Application Note 58

CHN Analysis: Handling challenging samples

The use of elemental microanalysis to determine percentage carbon, hydrogen and nitrogen (CHN) in samples and comparison of this data versus theoretical values is a well-established technique for determining sample purity. However not all sample types readily lend themselves to straight forward analysis by CHN microanalysis. CHN microanalysis of some sample types still requires the applications knowledge of a trained analyst and the use of a high performance CHN microanalysis system such is available from Exeter Analytical.

Over the last 10+ years – Exeter Analytical's team of experienced analysts have gained considerable knowledge and expertise in preparing and running a large variety of sample types for CHN analysis.

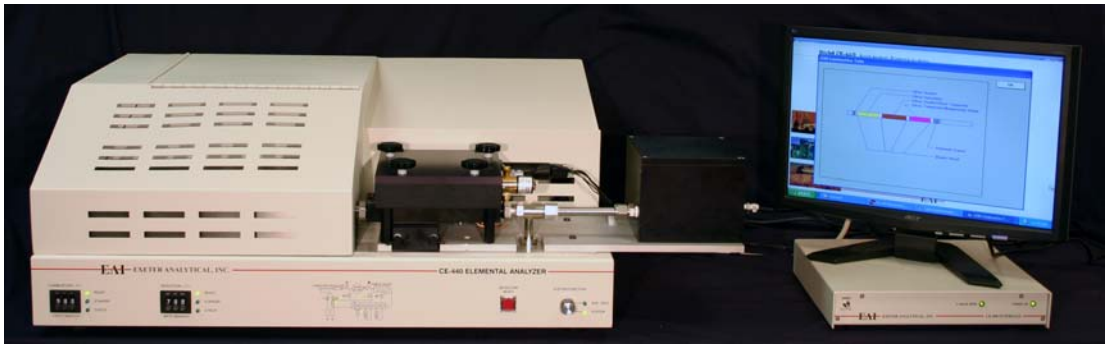
Introduction:

This application note has been written to share the knowledge and experience of our analysts which in conjunction with the unique features of the Exeter Analytical Model 440 CHN microanalysis system will enable you to produce consistent top quality data when faced with more challenging sample types. Sample types that can pose a challenge to generating accurate and precise CHN data include volatile materials, air sensitive samples, refractory materials, samples with elemental interferences and samples with a physical property making them problematic to weigh.

Benefits of a Horizontal furnace CHN Microanalysis System:

The Model 440 CHN microanalysis system (Figure 1), available from Exeter Analytical, has become the system of choice for many labs because of its proven ability to accurately analyse the widest range of sample types - quickly, easily and reliably. The Model 440 delivers unmatched productivity and top quality results as it benefits from a horizontal furnace design that prevents cross contamination and allows the running of a greater number of samples between combustion tube changes. Based upon a unique combined static / dynamic combustion technology, the Model 440 is able to precisely analyse the widest range of sample types from volatile liquids to difficult-to-combust materials including nitrides, graphite fibres, ceramics and even carbides with melting points of over 2000°C. Offering unmatched operating accuracy and precision, the fully automated Model 440 delivers simultaneous CHN analysis in less than 5 minutes.

Figure 1:



Expert Tips for handling challenging CHN microanalysis sample types:

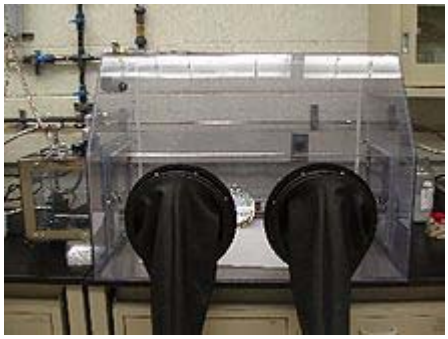
Volatile materials

It is important with volatile liquids that you achieve an air tight seal on the tin capsules in which the samples are weighed to prevent losses. The Exeter Analytical elemental analyser capsule sealer (Part No. 313-00012) was developed specifically for this purpose and is proven to reliably seal tin capsules with no volatile loss. Another option, especially useful for extremely volatile materials, is to use the Single Sample Interrupt (SSI) port on the Model 440 microanalysis system which enables samples to be analysed with minimal delay from the point of weighing. Where samples are either non-volatile or slightly volatile there is no disadvantage in sample introduction using the standard system autosampler.

Example of results achievable for analysis of volatile materials

Type of Sample	% Carbon	% Hydrogen	% Nitrogen
Fuel Oil	83.47 83.60	13.87 13.65	1 0

Air sensitive



Air sensitive samples should be weighed in an inert atmosphere to prevent the absorption of moisture from the air. If this precaution is not taken inaccurate results may result, particularly hydrogen due to the water molecules taken up by the sample. Precise analysis of air sensitive samples can be achieved using a glove box or glove bag filled with a gas such as argon. Samples should be carefully transferred into pre-weighed tin capsules and sealed within this environment. Once the samples are fully sealed they can then be removed from the glove box and the capsules re-weighed to obtain the sample weight.

Example of results achievable for the analysis of air sensitive samples

Type Of Sample	% Carbon	% Hydrogen	% Nitrogen
Air Sensitive Organometallic Compound	9.94 9.78	8.96 8.94	0.53 0.58

Refractory materials

Refractory materials such as nitrides, graphite fibres, ceramics and carbides can by their nature be difficult to combust. The Model 440 microanalysis system makes the analysis of these samples more routine. The Model 440 allows for complete control over combustion

parameters. The unique horizontal furnace design facilitates the use of combustion aids to achieve the high temperatures required for complete combustion of these difficult samples without the worry of residue build up in the combustion tube.

Example of results achievable for the analysis of a refractory material

Type of Sample	**% Carbon**
Silicon Carbide Reference standard 112b	*Theory 29.43%* Normal Temperature Operation - 20.11% ; 21.05% *High Temperature Operation – 29.37%, 29.60%*

Elemental interferences

Some materials, such as perfluorinated compounds and samples that can form carbides can cause potential elemental interference problems to the CHN analysis process. The Model 440 microanalysis system uses a range of specialised reagents to remove elemental interferences including Magnesium Oxide, Silver Tungstate, Silver Vanadate and Silver Oxide. Due to the unique design characteristics of the system, elemental interference removal reagents such as powdered magnesium oxide and 'red lead' can be used without the residue build up problems experienced by other designs of elemental analyser.

In the case of compounds containing fluorine, the magnesium oxide in the combustion tube reagent packing's absorbs fluorine as HF, and releases the hydrogen which enables accurate measurement of %CHN.

Example of results achievable for the analysis of perfluoro compounds

Type Of Sample	% Carbon	% Hydrogen	% Nitrogen
perfluoro-N-octanoic acid (95% purity)	22.15, 22.07 (Theory – 22.05%)	0.22, 0.25 (Theory – 0.24%)	0.0 0.0 (Theory - 0.0%)

Difficult to weigh samples

The physical nature of samples such as gums, viscous gels, low density materials and inhomogeneous samples can cause problems in CHN microanalysis.

Samples such as gums and viscous gels are difficult by their nature to remove from the sample vial in which they are stored. They are also problematic to weigh cleanly into tin capsules without losing some material in the process leading to inaccurate results. To resolve this problem our analysts have developed a proprietary technique involving the use of capillary tubes to measure controlled amounts of these substances and weigh them cleanly into tin capsules with minimal losses.

Typically low density samples, such as aerogels and freeze dried materials, can be quite bulky in nature making them difficult to weigh. To overcome this problem it is recommended to weigh these samples into larger tin capsules to obtain around 2mg in weight. Alternatively it is possible to carry out a specialised low weight calibration run for samples of this type enabling the analyst to take a lower weight than normal and still use standard tin capsules.

Some samples, such as environmental waste materials, are typically inhomogeneous. For such inhomogeneous samples it is vitally important that sample preparation methods, such as grinding, are used to homogenise the samples prior to analysis. In addition it is recommended that you weigh larger sample amounts to minimise the inhomogeneous nature of the sample.

Example of results achievable for Low density materials

Type Of Sample	**% Carbon**	**% Hydrogen**	**% Nitrogen**
Low density pharmaceutical sample	42.27 42.32	5.93 5.92	13.34 13.47

Conclusion

The expert application knowledge of our experienced analysts combined with the unique design of the Model 440 results in very accurate CHN analysis over a wide range of samples. Unlike other designs of CHN analyser the Model 440 microanalysis system will remove all residue between each analysis as opposed to simply combusting your sample on top of the old residue from previous analysis. This combined with absolute control over combustion parameters and static design of the Model 440 enables unsurpassed accuracy and precision.

For Further Information

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