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Operating instruction

XR 4.0 X-ray KBr-Crystal XR 4.0 X-ray LiF-Crystal XR 4.0 X-ray NaCl-Crystals



Fig. 1: 09056-01 KBr-Crystal, 09056-05 LiF-Crystal, 09058-02 Universal Crystal holder, 09058-01 NaCl-Crystals

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- **1 SAEFTY PRECAUTIONS**



- Optically polished windows from alkalihalide monocrystals (LiF, KBr, KCl, NaCl, CsJ, e.t.c.) are very sensitive and are to be protected against humidity, because they are soluble in water (but not hygroscopic). Use a desiccator (e.g. 34126-00) for storage.
- They shall be unpacked only by trained personnel familiar with crystals. Containers shall not be unpacked before windows have reached at least room temperature.

2 CALIBRATION

The crystals are used together with the goniometer 09057-10 and the XR 4.0 expert unit 09057-99.

In seldom cases, crystals for analysis may have a deviation of some 1/10° from the crystallographic main axis, so that the characteristic X-ray lines are not to be found at the expected Bragg angles. This concerns particularly the NaCI-crystals with the orientations 110 and 111. This fault can be corrected either manually or by means of the autocalibration function:

2.1 Automatic calibration

The anode material of the X-ray tube is automatically identified. The crystal must be manually set under "Menu", "Goniometer", "Parameter" (see section 5.5). For calibration, select "Menu", "Goniometer", "Autocalibration". The device now determines the optimal positions of the crystal and the goniometer to each other and then the positions of the peaks. The display shows the corresponding calibration curves. The newly configurated zero position of the goniometer system is saved even after switch-off of the X-ray unit.



2.2 Manual calibration

In seldom cases the automatic calibration does not lead to the desired results. In these cases, use the manual calibration to adjust the position of crystal and detector.

The crystal for analysis must be manually brought to the theoretical Bragg angle ϑ (counter tube correspondingly to 2ϑ). Now search for the intensity maximum of the line by iterative turning of the crystal and counter tube by a few $\pm 1/10^{\circ}$ around this angular position. Following this and in coupled mode, bring the crystal and counter tube to the particular zero position corrected by the error value and then confirm with "Menu", "Goniometer" and "Set to zero". Example:

If the position of detector and crystal to each other is not optimal then, after the optimization process described above, in coupled mode, one of the two is brought to the zero position. The other will now not be at zero but be somewhat over or under it. Now select (in coupled mode!) "Menu", "Goniometer" and "Set to zero". The display now shows 0.0 for both values. When the intensity maximum of a characteristic X-ray line is only above/below the theoretical Bragg angle by $\Delta \vartheta = \pm 0.X^{\circ}$, then the previous zero position is simply corrected by $\pm 0.X^{\circ}$ and then calibrated with "Set to zero" as described above. The new configuration of the zero position of the goniometer system is saved even after switch-off of the X-ray unit.

