DIRECT SOLID SAMPLE ANALYSIS USING A MODERN DC-ARC-OES INSTRUMENTATION

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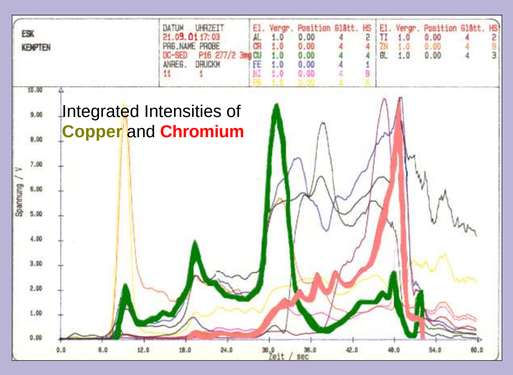
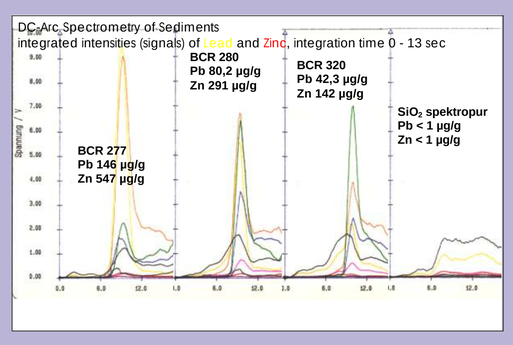
DC-arc: principle of operation

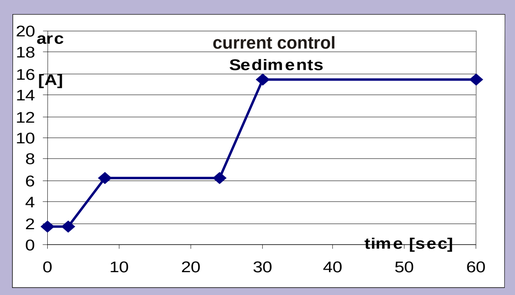
evaporation and excitation of the sample in a graphite-carrier-electrode by an arc-plasma under a shilding Argon-flow. Optical coupling to nearly any (simultaneous) OES-spectrometer by a UV-stabilised quartz-fibre-optic, synchronisation by electronic interface. Integrated microprocessor-control with graphic LCD-display for power and gas-flow. Electrode-gap adjustable (also while operation), bright focusing-screen for a good observation of the process.

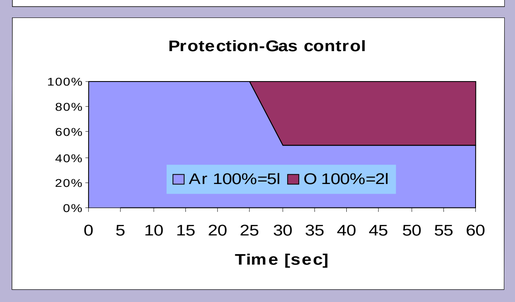
DC-Arc Spectrometry of Sediments

The experiments show the analytical possibilities in solid sample spectroscopy using dc-arc-OES. By separation of the integration-intervals for low- medium- and highboiling elements by a suited current- and gas-control (Ar resp. Ar/O2) good results are achievable in a easy way. The method is demonstrated with time-scans for the examples Zn and Pb (low boiling) and Cu, Cr (high boiling). The concentration-depending peak-areas (intensity versus time) are documentated for Pb and Zn.



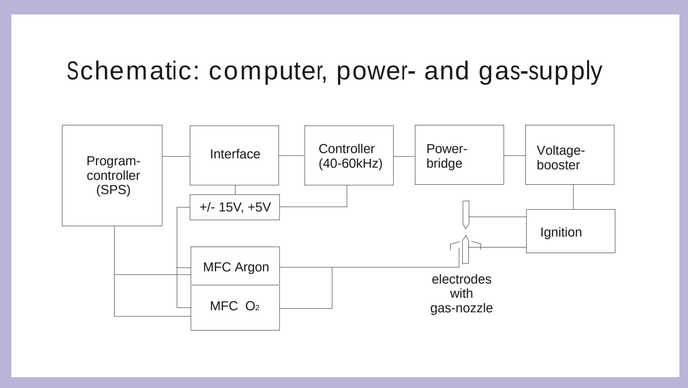






Experimental setup for dc-arc-OES:

Spectrometer: GDS-750, Leco Technik GmbH Vacuum-Polychromator (Paschen-Runge configuration),
39 elements, Argon rinsed.
Spectral data: Focal distance: 750mm, spectral range 150-456nm, lin.dispers. 0,55nm/mm
Signal detection: Photomultipliers, up to 1000 measurements/sec./element
dc-arc-unit: Jarrell Ash Fisher scintific, modified with fibre-optics.Power supply: P.Perzl; Spectral Systems; current 1,5-30 A, computer-controlled; electronic gas control



Double-fibre-optics: compensation of arc fluctuations

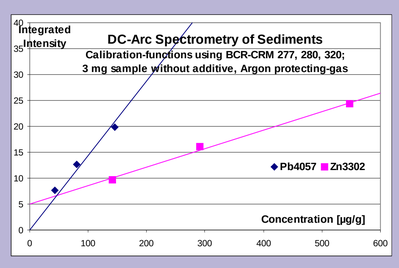
- homogeneous light-distribution at the spectrometer´s entrance
- compensation of local arc fluctuations
- flexible installation and adaption

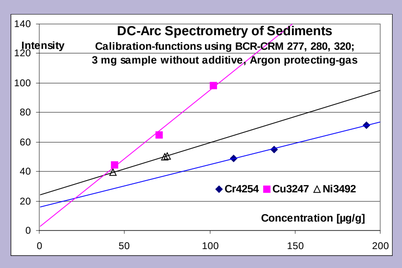
The Used Calibration Techniques

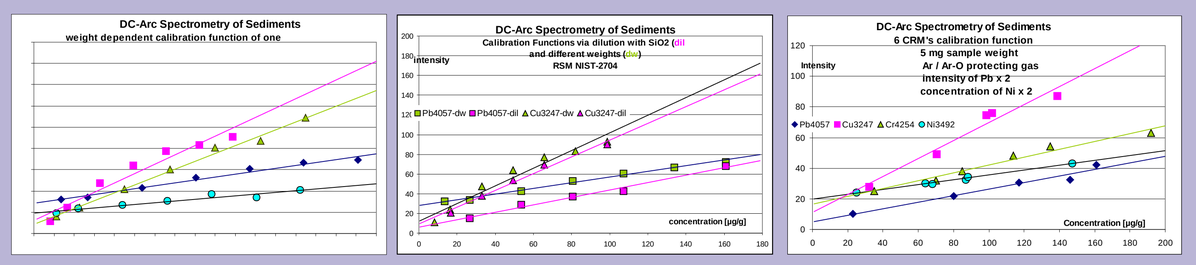
Standard Addition (sample dilution):
different behavior of diluted standards and solid samples in the heating process

Standard Reference Materials:
available only in a small range of concentrations

Different Weights of SRM:
the only correct final solution; the same matrix and the same behaviour of analytes



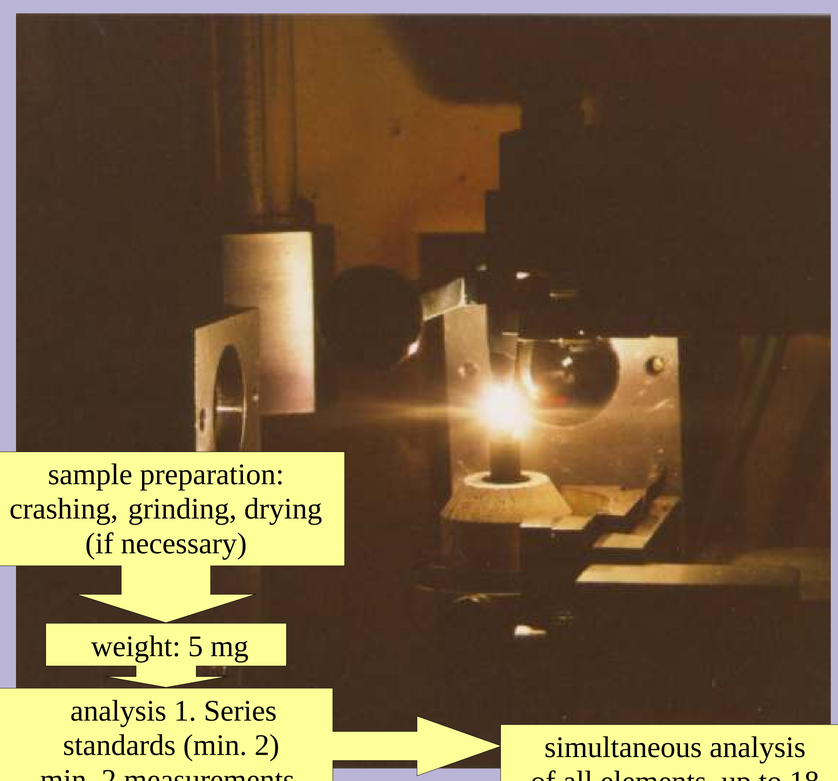




Concentrations of used standards

	BCR277	BCR280	BCR320	NBS2704	GSD5	GSD9I	SL1
Cr	192	114	138	135	70	85	104
Cu	102	70,5	44,1	98,6	137	32,1	30
Ni	43,4	73,6	75,2	44,1	34	32,3	44,9
Pb	146	80,2	42,3	161	112	23	37,7
Zn	547	291	142	438	243	78	223

DC-arc-trace-analysis: example for a typical procedure



sample preparation: crashing, grinding, drying (if necessary)

weight: 5 mg

analysis 1. Series standards (min. 2) min. 2 measurements

analysis of the samples 3 weights per sample)

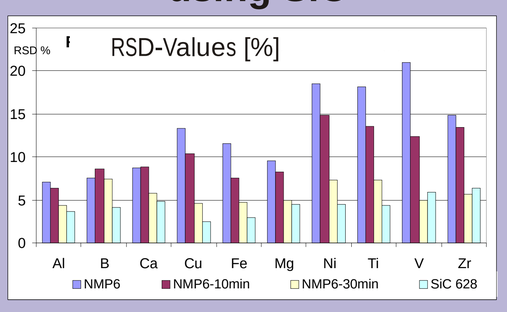
analysis 2. Series standards (min. 2) min. 2 measurements

simultaneous analysis of all elements, up to 18 spectral lines
evaluation of the signals
integration of the intensities
transfer of the results to evaluation-program

assessment of calibration-functions and control-analysis (standards)

documentation of the results

RSD-values dependent on grain-size in DC-arc-analysis using SiC



RSD depending on grain-size samples: NMP6 and SiC 628

Conclusion:

DC-arc in connection with a modern ICP-OES is a very sensitive and fast method in routine analysis. Precision and reproducibilities are convincing.
Commercial add-on-instruments with modern technology are an excellent completion of the possibilities of a good simultaneous spectrometer. The new optical coupling method of the arc to the spectrometer by special fiber-optics makes retrofits easy and gives high flexibility in changing from direct solid sampling to liquid analysis, the fluctuations of the arc on the electrode are partly compensated - this results in better RSD´s.

The presented results demonstrate the easy possibilities of dc-arc.The found limits of detection were less than 1-20µg/g dependent of analyt, less than 10% RSD are possible.

The field of applications ranges over nearly all solid samples, from simple qualitative tests up to trace-analysis. Examples are ceramics, metals, graphite, geological and environmental samples.

A major advantage of dc-arc is the complete absence of memories.