costech instruments

ECS 4010

Elemental Combustion System
CHNS-O

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ECS 4010

Innovation in elemental analysis

The **Elemental Combustion System 4010** was designed as an advanced analytical platform for CHNS-O elemental analysis and Nitrogen/ Protein determination. It is based on an automatic analytical unit whose operation - from

sampling up to signal detection - is microprocessor controlled. It represents an evolution in the technique of elemental analysis by "flash combustion/chromatographic separation and multi-detector techniques".

Traditionally, automatic instruments for CHNS-O elemental analysis have been divided into two groups; micro and macro elemental analysis. Samples for **micro EA** are easily analyzed by sophisticated automatic instruments using the technique of combustion/gas chromatography.

For the **analysis of stable isotopes of CHNS-O**, a sampling system is required which does not contaminate the sample with atmospheric nitrogen and oxygen, especially working at very low levels. Advanced, highly stable catalysts are required to avoid any material exchange to the combusted gases.

To increase throughput and minimize maintenance time and cost, a simple approach to the design of the analyzer is essential, especially for macro sample analysis where all combustion gases pass through the detector system without splitting or diluting.

Instrument description

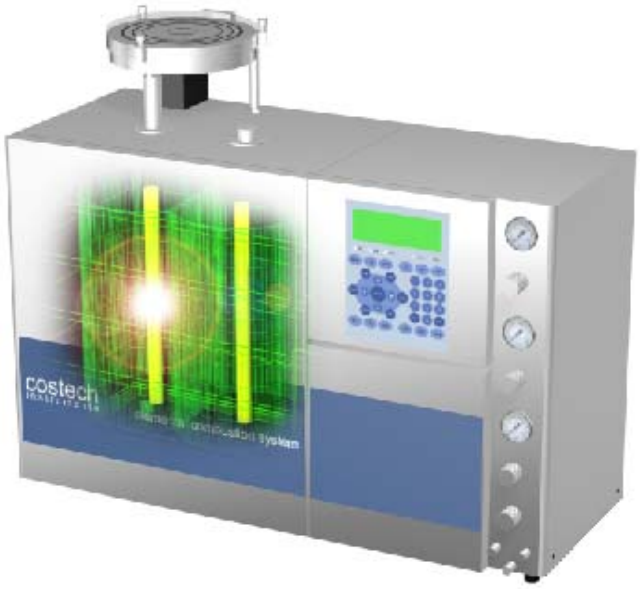
Starting from the basic furnace design; the ECS 4010 has two, large capacity (up to a 26 mm OD reactor) furnaces. Optional fittings let you quickly and easily switch between combustion/reduction reactors of different diameters. You can work with reactors from 10,5 mm to 26 mm OD.
The furnaces are mounted in a specially designed SS enclosure with an extremely efficient insulating material. This keeps the entire cabinet “cool to the touch” and allows the furnaces to heat from ambient to operating temperature in 1.5 hours. The electronics and power supplies are completely isolated from the furnace compartment and have their own separate cooling fan. The ECS monitors, and can display, the temperatures of the power supply, and electronics. It will also display the input line voltage supply.

The ECS 4010 consists of three modules;
The first module is the combustion system, the second is the detector system, and the third is the data handling system. When the three systems are combined the user can choose the required analytical configuration.

The first module consists of sampling, combustion and pneumatics. The autosamplers available are a sealed, “Zero Blank” sampler, and an open carousel sampler, where samples can be added continuously. The movement of the “Zero Blank” autosampler is controlled by a microstepping motor. You can choose either a 32 or 50 position carousel through the instrument software, and since it is completely sealed to atmosphere there is no contamination of the samples from nitrogen or oxygen. This is especially useful for stable isotope analysis. The second choice of sampler

has carousels which are open to the atmosphere, but samples can be added continuously and the number of sample positions is 147.

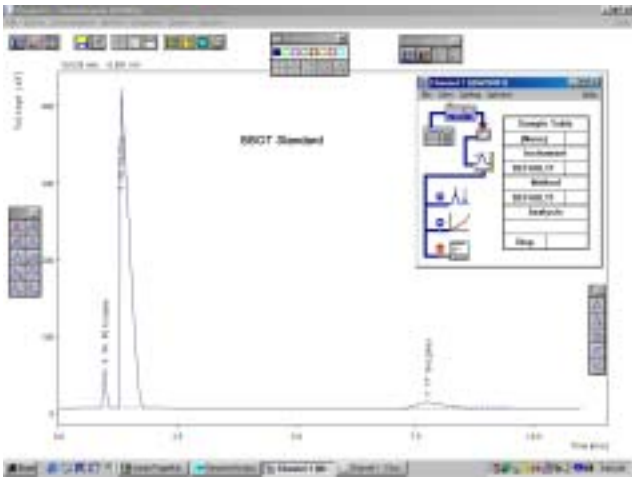
The ECS 4010 is equipped with two furnaces. Temperature control of the combustion furnace is up to 1100 ^{o}C, and the reduction furnace 800 ^{o}C.



The furnace dimensions and special fittings allows you to use a wide variety of reactors to tailor the analyzer to your laboratory needs (small and large samples, CHNS-O analysis, and N/Protein). The connection fittings for the quartz reactors make it fast and easy to replace the inserts in the combustion reactors (removal of ash and residue), for longer tube and catalyst life. The unique pneumatic system can be switched between three operating modes; Micro, Semi-Micro and Macro. By selecting a mode you choose the volume of oxygen for sample combustion. The gases (helium, oxygen and servo) are controlled by reliable SS diaphragm regulators for accurate flow control. Servo gas control is provided for use with our pneumatically actuated autosampler. A separate purge regulator is provided for flexibility in purge gas flow and volume.

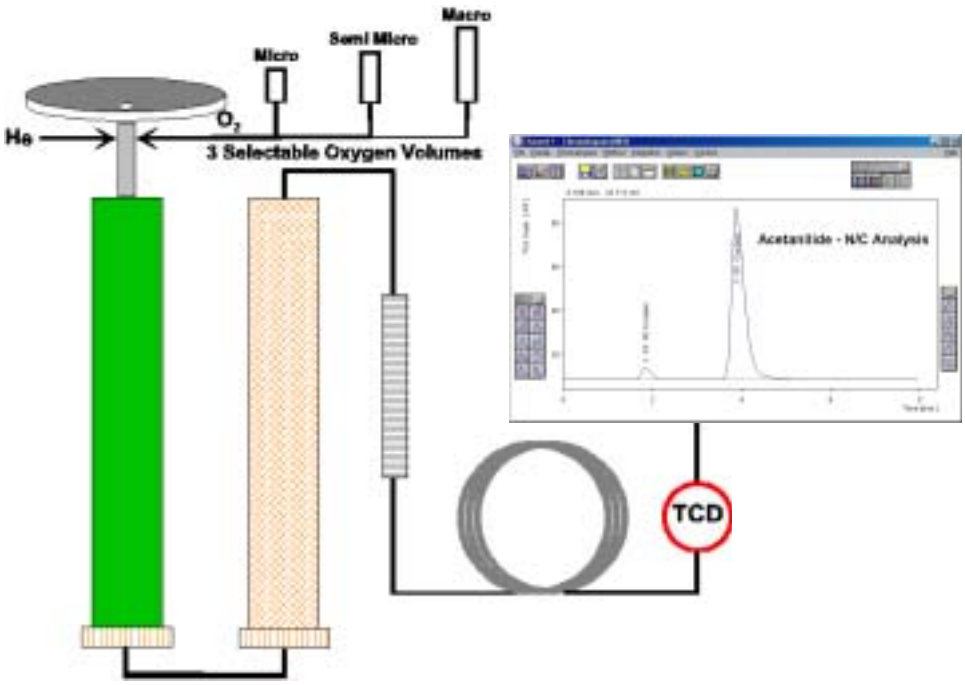
The second module is the detector system for measuring the combustion (or pyrolysis) products. These combustion gases can be separated by gas chromatography for CHNS-O analysis, or sent to a mass spectrometer for isotopic analysis. For the gas chromatographic technique a high stability oven, with temperature control from 30-110 ^{o}C, and a wide range of GC column lengths are available. The TCD (thermal conductivity detector) has been specially designed for this application, special features include; preheating of combustion gases before entering the detector to stabilize gas temperature, and a twin encapsulated filament which requires no reference gas flow. The advantages of the this are; simplicity of the combustion/detector system (low maintenance, minimum down time), efficiency of the combustion, and no splitting or dilution of the combustion gases, ***all combustion products are measured.***

The third module is EAS, our Windows™ based software package, for instrument control, data acquisition and report generation. A multichannel 24 bit A/D interface connects the ECS to a PC for data acquisition and instrument control. Sample information is entered, the software collects raw data from the TCD, creates the calibration, and generates reports including statistical information. Data can be easily exported to EXCEL™ in ASCII format for post-run calculations or exporting to LIMS. A reprocessing software option is available for setting up methods and transferring data from a remote computer, or working with data out of the laboratory. Interface boards can be ordered for 1,2, or 4 channels.



OPERATING PRINCIPLE

During normal system operation, helium carrier gas circulates within the analytical circuit which consists of a combustion reactor for CHNS and N/Protein, or a pyrolysis reactor for Oxygen. The carrier gas brings the products of combustion, or pyrolysis to a gas chromatographic separation column and TCD detector for CHNS-O analysis, or a multi-detector system for N/Protein determination.



C/H/N/S Elemental Analysis

At the start of the analytical cycle the helium carrier gas is switched to a volume of oxygen which is chosen by the operator depending on the size and composition of the sample. The samples are dropped sequentially into the combustion reactor prior to the arrival of oxygen. The sample and tin capsule react with oxygen and combust at temperatures of 1700-1800 °C and the sample is broken down into it's elemental components, N_2, CO_2, H_2O and SO_2. High performance copper wires absorb the excess oxygen not used for sample combustion. The gases flow through the gas chromatographic (GC) separation column which is kept at a constant temperature (+/- 0.1 °C). As they pass through the GC column, the gases are separated and are detected sequentially by the TCD. The TCD generates a signal, which is proportional to the amount of element in the sample. The EAS software compares the elemental peak to a known standard material (after calibration) and generates a report for each element on a weight basis. For Continuous Flow Isotope ratio Mass Spectroscopy, the separated gases are carried to the mass spectrometer interface and into the MS source.

Oxygen Analysis

Helium carrier gas circulates through the analytical circuit, which consists of a pyrolysis reactor which is filled with a special nickelized carbon wool contact material heated to 1080°C, a trap for acidic gases formed by the pyrolysis, a GC column which will separate the gas mixture, and a TCD. Samples are dropped automatically into the pyrolysis reactor, break down and the released oxygen reacts with the nickel carbon wool to form CO ($2C + O_2 = 2\ CO$) and N_2, if present in the sample. The mixture of N_2 and CO gases flow through the gas chromatographic (GC) separation column which is kept at a constant temperature (+/- 0.1 °C). As they pass through the GC column, the gases are separated and the CO peak is detected by the TCD. The TCD generates a signal which is proportional to the amount of CO in the sample. The EAS software compares the CO peak to a known standard material (after calibration) and generates a report for oxygen concentration on a weight basis.

ECS 4010

features and benefits

Sampling options
- "Zero Blank" sampler, 32 or 50 positions
- 150 position open carousel sampler
- Direct injection (syringe) of gases

Combustion and Reduction Tubes

- Diameters (OD)
  - 10.5 mm
  - 18 mm
  - 20 mm
  - 26 mm
- Simple, easy fittings to change tube diameters.
- Inserts for ash removal

G.C. Oven and Separation Column
- Temperature range 30-110 °C
- Temperature control +/- 0.1 °C
- Standard and custom column lengths for different applications

Dual filament TCD
- No reference gas required
- Pre-heating of combustion gases

EAS Software
- Windows™ 95/98/NT
- Instrument control via RS232
- Export data to Excel™ or LIMS
- Actualizes results

Mass Spectrometer or Other Detectors

AUTOSAMPLERS

The "Zero Blank" autosampler developed by Costech, uses a "sealed carousel" design. All samples are purged simultaneously with helium in an enclosed chamber. This eliminates any nitrogen background (from atmosphere) which is introduced in the open carousel sampler when it moves to drop the sample into the reactor.

The microstepper motor which drives the autosampler can switch from a 50 position carousel to a 32 position carousel for large or bulky samples.

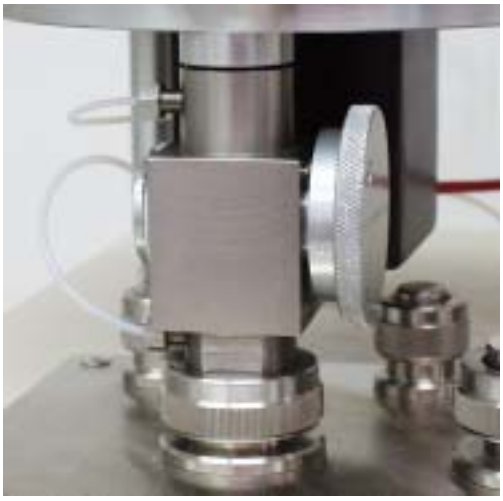
The autosampler controls (set in the ECS software) are; manual advance, carousel selection (50 or 32 position), and "jog", which is used to initially align the carousel.

Zero Blank Autosampler

Interior view - 50 position carousel

Optional Isolation Valve

An optional valve to minimize system changes is available. It replaces the standard drop tube and is the same length. The carrier gas connection is below the valve and a separate purge connection is provided above the valve. When opening the



carousel to add samples it is only necessary to turn the valve, isolating the entire carousel section. The carrier gas will continue to flow through the rest of the analytical circuit. After adding samples to the carousel, close the lid, turn on the purge flow and let the carousel purge for 2-5 minutes. Close the purge vent, turn off the purge flow, and open the isolation valve.

Pneumatic Autosampler

Our 150 position pneumatic autosampler has carousels (three) which are open to atmosphere, and samples can be added continuously while running. The sample holes in the carousels are 11.5 mm in diameter and can accommodate samples like glass fiber filters or large, bulky samples. A large viewing port allows you to clearly see the sample combustion during the analysis.

Continuous Flow EA-IRMS

The ECS 4010 uses the combustion/GC separation technique for elemental analysis. This makes it perfectly suitable for continuous flow, stable isotope analysis.

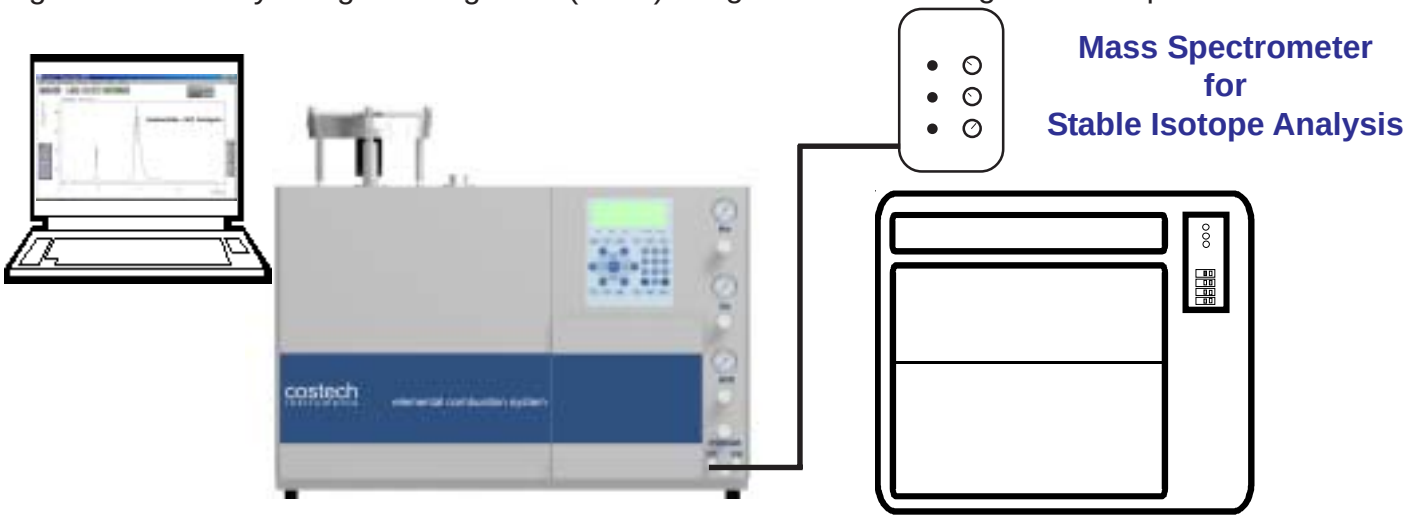
All instrument parameters are controlled by an internal microprocessor, no external computer or software is required when connected to a mass spectrometer. All that is needed for the EA/MS application is the ECS base unit and an autosampler. The ECS will accept an external start signal from any mass spectrometer, so the two instruments can function together.

Our Zero Blank autosampler is unique, and is especially suited for EA-IRMS:The sealed carousel design eliminates any nitrogen background (blank).

Two interchangeable carousels, 50 positions for normal size samples, and 32 positions for large samples, are available and can be changed in just 1 minute.

An optional isolation valve minimizes system changes when adding samples to the carousel.

To measure total CHNS-O, as well as the isotope ratios from the mass spectrometer simultaneously, our EAS software can be synchronized with the mass spectrometer so the two instruments run in the same time frame. This is because EAS will accept an external start signal; the mass spectrometer software starts the ECS, then the ECS sends an external start signal to EAS to begin data acquisition.



Mass Spectrometer for Stable Isotope Analysis

INSTRUMENT CONTROLS

Select Operational Mode
- CHNS or Oxygen Analysis
- Micro/Semi-Micro/Macro

Set Temperatures
- Combustion/Reduction Furnaces
- GC Oven

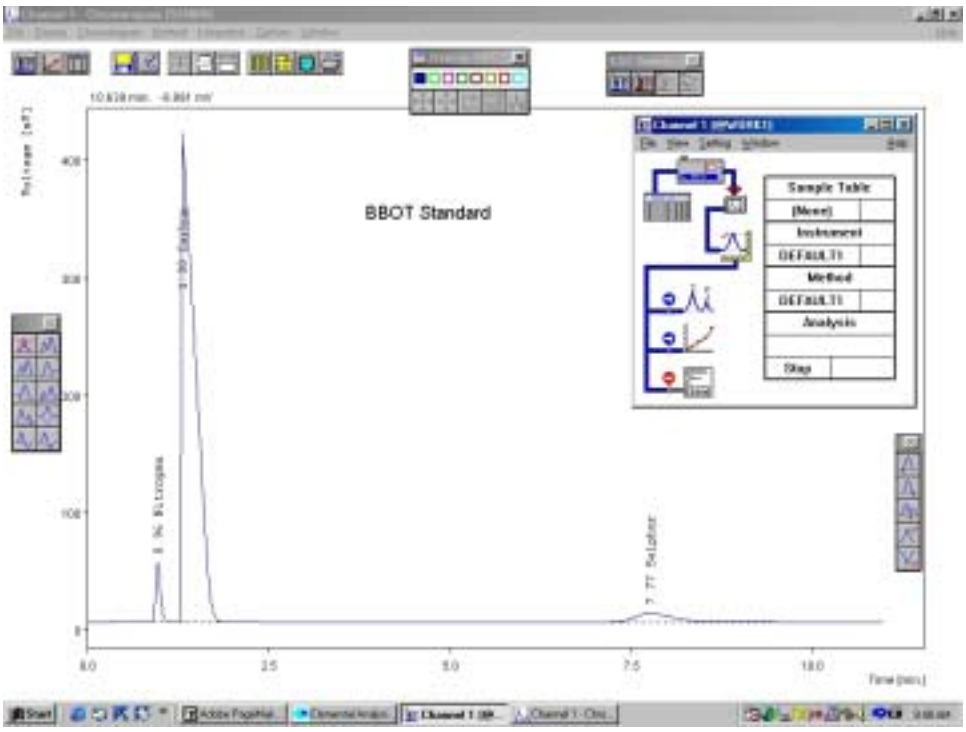
Program Timing Functions
- Run time
- Sample Start
- Oxygen injection

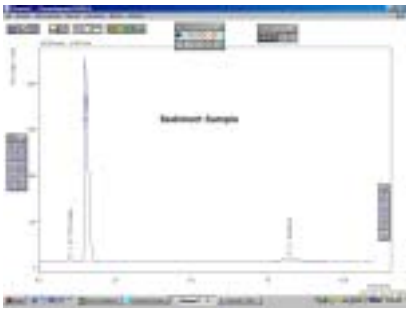
Monitor
- Line voltage
- Electronics & power supply temp.

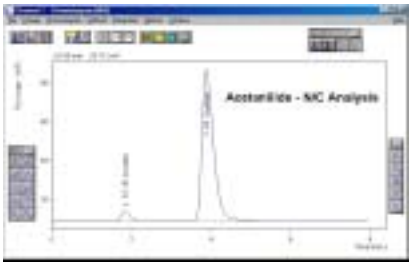
TCD
- Signal gain
- Electronic zero

TECHNICAL SPECIFICATIONS

Sample Size (density dependent)	Micro	<1-5 mg
	Semi-Micro	5-50 mg
	Macro	up to 500 mg of organic material
Measuring Range	0-100% CHNS-O	
Autosamplers	“Zero Blank”, sealed carousel, microstepper motor 147 position, open carousel, pneumatic	
Catalyst consumption	>1000 Analyses for Micro & Semi-Micro	
Dimensions	79 x 55 x 34 cm	
Power	220V 50/60 Hz	
Software	Windows™ 95/98/2000/NT	







EAS Software for Elemental Analysis

EAS is a data station developed for acquiring and evaluating data from the TCD (Thermal Conductivity Detector) in the ECS 4010 and reporting the elemental concentrations. A Sample Table and Calibration File are created containing information on standard reference materials (used for calibration) and unknown samples. After the calibration samples are analyzed and a calibration curve established, concentration results for unknown samples will be reported.

The **EAS** station is designed as a board to be installed in a PC computer and the corresponding software package for Windows™. The board contains one to four independent integrating A/D converters and controlling inputs and outputs. The station allows for simultaneous data acquisition from up to four detectors independently “in the background”, data analysis, baseline correction and for calibrated or non-calibrated data processing. Results may be printed on any printer with a wide range of optional print parameters, and tables, data, and chromatograms exported to files or directly to other programs running under Windows ™.
Calibration files enable you to carry out calibration-based calculations with an external standard method. Each calibration file may contain up to 20 concentration levels for each component.

Computer and operating system requirements

The **EAS** station may be run under any of the following systems: Windows 3.1, 3.11, Windows 95/98/ME and Windows NT/2000.
For Windows 3.1 and 3.11 with moderate demands on speed a 386/40 computer with 4 to 8 MB memory is sufficient. For Windows 95/98/ME we recommend at least 486/100 computer with 16 MB memory, and for Windows NT/2000 a Pentium/90 computer with 32 MB.

CONSUMABLES

Costech offers a complete line of consumables and accessories for its lines of analytical instruments. Capsules, quartz tubes, reagents, standard reference materials, oxidation and reduction catalysts, chromatographic columns, autosamplers, and data acquisition software packages.

To reduce the cost of sample analysis it is important to increase the number of samples which can be run using the minimum amount of catalysts, and to reduce the cost of disposal of the exhausted catalysts and reagents.

New, optimized catalysts are available for all configurations and applications. Catalysts are certified for physical/chemical properties, chemical purity and reactivity.

Quartz reactors
Tin and Silver Capsules
Catalysts
Copper Wires
Standards
Reagents
GC Separation Columns

PART NUMBERS

40.11.001	Base Unit configured for pneumatic autosampler
40.10.001	Base Unit configured for pneumatic and electronic autosampler
41.10.000	**Pneumatic autosampler**, w/1, 50 position carousel
41.10.100	Carousel number 1
41.10.200	Carousel number 2
41.10.300	Carousel number 3
41.00.001	**“Zero Blank” Autosampler**, w/50 position carousel
41.00.002	**“Zero Blank” Autosampler**, w/32 position carousel
41.00.100	Control module for “Zero Blank” Autosampler
41.00.200	50 position carousel for the Zero Blank autosampler
41.00.300	32 position carousel for the Zero Blank autosampler
41.00.110	**Isolation valve** for Zero Blank autosampler
41.00.120	**Gas injection device**, in-line
41.00.130	**Direct gas injection device**, for combustion tube
42.10.001	**EAS Software package**, includes single channel A/D board, software and cable
42.10.002	**EAS Software package**, includes 2 channel A/D board, software and cable
42.10.004	**EAS Software package**, includes 4 channel A/D board, software and cable
099006	**Sartorius M2P Microbalance**, 1 ug readability (0.001 mg)
43.13.001	Consumables kit for 1000 **Nitrogen** Analyses
43.13.002	Consumables kit for 1000 **Nitrogen/Carbon** Analyses
43.13.003	Consumables kit for 1000 **Nitrogen/Carbon/Hydrogen** Analyses
43.13.004	Consumables kit for 1000 **Nitrogen/Carbon/Sulfur** Analyses
43.13.005	Consumables kit for 1000 **Nitrogen/Carbon/Sulfur/Hydrogen** Analyses
43.13.006	Consumables kit for 1000 **Nitrogen/Carbon/Sulfur/Hydrogen- Oxygen** Analyses