CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Reproducibility of Automated Purge and 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 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* ELCD and a PID 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 for EPA analyses.

Figure 1 First Run in Series: HALL

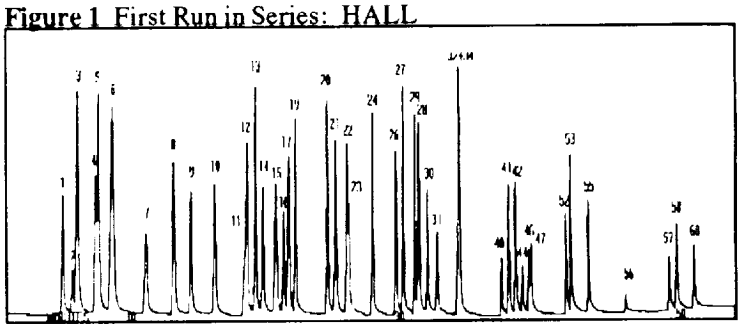

Figure 2 First Run in Series: PID

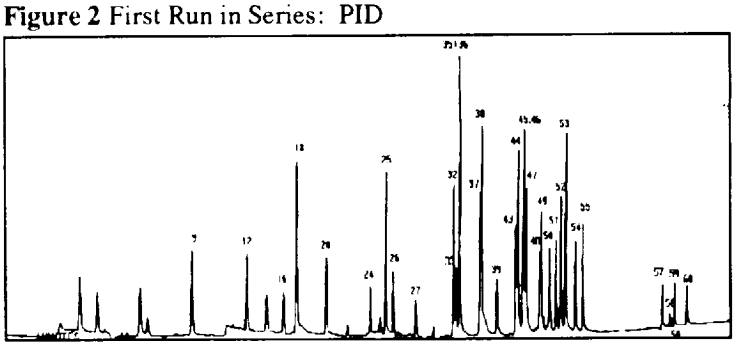

Figure 3 Fourteenth Run in Series: HALL

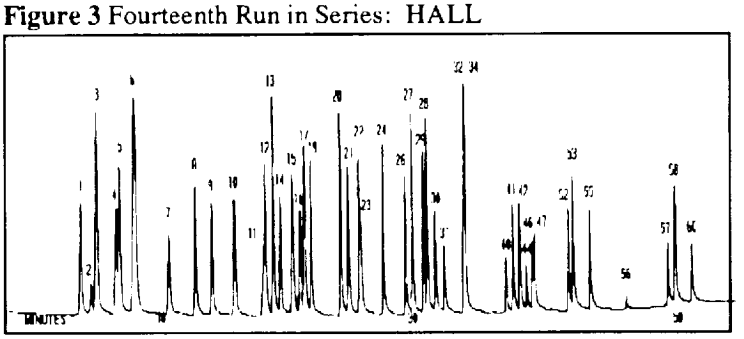

Figure 4 Fourteenth Run in Series: PID

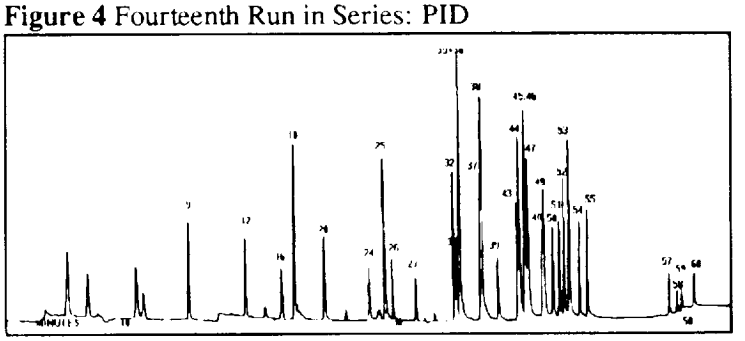

Table 1

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
24. cis-1,3-dichloropropene		6.4		3.1
25. toluene		ND		4.1
26. trans-1,3-dichloropropene		8.3		2.0
27. 1,1,2-trichloroethane		7.9		ND
28. 1,3-dichloropropane		7.7		ND
29. tetrachloroethene		5.2		ND
30. dibromochloromethane		7.5		ND
31. 1,2-dibromomethane		8.0		ND
32. chlorobenzene**		4.9		4.9
33. ethyl benzene		ND		4.3
34. 1,1,1,2-tetrachloroethane**		4.9		ND
35. m-xylene***		ND		4.3
36. p-xylene***		ND		4.3
37. o-xylene		ND		6.3
38. styrene		ND		6.0
39. isopropyl benzene		ND		7.5
40. bromoform		5.8		ND
41. 1,1,2,2-tetrachloroethane		8.4		ND
42. 1,2,3-trichloropropane		8.8		ND
43. n-propyl benzene		ND		6.5
44. bromobenzene		5.0		3.8
45. 1,3,5-trimethylbenzene		ND		5.4
46. 2-chlorotoluene		5.8		5.5
47. 4-chlorotoluene		5.7		5.6
48. tert-butlybenzene		ND		6.3
49. 1,2,4-trimethylbenzene		ND		5.2
50. sec-butylbenzene		ND		4.8
51. p-isopropyltoluene		ND		5.9
52. 1,3-dichlorobenzene		4.9		4.1
53. 1,4-dichlorobenzene		5.6		4.1
54. n-butylbenzene		ND		6.6
55. 1,2-dichlorobenzene		7.6		3.7
56. 1,2-dibromo-3-chloropropane		17.8		ND
57. 1,2,4-trichlorobenzene		7.2		5.7
58. hexachlorobutadiene		8.8		10.9
59. naphthalene		ND		16.4
60. 1,2,3-trichlorobenzene		10.2		9.9

*not separated **not separated ***not separated

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.

For more information on this and related applications, contact your local CDS representative, or contact CDS directly at the address below.

Produced by M.J. Matheson 593.

ABOUT CDS

CDS Analytical, Inc. is a leader in the design and manufacture of laboratory instruments for sample preparation and analysis. With 20 years experience in the field, CDS is dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of analytical pyrolysis, CDS manufactures the Pyroprobe 1000 and 2000 for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of purge and trap instruments for the analysis of volatile organic compounds in the environmental, food and pharmaceutical areas, as well as custom systems for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet their needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, or call us at 1 800 541 6593.

CDS Analytical, Inc. 7000 Limestone Rd., P.O Box 277. Oxford, PA USA 19363-0277 Ph: 215 932 3636