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Phil. Trans. R. Soc. Lond. A 1982 **307**, 491-501

doi: 10.1098/rsta.1982.0124

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Computer control of equipment and data handling

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In the 10 years since the first commercially available microprocessor was announced, nearly all commercial optical and infrared spectrometers have become computer-controlled. The advantages of such control and the cost of writing the necessary programs are discussed, and the industrial and academic situations contrasted. Data handling is discussed in terms of signal handling and the subsequent data-processing techniques available. Examples are given of the use of methods to aid visualization. Results are given of the application of the least-squares method to the measurement of a trace component, and an infrared interpretative search program is described.

INTRODUCTION

The results of a very recent computer search of *Chemical Abstracts* for the combination of the various forms of optical and infrared spectroscopy with 'computer' give some indication of the size and growth of the subject. A total of 600 references were found, covering the period from 1967 to date at a rate increasing from 3 per year to 80 per year.

In order to narrow this large field somewhat this paper concentrates mainly on microprocessor applications and, in this respect, it is worth noting that it is only just over 10 years since the first commercially available microprocessor was announced by Intel.

COMPUTER CONTROL

The growth in the use of microprocessors for instrument control is indicated in figure 1, which relates to i.r. instruments manufactured by one company (Perkin-Elmer Ltd). All other Perkin-Elmer analytical instruments have similar growth curves, and from the information available it appears true also for most other manufacturers, although there are some displacements along the time axis.

Clearly for such a rapid and complete change, the commercial advantages must have been great. The main advantages are listed in table 1 and are discussed below.

Mechanical simplification: the first applications of microprocessors were mainly the replacement of existing motors and gearboxes with stepper motors, still using cams and complex mechanical linkages to generate required functions. More recently, better use has been made of computing power by generating these functions in the microprocessor and using only the simplest possible mechanical drives. A typical example is a direct drive to scan any one of four diffraction gratings by using a single stepper motor and worm reduction gear. With 360 000 steps to a complete revolution, the angular resolution is better than 4". When compared with earlier systems used to perform the same function, the degree of simplification and consequential cost saving are very great.

For all forms of computer control both an actuator to perform the action and an encoder to measure the result are required; the stepper motor is a very useful device in this respect in that it can perform both functions.

[25]

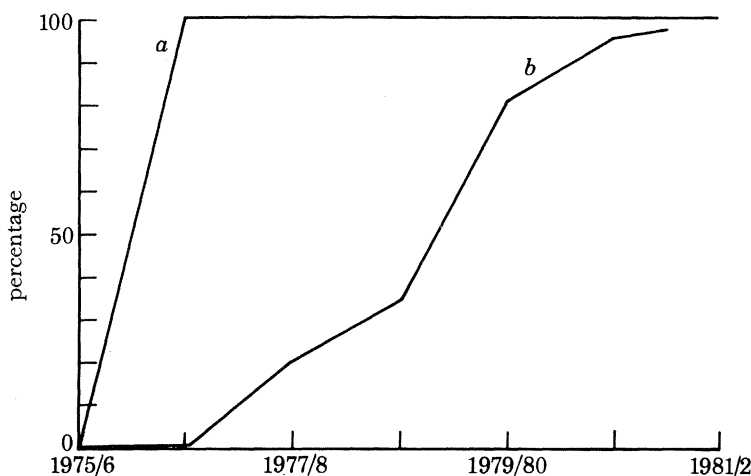


FIGURE 1. The percentage of infrared instruments from one manufacturer (Perkin-Elmer Ltd) using microprocessors. (a) New products; (b) total sales.

TABLE 1. COMMERCIAL JUSTIFICATIONS FOR THE USE OF MICROPROCESSORS TO CONTROL INSTRUMENTS

mechanical simplification: reduced cost
 more versatile control: easier to use
 increased automation: easier to use
 correction of systematic errors: better accuracy
 self-testing and fault diagnosis: cheaper to make and service
 easier to make functional improvements

The areas of convenience of use and presentation of data, that is the 'user interface', have tended to dominate recent commercial developments, as would have been obvious to anyone visiting this year's Pittsburgh symposium. A rapidly increasing proportion of instruments use visual display units (v.d.u.s) to guide the operator and give an initial display of the data. Often control is by use of 'soft' keys, that is keys whose function changes as indicated on the screen. This approach permits an unlimited range of operations to be handled without the use of a very complicated control panel or 'typed' commands.

Figure 2 shows the control panel and the lower part of the display screen of a modern i.r. instrument using such keys. The key functions displayed are the primary ones available initially to the operator.

Increased automation is another aspect of user convenience. There are two main aspects: (1) the automatic setting of parameters into self-consistent optimized sets such as scan speed (time) resolution and signal filtering, and (2) the ability to perform preselected sequences of operations. Figure 3 is a three-dimensional plot of phosphorescence against emission wavelength and delay time after excitation. The sequence of scans and presentation of data were performed without operator intervention, the instrument being controlled from an external, micro-processor-based 'data station'.

Such sequences of operations are also important for the automatic handling of multiple samples; not only may the scan sequence be controlled but the instrument conditions may be altered to suit the individual sample. Examples of this are the automatic selection of the optimum excitation or emission wavelength in fluorescence with scaling to fill the ordinate scale, or the setting of the baseline at a predetermined wavenumber in the i.r. Such functions

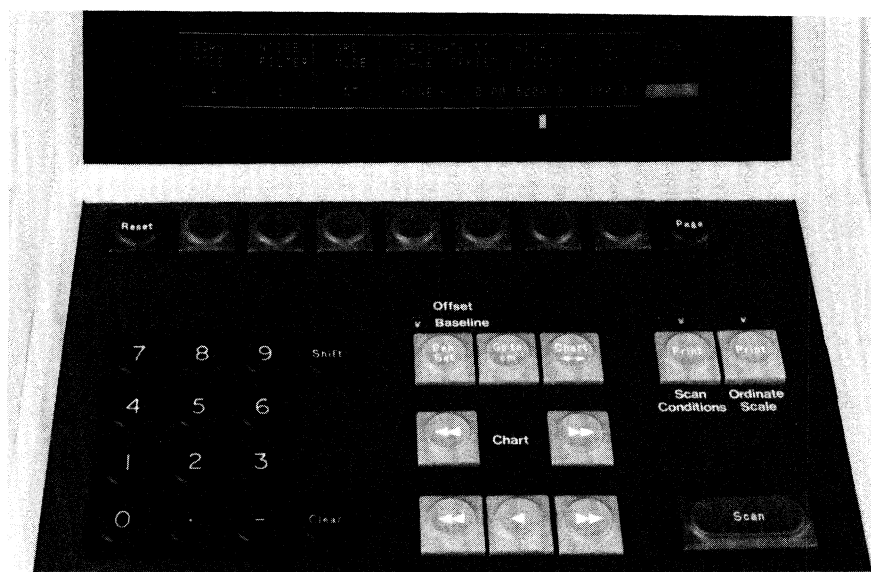


FIGURE 2. Control panel and lower part of screen of a modern dispersive infrared spectrophotometer, showing the use of 'soft' keys.

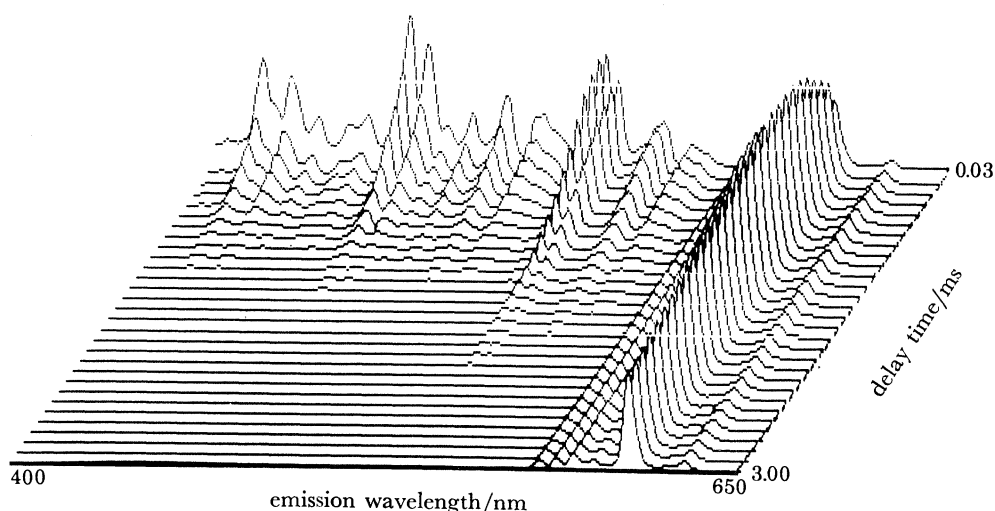


FIGURE 3. Plot of phosphorescence of a mixture of 0.5% Eu^{3+} in Y_2O_3 against emission wavelength and delay time after excitation. Excitation wavelength, 230 nm; delay time increment, 0.1 ms; number of scans, 30.

are often performed within modern instruments; more complex decisions can be made externally, for example optimizing the scanning conditions on the basis of a trial scan.

The correction of systematic errors is a less obvious but very important aspect of computer control. Provided that an effect is reproducible and can be measured, it can, in principle, be corrected. Typical examples are mechanical gear errors and effects of temperature, mains frequency and voltage and atmospheric pressure. Such correction is used in many commercial instruments and is generally better and cheaper than attempting to eliminate the errors at source.

Self-testing relates, at least in part, to this same question of error correction. So long as

suitable means of calibration can be built-in or provided, instruments can establish their own calibration factors. This can give a useful saving to the manufacturer as well as helping the user to maintain accuracy.

If any calibration factor goes outside a predetermined range the instrument may indicate a possible fault (self-diagnosis), and the nature of any malfunction, other than of the main microprocessor, can be often detected and displayed, permitting quick location of the cause. Some instruments can be coupled by telephone to the manufacturer's service department for detailed fault diagnosis.

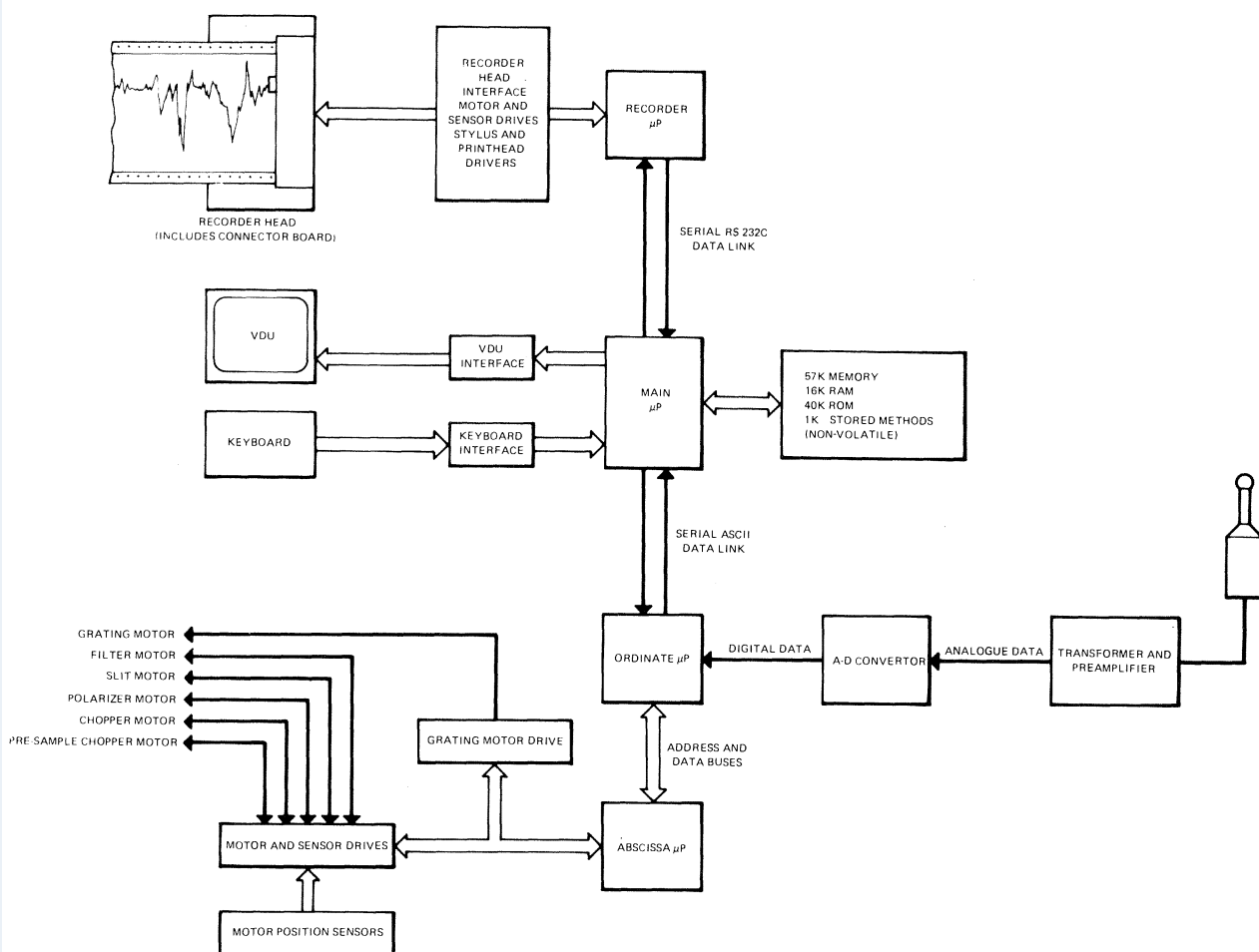


FIGURE 4. Control system of a dispersive infrared spectrophotometer showing the functions and relationships of the four microprocessors. μP , microprocessor; K, kilobytes; A-D, analogue-digital.

Finally, provided that consideration has been given to possible further developments in the initial design – e.g. by provision for enough memory – it is often possible to incorporate new developments by little more than a change of the program. The cost of development may still be high, but the speed and ease of introducing the change are greatly improved.

The complete control system for a recent dispersive i.r. spectrometer is shown in figure 4. There are four microprocessors: *abscissa* controlling the main scanning functions, *ordinate* performing the initial data processing, *main* controlling the v.d.u. and handling the user commands

as well as coordinating data transfer, and *recorder* controlling the printer plotter used to output spectra and other data.

The writing of the microprocessor programs for such an instrument is now a major part of the total development cost. It may already exceed 30% and is expected to rise to 50% in the future.

The magnitude of the programming task is illustrated in table 2. A very high proportion of the total relates to the user convenience features, handled by the main microprocessor. This is typically slow to write; being so subjective it can go through many iterations before the most acceptable result is achieved.

TABLE 2. THE SIZE OF PROGRAMS USED IN THE FOUR MICROPROCESSORS
IN A MODERN INFRARED SPECTROPHOTOMETER

(The programs were written in Motorola 6809 ASSEMBLER, taking $9\frac{1}{4}$ man years
at a cost of £259 000.)

microprocessor	program size (kbytes)
abscissa	11
ordinate	11
main	38
recorder	16
total	76

A very high proportion of commercial instrument control software is written in ASSEMBLER language. The efficiency in terms of speed and memory space used outweighs the extra programming cost, compared with a high-level language, when it is to be used in many hundreds of instruments. By contrast the typical academic development will be a single instrument, and efficiency in writing software will outweigh memory cost, provided that the lower speed can be tolerated. Therefore high-level languages are normally used, especially BASIC, even though this is typically 50–100 times slower in execution than ASSEMBLER.

Not surprisingly, because of the large commercial benefits of computer control, most work has been done by industry; however, many interesting academic applications have been published. An example is the work of Kasper *et al.* (1982) on computer control of broadly tunable lasers: the conversion of a colour-centre laser into a high-resolution laser spectrometer.

They describe the use of a minicomputer for the simultaneous control of three tuning elements that interact with each other, involving the use of both look-up tables – feed forward – and various forms of closed-loop feedback control. This work clearly illustrates many of the advantages of computer control.

DATA HANDLING

Data handling can be divided into two main areas: *signal handling*, which refers to the initial processing of the detector signal within the instrument, and is necessarily performed in ‘real time’, i.e. as it happens, and *data processing*, covering operations mainly performed on data prerecorded in some form of memory. The latter area can be further divided into four categories: manipulative, quantitative, qualitative, and bulk storage and retrieval.

Signal handling

Signal handling starts from the original digitization of the detector signal. There are some simple rules for digitization, which seem sometimes to be forgotten and are worth restating

briefly. (1) The smallest measurable increment must be less than the noise (by what factor is arguable). (2) The sampling rate must give at least two samples per cycle of the highest *noise* frequency. (3) The dynamic range must be sufficient to accept the largest signal. Provided that these conditions are met there should be no loss of information.

After initial digitization, the operations in a typical scanning spectrometer would be subtraction of 'zero', correction of transient effects due to scanning, ratioing in double-beam

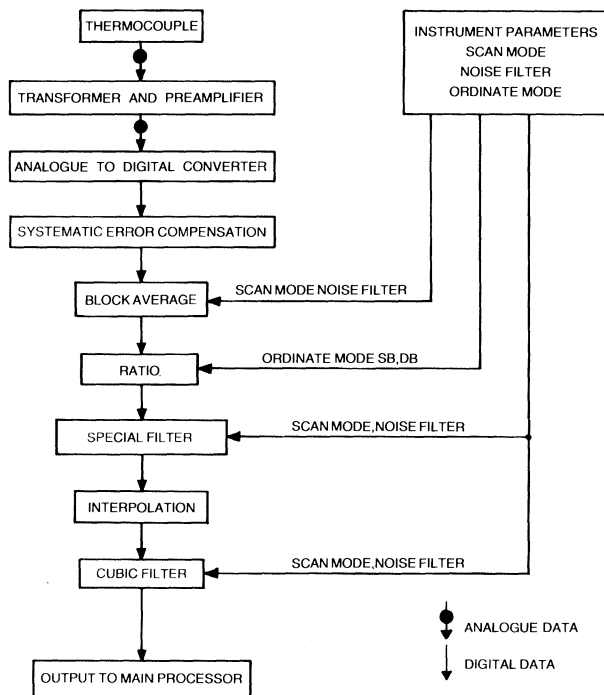


FIGURE 5. The ordinate data flow diagram of a modern infrared spectrophotometer.

instruments, filtering, correction of other systematic errors (e.g. baseline), absorbance conversion, scaling and interpolation for plotting or other display, and, of course, output to some suitable form of memory. The sequence of operations in a typical instrument is shown in figure 5. This type of time-critical operation is almost invariably programmed in ASSEMBLER and often demands the use of special fast algorithms. The special requirements of Fourier transform and photon correlation spectroscopy have been fully described elsewhere and will not be discussed here.

An interesting technique for handling the signal from a diode array detector has been described by Hicks *et al.* (1980). The spectrum is scanned across the array and each spectral element is measured in turn by each diode and the measurements co-added. The full multiplex advantage is obtained while avoiding the problems of variation of sensitivity of individual diodes. The system is used in an electron spectrometer but would be equally applicable to an optical spectrometer.

Data processing

Although some data-processing operations, particularly of the first category, may be performed within instruments, the majority of recent developments relate to the use of micro-processor-based data stations or minicomputers. It is these that are discussed below.

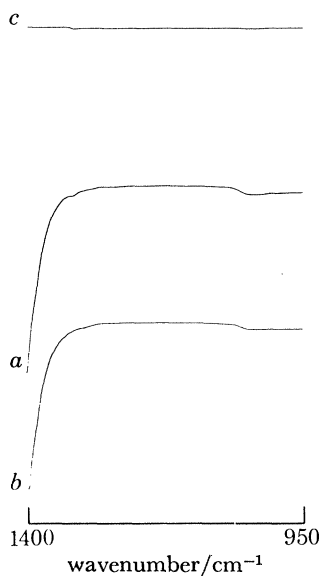


FIGURE 6. Spectra of (a) a $50 \mu\text{g g}^{-1}$ solution of the pesticide demeton-*S*-methyl sulphone in CS_2 ; (b) CS_2 blank; (c) computer-subtracted difference. Spectra (a) and (b) were each recorded in 3 min on a dispersive spectrophotometer.

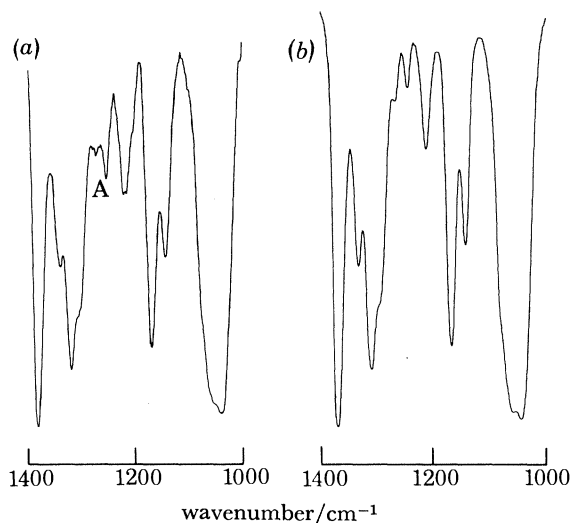


FIGURE 7. (a) Spectrum (c) of figure 6 expanded 400 times and smoothed; (b) the spectrum of a 20 times more concentrated solution expanded 20 times. Peak A is 10^{-4} absorbance.

Manipulative

This heading refers to procedures aimed at presenting data in a form most useful for the user to extract the relevant information, or for further data processing; it is rarely the spectrum itself that is required, but data extracted from it. Typical operations under this heading are: spectral subtraction in absorbance with one spectrum scaled, extra filtering, conversion between transmittance and absorbance, baseline flattening, generation of derivatives, deconvolution, scale expansion and a range of display techniques. Some of these functions may now be performed with parameters varied continuously while viewing the result on a screen. Nicolet have done

excellent work in this area, and both they and Digilab have developed special systems for dealing with the large quantities of data produced by Fourier transform i.r.-g.c. combinations.

One very important aspect of data-processing techniques is that, without them, it is very difficult to handle and interpret the increasing quantities of data produced by modern instruments using parallel detection techniques, such as diode arrays, vidicons and Fourier transformation (Fogarty *et al.* 1981). However, the special requirements of such instruments will not be discussed further here.

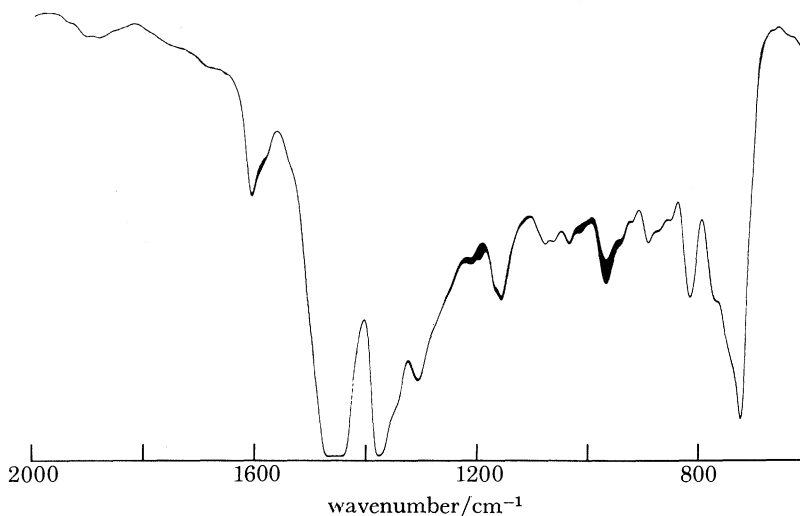


FIGURE 8. Superimposed spectra of a $1000 \mu\text{g g}^{-1}$ solution of tricresyl phosphate in a mineral oil and of the oil alone, indicating the areas of difference.

Figures 6 and 7 illustrate the use of spectral subtraction and expansion to obtain the i.r. spectrum of an extremely low concentration of an insecticide, which could not have been observed without the use of such techniques. The application of computer methods has greatly improved the sensitivity and precision of i.r. spectroscopy over recent years; for example the peak marked A in figure 7 is less than 10^{-4} absorbance units.

The ability to combine the various manipulative procedures in predetermined sequences together with the normal choice of mathematical operations is a very useful feature of some systems. This in effect provides the user with a spectrally oriented programming language, and can save much programming time.

Quantitative

Although the term covers a wide variety of measurements, discussion here has been restricted to quantitative chemical analysis. Many commercial instruments and some software packages exist for single- or multi-component analysis by 'traditional' techniques – peak height, area, etc. – but little has been done with the method of least squares.

The method, which is based on Legendre's principle, propounded in 1806, is described by Buckingham (1962) in his book on numerical methods. Undoubtedly the restriction in its use has been largely due to the extensive calculations that have to be performed, but these can now be done in an acceptable time by a microprocessor.

The technique is excellent for both multi-component and trace analysis (Haaland & Easter-

ling 1980). The measurement of a solution of $20 \mu\text{g g}^{-1}$ tricresyl phosphate (TCP) in a mineral oil was performed recently as a test of the sensitivity of the method. The very small difference between the i.r. spectra of the base oil and a $1000 \mu\text{g g}^{-1}$ solution of TCP in the oil is shown in figure 8.

The application of spectral subtraction techniques to the spectra of the $20 \mu\text{g g}^{-1}$ solution and the oil failed to produce any recognizable spectrum of TCP. However, the least-squares

TABLE 3. THE RESULT OF FIVE ANALYSES OF A $20 \mu\text{g g}^{-1}$ SOLUTION OF TCP, WITH BASE OIL AND A $166 \mu\text{g g}^{-1}$ SOLUTION AS STANDARDS

(Each spectrum used was recorded in 6 min on a dispersive spectrophotometer.)

base oil	$88.79 \pm 0.47 \%$	
solution	$11.21 \pm 0.47 \%$	
TCP	measured	actual
	$0.00186 \pm 0.00008 \%$	0.0020%

analysis gave the result in table 3. The standard deviation of the five separate measurements is remarkably small, although there is some indication of systematic error, possibly due to lack of temperature control. A check analysis of the base oil gave a TCP content of $0.8 \mu\text{g g}^{-1}$. The detection limit should thus be less than $2 \mu\text{g g}^{-1}$, equivalent to 20 ng TCP in the cell. The computation time in a microprocessor system was less than 20 s for each analysis.

Two special techniques are employed in the computation. Firstly, the presence of one extra component – a neutral absorber – is assumed. This has the effect of making it unnecessary to establish any zero absorbance baseline. (This can be extended from a neutral, i.e. zero order, absorber, to first, second or even higher order absorbers if sloping or curved baselines are encountered. In practice it seldom helps to use higher than first order.) Secondly, the data can be weighted according to the significance of each spectral interval; for an i.r. spectrum this would typically involve multiplication by the transmittance value of the unknown material being analysed. The former of these techniques has been used by H. A. Willis (personal communication 1981) and coworkers at I.C.I., but although the latter is described in mathematical terms by Buckingham I am not aware of its application to quantitative analysis. The technique is equally applicable to other forms of spectroscopy, e.g. u.v.–visible absorption and fluorescence, and has the important advantage of avoiding the need for the operator to make subjective decisions about the choice of wavelengths for use in the analysis.

Qualitative

A high proportion of the papers found in the search of *Chemical Abstracts* relate to methods of i.r. spectral library searching and computerized spectral interpretation as an aid to chemical structural determination, often in combination with mass spectroscopy or n.m.r. (Tomellini *et al.* 1981; Visser & Van der Mass 1981; Craver *et al.* 1981; Zupan *et al.* 1980; Schaarschmidt 1979; Small *et al.* 1979; Delany & Uden 1979; Tanabe *et al.* 1979; Gribov *et al.* 1979) and this is an area of continuing activity at a number of centres.

Commercial i.r. library search systems have existed for a number of years and an interpretative system was introduced by Perkin–Elmer in 1979. Since this system differs in its approach from most published work it is described here briefly.

This system was based on the recognition that, with over 5×10^6 chemical compounds already identified, the chance of finding the correct one in even a large spectral library was small. The

aim was therefore to identify closely related compounds. Experience with peak-matching routines indicated that they performed poorly in this respect, so it was decided to attempt to find some way of performing a match of major structural elements before performing a peak match. The method adopted was to identify, in the i.r. spectrum of the unknown, the presence or absence of bands, in certain spectral regions and within certain ranges of relative intensity, which were representative of specific chemical structures. Where ambiguities were observed in the interpretation, narrower categories were created until the ambiguity was resolved, if

TABLE 4. THE RESULT OF THE APPLICATION OF AN INTERPRETATIVE PROGRAM TO THE INFRARED SPECTRUM OF POLYVINYL CHLORIDE CONTAINING A PLASTICIZER

possible structural units: film 1	
202	alkyl group – general
208	alkyl group – methyl substituent
238	alkyl chain with alternating halogen substitution
316	halogenated hydrocarbon – multiple chloro substitution
1402	carboxylic acid ester – possibly aliphatic
4905	carbonyl compound – Class 5 (consult manual)
4925	carbonyl compound – possibly ester or ketone

Possible structural units above may be subject to interference. Consult manual.

necessary to the level of a single compound if this was anomalous. By using existing correlation tables and practical experience in spectral interpretation, 896 possible structural units were defined, each using, on average, 8 spectral regions, and these were classified into 48 major functional groups. The interpretation program compares the unknown with each of the 896 patterns in turn in a pass/fail test and lists the passes, this taking approximately 10 s on a data station.

The result of the application of this program to the spectrum of a polyvinyl chloride film containing a plasticizer is shown in table 4. In this instance the interpretation is virtually sufficient to identify the compound without the use of a library search.

The library contains the following information relating to each compound: code number, name, physical state, major constituent atoms, functional groups found by the interpretation program, and peak table with 1 cm^{-1} resolution. Data compression techniques are used to minimize memory requirements.

The significance of any match obtained by library searching can be expressed in terms of the ratio of the probability of the correct compound giving the observed degree of match to the probability of an unrelated compound giving the observed degree of match.

For given types of spectral data the latter can be calculated without having to make very gross assumptions, but the former depends greatly on the quality of data and, since the quality of spectral data in many libraries is very variable, little more than a qualified guess can be made at it. However, the expression is still a useful guide to the best algorithm for scoring 'degree of match' and between an 'unknown' and a library record and it was used in the choice of algorithms for scoring both functional group and peak matches.

Other than as a threshold defining the minimum intensity (relative to the strongest peak) below which peaks are not recorded, peak size is not used in the search, since there is sufficient selectivity without it. The average number of peaks in the search region of $1624\text{--}600\text{ cm}^{-1}$ is 16. Assuming random positioning, the chance of a perfect match of 16 peaks, allowing a $\pm 5\text{ cm}^{-1}$ tolerance, is less than 10^{-18} .

Bulk storage and retrieval

The final area of data processing, that of bulk storage and retrieval, is being worked on extensively by both commercial and non-commercial groups. However, there is little that is specific to spectroscopy. In addition to the laboratory-oriented systems now available, there are the major data bases such as the Chemical Abstracts Service of the American Chemical Society and the N.I.H., E.P.A. Chemical Information System, which can be accessed from a terminal by telephone from many countries. The full description of these systems is beyond the scope of this paper and readers are referred to the organizations concerned for further details.

SUMMARY

To summarize, this very wide field is one in which instrument manufacturers are playing a major part, mainly due to the sheer magnitude and cost of the programming effort involved. This can be justified for programs to be used by hundreds – possibly thousands – of users, in contrast to the academic situation where a program will often be used by only one person or group. However, some very interesting academic work is being undertaken, particularly in the area of computer-aided spectral interpretation in addition to special instrument control problems.

The author gratefully acknowledges help in the preparation of this paper from many colleagues in Perkin–Elmer, and from Professor J. T. Clerc of Berne University.

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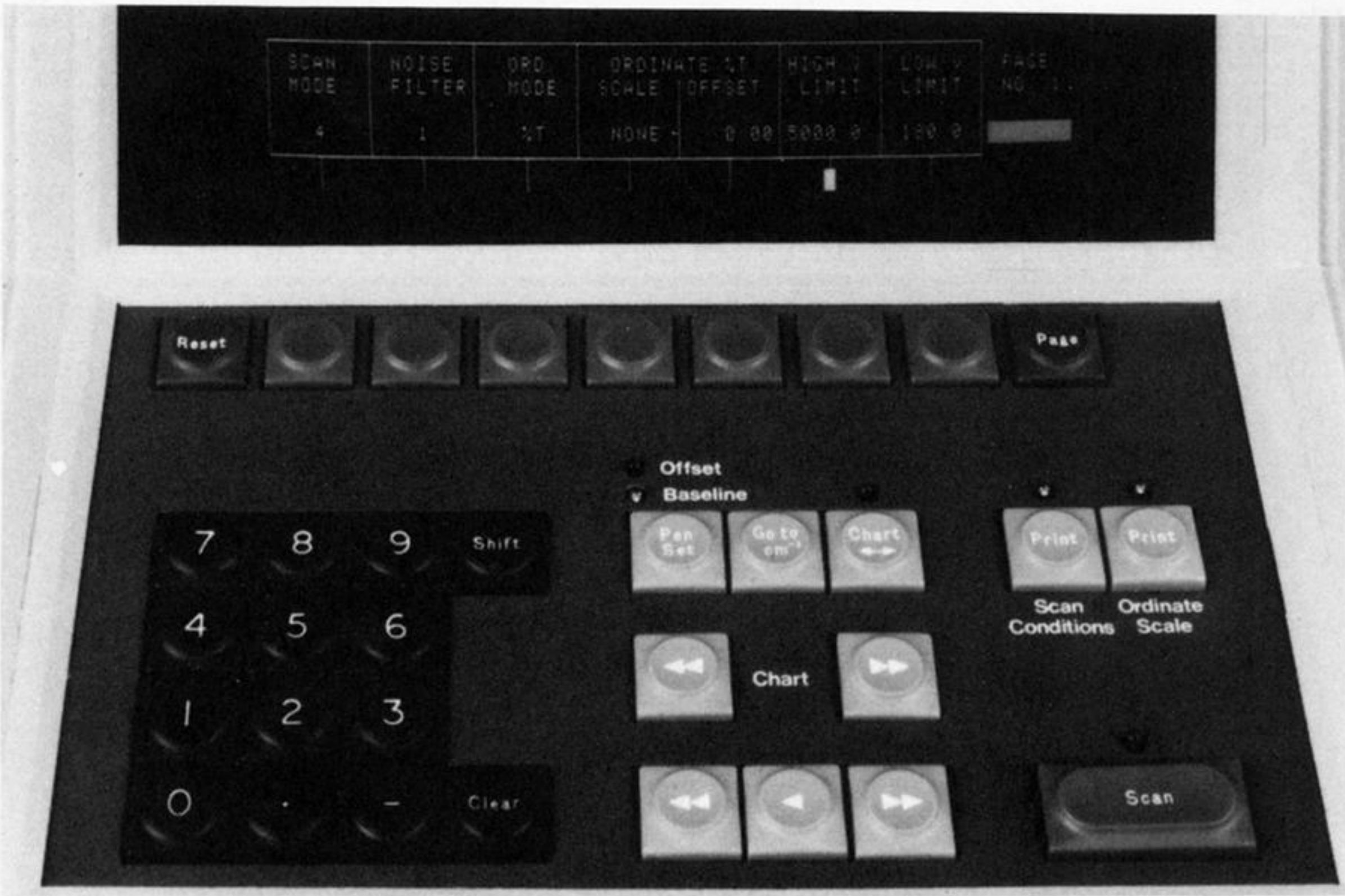


FIGURE 2. Control panel and lower part of screen of a modern dispersive infrared spectrophotometer, showing the use of 'soft' keys.