High-Speed Gas Chromatography

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Introduction

High speed gas chromatography (GC) has been the subject of an increasing number of papers in both journals and at conferences in recent years. These articles have progressed from concept papers mainly detailing the necessary instrumental modifications to the potential applications of high speed GC.

This article is focused on reported applications of what may be regarded as traditional high speed GC, i.e. short, wall-coated open tubular columns (generally of internal diameter (i.d.) <0.32 mm) operated at a higher than optimal carrier gas flow rate. The total chromatographic separation time is required to be less than 3–4 min. This eliminates some work on polychlorinated biphenyl separations where run times are approximately six times faster than conventional GC, but still take around 10 min. Other areas in high speed GC, such as rapid packed column separations, porous layer open tubular columns and the newly emerging imaging technique of solvating GC using packed columns will not be addressed. In addition, only those articles which specifically relate to the use of high speed GC for a particular analysis have been considered. The separation of a large number of compounds by high speed GC has been demonstrated in many research articles, but many of the mixtures separated are not specific to any particular problem or truly relevant to real world analysis.

High speed GC is still mainly an academic research technique. The number of published applications is small and review articles or books detailing these applications are nonexistent. For this reason this article draws mainly on journal papers.

High speed GC is not a new analysis technique. The first true high speed separations involving capillary columns were carried out nearly four decades ago by Desty. Although this clearly demonstrated that separations in seconds were possible, the instrumental demands that high speed GC makes upon the gas chromatograph made it unsuitable for general use at that time. In the following years several notable research groups around the world have made excellent progress both in solving some of the instrumental problems and in clarifying the theoretical considerations involved with high speed GC. In particular, the major contributions of Annino and Guiochon to theoretical developments and the research groups of both Cramers and Sacks for instrumental advances deserve specific mention.

There are several reasons for this slow transfer of high speed GC from the research laboratory to the analytical laboratory. One is the reluctance of application laboratories to substitute a new untried procedure for a known analytical method, even when the new procedure offers major time savings. New methods frequently encounter this hurdle and as more literature reports on high speed GC applications appear, this barrier should slowly crumble.

Another barrier, which will be harder to overcome, is the problem of sample preparation. The advantages of a very fast chromatographic separation are negated if the sample preparation step takes an order of magnitude or more longer than the separation. Further basic research is required in the sample preparation area to bring these two stages closer inline with each other. The workers who have pioneered high speed GC research in the last decade are already incorporating faster sample preparation steps into their research and it is likely that the coming years will see the progress needed in this area.

To date, most high speed GC applications have been in the analysis of volatile organic chemicals (VOCs) in either air or water. Other applications include chlorinated pesticides, polyaromatic hydrocarbons (PAHs), and the use of high speed GC to determine solvent purity. Each of these areas will be considered in more detail below.

Volatile Organic Chemicals

Air

Near real-time results may be particularly beneficial for a system where a centrally located high speed gas chromatograph is connected to a network of sampling lines. The high speed GC analysis of VOCs must be able to detect compounds at and below regulatory levels, such as the threshold limit value (TLV).

Original work utilized a valve loop system for sampling, but the use of mechanical valves may be a problem for an automated system. Valves may require periodic maintenance, or may change the sample through interaction with valve surfaces. In addition, changing the sample size requires either changing the loop or the use of several loop cycles. Ice formation which may plug the collection tube, resulting from water vapour in the air is another area of concern. However, studies to date have not shown this to be a problem.

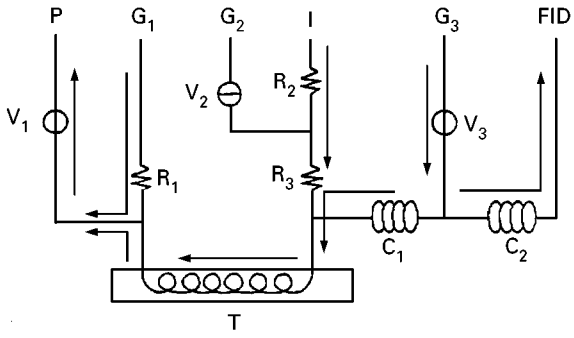

Figure 1 Complete high speed GC system with direct atmospheric sample collection. T, Cold trap system; V, valves; R, restrictors; P, vacuum pump; I, atmospheric pressure source; C, separation column; G, gas source. Arrows show flow directions during sample collection. (Reproduced with permission from Akard and Sacks, 1994.)

An alternative approach to achieve the detection limits required is a technique termed cryointegration. In this method, VOCs from an air sample, usually contained in a sampling loop, are cryofocused in a liquid nitrogen-cooled metal tube. Thus, dilute samples can be preconcentrated prior to high speed analysis. Resistive heating of the metal tube is used to ensure a narrow injection plug compatible with high speed GC requirements. This has been incorporated with a reversed-flow sample collection system. **Figure 1** shows the complete high speed GC system for an air monitor using this type of set-up.

The vacuum pump pulls sample and carrier gas through the cryotrap for the sample collection period. This time period can be adjusted to allow for more or less sample to be collected as required. After collection, the sample is introduced on to the separation column by heating the metal cryotrap tube.

The positioning of the sample near the column end of the cryotrap reduces band broadening and the potential for sample decomposition. The decreased bandwidth leads to narrower, taller peaks, which in turn improve the limit of detection (LOD). Although water vapour in the sample condensing in the cryotrap is not a problem, water vapour from the flame ionization detector (FID) is. For this reason, a gas source attached midway through the separation column has been used to prevent water vapour from the detector reaching the cryotrap.

Figure 2 shows representative chromatograms obtained using gasbag samples. The sampling flow rate is constant and about 0.3 mL min^{-1} once steady-state flow is achieved. There is, of course, a trade-off between reducing the LOD and loss of real-time data as the sampling time increases.

Tables 1 and **2** show the limit of detection values obtained using the conventional loop system and the reverse-flow collection system respectively. It can be seen that, as the sampling time increases with the cryointegration system, the LOD values approach those obtained using a fixed 1 mL loop. One would expect that further increases in sampling time would reduce the LOD even further. The disadvantage with further increases in sampling time is the loss of real-time information. These LOD values are already below regulatory or guideline concentrations such as the TLV and a further reduction in LOD would seem unnecessary.

Water

The apparatus used for VOC analysis in water by high speed GC is essentially the same as that used for the air analysis work. This is because, as sample preparation is conducted by a static headspace equi-

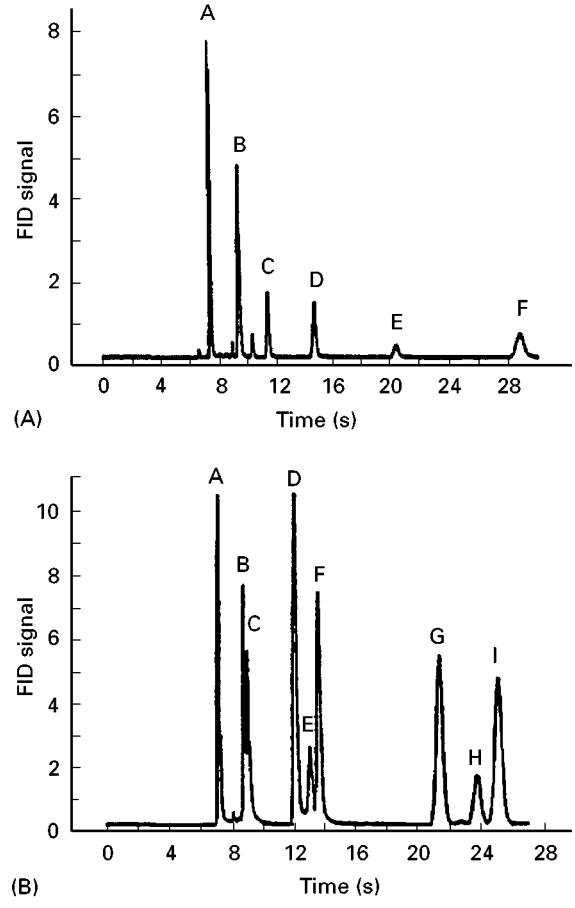

Figure 2 High speed chromatograms using gasbag samples and the reverse-flow collection instrument: (A) six-component mixture; (B) nine-component mixture. Peaks in (A) A, *n*-pentane; B, *n*-hexane; C, benzene; D, *n*-heptane; E, toluene; F, *n*-octane. Peaks in (B) A, *n*-hexane; B and C, isomers of 2-hexene; D, 1-heptene; E and F, isomers of 2-heptene; G, 1-octene; H and I, isomers of 2-octene. (Reproduced with permisssion from Akard and Sacks, 1994.)

Table 1 Limits of detection and quantitation measured for 13 common organic compounds

Compound	*LOD (p.p.b.v.)*	*LOQ (p.p.b.v.)*
Pentane	2	7
Hexane	3	10
Heptane	4	13
Octane	5	17
Benzene	8	27
Toluene	2	7
o-Xylene	50	170
m-Xylene	1	3
Dichloromethane	<1	<3
Chloroform	<1	<3
Tetrachloroethylene	<1	<3
1,1,2,2 Tetrachloroethane	2	7
1,2,3 Trichloropropane	<1	<3

All values are expressed in parts per billion by volume and are based on a sample volume of 1 mL. Values reported as <1 required extrapolation below sample volumes that were actually tested.

LOD, sample mass producing a peak height equal to a blank pus three standard deviations of the noise; LOQ, the sample mass producing a peak height equal to a blank plus 10 standard deviations of the noise. Reproduced with permission from Mouradian *et al*. (1991).

librium method, the actual sample analysed is an air sample.

Detection limits using an FID were found to be $<10\ \mu g\ L^{-1}$ for benzene, toluene, ethylbenzene and the xylenes (BTEX compounds). Again, by increasing the injection loop volume, these detection limits could be reduced further. Real samples from ground water obtained near a leaking underground storage tank have been analysed and preliminary comparisons to more established methods made. Full validation of the high speed method has not been carried out.

Table 2 Statistical data for air analysis using reverse-flow sample collection with two different sampling periods

Component	*Correlation coefficient*	*Log–log slope*	*LOD (p.p.b.)*	
			20	*110*
Benzene	0.993	0.99	26	4.6
n-Heptane	0.997	1.04	37	6.9
Toluene	0.999	1.07	25	4.0
Octane	0.995	1.07	47	8.4
p-Xylene	0.999	1.09	29	4.4

Detection limits (LOD) for a signal-to-noise ratio of 3.0 for both the 20 s and the 110 s sampling times. Reproduced with permission from Akard and Sacks (1994).

An alternative method for aqueous sample analysis combines solid-phase microextraction (SPME) with fast GC. The SPME extraction is conducted on the headspace above a water sample in a sealed vial. The analytes are then rapidly desorbed in a specially made injection port into a commercially available portable gas chromatograph. Separation of the BTEX compounds is achieved in less than 30 s. The photoionization detector for this work was not optimized for fast separations. Modification of the detector internal volume is required to reduce band broadening and provide performance suitable for use with high speed GC without the use of make-up gas. As the headspace above a sample is extracted with the SPME fibre, the method is easily adapted to soil sample analysis for VOCs.

One reason for the lack of published applications dealing with VOC analysis in water may be the large disparity between chromatographic analysis time and sample preparation time. Having a chromatographic time scale of 20 s (**Figure 3**), as is the case with this

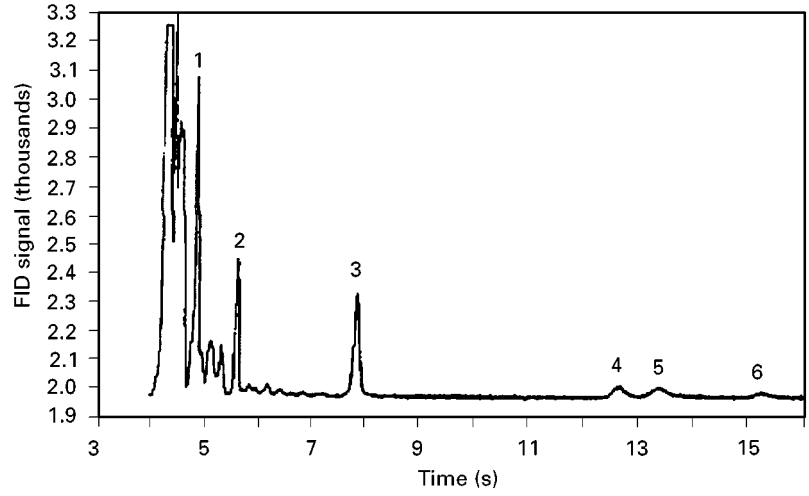

Figure 3 High speed chromatogram of the static headspace above a sample of gasoline-contaminated ground water. Peak identification. 1, methyl *t*-butyl ether (2370 $\mu g\ L^{-1}$); 2, benzene (97 $\mu g\ L^{-1}$); 3, toluene (109 $\mu g\ L^{-1}$); 4, ethylbenzene (2 $\mu g\ L^{-1}$); 5, co-eluting *m*- and *p*-xylenes (12 $\mu g\ L^{-1}$); 6, *o*-xylene ($<0.3\ \mu g\ L^{-1}$). (Reproduced with permission from Wang *et al*., 1991.)

BTEX analysis, has little advantage when the sample preparation time is between 20 and 60 min.

Pesticides

Due to concerns regarding adverse health effects resulting from pesticide residues in food or run-off from fields into the water supply, a large number of analytical methods have been published concerned with pesticide analysis. With the increase in studies linking pesticide residues to adverse human health effects comes a concurrent increase in the number of samples required to be analysed for pesticides.

Several approaches to speeding up pesticide analysis have been taken. One approach utilized the two-dimensional chromatographic approach for a comprehensive analysis of up to 17 pesticides. The two-dimensional approach has the advantage that if the two stationary phases have completely different retention mechanisms for the analytes then a truly orthogonal separation is achieved.

The instrumentation in the two-dimensional approach housed both GC columns (first column 2 m × 250 μm i.d., second column 0.80 m × 100 μm i.d.) in a single GC oven connected via a home-made thermal desorption modulator. The modulator collects the eluent from the first column and releases it on to the second column with a cycle time of 2.5 s. Thus, analysis in the first dimension is 4 min and 5 s in the second dimension. **Figure 4** shows the chromatogram obtained in the first dimension without any separation in the second dimension. Poor peak resolution is clearly seen.

Quantitation is achieved with one of two internal standards (IS). Using a FID, LOD values are between 1.8 and 3.8 pg of pesticide on-column and the relative standard deviation (RSD) of the response (in volume counts pg^{-1}) is less than 10% for all four pesticides reported.

This method has been used for a spiked human serum sample after supercritical fluid extraction. Although one of the IS peaks overlapped with a matrix component, quantitation was still possible using the other IS. This overlap can clearly be seen in **Figure 5**. The method allows for faster method development as rapid feedback on the performance of the extraction process is provided. This has been found to be very useful in this application as the supercritical fluid extraction method used only reliably extracted eight of the pesticides under investigation.

A second approach has been to use high speed GC as a screening tool for chlorinated pesticides in water samples. The rationale here is that many samples may contain pesticide residues at levels below those set as acceptable by regulatory agencies. By using a fast analysis method for screening, the number of samples requiring full analysis is reduced.

Using a conventional high speed GC system employing a cryotrap/thermodesorption system and a short 100 μm i.d. GC column, separation of 10

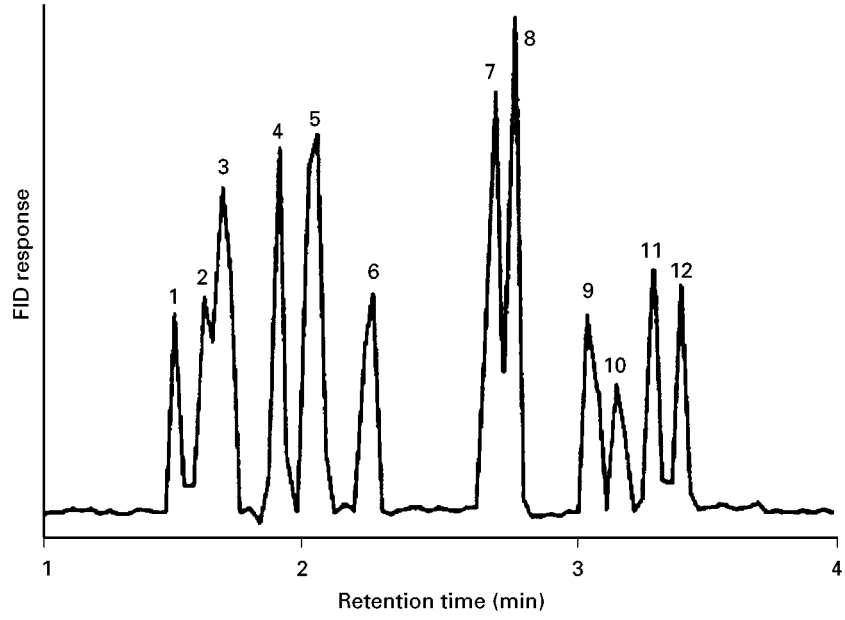

Figure 4 First-dimension chromatogram of a pesticide mixture. The GC oven was kept at 120°C for 0.3 min and then programmed at 15°C min^{-1} to 180°C. The modulator chamber was kept at 150°C isothermal. Peak identification : 1, dicamba; 2, trifluralin; 3, dicloran and phorate; 4, atrazine; 5, fonofos and diazinon; 6, terbufos; 7, alachlor; 8, metalaxyl; 9, malathion; 10, DCPA; 11, captan; 12, folpet. (Reproduced with permission from Liu *et al.*, 1994.)

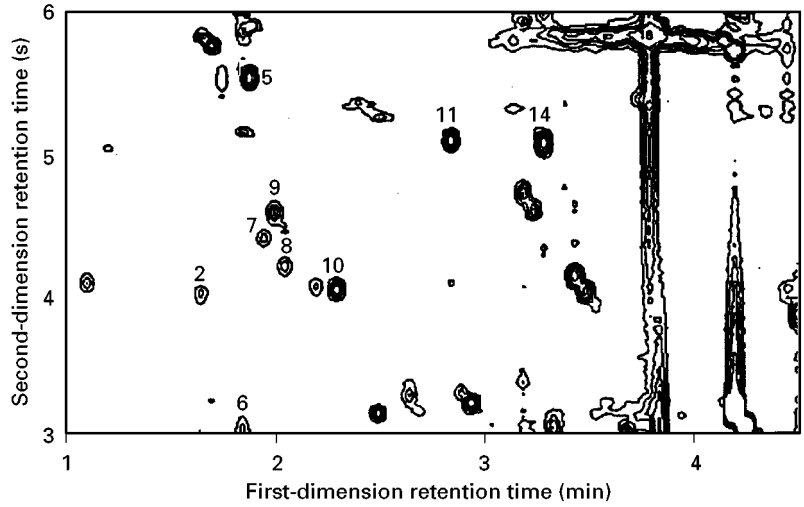

Figure 5 Two-dimensional gas chromatogram of a supercritical fluid extract of a spiked human serum sample. The GC oven was kept at 118°C for 0.5 min and then programmed at 15°C min^{-1} to 200°C. Peak identifications are the same as in Figure 4. (Reproduced with permission from Liu *et al*., 1994.)

chlorinated pesticides and an IS in under 2 min has been achieved.

The use of the pulsed-discharge detector operated in the electron capture mode gave on-column LOD values between 10 and 50 fg. This is an order of magnitude lower than that reported in previous work with high speed GC and a conventional radioactive electron-capture detector. These low LOD values allow reduced sample preparation and smaller sample volumes to be used prior to analysis.

The use of nonequilibrium solid-phase microextraction as the sample preparation method reduces the sample preparation time to just over 4 min per sample. The sample preparation step actually consists of a 2 min extraction stage and a 2 min desorption stage. LOD values for the complete method range from 10 to 20 ng L^{-1}, which is below current regulatory levels set by the Environmental Protection Agency in the USA for these chlorinated pesticides in water. **Table 3** shows the detection limits obtained with the fast screening method in comparison to other methods and regulatory levels.

Real river water samples spiked and analysed give good agreement between expected and actual values. **Figure 6** shows representative chromatograms obtained from spiked water and blank river water samples together with the focusing of septum bleed. RSD values were found to be high (20%). This was attributed to the very short extraction time. Despite this, the method offers a relatively simple way of screening a large number of samples for chlorinated pesticides.

Table 3 Results of the calibration curve obtained by solid-phase microextraction over the range 10–400 ng L^{-1}

	r^2	LOD (ng L^{-1})	From literature[a]	From literature[b]	EPA 508
α-BHC	*0.992*	*10*	*900*	*1*	*25*
β-BHC	*0.980*	*20*	*9000*	*1*	*10*
δ-BHC	*0.965*	*20*	*2000*	*2*	*10*
cis-chlordane	0.978	5	N/A	N/A	1.5
trans-chlordane	0.969	5	N/A	N/A	1.5
p, p′-DDE	0.908	10	100	1	10
p, p′-DDD/endrin	0.969	10 + 10[c]	60 + 200[c]	0.1 + 1[c]	2.5 + 15[c]
Endosulfan sulfate	0.929	10	50	0.6	15
Endrin ketone	0.964	10	500	1	N/A

[a] Solid-phase microextraction with GC-FID analysis.
[b] Solid-phase microextraction with GC-mass spectrometry analysis.
[c] Respective LODs for *p, p′*-DDD and endrin. Values obtained in this study are estimates based on co-eluted peaks obtained.
N/A, Not analysed.

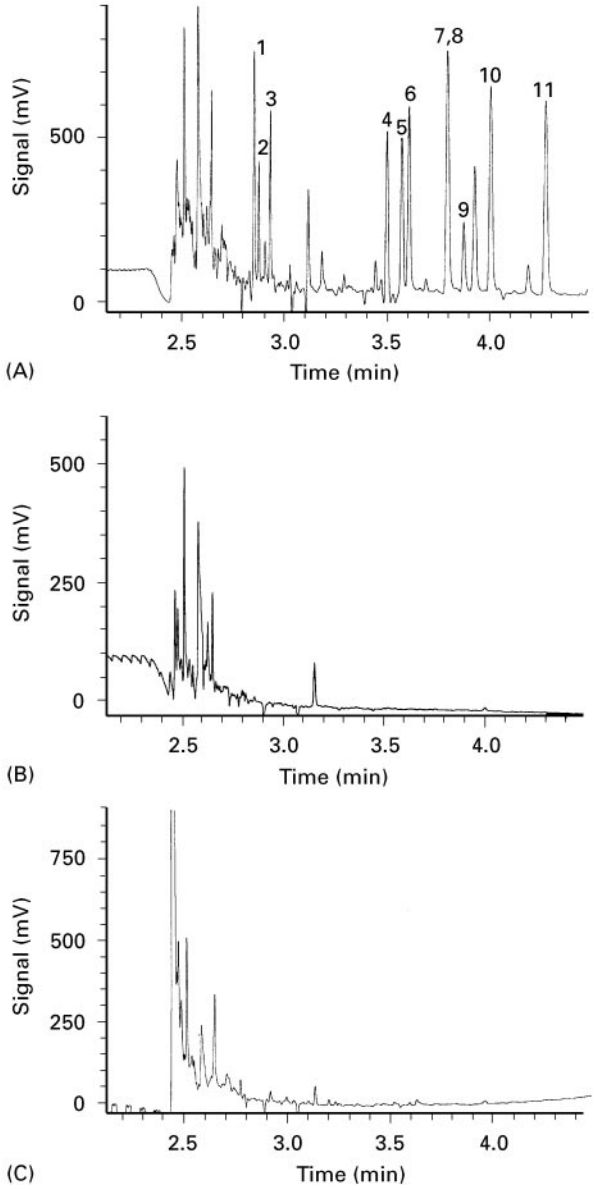

Figure 6 Chromatograms obtained after 2.3 min trapping at 60°C. (A) Solid-phase microextraction (SPME) fibre desorbed for 2 min after 2 min extraction of a 0.2 ng mL^{-1} spiked water sample. Peaks: 1, α-BHC; 2, β-BHC; 3, δ-BHC; 4, *cis*-chlordane; 5, *trans*-chlordane; 6, *p,p′*-DDE; 7, *p, p′*-DDD; 8, endrin; 9, endrin aldehyde; 10, endosulfan sulfate; 11, endrin ketone. (B) Blank run showing the extraneous peaks caused by septrum bleed being focused by the cryotrap. (C) SPME of Hocking river water; no pesticide peaks observed. (Reproduced with permission from Jackson and Andrews, 1998.)

PAHs

The use of high speed GC coupled with filter tape sampling and thermal desorption has been applied to the online assessment of PAHs in a combustion process.

Gas and particulate material are sampled directly from the emission stack using a null-type probe. A dilution method is used to cool the gas stream to below 50°C so that condensation of the PAHs occurs on particulates in the gas stream. These particulates are then retained on a glassfibre filter. **Figure** 7 shows the instrumental layout used for this work.

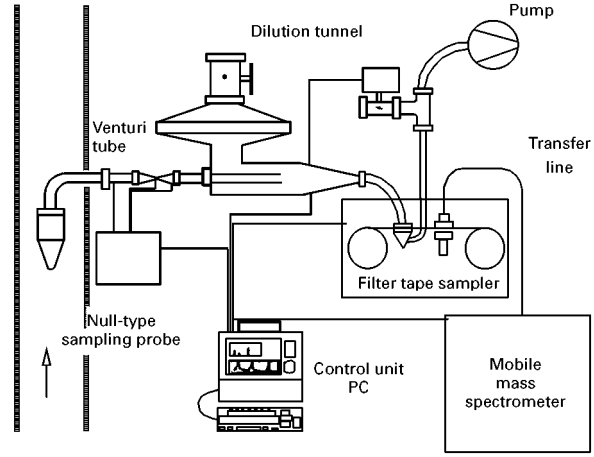

Figure 7 Overview of the sampling and analysis system for online measurement of PAHs in a combustion process. (Reproduced with permission from Munchmeyer *et al*., 1996.)

Once sampling is complete the PAHs are desorbed at 300°C by contact with a heated piston. The sample enters a short (1.6 m) metal capillary column, resistively heated, which allows for rapid cycling. The sample is detected using mass spectrometry. Complete separation of all PAH isomers is not achieved, although overlapping peaks of substances with different mass spectra can be resolved by correlation software. With a sample volume of 100 L the LOD values were found to be below the emission limits for carcinogenic compounds such as benzo[*a*]pyrene.

Solvent Purity

The analysis of impurities in high purity solvents is a very difficult task. By utilizing high speed GC instrumentation which has the ability to control the direction of carrier gas flow through the column this analysis can be greatly simplified.

The ability of dual-flow direction instrumentation allows for minor impurities in the solvent to be separated and identified by venting off the solvent peak only. After the majority of the solvent peak has passed off the column and into the detector, the carrier gas flow is reversed with subsequent refocusing of the residue left on the column in the cryotrap at the column head. This residue can then be reinjected.

This is well illustrated in **Figure 8**, which shows both 2-fluorotoluene and octane as impurities in toluene. Even with only 0.53 nL of sample on-column, the 2-fluorotoluene is still almost completely obscured in the

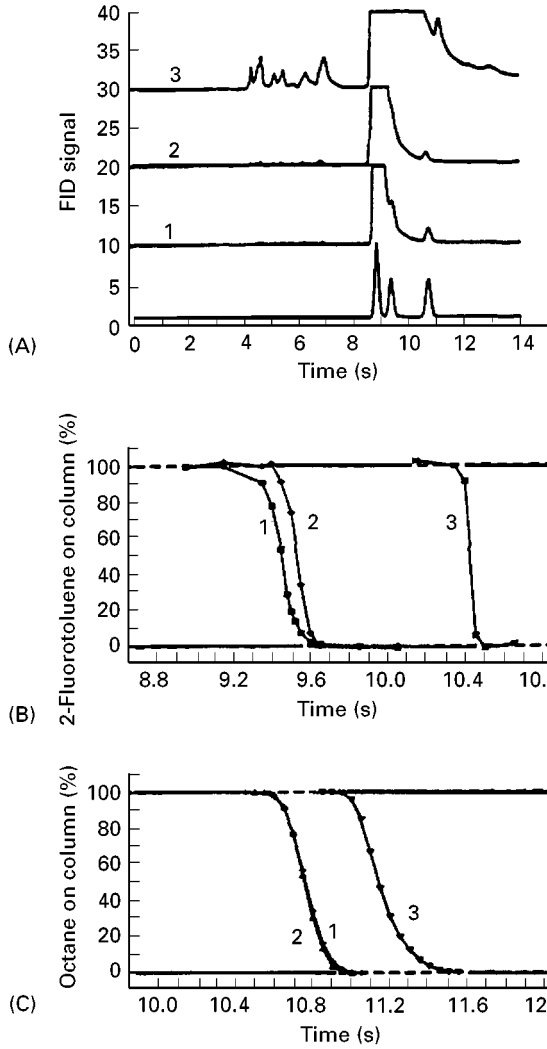

Figure 8 (A) Chromatograms showing the impurities in toluene. For plots 1, 2 and 3, 0.53, 2.67 and 13.3 nL of sample were collected in the cold trap. (B) and (C) show the percentage of impurity on column relative to time before refocusing commenced for plots 1, 2, and 3 from (A). This is the effect of solvent loading on the elution profiles of (B) 2-fluorotoluene and (C) octane in toluene. (Reproduced with permission from Klemp and Sacks, 1991.)

solvent peak. By venting the solvent and refocusing, the impurities can clearly be seen (Part (A), trace 1).

Stationary-phase film thickness is an important consideration in this type of fast analysis. Too thin a film will not sufficiently resolve impurities, which elute prior to the solvent peak. Too thick a film will degrade resolution of the peaks after the solvent peak, increase the size of the solvent tail and increase the total run time. An intermediate film thickness, such as 0.25 μm, is a satisfactory compromise between the two extremes.

Miscellaneous

One novel application of high speed GC has been in the rapid screening of soil gas samples for fuel-related

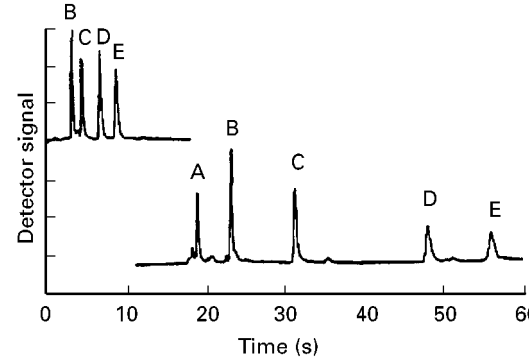

Figure 9 Comparison of high speed screening mode using the photoionization detector (top) and the advanced field analysis mode using the FID (bottom) for a test mixture containing; A, *n*-pentane; B, benzene; C, toluene; D, ethylbenzene; E, *o*-xylene. (Reproduced with permission from Sacks *et al*., 1996.)

hydrocarbons. The gas samples are continuously generated from a cone penetrometer (CPT) equipped with a heated probe tip. The GC instrument is located in the CPT truck and connected to a heated gas transport line. The whole instrument is intended to provide high spatial resolution for contaminated soil site characterization.

The instrument can operate in two modes: either a high speed mode to detect BTEX compounds using a photoionization detector, or an advanced field mode using a FID to obtain a finger print-type chromatogram for fuel-type identification. The time frame for the separation in the two modes is 10–20 s and up to 200 s respectively. **Figures 9** and **10** show representative chromatograms obtained in the two modes.

Another unusual application has been the coupling of multidimentional high speed GC with Fourier transform infrared (FTIR) detection. Using an offline interface solved the mismatch generated by the fast separation and the slow acquisition of the FTIR

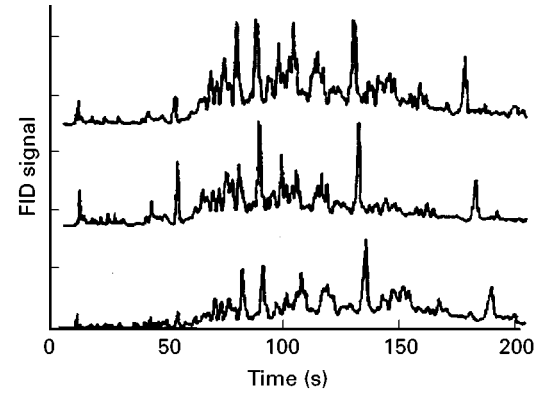

Figure 10 Advanced field analysis chromatograms of headspace from soil samples spiked with diesel fuel. (Top) Sand with 18% moisture; (middle) silty clay with 28% moisture; (bottom) silty loam with 38% moisture. (Reproduced with permission from Sacks *et al.*, 1996.)

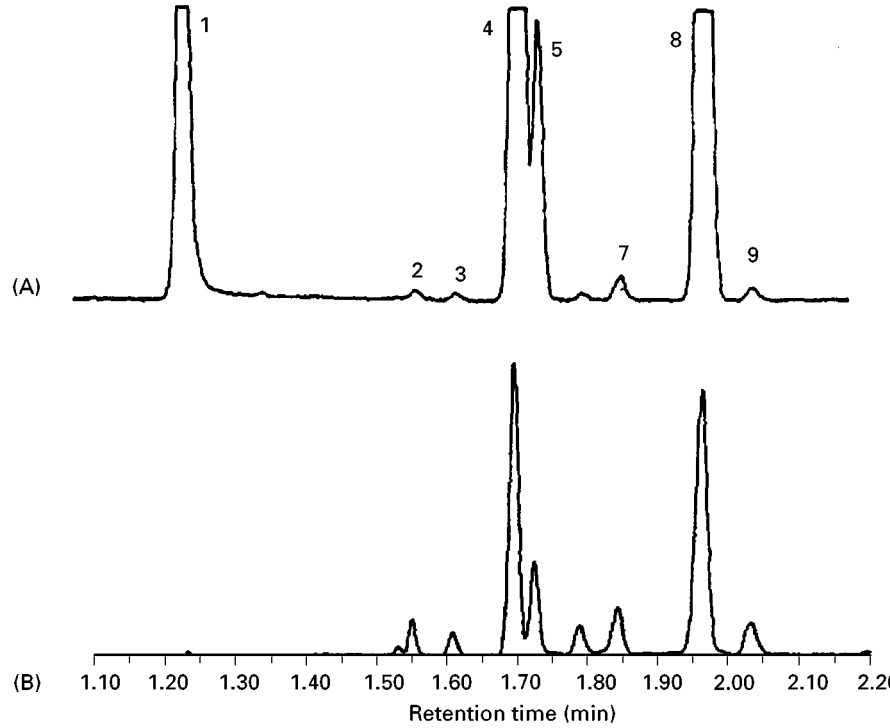

Figure 11 Secondary separation: (A) IR reconstructed chromatogram of a segment of a eucalyptus oil separation using a Rtx-1701 column and (B) FID trace. The secondary separation conditions were isothermal at 95°C with a linear carrier gas velocity of 90 cm s^{-1}. (Reproduced with permission from Ragunathan *et al*., 1994.)

detector. The eluent from the fast GC column was deposited on to a cooled rotating collector disc and actual analysis of the disc with the FTIR was undertaken after completion of the GC run.

The system was applied to the heart-cut analysis of essential oils from cascarilla and eucalyptus. A chromatogram of the eucalyptus analysis is shown in **Figure 11**. The use of FTIR detection provides structural information concerning the oil components, even the minor components present. Spectrum quality was found to be sufficient for component identification by library search methods. An infrared spectrum obtained from one of the components in Figure 11 is shown in **Figure 12**. One disadvantage is

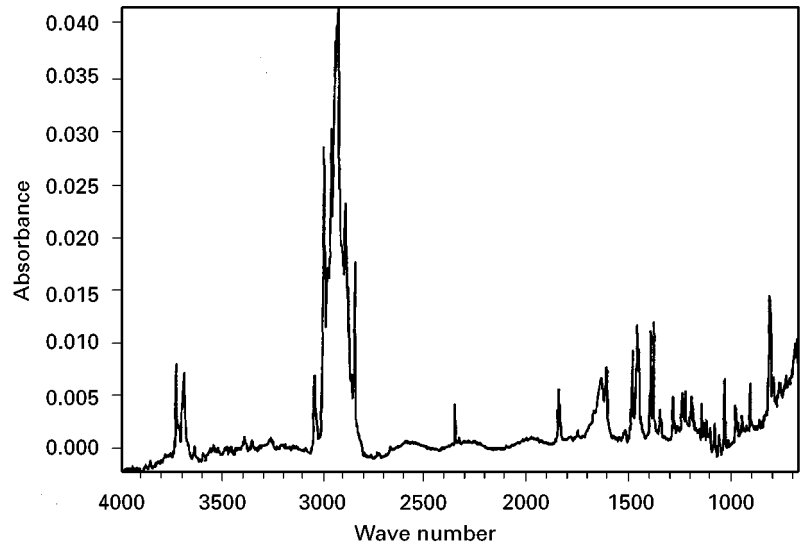

Figure 12 Infrared spectrum of the α-pinene peak (number 3 in Figure 11). The spectra acquisition conditions were 128 scans at 4 cm^{-1} resolution with a mirror velocity of 1.27 cm s^{-1}. The time for acquiring this spectrum was 120 s. The bands at $\sim$3700 and 1600 cm^{-1} are due to water. (Reproduced with permission from Ragunathan *et al*., 1994.)

that, for large sample sizes, some decomposition is seen from the reheating of the cryotrap region between the two columns.

Future Developments

High speed GC can probably be best described as currently being in an interim phase. It is in the process of moving from the research laboratory to the analysis laboratory. Most of the basic research demonstrating the feasibility of high speed GC and the instrumental modifications necessary for successful high speed analysis have been accomplished and the findings are well disseminated in the literature, yet the number of true applications remains small.

One technique, which has not been commented on in this article, is the combination of high speed GC with mass spectrometry (MS) as a detection method. The advent of fast scanning time-of-flight mass analysers has provided the data acquisition rates necessary for interfacing with high speed GC. Although the concept of coupling the two techniques together has been demonstrated, no real application articles using both techniques are currently available. Given the prominence of GC-MS analysis in many areas, this will surely change in the near future.

Other future developments will probably include the interfacing of other selective detectors with high speed GC and the subsequent expansion of high speed GC into the areas of analysis served by these selective detectors.

See also: **II/Chromatography: Gas:** Column Technology; Detectors: General (Flame Ionization Detectors and Thermal Conductivity Detectors); Detectors: Mass Spectrometry; Detectors: Selective; Historical Development; Multidimensional Gas Chromatography; Sampling Systems; Theory of Gas Chromatography.

Further Reading

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Historical Development

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In 1941 Martin and Synge published their classic paper on liquid–liquid partition chromatography in which they pointed our that there was no reason why the mobile phase should not be a gas. This suggestion was not followed up until 1952 when James and Martin published their paper on the separation of fatty acids by gas–liquid chromatography. This paper generated a frenzy of activity, particularly within the troleum industry where the method represented an