ELEMENTAL ANALYSIS PATHWAYS

Analytical Laboratory

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What was synthetized? → **SAMPLE** → **Structure confirmation (NMR, MS methods, etc.)**

SAMPLE → **ELEMENTAL ANALYSIS**
(elemental composition of prepared material and/or a proof purity of chemical individuum)

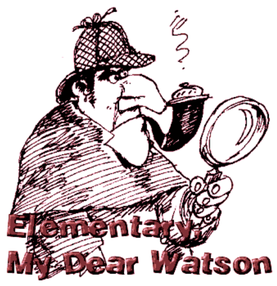
ELEMENTAL ANALYSIS → **FLUORINE**
Mineralisation and determination by using ion selective electrode.

ELEMENTAL ANALYSIS → **C H N (PE automatic analyzer)**

- If the difference from the theoretical composition is lower then 0,3%, the „theory“ is considered as confirmed.
- If the difference is bigger then 0.3%, analysis is repeated (difference of our results is usually lower then 0.3%).
- In case of totally unexpected results, the sample is controlled by ED-XRF analysis, which detectable/determinable elements are present.
  - When unexpected ED-XRF-detectable elements are identified - qualitative and (when possible) also quantitative ED-XRF analysis is performed.
    In case of an insoluble sample eventually classical titrimetric analysis is applied.

ELEMENTAL ANALYSIS → **OTHER ELEMENTS**

- **Sample soluble in MeOH**
  (in case of need after heatig, ultrasound treatment or after adding of low amount of other solvents - dioxane, toluene, ACN, DMF, pyridine...)
  Identification and quantitative analysis by ED-XRF (SPECTRO iQII) with a proper experimental method.
  If unexpected elements are present - their identification is done. Furthermore, quantitative analysis is performed, when a calibration method exists.
- **Sample insoluble in MeOH**
  Classical analysis - mineralisation and titration, (only P, S, Cl, Br, I; not in the presence of interfering elements).
  In case of evident difference from theoretical values a control qualitative ED-XRF analysis in suspension or powder form is provided.



Examples of difficult analysis:

Sample I:
Expected composition (rounded):
C 32% H 1% N 5% Br 52%,
structure confirmed by NMR and MS!
Results of CHN analysis:
C 0,8% H 0,05% N 0,2% ?! What was happend?
Sample insoluble in MeOH, we performed ED-XRF analysis in powder form. In measured spectra of sample very weak peak of Br was observed, but extremely strong signal of S was obtained.
After consultation with client: an unsuitable reduction reagent was used during synthesis, elemental sulfur was formed in reaction mixture (elemental sulfur is invisible by NMR and routine MS), sample contained ca. 98% of elemental S.

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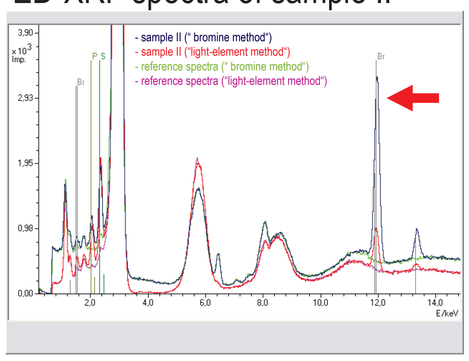
Sample II:
Expected composition:
C 32,44% H 4,42% N 9,46 % S 10,82%
Results of CHN analysis:
C 25,72% H 3,18% N 7,18%
- remarkable difference is observed.
Sample soluble in MeOH - results of ED-XRF S 7,79% and P 8,08%
(anticipated by the client), but also Br 20,85% - not expected - the reaction path was different than proposed.

ED-XRF spectra of sample II



Sample IIIA and IIIB:
Expected composition (both samples):
C 33,9% H 5,12% Br 45,31%
Results of CHN analysis:
IIIA: C 1,11% H 10,97%; IIIB: C 1,36% H 11,25%
Qualitative ED-XRF analysis:
IIIA: only traces of Br (0,4%); IIIB: Br not detected
Conclusion: after synthesis wrong fractions were isolated (dominant water matrix), the subsequent synthesis was succesfull.

Conclusion:
Elemental analysis is besides the established methods of structure elucidation (MS, NMR, IR and other spectral methods) very important analytical methodology for correct characterization of prepared substance. Its absence can lead to very substantial mistakes in interpretation of information obtained. Elemental analysis (with suitable combination of other available methods) is very important and irreplaceable tool for determination of purity of a chemical individuum.