O·I·Analytical

Reduced VOC Sample Analysis Times Using a New Dual Purge-and-Trap System

Application Note 19080203

Keywords
Dual Purge-and-Trap
Eclipse Sample Concentrator
Gas Chromatography
GC
GC/MS
Model 4560 Sample Concentrator
PT Express
Purge and Trap
VOC

Introduction

With gas chromatography (GC) cycle times of 15 minutes or less becoming the standard for VOC applications, the time required for chromatographic separation is no longer the rate-limiting step. Purge-and-trap (P&T) cycle times can be as much as two to three times longer than the fastest GC cycles, potentially causing the GC/MS to sit idle and unproductive, waiting for the sample preparation step to be completed. To make full use of short GC runs and to prevent idle downtime on the mass spectrometer (MS), many laboratories are turning to dual P&T configurations to maximize VOC sample throughput. The OI Analytical PT Express™ (Figure 1) facilitates coupling of two purge-and-trap sample concentrators to one GC. This new configuration eliminates GC/MS idle time and thereby nearly doubles laboratory productivity without the need for additional MS instrumentation.

A diagram of how the PT Express is configured with two sample concentrators and one GC is shown in Figure 2. Two P&T units with their associated autosamplers are “daisy-chained” together, allowing them to alternately deliver samples to one GC or GC/MS. When the first P&T begins desorption and starts the GC analysis of a sample, the second P&T immediately begins purging the second sample. While the first P&T continues desorption, baking of the trap, and rinsing the system (about half of the overall P&T cycle time), the second P&T is already half way into the cycle preparing the second sample. The result is that the second sample is ready for desorption to the GC column as soon as the first sample’s GC run is complete.

Figure 1. OI Analytical PT Express Automated Dual Purge-and-Trap Interface

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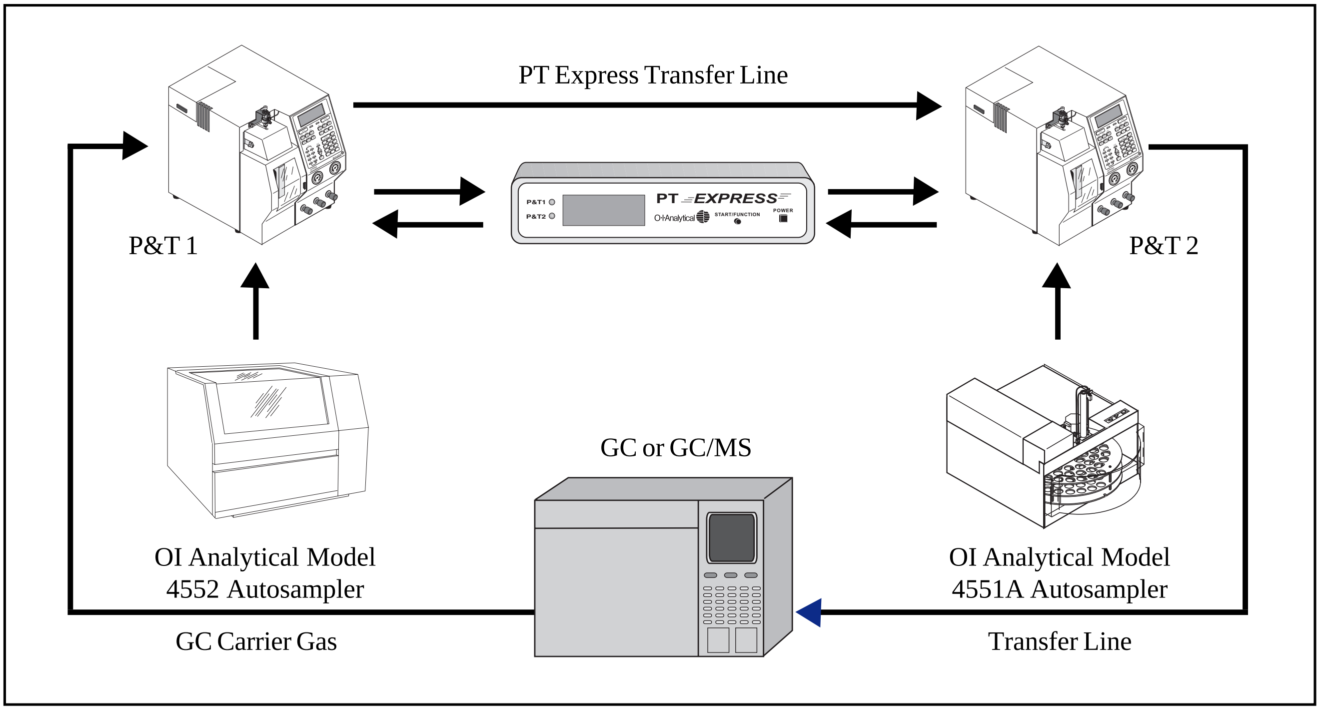


Figure 2. Configuration of the PT Express with Two P&T Sample Concentrators, Two Different Autosampler Types, and One GC. The PT Express Can Accommodate Any Combination of OI Analytical VOC P&T Autosamplers, Including the Model 4551A Liquids Autosampler, the Model 4552 Water/Soil Autosampler, the DPM-16 Discrete Purging Multisampler, and the Model 4506 Automated Multipoint Process Sampler (AMPS).

Recommended Operating Conditions

The 20 m x 0.18 mm I.D. fused silica capillary columns are becoming more common for VOC analyses and are usually necessary to achieve the 12–15 minute GC cycle times. The narrow bore columns also normally have a thinner phase, typically about 1 µm, which can be easily overloaded unless other operating parameters are adjusted appropriately. For example, by increasing the split ratio to 40:1 or higher, less mass is transferred to the column and there is a reduced chance of column overload. The higher split ratios have two additional advantages. They transfer less water to the MS, which can sometimes cause detector performance to deteriorate over time, and they increase P&T desorb flow rates significantly. The faster desorb flow rates transfer the VOC analytes to the column more quickly, minimizing the potential for band broadening and poor chromatography.

The reduced column dimensions and high split ratio also result in sharper, narrower peaks. While some sensitivity may necessarily be sacrificed with unusually high split ratios (e.g., 100:1), in many cases the improvements in peak height and resolution can actually improve the system's overall sensitivity in spite of the reduced mass. The stationary phases on the columns have been tuned by manufacturers to minimize peak co-elutions, which naturally increase as the analysis times are decreased, and the potential for co-elutions is minimized by the higher resolution of the narrower column I.D. Since these columns are used primarily for MS-based analyses and most analytes are quantified using a single ion, the co-elutions do not pose a significant problem. Figures 2 and 3 are examples of the type of excellent chromatography that can be achieved in less than 10 minutes with 20-meter columns from two different manufacturers. Each column was run using the manufacturer's recommended program, and both performed exceptionally well with some minor differences in the elution profiles.

The accompanying insert contains a full list of the optimized instrument settings recommended for use with the PT Express.

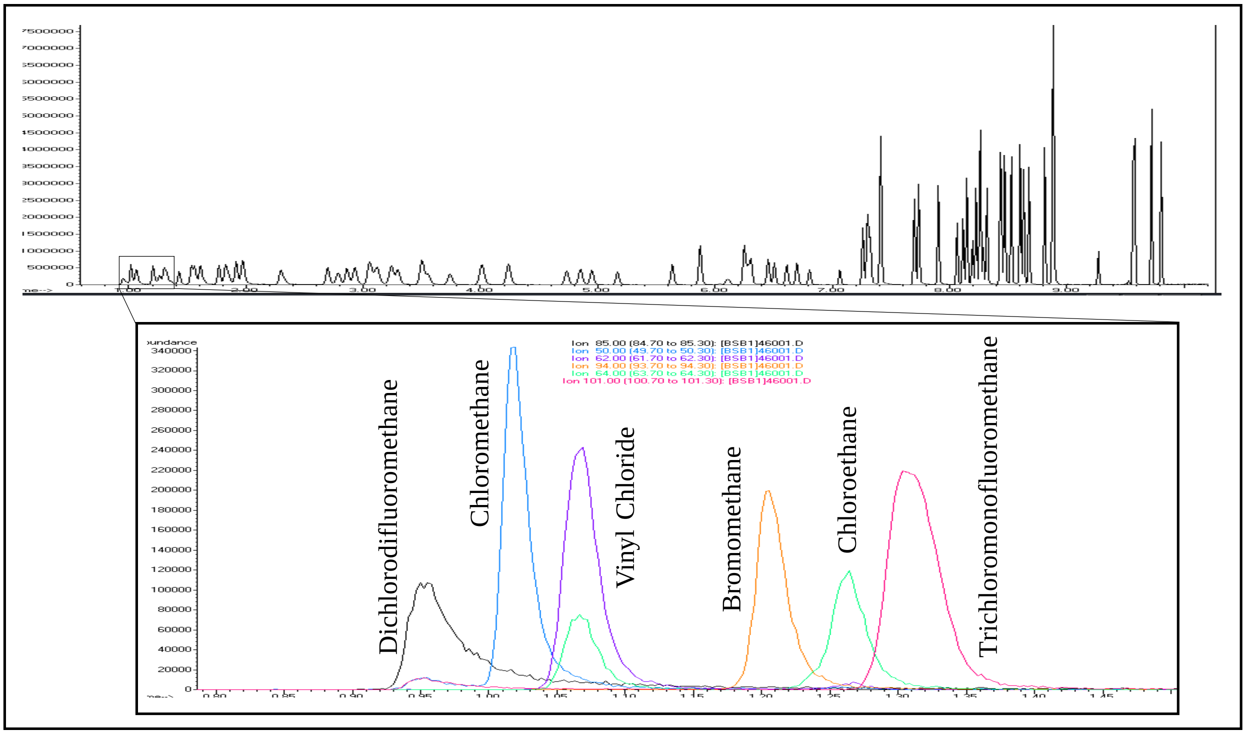


Figure 3. Chromatogram of USEPA Method 524.2 Standard Using the Model 4560 Sample Concentrator and a Restek Rtx-VMS Column. The Column (20 meter x 0.18 mm I.D. x 1.0 µm film) Was Run Using the Column Manufacturer's Recommended Program. The Expanded View Shows the Extracted Ion Profiles for the First Six Gases.

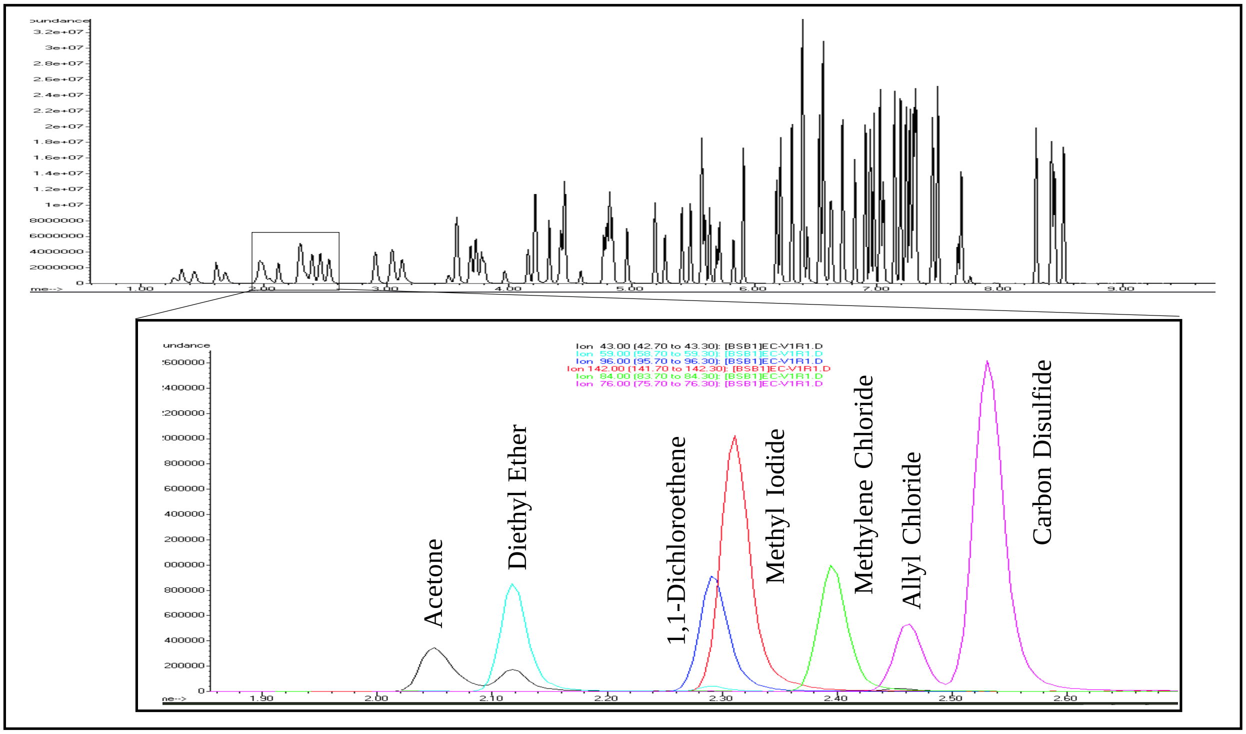


Figure 4. Chromatogram of USEPA Method 524.2 Standard Using the new OI Analytical Model 4660 Eclipse™ Sample Concentrator with an Agilent DB-VRX Column. The Column (20 meter x 0.18 mm I.D. x 1.0 µm film) Was Run Using the Column Manufacturer's Recommended Program. The Expanded View Shows the Extracted Ion Profile for a Group of Early Eluting Compounds that Includes Several of the Polar Analytes in the Method 524.2 List.

Experimental

To demonstrate performance, the PT Express was installed in two commercial laboratories that do high-volume throughput VOC analyses on a regular basis and one industry laboratory. The PT Express in Laboratory 1 was configured with two identical P&T systems, each comprised of a Model 4560 P&T sample concentrator and a Model 4552 Water/Soil autosampler. Laboratory 2 used a PT Express that was configured with two identical Model 4560 P&Ts, but with two different autosamplers; one used the Model 4552 Water/Soil autosampler and the other was mounted on a Model 4551A water-only autosampler. The industry laboratory used one Model 4560 P&T and the new OI Analytical Model 4660 Eclipse P&T, both with the water-only autosampler. The three different test configurations are summarized in Table 1.

Table 1. PT Express Configurations for Three Laboratory Installations

Laboratory	Sample Concentrators	Autosamplers
1	4560/4560	4552/4552
2	4560/4560	4552/4551A
3	4560/Eclipse	4551A/4551A

The PT Express system was put into service in the two environmental laboratories analyzing customer samples, and the instrumentation was run continuously for several months. During that time, all of the quality control (QC) data required by either client protocol or USEPA Method 524.2 or 8260 (BFB-tune checks, blanks, calibrations, CCVs, MS/MSDs, etc.) were acquired for both P&T/autosampler systems. A typical analytical sequence for a 12-hour period is shown in Table 2. The sequence can be streamlined by combining the BFB-tune check and the CCV into one analysis to make room for two additional non-QC samples. Using this sequence, between 21 and 23 analytical runs can be acquired from each P&T over a 12-hour period. This is nearly double the productivity of a single P&T run alone. The only downtime during the test period was for routine instrument maintenance.

Table 2. Typical Analytical Sequence for Two P&Ts Alternately Delivering Samples to One GC/MS Over a 12-hour Tune Period. The Sequence can be Streamlined by Combining the BFB-tune Check and the CCV into a Single Analysis and Results in up to 46 Analytical Runs Within 12 Hours.

	P&T 1	P&T 2
Run 1	BFB-tune check	—
Run 2	—	BFB-tune check
Run 3	CCV	—
Run 4	—	CCV
Run 5	System Blank	—
Run 6	—	System Blank
Run 7 to x (odd)	Samples + QC	—
Run 8 to y (even)	—	Samples + QC
Runs in 12 hours	21 to 23	21 to 23
Total runs in a 12-hour tune period = 42 to 46		

Results and Discussion

Data representative of the dual system performance are illustrated in Tables 3 and 4 and Figures 5 and 6. Table 3 contains the data from a pair of equivalent calibrations from the two systems run in Laboratory 1. The two 8-point calibrations each spanned a concentration range from 0.5–100 ppb, and met the ±20%RSD criteria specified in USEPA Method 524.2 for most analytes. Acetone and 2-butanone were calibrated using the alternative linear technique. All response factors in the Continuing Calibration Verification (CCV) standard also met the method criteria (30% for CCV) continuously for at least two weeks or longer.

Table 3. Equivalent Calibrations Acquired with the PT Express Dual P&T. Calibration Response Factors were Closely Matched on the Two P&Ts. The %RSDs on Both Instruments Fell Well Within the ±20% Criteria Specified in the Method Except for Acetone and 2-Butanone, Which Were Calibrated Using the Alternative Linear Procedure Allowed in the Method.

	Calibration Response Factor		**Calibration %RSD**	
Compound Name	**P&T 1**	**P&T 2**	**P&T 1**	**P&T 2**
Xylenes (total)	0.70181	0.68081	4.7	4.8
Dichlorodifluoromethane	0.19220	0.16376	12.1	10.7
Chloromethane	0.19023	0.19322	5.6	6.0
Vinyl chloride	0.18851	0.16618	2.9	4.3
Bromomethane	0.19630	0.15529	5.1	6.8
Chloroethane	0.16821	0.15961	4.9	3.1
Trichlorofluoromethane	0.46884	0.37686	2.5	3.9
Acetone	(Linear)	(Linear)	$R^2 = 0.99909$	$R^2 = 0.99851$
1,1-Dichloroethylene	0.38826	0.37497	5.4	7.1
Carbon disulfide	0.80342	0.74403	7.0	6.5
Methylene chloride	0.28073	0.29336	12.9	15.8
tert-Butyl methyl ether	0.74562	0.81193	8.9	9.4
trans-1,2-Dichloroethylene	0.26699	0.26643	5.1	4.3
Vinyl acetate	0.43189	0.48580	4.4	4.0
1,1-Dichloroethane	0.47577	0.48811	4.4	2.3
2-Butanone	(Linear)	0.11361	$R^2 = 0.99917$	11.7
cis-1,2-Dichloroethylene	0.29273	0.29836	5.7	4.9
2,2-Dichloropropane	0.41173	0.36750	1.9	4.4
Bromochloromethane	0.12396	0.13342	6.6	2.8
Chloroform	0.48448	0.50307	2.6	3.1
1,1,1-Trichloroethane	0.44933	0.43458	3.2	4.1
1,1-Dichloropropene	0.36944	0.36838	4.1	4.8
Carbon tetrachloride	0.37260	0.35484	5.7	6.1
1,2-Dichloroethane	0.36005	0.38895	2.9	3.3
Benzene	1.07006	1.08643	3.4	3.0
Trichloroethylene	0.27339	0.27719	5.6	3.6
1,2-Dichloropropane	0.25507	0.26126	2.9	5.2
Dibromomethane	0.13665	0.15285	6.5	3.3
Bromodichloromethane	0.35977	0.38124	4.5	3.7
cis-1,3-Dichloropropylene	0.41319	0.43873	3.9	4.6
4-Methyl-2-pentanone	0.25374	0.29588	7.3	3.7
Toluene	0.93599	0.90391	2.5	2.7
trans-1,3-Dichloropropylene	0.50964	0.54101	6.1	5.7
1,1,2-Trichloroethane	0.22750	0.23505	5.8	3.7
2-Hexanone	0.17387	0.20556	6.9	5.3
1,3-Dichloropropane	0.47308	0.51684	4.9	5.4
Tetrachloroethylene	0.30618	0.29063	5.2	5.0
Dibromochloromethane	0.30174	0.32180	8.7	7.6
1,2-Dibromoethane	0.26492	0.28932	5.6	4.6
Chlorobenzene	1.00724	1.00424	3.2	3.1
1,1,1,2-Tetrachloroethane	0.35141	0.35767	3.6	3.0
Ethylbenzene	1.79369	1.73782	2.6	3.7
m,p-Xylenes	0.70798	0.68523	5.0	5.1
o-Xylene	0.68945	0.67196	4.0	5.1
Styrene	1.10401	1.11479	5.3	2.8
Bromoform	0.15985	0.18179	13.2	10.9
Isopropylbenzene	1.85152	1.75365	2.1	2.2
1,1,2,2-Tetrachloroethane	0.31717	0.36328	3.6	3.5
1,2,3-Trichloropropane	0.17281	0.18476	8.3	4.4
Bromobenzene	0.76317	0.75495	6.4	8.4

Table 3. Equivalent Calibrations Acquired with the PT Express Dual P&T—Continued

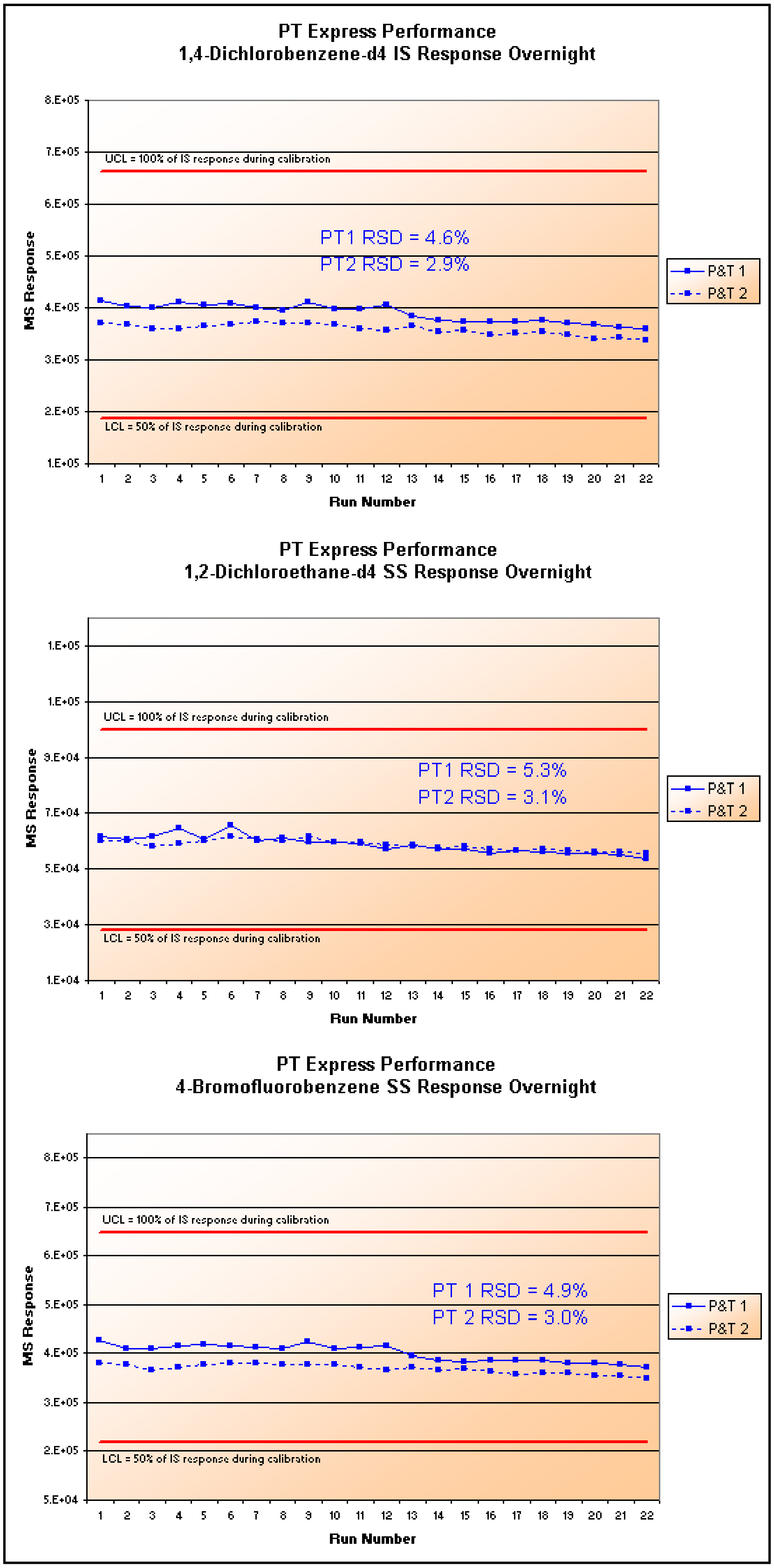
	Calibration Response Factor		**Calibration %RSD**	
Compound Name	**P&T 1**	**P&T 2**	**P&T 1**	**P&T 2**
n-Propylbenzene	3.88442	3.59669	5.8	6.0
2-Chlorotoluene	2.74918	2.56973	6.3	7.1
1,3,5-Trimethylbenzene	2.87768	2.71498	5.9	5.3
4-Chlorotoluene	2.47945	2.35732	6.4	5.4
tert-Butylbenzene	2.81534	2.62245	5.9	6.3
1,2,4-Trimthylbenzene	3.00925	2.80165	5.4	5.9
sec-Butylbenzene	3.81073	3.52464	4.6	6.0
4-Isopropyltoluene	3.07143	2.89017	5.4	6.0
1,3-Dichlorobenzene	1.50342	1.48765	6.0	8.1
1,4-Dichlorobenzene	1.50932	1.49491	5.1	11.2
n-Butylbenzene	2.81119	2.59261	4.6	5.9
1,2-Dichlorobenzene	1.39959	1.40604	4.3	6.2
1,2-Dibromo-3-chloropropane	0.11663	0.13092	7.3	15.7
1,2,4-Trichlorobenzene	1.00753	0.95084	4.9	6.3
Hexachlorobutadiene	0.63360	0.55052	4.7	6.2
Naphthalene	1.97950	2.14371	7.5	5.6
1,2,3-Trichlorobenzene	0.89102	0.84573	3.9	5.8
Dibromofluoromethane	0.23412	0.24064	2.9	2.6
Toluene-d8	1.30516	1.27467	2.2	3.1
Bromofluorobenzene	1.00445	0.97011	6.8	7.8

One concern with the dual system was that twice as much water would be transferred to the MS within a 12-hour tune period due to the increase in sample throughput, and the performance (i.e., sensitivity) of the MS would suffer. To monitor for this potential loss in sensitivity, many USEPA methods require that laboratories keep QC charts of internal standard (IS) responses for all samples. Figure 5 displays quality control charts for three Method 8260 internal standards run on a dual instrument system during a 12-hour tune period. These quality control charts demonstrate the excellent stability and repeatability that can be achieved with the system despite of the additional water from increased sample throughput. All responses were well within the QC criteria specified by the method, as indicated by the upper and lower control limits (100% and 50% of IS response during calibration, respectively), and had repeatability of 5.3% or better.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD) recoveries, such as those illustrated in Figure 6, are another common QC measurement used to check overall system performance. For this example, the MS/MSD standards were spiked into representative real-world matrices at a concentration of 5.0 µg/L and run on both P&T 1 and P&T 2. Concentrations were calculated based on the initial calibration for the individual instruments and percent recoveries were based on the original spike amount. All recoveries for the MS/MSD fell within the upper and lower control limits specified by the laboratory's in-house QC protocol and ranged from 88–112%.

Table 4 shows the %RSDs for all 86 compounds listed in USEPA Method 524.2 when run on the configuration shown for Laboratory 3. Ten replicate analyses of a 5-ppb standard (Method 524.2) were run on each P&T of the dual system (one Model 4560 and one Eclipse P&T) for a total of 20 analytical runs. The percent relative standard deviation (%RSD) was calculated for each target compound using the area of the quantitation ion. The asterisk in the last column indicates analytes that had equal or better performance on the Eclipse P&T.

The PT Express is fully compliant with all USEPA methodology. Soil samples can be run by USEPA Method 5035 in soil mode. Water samples do not have to be run as if they were soils, but can be run by USEPA Method 5030.



PT Express Performance
1,4-Dichlorobenzene-d4 IS Response Overnight

PT Express Performance
1,2-Dichloroethane-d4 SS Response Overnight

PT Express Performance
4-Bromofluorobenzene SS Response Overnight

Figure 5. Data from Laboratory 2 Showing the Stable Response of Method 8260 Internal Standards on Two Different P&T/Autosampler Combinations Over a 12-hour Period. The Upper and Lower Lines Indicate the Upper and Lower Control Limits.

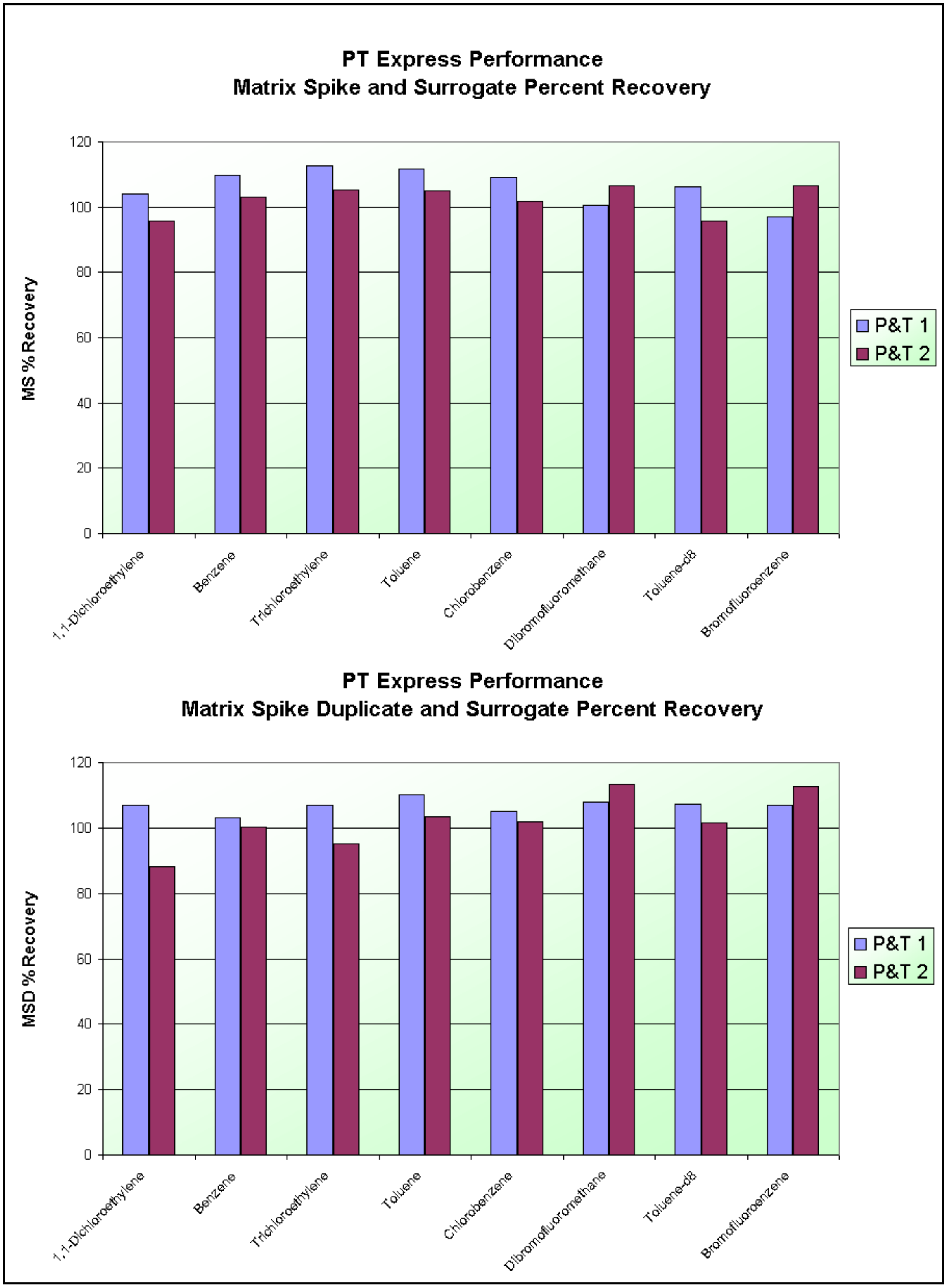


Figure 6. Data from Laboratory 1 Showing the Matrix Spike and Matrix Spike Duplicate (MS/MSD) Recoveries When Run on the PT Express. The Standards were Spiked into Representative Real-world Matrices at a Concentration of 5.0 µg/L.

Table 4. Laboratory 3 Data Listing %RSDs for All 86 USEPA Method 524.2 Compounds Run on the PT Express. Data Represent 10 Replicate Runs on Each System (20 Total Runs) with a 5-ppb Standard. The Asterisks in the Last Column Indicate Compounds with Lower %RSDs on the OI Analytical Eclipse Sample Concentrator.

Compound Number	**Compound Name**	**4560 %RSD**	**Eclipse %RSD**	
	Internal Standard			
33	Fluorobenzene	8.1	8.3	
	Target Compounds			
1	Dichlorodifluoromethane	3.3	4.7	
2	Chloromethane	11.7	8.7	*
3	Vinyl chloride	8.5	6.2	*
4	Bromomethane	8.4	4.3	*
5	Chloroethane	7.9	3.0	*
6	Trichloromonofluoromethane	3.4	4.1	
7	Diethyl ether	8.5	6.3	*
8	1,1-Dichloroethene	8.7	5.1	*
9	Carbon disulfide	9.1	9.0	*
10	Methyl iodide	8.2	5.2	*
11	Allyl chloride	9.4	7.5	*
12	Methylene chloride	8.2	5.7	*
13	Acetone	13.9	4.4	*
14	*trans*-1,2-Dichloroethene	7.7	5.4	*
15	Methyl-t-butyl ether	6.9	4.8	*
16	1,1-Dichloroethane	5.2	4.5	*
17	Acrylonitrile	7.3	5.3	*
18	*cis*-1,2-Dichloroethene	7.1	6.4	*
19	2,2-Dichloropropane	3.9	3.9	*
20	Bromochloromethane	7.7	7.7	*
21	Chloroform	4.8	3.6	*
22	Methyl acrylate	7.5	5.8	*
23	Carbon tetrachloride	6.6	4.7	*
24	Tetrahydrofuran	8.9	7.6	*
25	1,1,1-Trichloroethane	4.1	3.6	*
26	2-Butanone	13.0	9.1	*
27	1,1-Dichloropropene	5.7	5.5	*
28	1-Chlorobutane	6.3	6.1	*
29	Benzene	6.2	6.3	
30	Propionitrile	9.4	7.5	*
31	Methacrylonitrile	7.6	6.6	*
32	1,2-Dichloroethane	3.5	4.2	
34	Trichloroethene	7.8	8.1	
35	Dibromomethane	6.6	7.1	
36	1,2-Dichloropropane	7.6	7.7	
37	Bromodichloromethane	6.2	5.2	*
38	Methylmethacrylate	8.1	8.0	*
39	*cis*-1,3-Dichloropropene	7.2	6.6	*
40	Toluene	8.2	8.4	
41	Chloroacetonitrile	17.0	8.4	*
42	2-Nitropropane	4.8	4.3	*
43	1,1-Dichloropropanone	6.0	5.7	*
44	Tetrachloroethene	11.5	12.2	
45	4-Methyl-2-pentanone	6.7	7.4	
46	*trans*-1,3-Dichloropropene	6.1	5.9	*
47	1,1,2-Trichloroethane	6.7	7.3	
48	Ethyl methacrylate	7.2	7.3	
49	Dibromochloromethane	7.0	5.6	*
50	1,3-Dichloropropane	6.2	7.3	
51	1,2-Dibromoethane	7.0	7.6	
52	2-Hexanone	6.0	7.2	
53	Chlorobenzene	8.8	9.2	
54	Ethylbenzene	8.8	9.3	
55	1,1,1,2-Tetrachloroethane	7.4	6.8	*
56	*m/p*-Xylene	9.4	9.7	
57	*o*-Xylene	9.4	9.4	*
58	Styrene	8.8	9.3	
59	Bromoform	8.1	6.8	*
60	Isopropylbenzene	8.7	9.0	
61	4-Bromofluorobenzene (SS)	6.2	6.3	
62	Bromobenzene	10.3	10.4	
63	*n*-Propylbenzene	8.6	9.3	
64	1,1,2,2-Tetrachloroethane	7.2	7.3	
65	2-Chlorotoluene	8.3	8.6	
66	1,2,3-Trichloropropane	5.9	6.2	
67	1,3,5-Trimethylbenzene	8.7	9.3	
68	*trans*-1,4-dichloro-2-buten	7.9	9.5	
69	4-Chlorotoluene	8.4	8.6	
70	*tert*-Butylbenzene	8.5	9.6	
71	Pentachloroethane	8.9	8.4	*
72	1,2,4-Trimethylbenzene	8.8	9.4	
73	*sec*-Butylbenzene	8.3	9.5	
74	4-Isopropyltoluene	8.8	10.0	
75	1,3-Dichlorobenzene	10.4	10.6	
76	1,4-Dichlorobenzene	10.8	11.1	
77	*n*-Butylbenzene	8.6	9.3	
78	Hexachloroethane	8.7	8.7	*
79	1,2-Dichlorobezene-d4 (SS)	6.9	7.6	
80	1,2-Dichlorobenzene	11.1	11.0	*
81	1,2-Dibromo-3-chloropropan	6.4	6.2	*
82	Nitrobenzene	8.1	10.6	
83	Hexachlorobutadiene	8.7	8.8	
84	1,2,4-Trichlorobenzene	12.9	12.7	*
85	Naphthalene	9.4	8.1	*
86	1,2,3-Trichlorobenzene	12.8	12.7	*
	Average %RSD	**8.0**	**7.4**	*****

Sequence Integrity

Handling of the data from two blended sample sets into one continuous GC sequence is of critical importance. Because the two P&T systems are usually calibrated separately, care must be taken to ensure that each sample is associated with the correct set of QC data. Additionally, if one of the P&T/autosamplers fails (e.g., if the autosampler drops a vial), the integrity of the sample naming protocol must be maintained to comply with QC and audit requirements. The PT Express uses a microprocessor-based control unit that synchronizes the P&T and GC timing, preventing the defined GC sample sequences from getting out of order or two samples from being desorbed simultaneously. If one of the P&T/autosamplers fails, the PT Express inserts a blank run on the GC as a "placeholder" so that the integrity of the sample names remains intact. Sample sequences that become disordered can cost the laboratory in terms of labor and, in many cases, may not meet GLP or QC protocols. This unique design ensures that analytical runs cannot get scrambled or out of sequence in the GC's data system.

Cost/Benefit Analysis

The most costly part of any VOC analytical instrumentation is the GC/MS. Doubling the output of a single GC/MS with only minimal capital expense for a second P&T/autosampler can produce significant increases in revenue. Table 5 is an example of a cost/benefit analysis comparing the dual PT Express system to a single P&T system. With a capital outlay of only $30,000, less than one-third the cost of a complete VOC system, a commercial laboratory can nearly double productivity, resulting in as much as $1 million in additional revenue in just the first year. (Increases in revenue shown here are based on assumed average industry values for cost per sample and laboratory capacity and should be considered an example of the type of benefit that can be achieved. Individual experiences may vary.)

Table 5. Example of a Cost/Benefit Analysis that Can Be Made for Systems with the PT Express Compared to Single P&T Systems. Individual Experiences May Vary Depending on Cost Per Sample and Laboratory Capacity.

Parameter	**Single P&T + GC/MS**	**Dual P&T w/PT Express + GC/MS**
System Price	$100,000	$130,000
Revenue/Sample	$100	$100
Performance Criteria		
Time to Prep GC (min)	60	60
GC Cycle Time (min)	14	14
P&T Cycle Time (min)	27.5	NA
Runs/Day	50	99
Daily Sample Overview		
QA/QC Runs/Day	8	16
Revenue Samples/Day	42	83
Revenue/Day	$4,250	$8,300
Annual Financial Performance		
Working Days/Year	225	225
Revenue Samples/Year	9,450	18,675
Gross Revenue/Year	$945,000	$1,867,500
Increase in Revenue		**$922,500**

Conclusions

Today's production laboratories invest heavily in method development to reduce overall sample analysis cycle times and to increase instrument capacity. To make full use of their capital investment and to prevent idle downtime on the MS, many laboratories are turning to the use of dual purge-and-trap configurations such as the OI Analytical PT Express. In the three laboratory studies reported here, the dual system nearly doubled production, and, thus, also doubled laboratory revenue by analyzing up to twice as many samples as a single P&T system. All of this is possible without the need for additional expensive MS instrumentation.

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Parameter	Recommended Setting or Configuration	Comments
Trap	#10 trap (Tenax®/silica gel/carbon molecular sieve)	• The #10 trap provides the best all-around performance for VOC analysis, especially with MS systems. It is designed to work with the WMF to produce the best water management performance and optimum chromatography. • The #11 VOCARB trap can also be used but some changes to the operating conditions may be necessary.
Trap Temperatures	Recommended temperatures for a #10 trap: 20°C during purge 190°C during desorb 210°C during bake	• Setting the trap temperature to 20°C during purge will allow it to cool to ambient temperature. • Laboratory temperatures above ~35°C will hinder optimum cooling and can cause loss of light gases during purge.
Purge Time	11 min	• Specified in most USEPA VOC methods.
Purge Flow Rate	35–40 mL/min	• Most laboratories use helium as the purge gas.
Desorb Flow Rate	Minimum of 40 mL/min (when using a 20-m column)	• High flow rates minimize lateral diffusion of the analyte band during transfer to the GC column. The high desorb flow rate and rapid transfer to the GC column improve peak shape and height.
Desorb Time	0.5–4 min	• Desorption of 90–100% of all analytes can be achieved with a desorb time of 0.5–1 minute. • Desorb times of greater than two minutes do not improve recovery and can lead to excessive water transferred to the GC column.
Bake Time	2–5 min or more	• Baking the trap for 2–5 minutes between analyses is adequate for most applications. However, the bake time can be adjusted (longer or shorter) to balance P&T and GC cycle times. • For the Massachusetts VPH method an increase in bake temperature or time may be necessary.
6-Port Valve Temperature	110°–150°C	• Lower temperatures can cause water condensation. • Higher temperatures can degrade thermally labile compounds.
Transfer Line Temperature	110°–150°C	• Lower temperatures can cause water condensation. • Higher temperatures can be used, but watch for thermal degradation.

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Sample Temperature	40°–45°C	• Overall optimum performance for most analytes is achieved when the sample is heated to 40°–45°C. • At lower temperatures (e.g., ambient), polar, water-soluble compounds may have lower purge efficiencies. • At temperatures above 45°C, additional water is transferred to the trap and may cause performance problems. • Most current USEPA methods do not call for heating of the sample, but anticipated changes in some methods are expected to include this option (as of October 2002).
Sample Size	Water: 5 mL Soil: 5 g	• For high concentration discharge or ground water samples, a 5-mL sample size meets USEPA method guidelines and minimizes the potential for contamination from most samples. • For very high concentration or contaminated samples, dilution may be necessary. • For clean drinking water samples, a 25-mL sample size can be used to lower detection limits without fear of contamination. However, because of purge efficiency differences, not all analytes will have a five-fold increase in sensitivity. • Be sure to match the sparge vessel to the sample size. Analyzing a 5-mL sample in a 25-mL sparger reduces water carryover to the trap, but this procedure does not follow USEPA protocol specified in most methods and may result in slightly lower recoveries. • When using the Model 4552 Water/Soil Autosampler, water sample size can be varied from 1–25 mL and dilutions can be done automatically.
Water Management Fitting Temperatures	110°C during purge 0°C during desorb 240°C during bake	• Setting the WMF temperature to 0°C during desorb cools it to approximately ambient temperature and provides the best performance. • For analysis of some very polar, water-soluble compounds (e.g., California oxygenates method or USEPA Method 1666), using a slightly higher temperature during desorb (e.g., 50°–70°C) may improve recovery performance.

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Sparge Mount Temperature (Sample Inlet)	40°–45°C	• Cooler temperatures can cause water condensation and loss of brominated compounds and naphthalene. • Higher temperatures can transfer excessive water to the trap or cause degradation of some thermally labile compounds.
Prepurge	OFF	• This setting is normally used only with the Air-Tube Desorber Accessory to reduce moisture transferred to the trap.
Preheat	OFF	• This setting is not necessary when using the Infra-Sparge™ Sample Heater.
Dry Purge	OFF	• This setting is not necessary with a #10 trap. • For the VOCARB trap, a dry-purge of 1–6 minutes may improve performance but will extend the overall cycle time.
Desorb Preheat	Optional ON, 150°–175°C	• This function heats the trap to a desired temperature set point prior to rotation of the 6-port valve (i.e., no carrier gas flow is in line with the trap). • Desorb preheat is especially useful for analytes, such as trichloromonofluoromethane, that may be prone to peak splitting. • Desorb preheat temperatures that are too high can introduce chromatography problems such as tailing to other peaks.
Valve Manifold Temperature (2nd, F5)	50°C	• Do not use other temperatures.
Transfer Line/GC Inlet Interface	As short as possible; Insulated	• Make the connection as short as possible, ~1.5 inches, and insulate it to minimize water condensation. • **DO NOT** leave a 2–3 inch, noninsulated connection as recommended by other P&T manufacturers. This type of connection creates a cold spot and is intended to compensate for the lack of adequate water management. • Use inlet temperatures of 150°–200°C. • Capping off the septum purge vent may help reduce or minimize losses of the light components to the septum purge line. Individual results may vary.

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Split Ratio	40:1 or higher, up to 300:1 when using a 20-m capillary GC column	• The high split ratio has several advantages, such as increasing desorption flow rate, which improves peak shape and height. • Desorption time can be shortened. • Water transfer to the column is minimized. • Increases in peak height make up for any loss of mass to the column and potential increases in MDLs. • A 40:1 split works for a calibration range of ~0.5–100 ppb (5-mL sample size). • A 300:1 split works for a calibration range of ~10–200 ppb (5-mL sample size). • For drinking water analysis, use a 25-mL sample size and a 40:1 split ratio for the lowest detection limits.
Inlet Liner	1–2 mm I.D. straight tube liner	• Use the smallest I.D. liner available. • Minimizes lateral diffusion of the sample band and maintains peak shape during transfer to the column.
GC Column Dimensions	20-m length x 0.18-mm I.D. x 1-µm film thickness	• Most major column manufacturers (e.g., Agilent, Restek) have columns in these dimensions with phases specifically designed for VOC analysis. There are some specific differences in the phases. Contact the column manufacturer for details. • Dimensions are suitable for MS analysis only. Conventional detectors (PID, ELCD, etc.) have different requirements. • Results in the shortest overall cycle time when running dual P&T systems.

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