SYNTHESIS OF SODIUM PERTECHNETATE TETRAHYDRATE AND ITS THERMAL PROPERTIES

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No evidence has been given for the existence of sodium pertechnetate hydrates. Schwachau [1] reported the preparation of anhydrous sodium pertechnetate. We have found that passing 0.05 M aq. potassium pertechnetate through a cation-exchange resin in the H^+ form with subsequent neutralization of the eluate by the addition of NaOH and 10-fold evaporation gives the slow growth of large transparent crystals. Chemical analysis indicated that these crystals are sodium pertechnetate tetrahydrate. These crystals are rather stable in the mother liquor but gradually effloresce in the air or over $CaCl_2$ and completely lose their water of hydration over P_2O_5. The thermal properties of $NaTcO_4 \cdot 4H_2O$ were studied on a Paulik–Paulik–Erdey Q-1500D derivatograph. Upon heating to 313 K, this hydrate undergoes an endothermal phase transition ($\Delta S_{\beta \rightarrow \alpha}$ = 27.3 ± 5 J/mole·deg) and at 383 K, it undergoes one-step dehydration: α-$NaTcO_4 \cdot 4H_2O \rightarrow \gamma$-$NaTcO_4 + 4H_2O$ (ΔH = 77 ± 8 kJ/mole). Heat effects were not observed upon cooling γ-$NaTcO_4$, while the cooled sample has scheelite structure with parameters close to those reported by Spitsyn and Kuzina [2] as indicated by x-ray phase analysis. This sample is identical to that obtained from $NaTcO_4 \cdot 4H_2O$ upon efflorescence. Upon heating, γ-$NaTcO_4$ is converted to β-$NaTcO_4$ at 610 K (ΔS = 3.3 ± 0.5 J/mole·deg), while the latter is converted to α-$NaTcO_4$ at 635 K (ΔS = 18.7 ± 2 J/mole·deg). The melting of α-$NaTcO_4$ at 1063 K (ΔH = 8.3 ± 3 kJ/mole) is accompanied by a considerable increase in the volatility of sodium pertechnetate. Heating of sodium pertechnetate above 600 K is accompanied by the appearance of a yellow tint which disappears upon cooling.

LITERATURE CITED

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2. V. I. Spitsyn and A. F. Kuzina, Technetium [in Russian], Izd. Nauka, Moscow (1981), p. 60.

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