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# Experiment Report Form

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## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

| | | |
|---|---|---|
| ESRF | **Experiment title:**<br>Determination of cooling rate of pyroxenes from martian meteorites | **Experiment number**:<br>HS 2878 |
| **Beamline**:<br>ID13 | **Date of experiment**:<br>from: 29.08.2005 to:03.09.2005 | **Date of report**:<br>28.02.2006 |
| **Shifts:**<br>12 | **Local contact(s)**: M. Burghammer | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

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## Report:

**Introduction:** Clinopyroxenes from the martian meteorites NWA856 and SAU130 were studied by means of single crystal diffractometry with synchrotron radiation at ID13/ESRF. A structure refinement was performed and the cation distribution determined. In pyroxenes, the Mg and Fe2+ cations fractionate between the non-equivalent M1 and M2 sites. The fractionation value $K_D$ $(Fe^{2+}_{M1}\,Mg_{M2})$ /$(Fe^{2+}_{M2}\,Mg_{M1})$ is sensitive to temperature and thus serves as a recorder for the cooling rate of the rock. From structure refinements the $Mg/Fe^{++}$ site distribution, and Me-O bond distances, and, in turn, $K_D$ values were determined [1]. Closure temperatures were calculated to be $T_c$ = 520°C and 700°C for NWA856 and SAU130 [2], respectively, which are based on calibrations of intracrystalline geothermometry of meteoritic and terrestrial pigeonite samples known from literature[5]. Under certain assumptions these values of $T_c$ allow conclusions of the cooling rate of the host rock.

**Sample material:**

Crystals with edge lengths of <1µm - to 10µm were examined and only smallest crystals exhibit reflection profiles suitable for data collection and, most importantly, extraction of integrated intensity. Due to the very similar lattice constants of the intergrown pigeonite and augite lamellae an overlap of reflections from both phases occurs and the separation of reflections belonging to the two phases at low 2Θ angles proved to be impossible, especially if the mosaicity of the crystals is high. Pyroxene samples from martian meteorites have previously been considered as a hybrid between single crystals and textured powder in literature [2]. The crystal size seems to be the most important factor for determining the crystal quality. In practice, powdered sample material is placed on a kapton foil, which was scanned for good quality single grains. Data was collected at wavelengths between 0.73Å and 0.98Å and varying sample-CCD distances.

**Structure refinements**:

All our samples contain a main pigeonite phase and a minor part of augite phase as concluded from the x-ray patterns. Due to the very similar lattice constants of the intergrown pigeonite and augite lamellae, an overlap of reflections from both phases occurs and the separation of reflections belonging to the two phases at low 2Θ angles proved to be not completely possible, especially if the mosaicity of the crystals is high, i.e. higher than several degrees. Therefore, three reflections overlapping the strong augite reflections at low 2θ were excluded from the refinement (110, 020, 002, which have low influence on the $K_D$ value). For the reflections at higher angles, a large contribution of augite to the integrated intensities could be precluded, due to the integration method of profile fitting. Integral intensities were derived from data processing with the XDS program package [3]. The refinement was carried out only for the major pigeonite phase in the low-clino space group $P2_1/c$ using the Jana2000 software [4].

**Results: Closure temperature of Mg-Fe order-disordering in pigeonite bearing rocks**

Heating or even kinetic experiments on martian NWA pigeonites are not yet available. Therefore we used the geothermometric data published by Pasqual et al.[5] which have determined equilibrium temperatures of $Mg/Fe^{++}$ exchange of ureilite PCA82506 and (terrestrial) pigeonites BTS308. These samples contained up to 10% Wo, an amount which is comparable to that of the martian pigeonites. The geothermometric equations used by the authors are based on the Mueller rate equation which can be fitted by an Arrhenius equation. If we assume that these geothermometric equations are applicable to the NWA and SaU martian samples, we may get at least estimates of the closure temperatures of the exchange reactions in pigeonite of martian rocks. Table 1 includes values of calculated temperatures $T_c$ values using the constants given by Pasqual et al. [5]

| | SAU 130 | NWA 856 | | | | |
|---|---|---|---|---|---|---|
| | K310 | G1 | G2 | NWA722 | NWA470 | NWA337a |
| Lattice parameters | 9.650<br>8.884<br>5.204<br>108.475 | 9.732<br>8.964<br>5.255<br>108.6 | 9.747<br>8.989<br>5.263<br>108.636 | 9.693<br>8.928<br>5.221<br>108.726 | 9.685<br>8.909<br>5.218<br>108.656 | 9.722<br>8.960<br>5.231<br>108.428 |
| Cation Distribution | 0.12(4) | 0.049(5) | 0.05(1) | 0.044(6) | 0.048(8) | 0.05(2) |
| Tc | 700°C | From weighted average 0.049: 520°C | | | | |

Table 1.

**Crystallochemical (geometrical) parameters of pigeonite structure**

The topology of pyroxene structures can be described by alternating tetrahedral and octahedral layers parallel to the (100) plane. The octahedral layer contains the M1 and M2 sites. The $P2_1/c$ pigeonite space group is typical for low Ca pyroxenes and contains symmetrically distinct chains in adjacent tetrahedral layers. The more extended chain is denoted as A, with smaller tetrahedra. The O3 anions are bridging atoms, shared by neighbouring tetrahedra. The M2 is also coordinated by six anions, albeit in a very distorted octahedron. The mean M2-O radius increases with the radius of the cation – this distance is therefore an indicator of the cation distribution [6].

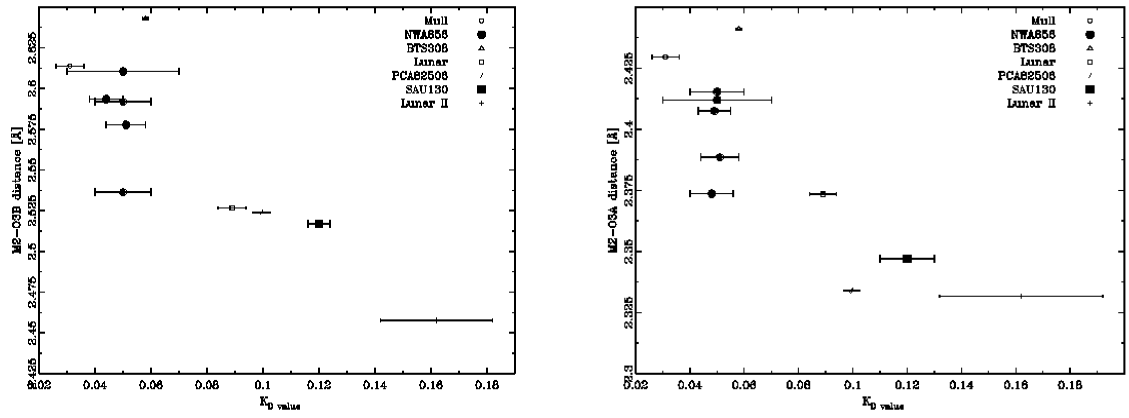


**Fig**. 1 Variation of M2-O3A (left) and M2-O3B (right) distance with cation distribution coefficient .Full symbols, NWA856 and SAU130

The structural data of the pyroxenes from martian meteorites SAU130 and NWA 856 are well within the trend of an almost linear dependency of Kd vs. M2-O3 distance.

## References

[1] E. Weidner, F. Frey, B. Pedersen and M. Burghammer, to be submitted
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[3] Kabsch, W. (1996), *J. Appl. Cryst.*, 26, 795
[4] Petricek, V. et al., (2000), *Inst. of Phys*.
[5] Pasqual, D. et al., (2000) *Am. Min.*, 85, 953
[6] Tribaudino, M. et al., (2003), *Min. Petr.*, 77, 161