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Application Note 160

The Analysis of Volatile Organic Compounds by Purge and Trap on the SPB™-624 Column

The SPB-624 column is an excellent choice for the separation of a wide range of volatile organic contaminants typically monitored for in air, soil and water.

Key Words:
● volatile ● desorption ● organic ● purge and trap

The SPB-624 column is an excellent choice for the separation of a wide range of volatile organic contaminants typically monitored for in air, soil and water. Often, this type of work is accomplished through purge and trap – GC/MS analysis. For GC/MS systems equipped with a jet separator, we recommend the 75m x 0.53mm ID, 3.0µm dimension. A 0.53mm ID column will accommodate faster flow rates than a 0.32 or 0.25mm. This faster flow rate will in turn allow a faster flow of gas through the purge trap during the desorption portion of the purge and trap cycle. A faster desorption flow rate will transfer the analytes to the head of the column in a tighter band resulting in better peak shapes. Since a GC/MS typically cannot accommodate flow rates in excess of 2-3 mL/min, a jet separator must be used to split the sample prior to entering the detector.

Poor trapping of the gaseous volatiles at the head of the column will result in wide and tailing peaks. Cryogenic cooling is used often to overcome this problem. With the 3.0µm film SPB-624, cryogenics are not necessary. Efficient focusing of the gaseous VOCs can be achieved at ambient temperature. Figure A illustrates a run of a 50ng volatile standard on the 75m x 0.53mm ID, 3.0µm SPB-624 column. Even at a starting temperature of 40°C with a hold time of only 2 minutes, the column was able to resolve the gases. For a production laboratory, this shorter run time can mean greater sample throughput. If one can sacrifice on the speed of the overall analysis, the response and peak shape of the gases can be improved by starting the run at 35°C and holding for a longer time.

Table 1 lists the average response factors and %RSD values obtained for 94 compounds in a calibration range of 50 – 1000ng on-column. The column showed excellent linearity for most compounds in the range of 50 to 1000ng on-column. Being able to calibrate up to 1000ng on-column can mean fewer sample reruns due to dilution (for samples falling outside the calibration range.)

Figure A. Purge and Trap Analysis of a 50ng VOA Standard on the SPB-624 Column

Sample:	50ng each component in 5ml of water
Purge and Trap:	Tekmar ® LSC 2000
Trap:	Vocarb™ 3000
Purge:	11 min, 40ml/min, ambient temp.
Dry Purge:	3 min.
Desorption Temp:	250°C for 4 min.
Bake:	280°C for 10 min.
Column:	**SPB-624 fused silica capillary column** 75m x 0.53mm ID, 3.0µm SPB-624
Cat. No.:	**25432**
Oven:	40°C (2 min.) to 65°C at 5°C/min, to 155°C at 12°C/min, to 210°C at 25°C/min
Carrier:	He, 10 ml/min
Detector:	MS, scan range 35-350 (With jet separator)

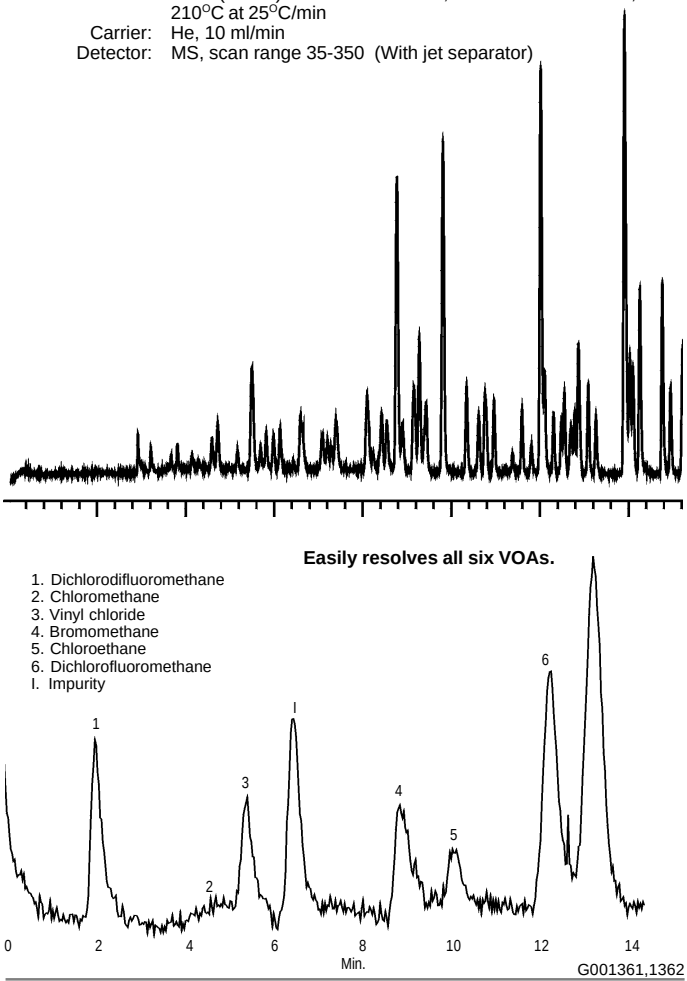

Table 1. Results of a VOA Calibration Curve From 50 – 1000ng on the SPB-624 Capillary Column

Compound	RT (min.)	Quant Ion	Avg. RRF	%RSD
Fluorobenzene	9.77	96		
Chlorobenzene-d5	13.90	117		
1,4-Dichlorobenzene-d4	16.62	152		
Dibromofluoromethane	8.74	113	0.4080	4.38
1,2-Dichloroethane-d4	9.25	102	0.0440	8.60
Toluene-d8	12.00	98	0.5800	4.35
Bromoflurobenzene	5.36	95	0.5670	20.06
Dichlorodifluoromethane	3.20	85	0.1550	10.26
Chloromethane	3.60	50	0.0970	4.89
Vinyl Chloride	3.66	62	0.1100	6.39
Bromomethane	4.11	94	0.0700	9.51
Chloroethane	4.26	64	0.0380	12.52
Dichloroflouromethane	4.54	67	0.3010	10.00
Trichloromonoflouromethane	4.64	101	0.2780	4.55
Diethyl Ether	5.20	74	0.0590	6.76
Acrolein	5.31	56	0.0290	8.34
1,1-Dichloroethene	5.44	96	0.1430	2.69
Trichlorotriflouroethane	5.47	151	0.2640	3.66
Acetone	5.58	58	0.0360	46.63
Iodomethane	5.65	142	0.3120	12.13
Carbon Disulfide	5.77	76	0.3490	3.58
Allyl Chloride	5.96	76	0.0770	12.45
Methylene Chloride	6.11	84	0.1430	6.68
tert-Butyl Alcohol	6.47	59	0.0450	11.39
Acrylonitrile	6.47	53	0.0560	4.53
trans-1,2-Dichloroethene	6.57	96	0.1600	4.81
Methyl-tert-Butyl Ether	6.67	73	0.3410	5.27
Hexane	7.05	57	0.1740	7.03
1,1-Dichloroethane	7.19	63	0.2680	6.11
Vinyl Acetate	7.30	43	0.2170	7.01
Chloroprene	7.36	53	0.2010	5.93
Disopropyl Ether	7.43	45	0.2380	5.26
cis-1,2-Dichloroethene	8.05	96	0.1620	4.67
2,2-Dichloropropane	8.09	77	0.2200	6.02
2-Butanone	8.11	72	0.0530	34.21
Propionitrile	8.12	54	0.0220	11.36
Ethyl Acetate	8.22	88	0.0070	41.25
Methacrylonitrile	8.38	41	0.1130	15.56
Bromochloromethane	8.40	128	0.1250	4.07
Chloroform	8.51	83	0.2910	5.44
Tetrahydrofuran	8.58	42	0.0840	5.88
1,1,1-Trichloroethane	8.86	97	0.2640	4.47
1,1-Dichloropropene	9.09	75	0.1860	5.26
Carbon Tetrachloride	9.13	117	0.2900	5.23
Isobutyl Alcohol	9.26	43	0.0250	14.30
1,2-Dichloroethane	9.35	62	0.1610	6.21
Benzene	9.39	78	0.3720	5.23
1-Butanol	10.21	56	0.0120	11.28
Trichloroethylene	10.31	95	0.2400	4.74
1,2-Dichloropropane	10.59	63	0.2050	5.30
Dibromomethane	10.73	93	0.2380	4.43
Methyl Methacrylate	10.78	69	0.1830	4.41
1,4-Dioxane	10.83	88	0.0090	17.89
Bromodichloromethane	10.94	83	0.3570	5.51
2-Nitropropane	11.20	43	0.0860	7.53
2-Chloroethyl Vinyl Ether	11.37	63	0.0950	7.17
cis-1,3-Dichloropropene	11.57	75	0.2830	5.65
Methyl Isobutyl Ketone	11.80	43	0.4050	5.39
Toluene	12.08	92	0.3180	5.29
trans-1,3-Dichloropropene	12.29	75	0.2520	6.96
Ethyl Methacrylate	12.47	69	0.3110	5.71
1,1,2-Trichloroethane	12.54	83	0.1890	5.59
1,3-Dichloropropane	12.77	76	0.2850	6.49
Tetrachloroethylene	12.85	164	0.2380	4.01
2-Hexanone	12.90	43	0.3130	6.60
Dibromochloromethane	13.08	129	0.4290	5.59
1,2-Dibromoethane	13.25	107	0.3770	5.71
Chlorobenzene	13.94	112	0.4800	4.17
1,1,1,2-Tetrachloroethane	14.02	131	0.2890	4.30
Ethylbenzene	14.09	91	0.6400	4.99
m-Xylene	14.25	106	0.5050	5.22
p-Xylene	14.25	106	0.5050	5.22
o-Xylene	14.76	106	0.2460	7.58
Styrene	14.76	104	0.4350	7.33
Bromoform	14.95	173	0.4380	6.89
Iso-Propylbenzene	15.22	105	0.7420	7.94
Cyclohexanone	15.26	55	0.3520	24.65
1,1,2,2-Tetrachloroethane	15.46	83	0.4830	6.54
trans-1,4-Dichloro-2-Butene	15.52	53	0.1020	9.45
1,2,3-Trichloropropane	15.53	75	0.5000	8.56
Bromobenzene	15.56	156	0.2930	10.85
n-Propylbenzene	15.69	91	0.7770	6.32
2-Chlorotoluene	15.78	91	0.6050	5.99
1,3,5-Trimethylbenzene	15.88	105	0.5580	7.73
4-Chlorotoluene	15.89	91	0.6590	6.99
tert-Butylbenzene	16.24	91	0.3940	7.74
1,2,4-Trimethylbenzene	16.28	105	0.5250	5.24
sec-Butylbenzene	16.46	105	0.9110	6.60
1,3-Dichlorobenzene	16.56	146	0.4230	9.25
Iso-Propyltoluene	16.60	119	0.7140	10.00
1,4-Dichlorobenzene	16.65	146	0.8870	1.74
n-Butylbenzene	17.01	91	1.0720	6.09
1,2-Dichlorobenzene	17.02	146	0.7780	1.42
1,2-Dibromo-3-chloropropane	17.78	75	0.2660	5.63
1,2,4-Trichlorobenzene	18.78	180	0.6990	13.09
Hexachlorobutadiene	19.02	225	0.5130	12.73
Naphthalene	19.09	128	1.5420	12.51
1,2,3-Trichlorobenzene	19.40	180	0.7120	10.16

internal standards

Trademarks

SPB – Sigma-Aldrich Co.
Vocarb – Sigma-Aldrich Co.
Tekmar – Tekmar Corp.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

Ordering Information:

Description	Cat. No.
SPB-624 Fused Silica Capillary Column	
75m x 0.53mm ID, 3.0µm film	**25432**

Note 160

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