

X-Ray-Fluorescence-Analysis of

Basalt-like material

using a PANalytical MINIPAL4 Spectrometer

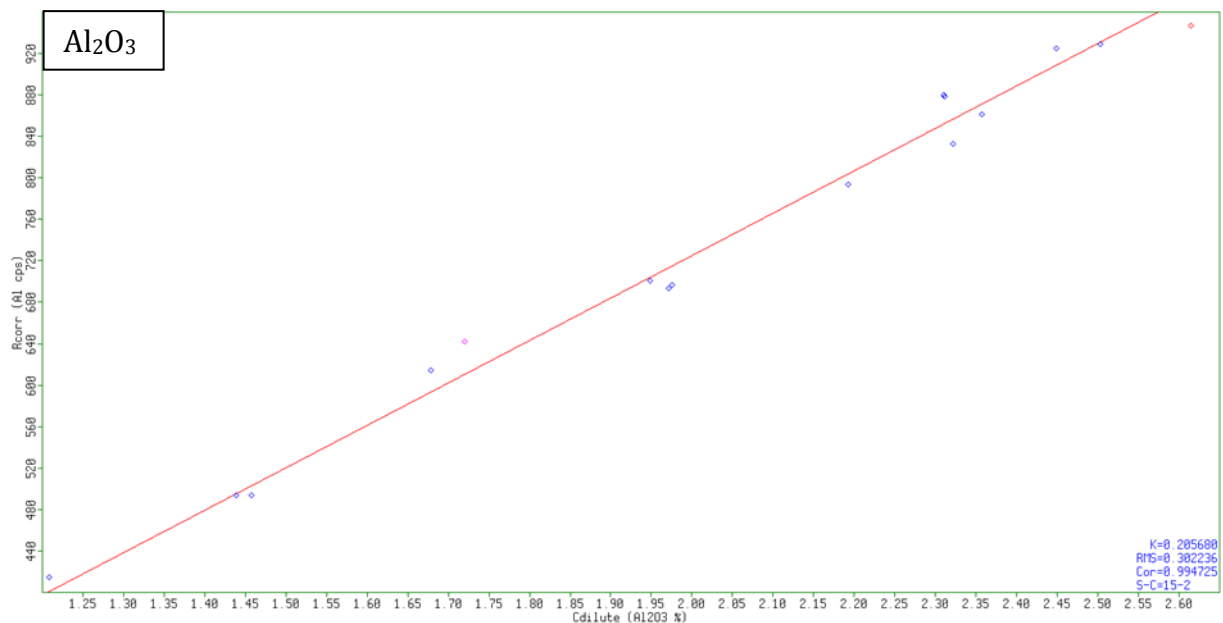
- Institute:** Institute for Geography and Geology,
Dept. for Geodynamics and Geomaterial Sciences
Würzburg University
Am Hubland
D-97074 Würzburg/Germany
- Sample Prep:** Sample powder one night in the drying cabinet at 105°C
600 mg sample powder,
3600 mg Merck Spectromelt A12 (66% di-lithium tetraborate + 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, 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:** Seventeen international standards (values from Geostandard Newsletter Vol. XIII, Spec. Issue 1989):
Basalt: BCR-1, BE-N, BHVO-1, BIR-1, BM, BR, GSR-3
Diabase: DNC-1, W-2
Gabbro/Norite: MRG-1, NIM-N
Diorite: DR-N
Syenite: SY-2, SY-3
Andesite: AGV-1, GSR-2
Quartz-Latite: QLO-1

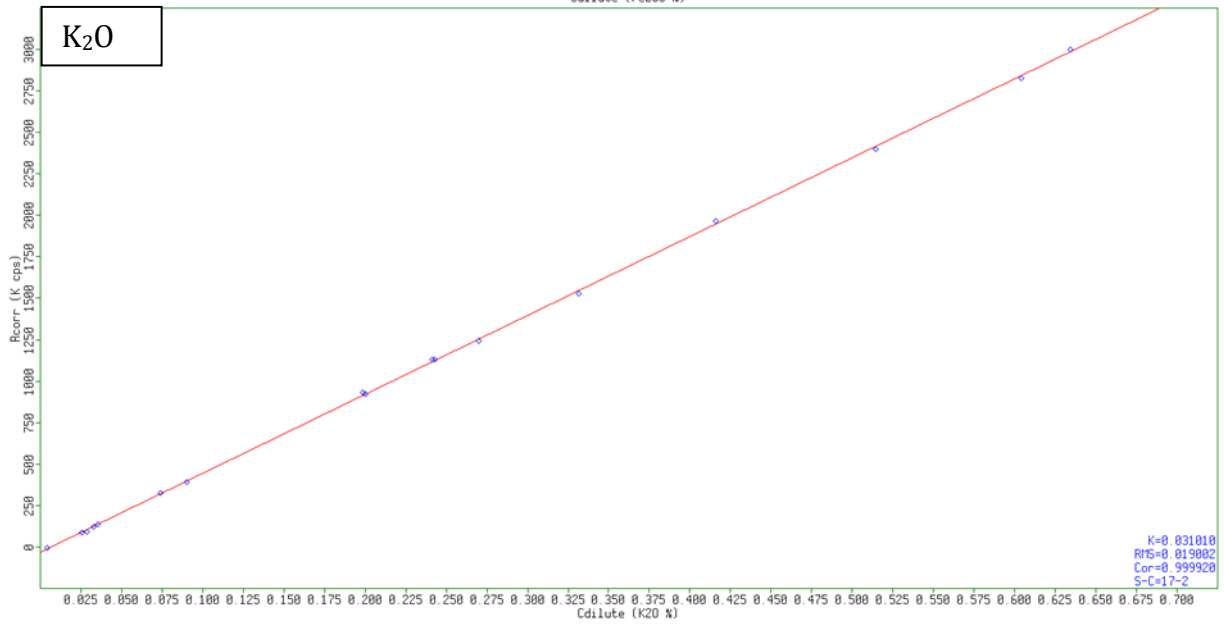
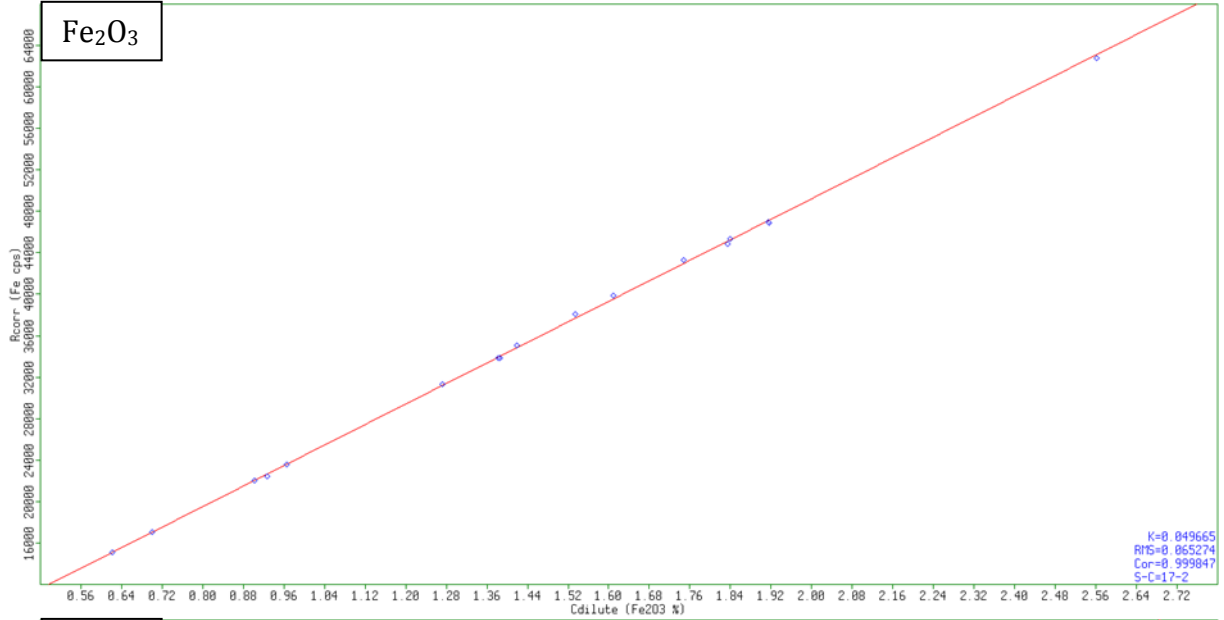
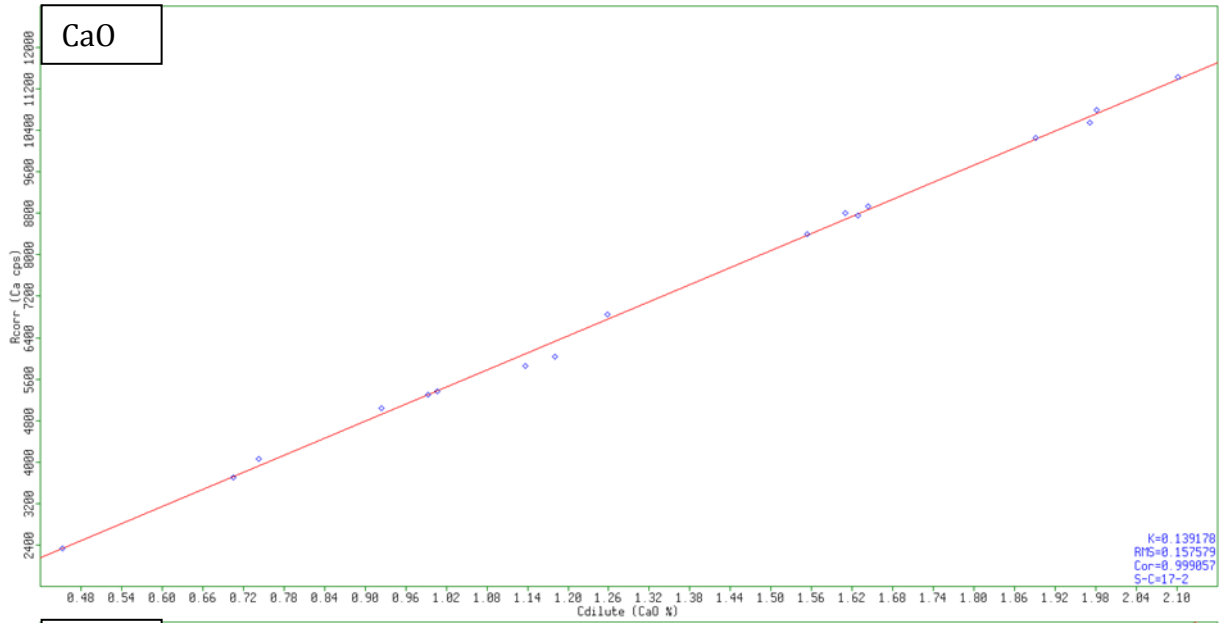
Calibration accuracy:

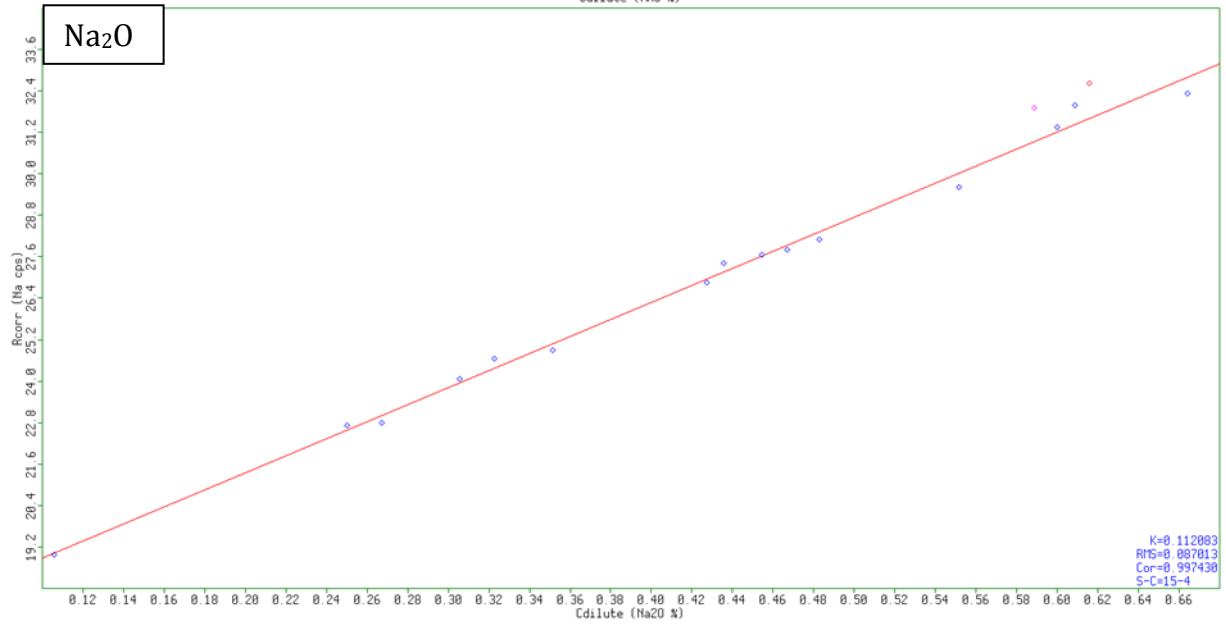
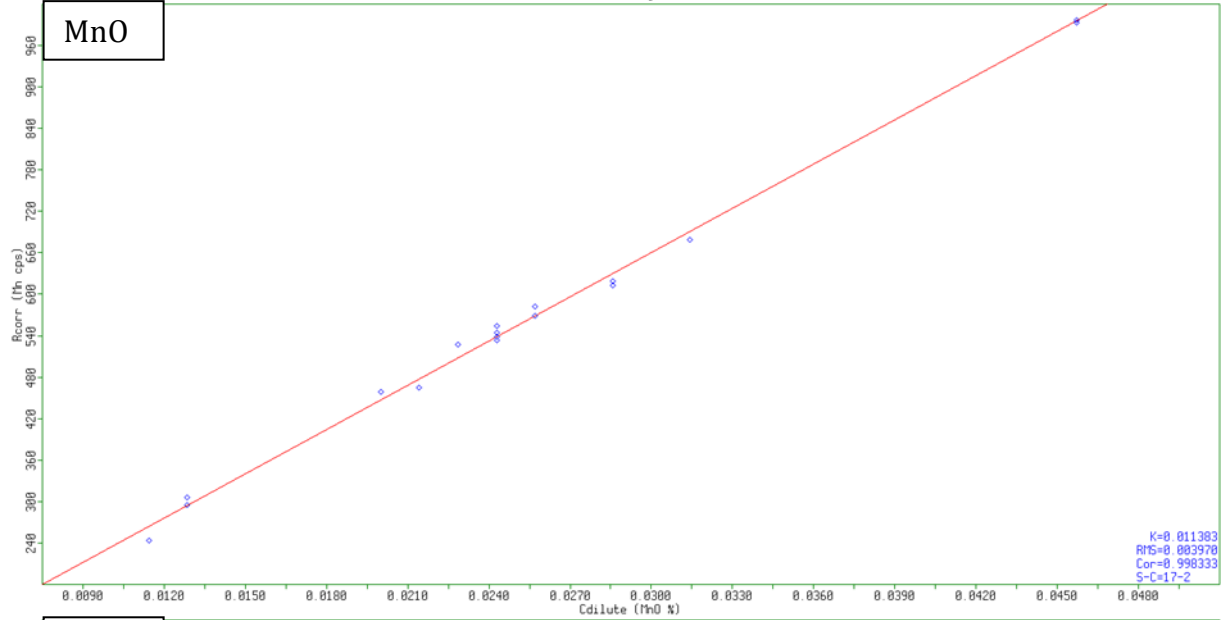
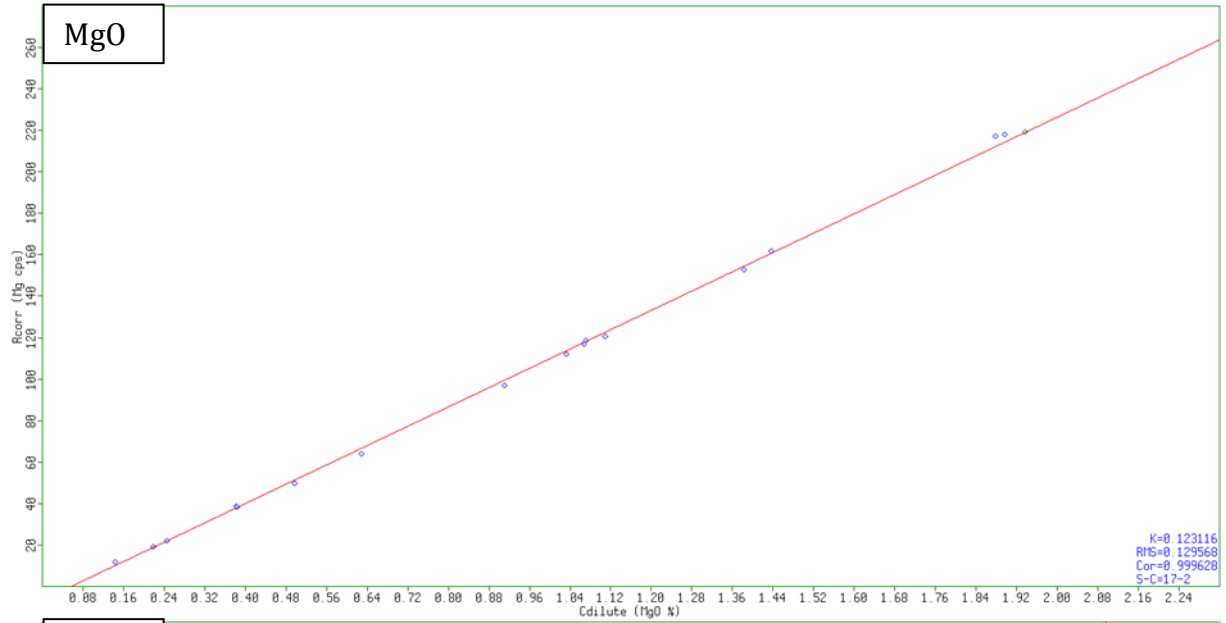
Oxide	Concentration range wt.%	RMS wt.%	Correlation	Standards choked
Al ₂ O ₃	8.63 – 18.30	0.302	0.9947	DNC-1, SY-2
CaO	3.17 – 13.87	0.158	0.9991	-
Fe ₂ O ₃	4.35 – 17.93	0.065	0.9998	-
K ₂ O	0.03 – 4.44	0.019	0.9999	-
MgO	1.00 – 13.55	0.130	0.9996	-
MnO	0.08 – 0.32	0.004	0.9983	-
Na ₂ O	0.74 – 4.65	0.087	0.9974	SY-2, SY-3
P ₂ O ₅	0.03 – 1.05	0.034	0.9962	-
SiO ₂	38.20 – 65.55	0.511	0.9982	-
TiO ₂	0.14 – 3.77	0.046	0.9992	-

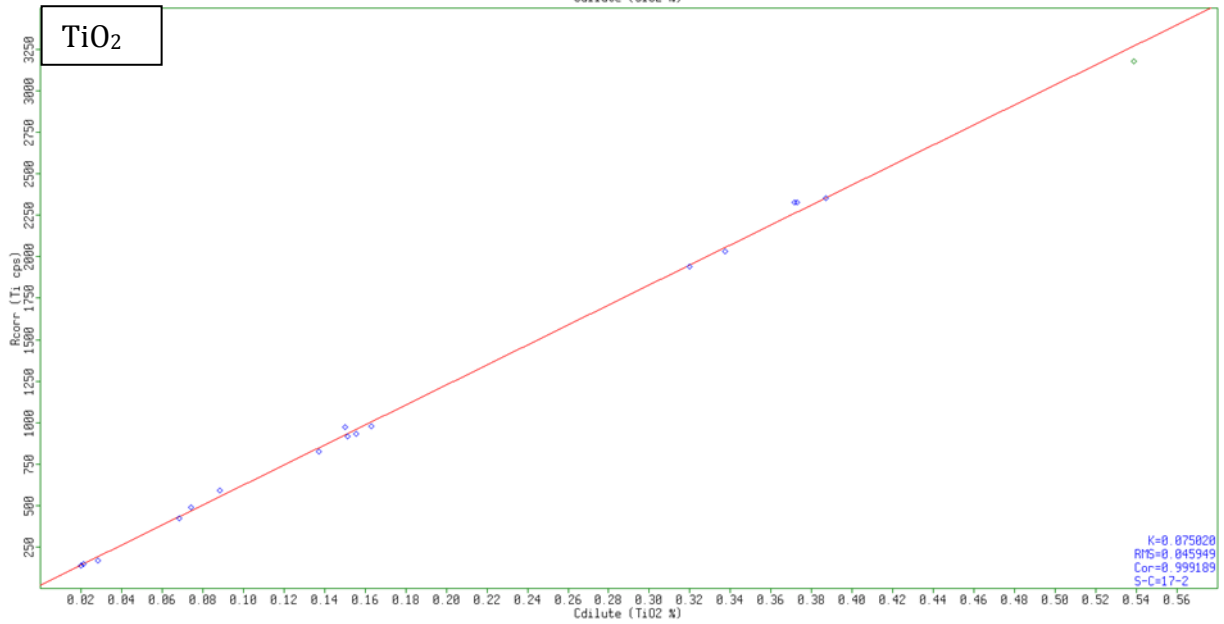
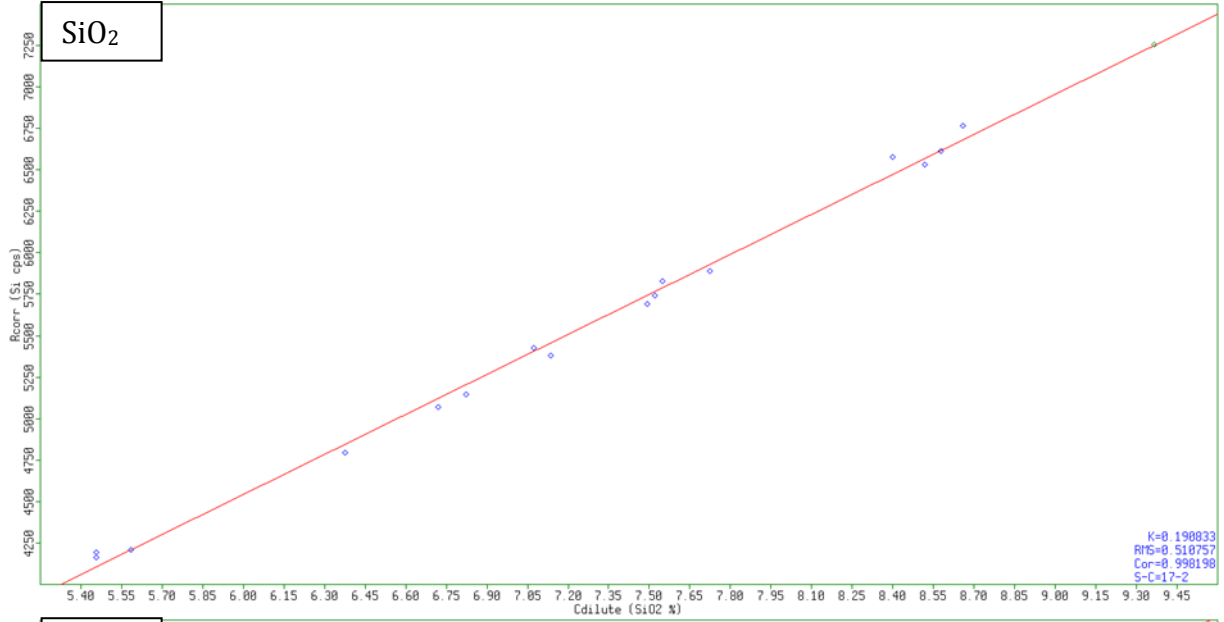
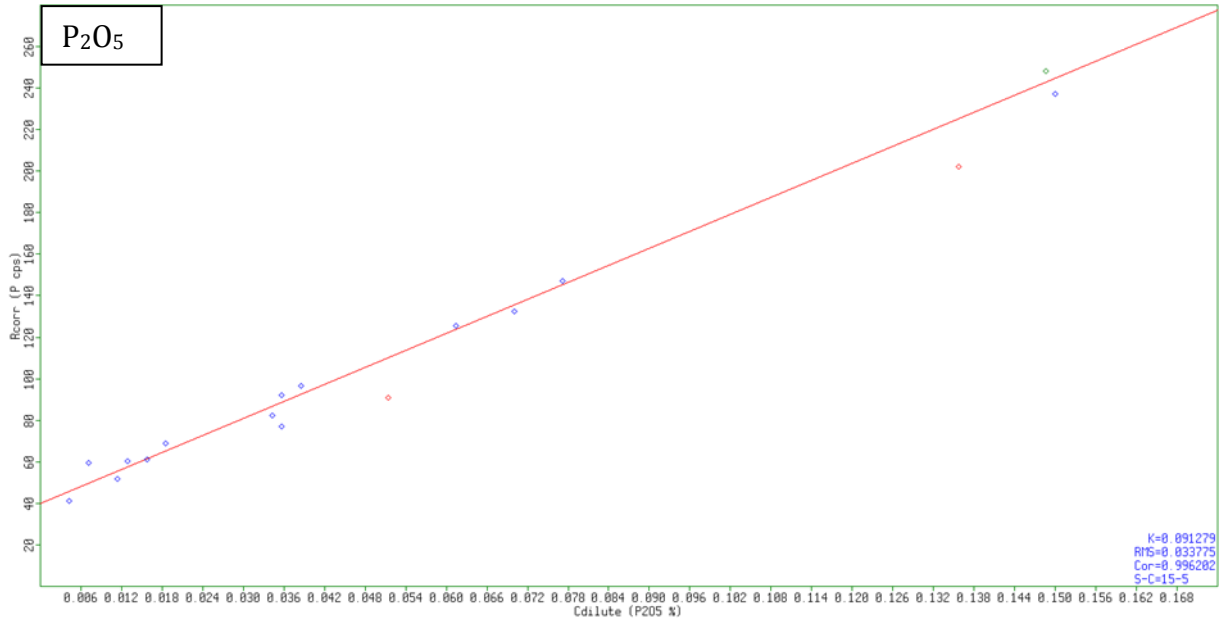
Typical examples for calibration lines:

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:

From one and the same sample powder, an alkaline continental within-plate basalt from Lindenstumpf in the Rhön volcanic chain, 23 fusion disks have been produced by 23 different persons (students who did this for the first time in the course of a lab practise).

The given error therefore comprises the analytical error of the measurements, but also the error of the weighing procedure and the error of the disc fusion procedure. i.e. the laboratory-internal analytical error.

n = 23	Minimum	Maximum	Average	1s (wt.%)	1s (relative %)
SiO ₂	42.29	42.82	42.54	0.15	0.34
TiO ₂	2.90	2.93	2.91	0.01	0.27
Al ₂ O ₃	12.93	13.09	12.99	0.04	0.34
Fe ₂ O ₃	12.14	12.33	12.23	0.05	0.43
MgO	10.21	10.46	10.37	0.06	0.56
CaO	11.25	11.33	11.29	0.02	0.20
MnO	0.17	0.18	0.17	0.00	2.01
Na ₂ O	2.74	3.05	2.92	0.08	2.62
K ₂ O	1.14	1.15	1.15	0.00	0.44
P ₂ O ₅	0.64	0.72	0.67	0.02	3.42

Instrumental analytical error:

One and the same sample (standard BIR-1) was repeatedly measured 25 times. As expected, the instrumental error of the Minipal4 is even lower than the laboratory-internal error:

n = 25	Minimum	Maximum	Average	1s (wt.%)	1s (relative %)
SiO ₂	47.08	47.36	47.20	0.08	0.17
TiO ₂	0.93	0.93	0.93	0.00	0.00
Al ₂ O ₃	15.10	15.20	15.13	0.03	0.20
Fe ₂ O ₃	11.29	11.34	11.32	0.01	0.09
MgO	9.43	9.60	9.51	0.05	0.53
CaO	13.25	13.28	13.27	0.01	0.08
MnO	0.17	0.17	0.17	0.00	0.00
Na ₂ O	1.65	1.91	1.76	0.06	3.41
K ₂ O	0.03	0.04	0.04	0.00	0.00
P ₂ O ₅	0.10	0.11	0.11	0.00	0.00