

A DEVICE FOR THE TRANSFER OF AN OPTICALLY ORIENTED MINERAL FRAGMENT TO A SINGLE-CRYSTAL X-RAY GONIOMETER HEAD¹

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ABSTRACT

A device, inexpensive to make and easy to use, has been designed to facilitate the transfer of an optically oriented crystal fragment for single crystal X-ray study. Except for isometric and triclinic crystals, a unique crystallographic axis is always associated with a principal optical vibration direction. Such a direction is first determined with the aid of a spindle stage, and the crystal is then transferred using the device to keep the unique axis either parallel or normal to the axis of rotation of a goniometer head. After transfer most crystals require an adjustment of less than three degrees of arc of the goniometer head to perfect the alignment.

INTRODUCTION

Mineral fragments separated from hard rocks often lack crystal faces or cleavages, making their orientation difficult and time consuming. Hence there seems to be a general lack of application of single-crystal X-ray methods in the study of rock-forming crystal fragments.

Hartshorne and Stuart (1960) described a method of transferring a crystal from a goniometer head to a spindle needle tip for optical study. A fairly elaborate piece of mounting apparatus with mounted eye piece is needed. The transfer also involves heating of dental wax with a micro flame. Since the needle is not preset, there is no sure way of correlating the optical orientation with that of the X-ray data if the crystal does not have any crystal faces. The procedure is also the reverse of that which we require.

The optical analyzer described by Donnay and Donnay (1957) and the stage goniometer and cell described by Jones (1960) were designed for checking the optical orientation of a crystal after it has been mounted on the goniometer head. Since the orientation of the crystal is limited by the movement of the goniometer head, these devices are useful provided only the desired symmetry directions are accessible. Using the above methods, it is extremely tedious if not impossible to correlate the optical orientation of a crystal fragment without crystal faces as reference directions with crystallography.

The primary objective of the procedure we describe here, involving the use of the transfer device, is to select suitable mineral *fragments* on the basis of their optical properties and take advantage of the known optical

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orientation to mount the crystal fragment for single crystal X-ray study. A basic objective is to correlate the optical orientation with crystallography so that in the transfer, the known optical orientation of crystal fragment is undisturbed. We do not propose to start with a crystal on the X-ray goniometer head to transfer it in order to get supplementary optical data, although there is no problem in reversing our procedure.

The instrument described here is made of materials which are inexpensive and readily available. The construction is simple. The required skill involved in the transfer procedure is no more than that required for the normal mounting of a crystal for precession work.

Briefly, a crystal is attached to the tip of a spindle with a water-soluble cement and oriented optically by spindle stage techniques (Wilcox, 1959). The spindle is then transferred to its groove in the device and rotated to the previously determined setting of the spindle arm. A silica glass fiber mounted in a brass nib for use with a goniometer head is rotated about the center of the transfer device to a position corresponding to the setting of the polarizing microscope stage. The spindle-mounted crystal and the end of the glass fiber are positioned so that they almost touch at the center of the device. The crystal is then joined to the glass fiber with a cement which is insoluble in water. When this cement has set, a drop of water is applied to dissolve the glue holding the crystal on the spindle. The brass pin with the oriented crystal attached to the end of the glass fiber may then be transferred to the goniometer head for X-ray studies.

Using this transfer device with tetragonal, trigonal, hexagonal, orthorhombic or monoclinic crystals it is a simple matter to select a crystal axis from the optical data and mount the crystal for X-ray study within a few degrees of the chosen crystallographic orientation. We have been using the transfer device with great success in our laboratory for the past year and a half in the study of fragments of shocked quartz, olivine and other minerals which exhibit no regular faces and can not be oriented with the usual two-circle goniometer. This technique should also be valuable with crystals which have been ground to spheres for purposes of absorption correction.

DESCRIPTION OF APPARATUS

The crystal transfer device is shown in Figures 1, 2 and 3. Referring to Figures 1 and 2c the circular baseplate A is 13.6 centimeters ($5\frac{3}{8}$ inches) in diameter and is cut from a sheet of 0.64 cm ($\frac{1}{4}$ in) Lucite or Plexiglas. Annular channels B, C and D, cut concentrically about the center of A, have outside diameters of 3.18 cm ($1\frac{1}{4}$ in), 2.46 cm ($\frac{3}{4}$ in) and 1.75 cm ($\frac{7}{16}$ in) respectively. Channels B and D lie 0.16 cm ($\frac{1}{16}$ in) below the top surface of A, and Channel C is 0.16 cm ($\frac{1}{16}$ in) deeper still. The center platform E has a diameter of 0.95 cm ($\frac{3}{8}$ in) and its surface is 0.08 cm ($\frac{1}{32}$ in) above that of Channel D. F is a groove drilled along

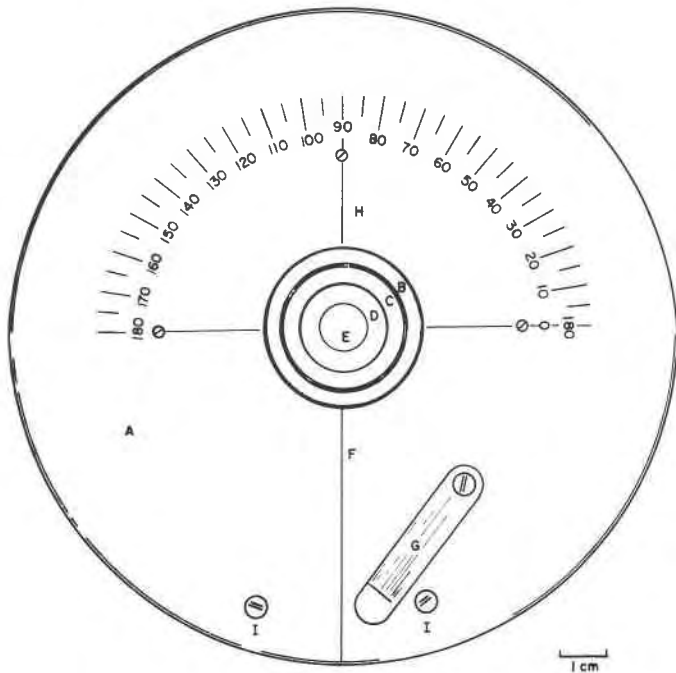


FIG. 1. Sketch of baseplate of transfer device, top view.

the radius of the baseplate to accommodate a 0.061 cm (24 mil) piano wire spindle. G is a clamp made of spring steel. It is attached to the baseplate by means of a screw, and swings over the spindle to hold it in place during the transfer operation. Three screws are used to attach the transparent plastic protractor H to the bottom of A and are countersunk so that they are flush with the bottom surface. The protractor is accurately aligned with its center under the center of the baseplate and its baseline perpendicular to the spindle groove. The slotted-head screws I are located equidistant from the spindle groove to accommodate the spindle stage protractor (Wilcox, 1959).

Figure 3a shows the device assembled for use, and Figure 3b is a view of the components disassembled. The holder for the brass nib glass fiber assembly (shown at the bottom of Figures 2a and 2b and at the right of Figure 3b) is a plastic ring made to fit into and rotate freely in Channel C. It has an outside diameter of 2.4 cm ($\frac{15}{8}$ in) and an inside diameter of 1.8 cm ($\frac{9}{5}$ in). For two-thirds of its circumference the ring is 0.24 cm ($\frac{3}{50}$ in) high, and for the other third it is 0.64 cm ($\frac{1}{4}$ in) high. At the midpoint of the higher segment a 0.32 cm ($\frac{1}{8}$ in) hole is drilled through the side of the ring to hold the brass nib. Attached to the top of this segment and centered over the hole is a pointer for use in determining settings on the large protractor scale.

The nib is made from 0.32 cm ($\frac{1}{8}$ in) brass rod and is 0.95 cm ($\frac{3}{8}$ in) in length. To hold the glass fiber a hole 0.035 cm (.014 in) in diameter is drilled along the axis of the cylinder for half its length. At the end of the nib near this hole there is cut a flat face 0.08 cm ($\frac{1}{32}$ in) in from the edge and 0.08 cm ($\frac{1}{32}$ in) along the length of the nib. This flat surface allows the nib to rest on the center platform E during the transfer operation and thus pre-

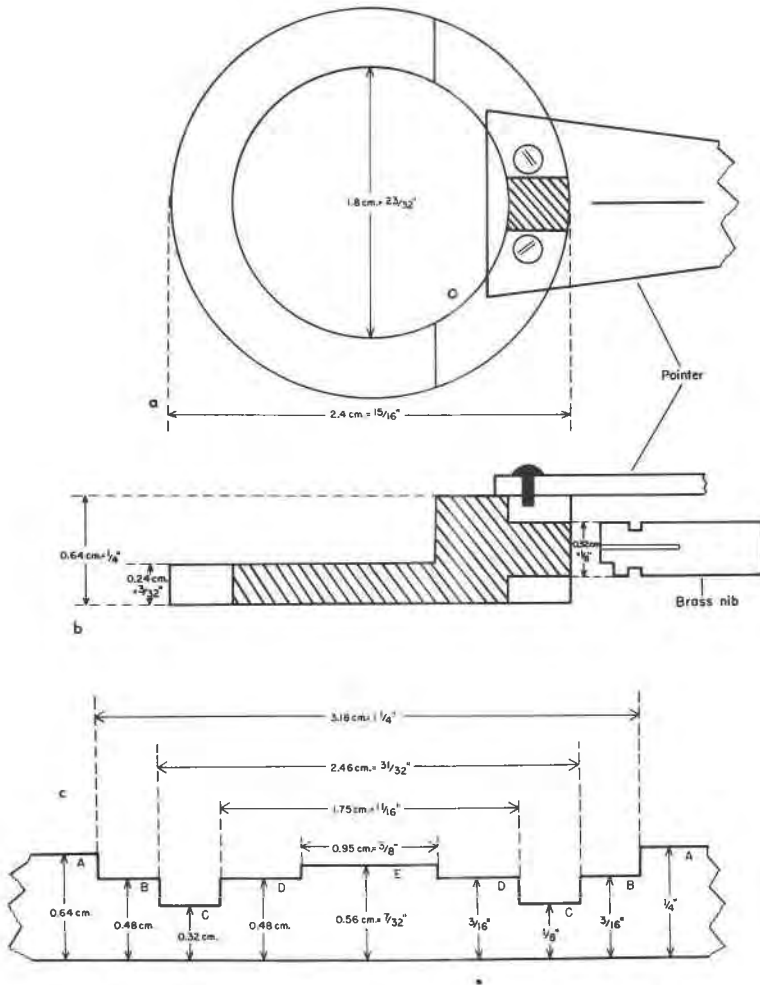
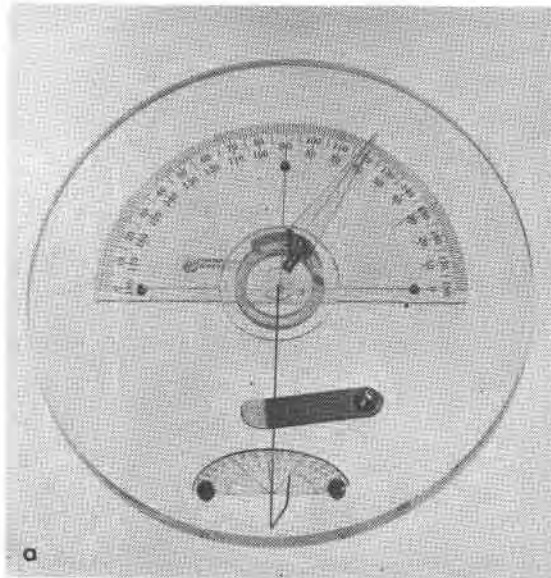


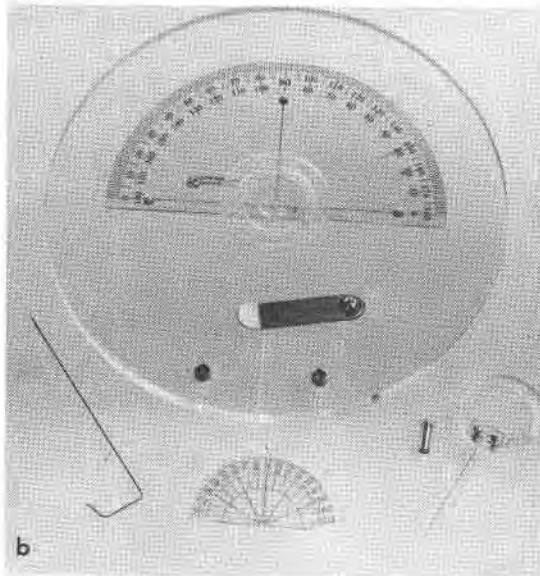
FIG. 2. Expanded view of components of transfer device, showing important dimensions (top to bottom):

- Holder for brass nib, top view.
- Holder and brass nib, side view.
- Center area of baseplate, side view.

vents any twisting of the nib while the cement joining the crystal and the glass fiber is setting. The selected crystallographic axis lies in a plane parallel to the flat face of the brass nib, thus providing a convenient reference direction after the crystal has been transferred to the goniometer head. For ease of gripping with tweezers a band 0.04 cm ($1/64 \text{ in}$) deep and 0.08 cm ($3/32 \text{ in}$) wide is machined into the nib slightly beyond the flat surface.



(a)



(b)

FIG. 3a. Photograph of transfer device with crystal on spindle ready for transfer.
 FIG. 3b. Component parts of the transfer device (left to right): spindle, baseplate,
 spindle stage protractor, brass nib with glass fiber, holder for brass nib.

TRANSFER PROCEDURE

Any single grain selected for study is first fastened to the tip of a spindle with a mixture of water-soluble carpenter's glue and molasses and mounted on the spindle stage for orientation of the crystal by the conoscopic method (Wilcox, 1959). The crystal is rotated about the spindle axis and the axis of the microscope stage until the desired principal vibration direction lies horizontal and NS. The spindle position and microscope stage setting are recorded.

A glass fiber is mounted in the brass nib so as to extend about 0.32 cm ($\frac{1}{8}$ in) beyond the end of the brass piece (in our laboratory beeswax is usually used to hold the fiber in place in the hole in the nib). The nib is then placed in the holder of the transfer device and the assembly inserted into Channel C (Figures 1 and 2) with the flat surface of the nib resting on the center platform E. The holder is rotated until its pointer indicates a reading on the protractor H corresponding to the setting of the microscope stage.

Next the spindle with crystal attached is removed from the spindle stage and the immersion oil rinsed from the surface of the crystal with acetone. The spindle is clamped in Groove F of the transfer device so that the crystal lies approximately over the center of platform E. The spindle stage protractor is placed in the slots of screws I and the spindle rotated to duplicate its position when it was on the microscope.

Finally the position of the brass nib is adjusted until the end of the glass fiber almost touches the crystal. A drop of colorless nail polish diluted with acetone is applied, using a dropper formed from a drawn capillary tube, to join the crystal to the glass fiber. After the nail polish has set for about 10 minutes, the carpenter's glue is dissolved with water, and the brass nib with its oriented crystal is ready for mounting on a goniometer head for X-ray study. (Care must be taken when applying the drop of nail polish that none runs over onto the glue joint, as this would prevent the water from dissolving the carpenter's glue once the nail polish has set.)

SPECIFIC EXAMPLES

Uniaxial Crystals (Tetragonal, Trigonal and Hexagonal). Since in uniaxial crystals the optic axis coincides with the c crystallographic axis it is a simple matter to orient such crystals with the optic axis horizontal and NS. The crystal fragment is transferred and mounted with the c -axis parallel to the length of the glass fiber.

Many shocked and unshocked quartz fragments were mounted during the past year for X-ray precession studies with the c -axis as the dial axis. A few setting photographs at ten-degree increments around the dial are usually sufficient to locate an a^* axis and therefore proceed to obtain a pattern for the $h_0 \cdot l$ or $ok \cdot l$ net.

Biaxial Crystals.

1. Orthorhombic. The orthorhombic case is again quite straightforward since the orthogonal crystallographic axes coincide with the principal vibration directions α , β and γ . From our experience in mounting olivine grains for study with the precession camera we have found the following procedure to work very well: the spindle stage and microscope stage settings are determined for α , β and γ and plotted on a stereogram. The crystal fragments is then transferred with any one of the three axes parallel to the length of the glass fiber, *i.e.* to serve as the dial axis. The dial setting for the second axis can then readily be determined with the aid of the stereographic projection and the flat surface of the brass nib as a reference direction. With this method, frequently two setting photographs are sufficient to align the desired net. In less than an hour, one can be ready to obtain a precession pattern for orthorhombic crystals.

2. Monoclinic. In monoclinic crystals one of the principal vibration directions always coincides with the b -axis, the unique crystallographic axis. Using the rock-forming clinopyroxenes as an example, either α or β coincides with b (for certain monoclinic amphiboles such as riebeckite, γ coincides with b). As a first step then, the crystal fragment can be mounted with say, β as the dial axis. If β does not turn out to be the unique crystallographic axis then α can be oriented horizontal on the spindle stage, transferred with the device and mounted as the dial axis. Once the unique axis has been located, for precession studies, setting pictures are then taken at 10° increments until the $a^* b^*$ or $b^* c^*$ net is located and properly oriented. As a rule, if the crystal exhibits any cleavages or crystal faces, this additional information plotted together with the optical triangle on a stereogram should of course be utilized to shorten the search around the dial axis for a^* or c^* .

3. Triclinic and isometric. Since in triclinic crystals none of the three principal vibration directions need necessarily coincide with a crystallographic axis, each triclinic case must be treated individually. Additional information such as cleavages must be available in order for this transfer technique to be useful for the triclinic system. For example in the triclinic plagioclase the c -axis always lies in the (010) cleavage plane and the cleavage trace (001) can also be observed. In this instance we plot two cleavage traces instead of the optical triangle for search of the c -axis. This method of approach is by no means always successful because of possible absence or misidentification of the cleavage traces on the fragment. If a triclinic crystal has no cleavage or parting at all, the transfer device will not be useful.

In isometric minerals the isotropic character is evident optically, so that orientation of such specimens must again depend on information from crystal faces or cleavages.

If a new transparent mineral is being investigated, the above procedures should be helpful since optical orientation is the best means of selecting the proper crystallographic reciprocal net for single crystal study. If a crystal is known, these techniques can be used expeditiously to locate a particular reflection or set of reflections which are of special interest, for example those that are indicators of composition or structural deformation.

For all crystal fragments which we have mounted with due care using the transfer device the orientation of the crystal has been good to within 2° or 3° of arc. The time saved with the aid of this method should make it possible and desirable for more investigators to employ single-crystal

techniques in addition to or in lieu of powder diffraction analyses in the study of rock-forming minerals of complex composition.

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