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Report by the Analytical Methods Committee

Evaluation of analytical instrumentation

Part XIV. Instrumentation for Fourier transform infrared spectrometry

Analytical Methods Committee†

The Royal Society of Chemistry, Burlington House, Piccadilly, London, UK W1V 0BN

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The Analytical Methods Committee has received and approved the following report from the Instrumental Criteria Sub-Committee.

Introduction

The following report was compiled by the above Sub-Committee of the AMC, which consisted of Professor S. Greenfield (Chairman), Mr D. C. M. Squirrell, Dr C. Burgess, Dr K. E. Jarvis, Professor S. J. Hill, Dr M. Barnard, Dr K. Altria and Dr M. Sargent, with Mr C. A. Watson as Honorary Secretary. The initial input of the features for consideration was undertaken by a working party chaired by Dr M. Barnard with Dr G. M. Barnard to whom the Committee expresses its thanks.

The purchase of analytical instrumentation is an important function of many laboratory managers, who may be called upon to choose between a wide range of competing systems which are not always easily comparable. The objectives of the Instrumental Criteria Sub-Committee are to tabulate a number of features of analytical instruments which should be considered when making a comparison between various systems. As is explained below, it is then possible to score these features in a rational manner, which allows a scientific comparison to be made between instruments.

The overall object is to assist purchasers in obtaining the best instrument for their analytical requirements. It is also hoped that this evaluation will, to some extent, also help manufacturers to supply the instrument best suited to their customers' needs. It is perhaps pertinent to note that a number of teachers have found the reports to be of use as teaching aids.

No attempt has been made to lay down a specification. In fact, the Committee considers that it would be invidious to do so: rather it has tried to encourage the purchasers to make up their own minds as to the importance of the features that are on offer by the manufacturers.

The XIVth report of the Sub-Committee deals with Fourier transform infrared spectrometry.

Notes on the use of this document

Column 1. The features of interest.

Column 2. What the feature is and how it can be evaluated.

Column 3. The Sub-Committee has indicated the relative importance of each feature and expects users to decide on a weighting factor according to their own application.

Column 4. Here the Sub-Committee has given reasons for its opinion as to the importance of each feature.

Column 5 onwards. It is suggested that scores are given for each feature of each instrument and that these scores are modified by a weighting factor and sub-totals obtained. The addition of the sub-totals will give the final score for each instrument.

Notes on scoring

1. (PS) Proportional scoring. It will be assumed, unless otherwise stated, that the scoring features will be by proportion, *e.g.*, from worst/0 to best/100.
2. (WF) Weighting factor. This will depend on individual requirements. An indication of the Sub-Committee's opinion of the relative importance of each feature is indicated as follows: VI (very important); I (important), NVI (not very important). A scale is chosen for the weighting factor which allows the user to discriminate according to needs, *e.g.*, $\times 1$ to $\times 3$, or $\times 1$ to $\times 10$. The factor could amount to total exclusion of an instrument.
3. (ST) Sub-total. This is obtained by multiplying PS by WF.

Fourier transform infrared spectrometry (FT-IR) is a well established analytical technique with applications in many areas. A wide range of instrumentation is available from different manufacturers. Systems range from instruments with a single beam splitter, a limited choice of detectors and limited resolution, to systems with a choice of beam splitter, detectors and variable resolution to enable a wider frequency range to be used. Systems can be controlled by the on-board microcomputer. However control and data collection is usually achieved by an external microcomputer which may also be used to control accessories and autosamplers. Selection of a suitable instrument for purchase is, therefore, not an easy task and the purpose of these notes is to provide some guidance to areas which should be considered, so that the choice is based on a full consideration of the available options. However, the performance of any FT-IR method depends primarily on the experimental conditions and thus on the sampling technique and spectroscopic parameters necessary to yield satisfactory data. The nature of the analyte will influence the choice of spectrometer and necessary sampling accessories.

† Correspondence should be addressed to the Secretary, Analytical Methods Committee, Analytical Divison, Royal Society of Chemistry, Burlington House, Piccadilly, London, UK W1V 0BN.

The first task in the selection of an instrument is to examine the range of analyses that it will be expected to perform. Care should be taken not to specify these requirements too closely as uses change with time. The analytical scientist should also not try to envisage every potential application or the selection criteria may become too detailed. The choice of mid-IR systems is discussed in this report. Systems for use in the NIR, coupled techniques *e.g.* GC-IR, or IR-microscopy are outside the scope of these guidance notes, but any specific requirements should be noted, such as special accessories or software.

With these requirements in mind, the user should then evaluate the instruments available on the market while bearing in mind the guidelines and any financial limitations. In many instances it will quickly become clear that a number of different instruments could be satisfactory and non-instrumental criteria may then be important. However, in some specialised cases only one or two instruments will have the necessary features to carry out the analysis.

The guidelines are intended to be used as a check list of features to be considered, mostly of the instrument itself, but some also of its service requirements and of the relationship of the user with the manufacturer. Their relative importance will depend on the installation requirements of the instrument as well as the uses to which it will be put. Therefore, to some extent, the selection process will inevitably be subjective, but if all the points have been considered, it should be an informed choice.

The Committee consider that, in general, FT-IR spectrometers are safe in operational use, but the manufacturer's safety instructions must be followed as the instrument incorporates a laser.

Finally, as many laboratories are now working to established quality standards, some consideration should be given to third party certification of the manufacturer to quality standards such as the ISO 9000 series. Such certification should extend to the service organisation.

Previous reports in this series from the Analytical Methods Committee

Evaluation of analytical instrumentation

Part I Atomic-absorption Spectrophotometers, Primarily for use with Flames, *Anal. Proc.*, 1984, **21**, 45. Revised 1997. *Analyst*, 1998, **123**, 1407.

Part II Atomic-absorption Spectrophotometers, Primarily for use with Electrothermal Atomisers, *Anal. Proc.*, 1988, **22**, 128. Revised 1997. *Analyst*, 1998, **123**, 1415.

Part III Polychromators for use in Emission Spectrometry with ICP Sources, *Anal. Proc.*, 1986, **23**, 109.

Part IV Monochromators for use in Emission Spectrometry with ICP Sources, *Anal. Proc.*, 1987, **24**, 3.

Part V Inductively Coupled Plasma Sources for Use in Emission Spectrometry, *Anal. Proc.*, 1987, **24**, 266.

Part VI Wavelength Dispersive X-Ray Spectrometers, *Anal. Proc.*, 1990, **27**, 324.

Part VII Energy Dispersive X-Ray Spectrometers, *Anal. Proc.*, 1991, **28**, 312.

Part VIII Instrumentation for Gas–Liquid Chromatography, *Anal. Proc.*,1993, **30**, 296.

Part IX Instrumentation for High-Performance Liquid Chromatography, *Analyst*, 1997, **122**, 397.

Part X Inductively Coupled Plasma-Mass Spectrometers, *Analyst*, 1997, **122**, 393.

Part XI Instrumentation for Molecular Fluorescence Spectrometry, *Analyst*, 1998, **123**, 1649.

Part XII Instrumentation for capillary electrophoresis, *Analyst*, 2000, **125**, 361.

Part XIII Instrumentation for UV–VIS–NIR spectrometry, *Analyst*, 2000, **125**, 367.

Instrumental criteria sub-committee evaluation form

Type of instrument FT–IR spectrometer						
Manufacturer:						
Model No.:						
Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
Non-instrumental criteria						
Selection of manufacturer	Laboratories in possession of other instruments should score highest for the manufacturer with the best past record based on the following sub-features:					
(*a*) Previous instruments						
(*i*) Innovation	Company's record for developing instruments with relevant innovative features.	I	The manufacturer should be aware of developments in optical design and detector technology.	PS WF ST		
(*ii*) Reliability record	Company's record for instrument reliability.	I	Reflects the company's ability for good design and manufacturing practices.	PS WF ST		
(*iii*) Confidence in the supplier	Confidence gained from past personal experience	I	Good working relationship already in place.	PS WF ST		
(b) Servicing	Score according to manufacturer's claims and past record, judged by the sub-features (*i*)–(*v*) below:	I				
(*i*) Service contract	Availability of suitable service contracts from the supplier, agent or third party contractor.	VI	Suggests long term commitment to user. Often ensures preferential service and guarantees a specific response time to call-outs.	PS WF ST		

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(*ii*) Availability and delivery of spares	Range of stock carried by, or quickly available to, the manufacturer/agent/ contractor.	VI	Rapid delivery of spares reduces downtime and cost of operating.	PS WF ST		
(*iii*) Call-out time	Adequate service personnel readily available minimising the call-out time.	I(VI)	Keeps laboratory in opertion by reducing down time [see also (*i*)].	PS WF ST		
(*iv*) Effectiveness of service engineers	The ability of the service engineers as judged from previous experience and reports of others, including the carrying of adequate spares.	I	Ability to repair on-site avoids return visit or removal of equipment for off-site repair so reduces down time and may reduce service cost.	PS WF ST		
(*v*) Cost of call-out and spares	Score for reasonable cost per hour and spares.	I	The proximity of the service centre may be a factor in travel costs.	PS WF ST		
(*c*) Technical support	As in (*b*) score in consideration of sub-features (*i*)–(*iv*) below.	VI for new user				
(*i*) Advice and training from the manufacturer	The quality of advice and training on the operation of the instrument from the manufacturer's applications department.	VI for new user	This helps in-house staff to make the most effective use of the equipment.	PS WF ST		
(*ii*) Technical literature	The range and quality of technical literature including the operating manual.	I	Guidance on optimum use of instrument suggests manufacturer's awareness of applications.	PS WF ST		
(*iii*) Telephone assistance	Willingness of the manufacturer or supplier or contractor to give effective advice over the telephone. This can normally only be evaluated by reference to existing users.	I	Rapidly available technical help reduces the number of call-outs and enhances productivity.	PS WF ST		
(*iv*) Customer maintenance	Score for the ability of the user to do verification and routine maintenance, *e.g.*, change a consumable.	I	Reduces call-out cost for a simple procedure.	PS WF ST		
Instrumental criteria 1. *General features* (*a*) Facilities required for:						
(*i*) Access and location of connections and controls on instrument	Score according to convenient access taking into account the proposed location of the instrument.	I	Depending on bench position and layout, connections and controls may limit accessibility for servicing and installation particularly at the rear of the instrument	PS WF ST		
(*ii*) Power requirements	Many systems require multiple power inputs. Score maximum for instruments with the minimum of separate power leads.	NVI	Excessive numbers of power cables may create hazards and make servicing more difficult.	PS WF ST		
(*iii*) Power failure effects	Score highest for systems that allow recovery from power failure with minimal data/control loss.	I(VI)	Down time is increased if power failure necessitates manual resetting of instrument control parameters.	PS WF ST		
(*iv*) Size of equipment *i.e.* 'benchprint'	Score according to convenience of installation.	I(VI)	Dimensions may be critical if space is limited.	PS WF ST		
2. *Spectral sources and detectors*	A variety of sources and detectors are available to cover the required spectral range.					
(*a*) IR source	Score highest for the source which has the highest energy output and the longest life. Score additionally for any warranty.	VI	Sources include glow bar which needs liquid cooling, lower energy filament source with a short lifetime and ceramic emitter optimised for mid-IR.	PS WF ST		
(*b*) Ease of source replacement	Score highest for easiest replacement and alignment of the source.	I	The user should be able to replace and align a new source without the need for a service engineer.	PS WF ST		
(*c*) IR detector	Score highest for the system which provides the most sensitive detector.	VI	Some low cost detectors are not linear at low energy throughput. The ability to use a cooled detector for energy limiting applications is advantageous.	PS WF ST		
3. *Interferometer design*						
(*a*) Internal construction	The performance of all interferometers relies on the maintained accuracy of the alignment between the fixed and moving mirror.	I				
(*b*) Sealing	Score zero if the interferometer is not sealed to minimise the interference of water and carbon dioxide.	I				
(*c*) Interferometer design	Score maximum for the system which provides the facility for dynamic alignment and auto-tune.	VI	These facilities compensate for wear in the bearings which can cause tilt and sheer between the mirrors.	PS WF ST		

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(*d*) Spectral range	Score highest for the system which has a beam splitter to cover the spectral range of interest.	VI	The choice of beam splitter dictates the spectral range. [**N.B.** Some beam splitters must only be used in a purged interferometer.]	PS WF ST		
(*e*) Spectral resolution	Score highest if the system allows the user to select the wavenumber resolution suitable for the application.	VI	1 cm^{-1} is adequate for many quality control (QC) applications but higher resolution is usually necessary for analytical work.	PS WF ST		
4. *Sample compartment*						
(*a*) Size	Score maximum for the availability of adequate space for appropriate accessories.	I	Convenience of interchangeability of accessories increases productivity and reduces errors.	PS WF ST		
(*b*) Accessibility	Score highest for interferometers which allow ready access for the installation of accessories.	VI	Ease of access is very important when using multi-samplers to ensure efficient operation.	PS WF ST		
(*c*) Gas purge	Score if this feature is provided.	VI	To purge the sample compartment with dry nitrogen to minimise the interference of water vapour and carbon dioxide which absorb in the mid-IR.	PS WF ST		
5. *Sample presentation*						
(*a*) Liquids						
(*i*) Cells for transmission/ absorbance	Score highest for the availability of cells at a selection of fixed pathlengths and appropriate crystals for the chosen spectral range.	VI	Fixed pathlength cells minimise errors due to varying pathlengths between the measurement of the solvent and sample. The correct choice of crystal material ensures that the spectral range is suitable for the sample.	PS WF ST		
(*ii*) Horizontal attenuated total reflectance accessory (HATR) with trough plate	Some highest for a horizontal attenuated total reflectance accessory (HATR) which on installation is automatically aligned by the system. Score additionally if a selection of crystals is available.	VI	The crystal used must be resistant to the chemical being measured. The depth of penetration of the sample varies depending on the refractive index of the crystal used. This is an excellent technique for highly absorbing samples as it only requires the sample to be placed in the trough.	PS WF ST		
(*b*) Solids						
(*i*) Horizontal attenuated total reflectance accessory (HATR) with flat plate	See (*ii*) above.	VI	The crystal used must be resistant to the chemical being measured. The depth of penetration of the sample varies depending on the refractive index of the crystal used. This is an excellent technique for highly absorbing samples as it only requires the sample to be placed on the crystal.	PS WF ST		
(*ii*) Diffuse reflectance accessory (DRIFT)	Score highest for a diffuse reflectance accessory (DRIFT) which on installation is automatically aligned by the system.	VI	Powders can be analysed without any sample preparation which minimises changes in crystal structure which sometimes occurs when the sample is ground and pressed to make a disc.	PS WF ST		
(*c*) Gas cells	Some highest if short and long pathlength gas cells are available where necessary. Score additionally for the availability of different window materials for these cells if it is important.	I	Gas cell pathlengths can vary from a few centimetres to metres long depending on the concentration of the gas. Different window materials are needed to cover the required spectral range.	PS WF ST		
(*d*) Specular reflectance accessories	Score only if appropriate for the availability of these accessories which enable the specular reflectance from materials to be measured.	I	These measurements are used to study the surface of a reflecting sample.	PS WF ST		
6. *Spectral performance*						
(*a*) Range	Score for the appropriate spectral range needed, *e.g.*, 7400–350 cm^{-1} for the mid-IR	VI	The spectral range will be determined by the choice of beam splitter and detector.	PS WF ST		
(*b*) Resolution	Score highest for the system which provides the resolution appropriate to the samples to be analysed.	VI	If gases are to be analysed it is necessary to select a system offering high resolution of approx. 0.125 cm^{-1}. A routine system for QC or quantitative work will offer resolution of 0.5–1.0 cm^{-1}.	PS WF ST		

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(*c*) Signal to noise ratio	Sore highest for the system offering the best signal to noise.	VI	The higher the signal to noise ratio the fewer scans are needed to acquire a quality spectrum.	PS WF ST		
(*d*) Linearity	Score highest for the system with the most linear detector system particularly for low energy applications.	VI	This is important when using some accessories where the energy throughput may be greatly reduced.	PS WF ST		
7. Instrument control and data collection						
(*a*) On-board computer	Score only if a simple system will provide all the data processing required.	NVI	The system will have a screen and the facilities provided will include basic data manipulation and usually the ability to print the data to a printer.	PS WF ST		
(*b*) External control of instrument parameters	Score highest for a comprehensive software package to control the spectrometer and collect the data.	VI	Ensures that the same analyses are always performed under identical conditions. This is vital if the system is in a regulated laboratory. Manufacturer supplied software will have to be validated. It is rarely cost effective to write one's own software.	PS WF ST		
(*c*) Instrument performance diagnostics	Score maximum for a system which self checks on power up and has a simple validation routine programmed into the software.	VI	As more systems are used in regulated laboratories it is vital that the system performs diagnostic checks on power up.	PS WF ST		
8. Data manipulation						
(*a*) Data collection software	Define the requirements before scoring these items. Most manufacturers offer software packages with routines for setting the instrument parameters and collecting the data. How the software runs can be a very personal choice. Make sure that the features offered are fully evaluated. The ease with which the data can be acquired and reports generated are of prime importance. Score only for the availability of essential routines.	VI	Control and data collection software options are essential for data integrity and must include all of the required routines. Software packages from the manufacturer are expensive, but the effort it will take to write and validate one's own software would prove to be extremely time-consuming and therefore more costly.	PS WF ST		
(*i*) Storage of data files	Score maximum for a system where all data collection parameters are stored with the ASCII data and ensure that the data format is compatible with any spectral library that will be used.	VI	ASCII data can be exported to and manipulated by many software packages. This expands the scope of data manipulation, spectral search and chemometric routines available for the user.	PS WF ST		
(*ii*) Display software routines	Score maximum for a comprehensive set of spectral display routines. Score additionally for the possibility of pseudo 3D contour plotting if this feature is considered to be desirable.	VI	Simple versatile spectral display routines are vital. Pseudo 3D contour plotting shows additional features especially if studying time resolved data.	PS WF ST		
(*b*) Data handling						
(*i*) Software to perform all arithmetic functions *e.g.* area, smoothing, derivatives, averaging, calculations on single points or spectra	Score according to the availability of these routines especially if spectral comparison measurements are to be made or multi-component calculations envisaged. For accessories score additionally for the ability to transform data, *e.g.*, Kubelka–Munk functions for reflectance data.	VI	The software enables routines to transform raw data without having to use third party software. This is particularly important for GLP, GMP and all regulated industries.	PS WF ST		
(*ii*) Quantitative analysis routines using single wavelength data or spectra for multi-component work	Score according to the availability of routines, for example, single wavelength quantitation. Score additionally if spectral data can be exported to be used in multi-component analysis sofware.	I(VI)	Linear and other non-linear functions should be provided for single wavelength quntitative analysis.	PS WF ST		

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(*iii*) Software to control any accessory and simul-taneously collect associated data	Score for the availability of routines to control and collect data directly from accessories, *e.g.*, computer controlled HATR or DRIFT accessories.	I	Simplifies the measurements and ensures that all data are compatible when doing the calculations.	PS WF ST		
(*iv*) Specific application routines, the ability to customise and record the settings	Score if this feature is present and appropriate.	(VI)	This facility enables routines to be written so that staff can perform the analysis routinely.	PS WF ST		
(*v*) Routines for checking the sensitivity of the system	Score maximum for the availability of a routine and an appropriate 'standard' sample to make these measurements.	VI	Keeping track of the system sensitivity is vital as it will show up a change in the system performance.	PS WF ST		
(*vi*) Validation software	Score maximum for a system which complies with the regulatory authorities' standards with validation routines as a standard feature.	VI	Essential in many laboratories for regulatory requirements for GLP, GMP and all regulated laboratories.	PS WF ST		
(*vii*) Instrument performance	Score maximum for an instrument which self-calibrates on power up and has a simple validation routine programmed into the software.	VI	As more instruments are used in regulated laboratories, it is vital that the system performs an effective calibration routine on switch on. This information *must* be stored.	PS WF ST		
(*viii*) Data collection software	Score maximum for a system where all data collection parameters are also stored with the ASCII data.	VI	Deviations must be flagged by the software. Essential for GLP, GMP and all regulated industries.	PS WF ST		
9. Hardware and output requirements						
(*a*) Computer	Score for compatibility with either existing or company selected computer.	VI	There may be a company requirement for uniformity. Speed and ability to upgrade are important.	PS WF ST		
(*b*) Output devices	Score maximum for a system which uses a standard printer, *e.g.,* laser, inkjet, dot matrix	VI	This facility has become very important for uniformity.	PS WF ST		
(*c*) Data storage	Score for possibility to store data on suitable media for future retrieval and use.	VI	This is very important as is the provision of a hard copy. Date and acquisition parameters must also be archived.	PS WF ST		
(*d*) Data output from simple instruments	Score if printed data output from the instrument is required.	(I)	It is beneficial if the system can be coupled to a standard printer to produce a hard copy.	PS WF ST		
(*e*) Ability to be networked		I(VI)	In many laboratories the instrument is run from a PC and at a suitable time the data is transferred to a server.	PS WF ST		
				Sum of sub-totals		
10. Value for money (Points per £)	Sum of the previous sub-totals divided by the purchase price of the instrument. Subject to proportional scoring and weighting factors, including ST in grand total	I	'Simple' instruments are often good value for money, whereas those with unnecessary refinements are often more costly.	PS WF ST		
				Grand total		

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