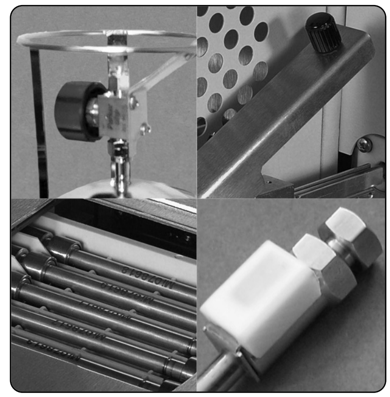
Laboratory-Based Air Monitoring Solutions from Agilent Technologies

Application Compendium



Abstract

Thermal desorption (TD) combined with GC/MS is a powerful technique for measuring organic vapors in air. Applications are wide-ranging, from environmental research to industrial hygiene. Agilent Technologies, a global leader in GC/MS, has partnered with Markes International, the world's leading supplier of analytical TD instrumentation, to provide state-of-the-art air analyzers for both environmental and workplace health and safety applications. This collection of Application Briefs demonstrates the wide range of analyses that can be successfully addressed with the Agilent TD-GC/MS systems.

Contents

Agilent Technologies

Introduction

Thermal desorption (TD) combined with GC/MS is now recognized as the technique of choice for measuring organic vapors in air. Agilent Technologies is a premier provider of TD-GC/MS systems for both environmental air monitoring and personal exposure monitoring. Systems are available to suit a wide range of air sampling media, including pumped sorbent tubes, canisters, axial/radial diffusive (passive) samplers and Tedlar bags. All offer full compliance with relevant international standard methods - ISO/EN 16017, ASTM D6196, U.S. EPA Methods TO-17 and TO-15, EN 14662 (parts 1 and 4), and NIOSH 2549.

Air monitoring applications are diverse and demanding, with analyte concentrations ranging from hundreds of ppm to sub-ppt, with the lowest-level target compounds often present in the most in complex and contaminated matrices. Agilent's products for air analysis offers the flexibility and performance to meet these challenges. Compatible applications include:

- Environmental research - global background pollution levels, stratospheric pollution, and other applications
- Air toxics in urban, industrial, and indoor air
- Stack and fugitive emissions
- Soil gas and vapor intrusion
- Odor monitoring
- Workplace/occupational hygiene - inhalation exposure and breath analysis for biological exposure

Innovation in air sampling and TD technology

Agilent Technologies has partnered with Markes International, the world's leading supplier of analytical thermal desorption (TD) instrumentation, to provide state-of-the-art products for air analysis. The combined systems offer best available "two-stage" TD performance, with none of the inconvenience of liquid cryogen. Two-stage systems provide better chromatographic resolution and sensitivity by focusing the desorbed species prior to introduction into the GC/MS.

With its uniquely wide application range and field-proven reliability, series 2 UNITY provides the TD platform in every Agilent air monitoring system. Optional components include the series 2 ULTRA with capacity for up to 100 sorbent tubes and/or the Canister Interface Accessory (CIA) for automated canister analysis.

The TD technology offered by Agilent air monitoring systems features many pioneering technical innovations. SecureTD-Q (quantitative sample re-collection for repeat analysis or validation) and electronic tube tagging are key examples that offer real benefits to busy commercial and regulatory labs.

"... Cryogen-free operation, simple water management, and application versatility (tubes, canisters/bags, ambient air toxics, and soil gas) ensure optimum productivity from your Agilent air monitoring system ..."

High-performance GC/MS

Agilent Technologies has established a proven track record for robust, long lasting high-performance systems and innovative features that ensure exceptional sensitivity and consistent compound identification in the most difficult matrices. The Agilent 7890A/5975C GC/MSD system represents an optimized union of advanced technologies for GC and MS. Built on the fifth generation of Electronic Pneumatic Control (EPC), the 7890A GC delivers analytes to the MSD with unequalled reproducibility. Improved backflush Capillary Flow Technology (CFT) provides increased protection for the MSD source. For the 5975C MSD, stable, maintenance-free, high-sensitivity operation and excellent mass accuracy are the net benefits of three unique components: an exceptionally inert source, a novel triple-axis HED-EM detector, and the industry's only heated, monolithic, quartz mass analyzer.

Monitoring Air Toxics using Canisters and Sorbent Tubes (U.S. EPA Methods TO-15/17)

Application Brief

In response to growing demand for measurement of ppb-level organic air toxics in urban and indoor air, cryogen-free thermal desorption technologies have now been developed that offer an automated method-compliant analytical platform for both tubes and canisters. Applicable GC/MS methods include U.S. EPA Method TO-17 for sorbent tubes and U.S. EPA Method TO-15 for canisters.

For the ultimate in air sampling flexibility - canisters, bags, and sorbent tubes - Agilent Technologies has TD and GC/MS products that comply fully with both Methods TO-15 and TO-17. Systems offer automated sequencing for up to eight canisters or bags, together with manual or automated tube desorption. The configurations incorporate Agilent's world-leading GC/MS system, which is comprised of an Agilent 7890A GC system and an Agilent 5975C Series GC/MSD. Electrically-cooled focusing (no liquid cryogen required), versatile water management, and uniquely efficient trap desorption all combine to minimize analysis costs, optimize uptime, and ensure uncompromised analytical performance, sensitivity, and repeatability. Systems that include automated tube desorption offer unattended operation all weekend, automated re-collection for repeat analysis, and the option of on-board electronic tag read/write for enhanced sample and tube traceability.

Configuration requirements

System configuration comprising: series 2 (ULTRA *50:50*/ISDP-) UNITY-CIA8 with 7890A GC and 5975C GC/MS

Recommended accessories:

Air Toxics Analyzer (ATA) starter kit comprising:

- 2 x ATA/SG focusing trap
- Pk 10 conditioned/capped ATA tubes
- Pk 10 BTX standards on tubes (100 ng level)
- CapLok tool

Agilent J&W DB-624 column: 60 m × 0.32 mm id × 1.8 µm

Available options:

Silcosteel canisters

TubeTAG starter kit

Pk 100 conditioned/capped ATA tubes

He leak detector

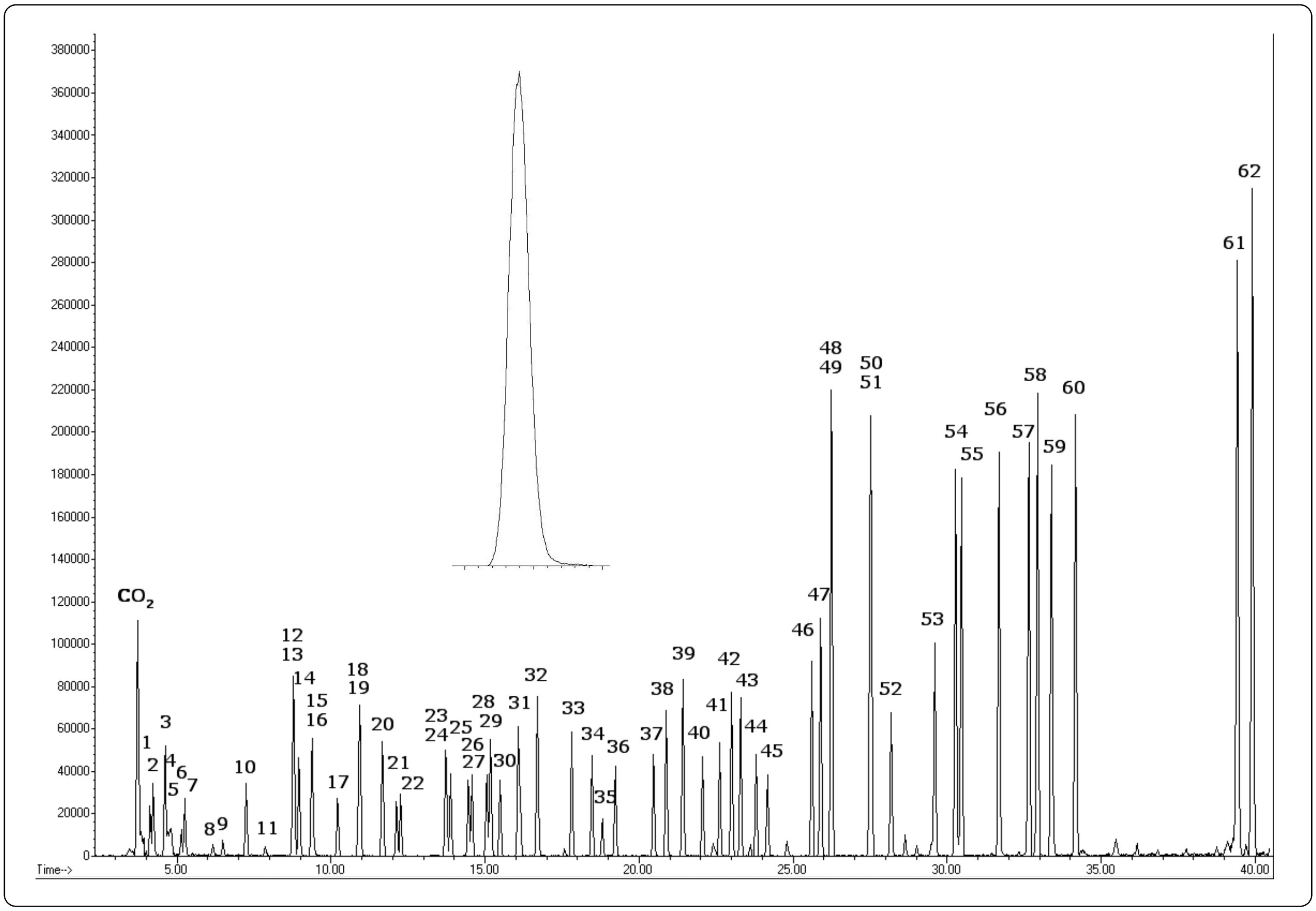
Gas standards for calibration (1ppm)

UNITY 2 routine maintenance kit

Pack 1 or 5 pumps

Sampling and analytical conditions

Sample volume:	100 mL to 1 L
Focusing trap:	25 °C, 40 °C/s to 320 °C (3 min)
Split ratio:	Splitless to 10:1 (depending on concentration)
TD flow path:	140 °C
Carrier gas:	Helium at constant pressure of 10 psi
GC oven:	35 °C (5 min), 5 °C/min to 230 °C
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 200 °C
Full scan:	35–300 *m/z*



1. Propylene
2. Dichlorodifluoromethane
3. 1,2-Dichlorotetrafluoroethane
4. Methyl chloride
5. 1,2,-Dichloroethane
6. 1,3-Butadiene
7. Vinyl chloride
8. Methyl bromide (bromomethane)
9. Chloroethane
10. Trichlorotrifluoroethane (Freon 113)
11. Ethanol
12. 1,2-Dichloroethylene
13. 1,1,2-Trichlorotrifluoroethane
14. Acetone
15. Carbon disulfide
16. Isopropyl alcohol
17. Methylene chloride
18. tert-Butyl methyl ether
19. *n*-Hexane
20. 1,1-Dichloroethane
21. Vinyl acetate
22. cis-1,2-Dichloroethylene
23. Methyl ethyl ketone
24. Ethyl acetate
25. Tetrahydrofuran
26. Chloroform
27. 1,1,1-Trichloroethane
28. Cyclohexane
29. Carbon tetrachloride
30. Benzene
31. *n*-Heptane
32. Trichloroethylene
33. 1,2-Dichloropropane
34. 1,4-Dioxane
35. Bromodichloromethane
36. trans-1,3-Dichloropropene
37. Methyl isobutyl ketone
38. Toluene
39. cis-1,3-Dichloropropene
40. trans-1,2-Dichloroethylene
41. 1,1,2-Trichloroethane
42. Tetrachloroethylene
43. Methyl n-butyl ketone
44. Dibromochloromethane
45. 1,2-Dibromoethane
46. Chlorobenzene
47. Xylene
48. Xylene
49. Xylene
50. Styrene
51. Tribromomethane
52. 1,1,2,2-Tetrachloroethane
53. 1,2,4-Trimethylbenzene
54. 1,3,5-Trimethylbenzene
55. 1-Ethyl-4-methylbenzene
56. Ethylbenzene
57. 1,2-Dichlorobenzene
58. 1,3-Dichlorobenzene
59. alpha-Chloromethylbenzene
60. 1,4,-Dichlorobenzene
61. 1,2,4-Trichlorobenzene
62. Hexachloro-1,3-butadiene

Figure 1. One liter of 62-component standard analyzed in splitless mode. Excellent peak shape ensures sub-100-ppt detection limits for all compounds. Inset shows close-up of peak shape for isopropyl alcohol.

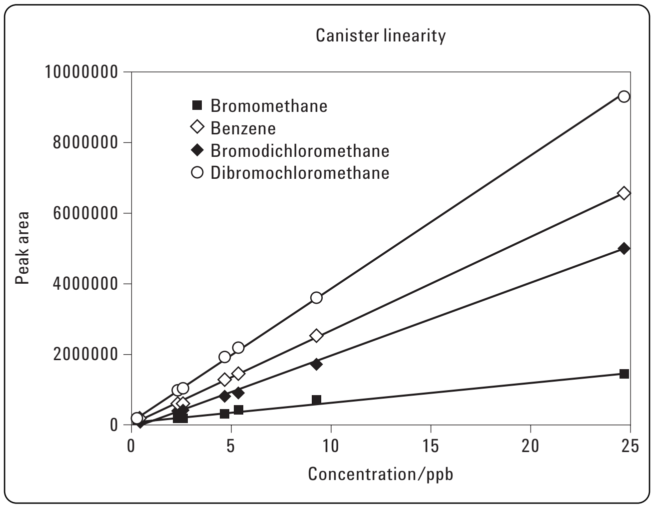


Figure 2. Linearity plots from 0.1 to 25 ppb.

Table 1. System Repeatability Demonstrated using Eight Compounds Covering the Air Toxics Range (n=5)

Compound	%RSD
Dichlorodifluoromethane	2.8
Isopropyl alcohol	4.0
Carbon disulfide	2.8
Dichloroethylene	3.3
Tetrahydrofuran	2.2
Benzene	1.9
Vinyl chloride	4.0
1,2,4-Trichlorobenzene	4.5

Monitoring Hazardous Air Pollutants using U.S. EPA Method TO-17
Application Brief

Volatile organic air toxics are monitored in many industrial, urban, and indoor environments as a measure of air quality. They range in volatility from methyl chloride to hexachlorobutadiene, and include polar as well as nonpolar species. Several national and international standard methods have been developed for air toxics and related applications. These include U.S. EPA Method TO-17, "Determination of volatile organic compounds in ambient air using active sampling onto sorbent tubes." Other relevant standards include EN ISO 16017 and ASTM D6196.

In response to increasing worldwide demand for measurement of ambient air toxics, Agilent Technologies has configured TD-GC/MS systems that operate cryogen-free and in full compliance with Method TO-17. Manual and automated versions are available, with the high-throughput systems offering capacity for up to 100 tubes and automated re-collection for repeat analysis. All systems feature versatile water management and Agilent's world-leading GC/MS system, which is comprised of an Agilent 7890A GC system and an Agilent 5975C Series GC/MSD.

Optional electronic tube tagging offers enhanced traceability for high-throughput labs and field-monitoring studies.

Configuration requirements for TO-17

System configuration comprising: series 2 (ULTRA *50:50/*ISDP-) UNITY with 7890A GC and 5975C GC/MS

Recommended accessories:

Air Toxics Analyzer (ATA) starter kit comprising:

- 2 x ATA/SG focusing trap
- Pk 10 conditioned/capped ATA tubes
- Pk 10 BTX standards on tubes (100 ng level)
- CapLok tool

Agilent J&W DB-624 column: 60 m × 0.32 mm id × 1.8 µm

Available options include:

TubeTAG starter kit

Additional conditioned/capped ATA or universal tubes

He leak detector

UNITY 2 maintenance kit

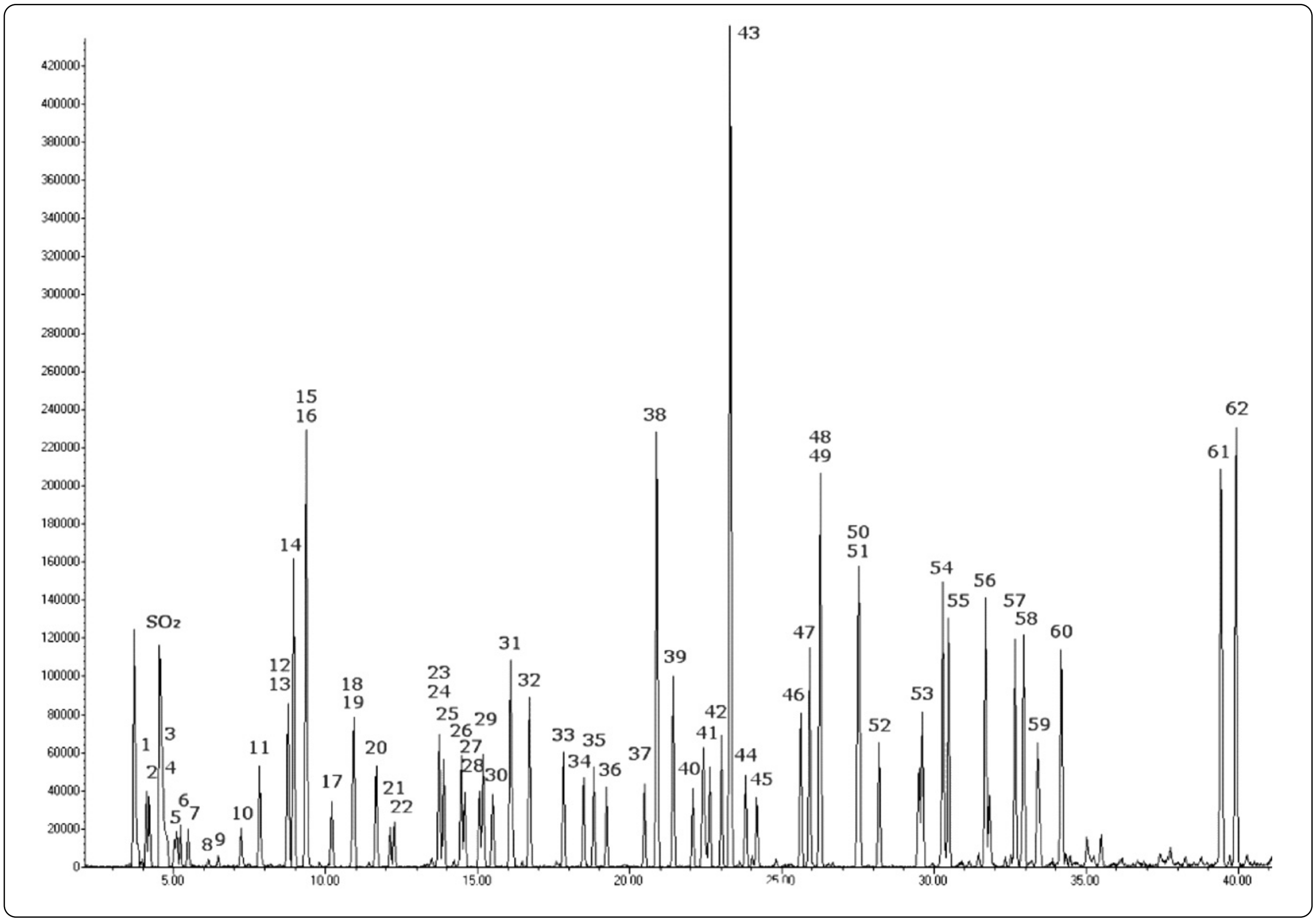
Gas standards for calibation 1ppm

Customized standard reference tubes (100 ng)

Pack 1 or 5 pumps

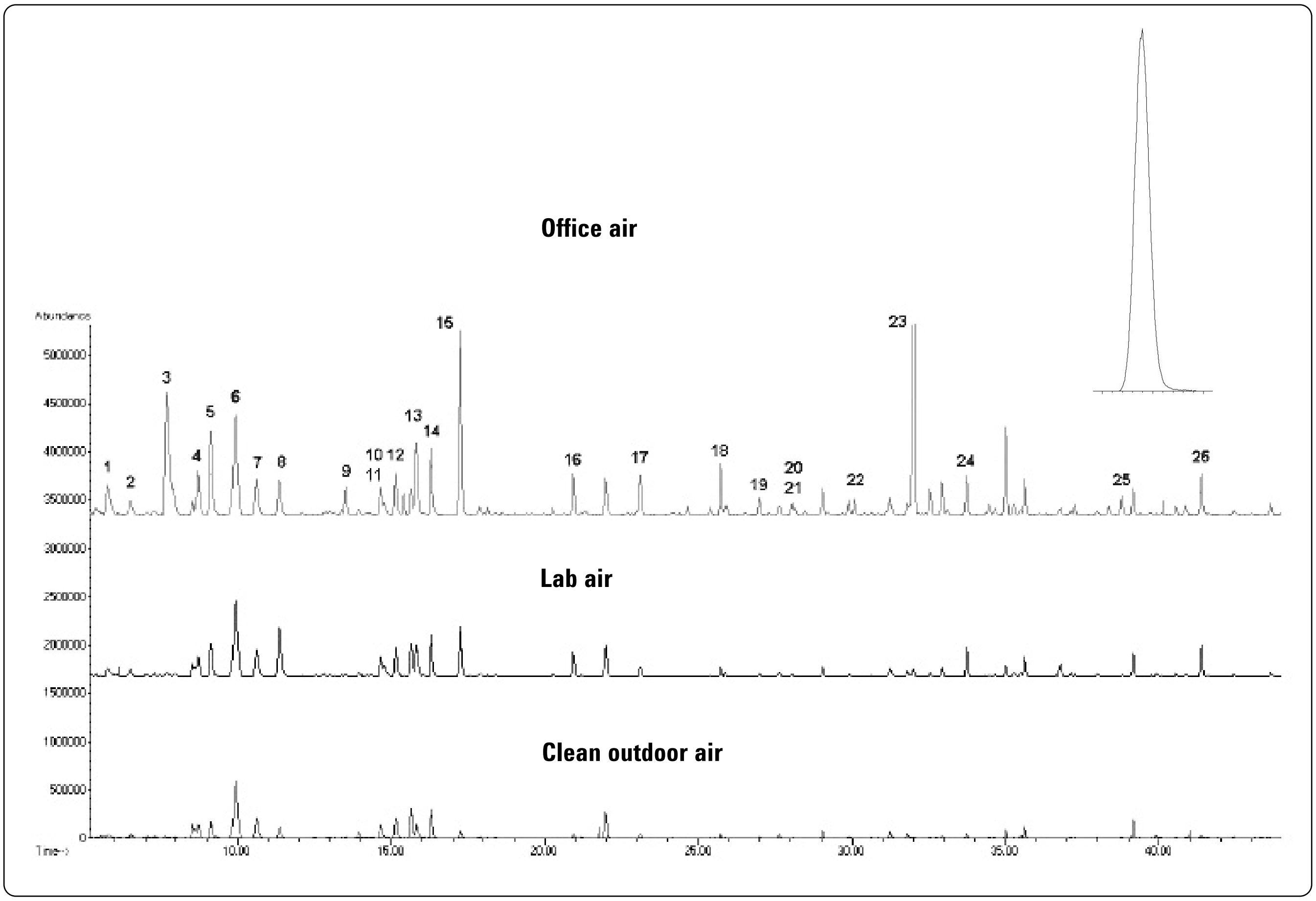
Sampling and analytical conditions

Sample volume:	Typically 1–5 L at 20–50 mL/min with ATA tubes
Tube desorb:	320 °C for 10 min
Focusing trap:	25 °C, 40 °C/s to 320 °C (3 min)
Split ratio:	Splitless to 10:1 (depending on concentration)
TD flow path:	140 °C
Carrier gas:	Helium at constant pressure of 10 psi
GC oven:	35 °C (5 min), 5 °C/min to 230 °C
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 200 °C
Full scan:	35–300 *m/z*



1. Propylene
2. Dichlorodifluoromethane
3. 1,2-Dichlorotetrafluoroethane
4. Methyl chloride
5. 1,2,-Dichloroethane
6. 1,3-Butadiene
7. Vinyl chloride
8. Methyl bromide (bromomethane)
9. Chloroethane
10. Trichlorotrifluoroethane (Freon 113)
11. Ethanol
12. 1,2-Dichloroethylene
13. 1,1,2-Trichlorotrifluoroethane
14. Acetone
15. Carbon disulfide
16. Isopropyl alcohol
17. Methylene chloride
18. tert-Butyl methyl ether
19. *n*-Hexane
20. 1,1-Dichloroethane
21. Vinyl acetate
22. cis-1,2-Dichloroethylene
23. Methyl ethyl ketone
24. Ethyl acetate
25. Tetrahydrofuran
26. Chloroform
27. 1,1,1-Trichloroethane
28. Cyclohexane
29. Carbon tetrachloride
30. Benzene
31. *n*-Heptane
32. Trichloroethylene
33. 1,2-Dichloropropane
34. 1,4-Dioxane
35. Bromodichloromethane
36. trans-1,3-Dichloropropene
37. Methyl isobutyl ketone
38. Toluene
39. cis-1,3-Dichloropropene
40. trans-1,2-Dichloroethylene
41. 1,1,2-Trichloroethane
42. Tetrachloroethylene
43. Methyl n-butyl ketone
44. Dibromochloromethane
45. 1,2-Dibromoethane
46. Chlorobenzene
47. Xylene
48. Xylene
49. Xylene
50. Styrene
51. Tribromomethane
52. 1,1,2,2-Tetrachloroethane
53. 1,2,4-Trimethylbenzene
54. 1,3,5-Trimethylbenzene
55. 1-Ethyl-4-methylbenzene
56. Ethylbenzene
57. 1,2-Dichlorobenzene
58. 1,3-Dichlorobenzene
59. alpha-Chloromethylbenzene
60. 1,4,-Dichlorobenzene
61. 1,2,4-Trichlorobenzene
62. Hexachloro-1,3-butadiene

Figure 1. One liter of 62-component air toxics standard (1 ppb) collected using ATA tubes and analyzed in splitless mode.



1. Methanol
2. 2-Methylbutane
3. Ethanol
4. Acetone
5. Isopropyl alcohol (IPA)
6. 2-Methylpentane
7. 3-Methylpentane
8. Hexane
9. Ethyl acetate
10. 2-Methylhexane
11. Cyclohexane
12. 3-Methylhexane
13. Heptane
14. Acetic acid
15. 1-Methyl-2-propanol
16. Toluene
17. Hexanal
18. Xylene
19. Xylene
20. alpha-Pinene
21. Cyclohexanone
22. alpha-Myrcene
23. *d*-Limonene
24. Phenol
25. Menthol
26. 2-Phenoxyethanol

Figure 2. Splitless analysis of three real air samples collected using universal tubes. Inset shows close-up of isopropyl alcohol, demonstrating excellent peak shape. Detection limits: below 0.1 ppb (100 ppt) for all compounds.

Soil Gas and Vapor-Intrusion Assessments Using a Specific "Soil Gas" Sorbent Tube

Application Brief

When investigating fuel-contaminated sites, soil gas measurements are used to assess the potential risk to human health from vapor intrusion into nearby buildings, and to identify fuel sources for mitigation and liability management. Measurement of the most toxic individual compounds (for example, benzene and naphthalene) and characterization of the total petroleum hydrocarbon (TPH) profile are usually both required.

Canisters and tubes have both been used to sample soil gas with subsequent analysis by TD-GC/MS, using procedures that are similar to U.S. EPA Methods TO-15 or TO-17. However, some of the "middle-distillate" fuels (diesel, jet fuel, kerosene, etc.) contain components that fall outside the scope of these methods. While sorbent tubes are compatible with high boilers (up to n-$C_{26/30}$) and facilitate complete recovery in a single TD-GC/MS analysis, canisters are prone to carryover of compounds higher than n-C_{10}. This can compromise results and lead to expensive and time-consuming canister cleaning issues or irreversible canister contamination.

To optimize and simplify this application, Agilent offers hydrophobic "soil gas" sampling tubes, with or without electronic tags, for use with either of the air toxic analyzers described above. Soil gas tubes allow quantitative retention and recovery of a wide range of organic contaminants, including both light and middle distillate fuels. They are ready for immediate reuse post-analysis; no additional conditioning is necessary. Second desorptions of the tubes used for soil gas sampling show no carryover across the volatility range.

Productivity and accuracy at minimal cost

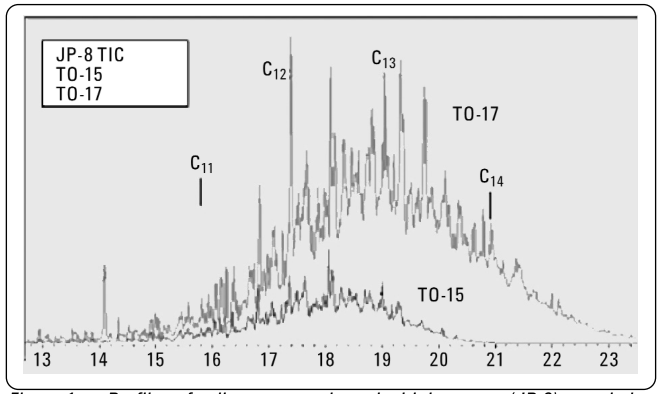


Figure 1. Profiles of soil gas contaminated with kerosene (JP-8) sampled using sorbent tubes (red) and canisters (blue). Data presented courtesy of Air Toxics Ltd., CA, U.S.A.

Configuration requirements

System configuration comprising: series 2 (ULTRA *50:50/*ISDP-) UNITY 2-(CIA 8) with 7890A GC and 5975C GC/MS

Recommended accessories:

Soil gas starter kit comprising:

- 2 x ATA/SG focusing trap
- Pk 10 conditioned/capped soil gas tubes
- Pk 10 BTX standard on tubes (100 ng level)
- CapLok tool

Agilent J&W DB-5 ms column: 30 m × 0.25 mm id × 0.25 µm

Available options include:

TubeTAG starter kit

Pk 100 conditioned/capped soil gas tubes

Pack 1 or 5 pumps

He leak detector

UNITY 2 maintenance kit

Analytical conditions

Hydrophobic soil gas tubes (Tenax/Carbopack X) sampled at 50 mL/min for 5 min

Tube desorption:	300 °C for 5 min
ATA trap:	25 °C, 310 °C (3 min) at maximum rate
Helium carrier:	13 psi, split 50:1 (trap only)
GC oven:	50 °C (1 min), 5 °C/min to 140 °C, 15 °C/min to 300 °C (1 min)
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 280 °C
Full scan:	30–450 *m/z*

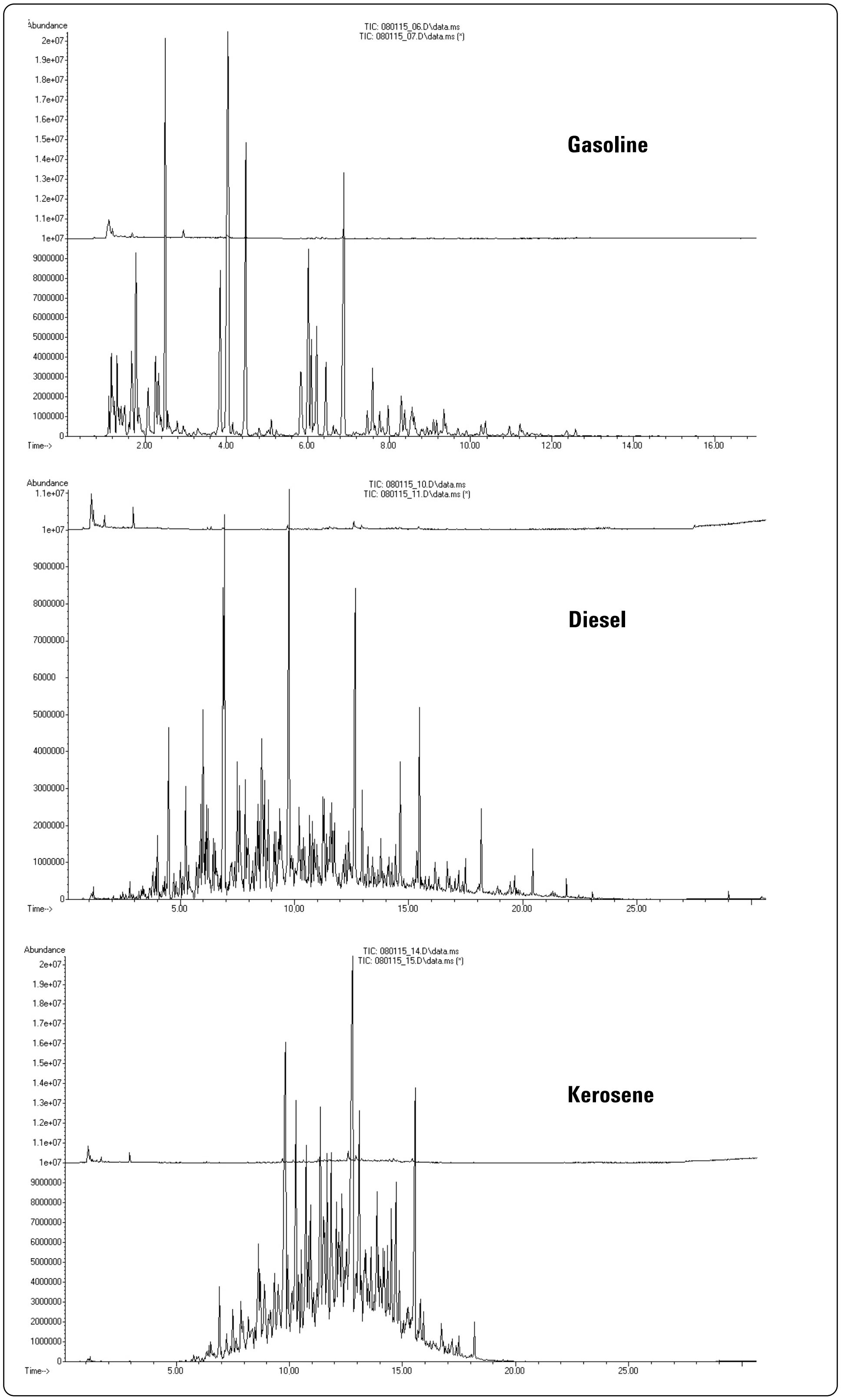


Figure 2.   First and second desorptions of soil gas tubes used to sample fuel vapors in contaminated soil.

Stack Emissions Testing
Application Brief

Stack gases are often aggressive, complex matrices that contain both high and low concentrations of organic vapors. High-level components are typically monitored on-line using continuous emission monitors (CEMs) built into the stack itself, but regulators around the world are increasingly concerned by lower-level emissions of toxic or odorous organic chemicals, which may need to be checked off-line for compliance with site operating permits.

Ultravolatile compounds of interest, such as freons, may be sampled using whole air/gas containers, but most stack-monitoring is carried out using sorbent tubes with subsequent analysis by thermal desorption or solvent extraction and GC/MS. TD is increasingly preferred for stack testing because it eliminates the need for manual sample preparation with toxic solvents such as CS_2, and the associated analytical interference. TD also offers 1000-fold greater sensitivity for trace analytes, while still accommodating concentrations in excess of 100 ppm.

The transfer of stack testing methods from solvent extraction to thermal desorption has been facilitated by the SecureTD-Q (re-collection for repeat analysis) capability of Agilent TD-GC/MS systems. Even manual systems offer SecureTD-Q as standard, and this facility can be automated for single- or double-splitting.

Configuration requirements

System configuration comprising: series 2 (ULTRA *50:50*/ISDP-) UNITY(-CIA8) with 7890A GC and 5975C GC/MS

Recommended accessories:

Pk 10 general purpose sorbent tubes (n-C_5 to n-C_{30}), conditioned and capped

CapLok tool

Spare general purpose focusing trap

Agilent J&W HP-5 MS column: 30 m × 0.25 mm id × 0.25 µm film

Available options include:

Pk 100 conditioned/capped general purpose tubes

TubeTAG starter kit

He leak detector

UNITY 2 maintenance kit

Second ULTRA for automated re-collection of both splits

Sampling and analytical conditions

Time-weighted average or grab sampling of 100 to 1500 mL stack gas (pump or large gas syringe)

Tube desorption:	5 min at 300 °C
Focusing trap:	25 °C to 300 °C (5 min) at maximum rate
Split ratio:	50:1 to 5,000:1 (depending on concentration)
TD flow path:	180 °C
Carrier gas:	Helium at constant pressure of 10 psi
GC oven:	40 °C (5 min), 10 °C/min to 280 °C (5min)
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 200 °C
Full scan:	30–450 *m/z*

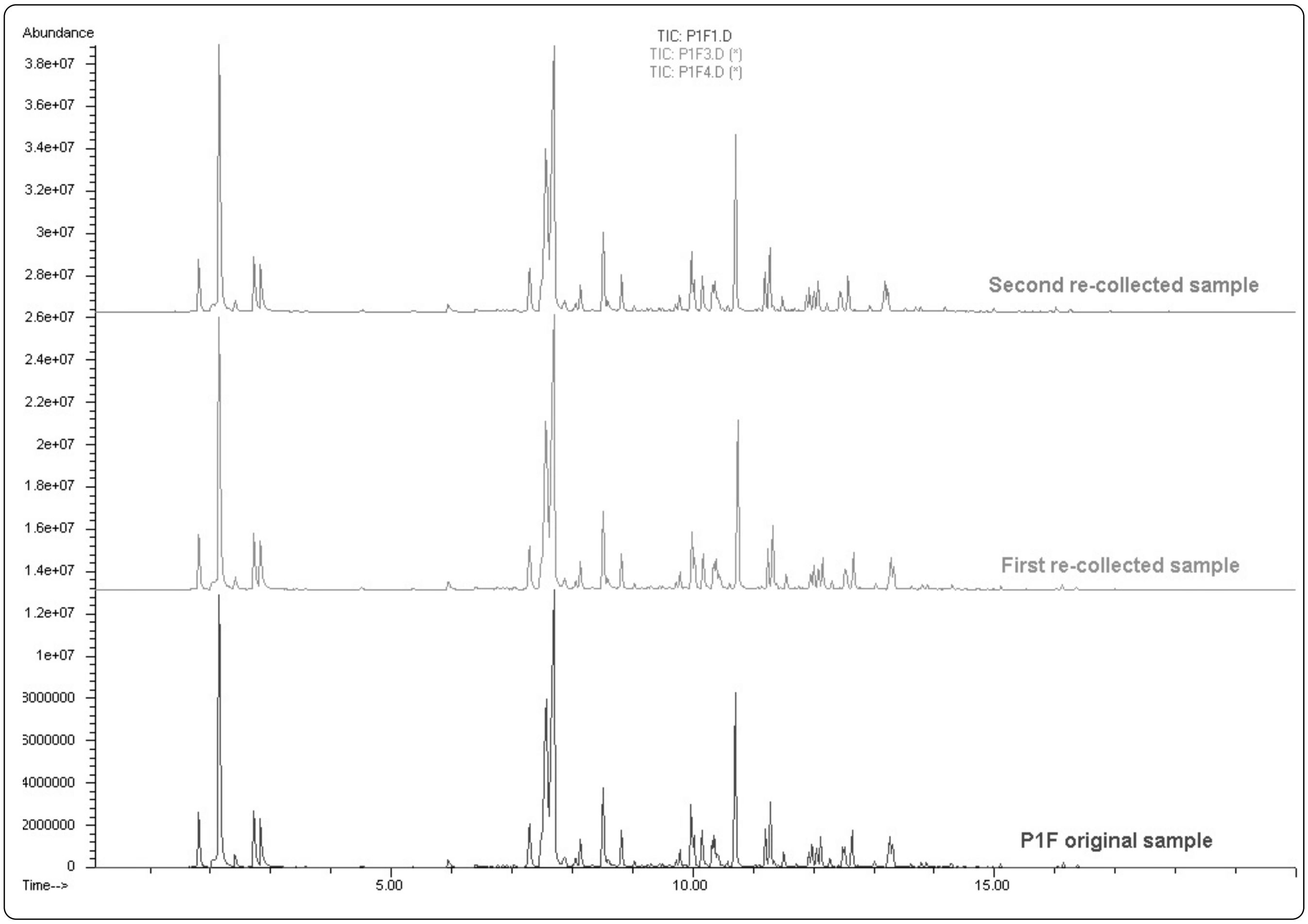


Figure 1. Three repeat analyses of a single 1.5-L stack gas sample analyzed using double splitting. Overall split ratio 3,000:1.

Standard Methods

Early TD methods for stack emissions specified large ("VOST") tubes for stack gas sampling. While these standards have now largely been superseded, they were nevertheless important for demonstrating the principle. More recent regulatory guidance, such as United Kingdom Environment Agency (UK EA) M2 and the latest method developments for stack testing (for example, prEN 13649) specify standard ¼-inch (6.4 mm) or 6 mm od tubes and allow both thermal desorption and solvent extraction options.

Table 1. Data from the Three Repeat Analyses Shown in Figure 1

Compound	Mass (µg) determined Run 1	Run 2	Run 3
MEK	580	583	580
Benzene	0.14	0.18	0.18
Toluene	94	91	93
Ethylbenzene	30	30	29
PGMEA	43	43	43
m/p-Xylene	261	262	258
o-Xylene	13	13	13
DMS	28	28	28
1,2,3-Trimethylbenzene	43	44	42

Occupational Hygiene/Workplace Air Monitoring
Application Brief

Legislation for health and safety in the workplace, such as the European Chemical Agents Directive and the U.S. Occupational Safety and Health Act/Hazard Communication Standard, requires industrial sites that use chemicals to carry out air monitoring and/or personal exposure assessment in the workplace to ensure compliance with regulated limits. Occupational hygiene demands unobtrusive samplers. For this reason, sorbent tubes and cartridges, operated diffusively (passively) or with small personal monitoring pumps, are almost universally preferred for organic vapors.

Inhalation exposure: TD-GC/MS has been widely used for workplace/personal air monitoring for many years, with relevant standard methods including ISO EN 16017, ASTM D6196, ENs 838/1076 and NIOSH 2549. Recent reductions in occupational limits have started to accelerate the move to thermal desorption and away from traditional charcoal tube/CS_2 extraction procedures. Solvent extraction has inherent limitations that include sensitivity (typically 1000-fold less sensitive than TD methods), variable or incomplete extraction efficiency, nonreusable tubes, and solvent issues (such as toxicity, disposal cost, and analytical interference).

Agilent TD-GC/MS products uniquely offer re-collection on both manual and automated configurations, thus allowing repeat analyses and overcoming the traditional "one-shot" limitation of older TD systems. The option of electronic tube-tagging for enhanced sample tube traceability is also of significant benefit to workplace studies.

Breath testing: Routes of exposure to chemicals at work may include skin absorption and ingestion, as well as inhalation. Recent development of TD-GC/MS-compatible breath testing, as a non-invasive alternative to blood or urine sampling, facilitates large-scale biological monitoring for the first time. Relevant applications include shoe workers who are exposed to solvents, staff at dry cleaning facilities, and monitoring medical personnel for exposure to anaesthetics.

Configuration requirements

System configuration comprising: series 2 (ULTRA 50:50/ISDP-) UNITY with 7890A GC and 5975C GC/MSD

Recommended accessories:

Workplace air starter kit (general) comprising:

- Pk 10 conditioned/capped universal tubes
- Pk 10 conditioned/capped Tenax tubes
- Pk 10 Diffusive caps and CapLok tool
- Pk 10 standard tubes (BTX) - 1 µg level
- Spare general purpose focusing trap

Agilent J&W DB-5ms column: 60 m × 0.25 mm id × 0.25 µm film

Available options include:

Pk 10/100 conditioned/capped tubes containing sorbent specific to target analytes

TubeTAG starter kit

Pk 10 Bio-VOC breath samplers

UNITY 2 maintenance kit

Helium leak detector

Pack 1 or 5 pumps

Sampling and analytical conditions

Sampling:	10 L of air sampled onto Tenax tube (or 8-hr diffusive sampling)
Tube desorption:	5 min at 300 °C
Standard trap:	–20 °C to 300 °C (5 min) at maximum rate
Split ratio:	50:1 to 500:1 (depending on concentration))
TD flow path:	160 °C
Carrier gas:	Helium at constant pressure of 20 psi
GC oven:	50 °C (5 min), 10 °C/min to 280 °C
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 200 °C
Full scan:	30–450 *m/z*

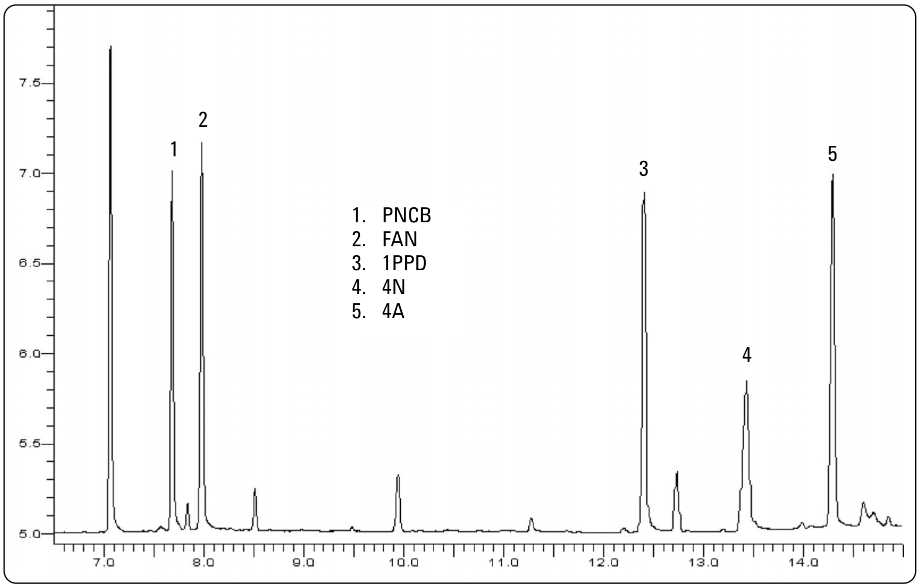


Figure 1. Analysis of reactive amines in workplace air.

Sampling and analytical conditions: breath

Sampling:	Bio-VOC™ breath sampler and Tenax tube
Tube desorption:	5 min at 250 °C
Standard trap:	–25 °C to 300 °C (3 min) at maximum rate
TD flow path:	150 °C
Carrier gas:	Helium at constant pressure of 20 psi
GC oven:	40 °C (5 min), 10 °C/min to 300 °C
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 200 °C
Full scan:	30–450 *m/z*

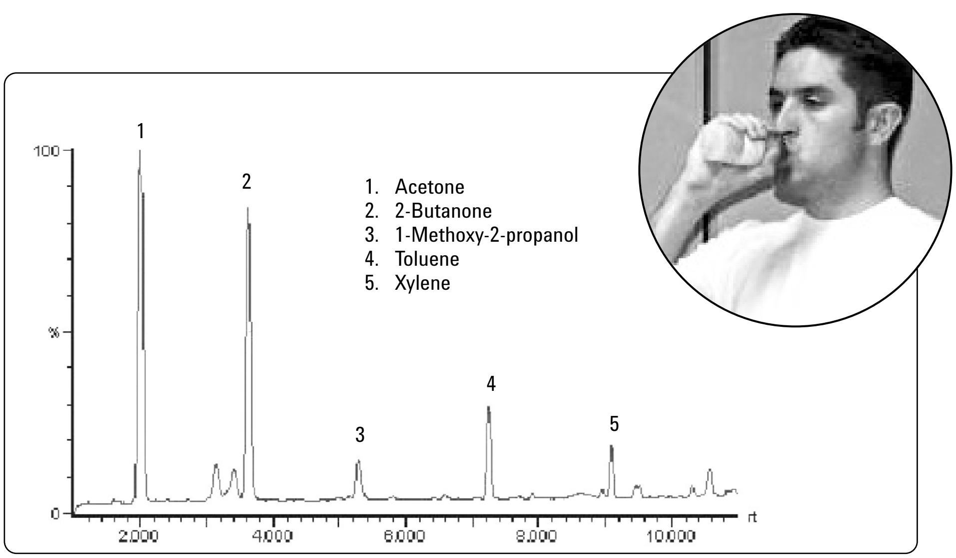


Figure 2. Skin-absorbed solvents in the breath of shoe workers, with sample collected using the Bio-VOC breath sampler and analyzed by TD-GC/MS.

Fugitive Emissions, Odor, and Fence-Line Monitoring
Application Brief

Some waste sites and industrial installations produce toxic and/or odorous organic vapors that can affect the health and quality of life of people who live and work in the immediate vicinity. Relevant regulations include the European Directive 1999/31/EC on the landfill of waste and U.S. EPA activity to control benzene concentrations at the perimeter of major oil refineries under the "Residual Risk" program.

Sorbent tubes provide the most versatile, cost-effective, and unobtrusive air sampling option in this case. Perimeter monitoring is typically best served by long-term diffusive sampling - allowing 1- or 2-week time-weighted average concentrations to be cost-effectively determined for key criteria pollutants such as benzene or naphthalene according to ISO EN 16017, ASTM D6196, and EN 14662-4.

Practical difficulties and the challenging nature of atmospheric conditions on landfill sites including high CO_2, elevated temperatures, high humidity, and contaminated background favor convenient grab sampling. This process entails pulling approximately 100 mL samples of landfill gas through inert tubes using low-cost bellows pumps or a large gas syringe. The United Kingdom Environment Agency (UK EA) guidance document on monitoring trace components in landfill gas complements the general-purpose standards TO-17, ISO EN 16017 and ASTM D6196 for this application.

The versatility of Agilent systems is of direct benefit to this field, with systems uniquely offering complete recovery of compounds ranging in volatility from C_2 to n-C_{40} and the option of low flow-path temperatures for compatibility with thermally labile, odorous compounds.

Configuration requirements

System configuration comprising: series 2 (ULTRA 50:50/ISDP-) UNITY with 7890A GC and 5975C GC/MS

Recommended options for landfill:

Pk 10 sulfur tubes (SilcoSteel, Tenax/Unicarb)

Two “Sulfur” focusing traps

Agilent J&W DB VXR column: 60 m × 0.25 mm id × 1.4 µm film

TubeTAG starter kit

He leak detector

Recommended options for refineries:

Pk 10 tubes (BTX) plus pk 10 tubes (1,3-butadiene)

Pk 10 diffusive caps

Spare general-purpose focusing trap

Agilent J&W HP-5 MS column: 60 m x 0.25 mm x 0.5 µm film

TubeTAG starter kit and He leak detector

Analytical conditions: landfill gas

Grab sampling of 100 mL gas onto “S” tubes

Tube desorption:	5 min at 300 °C
Focusing trap:	25 °C to 300 °C (5 min) at maximum rate
Helium carrier gas:	21.6 psi with a split ratio of 20:1
TD flow path:	120 °C
GC oven:	35 °C (5 min), 5 °C/min to 230 °C
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 200 °C
Full scan:	30–450 *m/z*

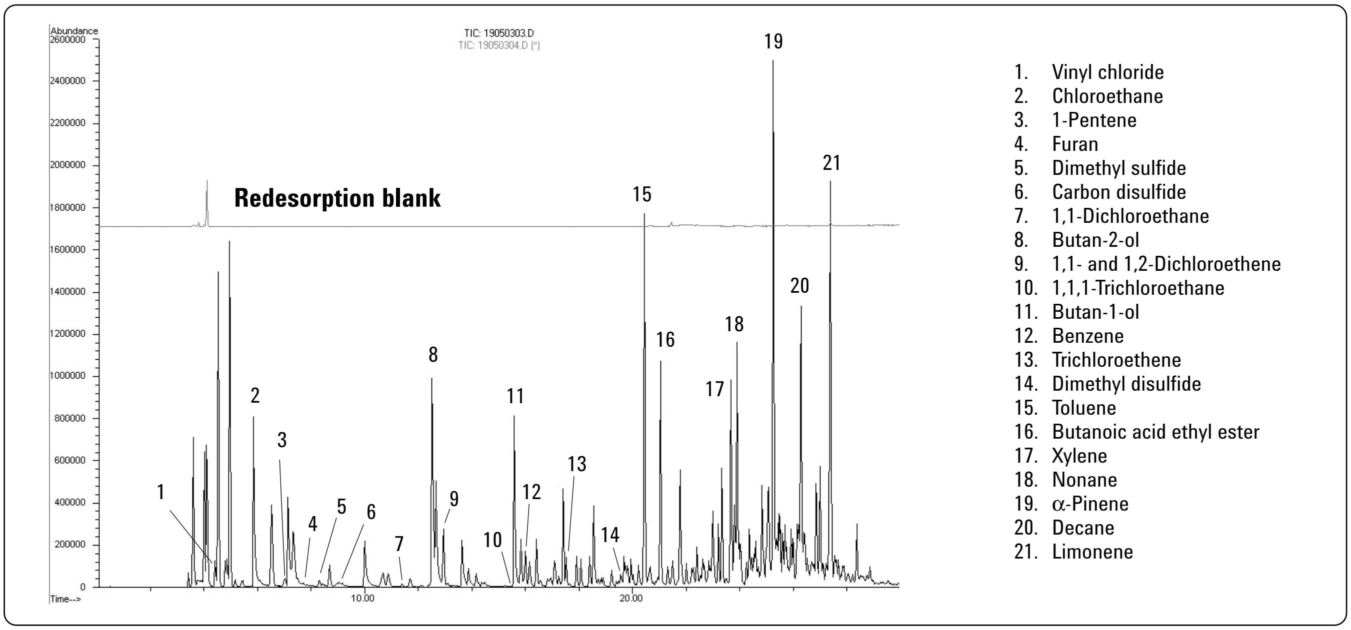


Figure 1. 100 mL landfill gas with trace target analytes and some other major background contaminants identified.

Analytical conditions: refinery perimeter

Sampling:	Two-week diffusive using Carbograph 1 TD tubes
Tube desorption:	5 min at 320 °C
Focusing trap:	Carbon general-purpose, –10 °C to 320 °C
Helium carrier gas:	21.6 psi with a split ratio of 10:1
TD flow path:	150 °C
GC oven:	35 °C (5 min), 5 °C/min to 230 °C
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 200 °C
Full scan:	30–450 *m/z*

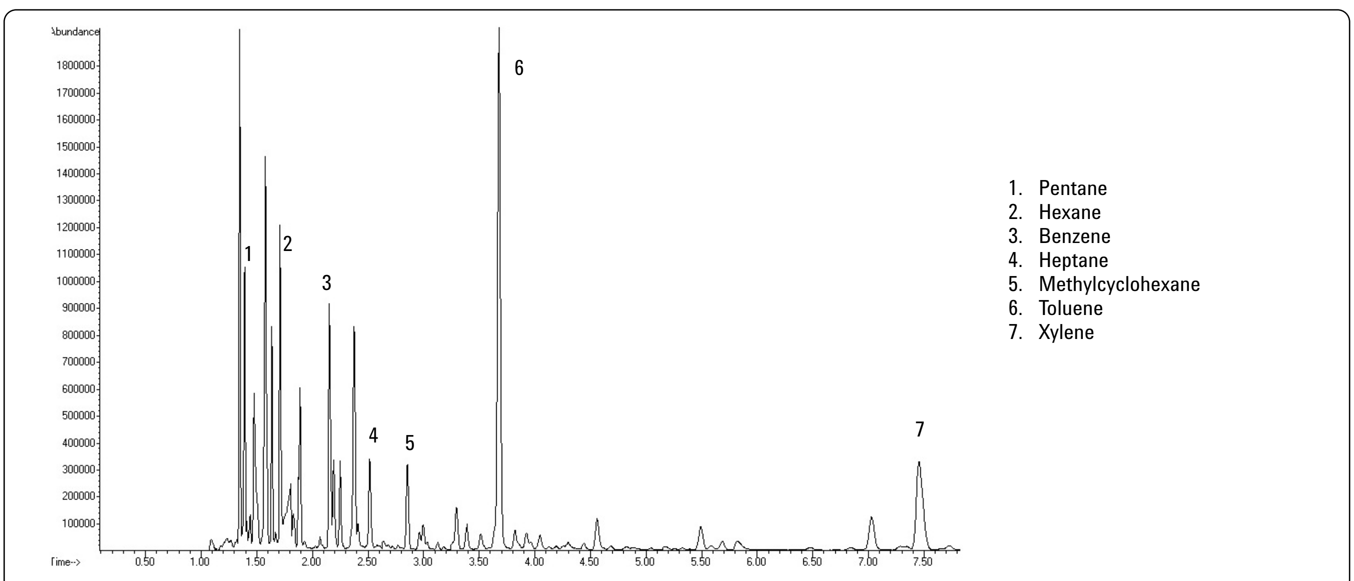


Figure 2. Two-week diffusive monitoring around a refinery perimeter. Volatile organic compounds (VOCs) detected include alkanes plus the aromatics benzene, toluene, and xylene. Reproduced here with the kind permission of BP.

Indoor Air Quality
Application Brief

People in the developed world spend around 90 percent of their time indoors or driving, and are subject to emissions from construction products, furnishings, and trim components from car interiors. Indoor and in-vehicle air quality are therefore key criteria for human health and comfort, especially when the most vulnerable members of society are taken into account.

Indoor air quality (IAQ) has become the focus of even more attention recently, as increased pressure on energy efficiency (for example, the new EC directive on Energy Performance of Buildings) has led to lower rates of indoor air change. Inadequate ventilation may allow chemicals (for example, from construction or cleaning products) to build up in the indoor environment, causing "Sick Building Syndrome" in extreme cases. Similar issues affect confined car cabins.

TD is used extensively for indoor air monitoring and for related applications such as tests of material emissions and tracer gas ventilation tests. IAQ sampling is typically carried out using pumped sorbent tubes per ISO 16000-6, ISO EN 16017, or ASTM D6196. Diffusive sampling provides an ideal option for indoor personal exposure assessment.

Offering best-in-class system sensitivity and simultaneous analysis of volatile and semivolatile organics, Agilent systems are particularly suited to IAQ applications. A wide portfolio of ancillary products complements the capabilities of Agilent's analytical system. Products include low-cost emissions screening tools and innovative data-mining software that enhances identification of trace toxic components in complex chromatographic profiles.

Configuration requirements

System configuration comprising: series 2 (ULTRA *50:50*/ISDP-) UNITY with 7890A GC and 5975C GC/MS

Recommended accessories:

IAQ starter kit comprising:

- Pk 10 conditioned/capped IAQ tubes
- Pk 10 conditioned/capped Tenax TA tubes
- CapLok tool
- 2 x IAQ focusing trap

Agilent J&W DB-5 ms column: 60 m × 0.25 mm id × 0.5 µm

Available options include:

Pk 100 conditioned/capped IAQ or Tenax tubes

TubeTAG starter kit

Pack 1 or 5 pumps

Pk 10 diffusive sampling caps

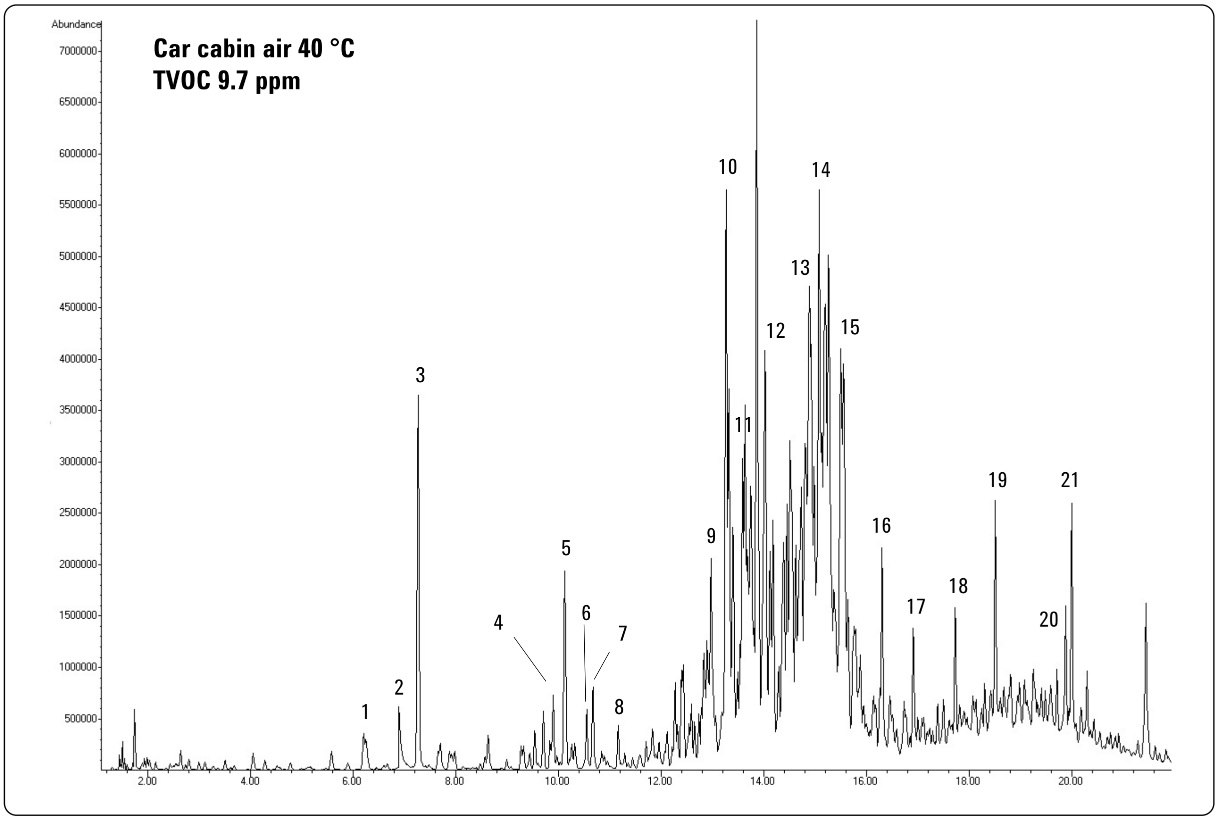
UNITY 2 maintenance kit

µ-CTE Micro-chamber/Thermal Extractor

DRS or TargetView "data mining" software

Analytical conditions: pumped sorbent tube monitoring of car cabin air at 40 °C

Sampling:	2 L sampled at 50 mL/min on IAQ tubes
Tube desorption:	6 min at 275 °C
Focusing trap:	30 °C to 300 °C (3 min) at maximum rate
Double split, total ratio:	100:1
TD flow path:	200 °C
Carrier gas:	Helium at constant pressure of 10 psi
GC oven:	40 °C (5 min), 10 °C/min to 200 °C (1 min)
MS temperatures:	Source 230 °C, quad 150 °C, transfer line 280 °C
Full scan:	45–350 *m/z*



1. Methylcyclohexane
2. N,N-DMF
3. Toluene
4. Ethylbenzene
5. *m*- and *p*-Xylene
6. Styrene
7. *o*-Xylene
8. *n*-Nonane
9. Trimethylbenzene
10. *n*-Decane
11. Dimethylbenzylamine
12. C_{11}
13. $C_{11/12}$ isomers
14. C_{12}
15. C_{13}
16. 2-(2-Butoxy-ethoxy)ethanol
17. Dodecane
18. Silyl ester
19. *n*-C_{13}
20. Copaene
21. *n*-C_{14}

Figure 1. Two liters of air from the cabin of a small car, showing a complex range of VOCs and high total-VOC levels.

Sampling and analytical conditions: diffusive sampling for indoor air quality studies

Sampling:	12 hour diffusive sampling of indoor air, outdoor air, and personal exposure using axial sorbent tubes
Tube desorption:	5 min at 300 °C
Focusing trap:	25 °C to 300 °C (5 min) at maximum rate
Split ratio:	10:1
TD flow path:	160 °C
Carrier gas:	Helium at constant pressure of 10 psi
GC oven:	35 °C (5 min), 5 °C/min to 230 °C
MS temperatures:	Source: 230 °C, quad 150 °C, transfer line 280 °C
Full scan:	45–450 *m/z*

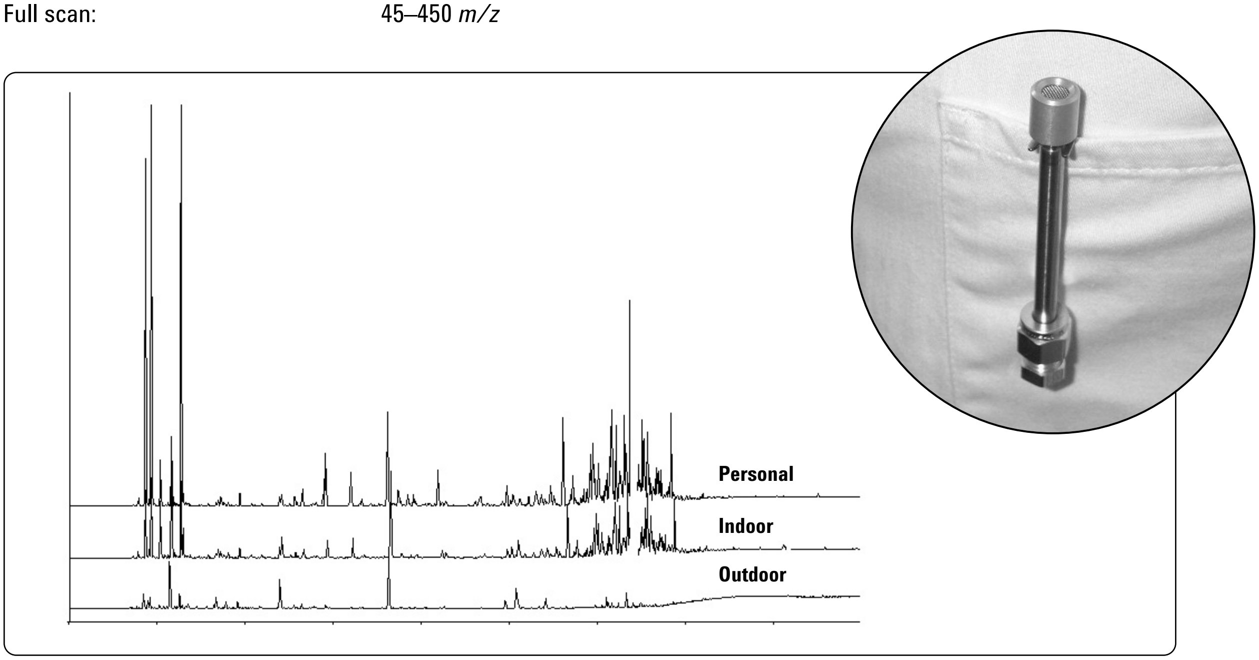


Figure 2. Poor indoor air quality and high personal exposure levels in this home were ultimately linked to a diesel car parked in an integral garage.

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