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by

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Synthesis of Calcium and Nickel Thiosulphates

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ABSTRACT

Calcium and Nickel Thiosulphate Hexahydrates were synthesized employing original procedures. Single crystals of the mentioned compounds were grown and some of their physical and chemical properties were determined.

INTRODUCTION

$CaS_2O_3.6H_2O$ and $NiS_2O_3.6H_2O$ have been synthesized previously by Petrovici [1] and Blesa [2] using $Na_2S_2O_3.H_2O$ and the the salts of calcium and nickel as starting substances.

In this work the starting substance was $BaS_2O_3.H_2O$ which has not so far been used.

EXPERIMENTAL

The starting substance of $BaS_2O_3.H_2O$ was synthesized using the following known process of Zhirov [3]:

$$Ba(NO_3)_2 + Na_2S_2O_3.5H_2O \rightleftarrows 2NaNO_3 + BaS_2O_3.H_2O$$

100 ml of each solutions of 0.2M $Na_2S_2O_3.5H_2O$ and 0.2M $Ba(NO_3)_2$ were thoroughly mixed and 100 ml 99 % ethanol was then added. $BaS_2O_3.H_2O$ precipitated, forming a white deposite, due to its very slight solubility in water and alcohol. The precipitation was extracted from the solution using a filter and then washed with 50 ml 99 % ethanol. In order to obtain high purity of $BaS_2O_3.H_2O$ the washing procedure was repeated three times.

$NiS_2O_3.6H_2O$ was produced according to the reaction:

$$BaS_2O_3 + Ni^{++} + SO^{=}_4 \rightleftarrows BaSO_4 + Ni^{++} + S_2O^{=}_3$$

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The stoichiometric amount of 5.101 g $BaS_2O_3.H_2O$ was added into the 100 ml 0.2M $NiSO_4.7H_2O$ solution and stirred by means of magnetic stirrer for about 2 hrs. At the end of the procedure the white $BaSO_4$ precipitation was separeted from the solution and $NiS_2O_3.6H_2O$ was extracted off the mother liquor by means of 80 % ethanol. Nickel thiosulphate solution left for a day yielded green needle like crystals which were rather stable at room temperature but decompose into NiS and S above 60°C [4].

$CaS_2O_3.6H_2O$ was obtained using the following process:

$$BaS_2O_3 + CaSO_4 \rightleftarrows Ca^{++} + S_2O^{=}_3 + BaSO_4$$

Equivalent grams of $BaS_2O_3.H_2O$ (5.346g) and $CaSO_4$(2.723g) were mixed 100 ml of water. White precipitate of $BaSO_4$ was formed within 30 minutes of stirring and separeted by a filter. $CaS_2O_3.6H_2O$ was extracted from the mother liquor with the aid of 200 ml 75 % ethanol and left for crystallization in 300 ml 99 % ethanol. The transparent prismatic crystals were formed within 3 days. The crystals tend to loose five water molecules out of six at room temperature [5].

The magnetic susceptibilities of both nickel and calcium thiosulphates were determined employing Gouy's method. As can be seen from the Table 1, the calcium salt is diamagnetic and the nickel salt is paramagnetic at room temperature.

The Na^+ and Ba^{2+}residues in both thiosulphates were analysed with a flame photometer and an energy-dispersive X-ray fluorescence spectrometer respectively [Table 2].

The solubilities of two thiosulphates in various solvents were investigated and the result given in Table 3 in a qualitative way.

DISCUSSION

As reported earlier [1,2,4,5] calcium and nickel thiosulphate hexahydrates have been obtained from $Na_2S_2O_3.5H_2O$ and chloride, nitrate, sulphate salts of calcium and nickel. The solubilities of calcium and nickel thiosulphates in water and alcohol are almost the same with those of the sodium salts produced in the reactions. Thus it becomes evident that it is rather difficult to extract these substances from their solutions in water and alcohol. Very slight

solubility of $BaSO_4$ in water enabled us to use $BaS_2O_3.H_2O$ with either $CaSO_4$ or $NiSO_4.7H_2O$ to obtain either calcium or nickel thiosulphates. So that the mentioned difficulty was overcome.

It must be noted that $NiSO_4.7H_2O$ and $CaS_2O_3.6H_2O$ were very slightly soluble in alcohol on contrary to the literature [6].

TABLE 1

The magnetic susceptibilities of $NiS_2O_3.6H_2O$ and $CaS_2O_3.6H_2O$ at room temperature.

SUBSTANCE	$\varkappa_g$ (cgs/g)	$\varkappa_m$(cgs/mole)
$NiS_2O_3.6H_2O$	1.40×10^{-5}	3.90×10^{-3}
$CaS_2O_3.6H_2O$	-1.70×10^{-7}	-4.42×10^{-5}

TABLE 2

Na^+ and Ba^{++} residues

SUBSTANCE	Na^+(%)	Ba^{++}(%)
$NiS_2O_3.6H_2O$	0.04	0.0110
$CaS_2O_3.6H_2O$	0.06	0.0113

TABLE 3

The Solubilities of $NiS_2O_3.6H_2O$ and $CaS_2O_3.6H_2O$ at room temperature.

SOLVENT	Water	Alcohol	Aceton	Methanol
$NiS_2O_3.6H_2O$	very soluble	insoluble	insoluble	soluble
$CaS_2O_3.6H_2O$	very soluble	insoluble	insoluble	insoluble

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ÖZET

Bu çalışmada $CaS_2O_3.6H_2O$ ve $NiS_2O_3.6H_2O$ orijinal yöntemlerle elde edilmiş, tek kristalleri büyütülerek bazı fiziksel ve kimyasal özellikleri belirlenmiştir.

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