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GC/MS Application Note

ENVIRONMENTAL

Quantitation of volatile organic compounds in drinking water using ITEX

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ENVIRONMENTAL

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SCIENTIFIC ANALYTICAL SOLUTIONS

Analysis of volatile organic compounds at low level in water using ITEX combined with a single quadrupole GC-MS

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System Configuration

TriPlus RSH

ITEX-Tool with Tenax GR Trap

Trace 1310 Gaschromatograph

ISQ (Single Quadrupole MS)

PTV injector with Tenax Liner

MEGA column 624 MS 60 m x 0.32 mm 1.8 µm

Chromeleon 7.3.1 Chromatography Data System

Goal

Demonstration of a routine analytical method for the quantitation of volatile organic compounds in drinking water, using the TriPlus RSH ITEX system along with a Thermo Scientific™ ISQ™ 7000 MS system coupled with a Thermo Scientific™ TRACE™ 1310 Gas Chromatograph (GC) and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS). Method linearity and detection limits were assessed to evaluate method performance.

Sample Preparation

Three working standards (0.1 µg/ml, 1 µg/ml and 10 µg/ml) were prepared in methanol. The calibration curve was prepared from 0.01 µg/l to 5 µg/l (parts per billion or ppb) for all compounds by adding 1 to 5 µl working standard in 10 ml pure water. Internal standards (Toluene D8 and 1,2-Dichloroethane D4) were prepared in methanol at concentration of 1 µg/ml, after which 5 µl was then mixed with each 10 ml sample.

Instrument Method

The injection was performed as a Large Volume Injection with solvent split to prevent air and moisture entering the system. The components are trapped on the Tenax liner and can be desorbed in splitless mode for good sensitivity.

TriPlus RSH ITEX Conditions	
Incubation	
Agitator temperature	70°C
Incubation time	20 min
Extraction	
Extraction volume	1000 µl
Aspiration flow	100 µl/s
Dispensation flow	100 µl/s
Pull-up delay	1 s
Number of strokes	50
Prefill ratio	10%
Syringe	
Syringe temperature	75°C
Trap pre cleaning temperatue	250°C
Trap pre cleaning time	120 s
Trap extraction temperatue	35°C
Trap purge time	0 s
Trap post cleaning temperatue	250°C
Trap post cleaning time	200 s
Injection	
Injection depth	30 mm
Injection speed	40 µl/s
Injection temperature	280°C
Penetration speed	50 mm/s
Injection aspiration flow rate	50 µl/s
Injection aspiration delay	1 s

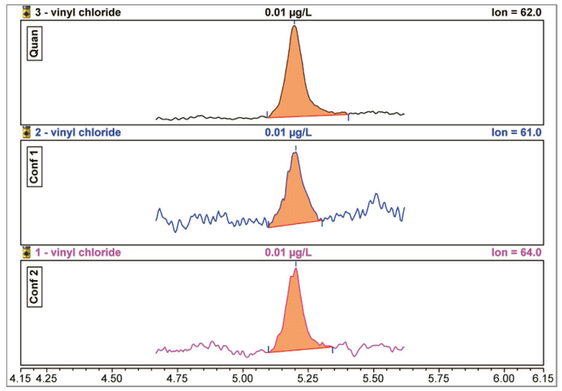
GC Conditions	
Column	MEGA 624 MS 60 m x 0.32 mm x 1.8 µm
Carrier gas	Helium, 1.5 ml/min
Oven profile	35°C, 1.5 min 2.5°C/min 50°C 5°C/min 200°C 20°C/min 240°C, 1 min
Inlet PTV	Injection: 30°C, solvent split 0.67 min 6ml, Transfer: 14.5°C/min 280°C 20 min Cleaning; 14.5°C/min 300°C 5 min Split 50 ml
Inlet PTV Liner	Tenax Liner

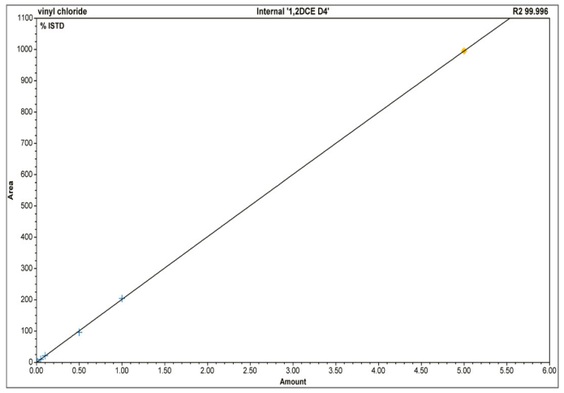
MS Conditions SIM Mode		
Temperature	Transferline 220°C, Source 280°C	
Component	RT (min)	Masses
dichlorodifluoromethane	4.6	85, 87
chloromethane	5.0	50, 52
vinyl chloride	5.2	61, 62, 64
bromomethane	5.8	94, 96
chloroethane	6.0	64, 66
trichlorofluoromethane	6.6	101, 103
ethene, 1,1-dichloro-	7.5	61, 96, 98
methylene chloride	8.4	49, 84, 86
ethylene, 1,2-dichloro-, (E)-trans	9.0	61, 96, 98
MTBE	9.0	73, 74
1,1-dichloroethane	9.9	63, 65, 83
ethylenes, 1,2-dichloro-, (Z)-cis	11.0	96, 98
2,2-dichloropropane	11.1	77, 79
bromochloromethane	11.5	49, 95, 130
chloroform	11.6	47, 83, 85
ethanes, 1,1,1-trichloro-	12.1	97, 99
1-propene, 1,1-dichloro-	12.6	75, 77, 110
carbon tetrachloride	12.6	67, 102
1,2DCE D4	12.6	117, 119
1,2-dichloroethane	12.8	62, 98
benzene	12.9	52, 77, 78
trichloroethylene	14.4	95, 130, 132
1,2-dichloropropane	14.8	63, 76
dibromomethane	15.0	93, 95, 173.8
bromodichloromethane	15.3	83, 85, 129
1-propene, 1,3-dichloro-, CIS	16.5	75, 110
toluene D8	17.2	98, 100
toluene	17.4	65, 91, 92
1-propene, 1,3-dichloro-, trans	17.8	110, 112
1,1,2-trichloroethanes	18.2	83, 97, 132
propane1,3-dichloro	18.7	76, 78
tetrachloroethylene	18.9	129, 131, 166
dibromochloromethane	19.2	79, 127, 129
dibromoethane	19.5	107, 109
chlorobenzene	20.9	51, 77, 112
1,1,1,2-tetrachloroethanes	21.1	117, 131, 133
ethylbenzene	21.3	77, 91, 106
m/p-xylenes	21.7	77, 91, 106
o-xylene	22.8	78, 103, 104
styrenes	22.8	91, 106
bromoform	23.1	91, 172.8, 251.8
isopropylbenzene	24.0	77, 105, 120
1,1,2,2-tetrachloroethanes	24.6	83, 85, 168
1,2,3-trichloropropane	24.7	110, 112
benzene, bromo-	24.8	156, 157
benzene, propyl-	25.3	91, 92, 120
2-chlorotoluene	25.5	91, 126, 128
4-chlorotoluene	25.8	91, 126, 128
benzene, 1,3,5 -trimethyl-	25.9	105, 119, 120
benzene, tert-butyl	26.9	91, 119, 134
benzene, 1,2,4-trimethyl-	27.0	105, 119, 120
benzene, (1-methylpropyl)-	27.5	91, 105, 134
benzene, 1,4-dichloro-	27.6	111, 146, 148
p-cymene	27.8	91, 119, 134
benzene, 1,3-dichloro-	27.9	111, 146, 148
benzene, 1,4-dichloro- D4	28.8	150, 152
benzene, 1,2-dichloro-	28.8	111, 146, 148
benzene, n-butyl	28.9	91, 92, 134
propanes, 1,2-dibromo-3-chloro	30.8	75, 155, 157
benzene, 1,2,4-trichloro	33.3	180, 182, 184
naphthalenes	34.0	102, 128, 129
1,3-butadiene, 1,1,2,3,4,4-hexachloro	34.0	189.9, 224.8, 261.9
bezene, 1,2,3-trichloro	34.7	180, 182

Linearity and Sensitivity

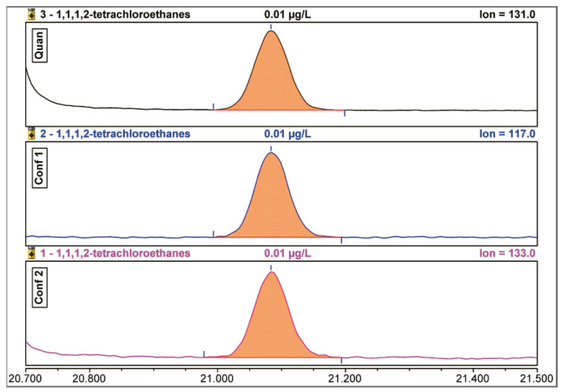
The calibration range of 0.01 µg/l to 5 µg/l was assessed for all compounds. The figures below demonstrate the quantitation of some tricky components at 0.01µg/L in a VOC standard and the calibration curve. More details shown in the table below.

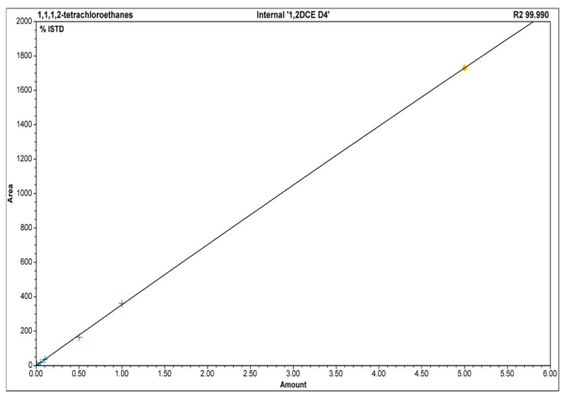
3 - vynil chlorid



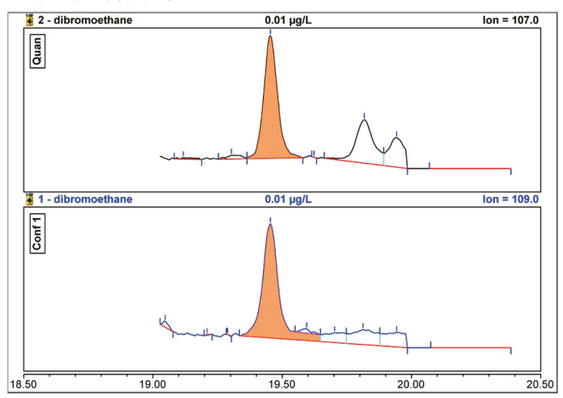


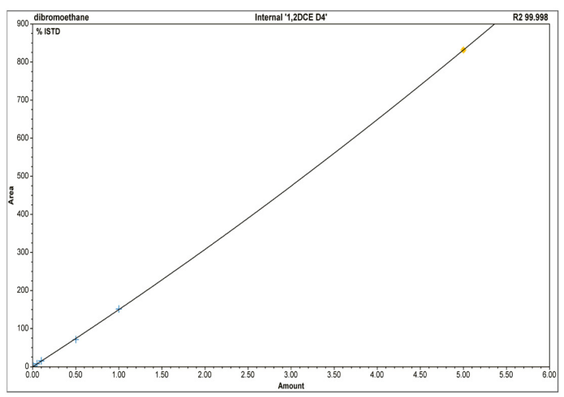
3 - 1,1,1,2-tetrachloroethanes



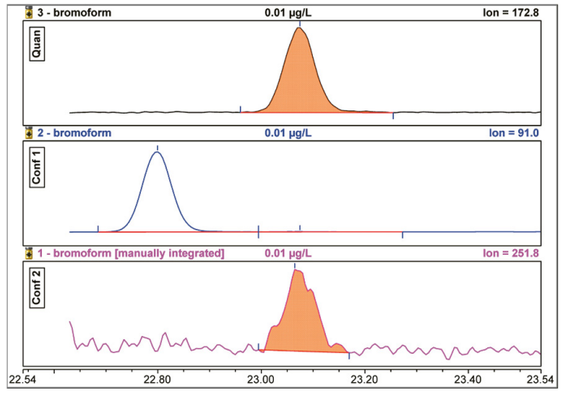


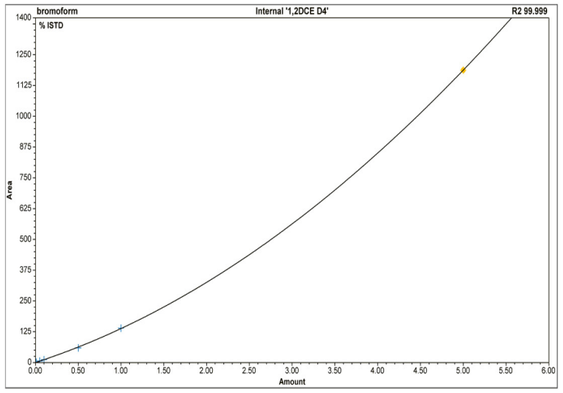
2 - dibromoethane



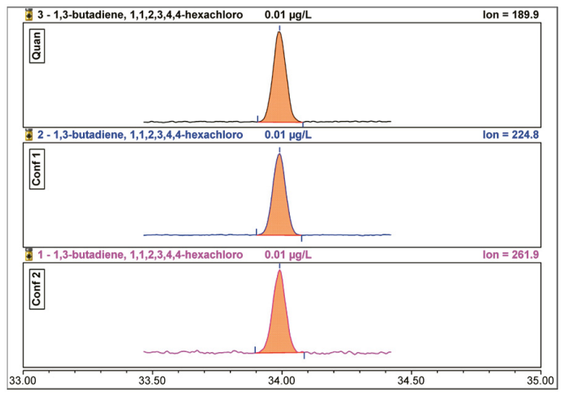


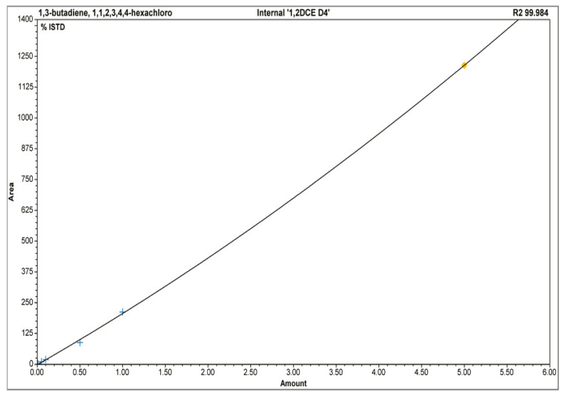
3 - bromoform





3 - 1,3-butadiene, 1,1,2,3,4,4-hexachloro





Discussion

The quantitation limit strongly depends on the ambient contamination in the lab and the chemicals used. The quantification limit of 0.01 µg/l is reachable for nearly all measured components. The limit of 0.05 µg/l is reachable for all components. MTBE and dichloromethane could not be measured due to a very significant contamination in the lab, but the detection of 0.02 µg/l is realistic.

Peak Name	Retention Time min	Quantitation Ion	R-Squared %	Cal.Type	Stand.Meth.
dichlorodifluoromethane	4.6	85	99.941	Quad	Internal 1,2DCE D4
chloromethane	4.93	50	99.993	Quad	Internal 1,2DCE D4
vinyl chloride	5.2	62	99.996	Quad	Internal 1,2DCE D4
bromomethane	5.8	94	99.955	Quad, WithOffset	Internal 1,2DCE D4
chloroethane	5.99	64	99.821	Quad	Internal 1,2DCE D4
trichlorofluoromethane	6.52	101	99.998	Quad	Internal 1,2DCE D4
ethene, 1,1-dichloro-	7.51	61	99.891	Quad	Internal TolueneD8
ethylene, 1,2-dichloro-, (E)-trans	9.04	98	99.999	Quad	Internal 1,2DCE D4
1,1-dichloroethane	9.85	83	99.989	Quad	Internal 1,2DCE D4
ethylenes, 1,2-dichloro-, (Z)-cis	11.03	96	100.000	Quad	Internal 1,2DCE D4
2,2-dichloropropane	11.03	79	99.988	Quad, WithOffset	Internal 1,2DCE D4
bromochloromethane	11.44	130	100.000	Quad	Internal 1,2DCE D4
chloroform	11.59	83	99.961	Quad, WithOffset	Internal 1,2DCE D4
ethanes, 1,1,1-trichloro-	12.17	99	99.998	Quad	Internal 1,2DCE D4
1-propene, 1,1-dichloro-	12.54	110	99.998	Quad	Internal 1,2DCE D4
carbon tetrachloride	12.58	117	99.993	Quad, WithOffset	Internal 1,2DCE D4
1,2-dichloroethane	12.81	62	99.977	Quad	Internal 1,2DCE D4
trichloroethylene	14.34	95	99.986	Quad	Internal 1,2DCE D4
1,2-dichloropropane	14.74	63	99.999	Quad	Internal 1,2DCE D4
dibromomethane	14.93	173.8	99.999	Quad	Internal 1,2DCE D4
bromodichloromethane	15.27	83	99.999	Quad	Internal 1,2DCE D4
1-propene, 1,3-dichloro-, CIS	16.45	75	99.989	Quad	Internal 1,2DCE D4
toluene	17.36	65	99.985	Quad, WithOffset	Internal TolueneD8
1-propene, 1,3-dichloro-, trans	17.74	110	99.974	Quad	Internal 1,2DCE D4
1,1,2-trichloroethanes	18.14	97	99.997	Quad	Internal 1,2DCE D4
propane1,3-dichloro	18.63	76	99.998	Quad	Internal 1,2DCE D4
tetrachloroethylene	18.82	129	99.999	Quad	Internal 1,2DCE D4
dibromochloromethane	19.11	127	99.996	Quad	Internal 1,2DCE D4
dibromoethane	19.45	107	99.998	Quad	Internal 1,2DCE D4
chlorobenzene	20.91	112	99.992	Quad	Internal 1,2DCE D4
1,1,1,2-tetrachloroethanes	21.08	131	99.990	Quad	Internal 1,2DCE D4
ethylbenzene	21.28	106	99.992	Quad, WithOffset	Internal TolueneD8
m/p-xylenes	21.61	77	99.991	Quad, WithOffset	Internal TolueneD8
styrenes	22.79	104	99.997	Quad, WithOffset	Internal TolueneD8
o-xylene	22.8	106	99.999	Quad, WithOffset	Internal TolueneD8
bromoform	23.07	172.8	99.999	Quad	Internal 1,2DCE D4
isopropylbenzene	24.01	105	99.998	Quad, WithOffset	Internal TolueneD8
1,1,2,2-tetrachloroethanes	24.56	83	99.949	Quad	Internal 1,2DCE D4
1,2,3-trichloropropane	24.74	112	99.999	Quad	Internal 1,2DCE D4
benzene, bromo-	24.78	156	99.999	Quad, WithOffset	Internal 1,2DCE D4
benzene, propyl-	25.28	91	99.997	Quad, WithOffset	Internal TolueneD8
2-chlorotoluene	25.47	128	99.998	Quad, WithOffset	Internal TolueneD8
4-chlorotoluene	25.79	128	99.999	Quad, WithOffset	Internal TolueneD8
benzene, 1,3,5 -trimethyl-	25.86	120	99.998	Quad, WithOffset	Internal TolueneD8
benzene, tert-butyl	26.81	134	99.996	Quad, WithOffset	Internal TolueneD8
benzene, 1,2,4-trimethyl-	26.93	105	99.993	Quad, WithOffset	Internal TolueneD8
benzene, (1-methylpropyl)-	27.42	105	99.992	Quad, WithOffset	Internal TolueneD8
benzene, 1,4-dichloro-	27.59	146	99.993	Quad, WithOffset	Internal TolueneD8
benzene, 1,3-dichloro-	27.82	146	99.994	Quad, WithOffset	Internal TolueneD8
p-cymene	27.83	134	99.992	Quad, WithOffset	Internal TolueneD8
benzene, 1,2-dichloro-	28.79	146	99.995	Quad, WithOffset	Internal TolueneD8
benzene, n-butyl	28.92	91.1	99.992	Quad	Internal TolueneD8
propanes, 1,2-dibromo-3-chloro	30.74	156.9	99.993	Quad	Internal 1,2DCE D4
benzene, 1,2,4-trichloro	33.31	180	99.992	Quad, WithOffset	Internal TolueneD8
naphthalenes	33.91	128	99.993	Quad, WithOffset	Internal TolueneD8
1,3-butadiene, 1,1,2,3,4,4-hexachloro	33.99	189.9	99.984	Quad, WithOffset	Internal 1,2DCE D4
bezene, 1,2,3-trichloro	34.66	180	99.990	Quad, WithOffset	Internal TolueneD8

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