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Review

Gas chromatography with Fourier transform infrared and mass spectral detection

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Abstract

For a number of years, the combination of gas chromatography with infrared and mass spectral detection has been of interest. Numerous applications have been reported and discussed in previous reviews. The present brief review covers recent developments in this area since 1997. It is clear that advances in computer technology combined with those in instrumentation make it only a matter of time until the goal of fully automated GC–Fourier transform IR–MS is realized.

Keywords: Detection, GC; Reviews; Infrared detection; Mass spectrometry

Contents

1. Background

Separation and identification of components in complex mixtures can be a daunting task. Hyphenated analytical methods are often used because multidimensional information can be obtained from a single analysis. Gas chromatography (GC) is the most common technique for separation of volatile and semi-volatile mixtures. It is well accepted that when GC is coupled with spectral detection methods, such as mass spectrometry (MS) or Fourier transform infrared (FT-IR) spectrometry, that the resulting combination is a powerful tool for the analysis of complex mixtures. In particular, multispectral analysis systems are even more definitive than those employing a single spectral detector. We

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have reviewed this topic several times during the past few years [1–3] – so the current brief review should be considered an update, covering new applications from the recent past. Because the previous three reviews were relatively thorough, only a brief reprise of the relevant theory and background will be provided here; readers desiring more complete information should consult the earlier reviews.

2. Gas chromatography–mass spectrometry

MS spectrometry is the most widely used spectral detection method for GC. Traditionally, quadrupole mass spectrometers equipped with electron ionization sources have been the most common instruments mated with gas chromatographs. Their popularity is due to their ease of operation, relatively low cost, simplicity, and ruggedness. Recently, there has been increased interest in the use of ion trap mass spectrometers for GC–MS. Such instruments share many of the advantages of the quadrupole instruments and, because of improved software, ease of operation, and compact design, such GC–MS systems are becoming more popular. The basics of mass spectrometer operation are beyond the scope of the present review, so that topic will not be discussed here; a good source of that type of information is a recent handbook [4]. Other references also explain mass spectrometers and the specific requirements for their use as chromatographic detectors [5,6]. For single spectral detection, reasons for choosing MS over FT-IR detection include superior detection limits and the availability of a great many standard GC–MS protocols, which have been developed for many routine applications. Furthermore, commercial libraries of electron impact ionization (EI) mass spectra are available and the number of spectra in such libraries exceed the number of IR spectra by at least an order of magnitude. Even so, because of the definitive information regarding functionality which IR spectra often provide, the need for large libraries of such spectra is diminished, because the matches which do occur can be used to advantage to simplify MS analysis and eliminate many MS library matches.

3. Gas chromatography–Fourier transform infrared spectrometry

Although GC–IR was first introduced in the 1960s [7], and its combination with MS suggested about the same time [8], its use did not become widespread until the 1980s. Several factors were responsible for its commercialization at that time. The key developments were sensitive mercury–cadmium–telluride (MCT) photodetectors and the light pipe flow cell [9]. Although the light pipe was the first commonly used GC–IR interface, three types of FT-IR interfaces are currently in use: light pipe [9], matrix isolation (MI) [10,11] and direct deposition (DD) [12–14]. Comparison studies of the different interfaces have been reported [15,16], and White has written an excellent book that addresses chromatography with IR detection in great detail [17].

The light pipe interface is the simplest design and the only one of the three that permits acquisition of gas phase infrared spectra. Typically, a light pipe flow cell is a gold-coated borosilicate glass capillary, usually about 1 mm in diameter and 10 cm long. GC effluent enters the light pipe and the IR beam is reflected through the cell, increasing the sample path length and, following Beer's law, allowing better detection limits. Even so, detection limits remain relatively poor (~10 ng), especially when compared with those achievable by MS. The volume of the flow cell, which is usually on the order of 100–150 μl, also imposes limits on the chromatographic resolution that can be maintained. Further details on the light pipe interface, its construction and optimization, together with leading references, can be found elsewhere [9,18].

DD- and MI-FT-IR are very similar, in that the sample is cryogenically frozen on a surface for FT-IR analysis. The major differences between the two are that in the DD–FT-IR interface, the surface is an IR transparent window, usually ZnSe. GC effluent is deposited onto upon this window and absorption spectra are subsequently acquired. A major advantage of this interface is that the spectra obtained are similar, if not identical, to KBr spectra [19], and therefore can be compared with those in standard computer-readable libraries of KBr spectra, which are much larger than those available for both

vapor phase (light pipe) and MI-FT-IR. In MI-FT-IR, the GC effluent is frozen, together with a matrix gas (usually 2–5% Ar, present in the carrier gas) on a reflective, gold surface and reflection-absorbance spectra are collected. This low temperature argon matrix partially isolates the individual molecules, resulting in IR spectra that exhibit very sharp features due to reduction of intramolecular interactions and elimination of much of the rotational and vibrational broadening. These sharp features facilitate spectral interpretation, although the MI-FT-IR library is relatively small (e.g. the Mattson Instruments library has 5000 spectra). With both DD- and MI-FT-IR detection, because the sample is frozen onto a substrate, off-line analysis, including signal averaging, can be performed. By utilizing signal averaging, detection limits of GC–FT-IR systems approach the picogram range typical of GC–MS. A practical limitation of such interfaces is their susceptibility to interferences from traces of water and carbon dioxide, which limit sensitivity unless precautions are taken to rigorously exclude them.

Because cryotrapping GC–FT-IR interfaces require no special sample handling and bring the detection limits of FT-IR and MS closer together, the popularity of obtaining both types of spectra from a GC separation has increased. Obviously, the combination of MS and FT-IR detection is a very powerful coupling because of the complementary nature of the data acquired. Mass spectra of isomers are often identical and IR spectra of homologues are very similar, making unambiguous identification difficult when only one type of spectrum is obtained. On the other hand, mass spectral measurements readily distinguish homologues and IR spectra of isomers often provide permit unique characterization. Thus, an analysis system providing both spectral capabilities can provide superior qualitative analysis.

4. Instrumental advances in GC–MS and GC–Fourier transform IR

Many of the recent primary improvements in GC–MS have evolved from the advances in GC, particularly in the areas of faster separations and field portable instrumentation. The fact that both topics were recently featured in Analytical Chemistry emphasizes the importance of those developments [20,21]. Although Desty suggested that high speed GC separations were possible in the early 1960s [22], it was not until the advent of faster electronics and computers that this area was extensively explored. When interfacing high speed separations to spectral detectors, many factors need to be considered. Most of these factors are related to instrumental dead volume and data acquisition rates. The speed of data acquisition rates is the limiting factor when interfacing fast separations to MS. Although fast GC has been interfaced with scanning mass spectrometers, such as the quadrupole [23,24] and ion trap [25] instruments, their scan rates limit their ability to provide high quality spectra for eluents from fast separations. For very high speed separations, a high speed detector, such as a time-of-flight (TOF) mass spectrometer, is necessary to successfully acquire mass spectral data [26]; such fast GC–TOF-MS systems are now commercially available.

Field-portable GC–MS systems are becoming more and more popular to reduce the number of samples sent back to the laboratory for complete analysis and to decrease the amount of sample handling and time between sample collection and analysis, both of which may alter results. With development of more compact electronics, as well as micromachining, GC, and even GC–MS systems have made the transition from benchtop to transportable and have even reached the truly portable stage.

Most of the recent advances in GC–FT-IR have been in the applications area, i.e. adapting the technique to make it more versatile for various types of analyses. For example, Visser and co-workers have developed an on-column interface to introduce large sample volumes for GC–FT-IR analysis [27] and utilized it for trace analysis of environmental contaminants [28]. Recently, we have reviewed other GC–FT-IR application advances [1–3] as have others [29,30].

5. Recent GC–Fourier transform IR–MS applications

As mentioned at the outset, this brief review is

intended to summarize, recent applications of GC–FT-IR–MS analysis. The main requirement for mention here is that both IR and MS spectra were collected from each component separated by GC, thus excluding papers where a separation stage does not precede spectral collection. Although it was required that both IR and MS spectra be obtained, it was not necessary that they be obtained simultaneously, i.e. we have included consideration of applications where separate GC–MS analysis and GC–FT-IR analyses were conducted. Because we recently reviewed this area [3], overlap is intentionally kept to a minimum. Although a great deal of work involving GC–MS and GC–FT-IR individually has been reported since the last review, research involving joint use of both methods of detection is more limited. From 1997 to the present, which is the time span this review covers, relatively few papers have been published utilizing GC–FT-IR–MS. Those that have will be discussed here.

5.1. GC–MS and lightpipe GC–IR results

Amenta et al. recognized the value of hyphenated techniques in the chemistry community and developed ways to incorporate GC–MS and GC–FT-IR into freshman and sophomore chemistry laboratories instead of waiting for an instrumental analysis course to introduce students to these techniques [31]. However, because they did not have access to a combined GC–IR–MS system, they used separate GC–MS and GC–IR instruments for experiments in which students both monitored reaction progress and characterized the products of a ferrocene synthesis. A lightpipe-based FT-IR was used for the IR measurements employed by the students. One of the pedagogical advantages of this approach was that it allowed the students to see the relative simplicity of gas phase IR spectra, compared with those of the solid products.

Basiuk and Navarro-González identified unusual products of silica-catalyzed amino acid condensation using GC–FT-IR–MS, as well as HPLC–particle beam MS [32]. For their GC–IR–MS analysis, they used the commercial lightpipe-based Hewlett-Packard GC–IR–MS system. Many of the products were first identified by interpreting their mass spectral fragmentation patterns with IR spectra being used to confirm these identifications. Additionally, computer-generated simulated IR spectra were employed for as additional confirmation of assigned identifications. In another paper, Basiuk et al. also studied amino acids and their pyrolysis products using GC–FT-IR–MS, as well as NMR [33]. Again, initial identifications were based upon the mass spectral data with IR spectra, both acquired and simulated, used for confirmation. Both this and the previously cited paper are good examples of applications where the analyst is not sample limited, and can therefore tolerate the sensitivity limitations of on-the-fly GC–IR. Fig. 1 shows the MS, FT-IR, and computer-generated IR spectra of pyrolysis products in their study. These spectral data, combined with GC retention times, provide a good deal of information about the identities of mixture components. As expected, more information leads to more confidence in the ultimate identifications.

Guitton et al. used GC–MS and GC–FT-IR to identify fentanyl metabolites [34]. Fentanyl is a synthetic opioid used for surgical analgesia and sedation. Their goal was to develop an analytical method to simultaneously detect and identify its metabolites. The subjects of their study were seven patients from an intensive care unit who had been receiving fentanyl infusion for less than 3 days. Twenty milliliter aliquots of 24-h urine samples were analyzed. Both stand-alone GC–MS and GC–IR instruments were used, in addition to a direct-linked GC–IR–MS system. Regardless of which system was used, the GC–IR spectra were obtained using a lightpipe interface. Interestingly, the primary use of the GC–IR–MS instrument, as reported, was to establish the necessary correspondence between the mass and infrared spectra for the same chromatographic peak. This observation highlights one of the primary advantages of an integrated system, which is the avoidance of any ambiguity regarding which mass and infrared spectra represent those of the same material. Obviously, even though one attempts to make chromatography identical, with separate stand-alone GC–MS and GC–IR systems it can be difficult to be absolutely certain that the correct pairings have been made. The authors also noted that allthough GC–MS had been previously used for this analytical problem, this was the first time GC–FT-IR was used to successfully confirm identification of nine of the

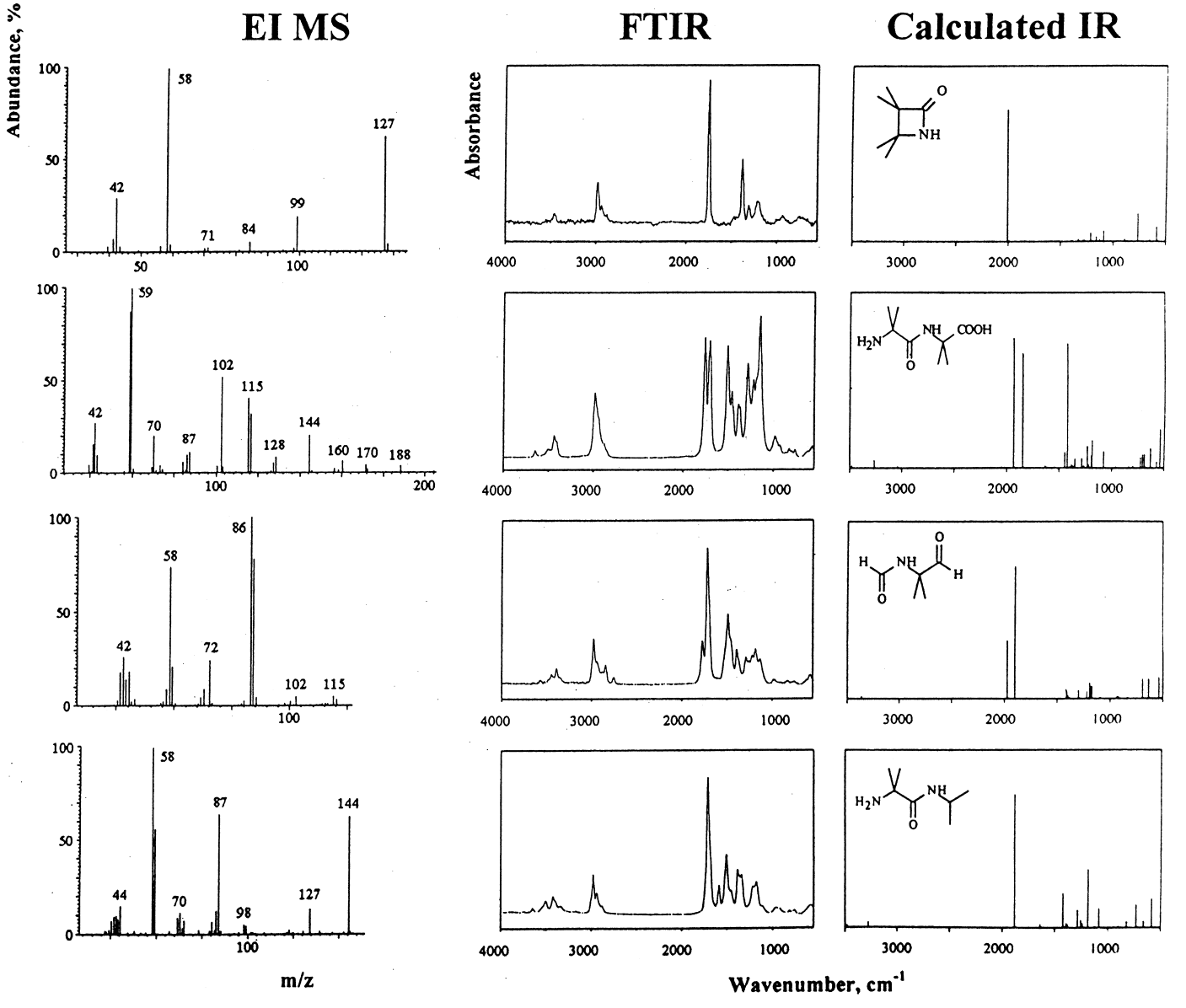

Fig. 1. Identification of chloroform-soluble products of α-aminoisobutryic acid pyrolysis. EI-MS, FT-IR, and calculated IR spectra are shown (from Ref. [33], with permission).

ten previously identified metabolites. Because of the lack of metabolite standards against which the previously obtained GC–MS data could be compared, this confirmatory evidence provided by GC–IR–MS was exceptionally useful.

As mentioned earlier, FT-IR is very useful for isomer analysis. Sommer et al. studied polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) extracted from municipal fly-ash using GC–FT-IR–MS analysis [35]. For this investigation, a linked GC–IR–MS system was used. The authors noted some difficulties resulting from the disparate flow-rate requirements of the IR and the MS detectors. This could be compensated for experimentally. FT-IR spectra are critical in this study because certain isomers are of toxicological importance. Furthermore, because many assignments for dioxin vibrations are uncertain in the literature, ab initio calculations were used on some dioxins and furans to obtain simulated IR spectra. These spectra coordinated well enough with measured spectra to allow definite assignment of the most important vibrations. Because the IR detector employed a light pipe instrument, sample preconcentration was necessary for adequate detection and detection limits of 10–20 ng were obtained, depending upon the analyte. One of the primary conclusions of this paper was that FT-IR spectrometry was a useful aid in dioxin and furan analysis, but that it could not replace GC–MS due to its lower sensitivity. It was also noted that the gas phase IR spectra were more susceptible to interferences from the fly-ash matrix than were the MI-FT-IR spectra from the literature which were used for comparisons.

Climent and Miranda used GC–light pipe FT-IR and GC–MS to study the photodegradation of two phenoxyalkanoic acids: 2-(2,4-dichlorophenoxy)-propionic acid and 2-naphthoxyacetic acid [36]. These acids and their esters are used as herbicides. Photodegradation is of concern because it controls the fate of chemicals in the environment. Furthermore, photolysis is involved in the activation and release of a number of bioactive molecules. By identifying the products of such degradation, information about the photolytic pathways can be obtained, which, in turn, can help establish ways to protect the environment. The authors irradiated sample solutions with simulated sunlight, both in air and in an argon environment, and analyzed the mixtures separately with by both GC–MS and GC–FT-IR. They were able to identify many of the products and establish photodegradation pathways. Although most of the pathways were similar to those already reported for other phenoxyalkanoic acids, they did discover some new processes which were not previously reported for pesticide photochemistry.

In the area of flavors and fragrances, Misharina and Golovnya studied volatile substances responsible for the flavors and fragrances of meat products [37]. Sulfur-containing compounds are key components of meat odor, and the same sulfur compounds and aldehydes are present in both beef and chicken flavors. The unique characteristics of each can be attributed to a difference in ratios of these compounds. The authors used mass spectral data for initial identifications but they were insufficient for identification of some components. For example, the molecular ions of unsaturated aldehydes with more than seven carbon atoms are not present in the mass spectra, making identification difficult. Also, many alkenals and alkadienals have very similar fragmentation patterns. For these components, retention indices were utilized to make primary identifications which were confirmed by interpreting lightpipe GC–IR spectra. Of seventy-two components identified from the meat and chicken flavorings, the authors confirmed the identification of twenty-two from their IR spectra. Fig. 2 shows IR spectra of six components in chicken flavoring. The numbers correspond to peak numbers in the chromatogram. Of particular importance, peak numbers 62 and 63 are of two isomers of 2,4-decadienals. Note that the spectra show some unique features, thus it is possible to distinguish between the two. Both qualitative and quantitative data from the flavorings compared favorably with those acquired from natural beef and chicken broth. Therefore, the study concluded that the flavorings are identical to those found in the natural products.

Tomlinson and Wilkins studied the presence of irritants in a variety of soaps utilizing multidimensional GC–FT-IR–MS [38]. With their lightpipe GC–IR–MS system, they analyzed the fragrance content, in order to determine whether or not six different irritants which can cause contact dermatitis were present. Many components which cause contact

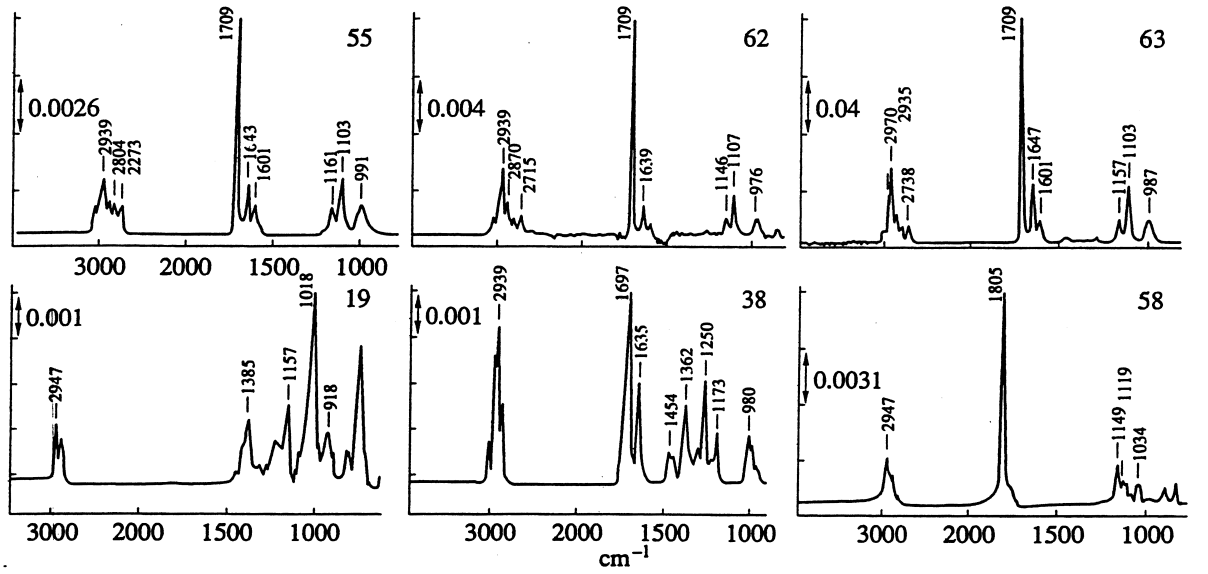

Fig. 2. FT-IR spectra of 2,4-nonadienal (55), two isomeric 2,4-decadienals (62,63), 2-111propylfuran (19), 3-octene-2-one (38), and γ-octalactone (58). Numbers correspond to peak number in the corresponding chromatogram. From ref. [37], with permission.

dermatitis are chiral, with one optical isomer sometimes being an irritant, while the other is not. Because fragrances are often chiral, one enantiomer, or a specific ratio of enantiomers, may be responsible for the characteristic smell. In this study, several components in the soaps investigated, including many enantiomers, were successfully separated. Mass and infrared spectra, as well as GC retention times were used to make identifications.

5.2. GC–MS and matrix isolation or direct deposition GC–IR results

Trichothecene mycotoxins, secondary fungal metabolites produced by species of mold, are a natural contaminant of feedstuffs and food. Because they can be toxic to humans and animals, their detection is important. Mossoba et al. utilized GC–MI-FT-IR and GC–MS to analyze grains for these contaminants [39]. Tricothecenes are large molecules with many functional groups, which make these compounds ideal for IR analysis. Previous studies utilized light pipe IR; however, the detection limits were poor, so the authors turned to MI-FT-IR. This paper presents some excellent examples of the advantages of the enhanced sensitivity and spectral detail available by use of matrix isolation FT-IR. Separate GC–IR and GC–MS instruments were used. Identifications were based upon GC retention times and IR spectral analyses. The sharp bands present in MI-FT-IR spectra, increasing the number of distinguishable bands, were used to advantage for qualitative analysis. For a compound to be identified, when its spectrum was compared to reference spectra all major characteristic FT-IR bands had to be present at correct wavenumbers, and the ratio of stronger to weaker bands also had to be similar. GC–MS was used to confirm identifications, as well as for quantitative purposes.

Söderström et al. used selective GC detection methods, namely nitrogen–phosophorous detection (NPD) and flame photometric detection (FPD), in addition to GC–MS and GC–IR to identify compounds relevant to chemical weapons [40]. They reported on the results of a trial proficiency test for laboratory procedures for chemical weapons analysis. The proficiency tests were organized by the Organization for the Prohibition of Chemical Weapons (OPCW). Four spiked samples (rubber, paint, and two soil samples) were analyzed. Retention indices were obtained using the selective detection methods (NPD and FPD) and both GC–

MS and direct deposition GC–IR were carried out. Retention indices were important to ensure that the correct chromatographic peaks were analyzed for each analysis, especially in the cases of very complex mixtures. To unambiguously identify a compound, it was required that at least two different spectroscopic methods agreed on the identification. Most compounds were identified through routine spectral searches and/or comparison with reference spectra. However, spectral interpretation was needed for a few compounds and references were synthesized, so that their spectra could be measured and compared with sample spectra.

6. Conclusion

From the information presented in this review, it is clear that GC–FT-IR–MS continues to be a powerful method for separation and identification of complex mixture components. Some of the main disadvantages of the technique are cost, time required for analysis, and the large amount of data generated. Recent literature supports the view that lightpipe GC–IR combined with MS will be most suitable for situations in which the sample is not limited. For applications which are sample-limited, the recent literature continues to document the advantages of direct deposition and matrix isolation GC–IR interfaces. It would also be possible to achieve improved sensitivity by modifications of other parts of the analytical procedure, such as the use of large-volume sampling methods. As computers and electronics decrease in price, it is expected cost of these systems also will decrease. This decrease in price will make the tradeoff between the cost of adding an additional spectral detector and the resulting information more favorable. As new systems employing faster separations are developed, the time required for analysis also will decrease. However, if fast GC is to become a viable technique for direct linkage with MS, less expensive time-of-flight mass analyzers will need to be mated with the fast GC technique, because the common GC–MS analyzers are simply not fast enough for this application. Finally, with the advances in computer software technology, much of the data analysis, such as spectral interpretation, peak finding, will be automated, moving the analytical procedure even closer to the ideal of a fully automated GC–FT-IR–MS system for the analysis of complex mixtures. Based upon the promising developments of the past few years, it is only a matter of time until this goal is realized.

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