

Application of gas chromatography in food analysis

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Gas chromatography (GC) is used widely in applications involving food analysis. Typical applications pertain to the quantitative and/or qualitative analysis of food composition, natural products, food additives, flavor and aroma components, a variety of transformation products, and contaminants, such as pesticides, fumigants, environmental pollutants, natural toxins, veterinary drugs, and packaging materials. The aim of this article is to give a brief overview of the many uses of GC in food analysis in comparison to high-performance liquid chromatography (HPLC) and to mention state-of-the-art GC techniques used in the major applications. Past and current trends are assessed, and anticipated future trends in GC for food applications are predicted. Among the several new techniques being developed, the authors believe that, in food analysis applications, fast-GC/mass spectrometry (MS) will have the most impact in the next decade. Three approaches to fast-GC/MS include low-pressure GC/MS, GC/time-of-flight (TOF)-MS and GC/supersonic molecular beam (SMB)-MS, which are briefly discussed, and their features are compared. © 2002 Published by Elsevier Science B.V. All rights reserved.

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1. Introduction

There is truth to the saying “We are what we eat.” Of course, most of us do not become a banana if we eat a banana, but, for good or for ill, the chemicals that we ingest must be incorporated, transformed, and/or excreted by our bodies. Food is an essential ingredient to life, and access to food is often the limiting factor in the size of a given population. There is some dispute among friends whether we “eat to live” or “live to eat” (and some people “are dying to eat” or “eat themselves to death”), but there is no denying the importance of food.

The only way to know which chemicals and how much of them are in food is through chemical analysis. Only then can we know the nutritional needs for the different chemicals or their effects on health. Through the ability to identify and to quantify components in food, analytical chemistry has played an important role in human development, and chromatography, in particular, has been critical for the separation of many organic constituents in food.

With the commercial introduction of gas chromatography (GC) 50 years ago, GC has been used to help determine food composition, discover our nutritional needs, improve food quality, and introduce novel foods. Furthermore, GC has been the only adequate approach to measure many of the organic contaminants that occur at trace concentrations in complex

food and environmental samples. GC has been instrumental in helping humans realize that we must use caution with agricultural and industrial chemicals to avoid harming our health, the food supply, and the ecosystem that we rely upon to sustain ourselves. The scientific discoveries made with the help of GC in agricultural and food sciences have contributed to more plentiful and healthier food, longer and better lives, and an expanding population of 6 billion people.

Other recent articles have reviewed the analytical chemistry of food analysis [1], and particular food applications involving GC, such as carbohydrates and amino acids [2], lipids and accompanying lipophilic compounds [3,4], aroma and flavors [5–8], and pesticide residues [9,10]. The purpose of this article is to mention the main applications of GC and discuss current trends in food analysis. We hope to provide insight into how state-of-the-art techniques may impact analytical food applications in the future. There is no space in this article to discuss all advances being made in GC of food applications, and we have chosen to focus on fast-GC/MS, which we believe is the developing technology that will have the most impact in the coming decade if it can be applied in routine food applications.

1.1. Needs for food analysis

Most needs for food analysis arise from nutrition and health concerns, but other reasons for food analysis include process-control or quality-assurance purposes, flavor and palatability issues, checking for food adulteration, identification of origin (pattern recognition), or “mining” the food for natural products that can be used for a variety of purposes. All analytical needs for food analysis originate from three questions:

1. What is the natural composition of the food(s)?
2. What chemicals appear in food as an additive or byproduct from intentional treatment, unintended exposure, or spoilage (and how much is there)?
3. What changes occur in the food from natural or human-induced processes?

We shall refer to the types of analyses that answer these questions as relating, respectively, to:

1. composition;
2. additives and contaminants; and,
3. transformation products.

These categories are not always clear or even important, but they are helpful for the purpose of describing the types of applications in food analysis that are the subject of this article.

1.2. Composition

Food is composed almost entirely of water, proteins, lipids, carbohydrates, and vitamins and minerals. Water is often a very large component of food, but it is generally removed by drying before compositional analysis is conducted. Mineral content (as measured by ash after burning) is generally a very small component of food, thus a compositional triangle of the remaining major components (lipids, proteins, and carbohydrates) can be devised as shown in Fig. 1 [11]. This food-composition triangle can be used to describe and categorize foods based on their chemical content, and the division of the triangle into nine sections, as shown, can be very helpful to the chemist in deciding the appropriate analytical techniques to use in making measurements [9].

Nutritional labeling laws in many countries require all processed foods to be analyzed and the reporting of their composition to the consumer. The food processor also has an interest (and necessity!) to analyze carefully the composition of its product, thus a great number of food compositional analyses are conducted every day. Although GC is rarely used in bulk compositional assays, it is the primary tool for analysis of fatty acids, sterols, alcohols, oils, aroma profiles, and off-flavors, and in other food-composition applications [12]. GC is also the method of choice for analysis of any volatile component in food.

1.3. Additives and contaminants

Many agrochemicals are used to grow the quantity and quality of food needed to sustain

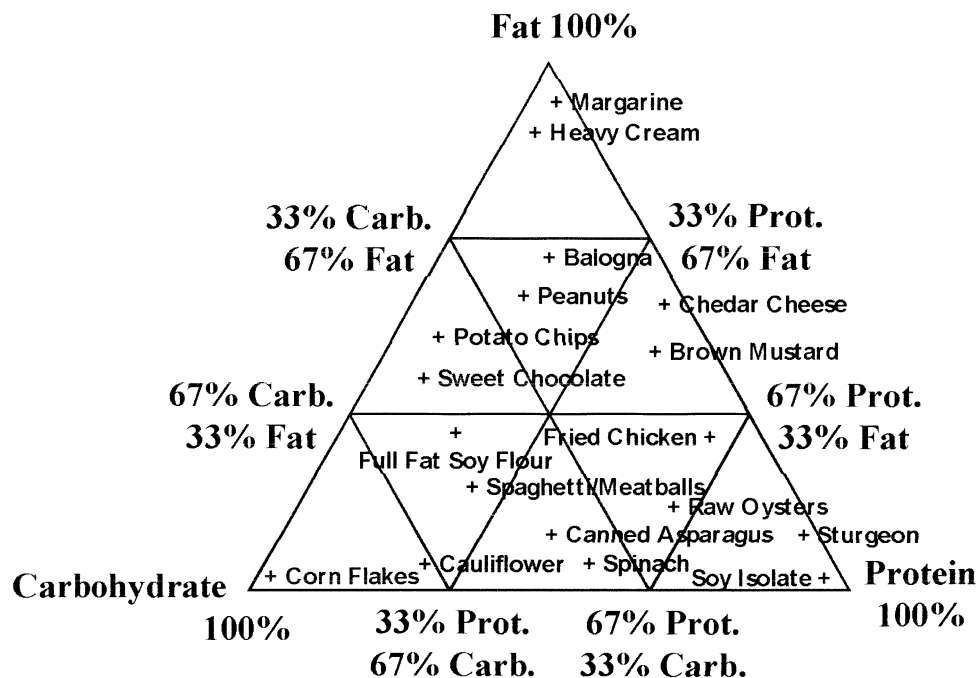


Fig. 1. Food-composition triangle divided into nine categories and examples of different foods in each category. Redrawn from [11] with permission from the author.

the human population. Many of the agrochemicals are pesticides (e.g. herbicides, insecticides, fungicides, acaricides, fumigants) that may appear as residues in the food. Other types of agrochemicals that may appear as residues in animal-derived foods are veterinary drugs (e.g. antibiotics, growth promotants, anthelmintics). Different types of environmental contaminants (e.g. polyhalogenated hydrocarbons, polycyclic aromatic hydrocarbons, organometallics) can appear in food through their unintended exposure to the food through the air, soil, or water. Food may also be contaminated by toxins from various micro-organisms, such as bacteria or fungi (e.g. mycotoxins), or natural toxins already present in the food or that arise from spoilage. Packaging components (e.g. styrenes, phthalates) can also leach into foods unintentionally.

In addition, chemical preservatives and synthetic antioxidants may be added after harvest or during processing of the food to extend storage time or shelf-life of food products. Other chemical additives (such as dyes, emulsifiers, sweeteners, synthetic flavor compounds, and taste enhancers) may be added to the food

to make it appear better to the consumer or to alter its taste or texture.

All these types of additives and contaminants are regulated by government agencies worldwide. Without doubt, more than a million analyses of food contaminants and additives are conducted worldwide per year by industry, government, academic, and contract laboratories. GC is the primary tool for the measurement of many chemical contaminants and additives.

1.4. Transformation products

Transformation products are those chemicals that may occur in food due to unintended chemical reactions (e.g. Maillard reactions, auto-oxidation), industrial processes (e.g. drying, smoking, thermal processing, irradiation), and/or other processes (e.g. cooking and spoilage). The types of chemicals that are categorized as transformational products (or endogenous contaminants arising from transformational processes) are polycyclic aromatic hydrocarbons, heterocyclic amines, urethane, nitrosamines,

chloropropanols, cholesterol oxides, irradiation products, microbial marker chemicals, and spoilage components, such as histamine and carbonyls, that cause rancidity. Some of these types of chemicals are also regulated, but the producers have no desire to market a spoiled, dangerous, or low-quality product. The bulk of analyses in this category are conducted in food-quality analytical laboratories by industry or research investigators.

2. Chromatographic analysis of foods

Typically, GC is useful for analyzing non-polar and semi-polar, volatile and semi-volatile chemicals. Without chemical derivatization, GC is often used for the analysis of sterols, oils, low chain fatty acids, aroma components and off-flavors, and many contaminants, such as pesticides, industrial pollutants, and certain types of drugs in foods. HPLC can be useful for separating all types of organic chemicals independent of polarity or volatility. But, because of the advantages of GC, HPLC has been primarily used for the analysis of polar, thermolabile, and/or non-volatile chemicals not easily done by GC. However, chemical derivatization of polar chemicals, such as amino acids, hydroxy (poly)carboxylic acids, fatty acids, phenolic compounds, sugars, vitamins, and many veterinary drugs, herbicides, and "natural" chemical toxins, is also performed to permit their analysis by GC methods. Only the non-volatile compounds, such as inorganic salts, proteins, polysaccharides, nucleic acids, and other large molecular weight organics, are outside the realm of GC, except through pyrolysis.

Although GC and HPLC are complementary techniques, the growth of HPLC in biochemical applications has led some analysts to use HPLC primarily, even in applications for which GC is advantageous. The major instrument manufacturers have focused more on HPLC applications in recent years, leaving smaller companies to take the lead in commercial advancements in GC injection, separations and detection.

An estimation and comparison of GC and HPLC chromatographic techniques used in food applications can be made fairly easily using PubMed, a free literature-search database provided by the US National Institutes of Health on the internet [13]. PubMed is an extensive database covering the major analytical and application journals, but it is designed for the biomedical researcher and not the analytical chemist or food scientist, thus the results presented here are not definitive. However, it serves the purpose of this article to display trends.

Fig. 2 gives the number of publications in the PubMed database in relation to the main food-application category, chromatographic technique, and year of the publication. Searches were limited by the terms, "GC OR gas chromatography" or "HPLC OR high performance liquid chromatography" AND "food." Thus, the search missed those papers in which the citation stated "high pressure" rather than "high performance" or "gas liquid chromatography (GLC)" instead of "gas chromatography (GC)." The caption gives the specific search terms used in each category to prepare Fig. 2.

Currently, the top GC applications for food analysis (according to the search parameters) involve: 1) lipids; 2) drugs; 3) pesticides; and, 4) carbohydrates. In the case of HPLC, the top applications involve: 1) drugs; 2) amino acids/proteins; 3) carbohydrates; and, 4) lipids.

In the case of GC, the number of publications in the food-composition category (striped regions in the Fig. 2) are approximately equal to the number of papers in the additive/contaminant category (shaded regions). But, in the case of HPLC, the food-composition papers are predominant. In both cases, applications involving transformation products barely register in comparison to the other two main needs for analysis.

As Fig. 2 shows, HPLC drew even with GC within 10 years of the commercialization of HPLC, and, during the 1990s, HPLC surpassed GC to become the more widely used tool in publications related to food applications (within the search parameters). Even for traditional

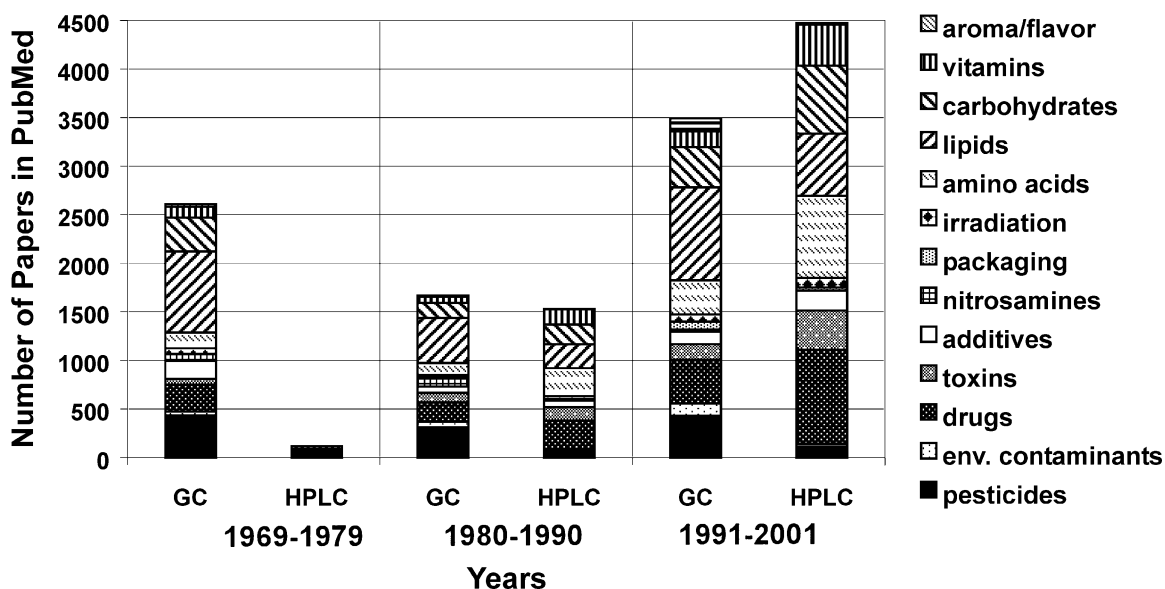


Fig. 2. Comparison of GC and HPLC in major food applications over three time periods (11 years each) of scientific literature abstracted in PubMed [13]. In addition to year of publication, all searches were limited by "GC OR gas chromatography" or "HPLC or high performance liquid chromatography" AND "food." Specific terms were used in the searches of each category as follows: 1) pesticides="pesticide OR herbicide OR insecticide OR fumigant OR fungicide"; 2) environmental contaminants="dioxin OR PAH OR PCB OR organometallic"; 3) drugs="pharmaceutical OR drug OR antibiotic OR hormone"; 4) toxins="toxin OR mycotoxin OR alkaloid"; 5) additives="additive OR preservative OR sweetener OR emulsifier"; 6,7,8) terms as listed were used for nitrosamines, packaging, and irradiation; 9) amino acids="amino acid OR protein"; 10) lipids="fat OR lipid OR oil OR fatty acid OR sterol OR cholesterol"; 11) carbohydrates="carbohydrate OR sugar OR fiber OR fibre"; 12) vitamins="vitamin OR nutrient OR mineral"; and, 13) aroma/flavor="aroma OR flavor".

GC applications, such as separations of lipids, HPLC has begun to rival GC in terms of publications.

2.1. Analytical trends

The future of analytical food applications is impossible to predict with certainty, but it is helpful in trying to predict the future by studying the past. The major goals in routine applications of analytical chemistry have always been the same: to achieve better accuracy, lower detection limits, and higher selectivity with faster, easier, and cheaper methods using more robust, highly versatile, and smaller instruments. The goals of lower detection limits and greater selectivity with smaller instruments have developed into actual trends, and, overall, many techniques today provide greater sample throughput with more ease (as a result of automation), but they are rarely cheaper!

Does this mean that only those techniques that meet the analytical quality objectives (lower detection limits with greater selectivity) will survive (at least until an even better approach comes along)? Can a faster, cheaper, easier method with a smaller instrument that gives lower quality results or lacks automation become widespread in useful applications?

A test case to answer these questions is solid-phase microextraction (SPME) [14–17]. In combination with GC, SPME is able to extract and to detect volatiles in food in an easy, and relatively fast and cheap approach. In the decade since its introduction, SPME has been the subject of nearly 1,000 publications, but because of complications in quantitation, strong dependence on matrix, and certain practical matters, some quality in the results is sacrificed for speed and ease. The strengths of SPME make it helpful in monitoring transformational changes or obtaining qualitative information,

but as Fig. 2 indicates, such transformational monitoring is a niche market. It will be interesting to see the status of SPME in 10 years.

2.2. Predictions from the 1980s

In 1982, Tanner [18] attempted to extrapolate the trends in food analysis for the 1980s. The major trend in GC at that time was that capillary columns were replacing packed columns, and it was an easy prediction to make that this trend would continue. In retrospect, another easy prediction was that the use of computers for instrument control and data processing would lead to time-saving and automated operation that would greatly increase sample throughput. The computer revolution has been essential in all aspects of science, and nearly all modern analytical instruments and many chemists could not function without computers.

However, Tanner also believed that, in food applications, the trend of lowering detection limits would not be as important in the 1980s. The more important factor was the accuracy of the determinations at the trace levels already being found. This is sometimes true in food-composition applications, and one could make the same argument today that food applications do not require lower limits of quantitation (LOQ).

During the last 20 years, the trend to lower LOQ has continued, and, even though lower detection limits may not be needed in some applications per se, lower LOQ enable the injection of more dilute samples, which is always a welcome feature, especially in GC (to reduce coinjection of non-volatiles). Instruments that give lower detection limits can also reduce the need for clean-up and solvent-evaporation steps. Indeed, the last 20 years have brought the analytical community away from multi-step, labor-intensive, large-volume, wet-chemical methods and into simpler, miniaturized approaches, in part because of the lower LOQ possible with modern instruments.

However, lower instrumental detection limits have no impact when matrix interferences are the limiting factor in detection limits for the

method. Thus, greater selectivity (in sample preparations, analytical separations, and detection techniques) is always another welcome feature that helps to provide better results at lower detection limits. The continuing ability to achieve lower detection limits with selective GC/MS(-MS) analysis, for example, has been a major advancement [19]. In industrialized nations, in addition to providing confirmatory results, GC/MS has become a primary GC tool for some food-analysis laboratories because of its ability to quantify many analytes at sufficiently low concentrations.

2.3. View from 1990s

If one was to predict the future in 1990, it may have been easy to make erroneous assessments of the impact of state-of-the-art techniques at the time. For example, the atomic emission detector (AED) was introduced [20] with a great deal of marketing and genuine scientific interest in 1990. The advantages related to the highly selective detection of several elements and simultaneously made the instrument potentially very powerful in many GC applications [21,22]. The reality was that the detection limits for important elements were not low enough in comparison to other element-selective detectors, and matrix interferences in other elemental channels limited the usefulness of these channels. The AED could provide key information to help in the identification of analytes [23], but MS by itself can provide structural elucidation and analyte identification. The cost of AED was much higher than the worth of the questionable benefits it could provide in most food applications. In 2001, the only commercial manufacturer of the AED announced the termination of the product.

The 1990s saw the rise and decline of other "advantageous" techniques with severe limitations in most food-analysis applications. A partial list includes supercritical fluid extraction, supercritical fluid chromatography, microwave assisted extraction, capillary electrophoresis, automated trace enrichment and dialysis, enzyme-linked immunosorbent assays, molecular

imprinted polymers, and matrix solid-phase dispersion. Of course, some of these techniques are continuing in certain analytical and/or non-analytical applications, but they are not used widely in food applications for which they were marketed.

2.4. Current and future trends

Any new approach has to compete in an uphill struggle with the “kings of the hill” in analytical chemistry. GC, HPLC, traditional selective detectors, MS, solid-phase extraction (SPE), and liquid-liquid extraction (LLE) are the current leading approaches in analytical food and agricultural applications. These techniques have usurped previous major analytical tools, such as thin-layer chromatography, Soxhlet extractions, tedious wet chemical methods, and non-selective GC detectors. The features and performance of the current leading technologies are established parameters, and any new technique will have to match or better them for a reasonable price. Are there any new technologies that can join, or even usurp, any of these “kings of the hill?”

Advantageous approaches that were introduced for bench-top operations in the last 15 years with strong applicability to food analysis include the major advances in HPLC/MS (and MSⁿ) and GC/MSⁿ, and other instrumental devices, such as programmable temperature vaporization (PTV), pulsed flame photometric detection (PFPD), halogen specific detection (XSD), and pressurized liquid extraction (PLE), which is also known as accelerated solvent extraction (ASE). Each of these techniques has been on the market for at least six years, and they provide benefits in breadth of scope, selectivity and/or detectability that are likely to make them useful for years to come.

Other potentially useful fairly new commercial devices for GC analysis of foods include large-volume injection (LVI), direct sample introduction (DSI) (commercially known as the ChromatoProbe), and resistively heated capillaries. These techniques are not yet established and it is not clear what their fate will be.

In the case of MS, its combination of qualitative and quantitative features gives it the advantages needed to become the biggest “king of the hill”, and some day, selective GC detectors will possibly be relegated to niche applications. The detectors with greater selectivity and/or sensitivity that complement MS, such as PFPD and XSD are likely to remain, and there is always a need for lower cost and reliable detectors that meet the needs of simpler analyses [24]. But the future of GC (and LC) detection and applications is tied with MS. The key question for MS will continue to be: how much extra capital expense will the laboratory pay to gain the benefits of MS?

3. Faster GC/MS

Increasing the speed of analysis has always been an important goal for GC separations. The time of GC separations can be decreased in a number of ways: 1) shorten the column; 2) increase carrier-gas flow; 3) reduce column-film thickness; 4) reduce carrier-gas viscosity; 5) increase column diameter; and/or, 6) heat the column more quickly. The trade-off for increased speed however is reduced sample capacity, higher detection limits, and/or worse separation efficiency. How much of these factors is the analyst willing to sacrifice for speed? Not much, apparently, because separation times in typical routine applications have been much the same for decades (20–50 min). Perhaps as more laboratories begin to use instruments with higher inlet-pressure limits, faster oven-temperature program rates, electronic pressure control, and faster electronics for detection, fast-GC with micro-bore columns will become more widely used, but the inherent trade-off will remain.

In practice, the GC conditions should be designed to give the shortest analysis time while still providing the necessary selectivity (i.e. separation of both analyte-analyte and matrix-analyte). The use of element-selective detectors may improve matrix-analyte selectivity, but, in that case, analyte-analyte selectivity must be

addressed solely by the separation. MS detection usually improves both types of selectivity (an exception includes dioxin and/or PCB analysis in which some congeners give similar mass spectra). Thus, GC/MS reduces the reliance on the GC separation and can lead to faster analysis times for a given list of analytes and matrices.

Chromatographers seem to have a dogma that each analyte in a separation should be baseline resolved, but MS provides an orthogonal degree of selectivity that is seldom used to its full potential in routine applications. The reliance on selective ion monitoring (SIM) and MS-MS, in which sequential segments are used in the analysis, also tends to extend chromatographic separations [25].

Full-scan mode is needed truly to meet the full potential of fast-GC/MS. Software programs, such as the automated mass deconvolution and identification system (AMDIS), which is available free from the US National Institute of Standards and Technology on the internet [26], have been developed to utilize the

orthogonal nature of GC and MS separations to provide automatically chromatographic peaks with background-subtracted mass spectra despite an incomplete separation of a complex mixture [27].

There are at least three approaches to fast-GC/MS: 1) use of micro-bore columns with time-of-flight (TOF)-MS [28–30]; 2) use of low-pressure (LP)-GC/MS to aid separations at increased flow rate [31–33]; and, 3) use of supersonic molecular beam (SMB)-MS (also known as Supersonic GC/MS), which can accept increased flow rates and short analytical columns [34–36]. The use of faster temperature programming in GC/MS with or without a shorter column is also always an option. Although fast-GC/MS is desirable in a variety of applications mentioned previously, these are newly developed approaches that have not been evaluated widely. One application for which each of these three approaches has been tested similarly is pesticide-residue analysis. As a result, the comparisons shown in Figs. 3–5 between

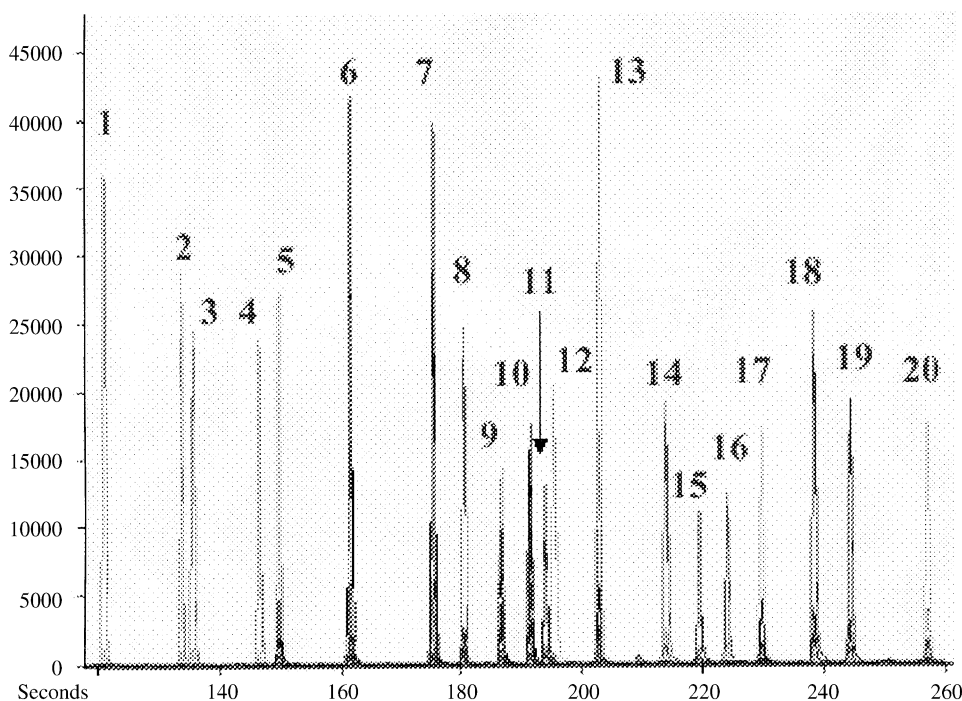


Fig. 3. Fast-GC/TOF-MS analysis of pesticides. 1) alpha-BHC, 2) gamma-BHC, 3) beta-BHC, 4) delta-BHC, 5) heptachlor, 6) aldrin, 7) isodrin, 8) heptachlor epoxide, 9) gamma-chlordane, 10) alpha-chlordane, 11) p,p'-DDE, 12) endosulfan I, 13) dieldrin, 14) p,p'-DDD, 15) endosulfan II, 16) p,p'-DDT, 17) endrin aldehyde, 18) endosulfan sulfate, 19) methoxychlor, 20) endrin ketone. Original figure from [29] provided by J. Cochran.

the different approaches are focused this application. The reader is directed to the literature for descriptions of other food applications [37–39].

3.1. GC/TOF-MS

An advantage of the micro-bore GC/TOF-MS method *versus* the other approaches is that separation efficiency need not be compromised for speed of analysis. Modern quadrupole instruments are capable of sufficiently fast scan rates for fast-GCMS [40], but quadrupole instruments cannot match the potential of TOF for this purpose. Rapid deconvolution of spectra (“scanrate”) with TOF-MS makes it the only MS approach to achieve several data points across a narrow peak in full scan operation. Fig. 3 gives an example of rapid GC/TOF-MS for the analysis of pesticides in a solution. However, the injection of complex extracts deteriorates the performance of micro-bore

columns quickly, and, since sample capacity is reduced by a cubed factor in relation to column diameter [41], increased LOQ and decreased ruggedness result, so such narrow columns can rarely be used in real-life applications.

TOF-MS can also give wide spectral mass range and/or exceptional mass resolution (typically at the expense of speed, however). Moreover, GC/TOF-MS techniques do not necessarily need to use short, micro-bore columns to achieve short analysis times. Short, wider columns, ballistic or resistive heating of columns, comprehensive 2-dimensional GC, and/or low pressure may become more suitable approaches to meet food-application needs in GC/TOF-MS in the future.

3.2. LP-GC/MS

LP-GC/MS, commercially known as Rapid-MS, is an interesting approach to speed the

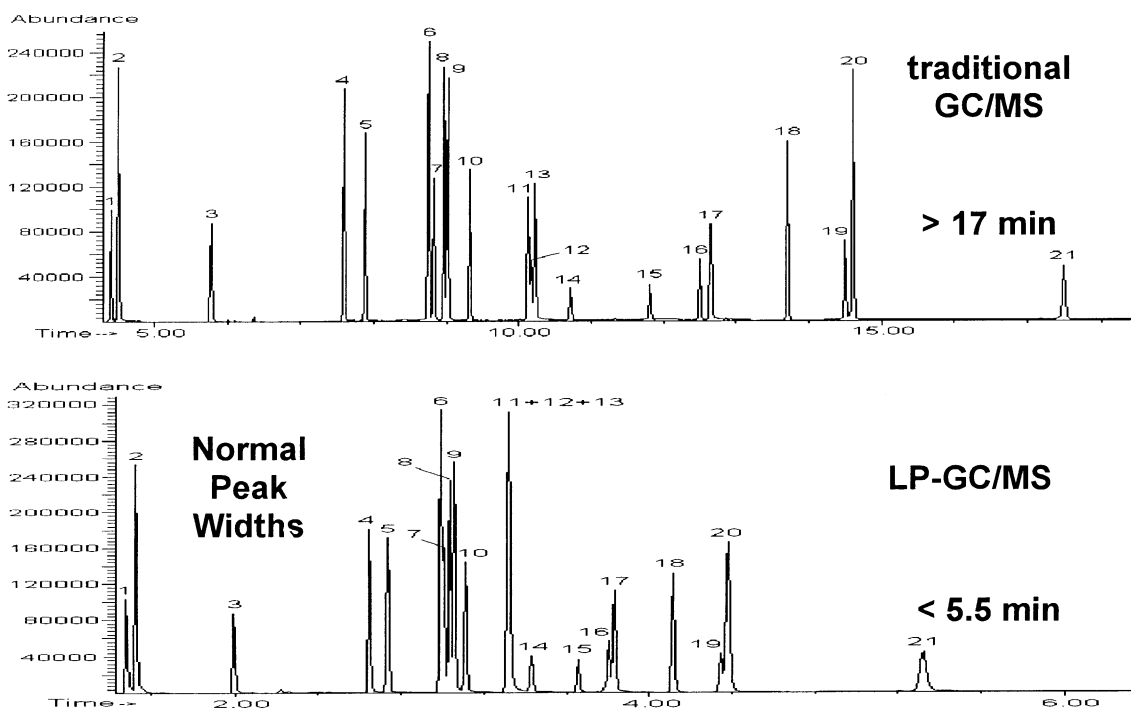


Fig. 4. Chromatogram of pesticides in toluene solution in conventional GC-MS and LP-GC/MS (5 ng injected). 1) methamidophos, 2) dichlorvos, 3) acephate, 4) dimethoate, 5) lindane, 6) carbaryl, 7) heptachlor, 8) pirimiphos-methyl, 9) methiocarb, 10) chlorpyrifos, 11) captan, 12) thiabendazole, 13) procymidone, 14) endosulfan I, 15) endosulfan II, 16) endosulfan sulfate, 17) propargite, 18) phosalone, 19) cis-permethrin, 20) trans-permethrin, 21) deltamethrin. Used from [32] with permission of the publisher.

analysis by which a relatively short (10 m) mega-bore (0.53 mm i.d.) column is used as the analytical column. The vacuum from the MS extends into the column, which leads to higher flow rate and unique separation properties. A restriction capillary (0.1–0.25 mm i.d. of appropriate length) is placed at the inlet end to provide positive inlet pressure and to allow normal GC injection methods. Advantages of LP-GC/MS include: 1) fast separations are achieved; 2) no alterations to current instruments are needed; 3) sample capacities and injection volumes are increased with mega-bore columns; 4) peak widths are similar to conventional separations to permit normal detection methods; 5) peak heights are increased and LOQ can be lower (depending on matrix interferences); 6) peak shapes of relatively polar analytes are improved;

and, 7) thermal degradation of thermally-labile analytes is reduced.

Fig. 4 shows how a three-fold gain in speed was made in the analysis of 21 representative pesticides using LP-GC/MS *versus* traditional GC/MS. Larger injection volume could be made in LP-GC/MS because of better focusing of the gaseous solvent at the higher head pressure and larger column capacity, so overall gains in sensitivity were achieved. However, reduced separation efficiency occurs with LP-GC/MS and ruggedness of the approach with repeated injections was no better than traditional methods with a narrow-bore analytical column.

3.3. GC/SMB-MS

GC/MS with current commercial instruments have a practical 2 mL/min flow limitation because of MS-instrument designs. GC/SMB-MS is a very promising technique and instrument that overcomes this flow rate limitation because SMB-MS requires a high gas-flow rate at the SMB interface. However, only a single prototype GC/SMB-MS instrument exists at this time, and the approach is not commercially available.

The advantages of GC/SMB-MS include: 1) the selectivity of the MS detection in electron-impact ionization is increased because an enhanced molecular ion occurs for most molecules at the low temperatures of SMB, so losses of selectivity in the GC separation can be made up by increased selectivity in the MS detection; 2) the use of very high gas-flow rates enables GC analysis of both thermally labile and non-volatile chemicals, thereby extending the scope of the GC/SMB-MS approach to many analytes currently done by HPLC; 3) the SMB-MS approach is compatible with any column dimension and injection technique; 4) reduced column bleed and matrix interference occurs because of the lower temperatures and enhanced molecular ions; and, 5) better peak shapes occur because tailing effects in MS are eliminated. Fig. 5 gives an example in the separation of diverse pesticides using GC/SMB-MS.

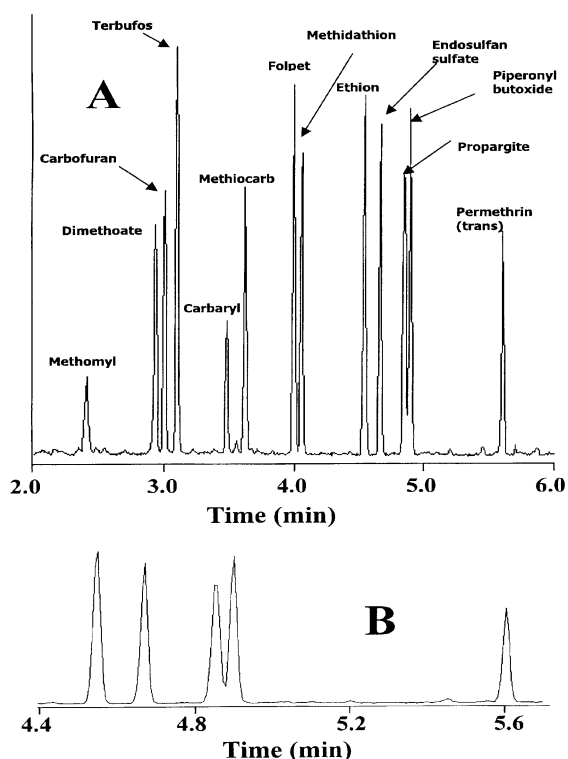


Fig. 5. Fast-GC/SMB-MS analysis of the indicated 13 pesticides in methanol (3–7 ng injected). Trace B is a zoom of the upper trace A in order to demonstrate the symmetric tailing-free peak shapes. A 6 m capillary column with 0.2 mm i.d., 0.33 μm DB-5ms film was used with 10 mL/min He flow rate. Used from [34] with permission of the publisher.

4. Conclusions

After 50 years of commercial GC, the technology and its applications have matured, but we have not reached an end of the possibilities made available by GC or the ever-expanding analytical needs it can address. There is always a need for higher quality and more practical GC methods in existing applications, and much remains to be discovered about the importance of chemicals on health and the environment.

As a result of the current emphasis by funding organizations and industry in biological and biochemical investigations, it may seem that HPLC is going to supplant most GC applications, but usually the reality is that "when GC can be used in a separation, GC should be used." No other current technique can match its combination of separation efficiency, instrument performance and reliability, range of detectors, analytical scope, understanding of the theory and practice, means to control separation, ease of use, diversity of features, reasonable cost, and the number of analysts experienced in the approach.

In the near future, GC/MS is expected to supplant many current methods for chemical contaminants using selective GC detectors, and GC/MS will be especially useful if it can be combined with fast-GC separations. The increased selectivity of MS reduces the need to achieve baseline-resolved separations as with selective detectors, so faster separations of lower chromatographic resolution are still useful. Three fast-GC/MS techniques that may become useful for this purpose are LP-GC/MS, GC/TOF-MS, and GC/SMB-MS, and it will be interesting to see which of these approaches will become the most widely used in food applications in the future.

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