X-ray diffraction analysis of the products of the high-pressure runs revealed, in addition to the two oxides, various hydroxides and oxyhydroxides. The water for the formation of these compounds comes from the decomposition at elevated temperature of the pyrophyllite gasket material. We have observed both of the previously reported hydroxides and the previously reported oxyhydroxide SmOOH. In addition, we have found a new phase which we call β-SmOOH.

Weight loss versus temperature curves from preparations showing only the X-ray diffraction lines of the new phase were obtained. These are shown in Figure 7. In each case the sample was heated in air for 1 hour at the indicated temperature, cooled, weighed and reheated to the next higher temperature. The weight loss begins above 300° C and constant weight is observed above 700° C.

In one case a small aliquot was taken for X-ray diffraction analysis after heating to 500° C. The curve shows this as a weight loss. The curve has not been renormalized for this loss since the displacement of the curve is small. Shown in the same figure is the expected weight loss assuming the starting material to be Sm_2O_3-H_2O. In a separate experiment a SmOOH was heated in vaccum to 700° C. The evolved gases were condensible at the temperature of liquid nitrogen and the weight loss was comparable to that reported above. Therefore, it is assumed that the new phase has the composition SmOOH.

Infrared measurements were made with a Perkin Elmer Model 221 spectrometer. The sample was scanned in the wavelength range from 2.5 to 16 microns using both KBr pellet and petrolatum mull techniques. The OH stretching vibration at 2.93 microns was observed together with the bending vibration at 6.8 microns. In addition, unassigned absorption peaks between 11 and 15 microns were observed.

Beta-SmOOH is light yellow in color. Since no single crystals could be isolated, optical properties could not be determined. It was, however, determined to be optically anisotropic with an average index of refraction of approximately 1.93.

The X-ray diffraction results are shown in Table V. These have been indexed on a tetragonal unit cell with $a_o = 8.102$, $c_o = 11.212$. The observed density, 6.62 grams per cc, gives 15.9 formula weights per unit cell. This is reasonable for a tetragonal structure. A structure with 16 formula units per unit cell and a theoretical density of 6.66 can therefore be assumed. As indicated above, no single crystals could be isolated and no more detailed studies could be performed.

However, it should be noted that by a 45-degree rotation around the C-axis, a nearly cubic cell can be obtained with sides averaging about 11.3 A which contains 32 formula units, that is $H_{32}Sm_{32}O_{64}$. This cube is only slightly larger than that of the defect fluorite lattice of cubic Sm_2O_3 and contains just enough excess oxygen ions to fill the vacancies in that structure. In fact, a fair fit of the stronger lines to a cube with $a_o = 5.70$ expedited the determination of the

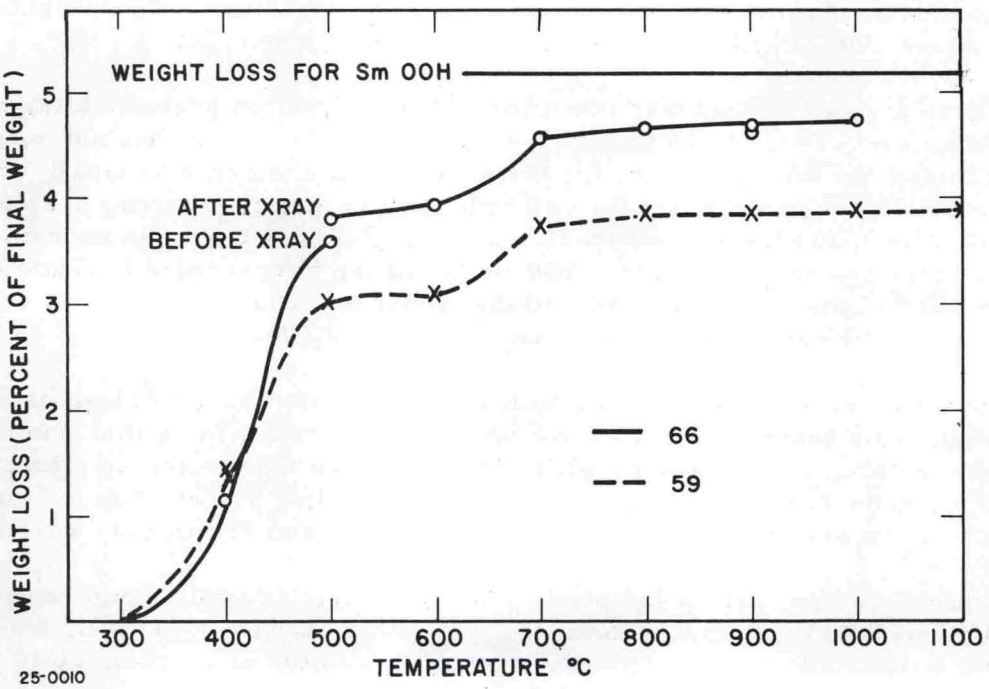

Figure 7 WEIGHT LOSS VERSUS TEMPERATURE FOR SmOOH