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XR 4.0 X-ray Goniometer

09057-10

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Fig: 09057-10 XR 4.0 X-ray Goniometer

Operating instructions

CE The unit complies with the corresponding EC guidelines.

CONTENTS

1 SAFETY PRECAUTIONS

- Carefully and completely read these operating instructions before operating the instrument. This is necessary for avoidance of damage as well as for user-safety.
- Only use the instrument for the purpose for which it was designed.
- Only use the instrument in dry rooms in which there is no risk of explosion.
- Take care that no liquid penetrates in through the housing openings.
- Only use the experimental set-up for the use it is intended for.
- Do not open up the instrument.

2 PURPOSE AND CHARACTERISTICS

The goniometer block is equipped with two stepping motors which operate independently of each other. They enable the sample holder (crystal or Compton scattering body) or the counter tube holder (Geiger-Mueller counter tube) to be turned either separately or coupled in an angle ratio of 2:1. The detector holder with slit diaphragm carrier for absorption foil mounting can be moved by sliding it to change the angular resolution. The goniometer is self-calibrating.

Plug & measure:

- Automatic identification of the goniometer
- With a light barrier system which limits the permitted swivelling range for the protection of detectors. Intuitive handling via operation directly via the device or via a PC

3 FUNCTIONAL AND OPERATING ELEMENTS

To be able to fully utilize the complete counter tube holder swivelling range of from -10° to +170°, we recommend the use of the Geiger-Mueller counter tube type B (article no. 09005-00). The counter tube is to be fitted in the slidable counter tube holder (1) to the stop and fixed in position with a milled screw (2).
The milled screw (*3*) of the holder serves to fix it on its guide rods. The counter tube holder is equipped with a diaphragm carrier (4) which is to be slid into its guide-way to the stop. Absorption foils (article no. 09056-02) can also be fitted in the slit diaphragm.
Sliding the counter tube holder on its guide rods (5) allows the angular resolution to be varied when required. The maximum angular resolution is reached when the counter tube holder is at the right-hand stop.
The scale (6) allows direct reading of the angle of the detector. The plug connections (7) at the centre of the goniometer axle are for the attachment of a universal crystal holder (09058-02) or a pre-mounted crystal (09056-01 and 09056-05).
The complete goniometer block can also be slid in the horizontal direction and be fixed in position with a milled screw (8). When the goniometer block is at the far right, the maximum swivel range of the counter tube holder is 170°. When it is at the far left, only an approx. 102° swivel is possible.
According to the intermediate positioning of the goniometer block, the range of swivel is approx. 135°, approx. 120° and approx. 112°. These ranges are not exceeded even when larger outlet angles are inadvertently selected in automatic operation. This limitation of the swivel range is effected by a light barrier system inside the goniometer block. It prevents the counter tube from striking against the inside wall of the housing.

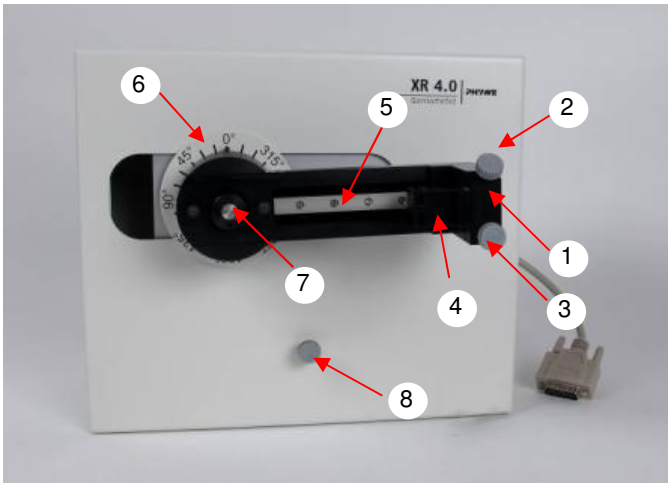

Fig. 2: Front view of the goniometer

4 NOTES ON OPERATION

The goniometer fulfils the technical requirements that are summarised in the current directives of the European Community.
The goniometer must be used under the supervision of an expert and in the electromagnetically controlled environment at research, teaching, and training facilities (schools, universities, institutes, and laboratories). This means that, in such an environment, radio transmission devices, e.g. mobile phones, should not be used in the direct vicinity of the unit. The connected cables must not be longer than 2 m.
Electrostatic charges or similar electromagnetic phenomena (HF, bursts, indirect lightning discharge, etc.) may affect the unit so that it will not work within the specified data range.
The following measures reduce or eliminate potential interferences: avoid carpets; provide equipotential bonding; perform the experiments on a conductive, earthed surface, use shields and shielded cables. Do not use radiofrequency transmitters (radio sets, mobile phones) in the direct vicinity of the unit.
After a total exit, perform a restart by briefly interrupting the power supply.

Fig. 3: Activation of the Goniometer-Mode

CAUTION:

The set-up is not to be moved when the goniometer is in the X-ray device. When transport is to be made, the goniometer must be taken out and separately transported.
The goniometer can only be operated when the sliding door is locked!

5 HANDLING

This section describes how to put goniometer 09057-10 into use and provides an overview of how to handle it. Please read this section attentively to avoid failure or improper operation.

The goniometer can only be operated when the sliding door is locked!

5.1 Installation of the goniometer

The goniometer can be connected anytime to the X-ray unit – even, when the XR 4.0 is switched on.

Two magnetic strips on the back of the goniometer enable it to be fixed in position in the X-ray expert unit (09057-99) far back in the back left corner.

Connection between the goniometer and the X-ray unit is made by plugging the permanently fixed data cable in the socket in the X-ray unit which is labelled “Goniometer”.

5.2 Activation of the goniometer mode

Select the menu by means of the button below the “Menu” display indicator (see Fig. 3). Use the arrow keys to select “Goniometer” and confirm it with “enter”, select “Mode” in the goniometer menu and again confirm with “enter”. In the Mode section select “on” and confirm with “enter”.

5.3 Manual operation

Turning only the sample holder or only the counter tube holder:
When the goniometer mode has been activated (see section 5.1), the display shows the actual counter tube and goniometer angle.
Press the “enter” key to select whether the crystal or the detector is to be moved, or if they are to be simultaneously

Fig. 4: top: coupled mode, left: rotate detector, right: rotate crystal.

moved in coupled mode.
In accordance with the selection, either one of the symbols is brightly highlighted or both of them are simultaneously (Fig. 4).

5.4 Goniometer menu overview

Menu prompt	Function
Parameter	Change parameters for an experiment → see section 5.5
Mode	Activate/switch off the goniometer mode
Set to zero	Define the actual positions of crystal and detector as being zero (“tare”) → see section 5.7.2
Reset to defaults	Again define the actual positions of crystal and detector based on the factory setting
Autocalibration	→ See section 5.7.1

5.5 Automatic operation

Select the menu by means of the button below the “Menu” display indicator (see Fig. 3). Use the arrow keys to select “Goniometer” and confirm it with “enter”. Select “parameters” in the goniometer menu and confirm with “enter”. To change parameters (Fig. 5), press the button under the “Modify” display indicator and use the “enter” key to select individual parameters. Use the arrow keys alongside the display on the right to change values. You can run through the parameters again by pressing the “redo“ button. Confirm your changes with “enter” and confirm the dialog which follows with “on”. Select “start” with the button below the “Menu” display indicator to start the experiment. You can stop the experiment by pressing the “halt” button below the display indicator. Confirmation of this with “esc” causes the experiment to be completely aborted.

Fig. 5: Parameter menu for the goniometer

Parameter:

- Scan-Type setting of measurement mode: Selection between three options:


Coupled mode


Firm detector, crystal is moved


Firm crystal, detector is moved:

- Further parameters


Crystal angle Start/Stop


Detector angle Start (is automatically set to 2 x crystal starting angle but can be changed)

In addition: Step width and integration time.

5.6 Operation with software assistance

For operation of the goniometer with software assistance, refer to the corresponding help function in the software. The goniometer can only be connected to a computer when it is connected to an XR 4.0 X-ray expert unit 09057-99.

5.7 Readjustment of the goniometer unit

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 fault can be corrected either manually or by means of the autocalibration function:

5.7.1. 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 go-

niometer 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.

5.7.2. Manual calibration
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 ±1/10° 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 $\Delta\vartheta = \pm 0.X°$, then the previous zero position is simply corrected by ±0.X° 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.

6 TECHNICAL SPECIFICATIONS

- Angle step width: 0,1°...10°
- Speed: 0.5...100 s/Step
- Swivelling range, sample: 0...360°
- Swivelling range, detector: -10°...+170°
- Dimensions (cm): 35 x 30 x 20
- Weight (kg): 5

7 PARTS SUPPLIED

- Complete with slit diaphragm for GM counter tube
- Operating instructions

8 ACCESSORIES

09056-01 XR 4.0 X-ray Potassium Bromide Crystal, mounted
09056-05 XR 4.0 X-ray Lithium fluoride crystal, mounted
09058-02 XR 4.0 X-ray Universal crystal holder for x-ray-unit
09058-04 XR 4.0 X-ray Compton attachment
09058-09 XR 4.0 X-ray holder for powder probes
09058-30 XR 4.0 X-ray energy detector (XRED)

9 NOTES ON THE GUARANTEE

We guarantee the instrument supplied by us for a period of 24 months within the EU, or for 12 months outside of the EU. Excepted from the guarantee are damages that result from disregarding the Operating Instructions, from improper handling of the instrument or from natural wear.
The manufacturer can only be held responsible for the function and technical safety characteristics of the instrument, when maintenance, repairs and alterations to the instrument are only carried out by the manufacturer or by personnel who have been explicitly authorized by him to do so.

10 WASTE DISPOSAL

The packaging consists predominately of environmentally compatible materials that can be passed on for disposal by the local recycling service.

Should you no longer require this product, do not dispose of it with the household refuse.

Please return it to the address below for proper waste disposal.

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APPENDIX

Symbols and safety designations

	CAUTION, general danger spot