Excerpt from

Geochemical

ATLAS

Federal Republic of Germany

Distribution of heavy metals in waters and stream sediments

HEINRICH FAUTH, ROLAND HINDEL, ULRICH SIEWERS & JÜRGEN ZINNER

Sampling

Rudolf Wewer, Erich Adick, Klaus Bieramperl, Hubert Böttcher, Wolfgang Bolbecher, Andreas Bruns, Rainer Büsing, Cornelius Carp, Michael Dempewolf, Jörg Detlef Eckhardt, Fred Flohr, Manfred Gläser, Abdul Hanan, Henner Hartmann, Wilfried Kramm, Michael Krieter, Rainer Kretschmann, Jörg Leopold, Wilhelm Nichstädt, Klaus Rust & Horst Wolfrum

Sample preparation

Horst Fechner, Margarete Gryschka, Wolfgang Jürgens, Heidemarie Klesper, Brigitte Meyer & Frank Weinert

Analytics

Hermann Fesser, Ulrich Brinkmann, Joachim Cramm, Wolfgang Glatte, Bodo Harazim, Klaus Last, Bodo Mieke, Jürgen Rausch, Klaus Tacke & Wilhelm Wisch

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Authors' addresses[1],[2]:

Dr. H. FAUTH, Dr. U. SIEWERS, J. ZINNER,
Bundesanstalt für Geowissenschaften und Rohstoffe,
Stilleweg 2, D-30655 Hannover.

Dr. R. HINDEL,
Niedersächsisches Landesamt für Bodenforschung,
Stilleweg 2, D-30655 Hannover

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[1] At the time of digital provision, the authors mentioned are no longer active in the BGR for reasons of age. The digital processing was carried out by U. Rauch, Bundesanstalt für Geowissenschaften und Rohstoffe, Dienstbereich Berlin, Wilhelmstraße 25-30, D-13593 Berlin, Email: uwe.rauch@bgr.de, geochemischeatlanten@bgr.de.

[2] The addresses were updated to the status of the year 2022.

1.2. Sampling and sample preparation

The samples were collected between May and September from 1977 to 1983. As a rule, the weather was not taken into consideration. Only during extreme weather conditions such as severe gales or thunderstorms was sample collecting interrupted. In the upper reaches of streams the water became clear relatively quickly after heavy rainstorms and no significant changes in the composition of the water were detected. The sampling sites were chosen using 1:25,000 topographic maps. The sites were selected to be as random as possible so that no one geological unit would be given preference. Industrial and residential areas (e.g. the Ruhr District, the Rhine-Main area, and Berlin) were not sampled.

Three field units were involved in sample collecting. At each sample site, one litre of stream, well or spring water was collected in a polyethylene bottle and about 500 g stream sediment in a kraft paper envelope. If the stream sediment was too coarse, it was sieved on site and the < 2-mm fraction retained as a sample.

At each site the width, depth and flow velocity of the stream were entered on a field-data card; any evidence that local contamination might be present was noted. The conductivity and pH of the water were also measured on the same day the samples were taken.

The water samples were analysed in a mobile laboratory, parked in each case in the middle of a 3000 - 4 000 km^2 working area. The stream sediment samples were dried in a drying cabinet at 90°C.

In Hannover, the sediment samples were split; one half was kept for reference and the other sieved to < 0.200 mm (80 mesh). About 2 g of the fine fraction was then ground to < 0.03 mm in an agate microgrinder.

After sieving and grinding, some of the material was kept for reference, although in some cases (about 5 % of all samples) the material was completely used up for repeat analyses.

At 6000 of the sites, two more samples were taken as a check; these duplicate samples were subjected to the same preparation procedure as described above.

1.3. Analytical methods

In 1976, the analytical equipment available to carry out the large number of analyses envisaged were an optical emission spectrometer (Jarrell-Ash Atomcomp 750) as the main instrument, an atomic absorption spectrometer (IL 751), a photometer, and a fluorimeter.

During the course of routine analysis with the optical emission spectrometer, however, it became apparent that due to matrix effects (the matrix of the sediment was difficult to evaluate), overlapping of lines, deflagration, and formation of carbides, the reproducibility was inadequate for the following trace elements Ag, As, B, Be, Bi, Cd, Ce, Ga, Ge, La, Li, Mo, Mn, Nb, Sb, Sc, Sn, W, Y, Zn, and Zr. Only the Ba, Co, Cr, Cu, Ni, Pb, Sr, and V analyses were sufficiently accurate.

It was then decided to carry out the bulk of the analyses with the atomic absorption spectrometer since this produced considerably more reliable results for Cd, Cu, Co, Ni, Pb, Zn, and Li dissolved by acid digestion, and for Sn using a special method of digestion. These AA analyses were used for the compilation of this Atlas; the optical emission data were used only for the elements Ba, Cr, Sr, and V.

F, U and W were determined using potentiometric, fluorimetric, and photometric methods, respectively.

All the methods were employed throughout the entire 7-year duration of the program in spite of the fact that improved techniques became available (e.g. ICP).

The detection limits and errors for the individual elements are given in Figures 1 - 3.

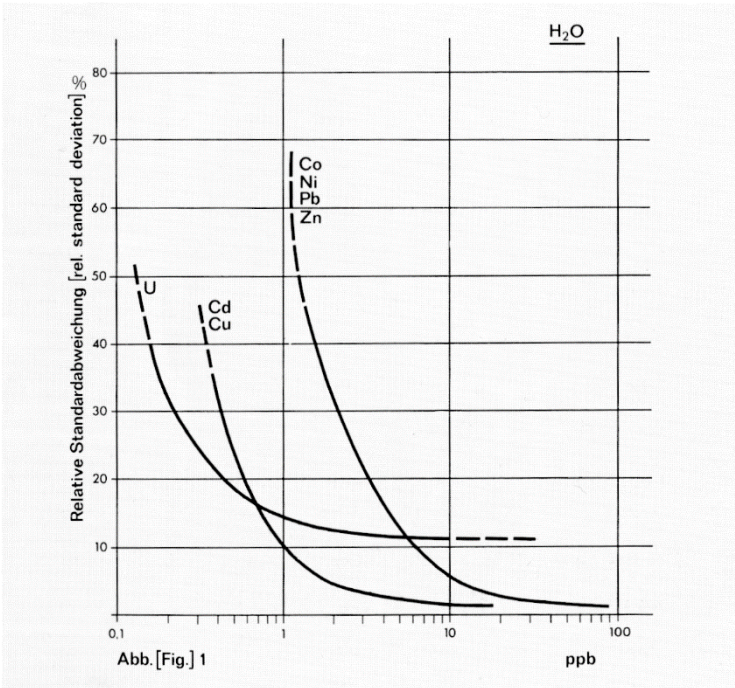

Figure 1. Analytical error (% rel. standard deviation in relation to the value), water

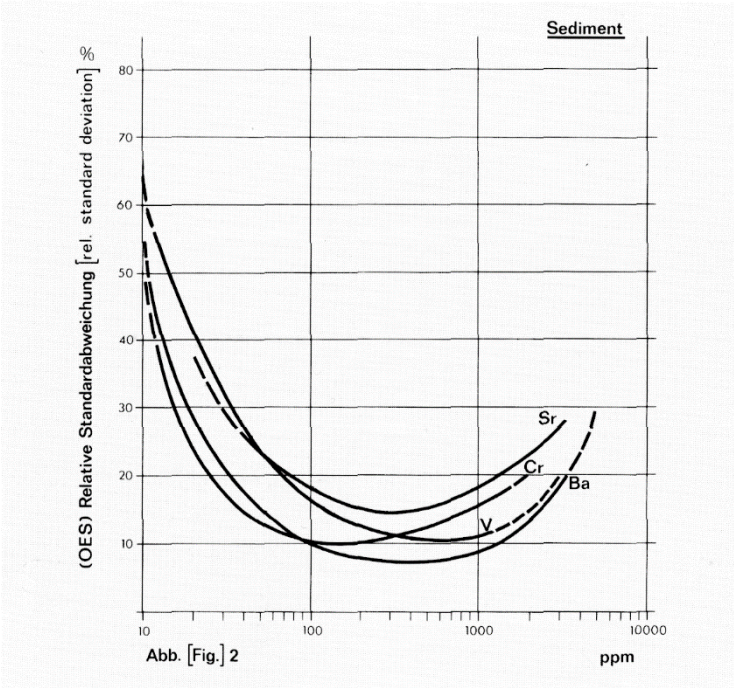

Figure 2. Analytical error (% rel. standard deviation in relation to the value), sediment, OES

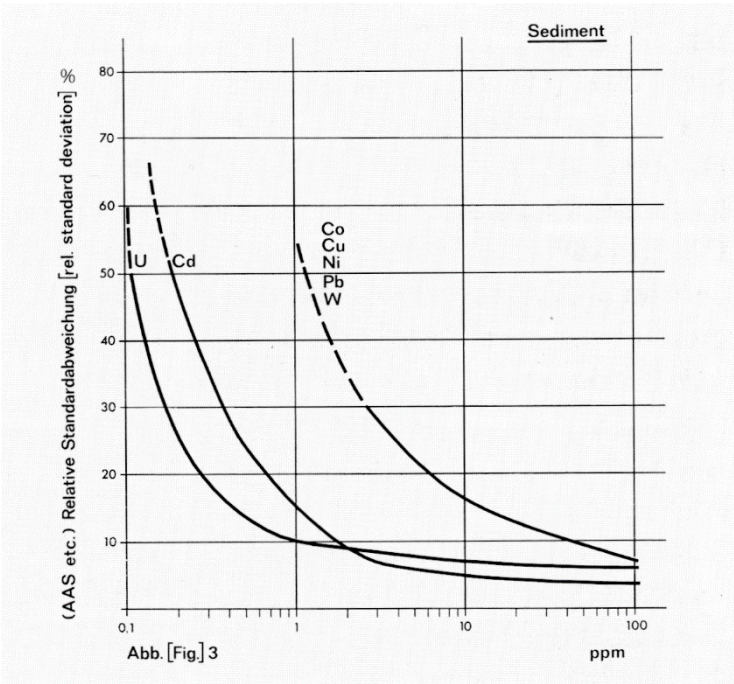

Figure 3. Analytical error (% rel. standard deviation in relation to the value), sediment, AAS e.t.c.

Trace elements in water

Cd, Cu, Co, Ni, Pb, and Zn ions were extracted from 400 ml water with dithizone/butyl acetate and determined by atomic absorption spectroscopy. Uranium was extracted from 100 ml water with tri-n-octylphosphinic oxide/cyclohexane and determined by fluorimetric analysis. Fluoride was analysed in 10 ml water using an ion selective electrode.

Trace elements in stream sediments

Ba, Cr, Sr, and V were determined using an optical emission spectroscopy quantometer on the lines 455.4, 424.5, 460.7 and 437.9 nm, respectively.

For Cd, Co, Cu, Ni, Pb, and Zn, the sample was first digested in 7 N HNO_3 (for Sn the sample was sub-limed with NH_4I) and then analysed by atomic absorption spectroscopy.

After acid digestion, U and F were determined in the same way as for the water samples.

For W, the sample was digested using HCl-$HClO_4$ and determined as a dithiol complex by photometry.

In most cases, the analytical measurements were stored by an online computer.