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DPT

DPT

Dinitro-pentamethylenetetramine
3,7-dinitro-1,3,5,7 tetraazabicyclo [3,3,1] nonane

$N_2(CH_2)_5(NNO_2)_2$

Since DPT is itself an explosive and can be prepared from a mixed H_2SO_4/HNO_3 nitration on hexamine, it is surprising that this compound is not more popular.

The best method of obtaining DPT consists in introducing hexamine dinitrate to 90% sulfuric acid at 8-15 °C. After 45 minutes all is poured on ice and the solution is filtrated. The filtrate is neutralized with 28% conc. ammonia to pH = 5.5-6.5 and DPT precipitated.

DPT can also be prepared from sulfamic acid, H_2N-SO_3H .

To 100 ml of HNO_3 (density 353g/ml or 53% conc.), cooled to +10 °C, was sprinkled 50g of sulfamic acid, a temperature rise is not observed. After mixing with stirring at 10° C for 30 minutes, 53g of DNU was made, the temperature is kept below 20 °. After holding at this temperature for 20 minutes, the reaction mixture was warmed to 30 °, then external heating was discontinued, but the reaction continued to increase in temperature by itself. After reaching 35-36 ° it was carried off to be gently cooled to 30 °C under running tap water. The temperature again continued to increase after reaching 35-37 °, and the mixture was again cooled to 30-33 °, and so on for as long as the temperature kept increasing. The reaction mixture was cooled to 15 °, and it becomes more viscous during this time. After dilution with 300ml of cold water, neutralization of sodium bicarbonate was carried out to pH = 6, by the end of the neutralization of the solution became dull, and formed a number of fine-grained sediment. After an exposure of 1 hour the solution was filtered, the precipitate was washed first with water and finally with isopropyl alcohol to facilitate drying. The mass of dry product was 3.5g or about 8% of the theoretical value (assuming the formation of DPT 1 mol to 1 mol DNU). The nature of combustion of the sample did not differ from the standard DPT. The measured melting point temperature of the sample amounted to about 195-198 °C. DPT obtained by the other more traditional method has a melting point of 200-201 °C.

Experiment 8

The concentration of nitric acid was about 63% (density 1.386g/ml). A mixture of 50 ml of nitric acid and 35 ml of sulfuric acid (92.5% concentration, density 1.825g/ml), which was cooled to 0 °C, was introduced with stirring 25 g of sulfamic acid. The temperature rose to 5 °C. After maintaining the temperature for 20 minutes at 0 °C, 27g of DNU started to be added in small portions with stirring, keeping the temperature below 15 °C. The reaction mixture was then kept at 5-10 °C for 40 minutes, while continuing stirring. The mixture was then gently heated to 30 °C, then heating was stopped, but the temperature continued to increase by itself, as in the previous experiments. Just as before, the temperature was allowed several times to reach 38-42 °C, and at such times the temperature was brought back down to 35 °C by cooling. When heat ceased to occur (in about an hour after heating to 30 °C), the reaction mixture was cooled to 15 °C, while it thickened and became similar to the reaction mixture during cooking. More PETN was diluted 1 to 2 cold porridge, prepared in advance by the freezing of 10% aqueous solution of ammonium nitrate. This was followed by neutralization with sodium bicarbonate to a pH of ~ 6, resulting in much sediment. This was filtered, then washed with water and isopropanol. After drying, the mass of sludge was 14.2g, or 65.1% of theory (DPT 1mol to 1mol DNU). The melting temperature of the product was 194-198 °C.

Experiment 9

The concentration of nitric acid 70% (density 1.411g/ml). All procedures are identical to experiment number 8, but with the following differences: the amount of sulfuric acid is reduced to 20 ml, after the termination of heat the reaction mixture of the porridge thickens much faster and at lower temperature, no more than about 25-30 ° C. The yield of DPT in this experiment was 21 g or 96.3% of the theoretical (assuming the formation of 1 mol DPT to 1 mol DNU). When doing this experiment two additional times, the yield was 20g and 20.5g, respectively, which was consistently above 90% yield. The products obtained by this method were in the temperature range of 198 ° C.

Kommentare