Smart Solid Sample Trace Analysis by ETV-ICP

Some examples for direct analysis by Electrothermal Vaporisation as add-on for ICP

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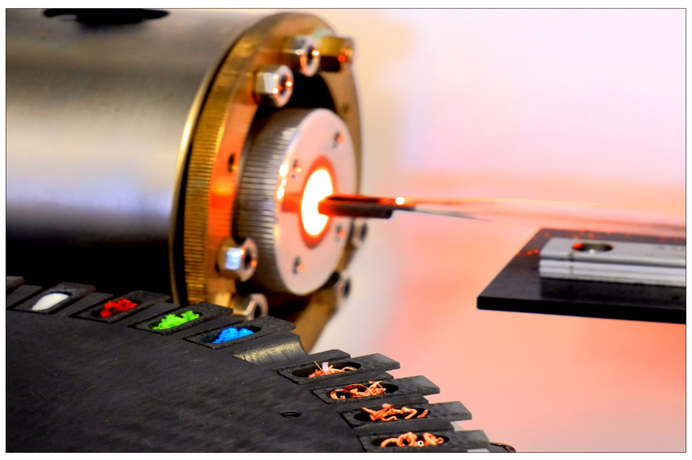
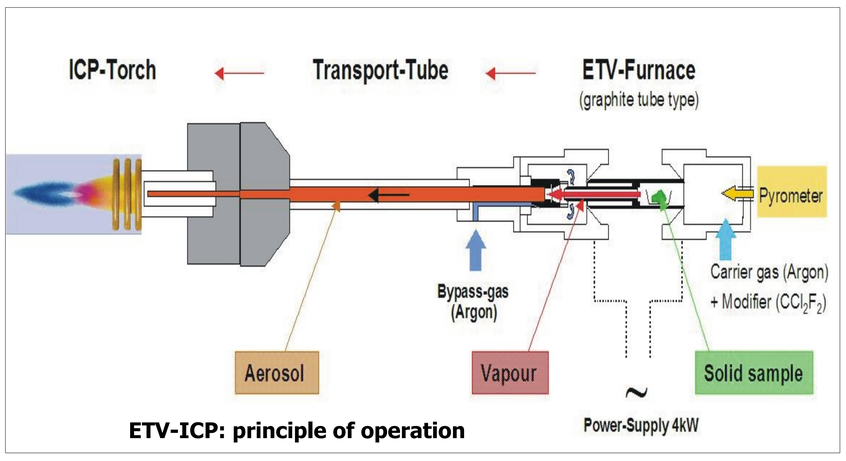
The method ETV - how does it work?

Instead of diluting solid samples (time consuming, pure and often dangerous agents needed, contaminations..) some mg (<1 to 20mg, up to 300mg for carbon matrices) are weighed directly into a graphite boat to be evaporated accordingly in a special graphite tube furnace, continuously adding some halogenated modifier gas. This halogenisation transfers carbide forming elements into volatile halogenides. Even elements like W, Mo, V can be analysed easily. The resulting vapour is transfered into an aerosol by an additional Argon stream and is lead directly into an ICP-injector by a short tube. Here the excitation takes place. Arbitrary temperature programs can be run to separate elements in time, for example to avoid spectral interferences or to determine species of an element (e.g. sulfur).

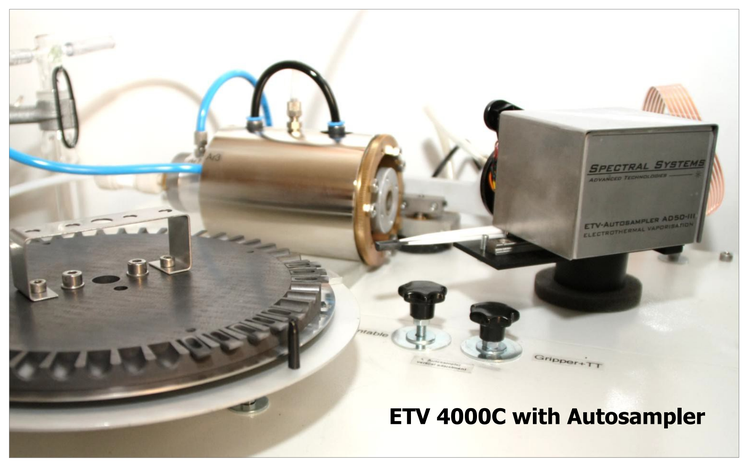
Calibration in ETV-analysis

One of the most important aspects of each analytical method are its possibilities of calibration, especially for solid sampling methods. How difficult is a calibration, are reference materials available, or is it even possible to work without solid SRM´s ? While liquid analysis (like ICP-OES) is mostly free of such issues, for many applications solid SRMs are not available. But in ETV calibration can be done easily using common aquaeous standards, dried directly in a boat, and frequently even without matrix-compensation.
The results below demonstrate that this easy procedure helps to achieve excellent results in a wide field of applications of ETV-ICP. Analysing different materials such as copper, graphite, SiC, boron-nitride and apple-leaves (as an example for many organic samples) the calibration-functions of some elements show that a convenient calibration using solid as well as aquaeous standards is common.





ETV-ICP: principle of operation



ETV 4000C with Autosampler

Instrumentation

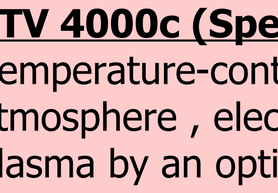
ETV 4000c (Spectral Systems):
Temperature-controlled evaporation of the sample (1-20 mg) in a graphite boat, placed in a graphite-tube furnace under argon atmosphere , electronic controlled gas-flow and addition of a halogenated reaction-gas (modifier). Transport of the aerosole to the ICP-plasma by an optimised gas guide with high transport-efficiency. Integrated microprocessor-control with graphic LCD-display, electronic gas-mixing, synchronisation with the spectrometer by electronic interface. Real-time touchless temperature-measurement of the boat itself (integrated pyrometer) and automatic temperature control (up to 3000°C). Compact modern power-supply (5 kW).

Autosampler AD-50-III (Spectral Systems):
Automatic boat handling from a tray with 50 boats, about 2 hours needed for analysis of 50 samples with arbitrary number of elements

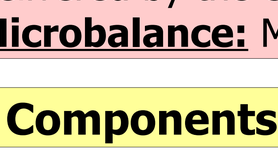
ICP-Spectrometer (here: OES):
IRIS intrepid (Thermo), Arcos-EOP (Spectro): simultaneous data acquisition, possibility to display and evaluate the transient signals delivered by the excited elements, start triggered by the ETV (for automatic runs)

Microbalance: Mettler M2P

Components



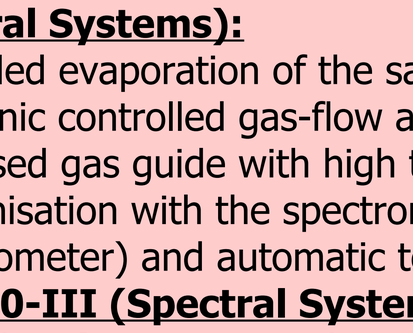
Graphite-Boats

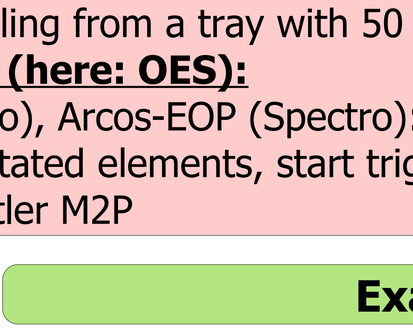


Graphite-Tubes

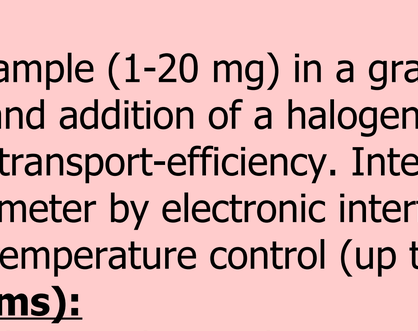
Examples for Metal, Graphite, Organic and Ceramic Material

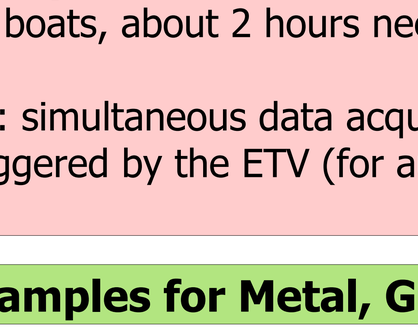
High-Purity Copper

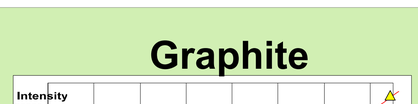




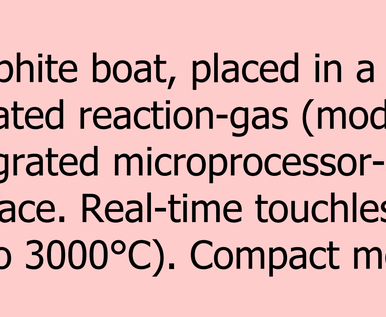
Graphite

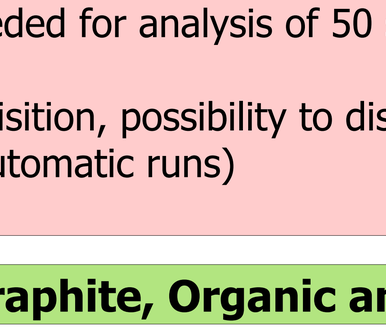


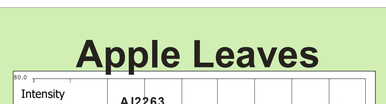




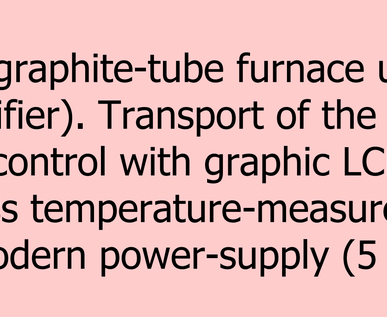
Apple Leaves

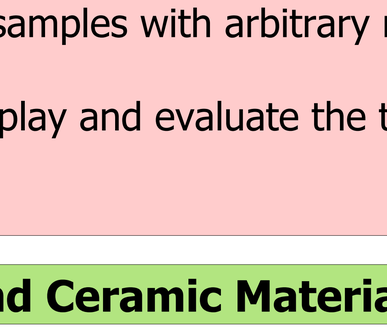


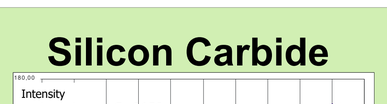




Silicon Carbide







Boron Nitride