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COMPREHENSIVE INTERFACED PYROLYSIS GAS CHROMATOGRAPHIC PEAK IDENTIFICATION SYSTEM

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SUMMARY

A versatile interfaced pyrolysis gas chromatographic peak identification system has been set up. It incorporates instrumentation for thermal degradation under slow or ultra-rapid temperature rise conditions. Evolved volatiles are transferred to a master trap manifold where precolumn procedures may be applied prior to gas chromatographic separation. Identification and analysis of individual peaks is then performed on-the-fly by vapor phase infrared spectrophotometry, mass (molecular weight) chromatography, elemental analysis, and functional group fingerprint fragmentation. An interfaced laboratory computer system provides for data acquisition, reduction and control.

INTRODUCTION

Gas chromatography (GC) has established a primary role as an analytical separation technique during a period of more than two decades, being amenable to any chemical species which maintains its integrity in the vapor phase. While separation and resolution with associated qualitative and quantitative analysis have always been paramount, it has always been clear that GC alone can provide absolute identification of species only indirectly or by inference. The use of retention data frequently provides adequate identification of unknowns whose probable molecular class or structure is already known or suspected, but is seldom sufficient in other circumstances. Two alternative approaches are available to provide unequivocal proof of identity of eluted species; the component peak may be collected in sufficient quantity to enable it to be subjected to subsequent identification procedures using any appropriate methodology; alternatively, eluate together with or without mobile phase may be passed directly into some identification device. The general applicability of such ancillary techniques has been well covered in the text by Ettre and McFadden[1]. and it is clear that virtually any physical or chemical identification method may be used in conjunction with GC.

The power of mass spectrometry (MS) has been effectively exploited with regard to the directly interfaced GC identification mode[2]. Normal criteria of mass spectral identification naturally apply and while confirmation of known species is often straightforward, problems inherent in the GC–MS interface or in the ionization

of the molecule itself may often preclude definitive conclusions. The simultaneous achievement of qualitative and quantitative data for multicomponent mixtures or labile species is often impossible. While such techniques as chemical ionization (CI) mass spectrometry and programmed multiple-ion-current monitoring are useful, they also add to the already considerable expense and complexity of the technique. Alternative on-the-fly measurements on eluates in GC have been much less explored[1], but a number offer substantial advantages notably when used in combination; additional facility for on-the-fly chemical reaction to be included may also be most helpful in elucidation of the data produced.

An area of particular importance where eluate identity is concerned is that of pyrolysis gas chromatography (PGC) or more generally evolved gas analysis (EGA), the latter having been primarily considered as the counterpart to thermal gravimetric analysis (TGA). PGC has been much applied in the elucidation of polymer degradation products[3,4], but the general application of the technique to gaseous, liquid, or solid samples can give valuable information on structure from observed fragmentation patterns[5,6]. A major problem, in common with most thermal analysis has been one of reproducibility, both qualitative and quantitative. Instrumentation for slow programmed thermal degradation is now available[7]. This method has been shown to produce minimal secondary fragmentation products. The alternative approach of using ultra-rapid rise-time temperature profiles can now give a high degree of reproducibility in pyrogram profiles[4].

The present paper describes the conception, implementation and some initial applications of a novel GC peak identification system. Volatile samples may be introduced directly, may be evolved by thermal degradation under slow temperature gradient conditions, or may be produced by pyrolysis with temperature rise rates of up to 20,000°/sec, in any desired atmosphere. Volatiles are transferred to a master trap manifold operable to 300° which provides for the selection of the course of the analytical sequence to be followed. On-line chemical reactions may be performed, components separated chromatographically, and the nature of individual peaks determined by means of the following valve selectable interfaced procedures:

(i) rapid scan vapor phase IR spectrophotometry
(ii) elemental analysis for carbon, hydrogen, oxygen, nitrogen, sulfur, etc.
(iii) functional group fingerprinting by vapor phase thermal cracking
(iv) molecular weight determination by differential gas density measurement.

Any or all of these techniques may be applied in series or in parallel for identification of a component peak. An interfaced laboratory computer system is available for data acquisition, reduction, and control. Central to the concept is the selectability of analytical configuration as most relevant to the problem in hand, based on information obtained during the course of the analysis. The analyst may choose the next identification step on the basis of data from the previous step. In addition, multiple flow paths also enable several routine analyses to be carried out simultaneously.

INSTRUMENTATION AND SYSTEM DESIGN

An overall system diagram is shown in Fig. 1. Samples may be introduced at several points; injection may be made into the MP-3, the CDS 820, the CDS 1200, the MC-2, or through an injection port in the trap manifold connected directly to the

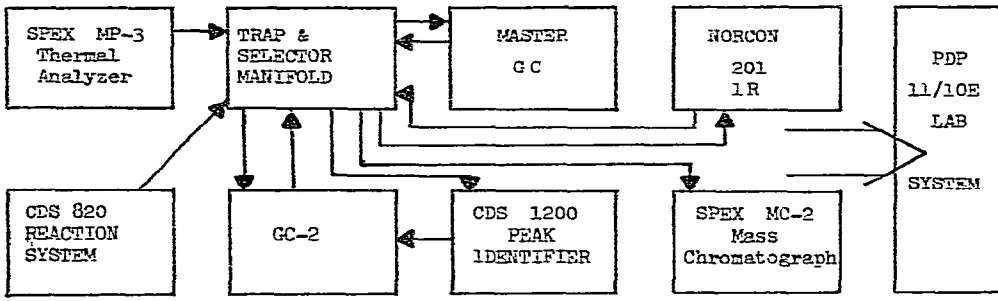

Fig. 1. System block diagram of the interfaced peak identification system.

master GC. Samples may be pyrolyzed in the MP-3, in the CDS 820 pyroprobe system, or in the trap manifold injection port. The MP-3 incorporates an internal gas chromatograph allowing stand-alone evolved GC, while the CDS 820 can utilize GC-2 for stand-alone operation. All samples enter the identification system through the trap manifold, where the course of the analysis is selected. The manifold also allows valve selection of several precolumn reactors and trapping sequences specific for individual problems. After GC separation, the peaks may first be characterized by vapor phase IR spectrophotometry, then return to the trap manifold, where either vapor phase pyrolysis (catalytic for elemental analysis or "thermal" for structural determination) in the CDS 1200, or molecular weight determination in the MC-2 may be carried out.

In order to make clear the operation of the system, a detailed discussion of individual instruments and system design follows. As noted above, the system comprises seven instruments, some appropriately modified. They comprise the following.

MP-3 multipurpose thermal analyzer (thermal chromatograph)

The MP-3 (Spex Industries), designed to produce evolved volatiles profiles with temperature increase followed by GC analysis of the profiles, was modified by the addition of two laminar flow controllers (HGC 187; Analabs) which provide controlled atmospheres in addition to helium as desired for thermal degradations. The three streams are all divertable for measurement of flow and meet at a mixing cross prior to the pyrolysis chamber. The three rear vents were removed and all streams pass to the trap manifold through standard transfer lines.

CDS 820 reaction system (incorporating CDS pyroprobe 100)

The CDS 820 (Chemical Data Systems) provides the controlled atmosphere for the Pyroprobe pyrolysis system. The standard heated interface for the pyroprobe was replaced by a Perkin-Elmer 900 series GC injection port which was installed in place of the reactor inlet of the CDS 820, allowing direct insertion of the probe. This solved leakage problems and allowed better control of pyroprobe temperature before pyrolysis. In addition, a water-cooled aluminum block can be placed over the exposed portion of the port to allow low probe temperatures to be maintained before pyrolysis. Further details of this cooled port design and operation will be reported elsewhere.

Master gas chromatograph

A Varian 2760 instrument with thermal conductivity and flame ionization detection was chosen for the large oven space and the ease with which major modifications could be made to the injector and detector systems. Injection ports were removed and two valves mounted to provide column switching and stop-flow capability, as shown in Fig. 2. The eight-port column switch and ten-port stop-flow valve are Valco Instruments HT series valves, heated by 50-W cartridge heaters controlled by the original Varian injection port heaters. Thermal safety switches were adjusted to prevent valve overheating. Swagelok bulkhead unions were mounted on an aluminum plate inside the oven to provide connections for two columns. Internal transfer lines were 0.016 in. O.D. stainless-steel (SS) tubing inside 1/8 in. O.D. copper tubing to ensure even heat transfer, bundled and heated with nichrome wire. The aluminum column bulkhead was heated by a 50-W cartridge heater to maintain efficiency for high-boiling species. The entire injection area was insulated with Johns Manville glass micro fibers. The detectors were arranged to provide three configurations, TC only, FID only, and TC split to FID, selected by the appropriate connection of the sample and reference streams from the ten-port valve to the detector fittings. Input and output are through the standard heated transfer lines.

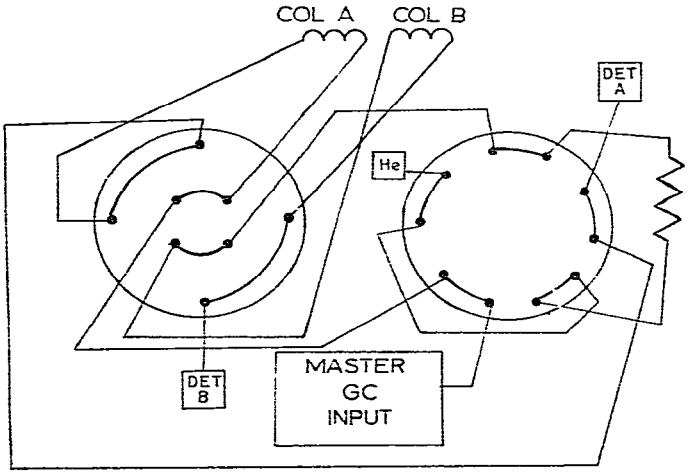

Fig. 2. Flow system of the master gas chromatograph stop-flow and column switching valve.

GC-2

The second gas chromatograph (a Varian 2760) has a detector arrangement identical to that of the master GC. It serves for analysis of samples from two sources, pyrolysis products from the CDS 820, and from the structural determination function of the CDS 1200. The former enter through a transfer line from the trap manifold and pass through the detector oven to a bulkhead fitting at the rear of the column oven. The latter pass through a transfer line from the CDS 1200 to a bulkhead fitting at the front of the column oven, the injection ports being moved to the right to accommodate this line in the usual column A position.

CDS 1200, functional group and elemental analyzer

The CDS 1200 (Chemical Data Systems) is present in the system with no modification. The instrumental transfer line is used for sample input, and a minimum length transfer line to GC-2 was made in the standard fashion.

MC-2 mass chromatograph

The MC-2 (Spex Industries) has been modified only by the addition of a 1/16 in. "tee" in the sample injection line for the transfer line input. The Spex transfer line enters through the detector section at the rear of the instrument. A helium purge flow is maintained through this transfer line and this interface does not interfere with the normal syringe injection of samples into the instrument.

Norcon 201, vapor phase IR spectrophotometer

The 201 (Norcon Instruments) has been modified to adapt it to the system. The output stream from the sample cell is returned to the trap manifold for further analysis. The original transfer lines were replaced with 3 1/16 in. SS lines in a single 3/8 in. heated copper tube to provide sample and reference flow and sample return. The return line is 0.043 in. I.D. to minimize back pressure, and all transfer lines are connected as close as possible to the "sample hold" valve in the 201. The computer data output and synchronizing signals provided were found not to be optimal for direct interfacing to the PDP-11/10e computer. An interface board was added, consisting of a voltage divider for the synch (VALID) signal and an adjustable gain amplifier (AD536J, Analog Devices), to invert the signal output and adjust it to make maximum use of the A/D converter sensitivity. This also facilitated auto gaining of data such that the switch to higher gains would occur near 100% T rather than near 0% T.

Transfer lines

These were constructed of 1/16 in. O.D. by 0.016 in. I.D. stainless steel where band broadening was a factor to be minimised, and of 1/16 in. O.D. by 0.043 in. I.D. stainless steel where samples were only to be trapped or where back pressure was a factor. Grade 304 stainless steel was used (Microweld) and welded 1/8 in. to 1/16 in. terminations (Microweld) were used for connections to 1/8 in. fittings in instruments. The 1/16 in. lines were passed through larger copper tubing to facilitate uniform heating. 1/8 in., 1/4 in., or 3/8 in. O.D. tubing was used to carry one, two or three, and more than three lines, respectively. The copper tubing was wrapped with glass electrical tape (Scotch No. 69, 3M Company) for electrical insulation, and a type J or K thermocouple (Omega Engineering) was installed in the line and insulated with tape. Heat was provided by 26 gauge glass insulated nichrome wire (Bergquist) wrapped tightly with one wire separation. Another layer of electrical tape was added and the line inserted into a silicone rubber sleeve for thermal insulation (Spex Industries).

Transfer line heating is controlled by Powerstat variable-voltage transformers mounted on aluminum chassis boxes. All lines are fused and the thermocouples are read on auxiliary positions on the various instruments; lines are normally maintained at approximately 300°. On lines longer than a few feet, the heating wire was divided at even intervals which were then connected in parallel to the powerstat. The hot lead

from the transformer was connected to the center of the lines and the common lead to the ends.

Trap manifold

This contains the valve oven where all sample switching is done. It also houses ports for ten traps and four precolumns and contains three sample loops. The six valves in the valve oven are Valco HT valves. The valve oven was constructed from a $21\frac{1}{2} \times 15 \times 13$ in. steel chassis box, and the valves mounted on aluminum bars insulated from the exterior of the cabinet by ceramic insulators. An aluminum plate mounted similarly provides support for the $28\frac{1}{8}$ in. SS bulkhead Swagelok unions to which traps and precolumns are attached. Transfer lines enter through the back and sides and the oven is insulated with glass microfibres held in place where necessary by $\frac{1}{2}$ in. galvanized wire cloth. An approximately 1 in. thickness of this insulation was found adequate to keep the exterior of the manifold cool. The oven is heated by a 700-W heater with an associated blower and temperature is controlled by a CDS 200 temperature controller employing a platinum resistance sensor (Omega Engineering). Temperature control for the traps and precolumns is provided by a trap monitor constructed in a separate chassis, containing a switching pyrometer (Assembly Products) to turn off trap heat at a set limit and a 12 position switch for selection of any of the traps or precolumns for readout. The switch also connects the output of a powerstat to the selected trap. A second powerstat provides heat for the precolumns but is not switched by the pyrometer, thus allowing continuous heating of the precolumns. A voltage meter is provided for resetting trap and precolumn voltages. A 28-V d.c. power supply in the monitor provides power for the necessary relays and indicators.

Traps and precolumns

The traps and precolumns used on the trap manifold were constructed of $\frac{1}{8}$ in. O.D. stainless steel, the construction paralleling that of the transfer lines except that no insulation was added over the nichrome wire, to allow more rapid heating and cooling. Traps have been packed with standard chromatographic column materials, 10% SE-30 on Chromosorb W, Porapak Q, glass microbeads, and a combination trap was constructed with 10% SE-30 followed by Porapak Q in the same trap. The latter has been found useful as a general purpose trap for most volatiles except light hydrocarbons and permanent gases at room temperature, even methane being retained for a short period.

PDP 11 data system

This consists of a PDP 11/10e laboratory computer system with the LPS data acquisition system. The LPS contains sixteen channels of multiplexed 12-bit A/D with the BA 408 switched gain multiplex system (Digital Equipment). Other peripherals include a single RK05 disk, a TU60 cassette, and a LA36 terminal. A Hewlett-Packard 130B oscilloscope is used for data display from the D/A converter of the LPS and analog output is also directed to an Omniscribe recorder (Houston Instruments) for hard copy. Input data are obtained from the computer outputs of the Varian 2760 gas chromatographs and from the previously described interface to the Norcon 201. The remaining instruments do not have direct computer output terminals and

data are presently obtained by connection across the detector attenuators. This approach will be modified as the data buffering requirements are determined.

System software includes the RT 11 foreground/background operating system, FORTRAN, and Lab Applications Program Library VO3. The latter includes SPARTA and THRU programs for preliminary data acquisition and modules for tailoring data acquisition to the needs of the system. These are being used to generate a program to acquire all data under one program in the foreground and perform peak integration and other data reduction functions. A new data acquisition program, IRFLAV, provides for IR data acquisition in the foreground with digital filtering. Another routine, IRSOUT, performs background subtraction and normalization, and prints out the spectrum to the recorder. This program may be operated under the RT 11 BATCH system for output of large accumulations of data. A FORTRAN program used for data reduction from the MC-2 mass chromatograph is discussed elsewhere[8].

System design

The design of the overall system grew from an in depth study of the application requirements of the seven component instruments. The trap manifold was designed to optimize flexibility and throughput of the instruments, consideration being given to the need for several users to work with different instruments with a minimal degree of interference and with no degradation of individual instrumental performance. The flow schematic of the valves in the trap manifold is shown in Figs. 3 and 4. The six valves have been divided for clarity between the two diagrams. Fig. 3 shows the input of samples into the system via the INPUT valve and the selection of samples for identification by means of the two remaining valves.

An important consideration in design of this type of interfaced system is the destination of all flowing streams in all switch positions. It is important that no flows to thermal conductivity detectors be stopped lest filaments be damaged, thus makeup flow must be provided and streams not selected for identification must be vented at

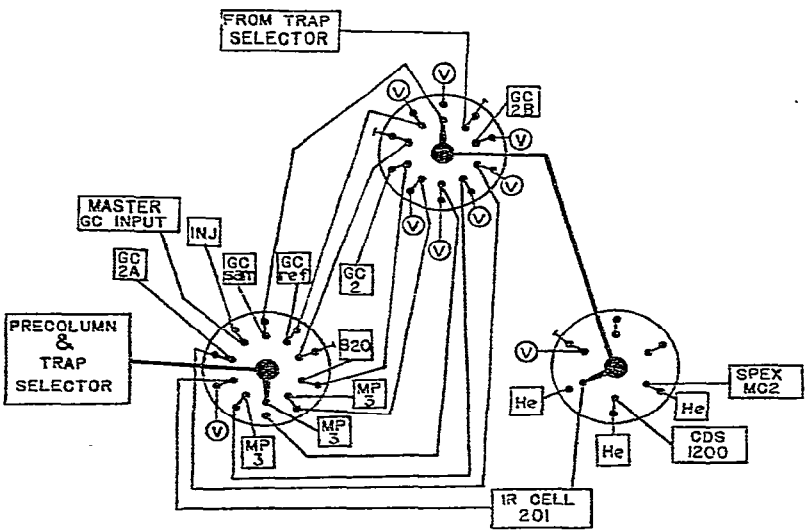

Fig. 3. Flow system of the input and identification selection valves of the trap manifold.

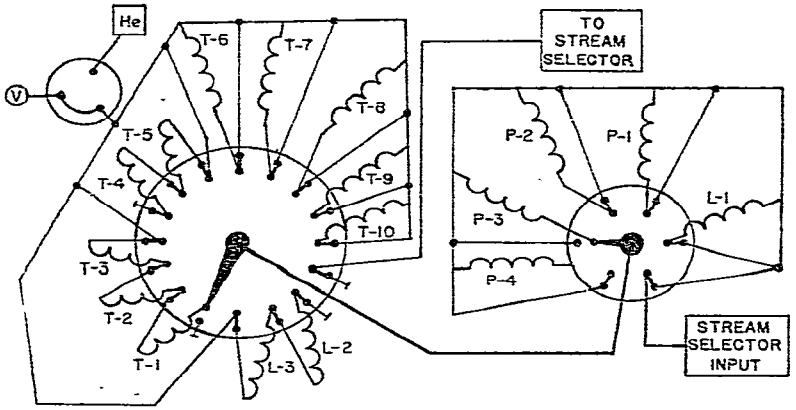

Fig. 4. Flow system of the precolumn and trap selection valves of the trap manifold.

all times. Samples entering the INPUT valve may either be selected for trapping or passed into the identification selection section. This arrangement allows effluent from the gas chromatographs to be trapped as well as identified, this being particularly useful with the precolumn reactions. It is also possible to pass an entire evolved gas envelope to any of the identification instruments without prior separation and trapping. Thus two valves are used first to select the stream for analysis and then the instrument for analysis. All streams not selected in this section pass to vents which are centrally located for convenient flow measurement. Two exceptions are the reference flow to the Norcon 201 which vents at the end of the reference cell, and the effluent from the CDS 820. Since the latter does not contain GC capability and does not therefore stand alone, its exit line leaves the selection system and passes to GC-2. These thus act together as a PGC system while the MP-3 is at the same time providing samples for identification to the remainder of the system.

The trapping and precolumn valving system shown in Fig. 4 was designed to provide maximum flexibility of analysis in complicated identification problems. The use of precolumns as subtractors or reactors has been demonstrated as valuable in complex analyses[1]; thus a switch selection of four precolumns is provided. In the bypass position these are completely isolated. A sample loop within the oven is also provided as part of this valving to allow 30-ml volumes of effluent to be held without trapping. Samples may be subjected to precolumn reactions either on the trap cycle or the backflush cycle or both. In addition, the bypass line from the trap valve to the selector valve allows samples to be passed through the precolumns and then directly to the identification function.

The selector valve for the traps allows any of the ten traps or two sample loops to be selected. Five of these traps are "single user" traps designed to allow various facilities for specific research areas; this aids in minimizing memory effects and carry over from one sample type to the next. The other five traps are general use system traps providing for various process chromatographic applications. Traps 6 and 7 and also traps 8, 9 and 10 are designed for series trapping followed by parallel backflush. In this mode a preliminary separation by either volatility or column affinity may be done at the trapping stage and then the appropriate column may be selected for anal-

ysis of the various fractions using the column selector valve. By determining the retention times of species on the traps, it is also possible to perform operations such as heart cutting using this valve system.

EVALUATION AND DISCUSSION

The quality of the chromatographic separations obtained in the interfaced system shows little or no deterioration from that obtained on the MP-3 alone or by the CDS 820 interfaced to the Varian 2760 gas chromatograph (GC-2). Preliminary evaluations were carried out using a mixture of normal hydrocarbons in the range C_7–C_{24}. This mixture was chromatographed in the master GC on a 6 ft. × $^1/_8$ in. O.D. column of 1.5% OV-101 on Chromosorb G, with a constant helium flow-rate of 20 ml/min and a 20°/min temperature program from 50–300°. Column efficiency was closely similar for the C_8 and the C_{24} alkanes and there was no evidence of band broadening or sample holdup in the transfer line from the MP-3 or in the trap manifold on the basis of plate number measurements, as compared with efficiency in the internal MP-3 gas chromatograph. The problem of hot spots in transfer lines due to excess insulation or passage through a bulkhead has not yet been addressed specifically using compounds known to be thermally unstable; however, the copper heat transfer tubes should minimize this problem as they do for the cold spots. In fact, only a single serious cold spot was found in the initial set up stage, at the aluminum column bulkhead of the master GC; this was satisfactorily corrected by incorporation of a 50-W cartridge heater at this point.

Further analysis of the *n*-alkane mixture injected into the MP-3 and initially trapped and separated in the master GC was carried out on the MC-2. The sample was transfered a second time through the trap manifold to the MC-2 traps and analyzed in the standard manner[8–10]. CO_2 and Freon 115 carrier gases were employed at flow-rates of 10 ml/min on matched 6 ft. × $^1/_8$ in. O.D. 8% Dexsil 300 GC columns on Chromosorb W-HP, using a program rate of 10°/min from 50–325°. The C_{12}–C_{24} portion of this chromatogram is shown in Fig. 5. Molecular weights calculated using the expression

$$M_x = \frac{(A_1/A_2)\,K \cdot M_{CG1} - M_{CG2}}{(A_1/A_2)\,K - 1}$$

where A_1/A_2 is the peak height response ratio of the compound in the two detectors, 1 and 2, M_{CG1} and M_{CG2} are the molecular weights of carrier gases 1 and 2, and K is the instrument calibration factor obtained by measuring responses for known compounds, and using the expression

$$K = (A_1/A_2)\left(\frac{MW_{Standard} - M_{CG1}}{MW_{Standard} - M_{CG2}}\right)$$

gave comparable precision for molecular weights to those obtained from direct injection of the hydrocarbon mixture into the MC-2 in the conventional way. The relative precision of the order of 2% so obtained may be improved by up to a factor of ten by using an iterative computer curve fitting technique to account for the variation of the instrumental K factor with molecular weight[8]. It is noteworthy that the

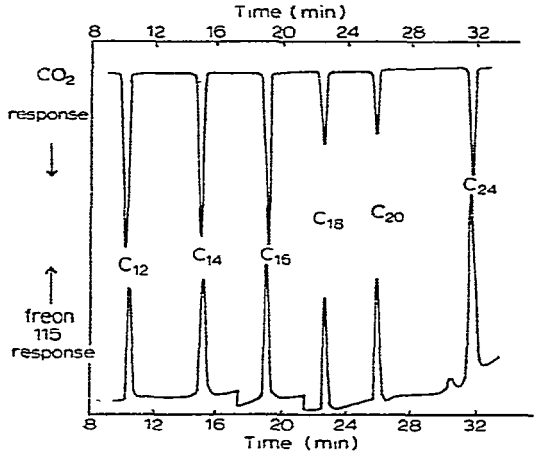

Fig. 5. Mass chromatogram of C_{12}–C_{24} *n*-alkanes initially injected into the MP-3 and transferred to the MC-2 as described in the text. Carrier gases, CO_2 and Freon 115; other chromatographic conditions, as given in the text.

flow and transfer path employed for the *n*-alkane sample in this particular example is as lengthy as is possible in the system, involving as it does transfer between three instruments, each involving passage through the trap manifold.

Several advantages have become immediately apparent in using this system. Firstly, the sample throughput for the MP-3 is virtually doubled by introducing the external trapping and gas chromatograph, since one sample may be pyrolyzed while another is chromatographed. Second is the advantage in the ability to rechromatograph the same sample several times by trapping the GC sample output. This allows analysis of a single sample before and after the use of a pre-column. It also allows the same sample to be sequentially analyzed on more than one GC column. This makes more efficient use of sample preparation time and helps eliminate problems of reproducing sampling and pyrolysis parameters.

An evaluation of the trapping and precolumn system was carried out using a sample of Green River oil shale which was pyrolysed separately in the MP-3 and in the CDS 820 and evolved volatiles passed through the various flow paths. The identical column was used (1.5% OV-101) as noted above with a carrier gas flow-rate and a temperature program of 12°/min from 30–300°. Fig. 6 shows the programmed temperature chromatogram obtained for the pyrolysis products produced from the thermal degradation of the oil shale in the MP-3 under helium with the 2-mg sample heated to 550° at 40°/min. Chromatogram A shows the separation of the original decomposition products, which consists of the typical unresolved component envelope upon which are superimposed a number of resolved and partially resolved components, considered to be primarily aliphatic, aromatic and unsaturated hydrocarbons[11]. The unresolved envelope is considered to comprise the non-hydrocarbon portion of the shale oil volatiles, including nitrogen, oxygen, and sulfur containing species. Chromatogram B is that of the eluent from A after passage through a Molecular sieve 5A 9 in. × $^1/_8$ in. O.D. SS precolumn at 275°. A large proportion of components in A are reproducibly removed; this type of precolumn has been shown to

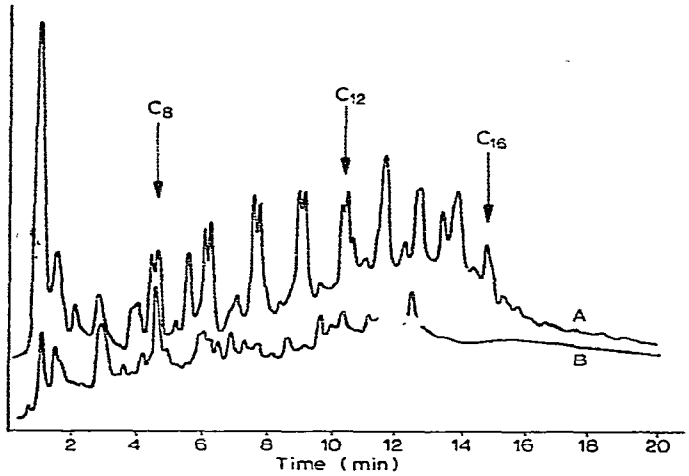

Fig. 6. Chromatograms of Green River oil shale pyrolyzates from the MP-3. (A) Volatiles transferred through the trap manifold and separated on the master GC. (B) Residual volatiles after passage of eluent from A through molecular sieve 5A trap. Chromatographic and pyrolysis conditions are given in the text.

remove preferentially normal and terminally branched molecules[12]. A variety of other specific precolumns have been used in this oil shale analysis[11,13] and are proving of great utility in the selective simplification of this exceedingly complex pyrolysis product mixture.

Preliminary data for the application of the Norcon 201 to the identification of unknowns is encouraging, very clean spectra of extracted material from shale oil having been obtained and presented elsewhere[11]. Data for methyl salicylate was obtained from a chromatographed peak of a 1% solution in acetone separated on the 1.5% OV-101 column at 130° with a 30 ml/min helium flow-rate of carrier gas. After determination of the delay time for transfer to the Norcon 201, scans were taken of the IR spectrum using the 6-sec scan mode and 3× scale expansion. Data were collected both on the Norcon 201 recorder and using the assembly language program IRFLAV. Spectra are presented in Fig. 7, the upper three traces showing output from the recorder directly while the lower trace shows the computer generated spectrum using IRFLAV and IRSOUT programs. This well defined spectrum corresponds to 5 μg of sample injected, and further optimization of the interface and of the Norcon 201 optical system should extend this limit to below the microgram level.

For the present the CDS 1200 unit has not been extensively utilized in the fully interfaced mode. Its operation and application have, however, been described by Liebman *et al.*[14], and we consider that the data obtained in the present configuration should be entirely equivalent to those reported by these authors.

CONCLUSIONS

The present paper is primarily devoted to discussion of the rationale behind the design and development of an interfaced vapor phase thermal analysis and GC peak identification system; the advantages of this type of approach to real laboratory

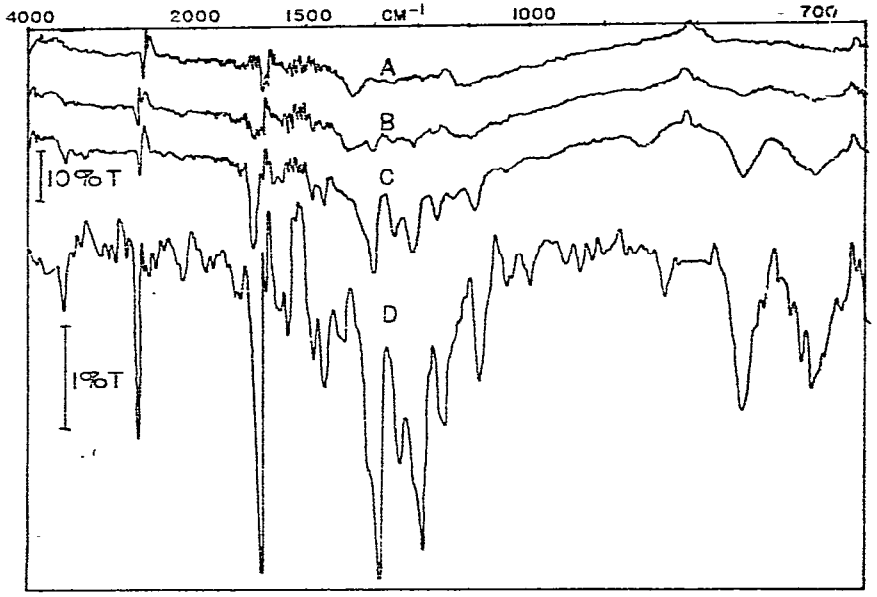

Fig. 7. Vapor phase IR spectra of eluted methyl salicylate peaks. Scan time on the Norton 201, 6 sec; range, 3×; other conditions, as given in the text. A = Background; B = 5 μg; C = 20 μg; D = computer printout of spectrum B after digital filtering, background subtraction, and normalization (from computer routines IRFLAV and IRSOUT).

problems are readily apparent. While a principal spur to the development of the system has been the interest in applications requiring pyrolysis or thermal degradation followed by evolved gas analysis by GC, a parallel involvement has been in the implementation of versatile on-the-fly peak identification procedures presently available but little developed or exploited in the interfaced mode.

Many interfaced systems utilize single identification instruments, but while this approach is sometimes adequate, it may lack sufficient versatility to provide adequate characterization of many unknowns particularly when present in poorly resolved multicomponent mixtures. The development of the interfaced system incorporating multiple identification capabilities is a logical advance, and when used in combination with on-line chemical precolumns and selective trapping devices it promises to be a very powerful and widely applicable system.

Thus far, development has concentrated primarily on the instrumental interfacing and the valving and peak transfer and reaction design. Some of the capabilities of the system have been assessed. Development is continuing and will be reported in future publications, as will present applications, particularly in the areas of oil shale analysis, polymer degradation, applications to fire retardant studies, and investigations of thermal properties of metal complexes and organometallics.

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