JSB is an authorised partner of Agilent Technologies Value Added Reseller

#032

Reproducibility of Automated Purge & Trap/GC: EPA Method 502.2

The reproducibility of the methods used for environmental analyses must be documented for EPA audits. In purge and trap/GC combined with an autosampler, there are numerous steps where error can be introduced, from errors in sampling, to variations in the sample volume n delivered by the autosampler to the purge vessel, to degradation of the detector sensitivity during a series of analyses. The *CDS Analytical EA-600* with a *Dynatech Autosampler* (the *CDS Analytical Auto-EA)* equipped with a *HALL* ELCO and a PIO was evaluated to determine the reproducibility of a test mixture of the compounds specified in EPA Method 502.2 over a 16 hour analysis period. The samples were 20 ppb in 5 ml water, using standard EPA Method 502.2 conditions (see back).

For Method 502.2, the relative standard deviations for all analytes must be within 20%. It is a particularly difficult analyses because of the number of analytes (60), the requirement to use two detectors in series, and the close elution time of many of the analytes. In addition, some of the analytes are very sensitive to factors such as the gradual buildup of water on the trap during a long series of analyses, and thus are more likely to result in standard deviations above the EPA mandated level.

Figures 1 through 4 are two pairs of the chromatograms obtained in this evaluation. The chromatograms at the beginning of the test series (Figures 1 and 2) are identical to those obtained after 14 analyses (Figure 3 and 4). The relative standard deviation for most analytes was from 4-6 %, and for all compounds tested was less than the EPA limit of 20% (Table 1). This indicates that the Auto-EA can be used with confidence of EPA analyses.

Figure 1 First Run in Series: HALL

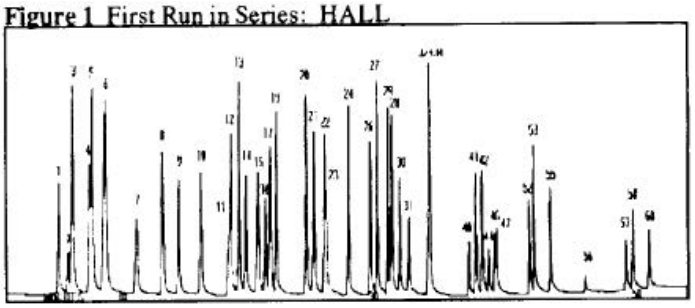

Figure 2 First Run in Series: PID

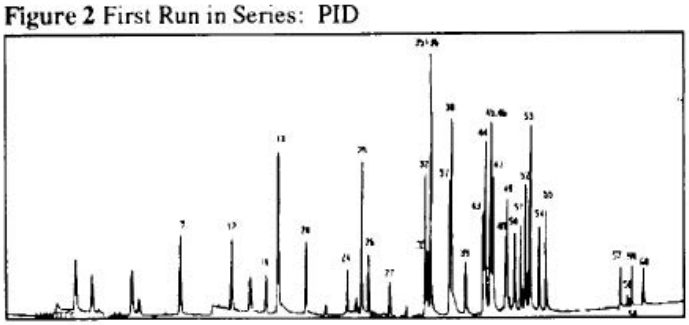

Figure 3 Fourteenth Run in Series: HALL

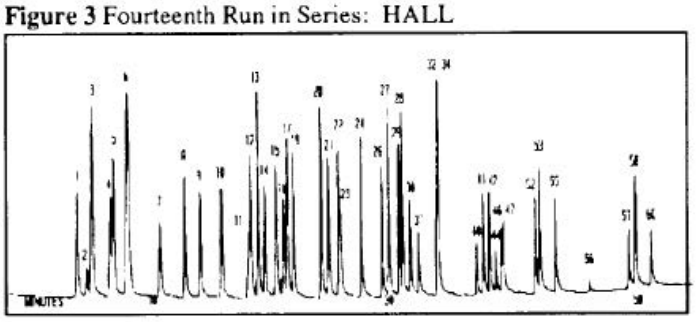

Figure 4 Fourteenth Run in Series: PID

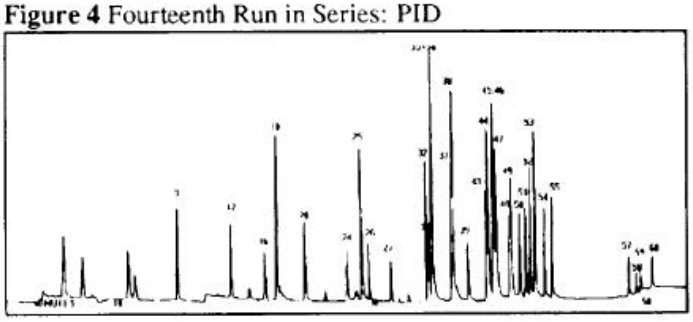

Table 1

Peak	HALL	%RSD	PID	%RSD
1. dichlorodifluoromethane		5.7		ND
2. chloromethane		8.2		ND
3. vinyl chloride		5.2		ND
4. bromomethane		6.3		ND
5. chloroethane		7.9		ND
6. trichlorofluoromethane		4.9		ND
7. 1, 1-dichloroethene		5.3		ND
8. methylene chloride		8.3		ND
9. trans-1, 2-dichloroethene		6.5		6.8
10. 1,1-dichloroethane		8.0		ND
11. 2, 2-dichloropropane •		6.6		ND
12. cis- 1 ,2-dichloroethene •		6.6		4.0
13. chloroform			4.2	ND
14. bromochloromethane		4.1		ND
15. 1, 1, 1-trichloroethane			4.1	ND
16. 1,1-dichloropropene		4.3		ND
17. carbon tetrachloride			5.0	ND
18. benzene			ND	3.6
19. 1,2-dichloroethane			7.7	ND
20. trichloroethene			4.6	3.8
21. 1 ,2-dichloropropane			5.6	ND
22. bromodichloromethane			5.0	ND
23. dibromomethane			6.6	ND
124. cis- 1 ,3-dichloropropene			6.4	3.1
125. toluene		ND		4.1
126. trans 1 ,3-dichloropropene			8.3	2.0
7. 1,1,2-trichloroethane		7.9		ND
128. 1 ,3-dichloropropane			7.7	ND
9. tetrachloroethene		5.2		NO
po. dibromochloromethane			7.5	ND
P 1. 1,2-dibromomethane		8.0		ND
P2. chlorobenzene			4.9	4.9
S3. ethyl benzene			ND	4.3
P4. 1,1, 1,2-tetrachloroethane			4.9	ND
S5. m-xylere •••			ND	4.3
P6. p-xylene •••			ND	4.3
p7. o-xylene			ND	6.3
pa. styrene			ND	6.0
P9. isopropyl benzene			ND	7.5
µ(), bromoform			5.8	ND

Analytical Conditions

Trap: Tenax-Silica Gel-Charcoal
Purge: 11 minutes
Flow: 38 cc/min HE
Trap temperature: 35 C
Desorb: 280 C, 3 min
Bake: 280 C, 2 min
GC Column: 105 m, 0.53mm ID
RTX Volatiles
GC Program: 30 C, hold 10 min;
4 C/min to 180 C, hold 5 min.
Sample: Restek Standard; 20 ppb in 5 ml water

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READING:

Air and Water Pollution: A Guide to Federal Regulations. J.J. Keller & Associates, Inc.

Sources of error in purge and trap analysis of volatile organic compounds. J.W. Washall, T.P. Wampler. American Lab, 22, 18 (1990) 38-44.

CDSolutions: *Water Management in Purge and Trap!GC.* M.J. Matheson, T.P. Wampler.

Produced by M.J. Matheson 593.

Headquarters
JSB International
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 251 47 53
F +31 (0) 40 251 47 58

Zoex Europe
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 257 39 72
F +31 (0) 40 251 47 58

Sales and Service

Netherlands
Apolloweg 2B
8239 DA Lelystad
T +31 (0) 320 87 00 18
F +31 (0) 320 87 00 19

Belgium
Grensstraat 7
Box 3 1831 Diegem
T +32 (0) 2 721 92 11
F +32 (0) 2 720 76 22

Germany
Max-Planck-Strasse 4
D-47475 Kamp-Lintfort
T +49 (0) 28 42 9280 799
F +49 (0) 28 42 9732 638

UK & Ireland
Cedar Court,
Grove Park Business Est.
White Waltham, Maidenhead
Berks, SL6 3LW
T +44 (0) 16 288 220 48
F +44 (0) 70 394 006 78

info@go-jsb.com
www.go-jsb.com