AMC
THE ANALYST
www.rsc.org/analyst

Report by the Analytical Methods Committee

Evaluation of analytical instrumentation. Part XV. Instrumentation for gas chromatography-ion-trap mass spectrometry

Analytical Methods Committee†

The Royal Society of Chemistry, Burlington House, Piccadilly, London W1J 0BA

Received 28th March 2001
First published as an Advance Article on the web 10th May 2001

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

Introduction

This report was compiled by the above Sub-Committee of the AMC which consisted of Professor S Greenfield (Chairman), Dr M Barnard, Dr C Burgess, Professor S J Hill, Dr K E Jarvis, Dr M Sargent and Mr D C M Squirrell with Mr C A Watson as Honorary Secretary. The initial input of the features for consideration and the reasons for their consideration was undertaken by a working party Chaired by Dr M Sargent with Dr A Edge, Dr G O'Connor and Dr K S Webb, to whom the committee express their thanks.

The purchase of analytical instrumentation is an important function of many laboratory managers, who may be called upon to choose between a wide variety 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 and as an aid to equipment qualification.

The overall object is to assist purchasers in obtaining the best instrument for their analytical requirements. It is hoped that this evaluation will, to some extent, also help manufacturers to supply the instrument best suited to their customer's needs. It is perhaps pertinent to note that a number of teachers have found the reports 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 various features of the equipment that is on offer by the manufacturers.

The XV

importance of each feature and expects the 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. It is suggested that scores are given for each feature of each instrument and that these scores are modified by the 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 of features will be by

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score
(				

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*v*) Dimensions	Score according to compatibility of dimensions (width and depth) with space available.	I	Availability of suitable bench space. This may be important in some circumstances.	PS WF ST				
2. *Gas supplies*								
(*a*) Gas control	Score maximum for a control system which gives the most stable gas flows under the required operating conditions.	VI	Control is needed to give a constant gas flow, upon which adequate precision and reproducibility are partially dependent.	PS WF ST				
(*i*) Flow controllers	Score maximum for the provision of the most accurate and precise mass-flow controllers.	VI	Useful for packed and wide-bore open tubular columns where back pressure variation may occur. Useful with open-tubular columns.	PS WF ST				
(*ii*) Pressure controllers	Score maximum for the provision of the most accurate and precise pressure controllers.	VI		PS WF ST				
(*iii*) Electronic control	Score for availability of electronic control of both flow and pressure.	VI	Electronic control is more precise and reproducible than manual control. This is particularly desirable if the settings will be changed frequently.					
(*iv*) Controlled temperature	Score additionally if the flow controllers are contained within a controlled temperature environment.	I	A controlled temperature environment will increase the stability of the system.	PS WF ST				
(*b*) Gas supply features								
(*i*) Carrier leak detector	Score zero if this feature is not present when combustible gases are to be used.	VI	Vital for safety if hydrogen is used as carrier gas to avoid build-up of gas in the oven in the case of a leak. Also to avoid wasting gas.	PS WF ST				
(*ii*) Detector 'make up gas'	Score if ancillary detectors are to be purchased.	I	Need to maintain a suitable gas flow through the detector when capillary columns are used.	PS WF ST				
(*iii*) Gas purity requirements	Score highest if operating specification can be achieved without ultra-pure gases.	I	Ultra-pure gases/gas purifiers are expensive.	PS WF ST				
(*iv*) Auto purge facility	Score for well-designed system.	I	Carrier gas may be wasted if it is of poor design, *e.g.* purge is left on indefinitely after a run.	PS WF ST				
(*c*) Connections								
(*i*) Gas supply lines	Score according to availability and ease of fitting of non-permeable gas lines. Also use of standard (or consistent) gas fittings.	I	Plastics can age with use and if exposed to sunlight. Metal tubing is more robust and essential if hydrogen is used. Different fittings on GC and MS complicates servicing and modification.	PS WF ST				
(*ii*) Gas purifiers	Score according to availability, stated efficiency, and ease of fitting of on-line traps, such as activated carbon or molecular sieves, into gas supply lines.	VI	Removal of oxygen and water from carrier gas is desirable for some sensitive stationary phases or for operation at high sensitivity. Oil may also need to be removed from air lines if a compressor is used.	PS WF ST				
3. *Injection ports*								
(*a*) General								
(*i*) Ease of cleaning	Score according to ease by which units can be dismantled and reassembled for cleaning.	I	The need to remove involatile residues.	PS WF ST				
(*ii*) Replaceable liners	Score for provision of replaceable liners for injection ports.	VI	Replacement of the liner removes involatile residues and reduces contamination.	PS WF ST				
(*iii*) Septum replacement	Score according to ease of removal and replacement of septa.	VI	Frequent changes of septa are necessary for satisfactory operation.	PS WF ST				
(*iv*) Septum purge	Score additionally for provision of bleed of carrier gas from just below septum.	I	Removes volatiles arising from degradation of the septum and reduces background peaks. Particularly needed for temperature programmed separation on open-tubular columns.	PS WF ST				
(*b*) Heaters								
(*i*) Injector heater control	Score highest for most stable control of temperature of injector heater unit. Score additionally for an independent temperature read out.	I	Injector temperature can affect volatilisation and sample stability on injection.	PS WF ST				

Feature		Definition and/or test procedures and guidance for assessment	Importance	Reason	Score
(*ii*)	Programmable cooling/ heating of injection zone	Score for availability of a programmable unit for temperature control of the injection zone.	I	Can be used in split/splitless injection (particularly important for	

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*c*) Oven programmers								
(*i*) Temperature/ gradient settings	Score highest for provision of digital control rather than analogue control for temperature gradient settings.	I	Digitally controlled temperature programmes are easier to reproduce.	PS WF ST				
(*ii*) Number of steps available	Score according to the numbers of separate delay periods and temperature ramps that can be programmed.	I (for complex samples)	The more steps available, the greater the flexibility. Desirable for complex samples, particularly to flush off involatiles. Most samples will only need a limited number of steps in the programme.	PS WF ST				
(*iii*) Heating rate	Score highest for the maximum ramp rate that the oven can achieve over the temperature range required.	I	High rates are needed for cold on-column injections or with short columns. Also needed when chromatograph is linked to autosampler to coordinate injection with temperature programme and data collection.	PS WF ST				
(*iv*) External control programme	If an external computer system is likely to be used, score for ability to control all necessary temperatures.	VI	An external computer may be useful for special purposes or remote operation.	PS WF ST				
(*v*) Reproducibility of programmed temperature	Score highest for the best reproducibility in temperature control on resetting programme.	VI	Programme reproducibility is more important than accuracy. Needed to ensure consistency of results.	PS WF ST				
(*d*) Column installation								
(*Column materials and stationary phases are outside the scope of this evaluation.*)								
(*i*) Column fittings	Score according to ease of changing columns.	I	Self evident.	PS WF ST				
(*ii*) Inter-changeability	Score for the ability to interchange between open-tubular, and wide-bore columns. When narrow open-tubular columns are to be used, score additionally for minimum dead volume in mass spectrometer interface.	I	Gives maximum flexibility in use of system, bearing in mind that most instruments are dedicated to one mode. Presence of large dead volumes degrades separation efficiency and may cause discrimination effects.	PS WF ST				
(*iii*) Ability to use wide-bore columns	If the application calls for the use of wide-bore columns, score additionally for the provision of this feature.	NVI	Allows greater column loadings.	PS WF ST				
5. *Autosampler*								
(*a*) General	Score for availability of the sub-features (*i*)–(*iv*) listed below.							
(*i*) Interfacing compatibility of micro-controller with the GC-MS computer system	Score for the ability of the autosampler to be controlled by the mass spectrometer system.	VI	Needed for reliable and flexible automatic operation and data collection. Some units operate only from their in-built controller which may have limited facilities.	PS WF ST				
(*ii*) Inter-changeability between sample injection ports	Score for ability of autosampler to inject into each of the available ports.	I	Needed in dual-column instruments so that either column position can be used.	PS WF ST				
(*iii*) Carousel sample capacity	Score for the maximum number of sample positions.	I	A large number of sample positions means that more samples can be analysed unattended.	PS WF ST				
(*iv*) Carousel temperature	Score for availability of temperature control for samples awaiting injection.	I	Permits pre-column derivatisation or cooling for thermally labile or volatile samples.	PS WF ST				
(*b*) Injection system								
(*i*) Injection volumes	Score maximum for greatest range of injection volumes that can be programmed.	I	If different volumes can be programmed for each injection this increases versatility so that different levels of analyte concentration can be handled.	PS WF ST				
(*ii*) Minimum sample size	Score maximum for system requiring minimum amount of sample in vial to flush needle and make injection.	VI	Sample size may be limited. Amount can be dependent on position of needle tip in vial and hence shape of vial.	PS WF ST				
(*iii*) Sample carry-over	Score highest for the system which minimises contamination of the next injection. Score additionally if needle wash is available.	VI	Avoidance of cross-contamination. Intermediate blank samples may otherwise be needed which will, however, increase analysis time.	PS WF ST				

Feature

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score
(*iv*) Changing between CI and EI	Score highest for instrumentation that allows the sources to be changed without having to re-evacuate the mass spectrometer. Score additionally for instrument that allows a combination of CI and EI in one chromatographic run paying particular attention to the reliability of the check valve system.	VI	EI and CI require different sources. These can either be combined on the same unit or be independent. Any leak or malfunction will cause serious operating/performance problems.	PS WF ST
(*c*) Mass analyser				
(*i*) Mass range	Score for the size of the range between the maximum and minimum mass detected. Most systems operate with			

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score
(*vii*) Ease of replacement	Score according to ease of replacement.	VI	Most multipliers are small and therefore difficult to work with. The problem is reduced for simple slot in type devices constructed on ceramic plates.	PS WF ST
(*viii*) Detector efficiency	This describes the percentage of incident radiation converted into secondary emissions at a given voltage. Score for the most efficient.	I	The higher the efficiency the greater the sensitivity and detector lifetime.	PS WF ST
(*e*) Operating characteristics				
(*i*) Tuning	Score for the ability to use an internal or external source to calibrate the instrumentation.	I	Provides increased flexibility of operation.	PS WF ST
	Score additionally for automatic tuning which generates a report of the settings which have been applied.	I	Knowledge of automatic tune settings reveals anomalies and is needed for QC purposes.	PS WF ST
(*ii*) Instrument stability	Score highest for a stable response over both short term (minutes) and long term (hours).	VI	Any drift in instrument response will degrade the quality of analytical results. Significant drift will necessitate frequent recalibration.	PS WF ST
(*iii*) Selective ion mode	The capability of acquiring data by repetitively monitoring a number of mass peaks selected by the operator anywhere within the instrument's mass response range. Score zero if this feature is absent.	VI	Operation in this mode maximises the data acquisition rate on mass peaks of interest. This improves the signal, increasing the sensitivity of the instrument to the selected ions.	PS WF ST
(*iv*) Isotope ratio measurement	The ability to calculate the abundance of one isotope relative to another. If relevant to the type of application, score for the availability of specialised software that allows data to be acquired and processed to maximise precision.			

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score
7. *Software*				
(*a*) General aspects				
(*i*) PC-based software for overall control and data processing	Score for availability of this feature, as well as individual control of the GC and the MS.	I	Most instruments utilise a PC for overall instrument operation and control but also incorporate separate microprocessor controllers for the GC and the MS. Availability of both allows maximum flexibility of operation.	
(*ii*) Ease of use	Score according to the ease which a new user can learn to use the software.	VI	This reduces the time spent learning how to use the instrumentation and also reduces operator errors.	PS WF ST
(*iii*) Availability of validatable software	Score for software that has been developed under a recognised quality system and fully documented.	VI (where applicable)	Essential for laboratories operating in a regulatory environment.	PS WF ST
(*iv*) Processing of results	Score for the degree of automation by which results can be processed and reported. Score additionally for full access to the raw data which has been			

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
8. *Additional features and accessories*	These features may be required for specific analytes or applications, and enquiries should be made as to the availability of suitable accessories. These features should only be scored when appropriate.							
(*a*) Column switching	Score for the ability to transfer eluent gas flow to second column and to reverse flow through column.	I	Can be used to facilitate heart-cut and back flush methods for complex samples.	PS WF ST				
(*b*) Headspace sampling unit	Score for the availability of this feature.	I	Extraction and injection of headspace vapour from above solid or liquid complex matrices. This is used for volatile analytes in comparatively less volatile matrices.	PS WF ST				
(*c*) Headspace analysis	Score if automated headspace analysis is required. Score according to the flexibility of the headspace analyser. Score higher for a combined shaker and heater system.	I (depending on application)	Useful introduction technique as it reduces sample preparation time.	PS WF ST				
	Score for the availability of this feature.	I	Ability to thermally degrade sample rapidly in inlet carrier gas flow is used to analyse involatile samples as characteristic volatile fragments.	PS WF ST				
(*e*) Thermal desorption unit.	Score for the ability to interface the chromatograph to a thermal desorption unit.	I	Thermal desorption units are often required for the analysis of volatile analytes in solid matrices including toxins from entrapped environmental air samples.	PS WF ST				
				Sum of sub totals				
9. *Value for money* (points per £)	Sum of the previous sub-totals divided by the price of the instrument. This sub-total should be subject to proportional scoring and weighting factors and included in grand total.	VI	‘Simple’ instruments are often good value for money, whereas those with unnecessary refinements are more costly.	PS WF ST				
				Grand total				