Report by the Analytical Methods Committee

Evaluation of analytical instrumentation. Part I. Revised 1998. Atomic absorption spectrophotometers, primarily for use with flames

Analytical Methods Committee

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A method is provided for comparing the features of atomic absorption spectrophotometers, primarily for use with flames.

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), Dr. C. Burgess, Dr. K. E. Jarvis, Dr. S. J. Hill, Dr. M. Barnard and Mr. D. Squirrell, 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 that are not always easily comparable. The objective of the Instrumental Criteria Sub-Committee is 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 between instruments to be made.

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 considered 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.

This report of the Sub-Committee, a revision of the first report published in 1984, deals with instrumentation primarily designed for Flame Atomic Absorption. There have been many advances since the first report, in particular the use of computers and software to control many instrument functions, to process data and provide data acquisition facilities. Use of boosted hollow cathode lamps and the determination of a number of elements by hydride generation has also been included.

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 a weighting factor according to their own needs.

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.*, $\times 1$ to $\times 3$, or $\times 1$ to $\times 10$. The factor could amount to total exclusion of the instrument.
3. (ST) Sub-total. This is obtained by multiplying PS by WF.

Flame AAS is now a very well established analytical technique with applications in many areas. An often bewildering range of instrumentation is available from well over twenty different manufacturers. Systems range from relatively simple instruments, with limited sample and data handling capabilities to powerful instruments with extensive data and automation capabilities.

Selection of a suitable instrument for purchase is, therefore, not an easy task, and the purpose of these notes is to provide some guidance to areas which should be considered so that the choice is based on a full consideration of the available options. However, the performance of any instrument used for trace metal analysis depends primarily on the preparation conditions and if test exercises are used in the evaluation, a reliable method of preparation and presentation for the levels being examined must be available. The type of instrument will also influence the sensitivity, although selectivity varies very little. A number of alternative instruments may thus be suitable, although different sample preparation procedures may be required for instruments sensitive to dissolved solids.

The first task in the selection of an instrument is to examine the range of analyses that it will be expected to perform. Care should be taken not to specify these requirements too closely as uses change with time. The analytical scientist should also not try to envisage every potential application or the selection criteria may become too detailed.

The choice of the introduction device (nebulizer/spray chamber/burner) and the available built-in source supplies are outside the scope of these guidance notes, but any specific requirements should be noted, such as the efficient use of small sample volumes.

With these requirements in mind, the user should then evaluate the instruments available on the market while bearing in mind the guidelines and any financial limitations. In many instances it will quickly become clear that a number of different instruments could be satisfactory and non-instrumental criteria may then be important. However, in some specialized cases only one or two instruments will have the ability or necessary features to carry out the assay. The guidelines are intended to be used as a check list of features to be considered, mostly of the instrument itself, but some also of its service requirements and of the relationship of the user with the manufacturer. Their relative importance will depend on the installation requirements of the instrument as well as the uses to which it will be put. Therefore, to some extent, the selection process will inevitably be subjective, but if all the points have been considered it should be an informed choice.

Finally, as many laboratories are now working to quality protocols and standards such as GLP/UKAS(NAMAS)/ISO 9000/FDA/EPA, some consideration should be given to third party recognition of the manufacturer to standards such as the appropriate ISO 9000 series. Such accreditation should extend to the service organization, which is particularly important when working to UKAS (NAMAS) or GLP criteria.

Previous reports in this series from the Analytical Methods Committee

Evaluation of Analytical Instrumentation

Part 1. Atomic absorption Spectrophotometers, Primarily for use with Flames, *Anal. Proc.*, 1984, **21**, 45. Revised in *Analyst*. 1998, 123, 1407.

Part 2. Atomic absorption Spectrophotometers, Primarily for use with Electrothermal Atomizers, *Anal. Proc.*, 1985, **22**, 128. Revised in *Analyst*, 1998, 123, 1415.

Part 3. Polychromators for use in Emission Spectrometry with ICP Sources, *Anal. Proc.*, 1986, **23**, 109.

Part 4. Monochromators for use in Emission Spectrometry with ICP Sources, *Anal. Proc.*, 1987, **24**, 3.

Part 5. Inductively Coupled Plasma Sources for use in Emission Spectrometry, *Anal. Proc.*, 1987, **24**, 266.

Part 6. Wavelength Dispersive X-ray Spectrometers, *Anal. Proc.*, 1990, **27**, 324.

Part 7. Energy Dispersive X-ray Spectrometers, *Anal. Proc.*, 1991, **28**, 312.

Part 8. Instrumentation for Gas–Liquid Chromatography, *Anal. Proc.*, 1993, **30**, 296.

Part 9. Instrumentation for High-performance Liquid Chromatography, *Analyst*, 1997, **122**, 387.

Part 10. Instrumentation for Inductively Coupled Plasma Mass Spectrometry, *Analyst*, 1997, **122**, 393.

Instrument Evaluation Form

Type of instrument: Flame AAS

Maufacturer:

Model No.:

Feature	Definition and/or test procedure and guidance for assessement	Importance	Reason	Score				
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:							
(1) Previous instruments								
(*a*) Innovation	Company's record for instruments with innovative features.	I	The manufacturer should be aware of recent developments in flame AAS.	PS WF ST				
(*b*) Reliability record	Company's record for instrument reliability.	I	Reflects the company's ability for good design and manufacturing practices.	PS WF ST				
(*c*) Confidence in the supplier	Confidence gained from past personal experience.	I	Good working relationship already in place.	PS WF ST				
(2) Servicing	Score according to manufacturers' claims and past record, judged by the sub-features (*a*) to (*e*) below:							
(*a*) Service contract	Availability of suitable service contracts from the supplier, agent or third party contractor.	I/NVI	Suggests long term commitment to user. This often ensures preferential service and guarantees a specific response time to call-outs.	PS WF ST				

Feature	Definition and/or test procedure and guidance for assessement	Importance	Reason	Score				
(*b*) Availability and delivery of spares	Range of stock carried by, or quickly available to the manufacturer/agent/contractor.	(VI)	Rapid delivery of spares, down time and operating costs.	PS WF ST				
(*c*) Call-out time	Adequate service personnel readily available, minimizing the call out time.	I(VI)	Keeps laboratory in operation by reducing down time [see also (*a*)].	PS WF ST				
(*d*) Effectiveness of service engineers	The ability of the service engineers, as judged from previous experience and reports of others, including the carrying of adequate spares.	I	Ability to repair on-site avoids return visit or removal of equipment for off-site repair, so reducing down time, and may also reduce service costs.	PS WF ST				
(*e*) Cost of call-out and spares	Score for reasonable cost per hour and spares.	I	The proximity of the service center may be a factor in travel costs.	PS WF ST				
(3) Technical support	As in (2) score in consideration of sub-features (*a*) to (*d*) below.							
(*a*) Advice from Applications Department	The advice and training available from the manufacturers' applications department.	VI for new users	This helps in-house staff to maximize the use of the equipment and with problems on new applications.	PS WF ST				
(*b*) Technical literature	The range and quality of technical literature including the operating manual.	VI	Guidance on optimum use of instrument suggests manufacturer's awareness of applications.	PS WF ST				
(*c*) Telephone assistance	Willingness of the manufacturer/supplier/contractor to give advice over the telephone. This can normally be evaluated by reference to existing users.	I	Rapidly available technical help reduces the number of call outs and enhances productivity.	PS WF ST				
(*d*) Customer's maintenance	Score for the ability of the user to perform routine maintenance such as cleaning and or replacing utility items, such as nebulizers and detectors.	I	Reduces call out costs for simple maintenance procedures.	PS WF ST				
Instrumental criteria								
(1) Hollow cathode lamp supply								
(*a*) Number of lamp stations	Number of lamps under operating conditions should be commensurate with the analytical requirements, bearing in mind the possible use of muti-element lamps.	I	Economic (speed of analysis *versus* financial commitment).	PS WF ST				
(*b*) Modulation	Type and frequency—score high for electronic modulation at non-multiples of mains frequency and also for the highest frequency.	I	Suppression of unwanted dc signals, and rejection of mains noise and low frequency noise from nebulizer and gas flows.	PS WF ST				
(*c*) Method of lamp alignment	Two-axis adjustment by accessible controls preferred. Score extra if this facility is automatic.	I	Accurate alignment of source on optical axis.	PS WF ST				
(*d*) Boosted hollow cathode lamp supply	An additional supply is required to run the boosted discharge lamps. Score maximum for best short and long term stability for a built-in unit.	I	A boosted lamp powers a secondary discharge to remove residual ground state atoms from the primary discharge. This results in narrower lines, which improve sensitivity, linearity and detection limit. They are particularly useful in the short ultraviolet region, where the increased brightness improves the signal to noise ratio.	PS WF ST				

Feature	Definition and/or test procedure and guidance for assessement	Importance	Reason	Score				
(2) Burner–nebulizer assembly								
(*a*) Range of fuels and oxidants	Maximum score for acetylene, hydrogen and propane, air, nitrous oxide and appropriate burner heads. Minimum score for air–acetylene only.	VI	Correct selection of fuel–oxidant permits the maximum analytical coverage and allows matrix effects to be minimized.	PS WF ST				
(*b*) Materials of construction	Maximum score for titanium burner with inert plastic spray chamber and tantalum nebuliser. Reduce score for other materials such as such as stainless steel or glass unless demanded by a special application.	VI	Safety, durability, resistance to corrosion and memory effects.	PS WF ST				
(c) Spray chamber characteristics	Maximum score for spray chamber with impact bead and baffle assembly, which produces the maximum number of small particles, with a narrow distribution.	VI	Small droplets minimize interferences and reduce noise.	PS WF ST				
(*d*) Method of positional adjustment of the burner	This function is often under software control, but there are situations where manual override is desirable. Score maximum for provision of software control, with manual override.	I	Alignment for maximum absorption and/or linear calibration.	PS WF ST				
(*e*) Nebulizer uptake adjustment	Maximum score for widest range. Reduce score for fixed uptake rate with minimum score for highest fixed uptake.	I	Uptake rate affects sensitivity, freedom from interferences and usage of sample. (*N.B.* Drop size and distribution are also affected by uptake rate.)	PS WF ST				
(*f*) Dissolved solids capability	Plot signal and standard deviations of a series of solutions containing 5 ppm of lead and plot signal and standard deviations of a series of solutions containing 0 to 20 ppm of magnesium chloride. Aspirate each solution for 5 min.	I	A low tolerance to solids will result in burner–nebulizer blockage with degradation of sensitivity and precision.	PS WF ST				
(3) Hydride generators								
(*a*) Availability	A means of producing volatile hydrides for elements, such as As, Se, Sb, Sn, Ge, Sn and Pb, with a facility for also determining Hg by measuring the mercury vapor produced under reducing conditions. Score for availability if required.	VI	Hydride-forming elements usually provide much better detection limits than are obtained in flames, as the residence times in the atom cell are much extended. This permits the determination of these elements at levels not dissimilar to furnace-AAS, which is not usually appropriate for these elements.	PS WF ST				
(*b*) Accessibility	If the device will need to be removed and refitted to the instrument on a regular basis, score more for an access system designed around a cassette system, which can be simply removed and replaced.	VI	Assemblies made up of a number of discrete components that require assembling and disassembling are inconvenient, time consuming and uneconomic to employ.	PS WF ST				
(4) Monochromator optics								
(*a*) Temperature stability	Score maximum for lowest value of $\Delta\lambda°\ C^{-1}$, change in wavelength per degree.	VI	Elimination of instrumental drift, particularly important when high-temperature flames are used.	PS WF ST				

Feature	Definition and/or test procedure and guidance for assessement	Importance	Reason	Score				
(*b*) Focal length and *f* number	If the instrument is required for trace analysis, score maximum for long focal length and high *f* number. For instruments required for analysis of major constituents, score maximum for short focal length and low *f* number.	I	Long focal lengths and high *f* number lead to a narrow beam in the flame. This results in only the central reducing region being examined and gives improved sensitivity and greater freedom from interferences. Short focal lengths and low *f* numbers give high Iight throughput and hence good signal to noise ratio, but this may be invalidated by increased shot noise due to extra flame emission reaching the detector.	PS WF ST				
(*c*) Slits	Score minimum for fixed slits, intermediate for stepwise adjustment and maximum for continuously variable. Additional score for height adjustment.	I	Spectral discrimination and control of luminous flux.	PS WF ST				
(*d*) Grating	Mount and blaze angle. Modified Czerny–Turner mount generally preferred to Ebert or Littrow, as stray light characteristics are better. Maximum score for blaze angle nearest to the wavelengths of most interest.	I	Suitable blaze angle required to ensure adequate radiation throughput throughout the range of interest. The useful working range is approximately from two thirds to three times the blaze wavelength, the fall of efficiency being particularly sharp at the short wavelength limit.	PS WF ST				
(*e*) Wavelength								
(*i*) Read-out precision	Four-figure digital read-out preferred for manual instruments.	I	Ease of re-setting instrument if it is not automatic.	PS WF ST				
(*ii*) Repeatability	Maximum score for smallest range of transmission values following re-setting to a previously located line.	I	Ability consistently to locate analytical wavelength.	PS WF ST				
(*f*) Number of reflective and refractive elements	Score maximum for minimum number of optical elements. Score extra for quartz coated optics.	I	Maximum energy throughput with minimum scatter. Coated optics will increase the useful life of the instrument	PS WF ST				
(*g*) Background correction	Score maximum for widest wavelength range for which background can be corrected. Additional score for ease of replacement of source if present. (See Note *iii*.)	I	Particularly important if ETA is contemplated or when samples with very high solids content are analysed by flame.	PS WF ST				
(*h*) Dispersion, resolution and resolving power	Score maximum for small angular deviation and high angular dispersion: also small reciprocal linear dispersion, high resolution and high resolving power.	NVI	Normally adequate for AAS, but important if emission measurements are contemplated.	PS WF ST				
(*i*) Slewing speed	Score maximum for maximum speed. For automatic instruments score maximum for speed and accuracy and ability to identify lines unambiguously.	NVI	The speed of analysis, for automatic instruments, will be higher if time is not wasted by slow slewing rates.	PS WF ST				
(*j*) Scanning speed	Score maximum for lowest speed and greatest range of speeds.	NVI	Normally adequate for 'peaking' in AAS, but important if emission measurements are contemplated	PS WF ST				
(*k*) Single or double beam	Double beam preferred for long continuous sample runs. Single beam for lower cost and better sensitivity.	NVI	Double beam eliminates any residual drift resulting from the source. This is only of importance when extended long continuous sample runs are contemplated, *e.g.*, auto-sampling. Single beam systems may be preferred for lower costs and minimum detection limits.	PS WF ST				

Feature	Definition and/or test procedure and guidance for assessement	Importance	Reason	Score				
(5) Gas control unit								
(*a*) Pressure for stable operation	Score maximum for wide range consistent with safe working practice. Score zero if minimum acetylene pressure required to operate the flame is above 9 lb in^{-2}.	VI	Wide range enables best fuel–oxidant usage under conditions that minimise fluctuations in flow rates. The operation of acetylene flames at over 9 lb in^{-2} is generally not permitted in the UK.	PS WF ST				
(*b*) Safety features	Score maximum for ‘Auto Shut Down’, ‘Pressure Drop Sensors’, ‘Gas Cylinder Valve Heaters’ and correct burner type and location sensor.	VI	Safe operation of instrument.	PS WF ST				
(*c*) Manual/ electronic	Electronic preferred in general.	I	Electronic control provides an extra element of safety and convenience for most situations, but may not be compatible with hydrogen fuel, as gases are shut off in the wrong order and many flame sensors fail to detect hydrogen flames.	PS WF ST				
(*d*) Number of fuel and support gas inlets	Score maximum for maximum number.	I	Enables instrument to be conveniently operated with desired fuel–oxidant combinations.	PS WF ST				
(*e*) Flow-rate indication	Score maximum for digital indication and wide range of flow for each gas line.	I	Ease of reproducing conditions.	PS WF ST				
(*f*) Auto-ignitors	Score according to preference.	NVI	Convenience of operation.	PS WF ST				
(6) Detectors	Score maximum for the availability of a photomultiplier tube which meets most requirements, and for the ease of interchange.	I	A suitable photomultiplier is required to cover the wavelength range of the elements of interest. Where one photomultiplier cannot give sufficient spectral range, ease of interchange is important, as is the ability of the replacement to attain working stability rapidly.	PS WF ST				
(7) EHT Supply								
(*a*) Voltage range	Score maximum for wide range and digital read-out applied voltage.	I	Reproducible instrument operation and minimum detector noise.	PS WF ST				
(*b*) Means of adjustment	Adjustment by calibrated manual control preferred. Automatic continuous adjustment of EHT is undesirable.	I	Consistent signal to noise ratio can only be achieved by operation at constant EHT, which should not be changed during the analysis.	PS WF ST				
(8) Amplifier								
(*a*) Type	Synchronously demodulated ‘lock-in’ normal; score maximum number for above type with largest number of attenuation ranges.	VI	Operational versatility and removal of unwanted dc signals. Note: Some instruments use digital data processing and the known timing of the readings permits the signal to be separated from the noise and the signal to be deconvoluted from the back-ground without the need for a lock-in amplifier.	PS WF ST				
(*b*) Time constants	Score maximum for widest available range and number.	I	Minimizing noise, consistent with signal type and efficient sample utilization.	PS WF ST				

Feature	Definition and/or test procedure and guidance for assessement	Importance	Reason	Score				
(*c*) Integration	Importance of ability to change integration times is dependent on the particular instrument.	I	If the signal contains a high proportion of (white) noise, integration will improve the precision. Signals that are dominated by proportional (pink) noise will not show much improvement in precision upon integration.	PS WF ST				
(9) Output								
(*a*) Read out type	Score maximum for availability of analogue, digital, printer and graphics output.	I	Digital read-out and printer are particularly suitable for quality control applications and measurement of small signals, while analogue and graphics outputs are beneficial when measuring transient peaks.	PS WF ST				
(*b*) Interface	Score maximum for suitable interface, *e.g.*, RS232, IEEE, BCD or ASCII.	I	Compatibility with available computers, printers or other data systems.	PS WF ST				
(10) Amenities These items will have varying importance to different users and should be scored and rated accordingly								
(*a*) Modular construction	Self explanatory.		Allows expansion of system to meet changing needs.	PS WF ST				
(*b*) Bench space required	Self explanatory.		The instrument must fit the laboratory or expensive modifications may be needed.	PS WF ST				
(*c*) Services	Electrical, plumbing, drainage.		Installation of additional services (*e.g.*, 3-phase power) will increase cost of installation.	PS WF ST				
(*d*) Automation	Various items such as sample presentation, lamp selection, wavelength setting, slit setting and burner operation may be automated.		Items such as auto-samplers are essential for some users (*e.g.*, ETA), while other automation may be desirable if large numbers of samples are to be handled. Automation also reduces operator errors and invariably improves precision.	PS WF ST				
(*e*) Availability of major accessories and updates	Enquire about manufacturer's policy on updating software and compatibility of present and future accessories.	I	Future analytical requirements.	PS WF ST				
(11) Over-all performance								
(*a*) Base line stability	Allow 30 min to warm up, then take readings at 1 min intervals for 30 min with only the lamp running (electronic stability). Repeat the above measurements with the flame running and water aspirating to give overall base line stability. Calculate the standard deviations and score accordingly.	VI	Affects accuracy and precision. A shorter warm-up period may be suitable for double-beam instruments, but although base lines reach a constant value more rapidly, sensitivity will still vary until the system has stabilised.	PS WF ST				
(*b*) Figures of merit	Use concentrations of test elements to give nominal absorbances of 0.0, 0.005, 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1.0 and 2.0 based on the manufacturers sensitivity data, assuming that a linear relationship exists. Measure each solution at least six times, using scale expansion for readings below 0.1. Solutions of absorbance 0.01 and 0.2 should be measured at 1 min intervals for 30 min to give precision data.							

Feature	Definition and/or test procedure and guidance for assessement	Importance	Reason	Score				
(*i*) Precision	Calculate standard deviation and score maximum for lowest.	VI	Self evident.	PS WF ST				
(*ii*) Sensitivity (slope of calibration curve)	Calculate slope of line and score maximum for greatest slope.	VI	Self evident.	PS WF ST				
(*iii*) Linear range	Calculate from calibration curve. Score high for widest linear range.	VI	Self evident.	PS WF ST				
(*iv*) Detection limit	Calculate from twice the standard deviation of the reagent blank, or any other factor preferred, providing that consistency is maintained. Score maximum for lowest.	VI	Self evident.	PS WF ST				
(*v*) Curve correction	Use the curve correction facility to linearise the calibration function in (*b*) above and spray a solution with a known concentration and a nominal absorbance of 1.2–1.5. Score maximum for the most accurate result.	NVI	Self evident.	PS WF ST Sum of sub totals				
(12) Value for money Points per £	Sum of previous sub-totals divided by the purchase price of the instrument. Subject to proportional scoring and weighting factor, as for previous features. 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				

Notes

(*i*) For instruments without built-in data handling, measurements can either be made using a recorder, which should be run for 10 s with a time constant of 1–2 s, or using integration and a digital read-out, in which case the integration time should be no more than 5 s, and should be as similar as possible for all of the instruments being evaluated. For instruments with data systems, similar tests may be carried out using the data system. This should not only evaluate the instruments' performance, but give some guidance as to how easy it is to set up to make non-routine measurements.

(*ii*) The user can employ any element(s) that are considered to be of importance; however, the following elements are thought to be particularly useful:
Arsenic at 193.7 nm—Evaluates performance at the far ultraviolet end of the instrument range.
Lead at 217.0 nm—Short wavelengths, widely determined, often difficult at levels of interest.
Nickel at 232.0 nm—Requires good resolution to achieve good sensitivity and linearity.
Aluminium at 309.3 nm—Uses N_2O–C_2H_2 flame which has high emission in this region. Will test flame stability and effectiveness of modulation in excluding flame noise.
Copper at 324.7 nm—Typical 'easy' mid-range element for which figures of merit are quoted by some manufacturers.
Calcium at 422.7 nm—Similar to copper, above, but uses N_2O–C_2H_2 flame.
Potassium at 766.7 nm—Most common long wavelength element, requiring either a red sensitive photomultiplier tube (PMT) or a wide range PMT with a red filter.

(*iii*) The efficiency of background correction depends on the availability of equal time constants in both channels of the amplifier and the ability to balance the source intensity for both channels. A test for the efficiency can be made by evaluating the positive interference caused by 10 000 ppm of sodium chloride on 5 ppm of lead at 217.0 nm and 10 ppm of arsenic at 193.7 nm.

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