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Base Hydrolysis of HMX/DMSO Solutions

Philip F. Pagoria

March 15, 1994

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Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

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Base Hydrolysis of HMX/DMSO Solutions

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March 15, 1994

We investigated the decomposition of HMX or PBX-9404 as a 25% solution in DMSO by treatment with aqueous base solutions. We investigated two concentrations of NaOH solutions, 40% NaOH and 2N NaOH. The method we chose to follow the decomposition was by ^1H -nmr spectroscopy, watching the disappearance of the $-\text{CH}_2-$ absorbance of HMX at 6.2 ppm. The 40% NaOH solution is initially immiscible with the DMSO solution but becomes miscible as it reacts. The reaction is exothermic with recorded temperatures after mixing reaching 90°C. The mixture does foam somewhat but this can be controlled by slowing down the stirring speed which reduces the foaming. The 2N NaOH solution is miscible with the HMX/DMSO solution but upon mixing the HMX is precipitated by the water present in the base solution. The mixing of the solutions is much less exothermic and the decomposition is slower than the 40% NaOH solution so the mixture is heated at 60 °C for 1h to facilitate the decomposition.

Procedure: A 3g (10.1 mmol) sample of HMX or PBX-9404 was dissolved in 12 mL of DMSO- d_6 . A 40% NaOD/D $_2$ O solution (6.1 mL) (61 mmol) was added and the biphasic mixture was stirred at room temperature. Upon stirring the mixture became homogenous, foamed slightly (this may be

controlled by slowing down the stirring which reduces the foaming), and warmed to 90°C. The mixture was stirred 10 min and an ^1H -nmr spectrum was taken of the still warm mixture. The ^1H -nmr spectrum indicated that all the HMX was decomposed (loss of peak at 6.2 ppm which corresponds to the $-\text{CH}_2-$ protons of HMX). The ^1H -nmr spectrum shows a peak at 8.5 ppm which we have assigned to sodium formate. It may be generated from the HMX decomposition by the reaction of formaldehyde with excess base present. The final solution is a thick, red, viscous suspension.

This procedure can also be done using 60 mmol of a 2N NaOH solution (30 mL) and heating the solution at 60°C for 1 h. Upon mixing the DMSO and base solutions a slurry is formed. After 1 h a clear yellow solution is realized. The final ^1H -nmr spectrum is almost identical to the one obtained from the decomposition using the 40% NaOD solution.

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