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Report by the Analytical Methods Committee

Evaluation of Analytical Instrumentation. Part V Inductively Coupled Plasma Sources for Use in Emission Spectrometry

Analytical Methods Committee
Royal Society of Chemistry, Burlington House, Piccadilly, London W1V 0BN

A method is provided for comparing the features of inductively coupled plasma sources for use in emission spectrometry.

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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, Professor L. Ebdon, Dr. E. J. Newman 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 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 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.

The fifth report of the Sub-Committee deals with ICP sources for use in emission 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 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.*, ×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: ICP source for use in emission spectrometry

Manufacturer:								
Model No:								
Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
1. *High frequency generators* (*a*) Type of oscillator	The oscillators differ mainly in the method which is used to control the frequency of oscillation and are variously known as free running, crystal controlled, Huth - Kühn and tuned line oscillators. Scoring may be inappropriate (see reason).	NVI	The primary requirement of a generator is stability (see below). There is no scientifically credible evidence that one type of generator offers superior performance. This presupposes that any generator in question meets the requirements for stability demanded by the user and complies with local or national regulations for HF shielding and					

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Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
			filtering. It is conceivable that newer types of generators based on solid-state devices will have some advantages, at least in terms of physical size.					
(*b*) Radiation shielding	All oscillators and torch boxes must be screened in order to prevent or minimise HF leakage and such screening must be guaranteed to comply with the regulations of the country of operation. Score maximum for the minimum leakage, measured with a suitable meter.	VI	The leakage of high frequency radiation presents a number of hazards. Also there are strict regulations in most, if not all, countries in the world with regard to the amount of energy of certain frequencies which may be radiated from an oscillator. Stray radiation not only interferes with communications but may also affect the detector electronics and hence the output of the instrument.	PS WF ST				
(*c*) HF filtering	There should be adequate filtering of HF signals on the power lines of the generator to prevent them from coupling with the power lines of any detection electronics. (There should be similar filters on the power lines of the detector electronics.) Score accordingly for the presence of such filters.	VI	If any HF leakage occurs within the generating unit, the power and earth lines can conduct HF signals. These signals can then couple with the detector electronics and affect the background noise. This, in turn, will affect the limits of detection obtainable. Leakage can readily couple with other local instrumentation and can modulate the signal affecting instrument performance.	PS WF ST				
(*d*) Frequency of operation	The usual frequency of operation of generators for spectroscopy is 27 MHz, but some generators operate at higher frequencies. It may be thought desirable to score greater for higher frequency (up to 100 MHz).	NVI	There is *some* evidence that higher frequency operation increases the signal to background ratio, although the excitation temperature is also reduced. In general, a higher frequency plasma is easier to start and sample introduction is easier than into one operated at lower frequency. The generator may also be more compact.	PS WF ST				
(*e*) Power available	The power in kilowatts which can be developed in a plasma by the generator. Score according to usage, bearing in mind that a high power generator may offer more flexibility if it can also be run at low power.	I	The power required will depend upon the torch to be used and on the purpose for which it is intended. Miniature torches require only a few hundred watts, but large torches call for several kilowatts of power. Intermediate size torches operate at around one kilowatt. Argon plasmas generally require lower power for their operation than do polyatomic gas plasmas. Higher power permits the introduction of organic solvents/samples and more flexibility of usage, as the torch is less prone to being extinguished by changes of solvent and the Swan bands are absent from the spectra at high powers.	PS WF ST				
(*f*) Power indication and setting	The power developed in the plasma should be indicated by a meter or calibrated control. According to application, score maximum for the system which gives the most accurate and complete information. Score additionally for reproducibility and flexibility of setting.	I	For comparison purposes in research work and in method development it is important to know the power developed in the plasma. For routine use it is sufficient to know the power developed at the work coil and to set this reproducibly. Most generators have a reflected power meter which gives the power into the impedance matching circuit. All meters and/or controls should have accompanying information, which gives the power in the	PS				

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Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
			plasma either from calculation or calibration.	WF ST				
(*g*) Coupling efficiency	This is the fraction of the power supplied to the coil that is transferred to the plasma. The power in the plasma can be determined directly by calorimetric measurement or indirectly by the use of a dummy load and calorimetry. Score maximum for highest efficiency.	I	For maximum efficiency, it is desirable to transfer the maximum amount of power available to the plasma.	PS WF ST				
(*h*) Power stability	The degree to which the power in the plasma varies from a set value. Score maximum for the highest degree of regulation of the d.c. voltage for a given mains variation. This figure should always be given by the manufacturers.	VI	Fluctuations are brought about by variations in line voltage and some form of feedback control should be incorporated in the generator. The emission intensity for elements introduced into an ICP is strongly dependent on the power in the plasma. Short term fluctuations in power are therefore clearly undesirable.	PS WF ST				
(*i*) Tuning (for crystal controlled generators)	Free running oscillators have their frequency fixed by the value of the components in the tank circuit. However, crystal controlled generators require re-tuning if the impedance changes. This is accomplished by a "match box" which should incorporate automatic tuning. Score maximum for the most rapid response to such changes and the widest range over which this can be achieved with minimal overshoot.	VI	Unless the matching is automatic and rapid, the plasma will be extinguished. A wide tuning range will accommodate the greatest changes in impedance, permitting the use of the widest range of gases.	PS WF ST				
(*j*) Water cooling	In most, if not all, plasma generators the work coil, and in some cases the oscillator valve, are water cooled. Score maximum for the system that requires the lowest flow and pressure of water and calls for the least treatment of the cooling water.	I	It is expensive to have to install high pressure mains supplies and to install water treatment in areas of hard water. Failure to treat water in such areas can lead to failure due to blocking of work coils.	PS WF ST				
(*k*) Air cooling	Some generators are air, rather than water, cooled. In such instances score maximum for the generators which achieve this with the lowest power requirement.	I	Instruments with high power requirements for cooling may be noisy and uneconomical.	PS WF ST				
(*l*) Interlocks	It should be impossible to gain access to the generator while the HF current is turned on. Score zero if this interlock is not effective.	VI	The voltages and currents involved in plasma generation are dangerous!	PS WF ST				
2. *Torch boxes* (*a*) Ease of access	Score according to the ease with which torches can be fitted and removed from the box and the ease with which coils can be fitted and removed. This to include centring of the torch within the coil.	VI	Breakage of torches can occur if it is difficult to fit them. Water leakage resulting from difficulty in tightening connections on work coils can cause damage. It is essential to ensure that the torch tube is centrally placed in the coil if a good, well positioned plasma is to be obtained. Off-centre torches will not only cause melting of the torch tube but will result in an off axis tailflame.	PS WF ST				
(*b*) Ease of observation	It desirable to be able to observe the plasma through an observation window which is screened against HF leakage and equipped with UV filters. Score according to the degree of convenience offered.	I	This facilitates observation of malfunctioning of the plasma torch, which can cause damage and will lead to unsatisfactory performance.	PS WF ST				

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Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
(*c*) Height of observation adjustment	It is essential to be able accurately to set the height of observation of the plasma tailflame and to be able to do so reproducibly. Score according to reproducibility and ease of re-setting.	VI	Owing to the spatial nature of the plasma, the emissions from the tailflame and interferences from easily ionised elements are very dependent on the height of observation.	PS WF ST				
(*d*) Mounting of the plasma torch	Score according to the ease with which the plasma torch can be held in a vertical position centrally within the work coil of the generator. Mounting devices with the minimum degrees of freedom of movement consistent with accurate positioning are to be preferred.	VI	The line of observation of the tailflame should not move off axis as the viewing height is altered or else misleading signals will be obtained. If the mounting has many degrees of freedom of movement, it becomes very difficult to ensure that off-axis observations do not occur.	PS WF ST				
(*e*) Exhaust systems	It is necessary to remove all gases from the torch box in such a manner so as not to disturb the plasma. Tests for gas flow and the presence of toxic substances should be augmented by visual observation of movement of the plasma when the exhaust is turned on. Score maximum for the efficient extraction combined with minimum observed movement when the exhaust is turned on.	VI	Hot and corrosive gases and toxic elements must be removed from the laboratory atmosphere, at the same time movement of the tailflame should be avoided if precision is to be maintained.	PS WF ST				
(*f*) Interlocks	It should not be possible to gain access to the torch box while the HF current is turned on or to be able to switch on the HF unless the water and gas supplies are on. The HF should also switch off automatically in the event of the plasma being extinguished. Score zero if this interlock is not effective.	VI	The voltages and currents involved in plasma production are dangerous! These interlocks will prevent accidental damage to the torch and load coil in the event of the failure of one of the supplies. They are also essential to ensure safe operation of the instrument.	PS WF ST				
3. *Gas controls*								
(*a*) Flow control	A constant mass flow of, particularly, the nebuliser gas, the plasma (intermediate) gas and, to a lesser degree, the coolant (outer) gas is highly desirable. Score highest for systems using electronic mass-flow controllers, less for spring loaded mass flow controllers and less still for systems using restrictors and up-stream pressure indicators. An even lower score should be given to the common method of measuring the gas from a two-stage regulator through a needle valve and float flow meter. There should be no interaction between the gas flows brought about by taking each flow from a common main. Increase the above score if gas controls are accurately calibrated.	VI	The emission from the tailflame of a plasma is very dependent on the gas flows, particularly the nebuliser gas flow. It is therefore essential to control these gas flows to a high degree. Any fluctuations in the nebuliser gas will affect the precision of the final signal. Any interactions caused by taking a relatively high gas flow from a common source, such as might be brought about by taking a coolant flow from the main which also supplies the much lower nebuliser flow, are to be avoided.	PS WF ST				
(*b*) Choice of gases	Gases other than argon may be useful under some circumstances. Score maximum for the widest choice of gases, ease of changeover and ability to provide other mixtures if required.	I	Economy as well as spectro-chemical reasons may often make it desirable to use alternative gases.	PS WF ST				
4. *Torches*	There are a variety of torches which can be used to produce plasmas for emission work. Score the highest for the system that allows the greatest flexibility in the choice of torch to be used.	I	Small diameter Fassel torches are the most commonly used as they require less gas and power than do larger torches and give a similar performance. They do, however, have a few disadvantages					

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Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score				
			when compared with large torches. Greater accuracy is required in their construction and they are less tolerant of manufacturing imperfections. They do not function well with many solutions of high solute content and the plasmas tend to be extinguished by ingress of air into the nebuliser, especially when working at low powers. The larger diameter Greenfield torches are of rugged design, fairly tolerant of manufacturing imperfections and able to work under more varied conditions, *e.g.*, with saturated solutions of many elements. They will tolerate the ingress of air and other gases such as hydrogen and nitrogen through the nebuliser without the plasma being extinguished. Laminar flow torches have the advantage of a lower noise than do the tangential flow torches; they are, however, relatively untried. Miniature torches use much less gas and power than all the torches previously mentioned but they also are relatively untried.	PS WF ST				
5. *Nebulisers and spray chambers (sample introduction devices)*	There are various sample introduction devices for conveying the sample to be analysed to the plasma. Score the highest for the plasma system which allows the greatest choice of such devices.	VI	Pneumatic, concentric or cross-flow nebulisers are the most commonly used in plasma work. However, they may not tolerate solutions with a high solute or particulate content. For these solutions it is usual to use the Babington nebulisers which will tolerate particulates (even slurries) and highly concentrated solutions. The spray chamber should effectively remove larger particles and minimise fluctuations from the nebuliser, while transporting the maximum amount of sample into the plasma. Electrothermal vaporisation and hydride generation devices are also available for use with ICP systems.	PS WF ST				
6. *Sample introduction pumps*	A pump with a wide range of stable pumping speeds, as free as possible from pulsations, is required. Score accordingly.	VI	It is often desirable to pump the sample solution to the nebuliser (this is essential for the Babington nebuliser). Pulsing from the pump impairs precision, this effect being minimised by the use of multi-roller pumps run at speed.					
7. *Amenities* (*a*) Bench/floor space/weight (floor loading)	Self-explanatory.	Varies with users circum-stances but may be VI	The instrument must be laboratory compatible or else expensive alterations will be required.	PS WF ST				

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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 (room temperature and humidity) necessary to enable the instrument to operate within its specification.	VI	Additional installation costs may be considerable if close control of environmental factors is required.	PS WF ST				
(*ii*) Electrical	Score maximum for compatibility with existing electrical supply, both with regard to loading and stability.	Varies with users circumstances	Additional power requirements may significantly increase installation costs.	PS WF ST				
(*c*) Servicing and spares	Enquire in detail as to local arrangements and score accordingly.	VI	Cost of spares, servicing and down-time may severely alter over-all running costs.	PS WF ST				
				Total				

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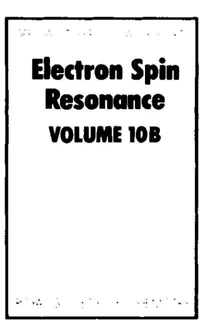

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