POSSIBILITIES OF HYDRAZINE SULPHATE SYNTHESIS BASED ON LOCAL RAW MATERIALS*

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Abstract

Currently, one of the key requirements of the state is the synthesis of globally competitive import-substituting new substances from local raw materials. This study explores the possibilities of synthesizing hydrazine sulfate from local resources. In alternative medicine, hydrazine sulfate is used in cancer treatment (Unconventional therapies for cancer: Hydrazine sulfate., Hydrazine and hydrazine sulfate.), and in chemical science, it is used in synthesizing heat-resistant polymers containing 1,3,4-oxadiazole rings. Due to the absence of hydrogen atoms and isomers in its composition, the symmetrical structure of 1,3,4-oxadiazole rings leads to greater stability of (1,3,4-oxadiazole) polymers when exposed to air heating compared to other polymers.

Keywords: hydrazine, hydrazine sulfate, urea, sodium hypochlorite.

Laboratory Procedures. Place 300 ml (ρ=1,17g/ml) of a 10% sodium hypochlorite (NaClO) solution into a one-liter glass conical flask and let it cool from 0°C to –15°C overnight. Next, prepare 39g of sodium hydroxide (NaOH) pellets, divide them into 2 portions, and dissolve each portion in 20 ml of distilled water. Mix the cooled sodium hypochlorite solution using a magnetic stirrer and place it in an ice bath. Gradually add one part of the sodium hydroxide solution to the sodium hypochlorite, maintaining the temperature between 10°C and 8°C. Then cool the sodium hypochlorite solution back to 0°C. If the temperature rises, sodium hypochlorite decomposes. After cooling, add the second part of the sodium hydroxide solution (solution 1) to the chilled sodium hypochlorite.

Dissolve 28.8g of urea and 0.842g of gelatin in 30ml of hot water, add it to the sodium hypochlorite solution added to sodium hydroxide at a temperature of 10°C, and mix uniformly (solution 2). Stir until bubbles are removed and cover the flask with a stopper. As a result, hydrazine gas is generated (Scheme 1). Then heat the solution to 95°C and maintain the temperature for 5 minutes. Cool again to 9°–10°C (Patent CN112047312A).

Add 100 ml of water to 55 ml (ρ = 1,84g/ml) of 95% sulfuric acid (forming 48% H_2SO_4 solution), and pour it by drops through a conical funnel into the cooled solution 2. As a result, based on the reaction of sodium carbonate formed in solution 2 with sulfuric acid, carbon dioxide (CO2) is released. Then, hydrazine gas reacts with sulfuric acid to form hydrazine sulfate ($N_2H_5 \cdot HSO_4$). Maintain the reaction temperature at 10°C. When the temperature decreases (to 8°C), sodium sulfate crystals form in the solution. At temperatures between 10–15°C, hydrazine sulfate crystals form and settle at the bottom of the flask. Then, filter the solution, wash it several times with distilled water, and dry the obtained hydrazine sulfate crystals in a desiccator. The resulting yield of the product is 60%.

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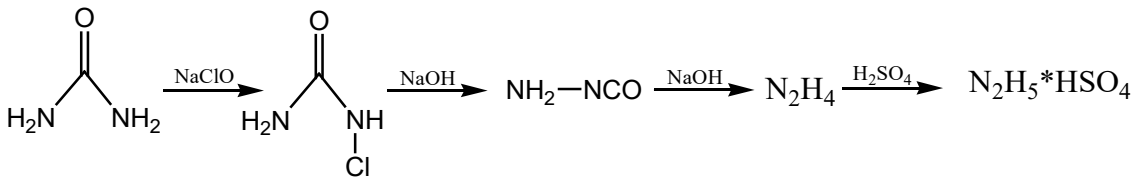
Scheme 1



Chemical Reactions Equations:

$$(NH_2)_2CO + NaClO + 2NaOH \longrightarrow N_2H_4 + NaCl + Na_2CO_3 + H_2O$$

$$Na_2CO_3 + H_2SO_4 = Na_2SO_4 + CO_2 + H_2O$$

$$N_2H_4 + H_2SO_4 \longrightarrow N_2H_5{*}HSO_4$$

Checking Hydrazine Sulfate

Place an equal amount of hydrazine sulfate and silver nitrate in a glass test tube and dissolve them in a small amount of water. Gradually add an aqueous solution of a small amount of ammonia. The result is a reaction of silver glass.

The structure of the synthesized hydrazine sulfate is confirmed using an Infrared (IR) spectrometer of the Nicolet IZ 10 type and XRD diffractometry of the D2PHASER type produced by Thermoscientific USA.

Infrared Spectrum of Hydrazine Sulfate

Sample preparation: Tablets were prepared from a mixture containing hydrazine sulfate ($N_2H_6SO_4$) and potassium bromide (KBr) in a mass ratio 1:9, and IR spectra were recorded (Figure 1) (Patent US3265602A).

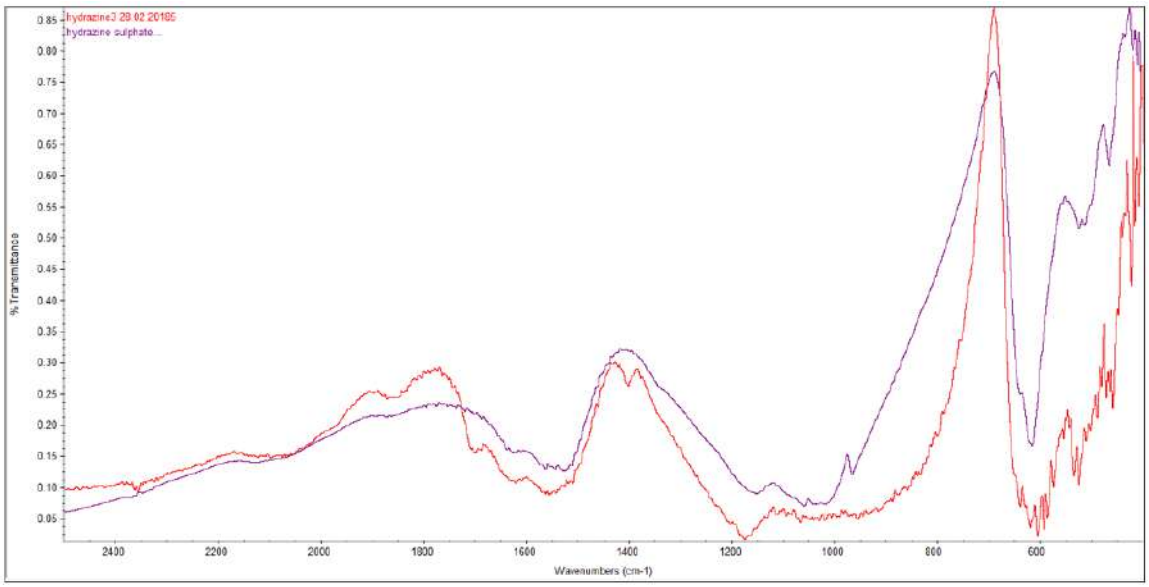


Figure 1. IR spectrum of hydrazine sulfate

As seen in Figure 1, the violet-colored spectrum corresponds to the spectrum of hydrazine sulfate. The red-colored spectrum corresponds to the spectrum of the synthesized hydrazine sulfate. Consequently, the correspondence between these two spectra confirms the correctness of the structure of the synthesized hydrazine sulfate (Patent US2682446A).

X-ray Diffraction Pattern of Hydrazine Sulfate

Sample preparation: A specific mass of hydrazine sulfate is ground and formed into a tablet placed in a cell. The X-ray diffraction pattern of the prepared tablet was examined (Figure 2), and its chemical composition was determined (Table).

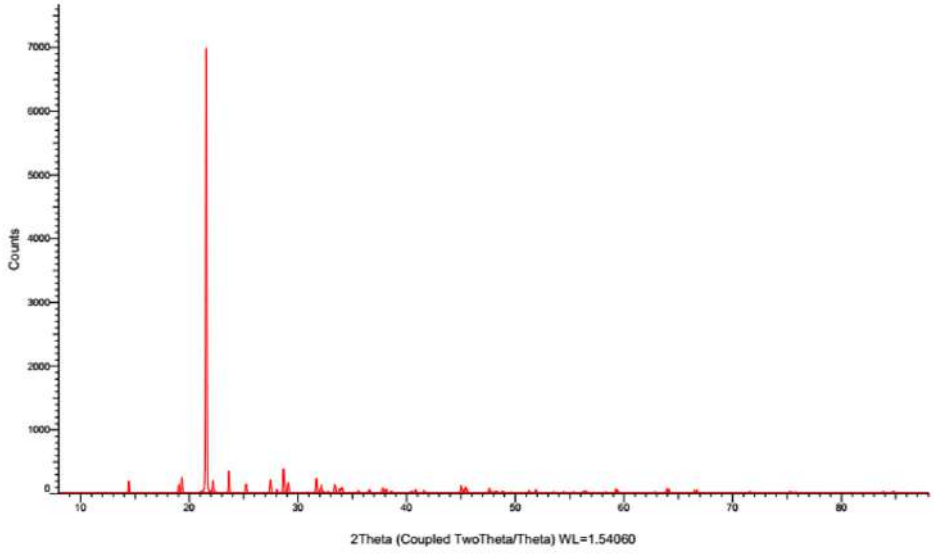


Figure 2. X-ray Diffraction Pattern of Hydrazine Sulfate

Table

Chemical Composition

Substance name	QTY %
Hydrazine Sulfate ($N_2H_6SO_4$)	96.2
Halite (NaCl)	3.8
Total	**100**

As seen from the table, the purity of the synthesized hydrazine sulfate is 96.2%, and the presence of halide (NaCl) is detected between levels 31-32 (2Theta).

CONCLUSION

1. Based on carbamide from the «Marycarbamide», «Tejencarbamide», and «Karabogazcarbamide» plants of the «Turkmenchemistry» State Concern and sulfuric acid from the Turkmenabat Chemical Plant named after S.A. Niyazov, hydrazine sulfate with a purity of 96.2% has been synthesized.
2. The composition and structure of the synthesized hydrazine sulfate have been confirmed through XRD diffraction and IR spectrum analysis.

LITERATURE

1. Patent CN112047312A, Preparation method of hydrazine sulfate.
2. Patent US2682446A, Process for making hydrazine sulfate.
3. Patent US3265602A, Method of producing hydrazine.