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A new method of reducing preferred orientation in diffractometer samples

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Abstract

In this simple method as little as 20 mg powdered mineral is mixed with an equal volume of fumed silica (Cab-O-Sil) and one or two drops of vegetable or mineral oil onto a glass slide which is directly inserted into the diffractometer. A comparison of observed relative intensities with those calculated from the crystal structure shows the new method quite effective in reducing preferred orientation.

Introduction

Published methods of reducing preferred orientation in powdered samples for diffractometer study include those of Flörke and Saalfeld (1955), Niskanen (1964), Lerz and Kramer (1966), and Bloss *et al.* (1967). Of these, the simplest and perhaps the best known is that of Bloss *et al.*

The method introduced here, like that of Bloss *et al.,* requires no additional or modified equipment. Furthermore, this sample preparation procedure is even simpler and faster, and it requires only one-tenth or less of the amount of powdered specimen required in their method. The only additional materials necessary are a few drops of cooking oil such as corn oil and a few milligrams of fumed silica. The latter is an extremely fine, snow-white, fluffy powder, with thickening control properties. When fumed silica is thoroughly dispersed in most liquid systems, a network of silica chains will form, due to the tendency of the fumed silica aggregates to link together through hydrogen bonding. Fumed silica is produced under the trade name of "Cab-O-Sil" by the Cabot Corporation, Boston, Massachusetts, and it is available in small quantities from laboratory supply companies at about $3.00 per 100 g.

Swanson *et al.* (1955, p.2) suggested that silica gel, when added to a diffractometer sample as a dilutent, may help eliminate preferred orientation. However, fumed silica or Cab-O-Sil is superior because only as little as three percent by weight is added to the sample, whereas silica gel, lacking chain-forming tendencies, must be added in relatively much larger amounts, thereby causing interference with the specimen diffraction pattern.

The proposed new method

A small amount of the powdered mineral (with particle/crystallite sizes of <45 microns for best results) is mixed with an equal volume of fumed silica on a glass slide. In most cases, 50 to 60 mg of powdered mineral will produce the best patterns, but in some cases as little as 15 mg of sample will produce adequate results. Two to three drops of vegetable oil are then worked into the mixed powders, spreading the resulting viscous mixture on the slide surface. No drying is required before the slide is inserted into the XRD goniometer, and the specimen may be rotated at any speed in its own plane, since the grains–silica mixture is firmly held on the slide. Cover glass spacers may be used to compensate for the thickness of the layer of sample on the slide or on a standard sample holder. After the analysis, the sample may be filed for further reference as a glass slide mount.

The fumed silica alone gives a relatively high background (low-angle peaks) in the 15–25°2θ (Cu $K\alpha$) region, but the background is considerably suppressed by absorption when the mineral sample is mixed with the silica. In cases where it is desirable to further reduce the background in the 15–25° region, an additional 2–3 mg of powdered mineral should be sprinkled on the surface of the silica–mineral mount.

Comparison of results

Figure 1 gives diffractograms for muscovite from Middletown, Connecticut, and shows the effect of different methods of sample preparation. The weight shown for the Cab-O-Sil method (Fig. 1B, 70 mg, and Fig. 1C, 35 mg) represents the weight of the mineral powder (<45 microns) before and after mixing

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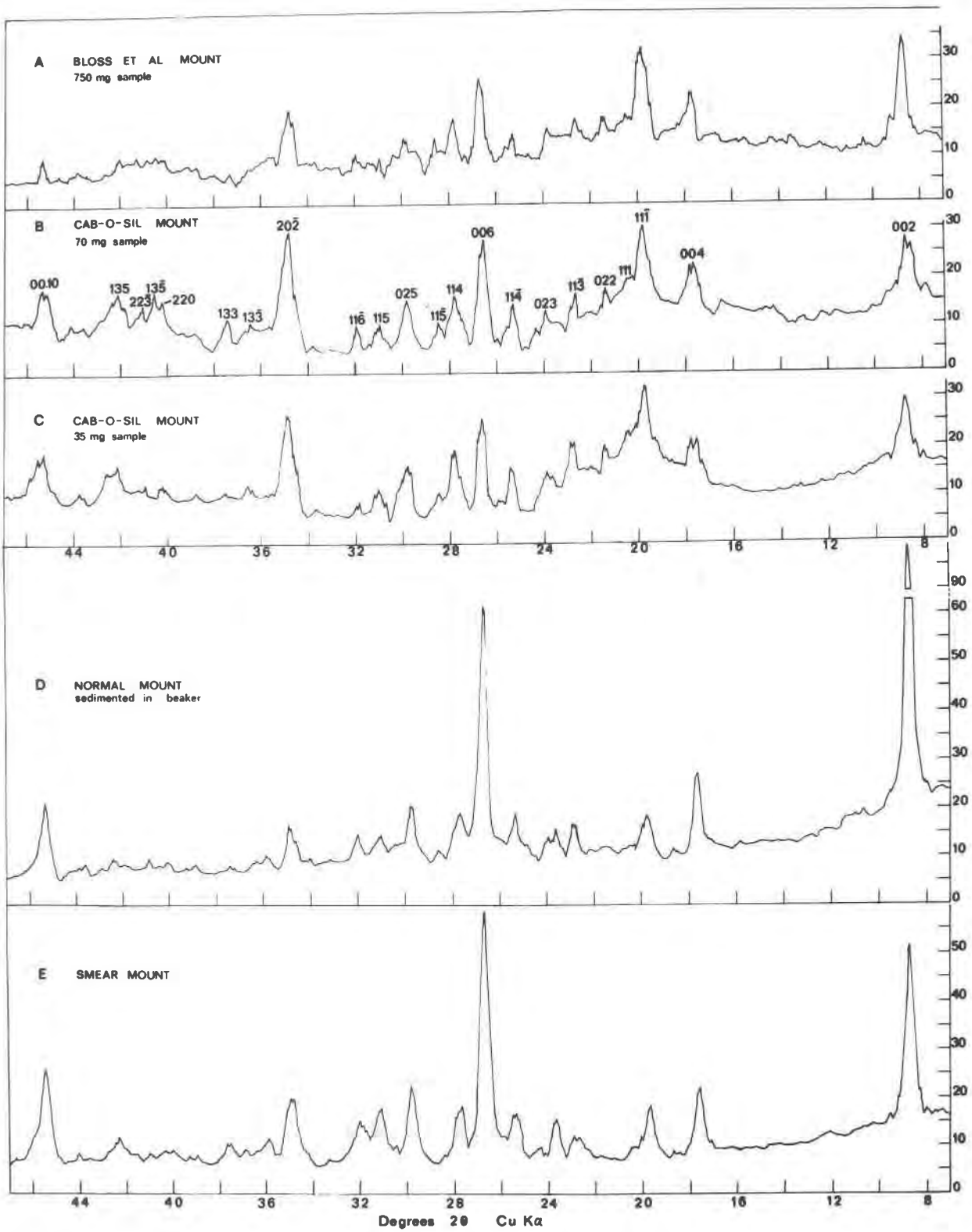

Fig. 1. X-ray diffractograms for muscovite from Middletown, Connecticut, illustrating the relationship between the degree of preferred orientation shown by a sample and the method of sample preparation used. All diffractograms represent identical settings on a G.E. XRD-5 unit.

Table 1. A comparison of observed relative intensities obtained with the Cab-O-Sil and the Bloss *et al.* (1967) methods with those calculated from the crystal structure

H K L	CALCULATED I(INT)*	OBSERVED Cab-O-Sil†	Bloss et al ††
002	97	112	162
004	27	66	92
11$\bar{1}$, 021	114	112	190
111	22	x	x
022	24	15	20
11$\bar{3}$	52	28	23
023	51	27	22
11$\bar{4}$	73	47	28
006, 024	142	142	142
114	74	71	62
11$\bar{5}$	6	22	x
025	79	62	38
11$\bar{5}$	54	29	22
11$\bar{6}$	44	18	x
20$\bar{2}$, 200, 13$\bar{1}$, 131, 116, 130	221	182	109
13$\bar{3}$, 202	65	45	x
133, 20$\bar{4}$	51	32	x
220, 22$\bar{1}$, 041	19	29	x
13$\bar{5}$, 221, 204, 042	31	38	x
22$\bar{3}$	10	x	x
135, 20$\bar{6}$, 043	77	70	x
00.10, 224, 13$\bar{7}$	62	60	22

*Data from Borg and Smith (1969, p. 477).
†Determined from pattern of Fig. 1B.
††Determined from pattern of Fig. 1A.
xPeak either absent or too weak to be accurately measured.

with fumed silica on the X-ray slide. The weight shown for the Bloss *et al.* method (Fig. 1A, 750 mg) represents the weight of the mineral powder (<45 microns) required in order to produce (according to the method's procedures) the X-ray slide used in the analysis. On the latter slide, the mineral grain-aggregates (115 mesh) weigh approximately 100 mg. For the normal mount (Fig. 1D) and the smear mount (Fig. 1E) 100 mg mineral powder (<45 microns) was used.

The Middletown muscovite was also used by Bloss *et al.*, who assessed the degree of preferred orientation by comparing the height of the 11$\bar{1}$ peak to that of the 002 and 006 basal peaks. On this basis, the Bloss *et al.* sample (Fig. 1A) and the two Cab-O-Sil samples (Fig. 1B and Fig. 1C) show approximately the same degree of preferred orientation since, in all cases, the top of the 111 peak falls above a reference line joining the tops of the 002 and 006 peaks. However, the two Cab-O-Sil samples (especially the 70 mg sample) produce far superior spectra in the region between 28° and 46°2θ, a region that may be critical for differentiating polytypes. As expected, the normal mount (Fig. 1D) and the smear mount (Fig. 1E) show the highest degree of preferred orientation.

The degree of preferred orientation was also assessed by comparing the observed relative intensities, obtained with the Cab-O-Sil and the Bloss *et al.* methods, with those calculated from the crystal structure (Table 1). The observed intensities were determined according to the method of Schultz (1964). The patterns were compared by setting the observed intensity of the 26.9° reflection (024, 006) equal to 142, which is the calculated value of the reflection obtained from Borg and Smith (1969). The data in Table 1 show that the basal reflections are somewhat enhanced relative to the nonbasal reflections in both sample preparation procedures, but that the Cab-O-Sil method is superior to that of Bloss *et al.* in reducing preferred orientation.

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