

Analytical Methods Committee

Evaluation of analytical instrumentation. Part XIX CHNS elemental analysers

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Abstract The reports of this series tabulate a number of features of analytical instruments that should be considered when making comparison between various systems. Scoring these features in a rational manner allows a scientific comparison to be made between instruments as an aid

to selection. This is the XIXth report of the series and deals with CHNS

are marked as VI (very important). A scale should be 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 the total exclusion of the instrument.

3. (ST) Sub-total. This is obtained by multiplying PS by WF.

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 specialised cases only one or two instruments will have the ability or necessary features to carry out the required analyses.

The guidelines are intended to be used as a checklist 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.

The Committee consider that instrumentation for CHNS analysis is safe in normal use, but care should be taken in handling toxic chemicals and high temperatures.

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 ISO 9001:2000. Such certification should extend to the service organisation.

Basis of instrumentation

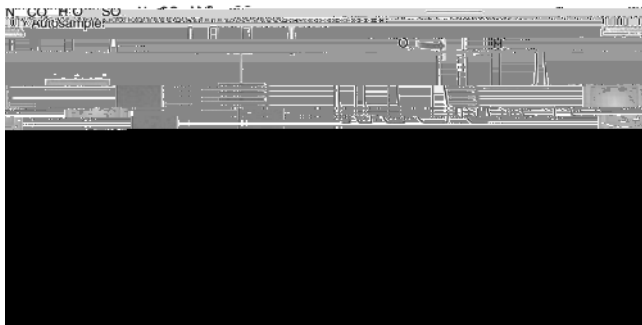
In its simplest form, simultaneous CHNS analysis requires high temperature combustion (ca 1000 °C furnace temperature) in an oxygen-rich environment. This combustion can be carried out under both static conditions i.e. introduction of a set volume of oxygen or dynamic conditions i.e. A constant flow of oxygen for a set period. Both types of technologies have or are being used by instrument manufacturers in elemental analysers at the present time. Often, catalysts are also added to the combustion tube in order to aid conversion.

Other elemental combinations can also be carried out using these elemental analysers: carbon alone; nitrogen alone; sulfur dynam3n6.5(bo4ulfu8e;)-640.1(i]TJ 0 -arbon)-63N

An overview of CHNS elemental analysers

In considering the use of elemental analysers to perform CHN and S analysis, it was decided that the remit should be restricted to only combustion systems as other techniques, particularly for sulphur analysis (ICP, XRF etc), are also available.

CHNS Elemental Analysers



Apparatus

As can be seen, there are a number of different formats which have been applied to combustion elemental analysers depending on the elements of interest; sample size; concentration range etc. Each of these different formats has its own advantages and disadvantages. The choice of analyser for particular applications will depend on a number of factors which are tabulated in the instrument evaluation form given below.

(i) *'Blank' contributions* When considering the accuracy of measurement, one of the important factors is an assessment of the blank contribution from the gases, the capsules used for sample containment and the instrument system.

In the case of the gases, the purity of the carrier gas (e.g. helium) and the oxygen are very important, the latter where a level of 99.9995% is advisable. Together with use of suitable GC gas traps to remove traces of organics, water etc, this minimises the contribution of nitrogen etc to the blank. Another important consideration is to reduce the length of piping from the cylinders to the instrument to a minimum and to avoid any jointing. This situation also allows effective flushing of the line when cylinders are

able and the organic content present. In addition, attempts have been made to improve limits of detection (LOD) etc by different modes of detection.

In the first approach, the combustion gases, after removal of excess oxygen and non-CHNS products, are fully separated on a GC column into nitrogen, carbon dioxide, water and sulfur dioxide. If the column deteriorates or is overloaded, the nitrogen and carbon dioxide peaks overlap resulting in incorrect data. Nowadays, the instrument is connected to a computer so that this problem is easily detected. If only trace nitrogen is being measured (0.02 percent m/m) in CHN mode, it is possible for the peak to be 'missed' by the computer if appropriate analytical conditions are not used.

In the second approach, the sample weight can be increased because only partial separation is required using a GC column. This is referred to as 'frontal' chromatography. In this case, all the CHN combustion products are collected in a gas-mixing chamber (impeller driven) and pressurised to 2 atmospheres (note: sulfur cannot be measured simultaneously in this mode of separation). At a set time interval, these gases flow through the GC column and continue to flow throughout the period of the analysis (approx. 4 minutes). This results in an accumulative stepwise trace which allows automated measurement of plateau heights for the three gases.

In the third approach, no separation step is included. This requires a more complicated detection system in that individual infra-red cells are used to quantify separately the carbon dioxide water and sulfur dioxide content with a thermal conductivity cell for nitrogen detection. This approach has the advantage that it allows separate measurement but is more complicated to use and more expensive in the first place.

Other approaches have also been used such as the separate adsorption of the gases followed by release and quantification in turn.

(iv) *Calibration procedures* Though there are different configurations of instrument, the calibration procedures are very similar in each case. Use is made of a number of certified reference materials for primary calibration e.g. acetanilide, benzoic acid, etc, which can be purchased from a number of suppliers. Acetanilide has traditionally been the compound of choice for CHN analysis as it can be produced with high purity.

In the case of instruments using GC separation, it is normal to run two or three 'bypass' acetanilide samples to condition the system before running three or four empty capsules to achieve 'blank' values. (The need for conditioning runs is primarily to stabilise the trace water content on the GC column.) Next, several weighed acetanilide samples are

Instrumental criteria sub-committee instrument evaluation form

Type of Instrument: Elemental Analyser (CHNS)					
Manufacturer					
Model No:					
Feature	Definition and/or test procedures and guidance for assessment	Importance	Reason	Score	
<i>A. non-instrumental criteria</i>					
<i>selection of manufacturer</i>					
(a) Previous instruments					
(i) Innovation	Laboratories in possession of other CHNS systems should score highest for the manufacturer with the best past record based on the following sub-features: Company's record for developing instruments with innovative features	I	The manufacturer should be alert to developments in technology	PS WF ST	
(ii) Reliability record	Company's record for instrument reliability. Score additionally if the manufacturing operation is accredited to a recognised Quality System e.g. ISO 9002	I	Indicates history of sound design and manufacturing concepts	PS WF ST	
(iii) Up-grading compatibility	Availability and ease of software and hardware upgrades and compatibility with earlier versions	I	Allows extension of instrument capabilities whilst ensuring the ability to reprocess old data files or methods. This is particularly important for laboratories that are accredited or regulated	PS WF ST	
(iv) Similarity of layout and design to instruments existing in laboratory	Consideration should be given to manufacturers of CHNS equipment who manufacture the other forms of elemental analysis instruments. For routine purposes this may be important. However, this may be less important for research applications	I	Similarity of layout means that operators can draw on in-house expertise, resulting in reduced training costs and time. It can also maximise the use of spares and fittings. Suites of elemental analysis equipment from a single manufacture imply similar computer software, reducing training and duplication of computer equipment	PS WF ST	
(v) Confidence in supplier	Confidence gained from past experience	I	Good working relationship already in place	PS WF ST	
(b) Servicing					
(i) Service contract	Score according to manufacturers claims and past record, judged by the sub-features (i)-(vi) below: Availability of suitable service contracts from the supplier, agent or third party contractor	VI	Suggests long commitment to user. Often ensures preferential service and can guarantee a specific response time to call-outs	PS WF ST	
(ii) Calibration	A certificate of calibration, including traceability of calibrants should be issued at any service	VI	The calibration department, on or off site, should be accredited as a calibration laboratory operating to ISO 17025	PS WF ST	
(iii) Availability and delivery of spares	Range of stock carried by, or quickly available to, the manufacturer/agent/contractor	I	Rapid delivery of spares reduces downtime	PS WF ST	
(iv) Call-out time	The time for an engineer to reach the laboratory following a call	I	Keeps laboratory in operation by reducing down time (see also (i) and (iii))	PS WF ST	
(v) 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. Training records of service engineers should be available on request	I	Ability to repair on-site avoids return visit or removal of equipment to supplier and reduces service time, costs and downtime	PS WF ST	
(vi) Cost of call-out and spares	It <i>may</i> be inappropriate to score this feature if downtime is not critical				

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