



Agri - Food



Chemicals



Geo - Environment



Energy

# Application Note

## CHN analysis of refinery and lubricating oils

Instrument: ECS 4024 or ECS 8020

Mode: CHN

Pretreatments: none



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**NC TECHNOLOGIES**

Innovative Elemental Analysis

# Application Note – CHN analysis of refinery and lubricating oils

## Mineral base oil

Crude oil results from physical and chemical processes acting over many million years on the buried remains of plants and animals.

Although crude oil is usually formed in fine-grained source rocks, it can migrate into more permeable reservoir rocks and large accumulations of petroleum, the oilfields, are accessed by drilling.

Different crude oils contain different proportions of classes of organic components and also vary in the boiling range distribution of their components. The main factors affecting crude oil selection for the manufacture of base oils are the following:

- ❖ content of material of a suitable boiling range for lubricants.
- ❖ yield of base oil after manufacturing processes.
- ❖ base oil product properties, both physical and chemical

Approximately 95 percent of the current lubricant market share is comprised of conventional (mineral-based) oils.

The petroleum that flows from a well in the form of crude oil comes in many varieties and types, ranging from light-colored oils containing mostly small hydrocarbon molecular chains to black, nearly solid asphalt-like large hydrocarbon chains.

These crude oils are very complex mixtures containing a plethora of different compounds made of hydrogen and carbon. These compounds (known as hydrocarbons) can range in size from methane (containing one carbon and four hydrogen atoms) to massive structures with 60 or more carbon atoms.



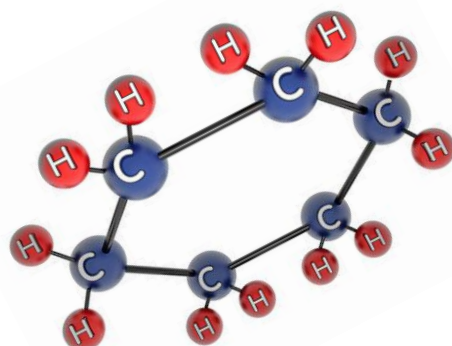
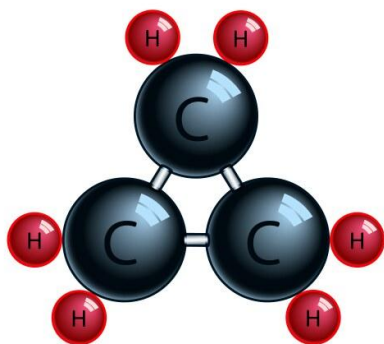
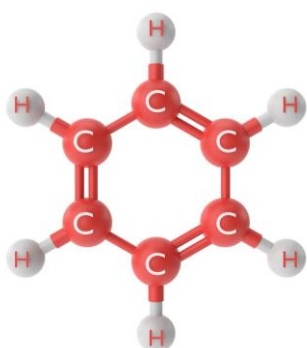
# Application Note – CHN analysis of refinery and lubricating oils

## The importance of refineries

Most lubricating oils come from petroleum or crude oil. In order to get a lubricating oil from a crude oil, the crude oil must be sent through a refinery. The refinery takes from the crude oil a lot of molecules of various sizes and structures that can be used for different things. For example, gasoline, diesel and kerosene are all derived from crude oil.

Lubricating oil relates to hydrocarbon molecules of a particular size (in the range from 26 to 40 carbons). Fairly large and heavy molecules are needed to work as lubricating oils.

The molecules that are used with gasoline and kerosene are smaller and have fewer carbons in the structure of the molecule. The refinery puts these molecules in little silos based on size and weight, and removes impurities, enabling each of the products from the crude oil to be utilized.



The basic function of a refinery is to separate the crude oil into its useful components and remove the components of unwanted materials. Base oils or base stocks, as they are sometimes called, are created from separating and cleaning up crude oil. They are one of several liquid components that are created from crude oil.

Gasoline is the lightest or smallest hydrocarbon component, followed by kerosene or jet fuel, diesel fuel, base oils, waxes and asphalt or bitumen, which is the heaviest, thickest material. Base oils are prepared from crude oils through the use of the following series of processes which, to some degree, must be applied to all crude oils for refining and also re-refining processes.

# Application Note – CHN analysis of refinery and lubricating oils

## Differences between oils, lubricants, and greases

In general, lubricating oil is a substance that reduces friction when placed between two surfaces with relative motion. There are lubricants with different grades and viscosities, although their functions have a lot of similarities as they:

- ✓ reduce friction between two surfaces in motion.
- ✓ protect mechanical components from wear and corrosion.
- ✓ clean and cool.
- ✓ act as a sealant between the segments/pistons and jackets in order to prevent gas leaks from occurring inside the combustion chamber.

What is the difference between oil and lubricant? A lubricant can be a fluid, solid, or semi-solid. Oil refers to a liquid lubricant with an oil base and additives. This is the best known type of lubricant.

A grease is a semi-fluid or semi-solid lubricant that consists of a mixture of a thickening agent with a liquid lubricant and other elements (additives), that give each grease its special properties

The structure of grease allows the lubricant to remain in a solid state until the shear force between surfaces reaches a certain level, and the grease begins to flow and becomes a mobile compound. Once the shear force is reduced, the grease regains its initial properties.

Due to its consistency it remains at the lubrication point, so surfaces remain protected against rust and corrosion, even when systems are shut down. On the other hand, greases act as a sealant, preventing leaks in the system and impurities from entering the lubrication point, absorbing external contamination, noise, and vibrations.



# Application Note – CHN analysis of refinery and lubricating oils

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## Quality control

CHN analysis in oils and lubricants determines the precise percentage of Carbon, Hydrogen, and Nitrogen, crucial for quality control, performance assessment, and refining potential, using the flash combustion analysis (Dumas method) to understand material composition, assess additives, and track degradation.

This elemental breakdown helps characterize crude oil, finished lubricants, biofuels, and even byproducts, ensuring product consistency and helping predict processing behavior.

The CHN analyses can be associated to other techniques such as NMR or FTIR in order to identify structures and chemical compositions, to perform mass balances or to highlight trends in lubricants behavior.

It is established that CHN analysis is the base for these studies: such importance has to be sustained by a high quality instrument able to produce precise data.

## ***What it Measures***

Carbon (C) & Hydrogen (H): indicates the base oil's hydrocarbon structure, useful for calculating the C/H ratio to assess refining potential, performance, and purity.

Nitrogen (N): reveals the presence of nitrogen-containing compounds, which can act as anti-wear additives or indicators of contamination/degradation.

## ***Why it's used in oils & lubricants***

- Quality Control: ensures base oils and finished lubricants meet specifications
- Performance: Nitrogen content can relate to additive effectiveness (like detergents, dispersants) and overall lubricant stability
- Processing: C/H ratio helps evaluate crude oil and fuels for upgrading processes
- Troubleshooting: identifies contamination or breakdown by monitoring changes in elemental composition

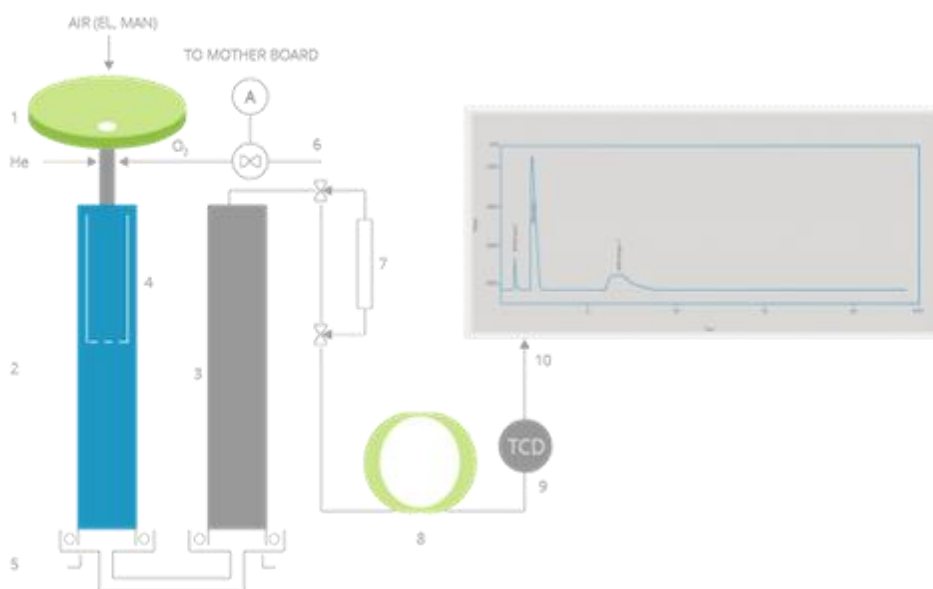
# Application Note – CHN analysis of refinery and lubricating oils

## Standard test method

The ASTM D5291 cover the instrumental determination of carbon, hydrogen, and nitrogen in laboratory samples of petroleum products and lubricants. Values obtained represent the total carbon, the total hydrogen, and the total nitrogen.

These standard method is applicable to samples such as crude oils, fuel oils, additives, and residues for carbon and hydrogen and nitrogen analysis.

In particular, the organic samples are packed into lightweight containers of oxidizable metal and dropped at preset times into a vertical quartz, inconel, or stainless steel reactor, heated at about 1050 °C, through which a constant flow of helium is maintained. When the samples are introduced, the helium stream is temporarily enriched with pure oxygen. Flash combustion takes place primed by the oxidation of the container. Quantitative combustion is then achieved by passing the gases over chromium trioxide and cupric oxide. The mixture of the combustion gases is transferred over copper at about 640 °C (840 °C in a steel reactor) to eliminate the excess of oxygen; then without stopping, it is introduced into the chromatographic column heated to about 120 °C. The individual components are then separated by elution in the order nitrogen, carbon dioxide, and water by a dedicated column and measured by a thermal conductivity detector. With dedicated software the percentage of elements present in the sample are calculated.



# Application Note – CHN analysis of refinery and lubricating oils

## Elemental analysers

The 40 and 80 series are the NC Technologies suitable analysers for this kind of analysis.

In particular, both ECS 4010 and the ECS 8020 are the better analytical choice.

### The **ECS 4010**

The ECS 4010 is recommended when you want to analyse particularly homogeneous samples (micro weighing). It is also recommended in laboratories where many samples are analysed, thanks to its high degree of automation.



|                              | ECS 4010              |
|------------------------------|-----------------------|
| Elements                     | C, H, N               |
| Autosampler                  | Single position       |
| Max sample weight            | Up to 200 mg**        |
| Max C content                | Up to 14 mg           |
| Nitrogen purification system | On board              |
| Operating mode               | CHN analysis in 1 run |
| Type of samples              | Liquids and solids    |

\*\* depending on C content

Both instruments are equipped with a PN 150 autosampler suitable for the analysis of both liquid and solid samples.

### The **ECS 8020**

The ECS 8020 is the high-throughput elemental analyser for inhomogeneous samples. Thanks to the double oven configuration, which involves the use of one combustion tube and one reduction tube, the operability of the instrument increases significantly; this translates into greater autonomy of the instrument.



|                              | ECS 8020            |
|------------------------------|---------------------|
| Elements                     | C, H, N             |
| Autosampler                  | Up to 147 positions |
| Max sample weight            | 300 mg**            |
| Max C content                | 20 mg               |
| Nitrogen purification system | On board            |
| Operating mode               | CHN in 1 run        |
| Type of samples              | Liquids and solids  |



# Application Note – CHN analysis of refinery and lubricating oils

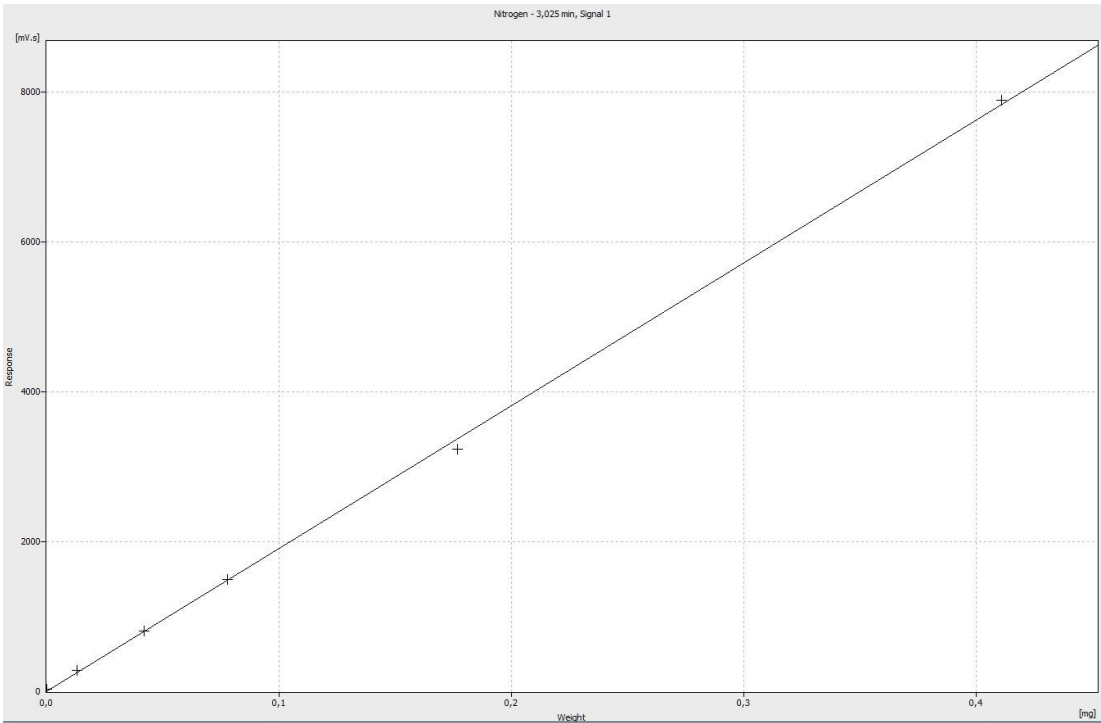
## Sample analysis – Example 1

In this example we tested Nitrogen in Lube Oil Additive in real samples provided by our customer.

| Instrument set up |                                     |
|-------------------|-------------------------------------|
| Instruments       | ECS 8020                            |
| Operative mode    | CN                                  |
| Left furnace      | Cr2O3+Co3O4 ; 18 mm diam; T = 980°C |
| Right furnace     | Cu 0,5 ; 18 mm diam: T = 650°C      |
| GC column         | T = 70°C; 4 m long.                 |
| TCD gain          | H6                                  |
| Carrier flow      | 80 ml/min                           |
| Oxygen amount     | fix 20 ml                           |

The aim of this configuration was to determine the nitrogen content. The dual furnace configuration let the user to be sure about the total conversion of elements in gases detected by on board TCD.

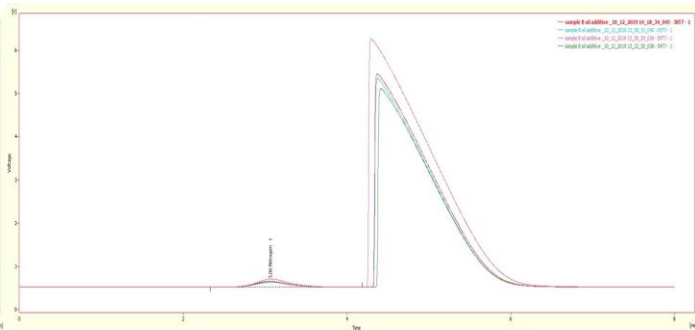
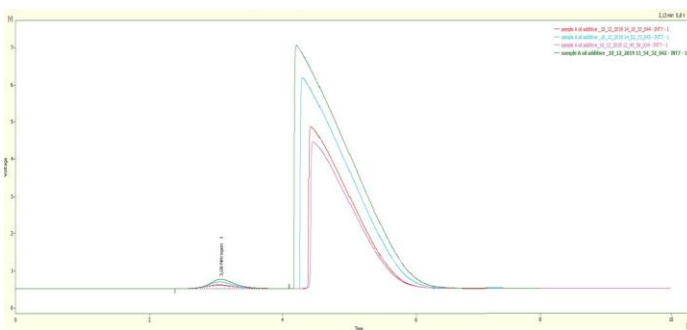
## Calibration curve for N



## Application Note – CHN analysis of refinery and lubricating oils

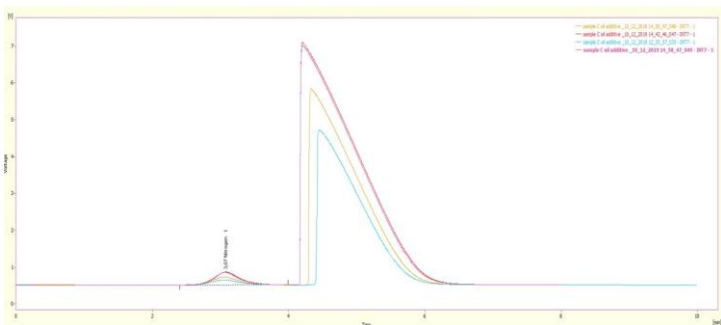
## Sample analysis – Example 1

## Results



| Sample ID          | Weight (mg) | % N    |
|--------------------|-------------|--------|
| Sample A batch 43k | 4,8         | 3,51   |
| Sample A batch 43k | 7,6         | 3,57   |
| Sample A batch 43k | 4,1         | 3,62   |
| Sample A batch 43k | 10,4        | 3,5    |
| Average            |             | 3,55   |
| Std Dev            |             | 0,056  |
| RSD %              |             | 1,5768 |

| Sample ID          | Weight (mg) | % N    |
|--------------------|-------------|--------|
| Sample B batch 85k | 8,1         | 2,63   |
| Sample B batch 85k | 7,7         | 2,73   |
| Sample B batch 85k | 10,4        | 2,76   |
| Sample B batch 85k | 7,2         | 2,65   |
| Average            |             | 2,6925 |
| Std Dev            |             | 0,0624 |
| RSD %              |             | 2,3169 |



| Sample ID          | Weight (mg) | % N    |
|--------------------|-------------|--------|
| Sample C batch 105 | 6,7         | 4,74   |
| Sample C batch 105 | 10,4        | 4,73   |
| Sample C batch 105 | 4,5         | 4,81   |
| Sample C batch 105 | 10,1        | 4,69   |
| Average            |             | 4,7425 |
| Std Dev            |             | 0,0499 |
| RSD %              |             | 1,0525 |

The content of nitrogen is quite low in every sample; this means there are low contaminant.

# Application Note – CHN analysis of refinery and lubricating oils

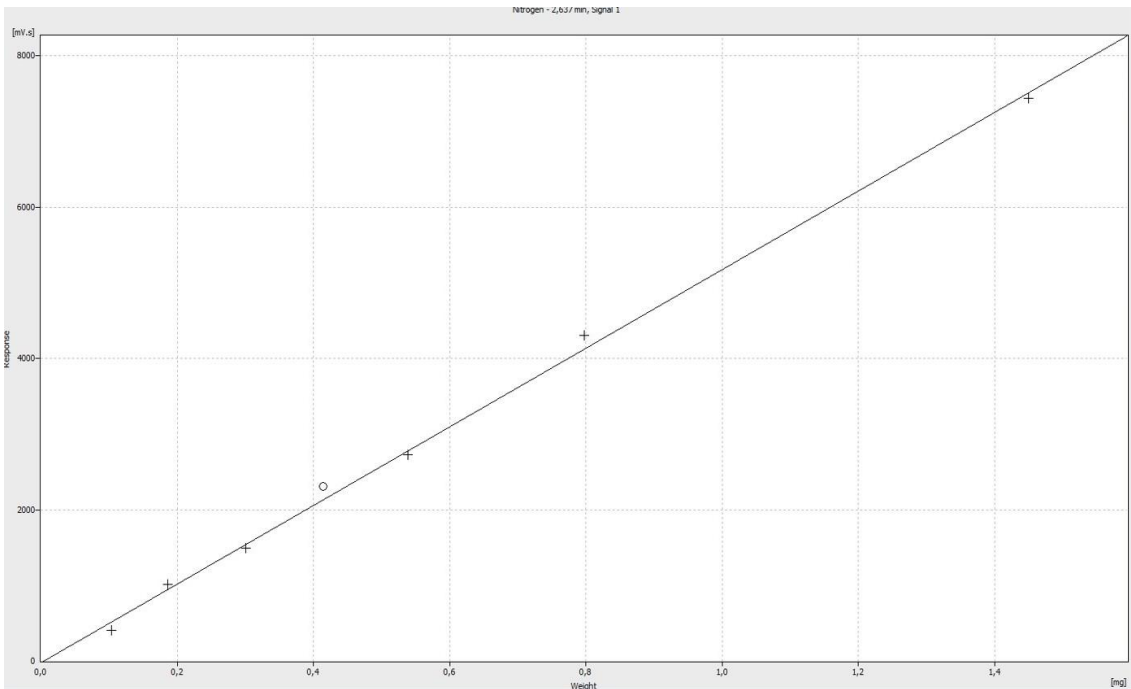
## Sample analysis – Example 2

In this example we analyse the CHN content in real samples provided by our customer.

| Instrument set up |                                     |
|-------------------|-------------------------------------|
| Instruments       | ECS 4010                            |
| Operative mode    | CHN                                 |
| Left furnace      | Cr2O3+Co3O4 ; 18 mm diam; T = 980°C |
| Right furnace     | Cu 0,5 ; 18 mm diam: T = 650°C      |
| GC column         | T = 70°C; 3 m long.                 |
| TCD gain          | L5                                  |
| Carrier flow      | 105 ml/min                          |
| Oxygen amount     | fix 40 ml                           |

The aim of this configuration was to determine the CHN content. The dual furnace configuration let the user to be sure about the total conversion of elements in gases detected by on board TCD.

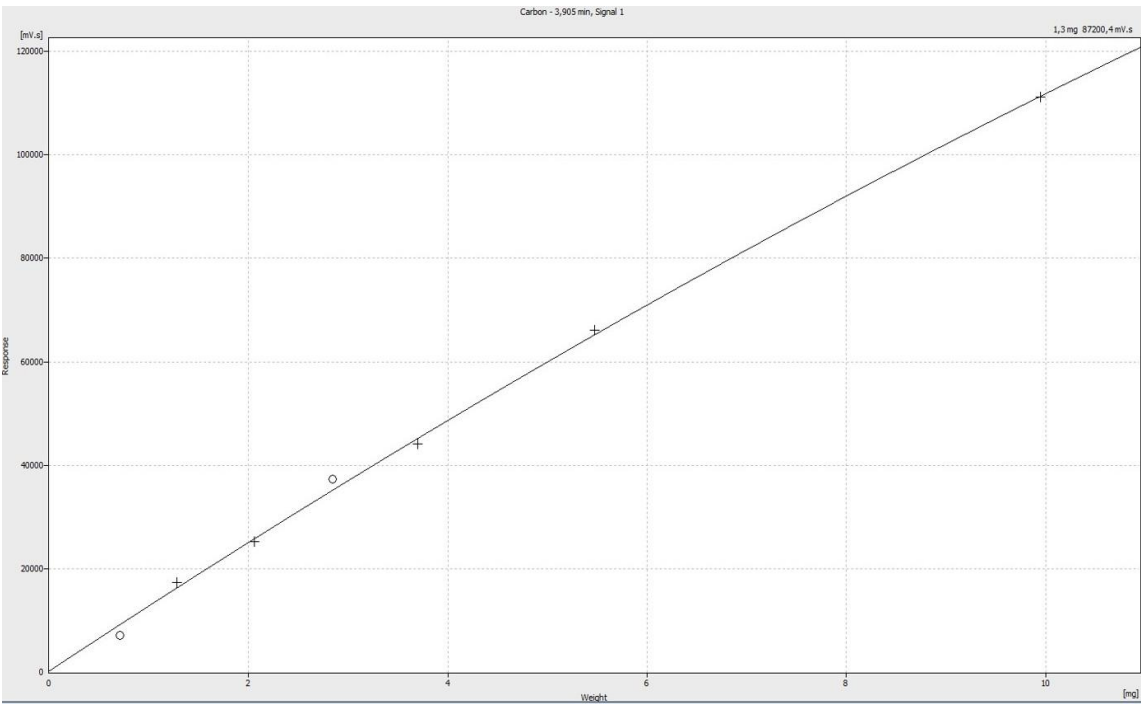
## Calibration curve for N



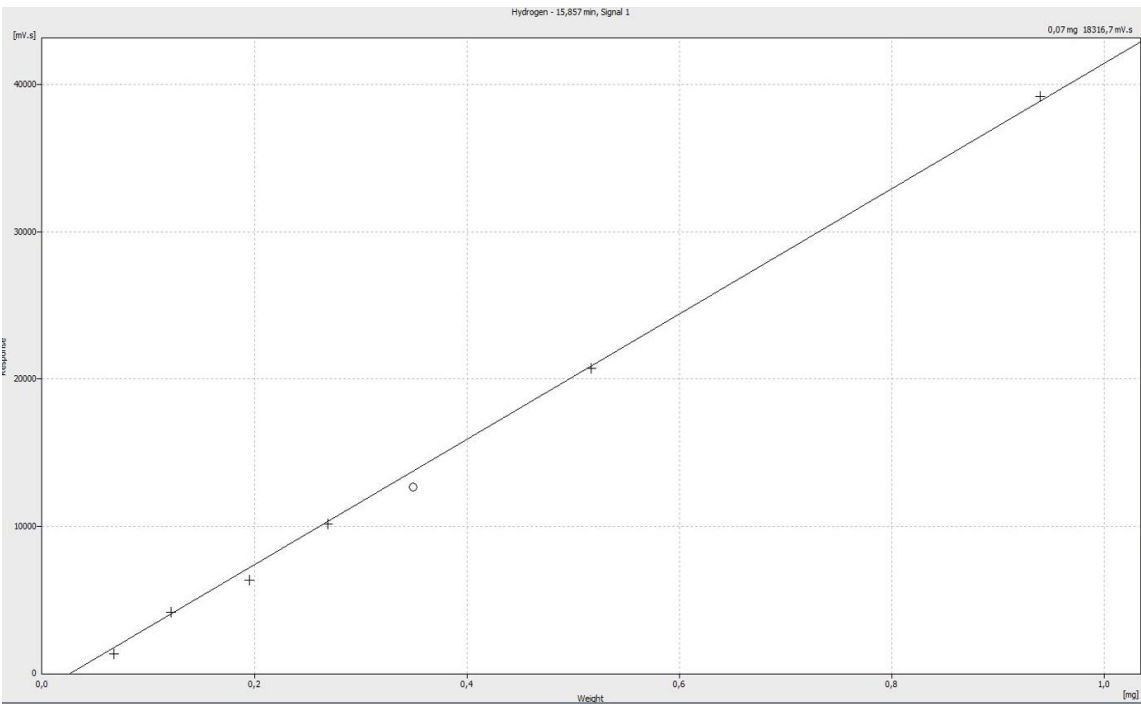
# Application Note – CHN analysis of refinery and lubricating oils

## Sample analysis – Example 2

Calibration curve for C



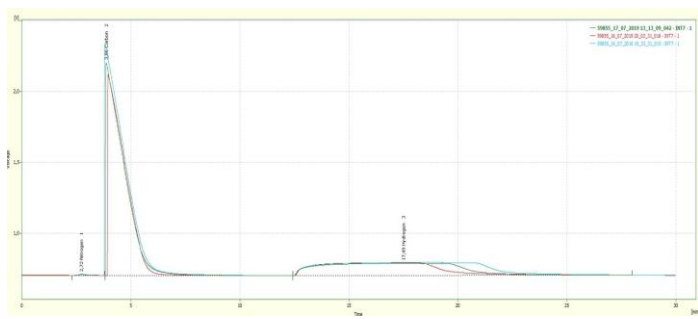
Calibration curve for H



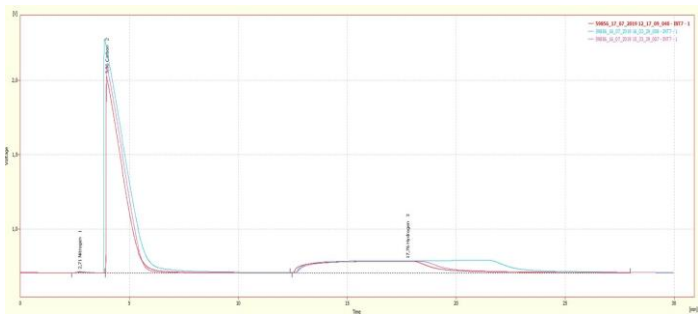
# Application Note – CHN analysis of refinery and lubricating oils

## Sample analysis – Example 2

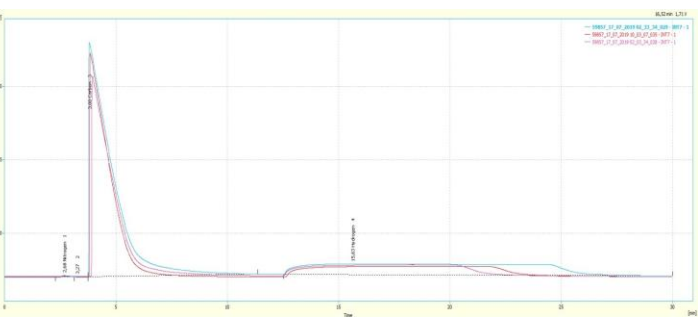
### Results



| Sample ID      | Weight (mg) | % N   | % C   | % H   |
|----------------|-------------|-------|-------|-------|
| ISS 2161-59855 | 8,7         | 0,43  | 84,99 | 10,96 |
| ISS 2161-59855 | 7,8         | 0,43  | 85,01 | 11,28 |
| ISS 2161-59855 | 10,4        | 0,45  | 85,25 | 11,2  |
| Average        |             | 0,44  | 85,08 | 11,15 |
| Std Dev        |             | 0,01  | 0,14  | 0,17  |
| RSD %          |             | 2,64% | 0,17% | 1,49% |



| Sample ID      | Weight (mg) | % N   | % C   | % H     |
|----------------|-------------|-------|-------|---------|
| ISS 2162-59856 | 6,8         | 0,41  | 83,17 | 10,72   |
| ISS 2162-59856 | 10,5        | 0,44  | 83,06 | 10,78   |
| ISS 2162-59856 | 7,6         | 0,47  | 83,43 | 10,45   |
| Average        |             | 0,44  | 83,22 | 10,65   |
| Std Dev        |             | 0,03  | 0,19  | 0,17578 |
| RSD %          |             | 6,82% | 0,23% | 1,65%   |



| Sample ID      | Weight (mg) | % N   | % C   | % H   |
|----------------|-------------|-------|-------|-------|
| ISS 2163-59857 | 11,9        | 0,35  | 84,55 | 11,23 |
| ISS 2163-59857 | 9,3         | 0,38  | 84,71 | 10,76 |
| ISS 2163-59857 | 8,8         | 0,39  | 84,69 | 10,74 |
| Average        |             | 0,37  | 84,65 | 10,91 |
| Std Dev        |             | 0,02  | 0,09  | 0,28  |
| RSD %          |             | 5,58% | 0,10% | 2,54% |