Loss on Ignition

1. Introduction

The Loss on Ignition method is performed in order to determine the percent mass that is lost when a coal combustion byproduct or rock is heated under controlled conditions in a pre-programmed furnace. The remaining residue is then used in the trace and major digestion methods (EGL Method 22 and EGL Method 12) which prepare samples for ICP-MS and ICP-OES analysis.

2. Interfaces with Other Methods

This method relies on:
EGL Method 29, Calibration of Laboratory Scales and Analytical Balances and
EGL Method 25, Method for Sample Login, Control, and Disposition.

3. Materials and Equipment

Furnace (programmed to run at both 525°C for 36 hours and 750°C for four hours) as well as ceramic crucibles.

4. Procedure

Weigh out approximately 1 gram of the sample material in a pre-weighed crucible and record this exact mass. Take the combined mass of the sample material and the crucible and subtract the mass of the crucible. Record this as the "as determined" mass. Insert the crucibles containing the sample material into a furnace and select the proper program.

A) For analysis of Major and Minor elements, heat the sample at 750°C for four hours
B) For analysis of Trace elements, heat the sample at 525°C for six hours.

After the selected program has been completed and the internal furnace temperature is below 100°C, remove the crucibles and place them into a desiccator until they reach room temperature. Once at room temperature, reweigh the crucibles containing the sample material and record this exact mass. Subtract this mass by the mass of the crucible and record this as the "dry" mass. The percent LOI can then be calculated.

Calculations:

$$LOI\ (\%) = ((A-B)/A) \times 100$$

Where A is the "as determined" mass and B is the "dry" mass.

5. Calibration and Quality Control Samples

Check the internal furnace temperature and program accuracy at least once annually with a thermocouple connected to a data logger. Adjust the program/settings control on the front of the furnace to ensure the correct program is selected.

Duplicate samples are analyzed to ensure the quality of data. Every job has at least one duplicate sample included and in larger jobs, a duplicate sample is included after every ten samples

6. Limits, Precautions, and Interferences

As a precaution, place the furnace under a hood to help dissipate heat and fumes.

7. Acceptance of Data

To determine if the data generated is acceptable, the duplicate sample mass must be within 20% of the original samples mass. If the relative deviation of the replicated samples exceeds 20%, the same samples will be re-heated at the same temperature for the same amount of time and re-weighed. If this does not result in masses which meet the acceptable data criteria, all of the samples will be discarded and new samples will be re-analyzed starting from the beginning of the procedure.

8. Data Handling and Transfer

The sample masses are transferred from the balance to an Excel™[1] template electronically. The Excel™ template calculates the LOI percent for each sample and the percentages are then transferred into a template which is saved in the data to be entered folder on the shared network drive.

9. References

American Society for Testing and Materials International [ASTM], 2007, Annual book of ASTM standards, section five, petroleum products, lubricants, and fossil fuels, Gaseous fuels; coal and coke: West Conshohocken, Pennsylvania, American Society for Testing and Materials International, v. 05.06, p. 680.

10. Attachments

There are no attachments included

[1] Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

11. History of Changes

Revision 0: initial issue