

使用Nutech预浓缩系统进行EPA TO-15A快速实验分析*

前言

使用三级冷阱预浓缩系统+GC/MS技术(全扫描和/或SIM)，在一次快速运行中分析了空气中的82种VOC化合物(TO-15A混合物由Linde提供)。结果表明，在0.1-20.0ppb范围内，线性标定、精密度、准确度、空白和MDL等都符合EPA TO-15A的要求。在SIM模式下，MDL可以达到1-2ppt水平。GC的运行时间可快至13.5分钟。Nutech的仪器系统是在环境空气中进行TO-15A目标VOCs分析的良好工具。

介绍

本应用尝试使用Nutech 8910仪器开发一个实验室分析方法，在一次快速实验中，涵盖所有82种VOC化合物。Nutech团队通过将Nutech预浓缩系统与GC/MS结合使用，在美国达拉斯和中国深圳实验室中进行了尝试。本应用简报是他们的近期成果。它显示GC运行时间只需13.5分钟，从第一次进样到下一次进样的整个循环时间可能短至18分钟。

实验

实验仪器配置

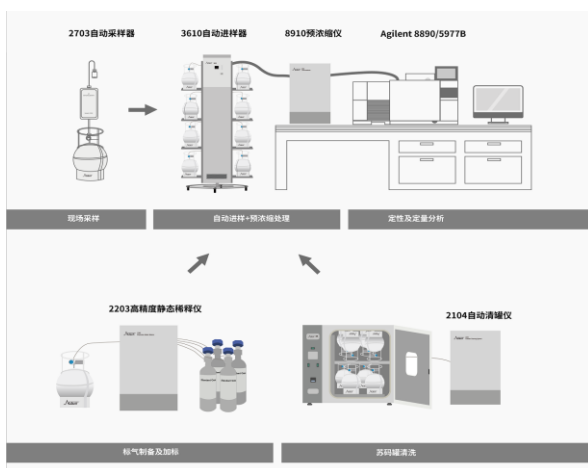
样品预处理仪器：

Nutech大气预浓缩系统，包括：Nutech8910预浓缩仪、Nutech3610 自动进样器、Nutech2203高精度静态稀释仪，Nutech2104自动清罐仪以及6L苏玛罐。

分析仪器：

Agilent 8890/5977B 单四极杆气质联用仪（可配置Deans Switch模块和FID检测器，但本项研究中未应用）

上述仪器工作的逻辑关系图如下：



实验标准气体

实验用到的标准气体全部来自Linde。

外标气体标准物质：

81种TO-15A气体标准物质，浓度1.00ppm；
环氧乙烷，浓度1.00 ppm。

由于环氧乙烷的稳定性，林德必须分别制作两瓶不同的标准气体。

内标气体标准物质：

一溴一氯甲烷、1,4- 二氟苯、氘代氯苯、4- 溴氟苯，
浓度都是1.0ppm

* 本应用实验分析由Nutech-美国达拉斯实验室、中国深圳实验室协同完成。

实验标气的配制

将两个高浓度标准气体和一个经认证的干净的6升苏玛罐连接到Nutech 2203稀释仪上，设置5 ppb为工作标气的最终浓度。按照稀释器操作手册制作工作标气。内标的操作相同，但浓度为50ppb。用50%的湿度对罐子进行加湿。

实验分析条件的设置

8910预浓缩仪分析条件:

一级冷阱（空）温度：-50℃，二级冷阱温度：-50℃。
一级到二级冷阱转移温度10℃，20毫升/分钟，5 分钟。

二级冷阱解析温度220℃。

三级冷阱聚焦温度-150℃，解析进样脉冲温度80℃，传输线温度40℃。

8890GC 分析条件:

进样口温度：250℃

分流比：分流/不分流

色谱柱：Restek Rtx-VHS，30m×0.25mm×1.4μm

中心切割阻尼柱：1.0m×0.18mm×0μm（如不使用中心切割，可不使用阻尼柱）

升温程序：40℃（保持1 min），然后15℃/min升至220℃（保持5min）

载气控制方式：恒流模式，流速1.8 ml/m

5977B MS分析条件:

离子源：320℃

接口温度：250℃

采集方式：全扫描（Full Scan）/选择离子扫描（SIM）

扫描范围：Full Scan: 25-300 amu;

SIM: 所有目标化合物的数量离子和95,128,130,114,117 amu。

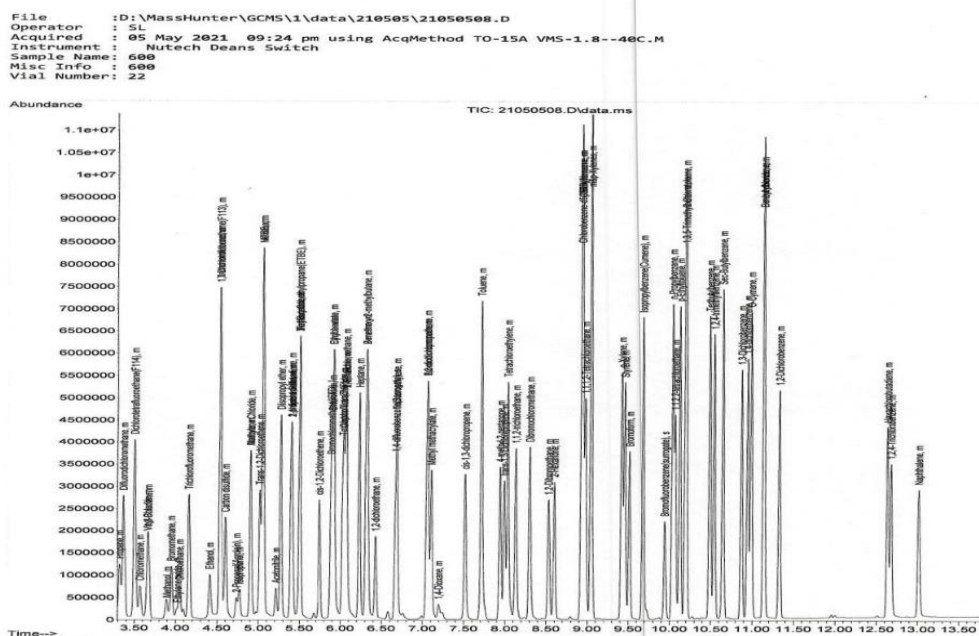
Initial Calibration

“3610自动进样器+8910预浓缩仪”依次进样30ml、60 ml、120 ml、240 ml、300 ml、600 ml，基准体300 ml。

使用5 ppb标准气，对应浓度为0.5 ppb、1.0 ppb、2.0 ppb、4.0 ppb、5.0 ppb、10.0ppb，以浓度为横坐标，响应值为纵坐标建立校准曲线。内标加载体积为30ml，浓度为5.0 ppb。

实验结果

82种TO-15A化合物图谱



线性 (1cal)

以0.5 ppb、1.0 ppb、2.0 ppb、4.0 ppb、5.0 ppb、10.0ppb建立标准曲线，线性范围为1: 20 。以一溴一氯甲烷、氘代氯苯两种物质作为内标(IS)，二氟苯、溴氯苯两种物质作为内标回收物(SS)，定量线性，定量数据列表如下：

标准曲线验证(CCV)

以5 ppb 浓度标准样品，进样120 ml为中间点，2.0ppb浓度标样，验证标准曲线 (CCV) 结果数据如下表：

Response Factor Report Nutech Deans Switch												
Method Path : D:\MassHunter\GCMS\1\												
Method File : TO15A-VMS-210505-1Cal-40C.M												
Title : TO-15A-VMS												
Last Update : Thu May 06 11:46:25 2021												
Response via : Initial Calibration												
Calibration Files												
1	2	3	4	5	6	Avg	VRSD					
Compound	1	2	3	4	5	6	Avg	VRSD				
-----ISTD-----												
1 I Bromochloromethane...												
2 m Propene	0.885	0.935	0.877	0.854	0.827	0.813	0.866	4.99				
3 m Difluorodichloro...	2.494	2.453	2.402	2.427	2.319	1.973	2.345	8.15				
4 m Dichlorotetra...	2.582	2.563	2.509	2.526	2.409	2.443	2.505	2.70				
5 m Chloromethane	0.997	1.020	0.961	0.960	0.912	0.916	0.961	4.45				
6 m Vinyl Chloride	1.105	1.130	1.074	1.076	1.021	1.025	1.072	4.02				
7 m 1,3-Butadiene	0.825	0.844	0.809	0.799	0.765	0.764	0.801	4.03				
8 m Ethylene oxide	0.209	0.228	0.201	0.174	0.190	0.192	0.192	13.49				
9 m Methanol	0.129	0.123	0.112	0.068	0.096	0.068	0.099	26.99				
10 m Bromomethane	0.930	0.945	0.903	0.897	0.873	0.844	0.899	4.10				
11 m Chloroethane	0.566	0.567	0.545	0.543	0.519	0.516	0.543	4.04				
12 m Trichlorofluor...	2.386	2.350	2.286	2.308	2.179	2.213	2.287	3.45				
13 m Ethanol	1.249	1.360	1.303	0.877	1.237	0.914	1.157	17.92				
14 m 1,1-Dichloroet...	1.771	1.600	1.710	1.718	1.631	1.639	1.678	3.86				
15 m Trichlorotrifu...	2.050	2.028	2.001	2.014	1.915	1.952	1.994	2.53				
16 m Carbon disulfide	3.045	3.027	2.963	2.971	2.893	2.896	2.959	2.70				
17 m 2-Propanol (Ac...	0.325	0.288	0.329	0.330	0.320	0.322	0.319	4.96				
18 m Isopropanol	0.927	1.000	0.980	0.756	0.973	0.750	0.998	12.76				
19 m Methylene Chloro...	1.365	1.407	1.300	1.289	1.223	1.219	1.301	5.76				
20 m Acetone	2.407	2.406	2.214	1.961	1.876	1.747	2.102	13.37				
21 m Trans-1,2-Dich...	1.673	1.653	1.596	1.612	1.513	1.544	1.598	3.85				
22 m MTBE	2.906	2.871	2.817	2.827	2.696	2.733	2.808	2.86				
23 m Hexane	2.548	2.586	2.468	2.491	2.381	2.383	2.474	3.40				
24 m Acetonitrile	0.682	0.743	0.680	0.632	0.665	0.641	0.674	8.87				
25 m Diisopropyl ether	3.928	4.031	3.775	3.793	3.578	3.588	3.782	4.77				
26 m Tert-Butanol	3.569	3.584	3.439	3.471	3.277	3.287	3.438	3.86				
27 m Vinyl acetate	2.939	3.073	2.908	2.912	2.769	2.771	2.895	3.96				
28 m 2-chloro-1,3-b...	2.250	2.315	2.226	2.256	2.158	2.178	2.230	2.55				
29 m 2-propenenitrile	2.250	2.315	2.226	2.256	2.158	2.178	2.230	2.55				
30 m 1,1-dichloroet...	2.006	2.026	1.941	1.946	1.835	1.848	1.934	4.08				
31 m 2-methoxy-2-met...	3.569	3.584	3.439	3.471	3.277	3.287	3.438	3.86				
32 m cis-1,2-Dichlor...	1.529	1.521	1.460	1.455	1.392	1.409	1.461	3.83				
33 m Chloroform	2.310	2.311	2.221	2.223	2.104	2.125	2.216	3.97				
34 m Cyclohexane	1.793	1.847	1.773	1.770	1.679	1.686	1.758	3.69				
35 m Ethyl acetate	3.105	3.121	2.879	2.841	2.679	2.673	2.883	6.82				
36 m Tetrahydrofura...	1.310	1.213	1.136	1.090	1.041	1.018	1.135	9.76				
37 m Carbon Tetrach...	2.022	2.085	2.054	2.136	2.031	2.110	2.073	2.19				
38 m 1,1,1-trichloro...	1.216	1.278	1.218	1.218	1.136	1.136	1.218	3.63				
39 m 2-Butanone	2.529	2.357	2.188	2.243	2.119	2.120	2.259	7.05				
40 m Heptane	2.076	2.162	2.031	2.048	1.928	1.926	2.028	4.46				
41 m Benzene	3.527	3.463	3.406	3.330	3.168	3.166	3.355	4.76				
42 m 2-methoxy-2-me...	3.216	3.212	3.112	3.164	3.013	3.041	3.126	2.76				
43 m 1,2-dichloroet...	1.423	1.447	1.361	1.356	1.273	1.283	1.357	5.21				
44 m 1,4-difluorobe...	3.965	3.974	3.871	3.846	3.877	3.962	3.932	1.17				
45 m Trichloroethyl...	1.353	1.314	1.318	1.329	1.274	1.303	1.315	2.00				
46 m 1,2-dichloropr...	1.335	1.318	1.254	1.267	1.197	1.215	1.264	4.33				
47 m Bromodichloro...	2.295	2.327	2.260	2.299	2.199	2.240	2.270	2.05				
48 m Methyl methacr...	1.413	1.471	1.407	1.456	1.379	1.422	1.425	2.36				
49 m 1,4-Dioxane	0.287	0.310	0.315	0.250	0.295	0.243	0.283	10.69				
50 m cis-1,3-dichlo...	1.929	1.933	1.875	1.906	1.819	1.812	1.889	2.29				
51 m Toluene	4.775	4.663	4.576	4.599	4.391	4.425	4.572	3.17				
52 m 4-methyl-2-pen...	2.258	2.330	2.231	2.286	2.140	2.189	2.239	3.04				
53 m trans-1,3-dich...	1.715	1.728	1.684	1.714	1.645	1.681	1.695	1.80				
54 m Tetrachloroeth...	1.665	1.634	1.597	1.613	1.549	1.586	1.607	2.50				
55 m 1,1,2-trichlor...	1.324	1.278	1.249	1.244	1.203	1.222	1.257	3.39				
56 m Dibromochlorom...	1.968	1.948	1.976	2.041	1.967	2.054	1.991	2.27				
57 m 1,2-Dibromoeth...	1.921	1.895	1.878	1.921	1.832	1.874	1.887	1.78				
58 m 2-Hexanone	1.986	2.031	1.946	1.990	1.857	1.891	1.950	3.37				
-----ISTD-----												
59 I Chlorobenzene-d5(I...												
60 m Ethylbenzene	1.382	1.348	1.397	1.332	1.291	1.312	1.337	2.46				
61 m Chlorobenzene	0.811	0.783	0.789	0.782	0.764	0.778	0.785	2.01				
62 m 1,1,1,2-Tetrac...	0.435	0.404	0.409	0.402	0.394	0.407	0.405	1.69				
63 m m,p-Xylenes	1.017	0.995	1.010	0.991	0.961	0.980	0.989	2.45				
64 m o-Xylene	1.014	0.987	1.005	0.988	0.962	0.981	0.990	1.84				
65 m Styrene	0.710	0.694	0.731	0.737	0.725	0.737	0.727	3.24				
66 m Bromoform	0.476	0.466	0.501	0.514	0.507	0.544	0.501	5.53				
67 m Isopropylbenze...	1.413	1.356	1.379	1.355	1.321	1.347	1.362	2.29				
68 m Bromofluorobe...	0.737	0.745	0.739	0.733	0.725	0.737	0.735	0.89				
69 m n-Propylbenzene	1.717	1.678	1.699	1.684	1.621	1.639	1.673	2.17				
70 m 1,1,2,2-tetrac...	0.721	0.711	0.721	0.704	0.682	0.694	0.706	2.20				
71 m p-Ethyltoluene	1.326	1.294	1.331	1.317	1.283	1.313	1.311	1.43				
72 m 1,3,5-Trimethy...	1.052	1.023	1.050	1.041	1.014	1.040	1.037	1.46				
73 m 2-Chlorotoluene	1.131	1.106	1.103	1.088	1.053	1.065	1.091	2.62				
74 m Tertbutylbenze...	0.969	0.936	0.963	0.967	0.938	0.970	0.955	1.61				
75 m 1,2,4-trimethy...	1.026	0.989	1.022	1.022	0.992	1.021	1.012	1.67				
76 m Sec-Butylbenze...	1.550	1.497	1.529	1.526	1.484	1.515	1.517	1.57				
77 m 1,3-Dichlorobe...	0.631	0.637	0.655	0.657	0.642	0.672	0.653	1.85				
78 m 1,4-dichlorobe...	0.653	0.634	0.656	0.668	0.646	0.681	0.656	2.49				
79 m o-Cymene	1.117	1.094	1.119	1.133	1.106	1.140	1.116	1.50				
80 m n-Butylbenzene	0.260	0.256	0.272	0.276	0.272	0.286	0.270	3.89				
81 m Benzyl chloride	0.172	0.167	0.179	0.182	0.179	0.190	0.178	4.47				
82 m 1,2-Dichlorobe...	0.592	0.583	0.595	0.598	0.582	0.610	0.593	1.74				
83 m Hexachlorobuta...	0.278	0.265	0.223	0.244	0.280	0.298	0.268	9.47				
84 m 1,2,4-Trichlor...	0.269	0.257	0.251	0.309	0.328	0.347	0.293	13.66				
85 m Naphthalene	0.520	0.507	0.557	0.653	0.702	0.746	0.614	16.32				
-----ISTD-----												
(8) = Out of Range												
TO15A-VMS-2...5-1Cal-40C.M Thu May 06 11:49:23 2021												
Evaluate Continuing Calibration Report												
Data Path : D:\MassHunter\GCMS\1\data\210505\												
Data File : 21050507.D												
Acq On : 05 May 2021 08:38 pm												
Operator : SL												
Sample : 300												
Misc : 300												
ALS Vial : 22 Sample Multiplier: 1												
Quant Time: May 06 10:10:39 2021												
Quant Method : D:\MassHunter\GCMS\1\TO15A-VMS-210505-1Cal-40C.M												
Quant Title : TO-15A-VMS												
QLast Update : Thu May 06 10:09:00 2021												
Response via : Initial Calibration												
Min. RRF	0.000	Min. Rel. Area	50%	Max. R.T.	Dev	0.50min						
Max. RRF Dev	25%	Max. Rel. Area	150%									
Compound	AvgRRF	CCRF	%Dev Area	%Dev(min)								
1 I Bromochloromethane (ISTD)	1.000	1.000	0.0	100	0.00							
2 m Propene	0.866	0.827	4.5	100	0.00							
3 m Difluorodichloromethane	2.345	2.319	1.1	100	0.00							
4 m Dichlorotetrafluoromethane(F	2.505	2.409	3.8	100	0.00							
5 m Chloromethane	0.961	0.912	5.1	100	0.00							
6 m Vinyl Chloride	1.072	1.021	4.8	100	0.00							
7 m 1,3-Butadiene	0.801	0.765	4.5	100	0.00							
8 m Ethylene oxide	0.193	0.190	1.6	100	0.00							
9 m Methanol	0.096	0.096	3.0	100	0.00							
10 m Bromomethane	0.899	0.873	2.9	100	0.00							
11 m Chloroethane	0.543	0.519	4.4	100	0.00							
12 m Trichlorofluoromethane	1.984	1.915	4.0	100	0.00							
13 m Ethanol	1.157	1.237	-6.9	100	0.00							
14 m 1,1-Dichloroet...	1.678	1.631	2.8	100	0.00							
15 m Trichlorotrifuoromethane(F11	2.479	2.383	4.8	100	0.00							
16 m Carbon disulfide	2.959	2.853	3.6	100	0.00							
17 m 2-Propanol (Acrolein)	0.319	0.320	-0.3	100	0.00							
18 m Isopropanol	0.973	0.973	0.0	100	0.00							
19 m Methylene Chloride	1.301	1.223	6.0	100	0.00							
20 m Acetone	2.102	1.746	10.8	100	0.00							
21 m Trans-1,2-Dichloroethane	1.598	1.513	5.9	100	0.00							
22 m MTBE	2.808	2.696	4.0	100	0.00							
23 m Hexane	2.476	2.381	3.8	100	0.00							
24 m Acetonitrile	3.782	3.578	5.4	100	0.00							
25 m Diisopropyl ether	3.438	3.277	5.4	100	0.00							
26 m Tert-Butanol	3.854	3.769	2.2	100	0.00							
27 m Vinyl acetate	2.230	2.158	3.2	100	0.00							
28 m 2-chloro-1,3-butanediol	2.230	2.158	3.2	100	0.00							
29 m 2-propenenitrile	2.230	2.158	3.2	100	0.00							
30 m 1,1-dichloroethane	1.461	1.392	4.7	100	0.00							
31 m 2-ethoxy-2-methylpropane(ET	3.438	3.277	4.7	100	0.00							
32 m 1,1,2-trichloroethane	1.461	1.392	4.7	100	0.00							
33 m Chloroform	2.425	2.349	3.6	100	0.00							
34 m Cyclohexane	1.758	1.679	4.5	100	0.00							
35 m Ethyl acetate	2.883	2.679	6.1	100	0.00							
36 m Tetrahydrofuran(THF)	1.310	1.213	7.8	100	0.00							
37 m Carbon Tetrachloride	2.073	2.031	2.0	100	0.00							
38 m 1,1,1-trichloroethane	2.013	1.930	4.1	100	0.00							
39 m 2-Butanone	2.048	1.939	5.8	100	0.00							
40 m Heptane	2.028	1.928	4.9	100	0.00							
41 m Benzene	3.355	3.388	5.0	100	0.00							
42 m 2-methoxy-2-methylbutane	3.126	3.013	3.8	100	0.00							
43 m 1,2-dichloroethane	1.357	1.273	6.2	100	0.00							
44 m 1,4-difluorobenzene(Surrogat												

加标回收率 % (盲样考核准确度)

TO-15A列出了大约20个实验室的目标化合物回收报告。浓度水平从0.2到0.9ppb。 在本实验中, 我们平均加标0.5 ppb, 并展示了那些相同的目标化合物的回收率。

重现性

重现性测试选择了0.5ppb和2.0ppb, 7个点。使用响应因子来显示平均和标准偏差以及RSD%来呈现重现性结果。数据显示, 大多数化合物的RSD%都小于10%。

Spike Recovery and RPD Summary Report - WATER										Response Factor Report Nutech Deans Switch									
Method Path : D:\MassHunter\GCMS\1\										Method Path : D:\MassHunter\GCMS\1\									
Method File : TO15A-VMS-210505-1Cal-40C.M										Method File : TO15A-VMS-210505-1Cal-40C-replicate.M									
Title : TO-15A-VMS										Title : TO-15A-VMS									
Last Update : Thu May 06 11:48:16 2021										Last Update : Thu May 06 14:41:13 2021									
Response Via : Initial Calibration										Response Via : Initial Calibration									
Datafile Path: D:\MassHunter\GCMS\1\data\210505\										Calibration Files									
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最小检出限 (MDL)

根据TO-15A参考MDL表, 本实验展示了我们采用全扫描和SIM方法得到的MDL结果。空白MDL以及TO-15A表上的参考MDL也列在下表中。结果显示, 我们的MDL与TO-15A选定的实验室结果相近。在全扫描下, 大多数化合物的MDL均低于0.03 ppb (30 ppt); 在SIM扫描下, 所列化合物的大多数MDL低至1至2 ppt水平。

全扫描模式下的MDL:

Nutech 8910/Agilent 8890/5977 TO-15A VOC Compounds MDL results					
MDL is based on 600mL Loading Volume with 7 replicate Run					
Number	Compound	Spike Conc.	Avg. Conc.	% RSD	MDL
		(ppbv)	(ppbv)		
1	Bromochloromethane(ISTD)	5.0	5.00		
2	Propene	0.5	0.51	3.74	0.03
3	Difluorodichloromethane	0.5	0.55	2.66	0.02
4	Dichlorotetrafluoroethane(F114)	0.5	0.53	2.65	0.02
5	Chloromethane	0.5	0.54	3.18	0.03
6	Vinyl Chloride	0.5	0.54	3.44	0.03
7	1,3-Butadiene	0.5	0.54	3.02	0.03
8	Ethylene oxide	0.5	0.60	11.55	0.11
9	Methanol	0.5	0.70	7.14	0.08
10	Bromomethane	0.5	0.54	2.94	0.02
11	Chloroethane	0.5	0.54	3.66	0.03
12	Trichlorofluoroethane	0.5	0.53	2.49	0.02
13	Ethanol	0.5	0.57	6.27	0.06
14	1,1-Dichloroethane	0.5	0.52	5.27	0.04
15	Trichlorotrifluoroethane(F113)	0.5	0.53	2.59	0.02
16	Carbon disulfide	0.5	0.53	2.88	0.02
17	2-Propenal(Acroline)	0.5	0.52	3.53	0.03
18	Isopropanol	0.5	0.57	4.30	0.04
19	Methylene Chloride	0.5	0.55	3.32	0.03
20	Acetone	0.5	0.58	1.33	0.01
21	Trans-1,2-Dichloroethene	0.5	0.53	2.70	0.02
22	MTBE	0.5	0.53	2.50	0.02
23	Hexane	0.5	0.53	2.45	0.02
24	Acetonitrile	0.5	0.55	4.39	0.04
25	Diisopropyl ether	0.5	0.54	3.12	0.03
26	Tert-Butanol	0.5	0.53	2.75	0.02
27	Vinyl acetate	0.5	0.53	3.50	0.03
28	2-chloro-1,3-butadiene	0.5	0.53	3.18	0.03
29	2-propenenitrile	0.5	0.53	3.18	0.03
30	1,1-dichloroethane	0.5	0.53	2.48	0.02
31	2-methoxy-2-methylpropane(ETBE)	0.5	0.53	2.75	0.02
32	cis-1,2-Dichloroethene	0.5	0.54	3.12	0.03
33	Chloroform	0.5	0.54	3.12	0.03
34	Cyclohexane	0.5	0.54	3.18	0.03
35	Ethyl acetate	0.5	0.54	3.09	0.03
36	Tetrahydrofuran(THF)	0.5	0.58	4.44	0.04
37	Carbon Tetrachloride	0.5	0.50	2.59	0.02
38	1,1,1-trichloroethane	0.5	0.53	2.95	0.02
39	2-Butanone	0.5	0.54	3.92	0.03
40	Heptane	0.5	0.54	3.34	0.03
41	Benzene	0.5	0.54	2.42	0.02

全扫描模式下的MDL: (继续)

42	2-methoxy-2-methylbutane	0.5	0.53	2.82	0.02
43	1,2-dichloroethane	0.5	0.53	2.66	0.02
44	1,4-difluorobenzene(Surrogate)	5.0	5.00	0.88	0.05
45	Trichloroethylene	0.5	0.53	2.50	0.02
46	1,2-dichloropropane	0.5	0.54	2.61	0.02
47	Bromodichloromethane	0.5	0.53	3.12	0.03
48	Methyl methacrylate	0.5	0.52	3.36	0.03
49	1,4-Dioxane	0.5	0.53	5.30	0.04
50	cis-1,3-dichloropropene	0.5	0.52	3.07	0.03
51	Toluene	0.5	0.53	2.58	0.02
52	4-methyl-2-pentanone	0.5	0.52	3.25	0.03
53	trans-1,3-Dichloropropene	0.5	0.52	3.07	0.03
54	Tetrachloroethylene	0.5	0.53	2.36	0.02
55	1,1,2-trichloroethane	0.5	0.53	2.78	0.02
56	Dibromochloromethane	0.5	0.50	2.89	0.02
57	1,2-Dibromoethane	0.5	0.52	2.43	0.02
58	2-Hexanone	0.5	0.53	3.30	0.03
59	Chlorobenzene-d5(ISTD)	5.0	5.00		
60	Ethylbenzene	0.5	0.53	2.85	0.02
61	Chlorobenzene	0.5	0.53	3.18	0.03
62	1,1,1,2-Tetrachloroethane	0.5	0.52	3.08	0.03
63	m,p-Xylenes	0.5	1.06	2.90	0.05
64	o-Xylene	0.5	0.52	2.73	0.02
65	Styrene	0.5	0.49	2.88	0.02
66	Bromoform	0.5	0.48	2.46	0.02
67	Isopropylbenzene(Cumene)	0.5	0.52	2.81	0.02
68	Bromofluorobenzene(surrogate)	5.0	5.00	0.51	0.04
69	n-Propylbenzene	0.5	0.52	2.92	0.02
70	1,1,2,2-tetrachloroethane	0.5	0.52	2.81	0.02
71	p-Ethyltoluene	0.5	0.52	2.46	0.02
72	1,3,5-Trimethylbenzene	0.5	0.51	2.57	0.02
73	2-Chlorotoluene	0.5	0.53	2.99	0.02
74	Tertbutylbenzene	0.5	0.51	2.61	0.02
75	1,2,4-trimethylbenzene	0.5	0.51	3.01	0.02
76	Sec-Butylbenzene	0.5	0.52	2.47	0.02
77	1,3-Dichlorobenzene	0.5	0.51	2.99	0.02
78	1,4-dichlorobenzene	0.5	0.50	2.93	0.02
79	O-Xylene	0.5	0.50	2.81	0.02
80	n-butylbenzene	0.5	0.49	2.55	0.02
81	Benzyl chloride	0.5	0.48	2.59	0.02
82	1,2-Dichlorobenzene	0.5	0.50	2.78	0.02
83	Hexachlorobutadiene	0.5	0.52	2.49	0.02
84	1,2,4-Trichlorobenzene	0.5	0.43	4.56	0.03
85	Naphthalene	0.5	0.40	3.55	0.02

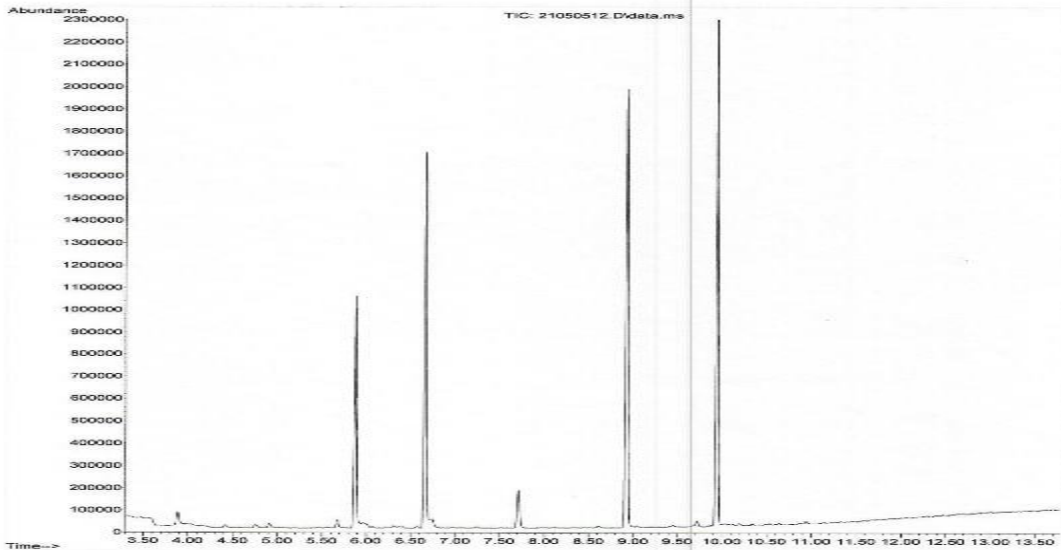
SIM模式下的MDL:

Nutech 8910/Agilent 8890/5977 TO-15A VOC Compounds MDL results									
MDL is based on 600mL Loading Volume with 7 replicate Run									
Number	Compound	Spike Conc.	Avg. Conc.	SD	MDL	Blank/Blank Report	Reference		
		(pptv)	(pptv)			MDL	MDL		
1	Difluorodichloromethane	10.00	9.78	0.25	0.79	0.00	1	2	
2	Chloromethane	10.00	9.85	0.30	0.93	1.37	1	4	
3	Dichlorotetrafluoroethane (F114)	10.00	9.82	0.33	1.04	0.00	1	2	
4	Vinyl Chloride	10.00	9.28	0.59	1.84	0.00	2	3	
5	1,3-Butadiene	10.00	11.08	1.17	3.69	0.00	4	7	
6	Bromomethane	10.00	10.34	0.69	2.16	0.00	2	2	
7	Chloroethane	10.00	9.33	0.45	1.41	0.00	1	1	
8	2-Propenal (Acrolein)	10.00	10.20	0.31	0.98	0.00	1	7	
9	Trichlorofluoroethane	10.00	10.29	0.52	1.65	0.00	2	2	
10	2-Propenenitrile	10.00	9.99	0.64	2.01	0.00	2	2	
11	1,1-Dichloroethane	10.00	9.42	0.41	1.30	0.00	1	1	
12	Methylene Chloride	10.00	11.41	0.36	1.14	0.86	1	3	
13	Trichlorotrifluoroethane (F113)	10.00	9.54	0.26	0.83	0.00	1	2	
14	1,1-dichloroethane	10.00	9.40	0.28	0.89	0.00	1	2	
15	cis-1,2-Dichloroethene	10.00	9.54	0.34	1.06	0.00	1	1	
16	Chloroform	10.00	9.78	0.28	0.87	0.72	1	2	
17	1,2-Dichloroethane	10.00	9.72	0.16	0.49	0.00	0.5	2	
18	1,1,1-Trichloroethane	10.00	10.01	0.25	0.78	0.00	1	1	
19	Benzene	10.00	12.04	0.41	1.29	2.04	2	2	
20	Carbon Tetrachloride	10.00	9.98	0.21	0.65	0.00	1	1	
21	1,2-Dichloropropane	10.00	8.66	0.53	1.65	0.00	2	1	
22	1,1,2-Trichloroethane	10.00	9.87	0.55	1.74	0.00	2	2	
23	cis-1,3-Dichloropropene	10.00	9.29	0.69	2.16	0.00	2	2	
24	Trans-1,3-Dichloropropene	10.00	9.38	0.54	1.69	0.00	2	2	
25	1,2,2-Trichloroethane	10.00	9.35	0.53	1.67	0.00	2	2	
26	Toluene	10.00	9.86	0.21	0.66	0.50	1	3	
27	1,2-Dibromoethane	10.00	8.86	0.13	0.42	0.00	0.4	2	
28	Tetrachloroethylene	10.00	9.66	0.47	1.48	0.00	1	2	
29	Chlorobenzene	10.00	10.02	0.39	1.23	1.07	1	2	
30	Ethylbenzene	10.00	9.72	0.32	1.02	0.48	1	1	
31	m,p-Xylenes	10.00	18.68	0.44	1.37	1.01	1	4	
32	Styrene	10.00	9.32	0.71	2.23	1.37	2	2	
33	o-Xylene	10.00	9.21	0.26	0.82	0.81	1	2	
34	1,1,2,2-Tetrachloroethane	10.00	9.56	0.31	0.96	0.00	1	2	
35	1,3,5-Trimethylbenzene	10.00	8.87	0.30	0.94	0.41	1	2	
36	1,2,4-Trimethylbenzene	10.00	8.78	0.68	2.13	0.00	2	6	
37	1,3-Dichlorobenzene	10.00	9.26	0.36	1.14	0.56	1	2	
38	1,4-Dichlorobenzene	10.00	9.26	0.36	1.14	0.56	1	2	
39	1,2-Dichlorobenzene	10.00	8.77	0.22	0.70	0.00	1	2	
40	1,2,4-Trichlorobenzene	10.00	8.10	0.30	0.93	0.96	1	4	
41	Hexachlorobutadiene	10.00	9.21	0.44	1.37	0.93	1	2	

空白

采用600毫升10 ppb 标样后直接进样300ml氮气空白，全部目标化合物低于检出限。全扫描模式和SIM模式的空白图谱显示如下。小峰为二氧化硅化合物，或是小于全扫描或SIM MDL的微量残留。

```
File       :D:\MassHunter\GCMS\1\data\210505\21050512.D
Operator   : SL
Acquired    : 06 May 2021 12:13 am using AcqMethod TO-15A VMS-1.8--40C.M
Instrument   : Nutech Deans Switch
Sample Name : Blank 300
Misc Info   : Blank 300
Vial Number: 1
```



结论

使用Nutech 8910/3610预浓缩系统与GC/MS全扫描和或选择性离子扫描，可以一次性快速测试82种目标化合物（TO-15A）。谱图时间短至13.5分钟，18分钟内可以做一个样。所有的QA/QC都符合EPA TO-15A的要求。在SIM模式下，通过预浓缩仪，EPA TO-15A所选化合物的MDL可低至1 ppt，这与TO15A中所列的参考实验室相同。在全扫描模式下，在全扫描下，大多数化合物的MDL均低于0.03 ppb。

本研究应用的方法无需配置Deans Switch（中心切割）模块和FID检测器，为现有仪器没有该等配置的实验室提供了很好的应用实例；与此同时，亦不需要FID相关的氢气、零空气和超纯氮气供应，可显著降低实验室运行成本、提供工作效率。

本研究应用及相应仪器配置方案线性范围（0.5-10ppb）优于同类仪器（1.25-10ppb）。另外的优势是稳定无残留。