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MEDINA salt of hydrazine

Neutralize MEDINA with hydrazine hydrate, forming the somewhat more stable salt. This ionic compound may be denser and more resistant to shock than the parent MEDINA; it has similar power. MEDINA is acidic and when neutralized by a base, it becomes more stable. A +2 ion and a -2 charged ion pack into dense, more poorly soluble crystals, usually.

Preparation of precursor: Methylenediformamide:

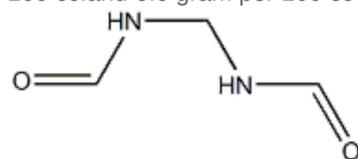
In a 1 liter reaction vessel place-A mixture of 70 grams of hexamine(0.5mole) and 270 grams(6mole) of freshly distilled formamide were heated to 140 deg C and maintained for 4 hours. On cooling the crystals were separated. Initial yield is 87.8 grams (29% yield)

To obtain more crop,the mother liquor was retained and to it was added 23.5 grams of hexamine and 90 grams of formamide.Heat the mixture to 140deg C for 5 hours.Cool the mixture and separate the resulting crystals. Successive yields range from between 80-100 grams for each reaction run. If it is desired to obtain more precursor, repeat procedure for 8 additional times using similar quantities hexamine and formamide (23.5grams and 90 grams respectively). This takes about 50 hours, consuming a total of 281.5 grams of hexamine and 1080 grams of formamide.The total yield of methylenediformamide is 1034 grams (78% yield).

Reaction equation



The pure product is a white crystalline material melting at 140-141degC.It is soluble in water at an extent of 100 grams per 100 cc.and 6.6 gram per 100 cc of isopropanol.



Preparation of Methylenedinitramine:

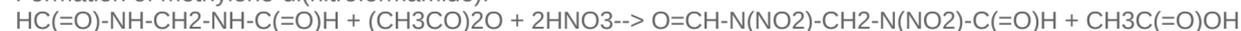
Place the 2-3 liter reaction vessel preferably in a dry ice-kerosene bath so as to much better control the temperature of the exothermic reaction between nitric acid,acetic anhydride and methylenediformamide. Nine moles(380 grams) of 100% HNO₃ was added as rapidly as possible at +-2deg C. to a well stirred suspension of 102 grams (1 mole) of methylenediformamide in a 380 ml(4 mole) of acetic anhydride. Upon completion of the nitric acid addition,remove the dry ice-kerosene bath,and replace it with an ice-bath. Allow the reaction to age over this ice bath for 4 hours. It will be noticed that after the application of the ice -bath, the temperature of the reaction will rise to 8deg C for 15-30 minutes and then fell to +2deg C where it remained during the aging period.

At the end of the aging period the reaction mixture was poured slowly with stirring into a mixture of one liter water and 1000-1500 grams of cracked ice. The methylenedi(nitroformamide) was immediately collected on a filter and washed with three 100 ml portions of ice -water.The filter cake was pressed with a rubber dam.The yield at this point was 190-220 grams of damp material.

This methylenedi(nitroformamide) is stirred into 100 ml of formic acid and the resulting paste allowed to stand overnight. The following day the mixture was warmed to 65 deg C to dissolve the methylenedinitramine which has crystallized. Filter and cool to ice temperature(and seed it preferably to cause crystallization.One way of doing this is to rub gently the end of the stirring rod inside the walls under the crystallizing solution).This crystallization will take 5-6 hours.The crystals were collected on a filter and washed with benzene (or toluene). The yield at this point is 70-80 grams of pure methylenedinitramine (50-60% based on methylenediformamide) To obtain a second extraction of methylenedinitramine,the mother liquor(with formic acid) was concentrated under reduced pressure and cooled to ice temperature and seeded to cause crystallization.The calculated total yield was 75-85% of theoretical based on the amount of methylenediformamide,about 100-115 grams of MEDINA.

Reaction equations

Formation of methylene-di(nitroformamide):

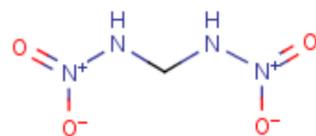


Hydrolysis with formic acid:



For further purification of Methylenedinitramine, recrystallize it from a 9/1 by volume mixture of either of these solvents composition: ethylenedichloride/isopropanol, ethylenedichloride/2-nitropropane, or ethylenedichloride/95%acetic acid

The purified methylenedinitramine (MEDINA) melts at 105-106deg C.It begins to decompose with the evolution of gas at 110degC. During detonation, MEDINA generates pressures of 345 kbar.



Comments