



Microanalytical Services

Introduction

Elemental analysis for carbon, hydrogen and nitrogen is widely performed to characterise and prove the chemical composition of organic or even inorganic materials. Other elements such as oxygen and sulphur can also be determined to provide additional information or confirmation.

The technique is quantitative in the range of 200ppm to 100% and typically uses sample weights in the order of 1 mg for typical pharmaceutical applications and up to 250 mg for soils and other heterogeneous materials. Analytical results on routine organic compounds have an uncertainty better than 0.25% for carbon, 0.05% for hydrogen and 0.10% for nitrogen.

Elemental Microanalysis Limited has provided this analytical service for industrial and academic customers for more than 25 years. Our accumulated analytical expertise is routinely applied to a wide diversity of sample types and applications. The analytical laboratory quality assures the company's own manufactured products and certifies internationally respected standard reference materials. Gaseous, liquid or solid samples (even air sensitive materials) are accepted for analysis.

Although the analysis service is primarily aimed at providing routine determinations on a wide variety of materials, the analytical laboratory is able to undertake specialist applications. Examples include nitrogen contamination in 2% sulphuric acid solution, low-level protein in serums and oxygen in gasolines. Please contact the laboratory to discuss your requirements.





Applications

These are a few of the many sample applications where elemental microanalysis is employed:

Petroleum Products

(coal, coke, fuel oils, gasolines, pitch, petrochemicals, lubricants etc)

Geological Samples

(kerogene, soils, sediments, shale, cement, clays etc)

Medical Samples

(blood, hair, nails, serum, urine etc)

Inorganic Materials

(carbon fibres, catalysts, refractories, cement, graphite, chemicals etc)

Organic Materials

(polymers, resins, detergents, chemicals, paint, rubber etc)

Plant Materials

(wood, cotton, paper, leaves, seeds, hay etc)

Environmental Samples

(contaminants, sludges, water, fertilisers, soils, composts)

Food Products

(flour, cereals, feeds, ingredients, milk, sugars, meat etc)

Schedule of Analytical Services

The analytical laboratory at Elemental Microanalysis Limited currently offers the following determinations:

CHN Carbon, Hydrogen and Nitrogen (automated simultaneous elemental analyser)

TOC Total Organic Carbon (sample acidification and automated elemental analyser)

N Protein (automated protein analyser)

O Oxygen (automated elemental analyser)

S Sulphur (automated elemental analyser)

Analyses are always carried out in duplicate (sample quantity permitting).

Submission of Samples for Analysis

Samples should be sent directly to the analytical laboratory in the UK.

Samples should be transported in secure and clearly labelled sample vials suitably packaged for transport by post or carrier. Sample vials should be proportional to the size of the contained sample. Identification should be unique where possible to avoid confusion caused by over simplified labeling (ie A, B, C, 1, 2, 3 etc).

Adequate sample should be provided (see recommended sample weights under each analysis description). Larger samples are preferred because our analysts must be able to collect the sample from the vial and transfer the material to small capsules. Surplus samples will be returned.

Air sensitive samples should be submitted in sealed glass ampoules or tubes (one provided for each analysis or group analysis such as CHN). Tubes should be of sufficient length to allow scoring and breaking at the middle point. The preferred size is 5mm bore and 100mm in length.

A sample Data Form should be completed for each sample submitted. These are generally self-explanatory. Information on the expected composition and the sample's elemental content is important to avoid errors introduced by known interferences. Data Forms can be downloaded from our website or supplied directly from Elemental Microanalysis Limited upon request.

Unless otherwise specified samples are analysed 'as received' without grinding or drying. We are able to dry or grind samples as required.

Our stated sample turn-round is less than one week but in reality we normally achieve 48 to 72 hours (depending on work load). Results are routinely reported by email but can be confirmed by fax, phone, and/or post if required. Although we do not formally offer an express service we can often provide a same-day service where there is a genuine requirement.

Please read our Terms and Conditions before submitting samples for analysis. Elemental Microanalysis Limited does not provide Expert Testimony in legal proceedings.



Carbon, Hydrogen and Nitrogen (CHN) Analysis

Total Carbon, Hydrogen and Nitrogen are determined by a commercial elemental analyser running under computer control.

Principle of method

Samples are precisely weighed into lightweight tin capsules and dropped at preset times into a combustion tube (at 1000°C) through which a constant stream of helium is maintained. Just prior to sample introduction the helium stream is replaced by pure oxygen for a brief period. The sample is instantaneously burned followed by intense oxidation of the tin capsule at 1800°C (flash combustion). The resulting combustion gases are passed over catalysts to ensure complete oxidation and absorption of halogens, sulphur and other interferences. Excess oxygen is removed as the gases are swept through a reduction tube containing copper at 650°C. Any oxides of nitrogen are reduced to nitrogen gas. Finally the gases are separated on a chromatographic column into nitrogen (N), carbon dioxide (C) and water vapour (H) and quantitatively measured by a thermal conductivity detector (TCD). The system response is calibrated to known calibration standards.

Where appropriate non-viscous liquid samples can be introduced into the system by means of an automatic liquid injection autosampler.

Sample weights

The required sample weights vary according to the sample type and the anticipated percentages of carbon, hydrogen and nitrogen.

<1% CHN	20mg	single analysis
1% to 5% CHN	10mg	single analysis
>5% CHN	1mg	single analysis

Limit of detection: 0.01% absolute approximately

Where samples are generally considered as heterogeneous at these sample sizes more material should be supplied (ie plant material). In this case larger samples sizes (up to 100 mg) might be used or the sample ground to provide a representative sample.

Interferences

The technique is generally free of interferences. A few silicon and organo-metallic compounds exhibit a tendency to form stable carbides. Samples containing phosphorus may not combust successfully and give low carbon values. The analysis of numerous fluorine containing compounds will cause errors in the hydrogen result. Fortunately all of these problems can be remedied by the addition of additives to the sample.

Oxygen (O) Analysis

Oxygen is determined by a commercial elemental analyser running under computer control.

Principle of Method

Samples are precisely weighed into lightweight silver capsules and dropped at preset times into a combustion tube (at 1050°C) through which a constant stream of helium is maintained. The resulting pyrolysed gases are passed over catalysed carbon to ensure complete conversion of oxygen in the sample to carbon monoxide. Finally the carbon monoxide is separated from other gases on a chromatographic column and quantitatively measured by a thermal conductivity detector (TCD). The system response is calibrated to known calibration standards.

Where appropriate non-viscous liquid samples can be introduced into the system by means of an automatic liquid injection autosampler.

Sample Weights

The required sample weights vary according to the sample type and the anticipated percentages of oxygen.

<1% O	40mg	single analysis
1% to 5% O	20mg	single analysis
>5% O	2mg	single analysis

Limit of detection: 0.05% absolute approximately

Where samples are generally considered as heterogeneous at these sample sizes more material should be supplied to provide a representative sample after grinding.

Interferences

The technique is generally free of interferences for compounds containing just carbon, hydrogen, nitrogen, oxygen, sulphur, chlorine, bromine or iodine. Fluorine interferes significantly due to its ability to liberate oxygen from the glass components in the system. Alkaline metals, alkaline earths, lanthanide and actinide series, silicon and other elements that form thermally stable oxides not reduced by carbon can induce significant errors. Phosphorus compounds can poison the carbon catalyst sufficiently to passivate its performance.

Total Organic Carbon (TOC) Analysis

Total Organic Carbon is determined by a commercial elemental analyser running under computer control.

Principle of Method

Samples are precisely weighed into lightweight silver boats or capsules, acidified by 1:1 hydrochloric acid and digested at 80°C. This effectively eliminates carbonates as carbon dioxide.

After final encapsulation the samples are dropped at preset times into a combustion tube (at 1000°C) through which a constant stream of helium is maintained. Just prior to sample introduction the helium stream is replaced by pure oxygen for a brief period. The sample is instantaneously burned. The resulting combustion gases are passed over catalysts to ensure complete oxidation and absorption of halogens, sulphur and other interferences. Excess oxygen is removed as the gases are swept through a reduction tube containing copper at 650°C. Finally carbon dioxide (C) is separated from other unwanted gases on a chromatographic column and quantitatively measured by a thermal conductivity detector (TCD). The system response is calibrated to known calibration standards.

Sample Weights

The required sample weights vary according to the sample type and the anticipated percentages of Total Organic Carbon.

<1% C	100mg	single analysis
1% to 5% C	20mg	single analysis
>5% C	5mg	single analysis

Limit of detection: 0.01% absolute approximately

Where samples are generally considered as heterogeneous at these sample sizes more material should be supplied (ie soil material). In this case larger samples sizes (up to 100 mg) might be used or the sample ground to provide a representative sample.

Interferences

The technique is generally free of interferences. A few silicon and organo-metallic compounds exhibit a tendency to form stable carbides. Samples containing phosphorus may not combust successfully and give low carbon values. Fortunately all of these problems can be remedied by the addition of additives to the sample. Even most inorganic compounds such as carbides and nitrides can be successfully analysed.

Sulphur (S) Analysis

Sulphur is determined by a commercial elemental analyser running under computer control.

Principle of Method

Samples are precisely weighed into lightweight tin capsules and dropped at preset times into a combustion tube (at 1000°C) through which a constant stream of helium is maintained. Just prior to sample introduction the helium stream is replaced by pure oxygen for a brief period. The sample is instantaneously burned followed by intense oxidation of the tin capsule at 1800°C (flash combustion). The resulting combustion gases are passed over catalysts to ensure complete oxidation. The gas stream is then dried by means of a water scrubber. Excess oxygen is removed as the gases are swept through a reduction tube containing copper at 650°C. Finally the sulphur dioxide is separated from other interfering gases on a chromatographic column and quantitatively measured by a thermal conductivity detector (TCD). The system response is calibrated to known calibration standards.

Where appropriate non-viscous liquid samples can be introduced into the system by means of an automatic liquid injection autosampler.

Sample Weights

The required sample weights vary according to the sample type and the anticipated percentages of sulphur.

<1% S	20mg	single analysis
1% to 5% S	10mg	single analysis
>5% S	2mg	single analysis

Limit of detection: 0.02% absolute approximately

Where samples are generally considered as heterogeneous at these sample sizes more material should be supplied to provide a representative sample after grinding.

Interferences

The technique is generally free of interferences. Some inorganic materials may not liberate sulphur immediately but this can be remedied by the addition of additives to the sample. A few compounds that contain phosphorus may form thermally stable compounds that may bind some sulphur.

Nitrogen (Protein) Analysis

Protein is determined as nitrogen by a commercial protein analyser running under computer control.

Principle of Method

Samples are precisely weighed into lightweight tin capsules and dropped at preset times into a combustion tube (at 950°C) through which a constant stream of helium is maintained. Just prior to sample introduction the helium stream is replaced by pure oxygen for a brief period. The sample is instantaneously burned followed by intense oxidation of the tin capsule at 1800°C (flash combustion). The resulting combustion gases are passed over catalysts to ensure complete oxidation and absorption of halogens, sulphur and other interferences. Excess oxygen is removed as the gases are swept through a reduction tube containing copper at 650°C. Any oxides of nitrogen are reduced to nitrogen gas. Water and carbon dioxide are removed by passing the gases through scrubbing tubes and the remaining nitrogen gas is quantitatively measured by a thermal conductivity detector (TCD). The system response is calibrated to known calibration standards. The protein value is normally calculated using a factor of 6.25 unless otherwise specified.

Sample Weights

The required sample weights vary according to the sample type and the anticipated percentages of nitrogen.

<1% N	300mg	single analysis
1% to 5% N	200mg	single analysis
>5% N	100mg	single analysis

Limit of detection: 0.03% absolute approximately

Where samples are generally considered as heterogeneous at these sample sizes more material should be supplied to provide a representative sample after grinding.

Interferences

The technique is generally free of interferences.



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"The centre of excellence in elemental analysis"

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