# MINIMIZING THE EFFECTS OF PREFERRED ORIENTATION IN X-RAY POWDER DIFFRACTION PATTERNS*

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ABSTRACT

A number of techniques to reduce preferred orientation in samples for *x*-ray powder diffraction patterns were studied. Minerals were mounted in ethyl cellulose spindles and spheres, on greased Lindemann glass rods, and in nylon tubes. Gross differences in intensities of diffraction lines are demonstrated on films showing random and preferred orientation of fibrous pectolite and platy paragonite mica. The sphere-mount method was shown to be the most successful and convenient method for achieving random orientation and thus for obtaining more nearly correct intensities of diffraction lines.

## INTRODUCTION

It has long been known that fine-grained aggregates of fibrous and platy minerals when dispersed in liquids and dried or when subjected to mild pressures tend to form oriented aggregates. Rod-shaped particles tend to align their long axes parallel and platy minerals tend to lie on their platy surfaces and form matted layers of overlapping flakes.

Several fibrous and platy minerals were studied by various methods in an attempt to reduce preferred orientation in *x*-ray powder diffraction mounts. Among these, fibrous pectolite and platy paragonite mica were chosen for further tests because optical examination showed that they retain their fibrous and platy character even when ground extremely fine. The platy character of several micas was found to diminish with decreasing particle size because the thickness of the plates increased relative to the dimensions across the flakes. Hence the effects of preferred orientation in the powder diffraction patterns of paragonite are not as great as in those of pectolite.

## PREPARATION OF SPHERE MOUNTS

The production of spheres in which preferred orientation in powder patterns of fibrous or platy minerals can be reduced to a minimum is an outgrowth of a method of preparing spindle mounts that was developed in the *x*-ray laboratory of the U. S. Geological Survey by several technicians over a period of about 12 years.

In this method, a small quantity of the mineral is finely powdered with a glass rod in a depression of a standard spot plate. A 10 per cent solution of granular, low-viscosity ethyl cellulose dissolved in toluene is used as a binder. A little xylene may be added when the amount of ma-

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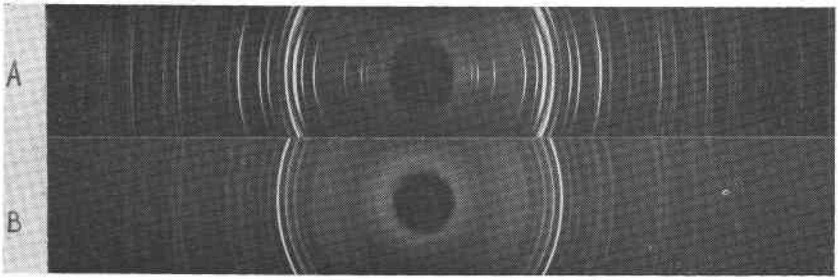


FIG. 1. Pectolite, Bergen Hill, New Jersey, USNM. 82452. A. Ethyl cellulose spindle 0.15 mm. in diameter showing preferred orientation, B. Ethyl cellulose sphere 0.29 mm. in diameter showing minimized preferred orientation.

terial is extremely small, in order to avoid too rapid drying. A very small amount of the binder is taken up on the end of a sewing needle and is thoroughly mixed with the powder until the mixture is no longer sticky, and a plastic, workable ball is obtained. This ball is quickly placed between a plate glass surface and a microscope slide and is rolled between the two pieces of glass in a circular motion, with the microscope slide resting on the edge of the glass plate as a pivot line. Whereas spindle type mounts conventionally used in powder diffraction cameras can easily be rolled to an optimum diameter of 0.10 mm. to 0.15 mm. because the excess material can squeeze out in the direction of the spindle length, the ultimate diameter of a spherical mount is determined by the initial amount of material used. It was found that the smallest spheres that could conveniently be made by rolling were 0.25 mm. to 0.30 mm. in diameter.

A sphere produced by this rolling technique is cemented to the end of an ethyl cellulose binder spindle simply by dipping the end of the spindle in toluene and touching it while still wet to the sphere. The spindle is then mounted in the camera in standard fashion, care being taken to project only the sphere into the collimating tube field.

## OTHER METHODS INVESTIGATED

Three other sample-mounting techniques investigated to minimize preferred orientation were as follows:

1) A short cylindrical section of the spindle used in preparing the pattern shown in Figure 1 A was cemented to the end of a binder spindle with its cylindrical axis normal to this supporting spindle. The mount was then placed in the camera in the same fashion as a sphere mount. The resulting diffraction pattern indicated that preferred orientation had been appreciably decreased, but not so strikingly as with sphere mounts.

TABLE 1. *X*-RAY DIFFRACTION DATA FOR PECTOLITE

| d(Å, meas.) | A | B | d(Å, meas.) | A | B |
|---|---|---|---|---|---|
| | Figure 1 A film 6509 | Figure 1 B film 6994 | | Figure 1 A film 6509 | Figure 1 B film 6994 |
| | I | I | | I | I |
| 7.83 | 7 | 5 | 1.661 | 7 | 4 |
| 7.03 | 7 | 5 | 1.603 | 3 | 4 |
| 5.50 | 7 | 5 | 1.570 | 6 | 2 |
| 4.98 | 3 | 1 | 1.554 | 5 | 5 |
| 4.55 | 1 | 1 | 1.521 | 2 | 2 |
| 4.00 | 2 | 2 | 1.490 | 8 | 5 |
| 3.90 | 8 | 6 | 1.475 | 5 | 5B |
| 3.77 | 2 | 2 | 1.465 | 2 | |
| 3.52 | 8 | 5 | 1.431 | 4 | 2 |
| 3.43 | 1 | 4 | 1.393 | 6 | 2 |
| 3.33 | 9 | 6B | 1.377 | 6 | 4 |
| 3.28 | 9 | | 1.356 | 6 | 3 |
| 3.16 | 4 | 5 | 1.329 | 1 | 1 |
| 3.10 | 10 | 8 | 1.312 | 1 | 1 |
| 2.921 | 6 | 10 | 1.299 | 6 | 2 |
| 2.739 | 8 | 6 | 1.285 | 5 | 1 |
| 2.637 | 1 | — | 1.265 | 2 | 1 |
| 2.600 | 8 | 6 | 1.245 | 3 | 2B |
| 2.495 | 3 | — | 1.220 | 2 | 1B |
| 2.430 | 3 | 5 | 1.180 | 3 | — |
| 2.368 | 4 | — | 1.169 | 5B | 4 |
| 2.338 | 6 | 5 | 1.149 | 2 | 2 |
| 2.298 | 9 | 6 | 1.136 | 2 | 4 |
| 2.270 | 2 | — | 1.114 | 2 | 1 |
| 2.227 | 3 | 2 | 1.105 | 5 | 5 |
| 2.191 | 6 | 6B | 1.093 | 5 | 2 |
| 2.166 | 2 | | 1.084 | 2 | 1 |
| 2.090 | 5 | 2 | 1.065 | 2B | 1B |
| 2.053 | 2 | 3B | 1.052 | 2B | — |
| 2.002 | 4 | | 1.043 | 3 | 2 |
| 1.945 | 5 | 2 | 1.032 | 1 | 1 |
| 1.926 | 5 | 4 | 0.999 | 4 | 2 |
| 1.877 | 2 | 4 | | | |
| 1.831 | 6 | 4 | | | |
| 1.781 | 3 | 3 | | | |
| 1.752 | 7 | 6 | | | |
| 1.716 | 5 | 6 | | | |
| 1.675 | 6 | 2 | | | |

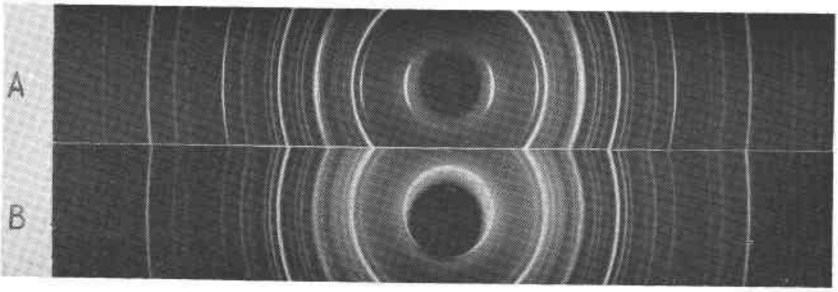


FIG. 2. Paragonite mica, Monte Campione, Switzerland, USNM. R4414, W. T. Schaller No. 19, D-1432-Pa 1. A. Ethyl cellulose spindle 0.16 mm. in diameter showing preferred orientation. B. Ethyl cellulose sphere 0.30 mm. in diameter showing minimized preferred orientation.

2) Lindemann glass rods 0.12 mm. in diameter coated with "celloseal" grease were dipped into the powdered mineral and mounted like a spindle in the camera. The resulting diffraction patterns of these also indicated that preferred orientation had been appreciably decreased. This method is probably not so advantageous as other methods not only because of the additional materials required for the sample mount but also because it is difficult to coat the greased rod uniformly with the powdered mineral and prevent broad bands contributed by the glass from showing up in the diffraction pattern.

3) A nylon tube with an inner diameter of 0.33 mm. was loosely filled with the mineral and mounted like a spindle in the camera. In the resulting pattern many strong broad bands contributed by the nylon almost completely obliterated the diffraction lines of the mineral, so that the method was not practical. A repetition of this test with a slightly larger nylon tube also gave unsatisfactory results. Nylon tubes with smaller diameters were not available.

## X-RAY DATA

In Figure 1 A, the diffraction pattern of pectolite made with a spindle, preferred orientation is best shown by unequal intensities of the diffraction lines along their arcs. In Figure 1 B, the diffraction pattern of a portion of the same mixture used in the spindle of Figure 1 A but rolled into a sphere, preferred orientation has been appreciably reduced, with a leveling off of the line intensities along their arcs as well as relative to each other. In Table 1, a comparison of the measured intensities of the two patterns of pectolite in Figure 1 shows the gross differences in intensities of identical diffraction lines. The line intensities shown by the sphere mount, Table 1 B, are the more nearly correct intensities for pectolite. Likewise, Figure 2 and Table 2 demonstrate preferred orientation in

TABLE 2. *X*-RAY DIFFRACTION DATA FOR PARAGONITE

| d(Å, meas.) | A<br>Figure 2 A<br>film 7026<br>I | B<br>Figure 2 B<br>film 7002<br>I | d(Å, meas.) | A<br>Figure 2 A<br>film 7026<br>I | B<br>Figure 2 B<br>film 7002<br>I |
|---|---|---|---|---|---|
| 9.7 | 9 | 8 | 1.834 | 3B | 3B |
| 4.96 | 2 | }6B | 1.774 | 2B | 2B |
| 4.85 | 7 | | 1.728 | 2B | 1B |
| 4.44 | 7 | 10 | 1.687 | 4B | 5B |
| 4.27 | 4 | 4 | 1.658 | 2 | 2 |
| 4.15 | 2 | 2 | 1.639 | 2 | 2 |
| 4.06 | 4 | 6 | 1.616 | 6B | 6B |
| 3.97 | 2 | — | 1.583 | 2B | 3B |
| 3.87 | 2 | — | 1.549 | 2B | 2B |
| 3.79 | 4 | 6 | 1.514 | 2B | 2B |
| 3.68 | 4 | 6 | 1.486 | 7B | 8B |
| 3.56 | 2 | — | 1.459 | 1 | — |
| 3.48 | 3 | 2 | 1.427 | 2 | 2 |
| 3.39 | 4 | 5 | 1.410 | 2 | 2 |
| 3.30 | 5 | 5 | 1.384 | 4 | 3 |
| 3.22 | 10 | 6 | 1.349 | 3B | 4B |
| 3.18 | 5 | 5 | 1.315 | 4B | 4B |
| 3.03 | 1 | — | 1.296 | 2 | — |
| 2.979 | 3 | — | 1.284 | 4 | 5 |
| 2.921 | 6 | 7 | 1.265 | 3B | 3B |
| 2.831 | 6 | 7 | 1.238 | 3B | 4B |
| 2.772 | 1 | — | 1.211 | 3 | 2 |
| 2.706 | 5 | 6 | 1.199 | 2 | 2 |
| 2.571 | 6 | 6 | 1.176 | 3B | 3B |
| 2.536 | 8 | 9 | 1.150 | 2 | 1 |
| 2.430 | 7 | 8 | 1.136 | 2 | 2 |
| 2.356 | 5B | 6B | 1.118 | 1B | 2B |
| 2.222 | 3 | 3 | 1.094 | 2B | 2B |
| 2.186 | 5 | 6B | 1.049 | 2B | 2B |
| 2.146 | 1B | 2B | 1.025 | 2B | 1B |
| 2.103 | 6B | 6B | 1.013 | 1B | 1B |
| 2.067 | 2 | 2 | 0.985 | 2B | 2B |
| 2.027 | 3 | 3 | 0.969 | 2B | 2B |
| 1.989 | 5 | 3 | 0.961 | 3B | 2B |
| 1.965 | 2 | — | 0.9022 | 3B | 2B |
| 1.933 | 8 | 6 | 0.8577 | 2B | 2B |
| 1.895 | 2 | 2 | | | |

paragonite, and Table 2 B shows the more nearly correct line intensities for paragonite.

Examination of Figure 1 shows that there is more broadening of lines in the sphere pattern than in the spindle pattern. Whether the greater diameter of the sphere is responsible for this broadening can not be established with certainty. Some of the other methods used to minimize preferred orientation also resulted in patterns in which there was broadening of lines with some loss of resolution. The best example of this was method (1) described under the heading "other methods investigated," where a very short section of the same spindle used to produce the pattern in Figure 1 A was mounted on a binder spindle. The resulting pattern showed more random orientation but also broadening of lines and poorer resolution. In view of the many factors involving both technique and instrumentation in the production of powder diffraction patterns, it is difficult to attribute broadening of lines and loss of resolution to any one factor.

In the patterns produced from sphere mounts it was noted that virtually all the lines except a few very weak ones were measurable both as to position and as to intensity in spite of broadening of lines and that these measurements agreed admirably with those made from patterns produced from spindle mounts although the spindle-mount patterns showed better resolution.

The diffraction patterns shown in Figures 1 and 2 were taken with North American Philips Debye-Scherrer powder cameras (114.59 mm. diameter) using the Straumanis and Wilson techniques with $CuK\alpha$ (Ni filter), $\lambda = 1.5418$ Å.

## Acknowledgments

The need for investigating new techniques for minimizing preferred orientation in powder diffraction films was pointed out by C. L. Christ, and an investigation of the problem was strongly urged by W. T. Schaller in connection with his studies of fibrous minerals. The minerals used in the tests described here are from Dr. Schaller's collection. Some of the grease-dipped rods were made by J. M. Axelrod in the early stages of the investigation.