PROGRESS REPORT
ORGANIC CHEMICALS DIVISION
TECHNOLOGY DEPARTMENT

CONFIDENTIAL

REPORTED BY	FOR	DETAILS	REPORT NO.	DATE
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LOCATION	JOB NOS
St. Louis - South Second Street	2-21-760.01-02031, 03790, 13480, 14490, 04050, 85010, 85050

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PROJECT TITLE

ENVIRONMENTAL ANALYTICAL PROGRAM - BIODEGRADATION STUDIES

PROJECT OBJECTIVE

Development and application of test procedures to measure the susceptibility of Organic Division products to primary biodegradation via the biological action of bacteria.

SUMMARY

Semi-continuous activated sludge (SCAS) testing on Aroclor 5432, Aroclor 5460, Santicizer 160, tolyl xylyl sulfone and trichlorodiphenyl ether are still in progress. Problems encountered with the quantitative recovery of Aroclor 5432 from mixed liquor together with the effects of contaminants have delayed the start of sampling on the Aroclor 5432 and Aroclor 5460 units. Work is under way to resolve these difficulties. On the basis of the data obtained thus far on Santicizer 160, tolyl xylyl sulfone, and trichlorodiphenyl ether, Santicizer 160 would be classified a Partially Degradable I, while both trichlorodiphenyl ether and tolyl xylyl sulfone would fall in the Refractory category. Limited testing will continue to determine if the disappearance rates will change significantly with time.

Testing of Santicizer 711, Santicizer 160, di(2-ethylhexyl) phthalate (DOP), Santicizer 140, and Santicizer 141 has been started using the standard screening protocol outlined last month. The screening data obtained on S-711 and S-160 will allow a comparison to the more extensive data previously obtained on these materials and will be used to experimentally establish the validity of the proposed protocols.

SCAS data obtained at a 0.1 CFH aeration rate on o-nitrochlorobenzene (ONCB), p-nitrochlorobenzene (PNCB), p-chloroaniline, and nitrated orthene showed some inconsistencies. It appears that insufficient agitation is a likely factor. An intermediate aeration rate, 0.25 CFH, will probably be the best compromise toward minimizing volatility losses, but maintaining adequate agitation.

River die-away tests on ONCB, PNCB, p-chloroaniline, and nitrated orthene are now in their third week. Interpretation of the results to date awaits completion of the tests.

FUTURE PLANS

Work Scheduled for Completion by 12/1/71

1. SCAS analytical method development for: Plasticizers - Di(2-ethylhexyl) phthalate (DOP); mixed cresyl phenyl phosphate (S-140); Diphenyl 2-ethylhexyl phosphate (S-141). Specialty Products - MCS 1109; MCS 1172.

2. Measurement of SCAS disappearance rates for: Specialty Products - Aroclor 5432, Aroclor 5460, S-160, S-711.

3. River die-away (RDA) testing of o-nitrochlorobenzene (ONCB), p-nitrochlorobenzene (PNCB), nitrated orthene, and p-chloroaniline.

V. W. Saeger

E. S. Tucker

db

DETAILS

Details and special study report references are given in the following section which is arranged according to the product group requesting the work.

I. General Chemicals

A. Product Tested: p-Chloroaniline (p-CA); p-Nitrochlorobenzene (PNCB); o-Nitrochlorobenzene (ONCB), and 3,4-Dichloronitrobenzene (nitrated orthene).

B. Discussion of Results

Because of the significant volatility losses for certain test compound, the effects of a five-fold reduction in aeration rate (0.5 to 0.1 CFH) were investigated. In terms of volatility losses, this decreased aeration rate as expected minimizes them. This is shown in Table I where volatility losses for ONCB and PNCB are shown for the 0.1 CFH aeration rate. The effect of the reduced aeration rate on the SCAS disappearance data is somewhat less certain. In Table II, the disappearance rate data is summarized. If a reduction in volatility loss were the only operative factor, one would expect a decrease in the disappearance rate. In the case of nitrated orthene, PNCB and ONCB, an increase in the disappearance rate was observed while for p-chloroaniline the rate decreased. The wide scatter in the p-CA data seemed to indicate that reduced agitation in the system might be significant. For this reason, an auxiliary stirrer was added to the unit. This resulted in a return to the previous level of disappearance rate. Further effort will be necessary to determine the exact effect of the reduced aeration rate. It would appear that a 0.25 CFH aeration rate is the best compromise toward minimizing volatility losses, but maintaining adequate agitation.

River die-away tests on ONCB, PNCB, p-CA and nitrated orthene are now in their third week. These are being carried out at the one ppm level using Mississippi River water. No conclusions can be made at this point. Samples are being analyzed on a weekly basis.

II. Plasticizers

A. Product Tested: Santicizer 160.

B. Discussion of Results

Disappearance rate data were obtained for S-160 over a 3-week sampling period at a feed rate of 10 mg/24-hour cycle. The mean disappearance rate and 95% confidence limits obtained were 65.8 ±17.3. These data would place S-160 in the Partially Degradable I category. Volatility losses appear to be insignificant for S-160.

Feeding of SCAS units with S-160, S-711, S-140, S-141, and DOP has been started using the standard screening protocol outlined last month. The screening data on S-160 and S-711 will permit a comparison to the more extensive data previously obtained and thus serve as a check on the validity of the screening protocol.

III. Specialty Products

A. Product Tested: Aroclor 5432, Aroclor 5460, trichlorodiphenyl ether, and tolyl xylyl sulfone.

B. Discussion of Results

1. Polychlorinated Terphenyls: Aroclor 5460 and Aroclor 5432

Because of apparent low recovery of Aroclor 5432 using benzene as the extracting solvent, recovery experiments with alternative solvents were carried out. In Table I, the solvents used together with some physical properties and the Aroclor 5432 and 5460 recovery data are given. The chloroform appears to give the highest recovery of Aroclor 5432. Excellent recoveries were obtained for Aroclor 5460 with all the solvents checked.

Additional problems encountered involved interferences from contaminants in the solvents and extraneous components from the mixed liquor. An alumina column treatment of the samples removes the interferences and will probably be needed for all the SCAS samples. Additional work is now in progress to refine the chloroform recovery data.

2. Trichlorodiphenyl Ether and Tolyl Xylyl Sulfone

Disappearance rate data was obtained over a one-month sampling period at a feed rate of 5 mg/48-hour cycle for trichlorodiphenyl ether and tolyl xylyl sulfone. The mean disappearance rates and 95% confidence limits obtained were 10.7 $\pm$11.6 for trichlorodiphenyl ether and 11.8 $\pm$7.9 for tolyl xylyl sulfone. On the basis of these data, both materials would be classified as Refractory.

A limited amount of additional testing will be carried out to determine if any significant change occurs as a function of time.

New Technology

In order to have comparative data on materials of known environmental behavior, SCAS testing on DDT and a chlorinated naphthalene are planned. Development of the analytical methods are now in progress.

TABLE I

SCAS VOLATILITY LOSSES
AT 0.1 CHF AERATION RATE

	Test Compound ONCB	PNCB
Mg in SCAS unit at 0 Time	10.76	10.07
Mg in SCAS unit at 21 Hrs.	5.38	-
Overall Disappearance Rate, %	50.0	-
Disappearance Rate due to Volatility	1.4	1.9

TABLE II

EFFECT OF AERATION RATE
ON SCAS DATA

Sampling Period (1971)	Aeration Rate	Type of Agitation	Disappearance Rate, % ONCB	PNCB	p-CA	Nitrated Orthene
5/17-6/21	0.5	Magnetic Stirrer	40.3 ±8.6	30.7 ±9.4	99.9±0	32.6±4.7
9/20-10/5	0.1	Magnetic Stirrer	47.5 ±8.1	65.7 ±14.3	62.0 ±27.1	63.9±5.4
10/11-10/15	0.1	Magnetic Stirrer & Blade Stirrer	-	-	97.6	-

TABLE III

AROCLOR 5432 AND 5460 RECOVERY
DATA

Solvent	Boiling Point, °C	Density, g/ml	H_2O Solubility %	% Recovery @ 0.5 PPM A-5432	A-5460
$CHCl_3$	61	1.49	0.82	64,83	96
CH_2Cl_2	40	1.34	2.0	59,78	95
Benzene	80	0.88	0.07	53,77	94
Hexane	67	0.66	i	42	-
Toluene	110	0.87	i	36	-
Ethyl Ether	35	0.71	7.5	27	-

Table V

(Revision VI) 12/3/71

Schedule for Biodegradation Program

Time Year	Method Development	Mean Weeks	Biodegradation Today	Mean Weeks
Dec, 1971 - Jan, 1972	Nippon 295, Nippon 296, Kureha Oil, MCS 1172	1-2	Aroclor 5432	1
	MCS 1207, MCS 1172		5460	1
	MCS 1168		S-160	1
			S-711	1
			DOP	1-2
			S-140	1-2
	Tetrathal	1	S-141	1-2
	S-79 TM	1	Nippon 295	1
	DUP	1	✓ Nippon 296	1
			✓ Kureha Oil	1
			MCS 1172	1
			✓ MCS 1207	1
			✓ MCS 1175	1
			✓ MCS 1168A	1
			Tetrathal	1-2
Feb Blanks	DOA	1-2	3-4, Dichlorodiphenyl methane	1-2
	MCS 1209		S-79 TM	1-2
	MCS 1210		DUP	1-2
	S-97 A	1-2		
	Polyethylene Glycol	1		
	UCON 50 HB-170			

50/50 Dec. Completion

Others

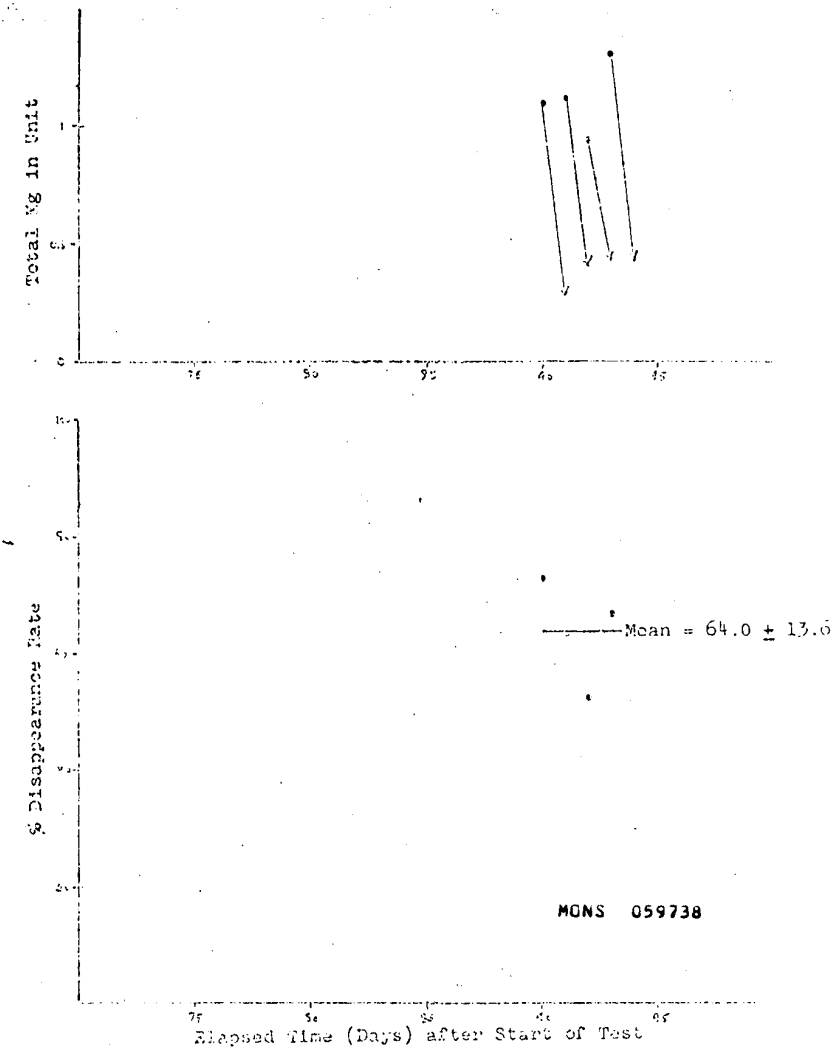
FID/GC [illegible] of sludge [illegible] for linear [illegible] esters [illegible]

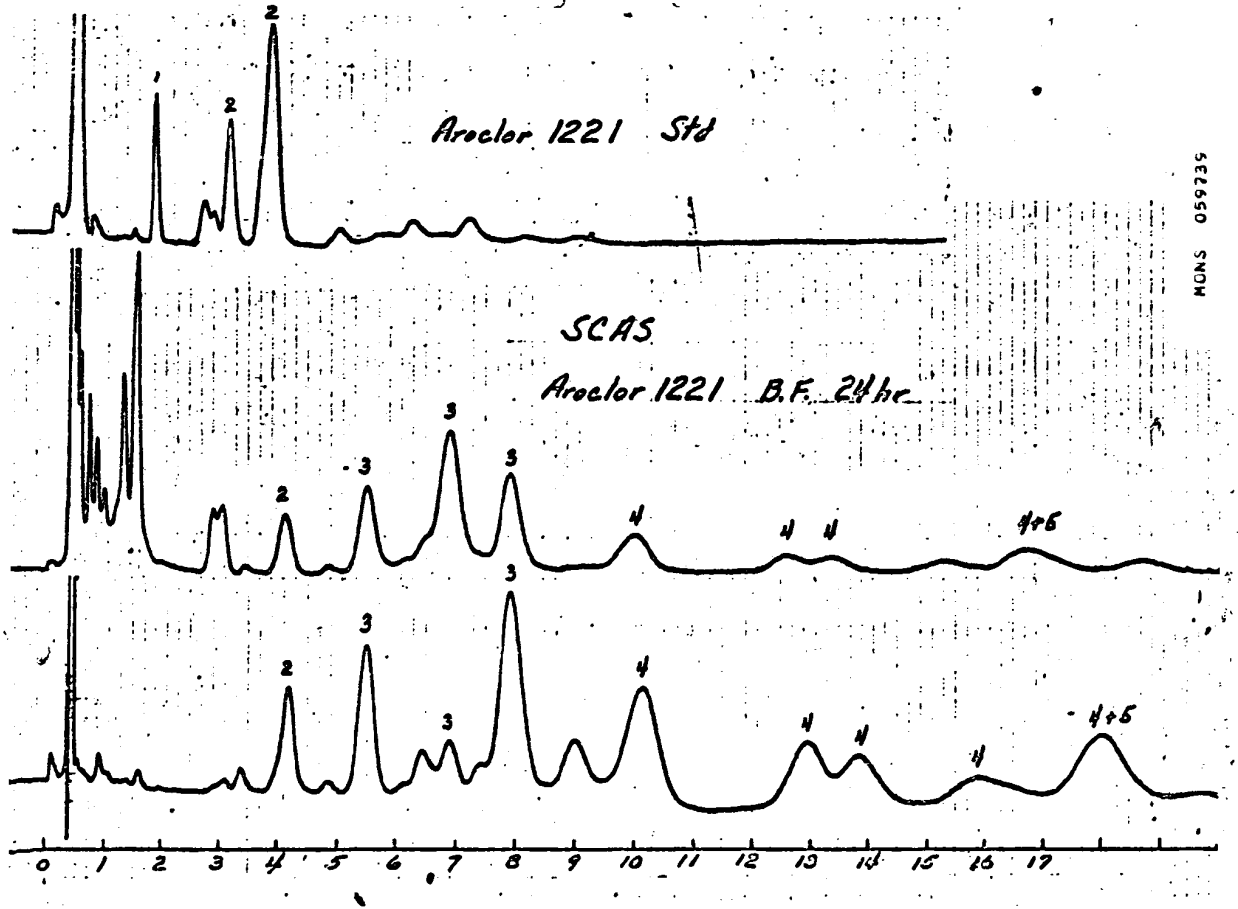
MONS 059737

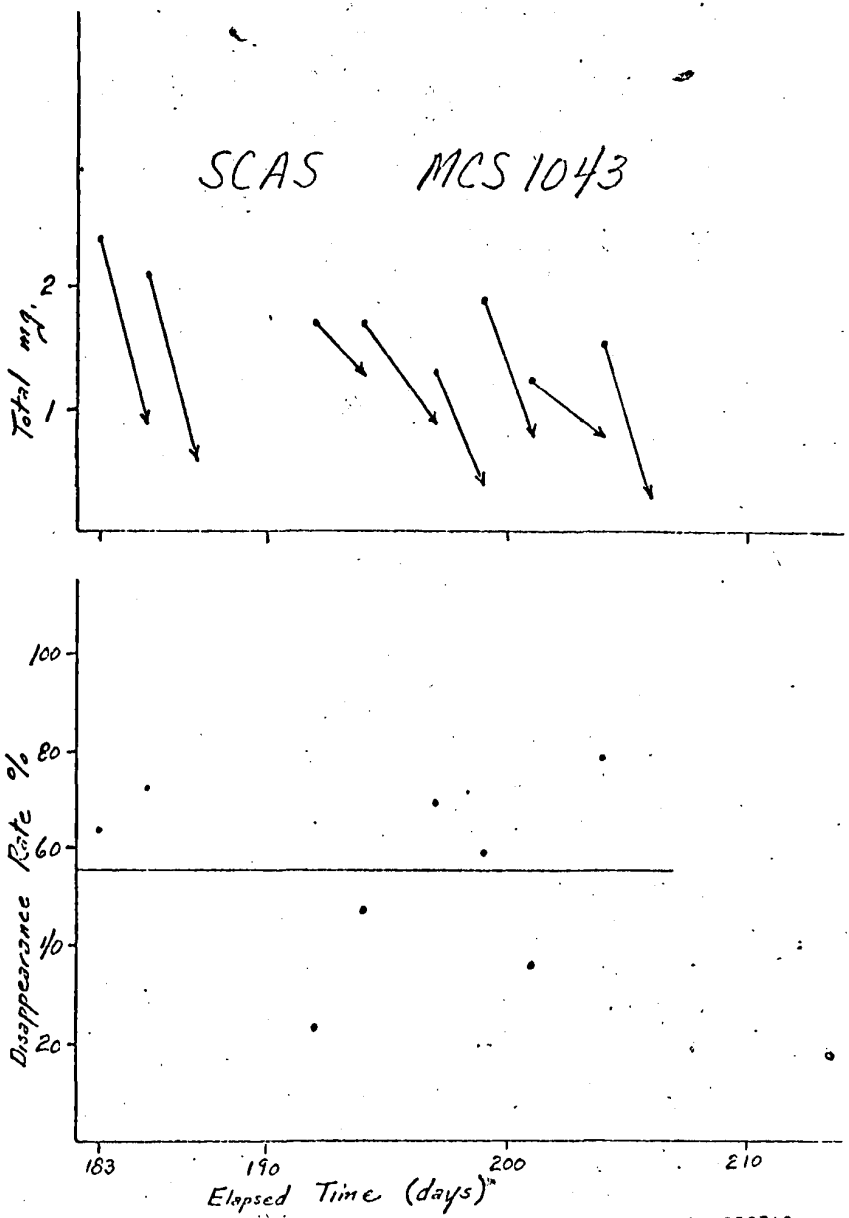
FIGURE 6

SCAS BIODEGRADATION DATA

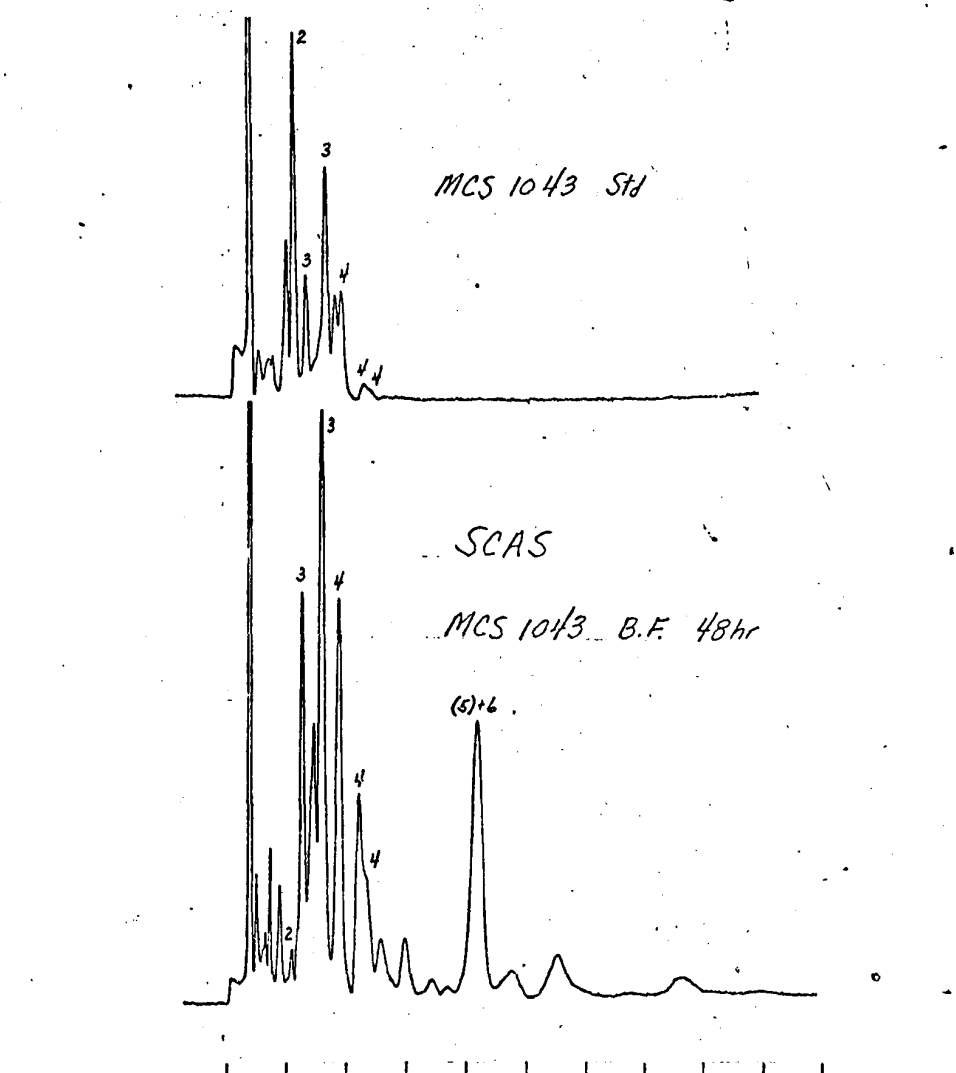
AROCLOR 1221




Aroclor 1221 Std
SCAS
Aroclor 1221 B.F. 24 hr
MONS 059739
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17


SCAS MCS 1043
Total mg.
2
1
100
80
60
40
20
Disappearance Rate %
183
190
200
210
Elapsed Time (days)

MONS 059740

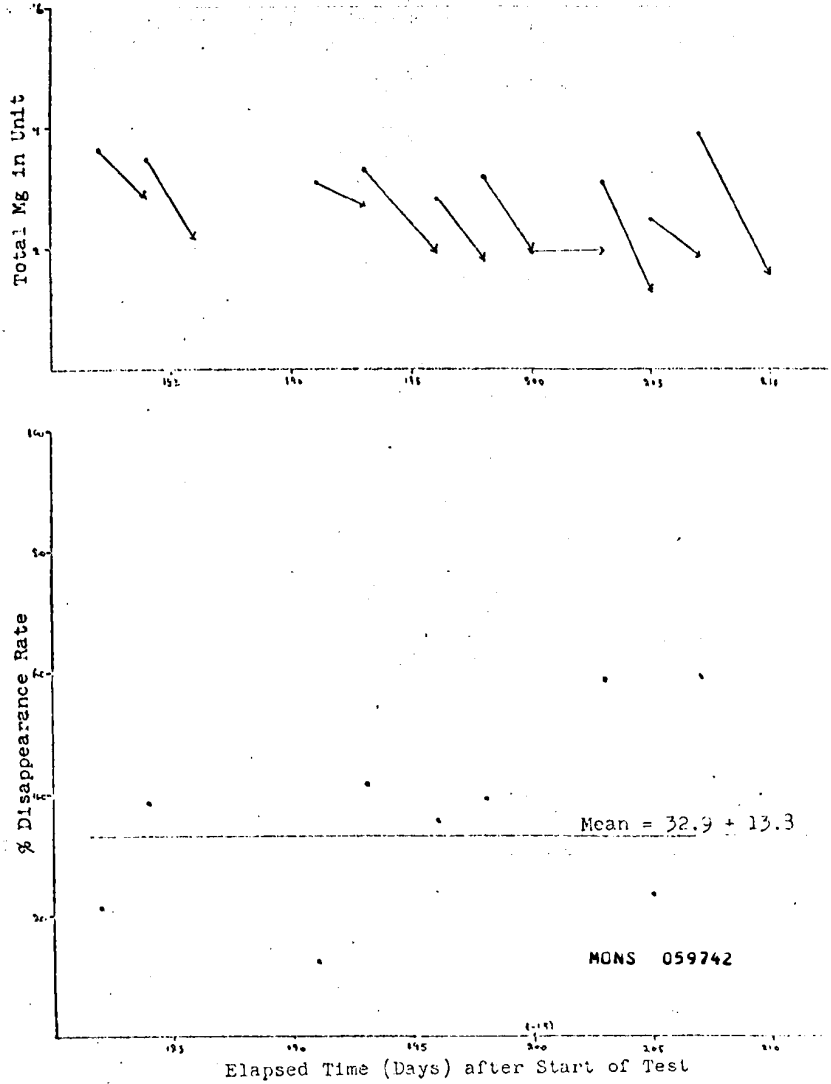

2
3
3
4
4
4
MCS 1043 Std
3
3
4
4
(5)+6
2
SCAS
MCS 1043 B.F. 48hr
0
1
2
3
4
5
6
7
8
9
10
minutes

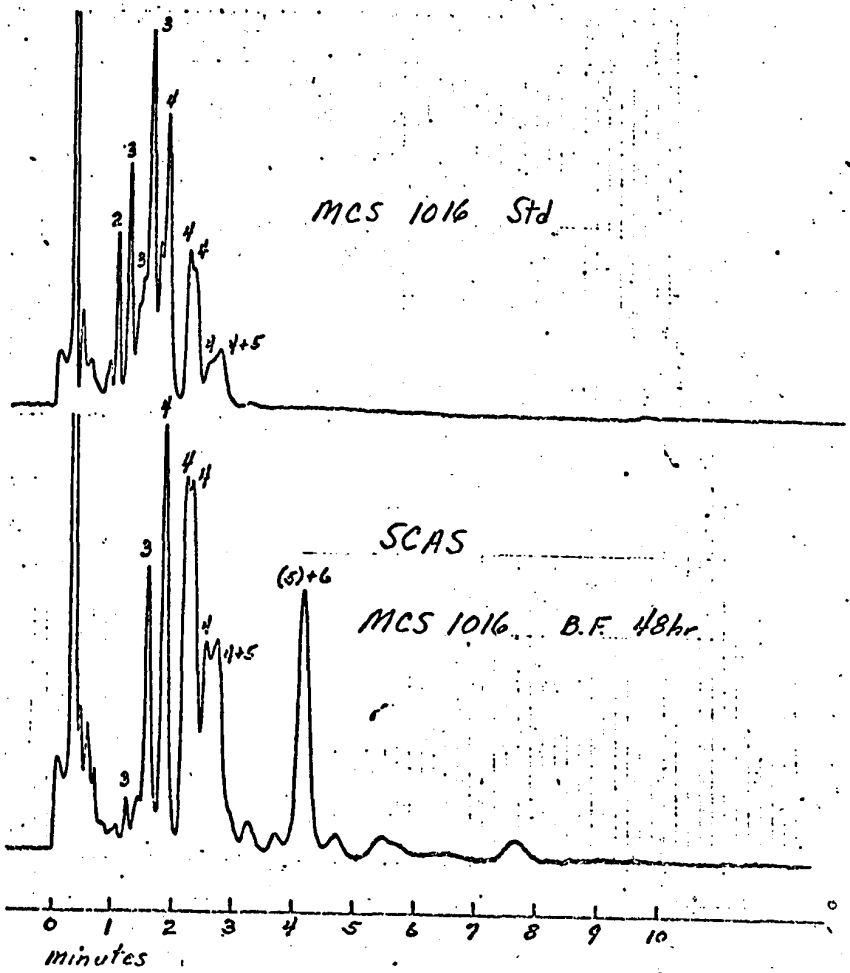
MONS 059741

FIGURE 3-2

SCAS BIODEGRADATION DATA

MCS 1016



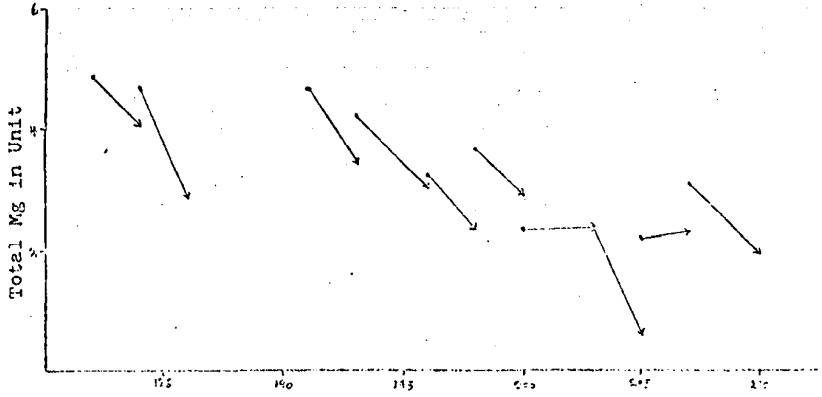

MCS 1016 Std
SCAS
MCS 1016 B.F. 48hr
(5)+6
4+5
0 1 2 3 4 5 6 7 8 9 10
minutes

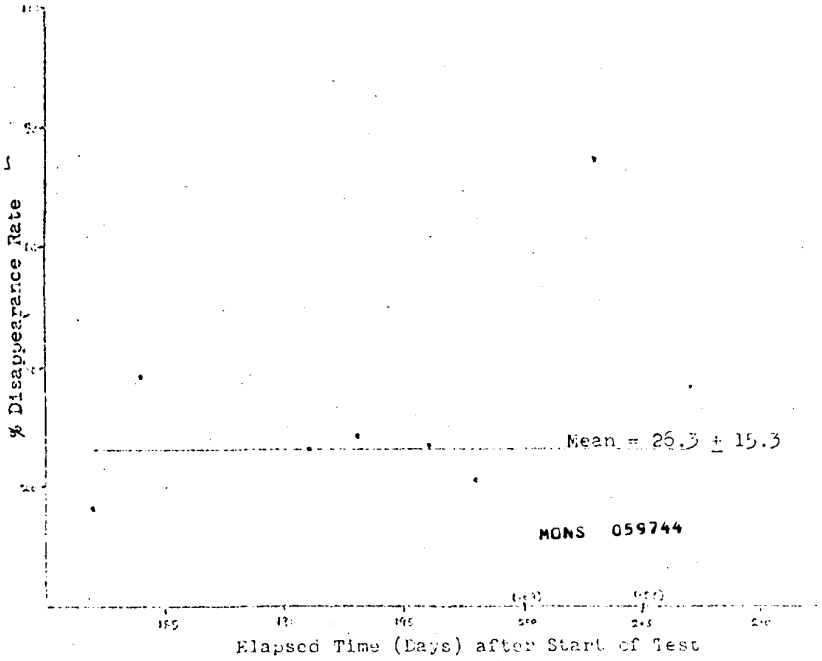
MONS 059743

FIGURE 2-2

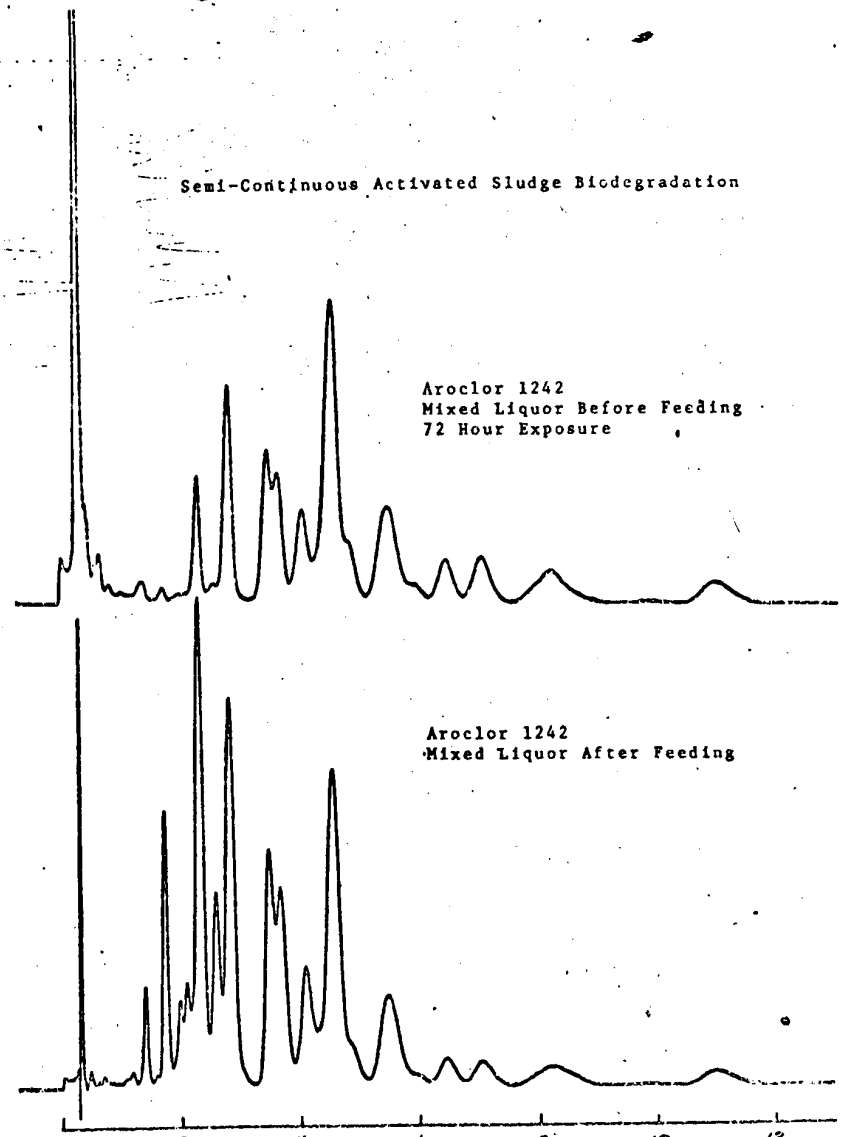
SCAS BIODEGRADATION DATA

AROCLOR 1242





Semi-Continuous Activated Sludge Biodegradation

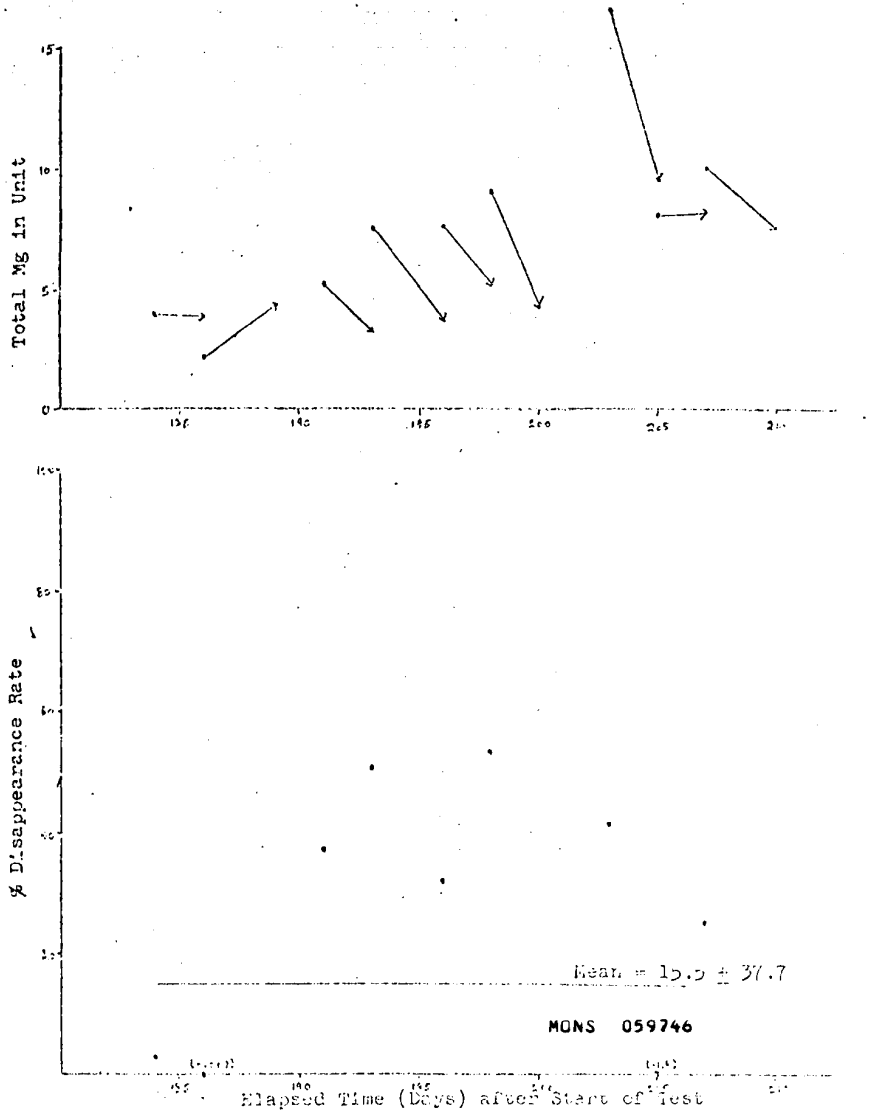


MONS 059745

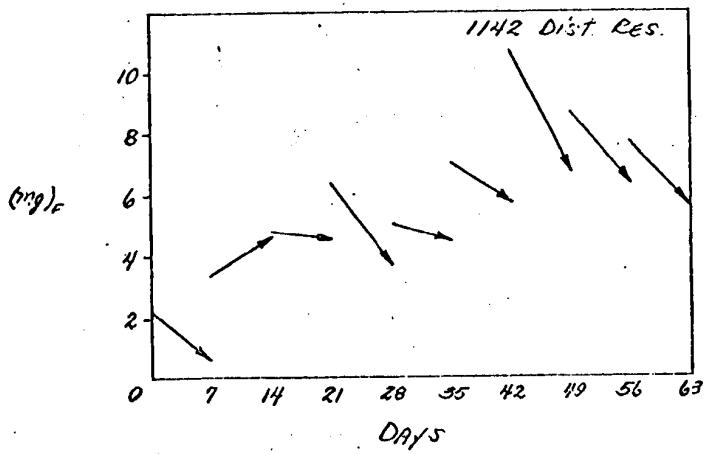
FIGURE 1-2

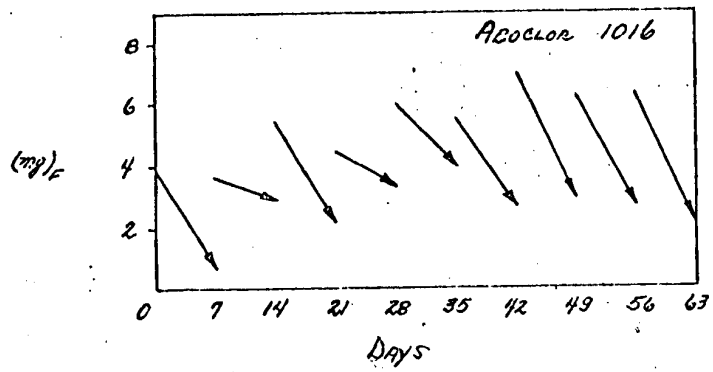
SCAS BIODEGRADATION DATA

AROCLOR 1254

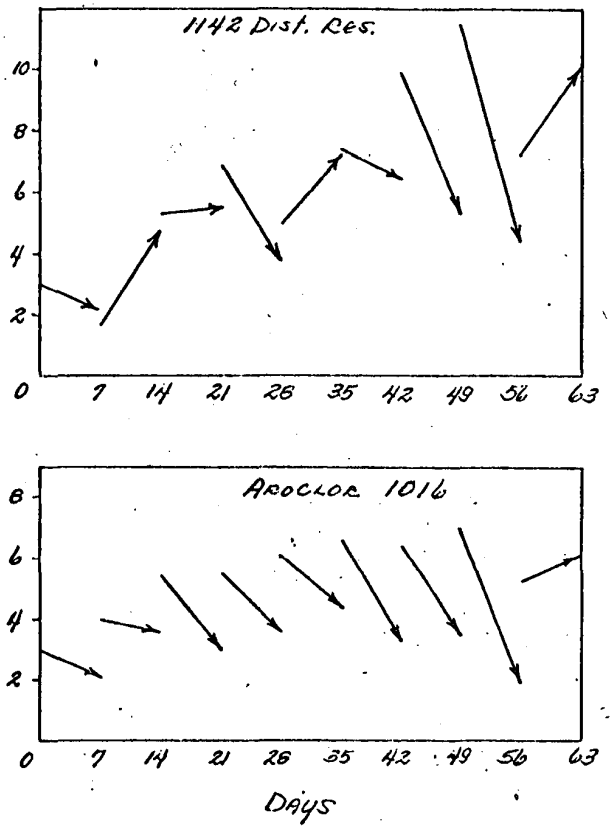


UV Data


1142 Dist. Res.
(mg)F
10
8
6
4
2
0
7
14
21
28
35
42
49
56
63
DAYS

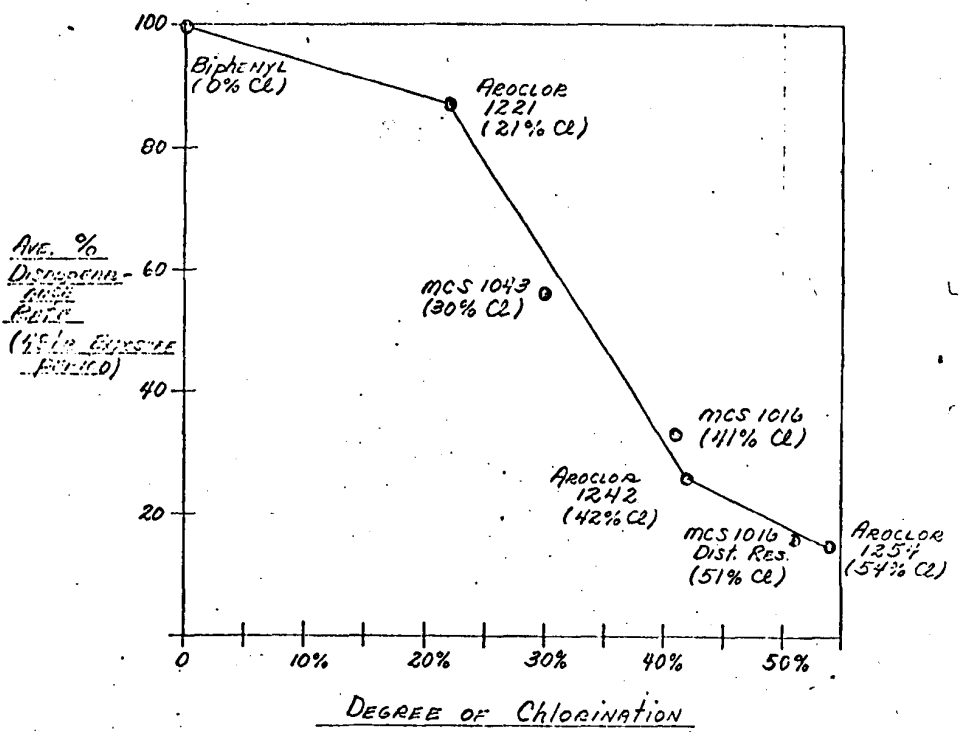

Aroclor 1016
(mg)F
8
6
4
2
0
7
14
21
28
35
42
49
56
63
Days

EC/GC DATA



(mV)$_F$

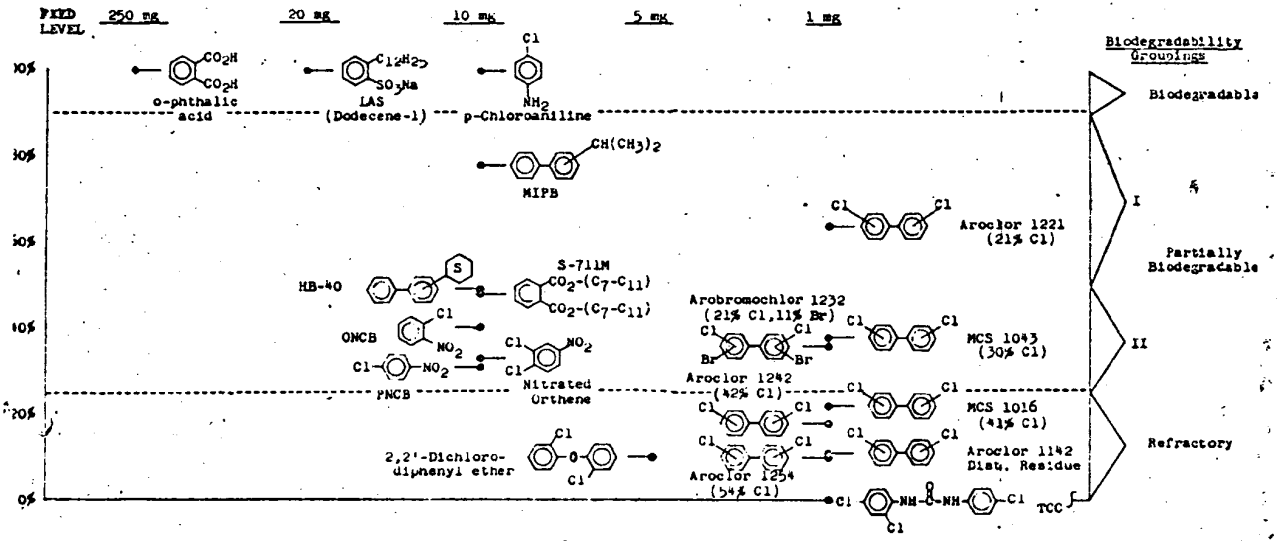
Semi-Continous Activated Sludge (SCAS)
Degradation of Polychlorinated Biphenyls



MONS 059749

FIGURE I

BIODEGRADABILITY OF ORGANIC DIVISION PRODUCTS



MONS 059750