(1)

(2)

cases still moist after the experiments. The formation of the trihydroxides or of the oxide hydroxides is dependent on the amount of solution used. In the reactions

$Ho_2O_3 + 3 H_2O = 2 Ho(OH)_3$

$Ho_2O_3 + H_2O = 2 HOOOH$

the volume difference between product and reaction mixture is greatest for (1), and the formation of the trihydroxide is to be expected, when sufficient solution is present. Powder patterns of all products were obtained as for the tetragonal modification of holmium oxide hydroxide, and the rare earth oxide hydroxide phases were indexed, using the tetragonal unit cell parameters given in Table 1.

Single crystals of tetragonal ytterbium oxide hydroxide were prepared from a mixture of ytterbium oxide and solid sodium hydroxide. The mixture was allowed to absorb humidity from the atmosphere for a few min, and was then placed in a platinum ampoule and treated in the high-pressure belt apparatus at 50 kb and 800°C for 1 h. The sample was then slowly cooled to 600°C over a period of 30 min, followed by fast cooling to room temperature. The crystals were washed with water and dried at room temperature.

A single crystal was investigated by precession methods using MoK α -radiation (λ =0.7107 Å). Photographs were taken of *hk*0, *hk*1, *hk*2, and *khl*. The reflections

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ALT: TA #

h00=2n+1 are absent. The symmetry of the photographs are in agreement with the space group $P\overline{42}_1m$ (No. 113). The crystal structure determination of the compound is in progress.

The present investigation shows, that high-pressure modifications of hydroxides can be obtained by hydrothermal synthesis at very high temperatures and pressures, and that a new experimental hydrothermal technique can be applied, using highpressure belt apparatus.

- 1. Christensen, A. N. Acta Chem. Scand. 19 (1965) 1391.
- Christensen, A. N. Acta Chem. Scand. 19 (1965) 1504.
- 3. Christensen, A. N. Acta Chem. Scand. 20 (1966) 896.
- 4. Christensen, A. N. and von Heidenstam, O. Acta Chem. Scand. 20 (1966) 2658.
- 5. Christensen, A. N., Hazell, R. G. and Nilsson, Å. Acta Chem. Scand. 21 (1967) 481.
- 6. Christensen, A. N. Naturwiss. 55 (1968) 386. 7. Wilhelmi, K.-A. Acta Chem. Scand. 23
- (1969) 419.
- Wilhelmi, K.-A. and Burger, W. Acta Chem. 8. Scand. 23 (1969) 414.
- 9. Waintal, A. Étude à haute et très haute pression de quelques transformations cristallographiques et contribution à l'étude des macles parfaites. Thesis, Grenoble 1969.

Received October 6, 1970.

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Acta Chem. Scand. 24 (1970) No. 8