1-31-00

MONSANTO COMPANY

№ 1999222

SUBJECT: Re-trial of extraction of oil (# 44) (see Page 1999216

JOB NO.	PREPARED BY (SIGNATURE)	DATE
13-027-760 60 3200100	David E. Kirk	8-26-8

Objective: to try a new method of cleanup to see if we can improve reproducibility. (see note on Page 1999230)

Sample: G. E. Transformer Oil # 44

Method: Do not use acid + Base washes. Cut sample size (= spikes) one half to allow alumina to handle PCB's better. Used a double column ~~setup~~ PCB to also allow better handling of PCB's on alumina.

Weigh out + Spike samples. Transfer to a regular Woelm Basic column with several rinses of 2% Methylene Chloride/Hexane. Elute column with 15 mls of 2% Methylene Chloride/Hexane. Save + Blow down to see how much PCB's came through. Elute same Column with 15 ml of 50% Methylene Chloride/Hexane. Blow to ~ 1-2 mls. ~~[illegible]~~ take This and go on second (fresh) Woelm basic column. Elute with 15 mls of 10% Methylene Chloride/Hexane. Blow down to check for PCB's.

Elute with second column with 15 mls of 50% Methylene Chloride/Hexane. add 150 µl of Tetradecane + blow to 150 µl.

Samples:			after 2%	after 50%	
3-44	Sample	0.30 gr	.21 gr	[illegible]	(3-44)B Sheet Shale
3-44R	Replicate	0.26 gr	.18 gr		
3-44 L	Low Spike	0.27 gr	[illegible]	[illegible]	[illegible]
3-44-H	High Spike	0.27 gr	[illegible]	[illegible]	[illegible]
3-44-S	Sample + Shale	0.26 gr	[illegible]	[illegible]	

All samples: 2.0 µl of 2.17 [illegible] TCDD + [illegible]

READ AND UNDERSTOOD BY: [illegible] Heleman

DATE: 6 [illegible]

MONS 220302

MONSANTO COMPANY

№ 2170506

SUBJECT Makeup of Mixed Dioxin Std

JOB NO.	PREPARED BY (SIGNATURE)	DATE
13-022-760-24 320.0100	David E. Kirk	Feb 24, 1982

Objective: to make up a standard of mixed Dioxins to be used for spiking samples and recovery studies.
Most of Std made Feb 23, 1982

Method: use old Cahn Microbalance Calibrate using documented 1 mg wt (
weigh documented @ .5147 weight and record weigh sample in Al boat. Put boats into 50 ml volumetric flask & bring to mark with o-xylene.

Sample		amount weighed	(.5147 weight)
2-Monochloro MCDD	RPE 16	631 µg	520 µg
2,7 Dichloro DCDD	RPE 25	619 µg	516 µg
1,2,4 Trichloro TRCDD	RPE 26	682 µg	514 µg
2,3,7,8 Tetrachloro TCDD	TD-194 Lt DDD5 Code	496 µg	521 µg
1,2,3,7,8 Pentachloro PCDD		598 µg	515 µg
1,2,3,4,7,8 Hexachloro	Code DDD6	575 µg	513 µg
1,2,3,4,6,7,8 Heptachloro	Code DDD7	626 µg	521 µg
Octachloro OCDD	RPE 17	500 µg	521 µg

Took 1 ml of the above mixture & put in a 10 ml vol flask. Go to the mark with dodecane to make a 1/10 dilution standard.

READ AND UNDERSTOOD BY	DATE
Fred Hileman	2 Mar 82

MONS 220303

MONSANTO COMPANY

№ 2170508

SUBJECT Preparation of New Mixed Furan Std

JOB NO.	PREPARED BY (SIGNATURE)	DATE
13-022-76024 320.0100	David E. Kirk	Feb 26, 1982

Objective: To make up a new mixed furan standard to be used for spiking samples and recoveries.

Used samples of furan cuts from Fred Heleman via Orville Hicks.
Used old calm microbalance + calibrated using .9974 std as 1 mg. weighed .5147 std to check balance for each sample. (did this for cuts 1 + 2 and octafuran) used 5 mg 5 class weight for cut three and weighed + recorded .9974 std.

(Cut #1)	0,1,2,3 Chlorinated dibenzofuran 1.7% Cl_0 31.8% Cl_1 50.5% Cl_2 15.7% Cl_3	870 µg	(.5147 std) 518 µg
(Cut #2)	3+4 Chlorinated 6% Cl_2 46.2% Cl_3 40.1% Cl_4 7.0% Cl_5	894 µg	511 µg
(Cut 3/3 cut)	30.7% Cl_5 56.5% Cl_6 12.2% Cl_7	2320 µg	(.9974 std) .9950
octachlorofuran RPE-19		929 µg	(.5147 std) 511 µg

Put in 25 ml flask + brought to mark with o-Xylene -

READ AND UNDERSTOOD BY	DATE
Fred D. Heleman	20 Mar 82

MONS 220304

NBP 2170508

	Cl_1	Cl_2	Cl_3	Cl_4	Cl_5	Cl_6	Cl_7	Cl_8
Cut 1	277	439	137	—	—	—	—	—
Cut 2	—	54	413	358	63	—	—	—
Cut 3	—	—	—	—	712	1311	283	—
Cut 4	—	—	—	—	"	—	—	92
Total µg	277	493	550	358	775	1311	283	92
µg/mL (25mL)	11.08	19.72	22.00	14.32	31.00	52.44	11.32	3.7

MONS 220305

NBP 2170506

ng/ml

	1/10	1/100	1/500
Cl_1	1.26	0.126	0.025
Cl_2	1.24	0.124	0.025
Cl_3	1.36	0.136	0.027
Cl_4	.992	0.0992	0.020
Cl_5	1.20	0.120	0.024
Cl_6	1.15	0.115	0.023
Cl_7	1.25	0.125	0.025
Cl_8	1.00	0.100	0.020

Dibenzofurans
NBP 2170508

Cl_1	1.11	0.111	0.022
Cl_2	1.97	0.197	0.039
Cl_3	2.20	0.220	0.044
Cl_4	1.43	0.143	0.029
Cl_5	3.10	0.310	0.062
Cl_6	5.24	0.524	0.105
Cl_7	1.13	0.113	0.023
Cl_8	3.72	0.372	0.074

MONS 220306

Fred,

Here are the 3 chlorinated dibenzofuran fractions we discussed over the phone. The percentage figures were determined by FID/GC - confirmed by GC/MS. You may want to compare/confirm in your laboratory, since I took fractions of semi-solids + solids; I may have "grabbed" a slightly different mix.

Ozzie Kaiser prepared our stock solutions back in Nov. 77 by weighing to constant weight (the two lower chlorinated fractions contain some benzene) - then dissolving in o-xylene + diluting to volume. We used o-xylene to minimize evaporative losses.

0, 1, 2, 3 Chlorinated Dibenzofurans

1.7% dibenzofuran
31.8% monochlorodibenzofuran
50.5% dichlorodibenzofuran
15.7% trichlorodibenzofuran

3+4 chlorinated dibenzofurans

6.0 % dichlorodibenzofuran
46.2 % tri "
40.1 % tetra "
7.0 % penta "

Last 43's cut

30.7 % pentachlorodibenzofuran
56.5 % hexa "
12.8 % hepta "

MONS 220307