

be examined for offsets. Single crystals are characterized by the absence of offsets occurring as the isochromes pass across the isogyres at 45° off extinction. The postulation is made that some values of 2V for natural micas may also be erroneous.

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NOTE ON "BIOTITE MICA EFFECT IN X-RAY SPECTROGRAPHIC ANALYSIS OF PRESSED ROCK POWDERS" BY A. VOLBORTH

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In Professor Volborth's recent paper in this journal, preliminary chemical values for "Q. Monzonite 84" (Volborth, 1964, Table 3, col. 6) as determined by us, are quoted. These values were obtained using W-1 and G-1 only for calibration. Because samples of this rock (now termed Standard #5, Pomona College) have been widely distributed to spectrographers it is important that newer, more precise determinations based upon multiple standards be given to avoid misuse of the analytical values in x-ray calibration procedures. Furthermore, the comparison of these newer values with Volborth's values adds emphasis to his conclusions concerning the chemical biases introduced by mica in rocks when using unfused samples for analysis by x-ray spectrography.

Revised values by oxide for this standard are shown in Table 1 where they may be compared with those of Volborth (1964, Table 3, col. 7). With the direct determination of oxygen in silicates now possible (Baird

TABLE 1. OXIDE WEIGHT PERCENTAGES IN Q. MONZONITE 84  
(Standard #5, Pomona College)

Oxide <sup>1</sup>	Preferred values <sup>2</sup> Pomona College	Volborth <sup>2</sup> 1964
SiO <sub>2</sub>	68.2(1)	66.60
TiO <sub>2</sub>	0.50	0.55
Al <sub>2</sub> O <sub>3</sub>	15.46	15.37
Fe <sub>2</sub> O <sub>3</sub>	3.03	3.46
MgO	0.85	1.23
CaO	2.06	2.00
K <sub>2</sub> O	3.39	3.48
Na <sub>2</sub> O	4.84	4.13
MnO	Not determined	0.122
P <sub>2</sub> O <sub>5</sub>	0.14	Not determined
Total	98.4(8)	96.94

<sup>1</sup> Oxides calculated from elemental percentages determined by *x*-ray spectrography (see Table 2).

<sup>2</sup> Water-free basis.

and Welday, 1963; Volborth and Banta, 1963; Henke, 1964; Sharma and Clayton, 1964) a more meaningful statement of the chemical composition of this rock is in weight percentages of elements (Table 2). The availability of the new analytical tools of soft *x*-ray spectrography and fast neutron activation make Eskola's (1954) strong arguments for presentation of rock analyses in ionic percentages even more compelling.

The revised values shown for Na through Fe were obtained from a re-calibration using 12 wet-chemically analyzed rocks generously provided by A. Volborth (University of Nevada), and I. Adler (U. S. Geological Survey), plus W-1 and G-1. The importance of relating *x*-ray calibrations to several rocks, rather than assuming linear calibrations between two points (*e.g.*, W-1 and G-1) has been discussed by Welday *et al.* (1964, p. 902). In this re-calibration all specimens were prepared by the Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> fusion technique previously described (Welday *et al.*, 1964). Determination of oxygen was performed on fine-ground (8 min., Pica Mill) rock powders. Intensity ratios, standard/quartz, were used in linear regression with oxygen values for the standards calculated by difference. The intensity ratios, P.C. #5/quartz, from 5 replicate samples were used in the regression equation to predict weight per cent of oxygen. Details of instrumental conditions for oxygen analysis have been described by Henke (1964); for instrumental conditions of Na through Fe analyses see Baird *et al.* (1963).

The approximate modal composition of Pomona College Standard #5

TABLE 2. ELEMENTAL WEIGHT PERCENTAGES IN Q. MONZONITE 84  
(Standard #5, Pomona College)  
determined by *x*-ray spectrography

Element	Mean Weight Per Cent	Analytical Precision (standard deviation)
O	48.0(2)	0.4
Na	3.59	0.02
Mg	0.51	0.01
Al	8.18	0.04
Si	31.8(8)	0.18
P	0.06	0.005
K	2.81	0.03
Ca	1.47	0.01
Ti	0.30	0.003
Fe	2.12	0.02
Total	98.9(4)	0.44 <sup>1</sup>

<sup>1</sup> Square root of the sum of variances.

has been estimated from stained slabs cut parallel to three directions at right angles from a 1 cubic foot volume of rock. Over 7000 points were counted using  $\frac{1}{16}$  inch square-grid Zip-A-Tone overlays viewed under a low-powered binocular microscope. The variation within any one orientation was too great to permit the detection of any variation between orientations. The very coarse-grained character of the rock (phenocrysts of potassic feldspar up to 3 cm. and an IC index of about 25) precluded the use of thin sections for modal analysis; thus quantitative information on trace mineral constituents is not available. The major mineral percentages are oligoclase 44.9%, microcline-perthite 26.9%, quartz 24.4% and biotite 3.8%.

A comparison of the chemical analyses calculated as oxides in Table 1 for the fusion technique (column 2) and the ground rock powder technique (column 3) supports the predictions of Volborth on the effect of mica, and extends the detection of the effect to a rock containing as little as 3.8% biotite. Those constituents of biotite which are relatively more abundant compared to the whole rock (Fe, Mg and K) yield higher results, and those which are relatively less abundant (Al, Si) yield lower results in the ground rock analyses. The effect in absolute amount seems most pronounced in Si but the percentage difference in Mg and Fe is very high. The latter two constituents occur only in the mica, except for trace minerals. As stated by Volborth (1964, p. 636) the effect is due to an increase in the total relative surface of biotite mica (compared to the other minerals present) with grinding and is enhanced by pressing the

powder at 30,000 psi to form a coherent specimen for *x*-ray analysis.

As we have pointed out previously (Welday *et al.*, 1964, p. 902) adequate rock standards for *x*-ray analysis are rare and the best (W-1 and G-1) are now in very short supply. In an effort to cooperate with other laboratories, limited samples of Pomona College Standard #5 will be made available upon request to those seriously interested. Larger supplies of Pomona College Standard #4 are available. This rock, another biotite quartz monzonite with a chemical composition similar to #5, has been analyzed and calibrated by the methods used for standard #5.

We thank Professor Volborth for reading the manuscript of this note, but we take full responsibility for the chemical values reported and the conclusions reached. This work was supported by National Science Foundation grants GP-1336 and GE-1038.

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## DISCUSSION OF "EMPRESSITE AND STUETZITE REDEFINED"

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I would like to make three comments regarding the very interesting paper on empressite and stuetzite from Colorado by Dr. R. M. Honea (1964, p. 325-338). These comments refer to:

- 1) Apparently incorrect statements regarding the assemblages  $Ag_{5-x}Te_3$ -petzite, and  $Ag_{5-x}Te_3$ -petzite-hessite.

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