Analytical Services for Stable Isotope Analysis and Elemental Analysis

Website: www.oealabs.com
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December 2015
Issue 3.02
Trusted Analytical Expertise

OEA Labs is an established commercial laboratory specialising in stable isotope and elemental analysis.

Our experience and fast turn-around means we can deliver real value to your research, processes or products.

Stable Isotope Analysis (SIA) for C, N, O and S

With two continuous-flow isotope ratio mass spectrometers interfaced with configured elemental analysers, OEA Labs routinely analyse the stable isotope ratios of carbon ($^{13}$C/$^{12}$C), nitrogen ($^{15}$N/$^{14}$N), oxygen ($^{18}$O/$^{16}$O) and sulphur ($^{34}$S/$^{32}$S) on solid or liquid samples.

Elemental Analysis (EA) for % C, H, N, S and O

Three CHNS/O elemental analysers routinely determine carbon, hydrogen, nitrogen, oxygen and sulphur in a wide range of organic and inorganic materials. OEA Labs analyse these elements from high percentages down to less than 0.01% on solid, liquid or even gaseous samples. We also determine total organic carbon (TOC), total inorganic carbon (TIC) and protein.

Elemental Analysis (EA) for F, Cl, Br, I and P

Hydro-combustion, acid digestion and oxygen flask techniques are employed to prepare samples for analysis by Ion Chromatography (IC), UV/Vis spectroscopy, titration and ion specific techniques to determine total fluorine, chlorine, bromine, iodine and phosphorus in both organic and inorganic samples. OEA Labs quantify these elements from percentage levels down to parts per million. We also analyse numerous anions such as nitrite, nitrate, sulphite, sulphate, phosphate and phosphite and undertake other non-routine analysis.

Industrial Production, Authenticity and Quality Control

Molecular and Biomolecular Chemistry

Geographical and Earth Sciences

Atmospheric and Marine Sciences

Environmental Chemistry and Monitoring

Food Web, Dietary and Metabolic Sciences

Medical and Pharmaceutical Sciences

Archaeology
About OEA Laboratories

OEA Labs is a trusted and established commercial laboratory providing analytical services in stable isotope and elemental analysis.

The analytical chemists and technicians at OEA Labs have many years of experience working in isotope and elemental analysis laboratories.

Our laboratories are equipped with modern isotope ratio mass spectrometers (IRMS), elemental analysers (EA), ion chromatographs (IC) and other supporting instrumentation and equipment.

We are committed to innovate and improve our analytical methods to the highest possible standard to meet or surpass the expectations of our customers.

Contact Details

Samples should be sent directly to the laboratories at OEA Labs:

Ship to: OEA Laboratories Limited
          Unit C2 Florence Road Business Park
          Kelly Bray, Callington, Cornwall, PL17 8EX
          United Kingdom

Phone: +44 (0) 1579 384174
Fax: +44 (0) 1579 384174
VAT No: GB 931 879 587
Website: http://www.oealabs.com

For technical information, quotation or other information: email stuart.carter@oealabs.com

Ordering

We accept orders by email, telephone, fax or post.

For analysis we do not necessarily need a purchase order number if payment can be made against our invoice. Please state your delivery address, invoice address, contact name and contact telephone number.

For intra-EU supplies, please state your VAT number or provide a VAT exemption certificate if your organisation is VAT exempt.

Sending Samples for Analysis to OEA Labs

Samples should be shipped 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 labelling (e.g. 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 recover sufficient sample from the vial to transfer into small capsules. We can return unused samples upon request.

For small numbers of submitted samples, sample information should be provided on one of our data forms or a covering letter. For larger numbers of submitted samples, sample information should be provided in table or spreadsheet format.

Unless otherwise specified samples are analysed as received without grinding or drying. At additional cost we are able to dry in air, under vacuum or grind samples as required. Please contact us for further information.

In accordance with our Terms and Conditions please note that OEA Labs does not provide Expert Testimony in legal proceedings.

Payment Methods

We can set up immediate 30 day invoice account facilities for bona fide companies, research organisations and academic institutions.

We also accept credit or debit card payments by telephone, post or email. We do not store card numbers although we do store other customer details in the normal course of our business. Your credit or debit card will not be debited until your samples have been analysed and reported. Please let us know if you wish to pay by this method at the time of ordering.
Stable Isotope Analysis (SIA) - $^{15}$N, $^{13}$C, $^{18}$O and $^{34}$S

With two continuous-flow isotope ratio mass spectrometers interfaced with configured elemental analysers, OEA Labs routinely analyse the stable isotope ratios of carbon ($^{13}$C/$^{12}$C, δ$^{13}$C), nitrogen ($^{15}$N/$^{14}$N, δ$^{15}$N), oxygen ($^{18}$O/$^{16}$O, δ$^{18}$O) and sulphur ($^{34}$S/$^{32}$S, δ$^{34}$S) on solid or liquid samples.

In stable isotope analysis, milligram amounts of samples are combusted or pyrolysed at high temperature in a helium carrier gas. After suitable preparation the measurable gases (N$_2$, CO$_2$, CO or SO$_2$) are separated on a chromatography column. A small proportion of this gas is fed into an evacuated ion source where the gas molecules are accelerated into a strong magnetic field. The gas species of different masses are subsequently deflected across a collector array where the individual masses are monitored and quantified against known references.

Both δ$^{15}$N and δ$^{13}$C can be analysed simultaneously if the sample can meet certain criteria. In order to achieve accurate results it is necessary to work within the linear response range of our isotope ratio mass spectrometers (IRMS). This necessitates having an absolute weight of 20 to 200 micrograms of nitrogen and 40 to 800 micrograms of carbon in a single weighed sample. If you know the elemental composition and the %C:%N ratio is less than 40 then it should be possible to weigh a sample within range. If this cannot be achieved then it is our practice to run nitrogen and carbon isotope analysis as two separate analysis runs.

Precision (as average standard deviation) on well prepared samples will generally be better than 0.3‰ for nitrogen and 0.2‰ for carbon. We report nitrogen results referenced to air and carbon to Vienna Pee Dee Belemnite (VPDB) and if required the %N and %C obtained from our total beam values.

Although we prefer to weigh your samples we are able to accept pre-weighed encapsulated samples for this service. We monitor our IRMS total beam values to ensure that the weight criteria is met and will inform you if there are any discrepancies.

Samples are precisely weighed to provide 20 to 200 micrograms of nitrogen for δ$^{15}$N analysis. Precision (as average standard deviation) on well prepared samples will generally be better than 0.3‰. We report nitrogen results referenced to air and if required the %N obtained from our total beam values.

Although we prefer to weigh your samples we are able to accept pre-weighed encapsulated samples for this service. We monitor our IRMS total beam values to ensure that the weight criteria is met and will inform you if there are any discrepancies.

Samples are precisely weighed to provide 40 to 800 micrograms of carbon for δ$^{13}$C analysis. Precision (as average standard deviation) on well prepared samples will generally be better than 0.2‰. We report carbon results referenced to Vienna Pee Dee Belemnite (VPDB) and if required the %C obtained from our total beam values.

Although we prefer to weigh your samples we are able to accept pre-weighed encapsulated samples for this service. We monitor our IRMS total beam values to ensure that the weight criteria is met and will inform you if there are any discrepancies.

Samples are precisely weighed to provide 20 to 200 micrograms of oxygen for δ$^{18}$O analysis. Precision (as average standard deviation) on well prepared samples will generally be better than 0.3‰. We report oxygen results referenced to Vienna Standard Mean Ocean Water (VSMOW) and if required the %O obtained from our total beam values.

Although we prefer to weigh your samples we are able to accept pre-weighed encapsulated samples for this service. We monitor our IRMS total beam values to ensure that the weight criteria is met and will inform you if there are any discrepancies.

Samples are precisely weighed to provide 20 to 100 micrograms of sulphur for δ$^{34}$S analysis. Precision (as average standard deviation) on well prepared samples will generally be better than 0.3‰. We report sulphur results referenced to Vienna Canyon Diablo Trolite (VCDT) and if required the %S obtained from our total beam values.

Although we prefer to weigh your samples we are able to accept pre-weighed encapsulated samples for this service. We monitor our IRMS total beam values to ensure that the weight criteria is met and will inform you if there are any discrepancies.
Sample Preparation for Stable Isotope Analysis

Because stable isotope analysis techniques typically use sample weights in the order of 1 to 5 milligrams, considerable attention should be given to sample preparation. To achieve reliable data samples must be of acceptable homogeneity and be representative of the whole. Samples should be dehydrated by freeze drying or oven drying at 40°C to 70°C (depending on material type) then finely ground using a mortar & pestle or a ball mill. Inorganic carbon in the form of carbonates can interfere with the measurements of organic $^{13}$C in soils or sediments. If inorganic carbon is present it can be eliminated by careful direct acidification with dilute hydrochloric acid or indirectly by exposure to concentrated sulphurous acid vapour. We are able to prepare or pre-treat your samples at additional cost. Please contact us to discuss any aspects of sample preparation that you may require.

Most of our customers submit dried and prepared samples for analysis in vials or bottles ready for us to weigh and run on our systems. In this case we will determine the workable weight ranges best suited for a particular analysis. Generally, if the %C:%N is less than 40, it is possible for us to determine $^{15}$N/$^{14}$N and $^{13}$C/$^{12}$C simultaneously on a single weighed sample. Please contact us to discuss your requirements.

Samples submitted for analysis should be catalogued (emailed Excel spread sheets are preferred when many samples are submitted).

Pre-weighed Sample Preparation for Stable Isotope Analysis

Some of our customers prefer to pre-weigh their own samples. Samples should be weighed into tin capsules (available from OEA Labs) using a 6 decimal place balance. The tops of the capsules should be folded over and the capsules formed into roughly spherical shapes using forceps. This will avoid jamming in our autosamplers. Care must be taken not to rupture the capsule to avoid loss of sample and consequential contamination in our systems.

Inorganic carbon, if present, in the form of carbonates will interfere with the measurement of organic $^{13}$C/$^{12}$C. The inorganic carbon can be removed by weighing the samples into silver capsules (tin capsules can decompose), slightly wetting with water and arranging in 96 well trays. The entire tray can be placed in a desiccator containing a beaker of concentrated sulphurous acid and leaving at room temperature for 48 hours. Samples should then be dried at 60°C and then sealed. Alternatively the whole silver capsule (which can become brittle after acid treatment) can be placed into a new tin capsule and sealed.

The sample weights should be catalogued (emailed Excel spread sheets are preferred when many samples are submitted) and indexed into 96 cell wells prior to submission. Capsules in various sizes and sample trays are available from us. Please enquire.

The optimum sample weight will depend on the type of material submitted for analysis. Our ideal target sample weights would contain 100 micrograms of nitrogen for $^{15}$N, 400 micrograms of carbon for $^{13}$C, 100 micrograms of oxygen for $^{18}$O or 30 micrograms of sulphur for $^{34}$S absolute. In practice there are ‘safe’ working limits on either side of these values. Please contact us if you have any questions.

For practical reasons maximum sample weights should not exceed 60mg because they may not fit our autosamplers. If samples fit loosely into a 96 cell tray (6mm diameter x 10mm deep) they will run on our systems. Samples containing more than 7mg absolute of carbon submitted for sulphur or nitrogen analysis should be avoided because the amount of available oxygen in our elemental analyser prep systems might be exceeded. In practice we can work outside of this range where necessary by the use of a large autosampler or oxygen dosing loop.

It is very useful for us to have a two sets of pre-weighed samples to allow us to run random QC check samples. This also ensures that we can keep our sample turn-around on schedule should we have any problems with our instrumentation or encounter other unforeseen failures.

### Weight Guide for Pre-Weighed Sample Submissions for $^{15}$N & $^{13}$C Stable Isotope Analysis

<table>
<thead>
<tr>
<th>Material</th>
<th>%N</th>
<th>%C</th>
<th>$^{15}$N</th>
<th>$^{13}$C</th>
<th>$^{15}$N &amp; $^{13}$C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Leaves</td>
<td>2-3</td>
<td>42-50</td>
<td>1-5mg</td>
<td>0.5-1.6mg</td>
<td>1-1.6mg</td>
</tr>
<tr>
<td>Root</td>
<td>0.8-1.3</td>
<td>36-40</td>
<td>2.5-11.5mg</td>
<td>0.6-2mg</td>
<td>2-2.5mg</td>
</tr>
<tr>
<td>Stems</td>
<td>0.4-0.8</td>
<td>46-48</td>
<td>5-18.8mg</td>
<td>0.4-1.7mg</td>
<td>not possible</td>
</tr>
<tr>
<td>Wood</td>
<td>0.02-0.06</td>
<td>40-44</td>
<td>60mg</td>
<td>0.5-1.8mg</td>
<td>not possible</td>
</tr>
<tr>
<td>Grain Flour</td>
<td>1.5-3.5</td>
<td>43-47</td>
<td>1.5-4.5mg</td>
<td>0.5-1.7mg</td>
<td>1.5-2mg</td>
</tr>
<tr>
<td>Grass</td>
<td>2.5-5</td>
<td>45-47</td>
<td>1-3mg</td>
<td>0.4-1.7mg</td>
<td>1-2mg</td>
</tr>
<tr>
<td>Soil – low organic</td>
<td>0.1-0.15</td>
<td>0.8-1.2</td>
<td>10-50mg</td>
<td>25-60mg</td>
<td>20-25mg</td>
</tr>
<tr>
<td>Soil – medium organic</td>
<td>0.2-0.3</td>
<td>2.5-3.5</td>
<td>10-20mg</td>
<td>8-23mg</td>
<td>10-23mg</td>
</tr>
<tr>
<td>Soil – high organic</td>
<td>0.3-1</td>
<td>10-14</td>
<td>7-15mg</td>
<td>2-5.7mg</td>
<td>5-7mg</td>
</tr>
<tr>
<td>Sediment</td>
<td>0.05-0.15</td>
<td>0.5-1.5</td>
<td>40-60mg</td>
<td>40-54mg</td>
<td>40-60mg</td>
</tr>
<tr>
<td>Animal Tissue</td>
<td>~10%</td>
<td>~40%</td>
<td>0.5-1.5mg</td>
<td>0.5-2mg</td>
<td>0.5-1.5mg</td>
</tr>
<tr>
<td>Hair</td>
<td>~14%</td>
<td>~47%</td>
<td>0.5-1mg</td>
<td>0.4-1.7mg</td>
<td>0.5-1.5mg</td>
</tr>
</tbody>
</table>

Note: Dual $^{15}$N and $^{13}$C can generally be determined on a single sample if the %N:%C ratio is less than 40
Elemental Analysis (EA) for % C, H, N, S and O

Our three CHNS/O elemental analysers routinely determine carbon, hydrogen, nitrogen, oxygen and sulphur in a wide range of organic and inorganic materials. OEA Labs analyse these elements from high percentages down to less than 0.01% on solid, liquid or even gaseous samples. We also determine total organic carbon (TOC), total inorganic carbon (TIC) and protein.

Elemental analysis (also known as elemental microanalysis) is a reliable and cost-effective technique used to assess the purity and chemical composition of research compounds or to provide information of the composition of materials.

In percentage elemental analysis (EA) of CHNSO, milligram amounts of samples are combusted or pyrolysed at high temperature in a helium carrier gas. After suitable preparation the measurable gases (CO$_2$, H$_2$O, N$_2$, SO$_2$, or CO) are separated on a chromatography column. The gases are passed in turn through a thermal conductivity detector (TCD) where the gases are quantified against known reference standards.

CHN This is probably the most common elemental analyser configuration which determines total carbon, hydrogen and nitrogen simultaneously. It is well suited to the analysis of most organics and inorganics from 0.01% to high percentage levels. The CHN method is limited to the analysis of relatively small sample weights because the resolution of the nitrogen and carbon peaks can be compromised.

CHNS The CHNS mode for elemental analysers determines total carbon, hydrogen, nitrogen and sulphur simultaneously from 0.01% (0.05% for sulphur) to high percentage levels. It is ideal for the analysis of most sulphur compounds and is widely used in the analysis of soils, sludges and plant material. As with the CHN method the analysis for CHNS is limited to relatively small sample weights because the resolution of the nitrogen and carbon peaks. Where sulphur is required at levels of less than 0.05% the sulphur method should be selected which allows the use of even larger samples or the more sensitive flame photometric detector (FPD).

NC The NC mode determines total nitrogen and carbon simultaneously from 0.001% (depending on sample types and weights) to high percentage levels. The method is reliable for almost all organic and inorganic compounds and is ideally suited to the analysis of plant material, animal tissue and soil samples. Unlike CHN or CHNS analysis the NC method has almost unlimited resolution of the nitrogen and carbon peaks which allows the analysis of much larger samples.

N The N mode determines total nitrogen from 0.001% (depending on sample types and weights) to high percentage levels. Protein can also be determined by this method. The use of large combustion tubes fitted to the elemental analyser allows protein determination on higher weights. The method is reliable for almost all organic and inorganic compounds and is ideally suited to the analysis of plant material, animal tissue and soil samples. The N method has almost unlimited resolution of the nitrogen which allows the analysis of much larger samples.

NCS The NCS mode determines total nitrogen, carbon and sulphur simultaneously from 0.01% (0.05% for sulphur) to high percentage levels. The method is reliable for almost all organic and inorganic compounds and is often applied to the analysis of plant material, animal tissue and soil samples where hydrogen analysis is not required. The method has some of the drawbacks of the CHNS method but can run slightly larger samples as it is not prone to water saturation. Where sulphur is required at levels of less than 0.05% the sulphur method should be selected which allows the use of even larger samples or the more sensitive flame photometric detector (FPD).

S The S mode determines total sulphur from a few 10’s of parts per million to high percentage levels depending on the sample type and the detection system used in the elemental analyser. The method is reliable for almost all organic and inorganic compounds and is often applied to the analysis of oils, minerals, plant material, animal tissue and soil samples. The method has none of the drawbacks of the CHNS and NCS methods so it can run larger samples. Where the S values are likely to be very low (in soils for example) it is possible to use higher sample weights, increase the gain of the instrument to achieve a better signal to noise ratio and/or switch to the more sensitive flame photometric detector (FPD).

O The O mode determines total oxygen from 0.01% (depending on sample types and weights) to high percentage levels. The method is generally applied to organic compounds but it can determine oxygen in many inorganic materials. The O method has almost unlimited resolution of the oxygen peak which allows the analysis of larger samples when required. Where the oxygen values are likely to be very low it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio.

TOC/TIC If inorganic carbon is present it can be eliminated by direct acidification with dilute hydrochloric acid or indirectly by exposure to concentrated sulphurous acid vapour. Where samples are submitted for total organic carbon (TOC) we will pre-treat your samples in this way to remove any inorganic carbon. Total inorganic carbon (TIC) is determined by difference from the total carbon and total organic carbon values.
Sample Preparation for % CHNSO Elemental Analysis

One of the great advantages of elemental analysis is the relatively small sample weights that are required for analysis. The sample weights used will vary according to the application, the sample type, detection limits and precision that needs to be achieved. Detection limits will normally be in the order of 0.01% or better but lower detection levels or precision can often be achieved by changing the analytical mode, increasing the sample weight and/or increasing the gain to achieve a better signal to noise ratio.

Please see below our weight guide to the sample sizes we require (per run) for each type of determination. Larger samples are preferred because our analysts must be able to recover sufficient sample from the vial to transfer into small capsules. It is very useful for us to have more sample to allow us to run random QC check samples. This also ensures that we can keep our sample turn-around on schedule should we have any problems with our instrumentation or encounter other unforeseen failures. We can return unused sample upon request.

Sample preparation might be required to obtain the best representation at the recommended weight levels. Unless otherwise requested samples are analysed 'as received'. Samples must be free from residual solvents or moisture to provide reliable results. Depending upon the sample type, samples should be finely ground using a mortar & pestle or ball mill to ensure that the sample is representative at the target sample weight.

If inorganic carbon is present it can be eliminated by direct acidification with dilute hydrochloric acid or indirectly by exposure to concentrated sulphurous acid vapour. Where samples are submitted for total organic carbon we will pre-treat your samples in this way to remove any inorganic carbon. Total inorganic carbon (TIC) is determined by difference from the total carbon and total organic carbon values.

We are able to prepare or pre-treat your samples at additional cost. We are able to accurately determine loss of weight on drying (on a micro scale) where required. Please contact us to discuss any aspects of sample preparation that you may require.

<table>
<thead>
<tr>
<th>Material / Method</th>
<th>CHN</th>
<th>CHNS</th>
<th>NC</th>
<th>NCS</th>
<th>N</th>
<th>O</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organic Compounds</td>
<td>0.4-2.5mg</td>
<td>1-2.5mg</td>
<td>0.4-14mg</td>
<td>1-7mg</td>
<td>1-20mg</td>
<td>0.5-10mg</td>
<td>0.4-2.5mg</td>
</tr>
<tr>
<td>Plant Material</td>
<td>0.4-3.3mg</td>
<td>2-3.3mg</td>
<td>0.4-18mg</td>
<td>2-9mg</td>
<td>1-60mg</td>
<td>0.3-5mg</td>
<td>10-60mg</td>
</tr>
<tr>
<td>Wood</td>
<td>0.6-3.8mg</td>
<td>2-3.8mg</td>
<td>0.6-20mg</td>
<td>2-10mg</td>
<td>15-60mg</td>
<td>0.3-5mg</td>
<td>10-60mg</td>
</tr>
<tr>
<td>Grain Flour</td>
<td>0.4-3.3mg</td>
<td>2-3.3mg</td>
<td>0.4-18mg</td>
<td>2-9mg</td>
<td>1-50mg</td>
<td>0.3-5mg</td>
<td>2.5-50mg</td>
</tr>
<tr>
<td>Soil - low organic</td>
<td>8-60mg</td>
<td>15-60mg</td>
<td>8-60mg</td>
<td>15-60mg</td>
<td>7-60mg</td>
<td>5-60mg</td>
<td>25-60mg</td>
</tr>
<tr>
<td>Soil - medium organic</td>
<td>3-40mg</td>
<td>10-40mg</td>
<td>3-60mg</td>
<td>10-60mg</td>
<td>4-60mg</td>
<td>2.5-50mg</td>
<td>2-60mg</td>
</tr>
<tr>
<td>Soil - high organic</td>
<td>1-10mg</td>
<td>5-10mg</td>
<td>1-50mg</td>
<td>5-20mg</td>
<td>2-60mg</td>
<td>1.7-30mg</td>
<td>1.2-0mg</td>
</tr>
<tr>
<td>Sediment</td>
<td>7-60mg</td>
<td>10-60mg</td>
<td>7-60mg</td>
<td>10-60mg</td>
<td>1-13mg</td>
<td>2.5-50mg</td>
<td>2.5-50mg</td>
</tr>
<tr>
<td>Animal Tissue</td>
<td>0.6-3.8mg</td>
<td>2-3.8mg</td>
<td>0.6-20mg</td>
<td>2-10mg</td>
<td>1-25mg</td>
<td>0.3-5mg</td>
<td>1.2-0mg</td>
</tr>
<tr>
<td>Hair</td>
<td>0.4-2.5mg</td>
<td>1-2.5mg</td>
<td>0.4-17mg</td>
<td>1-8mg</td>
<td>1-15mg</td>
<td>0.3-7mg</td>
<td>0.4-2mg</td>
</tr>
</tbody>
</table>

Petrochemical: coal/coke, oils, gasolines, tars, petrochemicals & lubricants
Geological: kerogens, soils, sediments, shale, cement & clays
Medical: blood, hair, nails, vaccines, serum & urine
Inorganics: carbon fibres, catalysts, refractories, graphite & chemicals
Organics: polymers, resins, detergents, chemicals, paint & rubber
Plant Materials: wood, cotton, paper, leaves, stems, roots & seeds
Environmental: contaminants, sludges, water, fertilisers, soils & composts
Food & Feed: flour, cereals, feeds, ingredients, milk, sugars & meat
Competitor Products: ingredients and composition
Elemental Analysis (EA) for % F, Cl, Br, I and P

Hydro-combustion, acid digestion and oxygen flask techniques are employed to prepare samples for analysis by Ion Chromatography (IC), UV/Vis spectroscopy, titration and ion specific techniques to determine total fluorine, chlorine, bromine, iodine and phosphorus in both organic and inorganic samples. OEA Labs quantify these elements from percentage levels down to parts per million. We also analyse numerous anions such as nitrite, nitrate, sulphite, sulphate, phosphite and phosphate and undertake other non-routine analysis.

The analysis of the halogens (fluorine, chlorine, bromine and iodine) and phosphorus in organic compounds has always been of interest to the organic chemist. When combined with other elemental data such as the percentages of C, H, N and S the overall purity of a compound can be assessed.

There are many techniques available to analyse these elements. Traditional micro methods such as oxygen flask combustion followed by titration, UV/Vis spectroscopy or ion selective electrode analysis are reliable and accurate for pure materials but are prone to interferences or suppression with ‘real world’ samples. For example, the analysis of chlorine, bromine or iodine by titration is widely used but the titrimetric analysis of these elements in admixture is not possible. The use of ion selective electrodes is also prone to errors with mixtures of these anions.

We have largely replaced these traditional methods with modern ion chromatography which is very specific, has a wide working range and is generally free from interferences. The analysis of fluorine is particularly reliable with this technique. Although relatively slow, ion chromatography is a powerful technique in the determination of a multitude of anions especially when used in conjunction with hydropyrolysis combustion sample preparation.

We are able to analyse many other anions with ion chromatography such as nitrite, nitrate, sulphite, sulphate, phosphite and phosphate. Please contact us to discuss your requirements.

Sample Preparation for % F, Cl, Br, I and P Elemental Analysis

Relatively small samples weights are required for analysis. The sample weights used will vary according to the application, the sample type, detection limits and precision that needs to be achieved.

For analysis at percentage levels sample sizes of 5 to 10 milligrams per analysis are adequate. For trace levels below 0.05% sample weights must be increased to 50 to 100 milligrams.

Where possible please submit more sample than required. This greatly assists our technicians to recover and transfer material into small capsules and allows us to run random QC check samples.

Sample preparation might be required to obtain the best representation at the recommended weight levels. Unless otherwise requested samples are analysed ‘as received’.

Samples must be free from residual solvents or moisture to provide reliable results. Depending upon the sample type, samples should be dehydrated by freeze drying or vacuum oven drying to constant weight. Samples should be finely ground using a mortar & pestle or a ball mill to ensure that the sample is representative at the target sample weight.

We are able to prepare or pre-treat your samples at additional cost. We are able to accurately determine loss of weight on drying (on a micro scale) where required. Please contact us to discuss any aspects of sample preparation that you may require.
Appendix

Appendix I - About Stable Isotopes

An atom is a basic unit of matter which contains a nucleus of protons and neutrons surrounded by a cloud of electrons. The number of protons in the nucleus defines the chemical element.

An isotope is an atom of a specific element that differs in atomic mass. This mass difference is due to a variation in the number of neutrons within the nucleus. For example, there are three isotopes of carbon – Carbon 12, 13 and 14. Carbon-12 (written as 12C) has 6 protons, 6 neutrons and 6 electrons. 13C has 6 protons and electrons but has 7 neutrons. Similarly, 14C has 6 protons and electrons with 8 neutrons.

Having fewer or more neutrons than protons in an atom can cause some isotopes (such as 13C) to be unstable. Such ‘radioisotopes’ as they are called will decay over a period of time to more stable forms. 12C and 13C do not decay because their particular combination of neutrons and protons are stable. These isotopes are referred to as stable isotopes.

The stable isotopes of nitrogen, carbon, sulphur, oxygen and hydrogen are referred to as light stable isotopes. These stable isotopes are of considerable interest in scientific research.

Stable isotope ratios in the environment can be influenced by natural processes involving biological, chemical and other physical transformations. This is known as isotope fractionation. Oxygen isotopes, for instance, can fractionate across continents as a result of meteorological processes, temperature, elevation and the distance from water bodies. From such information it is possible to determine water sources and age. The use of stable isotope analysis techniques provides a unique means of following these processes quantitatively and is often referred to as natural abundance isotope techniques.

Light isotope ratios in plant/animal matter, minerals/rocks and water can vary according to their geographical location. This is an interesting application of isotope data to ‘fingerprint’ origins of materials. Light isotope ratios have been deflected by the magnet. The heavier isotope molecules are deflected less than those containing the lighter isotopes. A strong magnet positioned over the flight tube deflects the path of the molecules according to their individual masses. The heavier isotope molecules are deflected less than those containing the lighter isotopes. A series of Faraday collectors at the end of the flight tube measures the intensity of each beam of ionised molecules which have been deflected by the magnet.

The mass spectrometer can be ‘tuned’ to different masses of gas molecules. In practice where a mass spectrometer is fitted with a fixed radius flight tube and a permanent magnet, this is accomplished by varying the acceleration voltage of the ion source. In systems fitted with electromagnets the magnetic field intensity can be varied.

As an example, nitrogen as N2 will produce masses of 28 (14N 14N), 29 (14N 15N) and 30 (15N 15N). Similarly carbon as CO2 will produce the common masses of 44 (12C 16O 16O), 45 (12C 16O 18O) and 46 (12C 18O 18O) although other variants (ie 12C 17O 16O) will also exist in small amounts but these small errors are corrected by the use of the Craig Algorithm. In most cases nitrogen and carbon can be determined from a single sample combustion because the gases produced are separated by the GC column. Nitrogen is eluted first followed by the carbon dioxide. The mass spectrometer ion source voltage is changed between the elution peaks so the isotopes of each gas align with the appropriate Faraday collector in turn.

Appendix II - Analysis of Stable Isotopes

The stable isotopes of nitrogen, carbon, sulphur, oxygen and hydrogen are analysed by converting a sample into a gas by combustion or pyrolysis (ie N2, CO2, SO2, CO or H2) and measuring the stable isotopes in the gaseous phase by mass spectrometry. This technique is referred to as Isotope Ratio Mass Spectrometry (IRMS), Elemental Analysis Isotope Ratio Mass Spectrometry (EA/IRMS) or Continuous Flow Isotope Ratio Mass Spectrometry (CF/IRMS).

The gases produced by the elemental analyser are carried by a helium stream into a gas chromatography (GC) column where the components (N2 and CO2 for example) are separated. These separated gases are then conveyed in series through a splitter/isolating valve on the mass spectrometer. A small portion of the gas is ‘sniffed’ or sucked into the mass spectrometer which is differentially pumped to a high vacuum by means of turbo and roughing vacuum pumps.

When this small portion of the gas enters the mass spectrometer it is ionised by the ion source. An electron is stripped from the gas molecule causing each molecule to become positively charged. These charged molecules are accelerated by high voltage, focussed and ‘fired’ into the flight tube which is curved to a specific radius. A strong magnet positioned over the flight tube deflects the path of the molecules according to their individual masses. The heavier isotope molecules are deflected less than those containing the lighter isotopes. A series of Faraday collectors at the end of the flight tube measures the intensity of each beam of ionised molecules which have been deflected by the magnet.

The mass spectrometer can be ‘tuned’ to different masses of gas molecules. In practice where a mass spectrometer is fitted with a fixed radius flight tube and a permanent magnet, this is accomplished by varying the acceleration voltage of the ion source. In systems fitted with electromagnets the magnetic field intensity can be varied.

Appendix III - Calibration of Isotope Ratio Mass Spectrometers

We calibrate our Isotope Ratio Mass Spectrometer systems by the following methods:

Primary calibration of IRMS systems is achieved by the use of international reference standards which are traceable to internationally accepted standards.

Secondary calibration of IRMS systems involves the use of control/working standards which are measured along with a series of unknown samples to verify the accuracy of a run sequence. The choice of which control/working standard to use will depend upon the sample types in a sequence. These secondary control/working standards have been certified against primary international standards.

Tertiary calibration of IRMS systems is provided by the injection of high purity reference gases such as nitrogen, carbon dioxide or sulphur dioxide of known isotopic values at the analysis time of each sample. This method provides a powerful tool to monitor the integrity of IRMS systems.

The performance of the elemental analyser (EA) or sample preparation system is monitored by quantifying the output of the EA thermal conductivity detector (TCD) and comparison to primary or secondary standards traceable to internationally accepted elemental analysis standards.
Appendix IV - Results for Stable Isotopes

Stable isotope analysis results are expressed as abundance ratios. These are expressed as the ratio of the two most abundant isotopes (heavy vs. light) in the sample compared to an internationally recognised sample using the ‘delta’ notation (δ). These ratios are very small and are expressed as parts per thousand or ‘per mil’ (%d) deviation from the standard.

For example carbon would be expressed as:

\[ \delta^{13}C = \left( \frac{^{13}C/^{12}C \text{ sample}}{^{13}C/^{12}C \text{ standard}} \right) - 1 \times 1000 \]

The standard is defined as 0‰ (zero per mil). In the case of carbon the international standard is Pee Dee Belemnite with an accepted value of \(^{13}C/^{12}C\) of 0.0112372. Samples with ratios of \(^{13}C/^{12}C\) greater than 0.0112372 have positive delta (δ) values and samples with ratios of \(^{13}C/^{12}C\) less than 0.0112372 have negative delta values. There can be more than one international standard for some elements.

Appendix V - CHN

This is probably the most common elemental analyser configuration which determines total carbon, hydrogen and nitrogen simultaneously. It is well suited to the analysis of organics and inorganics from 0.01% to high percentage levels. The CHN method is reliable for almost all organic and inorganic compounds.

The CHN method is limited to the analysis of relatively small sample weights because the resolution of the nitrogen and carbon peaks can be compromised. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the CHN values are likely to be very low it is possible to use higher sample weights and increase the gain of the instrument to achieve a better signal to noise ratio.

Solid samples are precisely weighed and sealed into tin capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to prevent the possible formation of metal or silicon carbides and to assist the combustion of some materials such as organometallics or inorganics.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into tin capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analysers autosampler carousel. The autosampler purges the sample chamber with oxygen to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started this helium carrier is replaced briefly by a measured dose of high purity oxygen. After a few seconds the oxygen dose arrives at the combustion zone in the upper half of the combustion tube operating at 1000°C. The autosampler drops the encapsulated sample into the combustion tube to meet the oxygen. The sample is burned instantaneously followed by the oxidation of the tin capsule giving a localised temperature in excess of 1800°C for a few seconds. This technique is known as flash combustion.

The resulting combustion gases are carried over a catalyst (chromium oxide) and an absorber (silvered cobaltous/cis oxide) in the lower half of the combustion tube. These materials ensure complete oxidation of the gases and also remove unwanted oxides of sulphur, halogens and other gaseous interfer- ences.

The gases are swept by the helium carrier into the reduction tube at 670°C. The reduction tube contains reactive copper which removes excess oxygen and also reduces any oxides of nitrogen that might have been formed.

A gas chromatography (GC) column separates and elutes the gaseous constituents into three peaks - elemental nitrogen (Nitrogen), carbon dioxide (Carbon) and water vapour (Hydrogen) which are quantified by a thermal conductivity detector (TCD). The peak responses are identified and integrated by the elemental analyser software and compared to known reference compounds traceable to international standards.

Appendix VI - CHNS

The CHNS mode for elemental analysers determines total carbon, hydrogen, nitrogen and sulphur simultaneously from 0.01% (0.05% for sulphur) to high percentage levels. It is ideal for the analysis of sulphur compounds and is widely used in the analysis of soils, sludges and plant material.

The CHNS method is reliable for almost all organic and inorganic compounds. The occasional analysis of fluorine compounds is quite successful but the repeated combustion of fluorine compounds attacks the silica combustion tube and leads to the gradual deterioration of the hydrogen value.

The CHNS method is limited to the analysis of relatively small sample weights because the resolution of the nitrogen and carbon peaks can be compromised. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the CHNS values are likely to be very low (in soils for example) it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio. Where sulphur is required at levels of less than 0.05% the sulphur method should be selected which allows the use of even larger samples or the more sensitive flame photometric detector (FPD).

Solid samples are precisely weighed and sealed into tin capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to prevent the possible formation of metal or silicon carbides and to assist the combustion of some materials such as organometallics or inorganics.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into tin capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with oxygen to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started this helium carrier is replaced briefly by a measured dose of high purity oxygen. After a few seconds the oxygen dose arrives at the combustion zone in the upper half of the combustion tube operating at 1000°C. The autosampler drops the encapsulated sample into the combustion tube to meet the oxygen. The sample is burned instantaneously followed by the oxidation of the tin capsule giving a localised temperature in excess of 1800°C for a few seconds. This technique is known as flash combustion.

The resulting combustion gases are carried over a catalyst (pure tungstic oxide) and high purity reactive copper in the lower half of the reaction tube. These materials ensure complete oxidation of the gases, remove excess oxy-
gen and other gaseous interferences and reduce any oxides of nitrogen that might have been formed.

A gas chromatography (GC) column separates and elutes the gaseous constituents into four peaks - elemental nitrogen (Nitrogen), carbon dioxide (Carbon), water vapour (Hydrogen) and sulphur dioxide (Sulphur) which are quantified by a thermal conductivity detector (TCD). The peak responses are identified and integrated by the elemental analyser software and compared to known reference compounds traceable to international standards.

**Appendix VII - NC**

The NC mode determines total nitrogen and carbon simultaneously from 0.001% (depending on sample types and weights) to high percentage levels.

The method is reliable for almost all organic and inorganic compounds and is ideally suited to the analysis of plant material, animal tissue and soil samples.

Unlike CHN or CHNS analysis modes the NC method has almost unlimited resolution of the nitrogen and carbon peaks which allows the analysis of much larger samples. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the NC values are likely to be very low (in soils for example) it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio.

Solid samples are precisely weighed and sealed into tin capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to prevent the possible formation of metal or silicon carbides and to assist the combustion of some materials such as organometallics or inorganics.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into tin capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with oxygen to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started this helium carrier is replaced briefly by a measured dose of high purity oxygen. After a few seconds the oxygen dose arrives at the combustion zone in the upper half of the combustion tube operating at 1000°C. The autosampler drops the encapsulated sample into the combustion tube to meet the oxygen. The sample is burned instantaneously followed by the oxidation of the tin capsule giving a localised temperature in excess of 1800°C for a few seconds. This technique is known as flash combustion.

The resulting combustion gases are carried over a catalyst (chromium oxide) and an absorber (silvered cobaltous/iodic oxide) in the lower half of the combustion tube. These materials ensure complete oxidation of the gases and also remove unwanted oxides of sulphur, halogens and other gaseous interferences.

The gases are swept by the helium carrier into the reduction tube at 670°C. The reduction tube contains reactive copper which removes excess oxygen and also reduces any oxides of nitrogen that might have been formed.

Water is removed from the gas stream by means of a drying tube which contains ecdronate or magnesium perchlorate.

A high efficiency gas chromatography (GC) column separates and elutes the gaseous constituents into two distinct peaks - elemental nitrogen (Nitrogen) and carbon dioxide (Carbon) which are quantified by a thermal conductivity detector (TCD). The peak responses are identified and integrated by the elemental analyser software and compared to known reference compounds traceable to international standards.

**Appendix VIII - N or Protein**

The N mode determines total nitrogen from 0.001% (depending on sample types and weights) to high percentage levels. Protein can also be determined by this method. The use of large combustion tubes fitted to the elemental analyser allows protein determination on higher weights.

The method is reliable for almost all organic and inorganic compounds and is ideally suited to the analysis of plant material, animal tissue and soil samples.

The N method has almost unlimited resolution of the nitrogen which allows the analysis of much larger samples. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the N values are likely to be very low it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio.

Solid samples are precisely weighed and sealed into tin capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives are not required for the analysis of nitrogen.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into tin capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with oxygen to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started this helium carrier is replaced briefly by a measured dose of high purity oxygen. After a few seconds the oxygen dose arrives at the combustion zone in the upper half of the combustion tube operating at 1000°C. The autosampler drops the encapsulated sample into the combustion tube to meet the oxygen. The sample is burned instantaneously followed by the oxidation of the tin capsule giving a localised temperature in excess of 1800°C for a few seconds. This technique is known as flash combustion.

The resulting combustion gases are carried over a catalyst (chromium oxide) and an absorber (silvered cobaltous/iodic oxide) in the lower half of the combustion tube. These materials ensure complete oxidation of the gases and also remove unwanted oxides of sulphur, halogens and other gaseous interferences. The use of platinumised alumina and copper oxide is sometimes used for protein analysis.

The gases are swept by the helium carrier into the reduction tube at 670°C. The reduction tube contains reactive copper which removes excess oxygen.
and also reduces any oxides of nitrogen that might have been formed.

Carbon dioxide is removed from the gas stream by means of a scrubbing tube containing soda lime. Similarly water is removed in a drying tube which contains edecrone or magnesium perchlorate.

A gas chromatography (GC) column separates and elutes a single constituent - elemental nitrogen (Nitrogen) which is quantified by a thermal conductivity detector (TCD). The peak response is identified and integrated by the elemental analyser software and compared to known reference compounds traceable to international standards.

Appendix IX - NCS

The NCS mode determines total nitrogen, carbon and sulphur simultaneously from 0.01% (0.05% for sulphur) to high percentage levels.

The method is reliable for almost all organic and inorganic compounds and is often applied to the analysis of plant material, animal tissue and soil samples where hydrogen analysis is not required.

The method has some of the drawbacks of the CHNS method but can run slightly larger samples as it is not prone to water saturation. The resolution of the nitrogen and carbon peaks can be compromised when running larger organic samples but this is not such an issue as with CHNS analysis. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the NCS values are likely to be very low (in soils for example) it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio.

Solid samples are precisely weighed and sealed into tin capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to prevent the possible formation of metal or silicon carbides when required to assist the combustion of some materials such as organometallics or inorganics.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into silver capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with oxygen to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started the autosampler drops the encapsulated sample into the pyrolysis tube. The sample is instantaneously pyrolysed.

The resulting pyrolysed gases are carried over a catalyst (nickelised carbon granules) in the lower half of the combustion tube. This material ensures complete conversion of any oxygen gases into carbon monoxide.

A gas chromatography (GC) column separates and elutes the carbon monoxide (Oxygen) which is quantified by a thermal conductivity detector (TCD). The peak responses are identified and integrated by the elemental analyser software and compared to known reference compounds traceable to international standards.

Appendix X - O

The O mode determines total oxygen from 0.01% (depending on sample types and weights) to high percentage levels.

The method is generally applied to organic compounds but it can determine oxygen in inorganic materials. Although many metal oxides can be determined, the analysis of refractory materials such as silica or alumina cannot be reduced under the operating conditions of the system. Samples containing fluoride do not give reliable results due to fluoride attack of the silica reaction tube. Phosphorus can also interfere due to the formation of thermally stable phosphates.

The O method has almost unlimited resolution of the oxygen peak which allows the analysis of larger samples when required. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the oxygen values are likely to be very low it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio.

Solid samples are precisely weighed and sealed into silver capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to assist the reduction of many metallic oxides.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into silver capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with helium to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started the autosampler drops the encapsulated sample into the pyrolysis tube operating at 1060°C. The sample is instantaneously pyrolysed.

The resulting pyrolysed gases are carried over a catalyst (nickelised carbon granules) in the lower half of the combustion tube. This material ensures complete conversion of any oxygen gases into carbon monoxide.

The method is reliable for almost all organic and inorganic compounds and is often applied to the analysis of oils, minerals, plant material, animal tissue and soil samples.

The method has none of the drawbacks of other sulphur methods (CHNS and NCS) so it can run larger samples. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the S values are likely to be very low (in soils for example) it is possible to use higher sample weights, increase the gain of the instrument to achieve a better signal to noise ratio and/or switch to the more sensitive flame photometric detector (FPD).

Solid samples are precisely weighed and sealed into tin capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to assist the combustion of some materials such as organometallics or inorganics.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into tin capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with helium to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started the autosampler drops the encapsulated sample into the pyrolysis tube operating at 1060°C. The sample is instantaneously pyrolysed.

The resulting pyrolysed gases are carried over a catalyst (nickelised carbon granules) in the lower half of the combustion tube. This material ensures complete conversion of any oxygen gases into carbon monoxide.

The method is generally applied to organic compounds but it can determine oxygen in inorganic materials. Although many metal oxides can be determined, the analysis of refractory materials such as silica or alumina cannot be reduced under the operating conditions of the system. Samples containing fluoride do not give reliable results due to fluoride attack of the silica reaction tube. Phosphorus can also interfere due to the formation of thermally stable phosphates.

The O method has almost unlimited resolution of the oxygen peak which allows the analysis of larger samples when required. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the oxygen values are likely to be very low it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio.

Solid samples are precisely weighed and sealed into silver capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to assist the reduction of many metallic oxides.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into silver capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with helium to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started the autosampler drops the encapsulated sample into the pyrolysis tube operating at 1060°C. The sample is instantaneously pyrolysed.

The resulting pyrolysed gases are carried over a catalyst (nickelised carbon granules) in the lower half of the combustion tube. This material ensures complete conversion of any oxygen gases into carbon monoxide.

The method is reliable for almost all organic and inorganic compounds and is often applied to the analysis of oils, minerals, plant material, animal tissue and soil samples.

The method has none of the drawbacks of other sulphur methods (CHNS and NCS) so it can run larger samples. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the S values are likely to be very low (in soils for example) it is possible to use higher sample weights, increase the gain of the instrument to achieve a better signal to noise ratio and/or switch to the more sensitive flame photometric detector (FPD).

Solid samples are precisely weighed and sealed into tin capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to assist the combustion of some materials such as organometallics or inorganics.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into tin capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with helium to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started the autosampler drops the encapsulated sample into the pyrolysis tube operating at 1060°C. The sample is instantaneously pyrolysed.

The resulting pyrolysed gases are carried over a catalyst (nickelised carbon granules) in the lower half of the combustion tube. This material ensures complete conversion of any oxygen gases into carbon monoxide.

The method is generally applied to organic compounds but it can determine oxygen in inorganic materials. Although many metal oxides can be determined, the analysis of refractory materials such as silica or alumina cannot be reduced under the operating conditions of the system. Samples containing fluoride do not give reliable results due to fluoride attack of the silica reaction tube. Phosphorus can also interfere due to the formation of thermally stable phosphates.

The O method has almost unlimited resolution of the oxygen peak which allows the analysis of larger samples when required. See the Weight Requirement Guide for Sample Submissions Table earlier in this section. Where the oxygen values are likely to be very low it is possible to use higher sample weights and/or increase the gain of the instrument to achieve a better signal to noise ratio.

Solid samples are precisely weighed and sealed into silver capsules. Microbalances provide milligram sample weights accurate to four decimal places (seven decimal places of a gram). Additives can be added to the sample capsule when required to assist the reduction of many metallic oxides.

Low viscosity liquid samples are directly injected into the elemental analyser using a liquid injection autosampler. High viscosity liquid samples can be sealed anaerobically into silver capsules. Gaseous samples are introduced into the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with helium to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started the autosampler drops the encapsulated sample into the pyrolysis tube operating at 1060°C. The sample is instantaneously pyrolysed.

The resulting pyrolysed gases are carried over a catalyst (nickelised carbon granules) in the lower half of the combustion tube. This material ensures complete conversion of any oxygen gases into carbon monoxide.
the elemental analyser by means of a fixed volume loop or by direct injection.

The weighed and encapsulated samples are loaded into the elemental analyser autosampler carousel. The autosampler purges the sample chamber with oxygen to exclude air and provides a means of introducing the sample into the combustion furnace tube without pressure loss.

A controlled flow of pure helium (carrier gas) is maintained through the elemental analyser analytical circuit. When the elemental analyser sequence is started this helium carrier is replaced briefly by a measured dose of high purity oxygen. After a few seconds the oxygen dose arrives at the combustion zone in the upper half of the combustion tube operating at 1000°C. The autosampler drops the encapsulated sample into the combustion tube to meet the oxygen. The sample is burned instantaneously followed by the oxidation of the tin capsule giving a localised temperature in excess of 1800°C for a few seconds. This technique is known as flash combustion.

The resulting combustion gases are carried over a catalyst (pure tungstic oxide) and high purity reactive copper in the lower half of the reaction tube. These materials ensure complete oxidation of the gases, remove excess oxygen and other gaseous interferences and reduce any oxides of nitrogen that might have been formed.

Water is removed from the gas stream by means of a drying tube which contains magnesium perchlorate.

A gas chromatography (GC) column separates and elutes the sulphur dioxide (Sulphur) which is quantified by a thermal conductivity detector (TCD) or flame photometric detector (FPD). The peak response is identified and integrated by the elemental analyser software and compared to known reference compounds traceable to international standards.

Appendix XII - Analysis of Ions by Ion Chromatography

The sample is combusted in a sealed oxygen flask or a hydropyrolysis combustion furnace. The combustion gases are absorbed in a known volume of absorption reagent. Inorganic materials can be taken into solution by dissolving, salt fusion, acid digestion or microwave digestion.

A two stage pump constantly pumps the eluent (usually a 9mM aqueous solution of sodium carbonate) at high pressure (~2000 psi) through the ion chromatography system.

The prepared sample solution is loaded into the sample loop (usually 25 microlitre capacity) from the autosampler.

When the analytical sequence is started the sample loop is switched into the eluent stream and the sample is injected into the analytical circuit.

The sample and eluent pass through a guard tube which removes contaminants that might poison the main separator column.

As the sample and eluent are pumped through the separator column (containing polymeric resins) the ions are separated by a process known as ion exchange. Different sample ions migrate through the ion chromatography column at different rates depending upon their interaction with the ion exchange sites.

After leaving the separator column the separated sample ions and eluent pass through a suppressor which selectively enhances the detection of sample ions while suppressing the conductivity of the eluent.

A thermostatically controlled conductivity cell measures the electrical conductance of the sample ions as they emerge in turn from the suppressor. This produces a signal (a peak) based on the chemical and physical properties of each individual analyte.

The data is processed by software which identifies the ions based on retention time and quantifies each analyte by integrating the peak area. The data is quantified by comparing the sample peak responses to those produced by known standard solutions traceable to international reference standards.
Terms and Conditions

1. Prices
Unless agreed in writing by OEA Laboratories, all quotations expire 30 days following the date of quotation. All prices are exclusive of VAT, duties and any other local taxes.

2. Handling and Use
Any chemicals supplied should be handled only by qualified individuals trained in laboratory procedures and familiar with their potential hazards. Some of the chemicals supplied may be extremely toxic, hazardous or flammable. Products are supplied on the condition that the customer accepts responsibility for any accident arising from their handling or use. All products are supplied for laboratory use only.

3. Disposal of Waste
The customer warrants that all chemicals and materials will be disposed of in a safe and environmentally friendly manner in line with current legislation.

4. COSHH
To comply with the customers own COSHH regulations, or similar such regulations, Material Safety Data Sheets (MSDS) are available for all appropriate products supplied by OEA Labs.

5. Payment Terms
OEA Laboratories payment terms are normally net 30 days unless otherwise quoted in writing. Credit or debit card payments will not be debited until your order is ready for dispatch or results reported.

6. Returns, Refunds or Incorrectly Ordered Goods
Our products have been developed and tested to perform to the highest standards in elemental microanalysis and isotope ratio mass spectrometry applications. All of our products are unconditionally guaranteed to meet your expectations. If we fail to meet your standards for whatever reason we will issue a refund in full. You can return such items within 14 days of delivery. Just call us and we will arrange collection free of charge and issue a full refund.

7. Cancellations
Orders may only be cancelled in writing unless otherwise agreed. Please telephone us in the first instance to discuss details. Cancellation of orders after the goods have been dispatched from OEA Laboratories can be accepted in legitimate circumstances but we may need to charge for the packing and shipping charges incurred.

8. Damaged Goods in Transit
In the event of goods being damaged during transit, OEA Laboratories must be informed within 7 days of receipt of the goods. When requested, damaged goods must be returned to OEA Laboratories for inspection before replacements are dispatched. Under no circumstances should a customer accept or sign for goods from a courier that are clearly damaged or potentially damaged. The customer must retain all packaging from damaged goods for inspection by an OEA Laboratories representative or a representative of the courier.

9. Title and Risk
Goods supplied by OEA Labs shall remain the property of, and be recovered by OEA Laboratories if any seals have been broken except by agreement.

10. Delivery
OEA Laboratories shall make every effort to meet any sample turn-around or delivery date quoted but shall not be liable for any loss or damage arising directly or indirectly from delay in delivery.

11. Shipping charges
All quotations given for international shipments are estimations given in good faith only. OEA Laboratories will invoice for such charges at cost and will provide proof of such charges if required. OEA Laboratories shall not be liable for additional charges incurred such as customs clearance, duty, local taxes and onward shipping charges.

12. Insolvency
In the event that the customer shall suffer insolvency, bankruptcy or a receiver be appointed, any goods that have not been paid for in full shall remain the property of, and be recovered by OEA Laboratories.

13. Public and Products Liability
Any claims against OEA Laboratories in respect of damage to property or persons shall be limited to the value of the contract.

14. Warranty
OEA Laboratories warrants its products against defects in materials, workmanship and design. No liability for loss or damage in excess of the purchase price of the goods will be accepted.

15. Severability and Waiver
In the event that one of these Terms and Conditions shall be found to be void, failure to enforce such condition shall not be regarded as a waiver of the remaining Terms and Conditions.

16. Relevant Law
These Terms and Conditions and the contracts to which they relate shall in all respects be governed and construed in accordance with the laws of England and Wales.

17. Acceptance
The placing of an order with OEA Laboratories implies acceptance of the terms and conditions.

18. Expert Testimony
OEA Laboratories does not provide Expert Testimony in legal proceedings.

19. Miscellaneous
The use of the term ‘OEA Laboratories’ or ‘OEA Labs’ in the above Terms and Conditions shall mean ‘OEA Laboratories Limited’.

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