

Investigations for Calibration in DC-ARC-OES

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Introduction

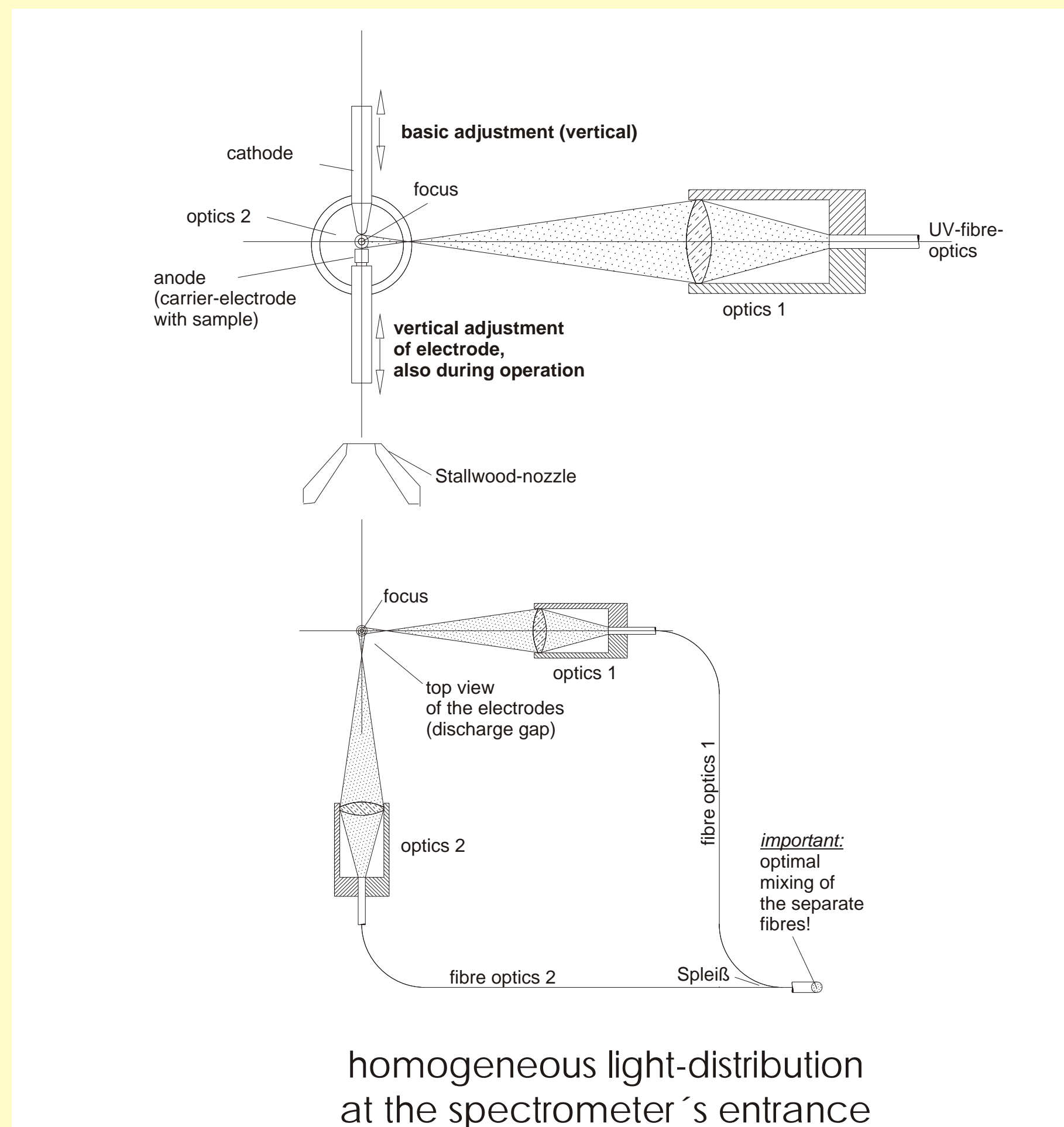
The common problem of all solid sampling spectrochemical methods is the adequate calibration. Mostly only a limited number of certified reference materials (CRM-s) is available. Therefore besides the use of solutions standards some other calibration procedures were proposed as the so called one-standard method. Here the change of the independent variable - the concentration - is achieved by applying different weights of one standard. The classical calibration with a set of calibration samples is compared with the one-standard-method in this work. In the past the bowl-electrodes were completely filled and crammed to ensure a constant sample weight. Today for the shown work - but also for routine analysis - the individual weights are taken by a microbalance with a precision of 0,001 mg. No additives have been added to the samples.

The results are shown as calibration functions for variable and constant weights. Some predetermining criteria (normality, homoscedasticity) and validation characteristics as linearity, goodness of fit, precision of the method, etc. were calculated in spectrochemical analysis of sediment's certified reference material (CRM's).

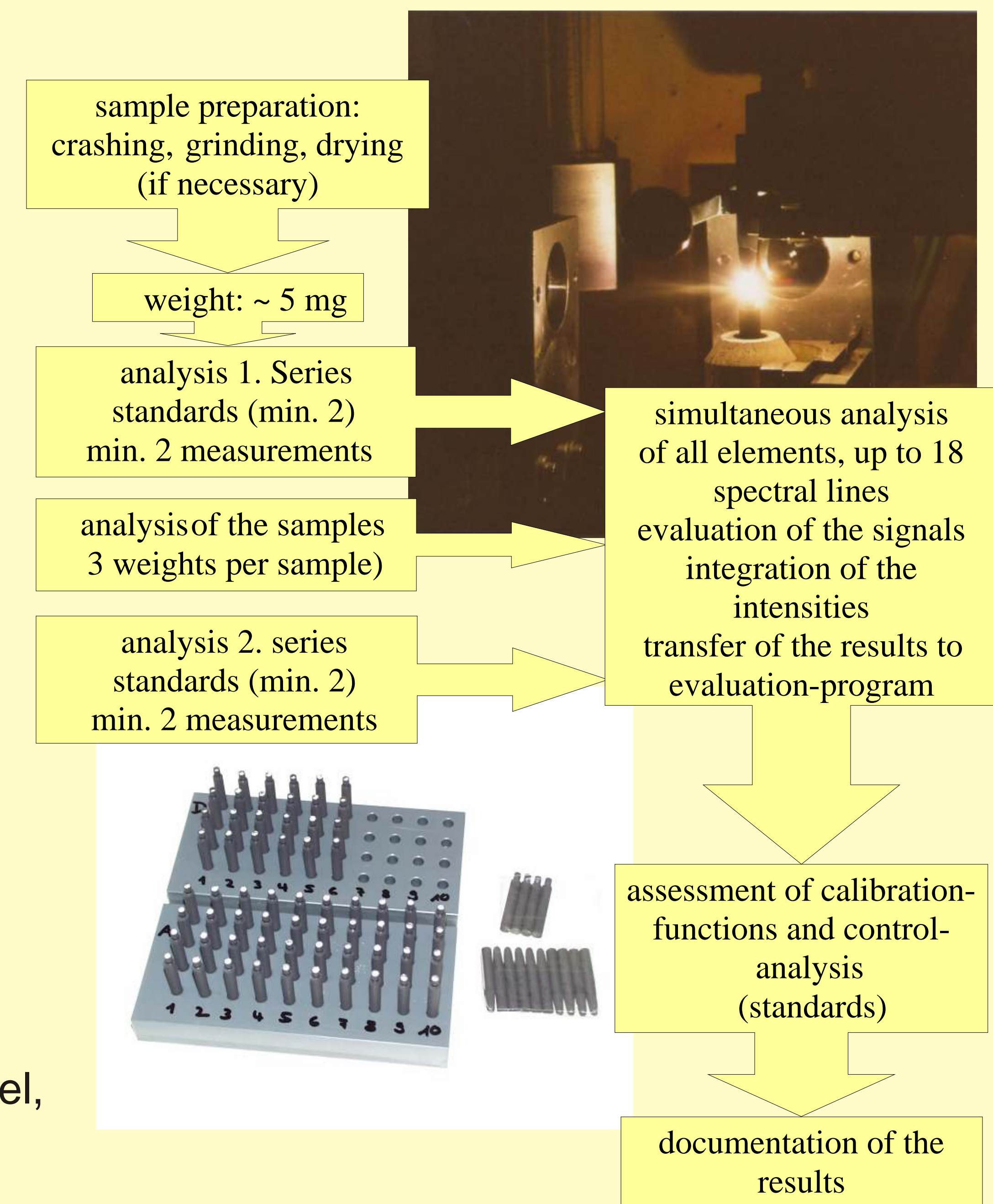
DC-arc-system DCA-301 (Spectral Systems) : principle of operation

Evaporation and excitation of the sample in a high-resistance graphite-carrier-electrode by an arc-plasma under a shielding argon/oxygen-flow. Optical coupling to nearly any (simultaneous) OES-spectrometer by a UV-stabilised quartz-fibre-optic. Double-view-optics for compensation of arc fluctuations and exclusion of emissions of the electrode tips. Synchronisation by electronic interface. Integrated microprocessor-control with graphic LCD-display for power and gas-flows. Electrode-gap adjustable during operation, bright focusing-screen for a good observation of the process.

Double-fibre-optics: compensation of arc fluctuations



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



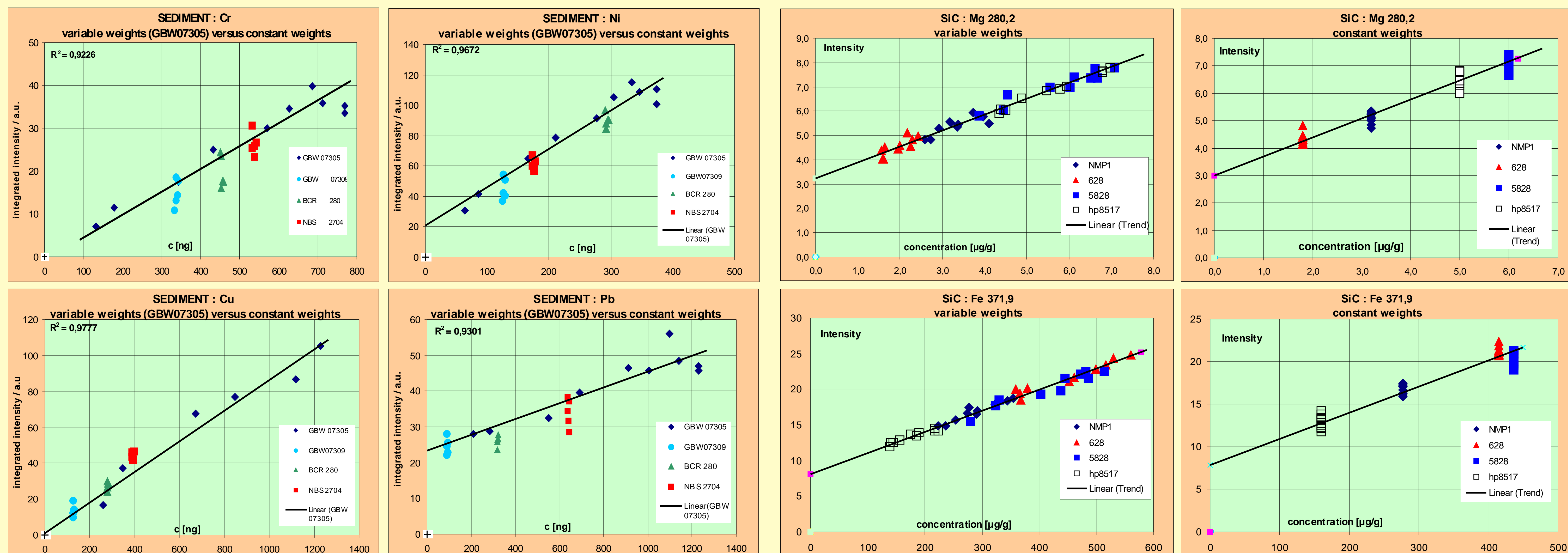
Spectrometer:

Two setups were used:

- Leco GDS-750 (Leco Technik GmbH) (Paaschen-Runge-polychromator, focal distance 750mm, dispersion (1.order) 0,55 nm/mm, 39 photomultipliers (=element channels)

- IRIS-AP (Thermo Jarrell Ash Co.) (Echelle-spectrometer with CID-camera 512x512 pixel, argon-rinsed, focal distance 381mm)

With both setups only the spectrometer-part of the instruments had been used (optical coupling via fibre-optics and adapter)



Sediments: Standard BCR 277, concentrations in [µg/g]				
Element	GBW 07305	GBW 07309	BCR 280	NBS 2704
Cr	425,4	70	85	114
Cu	324,7	137	32	70,5
Ni	349,2	34	32	73,6
Pb	405,7	112	23	80,2

SiC-home-standards, concentrations in [µg/g]				
Elements	NMP1	628	5828	8517
Mg	3,2	1,8	6	5
Fe	277	415	436	160
grain size	<15µm	<1µm	F500	F800
constant weights 5mg±0,1 resp. variable weights 3-7mg				

Conclusion: Calibrating by variable weights (weight depending calibration) can provide reasonable calibration functions. This easy and absolutely equivalent method needs only one (better two) standard materials (SRM's). For the individual material and analyte the working range for which the weight dependent calibration is valid (calibration-function) has to be checked. In the past the authors have examined sediments, sludges, B4C and SiC for the suitability of their calibration by variable weights for numerous elements. The presented examples are only a small part of the collected results.

