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Performance of the SOLATek 72 Multi-Matrix Vial Autosampler for the Analysis of VOCs

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OBJECTIVE

In this paper, the pertinent features of the SOLATek 72™ Multi-Matrix vial autosampler will be described. Data will be evaluated for linearity, precision, accuracy and carryover.

INTRODUCTION

In today's laboratories, increased efficiency and productivity are of extreme importance. Equally important is the ability to automate analyses without sacrificing sample integrity or data quality. The SOLATek 72 Multi-Matrix Vial Autosampler has been developed to fully automate purge & trap analysis of water, wastewater, and soil samples in accordance with current USEPA methods for volatile analysis. SOLATek 72 has a unique liquid sample pathway made of large bore tubing and large valve orifices that eliminate clogging of sample lines and valves often encountered when running particulated samples. The inert solid pathway is coated entirely with Silcosteel®. Variable mixing modes and speeds allow the analyst to customize their solids methods for different matrices. All sampling is followed by a complete hot water rinse of the systems pathway virtually eliminating carryover. SOLATek 72 can accommodate up to seventy-two 40 mL VOA vials, providing the highest sample capacity of any autosampler for VOC analysis.

The purpose of this study is to evaluate the performance of the SOLATek 72 in five key areas: minimum detection limit study, standard injection system, calibration, carryover, and sample delivery precision. Results from the performance evaluation will be discussed, and an overview of the system's features and reliability will be presented.

SOLATek 72: Water Mode	
Rinse Water	90°C
Sample Cup	30°C
Needle	30°C
Transfer Line	125°C
Sample Sweep Time	125°C
Needle Sweep Time	0.50 min.
Needle Rinse Volume	5 mL
Bake Rinse Volume	0.75 min.
Bake Sweep Time	0.50 min.
Bake Drain Time	0.50 min.
Bake Rinses	0

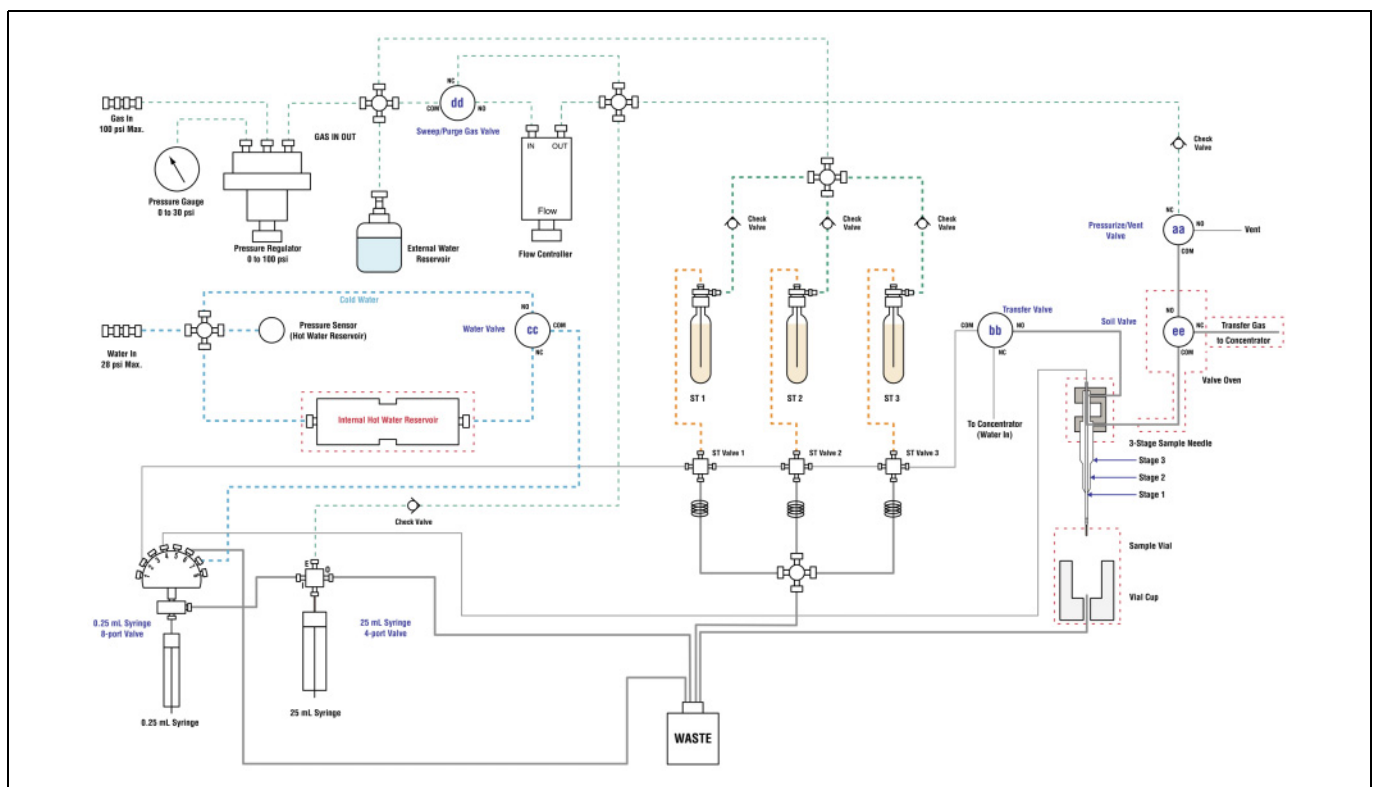
SOLATek 72: Soil Mode	
Rinse Water	90°C
Sample Cup	30°C
Needle	60°C
Transfer Line	125°C
Soil Valve	125°C
Sample Sweep Time	0.50 min.
Needle Rinse Volume	7 mL
Needle Sweep Time	0.75 min.
Purge Time	11 min.
Purge Stir	ON
Purge Mix Mode	Normal
Purge Stir Speed	5

Tekmar-Dohrmann Sample Concentrator	
Valve Temperature	150°C
Transfer Line Temperature	150°C
Mount Temperature	40°C
MCS Line Temperature	40°C
MCS Bake Temperature	320°C
Purge Ready Temperature	35°C
Trap	Vocarb 3000
Purge Time	11 min.
Dry Purge Time	2 min.
Desorb Preheat	245°C
Desorb Time	4 min.
Desorb Temperature	250°C
Bake Time	10 min.
Bake Temperature	280°C

Agilent 6890 GC/5973 MSD	
Injector	110°C; Split 20:1
Column	Restek Corp., Rtx-VMS, 60m x 0.25mm x 1.4 µm
Temperature Program	45°C (hold 10 min). Increase to 190°C @ 12°C/min., hold at 190°C for 2 min. Increase to 225°C @ 6°C/min, hold one min.
Carrier	Helium, at 1.2 mL/min.
MS Source Temperature	230°C
MS Quad Temperature	150°C
Electron Multiplier	1720 V
Mass Range Scanned	35-260 amu

The sample flow path in the SOLATek 72 is diagrammed in Figure 1.

Figure 1. SOLATek 72 Multi-Matrix Vial Autosampler



MINIMUM DETECTION LIMIT

A minimum detection limit (MDL) study was performed to evaluate the SOLATEk 72's accuracy, precision and cleanliness for low level analyses. The water MDL was established by running seven blank water replicates spiked manually with 0.5 ppb of the target analytes. The soil MDL was established by running seven blank water replicates spiked at 2.0 ppb and analyzed by a SOLATEk soil method. The following data represents MDLs at a 99% confidence level.

Table 1. Water MDLs

Compound	MDL (ppb)	Compound	MDL (ppb)	Compound	MDL (ppb)
dichlorodifluoromethane	0.022	trichloroethene	0.035	bromobenzene	0.017
chloromethane	0.092	1,2-dichloropropane	0.024	1,1,2,2-tetrachloroethane	0.043
vinyl chloride	0.024	dibromomethane	0.017	1,2,3-trichloropropane	0.051
bromomethane	0.025	bromodichloromethane	0.017	n-propyl benzene	0.017
chloroethane	0.036	cis-1,3-dichloropropene	0.017	2-chlorotoluene	0.030
trichlorofluoromethane	0.022	toluene	0.012	4-chlorotoluene	0.034
1,1-dichloroethene	0.022	trans-1,3-dichloropropene	0.025	1,3,5-trimethylbenzene	0.012
methylene chloride	0.057	1,1,2-trichloroethane	0.062	tert-butylbenzene	0.018
trans-1,2-dichloroethene	0.022	tetrachloroethene	0.035	1,2,4-trimethylbenzene	0.024
1,1-dichloroethane	0.015	1,3-dichloropropane	0.028	sec-butylbenzene	0.025
2,2-dichloropropane	0.022	dibromochloromethane	0.022	1,3-dichlorobenzene	0.031
cis-1,2-dichloroethene	0.017	1,2-dibromomethane	0.025	4-isopropyltoluene	0.034
bromochloromethane	0.025	chlorobenzene	0.012	1,4-dichlorobenzene	0.028
chloroform	0.025	1,1,1,2-tetrachloroethane	0.018	1,2-dichlorobenzene	0.031
1,1,1-trichloroethane	0.024	ethyl benzene	0.022	n-butylbenzene	0.034
1,1-dichloropropene	0.015	m,p-xylene	0.034	1,2-dibromo-3-chloropropane	0.034
carbon tetrachloride	0.012	o-xylene	0.034	1,2,4-trichlorobenzene	0.056
benzene	0.031	styrene	0.028	hexachlorobutadiene	0.062
1,2-dichloroethane	0.028	bromoform	0.018	naphthalene	0.030
		isopropyl benzene	0.015	1,2,3-trichlorobenzene	0.047

Table 2. Soil MDLs

Compound	MDL (ppb)	Compound	MDL (ppb)	Compound	MDL (ppb)
dichlorodifluoromethane	0.223	trichloroethene	0.252	bromobenzene	0.196
chloromethane	0.307	1,2-dichloropropane	0.182	1,1,2,2-tetrachloroethane	0.227
vinyl chloride	0.220	dibromomethane	0.190	1,2,3-trichloropropane	0.293
bromomethane	0.110	bromodichloromethane	0.277	n-propyl benzene	0.221
chloroethane	0.435	cis-1,3-dichloropropene	0.243	2-chlorotoluene	0.276
trichlorofluoromethane	0.128	toluene	0.246	4-chlorotoluene	0.300
1,1-dichloroethene	0.262	trans-1,3-dichloropropene	0.232	1,3,5-trimethylbenzene	0.103
methylene chloride	0.252	1,1,2-trichloroethane	0.125	tert-butylbenzene	0.195
trans-1,2-dichloroethene	0.301	tetrachloroethene	0.192	1,2,4-trimethylbenzene	0.262
1,1-dichloroethane	0.314	1,3-dichloropropane	0.288	sec-butylbenzene	0.252
2,2-dichloropropane	0.396	dibromochloromethane	0.209	1,3-dichlorobenzene	0.110
cis-1,2-dichloroethene	0.164	1,2-dibromomethane	0.220	4-isopropyltoluene	0.237
bromochloromethane	0.186	chlorobenzene	0.012	1,4-dichlorobenzene	0.191
chloroform	0.093	1,1,1,2-tetrachloroethane	0.178	1,2-dichlorobenzene	0.218
1,1,1-trichloroethane	0.160	ethyl benzene	0.134	n-butylbenzene	0.172
1,1-dichloropropene	0.287	m,p-xylene	0.210	1,2-dibromo-3-chloropropane	0.685
carbon tetrachloride	0.093	o-xylene	0.147	1,2,4-trichlorobenzene	0.188
benzene	0.160	styrene	0.233	hexachlorobutadiene	0.114
1,2-dichloroethane	0.291	bromoform	0.123	naphthalene	0.121
		isopropyl benzene	0.175	1,2,3-trichlorobenzene	0.213

The MDLs achieved for most compounds were at or below 0.10 ppb in water mode and 0.70 ppb in soil mode.

STANDARD INJECTION SYSTEM

The SOLATek 72 comes equipped with up to three standard injection systems. Each system is capable of delivering aliquots of 5, 10, 15, 20, or 25 μL . The volumes delivered are easily changed in the system software. Accuracy and precision were evaluated by performing 5 point BTEX calibrations using the injection system. All analyses for this evaluation were performed on a Varian 3400 GC with a FID.

Table 3. Standard Injection System Precision

	benzene	toluene	ethylben	chloroben	mp-xylene	o-xylene	1,3-dichlor	1,4-dichlor	1,2-dichlor
5 μL	45285	42175	27092	42299	79429	39794	21603	21900	21172
	47265	43693	26233	43096	81966	41492	22397	22746	21828
	45462	42101	28560	41333	78693	40113	21634	21801	21098
	46075	42263	28492	41452	78549	39932	21499	21918	21112
AVG	46022	42558	27594	42045	79659	40333	21783	22091	21303
%RSD	1.9	1.8	4.1	2.0	2.0	1.9	1.9	2.0	1.7
10 μL	88356	78976	52316	77111	146185	73916	39058	39558	38387
	89115	79273	52116	77022	145924	73963	38844	39232	38250
	87989	79340	52755	77561	147028	74454	39264	39682	38656
	88653	78934	51605	76894	145326	73502	38790	39174	37932
	89191	79197	52475	76958	145621	73986	39108	39624	38527
AVG	88661	79144	52253	77109	146017	73964	39013	39454	38350
%RSD	0.6	0.2	0.8	0.3	0.4	0.5	0.5	0.6	0.7
15 μL	128650	113503	74872	110995	211491	106202	55844	56651	54977
	129056	113752	75462	111052	211388	106174	55885	56513	55013
	129870	115150	75878	112522	214059	107517	56781	57491	55796
	130284	115936	75842	112893	213025	107528	56526	57170	55586
	129793	115300	75961	112565	214592	107568	56610	57230	55796
AVG	129531	114728	75603	112005	212911	106998	56329	57011	55434
%RSD	0.5	0.9	0.6	0.8	0.7	0.7	0.8	0.7	0.7
20 μL	171770	150452	99110	147812	281009	140892	74115	74986	72562
	170747	150690	98911	146058	278370	139537	73610	74509	72744
	170973	151649	99377	147680	281661	140945	73949	74857	72675
	169785	150006	98492	146643	279218	139313	73507	74360	72050
	175053	155567	102526	152137	292084	145703	76356	77500	75097
AVG	171666	151673	99683	148066	282468	141278	74307	75242	73026
%RSD	1.2	1.5	1.6	1.6	2.0	1.8	1.6	1.7	1.6
25 μL	216566	190440	124777	187093	357779	177674	93010	94211	91304
	207150	183316	119174	178602	339121	169658	89227	90443	87723
	211162	186051	121701	181534	345227	172542	90697	91700	88943
	211758	188581	123220	184859	351037	175617	92525	94033	91094
	210139	184581	121607	180325	343884	172116	90222	91622	88762
AVG	211355	186594	122096	182483	347410	173521	91136	92402	89565
%RSD	1.6	1.6	1.7	1.9	2.1	1.8	1.7	1.8	1.7
5	52215	46932	30046	44289	82595	41958	21187	21559	21435
10	94913	83309	54441	80122	150831	76402	39681	40328	39532
15	137003	120209	78403	115137	217224	109782	57329	58226	57029
20	179512	156894	101590	150891	285688	143557	74980	76081	74246
25	222365	196779	128400	190427	362993	182432	95452	96869	94748
CORR	1.0000	0.9998	0.9997	0.9997	0.9996	0.9996	0.9996	0.9996	0.9995

CALIBRATION

To evaluate the system's linearity, calibrations were performed using both the water and soil pathways. Listed below are the percent relative standard deviations obtained by analyzing five point calibrations. The concentration range is 1.0ppb - 200ppb.

Table 4. Water Calibration

Compound	%RSD	Compound	%RSD	Compound	%RSD
dichlorodifluoromethane	4.46	trichloroethene	2.51	bromobenzene	1.04
chloromethane	1.96	1,2-dichloropropane	1.52	1,1,2,2-tetrachloroethane	2.39
vinyl chloride	2.05	dibromomethane	2.15	1,2,3-trichloropropane	3.71
bromomethane	12.17	bromodichloromethane	4.45	n-propyl benzene	7.26
chloroethane	4.14	cis-1,3-dichloropropene	5.01	2-chlorotoluene	6.41
trichlorofluoromethane	2.73	toluene	9.22	4-chlorotoluene	6.74
1,1-dichloroethene	2.02	trans-1,3-dichloropropene	5.34	1,3,5-trimethylbenzene	6.43
methylene chloride	18.02	1,1,2-trichloroethane	1.51	tert-butylbenzene	4.59
trans-1,2-dichloroethene	2.21	tetrachloroethene	9.41	1,2,4-trimethylbenzene	7.74
1,1-dichloroethane	0.65	1,3-dichloropropane	1.56	sec-butylbenzene	6.86
2,2-dichloropropane	1.14	dibromochloromethane	6.63	1,3-dichlorobenzene	15.16
cis-1,2-dichloroethene	0.95	1,2-dibromomethane	1.44	4-isopropyltoluene	14.58
bromochloromethane	1.87	chlorobenzene	6.55	1,4-dichlorobenzene	9.33
chloroform	1.09	1,1,1,2-tetrachloroethane	2.86	1,2-dichlorobenzene	8.15
1,1,1-trichloroethane	1.77	ethyl benzene	6.58	n-butylbenzene	11.79
1,1-dichloropropene	3.51	m,p-xylene	12.47	1,2-dibromo-3-chloropropane	9.68
carbon tetrachloride	3.35	o-xylene	6.23	1,2,4-trichlorobenzene	12.21
benzene	2.14	styrene	2.43	hexachlorobutadiene	11.29
1,2-dichloroethane	1.77	bromoform	10.45	naphthalene	10.57
		isopropyl benzene	4.53	1,2,3-trichlorobenzene	8.77

Table 5. Soil Calibration

Compound	%RSD	Compound	%RSD	Compound	%RSD
dichlorodifluoromethane	14.36	trichloroethene	2.07	bromobenzene	2.79
chloromethane	13.26	1,2-dichloropropane	3.41	1,1,2,2-tetrachloroethane	4.24
vinyl chloride	6.06	dibromomethane	3.85	1,2,3-trichloropropane	2.06
bromomethane	3.16	bromodichloromethane	5.94	n-propyl benzene	1.93
chloroethane	11.06	cis-1,3-dichloropropene	6.44	2-chlorotoluene	2.53
trichlorofluoromethane	12.83	toluene	3.36	4-chlorotoluene	2.32
1,1-dichloroethene	6.98	trans-1,3-dichloropropene	8.76	1,3,5-trimethylbenzene	2.37
methylene chloride	6.73	1,1,2-trichloroethane	2.11	tert-butylbenzene	2.11
trans-1,2-dichloroethene	2.10	tetrachloroethene	0.98	1,2,4-trimethylbenzene	2.62
1,1-dichloroethane	1.81	1,3-dichloropropane	2.36	sec-butylbenzene	1.77
2,2-dichloropropane	2.14	dibromochloromethane	8.51	1,3-dichlorobenzene	1.45
cis-1,2-dichloroethene	1.95	1,2-dibromomethane	3.45	4-isopropyltoluene	2.69
bromochloromethane	1.86	chlorobenzene	2.27	1,4-dichlorobenzene	2.66
chloroform	1.82	1,1,1,2-tetrachloroethane	5.14	1,2-dichlorobenzene	3.07
1,1,1-trichloroethane	1.32	ethyl benzene	2.28	n-butylbenzene	4.26
1,1-dichloropropene	3.04	m,p-xylene	5.91	1,2-dibromo-3-chloropropane	16.09
carbon tetrachloride	0.95	o-xylene	2.62	1,2,4-trichlorobenzene	8.90
benzene	0.46	styrene	3.78	hexachlorobutadiene	3.78
1,2-dichloroethane	2.92	bromoform	13.25	naphthalene	11.21
		isopropyl benzene	2.11	1,2,3-trichlorobenzene	8.88

The overall %RSD for the water calibration data is 5.6% and the overall %RSD for the soil calibration data is 4.6%. Thus, good linearity is maintained over a wide concentration range.

CARRYOVER

To evaluate the system's carryover, 200 ppb standards were analyzed and followed by blank deionized water runs. Carryover was calculated based on raw area counts. The percent carryover for each compound is listed in Tables 6 and 7 for both waters and soils. The same evaluation was performed directly on the 3100 Sample Concentrator and it was found that carryover occurred primarily from the trap on the concentrator, not the autosampler.

Table 6. Water Carryover

Compound	%Carryover	Compound	%Carryover	Compound	%Carryover
dichlorodifluoromethane	0.01	trichloroethene	0.19	bromobenzene	0.14
chloromethane	ND	1,2-dichloropropane	ND	1,1,2,2-tetrachloroethane	ND
vinyl chloride	ND	dibromomethane	ND	1,2,3-trichloropropane	ND
bromomethane	0.12	bromodichloromethane	ND	n-propyl benzene	0.18
chloroethane	ND	cis-1,3-dichloropropene	ND	2-chlorotoluene	0.16
trichlorofluoromethane	0.09	toluene	0.16	4-chlorotoluene	0.32
1,1-dichloroethene	0.18	trans-1,3-dichloropropene	ND	1,3,5-trimethylbenzene	0.10
methylene chloride	ND	1,1,2-trichloroethane	ND	tert-butylbenzene	ND
trans-1,2-dichloroethene	0.12	tetrachloroethene	0.24	1,2,4-trimethylbenzene	ND
1,1-dichloroethane	ND	1,3-dichloropropane	ND	sec-butylbenzene	0.12
2,2-dichloropropane	ND	dibromochloromethane	0.25	1,3-dichlorobenzene	0.39
cis-1,2-dichloroethene	ND	1,2-dibromomethane	ND	4-isopropyltoluene	0.18
bromochloromethane	ND	chlorobenzene	ND	1,4-dichlorobenzene	0.38
chloroform	ND	1,1,1,2-tetrachloroethane	0.10	1,2-dichlorobenzene	0.15
1,1,1-trichloroethane	ND	ethyl benzene	0.14	n-butylbenzene	0.46
1,1-dichloropropene	ND	m,p-xylene	0.11	1,2-dibromo-3-chloropropane	0.09
carbon tetrachloride	0.06	o-xylene	0.04	1,2,4-trichlorobenzene	0.39
benzene	0.04	styrene	0.03	hexachlorobutadiene	0.56
1,2-dichloroethane	ND	bromoform	ND	naphthalene	0.30
		isopropyl benzene	0.06	1,2,3-trichlorobenzene	ND

Table 7. Soil Carryover

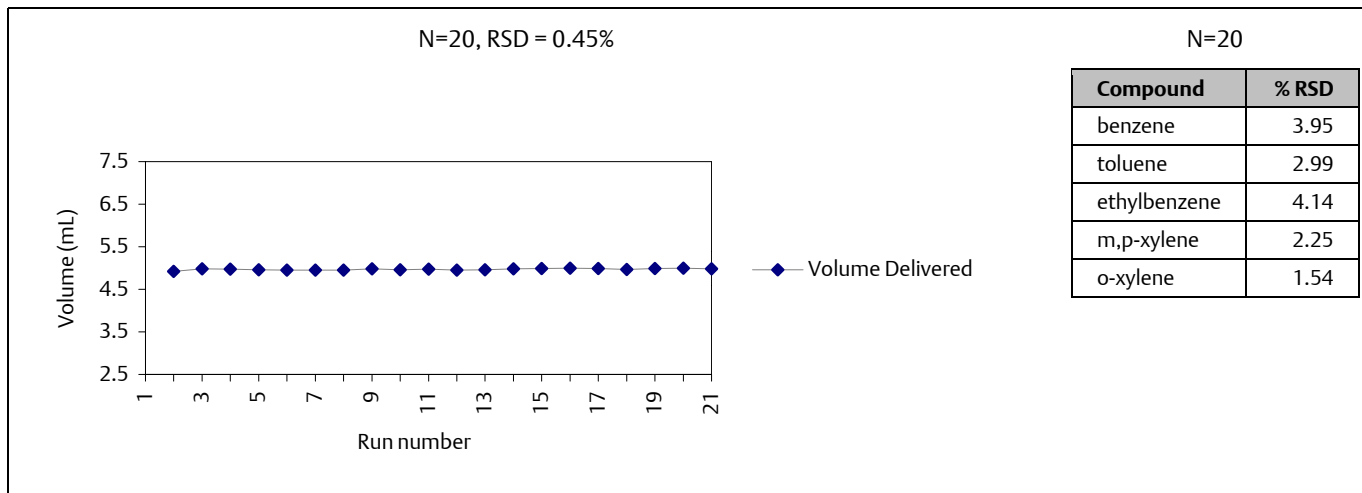
Compound	%Carryover	Compound	%Carryover	Compound	%Carryover
dichlorodifluoromethane	ND	trichloroethene	ND	bromobenzene	0.09
chloromethane	ND	1,2-dichloropropane	ND	1,1,2,2-tetrachloroethane	ND
vinyl chloride	ND	dibromomethane	ND	1,2,3-trichloropropane	ND
bromomethane	0.24	bromodichloromethane	ND	n-propyl benzene	ND
chloroethane	ND	cis-1,3-dichloropropene	ND	2-chlorotoluene	0.09
trichlorofluoromethane	ND	toluene	0.12	4-chlorotoluene	0.13
1,1-dichloroethene	ND	trans-1,3-dichloropropene	ND	1,3,5-trimethylbenzene	0.09
methylene chloride	ND	1,1,2-trichloroethane	ND	tert-butylbenzene	0.13
trans-1,2-dichloroethene	ND	tetrachloroethene	ND	1,2,4-trimethylbenzene	0.10
1,1-dichloroethane	ND	1,3-dichloropropane	ND	sec-butylbenzene	0.16
2,2-dichloropropane	ND	dibromochloromethane	ND	1,3-dichlorobenzene	0.16
cis-1,2-dichloroethene	ND	1,2-dibromomethane	ND	4-isopropyltoluene	0.12
bromochloromethane	ND	chlorobenzene	ND	1,4-dichlorobenzene	0.23
chloroform	ND	1,1,1,2-tetrachloroethane	ND	1,2-dichlorobenzene	0.19
1,1,1-trichloroethane	ND	ethyl benzene	0.07	n-butylbenzene	0.26
1,1-dichloropropene	ND	m,p-xylene	0.06	1,2-dibromo-3-chloropropane	0.05
carbon tetrachloride	ND	o-xylene	0.03	1,2,4-trichlorobenzene	0.34
benzene	0.06	styrene	0.07	hexachlorobutadiene	0.45
1,2-dichloroethane	0.03	bromoform	ND	naphthalene	0.30
		isopropyl benzene	0.02	1,2,3-trichlorobenzene	0.33

Carryover is minimized with the combination of high temperature OptiRinse™, multiple pressurized sweeps and a PEEK™/Teflon® tubing sample pathway. Most of the EPA 8260A volatile compound list analytes exhibited zero carryover following a 200 ppb standard for both water and soil analyses, with the exception of the higher molecular weight compounds. The maximum carryover for any compound was hexachlorobutadiene at 0.56%.

SAMPLE DELIVERY PRECISION

The precision of the SOLATEk 72's sample delivery was evaluated by two methods. The first test measured the actual volume of sample delivered by the autosampler to the concentrator. The second test measured the precision of the area counts obtained from BTEX analyses.

Figure 2.



CONCLUSIONS

Laboratories have productivity challenges that must be met to survive in their competitive markets. With a 72-vial capacity, the SOLATEk 72 provides the highest sample capacity of any VOC autosampler. Analytically, the SOLATEk 72 meets or exceeds the cleanliness, reproducibility, and durability required for a vial autosampler in a high quality environmental laboratory.

Linearity between the levels of 1.0ppb-200ppb was excellent, proving the inertness of both the water and soil pathways. The five point calibrations and the carryover evaluation were performed on an Agilent 6890/5973 MSD using a RTx-5 column under normal conditions.

Reproducibility and accuracy of the standard addition and sample delivery are excellent. This can best be seen by the %RSDs and correlation coefficients determined from the standard injection data. RSDs of the raw area counts were generally < 2%. Correlation coefficients of > 0.995 or better demonstrate the system's accuracy with multiple injections.

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