

Elemental Analysis of CHNS/S/O

1. Introduction

This method describes the simultaneous determination of carbon, hydrogen, nitrogen and sulfur (CHNS) present in a substance. Additionally, either sulfur (S) or oxygen (O) may be determined in a second, separate analysis. A wide variety of liquid or solid organic substances may be analyzed; most typically kerogens and oils are analyzed. This analysis quantitatively determines the weight percent of CHNSO. From this information hydrocarbon properties may be inferred, in particular elemental compositions help reveal the potential of the original source rock to form petroleum. (Durand et al, 1980, Orr et al, 1990 and Tissot et al, 1974)

2. Interfaces with Other Methods

EGL Method 29, Calibration of Laboratory Scales and Balances

3. Materials and Equipment

Instruments: Carlo Erba EA 1110 or Thermo Flash 2000¹
Sartorius MC-5 Microbalance

Materials: UHP He
UHP Oxygen
For oxygen analysis refer to:
Instruction Manual EA1110 Elemental Analyzers Rev. W06
0596mv, pg. 21 or Organic Elemental Analysis Flash 2000
Elemental Analyzer Operating Manual, 2010, pg. 62

For CHNS or S analysis refer to:
Organic Elemental Analysis Flash 2000 Elemental Analyzer
Operating Manual, 2010, pg. 61

¹ Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

4. Procedure

Turn on instrument and determine instrument is in ready state.

Run a leak check after reactors have been replaced.

Precisely weigh out samples in triplicate if sufficient sample is available. Use tin capsules for CHNS analysis and silver for oxygen analysis.

Place samples sequentially in autosampler and record sample weights in 'sample table'. Start each run by analyzing a blank cup. Check standards should be run at the beginning of each sequence to determine if calibration is valid (see section 5 below for validation requirements). Once check standards are validated, samples may be run. Check standards should be run minimally after every 6 unknowns. Each sequence should end with check standards.

5. Calibration and Quality Control Samples

Precisely weigh out 3-10 standards or reference materials into cups with weights ranging from 0.5 to 3 mg using a microbalance. Which standard(s) and number of standards to weigh out will depend upon type of unknowns to be analyzed; try to match unknowns to standard or reference materials similar to unknowns. Each calibration curve requires minimally 3 points. A linear calibration forced through zero is typically used; in instances where other calibration methods are used it is noted in the instrument notebook. The calibration curve is acceptable if calibration factor (CF) is 0.999 or better for each element. Instrument is recalibrated when reagents are depleted, or if the standards with known elemental weight percentages run as unknowns do not meet acceptance level ($\pm 10\%$ relative or ± 0.5 wt. % absolute whichever is greater).

A blank tin cup is run at the beginning of every batch of samples analyzed. BBOT (2,5-Bis(5-tert-butyl-benzoxazol-2yl)thiopene) for CHNS or Acetanilide are run as unknowns for CHNS and O respectively. BBOT and Acetanilide are commercially available compounds with fixed ratios of CHNSO. Standard percentages are maintained in the EGL database.

Replicate samples are analyzed when there is sufficient sample. If the standard deviation for the replicates is less than 0.5 wt. % the data is acceptable. If the standard deviation is greater than 0.5 wt. %, the sample is reanalyzed. If the second analysis has a standard deviation greater than 0.5 wt. % then it is noted in the comments; typically this occurs when samples are not homogeneous.

Duplicate samples are also analyzed; minimally one duplicate per sample set if there is sufficient sample. Duplicate samples are acceptable if the averaged results are ± 0.5 wt. % of one another. If samples are not within ± 0.5 , then a third analysis is required and the results are discussed with submitter to determine source of variability.

6. Limits, Precautions, and Interferences

Not all samples are homogeneous therefore replicate analysis are required when there is sufficient sample. Standard deviation and number of runs is reported to submitter.

Ralstonite: $\text{NaMg}[\text{Al}(\text{F},\text{OH})_6]\cdot\text{H}_2\text{O}$ may be formed during kerogen isolation. It is the submitters responsibility to remove Ralstonite from samples prior to submittal.

Lower Reporting Limits:

Nitrogen: 0.1 wt. %, Carbon: 0.15 wt. %, Hydrogen: 0.1 wt. %, Sulfur: 0.1 wt. %, Oxygen: 0.1 wt. %

7. Acceptance of Data

Standards with known elemental weight percentages run as unknowns must be within $\pm 10\%$ relative or ± 0.5 wt. % absolute, whichever is greater, for data to be acceptable. Standard deviation for unknown samples must be less than ± 0.5 wt. % absolute. See section 5 above for further information.

8. Data Handling and Transfer

Data is transferred via a floppy disk or flash drive from the instrument computer to a networked computer. Samples run multiple times are averaged using a locked Excel™ spreadsheet. The averaged value of the unknowns, blanks, duplicates and standards, along with the number of runs, and the standard deviation are all contained in the spreadsheet which is transferred into the EGL database.

9. References

Durand, B., Monin, J.C., Kerogen Insoluble Organic Matter From Sedimentary Rocks, 1980, pg. 113-141.

Instruction Manual EA1110 Elemental Analyzers Rev. W06 0596mv

Organic Elemental Analysis Flash 2000 Elemental Analyzer Operating Manual, 2010

Orr, W. L., Damste, Jaap S. Sinninghe, Geochemistry of Sulfur in Fossil Fuels, 1990, pg.2-29.

Tissot, B., Durand, B., Espitalie, J. and Combaz, A., (1974), *AAPG Bull.*, 58, 3, 499.

10. Attachments

None

11. History of Changes

R0: Initial Issue