

Formel: C₂H₂N₄O₃
Molgew.: 130.07g

Löslichkeit (g/l) @20°C: Aceton: 18g DMSO: 625g H₂O: 49g

Stoff:	OB:	Dichte:	VOD:	Druck:
NTO:	-24.60%	1.93 g/ml	8.752 m/s	37.46 Gpa
DTNTO:	-21.81%	2.08 g/ml	8.795 m/s	40.14 Gpa

Synthese:

The method adopted by Australian workers closely mirrored this route and involved the reaction of semicarbazide hydrochloride with formic acid followed by a mixed acid nitration, in a one-pot reaction. Formic acid (88%, 34.5 ml, 0.8 mol) was added to solid semicarbazide hydrochloride (33.45g, 0.3 mol) at ambient temperature and the resulting solution was then heated to 65°C and stirred to give the intermediate 4. A mixture of neat nitric (100ml) and 98% sulfuric acid (20ml) was then added and the temperature maintained at 65°C for between 1.5 and 2 hours. The mixture was then cooled and filtered, and the resulting solid washed with cold water. Recrystallisation from hot water gave the target NTO as a white crystalline solid in 77% yield. Other synthetic conditions varying the nitration method and reaction temperature were examined, but this route gave optimum yields and could easily be applied to larger scale reactions.

2,4-Dihydro-3H-1,2,4-triazol-3-one

Semicarbazide hydrochloride (6.6g) was added to the stirred solution of formic acid (6.9cm³). The reaction mixture was kept under stirring until all the semicarbazide hydrochloride dissolved completely. Subsequently, the excess of formic acid was removed by distillation until crystallization commenced, and then 10 cm³ of water was added. The distillation was continued to dryness and the residue was recrystallized from ethanol to obtain 1 as white solid (9.56g, 75%), having the melting point of 232°C.

5-Nitro-2,4-dihydro-3H-1,2,4-triazol-3-one

The synthesized TO (1.0g) was added to the stirred solution of nitric acid (10cm³) at 0°C, and the mixture was heated to 70°C for 16 h with constant stirring. An exothermic reaction occurred with the evolution of brown fumes. The product precipitated out, on completion of the reaction. The reaction mixture was allowed to cool down in ice bath. The precipitate was filtered and washed with water to remove the excess of nitric acid. The product obtained was recrystallised from water to obtain 2 (1.18g, 70%), having the melting point of 272-274°C.

2,4-Dinitramino-5-nitro-1,2,4-triazol-3-one

Acetic anhydride (1.56g) was added slowly to the stirred mixture of fuming nitric acid (0.580g) and HCl (0.124g). After stirring the reaction mixture for half an hour at 0°C, NTO (0.500g) was added in small portions. The reaction mixture was stirred overnight at room temperature and for 1-2 h at 35-40°C. The colour of the contents changed from red to white during the progress of the reaction monitored by TLC. After completion of reaction, the mixture was poured onto crushed ice. The precipitate was filtered and dried to obtain title compound (0.294g, 36%), having a melting point at 248-250°C.