

Method 525Date Extracted 11/21/01

Page _____

Lab #	Description	Sample vol/wt	Drip rate ml/min	Surr add	Spk add	IS add	Final vol.	T.V. pos.	pH 2	Count	Cart #	next
	LB	1L		1ml				7B			722501	
	MDL	1L			✓			10A				
	REF	1L			✓			7B				
	27984	1L						3B		12		
	27985	1L						7B		13		
	27986	1L						8A		14		
+	28043	1L						10B		15		✓
+	28044	1L						3A		16		
	28045	941ml						6A		17		
	28046	1L						6B		18		
	28047	981ml		↓				9A	↓	19	↓	
	28048 WBS 11/21/01											
	28049 WBS 11/21/01											
	28104 WBS 11/21/01											
	28105	996ml		1ml				2B		20	722501	
	28105-S	1L			✓			7B				
	28105-S0	1L			✓			8A				
+	28048	957ml						4B		1		
	28049	989ml						3B		2		
	28104	996ml						2B		3		
	28106	1L						6B		4		
	28164	1L						8A		5		
+	28165	1L						6A		6		
	28166	983ml						8A		7		
	28167	1L		↓				8A	↓	8	↓	
Remarks * 28048 and 28165 went to dry wash on Turnover re-extracted on 11/26/01 Study												
drip rate must be between 5-15 ml/min for liq-liq extractors Extraction type - <u>SPE</u> Solv. lot# <u>V43E07</u> NaCl lot# _____ Na2SO4 lot# <u>V32603</u> STANDARD INT STD LOG# _____ CALIBRATION _____ REF/SPIKE <u>2083a / 2084a / 2082a</u> MDL/MDRF <u>" / 1865c / "</u> Q. CHECK _____ INT. STD. <u>2118C</u> SURROGATE <u>2119h</u> LPC _____ ENDRIN/DDT _____												

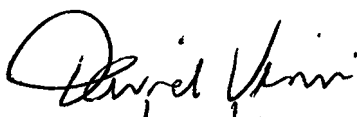
Reviewed by,

11/24/02Analyst D.W.M.
revision 4/12/01D.G.W.

Verified by _____

Reviewed by DNDate 1/30/2

All QC runs and samples associated with batches 11/21/01, and 12/19/01 gave typical results similar to all the previous batches analyzed on the new Agilent GCMS for method 525.2 with the following exception: the Endrin breakdown result was suddenly greater than 70%. Unfortunately this was not realized until 3 weeks after the injections were made. The instrument was removed from service and a thorough investigation was begun to attempt to determine and correct the causes of the high Endrin breakdown as well as low recoveries of several other less stable compounds. Involved in our investigation was Agilent staff, Finnigan staf., our 2 service consultants from MDL Systems, our gas suppliers - Air Gas Corporation, and members of our staff. Simultaneously, 4 other instruments in the laboratory suddenly experienced high Endrin breakdown also. Around January 22, the problems were isolated and for the most part corrected for each instrument. The causes were due to a combination of various problems, some which may have been building over time, i.e. bad vial of Endrin standard, contaminated gas lines and gas purifying traps, much too high injection port temperatures, bad lot of GC septa, improper design of Finnigan Ion Trap injection port, and inherently poor design of Agilent GC injection ports. Though we can not be conclusive as to which causes contributed the most to the problem, and which ones affected each instrument exactly, all in all with many corrections made, we believe we are now able to control the Endrin breakdown levels. In addition, we observed improved recoveries thus far for the 18 compounds analyzed by our method 525.2. In order for me to reaffirm the reliability of data obtained from the original injections for these 2 batches, we reinjected them and compared the new data against the original data. All results agree with improved responses for problem compounds and surrogates. The Endrin breakdown was 10.6%. Thus, I reported the originally obtained data but the newly obtained Endrin % breakdown result.


1/30/02

Sequence Name: C:\MSDchem\1\sequence\122801.s
Comment:
Operator: McLean
Data Path: C:\MSDCHEM\1\DATA\122801\
Pre-Seq Cmd:
Post-Seq Cmd:

Method Sections To Run On A Barcode Mismatch
(X) Full Method (X) Inject Anyway
() Reprocessing Only () Don't Inject

Original

Line	Type	Vial	DataFile	Method	Sample Name
1	Sample	1	DFTPP1	BIG3	dftpp +end
2	Sample	2	DDT1	BIG3	ddt
3	Sample	3	CAL2A	121401	5.0
4	Sample	4	MC	121401	
5	Sample	5	1219LB	121401	12/19
6	Sample	6	1219REF	121401	12/19
7	Sample	7	1219MDL	121401	12/19
8	Sample	8	30770	121401	12/19
9	Sample	9	30768	121401	12/19
10	Sample	10	30762	121401	12/19
11	Sample	11	30775	121401	12/19
12	Sample	12	30772	121401	12/19
13	Sample	13	30771	121401	12/19
14	Sample	14	30716S	121401	12/19 spk
15	Sample	15	30716SD	121401	12/19 spk dupe
16	Sample	16	MC2	121401	
17	Sample	1	DFTP2	BIG3	
18	Sample	2	DDT2	BIG3	
19	Sample	17	CAL3	121401	0.50
20	Sample	18	LB1219	121401	ucmr 12/19
21	Sample	19	REF1219	121401	ucmr 12/19
22	Sample	20	MDL1219	121401	ucmr 12/19
23	Sample	21	30739	121401	ucmr 12/19
24	Sample	22	30740	121401	ucmr 12/19
25	Sample	23	30741	121401	ucmr 12/19
26	Sample	24	30742	121401	ucmr 12/19
27	Sample	25	30743	121401	ucmr 12/19
28	Sample	26	30769	121401	ucmr 12/19
29	Sample	27	30740S	121401	ucmr 12/19 spk
30	Sample	28	30740SD	121401	ucmr 12/19 spk dupe
31	Sample	3	CAL6	121401	5.0
32					

Sequence Name: C:\MSDchem\1\sequence\012502a.s
 Comment:
 Operator: McLean
 Data Path: C:\MSDCHEM\1\DATA\012502A\
 Pre-Seq Cmd:
 Post-Seq Cmd:

Reinjection
Alm

Method Sections To Run On A Barcode Mismatch
 (X) Full Method (X) Inject Anyway
 () Reprocessing Only () Don't Inject

Line	Type	Vial	DataFile	Method	Sample Name
1	Sample	3	DFTPPB	012402DF	200 1 UL
2	Sample	4	DDT	012402DF	200 1 UL
3	Sample	5	C6	012402DF	5.0 CC
4	Sample	1	LB1121R	012402DF	
5	Sample	2	27984RR	012402DF	REINJ
6	Sample	3	27985RR	012402DF	REINJ
7	Sample	4	REF1121	012402DF	REINJ
8	Sample	5	MDL1121	012402DF	REINJ
9	Sample	6	28164RR	012402DF	REINJ
10	Sample	7	28166RR	012402DF	REINJ
11	Sample	8	28167RR	012402DF	REINJ
12	Sample	9	28044RR	012402DF	REINJ
13	Sample	10	28045RR	012402DF	REINJ
14	Sample	11	28046RR	012402DF	REINJ
15	Sample	12	28047RR	012402DF	REINJ
16	Sample	13	LB0111	012402DF	REINJ
17	Sample	14	28043RR	012402DF	REINJ
18	Sample	15	REF111	012402DF	REINJ 0111
19	Sample	16	DFTPP2	012402DF	
20	Sample	17	DDT2	012402DF	REINJ
21	Sample	18	C5	012402DF	CCV
22	Sample	19	MC2	012402DF	
23	Sample	20	28104RR	012402DF	REINJ
24	Sample	21	28105RR	012402DF	REINJ
25	Sample	22	28106RR	012402DF	REINJ
26	Sample	23	28049RR	012402DF	REINJ
27	Sample	24	28105SP	012402DF	REINJ
28	Sample	25	28105SD	012402DF	REINJ
29	Sample	26	REF0111D	012402DF	REINJ REF DUPE
30	Sample	27	1219REF	012402DF	REINJ REF
31	Sample	28	1219MDL	012402DF	REINJ REF
32	Sample	29	1219LB	012402DF	REINJ REF
33	Sample	30	30771R	012402DF	REINJ REF
34	Sample	31	30772R	012402DF	REINJ REF
35	Sample	32	30775R	012402DF	REINJ REF
36	Sample	33	30762R	012402DF	REINJ REF