



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 2010

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: _____

Vanda Gyimesi
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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Copy of the GLP Certificate
Chromatograms

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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 × 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 $\mu\text{g/ml}$ solutions except sodium nitrate and test item which were 500 and 20 $\mu\text{g/ml}$.

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.

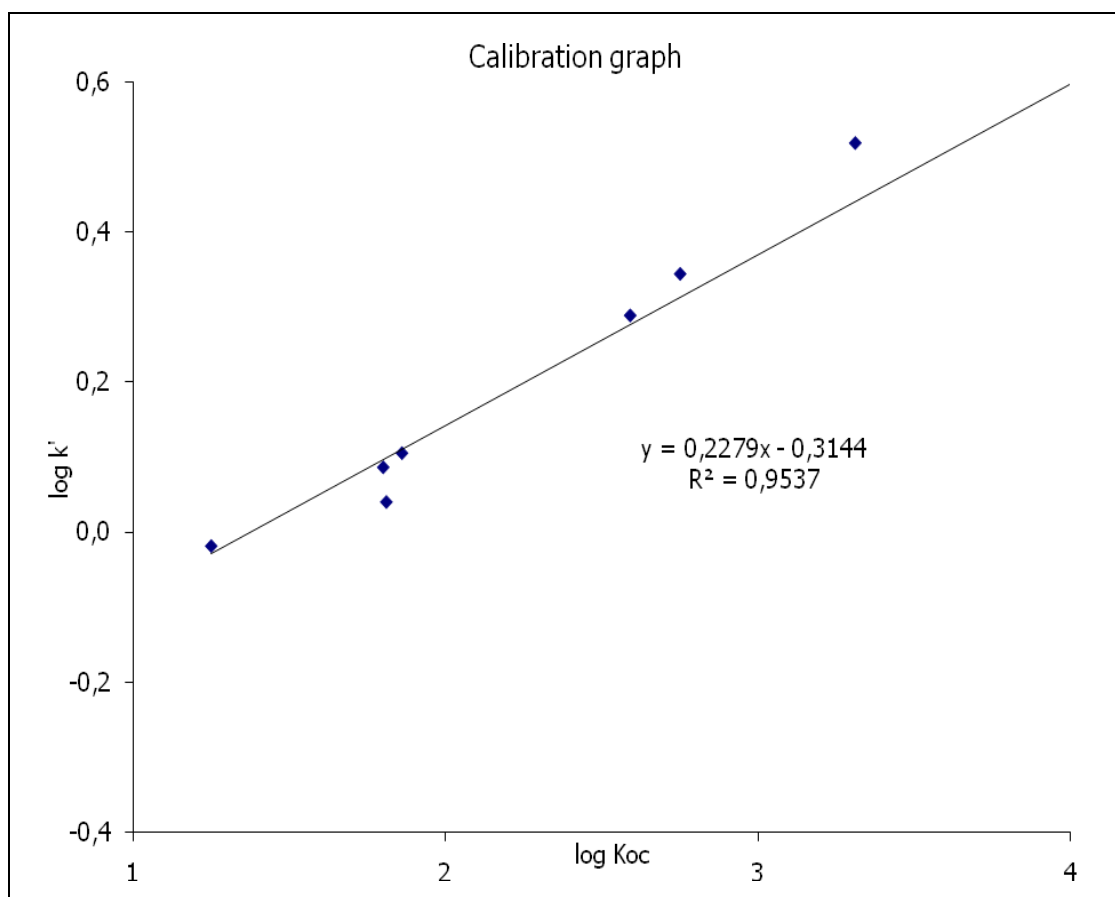


Figure 1. The calibration plot

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} | Retention time (min) | log k' | log K _{oc} | Accuracy log unit | Repeatability ΔlogK _{OC} , log unit |
|----------------|---------------------|----------------------|--------------------------------|---------------------|-------------------|--|
| | from OECD 121 | | 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 | 1.30 | | |
| 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 | 1.56 | | |
| CV% | 0.0 | - | - | | | |
| Methylbenzoate | 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 | 1.76 | | |
| CV% | 0.1 | - | - | | | |

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

| Chemical name | log K _{OC} | Retention time (min) | log k' | log K _{oc} | Accuracy log unit | Repeatability $\Delta\log K_{OC}$, log unit |
|-------------------------------------|---------------------|----------------------|--------------|---------------------|-------------------|--|
| | from OECD 121 | | 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 | 1.84 | | |
| | 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 | 2.65 | | |
| | 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 | 2.89 | | |
| | 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 | 3.66 | | |
| | 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 | 3.80 | | |
| | 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 | 1.91 | | |
| | 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

A P P E N D I X

Copy of the GLP-Certificate



ORSZÁGOS GYÓGYSZERÉSZETI INTÉZET
National Institute of Pharmacy

H-1051 Budapest, Zrínyi 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**

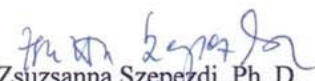
Based on the Inspection report and the discussion of follow up activities it is hereby certified that the test facility

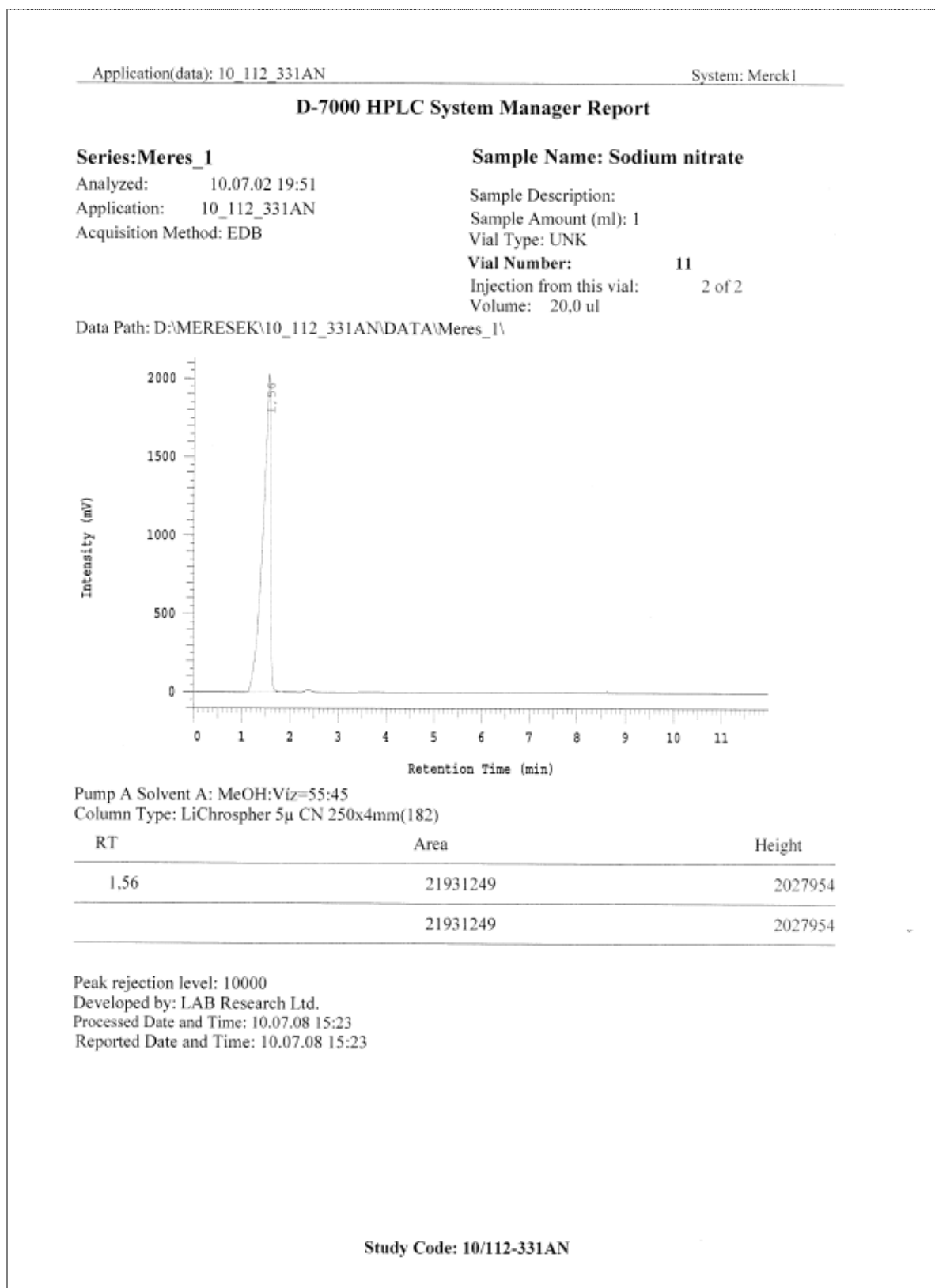
**LAB Research Ltd.
H-8201 Veszprém, Szabadságpuszta, Hungary**

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



CHROMATOGRAM 1.
Chromatogram of Sodium Nitrate

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report**Series:Merese_1****Sample Name: Acetanilide**

Analyzed: 10.07.02 20:18

Sample Description:

Application: 10_112_331AN

Sample Amount (ml): 1

Acquisition Method: EDB

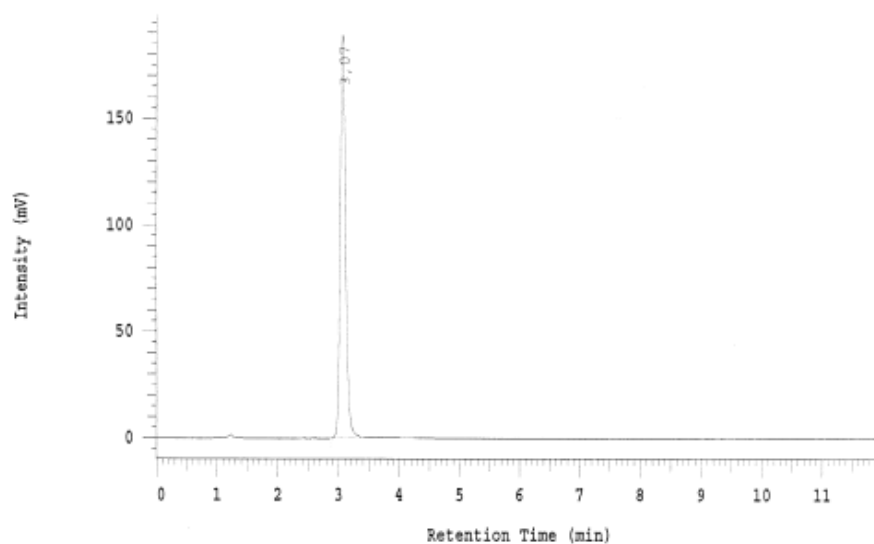
Vial Type: UNK

Vial Number: 12

Injection from this vial: 2 of 2

Volume: 20,0 ul

Data Path: D:\MERSEK\10_112_331AN\DATA\Merese_1\



Pump A Solvent A: MeOH:Viz=55:45

Column Type: LiChrospher 5µ CN 250x4mm(182)

| RT | Area | Height |
|------|---------|--------|
| 3,07 | 1141974 | 188684 |
| | 1141974 | 188684 |

Peak rejection level: 10000

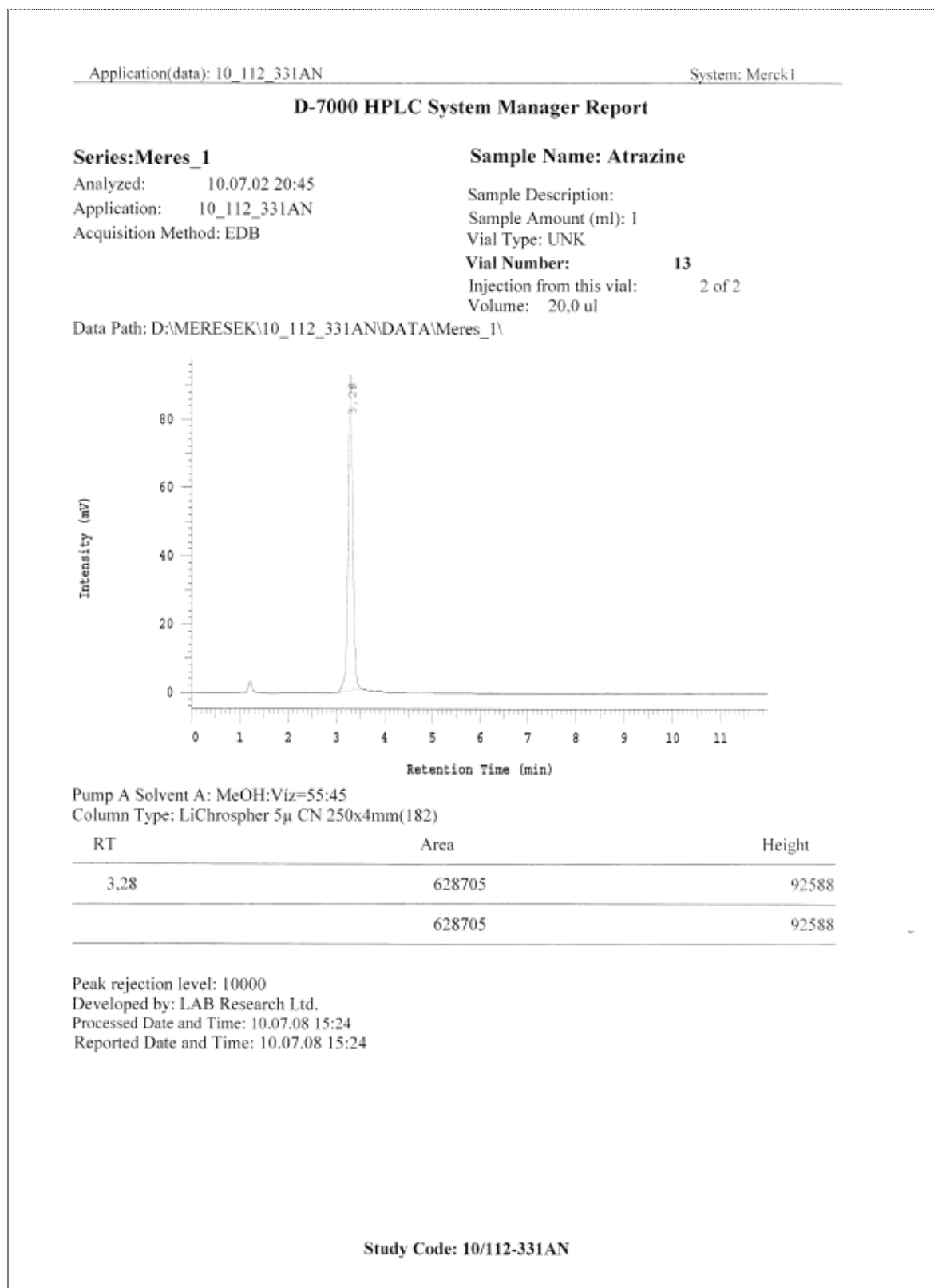
Developed by: LAB Research Ltd.

Processed Date and Time: 10.07.08 15:23

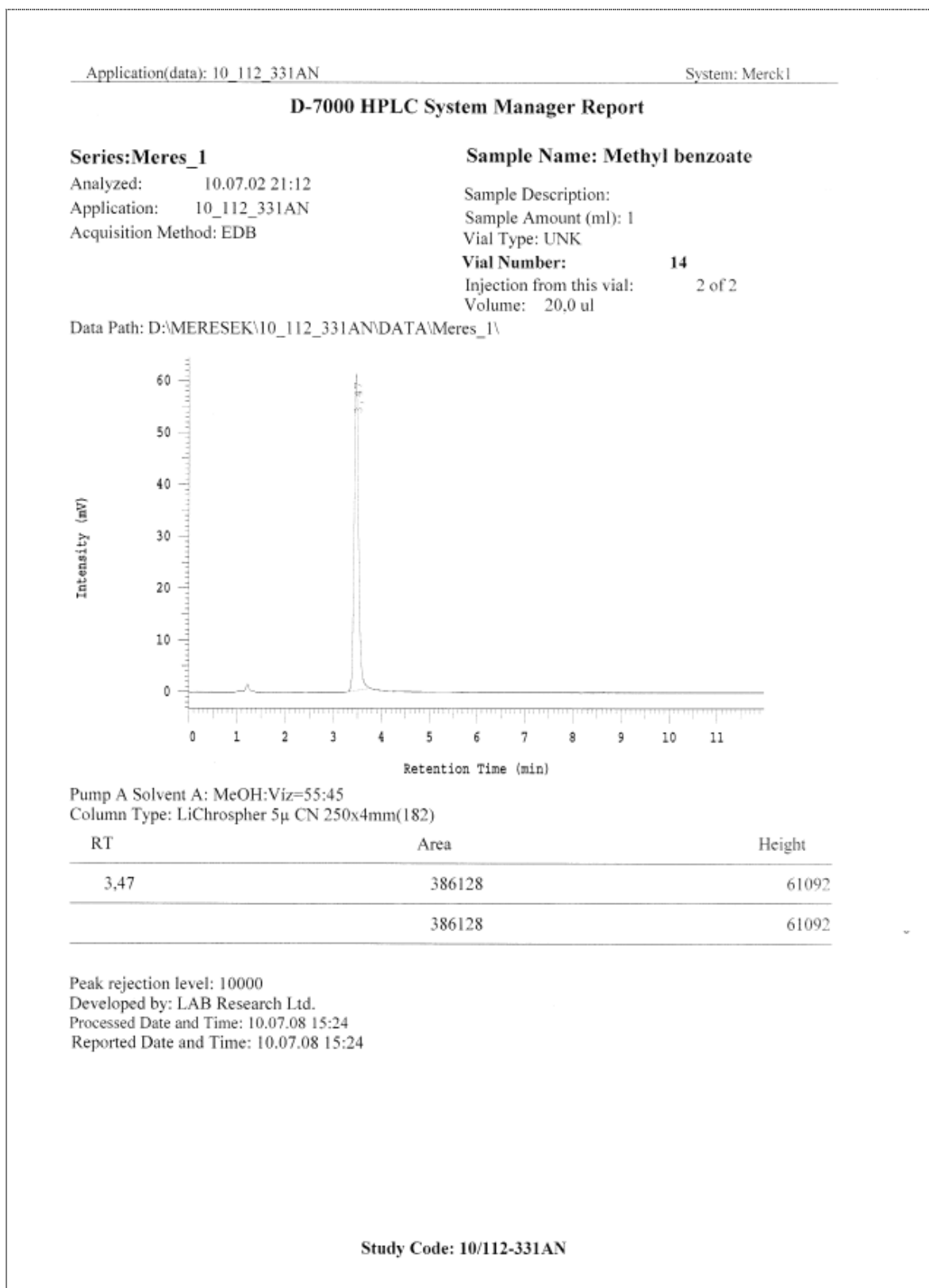
Reported Date and Time: 10.07.08 15:24

Study Code: 10/112-331AN

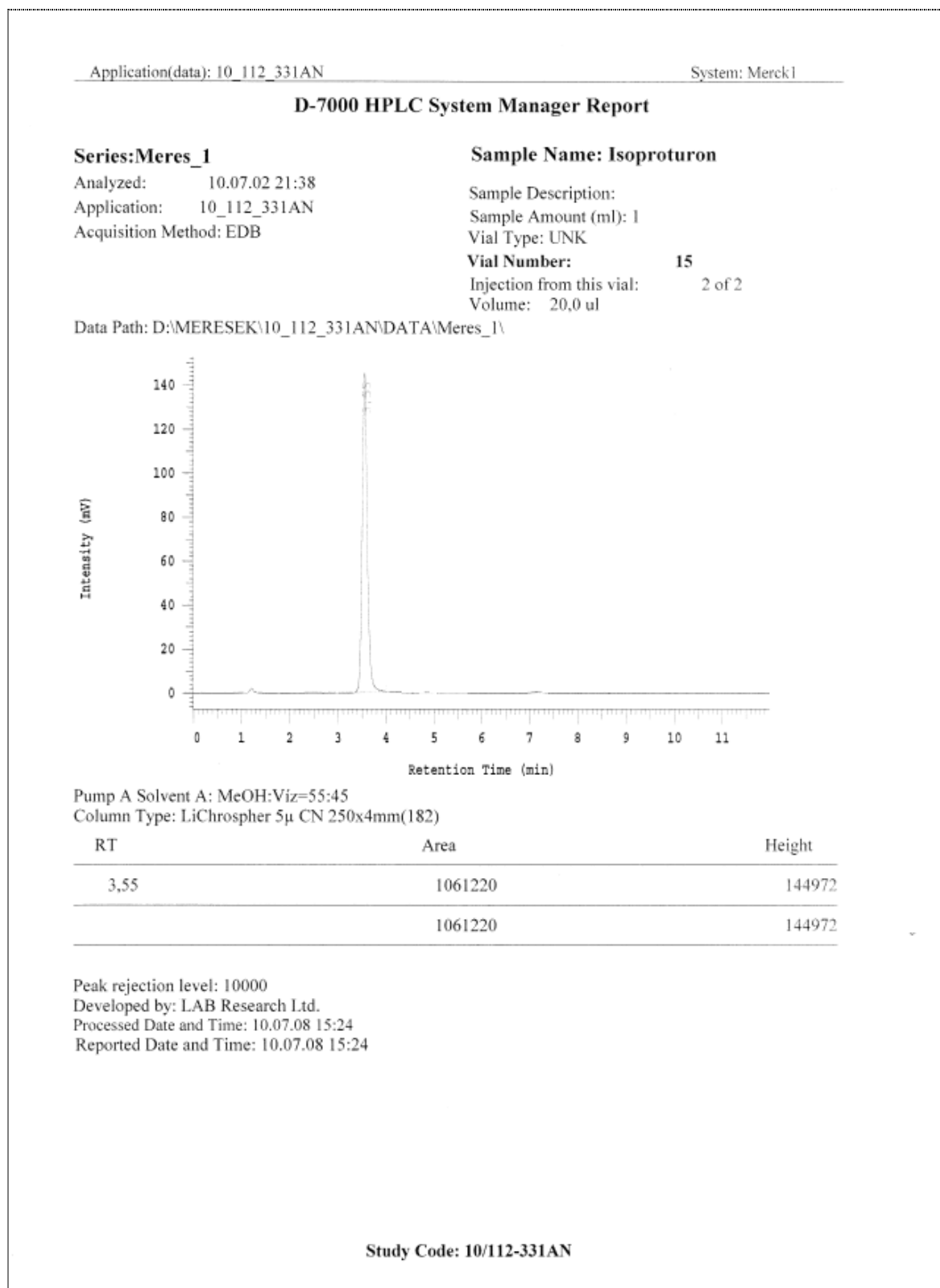
CHROMATOGRAM 2.
Chromatogram of Acetanilid



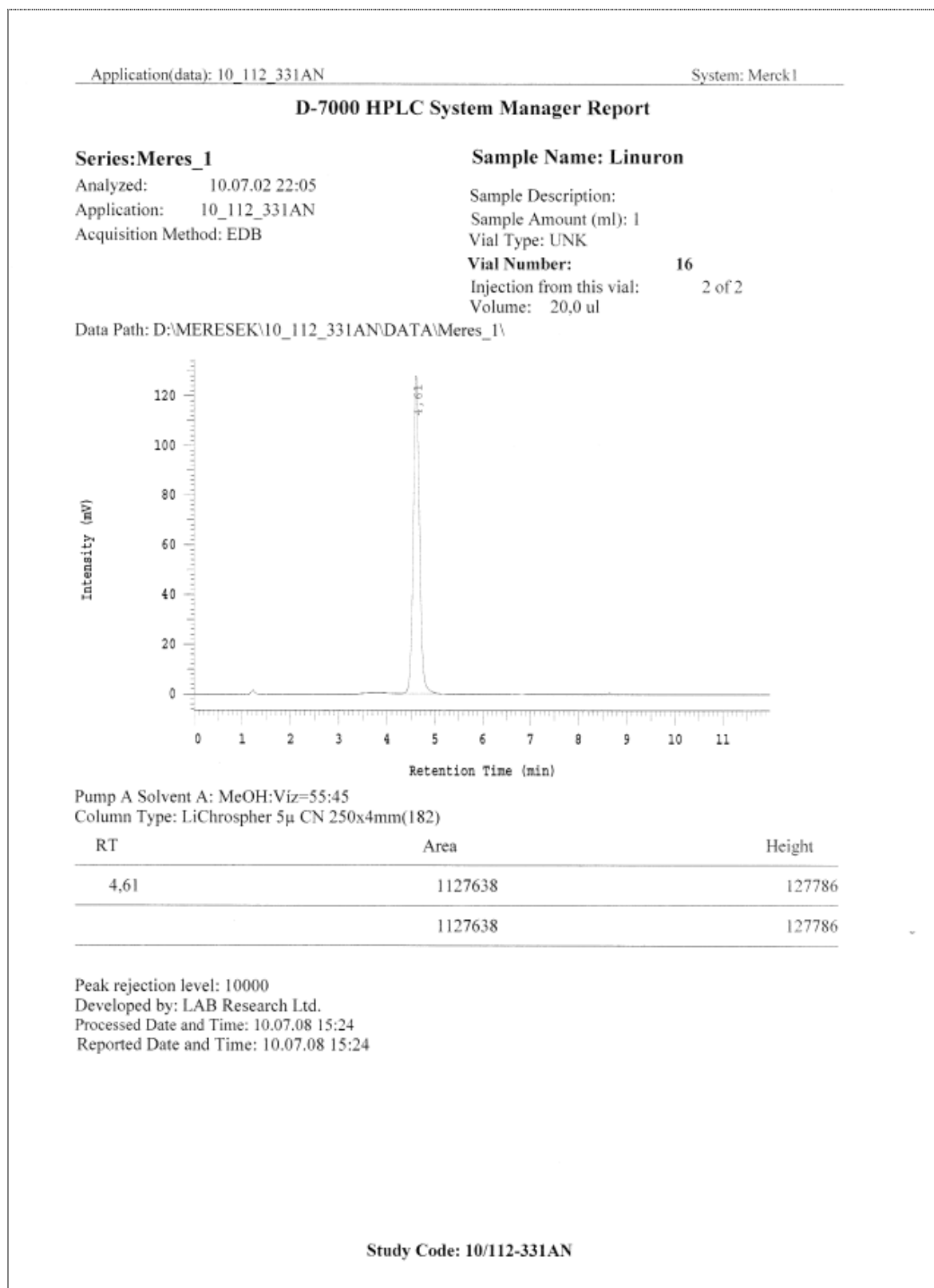
CHROMATOGRAM 3.
Chromatogram of Atrazine



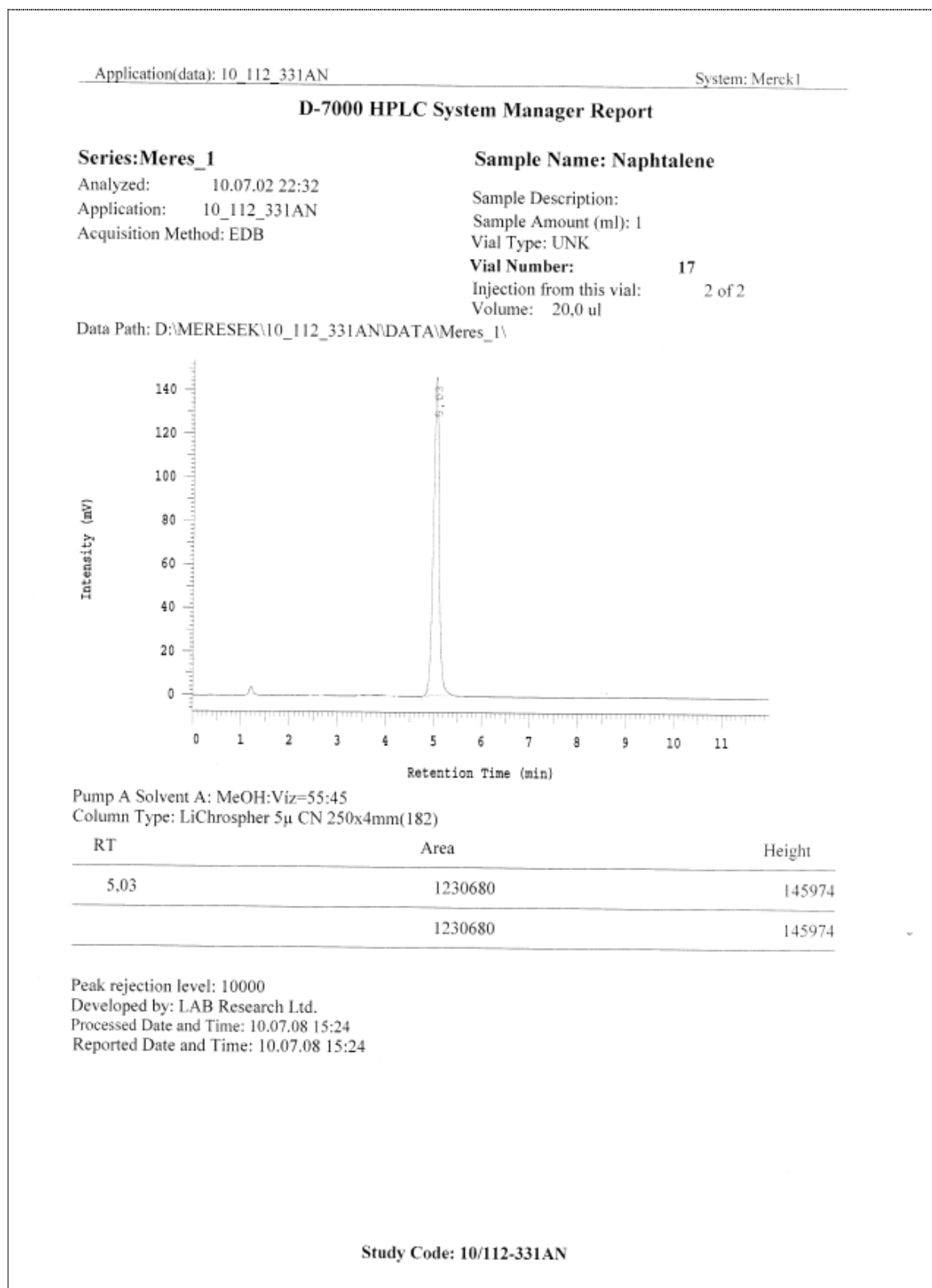
CHROMATOGRAM 4.
Chromatogram of Methyl benzoate



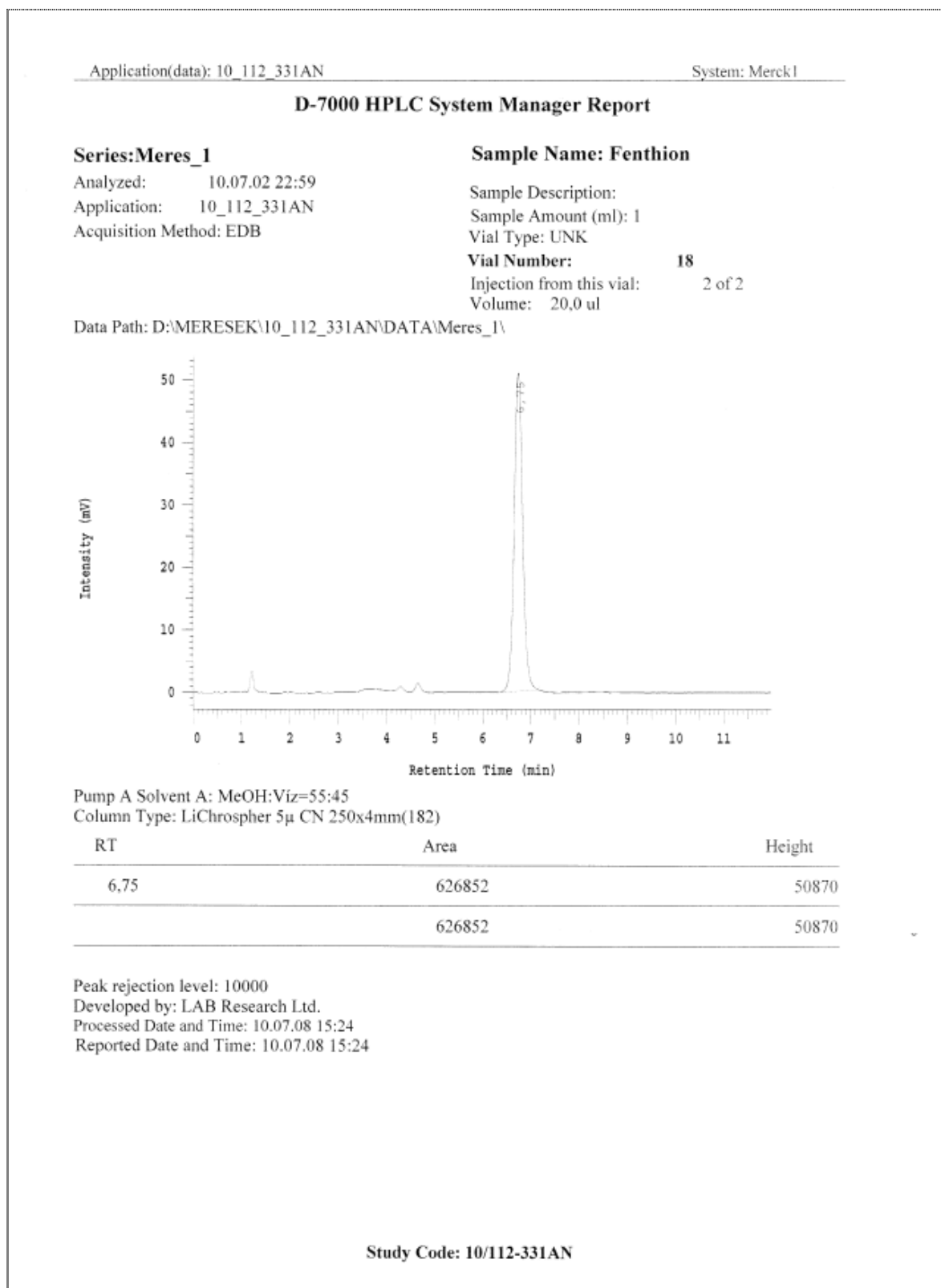
CHROMATOGRAM 5.
Chromatogram of Isoproturon



CHROMATOGRAM 6.
Chromatogram of Linuron



CHROMATOGRAM 7.
Chromatogram of Naphtalene



CHROMATOGRAM 8.
Chromatogram of Fenthion

Application(data): 10_112_331AN

System: Merck1

D-7000 HPLC System Manager Report**Series: Meres_1****Sample Name: Alfa-Endosulfan**

Analyzed: 10.07.02 23:25

Sample Description:

Application: 10_112_331AN

Sample Amount (ml): 1

Acquisition Method: EDB

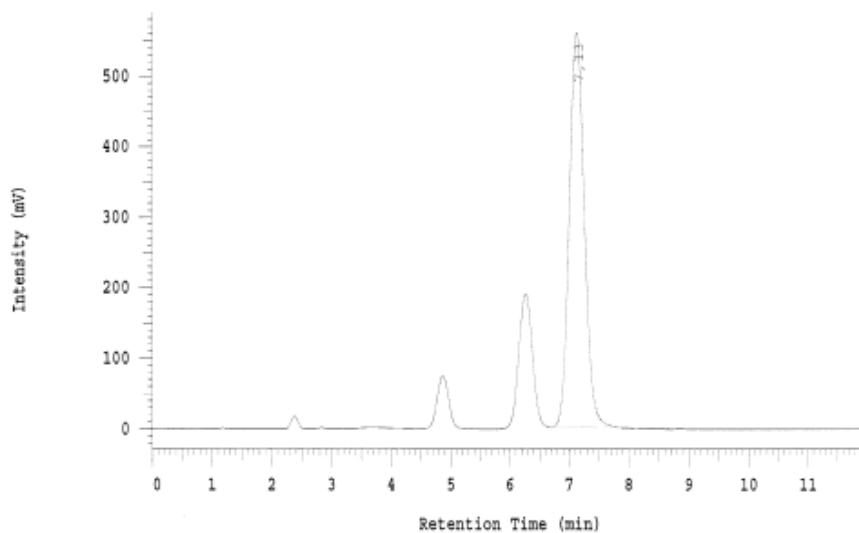
Vial Type: UNK

Vial Number: 19

Injection from this vial: 2 of 2

Volume: 20,0 ul

Data Path: D:\MERSEK\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 |
|------|---------|--------|
| 7,13 | 9777705 | 558414 |
| | 9777705 | 558414 |

Peak rejection level: 10000

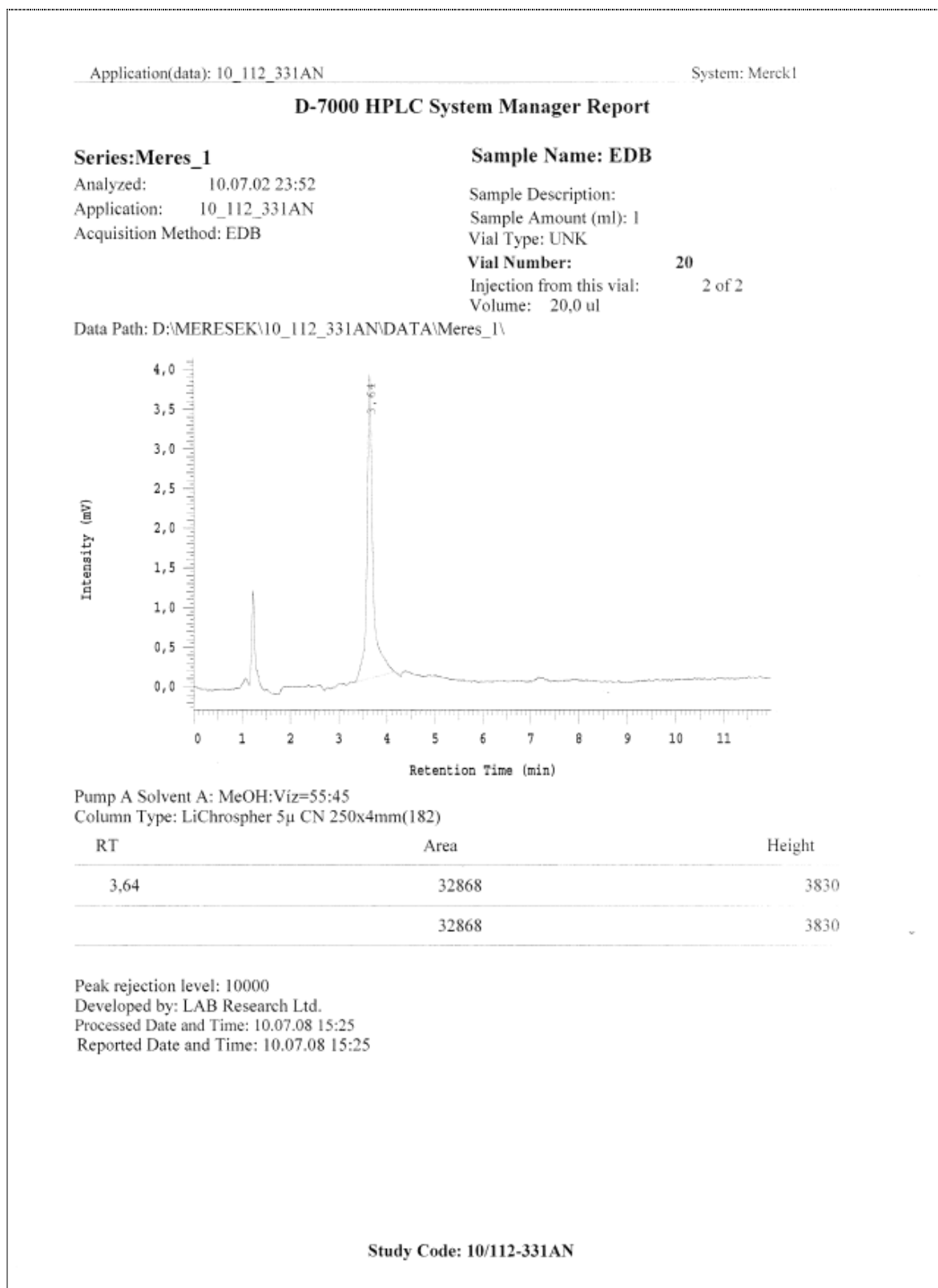
Developed by: LAB Research Ltd.

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



CHROMATOGRAM 9.
Chromatogram of Ethylene Dibromide Industrial (EDB)