INTER-OFFICE CORRESPONDENCE

Hooker Chemical Company

To: J. R. Hopper
H. K. Kaster
J. F. Knight
P. Fiedler

Date: March 30, 1981

From: J. G. Parsons

Subject: Shell Toll Fee

RECEIVED
APR 1 1981
J. R. HOPPER

Effective April 1, 1981, and until further notice, the toll fee for VCM from Shell will be 15.5¢/pound. This equates to a chlorine value, including pipeline charges, of $78/ton.

J. G. Parsons

JGP/akf
0429m

PC - JFK - file admin cont. 4-2-81

Hooker
RUCO DIVISION

INTER-OFFICE MEMORANDUM

VCM

To: D. A. Bloomfield

Copies to: B. Harrison, W. Howells, H. Kaster, A. Katona, J. Ruffing, M. Soble, A. Tybus, P. Weill, W. Wetzel

File Ref.: RJA: 293-74M

Date: April 17, 1974

From: R. J. Abramowitz

Div./Dep't: RUCO/Polymers

Subject: SPI AD HOC VCM/PVC MEETING

Location: Burlington

ABSTRACT

A meeting of the special SPI Ad Hoc VCM/PVC Group was held in Washington, D. C. on April 16th, 1974 for the purpose of organizing a coordinated industry response to the recent OSHA "Emergency Temporary Standard For Exposure to Vinyl Chloride." A copy of the agenda is attached. There were approximately 50 attendees.

Monitoring of VCM in plant areas and in polymers is being conducted vigorously in all plants. The feeling was that OSHA is moving towards a permanent standard of zero exposure, with a possible imminent revision of the temporary standard to a level somewhat below 50 ppm of VCM in work areas.

A committee was appointed to formulate a response to OSHA which will stress:

1. The satisfactory human experience in properly regulated VCM/PVC plants in operation for more than 20 years.
2. Time required by industry to conform to new OSHA regulations.
3. Support of additional animal exposure test at levels below 50 ppm of VCM.
4. Need for accurate definition of test methods, test equipment, protective devices, etc.
5. An economic impact statement regarding effect on the country of zero production.

The group unanimously agreed that the entire industry would shut down if the regulations called for a maximum VCM exposure of 1 ppm. Two or three producers thought they could live with 30 ppm (TWA), if absolutely necessary, while a majority agreed that they would find a way to cope with a level of 40-50 ppm.

It was agreed that more VCM data is required in PVC processing areas and this will be accumulated.

CBS is planning a one hour TV Documentary on the Vinyl Chloride-PVC problem. Industry was urged not to obstruct this effort for PR purposes.

RJA/kr

R. J. Abramowitz

OCC 1561

FORM NO. 98-086

Hooker
RUCO DIVISION

INTER-OFFICE MEMORANDUM

To: R. J. Abramowitz
C. W. Corbin
L. J. Friedman
Copies to: B. P. Hirsch
W. D. Howells
H. H. Kaster
A. W. Tybus

File Ref.: RVL:398-73M
Date: June 20, 1973
From: R. V. Lucke
Div./Dep't: RUCO/Polymers
Location: Burlington

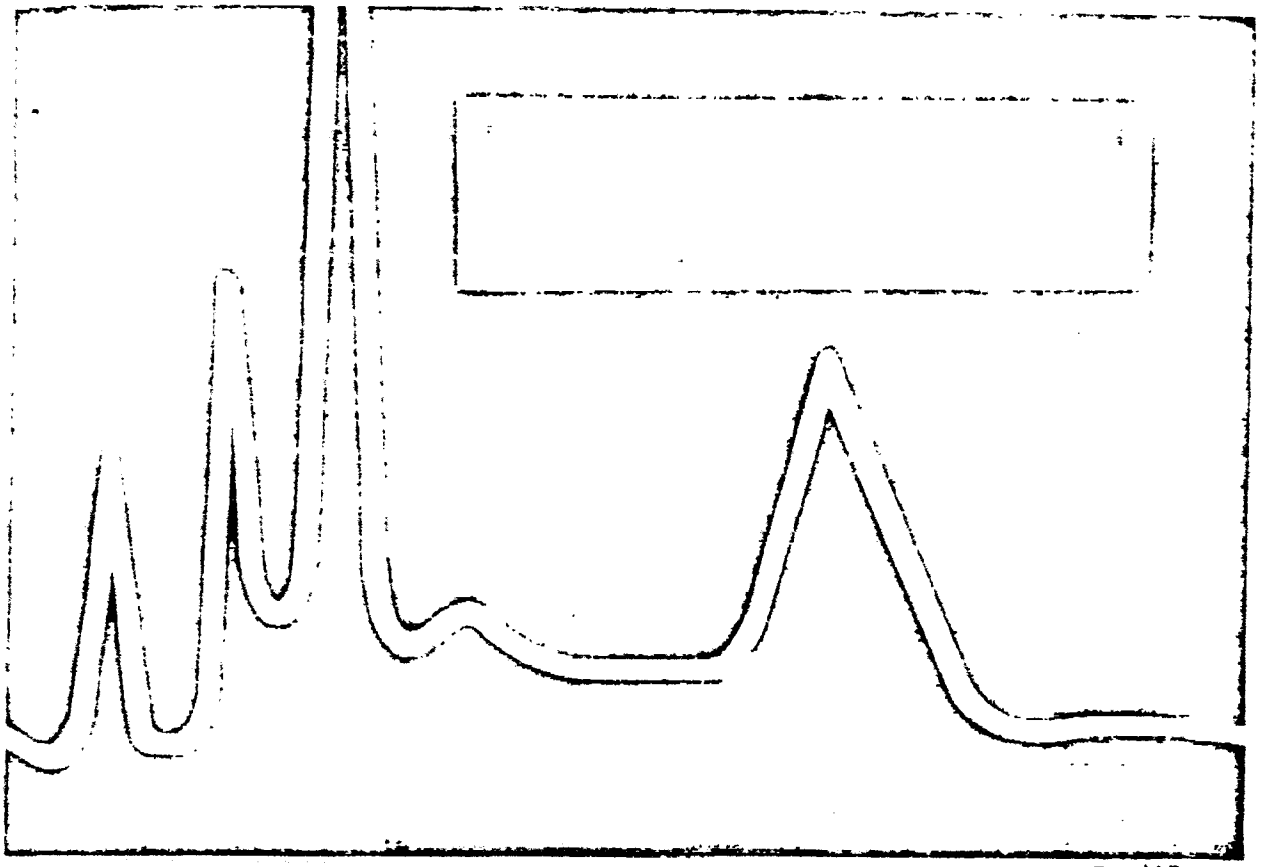
Subject: GC RECENT ARTICLES

Attached are several recent articles on GC utilization. Pages 34 and 42 have reasonable summaries for general review. I thought you might be interested in view of our heavy recent involvement in VCM quality and the FDA problem.

R. V. Lucke

RVL/cmc

OCC 1716



I-R Staff Drawing

HPLC—

Herbert L. Kahn and Zane Bitterfield
Perkin-Elmer Corp.

I-R UP-DATE

HPLC (Liquid Chromatography)

state of the art 1973

THE ANALYTICAL TECHNIQUE of chromatography is, by any standard, one of the most powerful that has ever been developed. Almost every analytical laboratory—and certainly any laboratory that is concerned with the analysis of organic materials (including, of course, those in the life sciences)—is equipped with some form of chromatographic instrumentation. It may be a gas chromatograph or the equipment to perform thin layer or paper chromatography.

And now increasing interest is being shown in liquid column chromatography, also known as high pressure liquid chromatography (HPLC). This was known formerly as liquid chromatography, but researchers prefer a more-precise term since other techniques also involve liquid in the process.

HPLC, the major subject of this article, has the curious distinction of being at once the oldest and the newest of the chromatographic techniques. In its simplest form, HPLC can be regarded as an increase in the sophistication of paper and thin layer techniques.

The sample is injected into a chromatographic column through which a suitable solvent is being pumped. Inside the column, the sample is separated into its components, which emerge from the end of the column at different times, and pass through a suitable detector.

Liquid column chromatography has been understood in principle for at least 50 years, but until recently suitable equipment, particularly pumps and detectors, has not been available.

A typical liquid chromatographic column is 500 mm long, has an inside diameter of about 2 mm, and is packed with spheres 10 to 30 microns in diameter. Unless high-pressure pumps are employed, the elution of a sample from such a column would take many hours or even days.

Now, however, it is common to use pump pressures of 1,000 to 3,000 psi (70 to 220 kg/cm²) and constant-flow pumps capable of 7,000 psi (500 kg/cm²) have recently been introduced. With such pumps, an analysis can be completed in several minutes.

A typical sample size for a high pressure liquid chromatograph is 10 to 20 μl, while solvent flow rates are of the order of 0.5 to 5 ml per minute. It requires only a simple computation to lead to the conclusion that the dilu-

tion of the sample is very great, placing great demands on the detector located at the end of the chromatographic column.

At present two types of detectors, RI and UV, are in general use. The refractive index detector measures differences in the optical refractive index, comparing the refractive index of the pure solvent to that of solvent plus sample. The output of the detector appears as a recorder peak where the area depends on the concentration of the sample component being measured, as well as on the difference between the refractive indices of the solvent and the sample component.

Until recently, refractive index detectors had mystifying problems and irritating instabilities. Now, however, detectors are available which can reliably measure differences of 10^{-6} units in refractive index. (As a standard of comparison, the refractive index of a vacuum is defined as 1.0, while that of ordinary window glass is approximately 1.5.)

Most materials have the ability to absorb radiation at ultraviolet wavelengths. Therefore an alternative detector, which frequently is used in HPLC, is an ultraviolet spectrophotometer. Since, again, tiny absorbances must be measured, the spectrophotometer that is used most commonly is a double-beam unit (for stability), employing a filter with a bandpass that peaks near 250 nm (for compactness and high throughput of light).

Again, the absorbance of the solvent is the reference cell is compared to that of solvent plus sample in the sample cell. UV spectrophotometers of good design can measure absorption differences of 0.01% with reasonable accuracy.

The search for sensitivity

High pressure liquid chromatography systems range in cost between $5,000 and $20,000. The price varies depending on complexity, convenience, versatility, and performance.

Sensitivity is one of the present limitations of HPLC instruments, particularly when compared to gas chromatographs. For this reason, many other types of detectors are under investigation, including molecular fluorescence and flame ionization units. One can also imagine that infrared spectrophotometers and atomic absorption instruments (for metal ligands) could be used.

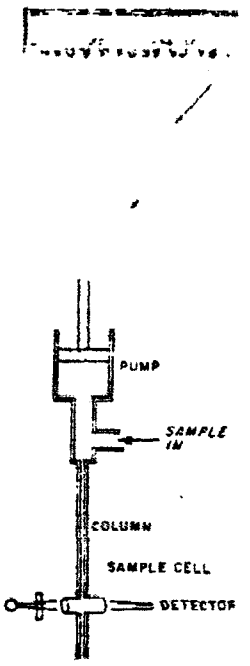
Another approach is the automatic addition of a color-forming reagent to the eluted sample in order to enhance its detectability. This is done in amino acid analyzers.

Despite its enormous promise, high pressure liquid chromatography is only starting to come into routine use in solving analytical problems. Part of the reason is that it has not been long since pumps and detectors have been brought under control, and it is even more recently that it has been possible to produce reliable and repeatable column packing materials.

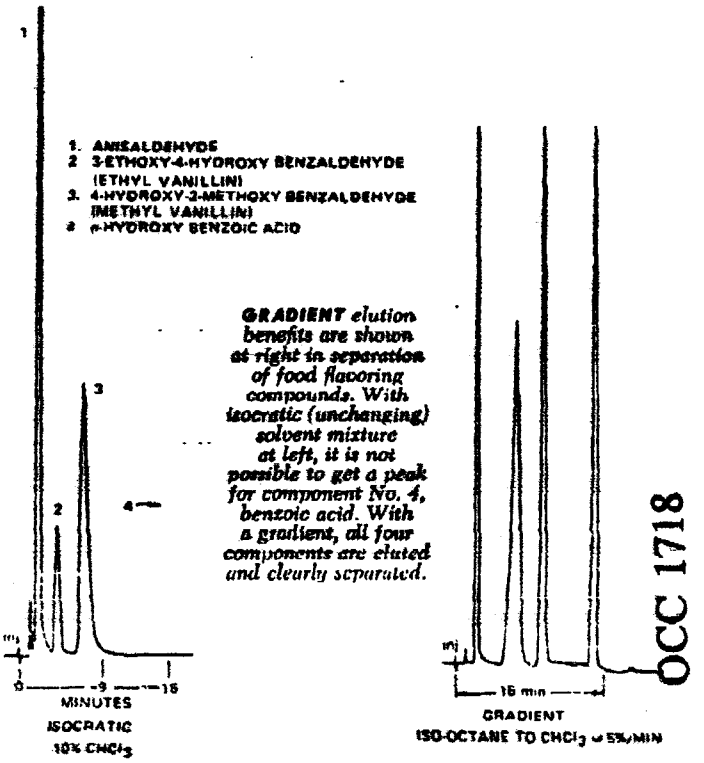
The latter is of extreme importance. Unless column packing materials are under good control, it is not possible to repeat with one instrument on one day an analysis that has been published on the basis of work done on another instrument on another day.

Another reason for the delayed acceptance is, paradoxically enough, the bewildering variety of opportunities that are open in high pressure liquid chromatography. Essentially, there are four different techniques possible: liquid-liquid chromatography, described earlier, which is known as partition; liquid-solid chromatography, where the substrate is a solid, and which therefore depends on selective adsorption; molecular exclusion, which represents an upgrading of gel permeation chromatography; and the well-known technique of ion exchange. Furthermore, there is an almost infinite choice of solvents and substrates.

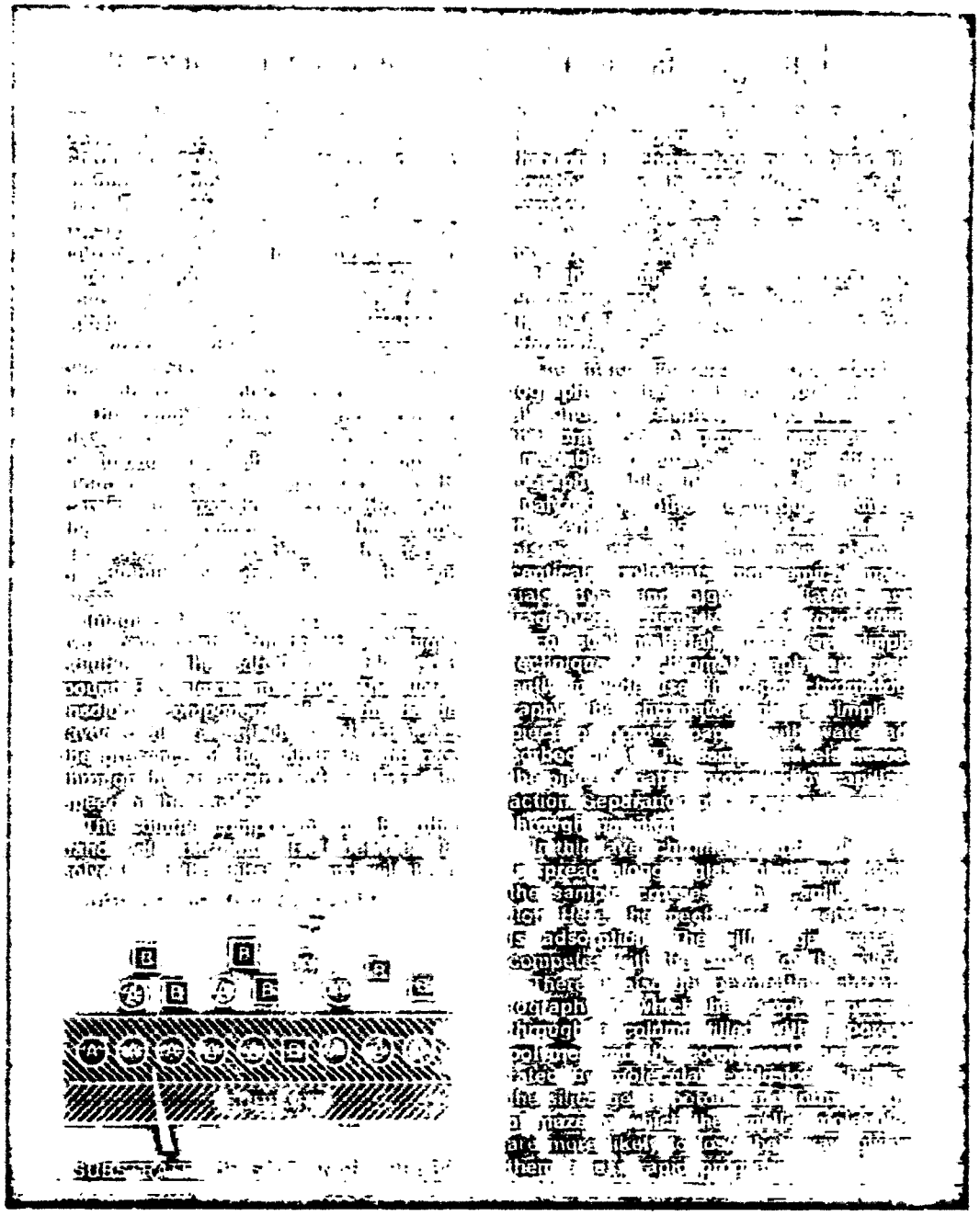
However, even now a substantial body of applications is emerging for which modern



a sleeping giant



GRADIENT elution benefits are shown at right in separation of food flavoring compounds. With isocratic (unchanging) solvent mixture at left, it is not possible to get a peak for component No. 4, benzoic acid. With a gradient, all four components are eluted and clearly separated.



highspeed liquid chromatography is the method of choice. In the production of polymers, highpressure molecular exclusion is used as the means to tell when the process should be stopped (conventional gel permeation is too slow). A similar method determines residual monomers in polymers.

Pharmaceutical houses use high pressure liquid chromatography for the determination of the presence and concentration of benzodiazopenes (tranquilizers) in tablets, thenothiazine (cough suppressant) in cough medicine, and for the measurement of decomposition products in quality control of stored pharmaceuticals.

Biochemical laboratories use it for the rapid isolation of viruses (interestingly enough, purely by size) such as Markoff's virus in chicken plasma. HPLC also is proving valuable for the analysis of dyestuffs and intermediates, which cannot generally be vaporized for use with gas chromatography.

Also, it is possible, though not yet proved, to make the generalization that modern high pressure liquid chromatographs can repeat and improve any analysis now performed by paper or thin layer chromatography. One can visualize the flat sheet used in the latter techniques being rolled up and inserted in a column, with an obvious gain in temperature control and isolation from the atmosphere.

Speed, separation, and repeatability are all

greatly improved, and there is a further benefit in that the columns are reuseable. Moreover, it is difficult to quantitate the results of the simpler techniques, and such devices as scanners are expensive and hard to use. Thus, if quantitative results are desired, modern liquid chromatographs can even have a cost advantage.

Don't dissolve the substrate

To select the conditions for an HPLC analysis, there are a few obvious basic conditions. If a refractive index detector is used, the solvent should—as far as possible—have a substantially different index of refraction from the sample components of interest.

For work with a UV detector, the solvent should have relatively low absorbance in the ultraviolet region. For obvious reasons, the stationary phase, or substrate, should not be readily soluble in the solvent. To keep the solvent from dissolving the substrate, one is generally chosen to be a highly polar liquid, while the other has very low polarity.

Commonly, the substrate is polar and the solvent is not. If it is the solvent that is polar, the technique is known as reverse-phase chromatography. Tables giving the polarities of different liquids, and hence their suitability for use together, are widely published and usually also show refractive index and UV transparency.

An opportunity to increase the power of high pressure liquid chromatography, at the cost of complicating the instrumentation, is known as "gradient elution." Veteran gas chromatographers will recognize this as the liquid chromatographic equivalent of column temperature programing. With gradient elution, an analysis starts out with Solvent A which, during the analysis, is gradually changed in a timed and predictable manner to Solvent B.

When gradient elution is properly used, it is possible to separate peaks which could not be separated with a single solvent or solvent mixture. It also is possible to speed up the analysis for sample components which are not very soluble in Solvent A, and to sharpen some of the later peaks, thereby increasing sensitivity.

Gradient elution requires a second pump, as well as a solvent mixing chamber. Obviously, if a gradient consisting of two solvents is good, a gradient of three solvents is better, but fortunately, so far, only a few appear to have carried this bright idea into practice.

R search into practice

Up to now, the overwhelming majority of users of high pressure liquid chromatographs are in government, industry, and university research laboratories. Because of the variety of opportunities that high pressure liquid chromatography offers, it is at least possible that any analytical problem, no matter how intractable, can be solved by the choice of the proper column, substrate, and solvent. Moreover, again because of the complexities among which one can wander, almost any work that is done probably is publishable.

Increasingly, however, useful general methods are emerging. As may be expected, instrument manufacturers are falling upon these with delight, and giving them the widest possible circulation.

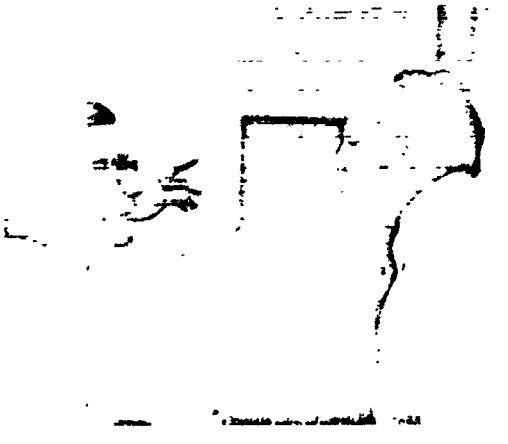
As a result high pressure liquid chromatography now is at the beginning of a period of explosive growth. As routine analytical methods become increasingly available, high pressure liquid chromatographs will take their places beside gas chromatographs in almost every type of analytical laboratory. □

SOLVENT POLARITY SCALE

Solvent	E° (Al O)	Viscosity (cP, 20°)	RI	UV Cutoff (mu)
Fluoroalkanes	−0.25		1.25	
n-Pentane	0.00	0.23	1.358	210
Hexane	0.00	0.33	1.375	210
Isooctane	0.01	0.3	1.404	210
Petroleum ether	0.01	0.3		210
Cyclohexane	0.04	1.00	1.427	210
Carbon Tetrachloride	0.18	0.97	1.466	265
Butyl chloride	0.25	0.47	1.436	220
i-Propyl ether	0.28	0.37	1.368	220
i-Propyl chloride	0.29	0.33	1.378	225
Benzene	0.32	0.65	1.501	280
Ethyl ether	0.38	0.23	1.353	220
Chloroform	0.40	0.57	1.443	245
Methylene chloride	0.42	0.44	1.424	245
Tetrahydrofuran	0.45	0.35	1.408	220
Methylethylketone	0.51	0.3	1.381	330
Acetone	0.56	0.32	1.359	330
Acetonitrile	0.65	0.37	1.344	210
i-propanol, n-propanol	0.82	2.3	1.38	210
Ethanol	0.88	1.20	1.361	210
Methanol	0.95	0.60	1.329	210
Acetic acid	Large	1.26	1.372	
Water	Larger		1.333	190

PROPERTIES *of some commercial solvents. Left hand numbers represent polarity measured by interaction with aluminum oxide. RI is refractive index. UV cutoff represents the wavelength above which the solvent is transparent.*

For abstracts of related articles from ARAC, NASA Regional Development Center, circle 678 on the reader service card.



THE AUTHORS

Zane L. Bitterfield is a chemistry graduate of Temple University and has experience at Drew Chemical and Wilson Pharmaceutical. His areas are quality control, lipids, and GC. Now he is working at Perkin-Elmer with Herbert L. Kahn in HPLC. Kahn appeared in I·R in February as product manager for spectroscopy. He has since moved to HPLC to be "where the action is." Zane, at left, is showing Herb where the sample goes in.

OCC 1720

QUALITY CONTROL *300 years ago was the scrutinizing eye of the alchemist. From the Fisher Scientific Co. collection, interpreted by I•R staff artist.*

quality

Patrick W. Byrnes
product manager
Fisher Scientific Co.

HOW CHROMATOGRAPHY meets the demands for many industrial quality control applications is a story as diverse as industry itself. Rather than attempt to cover the entire subject, this article will suggest some rules of thumb for the practical chemist to follow in assessing the applicability of gas chromatography to his quality control problems. Examples of procedures used in industry will be cited as further illustration of current techniques.

Gas chromatography is accurate, precise, and rapid. With the advent of digital integrators and computerized data reduction, interpretation and quantitation of results has been simplified. The introduction of automatic sampling devices has permitted automation of many GC procedures. Hence, GC can replace lengthy or cumbersome bench chemistry methods.

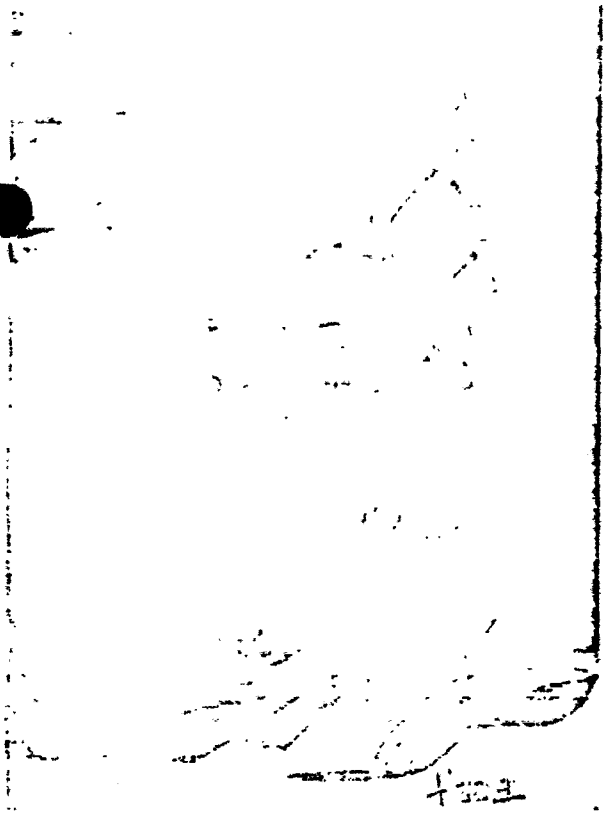
The problems of industrial quality control are numerous and varied. However a flexible GC instrument and an analyst with basic knowledge of GC technique can adapt procedures to solve almost any problem.

Consider the typical problem of accurately determining the NH_3 and CO_2 levels in aqueous ammonium carbonate solutions. Lengthy wet chemistry procedures including final titrations are necessary to report these compounds to the nearest 0.5% or better. Analysis time per sample exceeds 100 min.

By employing the correct column stationary phase and GC inlet system, the gas chromatographic determination of these components, including data interpretation, requires less than 8 minutes per sample. In this application, the gas chromatograph is set up with a thermal conductivity detector, tube pyrolyser inlet system, and 7 ft (2.13 m) x ⅛ in. diameter stainless steel column packed with Porapak or equivalent stationary phase.

The column is operated isothermally at 85 C with a helium carrier gas flow rate of about 40 ml/min. The detector is maintained at 150 C to prevent any sample vapors from condensing while being detected. Sampling is done by transferring exactly 5 μl of solution into a small steel boat which is placed inside the pyrolyser tube and sealed. While carrier gas is sweeping through the pyrolyser tube into the packed column, the tube is heated rapidly to 1000 C.

Under these conditions, the ammonium



quality control

ductivity detector, which will give a response to any type of compound, is most useful here.

There are many similar analytical quality control situations in the inorganic chemical industry that can be more easily handled by gas chromatography than by current wet techniques.

At the Chemical Manufacturing Division of Fisher Scientific Co., quality control chemists rely on GC for various quality control determinations. Fisher's 99% mole pure solvents are assayed by GC using either thermal conductivity or flame ionization detectors. The determination involves comparison of peak areas obtained from lot samples to those of standards having known mole purity. The following solvents are routinely assayed:

acetone	n,n dimethyl formamide
acetonitrile	1, 4 dioxane
analine	ethyl acetate
benzene	n hexane
n-butanol	iso octane
isobutanol	methanol
chloroform	carbon tetrachloride
toluene	methylene chloride
	2 propanol

In some cases, GC is used to determine the level of certain impurities. In production of carbonate stoichiometrically decomposes to NH_3, CO_2, and H_2O. These gases are swept out of the pyrolyser boat into the column where they are separated and detected separately. A digital integrator automatically determines the peak areas. These areas are compared to areas produced by standard samples for quantitation using this equation:

$$\frac{\text{Area of std.}}{\text{Area of unknown}} = \frac{\text{Conc. of std.}}{\text{Conc. of unknown}}$$

In this case, use of gas chromatography reduced analysis time from 100 min to less than 8 min while maintaining the integrity of the results. More important, proper control of high volume production facilities was made possible by fast reaction to results that were beyond specification. Employing GC allows corrections or alterations to be made in the production process without waiting 1.5 hours to find the product lot unacceptable.

A noteworthy point about this example is that the sample is an aqueous, inorganic salt solution—not normally thought of as subject to GC determination. The use of a pyrolysis inlet device permitted relatively easy and straight-forward GC work. A thermal conformic acid, for example, it sometimes is difficult to prevent build up of acetic acid. At Fisher, GC procedures for the determination of acetic acid impurity levels have replaced the old titration procedures. The GC procedure in this application offers three advantages.

First, it is far less time-consuming and cumbersome, Second, it has specificity—it eliminates errors caused by other acidic materials present in smaller quantities. Where the titration would give total acidic impurities, GC quantitates the acetic acid individually and unmistakably as a single, specific peak on the chromatogram. Third, GC's inherent sensitivity enables acetic acid detection at lower levels than previously possible by titration.

Turned on by hydrocarbons

A similar procedure is employed to determine the level of extractable organic substances in hydrochloric acid. The HCl is extracted with a suitable pure solvent and the extract is partially evaporated. The remainder is examined on a GC column and the separated hydrocarbon impurities detected by

OCC 1722

a matter of boiling points

GAS CHROMATOGRAPHY is simply a method for separating mixtures into their individual components. As with other chromatographic techniques, the sample is placed in a mobile phase and carried across the surface of some stationary phase.

The stationary phase is selected to interact more strongly with some components of the mixture than others and consequently retard their travel. This interaction, based on a difference of boiling point, electronegativity, solubility, molecular structure, and other factors, causes the various components to pass through the stationary phase at different rates and thus effectively separates them.

Gas chromatography, keeping to its name, employs a gas as the mobile phase. Samples are first converted into the vapor state. Next, they are swept by a constantly flowing carrier gas stream through a long narrow column which is packed tightly with the desired stationary phase.

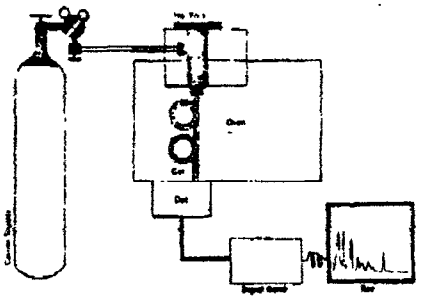
A gas chromatography system consists of several basic components joined together to:

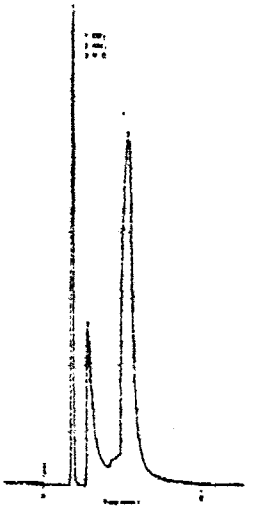
- provide a constant flow of carrier gas.
- permit introduction of sample vapors into the flowing gas stream.
- contain the appropriate length of narrow bore, packed column.
- maintain the column at a constant temperature sufficient to keep all samples in the vapor state.
- detect the components as they elute from the column and provide a readable signal proportional in size to the amount of component.

Detecting devices can be universal like the Thermal Conductivity Detector, or specific such as the Flame Ionization and Electron Capture Detectors.

The signal, or chromatogram, consists of a series of peaks, one for each component. The peak retention time, or time required for elution of the component, is used for qualitative identification of the component. The peak area is used as a relative measure of the amount of the component. Utilizing appropriate quantitative standards, the chromatographer can obtain precise and accurate quantitative data.

Gas chromatography is applicable to any substance which can undergo conversion to a stable, nonreactive vapor and then pass through a column. A vast array of organic materials as well as the fixed gases lend themselves to GC determination. Most inorganic components are not amenable to GC work since they resist conversion to a stable, non-reactive vapor. Some inorganic sulfates and nitrates, by contrast, will convert to stable fixed gases, and hence enable GC determination, providing an indirect analysis.





PYROLYSIS
of ammonium carbonate is an excellent example of how GC saves time, reducing a 100-min operation to less than 8 min.

a highly sensitive flame ionization detector (FID). The FID responds only to hydrocarbons and a well-designed FID will detect levels as low as 50 ppb.

In addition to being highly sensitive to hydrocarbons the FID does not respond to water. In some instances, where the hydrocarbons are moderately soluble in water, direct water injection eliminates the need for extraction. Even a considerable amount of water produces little or no response with the FID and, as a result, there is no interference with even small hydrocarbon peaks.

Perhaps the most predominant use of gas chromatography at the Fisher Chemical Division is for quality control of pesticide-free solvents. Following a procedure outlined by the Food and Drug Administration, samples from each lot of each solvent are analyzed by gas chromatography coupled with an electron capture detector—the most sensitive method used today for detecting and determining halogenated pesticide residues.

The detector sensitivity is adjusted so that 1 nanogram (10^{-9}gram) of heptachlor epoxide (chosen as an average standard by the FDA) per milliliter of solvent makes a half-scale deflection (50-division peak) on the chromatogram. To meet Fisher specifications, no solvent can contain existing impurities that produce a deflection of 1 mm on the chart. This means the impurities must be less than one part (heptachlor epoxide) in 50-billion parts. The solvents assayed in this manner include:

acetone	methylene chloride
acetonitrile	carbon tetrachloride
hexane	1-2-dichloroethane
methanol	n-pentane
benzene	petroleum ether
ethyl acetate	2-propanol

OCC 172

These features of currently-used GC procedures illustrate some of the criteria for selecting GC over some other technique:

- GC methods save time.
- GC procedures are more sensitive.
- GC is more specific.
- GC procedures are simple and rarely cumbersome.

When trying to decide between GC and bench chemistry procedures, weigh the benefits listed above against the initial capital outlay for the equipment. Gas chromatographic systems capable of a wide range of quality control work can be obtained for less than the cost of a technician for one year.

Chromatographic variety

There is certainly no shortage of gas chromatographs for the user to consider today. A wide selection of systems offering varying degrees of capability with one, two, and four columns, a choice of detectors, and other components, plus all the sophistication and complexity one could want, are on the market—each with its appropriate price.

Since most GC procedures require the capabilities of a dual column unit, discussion here will focus on these systems. There are, however, simpler procedures which can be carried out on a single column instument, but that's another subject. What then are the features and performance qualities that a gas chromatograph should have to represent a good value for the analyst in quality control?

Stability ranks as the most important single characteristic in selecting a gas chromatograph for quality control work. Good stability means repeatable retention time (or elution time) and this gives the analyst confident peak identification and precise results. Retention time, in turn, is grossly affected by temperature and flow fluctuations. The highly stable GC instrument of choice, therefore, should employ a differential flow controller for control of carrier gas to each column and should offer temperature stability of about ±0.5 C.

Most GC systems now available utilize state-of-the-art detectors and have adequate sensitivity to detect levels normally sought in quality control. A typical flame ionization detector, for example, will routinely detect less than 1 ppm of most hydrocarbons.

The second-most-important characteristic is reliability. The instrument must deliver consistent results around the clock. Instrument failure can delay an entire production facility and often can eliminate the only means of assaying product quality. Since down time is an inevitable occurence, however, the instrument should be easy to troubleshoot and repair.

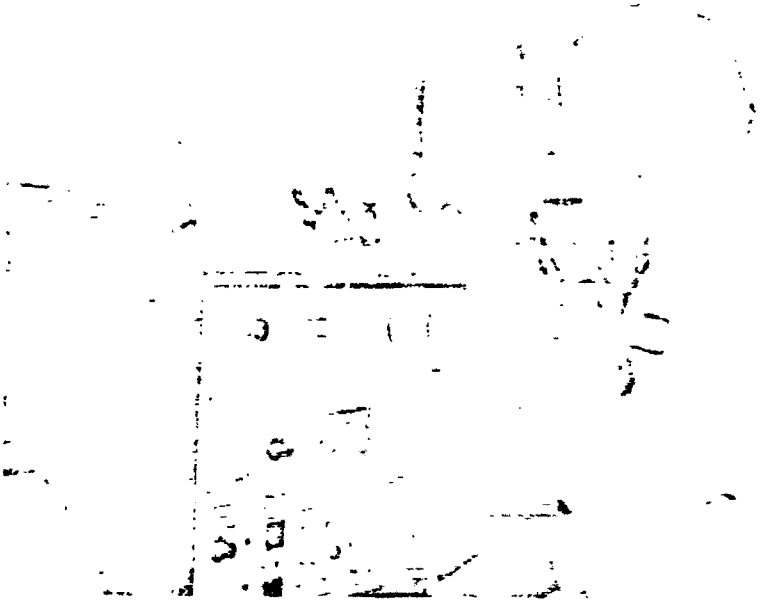
Before selecting a GC instrument consider the following questions to help minimize down time before it occurs. Is the instrument modular? Does it employ state-of-the-art solid-state electronics? Who services the warranty, and what is their reputation?

Several gas chromatograph manufacturers have recognized the potential of GC in industrial quality control and have developed instruments specifically to suit quality control needs. A recently-introduced gas chromatograph has a versatile dual column system which features multidetector capability. These include: dual flame ionization detector with standard dual channel electrometer; electron capture detector, supplied with an ECD Linearizer (displays linear response to concentration ranges of 2 x 10^4); and dual thermal conductivity detector, filament or thermistor type.

This choice of detectors combined with a large column oven introduces dozens of applications possibilities that were previously unavailable with an inexpensive GC system. Chromatographers can, for example, employ two dual detectors simultaneously enabling split effluent display (FID, ECD), or simultaneous TCD, FID operations, plus additional other operations.

Such a unit offers the temperature and flow stability needed for precise quality control work. In addition, the choice of temperature programming provides the chromatographer with all the versatility needed to accommodate analysts in almost any industry—chemicals to textiles. This economical instrument in the $3,000 price range, makes available a quality instrument capable of adapting to numerous QC procedures, including pyrolysis.

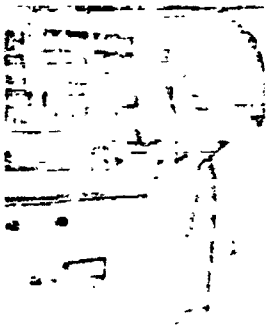
With instruments of similar description and price coming on the scene, the quality control chemist can upgrade many of his current bench procedures; often reduce production costs; and always save analysis time. ■



***INEXPENSIVE** gas chromatograph in use here is typical of units designed for quality control work. Its dual column system will accept a variety of sophisticated components.*

THE AUTHOR

Patrick W. Byrnes is gas chromatography product manager for the Analytical Instrument Div., Fisher Scientific Co. He previously managed their applications laboratory, and is a specialist in developing GC procedures.



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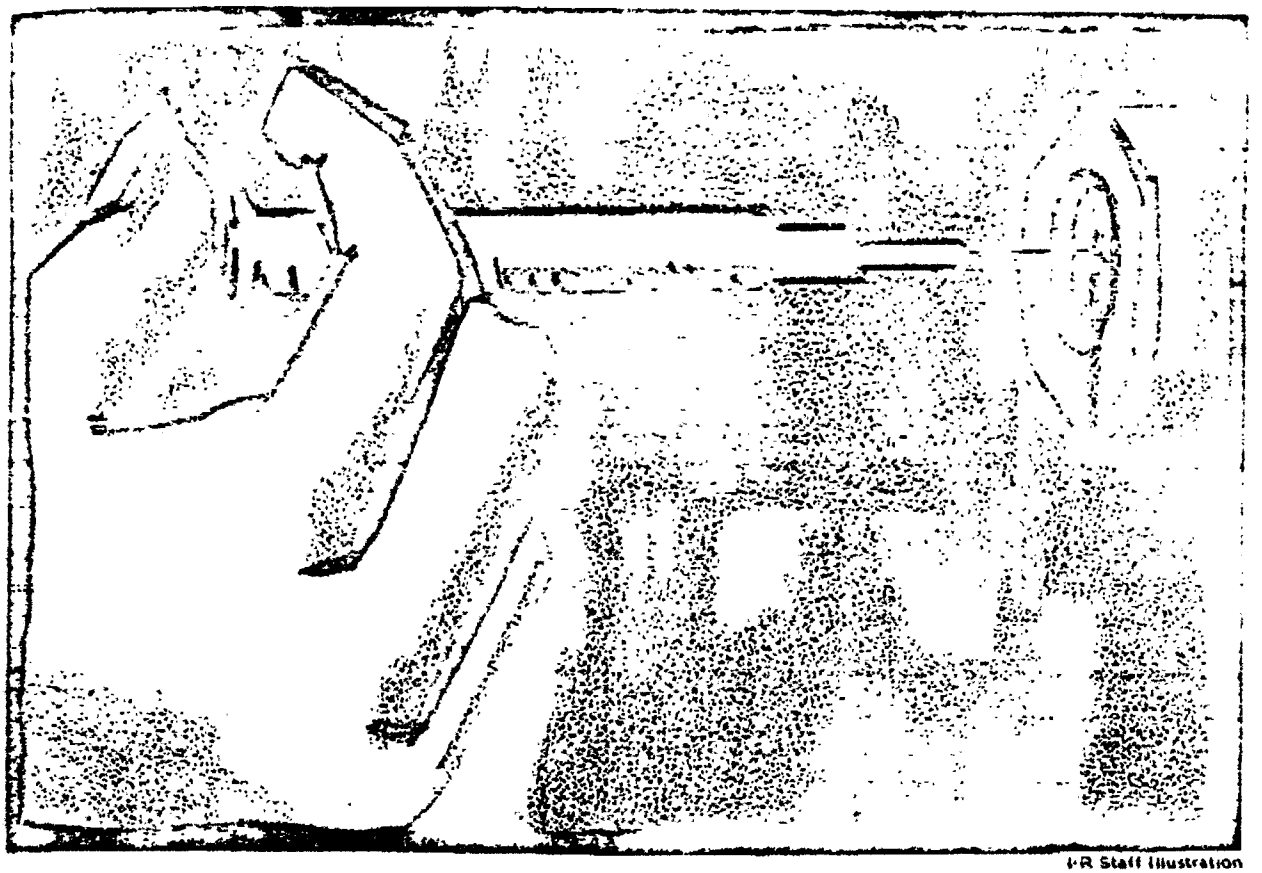

I-R Staff Illustration

chromatography

Dennis R. Gere
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Varian Associates

LET ME START by saying that this message is not being beamed out to the fat cats who have a fully-equipped lab and/or the capital equipment budget with elbow room to spare. Actually, this really is a moot point, for I suspect that all the fat cats died with the mid 1960s.

No longer can a laboratory justify the purchase of a "toy" which is fun to play with but does not justify its own existence on a month-to-month basis. It has been observed that this mechanism is not unique to the tools of the laboratory, but applies as well to project planning and even to the chemists themselves. Today, then, we have a much more sophisticated and discriminating group of people purchasing and justifying the purchase of laboratory equipment.

This trend towards a tougher management line on purchase of equipment is even more evident and significant in the smaller laboratories where funds are limited and competition for use of the funds is severe. What does a person in such a laboratory do if he is contemplating purchase of chromatography equipment?

First of all, rarely does one simply "contemplate the purchase of chromatography equipment" as an isolated incident. Usually, these days, a need indicates the purchase of equipment and if the proper tools are selected (and used properly) the needs will be fulfilled in a reasonable amount of time and everyone will come out ahead. There should be and is less of the "emotional" purchasing which usually spelled trouble for the lab user, the instrument manufacturer, and ultimately for the management which okayed the original purchase.

Instrumentation or classical analysis?

Let us examine, then, some representative approaches used to reach a sensible decision for purchase of chromatography instrumentation. To begin with, do you really need instrumentation?

This cannot be answered for all cases in general, but rather each case to be measured on its own merits. What one usually purchases these days in terms of chromatography equipment is not just a piece of hardware, but rather a "capability." This is often a complex and expensive capability and therefore requires some thought and effort to make sure the "capability provided" is well matched with the "capability needed."

OCC 1725

and the limited budget

Modern high-performance liquid chromatography, (HPLC) as used in the pharmaceutical industry provides several convincing arguments in favor of chromatography instrumentation over such wet chemical techniques as thin-layer chromatography (TLC). For a typical analysis, a total of six man-hours would be allotted to the TLC technique whereas a total of one man-hour is all that is required for the HPLC technique including precolumn work-up and post-separation data reduction.

Not only does one save time—but also new dimensions of resolution, precision, and accuracy are now attainable. The same is true in the comparison of the gas chromatography of 17-ketosteroids in pregnancy urines.

What are the choices?

So, you're convinced that the added speed, sensitivity, through-put, and resolution justify the capital expenditure to expand the capability of your limited budget lab. You now begin to explore the many pathways that could lead you to having such devices in your facility. If you simply walk in to your management and quote them typical prices "cold-turkey" for a fully loaded chromatograph, you can almost be assured they'll send you back to seek lower cost alternatives.

Perhaps they will suggest that you purchase a used model. Unless you are very lucky, that pathway is rarely worth pursuing. Used chromatographs are a tough commodity to find. It's something like buying and selling a used car. You must keep in mind there are several different terms used in describing the value: actual cash value, wholesale selling price, and retail buying price.

Whereas your management expects you to find the model to match your needs at perhaps 30% of the new price and only slightly used, you'll find a seven-year-old model selling at 70% of its full price and, upon close scrutiny, it doesn't have your solid-state electrometer which is now considered standard equipment and the temperature programer is a first generation design.

There's even the problem of locating a used device that will meet your needs. The instrument manufacturers rarely offer used equipment, for a variety of reasons. Used equipment usually means a demonstration device used in an application lab for no more than one year.

Usually, it is fully reworked in the factory before being shipped out, and then leaves with a new device warranty. This usually is a good buy from the standpoint of reliability and quality, but from an absolute dollar standpoint it will cost 85 to 95% of its original selling price.

A well-designed, useful chromatograph should have no trouble lasting at least ten years. That means that a laboratory rarely will give up one of these devices short of some disaster. If they do, it's because they want or need a more capable device (how does your chemical problem compare with theirs?), or because they have had a lot of downtime with it.

The best possibility you have of obtaining a bargain is visiting a laboratory which is being closed down. These days there are, admittedly, quite a number of these happenings. Even then, the good capital equipment is reallocated to the other parts of the company before the outside public is allowed to look over the used equipment.

Assuming that you can find a used piece of equipment (a used high-performance liquid chromatograph is impossible to find at this time), check carefully into a couple of other factors before you purchase it.

- ☐ Is the unit operational at this point?
- ☐ Will the seller aid you in making sure it is operational?
- ☐ Is the unit amenable to servicing?
- ☐ Are the replacement parts readily available today? Beware of the model whose manufacturer has ceased operation!

Leasing is for tax saving

Perhaps your next option would be to try and lease some equipment to avoid the capital outlay for the whole item at one time. This option rarely turns out to be of any value in easing the original justification for obtaining the equipment. Its biggest value is as a tax writeoff benefit.

If your management is unwilling to lay out the original purchase price at this point, the idea of signing up for 36 monthly lease payments without any gain in equity doesn't appeal to them either. (No, Virginia, there is no 6-months lease, where you can send it back if you don't like it at the end of the short term!)

If yours is a small independent laboratory, at this point of desperation you might consider going to a private bank to see what they will do for you. Usually this is a very discouraging ordeal. Bankers take a very conservative stance concerning scientific equipment. This means you must have 50% of the purchase price available in assests

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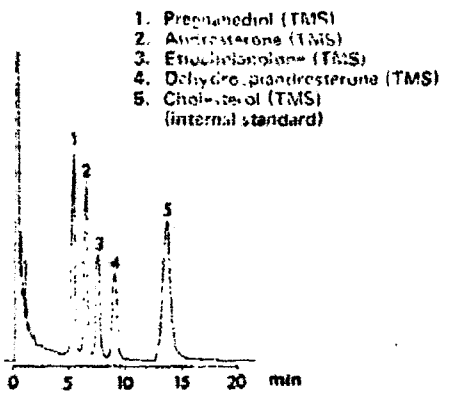
Key steroids form an important group of urinary components which can be analyzed very effectively with gas chromatography. The quantitation of key steroids in urine is usually performed by colorimetry. However, this method is not very accurate since only one steroid is determined for the total steroid concentration. The chromatogram shows the three common 17 ketosteroids in urine, analyzed as TMS derivatives, as well as pregnanediol. The internal standard is cholesterol.



Conditions: flame ionization detector; 6' x 1/4" glass column with 1.5% OV-17 on 100/120 HP Chromosorb G: 200°C; N_2 at 30 ml/min.

and the 50% you borrow should be paid back in no more than three years at stiff interest rates. Well, you're back to your own return on your assets calculation!

Oftentimes the next idea you have concerns buying components and putting them together yourself on a bare-bones allocation. The red flag really goes up here concerning man-power availability and hourly labor costs.

You must make sure you are buying the state-of-the-art components first of all. Secondly, you need someone available in your facility who can lash it all together and make it work, and service it when it is down.

A survey of a couple of the larger pharmaceutical firms which opted for this type of entree into high performance liquid chromatography indicates that this is not the best way for a fiscally-responsible organization to go. You'll find after a couple of years that the people you assigned to assemble and run the equipment have learned a lot about the technique, but meanwhile your competitors have turned out four times the analysis workload on equipment which was purchased with a systems responsibility (by the manufacturer).

Remember the commercial equipment is designed not only for ease of operation, but also very importantly, it is designed for serviceability. Spending some time comparing mean-time-to-failure and mean-down-time of commercial system vs. put-together components can be very revealing.

Make the salesman work

Finally, then, we come squarely back to the need for a systems responsibility. Justify your purchase on real need in your laboratory coupled with a capability which you don't possess at this time (or perhaps in not sufficient quantity).

You should be talking to a salesman in the early stages where he can help you define your needs in terms your management can easily recognize. He will also be helpful in referring you to current users of his equipment, competitor equipment, and even basic-component equipment. He can also put you in touch with other members of his own team (just who else is on his team and why?).

He'll let you talk to his development engineers and scientists who can tell you not only why a certain feature is available, but how a potential new feature could be added when the state-of-the-art advances. Concerning features of equipment, don't forget that the professional salesman can help you relate a hardware feature to a real benefit for you, the end-user, on your chemical problem. He also has access to an applications laboratory—a benefit which usually becomes apparent after the sale, during the critical startup period of new equipment.

Remember, it's not always fruitful to simply send in a customer sample to the applications laboratory and base your buying de-

Cost savings based on sample throughput

Item	Operator Costs* Manual	Integrator	Computer
Instrument supervision	$ .50	$ .10	$ .00
Area measurement	3.00	.30	.30
Composition calculation	1.67	1.67	.05
Report writing	1.00	1.00	.05
Total/sample	$6.17	$3.17	$ .45

*Based on 10 peaks, 10-minute chromatograms, $10/hour/operator

$$Payout/year = \frac{Savings/year}{System\ cost} = \frac{Samples/day \times savings/day \times work\ days/year}{System\ cost}$$

AUTOMATION *can come in various degrees. Depending on your needs, the more automation you have, the lower are your costs.*

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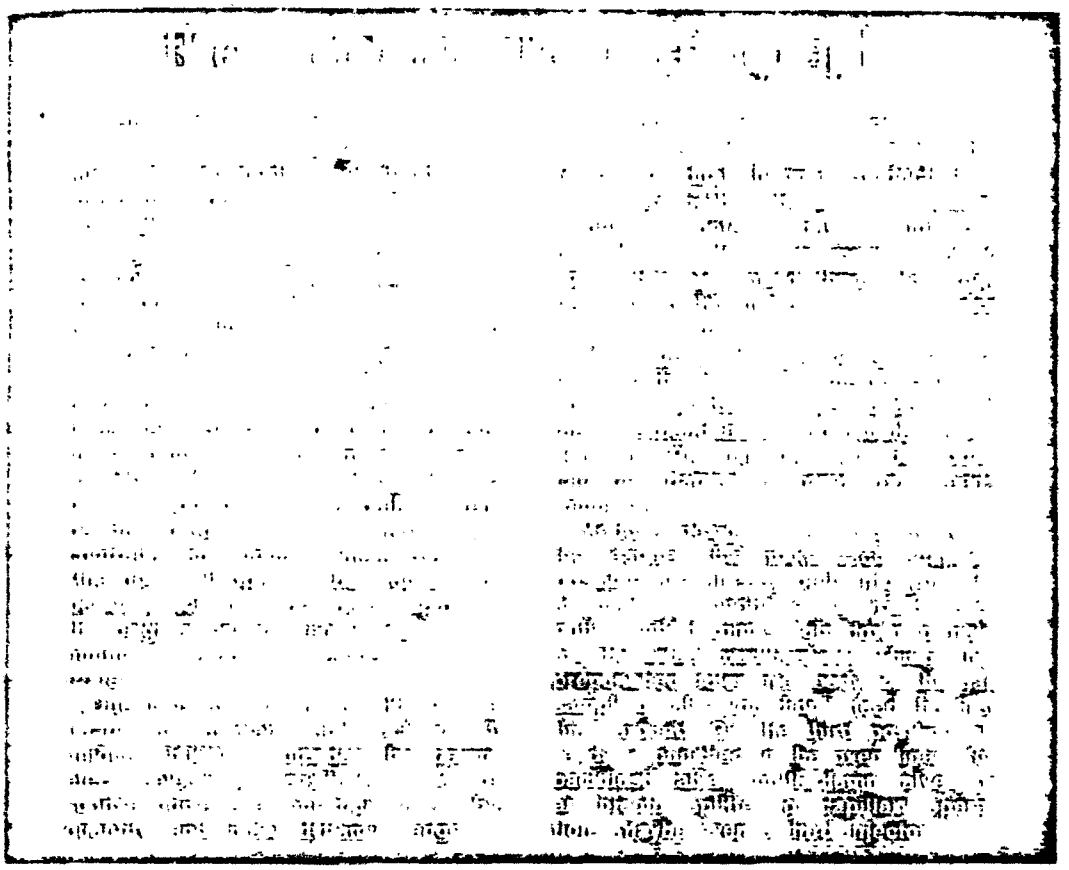

cision on the "beauty" of the chromatogram which it returns to you. Better that you arrange a visit to one of these laboratories and observe the equipment in action. Even better, get your own hands (or those of one of your laboratory personnel) on the equipment in the applications lab and operate if yourself.

Keep in mind, also, that the applications staff probably will be available to you for consultation after the sale, a very useful service. They can provide you with a continuing source of technical data on the care, feeding, and expanded use of your equipment. Oftentimes this can be in the form of short courses, basic how-to manuals, and continuing bibliographies.

There is also another group on the salesman's team that can be of vital assistance, the service engineers. At some time or other all equipment will develop a problem. The abilities of the service crew can substantially affect your minimum-down-time and frustration level. This capability can rapidly overcome the difference in original purchase price of a complete commercial system vs. the put-together-yourself component kit.

But there is one kind of do-it-yourself that can make sense. A good compromise in the approach of putting components together yourself is to purchase a chromatograph which is designed in a modular concept.

Finally, don't fail to consider automating features of a chromatograph. It may seem incompatible to consider an automated chromatograph when funds are limited. However, if a heavy workload is anticipated, automatic sample introduction and automated data handling can, in fact, pay for themselves. Operating expenses are reduced by the manpower savings resulting from one or more of the following areas: instrument supervision and maintenance, quantitative calculations, and report preparation. Faster sample through-put makes it possible to reduce the number of chromatographs required for a given work load.

Let your needs dictate your purchase requirements. Justify these needs as credibly as you can define them. Seek assistance in defining these needs and requirements from the instrument salesman and his team of experts. Avoid the pitfalls of buying for emotional reasons or on strictly price considerations.

Determine how your chromatograph can and will do the work for you. Despite what some bankers might think, there are few investments that pay back better or more dynamically than can a well designed chromatograph. ■

For abstracts of related articles from ARAC, NASA Regional Development Center, circle number 677 on the reader service card.

THE AUTHOR

Dennis R. Gere has worked six years for Varian Associates, most recently as manager of separation sciences in the applications lab. This month he is newly employed at Hewlett-Packard's Avondale Div.

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JOHN L. NELSON
DIVISION VICE PRESIDENT, MANUFACTURING

December 5, 1975

Mr. Raymond Abramowitz
Hooker Chemical Corporation
River Road
Burlington, New Jersey 08016

Dear Ray:

Enclosed is a copy of a report "Progress in Vinyl Chloride Containment", which was distributed yesterday in a media briefing in New York.

As a member of the Vinyl Chloride/Polyvinyl Chloride Producers Committee of SPI, you may be interested in some of the things B.F.Goodrich has done in meeting the challenge of the vinyl chloride health problem.

Yours very truly,

John Nelson

J.L.Nelson/ja

OCC 1730

hooker
RUCO DIVISION

INTER-OFFICE MEMORANDUM

To: H.H. Kaster

Copies to: R.A. Katherine

File Ref.: PLF-2-zc

Date: April 30, 1979

From: Phillip L. Friedman

Div./Dept: RUCO/New Ventures

Location: Burlington

Subject: VCM NOMINATION - 1980

This will confirm New Ventures' portion of the 1980 nomination for Shell VCM.

Please allow 12.0 MM pounds of VCM for re-sale and PVC tolling. The total nomination including Burlington should therefore be 171 MM pounds, allowing approximately 180 MM pounds maximum under our contract.

Phillip L. Friedman

PLF/zc

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FORM NO. 88-088