联和层析
应用文章精选
2018 | 08

联和层析致力于**提供无限可能的色谱解决方案**。十多年来，我们与全球多家专业色谱厂商保持良好合作伙伴关係，并将他们优势产品引进到国内。本着**诚恳、实在、专业、负责**的理念，联合层析在样品前处理及自动化的领域已占据领先地位。

为服务广大客户对于了解国际间最新色谱应用方向的需求，联和层析定期精选并翻译应用文章，为国内的科技服务提升，略尽绵薄之力！

《EPA 8260C 实例：CDS Analytical 7000C 与 PAL 系统进行吹扫捕集联用》

CDS Analytical 7000C 与 PAL 系统耦合是一种强大的清洗和捕集自动化解决方案。该配置能全自动清除并捕获痕量可溶挥发性有机化合物（VOCs）在水中的测量，符合 DIN EN ISO 15009，与 EPA 500 和 8000 系列对于 VOCs 在水中的方法规范。使用 7000C/PAL 系统得出的数据比 EPA 8260C 的性能标准要求精度更高。

【应用配置】

CDS Analytical 7000C 与 PAL RTC 耦合，两个仪器之间的通信是通过 TCP/IP 协议进行的。样品从 PAL RTC 吹扫捕集工具通过稀释模块传输到 CDS Analytical 7000C。CDS 专有的 X 类型捕集工具也被使用。

【实际应用与亮点】

7000C/PAL 系统在浓度范围为 0.5µg/L 至 200µg/L 的条件下，应用 EPA 8260C，能够得到非常好的主致密线。该系统的许多技术优点，包括自动稀释功能和内部标准模块，可为终端用户在仪器校准和样品测量中节省宝贵的时间。

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EPA Method 8260C Using CDS Analytical 7000C Purge and Trap with a PAL System

Application Note
Environmental

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Abstract

CDS 7000C Purge and Trap Concentrator coupled to a PAL System is a powerful Purge and Trap automation solution. This application demonstrates EPA Method 8260C using the 7000C Purge and Trap with the PAL System

Introduction

CDS Analytical's 7000C Purge and Trap concentrator designed for PAL System is the world's finest Purge and Trap automation solution. This instrumentation fully automates Purge and Trap for the trace measurement of purgeable volatile organic compounds (VOCs) in water, compliant with the official International Standard Organization method DIN-EN ISO 15009, U.S. EPA method 500 and 8000 series for VOCs in water. In this application note data is presented that the 7000C/PAL System exceeds the performance criteria set of EPA Method 8260C.

Experimental Conditions

A 7000C Purge and Trap concentrator connected to a PAL RTC Rail was used to collect the data. The Purge and Trap method parameters are shown in Table 1 which are standard for the analysis of VOCs defined in the EPA Method 8260C. The communications between the 7000C and RAL RTC were through TCP/IP protocol, and samples were transferred to the 7000C from RAL RTC Purge and Trap tool through a dilutor module. CDS's proprietary Type X trap was used.

Purge and Trap Model	**7000C-CTC PAL RTC**
Trap	Type X
Sample Size	5 mL
Purge Gas (He or N_2)	He
Purge Parameters:	
Valve Oven Temperature	130 °C
Transfer Line Temperature	130 °C
Hot Water Rinse Module Temperature	70 °C
Standby Flow	10 mL/min
Trap Ready Temperature	35 °C
Wet Trap Ready Temperature	45 °C
Sparge Vessel Heater	On
Purge Time	11 min
Purge Flow	40 mL/min
Purge Temperature	40 °C
Dry Purge Time	2 min
Dry Purge Flow	200 mL/min
Dry purge Temperature	35 °C
Foam Sensor	On
Desorb Parameters:	
Water Rinse Volume	5 mL
Number of Water Rinses	3

Table 1: Purge and Trap Method Parameters

Over Flow Sensor	On
Desorb Preheat Temperature	245 °C
GC Start Signal	Desorb
Desorb Time	6 min
Desorb Drain Flow	250 mL/min
Desorb Temperature	250 °C
Bake Parameters:	
Bake Time	4 min
Bake and Vessel Flow @MFC	200 mL/min
Trap Bake Temperature	260 °C
Wet Trap Bake Temperature	260 °C

Table 1: Purge and Trap Method Parameters, continued.

A Shimadzu single quad GCMS-QP 2010 was used. GC/MS conditions are listed in Table 2. The RTC rail was mounted directly on top of the GC. Carrier gas was supplied to the 7000C and a heated transfer line from the 7000C Concentrator was plumbed into the carrier supply line of the split/spitless inlet.

Gas Chromatograph:	Shimadzu GC 2010		
Analytical Column:	Rtx-VMS (30 m x 0.25 mm x 1.40 µm)		
Injector Temperature:	135 °C		
Carrier Gas:	Helium @ 1.0 mL/min		
Split Ratio:	40:1		
Oven Program:	Rate	Temperature	Hold Time
		35 °C	4 min
	5 °C/min	90 °C	0
	12 °C/min	150 °C	0
	30 °C/min	220 °C	2.67 min
Mass Spectrometer:	Shimadzu GCMS-QP 2010		
GC Transfer Line Temperature:	220 °C		
Ion Source Temperature:	200 °C		
Function Type:	Full Scan		
Solvent Delay:	1.0 min		
Scan Range:	m/z 35-260		
Scan Time:	0.3 sec		
Scan Speed:	833		

Table 2: GC/MS Conditions

The internal and external calibration standards were diluted from stock solutions using high precision Hamilton syringes and Class-A volumetric flasks. The external calibration standard contained a 50 component 8260 calibration mix (Supelco #500607) and a 6 component 502.2 calibration gas mix (Supelco #47408). The external standards were diluted to concentrations of 200 µg/L and 5 µg/L with deionized water, then added to two separate 40 mL VOC vials until full. The internal standard was a 3 component 8260 internal standard mix (Supelco #CRM861183) mixed with 3 component VOA surrogate (Supelco #861135) diluted to a concentration of 25 µg/L. 5 mL of this internal standard was added to the 7000C internal standard module reservoir #1 (2 reservoirs supported). The calibration levels (Table 3) used in this study were achieved with the auto dilution function embedded in the PAL Sample Control (PSC) software.

Calibration Level	Concentration (µg/L)	Preparation Method
1	0.5	Auto Dilution
2	1.5	Auto Dilution
3	5	Manual
4	20	Auto Dilution
5	60	Auto Dilution
6	200	Manual

Table 3:Calibration Levels

Results and Discussion

Figure 1 is the Total Ion Chromatogram (TIC) of a 200 µg/L calibration standard with internal standard and surrogates. All of the analytes are adequately resolved chromatographically. The chromatogram of the 6 gases is enlarged in the insert in order to show the excellent separation and peak shapes.

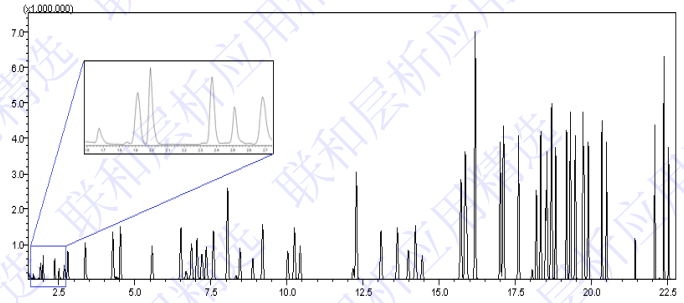

Figure 1. TIC of 8260C volatile organic standard mix at 200 µg/L with enlarged chromatogram of the 6 gasses.

Data summary Table 4 on the final pages lists the results for Retention Time (RT), Average Relative Response Factors (Avg RRF), Percent Relative Standard Deviation (% RSD) of the initial calibration, Method Detection Limits (MDL), along with method accuracy as Percent Recovery (% Rec) and as % RSD. All analytes exceed the EPA 8260C method requirements. MDL were determined by analyzing eleven replicate samples at a concentration of 1.0 µg/L. Precision and accuracy of recovery were measured by analyzing four replicates at a concentration of 5 µg/L.

The truncated TICs (18.5 min to 20 min) in Figure 2 illustrate the excellent repeatability at low concentration (1 µg/L). Figure 3 shows the six gases primary ion peaks at 0.5 µg/L concentration.

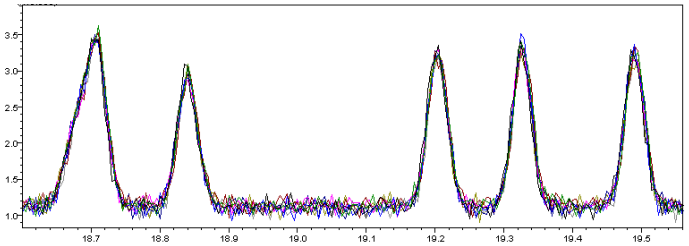

Figure 2. TICs overlaid from 18.5 min to 20 min.

The Internal Standard Module precisely delivered 1 µL of the pre-mixed internal standard solution to each sample. The reproducibility data from 8 runs is shown in Table 5. An excellent RDS < 2.4% is reported. Figure 4 is the time-shifted overlap of 8 1,4-Dichlorobenzene-d4 runs using the internal standard module.

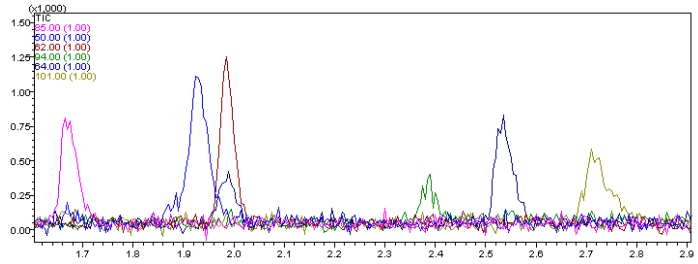

Figure 3. Six gases primary ion peaks at 0.5 µg/L concentration.

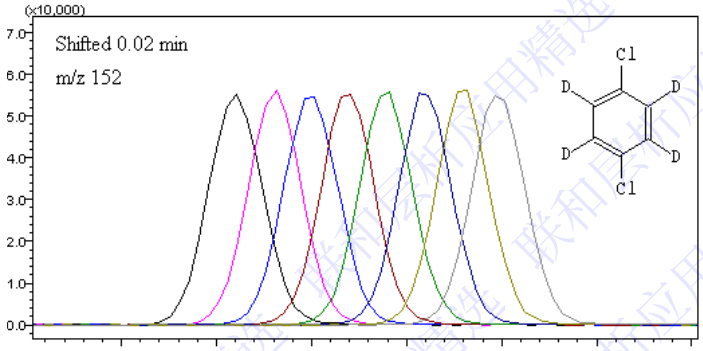

Figure 4. Overlap of eight 1,4-Dichlorobenzene-d4 runs from the internal standard module. The retention time of each peak has been shifted 1.2 seconds to show the consistency of the peak shape.

Conclusion

The 7000C Purge and Trap solution for PAL System easily meets and exceeds the EPA Method 8260C over a concentration range from 0.5 µg/L to 200 µg/L with excellent MDLs. Many of the technical advantages in the system, including the Auto Dilution function and the Internal Standard Module, are proven to be working to save precious time for end users in the instrument calibration and sample measurement.

No.	Compound	RT (min)	Avg RRF	RRF %RSD	MDL (µg/L)	Replicates (RSD%)	Recovery (%)
1	Dichlorodifluoromethane	1.639	0.287	2.43	0.06	2.3	99.6
2	Methane, chloro-	1.875	0.449	3.87	0.07	2.7	99.2
3	Vinyl chloride	1.952	0.376	3.67	0.06	2.3	99.3
4	Methane, bromo-	2.326	0.155	18.03	0.06	3.9	83.1
5	Ethyl Chloride	2.511	0.265	6.08	0.13	4.5	107.0
6	Trichloromonofluoromethane	2.653	0.310	2.79	0.09	4.1	96.0
7	Ethene, 1,1-dichloro-	3.317	0.301	3.88	0.09	3.7	96.6
8	Methylene Chloride	4.228	0.408	8.17	0.09	3.9	98.1
9	Ethene, 1,2-dichloro-, (trans)-	4.471	0.390	7.13	0.09	3.8	100.8
10	Ethane, 1,1-dichloro-	5.506	0.522	5.47	0.10	4.3	97.0
11	Ethene, 1,2-dichloro-, (cis)-	6.461	0.409	3.18	0.05	2.0	97.9
12	Propane, 2,2-dichloro-	6.6	0.291	5.74	0.06	4.6	90.9
13	Methane, bromochloro-	6.809	0.300	4.80	0.12	4.5	96.1
14	Trichloromethane	6.991	0.450	6.07	0.15	6.1	93.6
15	Carbon Tetrachloride	7.145	0.183	4.09	0.15	5.8	98.5
16	Ethane, 1,1,1-trichloro-	7.296	0.312	6.38	0.09	3.9	94.6
17	Dibromofluoromethane	7.369	Surrogate				
18	1-Propene, 1,1-dichloro-	7.531	0.392	6.26	0.07	3.1	95.5
19	Benzene	7.996	1.409	2.84	0.06	2.2	99.8
20	1,2-Dichloroethane-d4	8.327	Surrogate				
21	Ethane, 1,2-dichloro-	8.416	0.370	2.62	0.10	3.7	100.3
22	Benzene, fluoro-	8.83		Internal Standard			
23	Trichloroethylene	9.15	0.444	3.32	0.10	3.8	100.9
24	Methane, dibromo-	9.983	0.213	4.05	0.10	4.0	98.0
25	Propane, 1,2-dichloro-	10.205	0.396	2.73	0.11	4.5	99.8
26	Methane, bromodichloro-	10.393	0.359	6.65	0.06	2.6	91.2
27	Toluene-D8	12.168	Surrogate				
28	Toluene	12.236	2.008	5.73	0.09	3.1	109.3
29	Tetrachloroethylene	13.051	0.419	6.27	0.12	4.9	104.5
30	Ethane, 1,1,2-trichloro-	13.586	0.476	3.11	0.15	5.4	103.2
31	Methane, dibromochloro-	13.949	0.409	8.88	0.12	5.8	86.8
32	Propane, 1,3-dichloro-	14.181	0.852	4.14	0.09	3.4	101.6
33	Ethane, 1,2-dibromo-	14.41	0.480	3.79	0.12	4.9	95.6
34	Chlorobenzene-d5	15.649		Internal Standard			
35	Benzene, chloro-	15.685	1.370	3.34	0.06	2.1	103.4
36	Ethylbenzene	15.82	2.086	3.27	0.10	3.8	106.0
37	1,1,1,2-Tetrachloroethane	15.866	0.437	4.57	0.12	5.2	99.2
38	m,p-Xylene	16.148	3.317	4.98	0.08	2.8	108.3

Table 4. Initial Calibration Results for VOCs Listed at 0.5 – 200 µg/L

39	o-Xylene	16.975	1.693	3.88	0.08	3.0	105.0
40	Bromoform	17.065	0.261	12.24	0.09	5.6	83.8
41	Styrene	17.087	1.291	7.85	0.05	2.3	96.1
42	Cumene	17.584	1.999	5.87	0.07	2.7	105.8
43	Benzene, 1-bromo-4-fluoro-	18.034	Surrogate				
44	Benzene, bromo-	18.168	1.706	3.58	0.09	3.3	99.2
45	Benzene, propyl-	18.308	4.567	5.67	0.07	2.6	107.9
46	Ethane, 1,1,2,2-tetrachloro-	18.472	1.262	3.65	0.10	4.2	96.4
47	2-Chlorotoluene	18.516	2.873	3.85	0.07	2.4	107.4
48	1,2,3-Trichloropropane	18.639	1.358	3.25	0.08	3.0	104.3
49	Benzene, 1,3,5-trimethyl-	18.669	3.540	4.77	0.09	3.5	107.8
50	4-Chlorotoluene	18.801	2.973	4.70	0.07	2.8	106.7
51	Benzene, tert-butyl-	19.162	2.866	6.24	0.10	3.9	106.8
52	Benzene, 1,2,4-trimethyl-	19.284	2.469	5.09	0.11	3.2	106.8
53	Sec-Butylbenzene	19.447	3.908	8.00	0.07	3.0	108.3
54	p-Isopropyltoluene	19.697	3.300	7.32	0.07	2.9	106.6
55	Benzene, 1,3-dichloro-	19.729	2.129	4.36	0.09	3.5	102.1
56	1,4-Dichlorobenzene-d4	19.855		Internal Standard			
57	Benzene, 1,4-dichloro-	19.875	2.182	5.25	0.08	2.9	103.4
58	Benzene, butyl-	20.326	2.658	8.75	0.06	2.7	106.7
59	Benzene, 1,2-dichloro-	20.475	2.141	4.25	0.06	2.0	104.6
60	Propane, 1,2-dibromo-3-chloro-	21.424	0.399	6.40	0.21	10.6	85.7
61	Hexachlorobutadiene	22.057	0.353	17.88	0.13	5.8	107.5
62	Benzene, 1,2,4-trichloro-	22.075	1.080	7.18	0.09	3.8	98.6
63	Naphthalene	22.364	5.402	7.40	0.07	2.5	107.7
64	Benzene, 1,2,3-trichloro-	22.522	1.082	7.04	0.08	3.1	99.7

Table 4. Initial Calibration Results for VOCs Listed at 0.5 – 200 µg/L, continued.

Compound	**Fluorobenzene**	**Chlorobenzene-d5**	**1,4-Dichlorobenzene-d4**
RSD% (n=8)	1.449	1.478	2.338

Table 5: Reproducibility of Internal Standard Addition.