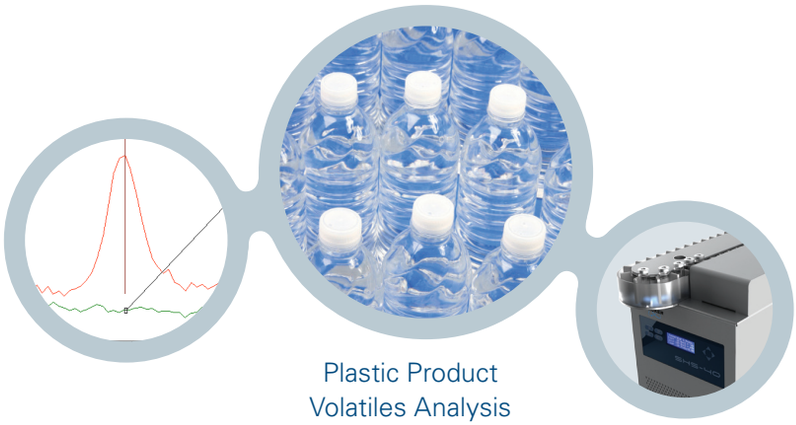


Plastic Product
Volatiles Analysis

Application Notes #283078

Direct Analysis of Finished Plastic Products by Heated Head Space and GC/MS

A sample of raw or finished material is directly analysed for volatile components using an automated head space auto sampler. Little or no sample preparation is required, making it an ideal technique for high throughput quality control testing in manufacturing. When operated in a synchronous SIM/Scan mode, the Bruker SCION SQ single quadrupole mass spectrometer provides quantitative data, along with tentatively identified compounds (non-target compounds).

Introduction

Material testing for outgassing volatile organic chemicals is required in many industries to ensure consumers are not being exposed to harmful contaminants. This is especially important when a material such as a plastic is exposed to excess heat with little or no ventilation. A good example would be plastic materials in a car such as dash boards, which are exposed to very high temperatures in direct sunlight. Volatile chemicals are also common in carpeting and polyvinyl chloride (PVC) piping, and may pose a risk in poorly ventilated homes.

Chlorinated solvents that outgas from materials are generally considered harmful, because they are classified as being carcinogenic or environmental hazards. Other compounds detected may be uncharacterized with little known about their toxicity. Phthalate esters, for example, have been linked to endocrine disruption in some animal species, and regulations are emerging to control these substances in plastic bottles (1).

Experimental

Samples of polypropylene from a car manufacturer and PVC pipe were cut into small pieces and placed directly into 20mL head space vials. About 500mg-1g was added. The Bruker SHS-40 Auto sampler conditions used are cited in Table 1.

Table 1. SHS-40 Sampling Conditions.

Parameter	Set Point
Oven Temp	70°C
Valve/Loop Temp	160°C
Transfer line Temp	125°C
Pressure	500psi
Loop Volume	1mL
PC (incubation) Time	30 min.
GC/MS Run Time	20 min.
Shake option	ON

Figure 1. Bruker SHS-40 Headspace Autosampler (left) with SCION GC/MS.

The SCION GC/MS column, oven program, and injector conditions:

Column:	BR-624ms, 20M x 0.18mm x 1.0um
Injector:	BR-1079, PTV injector with 3.4mm single goose-neck open split liner set at 200°C
Injector split ratio:	1:20
Column flow:	1mL/min
Oven program:	Initial 35°C hold 2 min; program to 170°C at 10°C/min; hold 0; program to 250°C at 50°C/min, hold 1 min, (total run time 17.9 min.)

Results

The polypropylene dash board sample was analysed in full scan. Figure 2 shows all compounds that were tentatively identified using an automated library search against the NIST 08 library.

Figure 2. Full Scan Identification of Compounds in Polypropylene Sample.

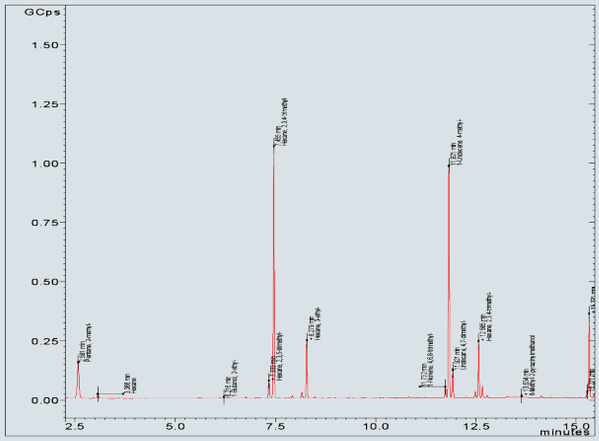


Table 2. Compounds detected in polypropylene dash board sample.

RT (min)	Peak Name	Result Type	Amt	Match	Result	Library
2.581	Pentane, 2-Methyl-	TIC	450552256	1	933	NIST
3.066	Hexane	TIC	43213484	1	903	NIST
6.216	1-Butanol, 2-Ethyl-	TIC	10450404	1	877	NIST
6.766	Toluene	TIC	10159705	1	901	NIST
7.333	Hexane, 2,3,5-Trimethyl-	TIC	139044608	1	925	NIST
7.455	Hexane, 2,3,4-Trimethyl-	TIC	1922249344	1	922	NIST
7.919	2,4-Dimethyl-1-Heptene	TIC	16034614	1	858	NIST
8.16	Nonane, 4-Ethyl-5-Methyl-	TIC	48203884	1	909	NIST
8.279	Hexane, 3-Ethyl-	TIC	418871424	1	921	NIST
11.732	1-Nonene, 4,6,8-Trimethyl-	TIC	85041800	1	876	NIST
11.821	1-Undecene, 4-Methyl-	TIC	1740321664	1	884	NIST
11.921	Undecane, 4,7-Dimethyl-	TIC	208282336	1	883	NIST
12.482	Octane, 2,3,6,7-Tetramethyl-	TIC	48176440	1	878	NIST
12.565	Hexane, 2,3,4-Trimethyl-	TIC	417671808	1	877	NIST
12.657	Hexane, 2,3,4-Trimethyl-	TIC	92484168	1	889	NIST
12.775	Undecane, 4-Methyl-	TIC	21904856	1	870	NIST
13.634	6-Methyl-2-Pyrazinylmethanol	TIC	15546811	1	766	NIST
14.128	Oxalic Acid, Isobutyl Nonyl Ester	TIC	11080139	1	885	NIST
15.271	1-Lodo-2-Methylundecane	TIC	40731856	1	882	NIST
15.321	Heptadecane, 2,6,10,14-Tetramethyl-	TIC	638716608	1	911	NIST

Table 3. Synchronous SIM/SCAN parameters.

Compound Name	Retention Time (RT)	RT Window	Scan Mode, Ions monitored	Dwell Time (ms)
Vinyl Chloride	3.10	1.0	SIM, 62, 64	49
1,2-Dichloroethane	4.67	1.0	SIM, 62, 64	49
Full Scan	NA	2.0-17.9	Full (m/z 35-300)	300

Table 4. Tentatively Identified compounds in PVC sample.

RT (min)	Peak Name	Result Type	Area	Amt	Match	Result	Library
1.184	1-Methyldodecylamine	TIC	6.79E+07	67918424	1	735	NIST
1.283	Hydrazinecarboxamide	TIC	6.61E+06	6613686	1	762	NIST
1.389	Ethyne, Fluoro-	TIC	546473	546473	1	665	NIST
2.159	Acetone	TIC	4.23E+06	4234303	1	867	NIST
3.116	Thiirane	TIC	4.68E+08	4.68E+08	1	907	NIST
3.583	Butane, 2-Nitro-	TIC	1.32E+07	13169822	1	760	NIST
5.659	Pentanal	TIC	5.34E+06	5341163	1	796	NIST
5.705	2-Propenoic Acid, 2-Methyl-, Methyl Este	TIC	5.40E+06	5398963	1	806	NIST
5.807	Acetaldehyde	TIC	5.83E+06	5825911	1	830	NIST
7.752	Hexanal	TIC	2.89E+07	28887902	1	886	NIST
9.732	Heptanal	TIC	6.03E+06	6025094	1	867	NIST
9.848	Benzene, (1-Methylethyl)-	TIC	6.89E+06	6891179	1	853	NIST
10.159	2,2,7,7-Tetramethyloctane	TIC	2.41E+07	24139264	1	872	NIST
10.348	1-Butanol, 3,3-Dimethyl-	TIC	5.30E+06	5302461	1	747	NIST
10.421	2,2,7,7-Tetramethyloctane	TIC	1.39E+07	13884072	1	846	NIST
10.516	Heneicosane, 11-(2,2-Dimethylpropyl)-	TIC	8.77E+06	8773806	1	824	NIST
10.657	Hexanal, 2-Ethyl-	TIC	5.78E+06	5782036	1	776	NIST
10.763	Tetradecane, 1-Iodo-	TIC	1.00E+07	9995009	1	793	NIST
10.787	Pentane, 2,3,4-Trimethyl-	TIC	5.01E+06	5005334	1	764	NIST
10.885	Pentanoic Acid, 1,1-Dimethylpropyl Ester	TIC	3.35E+06	3354330	1	733	NIST
11.25	Heneicosane, 11-(2,2-Dimethylpropyl)-	TIC	2.04E+07	20381632	1	890	NIST
11.409	Pentane, 3-Ethyl-2,2-Dimethyl-	TIC	8.05E+06	8048495	1	896	NIST
11.543	Octane, 2,4,6-Rrimethyl-	TIC	4.56E+07	45579500	1	867	NIST
11.802	Oxalic Acid, Isobutyl Nonyl Ester	TIC	6.71E+06	6708442	1	850	NIST
11.88	Pentane, 3-Ethyl-2,2-Dimethyl-	TIC	2.68E+07	26790618	1	886	NIST
11.919	Heneicosane, 11-(2,2-Dimethylpropyl)-	TIC	2.79E+06	2789403	1	795	NIST
11.983	Oxalic Acid, Isobutyl Octyl Ester	TIC	2.44E+07	24356798	1	886	NIST
12.224	Hexane, 2,4-Dimethyl-	TIC	2.94E+07	29438408	1	817	NIST
12.554	Hexane, 1,1-Dichloro-3-Methyl-	TIC	8.90E+06	8901146	1	656	NIST
13.033	Ethanone, 2,2-Dihydroxy-1-Phenyl-	TIC	1.32E+07	13228981	1	879	NIST
13.282	Nonanal	TIC	2.19E+07	21900612	1	865	NIST
13.367	Benzenemethanol, .Alpha.,.Alpha.-Dimethy	TIC	8.96E+06	8960782	1	727	NIST
13.63	6-Methyl-2-Pyrazinylmethanol	TIC	9.97E+06	9966499	1	741	NIST
14.874	2-Decen-1-Ol	TIC	1.31E+07	13070243	1	888	NIST

The PVC sample was run in SIM/Scan mode. Figure 3 shows detection of 1,2-dichloroethane, a target compound with qualifier ion.

Figure 3. SIM/Scan analysis of PVC sample.

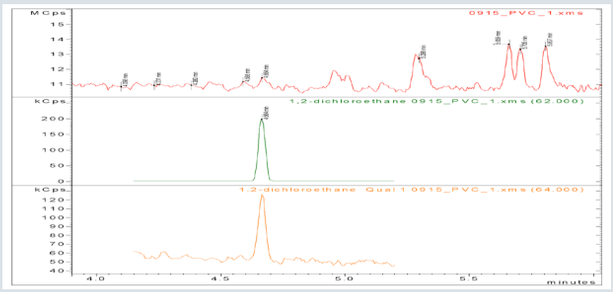
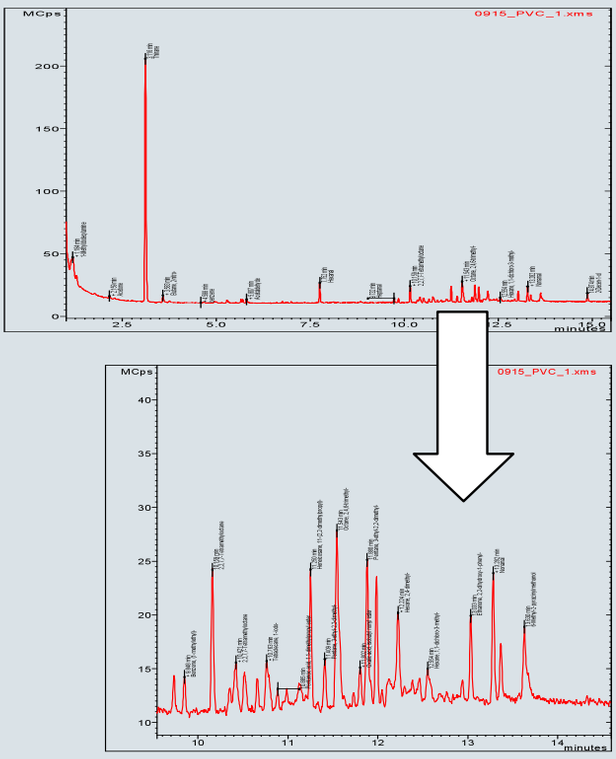


Figure 4. RIC of PVC sample.



The compounds were tentatively identified and are listed in Table 3. Lower display is magnification of peaks eluting between 10 and 14 min.

Conclusion

The Bruker SHS-40 headspace coupled with the SCION GC/MS is an excellent tool for qualitiative and quantitative identification of volatile compounds in raw materials. Using SIM, compounds can be selectively quantitated at very low concentrations. Full scan data can be interrogated for TICs and used for quality control "fingerprints".

References

(1) Developmental Effects of Endocrine-disrupting Chemicals in Wildlife and Humans; T. Colborn, F. S. vom Saal, and A. M. Soto, W. Alton Jones Foundation, Washington, DC 20037.

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