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CryPhysConcept
Mit Kristallphysik zum Zukunftskonzept elektrochemischer Energiespeicher

INFLUENCE OF ENVIRONMENTAL PARAMETERS VARIATIONS ON X-RAY BEAM INTENSITIES - A TIME-DEPENDENT ABSORPTION CORRECTION

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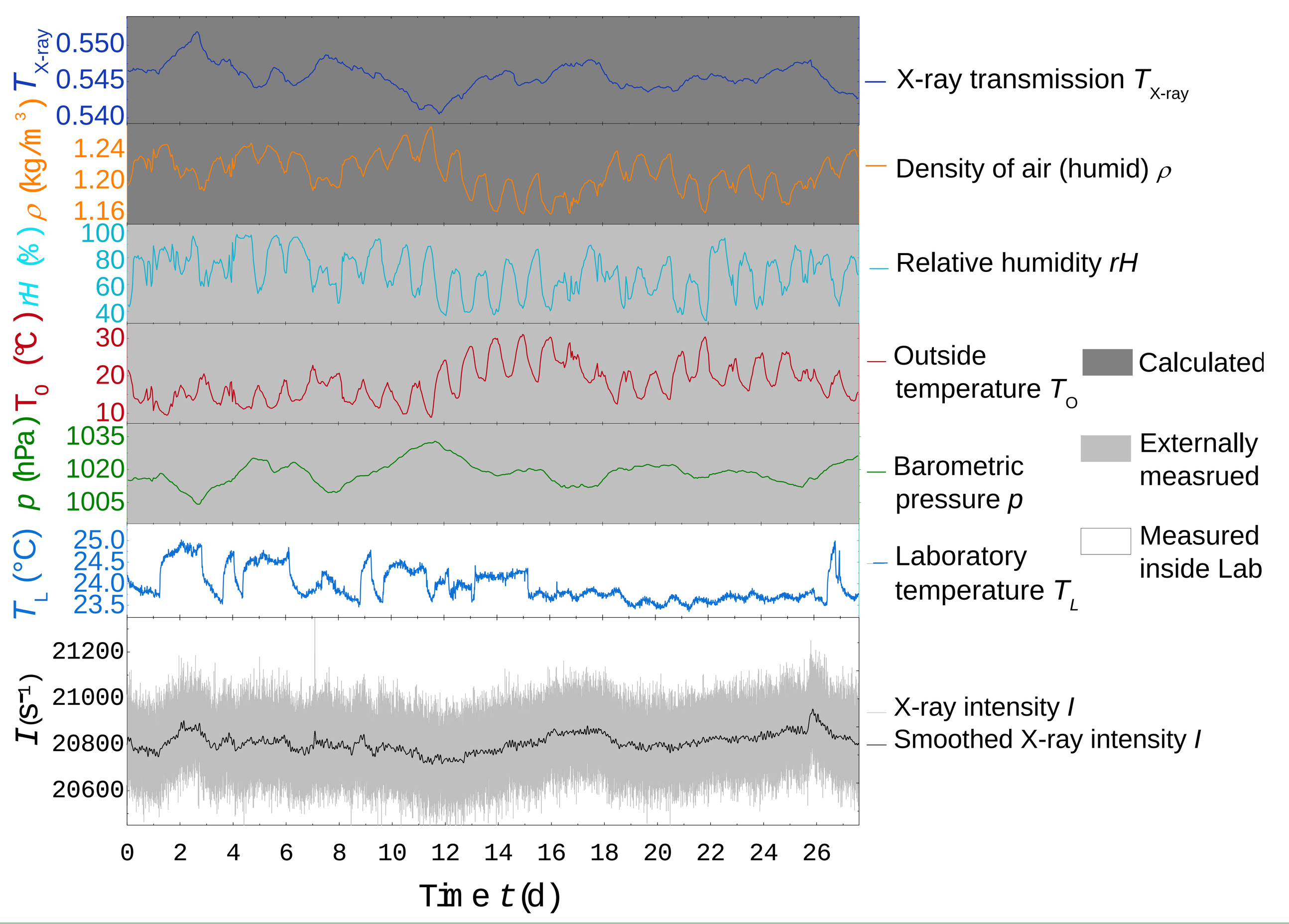
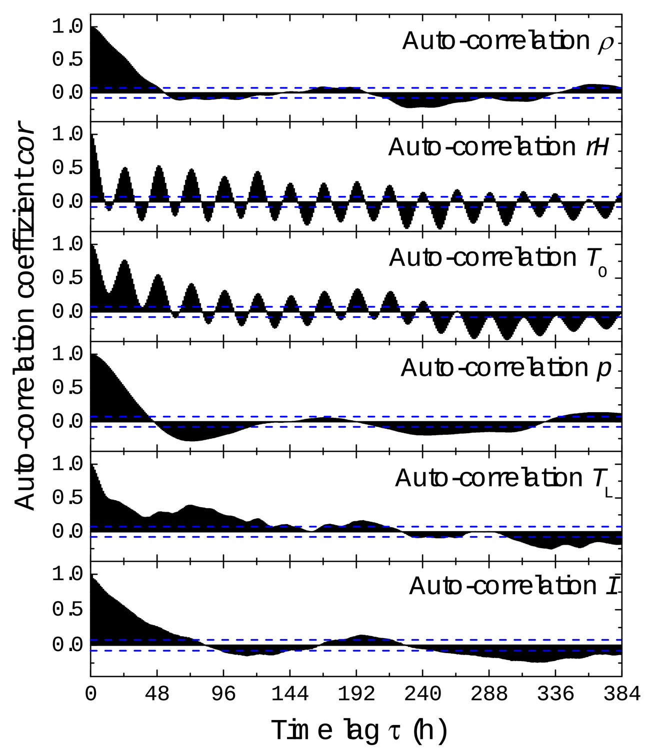
MOTIVATION

Essential for high precision X-ray diffraction or spectrometry analysis is a constant primary beam intensity. Besides electronically induced variations on X-ray primary intensity (X-ray generator electrical stability of 0.1 to 0.005 % [1]), ambient conditions are particularly important. This is barometric pressure, humidity and temperature, as well as cooling water, that affect the primary beam intensity. Changes of these parameters can influence the air density and thus the transmission of X-rays. This is important for long-term analysis, e.g. *in situ* investigation or high-precision structure data determination.

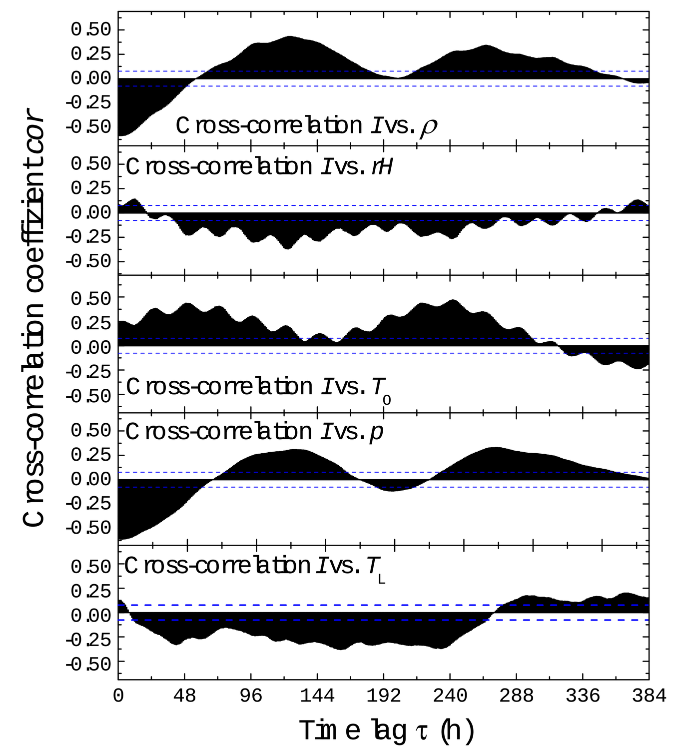
EXPERIMENTAL DATA



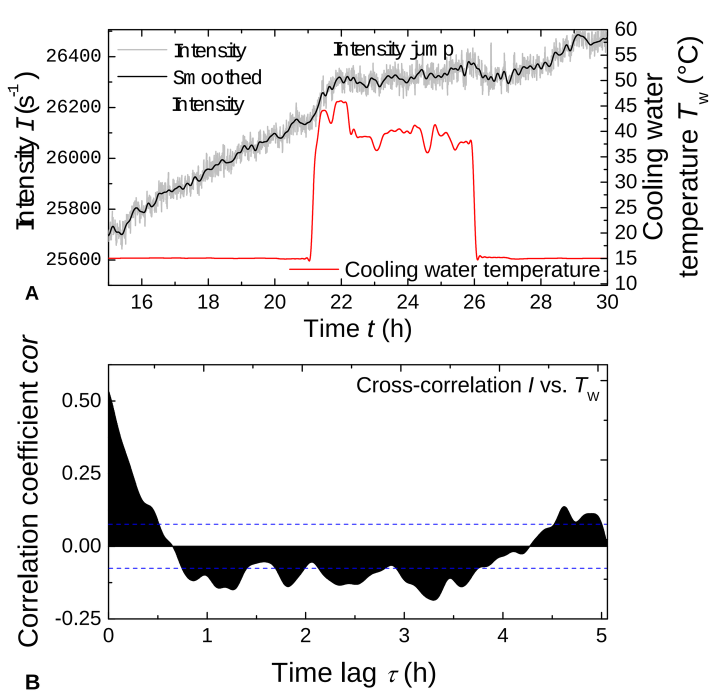
STATISTICAL ANALYSIS



The correlation of rH and T_O reflects day and night cycles. p shows no time-dependency. T_L has small day and night fluctuations, whereas a small time-dependency in the fine-structure of ρ and I is recognizable.



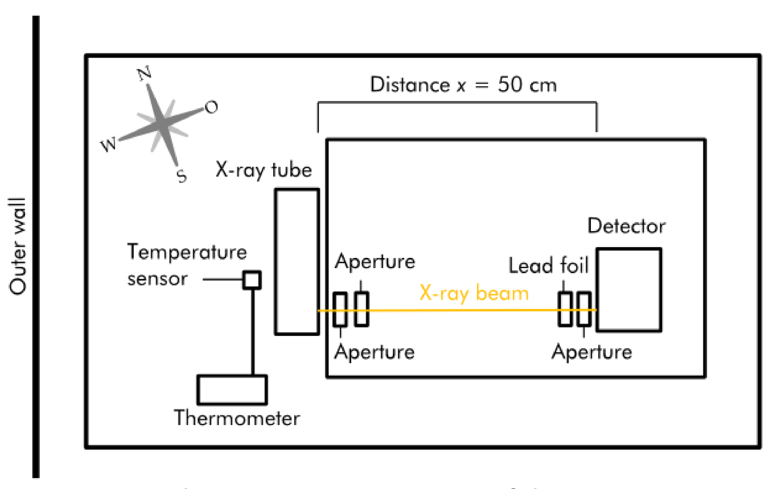
The highest correlation with cor = -0.61 at I vs. p and with cor = -0.58 at I vs. ρ. Correlations of I vs. rH and I vs. T_L are significant, but small. Day and night cycles reflected in I vs. rH and I vs. T_O. The ambient conditions influence I nearly instantaneous.



Self-induced cooling water temperature fluctuations: **A** Experimental data, $\Delta T_W = \pm 21.4$ K induce $\Delta I = \pm 0.5$ %. **B** Correlation analysis, T_W influenced the intensity instantaneously with cor = 0.53.

- Correlations were calculated with the statistical program R [4]
- Auto-Correlation shows the time-dependent correlation of particular parameters
- Cross-correlation shows the correlation between the parameters
- Analysis of Variance (ANOVA) used to indicate influence of daily and weekly cycles
  → changes of week and weekend have significant but small influence: during week I decreases, at weekend I increases
  → 12 hour periodic cycles influenced the intensity, but less than weekly cycles

EXPERIMENTAL DETAILS

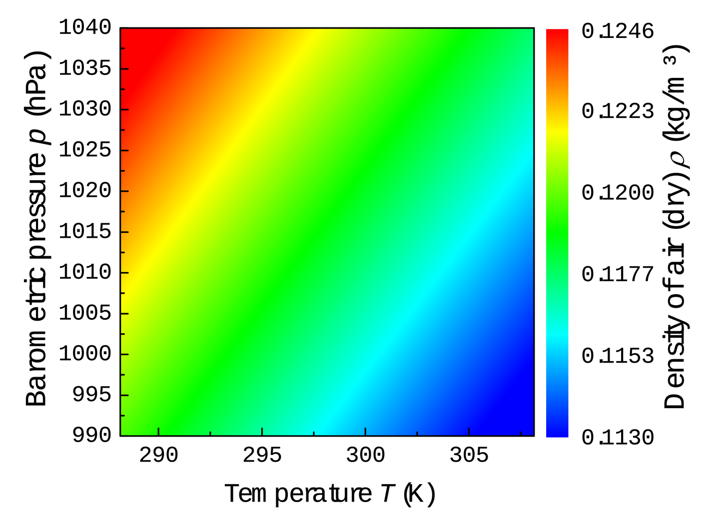


Schematic representation of the set-up.

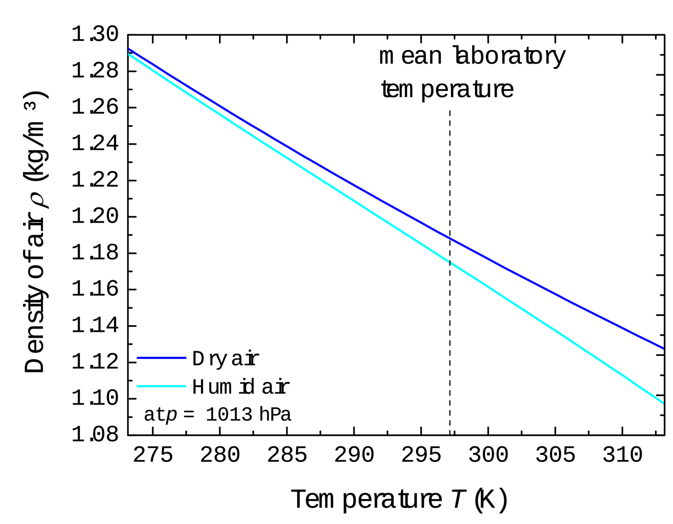
- X-ray source: water cooled sealed tube (Cu-Kα-radiation)
- Detector: scintillation counter
- Air conditioning supplied by a central facility
- Monitoring time: 28 days (664 hours)
- External data from weather station

THEORETICAL CONSIDERATIONS

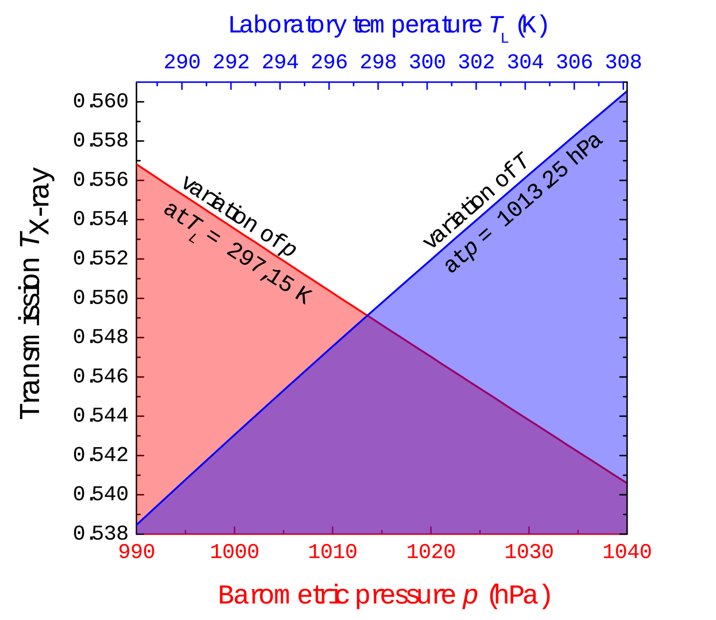
- Density of dry air calculated with ideal gas equation $\rho = \frac{p}{R_L \cdot T}$
- Humid air, the equation must be extended with the water content $\rho = \frac{p}{\left(1+0.608 \cdot 0.622 \cdot \frac{rH \cdot e}{p}\right) R_L \cdot T}$ [2]
- Transmission can be calculated by $T_{X\text{-ray}} = \frac{I}{I_0} = e^{-\mu(E)_{air} \cdot x}$
- Attenuation coeffcient $\mu(E)_{air}$ is expressed by $\mu(E)_{air} = \rho \cdot \sum_{i=1}^{N} g_i \cdot \mu_{m,i}(E)$ [1]



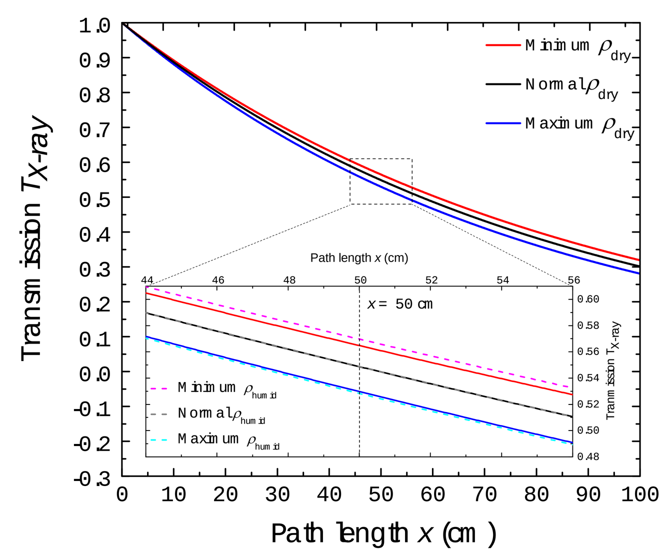
Influence of T and p on density of air. For low (high) temperature and high (low) pressure the density is maximal (minimal).



Influence of T on ρ at constant p. The lower ρ_{humid} corresponds to the light water molecules, whose amount is temperature-dependent [4].



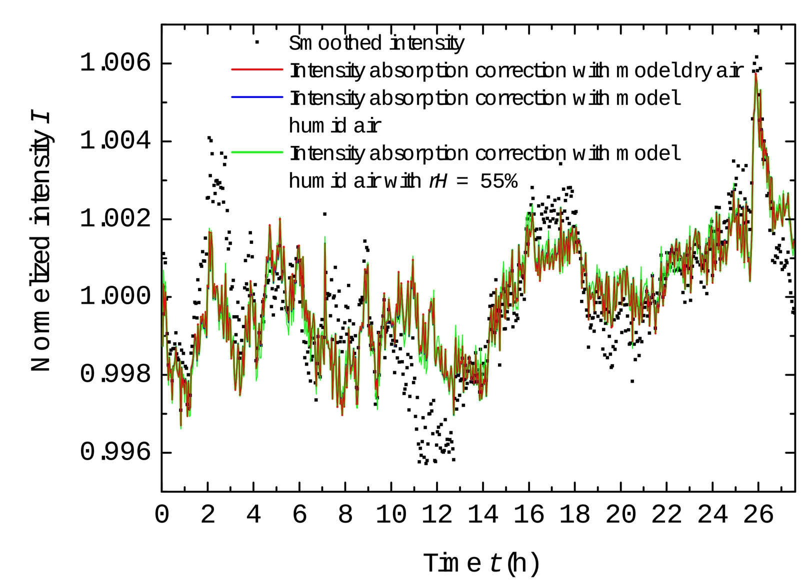
X-ray transmission in dependence of T and p for Cu-Kα. Changes of Δp = 50 hPa and ΔT = 20 K cause $\Delta T_{X\text{-ray}}$ changes of up to 1.6 % and 2.2 %.



Influence of ρ variations on $T_{X\text{-ray}}$ for Cu-Kα. At x = 50 cm, distance of usual experimental conditions, $\Delta T_{X\text{-ray}}$ are up to 3.5 % for ρ_{dry} and 4.1 % for ρ_{humid}.

CONCLUSION

- $\Delta I/I$ = 1.153 % induced by $\Delta T_{X\text{-ray}}$ = 1.137 % due to $\Delta\rho/\rho$ = 3.7 %
- Main impact on intensity variations has p
- Air conditioning system damped influence of T_L and rH, small correlations
- Fluctuations of T_W are small
- Weekly and 12 hour periodic cycles have small but significant influence on I
- Recording of ambient conditions can be used for time-dependent absorption correction of measured I, reduce variation due to 25 %



Time-dependent absorption correction of the measured I with the fit model $I_0(t) = \frac{I(t)}{A \cdot e^{-B \cdot \mu(E) \cdot x}}$. The parameters *A* and *B* have been introduced to allow the model to be flexible with respect to non-measured time-dependent effects. This correction reduced the intensity variations significantly, whereby the three models show similar results.

[1] Prince, E., et al., "International Tables for Crystallography" International Union of Crystallography, Volume C (2006).
[2] Etling, D., "Theoretische Meteorologie: Eine Einführung", Springer, Edition 3 (2008).
[3] Malberg, H., "Meteorologie und Klimatologie: Eine Einführung ; mit 56 Tabellen", Springer, Edition 5 (2007).
[4] R Core Team, "R: A Language and Environment for Statistical Computing" R Foundation for Statistical Computing.

T. Weigel, "Influence of environmental parameters variations on X-ray beam intensities - A time-dependent absorption correction" submitted to *Journal of Applied Crystallography* (2015)
TU Bergakademie Freiberg, "Vorrichtung und Verfahren für eine Absorptionskorrektur von Röntgenintensitäten", Deutsche Patentanmeldung: DE102015008975.7, Anmeldetag: 06.07.2015

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This work has been performed within the joint research project CryPhysConcept: Mit Kristallphysik zum Zukunftskonzept elektrochemischer Energiespeicher (03EK3029A) which is financially supported by the German Federal Ministry of Education and Research (BMBF).
We gratefully acknowledge Saxray GmbH.

Gefördert vom Bundesministerium für Bildung und Forschung
Europäische Union
Europa fördert Sachsen. ESF Europäischer Sozialfonds
Europäische Union
Europa fördert Sachsen. EFRE Europäischer Fonds für regionale Entwicklung