

X-Ray-Fluorescence-Analysis of **Granite-like material** using a PANalytical MINIPAL4 Spectrometer

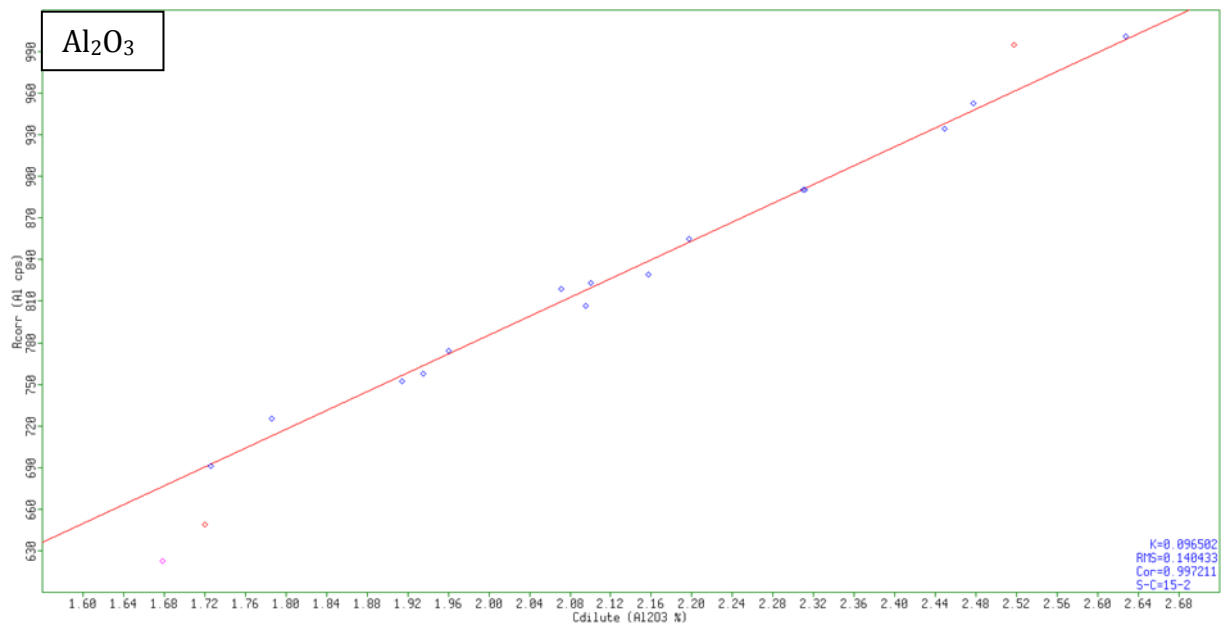
- Institute:** Institute for Geography and Geology,
Dept. for Geodynamics and Geomaterial Sciences
Würzburg University
Am Hubland
D-97074 Würzburg/Germany
- Sample Prep:** Sample powder one night in the drying cabinet at 105°C
600 mg sample powder
3600 mg Merck Spectromelt A12 (66% di-lithium tetraborate + 34%
lithium metaborate) as a flux
1000 mg NH₄NO₃ as an oxidant
Fusion disk: 5 min heating at 300°C, 5 min melting at 1000°C and 5 min
melting at 1080°C
- Machine:** PANalytical MINIPAL4 X-ray fluorescence spectrometer
Side-window Rh-tube with max 30 kV, max 1 mA and max 9 W.
Energy-dispersive Si drift detector.
Sample chamber with He-flush for better detection limits and low
analytical errors.
- Conditions:**
- | | | | | |
|-------|--------|----------------|------|--------------------------------|
| 09 kV | 350 µA | no filter | 600s | for Na, Mg, Al, Si, P, K, Ca, |
| 14 kV | 250 µA | thin Al-filter | 300s | for Ti, V, Cr, Mn, Fe, Co, Ni, |
| 30 kV | 150 µA | Al-filter | 300s | for Zn, Rb, Sr, Y, Zr, Nb. |
- The K α -line is used for measurements except for Na and P were
measurement integrates over a region of interest (ROI) enclosing the
K α -line.
- Calibration:** Eighteen international standards (values from Govindaraju, K. 1989:
Geostandard Newsletter Vol. XIII, Spec. Issue):
Granites: AC-E, G-2, GA, GH, GM, GS-N, GSR-1, MA-N, NIM-G
Granodiorites: GSP-1
Syenites: NIM-S, STM-1, SY-2, SY-3
Rhyolites: RGM-1
Andesites: AGV-1, GSR-2
Quartz-Latite: QLO-1

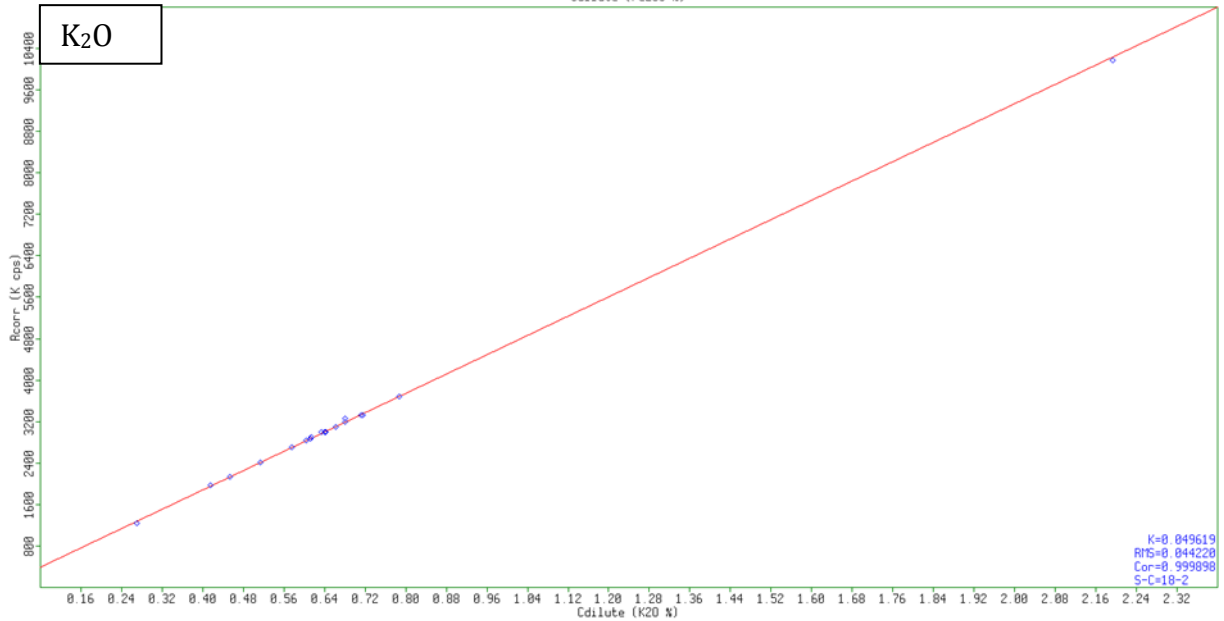
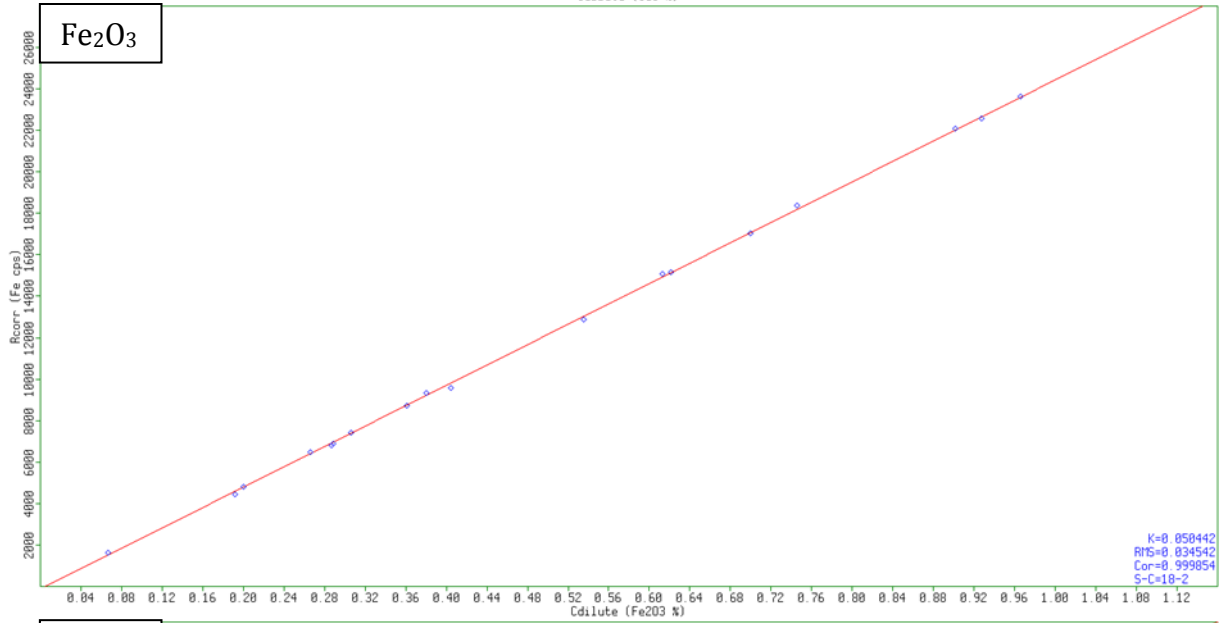
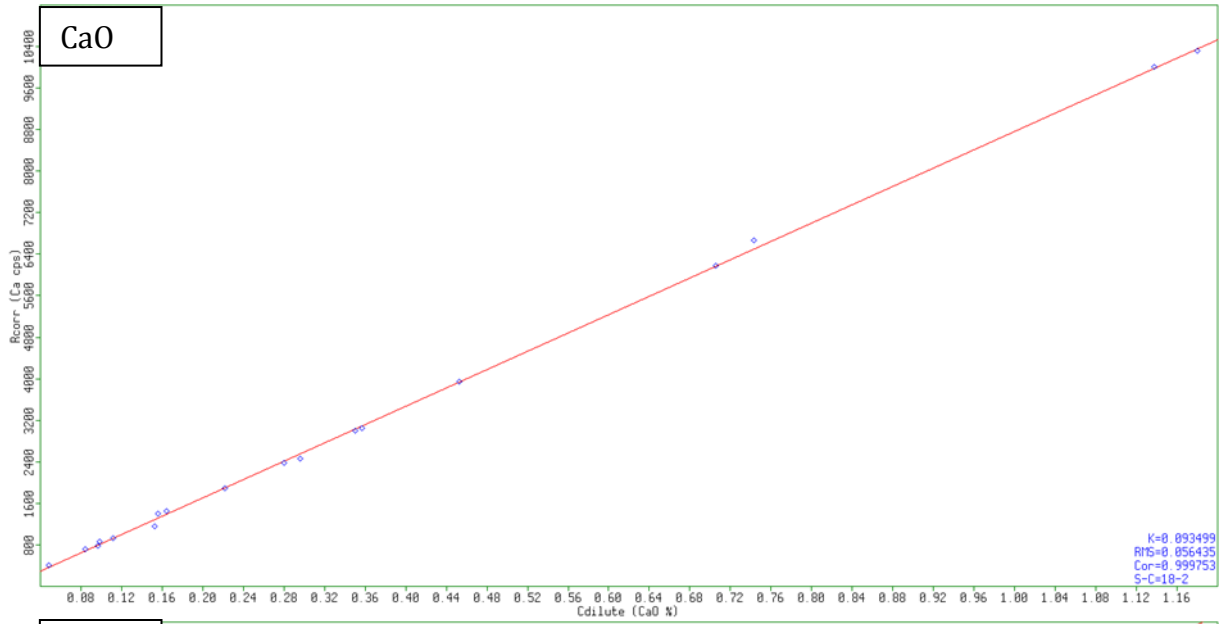
Calibration accuracy:

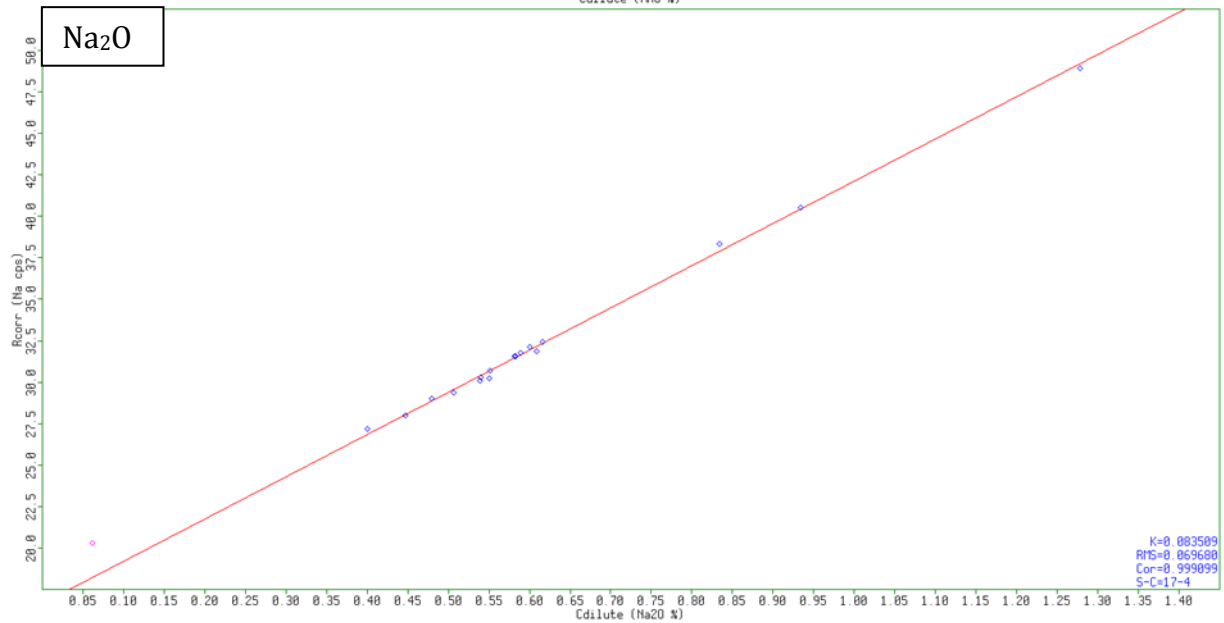
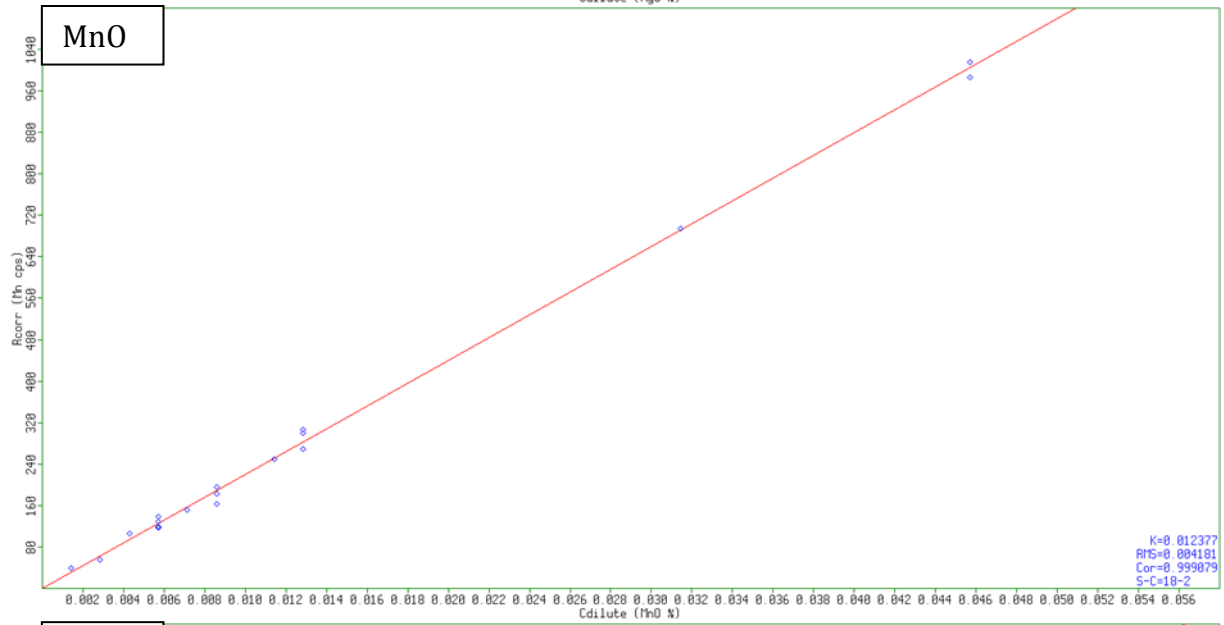
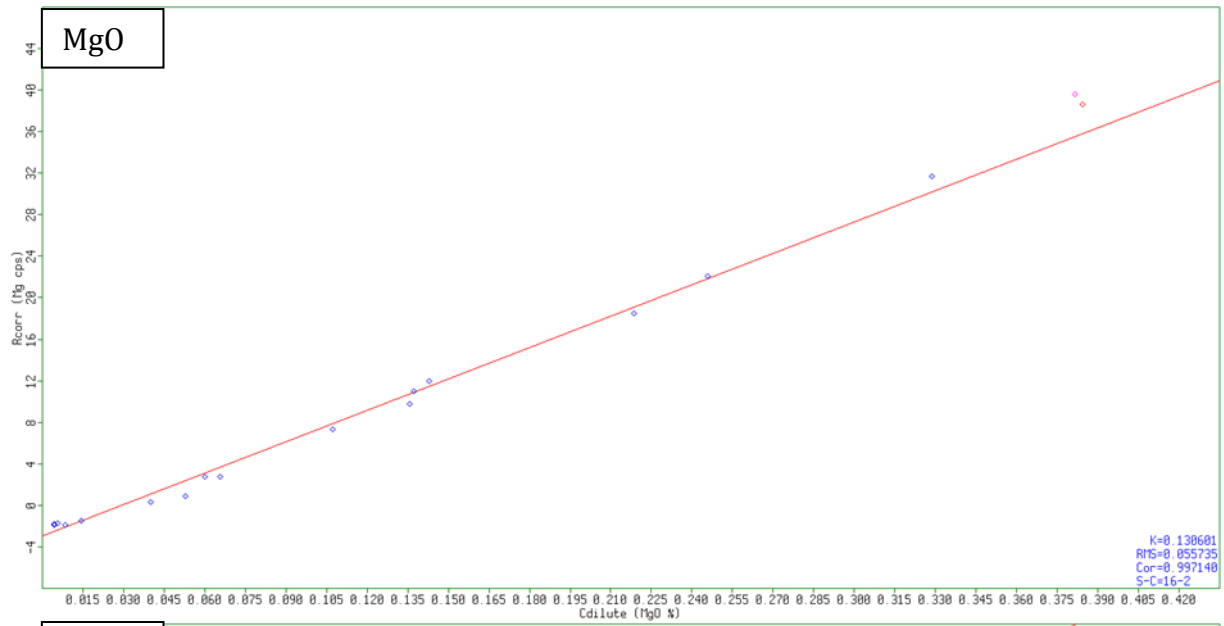
Oxide	Concentration range wt.%	RMS wt.%	Correlation	Standards choked
Al ₂ O ₃	11.7 – 18.4	0.140	0.9972	MA-N, SY-2, SY-3
CaO	0.3 – 8.3	0.056	0.9998	-
Fe ₂ O ₃	0.5 – 6.8	0.035	0.9999	-
K ₂ O	1.9 – 5.5 (15.4)	0.044	0.9999	-
MgO	0.03 – 2.7	0.056	0.9971	SY-2, SY-3
MnO	0.01 – 0.32	0.004	0.9991	-
Na ₂ O	2.8 – 9.0	0.070	0.9991	NIM-S
P ₂ O ₅	0.01 – 1.4	0.031	0.9965	-
SiO ₂	58.8 – 75.8	0.320	0.9985	-
TiO ₂	0.01 – 1.05	0.016	0.9986	G2, NIM-S

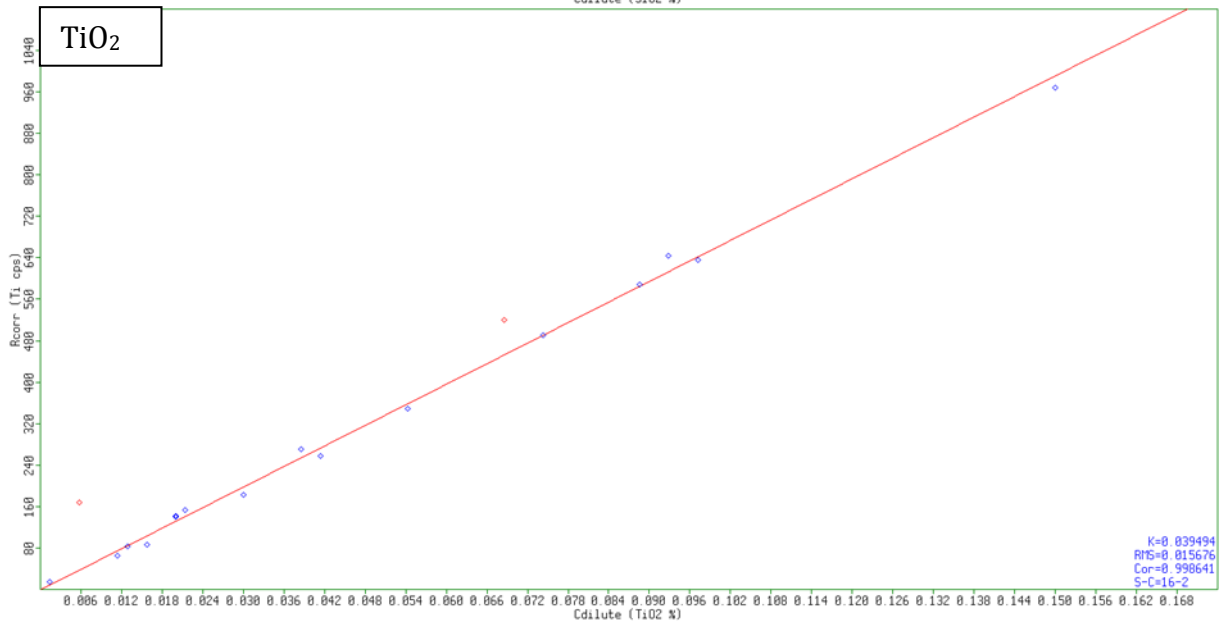
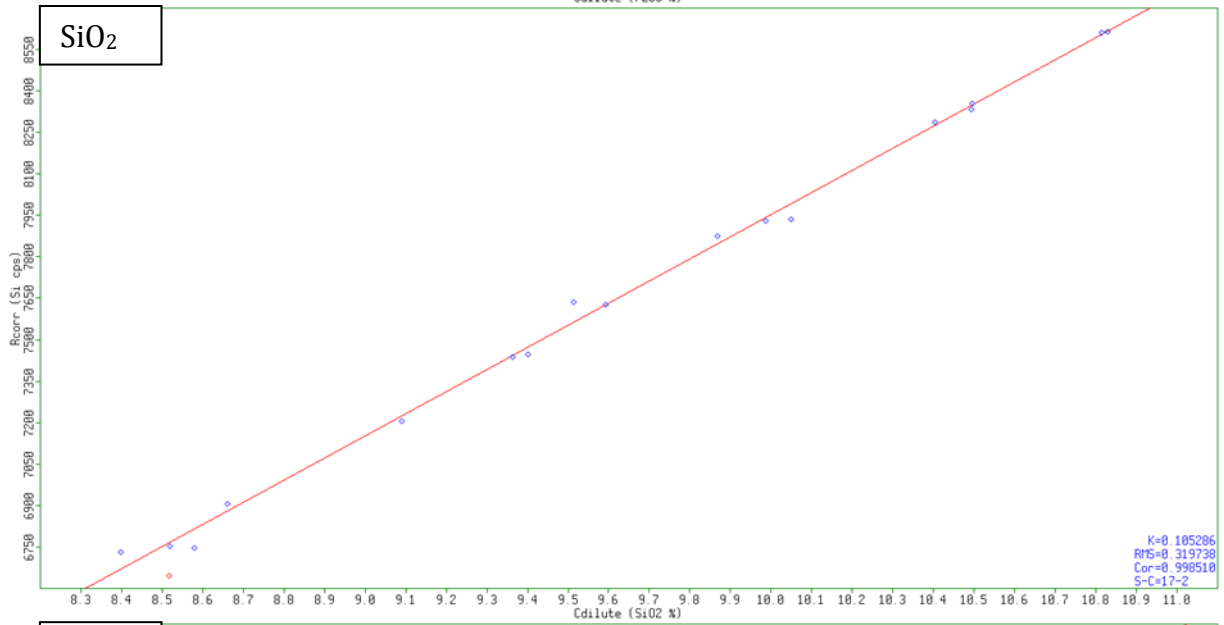
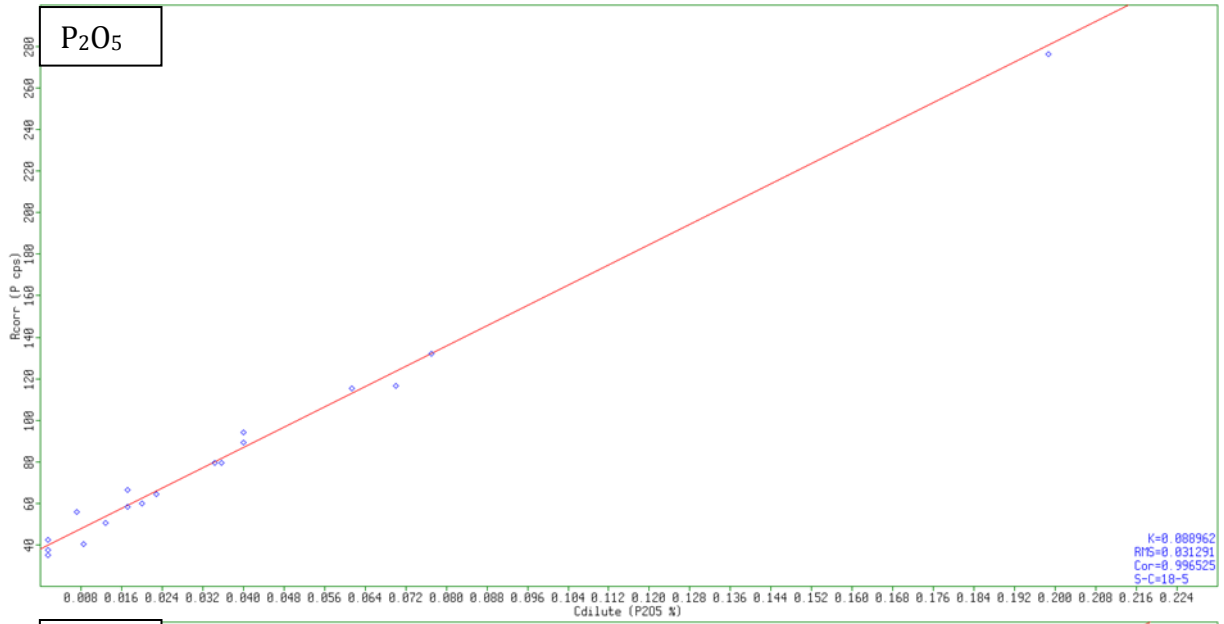
Typical examples for calibration lines:

The lines may slightly differ from re-calibration to re-calibration. A re-calibration is carried out after some weeks of downtime of the instrument. With a running machine, the calibration remains stable for several weeks. (red: standards were not used for calculating the correlation line)









Laboratory-internal analytical error for measurements of unknown samples:

This error was not determined so far for granite-like material. But see „X-Ray-Fluorescence-Analysis of Basalt-like material“