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ELECTRON PROBE ANALYSES OF COPPER IN MENEGHINITE

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Meneghinite was first described by Bechi (1852) from the Bottino Mine, near Tuscany, Italy. Based on chemical analyses and *x*-ray diffraction its space-group has been given as $D_{2h}^{16} = Pbnm$ with $a = 11.36$, $b = 24.04$, $c = 8.26$ Å, and the cell content, as $Pb_{26}Cu_2Sb_{14}S_{48}$ (Berry and Moddle, 1941). Two copper-free meneghinite formulas have been suggested, $Pb_4Sb_2S_7$ (von Rath, 1867) and $Pb_{13}Sb_7S_{23}$ (Palache *et al.*, 1938). Most analyses indicated an excess of Pb, and it seemed doubtful if the Cu (and much smaller amounts of Fe) really belonged in the structure. Ramdohr (1960) still quotes both possibilities.

In order to establish the distribution of Cu, two crystals of meneghinite from Bottino (specimen no. RH1407 in the Mineralogical Department of the Swedish Natural History Museum in Stockholm) were analyzed with an ARL electron microprobe *x*-ray analyzer. The quantitative results are shown in Table 1, and the distribution of Pb, Sb and Cu is demonstrated semi-quantitatively by beam scanning pictures, Fig. 1. The probe analysis agrees fairly well with the formula $Pb_{26}Cu_2Sb_{14}S_{48}$. The deviation as well as the low total, 95.6 per cent (no other elements were found in concentration above <0.1 per cent), is probably due to insufficiently known mass absorption and average atomic no. corrections (Ziebold and Ogilvie, 1963). The standards used were pure Sb-metal, galena (PbS), and chemically analysed bornite (Cu_5FeS_4).

It is immediately clear from Fig. 1 that although Pb, Sb and Cu are homogeneously distributed in the meneghinite, this mineral contains minute exsolved particles of a heavier mineral free from Cu and Sb. Probe analyses show this mineral to be galena (PbS). It is obvious that

TABLE 1. CHEMICAL COMPOSITION OF MENEGHINITE. WEIGHT %

	Theoretical ($Pb_{26}Cu_2Sb_{14}S_{48}$)	Probe Analysis[1] (Bottino Specimen)
Pb	61.5	58.7
S	17.6	16.7
Sb	19.5	19.0
Cu	1.45	1.15
Fe	0	0.04
Total	100.00%	95.6%
Pb/S	3.50	3.52
Pb/Sb	3.15	3.09

[1] Instrument: Applied Research Laboratories EMX (Univ. Calif. San Diego). X-ray intensity ratios were corrected for detector deadtime, background, self absorption. Fluorescence corrections were considered not to be necessary. Atomic number corrections were eliminated by the use of standards of similar chemical nature and average atomic number.

these micron-size particles could not easily be separated out, and an attempted chemical analysis would show an excess of Pb over Sb or S. Since Cu is evenly distributed in the meneghinite and since probe analysis does not show excess Pb, it may be concluded that the copper in Bottino meneghinite belongs to the structure.

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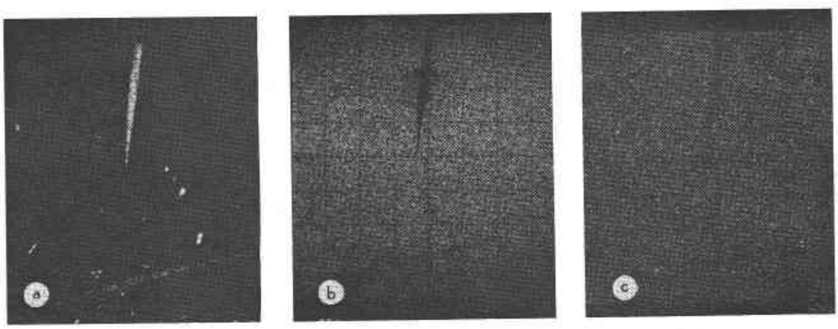

FIG. 1. Electron beam scanning pictures of 50×50 microns area on polished meneghinite from Bottino. a. Back scattered electron picture showing galena (PbS) inclusions white because this mineral has higher average atomic number than the meneghenite. b. Distribution of antimony; note the deficiency of this element in the galena inclusions. c. Distribution of copper; homogeneously in the meneghinite, lacking in the galena.

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SYNTHESIS OF THORIANITE CRYSTALS FROM BISMUTH OXIDE-LEAD FLUORIDE MELTS

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Oxides that can be readily grown as single crystals, and into which rare-earth ions can be incorporated, are of research interest and may be of value for certain electronic applications. Uranium and the lanthanides substitute for thorium in natural occurring thorianite (Palache *et al.*, 1944). The growth of thorianite by long continued fusion of ThO_2 in Borax has been previously described (Mellor, 1960). However, the synthesis of thorianite by this method results in either minute crystals or trellis-like aggregates of crystals. The purpose of this report is to describe a method for the growth of large thorianite crystals.

It has been found that ThO_2 crystals can be readily synthesized from the PbO-PbF_2, Bi_2O_3-PbF_2, and PbF_2 flux systems. The melts from which the largest ThO_2 crystals were grown contained 7 mole per cent ThO_2, 15 mole per cent Bi_2O_3, and 78 mole per cent PbF_2. Melts containing 10 mole per cent ThO_2 and 90 per cent PpF_2 also produced large crystals. The materials employed were 99.99 per cent pure ThO_2, reagent grade Bi_2O_3, and a purified grade of PbF_2. The powders were mechanically mixed (generally in 100 gram lots) and fused in tightly covered 50 ml platinum crucibles.

The crucibles were placed in two Super Kanthal heated horizontal muffle furnaces. The melts were held at 1250° C. for 4.5 to 8 hr and cooled