A54025

Translation of Document No. A48639

Title

Endosulfan - emulsifiable concentrate - (352 g/l)
(Code: Hoe 002671 00 EC33 B317)

Investigation of the degradation behaviour in soil
under field conditions

(Step 2 in accordance with Guideline BBA Part IV, 4 - 1)

Authors

H. Baedelt, Dr. H. Idstein, B. Krebs

Study Completion Date

02 September 1992

Testing Facility

Hoechst Aktiengesellschaft
Produktentwicklung Ökologie II
Hessendamm 1 - 3
6234 Hattersheim

Study No.:

ER90DEU720

STATEMENT OF COMPLIANCE

This study was conducted in compliance with the Principles of Good Laboratory Practice.

Note:
In this study soil samples were temporarily stored for two months at 0 °C instead of at least -18 °C, as provided for in the Study Plan. A storage stability study, however, had shown that residues remain stable under these conditions for more than two months and therefore the results of the tests are not affected.

No other circumstances were observed which might have affected the quality and integrity of the presented study.

Study Director: H. Baedelt

Signature: - signed - Date : 2 Sep 1992

Residue Analysis: Dr. H. Idstein

Signature: - signed - Date : 2 Sep 1992

Head of the Testing Facility : B. Krebs

Signature: - signed - Date : 2 Sep 1992

GLP Statement

Hoechst Aktiengesellschaft
Referat GLP

30 July 1992

Title : Endosulfan - Emulsifiable Concentrate 352 g/l
(Code: Hoe 002671 00 EC33 B317

Study No. : ER90DEU720

This study was inspected at periodical intervals, and written and properly signed records were submitted to the Testing Facility Management and the Study Director as shown below :

Inspection	Report
30.04.1990	30.04.1990
04.05.1990	04.05.1990
31.07.1990	31.07.1990
04.03.1991	04.03.1991
30.07.1992	30.07.1992
30.07.1992	30.07.1992

Signed : (i.A. M. Glöckner)
30 Jul 1992

Referat GLP

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Report HVA 2/91
Project ER 90 DEU 720
Examination of the residues of endosulfan (Hoe 002671) and endosulfan-sulfate (Hoe 051327) in soil
Author: G. Fuchsbichler
Bayerische Hauptversuchsanstalt für Landwirtschaft
der TUM-Weihenstephan
D-8050 Freising 1

Enclosure: 2

Report HVA 7/91
Hoe 002671 (endosulfan) and Hoe 051327 (endosulfan-sulfate) storage stability in soil
Author: G. Fuchsbichler
Bayerische Hauptversuchsanstalt für Landwirtschaft
der TUM-Weihenstephan
D-8050 Freising 1

Summary:

The outdoor degradation of 1056 g/ha endosulfan was examined at 2 locations after application of 3.0 l/ha of an emulsifiable concentrate with 352 g/l active ingredient (Code: Hoe 002671 00 EC33 B317). The product was applied to the bare soil with a plot-sprayer in 600 l/ha spray liquid.

Soil samples were taken from a depth of 0 - 20 cm and 20 - 40 cm immediately before and after application of the test substance, as well as at 9 other dates.

The determination of the residues of endosulfan was performed by GC-ECD using method AL 60/86 of Hoechst AG. The samples were analysed for residues of alpha-endosulfan (Hoe 052618), beta-endosulfan (Hoe 052619) and endosulfan-sulfate (Hoe 051327).

The limit of quantification (LOQ) was 0.01 mg/kg.

The results of the investigations of the residues and of the regression calculations (DT_{50}- and DT_{90}-values) are summarised in the following tables. The regression calculations have been performed for the active ingredient (sum of alpha- and beta-endosulfan) and for the metabolite endosulfan-sulfate and are related to a soil column of 20 cm.

For the sum of both stereo-isomers DT_{50} -values of 38 days (ER90DEU7200101) and of 17 days (ER90DEU7200301) were determined. A degradation of 90 % (DT_{90}) was reached after 425 days and 182 days. In both studies alpha-endosulfan showed considerably more rapid degradation than beta-endosulfan.

The common metabolite of alpha- and beta-endosulfan, i.e. endosulfan-sulfate, reached the maximum residue level in trial ER90DEU7200101 after 7 days. After 201 days the metabolite had decreased to 50 % and after 669 days to 90 %. For assessment of the degradation rate it must be taken into account that the regression calculation represents the resultant of the formation of the metabolite and of its degradation, i.e. degradation would proceed much more rapidly without new formation of the metabolite.

In trial ER90DEU7200301 great variations in the metabolite concentrations were observed. The correlation ($r < 0.7$) was too low to permit an assessment of the degradation rate.

Results of the residue analyses (Hoe 002671 mg/kg)

Trial No.: ER90DEU7200101
Location: 2093 Stelle (type of soil: loamy sand)

Sampling - days after application	Depth of soil cm	alpha-endosulfan	beta-endosulfan	endosulfan-sulfate
before application	0 - 20	< 0,010	< 0,010	0,021
after 0	0 - 20	0,168	0,144	0,083
7	0 - 20	0,144	0,125	0,137
14	0 - 20	0,131	0,139	0,145
28	0 - 20	0,080	0,123	0,175
56	0 - 20	0,073	0,141	0,159
84	0 - 20	0,027	0,046	0,054
148	0 - 20	0,028	0,055	0,088
273	0 - 20	0,013	0,023	0,044
336	0 - 20	0,019	0,039	0,052
424	0 - 20	< 0,010	0,036	0,047
before application	20 - 40	< 0,010	< 0,010	< 0,010
after 0	20 - 40	-	-	-
7	20 - 40	< 0,010	< 0,010	0,045
14	20 - 40	< 0,010	< 0,010	0,014
28	20 - 40	< 0,010	< 0,010	0,013
56	20 - 40	< 0,010	< 0,010	0,018
84	20 - 40	< 0,010	< 0,010	< 0,010
148	20 - 40	< 0,010	< 0,010	0,016
273	20 - 40	< 0,010	< 0,010	< 0,010
336	20 - 40	< 0,010	< 0,010	< 0,010
424	20 - 40	< 0,010	< 0,010	< 0,010

Results of the regression calculation:

	alpha- and beta-endosulfan	endosulfan-sulfate
DT_{50} (T/2) :	38 days	201 days
DT_{90} (T/10) :	425 days	669 days
Function :	root first order	first order

For comments on the basis of calculations see chapter 9

Results of the residue analyses (Hoe 002671 mg/kg)

Trial No.: ER90DEU7200301
Location: 8906 Gersthofen (type of soil: sandy loam)

Sampling - days after application	depth of soil cm	alpha - endosulfan	beta-endosulfan	endosulfan-sulfate
before application	0 - 20	< 0,010	< 0,010	< 0,010
after 0	0 - 20	0,544	0,309	0,309
7	0 - 20	0,076	0,075	0,082
14	0 - 20	0,123	0,162	0,164
28	0 - 20	0,063	0,156	0,180
59	0 - 20	0,016	0,039	0,070
86	0 - 20	< 0,010	0,019	0,032
154	0 - 20	< 0,010	0,017	0,034
323	0 - 20	< 0,010	0,019	0,047
363	0 - 20	< 0,010	0,035	0,235
457	0 - 20	< 0,010	0,010	0,038
before application	20 - 40	< 0,010	< 0,010	< 0,010
after 0	20 - 40	-	-	-
7	20 - 40	< 0,010	< 0,010	< 0,010
14	20 - 40	< 0,010	< 0,010	< 0,010
28	20 - 40	< 0,010	< 0,010	< 0,010
59	20 - 40	< 0,010	< 0,010	< 0,010
86	20 - 40	< 0,010	< 0,010	< 0,010
154	20 - 40	< 0,010	< 0,010	< 0,010
323	20 - 40	< 0,010	< 0,010	< 0,010
363	20 - 40	< 0,010	< 0,010	< 0,010
457	20 - 40	< 0,010	< 0,010	< 0,010

Results of the regression calculation:

	alpha- and beta-endosulfan	endosulfan-sulfate
DT_{50} (T/2) :	17 days	could not be evaluated *
DT_{90} (T/10) :	182 days	
Function :	root first order	

* correlation too low (r = < 0.7)
For comments on the basis of calculations see chapter 9

1. Organisation and personnel

Sponsor :

Hoechst Aktiengesellschaft
Geschäftsbereich Landwirtschaft
Produktentwicklung
6230 Frankfurt am Main 80

Study Director:

H. Baedelt

for address: see test institute
telephone-No.: 06190/803242

Testing Facility :

Hoechst Aktiengesellschaft
Geschäftsbereich Landwirtschaft
Produktentwicklung - Ökologie II
Hessendamm 1 - 3
6234 Hattersheim

Head: B. Krebs

Co-operating locations and responsible staff-members:

- Performance of the field trial

Hoechst Aktiengesellschaft
Pflanzenschutzforschung - Freilandversuchswesen
Hessendamm 1 - 3
6234 Hattersheim

- responsible staff members appointed as assistants to the Study Director:

H. Eickhoff trial location north 2093 Stelle
E. Knobloch trial location south 8906 Gersthofen

- Residue analyses:

Bayerische Hauptversuchsanstalt für Landwirtschaft
der TUM - Weihenstephan
D-8050 Freising 1

- Head of Test of the Institute of Analytical Examination

Dr. G. Fuchsbichler

- Monitoring analyst of Hoechst Aktiengesellschaft:

Dr. H. Idstein
Hoechst Aktiengesellschaft, Bereich Landwirtschaft
Produktentwicklung - Ökologie II
Gebäude G864
6230 Frankfurt/Main 80

- Archiving

Study Plan, raw data, specimens and the original of the final report are filed at the Department Produktentwicklung for at least the period prescribed in the GLP Guidelines. Specimens are to be stored only as long as their quality admits assessment. Samples are destroyed in accordance with the draft "Technical Rule - Storing of Specimen and Samples" after completion of the final report.

- Quality Assurance

Pharm. S. J. Harston
Hoechst Aktiengesellschaft
Referat GLP Gebäude H 823
Postfach 80 03 20
6230 Frankfurt am Main 80

-Time schedule

Approval of the study by the Study Director :	25.04.1990
Start of the experimental phase :	23.05.1990
Termination of the experimental phase :	23.08.1991(field part)
	24.03.1992(analysis)
Preparation of final report :	02.09.1992

2. Aim of the Test

Endosulfan is an active ingredient for the control of chewing and sucking insects in agricultural and horticultural crops, fruit, hops, ornamental plants and forestry.

In soil degradation and metabolism studies of the active ingredient endosulfan (alpha- and beta-endosulfan) DT90 values of 40 - > 365 days had been determined. The BBA - Guideline part IV, 4 - 1 (1986) requires field trials if the DT90-values are greater than 100 days.(2).

This investigation of the degradation behaviour of endosulfan and its persistence in soil under field conditions fulfils the requirements of a soil degradation study as specified in Stufe 2 of the above guideline.

3. Test substance

Trade name	:	Thiodan 35 liquid
Code	:	Hoe 002671 00 EC33 B317
Way of formulation	:	emulsifiable concentrate
Active ingredient and content	:	endosulfan 352 g/l (nominal)
Certificate of Analysis	:	No.: AZ 04063
dated	:	25.04.1989
issued by	:	Produktentwicklung Ökologie I
Active ingredient and content	:	endosulfan 361 g/l (determined)
Chemical name of the a.i. (IUPAC)	:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3-oxide

The active ingredient is composed of the two stereoisomers alpha-endosulfan (64 - 67 %) and beta-endosulfan (29 - 32 %).

Stability under storage conditions

According to the Certificate of Analysis the storage stability is to be 24 months at 25 °C.
Expiry date: 21 Apr 1991

Stability under conditions of application

Stability under conditions of application is guaranteed within the framework of formulation development.

Details on the purity of the test substance can be found in the documentation added to the certificate of analysis.

4. Test system

The test system consisted of two outdoor residue trials at different locations on bare agricultural or horticultural soils.

4.1. Trial locations

Trial number ER90DEU720	trial location	height above sea-level (m)
0101	trial point north 2093 Stelle	8
0301	trial point south, Helmhof 8906 Gersthofen	469

The test substance had not been applied to the trial areas for at least a year before the start of the trial.

The following parameters were determined at the start of the trial for all trial locations:

Trial No.	ER90DEU7200101		ER90DEU7200301	
depths of soil (cm)	0 - 20	20 - 40	0 - 20	20 - 40
pH - values (In Kcl)	5,7	4,3	5,65	5,75
content of org. carbon (%)	2,02	1,68	1,27	0,77
biomass (mg C/100 g soil	14,24	0,28	38,39	14,63
KUK (mval/100 g soil)	7,56	7,19	10,75	10,63
content of dispersible particles in percent by weight < 0,02 mm (%)	10,69	12,20	39,17	43,26
clay < 0,002 mm (%)	7,04	7,80	18,66	21,89
0,002 - 0,02 mm (%)	3,65	4,40	20,51	21,37
silt 0,02 - 0,063 mm (%)	5,73	5,96	18,55	16,60
0,063 - 0,2 mm (%)	36,04	36,30	20,83	20,17
sand 0,2 - 2,0 mm (%)	47,53	45,53	21,45	19,97
Species of soil *	lS	lS	sL	sL

* lS = loamy sand
sL = sandy loam

4.2. Trial layout

The trials consisted each of a treated area and an untreated area of corresponding size for control samples. The size of the plots was governed by the number of samples. (For details see 5.1)

4.3 Cultivation of the trials

During the trial period the trial areas were kept free of weed mechanically and by hand-hoe. The soil was not tilled to a depth greater than 5 cm. The rests of the plants remained on the trial area. Additional irrigation was not performed in either of the trials.

4.4. Record of the meteorological conditions

Air temperature and precipitation (mm) were recorded during the test period and the results are presented in the appendix (see Table 10).

5. Application of the test substance

5.1. Essential details of application

The test substance is applied at a maximum application rate of 3.0 l/ha (corresponding to 1056 g a.i./ha). This amount of expenditure was also used in the study, i.e. to the treated plot the test substance was applied once.

trial No. ER90DEU720		0101	0301
size of plot [m²]	:	66	109
application	:	11.06.1990	23.05.1990
date of application	:		
water volume [l/ha]	:	600	600
air temperature [°C]	:	16	20
relative humidity [%]	:	78	52
speed of wind [m/sec]	:	2	1
direction of wind [°]	:	350	210
1st start of sampling after application [h]	:	3	3,5
rainfall after appl. [h]	:	24	24
amount mm	:	-	-

5.2. Application

The trial areas were ploughed before application and were prepared to a fine crume as for preparing a seed-bed.

The trial substance was applied with a plot sprayer (van der Weij) with a 2m wide spray-boom and flat fan nozzles. The water volume was 600 l/ha.

6. Sampling

6.1. Sampling for characterisation of the soil

Before the application of the test substance samples were taken with a soil sampler, consisting of at least 20 cores from a depth of 0 - 20 cm and 20 - 40 cm, distributed over the treated and the untreated trial area, for determination of the soil parameters referred to under 4.1.

6.2. Collecting of soil samples for residue analysis

At the start of the study - before and after application of the test substance - soil samples for residue analyses were taken. Another 9 samplings took place after application at the following times:

- 1 week after application
- 2 weeks after application
- 1 month after application
- 2 months after application
- approx. 3 months after application
- approx. 5 months after application
- in the following year at begin of vegetation and
- in May
- in August

The samples were taken from a depth of 0 - 20 cm and 20 - 40 cm each. Deeper soil layers were not taken for account in sampling, because no residues were expected to occur there.

For each of the soil samples at least 20 soil cores were removed either with an open soil corer („Pürckhauer" auger) or with a closed soil corer with plastic tubes. The samples were distributed at random over the trial area. To avoid later samplings on the same places, the sampling sites were distributed over so-called "small areas" within the field, each of which was to be sampled only once (or the sampling site was suitably markered).

7. Preparation of samples, packing, storage and transport

The cores were carefully combined and homogenised for each soil layer separately (about 2 kg per sample) and packed in batches of approx. 1 kg in 1-litre PE containers lined with aluminium foil and labelled. The samples were deep frozen on the day of sampling (at -18 °C and lower).

Where the samples were taken with an auger fitted with plastic liners, the soil-filled liners were at first deep frozen. Division of the two soil layers, 0 - 20 cm and 20 - 40 cm, and subsequent preparation of the soil samples were carried out at Hattersheim before dispatch to the analytical institute.

The transport from the trial locations to Produktentwicklung - Ökologie II, Hattersheim was performed in the deep frozen state with refrigeration vans, or by car in insulated containers. At the destination the samples also were deep frozen at - 18 °C or lower and stored in the cold-storage depot.

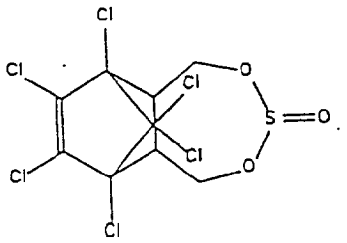
8. Residues analysis

The relevant residue of the tested substance Hoe 002671 00 EC33 B317 consists of the 2 stereo isomers alpha-endosulfan and beta-endosulfan and of the metabolite endosulfan-sulfate.

Hoe 052618 (alpha-endosulfan)

chemical name: 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3-oxide

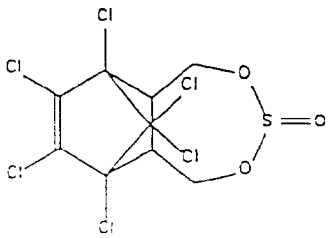
structural formula:



Hoe 052619 (beta-endosulfan)

chemical name: 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3-oxide

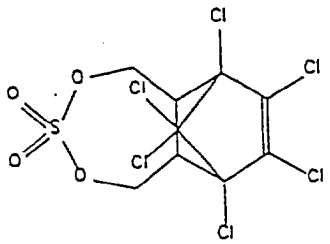
structural formula:



Hoe 051327 (endosulfan-sulfate)

chemical name: 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3,3-dioxide

structural formula:



8.1 Contract institute

The residues were analysed at the Bayerische Hauptversuchsanstalt für Landwirtschaft, Abteilung für Rückstandsanalytik, TUM-Weihenstephan in Freising. A GLP-attestation was issued to this institute a by the responsible supervising authority.

The monitoring analyst (see point 1 of this report) was appointed by the Sponsor as qualified contact person.

The analyses were performed at the contract institute under its Project No. HVA 2/91. The study plan was approved by the monitoring analyst and the study director. An original of this study plan is submitted as completion of the study plan to the raw data.

The samples were sent on 5 Feb 1991 and on 28 Oct 1991 in the deep frozen state with a refrigeration van to the contract institute.
In this study some of the soil samples were stored temporarily for two months at 0 °C, instead of at least -18 ° C as provided for in the study plan. During transport / delivery of the shipment of the samples of 5 Feb 1991 to the contract institute, the samples were placed in unscheduled intermediate storage at the transport company at 0 ° C. The delivery to the institute of analysis took place on 9 Apr 1991.

In an additional study it was investigated whether the stability of the residues of the test substance in soil decreases in storage at 0° C (see Appendix 2: Report HVA 7/91).

The performance of the analysis was inspected by the Unit for Quality Assurance of the contract institute and the compliance with the GLP - Guidelines was confirmed by this Unit in the appended final report of the contract institute. Additionally the monitoring analyst convinced himself of the quality of the analytical results on the basis of copies of all calculation sheets as well as of random test controls of chromatograms of untreated, treated and spiked samples.

8.2 Method of testing

The samples were examined for alpha-endosulfan (Hoe 052618), beta-endosulfan (Hoe 052619) and endosulfan-sulfate (Hoe 051327) by method AL 60/86 (3).

The determination of the residues was carried out by gas chromatography using an electron capture detector. The chromatographic conditions in method AL 60/86 (3) were modified at the contract institute for the chromatographic system used there.

There was no correction with recoveries. A short description of the method and the modification is given in the final report of the contract institute.

8.3 Final report of the contract institute

The results of the analytical procedure, including relevant dates for working up and measurement, are given in the final report of the contract institute (Appendix 1 Report HVA 2/91).

As an internal quality assurance for each sample material, this report also contains analytical results for untreated samples and details on recoveries in spiked untreated samples (Table 1). These correspond with the results obtained for the stated sample materials in a method validation conducted within the framework of the storage stability study (Appendix 2, Report HVA 7/91).

The lowest spiking level also defined the lower limit of the practical working range (5) for this analytical series which is used in this report synonymously with the term "determination limit".

9. Results

The results of the residue analyses are listed in Table 2 for the trial ER90DEU7200101 and in Table 3 for ER90DEU7200301. The residue substances alpha-endosulfan, beta-endosulfan as well as endosulfan-sulfate are given as equimolar mass of the reference substance endosulfan in mg/kg soil; The values refer to air-dried soil and are given for the soil layers 0 - 20 cm and 20 - 40 cm separately.

Analytical results below the determination limit were given as "< 0.01 mg/kg", the value "0.01 mg/kg" being the lower limit of the practical working range for the matrix in question.

A soil layer in which no residues are detected at any time is not taken in account for further evaluation (regression calculations).

Where the residues of one substance were distributed over several soil layers, the residue total for the separate soil layers (i.e. standardised to a 20 cm soil column) was obtained by the following procedure:

If the determination limit was exceeded at any measurement time in any soil layer, a value of half the determination limit was entered for this soil layer before and after the event and where necessary added to the value for the other soil layer. The other values below the determination limit were not taken into account for this calculation.

The isomers of the active ingredient (alpha-endosulfan and beta-endosulfan), standardised to the 20 cm soil column, were then totalled separately (see Tables 4 and 5).

The degradation kinetics of endosulfan were determined by the method of Timme et al. 1986 (7). These values were calculated with a PC evaluation program in the spreadsheet software Lotus 1-2-3 Version 2.1. The optimisation criterion was the minimum of the sum of the square of distances of the measuring points from the calculated kinetic function after reconversion to the original co-ordinates.

The evaluations for the active ingredient (sum of alpha- and beta-endosulfan) and of the metabolite endosulfan-sulfate are given in Table 6 -9. The graphical representations of the regression curve are shown in Figures 1 - 4.

The calculations of regression with the values corrected as explained above (Table 4 and 5) start off in each case with the significant maximum residue, i.e. for the initial residues of alpha and beta endosulfan in trials ER90 DEU 7200101 and ER90 DEU 7200301 on the day of application .

The residue maximum for the metabolite endosulfan-sulfate in trial ER90 DEU 7200101 was found on day 7. As the concentration values in trial ER90 DEU 7200301 varied too greatly and thus only permitted correlations at $r < 0.7$ to standard kinetical functions, it was not possible to calculate either the DT_{50} or the DT_{90} (Table 9).

The DT_{50} and DT_{90} results from the regression evaluations were rounded up or down to whole days.

10. Discussion

The test substance was applied at the maximum application rate of 3.0 l/ha recommended only for treatment of hops, which corresponds to 1056 g a.i./ha on bare fine-crume soil.

No endosulfan had been applied to these soils during the two years before the start of the study.

Analysis of the samples on day zero on location Stelle ER90DEU7200101 (loamy sand) before application in the treatment area, and after application in the untreated plot, yielded endosulfan-sulfate residues just above the determination limit (0.021 mg/kg and 0.015 mg/kg) in the 0 - 20 cm soil layer. In the untreated plot, endosulfan-sulfate was detectable at levels of 0.014 and 0.023 mg/kg after 7 and 14 days respectively. Beta-endosulfan was detected once at 0.011 mg/kg (day 14), see Table 2. During the further course of the study the untreated examined samples from this location remained below the limit of the practical working range.

As the residue results for the day 0 samples prior to application show, the soil of ER90DEU7200301 contained no residues of endosulfan before application (see Table 3).

Assuming a soil density of 1.5 kg/m^3 and a sampling depth of 20 cm, the theoretical initial concentration is calculated to be 0.352 mg endosulfan/kg soil. The experimentally determined initial concentrations were 0.312 mg/kg (ER90DEU7200101) and 0.853 mg/kg (ER90DEU7200301). In the latter trial a.i. residues of 0.151 mg/kg and 0.285 mg/kg were determined 7 and 14 days after application respectively.

The difference between the analytically determined residue and the theoretically calculated residue can be explained if it is taken into account that soil sampling is subject to considerable uncertainties, especially if the sampling takes place a relatively short time after application.

There are:

+ a soil density deviating from 1.5 kg/dm^3, in particular in the case of a loose seed bed which has been prepared a relatively short time before,

+ formation of a funnel around the sampling site and sinking down of the soil column in the sampler during removal (turning),

+ inaccuracies with regard to depth of removal, and

+ insufficient homogeneity of the soil sample, in particular if the contamination of soil particles from the surface and 20 cm soil depth differs considerably.

Without influence on the results of the study, however, was the 2-month intermediate storage of the soil-samples at 0 °C. A 3-month storage stability study at 0 °C (enclosure 2 HVA 7/91) showed that no degradation of alpha-endosulfan, beta-endosulfan and endosulfan-sulfate took place during the first two months. Levels between 83 % and 117 % of the initial concentration (0.1 mg/kg) were measured. Recoveries after 3 months were 44 % for alpha-endosulfan, 67 % for beta-endosulfan and 70 % for endosulfan-sulfate.

Graphs showing the degradation of the active ingredient (alpha- and beta-endosulfan) and the metabolite (endosulfan-sulfate) are given in Figures 1 - 4.

DT_{50}-values for the sum of the two stereo-isomers of the active ingredient were 38 days (ER90DEU7200101) and 17 days (ER90DEU7200301). A degradation of 90 % (DT_{90}) was reached after 425 days and 182 days. In both trials alpha-endosulfan showed a distinctly faster degradation than beta-endosulfan. A transportation of alpha- and beta-endosulfan to deeper layers of soil (20 - 40 cm) was not observed.

The common metabolite of alpha- and beta-endosulfan, i.e. endosulfan-sulfate, reached the residue-maximum in trial ER90DEU7200101 after 7 days. After another 201 days the metabolite had decreased by 50 % and after 669 days by 90 %. When evaluating the degradation rate of the metabolite it must be taken into account that the regression calculation shows the resultant of metabolite formation and degradation, i.e. degradation without formation of the metabolite would proceed much more rapidly.

In ER90DEU7200101 endosulfan-sulfate was determined in the soil-layer 20 - 40 cm in the time between days 7 and 148 after application (maximum day 7: 0.045 mg/kg, by day 148: 0.013 - 0.018 mg/kg). In the course of the additional samplings this soil layer was free of residues. Since the soil was regularly tilled, this might have resulted in a certain transportation of endosulfan-sulfate.

In trial ER90DEU7200301 major variations in the concentrations of the metabolite were observed, whereas no residues were found in the 20 - 40 cm soil layer. Already on day 0 after application a level of 0.309 mg/kg was found, which cannot be accounted for. A second excessive residue level of 0.235 mg/kg on day 363 after application cannot be attributed to different rates of formation and degradation of the metabolite in the course of the trial.

It is not possible to calculate a degradation in this study since the correlations were too low (r = < 0.7), and thus an optimum adaptation function cannot be selected.

Hoechst Aktiengesellschaft
Produktentwicklung Ökologie II

Authors:	2 Sep 1992	H. Baedelt	- signed -
	2 Sep 1992	Dr. H. Idstein	- signed -
	4 Sep 1992	B. Krebs	- signed -
Study Director:	2 Sep 1992	H. Baedelt	- signed -
Head of the Test Facility:	4 Sep 1992	B. Krebs	- signed -
Examined by Referat GLP:	30 Jul 1992	M. Glöckner	- signed -

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Januar 1990

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Table 1

Recoveries and limits of quantification (LOQ) of alpha-endosulfan, beta-endosulfan and endosulfan-sulfate ER90DEU720

active ingredient	Fortification mg/kg	recoveries mg/kg	%	LOQ mg/kg
alpha-endosulfan	0,5	0,459 /0,447	92 / 89	0,01
	0,1	0,093 /0,104	93 /104	
	0,05	0,049 /0,0495	98 / 99	
	0,02	0,0196/0,0183	98 / 92	
	0,02	0,0179	90	
	0,01	0,0096/0,0073	97 / 73	
Average recovery	:		93	
standard deviation s	:		8,1	
beta-endosulfan	0,5	0,449 /0,459	90 / 92	0,01
	0,1	0,088 /0,087	88*/ 87	
	0,05	0,0478/0,043	96 / 86	
	0,02	0,0208/0,015	104 / 75	
	0,01	0,0093/0,083	93 / 93	
Average recovery	:		90	
standard deviation s	:		7,5	
endosulfan-sulfate	0,5	0,522 /0,597	104*/119	0,01
	0,1	0,081 /0,085	81*/ 85	
	0,05	0,06 /0,0544	120 /109	
	0,02	0,0177/0,0228	89 /114	
	0,01	0,0088/0,104	88 /104	
Average recovery	:		101	
standard deviation s	:		14,5	

* corrected by blank values of control (Table 2)

Table 2 Results of the residue analyses of soil degradation
a) treated samples from 0 - 20 cm soil depth

Trial location : 2093 Stelle
Trial No. : ER90DEU7200101

Soil identification : 0 - 20 cm
Soil texture : ls
% org.C : 2.02
pH - value : 5.7
Suspended particles < 0.02 mm (%) : 10.69

Analysed substance(s) alpha-endosulfan (Hoe 052618), beta-endosulfan (Hoe 052619) and endosulfan-sulfate (Hoe 051327)
Analytical method AL60/86
Lower limit of determination 0.01 mg/kg
Recovery see Table 1
Investigating Laboratory: Bavarian Central Institute of Agriculture: of the TUM-Weihenstephan, Freising

Results of residue analyses (mg/kg) calculated as endosulfan (Hoe 002671)

date of *sampling*	days after *application*	soil depth *(cm)*	analytical sample *(g)*	alpha-*endosulfan*	beta-*endosulfan*	endosulfan-*sulphate*
11.06.90 *	before appl.	0 - 20	10	< 0,010	< 0,010	0,021
11.06.90 *	0	0 - 20	10	0,168	0,144	0,083
18.06.90 *	7	0 - 20	10	0,144	0,125	0,137
25.06.90 *	14	0 - 20	10	0,131	0,139	0,145
09.07.90 *	28	0 - 20	10	0,080	0,123	0,175
06.08.90 *	56	0 - 20	10	0,073	0,141	0,159
03.09.90 *	84	0 - 20	10	0,027	0,046	0,054
06.11.90 *	148	0 - 20	10	0,028	0,055	0,088
15.03.91	273	0 - 20	10	0,013	0,023	0,044
13.05.91	336	0 - 20	10	0,019	0,039	0,052
09.08.91	424	0 - 20	10	< 0,01	0,036	0,047

* intermediate storage at 0 °C in the period of 02/05/1991 - 04/09/1991

Table 2 (continued) Results of the residue analyses of soil degradation
b) treated samples 20 - 40 cm soil depth
Trial location : 2093 Stelle
Trial No. : ER90DEU7200101

Soil identification	: 20 - 40 cm	**Analysed substance(s)**	alpha-endosulfan (Hoe 052618), beta-endosulfan (Hoe 052619) and endosulfan-sulfate (Hoe 051327)
Soil texture	: ls		
% org.C	: 1.68	**Analytical method**	AL60/86
pH - value	: 4.3	**Lower limit of determination**	0.01 mg/kg
Suspended particles		**Recovery**	see Table 1
< 0.02 mm (%)	: 12.2	**Investigating Laboratory**:	Bavarian Central Institute of Agriculture: of the TUM-Weihenstephan, Freising

Results of residue analyses (mg/kg) calculated as endosulfan (Hoe 002671)

date of sampling	days after application	soil depth (cm)	analytical sample (g)	alpha-endosulfan	beta-endosulfan	endosulfan-sulphate
11.06.90 *	before appl.	20 - 40	10	< 0,01	< 0,01	< 0,01
11.06.90 *	0	20 - 40	10	-	-	-
18.06.90 *	7	20 - 40	10	< 0,01	< 0,01	0,045
25.06.90 *	14	20 - 40	10	< 0,01	< 0,01	0,014
09.07.90 *	28	20 - 40	10	< 0,01	< 0,01	0,013
06.08.90 *	56	20 - 40	10	< 0,01	< 0,01	0,018
03.09.90 *	84	20 - 40	10	< 0,01	< 0,01	< 0,01
06.11.90 *	148	20 - 40	10	< 0,01	< 0,01	0,016
15.03.91	273	20 - 40	10	< 0,01	< 0,01	< 0,01
13.05.91	336	20 - 40	10	< 0,01	< 0,01	< 0,01
09.08.91	424	20 - 40	10	< 0,01	< 0,01	< 0,01

* intermediate storage at 0 °C in the period of 02/05/1991 - 04/09/1991

Table 2 (Continued) Results of the residue analyses of soil degradation
c) - untreated samples - 0 - 20 cm and 20 - 40 cm soil depth
Trial location : 2093 Stelle
Trial No. : ER90DEU7200101

Soil identification	: 0 - 20 / 20 - 40 cm	**Analysed substance(s)**	alpha-endosulfan (Hoe 052618), beta-endosulfan (Hoe 052619) and endosulfan-sulfate (Hoe 051327)
Soil texture	: ls		
% org.C	: 2.02 / 1.68	**Analytical method**	AL60/86
pH - value	: 5.7 / 4.3	**Lower limit of determination**	0.01 mg/kg
Suspended particles		**Recovery**	see Table 1
< 0.02 mm (%)	: 10.69 / 12.20	**Investigating Laboratory**:	Bavarian Central Institute of Agriculture: of the TUM-Weihenstephan, Freising

Results of residue analyses (mg/kg) calculated as endosulfan (Hoe 002671)

date of sampling	days after application	soil depths (cm)	analytical sample (g)	alpha-endosulfan	beta-endosulfan	endosulfan-sulphate
11.06.90 *	0	0 - 20	10	< 0,01	< 0,01	0,015
18.06.90 *	7	0 - 20	10	< 0,01	< 0,01	0,014
25.06.90 *	14	0 - 20	10	< 0,01	0,011	0,023
09.08.91	424	0 - 20	10	< 0,01	< 0,01	< 0,01
11.06.90 *	0	20 - 40	10	< 0,01	< 0,01	< 0,01
18.06.90 *	7	20 - 40	10	< 0,01	< 0,01	< 0,01
25.06.90 *	148	20 - 40	10	< 0,01	< 0,01	< 0,01
11.03.91	273	20 - 40	10	< 0,01	< 0,01	< 0,01

When calculating the residual values and the recoveries, eventually existing blank values were considered.
* intermediate storage at 0 °C in the period of 02/05/1991 - 04/09/1991

Table 3 Results of the residue analyses of soil degradation
a) treated samples from 0 - 20 cm soil depth

Trial location :8906 Gersthofen
Trial No. :ER90DEU7200301

Soil identification	: 0 - 20 cm	**Analysed substance(s)**	alpha-endosulfan (Hoe 052618), beta-endosulfan (Hoe 052619) and endosulfan-sulfate (Hoe 051327)
Soil texture	: sL		
% org.C	: 1.27	**Analytical method**	AL60/86
pH - value	: 5.65	**Lower limit of determination**	0.01 mg/kg
Suspended particles		**Recovery**	see Table 1
< 0.02 mm (%)	: 39.17	**Investigating Laboratory**:	Bavarian Central Institute of Agriculture: of the TUM-Weihenstephan, Freising

Results of residue analyses (mg/kg) calculated as endosulfan (Hoe 002671)

date of sampling	days after application	soil depths (cm)	analytical sample (g)	alpha-endosulfan	beta-endosulfan	endosulfan-sulphate
22.05.90 *	before appl.	0 - 20	10	< 0,01	< 0,01	< 0,01
23.05.90 *	0	0 - 20	10	0,544	0,309	0,309
30.05.90 *	7	0 - 20	10	0,076	0,075	0,082
06.06.90 *	14	0 - 20	10	0,123	0,162	0,164
20.06.90 *	28	0 - 20	10	0,063	0,156	0,180
20.07.90 *	59	0 - 20	10	0,016	0,039	0,07
16.08.90 *	86	0 - 20	10	<0,01	0,019	0,032
23.10.90 *	154	0 - 20	10	<0,01	0,017	0,034
21.03.91	323	0 - 20	10	<0,01	0,019	0,047
21.05.91	363	0 - 20	10	<0,01	0,035	0,235
23.08.91	457	0 - 20	10	<0,01	0,010	0,038

Table 3 (Continued) Results of the residueanalyses of soil degradation
b) treated samples from 20 - 40 cm soil depth
Trial location : 8906 Gersthofen
Trial No. : ER90DEU7200301

Soil identification : 20 - 40 cm
Soil texture : suL
% org.C : 0.93
pH - value : 5.3
Suspended particles < 0.02 mm (%) : 35.4

Analysed substance(s) alpha-endosulfan (Hoe 052618), beta-endosulfan (Hoe 052619) and endosulfan-sulfate (Hoe 051327)
Analytical method AL60/86
Lower limit of determination 0.01 mg/kg
Recovery see Table 1
Investigating Laboratory: Bavarian Central Institute of Agriculture: of the TUM-Weihenstephan, Freising

Results of residue analyses (mg/kg) calculated as endosulfan (Hoe 002671)

date of sampling	days after application	soil depths (cm)	analytical sample (g)	alpha-endosulfan	beta-endosulfan	endosulfan-sulphate
22.05.90 *	before appl.	20 - 40	10	< 0,01	< 0,01	< 0,01
23.05.90 *	0	20 - 40	10	-	-	-
30.05.90 *	7	20 - 40	10	< 0,01	< 0,01	< 0,01
06.06.90 *	14	20 - 40	10	< 0,01	< 0,01	< 0,01
20.06.90 *	28	20 - 40	10	< 0,01	< 0,01	< 0,01
20.07.90 *	59	20 - 40	10	< 0,01	< 0,01	< 0,01
16.08.90 *	86	20 - 40	10	< 0,01	< 0,01	< 0,01
23.10.90 *	154	20 - 40	10	< 0,01	< 0,01	< 0,01
21.03.91	323	20 - 40	10	< 0,01	< 0,01	< 0,01
21.05.91	363	20 - 40	10	< 0,01	< 0,01	< 0,01
23.08.91	457	20 - 40	10	< 0,01	< 0,01	< 0,01

* intermediate storage at 0 °C in the period of 02/05/1991 - 04/09/1991

Table 4 Corrected values for regression calculation

Trial location : 2093 Stelle
Trial No. : ER90DEU7200101

Results of residue analyses (mg/kg) calculated as endosulfan (Hoe 002671)
soil layers 0 - 20 cm and 20 - 40 cm, and standardisation to 0 - 20 cm soil column (0 - 20 cm)

Days after application / soil depth cm	alpha - endosulfan			beta - endosulfan			alpha - and beta - endosulfan	endosulfan sulphate		
	0 - 20	20 - 40	(0 - 20)	0 - 20	20 - 40	(0 - 20)	(0 - 20)	0 - 20	20 - 40	(0 - 20)
before application	0,005	0	0,005	0,005	0	0,005	0,010	0,021	0,005	0,026
0	0,168	-	0,168	0,144	-	0,144	0,312	0,083	-	0,083
7	0,144	0	0,144	0,125	0	0,125	0,269	0,137	0,045	0,182
14	0,131	0	0,131	0,139	0	0,139	0,270	0,145	0,014	0,159
28	0,080	0	0,080	0,123	0	0,123	0,203	0,175	0,013	0,188
56	0,073	0	0,073	0,141	0	0,141	0,214	0,159	0,018	0,177
84	0,027	0	0,027	0,046	0	0,046	0,073	0,054	0,005	0,059
148	0,028	0	0,028	0,055	0	0,055	0,083	0,088	0,016	0,104
273	0,013	0	0,013	0,023	0	0,023	0,036	0,044	0,005	0,049
336	0,019	0	0,019	0,039	0	0,039	0,058	0,052	0	0,052
424	0,005	0	0,005	0,036	0	0,036	0,041	0,047	0	0,047

Values below the limit of quantification (LOQ) of 0.01 mg/kg are either filled in with half the LOQ, or they were not included and not taken into account for evaluation (0). For explanations see Section 9

The regression calculation was carried out for the standardised soil column (0 - 20 cm) with alpha- and beta- endosulfan and endosulfan-sulfate.

Table 5 Corrected values for regression calculation

Trial location : 8906 Gersthofen
Trial No. : ER90DEU7200301

Results of residue analyses (mg/kg) calculated as endosulfan (Hoe 002671)
(Distribution over the soil layers 0 - 20 cm and 20 - 40 cm, and standardisation to 0 - 20 cm soil column)

Days after application / soil depth cm	alpha - endosulfan			beta - endosulfan			alpha - and beta - endosulfan	endosulfan sulphate		
	0 - 20	20 - 40	(0 - 20)	0 - 20	20 - 40	(0 - 20)	(0 - 20)	0 - 20	20 - 40	(0 - 20)
before application	0,005	0	0,005	0,005	0	0,005	0,010	0,005	0	0,005
0	0,544	-	0,544	0,309	-	0,309	0,853	0,309	-	0,309
7	0,076	0	0,076	0,075	0	0,075	0,151	0,082	0	0,082
14	0,123	0	0,123	0,162	0	0,162	0,285	0,164	0	0,164
28	0,063	0	0,063	0,156	0	0,156	0,219	0,180	0	0,180
59	0,016	0	0,016	0,039	0	0,039	0,055	0,070	0	0,070
86	0,005	0	0,005	0,019	0	0,019	0,024	0,032	0	0,032
154	0	0	0	0,017	0	0,017	0,017	0,034	0	0,034
323	0	0	0	0,019	0	0,019	0,019	0,047	0	0,047
363	0	0	0	0,035	0	0,035	0,035	0,235	0	0,235
457	0	0	0	0,010	0	0,010	0,010	0,038	0	0,038

Values below the limit of quantification (LOQ) of 0.01 mg/kg are either filled in with half the LOQ, or they were not included and not taken into account for evaluation (0). For explanations see Section 9

The regression calculation was carried out for the standardised soil column (0 - 20 cm) with alpha- and beta- endosulfan and endosulfan-sulfate.

Table 6

Results of the regression calculations by Timme, et al., 1986 (7) for
Trial No.: ER90DEU7200101
- alpha- endosulfan (Hoe 052618) + beta- endosulfan (Hoe 052619) in soil -

Start of regression calculations on day 0

Initial concentration: [mg/kg]

time [days]	residue [%]	[mg/kg]
0	———	0,312
7	———	0,269
14	———	0,270
28	———	0,203
56	———	0,214
84	———	0,073
148	———	0,083
273	———	0,036
336	———	0,058
424	———	0,041

EVALUATION

Fit		Sum of least squares	Coefficient of correlation r		DT-50 [days]	DT-90 [days]
1st order		1,56E−02	−0,8904		142,5	473,5
1.5th order		1,57E−02	0,9019		111,4	581,6
2nd order		1,82E−02	0,8970		79,7	717,3
Root 1st order	Min.	1,13E−02	−0,9408	Max.	38,5	424,6
Root 1.5th order		1,48E−02	0,9316		12,3	336,2
Root 2nd order		2,07E−02	0,9083		0,1	5,0

Regression 1st order

	a1 =	−0,0049	
	a0 =	−1,4733	
r	^2 =	0,7927	
DT−	50 =	142,5	[days]
DT−	90 =	473,5	[days]

Regression root 1st order

	a1 =	−0,1117	
	a0 =	−1,0674	
r	^2 =	0,8851	
DT−	50 =	38,5	[days]
DT−	90 =	424,6	[days]

Regression 1.5th order

	a1 =	0,0078	
	a0 =	2,0908	
r	^2 =	0,8135	
DT−	50 =	111,4	[days]
DT−	90 =	581,6	[days]

Regression root 1.5th order

	a1 =	0,1746	
	a0 =	1,4806	
r	^2 =	0,8679	
DT−	50 =	12,3	[days]
DT−	90 =	336,2	[days]

Regression 2nd order

	a1 =	0,0532	
	a0 =	4,2435	
r	^2 =	0,8047	
DT−	50 =	79,7	[days]
DT−	90 =	717,3	[days]

Regression root 2nd order

	a1 =	1,1725	
	a0 =	0,2899	
r	^2 =	0,8250	
DT−	50 =	0,1	[days]
DT−	90 =	5,0	[days]

Table 7

Results of the regression calculations by Timme, et al., 1986 (7) for
Trial No.: ER90DEU7200101
- endosulfan sulphate (Hoe 051327) in soil -

Start of regression calculations on day 7

Initial concentration: [mg/kg]

time [days]	residue [%]	[mg/kg]
0	———	0,182
7	———	0,159
21	———	0,188
49	———	0,177
77	———	0,059
141	———	0,104
266	———	0,049
329	———	0,052
417	———	0,047

EVALUATION

Fit		Sum of least squares	Coefficient of correlation r	DT-50 [days]	DT-90 [days]
1st order	Min.	7,87E−03	−0,8601	201,4	669,0
1.5th order		8,12E−03	0,8719	180,8	943,6
2nd order		8,94E−03	0,8822	159,3	1433,9
Root 1st order		8,14E−03	−0,8858	82,2	906,6
Root 1.5th order		9,42E−03	0,8883	46,7	1271,3
Root 2nd order		1,13E−02	0,8893 Max.	16,3	1317,2

Regression 1st order

a1 = −0,0034
a0 = −1,8412
r ^2 = 0,7398
DT− 50 = 201,4 [days]
DT− 90 = 669,0 [days]

Regression root 1st order

a1 = −0,0765
a0 = −1,5785
r ^2 = 0,7846
DT− 50 = 82,2 [days]
DT− 90 = 906,6 [days]

Regression 1.5th order

a1 = 0,0058
a0 = 2,5234
r ^2 = 0,7602
DT− 50 = 180,8 [days]
DT− 90 = 943,6 [days]

Regression root 1.5th order

a1 = 0,1271
a0 = 2,0958
r ^2 = 0,7891
DT− 50 = 46,7 [days]
DT− 90 = 1271,3 [days]

Regression 2nd order

a1 = 0,0402
a0 = 6,4076
r ^2 = 0,7783
DT− 50 = 159,3 [days]
DT− 90 = 1433,9 [days]

Regression root 2nd order

a1 = 0,8746
a0 = 3,5269
r ^2 = 0,7908
DT− 50 = 16,3 [days]
DT− 90 = 1317,2 [days]

Table 8

Results of the regression calculations by Timme, et al., 1986 (7) for
Trial No.: ER90DEU7200301
- alpha- endosulfan (Hoe 052618) + beta- endosulfan (Hoe 052619) in soil -

Start of regression calculations on day 0

Initial concentration: [mg/kg]

time [days]	residue [%]	[mg/kg]
0	———	0,853
7	———	0,151
14	———	0,285
28	———	0,219
59	———	0,055
86	———	0,024
154	———	0,017
323	———	0,019
363	———	0,035
457	———	0,010

EVALUATION

Fit		Sum of least squares	Coefficient of correlation r		DT-50 [days]	DT-90 [days]
1st order		3,56E−02	−0,7639		105,2	349,5
1.5th order		4,86E−02	0,8171		79,3	413,7
2nd order		6,33E−02	0,8182		55,1	496,0
Root 1st order	Min.	2,90E−02	−0,8730		16,5	181,8
Root 1.5th order		4,58E−02	0,8790	Max.	2,8	75,1
Root 2nd order		6,40E−02	0,8360		Polstelle	lle

Regression 1st order

	a1 =	−0,0066
	a0 =	−1,7625
r	^2 =	0,5835
DT−	50 =	105,2 [days]
DT−	90 =	349,5 [days]

Regression root 1st order

	a1 =	−0,1708
	a0 =	−1,0465
r	^2 =	0,7621
DT−	50 =	16,5 [days]
DT−	90 =	181,8 [days]

Regression 1.5th order

	a1 =	0,0143
	a0 =	2,7345
r	^2 =	0,6676
DT−	50 =	79,3 [days]
DT−	90 =	413,7 [days]

Regression root 1.5th order

	a1 =	0,3487
	a0 =	1,3979
r	^2 =	0,7726
DT−	50 =	2,8 [days]
DT−	90 =	75,1 [days]

Regression 2nd order

	a1 =	0,1546
	a0 =	3,5208
r	^2 =	0,6695
DT−	50 =	55,1 [days]
DT−	90 =	496,0 [days]

Regression root 2nd order

	a1 =	3,5831
	a0 =	−4,0581
r	^2 =	0,6989
DT−	50 =	1,3 [days]
DT−	90 =	103,9 [days]

Table 9

Results of the regression calculations by Timme, et al., 1986 (7) for
Trial No.: ER90DEU7200301
- endosulfan sulphate (Hoe 051327) in soil -

Start of regression calculations on day 28

Initial concentration: [mg/kg]

time [days]	residue [%]	[mg/kg]
0	____	0,180
31	____	0,070
58	____	0,032
126	____	0,034
295	____	0,047
335	____	0,235
429	____	0,038

EVALUATION

Fit		Sum of least squares	Coefficient of correlation r		DT-50 [days]	DT-90 [days]
1st order	Min.	3,27E−02	−0,0543		2643,5	8781,5
1.5th order		3,40E−02	0,0731		2664,1	13907,3
2nd order		3,52E−02	0,0769		3569,3	32124,1
Root 1st order		3,46E−02	−0,1952		1089,0	12016,9
Root 1.5th order		3,55E−02	0,2172		1339,8	36511,0
Root 2nd order		3,64E−02	0,2220	Max.	2191,3	177496,9

Regression 1st order

	a1 =	−0,0003
	a0 =	−2,6628
r	^2 =	0,0029
DT−	50 =	2643,5 [days]
DT−	90 =	8781,5 [days]

Regression root 1st order

	a1 =	−0,0210
	a0 =	−2,4686
r	^2 =	0,0381
DT−	50 =	1089,0 [days]
DT−	90 =	12016,9 [days]

Regression 1.5th order

	a1 =	0,0006
	a0 =	4,0227
r	^2 =	0,0053
DT−	50 =	2664,1 [days]
DT−	90 =	13907,3 [days]

Regression root 1.5th order

	a1 =	0,0414
	a0 =	3,6597
r	^2 =	0,0472
DT−	50 =	1339,8 [days]
DT−	90 =	36511,0 [days]

Regression 2nd order

	a1 =	0,0050
	a0 =	17,9899
r	^2 =	0,0059
DT−	50 =	3569,3 [days]
DT−	90 =	32124,1 [days]

Regression root 2nd order

	a1 =	0,3242
	a0 =	15,1747
r	^2 =	0,0493
DT−	50 =	2191,3 [days]
DT−	90 =	177496,9 [days]

Table 10 Recording of meteorological conditions 1990/1991

Trial No. : ER90DEU7200101
Trial location : 2093 Stelle
Site of weather station: at trial location

Time	Treatment (B) Sampling (P)	Mean temperature (°C)	Precipitation (mm)
01.6.90		17,0	-
02.6.90		15,5	-
03.6.90		15,0	11,0
04.6.90		13,0	5,0
05.6.90		15,5	22,0
06.6.90		15,5	1,0
07.6.90		15,0	2,0
08.6.90		15,0	12,0
09.6.90		13,0	11,0
10.6.90		16,0	5,0
11.6.90	BP	18,5	-
12.6.90		15,5	-
13.6.90		13,5	-
14.6.90		13,0	-
15.6.90		12,5	-
16.6.90		13,5	-
17.6.90		14,5	-
18.6.90	P	14,5	-
19.6.90		19,0	-
20.6.90		17,0	6,0
21.6.90		20,5	7,0
22.6.90		19,0	-
23.6.90		18,0	7,0
24.6.90		17,5	7,0
25.6.90	P	16,5	1,0
26.6.90		24,0	-
27.6.90		18,5	-
28.6.90		21,0	7,0
29.6.90		20,5	-
30.6.90		22,0	6,0
Juni 90		16,7	110,0

Table 10 (Continued)

Trial No. : ER90DEU7200101
Trial location : 2093 Stelle
Site of weather station: at trial location

Time	Treatment (B) Sampling (P)	Mean temperature (°C)	Precipitation (mm)
01.07.90		20,0	2,0
02.07.90		19,0	1,0
03.07.90		16,5	2,0
04.07.90		17,0	3,0
05.07.90		16,0	-
06.07.90		12,5	7,0
07.07.90		16,5	18,0
08.07.90		15,0	-
09.07.90	P	18,5	1,0
Juli 90		18,7	42,0
06.08.90	P	14,5	-
August 90		17,3	74,0
03.09.90	P	16,5	1,0
September 90		12,2	120,2
Oktober 90		10,2	54,0
06.11.90	P	1,0	1,0
November 90		4,6	112,0
Dezember 90		1,8	71,0
Januar 91		1,9	61,0
Februar 91		-2,7	30,0
15.03.91	P	8,0	0,5
März 91		6,0	15,5
April 91		6,5	59,0
13.05.91	P	9,5	-
Mai 91		9,5	43,0
Juni 91		13,1	139,0
Juli 91		18,9	86,0
09.08.91	P	16,5	7,0
August 91		17,4	74,0

Table 10 (Continued)

Trial No. : ER90DEU7200301
Trial location : 8906 Gersthofen
Site of weather station: at trial location

Time	Treatment (B) Sampling (P)	Mean temperature (°C)	Precipitation (mm)
22.5.90	P	16,4	-
23.5.90	BP	17,0	-
24.5.90		14,2	-
25 5.90		11,8	-
26.5.90		11,1	-
27.5.90		10,9	-
28.5.90		9,1	-
29.5.90		8,6	-
30.5.90	P	11,0	-
31.5.90		12,7	-
Mai 90		13,9	30,0
1.6.90		18,0	-
2.6.90		17,3	0,9
3.6.90		13,8	-
4.6.90		12,0	-
5.6.90		14,8	-
6.6.90	P	15,0	2,0
7.6.90		15,0	12,0
8.6.90		14,3	14,0
9.6.90		12,5	8,0
10.6.90		12,3	1,5
11.6.90		12,5	-
12.6.90		12,0	-
13.6.90		12,8	-
14.6.90		12,5	-
15.6.90		13,0	-
16.6.90		12,3	-
17.6.90		15,0	-
18.6.90		16,8	-
19.6.90		19,5	0,8
20.6.90	P	15,8	22,0

Table 10 (Continued)

Trial No. : ER90DEU7200301
Trial location : 8906 Gersthofen
Site of weather station: at trial location

Time	Treatment (B) Sampling (P)	Mean temperature (°C)	Precipitation (mm)
21.6.90		15,3	-
22.6.90		16,0	5,0
23.6.90		15,8	6,6
24.6.90		15,5	-
Juni 90		15,7	85,3
20.07.90	P	19,0	-
Juli 90		17,2	35,2
16.08.90	P	19,5	-
August 90		19,3	104,8
September 90		12,2	98,2
23.10.90	P	-2,5	-
Oktober 90		10,4	55,9
November 90		3,9	89,0
Dezember 90		-0,8	39,7
Januar 91		0,8	36,6
Februar 91		-3,4	12,0
21.03.91	P	13,2	-
März 91		6,2	33,2
April 91		7,1	33,3
21.05.91	P	13,1	-
Mai 91		9,0	70,6
Juni 91		14,9	165,9
Juli 91		19,0	79,5
23.08.91	P	16,7	1,4
August 91		18,3	42,5

Figure 1

Graphic representation of the results

Trial No.: ER90DEU7200101
- alpha-endosulfan (Hoe 052618) + beta-endosulfan (Hoe 052619)

Degradation of endosulfan (alpha and beta) in soil

ER 90 DEU 720 0101, start of regression day 0, evaluation: root 1st order

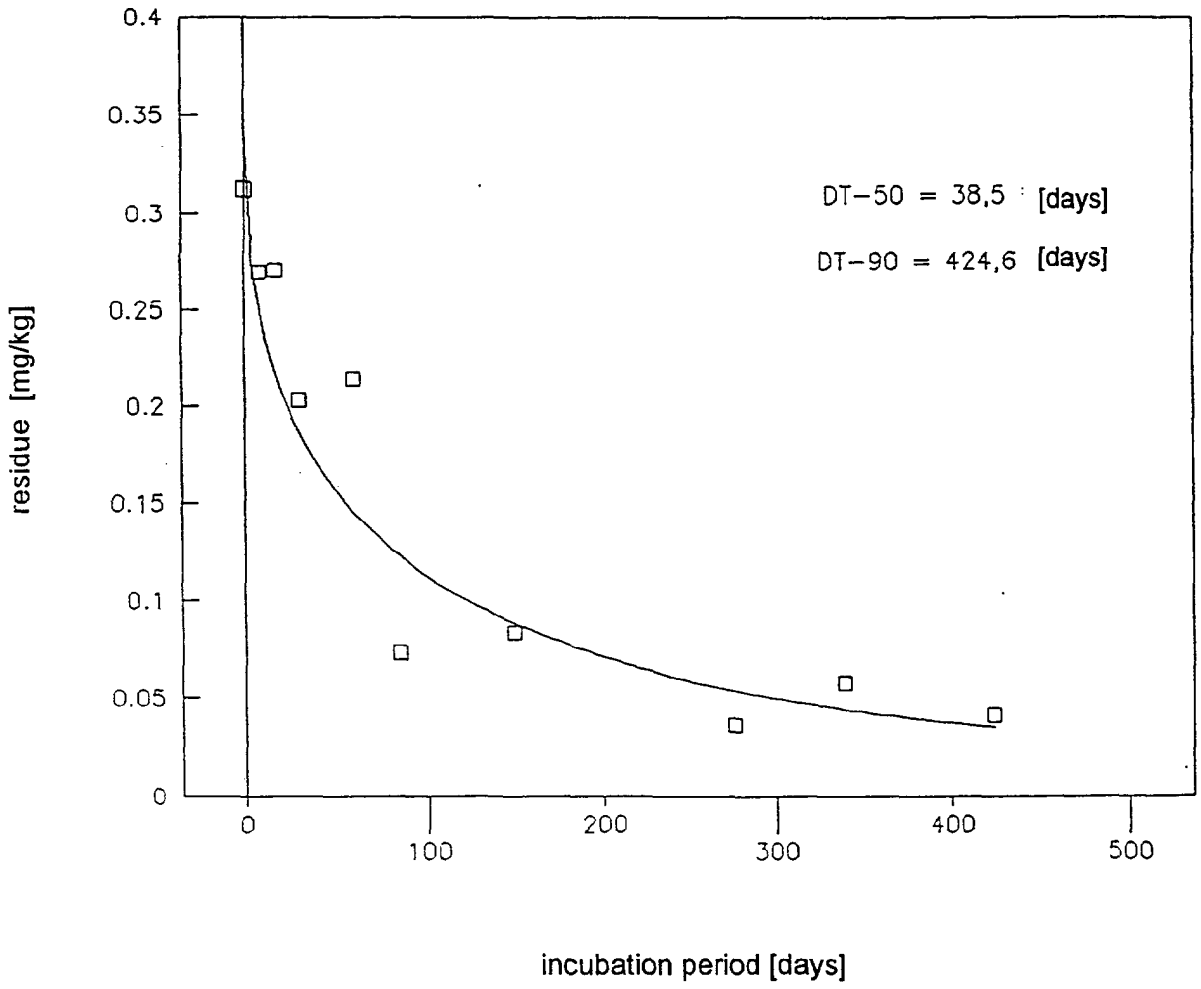


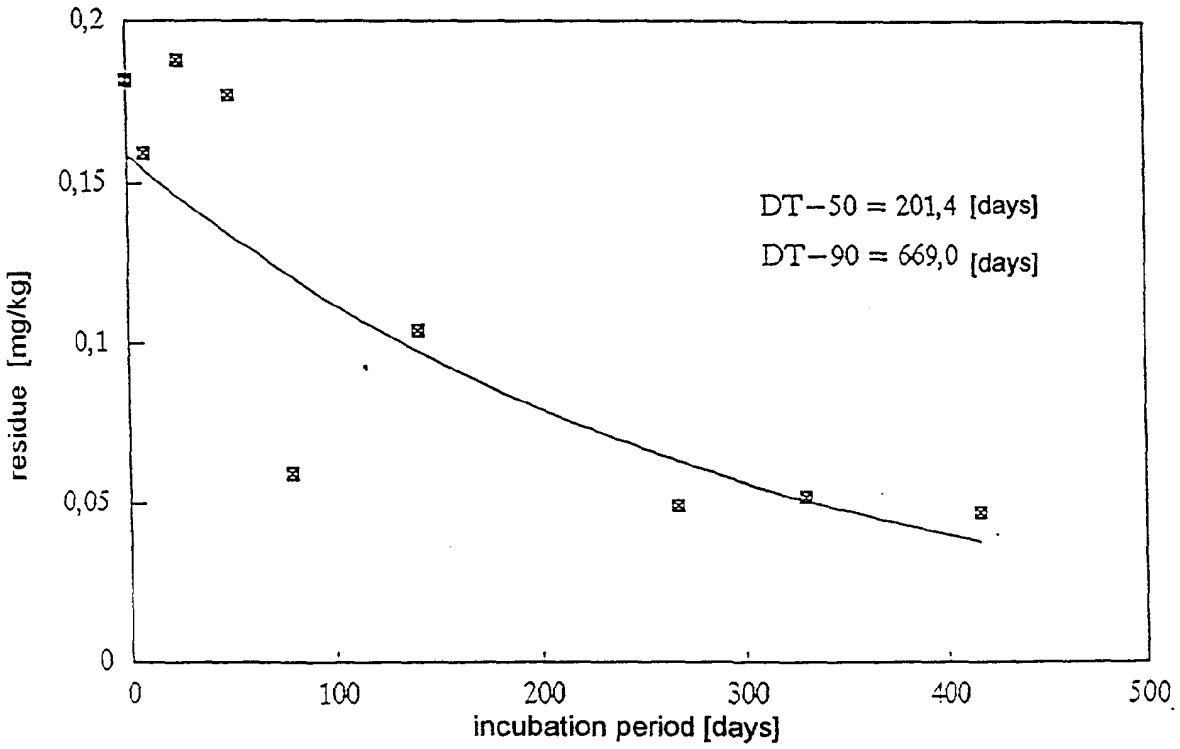
Figure 2

Graphic representation of the results

Trial No.: ER90DEU7200101
-endosulfan sulphate (Hoe 051327)

Degradation of endosulfan sulphate in soil

ER 90 DEU 720 0101, start of regression day 7, evaluation: 1st order



1 Apr 1992, Trial: ER 90 DEU 720 0101

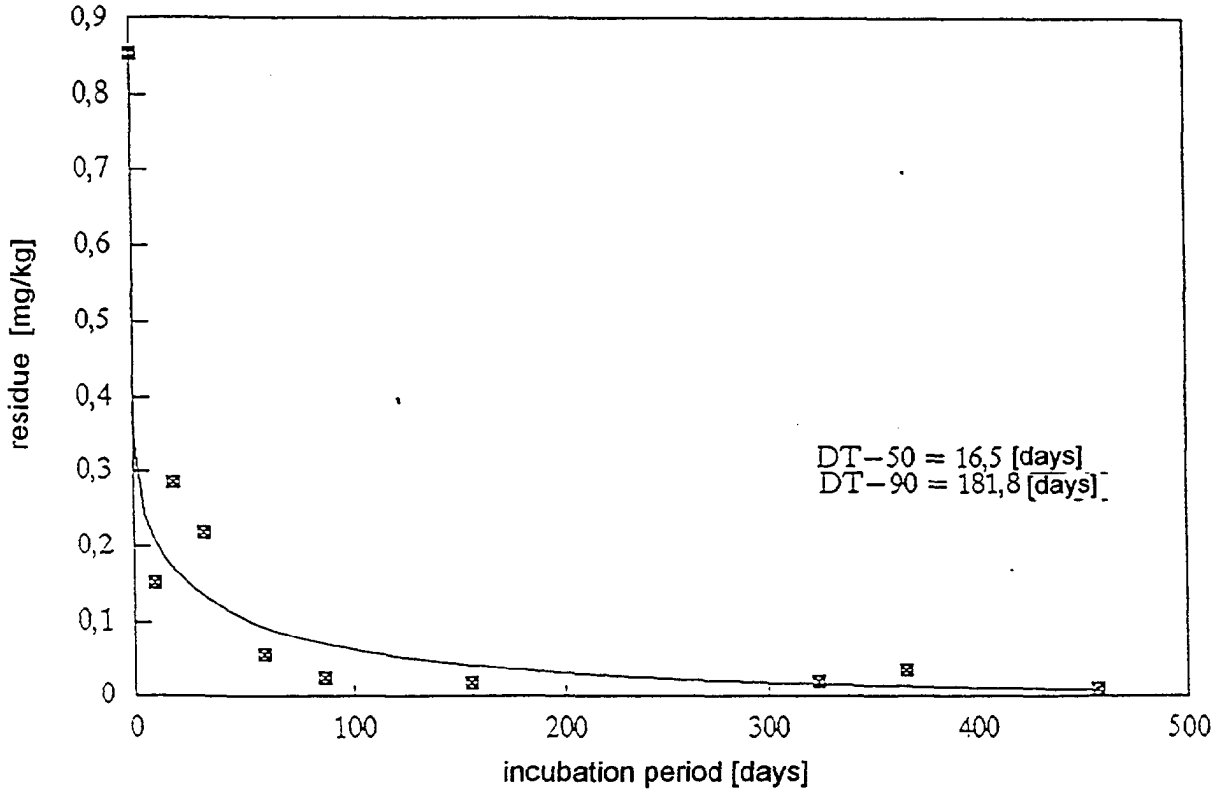
Figure 3

Graphic representation of the results

Trial No.: ER90DEU7200301
- alpha-endosulfan (Hoe 052618) + beta-endosulfan (Hoe 052619)

Degradation of endosulfan (alpha and beta) in soil

ER 90 DEU 720 0301, start of regression day 0, evaluation: root 1st order



28. Apr 1992, Trial: ER 90 DEU 720 0301

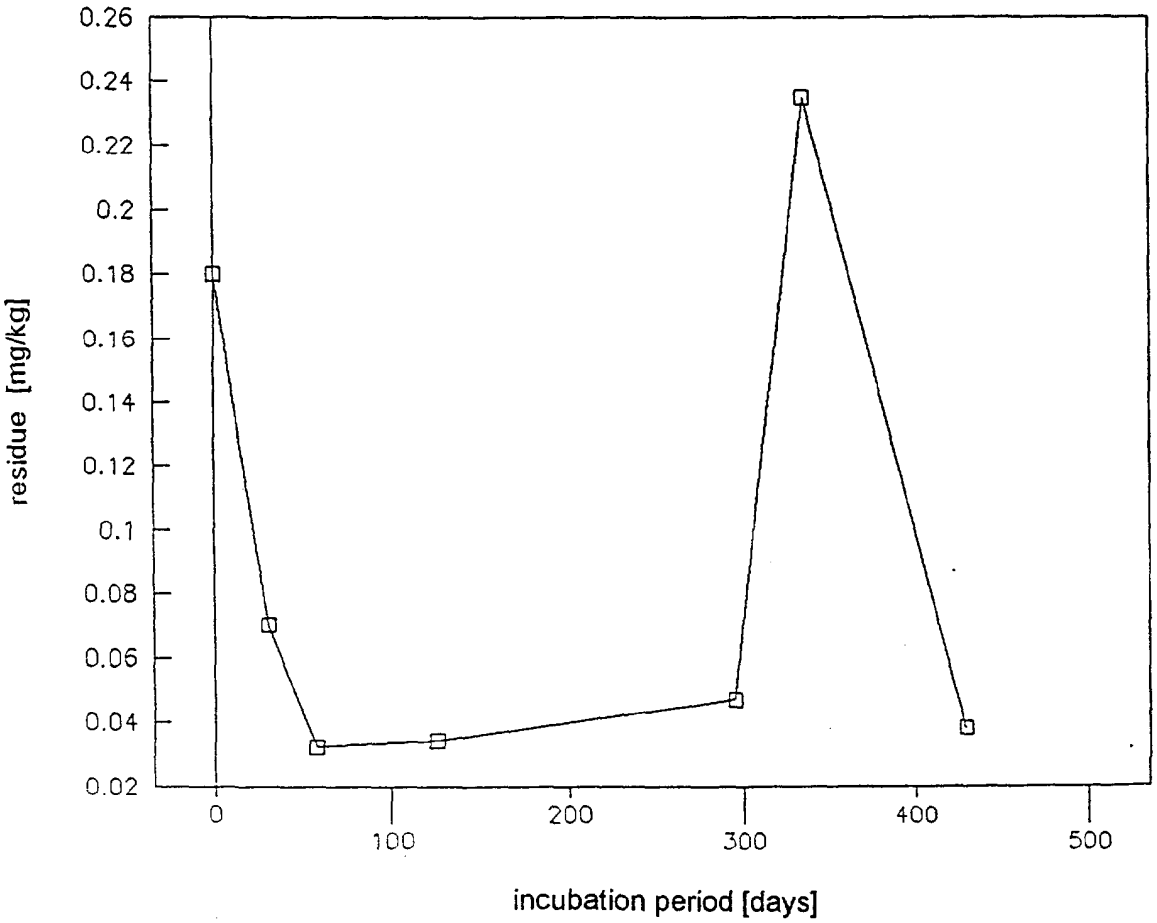
Figure 4

Graphic representation of the results

Trial No.: ER90DEU7200301
endosulfan sulphate (Hoe 051327)

Degradation of endosulfan sulphate in soil

ER 90 DEU 720 0301, start of regression day 28



A54025

Title

Project ER 90 DEU 720

Investigation of the residues of endosulfan (Hoe 002671)
and endosulfan-sulfate (Hoe 051327) in soil

Author

G. Fuchsbichler
15.04.1992

Testing Facility

Bayerische Hauptversuchsanstalt für Landwirtschaft
der TUM-Weihenstephan
D-8050 Freising 1

Study Plan No.

HVA 2/91

Statement

This study was conducted in compliance with the Principles of Good Laboratory Practice.

No unforeseen circumstances were observed which might have affected the quality and integrity of the presented study.

Study Director: Dr. G. Fuchsbichler

Signature: - signed - Date: 15 Apr 1992

Head of the Testing Institution: I. V. Dr. K. Teicher

Signature: - signed - Date: 15 Apr 1992

Quality Assurance Statement

The authorised representative for Quality Assurance of the laboratory periodically inspected the performed investigations as well as the final report. Records of these inspections were submitted to the Study Director.

Inspection	Report
22.01.91	22.01.91
31.10.91	31.10.91
16.04.92	16.04.92

Referat GLP: Dr. L. Nätscher

Signature: - signed - Date: 16 Apr 1992

1. Organisation and personnel

Sponsor:	Hoechst Aktiengesellschaft Geschäftsbereich Landwirtschaft Produktentwicklung Ökologie II Gebäude G864 6230 Frankfurt am Main 80
Monitoring analyst:	Dr. H. Idstein address: see Sponsor
Testing Facility:	Bayerische Hauptversuchsanstalt für Landwirtschaft D-8050 Freising 1 Tel.: 08161/713381 Head: Prof. Dr. K. Ranfft
Study Director:	Dr. G. Fuchsbichler address: see Testing Facility Tel.: 08161/713381
Quality Assurance:	Dr. R. Gerstl address: see Testing Facility Tel.: 08161/713381

Archiving:
Test-plan, raw data, and original final report are filed in the record office of the Testing Facility for at least the period prescribed in the GLP Guidelines. Subsequently the documents may only be destroyed with the consent of the Sponsor. If the record office is dissolved before termination of the prescribed storage period, the documents are to be returned to the Sponsor.

Time schedule:

Approval of the test-plan:	15.01.1991
Start of the experimental phase:	16.05.1991
End of the experimental phase:	09.04.1992
Completion of the final report:	15.04.1992

2. Residue Analysis

The laboratory samples were received on 9.04.1991 as well as on 31.10.1991 in the deep frozen state, after which they were registered and stored at -20°C until working up. Before working up was started, the frozen samples were thawed in the refrigerator at a temperature of 4° - 7° C.
Specifications on data of preparation and measurement of the samples can be found in Table 1.
In accordance with the study plan the same number of treated and untreated samples were taken. These were available as untreated controls, and - after fortification - for determination of the recovery rate. Further details will be found in Table 1.

2.1. Test substances and reference substances

2.1.1 Test substances

2.1.1.1 Hoe 052618

Common name:	alpha-endosulfan
Chemical name:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3-oxide (IUPAC, English)

2.1.1.2 Hoe 052619

Common name: beta-endosulfan
Chemical name: 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3-oxide (IUPAC, English)

2.1.1.3 Hoe 051327

Common name: endosulfan-sulfate
Chemical name: 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3,3-dioxide (IUPAC, English)

2.1.2 Reference substances

2.1.2.1 Hoe 052618

chem. name: 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3-oxide (IUPAC, English)
Empirical formula: $C_9H_6Cl_6O_3S$
Molar mass: 406.9 g/mol
Charge-No.: Hoe 052618 00 ZB 99 0004
Certificate No.: AZ 04153
dated: 14 Sep 1989
AZ 04457
dated: 08 Nov 1990

2.1.2.2 Hoe 052619

chem. name: 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin-3-oxide (IUPAC, English)
Empirical formula: $C_9H_6Cl_6O_3S$
Molar mass: 406.9 g/mol
Charge-No.: Hoe 052619 00 ZB 99 0003
Certificate No.: AZ 04152
dated: 14 Sep 1989
AZ 04636
dated: 01 Jul 1991

2.1.2.3 Hoe 051327

Chemical name:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxa-thiepin-3,3-dioxide (IUPAC, English)
Empirical formula:	$C_9H_6Cl_6O_4S$
Molar mass:	422.9 g/mol
Batch-No.:	Hoe 051327 00 ZB98 0002
Certificate No.:	AZ 04493 dated: 14 Jan 1991

in hexane in different concentrations
in acetone in different concentrations

2.2 Test methods

The samples were examined for Hoe 002671 (endosulfan) and Hoe 051327 (endosulfan-sulfate) by method AL 60/86 of Hoechst Aktiengesellschaft (Ref. 1.)

The active ingredients alpha-endosulfan, beta-endosulfan and endosulfan-sulfate are extracted from the sample material with acetone. An aliquot of the filtered extract is reconstituted with water and saturated sodium chloride solution and extracted with dichloromethane. The residue left over after evaporation of the organic phase is cleaned up by gel permeation-chromatography on SX3 Bio Beads. The final determination is carried out by gas chromatography using an electron capture detector. The residues are determined by the external standard method. There is no correction by recoveries.

Modifications

1. Point 6.1.2 of the Analytical Procedure
50 g sample are shaken with 100 ml acetone for 30 min. The filtrates are united and made up to 350 ml with acetone.

2. Point 6.2 of the Analytical Procedure
A 150 ml aliquot of the filtrate is transferred to a 500 ml separation funnel. After addition of 100 ml distilled water and 30 ml saturated sodium chloride solution, extraction is carried out with 100 ml dichloromethane. After separation of the phases, the organic phase is allowed to run off via a plaited filter filled with approx. 10 g thoroughly heated sodium sulphate; the filter is then rinsed with approx. 50 ml dichloromethane.
The organic phase is concentrated to a volume of approx. 5 ml on the rotary evaporator, the rest is blown to dryness under a gentle nitrogen stream.

3. Point 6.3 of the Analytical Procedure
The clean-up of the extract is conducted by gel chromatography. The residue was absorbed in 10 ml of elution mixture and the gel chromatograph (GPC) is loaded; in the process the equipment takes over 5 ml for cleaning, which corresponds to an aliquot of 75 ml.

Elution times:	Dump :	24 min
	Collect :	40 min

The collect phase was passed directly into round-bottom flask furnished with a column containing 3 g 15 % deactivated Florisil. Subsequently the collect phase is concentrated to a volume of approx. 5 ml on the rotary evaporator at a water bath temperature of 30 °C, and the rest of the dilution mixture is blown out to dryness under a gentle nitrogen stream.
The residue is absorbed in 2 ml hexane.

4. Point 6.4 of the Analytical Procedure

Gas chromatographic conditions

Equipment:	model 8521a (Dani)
detector:	electron capture detector (ECD) (Dani)
separation column:	Fused silica SE 54-CB-0.33 length: 50 m internal diameter: 0.33 mm thickness of film: 0.33 µm
gases:carrier gas:	helium 4.6, 3 ml/min
make-up gas:	nitrogen 5.0, 40 ml/min
Operating temperature:	injector: 250°C detector: 350°C temperature program oven: T_1: 150°C I_1: 1 min R_1: 15°C/min T_2: 245°C I_2: 30 min
Injection:	splitless, opening of split after 0.8 min
Injected volume:	1 µl

One representative chromatogram of the reference substance, of an untreated sample and of a spiked control sample are enclosed.

2.3 Lower limit of the practical working range

The lower limit of the practical working range for the sample material in this analytical series is defined as the lowest spiking level in untreated samples worked up and analysed together with treated samples:

	alpha-endosulfan	beta-endosulfan	endosulfan-sulfate
Matrix	Hoe 052618	Hoe 052619	Hoe 051327
soil	0.01 mg/kg	0.01 mg/kg	0.01 mg/kg

3.4 Recoveries and results for untreated samples :

As an internal quality assurance analyses of untreated samples and spiked untreated samples were carried out for the sample material using the substance under analysis. The recoveries obtained for alpha-endosulfan, beta-endosulfan and endosulfan-sulfate are listed in Table 2.

4. Results

The results of the residue analyses are given in mg/kg dry soil and are listed in Table 3. Results of analyses below the lower limit of the practical working range have been shown as < 0.01 mg/kg, 0.01 representing the lower limit of the practical working range in the sample material used.
The dry substance was determined by the VDLUFA method(2).

5. Discussion

The method used here enabled endosulfan and endosulfan-sulfate to be detected selectively. The untreated samples contained no substances interfering with determination. The lower limit of the practical working range and the recovery rates of parallel determinations of spiked and untreated samples (73 - 120%) permitted sensitive detection of endosulfan and endosulfan-sulfate.

Author:	15 Apr 1992	Fuchsbichler	(signed)
Study Director:	15 Apr 1992	Fuchsbichler	(signed)

Enclosures: chromatograms of representative samples
calculated results
calculated recoveries

References:

(1) H. - J. Werner, G. Klante, H. D. Merz
Hoe 002671 (Endosulfan)
Rückstandsbestimmung von Wirkstoff und Endosulfan-sulfat in Boden, Wasser, Urin und Pflanzen sowie von Endosulfandiol und Endosulfanlacton in Boden, Wasser und Urin
Method AL 60/86 dated 10/12/86

(2) VDLUFA-Mitteilungen 1987, Heft 3, 232 - 237

Table 1: Test-No. ER 90 DEU 720 0101; samples and working-up dates Sheet 1 (6)

Sample No.	Analysis-No.	Test-material	Rem.*	Lab code	Days after final appl.	Sample received	Start of working-up	Date of measurement
1	9171045	soil	u	B001	0	09.04.91	16.05.91	21.05.91
1	9171045	"	R	A001	0	"	16.05.91	19.05.91
2	9171046	"	u	B005	0	"	22.05.91	23.05.91
3	9171047	"	u	B002	7	"	16.05.91	21.05.91
4	9171048	"	u	B006	7	"	22.05.91	23.05.91
5	9171049	"	u	B009	14	"	10.09.91	12.09.91
5	9171049	"	R	A003	14	"	10.09.91	17.09.91
6	9171050	"	u		14	"	nicht analysiert	
7	9171051	"	u		28	"	nicht analysiert	
8	9171052	"	u		28	"	nicht analysiert	
9	9171053	"	u		56	"	nicht analysiert	
10	9171054	"	u		56	"	nicht analysiert	
11	9171055	"	u		84	"	nicht analysiert	
12	9171056	"	u		84	"	nicht analysiert	
13	9171057	"	u	B011	148	"	19.09.91	keine Auswert.**
13	9171057	"	R	A005	148	"	19.09.91	25.09.91
14	9171058	"	u	B015	148	"	27.01.92	keine Auswert.**
14	9171058	"	R	A009	148	"	27.01.92	keine Auswert.**
14	9171058	"	u	B017	148	"	10.03.92	07.04.92
14	9171058	"	R	A011	148	"	10.03.92	06.04.92
15	9172563	"	u		273	30.10.91	nicht analysiert	
16	9172564	"	u	B019	273	"	18.03.92	06.04.92
16	9172564	"	R	A012	273	"	18.03.92	06.04.92

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see Table 2)
Remark: the untreated samples were worked up in series together with the treated samples.
** no determination, see Enclosure 2

Table 1: Test-No. ER 90 DEU 720 0101; samples and working-up dates Sheet 2 (6)

Sample No.	Analysis-No.	Test-material	Rem.*	Lab code	Days after final appl.	Sample received	Start of working-up	Date of measurement
17	9172565	soil	u		336	30.10.91	nicht analysiert	
18	9172566	"	u		336	"	nicht analysiert	
19	9172567	"	u	B013	424	"	02.12.91	keine Auswert.**
19	9172567	"	u	B018	424	"	18.03.92	09.04.92
19	9172567	"	R	A007	424	"	02.12.91	10.12.91
20	9172568	"	u		424	"	nicht analysiert	
3	9171059	"	b	001	0 vor Anw.	09.04.91	16.05.91	21.05.91
4	9171060	"	b	007	0 vor Anw.	"	22.05.91	23.05.91
5	9171061	"	b	002	0 n. Anw.	"	16.05.91	22.05.91
5	9171061	"	b	032	0 n. Anw.	"	22.10.91	29.10.91
6	9171062	"	b	003	7	"	16.05.91	22.05.91
6	9171062	"	b	033	7	"	22.10.91	29.10.91
6	9171062	"	b	051	7	"	27.01.92	28.01.92
6	9171062	"	b	061	7	"	10.03.92	11.03.92
7	9171063	"	b	008	7	"	22.05.91	24.05.91
8	9171064	"	b	012	14	"	10.09.91	12.09.91
8	9171064	"	b	034	14	"	22.10.91	29.10.91
8	9171064	"	b	052	14	"	27.01.92	28.01.92
8	9171064	"	b	062	14	"	10.03.92	11.03.92
9	9171065	"	b	018	14	"	12.09.91	17.09.91
10	9171066	"	b	013	28	"	10.09.91	16.09.91
10	9171066	"	b	035	28	"	22.10.91	30.10.91
10	9171066	"	b	053	28	"	27.01.92	28.01.92
10	9171066	"	b	063	28	"	10.03.92	12.03.92

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see Table 2)
Remark: the untreated samples were worked up in series together with the treated samples.
** no determination, see Enclosure 2

Table 1: Test-No. ER 90 DEU 720 0101; samples and working-up dates Sheet 3 (6)

Sample No.	Analysis-No.	Test-material	Rem.*	Lab code	Days after final appl.	Sample received	Start of working-up	Date of measurement
11	9171067	soil	b	019	28	09.04.91	12.09.91	17.09.91
12	9171068	"	b	014	56	"	10.09.91	16.09.91
12	9171068	"	b	036	56	"	22.10.91	30.10.91
13	9171069	"	b	020	56	"	12.09.91	18.09.91
14	9171070	"	b	024	84	"	19.09.91	25.09.91
14	9171070	"	b	037	84	"	22.10.91	30.10.91
15	9171071	"	b	025	84	"	19.09.91	24.09.91
15	9171071	"	b	054	84	"	27.01.92	28.01.91
15	9171071	"	b	064	84	"	10.03.92	19.03.92
16	9171072	"	b	026	148	"	19.09.91	25.09.91
16	9171072	"	b	038	148	"	22.10.91	30.10.91
17	9171073	"	b	027	148	"	19.09.91	25.09.91
17	9171073	"	b	055	148	"	27.01.92	28.01.92
17	9171073	"	b	065	148	"	10.03.92	19.03.92
18	9172569	"	b	039	273	30.10.91	02.12.91	10.12.91
19	9172570	"	b	040	273	"	02.12.91	10.12.91
20	9172571	"	b	041	336	"	02.12.91	10.12.91
21	9172572	"	b	042	336	"	02.12.91	10.12.91
22	9172573	"	b	043	424	"	02.12.91	10.12.91
23	9172574	"	b	044	424	"	02.12.91	10.12.91

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see Table 2)
Remark: the untreated samples were worked up in series together with the treated samples.
** no determination, see Enclosure 2

Table 1: Test-No. ER 90 DEU 720 0101; samples and working-up dates Sheet 4 (6)

Sample No.	Analysis-No.	Test-material	Rem.*	Lab code	Days after final appl.	Sample received	Start of working-up	Date of measurement
1	9171074	soil	u	B003	0	09.04.91	16.05.91	22.05.91
2	9171075	"	u	B007	0	"	22.05.91	23.05.91
2	9171075	"	R	A002	0	"	22.05.91	25.05.91
3	9171076	"	u	B004	7	"	16.05.91	22.05.91
3	9171076	"	u	B012	7	"	22.10.91	30.10.91
3	9171076	"	R	A006	7	"	22.10.91	30.10.91
4	9171077	"	u	B008	7	"	22.05.91	23.05.91
5	9171078	"	u		14	"	nicht analysiert	
6	9171079	"	u		14	"	nicht analysiert	
7	9171080	"	u		28	"	nicht analysiert	
8	9171081	"	u	B010	28	"	12.09.91	18.09.91
8	9171081	"	R	A004	28	"	12.09.91	18.09.91
9	9171082	"	u		59	"	nicht analysiert	
10	9171083	"	u		59	"	nicht analysiert	
11	9171084	"	u		86	"	nicht analysiert	
12	9171085	"	u		86	"	nicht analysiert	
13	9171086	"	u	B016	154	"	28.01.92	30.01.91
13	9191086	"	R	A010	154	"	28.01.92	31.01.92
14	9171087	"	u		154	"	nicht analysiert	
15	9172575	"	u		323	30.10.91	nicht analysiert	
16	9172576	"	u		323	"	nicht analysiert	
17	9172577	"	u		363	"	nicht analysiert	

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see Table 2)
Remark: the untreated samples were worked up in series together with the treated samples.
** no determination, see Enclosure 2

Table 1: Test-No. ER 90 DEU 720 0101; samples and working-up dates Sheet 5 (6)

Sample No.	Analysis-No.	Test-material	Rem.*	Lab code	Days after final appl.	Sample received	Start of working-up	Date of measurement
18	9172578	soil	u	B014	363	30.10.91	04.12.91	11.12.91
18	9172578	"	R	A008	363	"	04.12.91	13.12.91
19	9172579	"	u		457	"	nicht analysiert	
20	9172580	"	u		457	"	nicht analysiert	
3	9171088	"	b	004	0 v.Anw.	09.04.91	16.05.91	keine Auswert.**
3	9171088	"	b	011	0 v.Anw.	"	22.05.91	24.05.91
4	9171089	"	b	009	0 v.Anw.	"	22.05.91	23.05.91
5	9171090	"	b	005	0 n.Anw.	"	16.05.91	23.05.91
5	9171090	"	b	066	0 n.Anw.	"	18.03.92	23.03.92
6	9171091	"	b	006	7	"	16.05.91	23.05.91
6	9171091	"	b	056	7	"	28.01.92	30.01.92
7	9171092	"	b	010	7	"	22.05.91	23.05.91
8	9171093	"	b	015	14	"	10.09.91	16.09.91
9	9171094	"	b	021	14	"	12.09.91	18.09.91
10	9171095	"	b	016	28	"	10.09.91	16.09.91
11	9171096	"	b	022	28	"	12.09.91	18.09.91
12	9171097	"	b	017	59	"	10.09.91	17.09.91
13	9171098	"	b	023	59	"	12.09.91	18.09.91
14	9171099	"	b	028	86	"	19.09.91	keine Auswert.**
14	9171099	"	b	057	86	"	28.01.92	30.01.92
15	9171100	"	b	029	86	"	19.09.91	keine Auswert.**
15	9171100	"	b	058	86	"	28.01.92	30.01.92

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see Table 2)
Remark: the untreated samples were worked up in series together with the treated samples.
** no determination, see Enclosure 2

Table 1: Test-No. ER 90 DEU 720 0101; samples and working-up dates Sheet 6 (6)

Sample No.	Analysis-No.	Test-material	Rem.*	Lab code	Days after final appl.	Sample received	Start of working-up	Date of measurement
16	9171101	soil	b	030	154	09.04.91	19.09.91	keine Auswert.**
16	9171101	"	b	059	154	"	28.01.91	31.01.92
17	9171102	"	b	031	154	"	19.09.91	keine Auswert.**
17	9171102	"	b	060	154	"	28.01.92	31.01.92
18	9172581	"	b	045	323	30.10.91	04.12.91	11.12.91
19	9172582	"	b	046	323	"	04.12.91	11.12.91
20	9172583	"	b	047	363	"	04.12.91	11.12.91
20	9172583	"	b	067	363	"	18.03.92	24.03.92
20	9172583	"	b	068	363	"	18.03.92	25.03.92
21	9172584	"	b	048	363	"	04.12.91	12.12.91
22	9172585	"	b	049	457	"	04.12.91	12.12.91
23	9172586	"	b	050	457	"	04.12.91	12.12.91

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see Table 2)
Remark: the untreated samples were worked up in series together with the treated samples.
** no determination, see Enclosure 2

Legend: 'nicht analysiert' - not analysed
'vor Anw.' - before application
'nach Anw.' - after application

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate of fortification [%]
soil	B001	0	<0,01	
"	B002	0	<0,01	
"	B003	0	<0,01	
"	B004	0	<0,01	
"	B005	0	<0,01	
"	B006	0	<0,01	
"	B007	0	<0,01	
"	B008	0	<0,01	
"	B009	0	<0,01	
"	B010	0	<0,01	
"	B012	0	<0,01	
"	B014	0	<0,01	
"	B016	0	<0,01	
"	B017	0	<0,01	
"	B018	0	<0,01	
"	B019	0	<0,01	

Sheet 2 (6)

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate of fortification [%]
soil	A001	0,50	0,459	92
"	A002	0,01	0,0096	97
"	A003	0,10	0,093	93
"	A004	0,05	0,049	98
"	A005	0,02	0,0196	98
"	A006	0,50	0,447	89
"	A007	0,05	0,0495	99
"	A008	0,01	0,0073	73
"	A010	0,02	0,0183	92
"	A011	0,02	0,0179	90
"	A012	0,10	0,104	104

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate of fortification [%]
soil	B001	0	<0,01	
"	B002	0	<0,01	
"	B003	0	<0,01	
"	B004	0	<0,01	
"	B005	0	<0,01	
"	B006	0	<0,01	
"	B007	0	<0,01	
"	B008	0	<0,01	
"	B009	0	0,011	
"	B010	0	<0,01	
"	B012	0	<0,01	
"	B014	0	<0,01	
"	B016	0	<0,01	
"	B017	0	<0,01	
"	B018	0	<0,01	
"	B019	0	<0,01	

Sheet 4 (6)

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate of fortification [%]
soil	A001	0,50	0,449	90
"	A002	0,01	0,0093	93
"	A003	0,10	0,088	88*
"	A004	0,05	0,0478	96
"	A005	0,02	0,0092	46**
"	A006	0,50	0,459	92
"	A007	0,05	0,043	86
"	A008	0,01	0,0093	93
"	A010	0,02	0,0208	104
"	A011	0,02	0,015	75
"	A012	0,10	0,087	87

* corrected by control
** no determination, the whole series was repeated

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate of fortification [%]
soil	B001	0	0,015	
"	B002	0	0,014	
"	B003	0	<0,01	
"	B004	0	<0,01	
"	B005	0	<0,01	
"	B006	0	<0,01	
"	B007	0	<0,01	
"	B008	0	<0,01	
"	B009	0	0,023	
"	B010	0	<0,01	
"	B012	0	<0,01	
"	B014	0	<0,01	
"	B016	0	<0,01	
"	B017	0	<0,01	
"	B018	0	<0,01	
"	B019	0	<0,01	

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate of fortification [%]
soil	A001	0,50	0,522	104*
"	A002	0,01	0,0088	88
"	A003	0,10	0,081	81*
"	A004	0,05	0,06	120
"	A005	0,02	0,0056	28**
"	A006	0,50	0,597	119
"	A007	0,05	0,0544	109
"	A008	0,01	0,0104	104
"	A010	0,02	0,0177	89
"	A011	0,02	0,0228	114
"	A012	0,10	0,085	85

* corrected by control
** no determination, the whole series was repeated

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
1	9171045	01 01	Boden 0-20 cm	0 unbeh.	<0,01	<0,01
2	9171046	01 01	Boden 20-40 cm	0 unbeh.	<0,01	<0,01
3	9171047	01 01	Boden 0-20 cm	7 unbeh.	<0,01	<0.01
4	9171048	01 01	Boden 20-40 cm	7 unbeh.	<0,01	<0,01
5	9171049	01 01	Boden 0-20 cm	14 unbeh.	<0,01	<0,01
6	9171050	01 01	Boden 20-40 cm	14 unbeh.	nicht analysiert	
7	9171051	01 01	Boden 0-20cm	28 unbeh.	nicht analysiert	
8	9171052	01 01	Boden 20-40 cm	28 unbeh.	nicht analysiert	
9	9171053	01 01	Boden 0-20 cm	56 unbeh.	nicht analysiert	
10	9171054	01 01	Boden 20-40 cm	56 unbeh.	nicht analysiert	
11	9171055	01 01	Boden 0-20 cm	84 unbeh.	nicht analysiert	
12	9171056	01 01	Boden 20-40 cm	84 unbeh.	nicht analysiert	

For legend: see page 2

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
13	9171057	01 01	Boden 0-20 cm	148 unbeh.	nicht analysiert	
14	9171058	01 01	Boden 20-40 cm	148 unbeh.	<0,01	< 0,01
15	9172563	01 01	Boden 0- 20 cm	273 unbeh.	nicht analysiert	
16	9172564	01 01	Boden 20-40 cm	273 unbeh.	<0,01	<0,01
17	9172565	01 01	Boden 0-20 cm	336 unbeh.	nicht analysiert	
18	9172566	01 01	Boden 20-40 cm	336 unbeh.	nicht analysiert	
19	9172567	01 01	Boden 0-20 cm	424 unbeh.	<0,01	<0,01
20	9172568	01 01	Boden 20-40 cm	424 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed,
'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
3	9171059	01 01	Boden 0-20 cm	0 vor Anw.	<0,01	<0,01
4	9171060	01 01	Boden 20-40 cm	0 vor Anw.	<0,01	<0,01
5	9171061	01 01	Boden 0-20 cm	0 nach Anw.	0,136/0,199	0,168
6	9171062	01 01	Boden 0-20 cm	7	0,119/0,166/0,146	0,144
7	9171063	01 01	Boden 20-40 cm	7	<0,01	<0,01
8	9171064	01 01	Boden 0-20 cm	14	0,129/0,143/0,120	0,131
9	9171065	01 01	Boden 20-40 cm	14	<0,01	<0,01
10	9171066	01 01	Boden 0-20 cm	28	0,114/0,048/0,077	0,080
11	9171067	01 01	Boden 20-40 cm	28	<0,01	<0,01
12	9171068	01 01	Boden 0-20 cm	56	0,075/0,071	0,073
13	9171069	01 01	Boden 20-40 cm	56	<0,01	<0,01
14	9171070	01 01	Boden 0-20 cm	84	0,027	0,027
15	9171071	01 01	Boden 20-40 cm	84	<0,01	<0,01

for legend: see page 2

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Residue (mg/kg TS) Mean value
16	9171072	01 01	Boden 0-20 cm	148	0,028	0,028
17	9171073	01 01	Boden 20-40 cm	148	<0,01	<0,01
18	9172569	01 01	Boden 0-20 cm	273	0,013	0,013
19	9172570	01 01	Boden 20-40 cm	273	<0,01	<0,01
20	9172571	01 01	Boden 0-20 cm	336	0,019	0,019
21	9172572	01 01	Boden 20-40 cm	336	<0,01	<0,01
22	9172573	01 01	Boden 0-20 cm	424	<0,01	<0,01
23	9172574	01 01	Boden 20-40 cm	424	<0,01	<0,01

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
1	9171074	03 01	Boden 0-20 cm	0 unbeh.	<0,01	<0,01
2	9171075	03 01	Boden 20-40 cm	0 unbeh.	<0,01	<0,01
3	9171076	03 01	Boden 0-20 cm	7 unbeh.	<0,01/<0,01	<0,01
4	9171077	03 01	Boden 20-40 cm	7 unbeh.	<0,01	<0,01
5	9171078	03 01	Boden 0-20 cm	14 unbeh.	nicht analysiert	
6	9171079	03 01	Boden 20-40 cm	14 unbeh.	nicht analysiert	
7	9171080	03 01	Boden 0-20 cm	28 unbeh.	nicht analysiert	
8	9171081	03 01	Boden 20-40 cm	28 unbeh.	<0,01	<0,01
9	9171082	03 01	Boden 0-20 cm	59 unbeh.	nicht analysiert	
10	9171083	03 01	Boden 20-40 cm	59 unbeh.	nicht analysiert	
11	9171084	03 01	Boden 0-20 cm	86 unbeh.	nicht analysiert	
12	9171085	03 01	Boden 20-40 cm	86 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed,
'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
13	9171086	03 01	Boden 0-20 cm	154 unbeh.	<0,01	<0,01
14	9171087	03 01	Boden 20-40 cm	154 unbeh.	nicht analysiert	
15	9172575	03 01	Boden 0-20 cm	323 unbeh.	nicht analysiert	
16	9172576	03 01	Boden 20-40 cm	323 unbeh.	nicht analysiert	
17	9172577	03 01	Boden 0-20 cm	363 unbeh.	nicht analysiert	
18	9172578	03 01	Boden 20-40 cm	363 unbeh.	<0,01	<0,01
19	9172579	03 01	Boden 0-20 cm	457 unbeh.	nicht analysiert	
20	9172580	03 01	Boden 20-40 cm	457 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
3	9171088	03 01	Boden 0-20 cm	0 vor Anw.	<0,01	<0,01
4	9171089	03 01	Boden 20-40 cm	0 vor Anw.	<0,01	<0,01
5	9171090	03 01	Boden 0-20 cm	0 nach Anw.	0,322/0,766	0,544
6	9171091	03 01	Boden 0-20 cm	7	0,070/0,081	0,076
7	9171092	03 01	Boden 20-40 cm	7	<0,01	<0,01
8	9171093	03 01	Boden 0-20 cm	14	0,.123	0,123
9	9171094	03 01	Boden 20-40 cm	14	<0,01	<0,01
10	9171095	03 01	Boden 0-20 cm	28	0,063	0,063
11	9171096	03 01	Boden 20-40 cm	28	<0,01	<0,01
12	9171097	03 01	Boden 0-20 cm	59	0,016	0,016
13	9171098	03 01	Boden 20-40 cm	59	<0,01	<0,01
14	9171099	03 01	Boden 0-20 cm	86	<0,01	<0,01
15	9171100	03 01	Boden 20-40 cm	86	<0,01	<0,01

for legend: see page 2

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
16	9171101	03 01	Boden 0-20 cm	154	<0,01	<0,01
17	9171102	03 01	Boden 20-40 cm	154	<0,01	<0,01
18	9172581	03 01	Boden 0-20 cm	323	<0,01	<0,01
19	9172582	03 01	Boden 20-40 cm	323	<0,01	<0,01
20	9172583	03 01	Boden 0-20 cm	363	<0,01	<0,01
21	9172584	03 01	Boden 20-40 cm	363	<0,01	<0,01
22	9172585	03 01	Boden 0-20 cm	457	<0,01	<0,01
23	9172586	03 01	Boden 20-40 cm	457	<0,01	<0,01

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
1	9171045	01 01	Boden 0-20 cm	0 unbeh.	<0,01	<0,01
2	9171046	01 01	Boden 20-40 cm	0 unbeh.	<0,01	<0,01
3	9171047	01 01	Boden 0-20 cm	7 unbeh.	<0,01	<0.01
4	9171048	01 01	Boden 20-40 cm	7 unbeh.	<0,01	<0,01
5	9171049	01 01	Boden 0-20 cm	14 unbeh.	0,011	0,011
6	9171050	01 01	Boden 20-40 cm	14 unbeh.	nicht analysiert	
7	9171051	01 01	Boden 0-20cm	28 unbeh.	nicht analysiert	
8	9171052	01 01	Boden 20-40 cm	28 unbeh.	nicht analysiert	
9	9171053	01 01	Boden 0-20 cm	56 unbeh.	nicht analysiert	
10	9171054	01 01	Boden 20-40 cm	56 unbeh.	nicht analysiert	
11	9171055	01 01	Boden 0-20 cm	84 unbeh.	nicht analysiert	
12	9171056	01 01	Boden 20-40 cm	84 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed,
'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
13	9171057	01 01	Boden 0-20 cm	148 unbeh.	nicht analysiert	
14	9171058	01 01	Boden 20-40 cm	148 unbeh.	<0,01	<0,01
15	9172563	01 01	Boden 0-20 cm	273 unbeh.	nicht analysiert	
16	9172564	01 01	Boden 20-40 cm	273 unbeh.	<0,01	<0,01
17	9172565	01 01	Boden 0-20 cm	336 unbeh.	nicht analysiert	
18	9172566	01 01	Boden 20-40 cm	336 unbeh.	nicht analyisert	
19	9172567	01 01	Boden 0-20 cm	424 unbeh.	<0,01	<0,01
20	9172568	01 01	Boden 20-40 cm	424 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
3	9171059	01 01	Boden 0-20 cm	0 vor Anw.	<0,01	<0,01
4	9171060	01 01	Boden 20-40 cm	0 vor Anw.	<0,01	<0,01
5	9171061	01 01	Boden 0-20 cm	0 nach Anw.	0,114/0,173	0,144
6	9171062	01 01	Boden 0-20 cm	7	0,111/0,160/0,103	0,125
7	9171063	01 01	Boden 20-40 cm	7	<0,01	<0,01
8	9171064	01 01	Boden 0-20 cm	14	0,138/0,146/0,113	0,139
9	9171065	01 01	Boden 20-40 cm	14	<0,01	<0,01
10	9171066	01 01	Boden 0-20 cm	28	0,177/0,082/0,111	0,123
11	9171067	01 01	Boden 20-40 cm	28	<0,01	<0,01
12	9171068	01 01	Boden 0-20 cm	56	0,167/0,114	0,141
13	9171069	01 01	Boden 20-40 cm	56	<0,01	<0,01
14	9171070	01 01	Boden 0-20 cm	84	0,046	0,046
15	9171071	01 01	Boden 20-40 cm	84	<0,01	<0,01

for legend: see page 2

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
16	9171072	01 01	Boden 0-20 cm	148	0,055	0,055
17	9171073	01 01	Boden 20-40 cm	148	<0,01	<0,01
18	9172569	01 01	Boden 0-20 cm	273	0,023	0,023
19	9172570	01 01	Boden 20-40 cm	273	<0,01	<0,01
20	9172571	01 01	Boden 0-20 cm	336	0,039	0,039
21	9172572	01 01	Boden 20-40 cm	336	<0,01	<0,01
22	9172573	01 01	Boden 0-20 cm	424	0,036	0,036
23	9172574	01 01	Boden 20-40 cm	424	<0,01	<0,01

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
1	9171074	03 01	Boden 0-20 cm	0 unbeh.	<0,01	<0,01
2	9171075	03 01	Boden 20-40 cm	0 unbeh.	<0,01	<0,01
3	9171076	03 01	Boden 0-20 cm	7 unbeh.	<0,01/<0,01	<0,01
4	9171077	03 01	Boden 20-40 cm	7 unbeh.	<0,01	<0,01
5	9171078	03 01	Boden 0-20 cm	14 unbeh.	nicht analysiert	
6	9171079	03 01	Boden 20-40 cm	14 unbeh.	nicht analysiert	
7	9171080	03 01	Boden 0-20 cm	28 unbeh.	nicht analysiert	
8	9171081	03 01	Boden 20-40 cm	28 unbeh.	<0,01	<0,01
9	9171082	03 01	Boden 0-20 cm	59 unbeh.	nicht analysiert	
10	9171083	03 01	Boden 20-40 cm	59 unbeh.	nicht analysiert	
11	9171084	03 01	Boden 0-20 cm	86 unbeh.	nicht analysiert	
12	9171085	03 01	Boden 20-40 cm	86 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed,
'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
13	9171086	03 01	Boden 0-20 cm	154 unbeh.	<0,01	<0,01
14	9171087	03 01	Boden 20-40 cm	154 unbeh.	nicht analysiert	
15	9172575	03 01	Boden 0-20 cm	323 unbeh.	nicht analysiert	
16	9172576	03 01	Boden 20-40 cm	323 unbeh.	nicht analysiert	
17	9172577	03 01	Boden 0-20 cm	363 unbeh.	nicht analysiert	
18	9172578	03 01	Boden 20-40 cm	363 unbeh.	<0,01	<0,01
19	9172579	03 01	Boden 0-20 cm	457 unbeh.	nicht analysiert	
20	9172580	03 01	Boden 20-40 cm	457 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
3	9171088	03 01	Boden 0-20 cm	0 vor Anw.	<0,01	<0,01
4	9171089	03 01	Boden 20-40 cm	0 vor Anw.	<0,01	<0,01
5	9171090	03 01	Boden 0-20 cm	0 nach Anw.	0,232/0,386	0.309
6	9171091	03 01	Boden 0-20 cm	7	0,068/0,082	0,075
7	9171092	03 01	Boden 20-40 cm	7	<0,01	<0,01
8	9171093	03 01	Boden 0-20 cm	14	0.162	0,162
9	9171094	03 01	Boden 20-40 cm	14	<0,01	<0,01
10	9171095	03 01	Boden 0-20 cm	28	0,156	0,156
11	9171096	03 01	Boden 20-40 cm	28	<0,01	<0,01
12	9171097	03 01	Boden 0-20 cm	59	0,039	0,039
13	9171098	03 01	Boden 20-40 cm	59	<0,01	<0,01
14	9171099	03 01	Boden 0-20 cm	86	0,019	0,019
15	9171100	03 01	Boden 20-40 cm	86	<0,01	<0,01

for legend: see page 2

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
14	9171101	03 01	Boden 0-20 cm	154	0,017	0,017
15	9171102	03 01	Boden 20-40 cm	154	<0,01	<0,01
16	9172581	03 01	Boden 0-20 cm	323	0,019	0,019
17	9172582	03 01	Boden 20-40 cm	323	<0,01	<0,01
18	9172583	03 01	Boden 0-20 cm	363	0,011/0,047/0,048	0,035
19	9172584	03 01	Boden 20-40 cm	363	<0,01	<0,01
20	9172585	03 01	Boden 0-20 cm	457	0,01	0,01
21	9172586	03 01	Boden 20-40 cm	457	<0,01	<0,01

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
1	9171045	01 01	Boden 0-20 cm	0 unbeh.	0,015	0,015
2	9171046	01 01	Boden 20-40 cm	0 unbeh.	<0,01	<0,01
3	9171047	01 01	Boden 0-20 cm	7 unbeh.	0,014	0.014
4	9171048	01 01	Boden 20-40 cm	7 unbeh.	<0,01	<0,01
5	9171049	01 01	Boden 0-20 cm	14 unbeh.	0,023	0,023
6	9171050	01 01	Boden 20-40 cm	14 unbeh.	nicht analysiert	
7	9171051	01 01	Boden 0-20cm	28 unbeh.	nicht analysiert	
8	9171052	01 01	Boden 20-40 cm	28 unbeh.	nicht analysiert	
9	9171053	01 01	Boden 0-20 cm	56 unbeh.	nicht analysiert	
10	9171054	01 01	Boden 20-40 cm	56 unbeh.	nicht analysiert	
11	9171055	01 01	Boden 0-20 cm	84 unbeh.	nicht analysiert	
12	9171056	01 01	Boden 20-40 cm	84 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
13	9171057	01 01	Boden 0-20 cm	148 unbeh.	nicht analysiert	
14	9171058	01 01	Boden 20-40 cm	148 unbeh.	<0,01	<0,01
15	9172563	01 01	Boden 0-20 cm	273 unbeh.	nicht analysiert	
16	9172564	01 01	Boden 20-40 cm	273 unbeh.	<0,01	<0,01
17	9172565	01 01	Boden 0-20 cm	336 unbeh.	nicht analysiert	
18	9172566	01 01	Boden 20-40 cm	336 unbeh.	nicht analyisert	
19	9172567	01 01	Boden 0-20 cm	424 unbeh.	<0,01	<0,01
20	9172568	01 01	Boden 20-40 cm	424 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed,
'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
3	9171059	01 01	Boden 0-20 cm	0 vor Anw.	0,021	0,021
4	9171060	01 01	Boden 20-40 cm	0 vor Anw.	<0,01	<0,01
5	9171061	01 01	Boden 0-20 cm	0 nach Anw.	0,043/0,122	0,083
6	9171062	01 01	Boden 0-20 cm	7	0,185/0,089/0,138	0,137
7	9171063	01 01	Boden 20-40 cm	7	0,045	0,045
8	9171064	01 01	Boden 0-20 cm	14	0,180/0,134/0,120	0,145
9	9171065	01 01	Boden 20-40 cm	14	0,014	0,014
10	9171066	01 01	Boden 0-20 cm	28	0,245/0,109/0,172	0,175
11	9171067	01 01	Boden 20-40 cm	28	0,013	0,013
12	9171068	01 01	Boden 0-20 cm	56	0,144/0,174	0,159
13	9171069	01 01	Boden 20-40 cm	56	0,018	0,018
14	9171070	01 01	Boden 0-20 cm	84	0,054	0,054
15	9171071	01 01	Boden 20-40 cm	84	<0,01	<0,01

for legend: see page 2

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Residue (mg/kg TS) Mean value
16	9171072	01 01	Boden 0-20 cm	148	0,088	0,088
17	9171073	01 01	Boden 20-40 cm	148	0,016	0,016
18	9172569	01 01	Boden 0-20 cm	273	0,044	0,044
19	9172570	01 01	Boden 20-40 cm	273	<0,01	<0,01
20	9172571	01 01	Boden 0-20 cm	336	0,052	0,052
21	9172572	01 01	Boden 20-40 cm	336	<0,01	<0,01
22	9172573	01 01	Boden 0-20 cm	424	0,047	0,047
23	9172574	01 01	Boden 20-40 cm	424	<0,01	<0,01

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
1	9171074	03 01	Boden 0-20 cm	0 unbeh.	<0,01	<0,01
2	9171075	03 01	Boden 20-40 cm	0 unbeh.	<0,01	<0,01
3	9171076	03 01	Boden 0-20 cm	7 unbeh.	<0,01/<0,01	<0,01
4	9171077	03 01	Boden 20-40 cm	7 unbeh.	<0,01	<0,01
5	9171078	03 01	Boden 0-20 cm	14 unbeh.	nicht analysiert	
6	9171079	03 01	Boden 20-40 cm	14 unbeh.	nicht analysiert	
7	9171080	03 01	Boden 0-20 cm	28 unbeh.	nicht analysiert	
8	9171081	03 01	Boden 20-40 cm	28 unbeh.	<0,01	<0,01
9	9171082	03 01	Boden 0-20 cm	59 unbeh.	nicht analysiert	
10	9171083	03 01	Boden 20-40 cm	59 unbeh.	nicht analysiert	
11	9171084	03 01	Boden 0-20 cm	86 unbeh.	nicht analysiert	
12	9171085	03 01	Boden 20-40 cm	86 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
13	9171086	03 01	Boden 0-20 cm	154 unbeh.	<0,01	<0,01
14	9171087	03 01	Boden 20-40 cm	154 unbeh.	nicht analysiert	
15	9172575	03 01	Boden 0-20 cm	323 unbeh.	nicht analysiert	
16	9172576	03 01	Boden 20-40 cm	323 unbeh.	nicht analysiert	
17	9172577	03 01	Boden 0-20 cm	363 unbeh.	nicht analysiert	
18	9172578	03 01	Boden 20-40 cm	363 unbeh.	<0,01	<0,01
19	9172579	03 01	Boden 0-20 cm	457 unbeh.	nicht analysiert	
20	9172580	03 01	Boden 20-40 cm	457 unbeh.	nicht analysiert	

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed,
'vor Anw.' - before application, 'nach Anw.' - after application

Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
3	9171088	03 01	Boden 0-20 cm	0 vor Anw.	<0,01	<0,01
4	9171089	03 01	Boden 20-40 cm	0 vor Anw.	<0,01	<0,01
5	9171090	03 01	Boden 0-20 cm	0 nach Anw.	0,227/0,390	0,309
6	9171091	03 01	Boden 0-20 cm	7	0,081/0,082	0,082
7	9171092	03 01	Boden 20-40 cm	7	<0,01	<0,01
8	9171093	03 01	Boden 0-20 cm	14	0.164	0,164
9	9171094	03 01	Boden 20-40 cm	14	<0,01	<0,01
10	9171095	03 01	Boden 0-20 cm	28	0,180	0,180
11	9171096	03 01	Boden 20-40 cm	28	<0,01	<0,01
12	9171097	03 01	Boden 0-20 cm	59	0,070	0,070
13	9171098	03 01	Boden 20-40 cm	59	<0,01	<0,01
14	9171099	03 01	Boden 0-20 cm	86	0,032	0,032
15	9171100	03 01	Boden 20-40 cm	86	<0,01	<0,01

for legend: see page 2

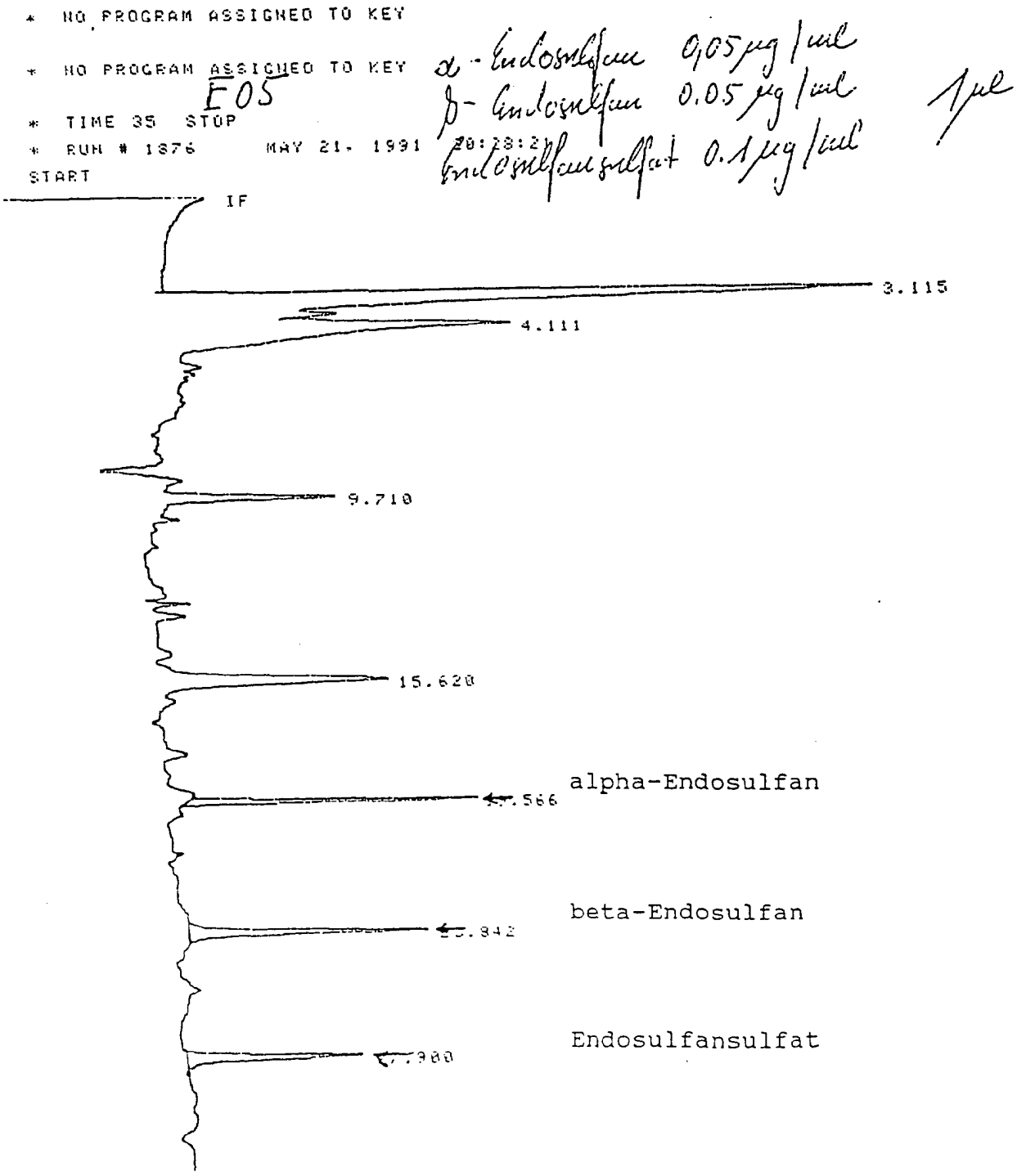
Table 3: Trial No. ER 90 DEU 720 0101; Residue values of alpha-endosulfan in soil

Sample No.	Analysis No.	Trial	Sample material	Days after application	Residue (mg/kg TS) Single value	Mean value
14	9171101	03 01	Boden 0-20 cm	154	0,034	0,034
15	9171102	03 01	Boden 20-40 cm	154	<0,01	<0,01
16	9172581	03 01	Boden 0-20 cm	323	0,047	0,047
17	9172582	03 01	Boden 20-40 cm	323	<0,01	<0,01
18	9172583	03 01	Boden 0-20 cm	363	0,225/0,271/0,210	0,235
19	9172584	03 01	Boden 20-40 cm	363	<0,01	<0,01
20	9172585	03 01	Boden 0-20 cm	457	0,038	0,038
21	9172586	03 01	Boden 20-40 cm	457	<0,01	<0,01

Legend: 'Boden' - soil, 'unbeh.' - untreated, 'nicht analysiert - not analysed, 'vor Anw.' - before application, 'nach Anw.' - after application

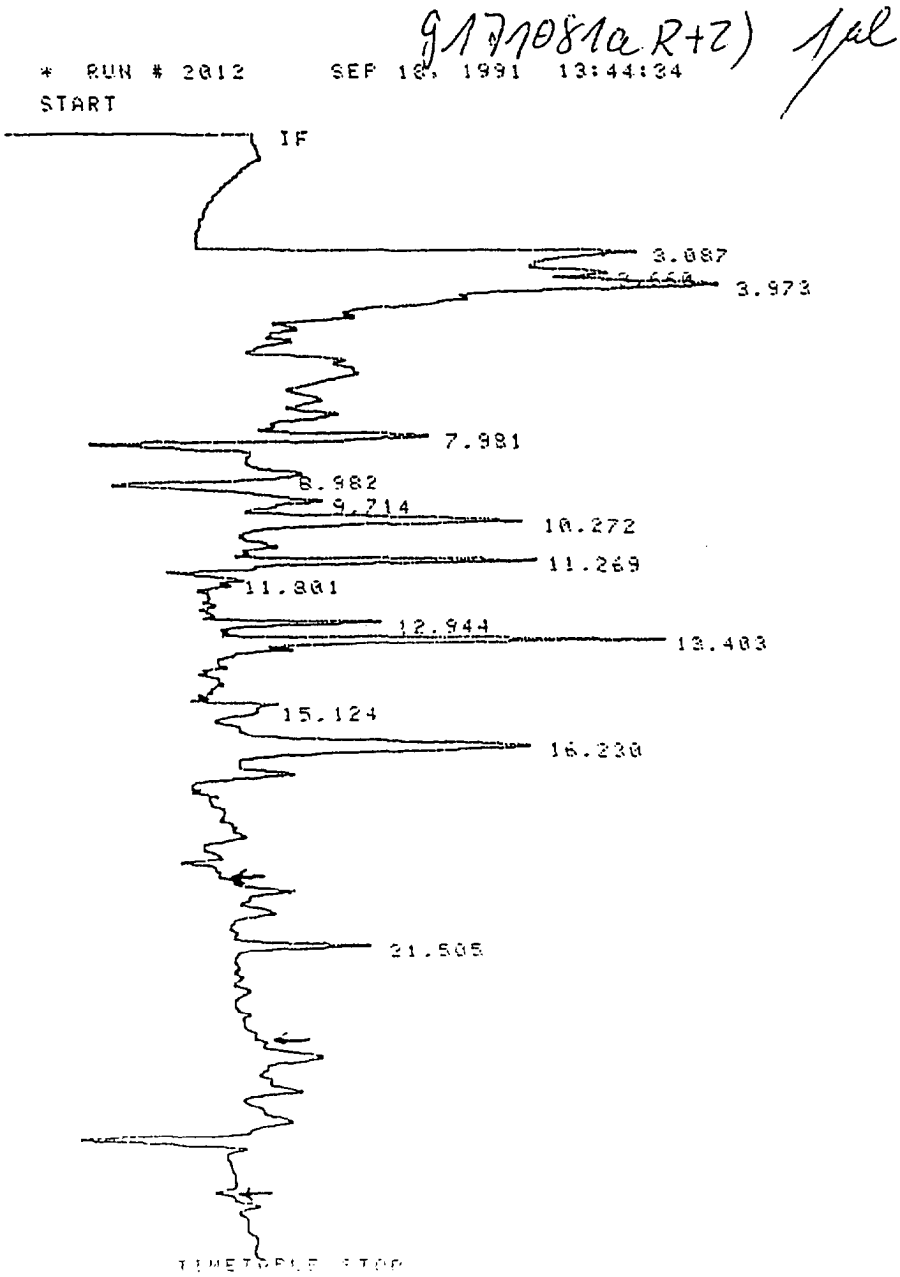
Enclosure 1: Figure 1 Sheet 1 (3)

typical chromatogram
standard solution
concentr.: alpha-endosulfan 0.05 µg/ml
beta-endosulfan 0.05 µg/ml
endosulfan-sulfate 0.10 µg/ml
GC-conditions: see chapter 2.2
injected volume: 1 µl



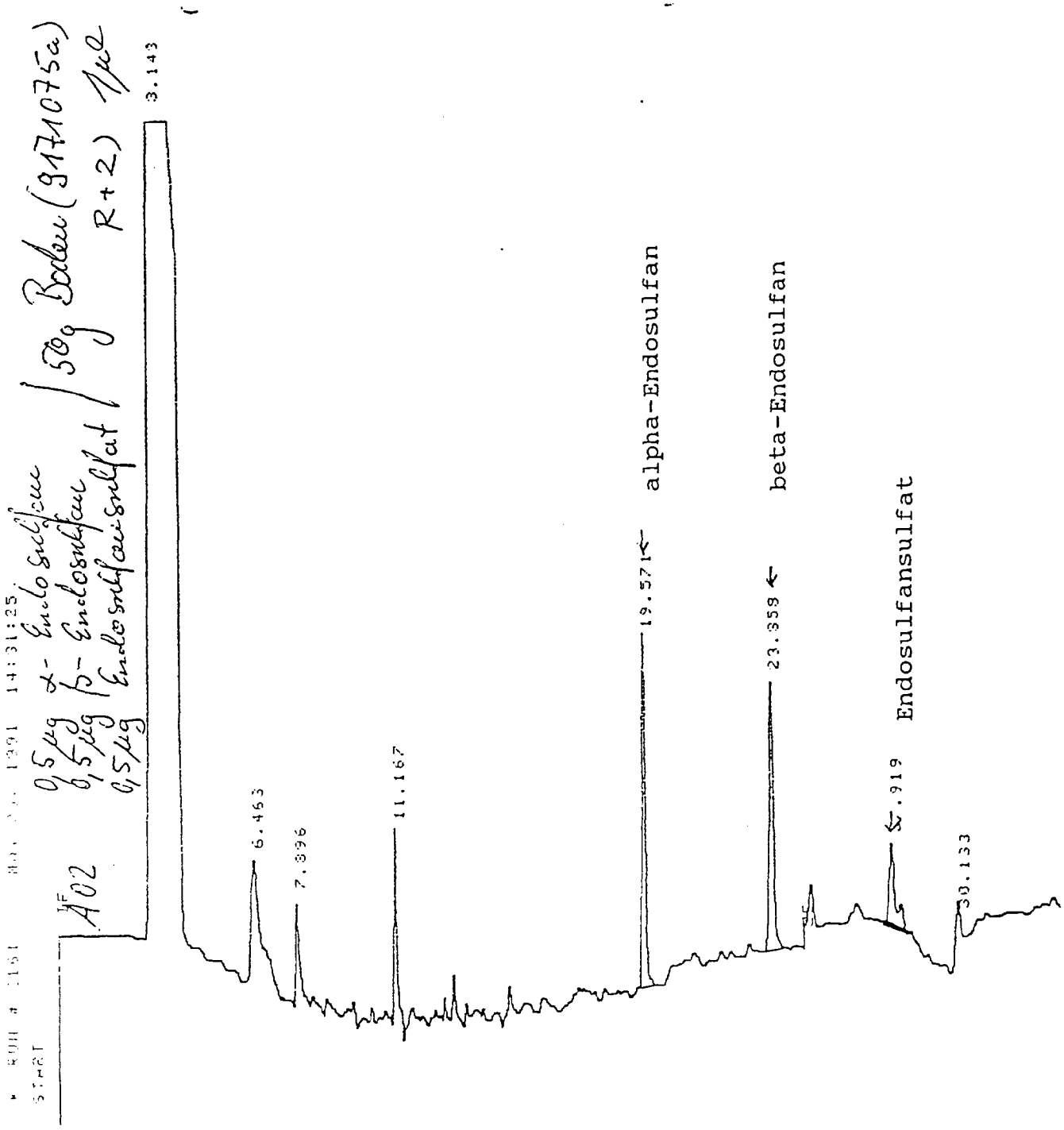
Enclosure 1: Figure 2 Sheet 2 (3)

typical chromatogram
untreated sample B010
50 g ---> 350 ml / 75 ml ---> 2 ml
GC-conditions: see chapter 2.2
injected volume: 1 µl



Enclosure 1: Figure 3 Sheet 3 (3)

typical chromatogram
untreated sample of soil
spiked control-sample A002
control + 0.1 mg/kg alpha-endosulfan
+ 0.1 mg/kg beta-endosulfan
+ 0.1 mg/kg endosulfan-sulfate
50 g ---> 350 ml / 75 ml ---> 2 ml
GC-conditions: see chapter 2.2
injected volume: 1 µl



Enclosure 2 : Calculation of results

The calculation of residues N_t in the soil is performed according to following equation:

$$N_t = \frac{F_p \times M_t \times V_1 \times V_2 \times 100}{F_s \times T_1 \times W \times TS}$$

with

F_p = Peak-height of sample (cm)
F_s = Peak-height of standard (cm)
M_t = concentration of standard (µg/ml)
V_1 = volume of solution for GC (ml)
V_2 = total volume (ml)
T_1 = partly volume of V2 (ml)
W = weight of sample taken (g)
TS = dry substance (%)

Enclosure 2: Calculation of results alpha-endosulfan

Sheet 1 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	a-endo-sulfan mg/kg TS Nt
21.05.91	9171045a	B001	5	4.6	-	0.05	2	350	75	50	87	-
23.05.91	9171046a	B005	15	4.3	-	0.05	2	350	75	50	87	-
21.05.91	9171047a	B002	5	4.6	-	0.05	2	350	75	50	89	-
23.05.91	9171048a	B006	15	4.3	-	0.05	2	350	75	50	89	-
12.09.91	9171049a	B009	26	4.6	-	0.05	2	350	75	50	87	-
24.09.91	9171057a	B011	no determination, bad recovery									
28.01.92	9171058a	B015	no determination, GPC defect									
07.04.92	9171058b	B017	139	6.1	-	0.025	2	350	75	50	88	-
21.05.91	9171059a	001	5	4.6	-	0.05	2	350	75	50	86	-
23.05.91	9171060a	007	15	4.3	-	0.05	2	350	75	50	86	-
22.05.91	9171061a	002	8	7.4	9.3	0.10	10	350	75	50	86	0.136
29.10.91	9171061b	032	70	6.2	7.1	0.10	16	350	75	50	86	0.199
22.05.91	9171062a	003	8	7.4	8.5	0.10	10	350	75	50	90	0.119
29.10.91	9171062b	033	70	6.2	6.2	0.10	16	350	75	50	90	0.166
28.01.92	9171062c	051	no determination, GPC defect									
11.03.92	9171062d	061	110	11.7	9.7	0.10	20	350	75	50	90	0.172
11.03.92	9171062d	061	112	8.4	9.7	0.05	20	350	75	50	90	0.120
24.05.91	9171063a	008	19	4.9	2.9	0.05	2	350	75	50	88	0.006
12.09.91	9171064a	012	27	8.2	6.6	0.10	16	350	75	50	86	0.139
29.10.91	9171064b	034	70	6.2	5.1	0.10	16	350	75	50	86	0.143
28.01.92	9171064c	052	no determination, GPC defect									
11.03.92	9171064d	062	113	7.5	8.3	0.05	20	350	75	50	86	0.120
17.09.91	9171065a	018	40	5.0	0.6	0.05	2	350	75	50	87	0.001

Enclosure 2: Calculation of results alpha-endosulfan

Sheet 2 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	a-endo-sulfan mg/kg TS Nt
16.09.91	9171066a	013	28	5.9	4.3	0.10	16	350	75	50	86	0.127
16.09.91	9171066a	013	29	3.7	4.3	0.05	16	350	75	50	86	0.101
30.10.91	9171066b	035	74	4.7	5.2	0.05	8	350	75	50	86	0.048
28.01.92	9171066c	053	no determination, GPC defect									
12.03.92	9171066d	063	116	10.2	8.8	0.10	10	350	75	50	86	0.094
12.03.92	9171066d	063	115	7.9	8.8	0.05	10	350	75	50	86	0.060
17.09.91	9171067a	019	40	5.0	0.4	0.05	2	350	75	50	86	0.001
16.09.91	9171068a	014	31	6.2	5.7	0.05	16	350	75	50	92	0.075
30.10.91	9171068b	036	74	4.7	4.1	0.05	16	350	75	50	92	0.071
18.09.91	9171069a	020	43	5.2	1.9	0.05	2	350	75	50	89	0.004
25.09.91	9171070a	024	no determination, bad recovery									
30.10.91	9171070b	037	76	3.9	4.9	0.05	4	350	75	50	86	0.027
24.09.91	9171071a	025	keine Auswertung, schlechte Wiederfindungsrate									
28.01.92	9171071b	054	keine Auswertung, GPC defekt									
19.03.92	9171071c	064	120	5.4	1.0	0.025	2	350	75	50	86	0.001
25.09.91	9171072a	026	no determination, bad recovery									
30.10.91	9171072b	038	76	3.9	5.0	0.05	4	350	75	50	85	0.028
25.09.91	9171073a	027	no determination, bad recovery									
28.01.92	9171073b	055	no determination, GPC defect									
19.03.92	9171073c	065	120	5.4	1.3	0.025	2	350	75	50	86	0.001
22.05.91	9171074a	B003	7	5.4	-	0.05	2	350	75	50	90	-
23.05.91	9171075a	B007	17	4.4	-	0.05	2	350	75	50	85	-
22.05.91	9171076a	B004	9	4.3	0.6	0.05	2	350	75	50	88	0.001
30.10.91	9171076b	B012	76	3.9	0.8	0.05	2	350	75	50	88	0.002

Enclosure 2: Calculation of results alpha-endosulfan

Sheet 3 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	a-endo-sulfan mg/kg TS Nt
23.05.91	9171077a	B008	17	4.4	-	0.05	2	350	75	50	86	-
18.09.91	9171081a	B010	43	5.5	-	0.05	2	350	75	50	92	-
31.01.92	9171086a	B016	106	5.2	-	0.05	2	350	75	50	88	-
23.05.91	9171088a	004	spilled during processing									
24.05.91	9171088b	011	19	4.9	-	0.05	2	350	75	50	87	-
23.05.91	9171089a	009	17	4.4	-	0.05	2	350	75	50	86	-
23.05.91	9171090a	090	13	8.6	9.1	0.10	30	350	75	50	92	0.322
23.03.92	9171090b	066	123	8.1	9.4	0.05	120	350	75	50	92	0.706
23.05.91	9171091a	006	13	8.6	11.4	0.10	5	350	75	50	88	0.070
30.01.92	9171091b	056	103	10.0	9.6	0.10	8	350	75	50	88	0.081
23.05.91	9171092a	010	17	4.4	2.6	0.05	2	350	75	50	86	0.006
16.09.91	9171093a	015	32	3.6	5.0	0.02	32	350	75	50	83	0.100
16.09.91	9171093a	015	31	6.2	5.0	0.05	32	350	75	50	83	0.145
18.09.91	9171094a	021	44	5.1	2.8	0.05	2	350	75	50	86	0.006
16.09.91	9171095a	016	31	6.2	5.3	0.05	16	350	75	50	86	0.074
16.09.91	9171095a	016	32	3.6	5.3	0.02	16	350	75	50	86	0.051
18.09.91	9171096a	022	44	5.1	1.4	0.05	2	350	75	50	86	0.003
17.09.91	9171097a	017	35	5.0	3.6	0.05	4	350	75	50	86	0.016
18.09.91	9171098a	023	44	5.1	0.7	0.05	2	350	75	50	86	0.001
25.09.91	9171099a	028	no determination, bad recovery									
30.01.92	9171099b	057	104	4.5	3.2	0.05	2	350	75	50	87	0.008
26.09.91	9171100a	029	no determination, bad recovery									
30.01.92	9171100b	058	104	4.5	0.5	0.05	2	350	75	50	86	0.001

Enclosure 2: Calculation of results alpha-endosulfan

Sheet 4 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	a-endo-sulfan mg/kg TS Nt
26.09.91	9171101a	030	no determination, bad recovery									
31.01.92	9171101b	059	106	5.2	2.2	0.05	2	350	75	50	86	0.007
27.09.91	9171102a	031	no determination, bad recovery									
31.01.92	9171102b	060	106	5.2	-	0.05	2	350	75	50	86	-
06.04.92	9172564a	B019	133	8.1	-	0.05	2	350	75	50	87	-
09.04.92	9172567b	B018	143	5.2	-	0.025	2	350	75	50	87	-
10.12.91	9172569a	039	84	12.0	7.8	0.10	2	350	75	50	88	0.014
10.12.91	9172569a	039	83	6.7	7.8	0.05	2	350	75	50	88	0.012
10.12.91	9172570a	040	83	6.7	-	0.05	2	350	75	50	88	-
10.12.91	9172571a	041	85	6.5	5.9	0.05	4	350	75	50	89	0.019
10.12.91	9172572a	042	85	6.5	-	0.05	2	350	75	50	87	-
10.12.91	9172573a	043	85	6.5	5.5	0.05	2	350	75	50	86	0.009
10.12.91	9172574a	044	85	6.5	-	0.05	2	350	75	50	88	-
11.12.91	9172578a	B014	89	7.5	-	0.05	2	350	75	50	85	-
11.12.91	9172581a	045	89	7.5	2.6	0.05	2	350	75	50	84	0.004
11.12.91	9172582a	046	89	7.5	-	0.05	2	350	75	50	85	-
11.12.91	9172583a	047	89	7.5	3.8	0.05	2	350	75	50	87	0.005
24.03.92	9172583b	067	127	8.5	2.6	0.05	4	350	75	50	87	0.007
25.03.92	9172583c	068	130	8.4	5.4	0.05	2	350	75	50	87	0.007
12.12.91	9172584a	048	94	5.0	-	0.05	2	350	75	50	85	-
12.12.91	9172585a	049	94	5.0	0.8	0.05	2	350	75	50	87	0.002
12.12.91	9172586a	050	94	5.0	-	0.05	2	350	75	50	85	-

Enclosure 2: Calculation of results beta-endosulfan

Sheet 5 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	b-endo-sulfan mg/kg TS Nt
21.05.91	9171045a	B001	5	3.8	2.0	0.05	2	350	75	50	87	0.006
23.05.91	9171046a	B005	15	3.2	1.5	0.05	2	350	75	50	87	0.005
21.05.91	9171047a	B002	5	3.8	1.7	0.05	2	350	75	50	89	0.005
23.05.91	9171048a	B006	15	3.2	0.8	0.05	2	350	75	50	89	0.003
12.09.91	9171049a	B009	26	3.2	3.3	0.05	2	350	75	50	87	0.011
24.09.91	9171057a	B011	no determination, bad recovery									
28.01.92	9171058a	B015	no determination, GPC defect									
07.04.92	9171058b	B017	139	5.4	3.3	0.025	2	350	75	50	88	0.003
21.05.91	9171059a	001	5	3.8	2.0	0.05	2	350	75	50	86	0.006
23.05.91	9171060a	007	15	3.2	1.1	0.05	2	350	75	50	86	0.004
22.05.91	9171061a	002	8	5.5	5.8	0.10	10	350	75	50	86	0.114
29.10.91	9171061b	032	70	5.7	5.7	0.10	16	350	75	50	86	0.173
22.05.91	9171062a	003	8	5.5	5.9	0.10	10	350	75	50	90	0.111
29.10.91	9171062b	033	70	5.7	5.5	0.10	16	350	75	50	90	0.160
28.01.92	9171062c	051	no determination, GPC defect									
11.03.92	9171062d	061	112	7.4	7.4	0.05	20	350	75	50	90	0.103
24.05.91	9171063a	008	19	3.5	2.8	0.05	2	350	75	50	88	0.008
12.09.91	9171064a	012	27	6.8	5.4	0.10	16	350	75	50	86	0.138
29.10.91	9171064b	024	70	5.7	4.8	0.10	16	350	75	50	86	0.146
28.01.92	9171064c	052	no determination, GPC defect									
11.03.92	9171064d	062	113	7.0	7.3	0.05	20	350	75	50	86	0.113
17.09.91	9171065a	018	40	3.9	2.8	0.05	2	350	75	50	87	0.008
16.09.91	9171066a	013	28	4.7	4.8	0.10	16	350	75	50	86	0.177
30.10.91	9171066b	035	75	6.0	5.7	0.10	8	350	75	50	86	0.082

Enclosure 2: Calculation of results beta-endosulfan

Sheet 6 (12)

Date	Analysis -No.	Lab. code	Peak-No.	Peak height std. sol.	Peak height sample	Conc. std. sol.	Volume sol. for GC	Total vol.	Part-vol. of V2	Weight sample	TS	b-endo-sulfan mg/kg TS
			E	cm Fs	cm Fp	µg/ml Mt	ml V1	ml V2	ml T1	g W	% TS	Nt
28.01.92	9171066c	053		no determination, GPC defect								
12.03.92	9171066d	063	116	9.5	9.7	0.10	10	350	75	50	86	0.111
17.09.91	9171067a	019	40	3.9	1.8	0.05	2	350	75	50	86	0.005
16.09.91	9171068a	014	33	7.0	7.2	0.10	16	350	75	50	92	0.167
30.10.91	9171068b	036	75	6.0	4.4	0.10	16	350	75	50	92	0.119
30.10.91	9171068b	036	76	3.3	4.4	0.05	16	350	75	50	92	0.108
18.09.91	9171069a	020	43	3.9	2.7	0.05	2	350	75	50	89	0.007
25.09.91	9171070a	024		no determination, bad recovery								
30.10.91	9171070b	037	75	6.0	6.4	0.10	4	350	75	50	86	0.046
24.09.91	9171071a	025		no determination, bad recovery								
28.01.92	9171071b	054		no determination, GPC defect								
19.03.92	9171071c	064	120	4.7	4.3	0.025	2	350	75	50	87	0.005
25.09.91	9171072a	026		no determination, bad recovery								
30.10.91	9171072b	038	75	6.0	7.5	0.10	4	350	75	50	85	0.055
25.09.91	9171073a	027		no determination, bad recovery								
28.01.92	9171073b	055		no determination, GPC defect								
19.03.92	9171073c	065	120	4.7	6.5	0.025	2	350	75	50	86	0.007
19.03.92	9171073c	065	119	7.5	6.5	0.05	2	350	75	50	86	0.009
22.05.91	9171074a	B003	7	4.1	0.8	0.05	2	350	75	50	90	0.002
23.05.91	9171075a	B007	17	3.1	-	0.05	2	350	75	50	85	-
22.05.91	9171076a	B004	9	2.8	-	0.05	2	350	75	50	88	-
30.10.91	9171076b	B012	76	3.3	0.7	0.05	2	350	75	50	88	0.002
23.05.91	9171077a	B008	17	3.1	-	0.05	2	350	75	50	86	-

Enclosure 2: Calculation of results beta-endosulfan

Sheet 7 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	b-endo-sulfan mg/kg TS Nt
18.09.91	9171081a	B010	43	3.9	-	0.05	2	350	75	50	92	-
31.02.92	9171086a	B016	106	4.2	1.0	0.05	2	350	75	50	88	0.002
23.05.91	9171088a	004	spilled during processing									
24.05.91	9171088b	011	19	3.5	-	0.05	2	350	75	50	87	-
23.05.91	9171089a	009	17	3.1	-	0.05	2	350	75	50	86	-
23.05.91	9171090a	005	13	7.2	5.5	0.10	30	350	75	50	92	0.232
23.03.92	9171090b	066	124	5.2	6.6	0.025	120	350	75	50	92	0.386
23.05.91	9171091a	006	13	7.2	9.3	0.10	5	350	75	50	88	0.068
30.01.92	9171091b	056	103	9.1	8.8	0.10	8	350	75	50	88	0.082
23.05.91	9171092a	010	17	3.1	1.9	0.05	2	350	75	50	86	0.007
16.09.91	9171093a	015	31	5.0	4.5	0.05	32	350	75	50	83	0.162
18.09.91	9171094a	021	44	3.8	2.4	0.05	2	350	75	50	86	0.007
16.09.91	9171095a	016	33	7.0	6.3	0.10	16	350	75	50	86	0.156
18.09.91	9171096a	022	44	3.8	1.6	0.05	2	350	75	50	86	0.005
17.09.91	9171097a	017	36	4.7	4.2	0.05	8	350	75	50	86	0.039
18.09.91	9171098a	023	44	3.8	1.7	0.05	2	350	75	50	86	0.005
26.09.91	9171099a	028	no determination, bad recovery									
30.01.92	9171099b	057	103	9.1	6.8	0.10	2	350	75	50	87	0.016
30.01.92	9171099b	057	104	3.4	6.8	0.05	2	350	75	50	87	0.021
26.09.91	9171100a	029	no determination, bad recovery									
30.01.92	9171100b	058	104	3.4	1.3	0.05	2	350	75	50	86	0.004
27.09.91	9171101a	030	no determination, bad recovery									
31.01.91	9171101b	059	106	4.2	6.8	0.05	2	350	75	50	86	0.018
31.01.92	9171101b	059	105	9.6	6.8	0.10	2	350	75	50	86	0.015

Enclosure 2: Calculation of results beta-endosulfan

Sheet 8 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	b-endo-sulfan mg/kg TS Nt
27.09.91	9171102a	031	no determination, bad recovery									
31.01.92	9171102b	060	106	4.2	-	0.05	2	350	75	50	86	-
06.04.92	9172564a	B019	133	7.3	2.3	0.05	2	350	75	50	87	0.003
09.04.92	9172567b	B018	143	4.6	-	0.025	2	350	75	50	87	-
10.12.91	9172569a	039	84	10.5	11.5	0.10	2	350	75	50	88	0.023
10.12.91	9172570a	040	83	5.2	3.1	0.05	2	350	75	50	88	0.006
10.12.91	9172571a	041	84	10.5	9.7	0.10	4	350	75	50	89	0.039
10.12.91	9172572a	042	83	5.2	2.5	0.05	2	350	75	50	87	0.005
10.12.91	9172573a	043	86	10.4	8.6	0.10	4	350	75	50	86	0.036
10.12.91	9172574a	044	85	5.2	3.4	0.05	2	350	75	50	88	0.007
11.12.91	9172578a	B014	89	5.9	-	0.05	2	350	75	50	85	-
11.12.91	9172581a	045	88	11.2	9.6	0.10	2	350	75	50	84	0.019
11.12.91	9172581a	045	89	5.9	9.6	0.05	2	350	75	50	84	0.018
11.12.91	9172582a	046	89	5.9	-	0.05	2	350	75	50	85	-
11.12.91	9172583a	047	91	8.1	8.0	0.05	2	350	75	50	87	0.011
24.03.92	9172583b	067	127	7.7	5.6	0.05	12	350	75	50	87	0.047
25.03.92	9172583c	068	130	7.6	5.7	0.05	12	350	75	50	87	0.048
12.12.91	9172584a	048	94	4.3	0.6	0.05	2	350	75	50	85	0.001
12.12.92	9172585a	049	94	4.3	3.9	0.05	2	350	75	50	87	0.010
12.12.91	9172586a	050	94	4.3	0.6	0.05	2	350	75	50	85	0.002

Enclosure 2: Calculation of results endosulfan-sulphate

Sheet 9 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	endos.-sulphate mg/kg TS Nt
21.05.91	9171045a	B001	5	2.8	1.9	0.10	2	350	75	50	87	0.015
23.05.91	9171046a	B005	15	2.1	–	0.10	2	350	75	50	87	–
21.05.91	9171047a	B002	5	2.8	1.9	0.10	2	350	75	50	89	0.014
23.05.92	9171048a	B006	15	2.1	–	0.10	2	350	75	50	89	–
12.09.91	9171049a	B009	26	2.6	2.8	0.10	2	350	75	50	87	0.023
24.09.91	9171057a	B011	no determination, bad recovery									
28.01.92	9171058a	B015	no determination, GPC defect									
07.04.92	9171058b	B017	139	5.4	2.8	0.05	2	350	75	50	88	0.005
21.05.91	9171059a	001	5	2.8	2.7	0.10	2	350	75	50	86	0.021
23.05.91	9171060a	007	15	2.1	0.9	0.10	2	350	75	50	86	0.009
22.05.91	9171061a	002	7	2.9	2.3	0.10	5	350	75	50	86	0.043
29.10.91	9171061b	032	70	4.8	3.2	0.20	8	350	75	50	86	0.116
29.10.91	9171061b	032	71	2.2	3.2	0.10	8	350	75	50	86	0.126
22.05.91	9171062a	003	8	3.3	5.9	0.20	5	350	75	50	90	0.185
29.10.91	9171062b	033	71	2.2	2.7	0.10	8	350	75	50	90	0.102
29.10.91	9171062b	033	70	4.8	4.4	0.20	4	350	75	50	90	0.076
28.01.92	9171062c	051	no determination, GPC defect									
11.03.92	9171062d	061	112	2.4	1.6	0.10	20	350	75	50	90	0.138
24.05.91	9171063a	008	18	2.8	3.0	0.20	2	350	75	50	88	0.045
12.09.91	9171064a	012	26	2.6	2.7	0.10	16	350	75	50	86	0.180
29.10.91	9171064b	034	71	2.2	1.7	0.10	16	350	75	50	86	0.134
28.01.92	9171064c	052	no determination, GPC defect									
11.03.92	9171064d	062	113	3.8	2.1	0.10	20	350	75	50	86	0.120

Enclosure 2: Calculation of results endosulfan-sulphate

Sheet 10 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	endos.-sulphate mg/kg TS Nt
17.09.91	9171065a	018	40	3.2	2.1	0.10	2	350	75	50	87	0.014
16.09.91	9171066a	013	29	1.7	2.4	0.10	16	350	75	50	86	0.245
30.10.91	9171066b	035	74	2.8	3.5	0.10	8	350	75	50	86	0.109
28.01.92	9171066c	053		no determination, GPC defect								
12.03.92	9171066d	063	115	2.5	4.2	0.10	10	350	75	50	86	0.182
12.03.92	9171066d	063	116	5.6	4.2	0.20	10	350	75	50	86	0.162
17.09.91	9171067a	019	41	3.5	2.1	0.10	2	350	75	50	86	0.013
16.09.91	9171068a	014	31	5.2	4.6	0.10	16	350	75	50	92	0.144
30.10.91	9171068b	036	74	2.8	3.0	0.10	16	350	75	50	92	0.174
18.09.91	9171069a	020	43	3.4	2.9	0.10	2	350	75	50	89	0.018
25.09.91	9171070a	024		no determination, bad recovery								
30.10.91	9171070b	037	74	2.8	3.5	0.10	4	350	75	50	86	0.054
24.09.91	9171071a	025		no determination, bad recovery								
28.01.92	9171071b	054		no determination, GPC defect								
19.03.92	9171071c	064	120	4.4	2.8	0.05	2	350	75	50	87	0.007
25.09.91	9171072a	026		no determination, bad recovery								
30.10.91	9171072b	038	77	5.5	5.5	0.20	4	350	75	50	85	0.088
25.09.91	9171073a	027		no determination, bad recovery								
28.01.92	9171073b	055		no determination, GPC defect								
19.03.92	9171073c	065	120	4.4	6.3	0.05	2	350	75	50	86	0.016
22.05.92	9171074a	B003	9	1.5	0.3	0.10	2	350	75	50	90	0.004
23.05.91	9171075a	B007	17	2.3	-	0.10	2	350	75	50	85	-
22.05.91	9171076a	B004	9	1.5	-	0.10	2	350	75	50	88	-
30.10.91	9171076b	B012	74	2.8	0.3	0.10	2	350	75	50	88	0.002

Enclosure 2: Calculation of results endosulfan-sulphate

Sheet 11 (12)

Date	Analysis -No.	Lab. code	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Weight sample g W	TS % TS	endos.-sulphate mg/kg TS Nt
23.05.91	9171077a	B008	17	2.3	-	0.10	2	350	75	50	86	-
18.09.91	9171081a	B010	43	3.4	-	0.10	2	350	75	50	92	-
31.01.92	9171086a	B016	105	7.8	0.5	0.20	2	350	75	50	88	0.002
23.05.91	9171088a	004		spilled during processing								
24.05.91	9171088b	011	19	1.6	-	0.10	2	350	75	50	87	-
23.05.91	9171089a	009	17	2.3	-	0.10	2	350	75	50	86	-
23.05.91	9171090a	005	11	5.5	4.1	0.20	15	350	75	50	92	0.227
23.03.92	9171090b	066	123	5.0	4.8	0.10	40	350	75	50	92	0.390
23.05.91	9171091a	006	13	6.0	4.6	0.20	5	350	75	50	88	0.081
30.01.92	9171091b	056	103	6.0	2.9	0.20	8	350	75	50	88	0.082
23.05.91	9171092a	010	17	2.3	0.5	0.10	2	350	75	50	86	0.005
16.09.91	9171093a	015	32	2.9	3.3	0.04	32	350	75	50	83	0.164
18.09.91	9171094a	021	44	3.5	1.6	0.10	2	350	75	50	86	0.009
16.09.91	9171095a	016	31	5.2	5.4	0.10	16	350	75	50	86	0.180
18.09.91	9171096a	022	44	3.5	0.5	0.10	2	350	75	50	86	0.003
17.09.91	9171097a	017	36	4.6	3.7	0.10	8	350	75	50	86	0.07
18.09.91	9171098a	023	44	3.5	1.4	0.10	2	350	75	50	86	0.009
26.09.91	9171099a	028		no determination, bad recovery								
30.01.92	9171099b	057	103	6.0	4.5	0.20	2	350	75	50	87	0.032
26.09.91	9171100a	029		no determination, bad recovery								
30.01.92	9171100b	058	103	6.9	0.7	0.20	2	350	75	50	86	0.004
27.09.91	9171101a	030		no determination, bad recovery								
31.01.92	9171101a	059	105	7.8	6.1	0.20	2	350	75	50	86	0.034

Enclosure 2: Calculation of results endosulfan-sulphate

Sheet 12 (12)

Date	Analysis -No.	Lab. code	Peak-No.	Peak height std. sol.	Peak height sample	Conc. std. sol.	Volume sol. for GC	Total vol.	Part-vol. of V2	Weight sample	TS	endos.-sulphate
			E	cm Fs	cm Fp	µg/ml Mt	ml V1	ml V2	ml T1	g W	% TS	mg/kg TS Nt
27.09.91	9171102a	031		no determination, bad recovery								
31.01.92	9171102b	060	105	7.8	-	0.20	2	350	75	50	86	-
06.04.92	9172564a	B019	133	5.8	2.2	0.10	2	350	75	50	87	0.008
09.04.92	9172567b	B018	143	4.9	-	0.05	2	350	75	50	87	-
10.12.91	9172569a	039	84	2.4	2.5	0.20	2	350	75	50	88	0.044
10.12.91	9172570a	040	84	2.4	-	0.20	2	350	75	50	88	-
10.12.91	9172571a	041	84	2.4	3.0	0.20	2	350	75	50	89	0.052
10.12.91	9172572a	042	85	1.7	0.5	0.10	2	350	75	50	87	0.006
10.12.91	9172573a	043	86	3.8	4.1	0.20	2	350	75	50	86	0.047
10.12.91	9172574a	044	86	3.8	-	0.20	2	350	75	50	88	-
11.12.91	9172578a	B014	89	1.3	-	0.10	2	350	75	50	85	-
11.12.91	9172581a	045	90	4.8	5.1	0.20	2	350	75	50	84	0.047
11.12.91	9172582a	046	91	3.3	-	0.10	2	350	75	50	85	-
11.12.91	9172583a	047	90	4.8	6.3	0.20	8	350	75	50	87	0.225
24.03.92	9172583b	067	126	7.4	7.8	0.20	12	350	75	50	87	0.271
25.03.92	9172583c	068	131	9.1	7.8	0.20	12	350	75	50	87	0.220
25.03.92	9172583c	068	130	5.0	7.8	0.10	12	350	75	50	87	0.210
12.12.91	9172584a	048	94	1.7	0.3	0.10	2	350	75	50	85	0.004
12.12.91	9172585a	049	94	1.7	3.2	0.10	2	350	75	50	87	0.040
12.12.91	9172585a	049	93	3.9	3.2	0.20	2	350	75	50	87	0.035
12.12.91	9172586a	050	94	1.7	-	0.10	2	350	75	50	85	-

Enclosure 3: Calculation of recoveries

The calculation of the recoveries in % is evaluated according to following equations:

$$M_f = \frac{F_p \times M_t \times V_1 \times V_2}{F_s \times T_1}$$

$$F = \frac{M_f \times 100}{M_1}$$

with

F_p = Peak-height of sample (cm)
F_s = Peak-height of standard (cm)
M_t = concentration of standard (µg/ml)
V_1 = volume of solution for GC (ml)
V_2 = total volume (ml)
T_1 = partly volume of V2 (ml)
M_f = found amount (µg)
M_1 = added amount (µg)

Enclosure 3 : Calculation recovery-rate of alpha-endosulfan

Sheet 1 (3)

Date	Material	Lab code	Alpha-endos. added	Peak-No.	Peak-height std. sol.	Peak-height sample	Conc. std. sol.	Volume sol. for GC	Total vol.	Part. vol. of V2	Alpha-endos. found	Recovery
			µg		cm	cm	µg/ml	ml	ml	ml	µg	%
		A	M1	E	Fs	Fp	Mt	V1	V2	T1	Mf	F
19.05.91	Boden (9171045)	01	25.0	2	12.2	12.0	0.20	25	350	75	22.95	92
25.05.91	Boden (9171075)	02	0.5	22	5.6	5.8	0.05	2	350	75	0.48	97
17.09.91	Boden (9171049)	03	5.0	37	9.1	9.1	0.10	10	350	75	4.67	93
18.09.91	Boden (9171081)	04	2.5	45	8.2	8.6	0.10	5	350	75	2.45	98
25.09.91	Boden (9171057)	05	1.0	56	6.2	6.5	0.10	2	350	75	0.98	98
30.10.91	Boden (9171076)	06	25.0	77	6.1	7.3	0.10	40	350	75	22.34	89
10.12.91	Boden (9172567)	07	2.5	83	6.7	8.5	0.05	8	350	75	2.37	85
		07	2.5	82	12.3	8.5	0.10	8	350	75	2.58	103
13.12.91	Boden (9172578)	08	0.5	99	7.7	6.0	0.05	2	350	75	0.36	73
28.01.92	Boden (9171058)	09	1.0		no determination							
31.02.92	Boden (9171086)	10	1.0	109	5.9	5.8	0.10	2	350	75	0.92	92
06.04.92	Boden (9171058)	11	1.0	134	9.9	9.5	0.10	2	350	75	0.90	90
06.04.92	Boden (9172564)	12	5.0	135	9.9	11.0	0.10	10	350	75	5.19	104

Enclosure 3 : calculation recovery-rate of beta-endosulfan

Sheet 2 (3)

Date	Material	Lab code A	Alpha-endos. added µg M1	Peak-No. E	Peak-height std. sol. cm Fs	Peak-height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part. vol. of V2 ml T1	beta-endos. found µg Mf	Recovery % F
19.05.91	Boden (9171045)	01	25.0	2	10.7	10.3	0.20	25	350	75	22.46	90
25.05.91	Boden (9171075)	02	0.5	22	4.4	4.4	0.05	2	350	75	0.47	93
17.09.91	Boden (9171049)	03	5.0	37	7.4	7.7	0.10	10	350	75	4.39*	88
18.09.91	Boden (9171081)	04	2.5	45	8.4	8.6	0.10	5	350	75	2.39	96
25.09.91	Boden (9171057)	05	1.0	56	6.1	6.0	0.10	2	350	75	0.46*	46
30.10.91	Boden (9171076)	06	25	77	5.7	7.0	0.10	40	350	75	22.93	92
10.12.91	Boden (9171567)	07	2.5	83	5.3	6.1	0.05	8	350	75	2.15	86
13.12.91	Boden (9171578)	08	0.5	99	6.5	6.5	0.05	2	350	75	0.47	93
28.01.92	Boden (9171058)	09	1.0		no determination							
31.02.92	Boden (9171086)	10	1.0	109	5.4	6.0	0.10	2	350	75	1.04	104
06.04.92	Boden (9171058)	11	1.0	134	10.0	8.0	0.10	2	350	75	0.75	75
06.04.92	Boden (9172564)	12	5.0	135	10.3	9.6	0.10	10	350	75	4.35	87

Enclosure 3 : Calculation recovery-rate of endosulfan-sulfate

Sheet 3 (3)

Date	Material	Lab code	Alpha-endos. added	Peak-No.	Peak-height std. sol.	Peak-height sample	Conc. std. sol.	Volume sol. for GC	Total vol.	Part. vol. of V2	endos.-sulphate found	Recovery
			µg		cm	cm	µg/ml	ml	ml	ml	µg	%
		A	M1	E	Fs	Fp	Mt	V1	V2	T1	Mf	F
19.05.91	Boden (9171045)	01	25.0	1	5.5	6.3	0.20	25	350	75	26.09*	104
25.05.91	Boden (9171075)	02	0.5	24	1.1	1.3	0.04	2	350	75	0.44	88
17.09.91	Boden (9171049)	03	5.0	36	4.7	5.1	0.10	10	350	75	4.05*	81
18.09.91	Boden (9171081)	04	2.5	44	3.5	4.5	0.10	5	350	75	3.00	120
25.09.91	Boden (9171057)	05	1.0	56	5.8	6.0	0.20	2	350	75	0.28*	28
30.10.91	Boden (9171076)	06	25.0	77	5.5	4.4	0.20	40	350	75	29.87	119
09.12.91	Boden (9171567)	07	2.5	80	3.6	3.5	0.20	3	350	75	2.72	109
13.12.91	Boden (9171578)	08	0.5	99	1.8	1.0	0.10	2	350	75	0.52	104
28.01.92	Boden (9171058)	09	1.0		no determination							
31.01.92	Boden (9171086)	10	1.0	107	8.0	3.8	0.20	2	350	75	0.887	89
06.04.92	Boden (9171058)	11	1.0	133	5.8	7.1	0.10	2	350	75	1.14	114
06.04.92	Boden (9172564)	12	5.0	136	7.4	6.7	0.10	10	350	75	4.23	85

A54025

Title

Project ER 90 DEU 720

Hoe 002671 (endosulfan) and Hoe 051327 (endosulfan-sulfate)
Storage stability in soil

Author

G.Fuchsbichler
21 February 1992

Test Facility

Bayrische Hauptversuchsanstalt für Landwirtschaft
TUM-Weihenstephan
D-8050 Freising 1

Study Plan No.
HVA 7/91

Statement of Compliance:

This study was performed in accordance with the Principles of Good Laboratory Practice.

No unforeseen circumstances were observed which might have affected the quality and integrity of the presented study.

Study Director: Dr. G. Fuchsbichler

Signature: - signed - Date: _21 Feb 1992_

Head of the Test Facility: Prof. Dr. K. Ranfft

Signature: - signed - Date: 21 Feb 1992

Quality Assurance Statement

The authorized representative for quality assurance of the laboratory periodically inspected the performed examinations as well as the final report and a report of these inspections was submitted to the Study Director.

Day of inspection	Report
03.05.91	03.05.91
08.08.91	09.08.91
28.02.92	03.03.92

Referat GLP: Dr. R. Gerstl

Signature: - signed - Date: 03 Mar 1992

1. Organisation and personnel

Sponsor:	Hoechst Aktiengesellschaft Business Unit Agriculture Product Development Ecology II Building G864 6230 Frankfurt am Main 80
Monitoring analyst:	Dr. H. Idstein for address; see sponsor
Test Facility:	Bayerische Hauptversuchsanstalt für Landwirtschaft D-8050 Freising 1 Tel.: 08161/713381 Head: Prof. Dr. K. Ranfft
Study Director:	Dr. G. Fuchsbichler for address; see Test Facility Tel.: 08161/713381
Quality Assurance:	Dr. R. Gerstl for address; see Test Facility Tel.: 08161/713381

Archiving:
The study plan, raw data, and original final report will be filed in the archive of the Test Facility for at least the period prescribed in the GLP-Guidelines. Subsequently, the documents may only be destroyed with the consent of the sponsor. If the archive is dissolved before termination of the prescribed filing period, the documents are to be returned to the sponsor.

Time schedule:

Drawing-up of study plan:	16.04.1991
Start of the experimental phase:	08.05.1991
End of the experimental phase:	20.08.1991
Completion of the final report:	21.02.1992

2. Performance of the study

The laboratory samples of the project ER20DEU720 were received on 09 April 1991 in the deep-frozen state, were registered and stored at -20°C until preparation of the study. Before the start of the analytical preparations the frozen, untreated samples were defrosted in the refrigerator at a temperature of 4° - 7° C and spiked with alpha-endosulfan, beta-endosulfan and endosulfan-sulfate. Additionally 2 parallel blank samples were stored for each sampling date.
The dates on which the samples were worked up and measured are set out in Table 1.
Untreated controls were first spiked and then used as fresh laboratory spiking for the determination of recoveries.
More details will be found in Table 1.

2.1 Analytical preparations

2.1.1. Production of the standard solutions

Alpha-endosulfan, beta-endosulfan and endosulfan-sulfate were dissolved separately in acetone. Solutions were prepared by dilution, containing 5 µg alpha-endosulfan, 5 µg beta-endosulfan and 5 µg endosulfan sulfate/ml.

2.1.2 Spiking
50 g soil were weighed into screw-top flasks and each mixed separately with 1 ml of the standard solution from 2.1.1. The flasks were then closed and the content shaken carefully to obtain a largely homogeneous distribution of the test substances. Untreated blank samples were prepared at the same time.

2.1.3 Storage Conditions
The samples were stored at 0°C in darkness

2.1.4 Sampling
The samples were analysed after 1, 2 and 3 months. For each date of sampling two spikings of alpha-endosulfan, beta-endosulfan and endosulfan sulfate were analysed as well as two blank samples stored in parallel. At each date a fresh laboratory spiking was also examined.

Analytical preparations (date)	Start of processing
08.05.91	06.06.91
08.05.91	03.07.91
08.05.91	13.08.91

3 ANALYSIS OF RESIDUES

3.1. Test substances

3.1.1. Hoe 052618 (alpha-endosulfan)
Empirical formula: $C_9H_6Cl_6\ O_3S$
Molar mass: 4069 g/mol
Name of compound: 6,7,8,9,10,10,-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzo-dioxathiepin-3-oxide (IUPAC, English)

Purity: 99.8%
No. of batch: 01290
No. of certificate: AZ 04457

3.1.2. Hoe 052619 (beta-endosulfan)

Empirical formula:	$C_9H_6Cl_6O_3S$
Molar mass:	406.9 g/mol
Name of compound:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzo-dioxathiepin-3-oxide-beta-isomer (IUPAC, English)
Purity:	99.3 %
No. of batch:	90790
No. of certificate:	AZ 04152

3.1.3. Hoe 051327 (endosulfan-sulfate)

Empirical formula:	$C_9H_6Cl_6O_4S$
Molar mass:	422.9 g/mol
Name of compound:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzo-dioxathiepin-3,3-dioxide (IUPAC, English)
Purity:	97.9 %
No. of batch:	01480
No. of certificate:	AZ 04493

3.2. Reference substances:

3.2.1. Hoe 052618 (alpha-endosulfan)

Empirical formula:	$C_9H_6Cl_6O_3S$
Molar mass:	406.9 g/mol
Name of compound:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzo-dioxathiepin-3-oxide-alpha-isomer (IUPAC, English
Purity:	99.8 %
No. of batch:	01290
No. of certificate:	AZ 04457

3.2.2. Hoe 052619 (beta-endosulfan)

Empirical formula:	$C_9H_6Cl_6O_3S$
Molar mass:	406.9 %
Name of compound:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzo-dioxathiepin-3-oxide-beta-isomer (IUPAC, English)
Purity:	99.3 %
No. of batch:	90790
No. of certificate:	AZ 04152

3.2.3. Hoe 051327 (endosulfan-sulfate)

Empirical formula:	$C_9H_6Cl_6O_4S$
Molar mass:	422.9 g/mol
Name of compound:	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzo-dioxathiepin-3,3-dioxide
Purity:	97.9 %
No. of batch:	01480
No. of certificate:	AZ 04493

in hexane in different concentrations
in acetone in different concentrations

3.3 Test methods

The method of analysis AL 60/86 of Hoechst AG was used for detection of endosulfan. The active ingredient and its metabolite were extracted with acetone from the samples. After clean-up by gel permeation chromatography on Bio-Beads SX-3, the final determination was carried out by gas chromatography with an electron capture detector (ECD). Determination of the residues was conducted by the external standard method. There was no correction with recoveries.

Modifications

1. Point 6.1.2 of the analytical procedure
   Filtration is carried out into a 500 ml mixing cylinder and the volume is made up to 300 - 350 ml. An aliquot of 75 - 150 ml is processed.

2. Point 6.2.1 of the analytical procedure
   The organic phase is concentrated to a volume of about 5 ml on the rotary evaporator, the rest is blown to dryness under a gentle nitrogen stream. The residue was absorbed in 10 ml GPC mixture of cyclohexane/dichloromethane = 85/15.

3. Point 6.3.1 of the analytical procedure

GPC conditions:
Elution mixture: cyclohexane/dichloromethane = 85/15.
Dump : 24 min
Collect : 40 min
Post-column: 3 g 15 % deactivated Florisil
The collect phase was conducted directly in an on-line procedure via a column filled with 3 g 15 % deactivated Florisil.

4. Point 6.3.2 of the analytical procedure
   Clean-up by silica gel mini column omitted

5. Point 6.4.1 of the analytical procedure
   Gas chromatographic conditions

Equipment:	model 8521a (Dani company)
detector:	electron capture detector (ECD) (Dani company)
separation column:	Fused silica FS 54 length: 50 m internal diameter: 0.22 mm thickness of film: 0.33 µm
gases:carrier gas:	helium 4.6, 4 ml/min
make-up gas:	nitrogen 5.0, 45 ml/min

Operating temperature: injector: 250°C
detector: 300°C
temperature program oven:
T_1: 150°C
I_1: 1 min
R_1: 15°C/min
T_2: 245°C
I_2: 30 min

Injection: splitless, opening of split after 0.8 min

Injected volume: 1 µl

One representative chromatogram of the reference substance, of an untreated sample and of a spiked untreated sample have been enclosed.

3.4 Recoveries and results for untreated samples :

As an internal quality assurance analyses of untreated samples and spiked untreated samples were carried out for the sample material using the substance under analysis. The recoveries obtained for alpha-endosulfan, beta-endosulfan and endosulfan-sulfate are listed in Table 2.

4. RESULTS

The results of the residue analyses are given in mg/kg in Table 3 as well as in % of initially applied content in Table 4. Results of analyses below the lower limit of the practical working range have been shown as < 0.01 mg/kg.

5. DISCUSSION

The method used here enabled endosulfan and endosulfan-sulfate to be detected selectively. The untreated samples contained no substances interfering with determination. It was shown that under the given storage conditions no decrease of endosulfan and endosulfan-sulfate was recorded during the first 2 months. Between 83 % and 117 % of the applied initial concentration could be detected. After a storage time of 3 months the recovery of alpha-endosulfan was 44 %, of beta-endosulfan 67 % and of endosulfan-sulfate 70 %.

Author:	21 Feb 1992	(Signed)
Study Director:	21 Feb 1992	(Signed)

Enclosures: chromatograms of representative samples
calculated results
calculated recoveries

Literature:

(1) H. - J. Werner, G. Klante, H. D. Merz
Hoe 002671 (Endosulfan)
Rückstandsbestimmung von Wirkstoff und Endosulfansulfat in Boden, Wasser, Urin und Pflanzen sowie von Endosulfandiol und Endosulfanlacton in Boden, Wasser und Urin
Method AL 60/86 dated 10/12/86

Table 1: Test-No. ER 90 DEU 720; samples and working-up dates Sheet 1 (2)

Analysis No.	Test-material	Rem.*	Lab code	Months after final appl.	Sample received	Preparation of test	Start of working-up	Date of measurement
9171200	soil	u	B001	1	09.04.91	08.05.91	06.06.91	09.06.91
9171204	"	u	B002	1	"	"	06.06.91	09.06.91
9171208	"	b	L001	1	"	"	06.06.91	08.06.91
9171214	"	b	L002	1	"	"	06.06.91	08.06.91
9171220	"	b	L003	1	"	"	06.06.91	08.06.91
9171226	"	b	L004	1	"	"	06.06.91	08.06.91
9171232	"	b	L005	1	"	"	06.06.91	09.06.91
9171238	"	b	L006	1	"	"	06.06.91	no determination
	"	R	A001	1	"	"	06.06.91	12.06.91
	"	R	A001	1	"	"	06.06.91	14.06.91
9171201	"	u	B003	2	"	"	03.07.91	09.07.91
9171205	"	u	B004	2	"	"	03.07.91	09.07.91
9171209	"	b	L007	2	"	"	03.07.91	09.07.91
9171215	"	b	L008	2	"	"	03.07.91	09.07.91
9171221	"	b	L009	2	"	"	03.07.91	09.07.91
9171227	"	b	L010	2	"	"	03.07.91	09.07.91
9171233	"	b	L011	2	"	"	03.07.91	10.07.91
9171239	"	b	L012	2	"	"	03.07.91	10.07.91
	"	R	A002	2	"	"	03.07.91	09.07.91
	"	R	A002	2	"	"	03.07.91	10.07.91

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see Table 2)
Remark: the untreated samples were worked up in series together with the treated samples.

Table 1: Test-No. ER 90 DEU 720; samples and working-up dates Sheet 2 (2)

Analysis No.	Test-material	Rem.*	Lab code	Months after final appl.	Sample received	Preparation of test	Start of working-up	Date of measurement
9171202	"	u	B005	3	09.04.91	08.05.91	13.08.91	20.08.91
9171206	"	u	B006	3	"	"	13.08.91	20.08.91
9171210	"	b	L013	3	"	"	13.08.91	20.08.91
9171216	"	b	L014	3	"	"	13.08.91	20.08.91
9171222	"	b	L015	3	"	"	13.08.91	20.08.91
9171228	"	b	L016	3	"	"	13.08.91	20.08.91
9171234	"	b	L017	3	"	"	13.08.91	20.08.91
9171240	"	b	L018	3	"	"	13.08.91	20.08.91
	"	R	A003	3	"	"	13.08.91	20.08.91

* b: treated sample, u: untreated sample, R: untreated sample spiked for recovery (see table 2)
Remark: the untreated samples were worked up in series together with the treated samples.

Sheet 1 (3)

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate [%]
soil	B001	0	<0,01	
"	B002	0	<0,01	
"	B003	0	<0,01	
"	B004	0	<0,01	
"	B005	0	<0,01	
"	B006	0	<0,01	
"	A001	0,10	0,076	76
"	A002	0,10	0,111	111
"	A003	0,10	0,076	76

Sheet 2 (3)

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate [%]
soil	B001	0	<0,01	
"	B002	0	<0,01	
"	B003	0	<0,01	
"	B004	0	<0,01	
"	B005	0	<0,01	
"	B006	0	0,021	
"	A001	0,10	0,081	81
"	A002	0,10	0,117	117
"	A003	0,10	0,086	86

Sheet 3 (3)

Table 2: Test-No. ER 90 DEU 720; results of untreated samples and recovery-rates

Testmaterial	Laboratory code	Admixture alpha-endos. [mg/kg]	Found alpha-endos. [mg/kg]	Recovery-rate [%]
soil	B001	0	<0,01	
"	B002	0	<0,01	
"	B003	0	<0,01	
"	B004	0	<0,01	
"	B005	0	<0,01	
"	B006	0	<0,01	
"	A001	0,10	0,093	93
"	A002	0,10	0,096	96
"	A003	0,10	0,078	78

Sheet 1 (2)

Table 3: Test-No. ER 90 DEU 720; results storage stability of endosulfan in soil

Laboratory code	Analysis-No.	Storage period	Spiking	Alpha-endosulfan	Spiking	Beta-endosulfan	Spiking	Endosulfan-sulfate
		months	mg/kg	recorded mg/kg	mg/kg	recorded mg/kg	mg/kg	recorded mg/kg
L001	9171208	1	0,10	0,091				
L002	9171214	1	0,10	0,085				
L007	9171209	2	0,10	0,093				
L008	9171215	2	0,10	0,099				
L013	9171210	3	0,10	0,040				
L014	9171216	3	0,10	0,048				
L003	9171220	1			0,10	0,106		
L004	9171226	1			0,10	0,117		
L009	9171221	2			0,10	0,096		
L010	9171227	2			0,10	0,109		
L015	9171222	3			0,10	0,068		
L016	9171228	3			0,10	0,065		

Sheet 2 (2)

Table 3: Test-No. ER 90 DEU 720; results storage stability of endosulfan in soil

Laboratory code	Analysis-No.	Storage period	Spiking	Alpha-endosulfan	Spiking	Beta-endosulfan	Spiking	Endosulfan-sulfate
		months	mg/kg	recorded mg/kg	mg/kg	recorded mg/kg	mg/kg	recorded mg/kg
L005	9171232	1					0,10	0,116
L006	9171238	1					sample spilled	
L011	9171233	2					0,10	0,093
L012	9171239	2					0,10	0,083
L017	9171234	3					0,10	0,066
L018	9171240	3					0,10	0,073

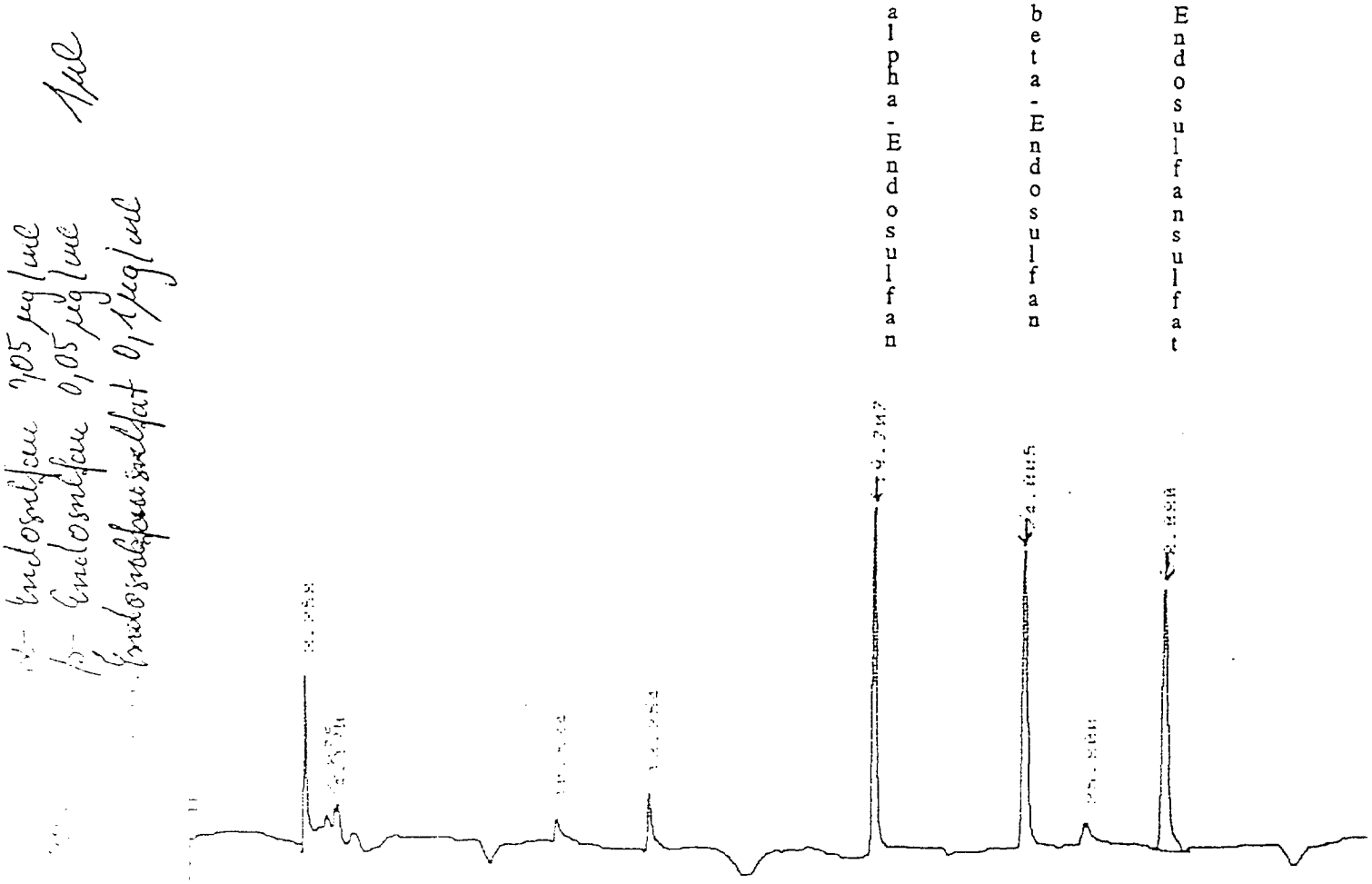
Sheet 1 (1)

Table 4: Test-No. ER 90 DEU 720; storage stability endosulfan in soil
results in % of initial concentration

period of storage	alpha-endosulfan			beta-endosulfan			endosulfan-sulfate		
	1	2	x	1	2	x	1	2	x
1 month	91	85	88	106	117	112	116	-	116
2 months	93	99	96	96	109	103	95	83	89
3 months	40	48	44	68	65	67	66	73	70

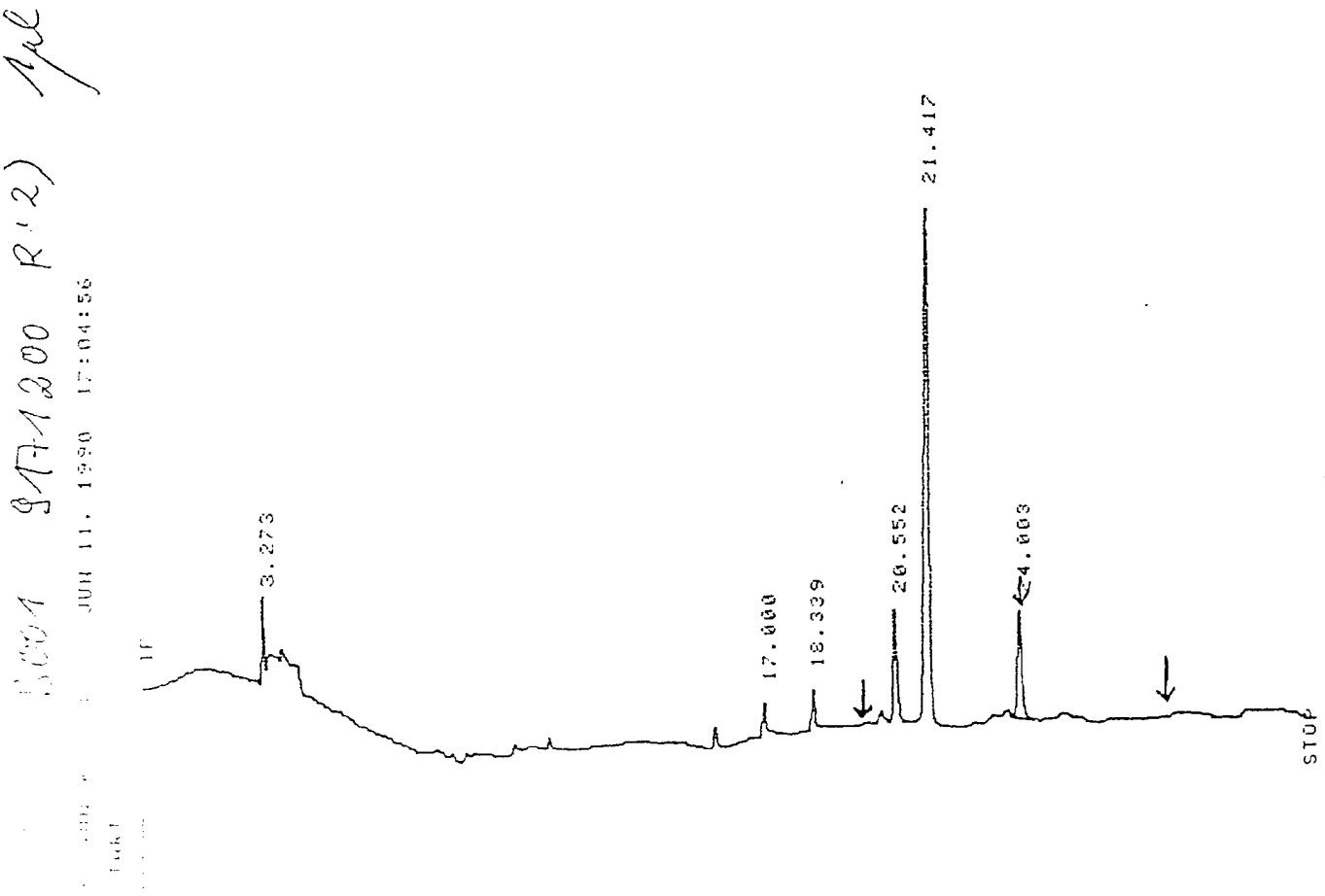
Enclosure 1: Figure 1 Sheet 1 (3)

typical chromatogram
standard solution
concentr.: alpha-endosulfan 0.05 µg/ml
beta-endosulfan 0.05 µg/ml
endosulfan-sulfate 0.10 µg/ml
GC-conditions: see chapter 3.3
injected volume: 1 µl



Enclosure 1: Figure 2 Sheet 2 (3)

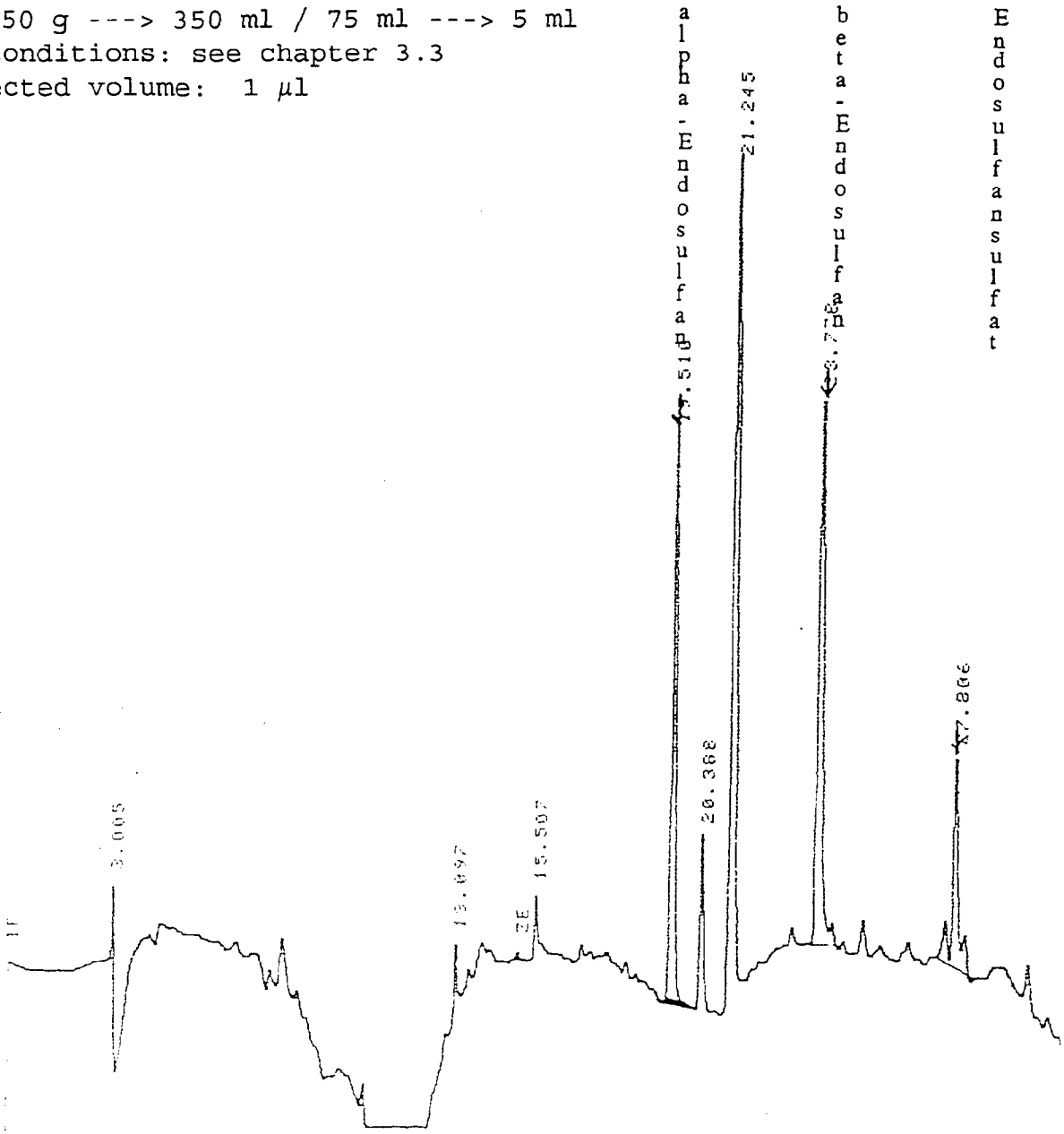
typical chromatogram
untreated sample of soil B001
50 g ---> 300 ml / 150 ---> 2 ml
GC-conditions: see chapter 3.3
injected volume: 1 µl



Enclosure 1: Figure 3

Sheet 3 (3)

typical chromatogram
untreated sample of soil
spiked control-sample A003
control + 0.1 mg/kg alpha-endosulfan
+ 0.1 mg/kg beta-endosulfan
+ 0.1 mg/kg endosulfan-sulfate
50 g ---> 350 ml / 75 ml ---> 5 ml
GC-conditions: see chapter 3.3
injected volume: 1 µl



Enclosure 2 : Calculation of results

The calculation of residues F in the soil is performed according to following equation:

$$F = \frac{F_p \times M_t \times V_1 \times V_2}{F_s \times T_1 \times W}$$

with

F_p = Peak-height of sample (cm)
F_s = Peak-height of standard (cm)
M_t = concentration of standard (µg/ml)
V_1 = volume of solution for GC (ml)
V_2 = total volume (ml)
T_1 = partly volume of V2 (ml)
W = weight of sample taken (g)

Enclosure 2: Calculation of results alpha-endosulfan

Sheet 1 (3)

Date	Analysis -No.	Lab. code	Alpha endos. added µg M1	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Alpha-endos. found µg Mf	Weight sample g W	Recovery mg/kg F
08.06.91	9171208	L001	5,0	2	3,4	3,1	0,10	25	300	150	4,56	50	0,091
08.06.91	9171214	L002	5,0	3	7,3	6,2	0,10	25	300	150	4,25	50	0,085
09.06.91	9171204	B002	-	6	6,0	-	0,05	2	300	150	-	50	-
09.06.91	9171200	B001	-	7	5,2	-	0,05	2	300	150	-	50	-
09.07.91	9171209	L007	5,0	16	11,3	10,5	0,10	25	300	150	4,65	50	0,093
09.07.91	9171215	L008	5,0	18	11,3	11,2	0,10	25	300	150	4,96	50	0,099
09.07.91	9171201	B003	-	19	7,4	-	0,05	2	300	150	-	50	-
09.07.91	9171205	B004	-	19	7,4	-	0,05	2	300	150	-	50	-
20.08.91	9171210	L013	5,0	28	7,8	6,7	0,10	5	350	75	2,00	50	0,040
20.08.91	9171216	L014	5,0	2	7,8	8,1	0,10	5	350	75	2,42	50	0,048
20.08.91	9171202	B005	-	29	8,0	-	0,10	2	350	75	-	50	-
20.08.91	9171206	B006	-	29	8,0	-	0,10	2	350	75	-	50	-

Enclosure 2: Calculation of results beta-endosulfan

Sheet 2 (3)

Date	Analysis No.	Lab. code	Alpha endos. added µg M1	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Alpha-endos. found µg Mf	Weight sample g W	Recovery mg/kg F
08.06.91	9171220	L003	5,0	3	6,4	6,8	0,10	25	300	150	5,30	50	0,106
08.06.91	9171226	L004	5,0	3	6,4	7,5	0,10	25	300	150	5,86	50	0,117
09.06.91	9171204	B002	-	6	4,8	1,9	0,05	2	300	150	0,079	50	0,002
09.06.91	9171200	B001	-	7	4,2	1,5	0,05	2	300	150	0,071	50	0,002
09.07.91	9171221	L009	5,0	18	7,7	7,4	0,10	25	300	150	4,81	50	0,096
09.07.91	9171227	L010	5,0	18	7,7	8,4	0,10	25	300	150	5,45	50	0,109
09.07.91	9171201	B003	-	19	4,7	3,6	0,05	2	300	150	0,15	50	0,003
09.07.91	9171205	B004	-	19	4,7	1,9	0,05	2	300	150	0,081	50	0,002
20.08.91	9171222	L015	5,0	27	8,7	7,5	0,20	5	350	75	4,0	50	0,080
20.08.91	9171222	L015	5,0	28	6,2	7,5	0,10	5	350	75	2,82	50	0,056
20.08.91	9171228	L016	5,0	27	8,7	7,1	0,20	5	350	75	3,80	50	0,076
20.08.91	9171228	L016	5,0	28	6,2	7,1	0,10	5	350	75	2,67	50	0,053
20.08.91	9171202	B005	-	29	6,3	7,1	0,10	2	350	75	1,05	50	0,021
20.08.91	9171206	B006	-	29	6,3	1,7	0,10	2	350	75	0,25	50	0,005

Enclosure 2: Calculation of results endosulfane-sulfate

Sheet 3 (3)

Date	Analysis -No.	Lab. code	Alpha endos. added µg M1	Peak-No. E	Peak height std. sol. cm Fs	Peak height sample cm Fp	Conc. std. sol. µg/ml Mt	Volume sol. for GC ml V1	Total vol. ml V2	Part-vol. of V2 ml T1	Alpha-endos. found µg Mf	Weight sample g W	Recovery mg/kg F
09.06.91	9171232	L005	5,0	5	3,8	4,4	0,10	25	300	150	5,79	50	0,116
09.06.91	9171238	L006	5,0		sample spilled during treatment								
09.06.91	9171204	B002	-	6	2,0	-	0,10	2	300	150	-	50	-
09.06.91	9171200	B001	-	7	2,0	-	0,10	2	300	150	-	50	-
09.07.91	9171201	B003	-	19	1,6	-	0,10	2	300	150	-	50	-
09.07.91	9171205	B004	-	19	1,6	-	0,10	2	300	150	-	50	-
10.07.91	9171233	L011	5,0	23	4,9	5,8	0,20	5	300	75	4,73	50	0,095
10.07.91	9171239	L012	5,0	23	4,9	5,1	0,20	5	300	75	4,16	50	0,083
20.08.91	9171234	L017	5,0	29	3,7	2,6	0,20	5	350	75	3,28	50	0,066
20.08.91	9171240	L018	5,0	29	3,7	2,9	0,20	5	350	75	3,66	50	0,073
20.08.91	9171202	B005	-	29	3,7	-	0,20	2	350	75	-	50	-
20.08.91	9171206	B006	-	29	3,7	-	0,20	2	350	75	-	50	-

Enclosure 3: Calculation of recoveries

The calculation of the recoveries in % is evaluated according to following equations:

$$M_f = \frac{F_p \times M_t \times V_1 \times V_2}{F_s \times T_1}$$

$$F = \frac{M_f \times 100}{M_1}$$

with

F_p = Peak-height of sample (cm)
F_s = Peak-height of standard (cm)
M_t = concentration of standard (µg/ml)
V_1 = volume of solution for GC (ml)
V_2 = total volume (ml)
T_1 = partly volume of V2 (ml)
M_f = found amount (µg)
M_1 = added amount (µg)

Enclosure 3 : Calculation recovery-rate of alpha-endosulfan

Sheet 1 (3)

Date	Material	Lab code	Alpha-endos. added	Peak-No.	Peak-height std. sol.	Peak-height sample	Conc. std. sol.	Volume sol. for GC	Total vol.	Part. vol. of V2	Alpha-endos. found	Recovery
			µg		cm	cm	µg/ml	ml	ml	ml	µg	%
		A	M1	E	Fs	Fp	Mt	V1	V2	T1	Mf	F
06/14/91	soil	001	5	13	5,5	5,2	0,2	5	300	75	3,78	76
07/09/91	soil	002	5	20	11,7	13,0	0,1	25	300	150	5,56	111
08/20/91	soil	003	5	27	10,5	8,5	0,2	5	350	75	3,78	76

enclosure 3 : calculation recovery-rate of beta-endosulfan

Sheet 2 (3)

Date	Material	Lab code	Alpha-endos. added	Peak-No.	Peak-height std. sol.	Peak-height sample	Conc. std. sol.	Volume sol. for GC	Total vol.	Part. vol. of V2	Alpha-endos. found	Recovery
			µg		cm	cm	µg/ml	ml	ml	ml	µg	%
		A	M1	E	Fs	Fp	Mt	V1	V2	T1	Mf	F
06/14/91	soil	001	5	13	4,15	4,2	0,2	5	300	75	4,05	81
07/09/91	soil	002	5	20	8,10	9,5	0,1	25	300	150	5,86	117
08/20/91	soil	003	5	27	8,70	8,0	0,2	5	350	75	4,29	86

Enclosure 3 : Calculation recovery-rate of endosulfan-sulfate

Sheet 3 (3)

Date	Material	Lab code	Alpha-endos. added	Peak-No.	Peak-height std. sol.	Peak-height sample	Conc. std. sol.	Volume sol. for GC	Total vol.	Part. vol. of V2	Alpha-endos. found	Recovery
			µg		cm	cm	µg/ml	ml	ml	ml	µg	%
		A	M1	E	Fs	Fp	Mt	V1	V2	T1	Mf	F
06/14/91	soil	001	5	10	4,4	5,1	0,2	5	300	75	4,63	93
07/09/91	soil	002	5	23	4,9	5,9	0,2	5	300	75	4,82	96
08/20/91	soil	003	5	28	3,7	3,1	0,2	5	350	75	3,91	78