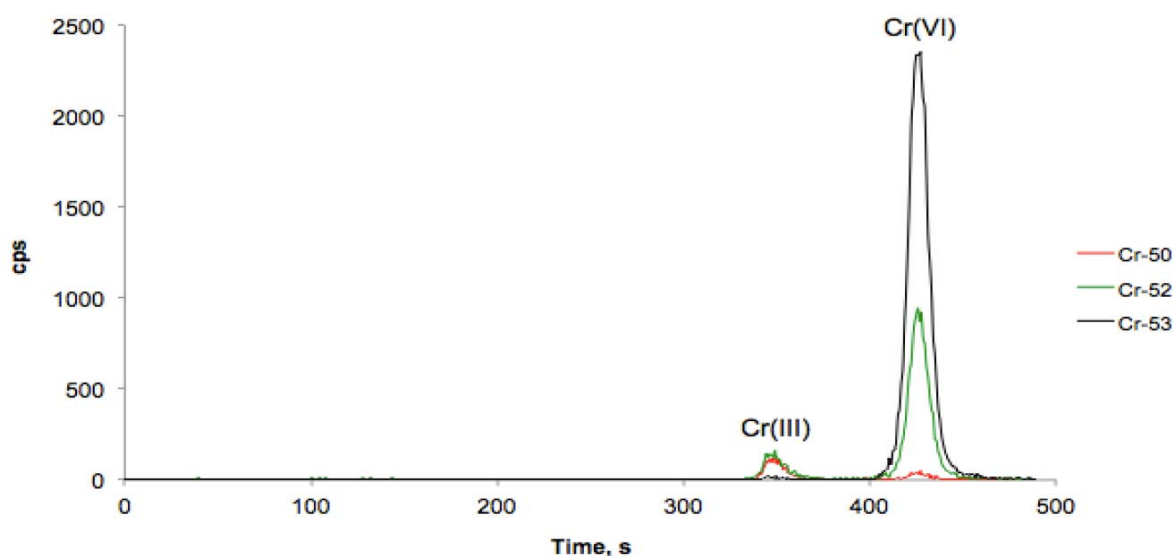


Determination of soluble Cr(III) and Cr(VI) in alkaline soil extract applying IC-ICP/MS



Separation of Cr(III) and Cr(VI) in a soil extract using alkaline solution of EDTA after double-spiking the soil with $^{53}\text{Cr(VI)}$ and $^{50}\text{Cr(EDTA)}$

Soil usually contains small amounts of chromium that stem from weathering rocks or anthropogenic sources. The speciation between trivalent chromium – Cr(III) – and hexavalent chromium – Cr(VI) – is important due to the toxic nature of the latter. Here, the two species are separated as Cr(III)-EDTA complex and chromate on a Metrosep A Supp 4 - 250/4.0 column. For detection, speciated isotope dilution mass spectrometry is applied.

Results

	Cr(III) [$\mu\text{g/g}$]	Cr(VI) [$\mu\text{g/g}$]
Soil (extracted)	11.3	10.1

Sample

Soil (standard reference material)

Sample preparation

Alkaline extraction with EDTA, direct injection after filtration (0.45 µm)

Columns

Metrosep A Supp 4 - 250/4.0	6.1006.430
Metrosep A Supp 4/5 Guard/4.0	6.1006.500

IC Solutions

Eluent	2.0 mmol/L EDTA pH = 10
Extraction solution	50 mmol/L EDTA pH = 10

Parameters

Flow rate	0.8 mL/min
Injection volume (MiPT)	100 µL
P _{max}	12 MPa
Recording time	10 min

Parameters ICP/MS

RF power	1550 W
Plasma gas flow rate	15 L/min
Carrier gas flow rate	0.95 L/min
Makeup gas flow rate	0.15 L/min
Collision gas (He) flow rate	4.0 mL/min
Sampling depth	8.0 mm
Spray chamber temperature	2 °C
Tuning solution	1 µg/L Li, Co, Y, Ce Ti in 2% HNO ₃ solution
Acquisition mode	Spectrum and time resolved analysis
Monitoring masses	50, 52, and 53 amu

Analysis

ICP/MS detection

Instrumentation

940 Professional IC Vario ONE	2.940.1100
ICP-MS Agilent 7700	
858 Professional Sample Processor	2.858.0020
Remote box	6.2148.010
Remote cable Professional IC - MS- Detector (Agilent)	6.2141.380

