Report by the Analytical Methods Committee

Evaluation of Analytical Instrumentation. Part VI Wavelength Dispersive X-ray Spectrometers

Analytical Methods Committee
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A method is provided for comparing the features of wavelength dispersive X-ray spectrometers.

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), Professor E. Bishop, Dr. P. Potts, Mr. D. Squirrell and Mr. P. Warren, with Mr. C. A. Watson as Honorary Secretary.

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 possible then to score these features in a rational manner, which allows a scientific comparison to be made between instruments.

The over-all object is to assist purchasers in obtaining the best instrument for their analytical requirements. It is also hoped that, to a degree, it will help manufacturers to supply the instrument best suited to their customers' needs.

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 manufacturers.

The sixth report of the Sub-Committee deals with X-ray fluorescence spectrometers that are designed for use as sequential instruments.

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 applications.

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 of features will be by proportion, *e.g.*, 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 will be indicated by the abbreviations VI (very important), I (important) and NVI (not very important). A scale is chosen for the weighting factor which allows the user to discriminate according to needs, *e.g.*, ×1 to ×3, or ×1 to ×10. The factor could amount to total exclusion of the instrument.
3. (ST) Sub-total. This is obtained by multiplying PS by WF.

INSTRUMENTAL CRITERIA SUB-COMMITTEE INSTRUMENT EVALUATION FORM

Type of Instrument: Wavelength dispersive X-ray spectrometer

Manufacturer:								
Model No:								
Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
1. *Excitation* (*a*) Generator (*i*) Conventional	These are based on vacuum tube technology, use relatively large and heavy transformers and require water cooling.							
(*ii*) Solid State	These are based on solid-state circuits, have higher frequency oscillators and, as they use smaller transformers, are more compact. Both types are satisfactory and it may be inappropriate to score this item.		This item is only significant if physical size or weight and/or cooling water capacity limits application.					
(*iii*) Stability	Score maximum for the best available.	VI	The output in current and voltage is subject both to long term drift and short term fluctuations with mains voltage. Either source of instability will affect the frequency of re-calibration and the over-all performance of the instrument.	PS WF ST				
(*b*) X-ray tubes (*i*) Type	These may have a side or end window with a range of metal anodes to permit optimisation of excitation conditions. Both types are satisfactory but it may still be appropriate to score this feature, depending on application and range of elements required.		3 kW-side-window tubes can be operated at up to 100 kV and the design permits close coupling to the sample and hence high excitation efficiency. This is partly counteracted by the need for a relatively thick beryllium exit window to accommodate the heating effect of backscattered electrons from the anode which is held at ground potential. Dual anode tubes are available to compensate for poorer excitation efficiency for the lightest elements resulting from attenuation of the low energy continuum by the relatively thick windows required. Normal drinking quality water can be used for cooling. With 3 kW end-window tubes the maximum operational voltage is at present limited to 75 kV. However, as the anode is maintained at a high positive potential, electron backscatter is inhibited and the consequential window heating effect is reduced. A thinner window can thus be used, increasing the efficiency of excitation for low atomic number elements. The cooling system requires de-ionised water or oil for the primary cooling circuit coupled to a heat exchanger that requires a normal water supply for heat removal.	PS WF ST				

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*ii*) Ease of tube inter-change/ replace-ment	Score maximum for convenient tube change procedure.		This may be important in a non-routine environment, but suitable selection of anode material and filters may render this operation less common than in the past. If a very wide range of analytes are being examined, more than one tube may be required. Tubes also have a limited life and require replacement. Therefore, it is important that this task should not be excessively time consuming or difficult.	PS WF ST				
(*iii*) Cooling and water supplies	Score maximum for minimum flow-rate and widest tolerance to temperature, supply pressure, and dissolved salts, without impairing cooling efficiency.	I	All tubes and some generators require cooling water. A tight specification for water hardness, temperature and flow will increase installation and running costs.	PS WF ST				
(*iv*) Maximum power	Score maximum for the highest power available for a tube with the anode of interest.	I	In general, higher power will increase sensitivity or reduce analysis times.	PS WF ST				
(*v*) Maximum voltage	Score maximum for the highest voltage available for a tube with the anode of interest.	I	For certain elements, *e.g.*, z = 42–58 (Mo–Ce), for some applications, there may be an advantage in using K lines which are more efficiently excited by tubes operated at higher voltages.	PS WF ST				
(*c*) Filters	Score highest for the maximum number of available filters, which are a selection of metal foils placed in the primary X-ray beam to remove unwanted lines.	I	Filters are used to remove from the primary X-ray beam exciting the sample the characteristic lines from the X-ray tube anode which would cause spectrum overlap interferences. Improvements in signal to background ratio may also be achieved by removal of continuum radiation.	PS WF ST				
(*d*) Safety inter-locks								
(*i*) Operator protection	Safety interlocks are devices to prevent injury from ionising radiation or hazardous voltages during stand-by, operation, or maintenance of the instrument and must satisfy current international regulations and be guaranteed by the manufacturers. Failure to meet national safety standards would preclude the legal operation of the instrument.	VI	Safety devices will include warning lights, appropriate safety switches and interlocks to prevent access to any potential source or area of radiation whilst the X-ray tube is energised and also access to high voltage cable connections or mains voltage cables while electrically energised or operation of the tube unless it is secured in its normal position within the instrument.	PS WF ST				
(*ii*) Instrument protection	The flow of cooling water must be monitored and facilities provided for automatic safe shut-down in the event of supply failure. Score additionally if system also monitors water temperature and, where applicable, conductivity of deionised water supply.	VI	Efficient cooling is essential to prevent tube deterioration and/ or burn-out. A rise in water temperature will give early warning of partial blockage in supply lines or malfunction of the heat exchanger in a circulatory system. A rise in conductivity indicates loss of efficiency of the deioniser (if fitted), which, if allowed to continue, could lead to insulation breakdown by the high voltages present in the end window tube.	PS WF ST				

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
2. *Sample changer and presentation*								
(*a*) Number of samples								
(*i*) Internal	Score maximum for the maximum number of samples which can be accommodated.	I	If more than two positions are available, some position(s) can be used for intensity (or re-calibration) monitors, thus reducing any possibility of monitor contamination.	PS WF ST				
(*ii*) External sample changer	Score maximum for the sample changer which can comfortably accommodate the maximum number of samples that are likely to be encountered in an analytical programme. Score extra if sample changer can be retrofitted.	NVI	An external auto-sampler is often needed to give flexible efficient operation. This may be very expensive if not easy to retrofit.	PS WF ST				
(*iii*) Sample identification	Score maximum for a system of automatic identification which enables the sample to be positively identified and the analytical sequence to be defined to maximise operator convenience.	VI	Essential if instrument is being used for routine analysis, particularly if unattended operation is envisaged.	PS WF ST				
(*b*) Vacuum system								
(*i*) Reproducibility	Providing a level of less than 1 torr can be reached, scoring is inappropriate.	VI	Adequate vacuum within the spectrometer is essential to prevent attenuation of the fluorescence signal if the determination of light elements is envisaged ($z < 20$, Ca).					
(*ii*) Pre-analysis pump down	Score maximum for minimum pump down time, if intermediate evacuation compartment is not available between the external sample changer and the analytical chamber.	I	In the absence of an evacuation compartment, pump down time must be added to sample counting time when calculating total analysis time. This may be further extended if delays are encountered in achieving operating vacuum because of samples outgassing. Such delay is unlikely to be a problem with an automated system with an intermediate pump down compartment.	PS WF ST				
(*iii*) Helium path option	Score maximum for the facility to interchange an atmospheric pressure of helium within the spectrometer as an alternative to vacuum. Score additionally for economical use of helium.	Depends on sample type	This facility is essential if light elements are to be determined in volatile liquid samples and/or finely divided loose powders. Spectrometer sample chambers cannot then be evacuated without disrupting the sample, which may then contaminate and/or cause damage to the instrument.	PS WF ST				
(*c*) Reference sample	Score maximum for the facility to maintain a reference sample or intensity drift monitor within the spectrometer.	I	To obtain the highest precision, allowance must be made for the drift which occurs during instrumental operation. The precision of the analytical results can be improved by ensuring that such drift is detected and appropriate corrections made to the data.	PS WF ST				

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*d*) Positioning and alignment of sample	Score maximum for the best mechanical precision obtained when presenting a sample in each position of the carousel and in each specimen holder.	VI	Discrepancies in the mechanical alignment will affect precision of measurements, particularly as de-focusing can occur when a fine collimator is used. Any displacement (height, angular or lateral) of the sample will affect both excitation and counting efficiency, causing alterations to the measured count rate.	PS WF ST				
(*e*) Sample spinning	Score maximum for the ability to rotate the sample continuously throughout the analysis period.	VI	Averaging the excitation induced at the sample surface will compensate for effects caused by minor sample inhomogeneity and surface defects.	PS WF ST				
(*f*) Sample area irradiated and masking								
(*i*) Sample holder	Score maximum for the maximum proportion of the sample area that can be excited.	I	Maximising the area on the sample from which fluorescence is detected will reduce the effects of sample inhomogeneity and enhance the count rates.	PS WF ST				
(*ii*) Materials and design of sample holders	Score maximum for the availability of sample holders in the widest range of materials and designs.	Varies according to sample types to be analysed. Important for some sample types.	The sample holders may yield an unwanted fluorescent signal, particularly if liquid or thin film samples are analysed. Selection of holders from a range of available materials will enable the user to avoid such interferences. A range of different sample holder designs are also required if non-standard or small samples are to be analysed.	PS WF ST				
(*iii*) Masking	Score maximum for the availability of suitable masks or apertures to match each sample size which may be used.	I	The use of suitable masks will minimise the detection of scattered radiation and unwanted fluorescence from the sample holder. Masking is particularly important when analysing small samples in order to avoid excitation of the sample holder.	PS WF ST				
3. *Spectrometer*								
(*a*) Primary collimator	Parallel blade collimator (Soller slits) is provided to restrict the angular dispersion of fluorescent radiation reaching the analyser crystal. Score maximum for the maximum choice of blade spacings that can be installed within the instrument consistent with ease of change over.	VI	Finer collimators improve spectrometer resolution, minimising line overlaps, but at the expense of count rate. A choice is, therefore, advantageous so that an acceptable compromise between resolution and count rate can be selected as appropriate to the application.	PS WF ST				
(*b*) Goniometer								
(*i*) Angular reproducibility	Score maximum for the system that can achieve the highest precision on re-setting to a particular wavelength from either direction.	VI	Precise intensity measurements on a series of samples can only be achieved if the spectrometer re-sets to precisely the selected wavelength. Correction for spectrum overlap interferences and operation under high resolution conditions place considerable demands on the angular reproducibility of the instrument. It is advantageous to locate the measurement angle from either direction with negligible backlash.	PS WF ST				

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*ii*) Angular movement	Score maximum for the maximum angular range (2θ) in combination with high slew speed.	I	Wide angular movement extends the range of lines that can be measured with a given diffraction crystal whilst a rapid slew speed reduces analytical measurement time.	PS WF ST				
(*iii*) Qualitative scanning	Score maximum for the highest scan rate together with a wide range of speeds. Score extra for the provision of a continuous scan mode in addition to the standard step scan operation.	I	High scan speeds reduce analytical time for rapid identification of major elements. A range of speeds is required to enable searches for trace elements to be made under high resolution conditions. A continuous scan mode enables a wide range of elements to be scanned in the minimum time.	PS WF ST				
(*c*) Analysing crystals	Score maximum for the widest range of crystals available from the manufacturer and the maximum number that can be fitted to a given instrument.	VI	A number of crystals are essential to provide acceptable resolution and intensities over the complete spectral range. The crystals required to be mounted within the instrument may vary with the elements of interest and the application envisaged by the analyst.	PS WF ST				
(*d*) Detectors	Score maximum for a detector system which effectively covers the whole wavelength range. This system may comprise a flow proportional counter and a scintillation counter which can be mounted either separately or in tandem. Where the detectors are mounted separately a sealed proportional counter may also be fitted. Score accordingly.	VI	The ideal detector system would consist of a scintillation counter for heavy elements, a sealed proportional counter for 1st row transition elements and a flow proportional counter for light elements. On some instruments the addition of the sealed proportional counter is avoided by the ability to use the scintillation and flow counters in tandem. In such instances an auxiliary collimator should be used between detectors. This option allows better resolution for short wavelength lines of many elements but with some loss in sensitivity.	PS WF ST				
(*i*) Flow proportional counter	Score maximum for counter giving highest count rate and best resolution for elements z = 9 to 20 (F to Ca) using a coarse collimator and thinnest entrance window. The system should be operable with the spectrometer under either vacuum or helium flow conditions. Score additionally for a choice of window thicknesses and ease of replacement, together with availability of efficient temperature/pressure/flow control of counter gas.	VI	Thin polymer film windows are necessary to reduce absorption of long wavelength radiation and give high count rates for the light elements. In the event of window failure, ease of replacement is important. Good resolution is necessary to assist the discrimination of analyte lines from background and neighbouring radiation. A high count rate improves sensitivity and precision. Effective gas flow pressure control is required to compensate for changes in the pulse height with atmospheric pressure.	PS WF ST				
(*ii*) Scintillation counter	Score maximum for highest count-rate and resolution from Kα z = 26 (Fe) to Kα z = 56 (Ba).	VI	Scintillation counters should operate efficiently over the wavelength range Fe Kα to Ba Kα. High count rates contribute towards improved precision and/or allow the spectrometer to be operated under high resolution conditions (fine collimator).	PS WF ST				

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*iii*) Sealed proportional counters	Score maximum for the highest count rate and resolution for Kα lines for z = 22 (Ti) to z = 33 (As).		The provision of a sealed counter improves the performance of the instrument in the middle wavelength range and is of particular benefit if no facility for tandem operation of the scintillation and flow proportional counters is available.	PS WF ST				
4. *Electronics* (*a*) Pulse processing								
(*i*) Window selection (discriminator)	This is the ability to select a suitable electronic window encompassing the X-ray pulses. Providing this facility is available it may be inappropriate to score this feature.		A minimum voltage threshold must be set to exclude electronic noise. It is beneficial to set a maximum to exclude pulses from higher order diffractions which will contribute to the background count rate.					
(*ii*) Pulse height depression	Score for the provision of effective electronic compensation for change of pulse height distribution as a function of count rate.	VI	Pulse height depression occurs progressively at count rates above about 10^4 due to the changes in the electric fields within gas proportional counters. The depression may cause the analytical signal to drift out of an electronic window set up on the pulse height analyser when samples of varying count rate are measured.	PS WF ST				
(*iii*) Maximum usable count rate	Score maximum for the highest count rate for which the instrument will yield quantitative data.	VI	The ability to produce a linear response over a wide dynamic range, permitting a wide range of elemental concentrations to be analysed without changing the excitation conditions, minimises analysis time, as excitation conditions do not need to be compromised by a need to constrain count rates from the more concentrated samples.	PS WF ST				
(*iv*) Dead time correction and linearity	Score highly for accurate dead time correction up to the maximum usable count rates.*	VI	Dead time arises from the inability of the counter to respond to a second X-ray event detected a short time after the first and the inability of the amplifier to distinguish between two events occurring within the resolving time set by the selected time constant. Both of these effects could lead to systematically low measurements at high count rates, unless the appropriate correction is made.	PS WF ST				
(*v*) Total count capacity	Score maximum for the maximum number of counts that can be registered for a single measurement.	NVI	There may be a limitation on the maximum numbers of events that can be processed for a single measurement. This will limit the ability of the instrument to make convenient measurements under conditions of high sensitivity or high concentration.	PS WF ST				

* This can be tested by checking the linear range of the instrument by plotting count rate *versus* tube current. It must be realised that this perceived linearity will also be affected by the linearity of the generator.

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
5. *Computer* (*a*) Automation (*i*) Instrument control	Score maximum for the greatest number of instrument features which are under computer control.	VI	Computer control of instrumental parameters ensures reliable and reproducible operation of the instrument by well trained, but non-expert, operators. This prevents instrumental errors (conflicting settings), which could lead to instrumental malfunction, or even damage, and, most importantly, permits unattended operation for prolonged periods even when a number of parameters need re-setting during such a period of automatic operation.	PS WF ST				
(*ii*) Operating programme	Score maximum for a user friendly operating system which permits the user to develop versatile application programmes.	I	Difficult interaction and complex access codes can lead to errors. The use of "soft keys" or other means of reducing setting-up time and minimising complexity minimises training effort.	PS WF ST				
(*iii*) Instrument	Score maximum for the most comprehensive display of status of instrument parameters and effective monitoring for instrumental faults.	I	Rapid and effective monitoring of faults will minimise instrument down time, while a comprehensive display of instrument parameters will permit the operator to be confident that the required conditions are being met.	PS WF ST				
(*b*) Data processing (*i*) Quantitative	Score maximum for the provision, as appropriate to the application, of such features as: 1, Background correction; 2, Peak overlap correction; 3, Spectrum subtraction; 4, Calibration using various data fitting modes for linear and non-linear functions; 5, Standardless calibration; 6, Matrix correction for absorption and enhancement effects by empirical and fundamental parameter corrections; and 7, Output, storage for further processing and comparison, editing and report formatting.	VI	Raw data can rarely be used to produce quantitative analyses directly. Provision of these various functions enables the instrument to produce meaningful outputs.	PS WF ST				
(*ii*) Qualitative analysis	Score maximum for the provision of features such as: 1, Automatic scanning over specified wavelength ranges or elements; 2, Digital manipulation of spectra such as smoothing background correction and overlays, leading to element identification. 3, Output, quality of hard copy and provision of storage for further manipulation.	I	These features are required for effective programming for both routine and research use. Effective characterisation of unknown sample and efficient identification of possible analytes in unknown samples is a prerequisite of satisfactory quantitative analysis.	PS WF ST				
6. (*a*) Instrument footprint	The bench and/or floor space and the floor loading of the instrument.	Varies with location but may be VI	The instrument must be laboratory or plant compatible to avoid expensive alterations.	PS WF ST				

Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*b*) Services								
(*i*) Environmental control	Score maximum for minimum requirements for environmental control (temperature, humidity, vibration, *etc.*) necessary to enable the instrument to operate within its specification.	VI	Additional costs may be considerable if close control of environmental factors is required. This may be particularly important if the instrument is intended to be operated in a plant environment.	PS WF ST				
(*ii*) Electrical	Score maximum for compatability with existing electrical supply with regard to loading, stability and tolerance of transients.	Varies with users circumstances	Provision of alternative power supplies may significantly increase installation costs.	PS WF ST				
(*iii*) Cooling water	Evaluate costs for the provision of cooling water supply and score maximum for the lowest costs.	I	Requirements for large amounts of cooling water or the installation of a recirculating system can significantly increase running costs.	PS WF ST				
(*c*) Service and spares	Enquire in detail as to local arrangements and score accordingly.	VI	Cost of spares and availability of service may affect down time and running costs.	PS WF ST				
(*d*) Training facilities, effective documentation and technical support	Enquire as to local arrangements and score accordingly.	I	Good technical support can reduce commissioning time and improve analytical effectiveness of the instrument.	PS WF ST				
7. *Safety considerations*	In the UK, construction of the instrument and all safety interlocks must comply with appropriate regulations including the Ionising Radiations Regulations 1985. Other countries have similar National Regulations with which instrumentation must comply.	VI	Apart from the obvious danger, operation of instruments in contravention of statutory regulations is illegal.	PS WF ST				
8. *Extraneous leakage of radiation*	The lowest possible leakage of radiation from the equipment should be aimed for. Score additionally for extra features which minmise extraneous radiation.	I	Exposure to any radiation is undesirable and legally enforceable limits apply in most countries. Specified maximum radiation below legal requirements should be scored additionally.	PS WF ST				
9. *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 include ST in grand total.	I	"Simple" instruments are often good value for money, whereas those with many refinements are often costly.	PS WF ST				
				Grand Total				

Overall Performance

Figures of Merit

This test measures the capability of an instrument to detect trace elements across the complete spectrum range. Use a sample containing a small concentration of the element being tested, preferably in a matrix matched to that of the samples and which contains no interfering elements.

1. Low *z* (long wavelength), *e.g.*, Mg Kα: Tests multi-layer diffracting element, flow counter and coarse collimator combination.
2. (*a*), Medium *z* (medium wavelength), *e.g.*, Zn Kα: Tests LiF 200 crystal, flow or scintillation counter and fine collimator. (*b*), Nb Kα: Tests LiF 200 crystal, scintillation counter and fine collimator.
3. High *z* (short wavelength), *e.g.*, Ba Kα: Tests LiF 200 crystal, scintillation counter, fine collimator and beam filter.

Collect data for both the gross peak and background count rates. Calculate the detection limit (3 sigma).

Detection limit

$$DL = \frac{3\sqrt{2}}{m} \times \sqrt{\frac{R_b}{T_b}}$$

where R_b is the count rate at background, T_b the counting time on background and m the sensitivity (counts s^{-1}, p.p.m. or %). The lower the detection limit value the better.

Also calculate the factor (F) (often referred to as a figure of merit) which is used for optimising instrument operating conditions.

$$F = \sqrt{R_p} - \sqrt{R_b}$$

where R_p is the count rate at peak.

The higher the value of F, the better.

Stability

This test checks the degree of long-term drift under defined conditions. Perform this test on an overnight (14 h) run. Choose a sample containing elements representative of the full wavelength range and repeat the measurement cycle over the necessary time span. The sequential measurement of each element should involve changing crystal, detector and collimator. If possible, the position of the sample in the sample chamber should also be changed at the end of each measurement cycle.

Separate runs should be used to monitor performance under vacuum, helium (and air) conditions as appropriate to the application. Perform a statistical analysis of each series of data as follows: calculate the spread of the measurements (difference between the maximum and minimum count expressed as a percentage of the mean). Calculate the standard deviation of the mean (expressed as relative standard deviation) and note any drift of the count rate with time. The smaller the value of these parameters, the better.

Spectrometer Resolution

The resolution of an X-ray spectrometer depends on a number of factors, including the choice and quality of the diffraction crystal, choice of collimators (coarse or fine) and spectrometer angle. Some of these parameters are fixed during instrument manufacture, others are selectable by the user. Operation under conditons of high resolution is invariably accompanied by a reduction in measured count rates. Unlike atomic emission applications, spectrometers that are designed to give best resolution will not necessarily give optimum over-all analytical performance in a particular application. Analytical judgement must be used in specifying the most appropriate compromise between high spectrometer resolution (so minimising spectrum interferences) and count rate performance (which influences the precision of measurements).

With these limitations in mind, spectrometer resolution can be measured by exciting a sample with elements which give fluorescent lines that differ in wavelength by about 0.002 nm.

1. Cl Kα (at 0.473 nm) and Ag Lα (at 0.471 nm), use Ge crystal and fine collimator.
2. Ba Lα (at 0.278 nm) and Ti Kα (at 0.275 nm), use LiF (200) crystal and fine collimator.
3. Th Lα_1 (0.095 6) and Rb Lα_2 (0.093 0 nm) or Pb Lα_1 (0.084 0 nm), Y K$\alpha_{1,2}$ (0.083 04 nm), use LiF (220) and fine collimator.

Ideally, the instrument should achieve baseline separation between the two peaks in each instance.

Special Purity

This test checks for the presence of stray lines generated from contaminants in the X-ray tube or by fluorescence of spectrometer components. Any significant peaks make the interpretation of trace amounts in qualitative scans more difficult. High blank values also reduce the detection limit of the instrument. Perform a full spectral scan by exciting a sample of graphite on silica that itself contains negligible trace element contaminants. Examine this scan for the presence of fluorescence lines scattered off the sample, particularly of the transition metals (Cr–Zn).