Accred Qual Assur (2008) 13:453–464
DOI 10.1007/s00769-008-0358-x

GENERAL PAPER

Evaluation of analytical instrumentation. Part XXIII. Instrumentation for portable X-ray fluorescence spectrometry

Analytical Methods Committee, Royal Society of Chemistry

Received: 10 January 2008 / Accepted: 11 January 2008 / Published online: 4 March 2008

Abstract The reports of this series tabulate a number of features of analytical instruments that should be considered when making comparison between various systems. Scoring these features in a rational manner allows a scientific comparison to be made between instruments as an aid to selection. This is the XXIII report of the series and deals with instrumentation for portable X-ray fluorescence spectrometry.

Keywords Instrumentation · Overview · Evaluation · X-ray fluorescence spectrometry

Introduction

The following report was compiled by the above Sub-Committee of the AMC, which consisted of Professor S. Greenfield (Chairman), Dr. M. Barnard, Dr. C. Burgess, Mr. D. Edwards, Professor S. J. Hill, Dr. K. E. Jarvis, Dr. G. Lord, Dr. M. Sargent, Professor P. J. Potts, Dr. J. Price and Dr. M. West with Dr. E. J. Newman as Secretary. The initial input of the features for consideration was undertaken by a working party comprising Professor P. J. Potts and Dr. M. West 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 wide ranges of competing systems that are not always easily comparable. The objectives of the Instrumental Criteria Sub-Committee are to tabulate a number of features of analytical instruments that 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 as an aid to selection.

The over-all 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 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 XXIII report of the Sub-Committee deals with instrumentation for portable X-ray fluorescence 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.

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

Analytical Methods Committee, Royal Society of Chemistry
The Royal Society of Chemistry, Burlington House,
Piccadilly, London W1V 0BN, UK
e-mail: vandenewman@tiscali.co.uk

Column 5 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 grand total will give the final score that can contribute to the selection of the instrument that best suits the user's requirements.

Notes on Scoring

1. (PS) Proportional scoring. It will be assumed, unless otherwise stated, that the scoring of features will be by proportion, e.g., Worst/0 to Best/100.
2. (WF) Weighting factor. This will depend on individual requirements. All features mentioned in the tables have some importance. If, in Sub-Committee's opinion, some features are considered to be of greater importance they are marked I. Those features of greatest importance are marked as VI (very important). A scale should be chosen for the weighting factor that allows the user to discriminate according to needs, e.g., x1 to x3 or x1 to x10.
3. (ST) Sub-total. Multiplying PS by WF obtains this.
4. In some circumstances, where there is a fundamental incompatibility between a feature of the instrument and the intended application, it may be necessary to exclude an instrument completely from further consideration.

With these requirements in mind, the user should then evaluate the instruments available on the market taking into account the following 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 become important. However, in some specialized cases only one or two instruments will have the ability or necessary features to be used in the intended application.

The guidelines are intended to be used as a checklist of features to be considered, mostly of the instrument itself, but also of service requirements and any existing relationship between the user and manufacturer. The relative importance of these features will depend on a number of factors, which in some circumstances could be subjective. However, if all the points have been considered, the choice should be informed.

The Committee consider that instrumentation for portable X-ray spectrometry is safe in normal use, but care should be taken to avoid exposure to X-ray radiation by ensuring that all safety features are fully operational and that instrumentation is used strictly in accordance with the manufacturer's instructions.

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

An overview of portable X-ray fluorescence spectrometry (PXRF)

Portable X-ray fluorescence analysis offers a combination of unique advantages that cannot be rivalled by any other analytical technique:

- in situ investigation
- multi-element analytical capability
- non-destructive nature
- immediate availability to the operator of information on the chemical composition of a sample
- immediate development on a judgemental sampling strategy.

Many analytical techniques may be considered "portable" in the sense that they can be operated in a mobile laboratory that is capable of being transported to a field site to provide an immediate laboratory analytical facility. However, the particular advantage of PXRF is the capability of the technique to undertake in situ analytical measurements where the PXRF analyser is taken to, and placed in direct contact with the sample. No sample preparation is involved, the only flexibility available to the operator being sample *selection*. An analytical measurement is undertaken and the result is immediately available to the operator. The operator may then decide what to analyse next, giving rise to the concept of an "interactive sampling and analysis" capability that few other techniques are capable of delivering. PXRF is, therefore, normally used as a surface analytical technique. However, careful interpretation of results may be necessary in circumstances where the objective is to measure the bulk composition of a sample as the operator may need to take into account surface alteration effects such as roughness and moisture.

PXRF systems use either a miniature X-ray tube or a radioactive sealed source to excite the sample with X-ray photons. These primary X-ray photons are capable of exciting secondary X-ray photons that are characteristic of the atoms present in the sample. The resultant X-ray spectrum is then recorded with a suitable detector such as silicon (Si-PIN) diodes, silicon drift, mercury (II) iodide (HgI_2) or a cadmium zinc telluride (CZT) device. The spectrometer usually incorporates a pre-calibration facility such that the instrument may be configured for a particular application such as:

- analysis of contaminated land
- workplace monitoring

- particulates on filters
- analysis of surfaces, coatings and paint
- metal and alloy sorting
- architectural building or monumental stone studies
- geochemical prospecting
- archaeological investigations
- studies on works of art and archaeological artefacts
- extra-terrestrial explorations.

In theory, the technique is capable of measuring almost all the elements in the periodic table. However, as PXRF measurements are normally undertaken in air, severe attenuation of the low energy fluorescence X-rays occurs so that elements below about silicon (depending on instrument design) cannot be effectively detected. Some instruments, however, offer a partial vacuum facility to report multi-element analysis from magnesium to uranium. Elements that can be detected with highest sensitivity are those measured from the K line series with absorption edges just below the energy of the emissions from the excitation source. These are normally elements up to about molybdenum in the periodic table, but depend on the excitation source selected. Higher atomic elements can normally be determined, but at a lower sensitivity as the L-series lines must be used for detection.

The mass of sample analysed depends on the energy of the characteristic fluorescent X-ray and its associated critical penetration depth within the sample. Critical penetration depths for the lower atomic number elements are in the μm range, and if higher atomic elements determined from the K-line, 1–2 mm (for Mo K in soil) or more is the relevant figure. The best estimate of analysed mass is, therefore, in the mg to g range.

Modern instruments are extremely compact and light in weight as illustrated below.

The apotheosis of such instrumentation are systems designed for extra-terrestrial measurements, such as the Beagle 2 PXRF, of mass 280 g, designed for measurements of rock and soil on the surface of Mars.

All instruments are designed and built to satisfy statutory, international X-ray safety legislation with appropriate interlocks and adequate radiation shielding. In addition, instruments fitted with radioactive sealed source(s) must comply with regulations covering the transportation of such devices (Table 1).

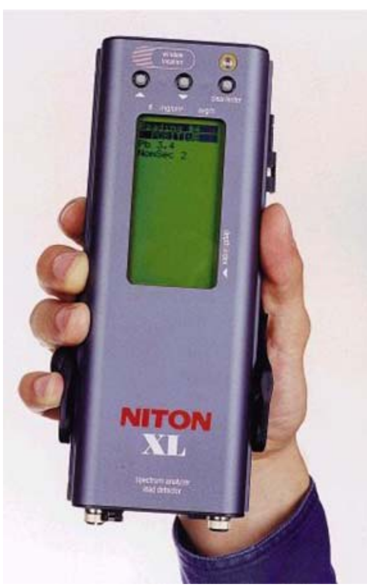

Table 1 Instrumental criteria sub-committee evaluation form

Type of Instrument: Portable X-ray fluorescence spectrometer						
Manufacturer:						
Model No:						
Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
A. Non Instrumental Criteria Selection of manufacturer	Laboratories in possession of other spectrometers 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 innovative features	I	The manufacturer should be alert to developments in design, excitation and detector technologies	PS WF ST		
(ii) Reliability record	Company's record for instrument reliability	I	Indicates history of sound design / manufacturing concepts	PS WF ST		
(iii) Similarity of operation, layout and design (including software) to existing instruments in the laboratory	For routine purposes, this may be important. However, this may be less important for research applications	I	Similarity of design and operation means that operators can draw on in-house expertise, resulting in reduced costs and time for training. It may also maximise the use of spares and fittings	PS WF ST		
(iv) Confidence in the supplier	Confidence gained from past personal experience	I	The benefits arising from good working relationship already in place	PS WF ST		
(b) Servicing	Score according to manufacturers' claims and past record, judged by the sub-features (i) to (iii) below					
(i) Service support	Availability of a suitable service support from the supplier, agent or third party contractor	VI	Essential to ensure reliable operation over the planned working life of the instrument. Often available as trouble-shooting self-help documentation and off-site repair	PS WF ST		
(ii) Availability, cost and delivery of spares	Range of stock carried by, or quickly available from the manufacturer / agent / contractor	VI	Rapid delivery of spares reduces down time	PS WF ST		
(iii) Effectiveness of service support	The ability of the supplier to identify and repair faults as judged from previous experience and reports of others	I	A rapid response reduces down time. Note that the guaranteed response may depend on the proximity of the service centre for off-site repairs and that travel/transport costs may be a factor	PS WF ST		
(c) Technical support	Score according to manufacturers' claims and past record, judged by the sub-features (i) to (vi) below					
(i) Applications department	The advice and training available from the manufacturer's applications department	I	This helps in-house staff to optimise measurements for new applications	PS WF ST		
(ii) Technical literature	The range and quality of technical literature including the operating manual	I	The availability of quality technical literature helps operators optimise the use of the instrument. Note that on many modern instruments, operating instructions are available online as part of the instrument operating software	PS WF ST		
(iii) Telephone and internet assistance	Willingness of the manufacturer, supplier or contractor to give effective advice over the telephone or internet. This can normally be evaluated by reference to existing users	I	Rapidly available technical help reduces errors and enhances productivity	PS WF ST		
(iv) Training	This includes initial training when setting up the instrumentation and subsequent support	VI	A comprehensive training scheme will ensure that operators and instrumentation are working effectively	PS WF ST		
(v) User Group Meetings	Meetings and technical briefings organised for users of instrumentation by the manufacturer or third party	I	Other users are often the best source of advice on problems, solutions and applications	PS WF ST		

Table 1 continued

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(vi) Risk assessment	Score according to the support available from manufacturers in the development of appropriate risk assessments	I	Because of the mode of operation and the intimate interaction between instrument and operator, special consideration is required for the development of risk assessment to comply with legislation designed to minimise the risk of accidental exposure to ionising radiation	PS WF ST		
B. Instrumental Criteria	The specific tests recommended in this report should be discussed with the instrument manufacturer in advance of any evaluation to ensure that any testing is undertaken in a practical and effective manner, taking account of effective health and safety procedures especially with respect to ionising radiation					
1. *General Features Facilities required for:*						
(a) Size and portability of equipment	Score according to weight and practical portability for envisaged use off-site	I /VI	The instrument and any accessories may need to be carried and used in hand held mode at field sites for extended periods of time. The comfort and effective use by the operator will be influenced by the weight of the instrument and various ergonometric design features that promote hand held operation	PS WF ST		
(b) Suitability for field use	Score according to design features that ensure effective field use, including ruggedness (as evidenced by drop test data), water proofing and dust proofing, and effective operation at the range of temperatures that will be encountered at a field site, taking account of the envisaged use	I /VI	PXRF instrumentation may be used in some demanding environments including outdoors (including in wet weather) and at various types of industrial sites. Features will then need to be designed into instrumentation to ensure the effective and enduring operation of the instrumentation	PS WF ST		
(c) Shape and size of analytical window and contact surface	Score according to the shape and size of the analysis window and analytical plane of the instrument which is placed in contact with the sample surface to be analysed	I	Instruments that are to be used to analyse surfaces where there is restricted access (for example welds) will benefit from a narrow 'snout' to facilitate contact when there is restricted access. Instruments for use in analysing large flat surfaces will benefit from a larger window and flat surface that can easily be registered in contact with the sample surface. An appropriate compromise will need to be made for general purpose instruments	PS WF ST		
(d) Self supporting field and laboratory operation	Score for features that allow the instrument to be operated in the field in a self supported mode and accessories that allow the instrument to analyse samples safely and effectively in the laboratory if these modes of operation are important to the intended application		In some applications, for example in the analysis of soil, it is convenient to place the instrument on the sample and operate it remotely, rather than the operator needing to hold the instrument with the analysis trigger being permanently depressed for the duration of the analysis. In this mode of operation, the instrument must be designed to be self supporting (some instruments will fall over!). For the most versatile use, it is often convenient to be able to analyse samples in the laboratory with the instrument supported in an appropriate stand and the analysis window enclosed in a suitable lid or enclosure. In both modes of operation, consideration will need to be made for the appropriate use of radiation interlocks. Scoring is important if these modes of operation are important in the intended application			

Table 1 continued

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(e) Accessory to analyse low atomic number elements	If relevant to the application, score according to the availability of accessory to promote the detection and analysis of low atomic number elements	I	Laboratory XRF is routinely used to determine elements down to Na in the periodic table, however this can only be achieved by minimising the severe attenuation of the corresponding low energy characteristic X-rays in air by evacuating the sample chamber. This approach is obviously not possible for portable *in situ* field measurements. However, devices are available to reduce the air path by evacuating the source/detector part of the instrument using a small vacuum pump or by providing a helium flush facility to facilitate the detection of low atomic number elements			
(f) Connectivity	Score according to convenient and effective means of connecting the PXRF to laptop or desktop computers to allow analytical results recorded in the field to be downloaded for further manipulation	VI	Analytical data stored in the instrument during field use can be downloaded to a PC via a USB cable or a wireless device such as Bluetooth	PS WF ST		
(g) Battery	Score for battery life, low temperature tolerance (if relevant) and ease of change in the field without loss of stored results or operating conditions	VI	Most instruments use Li-ion batteries that are easily changed in the field to ensure extended operation. The lowest operating temperature of the instrument is normally set by battery tolerance and may be relevant if the instrument is to be used in extreme environments	PS WF ST		
(h) Safety keys and interlocks	Safety features, which include operating keys, warning lights, appropriate safety switches, interlocks and proximity detectors, are devices to prevent accidental exposure to ionising radiations during operation or maintenance of the instrument. The provision and operation of safety interlocks must satisfy current national and international regulations and be maintained in full working order	VI	Failure to meet these standards precludes legal operation of instrumentation. Operation of any X-ray tube must be inhibited unless mounted in the instrument with all shielding in place. In some applications, for example where the instrument is to be used to analyse delicate, fragile or valuable sample surfaces, proximity detectors that make physical contact with the sample surface may be unacceptable	PS WF ST		
(i) Provision of GPS	Score according to the availability of a facility to tag analytical results with global positioning system data, if appropriate to the application	I	In some applications there is considerable benefit in tagging analytical results with GPS locational data to allow spatial distributions to be readily reconstructed. This facility can be provided by incorporation of an on-board GPS provision, or connectivity to an external GPS instrument	PS WF ST		
2. *Choice of category of instrument*	Portable X-ray fluorescence instruments are available with a number of different excitation sources and application-specific software. These design characteristics are summarised in the following entries and should be scored in relation to the advantages they offer in the intended application of the instrument as judged from the details given below	VI	The different excitation sources and detection systems offer different analytical characteristics that can give analytical advantages in a particular application	PS WF ST		
(a) X-ray tube excitation	The development of miniaturisation technology has led to the introduction of miniature X-ray tubes with performance and excitation characteristics that are largely analogous to higher powered tubes used in conventional XRF instrumentation.	VI	Low powered tubes typically operate at 35 to 40 kV and 50 μA providing 8 h use before the instrument's Li-ion battery needs recharging. This excitation source offers the principal advantage of providing a 'bright' emission characteristics and	PS WF ST		

Table 1 continued

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
			does not require a licence under radioactive substances legislation for sealed sources thus avoiding restrictions on transport and use across national boundaries.			
(b) Radioisotope excitation	The sample is excited using an appropriate sealed radioactive source such as ^{55}Fe, ^{244}Cm, ^{109}Cd and/or ^{241}Am.	VI	Radioactive source excitation is an effective way of meeting criteria for minimising the power consumption of PXRF instrumentation and facilitates the development of compact instrumentation optimised for a specific application. More than one source (with the complication of an exchange mechanism) may be required for multi-element applications. Effective shielding must be provided even when the instrument is not in use and ownership of the instrument requires a licence under radioactive substances legislation that places restrictions on the use, storage, custody and transport of the instrument within national boundaries.	PS WF ST		
Particular features relevant to each of these categories of instrumentation will now be considered as follows: 3. *X-ray tube excitation* (a) X-ray tube	Miniature low-power X-ray tubes provide a potential flexible excitation source. Score according to the availability of a tube with an anode appropriate for the intended application		An X-ray source provides a bright source of excitation. Since the range of elements that can be excited with optimum sensitivity is largely dictated by the tube anode it is important that the appropriate anode is available. (Au offers improved sensitivity for toxic elements such as Cd, whereas a Ag anode may be preferred for "lighter" analytes)			
(b) Ease of replacement	Score according to the ease of the tube exchange procedure. Safety considerations may restrict this activity to trained service personnel, in which case, speed, convenience and cost will be the features to be scored	I	Occasional replacement may be necessary following tube failure. It is important that these operations can be undertaken simply. Changing tubes should only be undertaken by a fully qualified operator	PS WF ST		
(c) Power settings	Score if user optimisation of tube kV and mA are important in the planned activity	I	In simple applications, the X-ray tube may be operated under fixed conditions of applied kV and mA and scoring may then be inappropriate. However, X-ray tubes can be optimised for the excitation of different ranges of elements by adjusting the tube kV (and mA)	PS WF ST		
(d) Primary beam filters	Score if optimisation of excitation conditions by use of a primary beam filter is important in the intended application. In the most demanding applications, primary beam filters may offer advantage	I	Primary beam filters are thin metallic (or polymer) foils placed between tube and sample to modify the spectrum available to excite the sample. Filters can be used to improve detection limits by removing a proportion of the tube continuum that would otherwise be scatteredoff the sample into the detector, degrading the signal to background ratio of elements of interest	PS WF ST		

Table 1 continued

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
4. *Radioisotope excitation*						
(a) Choice of isotope	The selected radioisotope source(s) must be capable of effectively exciting the range of elements relevant to the application. However, as appropriate sources are likely to be available from all manufacturers, it is inappropriate to score this feature. If provision is made in the instrument for installing multiple sources with automatic source exchange to extend the range of elements that may be determined and this facility is relevant to the application, score accordingly		Individual radioisotope sources are only capable of exciting effectively a restricted range of elements and are, therefore, normally less versatile excitation sources than an X-ray tube. Choice of radioisotope source must be made according to application, normally from the following list (characteristic X-ray emissions in parenthesis): ^{55}Fe (Mn K lines), ^{244}Cm (Pu L lines), ^{109}Cd (Ag K lines), ^{241}Am (59.5 keV gamma emission lines and Np L X-rays). Note that it is not possible to replicate the excitation characteristics of a ^{241}Am source using a miniature X-ray tube, giving that source a potential advantage in the excitation of the K-lines of higher atomic number elements			
(b) Activity of sources	The activity of a radioactive source is specified in megabecquerels (MBq). If the source has sufficient activity to excite sufficient sensitivity for the range of elements relevant to the application, it is inappropriate to score this feature		The intensity of X-ray emission from a radioactive source depends on the decay characteristics of the selected isotope, but is directly proportional to source activity. A compromise must be achieved between adequate excitation capability, cost and safety associated with the need to provide adequate shielding to restrict radiation leakage and consequent operator exposure			
(c) Useful working life	The effective working life of a source depends on its initial activity and intrinsic half-life. It is inappropriate to score this feature		The half-life of commonly used sources vary as follows: ^{55}Fe (2.7 years), ^{244}Cm (18 years), ^{109}Cd (1.3 years), ^{241}Am (458 years). Users should be aware that instrument performance (in terms of excitation efficiency) will decline with age of source. When quantifying analytical results, source decay is normally accounted for by a simple mathematical correction based on the source half life to compensate for radioactive decay			
(d) Cost of replacing sources	Score maximum for the most cost effective service offered by the manufacturer. Score additionally for a disposal service offered by the manufacturer following source replacement	I	This operation should be entrusted to the manufacturer and may contribute to the longer term running costs of instrumentation. If the manufacturer does not dispose of the source, the user will have to pay a specialist contractor to undertake this task	PS WF ST		
(e) Safety considerations	In addition to the provision of safety interlocks for any PXRF instrument, sealed sources must be retractable when the instrument is not in use. As these safety features are a legal requirement, it is inappropriate to score this feature	VI	Any exposure to ionising radiations represents a potential hazard to health. The safety feature descibed here must be fail-safe in design to accommodate the unexpected failure of the power supply			
5. *Choice of detectors*	The following is provided as additional information., In practice a PXRF instrument will be configured by the manufacturer for a specific application. Several categories of energy dispersive detector are available. Scoring may be inappropriate unless particular characteristics of the detector type are relevant to the application. Specific features of detectors are referred to in detail in the ED-XRF report see Accred. Qual. Assur 2006, 11,610-624	depends on application	Si-PIN diodes, Si-drift, HgI_2 detectors and CZT devices all possess some distinctive characteristics that may offer advantages in particular applications as summarised in the next paragraphs			

Table 1 continued

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(a) Si(PIN) detector	A detector comprising high purity silicon wafer of typical thickness 300 μm and active area 7 to 25 mm^2		Si(PIN) detectors are compact and offer good performance characteristics, without the need for cryogenic cooling (a small degree of Peltier cooling is sufficient). Best resolutions are in the order of 180 eV full width of peak at half maximum (FWHM) at 5.9 keV with detector effective energy range of 1 to 20 keV			
(b) Silicon drift detector	A particular design of detector in which the capacitance is minimised by the overall detector design and by the integration of the first stage signal amplification onto the detector wafer. This detector is normally designed on a wafer 300 μm thick		Silicon drift detectors offer significant advance in performance, in particular overcoming the principal maximum count rate restrictions of a Si(PIN) device However, these detectors are significantly more expensive, requiring Peltier cooling,. This type can offer resolutions of better than 140 eV FWHM at 5.9 keV and because of its low inherent capacitance, will operate successfully at input count rates in excess of 10^5 s^{-1}			
(c) Mercuric iodide detector	An energy dispersive X-ray detector based on a high purity mercury (II) iodide semiconductor crystal		Mercuric iodide detectors represent an evolving technology in which further improvements are likely to occur. The principal property of these devices is that they offer a reasonably good resolution response (about 250 eV at 5.9 keV) with a small degree of Peltier cooling. Furthermore, detection efficiency extends to significantly higher energies than silicon devices allowing their use in measuring the higher energy fluorescence spectrum. Escape peaks from Hg (L lines) and I (K lines) may cause spectral overlap interferences in some applications			
(d) CZT	Cadmium-zinc-telluride represents one of a number of semiconductor materials that are being investigated for their X-ray detection properties		New detector materials will need to show clear advantages in detection characteristics, longevity and/or robustness compared with the detector types described in the above sections before they are likely to find application in commercial instrumentation	PS WF ST		
6. *Quantification and analysis software*						
(a) Quantitative analysis	Score for the provision of applications packages that will allow the user to analyse all the elements of interest in the matrices of interest. Evaluate this feature by the analysis of a series of test samples having the relevant matrix and of know composition that covers the complete composition range of interest. Evaluate the quality of data (in particular bias and precision) in relation to fitness for purpose criteria for the applications of interest	VI	PXRF instruments often use a fundamental parameter-based matrix correction algorithm which must often be optimised for the matrix type of interest. Whereas it is usual to be able to pre-programme instruments with several matrix correction applications, the accuracy of analytical data is likely to be degraded if the application is not optimised for the sample matrices of interest	PS WF ST		
	Score as appropriate to the application, of firmware features such as: (1) Calibration using various data fitting modes for linear and non-linear functions. (2) Matrix correction for absorption and enhancement effects by empirical and/or fundamental parameter procedures. (3) Options to store results for further processing and		Calibration functions and matrix corrections must be applied to raw intensity data to obtain quantitative analysis results. In many applications, results must be supplied to the user in a standardised report format. To satisfy quality control criteria,			

Table 1 continued

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
	comparison as well as editing and outputting data in a report format. (4) The facility to transfer files of results in the appropriate computer format to an external device. (5) Statistical process control software for quality control applications		statistical treatment of sets of data is essential before results can be released to the user for interpretation			
(b) Semi-quantitative analysis	If relevant to the application, score for the capability of rapid visual appraisal of the estimated composition of a sample. Score additionally for display of precision data that allows an analysis to be terminated once adequate confidence has been achieved in sample identification. Score for the provision of a firmware package capable of estimating concentration levels from measured intensity data without recourse to calibration using a full elemental set of standard materials	VI	Results that are only displayed at the completion of a preset count time may not allow instrument use to be optimised when the aim is to qualitatively identify the material. Some operating systems allow the current estimate of the composition of a sample to be displayed in real time during the course of a measurement. In applications where limited numbers of calibration standards are available, the user's requirements may be satisfied by a semi-quantitative analysis in which intensity data are converted to concentrations using a theoretical correction based on fundamental equations of excitation and absorption	PS WF ST		
7. *Instrument control, data acquisition and measurement*						
(a) Operating program	Score maximum for a user-friendly interface, that allows the user to develop versatile analytical programmes tailored to the application	VI	Difficulty of use can lead to operator frustration and errors. The use of a graphical user interface (e.g., pull-down menu options) and/or soft keys or other devices for reducing setting up times and initiating analytical programmes reduces training requirements.	PS WF ST		
(b) Instrument status	Score maximum for an adequate display of instrument status parameters and alarm functions that monitor whether the instrument is operating within its design envelope for the selected analytical programme	VI	A display of instrument parameters will confirm to the operator that the required analytical programme is being followed. Effective monitoring of instrument status may give early warning of malfunction.	PS WF ST		
(c) Instrument performance diagnostics	Score for an instrument with the most comprehensive set of self-checks on power up and easy to use qualification and validation routines	I	It is vital that the system performs 'fail safe' diagnostic checks on power up; this information must be recorded. Essential in many laboratories for regulatory requirements, e.g., GLP, GMP, MCERTS and all regulated industries	PS WF ST		
(d) Data collection and application software	Define the requirements before scoring these items					
(i) Specialised options on purchase	Score, where appropriate for the application, for the provision of firmware enabling the instrument to be used for a specialised task such as: (1) Alloy/metal sorting. (2) Compliance screening for restriction of the use of certain hazardous substances in electrical and electronic equipment (RoHS). (3) Thickness measurements of multilayer samples. (4) Analysis of dust. (5) Other finger printing applications. It is important that such software packages are verified using a range of suitable test samples	VI (depending on application)	The provision of appropriate software enhances operator training when specialised applications are envisaged	PS WF ST		
(ii) Display software routines	Score maximum for the most versatile set of spectral display routines	I	Simple versatile spectral display routines are useful	PS WF ST		

Table 1 continued

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score		
(iii) Intuitive and flexible firmware	Score for firmware that is user friendly, versatile and flexible. Score highly for a system that offers help functions, error messages, complete libraries of relative intensity, interferences and corrections. Score additionally for databases of matrices, methods, results and QC standards	VI	Versatile and user friendly application software varies considerably between manufacturers. It is often useful to communicate with existing users when evaluating. Software that enables a choice of matrix correction procedures and reanalysis of acquired data under amended matrix or calibration parameters provides the user with valuable flexibility. In addition, quality control procedures such as re-analysis options based on periodic QC standards, sample out of calibration range protocols and calibration failure protocols e.g. re-calibrate, ignore erroneous standards, etc., facilitate the validation of the method Ideally, the software should be intuitive, and a comprehensive help function is highly desirable. Meaningful error messages rather than merely a non-specific error number are also a huge advantage	PS WF ST		
(iv) Custom reporting	Score for a flexibility of reporting format. Score additionally when reports may be generated from stored data	I	The possibility of different reporting formats, e.g., full statistics with replicates and methods, full statistics alone or only a summary report gives the analyst flexibility. The capacity to generate a report from stored data extends the flexibility further	PS WF ST		
(e) Data storage	Data may be stored on various media and increasingly via networks. Score for compatibility with existing data processing facilities and requirements	VI	The vast majority of modern instruments have data storage options that enable further data processing to be easily performed. ASCII data can be exported to and manipulated by many external software packages. The nature of the data output may need to be compatible with LIMS systems, if in use. This expands the scope of data manipulation and chemometric routines available to the user and provides security and backup	PS WF ST		
8. *Safety considerations* (a) Regulatory compliance	In the UK, design and construction of instrumentation and all safety features, including interlocks, must comply with the appropriate legislation. Other countries have similar national regulations with which instrument design and construction must comply. Reject any instrument that does not comply with any of these requirements		Apart from the obvious hazard, operation of instrumentation in contravention of statutory regulations is illegal	PS WF ST		
(b) Shielding of ionising radiations	In the UK, to comply with the Ionising Radiations Regulations 2000, leakage of ionising radiation from an instrument must fall below a specified maximum value. Score for shielding and additional features that minimise leakage of extraneous radiation below the regulatory limits		Any exposure to ionising radiation is potentially hazardous. Instruments must be designed to ensure that any external leakage is as low as reasonably practicable	PS WF ST		
9. *Value for money (points per currency unit)*	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		'Simple' instruments are often good value for money, whereas those with unnecessary refinements are often more costly	**Sum of sub-totals** **Grand Total**		

Other reports

The Analytical Methods Committee has published the following reports in the series:

Part I Atomic absorption spectrophotometers, primarily for use with flames (1984) Anal Proc 21:45. Revised in (1998) Analyst 123:1407

Part II Atomic absorption spectrometers, primarily for use with electrothermal atomizers (1985) Anal Proc 22:128. Revised in (1998) Analyst 123:1415

Part III Polychromators for use in emission spectrometry with ICP sources (1986) Anal Proc 23:109

Part IV Monochromators for use in emission spectrometry with ICP sources (1987) Anal Proc 24:3

Part V Inductively coupled plasma sources for use in emission spectrometry (1987) Anal Proc 24:266

Part VI Wavelength dispersive X-ray spectrometers (1990) Anal Proc 27:324

Part VII Simultaneous wavelength dispersive X-ray spectrometers (1991) Anal Proc 28:312

Part VIII Instrumentation for gas–liquid chromatography (1993) Anal Proc 30:296

Part IX Instrumentation for high-performance liquid chromatography (1997) Analyst 122:387

Part X Instrumentation for inductively coupled plasma mass spectrometry (1997) Analyst 122:393

Part XI Instrumentation for molecular fluorescence spectrometry (1998) Analyst 123:1649

Part XII Instrumentation for capillary electrophoresis (2000) Analyst 125:361

Part XIII Instrumentation for UV–VIS–NIR spectrometry (2000) Analyst 125:367

Part XIV Instrumentation for Fourier transform infrared spectrometry (2000) Analyst 125:375

Part XV Instrumentation for gas chromatography-ion trap mass spectrometry (2001) Analyst 126:953

Part XVI Evaluation of general user NMR spectrometers (2006) Accred Qual Assur 11:130–137

Part XVII Instrumentation for inductively coupled emission spectrometers. (2005) Accred Qual Assur 10:155–159

Part XVIII Instrumentation for differential scanning calorimetry (2005) Accred Qual Assur 10:160–163

Part XIX CHNS elemental analysers (2006) Accred Qual Assur 11:569–576

Part XX Instrumentation for energy dispersive X-ray fluorescence spectrometry (2006) Accred Qual Assur 11:610–624

Part XXI NIR Instrumentation for process control (2006) Accred Qual Assur 11:236–237

Part XXII Instrumentation for liquid chromatography/mass spectrometry (2007) Accred Qual Assur 12:3–11

Part XXIII Instrumentation for portable X-ray fluorescence spectrometry (2008) Accred Qual Assur. doi:10.1007/s00769-008-0358-7