



Technical Service Report

Report No. 431-81-804-8

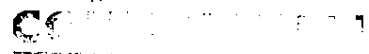
To E. L. Sones

From C. B. Herrin

Date April 8, 1981

Subject PURGEABLE PRIORITY POLLUTANTS IN SAMPLE VOA 4236-93-13 LCCP 002
ANALYTICAL NUMBER 16998

Conoco Inc.
Research and Development Department
Analytical Research Section
Ponca City, Oklahoma



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Conclusion

The following priority pollutants were found:

1) methylene chloride	1 ppb
2) chloroform	0.9 ppb
3) 1,2 dichloroethane	3 ppb
4) benzene	244 ppb
5) toluene	60 ppb
6) ethyl benzene	2 ppb.

Other components found in "significant" amounts were butane and hexane and butanol and hexanol.

Experimental

The sample was run on the Finnigan OWA-30 GC/MS/DS using a Tekmar LSC-2 automatic purge-and-trap apparatus. The conditions were:

Sample Concentrator

Purge gas	He @ 40 mL/min
Purge time	12 min
Desorption time	4 min
Desorption temperature	180°C
Sample size	5 mL
Internal standards	1,4 dichlorobutane and bromochloromethane.

Gas Chromatograph

Carrier	He @ 20 mL/min
Column	6 ft, 1% SP 1000 on Carbopak B
Oven temperature program	30°C for 4 min initial hold then 8°/min up to 180°C then hold for 120 min.

Mass Spectrometer

Scan 40-250 amu in 4 sec
1000 scans collected.

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The sample was removed from the cold room and allowed to come to room temperature before it was run. Methanolic solutions of the internal standards were injected into a syringe containing 5 mL of sample. Calibration was accomplished by dosing boiled-and-sparged Milli-Q water with the internal standards and a blend of EPA Consent Decree volatile standards prepared by Radian Corp. and purchased from Regis Chemical Co. This dosed water was run under the same conditions as the sample, and the data obtained were fed into two of the OWA system libraries. The data from the sample were run through the OWA system automatic quantification program which employed the two libraries in the reverse search mode. The standard blend did not contain any compounds more volatile than methylene chloride, so a manual search was performed for the five gaseous priority pollutants. None were detected.

Discussion of Results

The sample contained non-priority pollutant components which appeared to be in excess of 10 ppb. These components were forward searched using the NBS library and an automatic search routine. Good matches with reference spectra were obtained for the compounds listed in the Conclusion section. Xylenes were not detected. The automatic quantification program gave hits for trichloroethylene and tetrachloroethylene. These may have been false positives. The spectra were not good enough to make a positive identification. The automatic system also gave a false negative for methylene chloride. The methylene chloride value was calculated manually.

C. B. Herrin

C. B. Herrin
Research Group Supervisor
Gas Chromatography Group
Analytical Research Section

BW
Attachments
Copies to

FK EAS Lib.

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SCAN 1 OF 1000

3/26/81 15:45:16

ACQ STARTED

UNT 01619013

SAMPLE: LCCF002

SUBMITTED BY: ELS

ANALYST: CBH

COMMENTS: 4236-93-13 LCCF 002 VOA 3/25/81 5 ML SAMPLE +698 NG 14DCB
AND 564 NG BOM PURGE & TRAP

GC BYPASSED

INJ. MODE: FID

1 SEC = DV OPEN

FTL/MUL MODE: AUTO

SCAN FROM 40 AMU TO 250 AMU IN 4.0 SEC

3/26/81 15:53:31
ACQUISITION COMPLETED

UNKNOWN SAMPLE QUANTITATION

OWA REVERSE SEARCH STATUS REPORT

THE INTERNAL STANDARD AREA HAS A DIFFERENCE OF: -100 PERCENT OF THE LAST STANDARD
INTERNAL STANDARD FOUND FOR LIBRARY: LIBRARYV1

COMPOUNDS QUANTITATED BY INTERNAL STANDARD USING PARAMETERS IN LIBRARYV1

REPORT ENTRY NO.	EXPECTED SCAN	BEST SCAN	FIT	PURITY	LIBRARY ENTRY NO	PEAKS FOUND	PEAKS QUANT	SATURATED PEAKS
						**		
1	212	215	993	672	1	14	1	0
12	244	247	998	281	12	1	1	0
13	256	258	964	307	13	1	1	0

NUMBER OF COMPOUNDS IDENTIFIED 3

DATA PROCESSING OF GL9313 COMPLETED ON 4/06/81 19.33:17

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MINNICH TARGET COMPOUND ANALYSIS
QUANTIFICATION REPORT FILE: GL9313

DATA: GL9313 T1
03/26/91 15:34:00
SAMPLE: LCCP002
SUBMITTED BY: ELS

ANALYST: CBH

AMOUNT=AREA(HGHT) * REF. AMNT/(REF. AREA(HGHT)* RESP. FACT)
RESP. FAC. FROM LIBRARY ENTRY

- | NO | NAME |
|----|--|
| 1 | BROMOCHLOROMETHANE (INTERNAL STANDARD) |
| 2 | CHLOROMETHANE |
| 3 | DICHLORODIFLUOROMETHANE |
| 4 | BROMOMETHANE |
| 5 | VINYL CHLORIDE |
| 6 | CHLOROETHANE |
| 7 | METHYLENE CHLORIDE |
| 8 | TRICHLOROFLUOROMETHANE |
| 9 | 1,1-DICHLOROETHYLENE |
| 10 | 1,1-DICHLOROETHANE |
| 11 | TRANS-1,2-DICHLOROETHYLENE |
| 12 | CHLOROFORM |
| 13 | 1,2-DICHLOROETHANE |
| 14 | 1,1,1-TRICHLOROETHANE |
| 15 | CARBON TETRACHLORIDE |
| 16 | BROMODICHLOROMETHANE |

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA(HGHT)	AMOUNT	%TOT
1	180	215	14.31	1	1.000	A BB	138019.	100.000 UG/L	96.05
2	NOT FOUND								
3	NOT FOUND								
4	NOT FOUND								
5	NOT FOUND								
6	NOT FOUND								
7	NOT FOUND								
8	NOT FOUND								
9	NOT FOUND								
10	NOT FOUND								
11	NOT FOUND								
12	83	247	16.40	1	1.149	A BB	3155.	0.847 UG/L	0.81
13	95	258	17.25	1	1.200	A BB	797	3.266 UG/L	3.14
14	NOT FOUND								
15	NOT FOUND								
16	NOT FOUND								

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PINNIGAN TARGET COMPOUND ANALYSIS
QUANTITATION REPORT FILE: GL7313

DATA: GL7313.01
03/26/91 15:34:00
SAMPLE: LCCP002
SUBMITTED BY: ELS

ANALYST: CBH

AMOUNT=AREA(HGHT) * REF. AMNT/(REF. AREA(HGHT)* RESP. FACT)
RESP. FAC. FROM LIBRARY ENTRY

NO	NAME
1	1,4-DICHLOROBUTANE (INTERNAL STANDARD)
2	1,2-DICHLOROPROPANE
3	TRANS-1,3-DICHLOROPROPENE
4	TRICHLOROETHYLENE
5	BENZENE
6	CIS-1,3-DICHLOROPROPENE
7	1,1,2-TRICHLOROETHANE
8	DIBROMOCHLOROMETHANE
9	BROMOFORM
10	1,1,2,2-TETRACHLOROETHYLENE
11	1,1,2,2-TETRACHLOROETHANE
12	TOLUENE
13	CHLOROBENZENE
14	ETHYLBENZENE

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA(HGHT)	AMOUNT	%OT
1	55	440	29:42	1	1.000	A BB	328818.	140.000 NG/ML	31.41
2	NOT FOUND								
3	NOT FOUND								
4	130	323	21:48	1	0.734	A BB	1008.	0.267 NG/ML	0.06
5	78	331	22:21	1	0.752	A BB	2065130.	243.561 NG/ML	54.65
6	NOT FOUND								
7	NOT FOUND								
8	NOT FOUND								
9	NOT FOUND								
10	124	431	29:56	1	0.980	A BB	86.	0.025 NG/ML	0.01
11	NOT FOUND								
12	52	476	32:08	1	1.082	A BB	287352.	59.535 NG/ML	13.36
13	NOT FOUND								
14	103	636	42:56	1	1.445	A BB	5914	2.306 NG/ML	0.52

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THE INTERNAL STANDARD AREA HAS A DIFFERENCE OF: 16 PERCENT OF THE LAST STANDARD
INTERNAL STANDARD FOUND FOR LIBRARY: LIBRARYV2

COMPOUNDS QUANTITATED BY INTERNAL STANDARD USING PARAMETERS IN LIBRARYV2

REPORT ENTRY NO.	EXPECTED SCAN	BEST SCAN	FIT	PURITY	LIBRARY ENTRY NO.	PEAKS FOUND	PEAKS QUANT	SATURATED PEAKS
1	438	440	989	476	1	1	1	0
4	321	323	993	6	4	1	1	0
5	329	331	1000	483	5	1	1	0
10	429	431	921	33	10	1	1	0
12	475	476	996	641	12	1	1	0
14	634	635	997	198	14	1	1	0

NUMBER OF COMPOUNDS IDENTIFIED 6

DATA PROCESSING OF GL9313 COMPLETED ON 4/06/81 20:48:27

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