



LAB Research Ltd.

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FINAL REPORT

**ESTIMATION OF THE
ADSORPTION COEFFICIENT (K_{oc})
OF ETHYLENE DIBROMIDE
INDUSTRIAL (EDB)**

STATEMENT OF STUDY DIRECTOR

This study has been performed in accordance with the study plan, and the Principles of Good Laboratory Practice (Hungarian GLP Regulations: 9/2001 (III. 30) EüM-FVM joint decree of the Minister of Health and the Minister of Agriculture and Regional Development which corresponds to the OECD GLP, ENV/MC/CHEM (98) 17.).

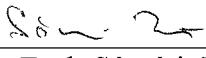
I the undersigned declare that this report constitutes a true record of the actions undertaken and the results obtained in the course of this study.

Summary of the results:

In the course of this study the estimated adsorption coefficient (K_{oc}) of Ethylene Dibromide Industrial (EDB) was determined using high performance liquid chromatography.

The estimated $\log K_{oc}$ 1.91.

The estimated K_{oc} 82.

Signature: 
Zsolt Sárvári, M.Sc.
Study Director

Date: 28 July 2010

STATEMENT OF THE MANAGEMENT

According to the conditions of the research and development agreement between Chemtura Corporation (as Sponsor) and LAB Research Ltd. (as Testing Facility) "Estimation of the Adsorption Coefficient (K_{oc}) of Ethylene Dibromide Industrial (EDB)" has been performed in compliance with the study plan and the Principles of Good Laboratory Practice.

Signature: 
Christopher Banks, DABT
Managing Director

Date: 28 July 2000

QUALITY ASSURANCE STATEMENT

Study Code: 10/112-331AN

Study Title: Estimation of the Adsorption Coefficient (K_{oc}) of Ethylene Dibromide Industrial (EDB)

Test Item: Ethylene Dibromide Industrial (EDB)

This study has been inspected, and this report audited by the Quality Assurance Unit in compliance with the Principles of Good Laboratory Practice. As far as it can be reasonably established, the methods described and the results incorporated in this report accurately reflect the raw data produced during this study.

All inspections, data reviews and the report audit were reported in writing to the study director and to management. The dates of such inspections and of the report audit are given below:

Date of Inspection	Phase(s) Inspected / Audited	Date of report to	
		Management	Study Director
28 June 2010	Study Plan	28 June 2010	28 June 2010
02 July 2010	Solution preparation, identification	02 July 2010	02 July 2010
19 July 2010	Draft Report	19 July 2010	19 July 2010
28 July 2010	Final Report	28 July 2010	28 July 2010

Signature: Gyimesi · Vanda
Vanda Gyimesi, M.Sc.
On behalf of QAU

Date: 28 July 2010

STUDY TITLE: Estimation of the Adsorption Coefficient (K_{oc}) of Ethylene Dibromide Industrial (EDB)

SPONSOR: CHEMTURA CORPORATION
Address: 199, Benson Road,
Middlebury,
Connecticut 06749
USA

STUDY PERFORMED BY: LAB Research Ltd.
Address: H-8200 Veszprém, Szabadságpuszta
Hungary
Phone: +36 88 545 300
Fax: +36 88 545 301

STUDY DIRECTOR: Zsolt Sárvári, M.Sc.

ASSISTANT SCIENTIST: György Kiss, M.Sc.

QUALITY ASSURANCE: Vanda Gyimesi, M.Sc.
On behalf of QAU

BASIS OF STUDY: OECD Guideline for Testing of Chemicals No. 121., "Estimation of the Adsorption Coefficient (KOC) on Soil and on Sewage Sludge using High Performance Liquid Chromatography (HPLC)" (Original Guideline, adopted 22nd January 2001),

European Economic Community (EEC) directive 2001/59, Annex V, C.19 „Estimation of the Adsorption Coefficient (Koc) on Soil and on Sewage sludge using High Performance Liquid Chromatography (HPLC)“, EEC Publication No. O.J. L225, 2001

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1. OBJECTIVE OF STUDY

The purpose of the study was to estimate the adsorption coefficient, K_{oc} of Ethylene Dibromide Industrial (EDB) using the HPLC method.

2. MATERIALS

2.1. TEST ITEM

Name:	Ethylene Dibromide Industrial (EDB)
Chemical name:	1,2-Dibromoethane
Batch No.:	510100003
Active component:	>99.94 % 1,2-Dibromoethane (CAS 106-93-4)
Description:	clear to amber liquid
Manufacture date:	February 2010
Expiry date:	February 2011
Storage:	room temperature, protected from light
Safety Precautions:	see Safety Data Sheet
Manufacturer:	Chemtura Manufacturing UK Limited
	Address: Tenax Road, Trafford Park
	Manchester
	United Kingdom
	M17 1WT

2.1.1. Receipt

The test item and safety instructions, required for the handling and disposal of the test substance, were received from the Sponsor.

Identification of test item was carried out by Central Dispensary Unit of LAB Research Ltd. and was based on its appearance and colour.

2.2. REAGENTS AND MATERIALS

Methanol:	for HPLC, Carlo Erba, Batch No.: V9M706099M
Ultra pure water (ASTM Type I):	prepared by Direct-Q 3 system, Millipore

3. TEST METHODS AND RESULTS

Start of experiment: 02 July 2010
End of experiment: 03 July 2010

3.1. PRINCIPLE OF THE TEST

The method uses HPLC for estimation of the adsorption coefficient, K_{oc} . The dual composition of the stationary phase having polar and non-polar sites allows for interaction of polar and non-polar groups of a molecule in a similar way, as in the case for organic matter in soil or sewage sludge matrices. This enables the relationship between the retention time on the column and the adsorption coefficient on organic matter to be established.

3.2. APPARATUS

Stationary Phase LiChrospher 100 CN 250 x 4 mm, 5 μ m HPLC Column
No.: 540779-1

HPLC system: Merck-Hitachi LaChrom HPLC system:
D-7000 Interface, No.: 0811-181
L-7100 HPLC Pump, No.: 0823-059
L-7200 Autosampler, No.: 0606-021
L-7400 UV Detector, No.: 0928-058
655A-52 Column Oven, No.: 6211-016
L-7612 Degasser, No.: 012-05

Ultrasonic bath: Elmasonic S300H, ELMA, No.: 010890105
Balance: BP221S Sartorius, No.: 11809117
Refrigerator: Zanussi No.: ZLKI-262
Water purification system: MILLIPORE, DIRECT Q3, FOMNO 7334I

HPLC Conditions:

Detector: UV at 207 nm
Column: LiChrospher 100 CN 250 x 4 mm, 5 μ m No.: 540779-1
Mobil Phase: Methanol : water = 55 : 45
Flow: 1 ml/min.
Injection volume: 20 μ l
Temperature: 25 °C

3.3. REFERENCE SUBSTANCES

Seven chemicals for which $\log K_{oc}$ has been reported were used to calibrate the elution time in units of $\log K_{oc}$.

Dead time, t_0 was determined by the injection of sodium nitrate solution.

Table 1. Reference substances used for calibration

Chemical name	CAS Number	$\log K_{oc}$	Purity, Supplier	Batch No.
Sodium nitrate	7631-99-4	t_0	100 % Merck KGaA	A980637
Acetanilide	103-84-4	1.25	99.9 % Merck KGaA	S4715244
Methyl benzoate	93-58-3	1.80	99.9 % Merck KGaA	S4748230
Atrazine	1912-24-9	1.81	97.5 % Sigma-Aldrich	8087X
Isoproturon	34123-59-6	1.86	99.8 %, Sigma Aldrich	3209X
Linuron	330-55-2	2.59	99.7 %, Sigma Aldrich	5320X
Naphthalene	91-20-3	2.75	99.9 % Merck KGaA	S4767446
Fenthion	55-38-9	3.31	97.8 %, Sigma Aldrich	6193X
α -Endosulfan	959-98-8	4.09	99.6 % Sigma-Aldrich	SZE8289X

3.4. PERFORMANCE OF THE TEST

Preparation of the Test Solutions:

Reference substances and test item were dissolved in methanol. Concentration of these stock solutions was about 1 mg/ml.

These stock solutions were diluted with the mobile phase resulting in about 10 μ g/ml solutions except sodium nitrate and test item which were 500 and 20 μ g/ml.

Determination of the retention times:

Three series of the above reference solutions and the test item solution were measured alternately, with two replicate injections.

For the determination of t_0 (t_0 = retention time of the unretarded component) sodium nitrate solution was injected.

The retention times were determined.

3.5. EVALUATION

The capacity factors, k' were calculated from the retention times (t_R) of the selected reference substances and the dead time (t_0):

$$k' = \frac{t_R - t_0}{t_0}$$

A calibration plot of $\log k'$ versus $\log K_{oc}$ was prepared. (See Figure 1.) The calibration equation was determined by linear regression using the least squares method.

Repeatability is the difference of maximum and minimum values of $\log K_{oc}$ derived from individual measurements.

Accuracy is the difference between the estimated $\log K_{oc}$ and the $\log K_{oc}$ value determined by the batch equilibrium method.

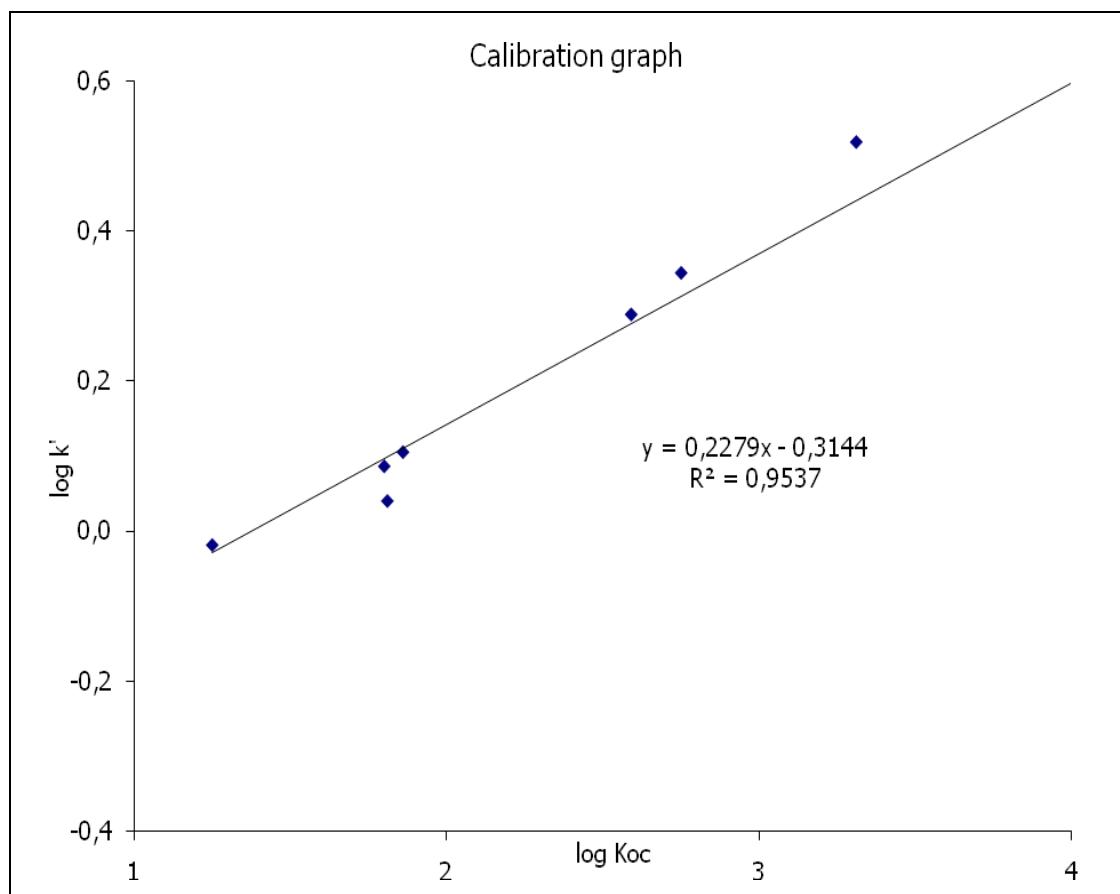


Figure 1. The calibration plot

3.6. RESULTS

The three calibration series and three test item series were measured alternately. The adsorption coefficient K_{oc} was determined based on a calibration curve using 8 reference items.

The capacity factors k' were calculated from the dead time and retention times of the reference substances. The log k' data of the reference substances were plotted against their log K_{oc} values. The resulting calibration curve and the respective data are given in Figure 1 and Table 1, respectively. The equation of the curve fitted to the calibration points is as follows:

$$\log k' = 0.228 \times \log K_{oc} - 0.314 \quad (R_{sq} = 0.954)$$

Table 2: Measured and calculated data of the reference substances and test item

Chemical name	log K_{oc} from OECD 121	Retention time (min)	log k'	log K_{oc}	Accuracy log unit	Repeatability $\Delta \log K_{OC}$, log unit
			Calculated	Calculated		
Sodium nitrate	-	1.56			for determination of dead time	
		1.56				
		1.56				
		1.56				
		1.57				
		1.57				
		Mean	1.56			
	CV%	0.3				
Acetanilide	1.25	3.06	-0.019	1.30	0.05	0.01
		3.06	-0.019	1.30		
		3.06	-0.019	1.30		
		3.07	-0.016	1.31		
		3.07	-0.016	1.31		
		3.06	-0.019	1.30		
		Mean	3.06	-0.018		
	CV%	0.2	-	-		
Atrazine	1.81	3.28	0.041	1.56	0.25	0.00
		3.28	0.041	1.56		
		3.28	0.041	1.56		
		3.28	0.041	1.56		
		3.28	0.041	1.56		
		3.28	0.041	1.56		
		Mean	3.28	0.041		
	CV%	0.0	-	-		
Methyl-benzoate	1.80	3.47	0.086	1.76	0.04	0.01
		3.47	0.086	1.76		
		3.48	0.088	1.77		
		3.47	0.086	1.76		
		3.47	0.086	1.76		
		3.48	0.088	1.77		
		Mean	3.47	0.087		
	CV%	0.1	-	-		

Table 2: Measured and calculated data of the reference substances and test item (continued)

Chemical name	log K _{OC} from OECD 121	Retention time (min)	log k'	log K _{OC}	Accuracy log unit	Repeatability ΔlogK _{OC} , log unit
			Calculated	Calculated		
Isoproturon	1.86	3.56	0.106	1.85	0.02	0.01
		3.56	0.106	1.85		
		3.56	0.106	1.85		
		3.55	0.104	1.84		
		3.56	0.106	1.85		
		3.56	0.106	1.85		
		Mean	3.56	0.106		
	CV%	0.1	-	-		
Linuron	2.59	4.60	0.288	2.65	0.06	0.01
		4.60	0.288	2.65		
		4.61	0.290	2.65		
		4.61	0.290	2.65		
		4.61	0.290	2.65		
		4.61	0.290	2.65		
		Mean	4.61	0.289		
	CV%	0.1	-	-		
Naphthalene	2.75	5.01	0.343	2.89	0.14	0.01
		5.01	0.343	2.89		
		5.03	0.346	2.90		
		5.03	0.346	2.90		
		5.02	0.345	2.89		
		5.02	0.345	2.89		
		Mean	5.02	0.345		
	CV%	0.2	-	-		
Fenthion	3.31	6.70	0.517	3.65	0.35	0.02
		6.70	0.517	3.65		
		6.74	0.520	3.66		
		6.75	0.521	3.67		
		6.74	0.520	3.66		
		6.73	0.519	3.66		
		Mean	6.73	0.519		
	CV%	0.3	-	-		
α-Endosulfan	4.09	7.12	0.551	3.80	0.29	0.01
		7.11	0.550	3.79		
		7.13	0.552	3.80		
		7.13	0.552	3.80		
		7.10	0.549	3.79		
		7.10	0.549	3.79		
		Mean	7.12	0.550		
	CV%	0.2	-	-		
Ethylene Dibromide Industrial (EDB)	-	3.63	0.121	1.91	-	0.01
		3.63	0.121	1.91		
		3.63	0.121	1.91		
		3.64	0.123	1.92		
		3.63	0.121	1.91		
		3.63	0.121	1.91		
		Mean	3.63	0.122		
	CV%	0.1	-	-		

The capacity factors k' and $\log K_{oc}$ for Ethylene Dibromide Industrial (EDB) were calculated based on the calibration data obtained with the reference substances.

The estimated $\log K_{oc}$ 1.91.

The estimated K_{oc} 82.

4. ARCHIVES

The study documents:

- study plan
- all raw data
- sample of the test item
- study report and any amendment(s)
- correspondence

are stored in the archives of LAB Research Ltd., 8200 Veszprém, Szabadságpuszta, Hungary, according to the Hungarian GLP and our SOPs.

5. DISTRIBUTION OF THE FINAL REPORT

Sponsor:

- 1 × copy, bound
- 1 × copy, unbound
- 1 × pdf-file

Archives:

- 1 × original, bound

APPENDIX

Copy of the GLP-Certificate



ORSZÁGOS GYÓGYSZERÉSZETI INTÉZET
National Institute of Pharmacy

H-1051 Budapest, Zrinyi u. 3.

Mail: 1372 P.O. Box 450.

Phone: +36 1 8869-300

Fax: +36 1 8869-460

E-mail: ogyi@ogyi.hu

Budapest, 20th December 2008

No: 38625/48/2007

Our ref.: Szilvia Karsai

Subject: GLP Certificate

GOOD LABORATORY PRACTICE (GLP) CERTIFICATE

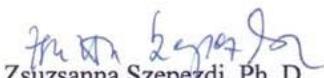
Based on the Inspection report and the discussion of follow up activities it is hereby certified that the test facility

LAB Research Ltd.
H-8201 Veszprém, Szabadságpuszta, Hungary

is able to carry out **Physical-chemical testing, Toxicity studies, Mutagenicity studies, Environmental toxicity studies on aquatic and terrestrial organisms, Studies on behaviour in water, soil and air; bioaccumulation, Bioanalytical, Analytical and clinical chemistry testing** compliance with the Principles of GLP (Good Laboratory Practice).

Date of the inspection: **13-22 October 2008**.

This GLP Certificate is valid for 2 years.


Zsuzsanna Szepezdi, Ph. D.
Director-General

Application(data): 10_112_331AN

System: Merck1

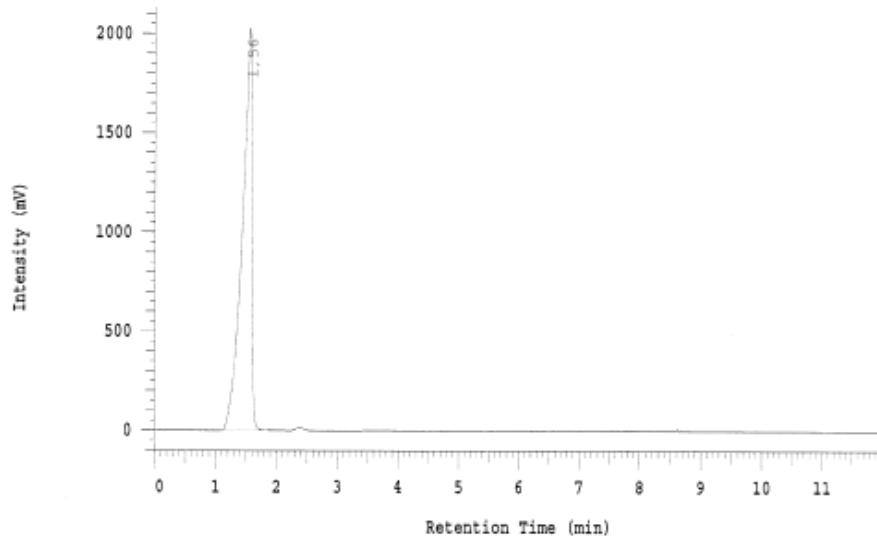
D-7000 HPLC System Manager Report**Series:Merces_1**

Analyzed: 10.07.02 19:51
Application: 10_112_331AN
Acquisition Method: EDB

Sample Name: Sodium nitrate

Sample Description:
Sample Amount (ml): 1
Vial Type: UNK
Vial Number: 11
Injection from this vial: 2 of 2
Volume: 20.0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
1.56	21931249	2027954
	21931249	2027954

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:23
Reported Date and Time: 10.07.08 15:23

Study Code: 10/112-331AN

CHROMATOGRAM 1.
Chromatogram of Sodium Nitrate

Application(data): 10_112_331AN

System: Merck1

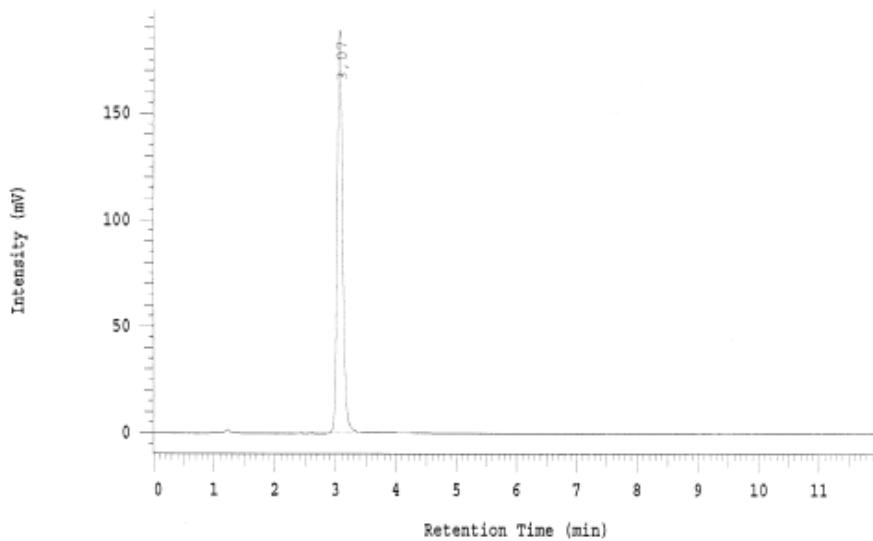
D-7000 HPLC System Manager Report**Series:Merces_1**

Analyzed: 10.07.02 20:18
Application: 10_112_331AN
Acquisition Method: EDB

Sample Name: Acetanilide

Sample Description:
Sample Amount (ml): 1
Vial Type: UNK
Vial Number: 12
Injection from this vial: 2 of 2
Volume: 20,0 μ l

Data Path: D:\MERESEK\10_112_331AN\DATA\Meres_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
3.07	1141974	188684
	1141974	188684

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:23
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

CHROMATOGRAM 2.
Chromatogram of Acetanilid

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report**Series:Merces_1**

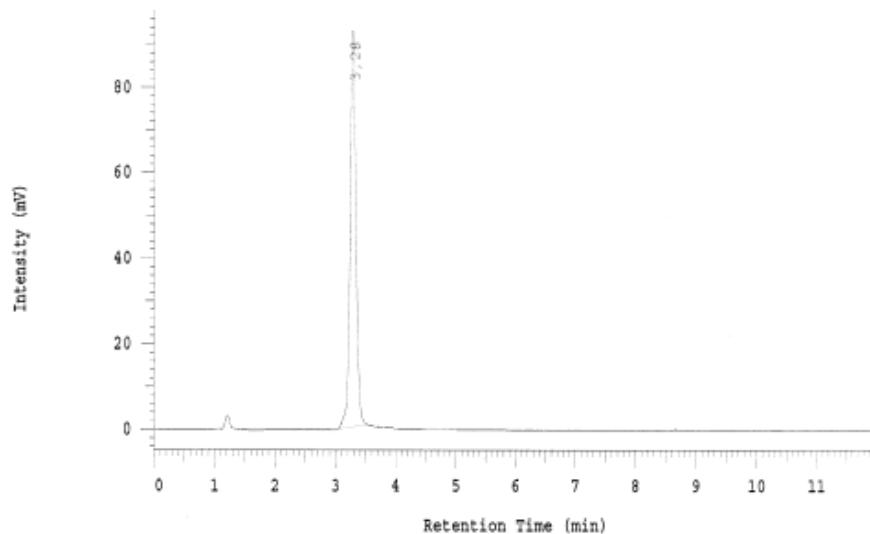
Analyzed: 10.07.02 20:45
Application: 10_112_331AN
Acquisition Method: EDB

Sample Name: Atrazine

Sample Description:
Sample Amount (ml): 1
Vial Type: UNK

Vial Number: 13
Injection from this vial: 2 of 2
Volume: 20,0 μ l

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
3,28	628705	92588
	628705	92588

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:24
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

CHROMATOGRAM 3.
Chromatogram of Atrazine

Application(data): 10_112_331AN

System: Merck1

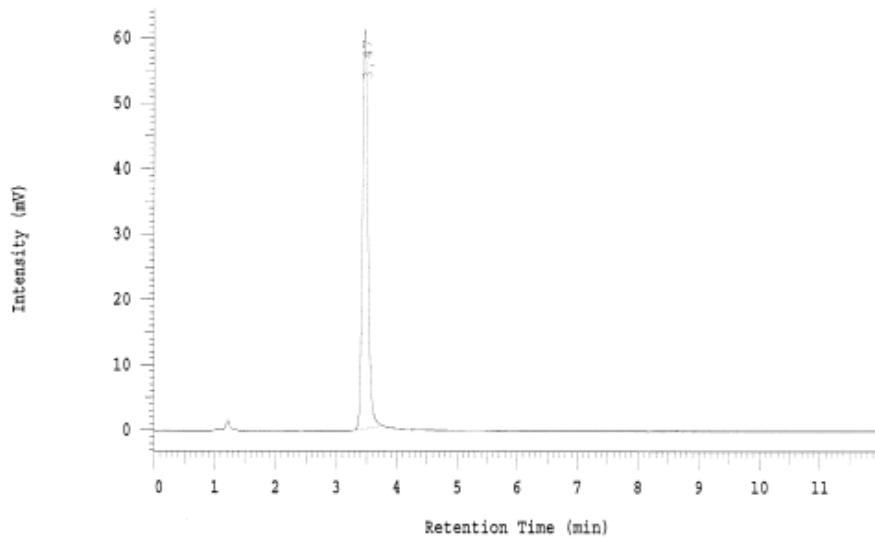
D-7000 HPLC System Manager Report**Series:Merces_1**

Analyzed: 10.07.02 21:12
Application: 10_112_331AN
Acquisition Method: EDB

Sample Name: Methyl benzoate

Sample Description:
Sample Amount (ml): 1
Vial Type: UNK
Vial Number: 14
Injection from this vial: 2 of 2
Volume: 20,0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
3,47	386128	61092
	386128	61092

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:24
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

CHROMATOGRAM 4.
Chromatogram of Methyl benzoate

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report

Series:Merces_1

Analyzed: 10.07.02 21:38

Application: 10_112_331AN

Acquisition Method: EDB

Sample Name: Isoproturon

Sample Description:

Sample Amount (ml): 1

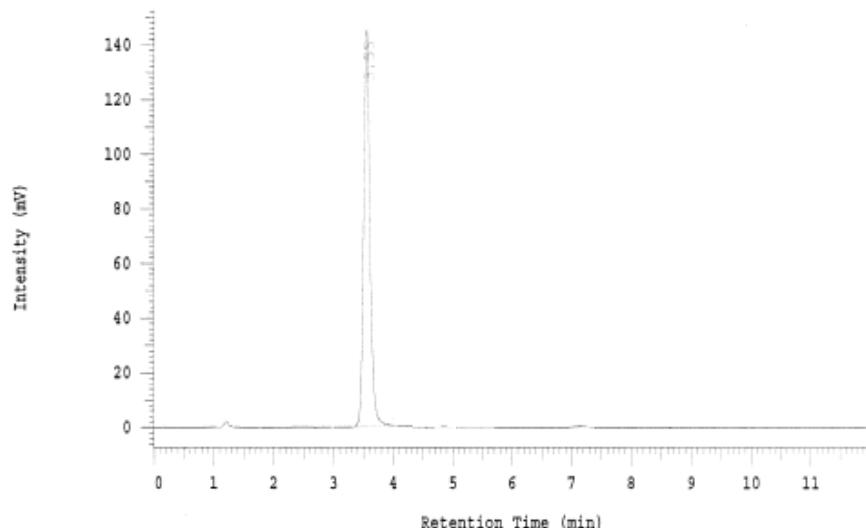
Vial Type: UNK

Vial Number: 15

Injection from this vial: 2 of 2

Volume: 20,0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
3,55	1061220	144972
	1061220	144972

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:24
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

CHROMATOGRAM 5.
Chromatogram of Isoproturon

Application(data): 10_112_331AN

System: Merck1

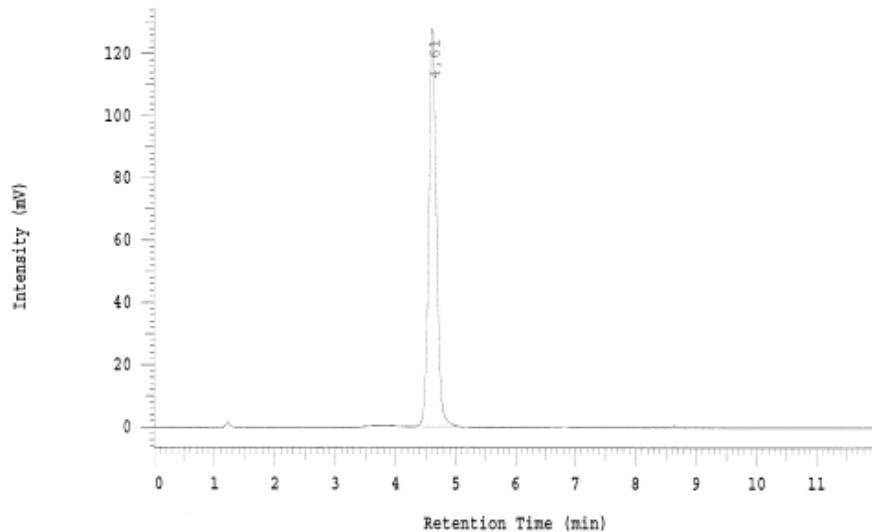
D-7000 HPLC System Manager Report**Series: Meres_1**

Analyzed: 10.07.02 22:05
Application: 10_112_331AN
Acquisition Method: EDB

Sample Name: Linuron

Sample Description:
Sample Amount (ml): 1
Vial Type: UNK
Vial Number: 16
Injection from this vial: 2 of 2
Volume: 20,0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Meres_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
4,61	1127638	127786
	1127638	127786

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:24
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

CHROMATOGRAM 6.
Chromatogram of Linuron

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report

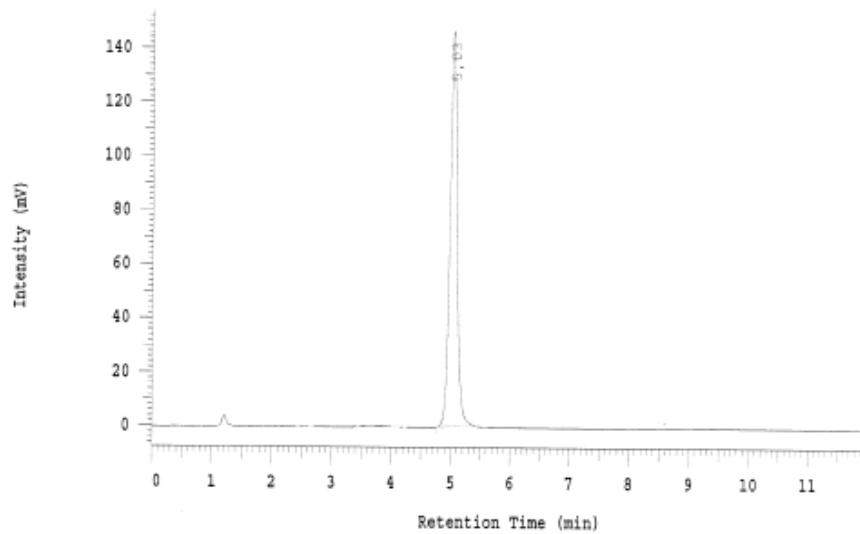
Series:Merces_1

Analyzed: 10.07.02 22:32
Application: 10_112_331AN
Acquisition Method: EDB

Sample Name: Naphthalene

Sample Description:
Sample Amount (ml): 1
Vial Type: UNK
Vial Number: 17
Injection from this vial: 2 of 2
Volume: 20.0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
5.03	1230680	145974
	1230680	145974

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:24
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

CHROMATOGRAM 7.
Chromatogram of Naphthalene

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report

Series:Merces_1

Analyzed: 10.07.02 22:59

Application: 10_112_331AN

Acquisition Method: EDB

Sample Name: Fenthion

Sample Description:

Sample Amount (ml): 1

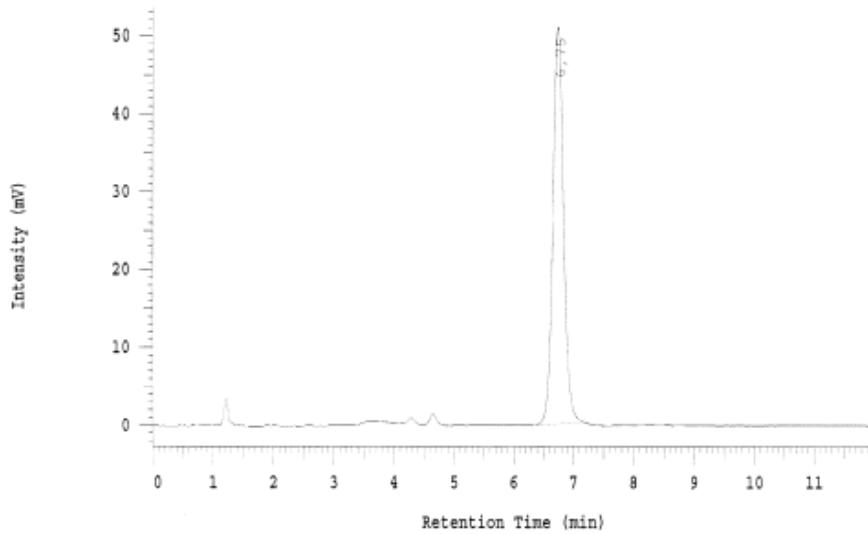
Vial Type: UNK

Vial Number: 18

Injection from this vial: 2 of 2

Volume: 20,0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
6.75	626852	50870
	626852	50870

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:24
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

CHROMATOGRAM 8.
Chromatogram of Fenthion

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report

Series:Merces_1

Analyzed: 10.07.02 23:25

Application: 10_112_331AN

Acquisition Method: EDB

Sample Name: Alfa-Endosulfan

Sample Description:

Sample Amount (ml): 1

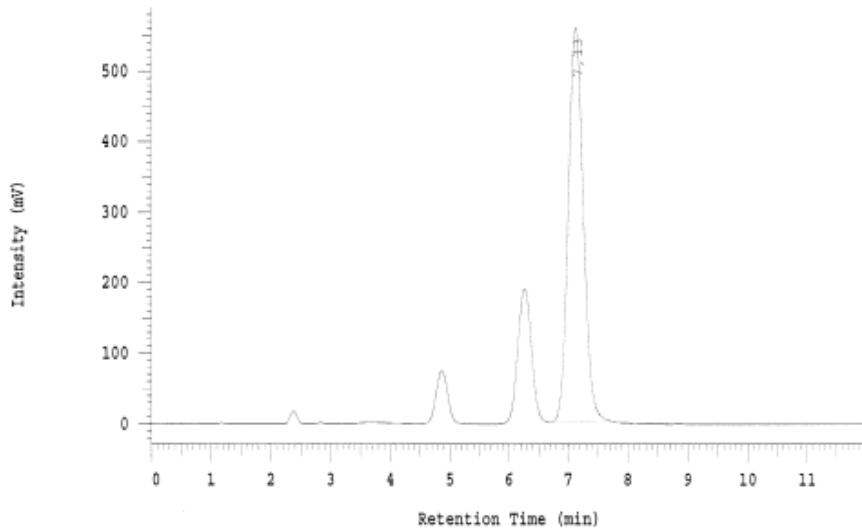
Vial Type: UNK

Vial Number: 19

Injection from this vial: 2 of 2

Volume: 20,0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
7.13	9777705	558414
	9777705	558414

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:25
Reported Date and Time: 10.07.08 15:25

Study Code: 10/112-331AN

CHROMATOGRAM 9.
Chromatogram of α -Endosulfan

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report**Series:Merces_1**

Analyzed: 10.07.02 23:52

Application: 10_112_331AN

Acquisition Method: EDB

Sample Name: EDB

Sample Description:

Sample Amount (ml): 1

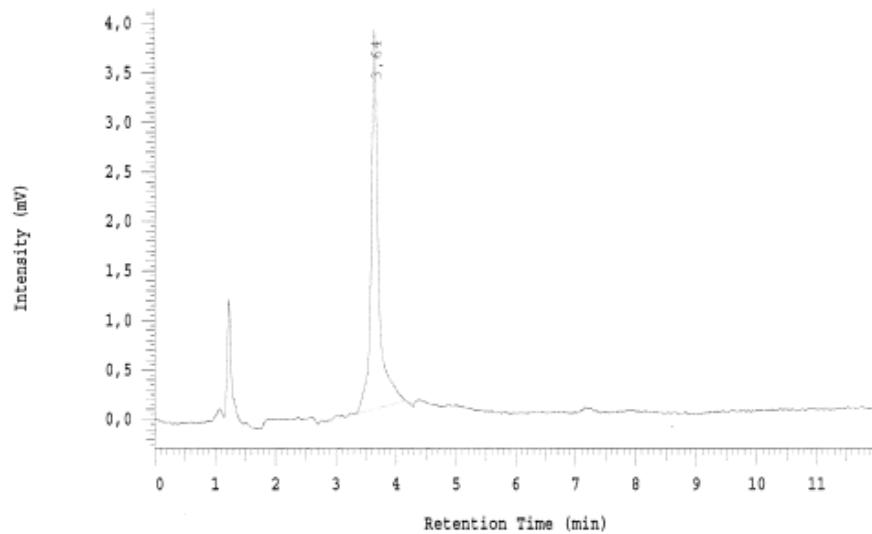
Vial Type: UNK

Vial Number: 20

Injection from this vial: 2 of 2

Volume: 20,0 ul

Data Path: D:\MERESEK\10_112_331AN\DATA\Merces_1\



Pump A Solvent A: MeOH:Viz=55:45
Column Type: LiChrospher 5 μ CN 250x4mm(182)

RT	Area	Height
3,64	32868	3830
	32868	3830

Peak rejection level: 10000
Developed by: LAB Research Ltd.
Processed Date and Time: 10.07.08 15:25
Reported Date and Time: 10.07.08 15:25

Study Code: 10/112-331AN

CHROMATOGRAM 9.
Chromatogram of Ethylene Dibromide Industrial (EDB)