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Corresponds to study #22 in Attachment A of transmittal memo on CBI
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BASF Aktiengesellschaft
Ecology and Environmental Analytics
Laboratory of Ecology
D-67056 Ludwigshafen

Deutscher Akkreditierungs Rat
DAR
DAC-P-0030-97-02

FINAL REPORT

Determination of the Biodegradability of

Perylimid F

in the Manometric Respirometry Test

according to **GLP, EN 45001 and ISO 9002**

Project Number

98/0291/26/1

Completion date: July 1999

Study Director: [redacted]

1 LIST OF CONTENTS

2 QUALITY CRITERIA

This project was performed according to the following quality criteria:

- European Standard EN 45001 "General criteria for the operation of testing laboratories"
- International Standard ISO 9002 "Quality systems - Model for quality assurance in production installation and servicing"
- OECD Principles of Good Laboratory Practice (GLP).

The Laboratory of Ecology is a part of the "Ecology and Environmental Analytics" test facility of BASF AG in Ludwigshafen
- accredited to [redacted] see number [redacted]-0030-97-02 and
- registered for GLP with the name "BASF [redacted]" in Bundesanzeiger (German Federal Gazette) No. 140 page 8633 of 30 July 1996.

Copies of the certificates can be provided by request.

According to the International Standard ISO 31-0 the decimal sign is a comma.

3 SUMMARY

Determination of Biodegradability of Perylimid F in the Manometric Respirometry Test according to Annex of EEC-Directive 92/69; corresponds to OECD Guideline 301 F and ISO Standard 9408.

TEST RESULT

Biodegradation degree (BOD of ThOD) after 28 days : 0-10 %

EVALUATION OF THE TEST RESULT

The test substance is in this test poorly biodegradable
and not readily biodegradable (according to OECD criteria).

Test concentration: 100 mg/l

Inoculum:
Municipal activated sludge from laboratory wastewater treatment plants fed with municipal sewage.
Concentration of dry substance 30 mg/l.

4 GLP STATEMENT OF COMPLIANCE AND SIGNATURES

This study was performed according to the principles of good laboratory practice (GLP) of the OECD, published February 04, 1983 and the German Chemical Law (July 25, 1994). This is confirmed by the signature of the study director. All data and the test substances are stored in the Ecology Department ([redacted]) in Building [redacted] of BASF Aktiengesellschaft in Ludwigshafen/Rhein (Germany).

Date: 13 July 1999 15 July 1999 July 30th 1999

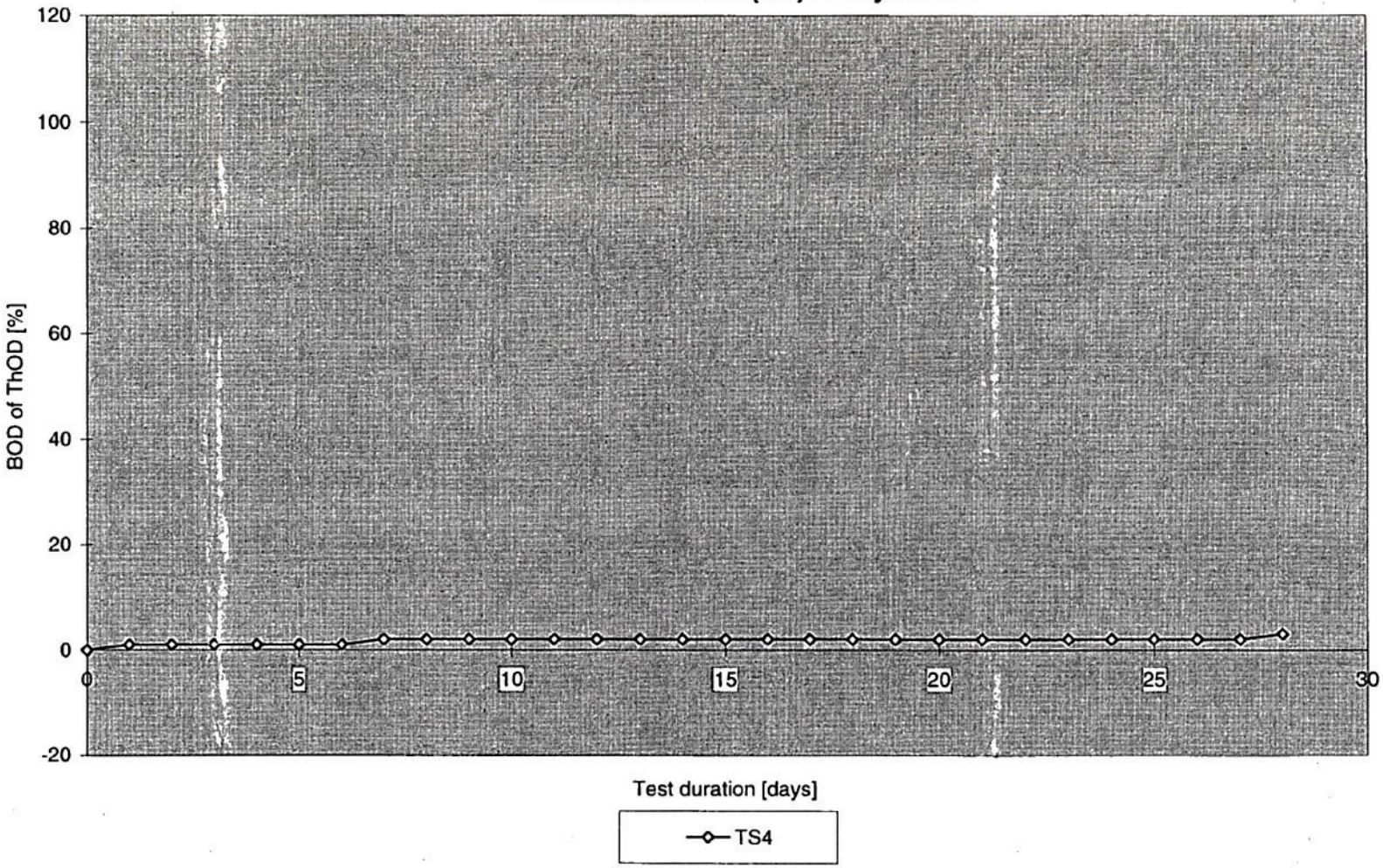
Signature: [redacted]

Name: [redacted]

Function: [redacted]

Manometric Respirometry Test

Test substance(TS): Perylimid F



5 STATEMENT OF THE QUALITY ASSURANCE UNIT

MSS-Number: 99/243
Test substance: Perylimid F
Title: Determination of the Biodegradability in the Manometric Respirometry Test

The Quality Assurance Unit inspected the study, audited the final report and reported findings to the study director and to the management.
Laboratories and short term studies are inspected in regular intervals.

Last procedure audit: 24.06.99

Inspection	Date of inspections (dd.mm.yy)	Reported at
Study plan	26.05.99	
Study audit	24.06.99	
Draft	13.07.99	
Final report	21.07.99	21.07.99

Ludwigshafen, 21.07.99

[redacted]

6 DESCRIPTION OF THE TEST METHOD

Test principle of the Manometric Respirometry Test

The Manometric Respirometry Test is a static method for testing the ultimate aerobic biodegradability of a test substance in water. Mixtures of the test substance, a defined inorganic medium and an inoculum, which is not pre-adapted (e.g. activated sludge or effluent of a municipal or laboratory waste water treatment plant), are incubated and aerated in a respirometer (Sapromat) at room temperature up to 28 days. The oxygen used for the biodegradation of the test substance (biochemical oxygen demand, BOD) is continuously produced and measured by the test apparatus.

In addition to the test substance the following investigations are required: blank values (BC), biodegradation of a reference substance (RS), inhibition of the inoculum (IH) and abiotic elimination (PC).

Evaluation of Test Results

The biochemical oxygen demand gives unequivocal evidence for biodegradation. The measured BOD is compared with the calculated theoretical oxygen demand (ThOD) or the measured chemical oxygen demand (COD) of the test substance and indicated as biodegradation degree in per cent. Since the microorganisms oxidize only part of the test substance and incorporate the rest as biomass, substances with a degree of >60 % biodegradation are considered to be sufficiently biodegradable. The relation BOD to ThOD or COD is classified as follows:

>60 % in 10 days	"readily biodegradable (OECD criteria)"
>60 %	"biodegradable"
20-60%	"in this test moderately or partly biodegradable"
<20%	"in this test poorly biodegradable"

One of the OECD criteria for ready biodegradable substances is the threshold value of 60% biodegradation within a period of 10 days during a total test period of 28 days. If a test substance is biodegradable in this test, it can be assumed that it will be biodegraded in the environment, especially in wastewater treatment plants and surface waters. If insufficient biodegradation was measured, the test substance may nevertheless be inherently biodegradable. This has to be shown by further investigations.

7 TEST METHODS

Annex to EEC- Directive 92/69/EEC from 31 July 1992
Manometric Respirometry (Method C.4-D)
Official Journal of the European Communities L383 A, 29 December 1992

Organisation for Economic Cooperation and Development - OECD
Guidelines for Testing of Chemicals
Ready Biodegradability - Manometric Respirometry Test 301 F; Paris 1993.

International Standard ISO 9408:1991 .
Water Quality - Evaluation in an aqueous medium of the "ultimate" aerobic biodegradability of organic compounds - Method by determining the oxygen demand in a closed respirometer.

8 ORDER INFORMATION

Sponsor: [redacted]
Department code:
Telephone: 99333
Date of order: 30 Apr 1998

9 SPECIFICATION OF THE TEST SUBSTANCE BY THE SPONSOR

Name of test substance	:	Perylimid F
Chemical name	:	Perylene-3,4,9,10-Tetracarboxydiimide
Batch number	:	Partie 18
Date of production	:	2.Quartal 96
Product number	:	073209
CAS number	:	81-33-4
Molecular formular	:	C24 H10 N2 O4
Molecular weight [g/mol]	:	390.36
Aggregate state	:	solid
Density [kg/l]	:	0,3 (20°C)
Water-solubility	:	< 500 mg/l [1]
Colour	:	violet/red
Purity of the test substance [%]:		98,9% [3]
Impurities	:	no data
Homogeneity	:	yes
Instability against	:	Heat: No Light: No Oxygene: No
		Water: No Acid: No Alcali: No
Limited storage	:	No
Special storage conditions	:	ca. 4°C: No under N2: No
		<= -15°C: No Light exclusion: No
Further remarks	:	none

Origin of data of purity and homogeneity:

[1]) Observation of the Laboratory of Ecology
[2]) Report of the Analytical Laboratory No
[3]) Report of the sponsor No: analysis from 06 June 1996

10 PRELIMINARY INVESTIGATIONS

Summary parameters	TOC	DOC	C*	H*	O*	N*	ThOC*	ThOD+
[mg/g]	---	---	724	---	---	---	724	1865

(+) calculated by: X summary formula at 98,9 % purity
( ) calculated by: X Analysis of the elements No. 98L00694

Inhibition of bacteria Activated Sludge Respiration Inhibition Test project no.: 98/0291/08/1	EC20 : --- mg/l	X no inhibition up to 1000 mg/l O not ready yet

11 TEST INFORMATION

Experimental phase :	27 May 1999 – 24 June 1999
Begin of the study :	25 May 1999

Test duration (days)	: 28
Duration of adaptation phase (days)	: ---
Duration of degradation phase (days)	: ---
Degradation of the test substance at the end of the ten-day window (% BOD/ThOD)	: ---
Degradation degree of the test substance after 28 days (% BOD/ThOD)	: 0-10
Degradation degree of the test substance at the end of the test (% BOD/ThOD)	: 0-10
Degree of degradation of the test substance in the abiotic control at the end of the test (% BOD/ThOD)	: 0-10

Reference substance	: aniline
Degradation degree of the reference substance after 14 days (% BOD/ThOD)	: 70-80
Degradation degree in the inhibition control after 14 days (% BOD/ThOD)	: 40-50

12 VALIDITY CRITERIA

Deviation of the degradation degree of the test substance in the plateau phase <20%	: yes
Degradation degree of the reference substance >60% after 14 days	: yes
Degradation degree in the inhibition control >25 % after 14 days	: yes
Oxygen demand in the blank control < 60 mg/l at the end of the test	: yes
pH values in the test assays ranged from pH 6 to 8,5 at the end of the test	: yes
The test is valid	**: yes**

Proof of ready biodegradability according to OECD criteria

Degradation degree of the test substance >60% within 28 days	: no
10-day window met	: no
No pre-adapted inoculum used	: yes
Test substance is readily biodegradable according to OECD criteria	**: no**

13 ACCOUNT OF THE TEST RESULTS

Manometric Respirometry Test

Sapromat No.: 8 **Test Details**

Test substance(TS): Perylimid F **Reference substance (RS): Aniline**

Direct addition:

Stock solution:

Mass	:	2500 mg
Volume	:	1000 ml
ThOC	:	774 mg/g
DOC	:	749 mg/g

ThOC : 724 mg/g*
ThOD : 1865 mg/g (summary formula)

ThOD : 2393 mg/g
ThOD of IH : 2127 mg/g

*) by analysis of the elements

Assay	Inorgan. medium	Inoculum	Aniline	Aniline	TS	TS	TS	TS	$HgCl_2$	pH	pH
	ml	ml	ml/A	mg/l	ml/A	mg/l	mg/A	µl/A	µl	start	end
BC 1	240	10	-	-	-	-	-	-	-	7,1->7,3	7,5
BC 2	240	10	-	-	-	-	-	-	-	7,1->7,4	7,5
RS	230	10	10	100	-	-	-	-	-	7,2	9,2
IH	230	10	10	100	-	101,6	25,4	-	-	7,1->7,4	8,2
PC	250	-	-	-	-	100,0	25,0	-	250	7,2	7,5
TS 1	240	10	-	-	-	98,8	24,7	-	-	7,2	7,5
TS 2	240	10	-	-	-	101,6	25,4	-	-	7,2	7,5
TS 3	240	10	-	-	-	100,4	25,1	-	-	7,1->7,3	7,5
TS 4	240	10	-	-	-	100,8	25,2	-	-	7,1->7,3	7,5
TS 5	240	10	-	-	-	101,2	25,3	-	-	7,1->7,4	7,5
TS 6	240	10	-	-	-	102,0	25,5	-	-	7,1->7,3	7,5
TS 7	240	10	-	-	-	98,4	24,6	-	-	7,2	7,4

Remarks: BC= Blank control IH= Inhibition control PC= Abiotic elimination

Inoculum: Municipal activated sludge from laboratory wastewater treatment plants fed with municipal sewage. Concentration of dry substance 30 mg/l.

Manometric Respirometry Test
BOD values [mg/l] per day (accumulated)

Test substance: Perylimid F
Start of the test: 27.5.99
Project technician: [redacted]

Days	BC1 1	BC2 2	RS 3	IH 4	PC 5	TS1 6	TS2 7	TS3 8	TS4 9	TS5 10	TS6 11	TS7 12
0	0	0	0	0	0	0	0	0	0	0	0	0
1	1	0	0	0	0	0	0	0	2	1	2	0
2	1	9	0	0	0	0	1	1	3	2	4	0
3	3	9	1	2	0	0	3	2	5	3	6	2
4	4	9	50	136	0	3	4	3	6	5	8	4
5	5	14	141	140	0	3	5	3	7	5	10	4
6	6	16	148	146	0	3	5	5	8	7	12	6
7	6	16	155	150	0	3	5	5	9	7	14	8
8	7	16	166	154	0	6	7	7	10	8	14	8
9	7	16	173	158	0	6	8	7	10	9	16	8
10	8	16	177	166	0	6	9	7	11	10	18	10
11	9	21	179	172	0	6	9	7	12	10	20	10
12	9	27	181	176	0	6	9	9	12	11	20	12
13	10	43	183	182	0	6	9	9	13	11	22	12
14	10	43	183	184	0	6	9	9	13	12	24	12
15	10	43	184	188	0	6	11	9	14	13	25	14
16	11	43	184	190	0	6	12	9	14	13	26	14
17	11	43	186	192	0	6	12	9	15	13	27	14
18	11	43	186	194	0	6	12	11	15	14	28	14
19	11	43	186	194	0	6	12	11	15	14	30	16
20	12	47	186	196	10	9	12	11	16	14	31	16
21	12	48	187	198	10	9	12	11	16	15	33	18
22	13	48	187	198	10	9	12	13	17	16	35	18
23	13	48	187	200	10	9	12	13	17	16	35	18
24	13	48	187	202	10	9	12	13	17	16	35	18
25	13	48	188	202	10	9	12	13	17	16	37	18
26	13	48	190	202	10	9	14	13	17	16	38	18
27	14	57	191	204	10	9	15	13	18	17	40	20
28	14	59	192	204	10	9	15	13	19	17	41	20

Remark: For unknown reasons an extraordinary high BOD was measured in the blank assay BC2.
Therefore this assay is regarded as invalid.

Validated : [redacted]

13 July 1999
Date:

Manometric Respirometry Test
Calculated specific BOD values [mg/g]

Test substance: Perylimid F

Date	Days	BC 1	RS	IH	PC	TS1	TS2	TS3	TS4	TS5	TS6	TS7
27.5.99	0	0	0	0	0	0	0	0	0	0	0	0
28.5.99	1	1	-10	-5	0	-10	-10	-10	10	0	10	-10
29.5.99	2	1	-10	-5	0	-10	0	0	20	10	29	-10
30.5.99	3	3	-20	-5	0	-30	0	-10	20	0	29	-10
31.5.99	4	4	460	655	0	-10	0	-10	20	10	39	0
1.6.99	5	5	1360	670	0	-20	0	-20	20	0	49	-10
2.6.99	6	6	1420	694	0	-30	-10	-10	20	10	59	0
3.6.99	7	6	1490	714	0	-30	-10	-10	30	10	78	20
4.6.99	8	7	1590	729	0	-10	0	0	30	10	69	10
5.6.99	9	7	1660	749	0	-10	10	0	30	20	88	10
6.6.99	10	8	1690	784	0	-20	10	-10	30	20	98	20
7.6.99	11	9	1700	809	0	-30	0	-20	30	10	108	10
8.6.99	12	9	1720	828	0	-30	0	0	30	20	108	30
9.6.99	13	10	1730	853	0	-40	-10	-10	30	10	118	20
10.6.99	14	10	1730	863	0	-40	-10	-10	30	20	137	20
11.6.99	15	10	1740	883	0	-40	10	-10	40	30	147	41
12.6.99	16	11	1730	888	0	-51	10	-20	30	20	147	30
13.6.99	17	11	1750	898	0	-51	10	-20	40	20	157	30
14.6.99	18	11	1750	908	0	-51	10	0	40	30	167	30
15.6.99	19	11	1750	908	0	-51	10	0	40	30	186	51
16.6.99	20	12	1740	913	100	-30	0	-10	40	20	186	41
17.6.99	21	12	1750	923	100	-30	0	-10	40	30	206	61
18.6.99	22	13	1740	918	100	-40	-10	0	40	30	216	51
19.6.99	23	13	1740	928	100	-40	-10	0	40	30	216	51
20.6.99	24	13	1740	938	100	-40	-10	0	40	30	216	51
21.6.99	25	13	1750	938	100	-40	-10	0	40	30	235	51
22.6.99	26	13	1770	938	100	-40	10	0	40	30	245	51
23.6.99	27	14	1770	942	100	-51	10	-10	40	30	255	61
24.6.99	28	14	1780	942	100	-51	10	-10	50	30	265	61

Remark: For unknown reasons an extraordinary high BOD was measured in the blank assay BC2.
Therefore this assay is regarded as invalid.

mv = mean value

Manometric Respirometry Test

Biodegradation in relation to the ThOD [%]

Test substance: Perylimid F

Date	Days	RS	IH	PC	TS1	TS2	TS3	TS4	TS5	TS6	TS7
27.5.99	0	0	0	0	0	0	0	0	0	0	0
28.5.99	1	0	0	0	-1	-1	-1	1	0	1	-1
29.5.99	2	0	0	0	-1	0	0	1	1	2	-1
30.5.99	3	-1	0	0	-2	0	-1	1	0	2	-1
31.5.99	4	19	31	0	-1	0	-1	1	1	2	0
1.6.99	5	57	32	0	-1	0	-1	1	0	3	-1
2.6.99	6	59	33	0	-2	-1	-1	1	1	3	0
3.6.99	7	62	34	0	-2	-1	-1	2	1	4	1
4.6.99	8	66	34	0	-1	0	0	2	1	4	1
5.6.99	9	69	35	0	-1	1	0	2	1	5	1
6.6.99	10	71	37	0	-1	1	-1	2	1	5	1
7.6.99	11	71	38	0	-2	0	-1	2	1	6	1
8.6.99	12	72	39	0	-2	0	0	2	1	6	2
9.6.99	13	72	40	0	-2	-1	-1	2	1	6	1
10.6.99	14	72	41	0	-2	-1	-1	2	1	7	1
11.6.99	15	73	42	0	-2	1	-1	2	2	8	2
12.6.99	16	72	42	0	-3	1	-1	2	1	8	2
13.6.99	17	73	42	0	-3	1	-1	2	1	8	2
14.6.99	18	73	43	0	-3	1	0	2	2	9	2
15.6.99	19	73	43	0	-3	1	0	2	2	10	3
16.6.99	20	73	43	5	-2	0	-1	2	1	10	2
17.6.99	21	73	43	5	-2	0	-1	2	2	11	3
18.6.99	22	73	43	5	-2	-1	0	2	2	12	3
19.6.99	23	73	44	5	-2	-1	0	2	2	12	3
20.6.99	24	73	44	5	-2	-1	0	2	2	12	3
21.6.99	25	73	44	5	-2	-1	0	2	2	13	3
22.6.99	26	74	44	5	-2	1	0	2	2	13	3
23.6.99	27	74	44	5	-3	1	-1	2	2	14	3
24.6.99	28	74	44	5	-3	1	-1	3	2	14	3

Manometric Respirometry Test
Test substance(TS): Perylimid F
Reference substance: Aniline