MONSANTO COMPANY

№ 2431001

SUBJECT Chlorodibenzofurans in PCBs

JOB NO	PREPARED BY (SIGNATURE)	DATE
8640045	Paul R. Michael	12/9/82

Weighed out five samples of Aroclors into 2 ml vol flask & diluted to mark with hexane

Batch ID	Aroclor	Lot No	ES Log #	Quantity (g)
I	1254	KI 03-606	2091308	1.119
II	1254	AK-38		1.147
III	1254	KI 02-602	2091306	1.012
IV	1248	KE-528		1.014
V	1016	KBC 6-706		1.001

To check to see how much PCB interference comes through after first alumina column (step #6 NBS 2286798) the ~~into~~ solution from step #9 from sample DI was concentrated to 100 µl and run on GC/MS, using conditions for CDF analysis. Very little interference appears ~~to~~ at even the 2nd alumina column is not necessary.

In the absence of a high purity radiolabled CDF isomer, anthracene will be used as an internal standard to determine if that will improve precision at least in the GC/MS step. Anthracene is aromatic, has a strong molecular ion, and is well separated (on 60M DB 1) from CDF's.

Performed short BATCH run using CDF standards (see NBS 2286796 + facing blank page) with anthracene added at the 0.5 ppm level.

Results - precision seems to be much better, but results not calculated.

READ AND UNDERSTOOD BY	DATE
K Kuehl	8/30/83

MONSANTO COMPANY

№ 2431002

SUBJECT CDF's in PCB's

JOB NO	PREPARED BY (SIGNATURE)	DATE
5640045	Paul R. Michael	12/10/82

Using procedure on NBP 2286798, steps ① - ⑥, aliquots of sample I, II, III, IV, V (see previous NBP) were prepared for analysis.
Also aliquots 6 & 7 were taken from solutions A, B, and D (NBP 2286777)
These aliquots (6 total) were prepared as I - V above

As the final step in the above preparation, the remaining sol'n was transferred quantitatively using 2 two ml 50% CH$_2$Cl$_2$/hex rinses) to a micro K-D and blown down to 0.10 ml using N_2. 100 ul of 0 xylene had been previously added to the solution.

Solutions transferred to mini-to autosampler vials (screw-cap w/ Teflon faced septa) and 5 ul of 10 ppm anthracene was added.

BATCH run set up on HP 5985 to analyze for odd CDF's

12/1 Previous run was successful and new run was started to analyze for even CDF's

READ AND UNDERSTOOD BY	DATE
K Kuehne	8/30/83

MONSANTO COMPANY

№ 2431003

SUBJECT CDF's in PCBs

JOB NO	PREPARED BY (SIGNATURE)	DATE
8640045	Paul R. Michael	12/15-16/82

12/15 Ran alumina column prep on A8, B8, C6, C7, C8, and D8.

12/16 Ran alumina column prep on A9, A10, B9, B10, C9, C10, D9, D10, Solvent Blank 1 and Solvent Blank 2.

Concentrated all solutions from 12/15 and today down to ~200 ml in micro K&D. During concentration step, B8 was lost.

READ AND UNDERSTOOD BY	DATE
K. Kuehnel	8/30/83

MONS 219918

MONSANTO COMPANY

№ 2431007

SUBJECT: CDF's in PCB's

JOB NO.	PREPARED BY (SIGNATURE)	DATE
	R. R. Michael	1/27-1/28/83

Took concentrates from 12/10 (NBP 2431003) and added 5 µl of 9.88 ppm anthracene. Rinsed down sides of K+D with a few drops of o-xylene and concentrated solutions down to 100 µl.

For standards (NBP 2286796), 5 µl of the anthracene soln was added to 100 µl autosampler vial followed by 100 µl of standard.

Samples transferred quantitatively to autosampler vials. All vials capped and sealed with Teflon lined septa.

Made several manual runs on HP5985 GC/MS to assess the state of the instrument and the retention times of the CDF isomers. During 1st run, column bleed was high. Bleed was reduced by leaving column at 320° for several hours.

Sensitivity (except for hepta CDF) looks good, also, resolution and retention time.

Setup batch run using sequence in file folder.

1/28/83 Batch run appeared to finish successfully, but on examination of the data files there was no measurable signal – just noise. Autosampler checked out OK and all vials were pierced. There is something wrong with the capillary or the MS

1/31 Ran 5 µl 9.88 ppm anthracene standard in splitless mode. Peak shape looked good but only got ~500 counts for m/z = 178 and 300 eV
Terry Hunter ran "AUTOTUNE" with PFTBA and got poor results. ∴ An MS problem.

READ AND UNDERSTOOD BY	DATE
R Michael	8/24/83

MONS 219919

MONSANTO COMPANY

№ 2431011

SUBJECT CDF's in PCB's

JOB NO.	PREPARED BY (SIGNATURE)	DATE
	R.R. Michal	2/25/83

Solutions prepared ~2 months ago (on NBP2431003) had dried up awaiting analysis by GC/MS and were of questionable value. Therefore they were discarded and new solutions prepared.

Using identical procedure (200µl spiked Aroclor/hexane solution, 1g alumina column, 10ml 2% CH$_2$Cl$_2$/hexane, 10ml 50% CH$_2$Cl$_2$/hexane) prepared aliquots A11, A12, A13, B11, B12, B13, C11, C12, C13, C14, C15, D11, D12, D13, solvent blank 1 and solvent blank 2.

The 50% fractions were blown down to ~1ml while still in the 4dram vial. A13, B13, D13, sol. bl. 2 were blown dry by mistake. The remaining solutions were transferred to micro K&D containing ~200µl o-xylene. Each vial was rinsed twice with ~0.5ml 50% CH$_2$Cl$_2$/hexane to make a quantitative transfer.

~~Each~~ K+D's placed in beakers and covered with Aluminum foil

READ AND UNDERSTOOD BY	DATE
K Kuchar	8/30/83

MONS 219920

MONSANTO COMPANY

№ 2431014

SUBJECT [illegible]

JOB NO.	PREPARED BY (SIGNATURE)	DATE
	[illegible]	[illegible]

(NBP24310A)

All solutions prepared on 2/25 were blown down to 100 µl. 5 µl anthracene std was put in 200 µl autosampler vial followed by quantitative transfer of sample to vial. Vial sealed with Teflon lined septum and screw cap.

Standards prepared in like manner.

Made manual runs in SIM mode to checkout GC/MS. looks good!

Set up batch run to measure even CDF's

Run was successful.

3/3 Set up batch run to measure odd CDF's

Sensitivity of GC/MS system decreased drastically during first few injections (as determined by looking at anthracene peak)

Data discarded

READ AND UNDERSTOOD BY	DATE
R Kuehnel	8/30/83

MONS 219921

MONSANTO COMPANY

№ 2431015

SUBJECT CDF's in PCB's

JOB NO.	PREPARED BY (SIGNATURE)	DATE
	R.R. Michael	3/9/83

After cleaning + adjusting GC/MS, sensitivity is much improved.

Using solutions run previously (NBP 2431014), set up batch run for odd CDF's

Run was successful, however because of low solution levels in some of the vials (this is the third run on the original 100 ul of solutions) A13, C13, C14 did not get a full injection or got no injection at all (as determined by monitoring anthracene internal standard).

These solutions (A13, C13, C14) will not be repeated.

Results of Recovery studies are summarized below. In the future, an additional spiking experiment should be done at much higher levels (~10x or more). This would get the CDF's significantly above the levels that are already present.

	Cl_1	Cl_2	Cl_3	Cl_4	Cl_5	Cl_6	Cl_7	Cl_8
Spike (ppm)	1.11	1.97	2.2	1.43	3.1	5.24	1.13	3.72
ave % recovery	39.0	59.4	58.5	66.0	52.8	79.1	68.8	71.3
%RSD	28	10	7.7	32	112	74	50	25
Spike (ppm)	0.111	0.197	0.22	0.143	0.31	0.524	0.113	0.372
ave % recovery	49.2	49.3	67.0	①	①	①	①	77.5
%RSD	71	56	~~227~~ 78					53
Spike (ppm)	0.056	0.099	0.11	0.072	0.16	0.26	0.057	0.19
ave % recovery	46.4	63.4	71.1	①	①	①	①	70.5
%RSD	81	29	58					104
PPM found in nonspiked A1254	0.018②	0.028②	0.118	0.592	7.83	8.32	162	0.165②

① Spiking level much less than level already present. Compounds not found, numbers based on S/N ratio

READ AND UNDERSTOOD BY	DATE
R Kuehnel	8/30/83

MONS 219922