Summary of CHNS Elemental Analysis

Overview

Our instrument setup uses dynamic flash combustion to analyse the following elements with an accuracy of 0.3% absolute: carbon, hydrogen and nitrogen (default) and, if requested, sulphur. We cannot analyse other elements or trace levels (<0.3%) of elements. Due to the additional time and setup required for sulphur analysis, these are done on a monthly basis. Samples for sulphur analysis must be submitted by the end of the first week of each month and will be run on the second week. Samples not requiring a measurement for sulphur are run more frequently based on demand. Due to a restriction of six air-sensitive samples that can be run per batch, there may be a longer wait if there are a large number of air-sensitive samples submitted.

Samples

A minimum of 5mg per sample is required for elemental analysis. The sample should be as pure as possible and completely dry. Any impurities or trace solvents will adversely affect the results. Ideally solid samples should be submitted in a powder form. Ensure you list ALL elements in the submission form. Samples containing fluorine or sulphur must be treated differently to ensure no carry-over into the next sample (regardless of whether an analysis for sulphur is requested). For air sensitive samples, fill out the submission form selecting "weigh under N_2"; you will be contacted the day before analysis to bring the sample down to the lab. Sulphur will not be analysed unless specified on the submission form. Please try NOT to use large vials to store the sample; it is very time consuming to pick out a single crystal or drop of liquid from large sample bottles.

QA/QC

Measured CHNS must be within 0.40% (absolute) of the calculated values for ACS journal publications. To ensure accurate results, samples are run twice to ensure reproducibility (repeated if necessary), and certified standards are run every two samples in order to ensure the calibration curve has not drifted. The instrument will be recalibrated if the deviation on the standard greater than 0.30% (absolute). All standards used are certified Organic Analytical Standards purchased from Elemental Microanalysis.

Common Problems

- Insufficient sample will not allow for replication and accuracy cannot be guaranteed. Ensure there is at least 5mg of material.
- Too little sample for the size of the vial makes it difficult and even impossible to get sufficient material into the tin capsule for analysis even if 5mg is present. Provide more material or use a smaller vial.
- Impure samples will adversely affect the results. Samples should be as pure as possible with no residual solvent.

Example: Phenyl hydrazine ($C_6H_8N_2$ – 108.14 g/mol) with 2% (w/w) methanol impurity

Mass: 1.5000 mg, but the sample is really:
1.4700 mg phenyl hydrazine (1.359e-2 mmol)
0.0300 mg methanol (9.375e-4 mmol)

Theoretical formula: $C_6H_8N_2$
Actual formula: $C_6H_8N_2 \cdot 0.069$ MeOH ie: $C_{6.069}H_{8.276}N_2O_{0.069}$

	%C	%H	%N
Theoretical	66.64	7.46	25.90
Actual	66.06	7.56	25.39
Deviation	**-0.58**	+0.10	**-0.51**

With values not within 0.40% of the theoretical, these results would not be sufficient for publication and the sample would have to be purified, properly dried and re-run. It does not take much to throw the results off.

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