GENERAL PAPER

Evaluation of analytical instrumentation. Part XXIII0ihce0 -1 efeatures of anal

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 m 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.

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

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. I[illegible]ta C[illegible]a	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. *G[illegible]a F[illegible]a[illegible] Fac[illegible]* [illegible]:						
(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	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 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) [illegible] 1i9.2(andards)]T8.6757 [illegible] TD0 [illegible] 1(ring)]T0 -1.1481 TD-0.ndards)]T8.675i( )-9mex)]TTju)-10hangen of the
xtitSpl rplacrw[at op-9.1(i)-9(an9on--9.1(ring)]T0 -1.1501 Tot)-9(n x)]TTjp[(irat op-9, w[m)8e envi)9(with the)]T0 -1.5oDn(deiv)-9.2(m)9(it)ctfmee1(hts, TD[n of)-9(the) 5proxi)-TD0 Tw[instru)-9.19(nmen

Table 1 continued

		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]	

Table 1 continued

Feature	Definition and/or test procedures and guidance	Importance	Reason	Score			
4. *Ra c tat*							
(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 ga Tmo6.48j-0.(5 375.3611 59o(nuu L)TJu Tm-0s a-1.rp (X9(m)-9.1(an X-ra)-1ine				

X4iaiadiogl

Table 1 continued

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

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	PS WF ST		

Table 1 continued

Feature	Definition and/or test procedures and guidance	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,	PS WF ST		

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