / C18-cartirdge with 10 mL methanol/water (30:70, v/v).
	Eluate AE F115008 and AE F075736 with 15 mL methanol/water (60:40, v/v)
	Reduce the eluate to dryness using a vacuum rotary evaporator (bath temperature ca. 50 °C).
Silicagel-cartridge clean-up	Dissolve the residue in 20 mL toluene (if necessary use an ultrasonic bath) suck through a Silicagel-cartridge (conditioned with 5 mL toluene)
	Discard the eluate, suck the Silicagel-cartridge to dryness
	Wash the round-bottom flask with 30 mL toluene/methanol (95:5, v/v) and elute AE F115008 and AE F075736 with this solution Reduce the eluate to dryness using a vacuum rotary evaporator (bath temperature ca. 40 °C).
HPLC	Dissolve the residue in 1.0 mL water/acetonitile (1/1, v/v) for quantification with UV/HPLC

1 Organization and Personnel

Sponsor:	AgrEvo GmbH D - 65926 Frankfurt am Main
Test facility:	AgrEvo GmbH Rückstände und Verbrauchersicherheit D - 65926 Frankfurt am Main Head: Dr. M. Uihlein
Study No.:	CR 99/029
Study director:	Dr. A. Wrede Address: see Test facility
Method No.:	EM F 12/99-0

Archiving:
The study plan, the raw data and the original report will be archived at

Hoechst Schering AgrEvo GmbH
Rückstände und Verbrauchersicherheit
D-65926 Frankfurt am Main

Retained samples of the test and reference substances for the analytical work will be archived at

Hoechst Schering AgrEvo GmbH
Produktanalytik
D-65926 Frankfurt am Main

for at least the duration of the period prescribed in the GLP guidelines.

Quality assurance:

Hoechst Schering AgrEvo GmbH
GLP-Qualitätssicherungseinheit
D-65926 Frankfurt am Main

Time schedule:

Study plan signed by study director:	29 Sep 1999
Start of analysis:	22 Nov 1999
End of analysis:	25 Jan 2000

2 Objectives

The objective of this study was the development and validation of an analytical method for the determination of residues of metsulfuron-methyl (AE F 075736) in drinking water and metsulfuron-methyl (AE F075736) and iodosulfuron-methyl-sodium (AE F115008) in surface water by HPLC using UV-detection.
The validation for AE F115008 in drinking water is given in the analytical method EM F 01/98 – 0 (ref. 1).

3 Test commodities

The drinking water used for the validation was Vittel.
The samples of surface water used for the validation were taken from the small lake at building F821 (Industriepark Höchst).

The characteristics [1] of the surface water are:

pH	7.90 ± 0.02
DOC[1] [mg/L][1]	7.3 ± 0.3
total hardness [° dH][2]	10.1

[1] dissolved organic content
[2] degree german hardness [mg CaO+MgO / 100 mL water]

4 Relevant residue and reference substances

4.1 Relevant residue

The relevant residue consists of the metabolite metsulfuron-methyl (AE F075736) and the parent compound iodosulfuron-methyl-sodium (AE F115008).

[1] Determination of the characteristics of the surface water was not done under GLP.

4.2 Test and reference substances

Iodosulfuron-methyl-sodium (AE F115008)

Chemical name (IUPAC): methyl 4-iodo-2-[3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)ureido-sulfonyl]benzoate, sodium salt

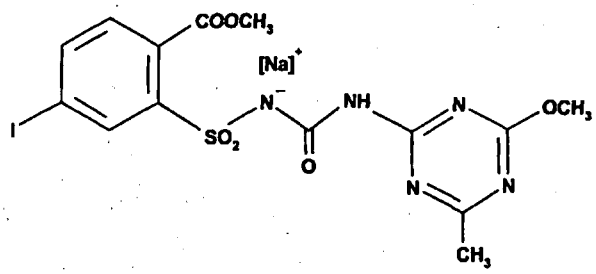
Empirical formula: $C_{14}H_{13}IN_5NaO_6S$

Molecular weight: 529.3

Solubility (20 °C):

Solvent	Solubility		Source
acetone	> 380	g/L	ref. 2
dichloromethane	> 500	g/L	ref. 2
ethyl acetate	23	g/L	ref. 2
n-hexane	$1.2 \bullet 10^{-3}$	g/L	ref. 2
methanol	12	g/L	ref. 2
n-heptane	$1.1 \bullet 10^{-3}$	g/L	ref. 2
2-propanol	4.4	g/L	ref. 2
toluene	2.1	g/L	ref. 2
acetonitrile	52	g/L	ref. 2
DMSO	> 500	g/L	ref. 2
PEG	87	g/L	ref. 2

Structural formula:



Certificate of analysis: AZ 07931

Drawn up by: Hoechst Schering AgrEvo GmbH
Produktanalytik
D-65926 Frankfurt am Main, Germany

Purity: 97.3 % (w/w)

Expiry date (d/m/y): 30 May 2000

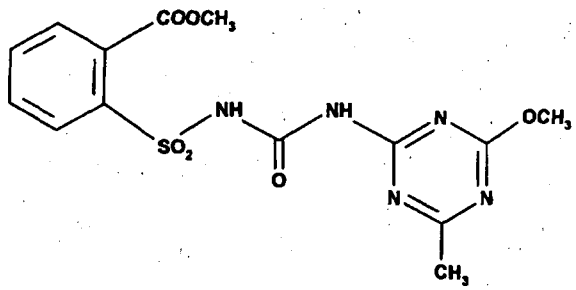
Metsulfuron-methyl (AE F075736)

Chemical name (IUPAC): methyl 2-[3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl) ureidosulfonyl]-benzoate

Empirical formula: $C_{14}H_{15}N_5O_6S$

Molecular weight: 381.4

Structural formula:



Certificate of analysis: AZ 06892
Drawn up by: Hoechst Schering AgrEvo GmbH
Produktanalytik
D-65926 Frankfurt am Main, Germany
Purity: 98.4 % (w/w)
Expiry date (d/m/y): 07 May 2000

5 Procedures

5.1 Principle of analytical method

The method flow sheets are presented in Appendix I.

Metsulfuron-methyl in drinking water:

The water sample is adjust to pH 2.5 with phosphoric acid (2 N) and sucked through an C18-cartridge (conditioned with 5 mL methanol and 5 mL water). AE F075736 is eluated with 5 mL methanol. AE F075736 in the final solution in acetonitrile/water (1/1, v/v) is determined by HPLC/UV.

Metsulfuron-methyl and iodosulfuron-methyl-sodium in surface water:

The water sample is adjust to pH 2.5 with phosphoric acid (2 N) and filtered through a glass microfibre filter and a cellulose nitrate filter (0.45 µm). The sample is sucked through a NH2 / C18-cartridge (conditioned with 5 mL methanol and 5 mL water). AE F115008 and AEF075736 are eluated with 15 mL methanol/water (60:40, v/v). After reducing to dryness, the residue is dissolved in 20 mL toluene and sucked through a Silicagel-cartridge (conditioned with 5 mL toluene). AE F115008 and AE F075736 are eluted with 30 mL toluene/methanol (95:5, v/v). AE F115008 and AE F075736 in the final solution in acetonitrile/water (1/1, v/v) are determined by HPLC/UV.

5.2 Reagents

- methanol Chromasolv, cat. No. 34860 (Riedel-de Haën, Germany)
- acetonitrile Chromasolv p.A., cat. No. 34851(Riedel-de Haën, Germany)
- deionized water
- water (e.g. prepared with Milli-Q-Plus, Millipore)
- phosphoric acid 2 N, cat. No. 30417 (Riedel-de Haën, Germany)
- toluene Pestanal, cat. No. 34494 (Riedel-de Haën, Germany)
- AE F075736, analytical standard (AgrEvo GmbH, Germany)
- AE F115008, analytical standard (AgrEvo GmbH, Germany)
- C18 – cartridge, 500 mg, cat. No. 730013 (Chromabond)
- Glass microfibre filter, cat. No. 1827070 (Whatman)
- Cellulose nitrate filter (0.45 µm), cat. No. 11306-50-N (Satorius)
- NH2/C18 – cartridge, 500mg NH2, 500mg C18, cat. No. 730618 (Chromabond)
- Silicagel-cartridge, 500 mg, ISOLUTE, cat. No. 460-0050-H (ICT)

Stock solutions of the analytical standards were prepared by dissolving about 50 mg of analytical standard of AE F075736 and 50 mg of the analytical standard of AE F115008 in ca. 50 mL acetonitrile / triethylamine (0.02 mol/L), 4:1, v/v. Concentration of the stock solutions was 1.0 mg/mL. Working solutions were prepared from the stock solution by further dilution with acetonitrile / water, 1:1, v/v.

5.3 Apparatus

The following list contains the apparatus used in the laboratory of the author for validation. Suitable alternatives can be taken.

- standard laboratory glassware
- rotary vacuum evaporator with water bath
- HPLC system with UV-detector
- chromatography column, Prodigy ODS, 150 mm x 4.6 mm, 5 µm
- chromatography column, Nucleosil C18, 5 µm, 250 mm x 4 mm (confirmation method)

5.4 Preparation of samples and storage

The samples of drinking water (Vittel) were bought November 1999.
The samples of surface water were taken from the small lake at building F821 (Industriepark Höchst) on 14 Jan 2000.

Samples were stored at room temperature.

5.5 Laboratory steps

5.5.1 Metsulfuron-methyl in drinking water

5.5.1.1 Extraction and C18-cartridge clean up

1000 mL of the water sample is adjust to pH 2.5 with phosphoric acid (2 N) and sucked through a Chromabond 500 mg C18-cartridge (conditioned with 5 mL methanol and 5 mL water) with a flow rate of ca. 10 – 20 mL/min. Wash used glassware with 200 mL Millipore water and suck the washing water through the cartridge. Suck the C18-cartridge to dryness within ca. 5 min. Eluate AE F075736 with 5 mL methanol into a test tube. Reduce the eluate to dryness using a vacuum rotary evaporator (bath temperature ca. 50 °C).

5.5.1.2 Preparation of the final solution

Dissolve the residue in 1.0 mL acetonitrile/water (1/1, v/v).

5.5.1.3 Determination of residues

The following conditions have been used successfully during validation of this analytical method. If different equipment and columns are used, modifications of the given conditions may be necessary.

HPLC-conditions

Instrument:	Beckmann
System	IBM-PC System 2 8570 Model 70386
Pump:	226 Beckmann
Detector:	Diode Array Detector 168 Beckmann
Injector:	Autosampler 507 Beckmann
Injection volume:	100 µL
Column temperature:	30 °C
Column	Prodigy ODS, 5 µm, 150 mm x 4,6 mm
Wavelength:	233 nm
Flow rate:	1.0 mL/min
Mobile phase:	
Eluent A	Acetonitrile Chromasolv
Eluent B	Phosphoric acid c_{H3PO4}= 0.01mol/L

Gradient program for the determination of AE F075736

Time [min]	Total flow pump A + B [mL/min]	Pump A (eluent A) Acetonitrile Chromasolv [%]	Pump B (eluent B) phosphoric acid c_{H3PO4}= 0.01mol/L [%]
0	1.0	20	80
10	1.0	50	50
20	1.0	50	50
30	1.0	80	20
35	1.0	80	20
45	1.0	20	80
47	1.0	20	80
55	1.0		

Under these conditions the retention time for AE F075736 is about 21.0 min.

The chromatography data were recorded and evaluated with TURBOCHROM© Client/Server system, PERKIN ELMER.

5.5.2 Iodosulfuron-methyl-sodium and metsulfuron-methyl in surface water

5.5.2.1 Extraction and NH2/C18-cartridge clean up

1000 mL of the water sample is adjust to pH 2.5 with phosphoric acid (2 N) and filtered through a glass microfibre filter and a cellulose nitrate filter (0.45 µm). The sample is sucked through a Chromabond 500 mg NH2 / 500 mg C18-cartridge (conditioned with 5 mL methanol and 5 mL water) with a flow rate of ca. 10 – 20 mL/min. Wash used glassware with 200 mL Millipore water and suck the washing water through the cartridge. Suck the NH2 / C18-cartridge to dryness within ca. 5 min. and wash the NH2 / C18-cartirdge with 10 mL methanol/water (30:70, v/v). Eluate AE F115008 and AE F075736 with 15 mL methanol/water (60:40, v/v) into a round-bottom flask. Reduce the eluate to dryness using a vacuum rotary evaporator (bath temperature ca. 50 °C).

5.5.2.2 Silicagel-cartridge clean up

Dissolve the residue in 20 mL toluene (if necessary use an ultrasonic bath) and suck through a Silicagel-cartridge (conditioned with 5 mL toluene). Discard the eluate, suck the Silicagel-cartridge to dryness. Wash the round-bottom flask with 30 mL toluene/methanol (95:5, v/v), if necessary use an ultrasonic bath, and elute AE F115008 and AE F075736 with this solution. Reduce the eluate to dryness using a vacuum rotary evaporator (bath temperature ca. 40 °C).

5.5.2.3 Preparation of the final solution

Dissolve the residue in 1.0 mL acetonitrile/water (1/1, v/v).

5.5.2.4 Determination of residues

The following conditions have been used successfully during validation of this analytical method. If different equipment and columns are used, modifications of the given conditions may be necessary.

HPLC-conditions

Instrument:	Beckmann
System	IBM-PC System 2 8570 Model 70386
Pump:	226 Beckmann
Detector:	Diode Array Detector 168 Beckmann
Injector:	Autosampler 507 Beckmann
Injection volume:	100 µL
Column oven:	Beckmann
Column temperature:	30 °C
Column	Prodigy ODS, 5 µm, 150 mm x 4,6 mm (validation)
Column	Nucleosil C18, 5 µm, 250 mm x 4 mm (confirmation method)
Wavelength:	233 nm
Flow rate:	1.0 mL/min
Mobile phase:	
Eluent A	Acetonitrile Chromasolv
Eluent B	Phosphoric acid c_{H3PO4}= 0.01mol/L

Gradient program for the determination of AE F075736

Time [min]	Total flow pump A + B [mL/min]	Pump A (eluent A) Acetonitrile Chromasolv [%]	Pump B (eluent B) phosphoric acid c_{H3PO4}= 0.01mol/L [%]
0	1.0	20	80
10	1.0	50	50
20	1.0	50	50
30	1.0	80	20
35	1.0	80	20
45	1.0	20	80
47	1.0	20	80
55	1.0		

Under these conditions the retention time for AE F075736 is about 21.0 min and for AE F115008 about 25.4 min.

The chromatography data were recorded and evaluated with TURBOCHROM© Client/Server system, PERKIN ELMER.

Confirmatory method
For confirmatory purposes a different stationary phase was used:

HPLC-Column: Nucleosil C18, 5 µm, 250 mm x 4 mm

Under these conditions the retention time for AE F075736 is about 21.5 min and for AE F115008 about 25.5 min.

5.6 Calibration

The concentration of AE F075736 and AE F115008 were calculated using external standards at 4 different concentrations over a range from 0.1 ng/µL up to 1 or 2 ng/µL. The lowest concentration was 0.1 ng/µL. The highest concentration was 2 ng/µL.

The recommended order of samples / test solutions for setting up a sequence for HPLC-determination is 'test solution – sample - test solution - sample '. If different equipment is used and /or more or less samples are worked up, modifications of this order may be necessary.

5.7 Calculations

Determination of concentration of the analytical target in the final solution
The concentrations of the analytes in control samples, fortified samples and treated samples were calculated using external standard procedures with multi level or single level calibration.

Single level calibration (one point calibration):

$$C_S = \frac{P_S}{P_R} \bullet C_R \bullet \frac{I_R}{T_4} \qquad \left[pg/\mu L = \frac{counts}{counts} \bullet pg/\mu L \bullet \frac{\mu L}{\mu L} \right] \qquad (1)$$

C_S	Concentration in final sample solution V_{end} (identical with conc. in T_4) (treated, untreated and recovery)	[pg/µL] = [ng/mL]
C_R	Concentration in reference solution	[pg/µL] = [ng/mL]
P_S	Peak area or peak height of the sample solution	[counts]
P_R	Peak area or peak height of the reference solution	[counts]
T_4	Injection volume of the sample solution	[µL]
I_R	Injection volume of the reference solutions	[µL]

Multi level calibration (calibration curve):

For the calibration peak areas (heights) of the standards were plotted versus the corresponding concentrations. An optimized calibration curve of the following form

$$f(C_S) = P = a + bC_S + cC_S^2 \qquad (2)$$

is calculated, where $f(C_S)$ is the peak area (height), C_S the concentration of the analyte in the final sample extract and a, b, c are constants.

Determination of residues

Calculation of residues was carried out by a data handling software according to the following procedure

$$Res = \frac{C_S \bullet V_{end} \bullet f}{W} \qquad \left[\mu g/L = \frac{(ng/mL) \bullet mL \bullet 1}{mL}\right] \qquad (3)$$

$$f = \frac{V_1 \bullet V_2 \bullet V_n}{T_1 \bullet T_2 \bullet T_n} \qquad \left[1 = \frac{mL \bullet mL \bullet mL}{mL \bullet mL \bullet mL}\right] \qquad (4)$$

Res	Residue	[µg/L]
C_S	Concentration in final sample solution V_{end} (treated, untreated and recovery)	[ng/mL]
W	Sample weight	[mL]
f	Dilution factor	without dimension
V_1	Volume for primary extraction	[mL]
V_2	Volume after making up of aliquot T_1	[mL]
V_n	Volume after making up of aliquot T_{n-1} (n = 3, 4 and so on)	[mL]
V_{end}	Final sample solution (identical with V_2 or V_3 or V_n depending on the method)	[mL]
T_1	Aliquot of V_1	[mL]
T_2	Aliquot of V_2	[mL]
T_n	Aliquot of V_n (n = 3, 4 and so on)	[mL]

Determination of recovery rates

Calculation of recovery rates were carried out by a data handling software according to the following procedure

$$Res_d = Res_{(Rec)} - Res_{(Unt)} \qquad \left[\frac{\mu g}{L} = \frac{\mu g}{L} - \frac{\mu g}{L}\right] \qquad (5)$$

$$Rec = \frac{Res_d}{Res_f} \bullet 100 \qquad \left[\% = \frac{\mu g/L}{\mu g/L} \bullet \%\right] \qquad (6)$$

$Res_{(Rec)}$	Residue in the sample solution of the recovery test calculated with equation (3) and (4)	[µg/L]
$Res_{(Unt)}$	Residue in the sample solution of the corresponding untreated control sample calculated with equation (3) and (4)	[µg/L]
Rec	Recovery rate	[%]
Res_f	Concentration spiked for fortification	[µg/L]
Res_d	Concentration detected by analytical method	[µg/L]

6 Results

Calculation sheets and calibration curves for all laboratory analyses are given in Annex II and III with examples of chromatograms given in Annex VI.

6.1 Recoveries

Recovery experiments for method validation were conducted at 0.1 and 1.0 µg/L. At each level for drinking water or surface water a min. of 5 recovery values were determined. Recoveries are calculated with calibration curve according to the procedure described in section 5.8 with correction for any apparent residues.

The mean recoveries for AE F075736 in **drinking water** were found at 79 and 92 %, with an RSD of up to 9 %.

In **surface water** the mean recoveries for AE F075736 were found at 85 and 105 %, with an RSD of up to 10 %, the confirmation method gave mean recovery values of 92 and 106 %, with an RSD of up to 18 %.

In **surface water** the mean recoveries for AE F115008 were found at 92 and 103 %, with an RSD of up to 9 %, the confirmation method gave mean recovery values of 85 and 96 %, with an RSD of up to 20 %.

A summary of all recoveries for AE F075736 and AE F115008 are given in the following tables.

Summary table of recoveries:

Analyte: AE F075736

Matrix	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
Drinking water (Vittel)	0.10	82 75 87 83 70	79	9	5
	1.0	96 91 87 93 93	92	4	5
Surface water	0.10	90 78 91 92 74	85	10	5
	1.0	98 102 105 109 109	105	5	5
Surface water Confirmation method	0.10	74 110 92 106 108 90 67 35 (b)	92	18	7
	1.0	102 106 106 108 109	106	3	5

The calculation was done with calibration function of peak areas.

a) RSD = S.D. / Mean Recovery • 100 %

$$S.D. = \left[\frac{\Sigma (R_i - R_m)^2}{n - 1}\right]^{1/2}$$

R_i: recovery
R_m: mean recovery
n: number of recoveries

b) Outliner Injection problems, HPLC system used to much solution for washing, so the injection volume was too small

Summary table of recoveries:

Analyte: AE F115008

Matrix	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
Surface water	0.10	108 92 94 106 113	103	9	5
	1.0	89 90 90 94 95	92	3	5
Surface water Confirmation method	0.10	75 78 74 114 138 (b) 98 73 36 (c)	85	20	6
	1.0	92 97 97 97 98	96	2	5

The calculation was done with calibration function of peak areas.

a) RSD = S.D. / Mean Recovery • 100 %

$$S.D. = \left[\frac{\Sigma (R_i - R_m)^2}{n - 1}\right]^{1/2}$$

R_i: recovery
R_m: mean recovery
n: number of recoveries

b) Outliner Unexpected high value (results including this value: mean recovery = 93 %, RSD = 27 %, n = 7)

c) Outliner Injection problems, HPLC system used to much solution for washing, so the injection volume was too small

6.2 Limit of quantification (LOQ)

Drinking water:

The lowest level at which metsulfuron-methyl has been quantified in this study is 0.1 µg/L. This level is therefore considered to be an appropriate limit of quantification for AE F075736 in drinking water.

The LOQ (0.1 µg/L) for AE F115008 in drinking water is given in the analytical method EM F 01/98 – 0 (ref. 1).

Surface water

The lowest level at which iodosulfuron-methyl-sodium and metsulfuron-methyl have been quantified in this study is 0.1 µg/L. This level is therefore considered to be an appropriate limit of quantification for AE F115008 and AE F075736 in surface water.

6.3 Blank values

Drinking water:
Analysis of control samples has shown that apparent residues of AE F075736 observed were n.d. (not detectable, < 0.3 x LOQ). This demonstrates that 0.1 µg/L is a feasible level for recognition of residues with reasonable certainty.

Surface water:
Analysis of control samples has shown that apparent residues of AE F115008 and AE F075736 observed were n.d. (not detectable, < 0.3 x LOQ). This demonstrates that 0.1 µg/L is a feasible level for recognition of residues with reasonable certainty.

6.4 Critical steps of the method

There are no critical steps of the method.

6.5 Time for analysis

From extraction of the samples to preparation of the final solutions for HPLC/UV determination, it is normally possible to analyse a batch of 14 samples in three day.

REFERENCES

No	Doc No	Report No	**Author(s)** Title Source and Date
1	C000710	EM F 01/98 - 0	Wrede A., 1998 Code AE F115008 Analytical method and validation for the determination of residues of AE F115008 and its metabolite AE F075736 in water using HPLC
2	A54684	CP 94/070	Sadowsky-Dunkmann I., Schmidt W., 1995 Substance, pure Code: HOE 115008 00 ZB97 0001 Solubility in organic solvents/vehicles according to Commission Directive 94/37/EEC (1994)

Annex I: Analytical method flow sheet

Metsulfuron-methyl in drinking water

Extraction AE F075736 and C18-cartridge clean-up

1000 mL water
is adjust to pH 2.5 with phosphoric acid (2 N)
and sucked through an Chromabond 500 mg C18-cartridge
(conditioned with 5 mL methanol and 5 mL water)
with a flow rate of ca. 10 – 20 mL/min

↓

Wash used glassware with 200 mL Millipore water
and suck the washing water through the cartridge.

↓

Suck the C18-cartridge to dryness within ca. 5 min.

↓

Eluate AE F075736 with 5 mL methanol
into a test tube

↓

Reduce the eluate to dryness using a vacuum rotary evaporator
(bath temperature ca. 50 °C).

↓

HPLC

dissolve the residue in 1.0 mL water/acetonitrile (1/1, v/v) for quantification
with UV/HPLC

Iodosulfuron-methyl-sodium and metsulfuron-methyl in surface water

Extraction AE F115008 and AE F075736 and NH2 / C18-*cartridge clean-up*	1000 mL water is adjust to pH 2.5 with phosphoric acid (2 N), filtered through a glass microfibre filter and a cellulose nitrate filter (0.45 µm) the sample is sucked through a Chromabond 500 mg NH2 / 500 mg C18-cartridge (conditioned with 5 mL methanol and 5 mL water) with a flow rate of ca. 10 – 20 mL/min
	Wash used glassware with 200 mL Millipore water and suck the washing water through the cartridge.
	Suck the NH2 / C18-cartridge to dryness within ca. 5 min.
	Wash the NH2 / C18-cartirdge with 10 mL methanol/water (30:70, v/v).
	Eluate AE F115008 and AE F075736 with 15 mL methanol/water (60:40, v/v)
	Reduce the eluate to dryness using a vacuum rotary evaporator (bath temperature ca. 50 °C).
Silicagel-cartridge clean-up	Dissolve the residue in 20 mL toluene (if necessary use an ultrasonic bath) suck through a Silicagel-cartridge (conditioned with 5 mL toluene)
	Discard the eluate, suck the Silicagel-cartridge to dryness
	Wash the round-bottom flask with 30 mL toluene/methanol (95:5, v/v) and elute AE F115008 and AE F075736 with this solution Reduce the eluate to dryness using a vacuum rotary evaporator (bath temperature ca. 40 °C).
HPLC	dissolve the residue in 1.0 mL water/acetonitile (1/1, v/v) for quantification with UV/HPLC

Annex II: Calibration curves

AE F075736
I

(drinking water)

Calibration Results

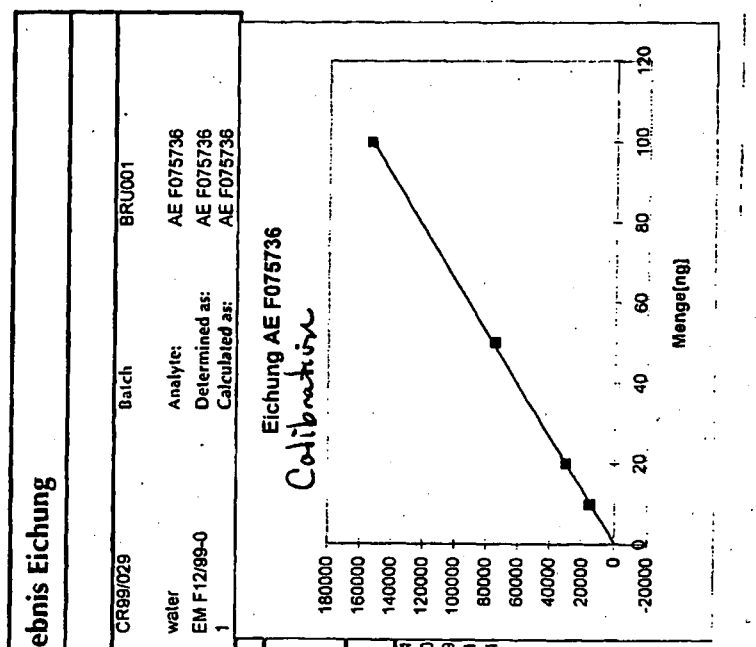
Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU001
Submission ID:	100000346	Crop:			
Operation:	AE F075736(WASSER/MP/EM F1	Commudity:	water	Analyte:	AE F075736
Date:	23.11.1999	AL-Method:	EM F12/99-0	Determined as:	AE F075736
Filename:		Factor:	1	Calculated as:	AE F075736

y=A+B*x, Weighting: None

A: -4.33E+02
B: 1.54E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng	Sign.(exp)	Sign.(ber)
E001	10	15087	14924
E002	20	30083	30280
E003	50	74944	76349
E004	100	153777	153131
E005	10	15716	14924
Stdabw:	1014		



Calibration curves

AE F075736
II

(drinking water)

Calibration Results

Ergebnis Eichung

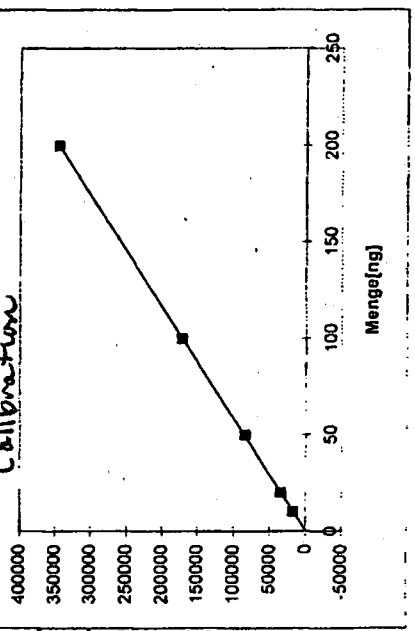
Analyst:	BRUEGGEMANN	Study ID:	CR99/028	Batch	BRU003
Submission ID:	100000346	Crop:			
Operation:	AE F075736(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F075736
Date:	09.12.1999	AL-Method:	EM F12/99-0	Determined as:	AE F075736
Filename:		Factor:	1	Calculated as:	AE F075736

y=A+B*x, Weighting: None

A: -3.51E+02
B: 1.73E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng	Sign.(exp)	Sign.(ber)
E018	10	17841	16934
E019	20	34251	34220
E020	50	84410	86076
E021	100	172917	172502
E022	200	345642	345356
E023	20	34933	34220
E024	50	85489	86076
E025	100	172402	172502
Stdabw:	886		

Eichung AE F075736
Calibration



Calibration curves

AE F075736
III

(surface water)

Calibration Results

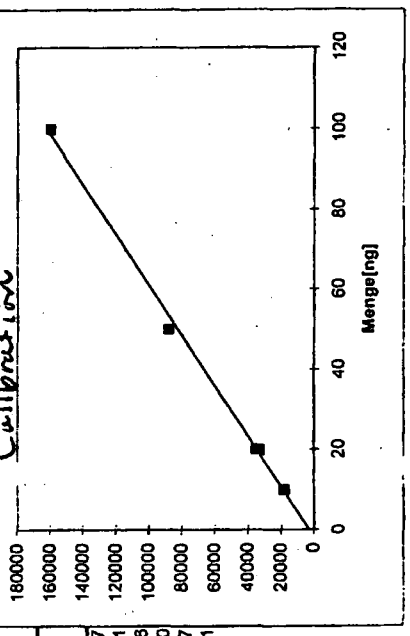
Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU007
Submission ID:	100000348	Crop:			
Operation:	AE F075736(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F075736
Date:	24.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F075736
Filename:		Factor:	1	Calculated as:	AE F075736

y=A+B*x, Weighting: None

A: 3.07E+03
B: 1.59E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng	Sign.(exp)		Sign.(ber)
E054	10		18536	18977
E055	20		32959	34881
E056	50		88301	82596
E057	100		159773	162120
E058	10		17758	18977
E059	20		35105	34881
Stdabw:	3297			



Calibration curves

AE F075736
IV

(surface water)

Calibration Results

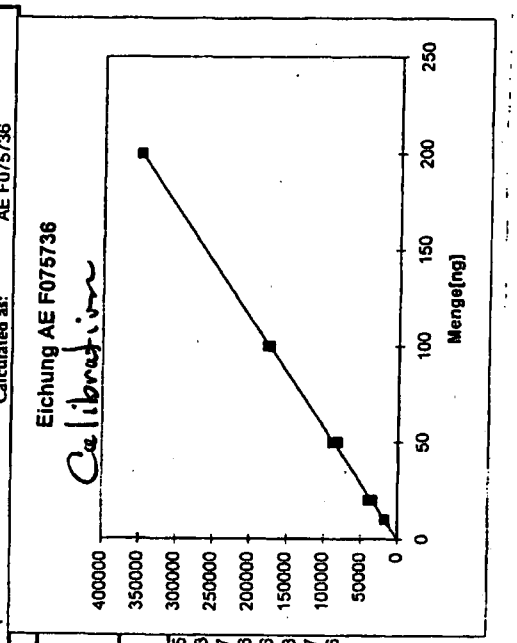
Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU008
Submission ID:	100000346	Crop:			
Operation:	AE F075736(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F075736
Date:	25.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F075736
Filename:		Factor:	1	Calculated as:	AE F075736

y=A+B*x, Weighting: None

A: 3.57E+02
B: 1.75E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng	Sign.(exp)		Sign.(ber)	
E062	10		17826		17815
E063	20		34416		35273
E064	50		82336		87647
E065	100		176646		174936
E066	200		349915		349516
E067	20		39681		35273
E068	50		88413		87647
E069	100		174010		174936
Sdabw:	2970				



Calibration curves

AE F075736
V

(surface water)
(confirmation method)

Calibration Results

Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU005
Submission ID:	100000346	Crop:			
Operation:	AE F075736(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F075738
Date:	18.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F075736
Filename:		Factor:	1	Calculated as:	AE F075736

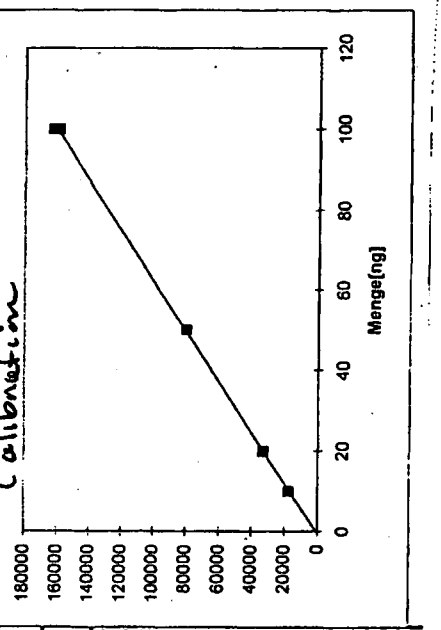
y=A+B*x, Weighting: None

A: 1.11E+03
B: 1.60E+03
C: 0.00E+00

excl.	Standard No	Menge(exp) ng		Sign.(exp)		Sign.(ber)
	E038		10		17428	17066
	E039		20		32714	33028
	E040		50		80124	80912
	E041		100		159251	160718
	E042		10		17898	17066
	E043		20		33157	33028
	E044		50		78847	80912
	E045		100		163029	160718
	Stdabw:		1303			

Eichung AE F075736

Calibration



Calibration curves

AE F075736
VI

(surface water)
(confirmation method)

Calibration Results

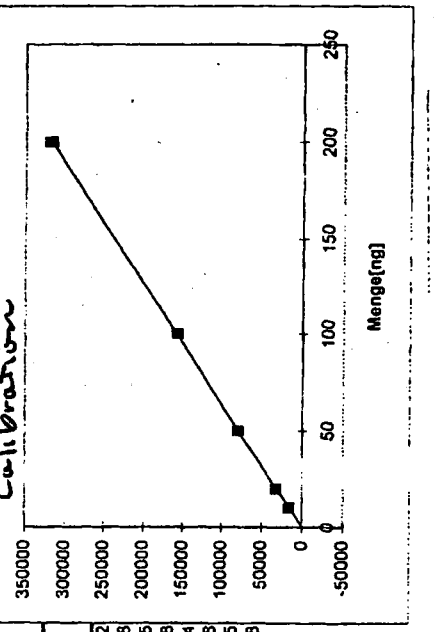
Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU008
Submission ID:	100000346	Crop:			
Operation:	AE F075736(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F075736
Date:	20.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F075736
Filename:		Factor:	1	Calculated as:	AE F075736

y=A+B*x, Weighting: None

A: -3.69E+02
B: 1.59E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng		Sign.(exp)		Sign.(ber)
E046		10		16425	15552
E047		50		79812	79238
E048		100		156679	158845
E049		200		316445	318058
E050		20		31939	31474
E051		50		78836	79238
E052		100		158256	158845
E053		200		320915	318058
Stdabw.		1697			



Calibration curves

AE F075736
VII

(surface water)
(confirmation method)

Calibration Results

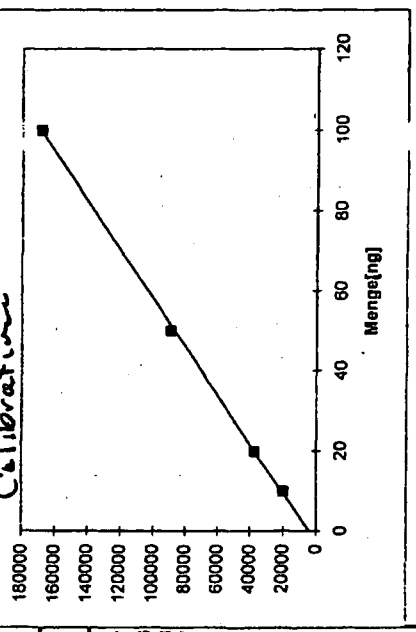
Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR98/029	Batch	BRU009
Submission ID:	100000346	Crop:			
Operation:	AE F075736(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F075736
Date:	27.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F075736
Filename:		Factor:	1	Calculated as:	AE F075736

y=A+B*x, Weighting: None

A: 4.32E+03
B: 1.65E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng		Sign.(exp)		Sign.(ber)
E070		10		19869	20821
E071		20		37294	37317
E072		50		89098	88806
E073		100		168310	169288
E074		20		36979	37317
Stdabw:		1552			



Calibration curves

AE F115008
I

(surface water)

Calibration Results

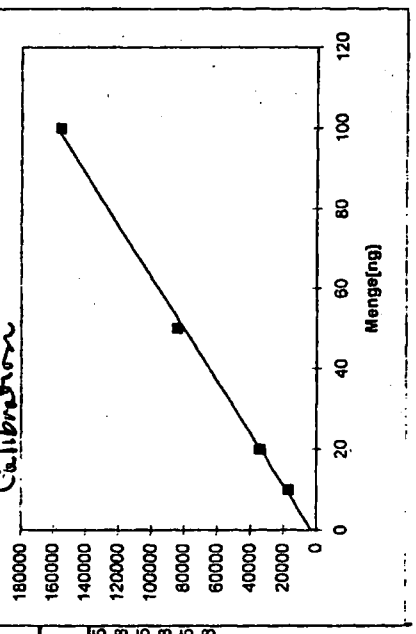
Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU007
Submission ID:	100000346	Crop:			
Operation:	AE F115008(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F115008
Date:	24.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F115008
Filename:		Factor:	1	Calculated as:	AE F115008

y=A+B*x, Weighting: None

A: 3.21E+03
B: 1.55E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng		Sign.(exp)	Sign.(ber)
E054		10	17265	18665
E055		20	34416	34118
E056		50	84508	80475
E057		100	155883	157738
E058		10	17166	18665
E059		20	34562	34118
Sdabw:		2463		



Calibration curves

AE F115008
II

(surface water)

Calibration Results

Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU008
Submission ID:	100000346	Crop:			
Operation:	AE F115008(WASSER/MIP/EM F1	Commodity:	water	Analyte:	AE F115008
Date:	25.01.2000	AL-Method:	EM F12/98-0	Determined as:	AE F115008
Filename:		Factor:	1	Calculated as:	AE F115008

Y=A+B*x, Weighting: None

A: 1.36E+03
B: 1.53E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng	Sign.(exp)		Sign.(ber)	
E062		10	16371		16670
E063		20	31363		31984
E064		50	79517		77927
E065		100	155876		154499
E066		200	308397		307643
E067		20	32420		31984
E068		50	78680		77927
E069		100	150710		154499
Sdabw.		1829			

Calibration curves

AE F115008
III

(surface water)
(confirmation method)

Calibration Results

Ergebnis Eichung

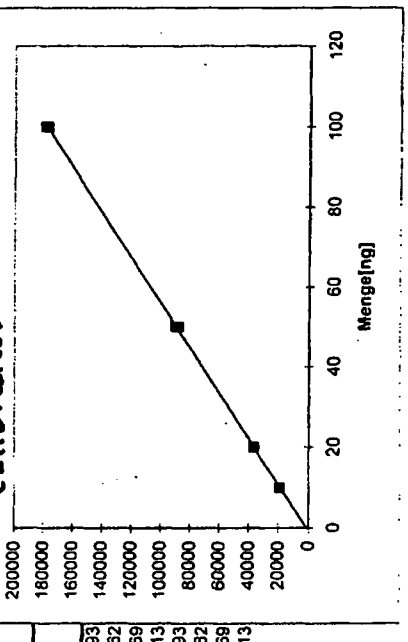
Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU005
Submission ID:	100000346	Crop:			
Operation:	AE F115008(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F115008
Date:	18.1.2000	AI-Method:	EM F12/99-0	Determined as:	AE F115008
Filename:		Factor:	1	Calculated as:	AE F115008

y=A+B*x, Weighting: None

A: 1.32E+03
B: 1.77E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng		Sign.(exp)		Sign.(ber)
E038		10		19350	18993
E039		20		36430	36662
E040		50		87795	89669
E041		100		177369	178013
E042		10		19501	18993
E043		20		36790	36682
E044		50		90154	89669
E045		100		179285	178013
Stdabw:		1019			

Eichung AE F115008
Calibration



Calibration curves

AE F115008 (surface water)
IV (confirmation method)

Calibration Results

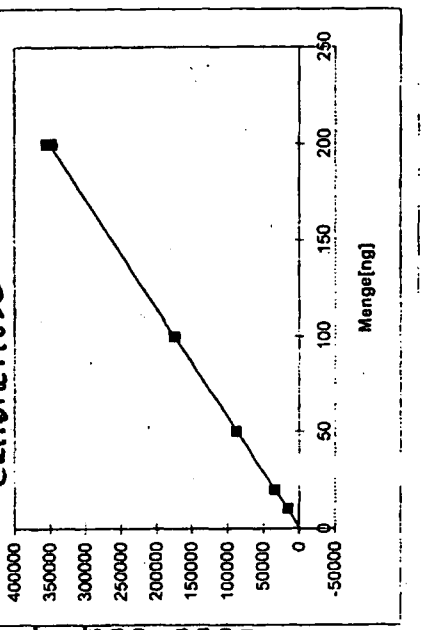
Ergebnis Eichung

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch	BRU006
Submission ID:	10000346	Crop:			
Operation:	AE F115008(WASSER/MP/EM F1	Commodity:	water	Analyte:	AE F115008
Date:	20.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F115008
Filename:		Factor:	1	Calculated as:	AE F115008

Y=A+B*x, Weighting: None

A: -6.43E+02
B: 1.76E+03
C: 0.00E+00

ext. Standard No	Menge(exp) ng	Sign.(exp)	Sign.(ber)
E046	10	16322	16925
E047	50	88019	87198
E048	100	174614	175039
E049	200	346719	350721
E050	20	34832	34483
E051	50	87945	87198
E052	100	173647	175039
E053	200	355236	350721
Stdabw:	2589		



Calibration curves

AE F115008 (surface water)
V (confirmation method)

Analyst: Weisel
Submission ID: 100000346
Operation:
Date: 27.1.2000
Filename:

Study ID: CR99/029
Crop: water
Commodity: water
AL-Method: EM F12/99-0
Factor: 1

Batch BRU009
Analyte: AE F115008
Determined as: AE F115008
Calculated as: AE F115008

y=A+B*x Weighting: None

A: 3.90E+03
B: 1.81E+03

ext.Standard No	Menge(exp) [ng]	Signal(exp)	Signal(theo)
E070	10.00	19691	22003
E071	20.00	40762	40110
E072	50.00	101176	94432
E073	100.00	182152	184970
E074	20.00	37844	40110
Stdabw:	4630.29		
Fehler-Summe	0.00		

Annex III: Calculation sheets

AE F075736
Drinking water

0.1 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst: BRUEGGEMANN Study ID: CR99/029 Batch: BRU001 SEND LOQ: 0.1 0.3*LOQ 0.03
Submission ID: 10000346 Crop: mean Blank: 0.01069 STD: 0.01512
Operation: AE F075736(WASSER/W Sample Mat.: water Analyte: AE F075736 mean Recov: 79 STD: 6.93
Date: 23.11.1999 AL-Method: EM F12/99-0 Determined as: AE F075736 RSD: 9
Filename: BR094604 Factor: 1.00 Calculated as: AE F075736 mean Lstab: 0 STD:

Sample_ID	Type	Task-ID	Lab Code	W	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 µl	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Resid ug/l	< LOQ	spiked Sample	Spiked ug/l	Rec %
200016106	BLI	300030073	U001	1	1	1	1	1	1	1	1	100	0	U001	-4.33E+02	1.54E+03	0.00E+00	0.000	n.d.			
200016107	BLI	300030074	U002	1	1	1	1	1	1	1	1	100	2850	U002	-4.33E+02	1.54E+03	0.00E+00	0.021	n.d.			
200016130	AUF	300030068	R001	1	1	1	1	1	1	1	1	100	13841	R001	-4.33E+02	1.54E+03	0.00E+00	0.093		MBV	0.1	82
200016131	AUF	300030071	R002	1	1	1	1	1	1	1	1	100	12673	R002	-4.33E+02	1.54E+03	0.00E+00	0.085		MBV	0.1	75
200016132	AUF	300030069	R003	1	1	1	1	1	1	1	1	100	14513	R003	-4.33E+02	1.54E+03	0.00E+00	0.097		MBV	0.1	87
200016133	AUF	300030072	R004	1	1	1	1	1	1	1	1	100	14017	R004	-4.33E+02	1.54E+03	0.00E+00	0.094		MBV	0.1	83
200016134	AUF	300030070	R005	1	1	1	1	1	1	1	1	100	11920	R005	-4.33E+02	1.54E+03	0.00E+00	0.080		MBV	0.1	70

Calculation sheets

AE F075736
Drinking water

1.0 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

| | | |

Calculation sheets

AE F075736
Surface water

0.1 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch:	BRU007 SEND	LOQ: 0.1		0.3*LOQ	0.03
Submission ID:	100000346	Crop:	water			mean Blank:	0	STD:	0
Operation:	AE F075736(WASSER/W	Sample Mat.:	water	Analyte:	AE F075736	mean Recov:	85	STD:	8.48
Date:	24.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F075736			RSD:	10
Filename:	BR061331	Factor:	1.00	Calculated as:	AE F075736	mean Lstab:	0	STD:	

Sample_ID	Type	Task-ID	Lab Code	W l	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 ul	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Resid ug/l	< LOQ	spiked Sample	Spiked ug/l	Rec %
200016122	BLI	300031929	U013	1	1	1	1	1	1	1	1	100	0	U013	3.07E+03	1.59E+03	0.00E+00	0.000	n.d.			
200016123	BLI	300031931	U014	1	1	1	1	1	1	1	1	100	0	U014	3.07E+03	1.59E+03	0.00E+00	0.000	n.d.			
200016915	AUF	300031919	R027	1	1	1	1	1	1	1	1	100	17441	R027	3.07E+03	1.59E+03	0.00E+00	0.090		MBV	0.1	90
200016916	AUF	300031925	R028	1	1	1	1	1	1	1	1	100	15474	R028	3.07E+03	1.59E+03	0.00E+00	0.078		MBV	0.1	78
200016917	AUF	300031921	R029	1	1	1	1	1	1	1	1	100	17604	R029	3.07E+03	1.59E+03	0.00E+00	0.091		MBV	0.1	91
200016918	AUF	300031927	R030	1	1	1	1	1	1	1	1	100	17685	R030	3.07E+03	1.59E+03	0.00E+00	0.092		MBV	0.1	92
200016919	AUF	300031923	R031	1	1	1	1	1	1	1	1	100	14837	R031	3.07E+03	1.59E+03	0.00E+00	0.074		MBV	0.1	74

Calculation sheets

AE F075736
Surface water

1.0 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst:	BRUEGGEMANN	Study ID:	CR99/029		Batch:			BRU008	SEND							LOQ: 0.1				0.3*LOQ	0.03						
Submission ID:	100000348	Crop:	water													mean Blank:	0			STD:	0						
Operation:	AE F075736(WASSER/N	Sample Mat.:	water		Analyte:			AE F075736								mean Recov:	105			STD:	4.83						
Date:	25.01.2000	AL-Method:	EM F12/99-0		Determined as:			AE F075736												RSD:	5						
Filename:	BR050443	Factor:	1.00		Calculated as:			AE F075736								mean Lstab:	0			STD:							
Sample_ID	Type	Task-ID	Lab Code	W	V1	T1	V2	T2	V3	T3	V4	T4	Sample	File	A	B	C	Resid	<	spiked	Spiked	Rec					
				l	ml	ml	ml	ml	ml	ml	ml	ul	Counts		counts	counts/ng	counts/ng*ng	ug/l	LOQ	Sample	ug/l	%					
200016124	BLI	300032117	U015	1	1	1	1	1	1	1	1	100	0	U015	3.57E+02	1.75E+03	0.00E+00	0.000	n.d.								
200016125	BLI	300032119	U016	1	1	1	1	1	1	1	1	100	0	U016	3.57E+02	1.75E+03	0.00E+00	0.000	n.d.								
200016967	AUF	300032107	R032	1	1	1	1	1	1	1	1	100	171221	R032	3.57E+02	1.75E+03	0.00E+00	0.98		MBV	1		98				
200016968	AUF	300032113	R033	1	1	1	1	1	1	1	1	100	178003	R033	3.57E+02	1.75E+03	0.00E+00	1.0		MBV	1		102				
200016969	AUF	300032109	R034	1	1	1	1	1	1	1	1	100	183506	R034	3.57E+02	1.75E+03	0.00E+00	1.0		MBV	1		105				
200016970	AUF	300032115	R035	1	1	1	1	1	1	1	1	100	190084	R035	3.57E+02	1.75E+03	0.00E+00	1.1		MBV	1		108				
200016971	AUF	300032111	R036	1	1	1	1	1	1	1	1	100	191374	R036	3.57E+02	1.75E+03	0.00E+00	1.1		MBV	1		109				

Calculation sheets

AE F075736
Surface water (confirmation method)

0.1 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst: BRUEGGEMANN Study ID: CR99029 Batch: BRU005 SEND LOQ: 0.1 0.3*LOQ 0.03
Submission ID: 100000346 Crop: water mean Blank: 0.0043 STD: 0.00609
Operation: AE F075736(WASSER/N Sample Mat.: water Analyte: AE F075736 mean Recov: 98 STD: 15.11
Date: 18.01.2000 AL-Method: EM F12/99-0 Determined as: AE F075736 RSD: 15
Filename: BR120447 Factor: 1.00 Calculated as: AE F075736 mean Lstab: 0 STD:

Sample_ID	Type	Task-ID	Lab Code	W	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 µl	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Readl ug/l	< LOQ	spiked Sample	Spiked ug/l	Rec %
200016118	BLI	300031470	U009	1	1	1	1	1	1	1	1	100	0	U009	1.11E+03	1.60E+03	0.00E+00	0.000	n.d.			
200016119	BLI	300031472	U010	1	1	1	1	1	1	1	1	100	2479	U010	1.11E+03	1.60E+03	0.00E+00	0.009	n.d.			
200016484	AUF	300031460	R017	1	1	1	1	1	1	1	1	100	13586	R017	1.11E+03	1.60E+03	0.00E+00	0.078		MBV	0.1	74
200016485	AUF	300031466	R018	1	1	1	1	1	1	1	1	100	19301	R018	1.11E+03	1.60E+03	0.00E+00	0.11		MBV	0.1	110
200016486	AUF	300031462	R019	1	1	1	1	1	1	1	1	100	16461	R019	1.11E+03	1.60E+03	0.00E+00	0.096		MBV	0.1	92
200016487	AUF	300031464	R020	1	1	1	1	1	1	1	1	100	18728	R020	1.11E+03	1.60E+03	0.00E+00	0.11		MBV	0.1	106
200016488	AUF	300031468	R021	1	1	1	1	1	1	1	1	100	18972	R021	1.11E+03	1.60E+03	0.00E+00	0.11		MBV	0.1	108

Calculation sheets

AE F075736 1.0 µg/L
Surface water (confirmation method)

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst: BRUEGGEMANN Study ID: CR99/029 Batch: BRU006 SEND LOQ: 0.1 0.3*LOQ 0.03
Submission ID: 100000346 Crop: water mean Blank: 0.0088 STD: 0.01217
Operation: AE F075736(WASSER/W Sample Mat.: water Analyte: AE F075736 mean Recov: 106 STD: 2.6
Date: 20.01.2000 AL-Method: EM F12/99-0 Determined as: AE F075736 RSD: 2
Filename: BR083833 Factor: 1.00 Calculated as: AE F075736 mean Lstab: 0 STD:

Sample_ID	Type	Task-ID	Lab Code	W	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 ul	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Resid ug/l	< LOQ	spiked Sample	Spiked ug/l	Rec %
200016120	BLI	300031620	U011	1	1	1	1	1	1	1	1	100	2371	U011	-3.69E+02	1.59E+03	0.00E+00	0.017	n.d.			
200016121	BLI	300031622	U012	1	1	1	1	1	1	1	1	100	0	U012	-3.69E+02	1.59E+03	0.00E+00	0.000	n.d.			
200016828	AUF	300031610	R022	1	1	1	1	1	1	1	1	100	183250	R022	-3.69E+02	1.59E+03	0.00E+00	1.0		MBV	1	102
200016829	AUF	300031616	R023	1	1	1	1	1	1	1	1	100	170449	R023	-3.69E+02	1.59E+03	0.00E+00	1.1		MBV	1	106
200016830	AUF	300031612	R024	1	1	1	1	1	1	1	1	100	169455	R024	-3.69E+02	1.59E+03	0.00E+00	1.1		MBV	1	106
200016831	AUF	300031618	R025	1	1	1	1	1	1	1	1	100	172371	R025	-3.69E+02	1.59E+03	0.00E+00	1.1		MBV	1	108
200016832	AUF	300031614	R026	1	1	1	1	1	1	1	1	100	174106	R026	-3.69E+02	1.59E+03	0.00E+00	1.1		MBV	1	109

Calculation sheets

AE F075736
Surface water (confirmation method)

0.1 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst: BRUEGGEMANN Study ID: CR99/029 Batch: BRU009 SEND LOQ: 0.1 0.3*LOQ 0.03
Submission ID: 100000346 Crop: water mean Blank: 0 STD:
Operation: AE F075736(WASSER/V Sample Mat.: water Analyte: AE F075736 mean Recov: 84 STD: 27.75
Date: 27.01.2000 AL-Method: EM F12/99-0 Determined as: AE F075736 RSD: 43
Filename: BR082325 Factor: 1.00 Calculated as: AE F075736 mean Lstab: 0 STD:

Sample_ID	Type	Task-ID	Lab Code	W	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 ul	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Resid ug/l	< LOQ	spiked Sample	Spiked ug/l	Rec %
200016126	BLI	300032200	U017	1	1	1	1	1	1	1	1	100	0	U017	4.32E+03	1.65E+03	0.00E+00	0.000	n.d.			
200016983	AUF	300032194	R037	1	1	1	1	1	1	1	1	100	19134	R037	4.32E+03	1.65E+03	0.00E+00	0.090		200016126	0.1	90
200016984	AUF	300032196	R038	1	1	1	1	1	1	1	1	100	15440	R038	4.32E+03	1.65E+03	0.00E+00	0.067		200016126	0.1	67
200016985	AUF	300032198	R039	1	1	1	1	1	1	1	1	100	10033	R039	4.32E+03	1.65E+03	0.00E+00	0.035		200016126	0.1	35

Calculation sheets

AE F115008
Surface water

0.1 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

| | | |

Calculation sheets

AE F115008
Surface water

1.0 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst: BRUEGGEMANN Study ID: CR99029 Batch: BRU008 SEND LOQ: 0.1 0.3*LOQ 0.03
Submission ID: 10000346 Crop: water mean Blank: 0 STD: 0
Operation: AE F115008(WASSER/W Sample Mat.: water Analyte: AE F115008 mean Recov: 82 STD: 2.67
Date: 25.01.2000 AL-Method: EM F12/99-0 Determined as: AE F115008 RSD: 3
Filename: BR080935 Factor: 1.00 Calculated as: AE F115008 mean Lstab: 0 STD:

Sample_ID	Type	Task-ID	Lab Code	W	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 µl	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Resid µg/l	< LOQ	spiked Sample	Spiked µg/l	Rec %
200016124	BLI	300032116	U015	1	1	1	1	1	1	1	1	100	0	U015	1.36E+03	1.53E+03	0.00E+00	0.000	n.d.			
200016125	BLI	300032118	U016	1	1	1	1	1	1	1	1	100	0	U016	1.36E+03	1.53E+03	0.00E+00	0.000	n.d.			
200016967	AUF	300032106	R032	1	1	1	1	1	1	1	1	100	137822	R032	1.36E+03	1.53E+03	0.00E+00	0.89		MBV	1	89
200016968	AUF	300032112	R033	1	1	1	1	1	1	1	1	100	138633	R033	1.36E+03	1.53E+03	0.00E+00	0.90		MBV	1	90
200016969	AUF	300032108	R034	1	1	1	1	1	1	1	1	100	139105	R034	1.36E+03	1.53E+03	0.00E+00	0.90		MBV	1	90
200016970	AUF	300032114	R035	1	1	1	1	1	1	1	1	100	145513	R035	1.36E+03	1.53E+03	0.00E+00	0.94		MBV	1	94
200016971	AUF	300032110	R036	1	1	1	1	1	1	1	1	100	146342	R036	1.36E+03	1.53E+03	0.00E+00	0.95		MBV	1	95

Calculation sheets

AE F115008
Surface water (confirmation method)

0.1 µg/L

Calculation of Calibration Curve

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst:	BRUEGGEMANN	Study ID:	CR99/029	Batch:	BRU005 SEND	LOQ: 0.1		0.3*LOQ	0.03
Submission ID:	100000346	Crop:	water			mean Blank:	0	STD:	0
Operation:	AE F115008(WASSER/W	Sample Mat.:	water	Analyte:	AE F115008	mean Recov:	96	STD:	28.8
Date:	18.01.2000	AL-Method:	EM F12/99-0	Determined as:	AE F115008			RSD:	30
Filename:	BR095153	Factor:	1.00	Calculated as:	AE F115008	mean Lstab:	0	STD:	

Sample_ID	Type	Task-ID	Lab Code	W l	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 ul	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Resid ug/l	< LOQ	spiked Sample	Spiked ug/l	Rec %
200016118	BLI	300031469	U009	1	1	1	1	1	1	1	1	100	0	U009	1.32E+03	1.77E+03	0.00E+00	0.000	n.d.			
200016119	BLI	300031471	U010	1	1	1	1	1	1	1	1	100	0	U010	1.32E+03	1.77E+03	0.00E+00	0.000	n.d.			
200016484	AUF	300031459	R017	1	1	1	1	1	1	1	1	100	14508	R017	1.32E+03	1.77E+03	0.00E+00	0.075		MBV	0.1	75
200016485	AUF	300031465	R018	1	1	1	1	1	1	1	1	100	15150	R018	1.32E+03	1.77E+03	0.00E+00	0.078		MBV	0.1	78
200016486	AUF	300031461	R019	1	1	1	1	1	1	1	1	100	14341	R019	1.32E+03	1.77E+03	0.00E+00	0.074		MBV	0.1	74
200016487	AUF	300031463	R020	1	1	1	1	1	1	1	1	100	21408	R020	1.32E+03	1.77E+03	0.00E+00	0.11		MBV	0.1	114
200016488	AUF	300031467	R021	1	1	1	1	1	1	1	1	100	25649	R021	1.32E+03	1.77E+03	0.00E+00	0.14		MBV	0.1	138

Calculation sheets

AE F115008
Surface water (confirmation method)

1.0 µg/L

Calculation of Calibration Curve.

Auswertung(Eichkurve: Counts=A+B*Conc+C*Conc*Conc)

Analyst: BRUEGGEMANN Study ID: CR99029 Batch: BRU006 SEND LOQ: 0.1 0.3*LOQ 0.03
Submission ID: 10000346 Crop: water mean Blank: 0 STD: 0
Operation: AE F115008/WASSER/W Sample Mat.: water Analyte: AE F115008 meen Recov: 96 STD: 2.46
Date: 20.01.2000 AL-Method: EM F12/99-0 Determined as: AE F115008 mean Lsab: 0 RSD: 3
Filename: BR094630 Factor: 1.00 Calculated as: AE F115008 STD:

Sample_ID	Type	Task-ID	Lab Code	W l	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 µl	Sample Counts	File	A counts	B counts/ng	C counts/ng*ng	Resid ug/l	< LOQ	spiked Sample	Spiked ug/l	Rec %
200016120	BLI	300031619	U011	1	1	1	1	1	1	1	1	100	0	U011	-6.43E+02	1.76E+03	0.00E+00	0.000	n.d.			
200016121	BLI	300031621	U012	1	1	1	1	1	1	1	1	100	0	U012	-6.43E+02	1.76E+03	0.00E+00	0.000	n.d.			
200016828	AUF	300031609	R022	1	1	1	1	1	1	1	1	100	160784	R022	-6.43E+02	1.76E+03	0.00E+00	0.92		MBV	1	92
200016829	AUF	300031615	R023	1	1	1	1	1	1	1	1	100	169113	R023	-6.43E+02	1.76E+03	0.00E+00	0.97		MBV	1	97
200016830	AUF	300031611	R024	1	1	1	1	1	1	1	1	100	168966	R024	-6.43E+02	1.76E+03	0.00E+00	0.97		MBV	1	97
200016831	AUF	300031617	R025	1	1	1	1	1	1	1	1	100	169945	R025	-6.43E+02	1.76E+03	0.00E+00	0.97		MBV	1	97
200016832	AUF	300031613	R026	1	1	1	1	1	1	1	1	100	172108	R026	-6.43E+02	1.76E+03	0.00E+00	0.98		MBV	1	98

Calculation sheets

AE F115008
Surface water (confirmation method)

0.1 µg/L

Analyst:		Weisel	Study ID:	CR99/029	Batch:	BRU009	LOD:	0.1	0.3*LOQ	0.03			
Submission ID:	100000346		Crop:	water			mean Blank:	0	STD:				
Operation:			Commodity:	water	Analyte:	AE F115008	mean Recov:	69	STD:	31.23			
Date:	27.1.2000		AL-Method:	EM F12/99-0	Determined as:	AE F115008			RSD:	45			
Filename:			Factor:	1.00	Calculated as:	AE F115008	mean Lstab:	0	STD:				

Lab Code	Spiked Code	W g	V1 ml	T1 ml	V2 ml	T2 ml	V3 ml	T3 ml	V4 ml	T4 ul	Sample Counts	Channel/ Sequenz	A	B	C	Residue mg/kg	< LOD	Spiked mg/kg	Rec %
U017			1	1	1	1	1	1	1	100	0	U017	3895.1481	1810.746	0	0.000	n.d.		
R037	U017		1	1	1	1	1	1	1	100	21588	R037	3895.1481	1810.746	0	0.098		0.100	98
R038	U017		1	1	1	1	1	1	1	100	17120	R038	3895.1481	1810.746	0	0.073		0.100	73
R039	U017		1	1	1	1	1	1	1	100	10357	R039	3895.1481	1810.746	0	0.036		0.100	36

Annex IV: Sample history and recovery efficiencies

Analyte: AE F075736

Matrix	Lab code	Start of sample work-up	Date of data acquisition	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
Drinking water (Vittel)	R001	22.11.1999	23.11.1999	0.10	82			
	R002	22.11.1999	23.11.1999	0.10	75			
	R003	22.11.1999	23.11.1999	0.10	87			
	R004	22.11.1999	23.11.1999	0.10	83			
	R005	22.11.1999	23.11.1999	0.10	70	79	9	5
	R009	06.12.1999	08.12.1999	1.0	96			
	R010	06.12.1999	08.12.1999	1.0	91			
	R011	06.12.1999	08.12.1999	1.0	87			
	R012	06.12.1999	08.12.1999	1.0	93			
	R013	06.12.1999	08.12.1999	1.0	93	92	4	5

The calculation was done with calibration function of peak areas.

a) RSD = S.D. / Mean Recovery • 100 %

$$S.D. = \left[\frac{\Sigma (R_i - R_m)^2}{n - 1}\right]^{1/2}$$

R_i: recovery
R_m: mean recovery
n: number of recoveries

Analyte: AE F115008

Matrix	Lab code	Start of sample work-up	Date of data acquisition	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
Surface water	R027	20.01.2000	21.01.2000	0.1	108			
	R028	20.01.2000	21.01.2000	0.1	92			
	R029	20.01.2000	21.01.2000	0.1	94			
	R030	20.01.2000	22.01.2000	0.1	106			
	R031	20.01.2000	22.01.2000	0.1	113	103	9	5
	R032	24.01.2000	24.01.2000	1.0	89			
	R033	24.01.2000	25.01.2000	1.0	90			
	R034	24.01.2000	25.01.2000	1.0	90			
	R035	24.01.2000	25.01.2000	1.0	94			
	R036	24.01.2000	25.01.2000	1.0	95	92	3	5

The calculation was done with calibration function of peak areas.

a) RSD = S.D. / Mean Recovery • 100 %

$$S.D. = \left[\frac{\Sigma (R_i - R_m)^2}{n - 1}\right]^{1/2}$$

R_i: recovery
R_m: mean recovery
n: number of recoveries

Analyte: AE F075736

Matrix	Lab code	Start of sample work-up	Date of data acquisition	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
Surface water	R027	20.01.2000	21.01.2000	0.1	90			
	R028	20.01.2000	21.01.2000	0.1	78			
	R029	20.01.2000	21.01.2000	0.1	91			
	R030	20.01.2000	22.01.2000	0.1	92			
	R031	20.01.2000	22.01.2000	0.1	74	85	10	5
	R032	24.01.2000	24.01.2000	1.0	98			
	R033	24.01.2000	25.01.2000	1.0	102			
	R034	24.01.2000	25.01.2000	1.0	105			
	R035	24.01.2000	25.01.2000	1.0	109			
	R036	24.01.2000	25.01.2000	1.0	109	105	5	5

The calculation was done with calibration function of peak areas.

a) RSD = S.D. / Mean Recovery • 100 %

$$S.D. = \left[\frac{\Sigma (R_i - R_m)^2}{n - 1}\right]^{1/2}$$

R_i: recovery
R_m: mean recovery
n: number of recoveries

Analyte: AE F115008

Matrix	Lab code	Start of sample work-up	Date of data acquisition	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
Surface water Confirmation method	R017	17.01.2000	17.01.2000	0.1	75			
	R018	17.01.2000	17.01.2000	0.1	78			
	R019	17.01.2000	18.01.2000	0.1	74			
	R020	17.01.2000	18.01.2000	0.1	114			
	R021	17.01.2000	18.01.2000	0.1	138 (b)			
	R037	25.01.2000	25.01.2000	0.1	98			
	R038	25.01.2000	25.01.2000	0.1	73			
	R039	25.01.2000	25.01.2000	0.1	36 (c)	85	20	6
	R022	19.01.2000	19.01.2000	1.0	92			
	R023	19.01.2000	19.01.2000	1.0	97			
	R024	19.01.2000	20.01.2000	1.0	97			
	R025	19.01.2000	20.01.2000	1.0	97			
	R026	19.01.2000	20.01.2000	1.0	98	96	2	5

The calculation was done with calibration function of peak areas.

a) RSD = S.D. / Mean Recovery • 100 %

$$S.D. = \left[\frac{\Sigma (R_i - R_m)^2}{n - 1}\right]^{1/2}$$

R_i: recovery
R_m: mean recovery
n: number of recoveries

b) Outliner Unexpected high value (results including this value: mean recovery = 93 %, RSD = 27 %, n = 7)

c) Outliner Injection problems, HPLC system used to much solution for washing, so the injection volume was too small

Analyte: AE F075736

Matrix	Lab code	Start of sample work-up	Date of data acquisition	Fortification level [µg/L]	Recovery [%]	Mean recovery [%]	RSD (a) [%]	n
Surface water Confirmation method	R017	17.01.2000	17.01.2000	0.1	74			
	R018	17.01.2000	17.01.2000	0.1	110			
	R019	17.01.2000	18.01.2000	0.1	92			
	R020	17.01.2000	18.01.2000	0.1	106			
	R021	17.01.2000	18.01.2000	0.1	108			
	R037	25.01.2000	25.01.2000	0.1	90			
	R038	25.01.2000	25.01.2000	0.1	67			
	R039	25.01.2000	25.01.2000	0.1	35 (b)	92	18	7
	R022	19.01.2000	19.01.2000	1.0	102			
	R023	19.01.2000	19.01.2000	1.0	106			
	R024	19.01.2000	20.01.2000	1.0	106			
	R025	19.01.2000	20.01.2000	1.0	108			
	R026	19.01.2000	20.01.2000	1.0	109	106	3	5

The calculation was done with calibration function of peak areas.

a) RSD = S.D. / Mean Recovery • 100 %

$$S.D. = \left[\frac{\Sigma (R_i - R_m)^2}{n - 1}\right]^{1/2}$$

R_i: recovery
R_m: mean recovery
n: number of recoveries

b) Outliner Injection problems, HPLC system used to much solution for washing, so the injection volume was too small

Annex V: Sample history and apparent residue levels

Analyte: AE F075736

Matrix	Lab code	Start of sample work-up	Date of data acquisition	Apparent residue level AE F075736 [µg/L]	Mean apparent residue level AE F075736 [µg/L]
Drinking water (Vittel)	U001	22.11.1999	22.11.1999	0.000	
	U002	22.11.1999	22.11.1999	0.021	n.d.
	U005	06.12.1999	08.12.1999	0.019	
	U006	06.12.1999	08.12.1999	0.018	n.d.
Surface water	U013	20.01.2000	21.01.2000	0.000	
	U014	20.01.2000	21.01.2000	0.000	n.d.
	U015	24.01.2000	24.01.2000	0.000	
	U016	24.01.2000	24.01.2000	0.000	n.d.
Surface water Confirmation method	U009	17.01.2000	17.01.2000	0.000	
	U010	17.01.2000	17.01.2000	0.009	n.d.
	U011	19.01.2000	19.01.2000	0.017	
	U012	19.01.2000	19.01.2000	0.000	n.d.
	U017	25.01.2000	25.01.2000	0.000	n.d.

Analyte: AE F115008

Matrix	Lab code	Start of sample work-up	Date of data acquisition	Apparent residue level AE F115008 [µg/L]	Mean apparent residue level AE F115008 [µg/L]
Surface water	U013	20.01.2000	21.01.2000	0.000	
	U014	20.01.2000	21.01.2000	0.000	n.d.
	U015	24.01.2000	24.01.2000	0.000	
	U016	24.01.2000	24.01.2000	0.000	n.d.
Surface water Confirmation method	U009	17.01.2000	17.01.2000	0.000	
	U010	17.01.2000	17.01.2000	0.000	n.d
	U011	19.01.2000	19.01.2000	0.000	
	U012	19.01.2000	19.01.2000	0.000	n.d
	U017	25.01.2000	25.01.2000	0.000	n.d.

Annex VI: Typical chromatograms

The following chromatograms are given as examples of characteristic recorder plots.
The retention time of the target signal varied to a certain extent as the chromatographic system had to be fitted to the particular situation.
The computer plot offers the possibility of presenting only the important cut of the chromatogram with additional stretching of time or height. The electronic plots show the peak height in counts plotted vs. the retention time in minutes.

Sample material	Lab code	Description		Page
–	E001	Test	0.10 µg AE F075736/mL	61
	E002	Test	0.20 µg AE F075736/mL	62
	E003	Test	0.50 µg AE F075736/mL	63
	E004	Test	1.0 µg AE F075736/mL	64
Drinking water (Vittel)	U001	Control	–	65
	R001	Recovery	0.10 µg AE F075736/L	66
	R002	Recovery	0.10 µg AE F075736/L	67
	U005	Control	–	68
	R009	Recovery	1.0 µg AE F075736/L	69
	R010	Recovery	1.0 µg AE F075736/L	70
–	E054	Test	0.10 µg (AE F115008 + AE F075736)/mL	71
	E055	Test	0.20 µg (AE F115008 + AE F075736)/mL	72
	E056	Test	0.50 µg (AE F115008 + AE F075736)/mL	73
	E057	Test	1.0 µg (AE F115008 + AE F075736)/mL	74
Surface water	U013	Control	–	75
	R027	Recovery	0.10 µg (AE F115008 + AE F075736)/L	76
	R028	Recovery	0.10 µg (AE F115008 + AE F075736)/L	77
	U015	Control	–	78
	R032	Recovery	1.0 µg (AE F115008 + AE F075736)/L	79
	R033	Recovery	1.0 µg (AE F115008 + AE F075736)/L	80
–	E038	Test	0.10 µg (AE F115008 + AE F075736)/mL	81
(Confirmation method)	E041	Test	1.0 µg (AE F115008 + AE F075736)/mL	82
Surface water	U009	Control	–	83
(Confirmation method)	R017	Recovery	0.10 µg (AE F115008 + AE F075736)/L	84
	R018	Recovery	0.10 µg (AE F115008 + AE F075736)/L	85

Lab code **Description**

E001 Test 0.10 µg AE F075736/mL

Software Version	: 6.1.0.2:G07	Date	: 22.11.1999 13:58:33
Operator	: Brueggemann	Sample Name	: E001
Sample Number	: 1	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 22.11.1999 13:08:46	Cycle	: 1

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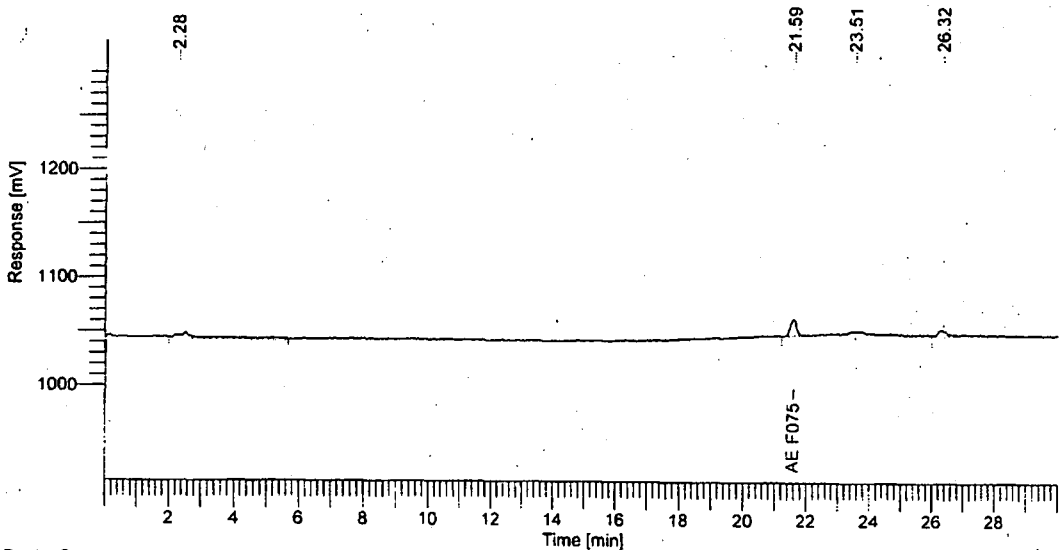
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru001.seq
Sample Notes:
10 ng AE F075736 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.589	211310	15087

Lab code	Description	
E002	Test	0.20 µg AE F075736/mL

Software Version	: 6.1.0.2:G07	Date	: 22.11.1999 15:39:02
Operator	: Brueggemann	Sample Name	: E002
Sample Number	: 3	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 22.11.1999 15:04:01	Cycle	: 3

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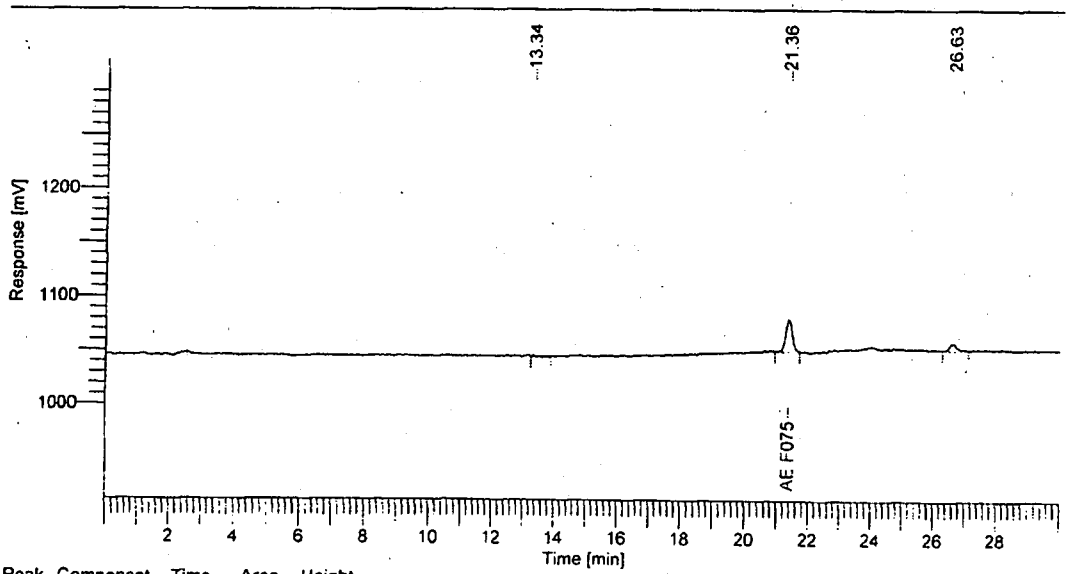
Sample Notes:
20 ng AE F075736 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.359	420875	30083

Lab code **Description**

E003 Test 0.50 µg AE F075736/mL

Software Version	: 6.1.0.2:G07	Date	: 23.11.1999 08:00:10
Operator	: Brueggemann	Sample Name	: E003
Sample Number	: 5	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 22.11.1999 16:59:17	Cycle	: 5

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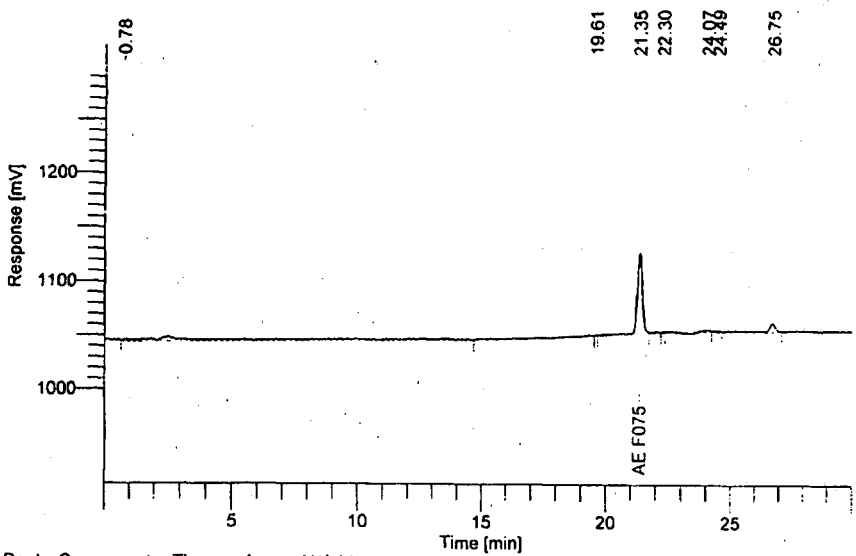
Sample Notes:
50 ng AE F075736 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
3	AE F075736	21.352	995790	74944

Lab code	Description	
E004	Test	1.0 µg AE F075736/mL

Software Version	: 6.1.0.2:G07	Date	: 23.11.1999 08:01:43
Operator	: Brueggemann	Sample Name	: E004
Sample Number	: 7	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 22.11.1999 18:54:36	Cycle	: 7

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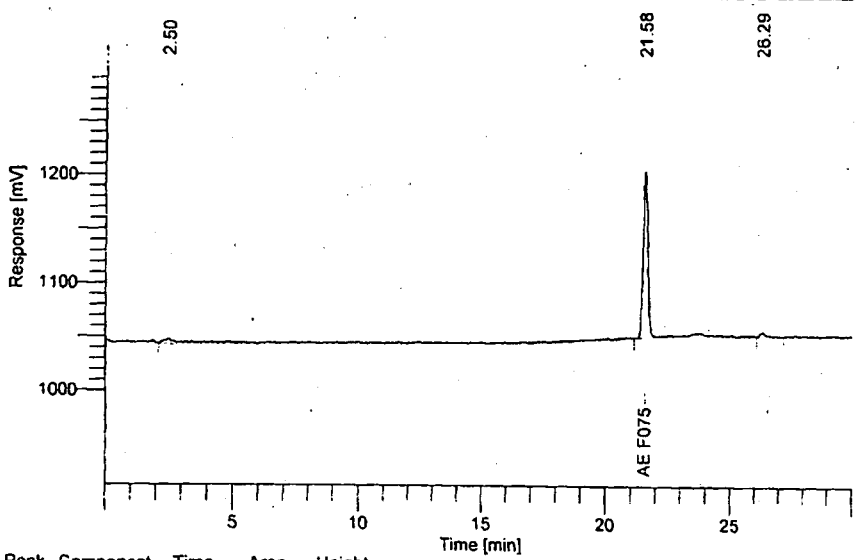
Sample Notes:
100 ng AE F075736 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.575	2005100	153777

Sample material	Lab code	Description
Drinking water (Vittel)	U001	Control –

Software Version	: 6.1.0.2:G07	Date	: 23.11.1999 08:44:00
Operator	: Brueggemann	Sample Name	: U001
Sample Number	: 2	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 22.11.1999 14:06:23	Cycle	: 2

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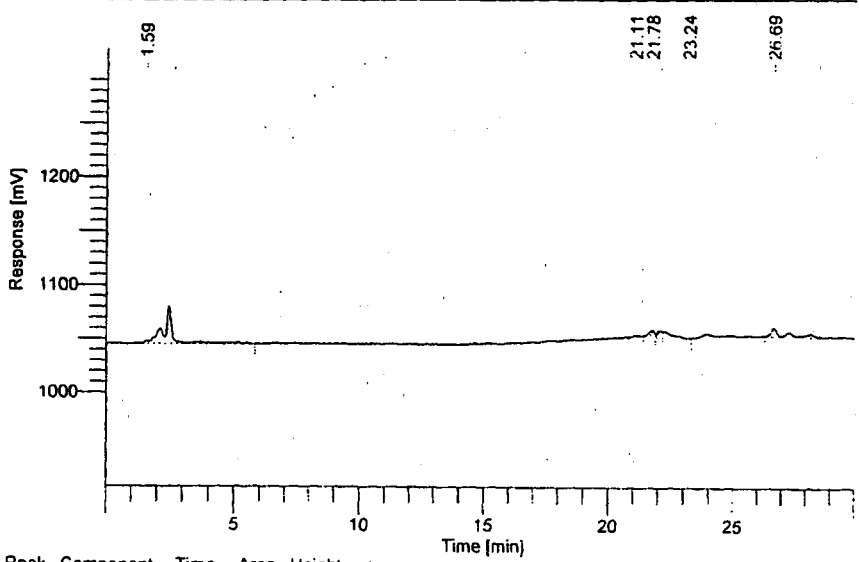
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
-	AE F075736	21.500	0	0

Sample material	Lab code	Description
Drinking water (Vittel)	R001	Recovery 0.10 µg AE F075736/L

Software Version	: 6.1.0.2:G07	Date	: 23.11.1999 08:01:17
Operator	: Brueggemann	Sample Name	: R001
Sample Number	: 6	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 22.11.1999 17:56:56	Cycle	: 6

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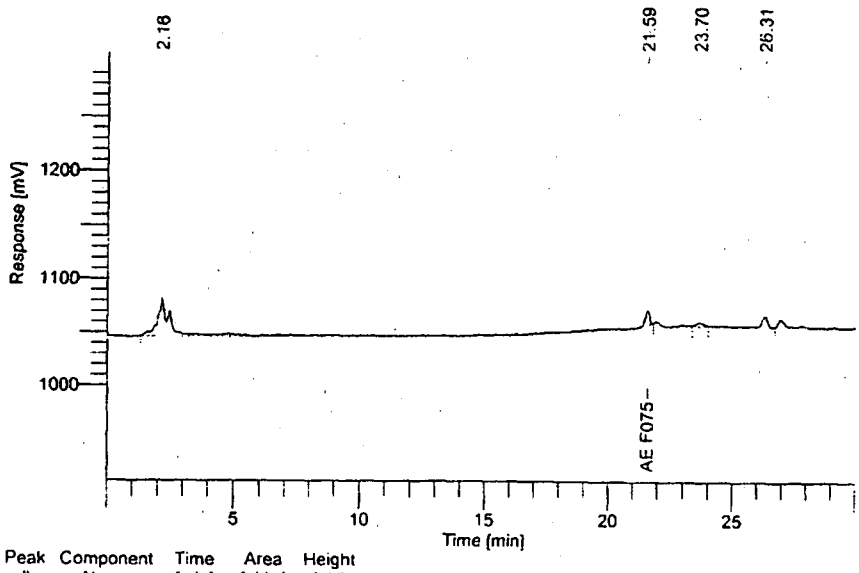
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.587	170540	13841

Sample material	Lab code	Description
Drinking water (Vittel)	R002	Recovery 0.10 µg AE F075736/L

Software Version	: 6.1.0.2:G07	Date	: 23.11.1999 08:03:17
Operator	: Brueggemann	Sample Name	: R002
Sample Number	: 8	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 22.11.1999 19:52:16	Cycle	: 8

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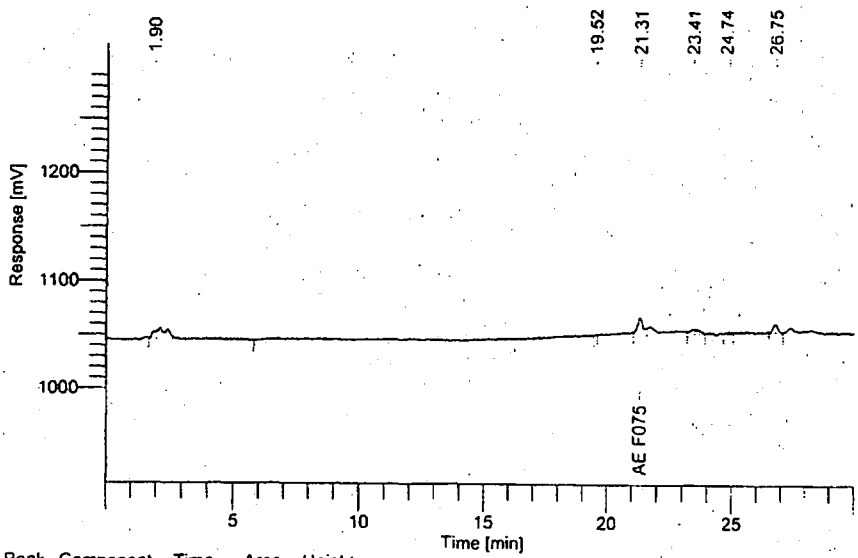
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru001.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
3	AE F075736	21.313	138420	12673

Sample material	Lab code	Description
Drinking water (Vittel)	U005	Control --

Software Version	: 6.1.0.2:G07	Date	: 08.12.1999 14:14:58
Operator	: Brueggemann	Sample Name	: U005
Sample Number	: 2	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 08.12.1999 09:42:55	Cycle	: 2

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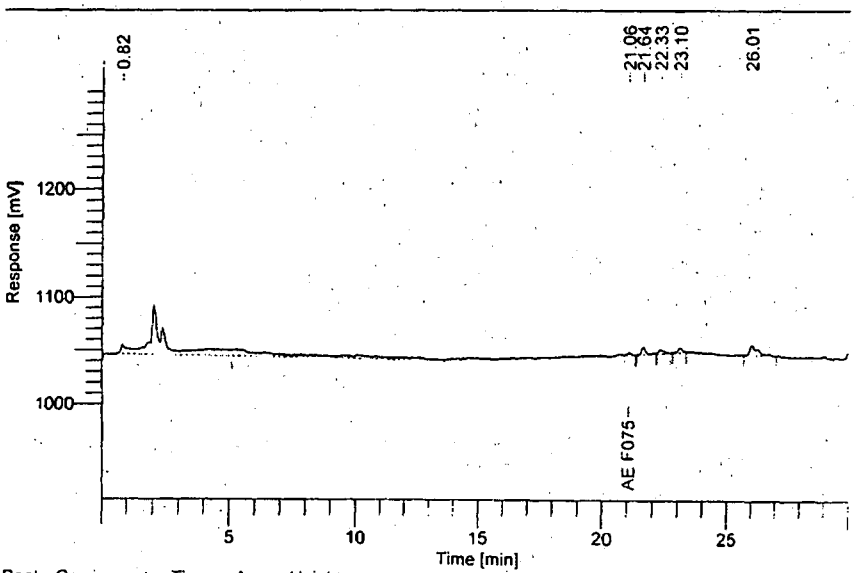
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru003.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.065	38485	2869

Sample material	Lab code	Description	
Drinking water (Vittel)	R009	Recovery	1.0 µg AE F075736/L

Software Version	: 6.1.0.2:G07	Date	: 08.12.1999 14:17:04
Operator	: Brueggemann	Sample Name	: R009
Sample Number	: 6	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 08.12.1999 13:33:00	Cycle	: 6

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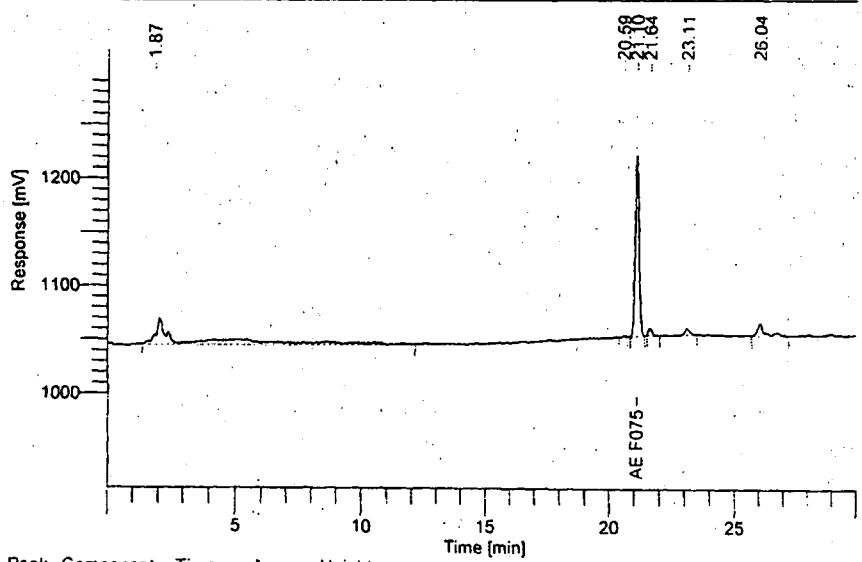
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru003.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
3	AE F075736	21.096	1829750	168915

Sample material	Lab code	Description
Drinking water (Vittel)	R010	Recovery 1.0 µg AE F075736/L

Software Version	: 6.1.0.2:G07	Date	: 09.12.1999 10:26:13
Operator	: Brueggemann	Sample Name	: R010
Sample Number	: 8	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 08.12.1999 15:28:06	Cycle	: 8

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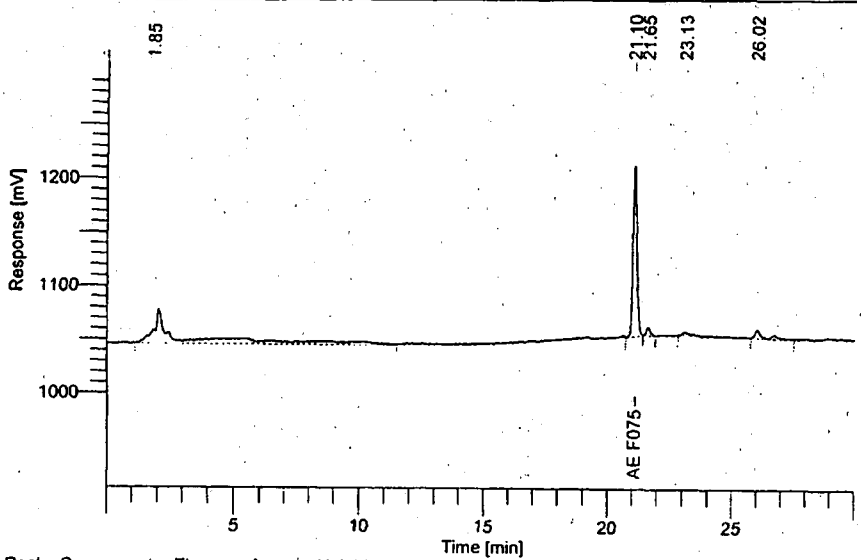
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru003.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.096	1744620	159862

Lab code	Description	
E054	Test	0.10 µg (AE F115008 + AE F075736)/mL

Software Version	: 6.1.0.2:G07	Date	: 21.01.2000 15:05:50
Operator	: Brueggemann	Sample Name	: E054
Sample Number	: 1	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 21.01.2000 14:24:55	Cycle	: 1

Raw Data File : \\adeft003\Turbochrom Daten\Brueggemann\Chromatogramme\CR99029\bru007\E054.raw
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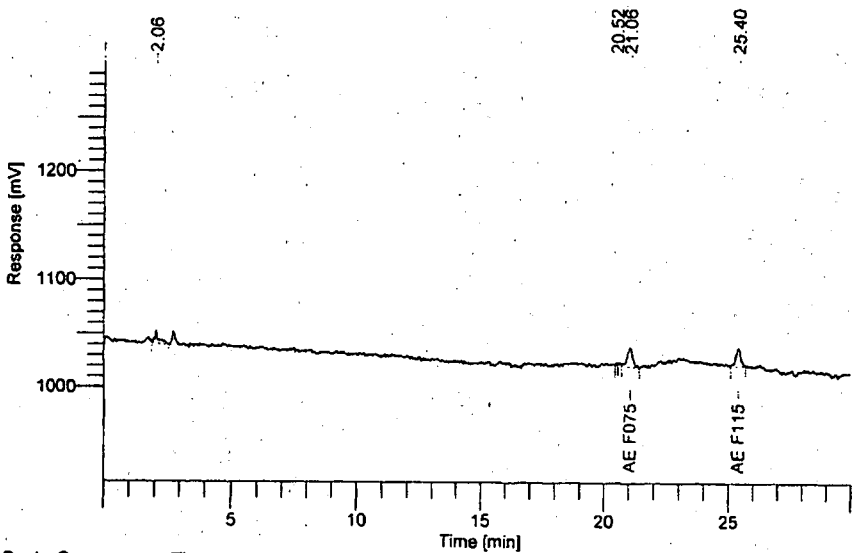
Calib Method : \\adeft003\Turbochrom Daten\brueggemann\methoden\115008emf1299 from \\Adeft003\turbochrom daten\Brueggemann\Chromatogramme\CR99029\bru007\E054.rst
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru007.seq
Sample Notes:
10 ng AE F075736, 10 ng AE F115008 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
3	AE F075736	21.062	291500	18536
4	AE F115008	25.403	251150	17265

Lab code	Description	
E055	Test	0.20 µg (AE F115008 + AE F075736)/mL

Software Version	: 6.1.0.2:G07	Date	: 24.01.2000 07:47:54
Operator	: Brueggemann	Sample Name	: E055
Sample Number	: 3	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 21.01.2000 16:20:00	Cycle	: 3

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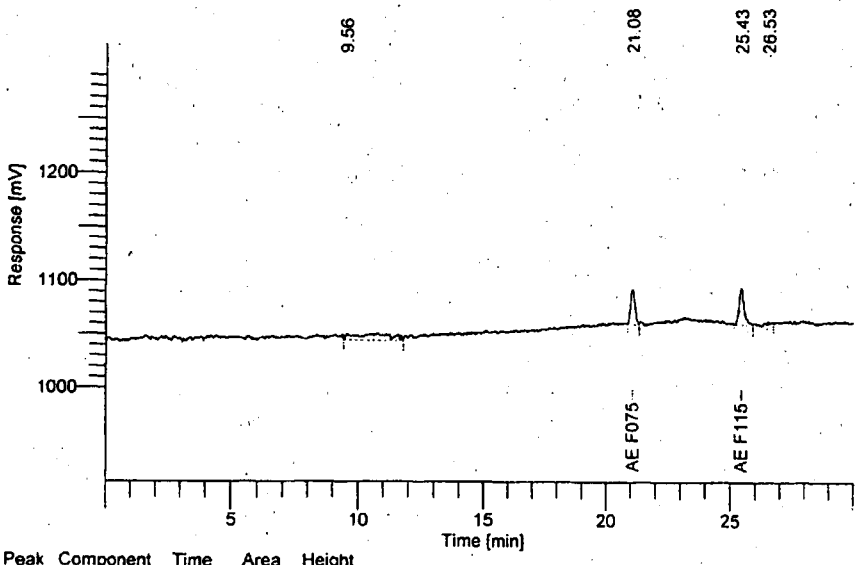
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru007.seq
Sample Notes:
20 ng AE F075736, 20 ng AE F115008 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.080	412925	32959
3	AE F115008	25.431	482305	34416

Lab code	Description	
E056	Test	0.50 µg (AE F115008 + AE F075736)/mL

Software Version	: 6.1.0.2:G07	Date	: 24.01.2000 07:48:57
Operator	: Brueggemann	Sample Name	: E056
Sample Number	: 5	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 21.01.2000 18:15:05	Cycle	: 5

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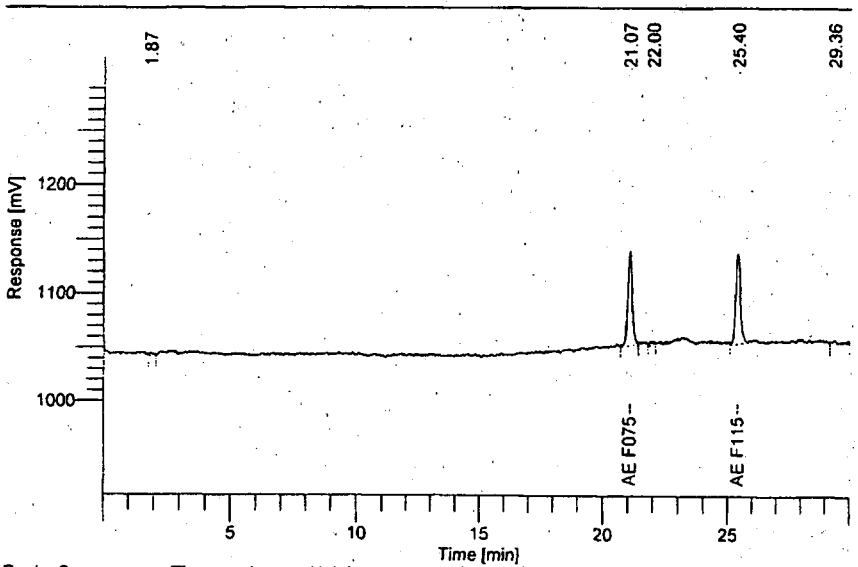
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru007.seq
Sample Notes:
50 ng AE F075736, 50 ng AE F115008 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.070	1050150	88301
4	AE F115008	25.405	1161025	84508

Lab code **Description**

E057 Test 1.0 µg (AE F115008 + AE F075736)/mL

Software Version	: 6.1.0.2:G07	Date	: 24.01.2000 07:51:02
Operator	: Brueggemann	Sample Name	: E057
Sample Number	: 7	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 21.01.2000 20:10:09	Cycle	: 7

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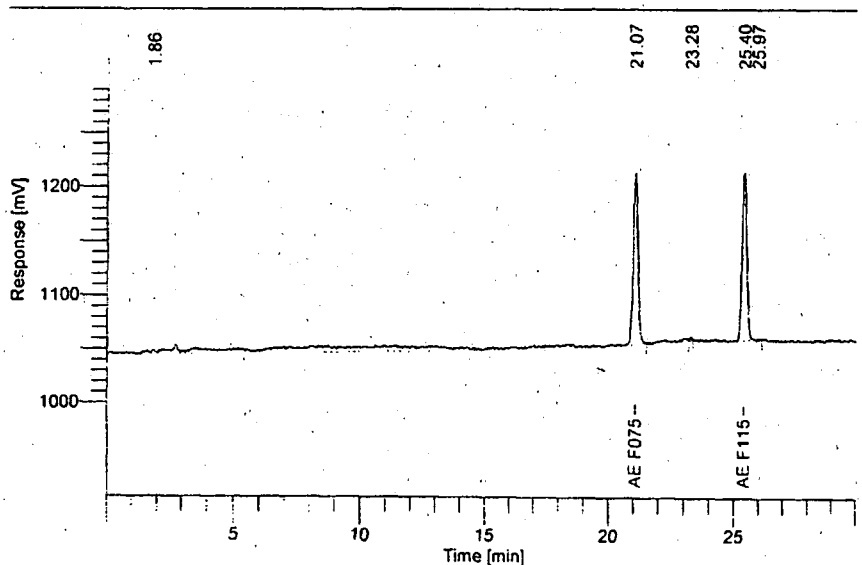
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru007.seq
Sample Notes:
100 ng AE F075736, 100 ng AE F115008 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.067	2091435	159773
4	AE F115008	25.397	2136590	155863

Sample material	Lab code	Description
Surface water	U013	Control

Software Version	: 6.1.0.2:G07	Date	: 24.01.00 08:02:55
Operator	: Brueggemann	Sample Name	: U013
Sample Number	: 2	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0,00 min	End Time	: 30,00 min
Sampling Rate	: 1,0000 pts/s		
Volume Injected	: 100,000000 µL	Area Reject	: 0,000000
Sample Amount	: 1,0000	Dilution Factor	: 1,00
Data Acquisition Time	: 21.01.00 15:22:27	Cycle	: 2

Raw Data File : \\adeft003\Turbochrom Daten\Brueggemann\Chromatogramme\CR99029\bru007\U013.raw
Result File : \\adeft003\Turbochrom Daten\Brueggemann\Chromatogramme\CR99029\bru007\U013.rst
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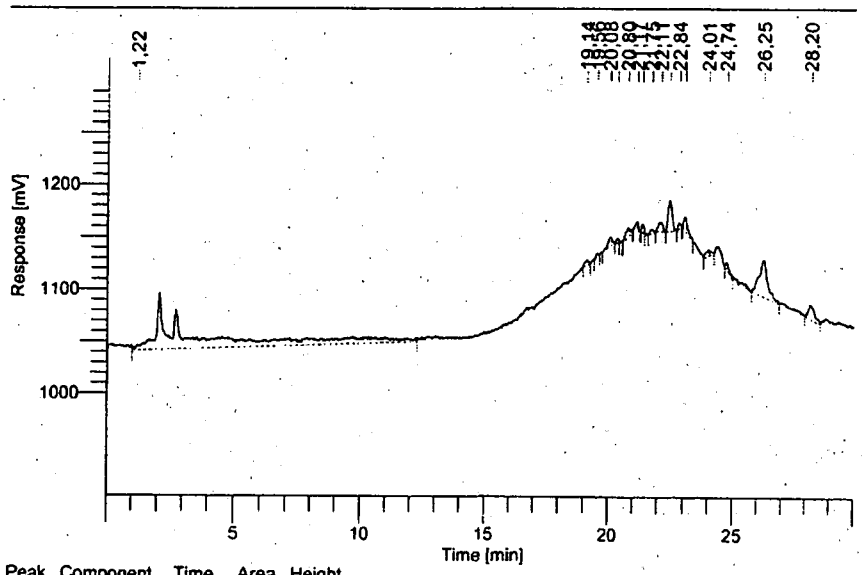
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
-	AE F075736	21,070	0	0
-	AE F115008	25,400	0	0

Sample material	Lab code	Description	
Surface water	R027	Recovery	0.10 µg (AE F115008 + AE F075736)/L

Software Version	: 6.1.0.2:G07	Date	: 24.01.2000 07:50:39
Operator	: Brueggemann	Sample Name	: R027
Sample Number	: 6	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 21.01.2000 19:12:38	Cycle	: 6

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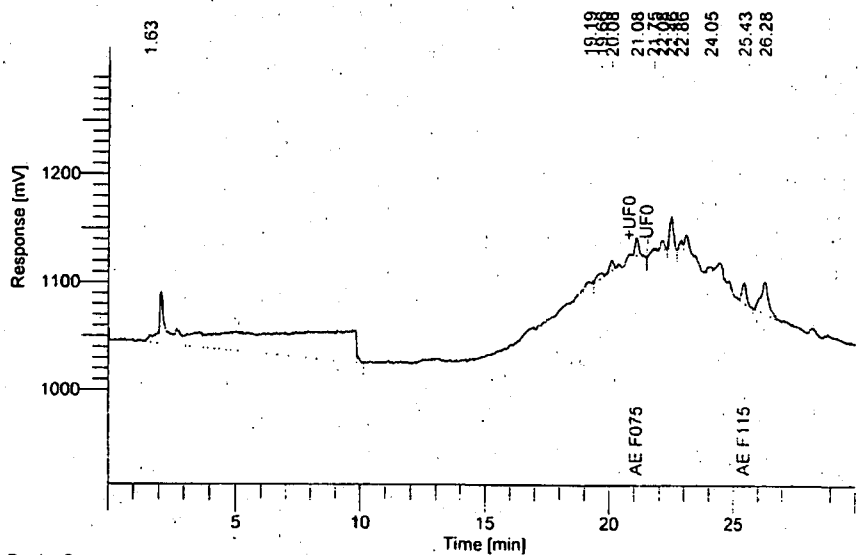
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru007.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
5	AE F075736	21.083	187581	17441
12	AE F115008	25.429	256350	19968

Sample material	Lab code	Description	
Surface water	R028	Recovery	0.10 µg (AE F115008 + AE F075736)/L

Software Version	: 6.1.0.2:G07	Date	: 24.01.2000 07:51:28
Operator	: Brueggemann	Sample Name	: R028
Sample Number	: 8	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 21.01.2000 21:07:41	Cycle	: 8

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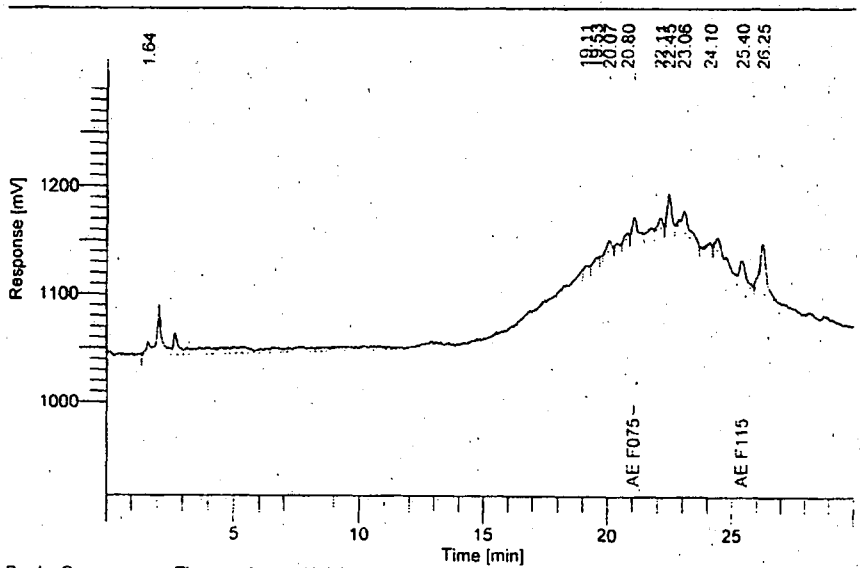
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru007.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
6	AE F075736	21.066	169860	15474
12	AE F115008	25.404	230545	17369

Sample material	Lab code	Description	
Surface water	U015	Control	--

Software Version	: 6.1.0.2:G07	Date	: 25.01.00 07:56:33
Operator	: Brueggemann	Sample Name	: U015
Sample Number	: 2	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0,00 min	End Time	: 30,00 min
Sampling Rate	: 1,0000 pts/s		
Volume Injected	: 100,000000 µL	Area Reject	: 0,000000
Sample Amount	: 1,0000	Dilution Factor	: 1,00
Data Acquisition Time	: 24.01.00 18:52:43	Cycle	: 2

Raw Data File : \\adeft003\Turbochrom Daten\Brueggemann\Chromatogramme\CR99029\bru008\U015.raw
Result File : \\adeft003\Turbochrom Daten\Brueggemann\Chromatogramme\CR99029\bru008\U015.rst
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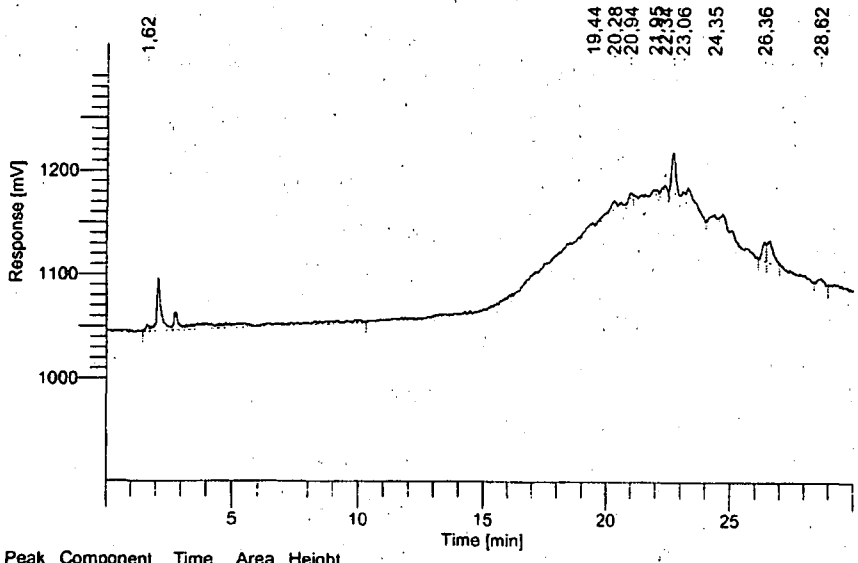
Calib Method : \\adeft003\Turbochrom Daten\Brueggemann\Methoden\115008emf1299.mth
Sequence File : \\adeft003\PenExe\TcCS\Ver6.1.0\Temp\U015-2010099093-20000125-075306.idx
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
-	AE F075736	21,500	0	0
-	AE F115008	25,400	0	0

Sample material	Lab code	Description	
Surface water	R032	Recovery	1.0 µg (AE F115008 + AE F075736)/L

Software Version	: 6.1.0.2:G07	Date	: 25.01.2000 07:37:00
Operator	: Brueggemann	Sample Name	: R032
Sample Number	: 6	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 24.01.2000 22:42:53	Cycle	: 6

Raw Data File : \\adeft003\Turbochrom Daten\Brueggemann\Chromatogramme\CR99029\bru008\R032.raw
Result File : \\Adeft003\turbochrom daten\Brueggemann\Chromatogramme\CR99029\bru008\R032.rst
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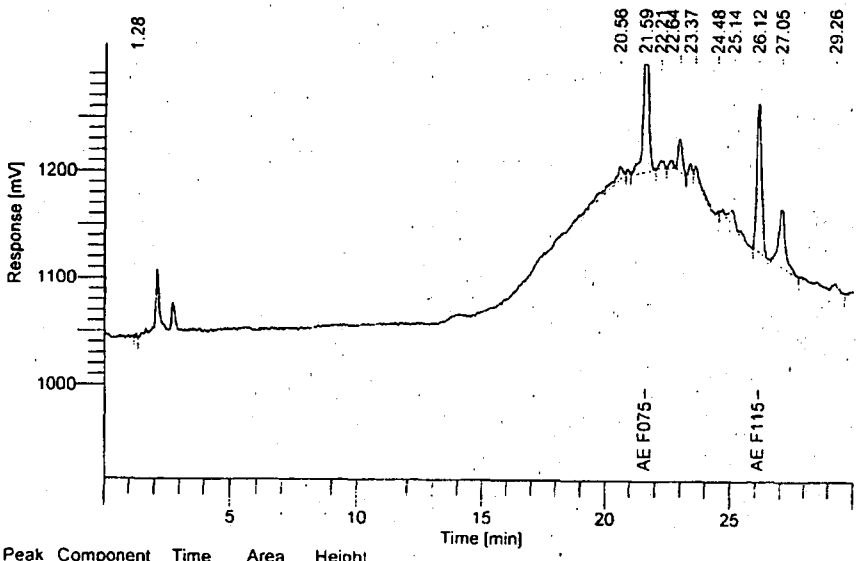
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru008.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
3	AE F075736	21.586	2205040	171221
11	AE F115008	26.118	1897340	137822

Sample material	Lab code	Description	
Surface water	R033	Recovery	1.0 µg (AE F115008 + AE F075736)/L

Software Version	: 6.1.0.2:G07	Date	: 25.01.2000 07:38:17
Operator	: Brueggemann	Sample Name	: R033
Sample Number	: 8	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 25.01.2000 00:37:58	Cycle	: 8

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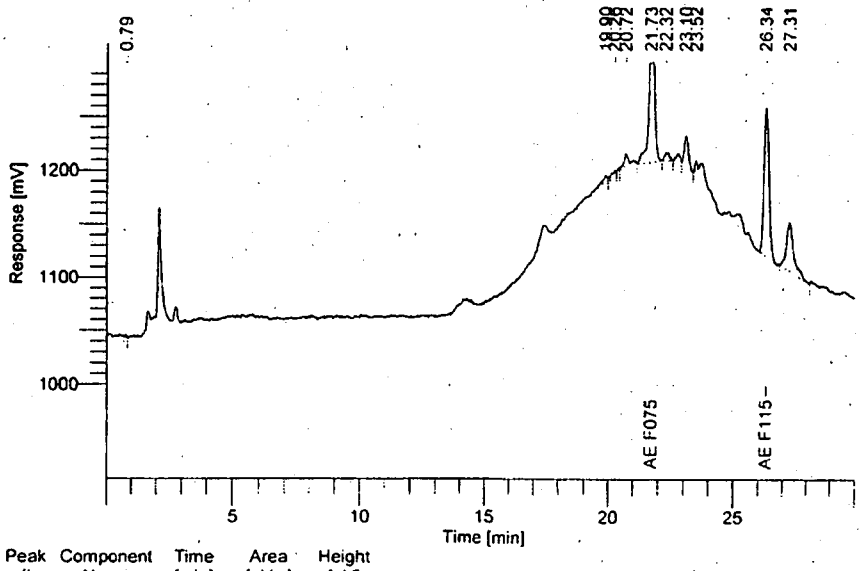
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru008.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
5	AE F075736	21.731	2364410	178003
9	AE F115008	26.344	1973825	138633

Lab code **Description**

E038 Test 0.10 µg (AE F115008 + AE F075736)/mL
Confirmation method

Software Version	: 6.1.0.2:G07	Date	: 18.01.2000 07:39:26
Operator	: Brueggemann	Sample Name	: E038
Sample Number	: 1	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 17.01.2000 16:54:51	Cycle	: 1

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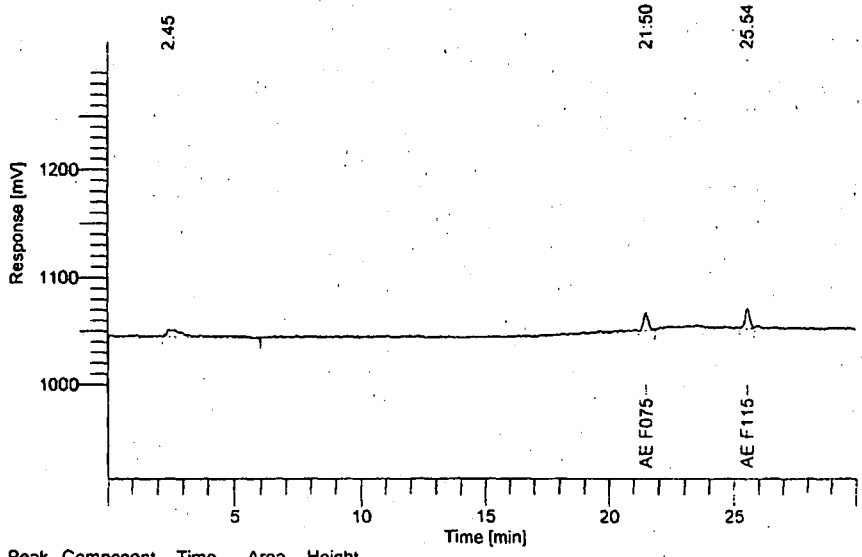
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru005.seq
Sample Notes:
10 ng AE F075736, 10 ng AE F115008 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.503	226270	17428
3	AE F115008	25.543	245910	19350

Lab code **Description**

E041 Test 1.0 µg (AE F115008 + AE F075736)/mL
Confirmation method

Software Version	: 6.1.0.2:G07	Date	: 18.01.2000 07:47:40
Operator	: Brueggemann	Sample Name	: E041
Sample Number	: 7	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 17.01.2000 22:40:09	Cycle	: 7

Raw Data File : \\adeft003\Turbochrom Daten\Brueggemann\Chromatogramme\CR99029\bru005\E041.raw
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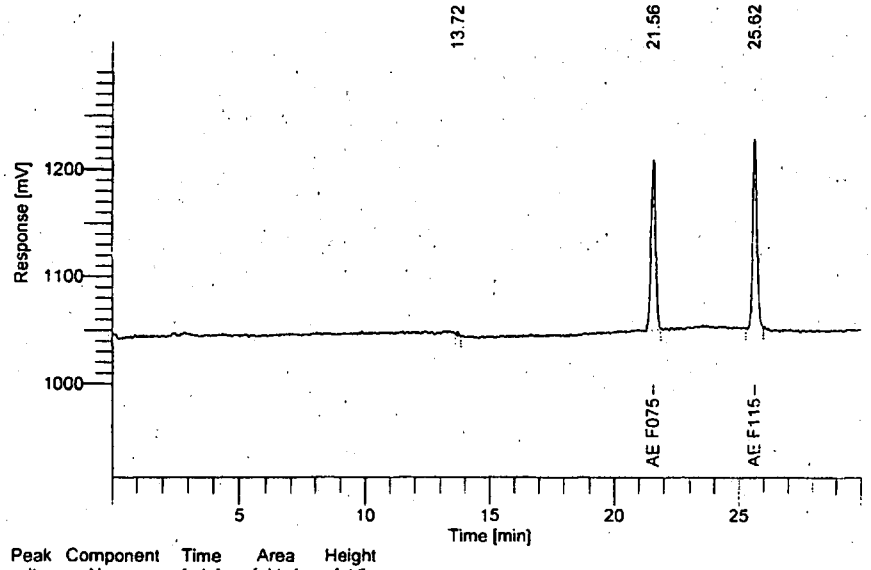
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru005.seq
Sample Notes:
100 ng AE F075736, 100 ng AE F115008 in 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
2	AE F075736	21.562	2029860	159251
3	AE F115008	25.622	2181430	177369

Sample material	Lab code	Description
Surface water	U009	Control

Confirmation method

Software Version	: 6.1.0.2:G07	Date	: 18.01.00 08:13:16
Operator	: Brueggemann	Sample Name	: U009
Sample Number	: 2	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0,00 min	End Time	: 30,00 min
Sampling Rate	: 1,0000 pts/s		
Volume Injected	: 100,000000 µL	Area Reject	: 0,000000
Sample Amount	: 1,0000	Dilution Factor	: 1,00
Data Acquisition Time	: 17.01.00 17:52:25	Cycle	: 2

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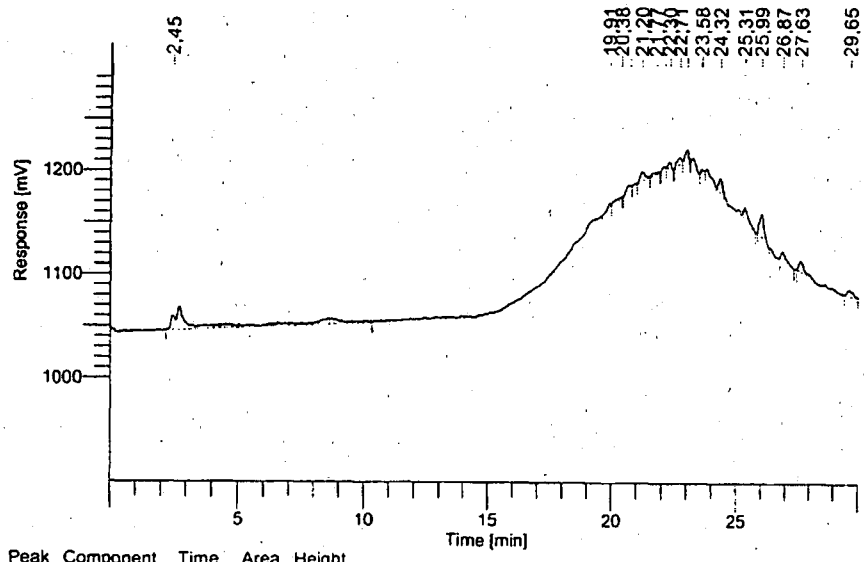
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
-	AE F075736	21,600	0	0
-	AE F115008	25,500	0	0

Sample material	Lab code	Description	
Surface water	R017	Recovery	0.10 µg (AE F115008 + AE F075736)/L
Confirmation method			

Software Version	: 6.1.0.2:G07	Date	: 18.01.2000 08:22:17
Operator	: Brueggemann	Sample Name	: R017
Sample Number	: 6	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 17.01.2000 21:42:36	Cycle	: 6

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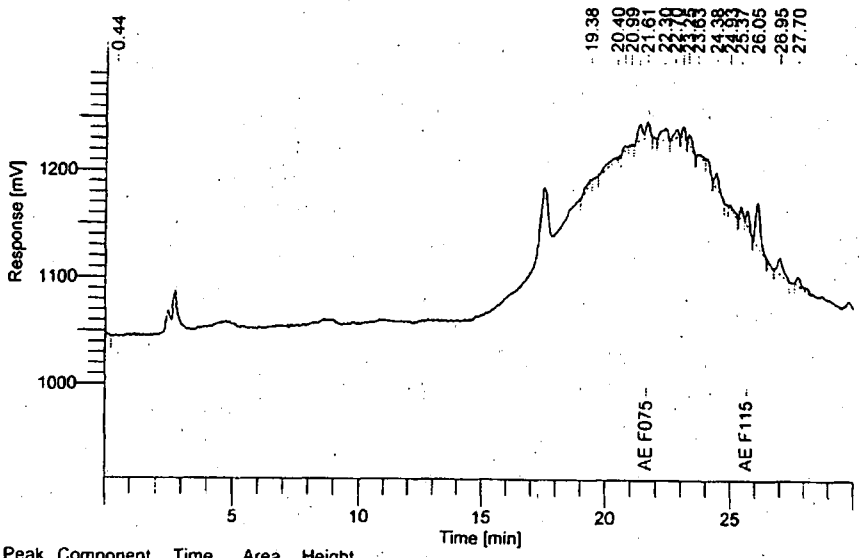
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
7	AE F075736	21.610	127750	13586
16	AE F115008	25.641	142835	14508

Sample material	Lab code	Description	
Surface water	R018	Recovery	0.10 µg (AE F115008 + AE F075736)/L

Confirmation method

Software Version	: 6.1.0.2:G07	Date	: 18.01.2000 07:48:39
Operator	: Brueggemann	Sample Name	: R018
Sample Number	: 8	Study	: CR99/029
AutoSampler	: NONE	Rack/Vial	: 0/0
Instrument Name	: 101	Channel	: A
Interface Serial #	: 5237270013	A/D mV Range	: 10000
Delay Time	: 0.00 min	End Time	: 30.00 min
Sampling Rate	: 1.0000 pts/s		
Volume Injected	: 100.000000 µL	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 17.01.2000 23:37:41	Cycle	: 8

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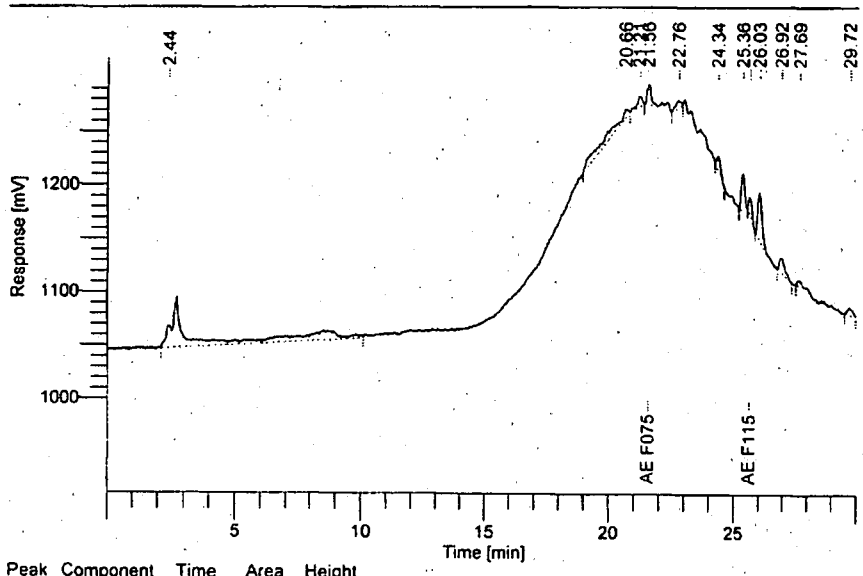
Sequence File : \\adeft003\Turbochrom Daten\Brueggemann\Sequenzen\CR99029\bru005.seq
Sample Notes:
1 mL / 100 µL



Peak #	Component Name	Time [min]	Area [µV·s]	Height [µV]
4	AE F075736	21.564	209790	19301
8	AE F115008	25.627	154250	15150

HESSISCHES MINISTERIUM
FÜR UMWELT, ENERGIE,
JUGEND, FAMILIE UND
GESUNDHEIT

GLP-Bescheinigung

Bescheinigung

Hiermit wird bestätigt, daß die Prüfeinrichtung
Rückstände und Verbrauchersicherheit
in 65929 Frankfurt am Main
Brüningstraße 50
(Ort, Anschrift)
der Hoechst Schering AgrEvo GmbH, Werk Höchst
(Firma)
am 06.06.1997 und 10.09.1997
(Datum)

von der für die Überwachung zuständigen Behörden über die Einhaltung der Grundsätze der Guten Laborpraxis inspiziert worden ist.

Es wird hiermit bestätigt, daß folgende Prüfungen in dieser Prüfeinrichtung nach den Grundsätzen der Guten Laborpraxis durchgeführt werden:

Prüfungen zur Bestimmung von Rückständen

Certificate

It is hereby certified that the test facility
Rückstände und Verbrauchersicherheit
in Frankfurt am Main
Brüningstraße 50
(location, address)
of Hoechst Schering AgrEvo GmbH, Werk Höchst
(company name)
on 06.06.1997 und 10.09.1997
(date)

was inspected by the competent authority regarding compliance with the Principles of Good Laboratory Practice.

It is hereby certified that studies in this test facility are conducted in compliance with the Principles of Good Laboratory Practice:

Residues

Im Auftrag

Dr. Hecker

(Dr. Hecker) Wiesbaden, den 11 Februar 1998