Full Paper

Preparation of HMX by Perfluorooctanesulfonic Acid Catalyzed Nitrolysis of Hexamethylenetetramine in Fluorous Media

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Abstract

Perfluorooctanesulfonic acid ($CF_3(CF_2)_7SO_3H$, PFOS) efficiently catalyzes the nitrolysis of hexamethylenetetramine in fluorous media, affording a good yield of the explosive HMX. The reaction can be carried out without acetic anhydride or acetic acid. The fluorous phase containing the catalyst could be easily separated and can be reused several times without significant loss of catalytic activity.

Keywords: Fluorous Media, Hexamethylenetetramine, HMX, Perfluorooctanesulfonic Acid

1 Introduction

Nitrolysis is one of the key processes in synthetic chemistry and is widely used industrially [1]. Traditional nitrolysis in the manufacturing process requires large amounts of concentrated or fuming nitric acid, leading to excessive acid waste. Thus, nitrolysis is one of the most troublesome functionalizations in the context of green chemistry [2]. Despite some breakthroughs based on the use of various catalysts [3] and the use of $NO^+BF_4^-$ [4], $N_2O_5/$ HNO_3, N_2O_5/halobenzenes, $N_2O_5/(CF_3CO)_2O$, $N_2O_5/(CF_3SO_2)_2O$ [5] as a nitrolysis agent, major problems remain especially when nitrolysis is required for the production of energetic materials.

HMX, named octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazine or cyclotetramethylenetetranitramine, a high explosive energetic material, plays an important role in defence industry in many countries [3]. In the past decades, many methods for its preparation have been reported in the literature. These include Urotropine (hexamethylenetetramine) [1], DPT (1,5-endomethylene-3,7-dinitro-1,3,5,7-tetrazacyclooctane) [1b], TAT (1,3,5,7-tetracetyl-1,3,5,7-tetrazacyclooctane) [6], DADN (1,5-diacetyl-3,7-dinitro-1,3,5,7-tetrazacyclooctane) [7], and DANNO (1,5-diacetyl-3-nitro-5-nitroso-1,3,5,7-tetrazacyclooctane) methods [8],

comprehensive process methods [9], smaller molecular synthetic methods [10], and others. Traditional synthesis of HMX in the manufacturing process requires the use of large amount of acetic anhydride (10–12 equiv.) with acetic acid (24–28 equiv.), leading to excessive acid waste streams and added expense.

Inspired by the recent work on polynitration [11] in the presence of perfluorooctanesulfonic acid (PFOS), a strong monobasic acid with an acidity function H_0 of -12.08 (that of sulfuric acid is -11.90) [12] and especial solubility in fluorous solvents [perfluorocarbons (PFC)] [11, 13], we have applied the catalytic system PFOS–PFC to the nitrolysis of hexamethylenetetramine. In this catalytic system, hexamethylenetetramine could be converted into the explosive hexahydro-1,3,5-trinitro-1,3,5-triazine or cyclotrimethylenetrinitramine (RDX) in rather high yields in the presence of catalytic amounts of ammonium nitrate (20–50 mol-%) at room temperature [14]. In this paper, it was exciting to find that good yields of the high explosive HMX were obtained in the presence of 3–5 equiv. of ammonium nitrate and 25–50 mol-% of metaformaldehyde. The fluorous phase containing the catalyst could be easily reused by simple phase separation. We would like to report herein the work on this new application of the catalytic system.

2 Experimental

2.1 General Remarks

Melting points were obtained with Shimadzu DSC-50 thermal analyzer. ^{19}F NMR spectra were characterized with a Bruker Advance RX300 spectrometer. IR spectra were recorded on a Bomen MB154S infrared analyzer. UV–Vis spectra were obtained with a UV-1601 apparatus. Mass spectra were recorded on a Saturn 2000GC/MS instrument. Elemental analyses were performed on a Yanagimoto MT3CHN recorder. PFOS was commercially obtained

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from ARCOS Co. All fluorous solvents were purchased from Aldrich Co. Commercially available reagents were used without further purification.

2.2 Typical Procedure for Nitrolysis in Fluorous Media

Hexamethylenetetramine (1 g, 7.1 mmol) was added to a mixture of metaformaldehyde (0.16 g, 1.8 mmol), PFOS (0.36 g, 0.71 mmol), and perfluorodecalin ($C_{10}F_{18}$, *cis* and *trans*-mixture, 15 mL) in a glass flask under vigorous stirring. After 10 min, a solution of 95% HNO_3 (1.4 mL, 35.5 mmol) containing ammonium nitrate (1.7 g, 21.3 mmol) was introduced into the flask within 30 min at 45 °C. The mixture was kept at 45 °C for 45 min, and then refluxed at 108 °C for 20 min. When the reaction was finished, the reaction mixture was cooled to 0–5 °C and filtered. The solid obtained was washed with warm water (5 mL × 2), and dried to give a white product (1.08 g, 51.6%) with a m.p. of 258–264 °C. The product was recrystallized from DMF (4 mL). When the obtained solid was stirred in boiled water for 0.5 h, cooled, and filtered, a 93.4% recovery of HMX (1.01 g, based on the crude product) with a m.p. of 278–280 °C was obtained. IR spectra of the product are identical with the authentic sample. $C_4H_8N_8O_8$: anal. calcd. C 16.22, H 2.70, N 37.84; found C 16.20, H 2.75, N 37.80.

2.3 Typical Procedure for Catalyst Recycling

After the reaction as described above, the filtrate from the reaction mixture was allowed to stand for ca. 1 h without stirring at 0–5 °C, and the water phase on the top layer was separated using a pipette. The resulting fluorous phase was ready for further runs which means the reaction can be performed again by adding hexamethylenetetramine (1 g, 7.1 mmol), metaformaldehyde (0.17 g, 1.9 mmol), ammonium nitrate (1.7 g, 21.3 mmol) in 95% HNO_3 (1.4 mL, 35.5 mmol) to the fluorous phase.

## 2.4 Typical Procedure for the Test of Leaching and Catalyst Recovery

The above-mentioned water phase was shaken with hot $CF_3C_6H_5$ (5 mL, 40–60 °C), and the $CF_3C_6H_5$ layer was separated. An aliquot (0.8 mL) was analyzed by GC-MS equipped with an FID. Injection port temperature was 250 °C, detector temperature was 270 °C, and carrier gas was nitrogen. The recycled fluorous phase was evaporated under reduced press to remove and recover perfluorodecalin. The residue (PFOS) was analyzed by UV–Vis or ^{19}F NMR. For the measurement of UV–Vis, pure water (10 mL) was added to the residue and an aliquot (4 mL) was conducted on the UV-apparatus. In the measurement of ^{19}F NMR, a solution of C_6F_6 (0.1628 g, 0.876 mmol) in $CF_3C_6H_5$ (15 mL) was prepared. The residue was dissolved in the solution for NMR analysis; ^{19}F NMR (300 MHz, $CF_3C_6H_5$): δ (ppm) =

−80.9 (CF_3), −112.7 ($\alpha - CF_3$), −119.8 ($\beta - CF_3$), −121.1 ($\gamma - CF_3$), 122.4 ($3X - CF_2-$), 125.8 ($\theta - CF_3$). The total intensities of all $(CF_2)_7CF_3$-based CF_3 triplets were integrated against the C_6F_6 signal.

3 Results and Discussion

The nitrolysis of hexamethylenetetramine was carried out in perfluorodecalin using 95% nitric acid as a nitrolysis agent (Scheme 1). We first screened a variety of nitrolysis conditions (Table 1). Accordingly, the nitrolysis could be divided into three processes, that is, feeding, keeping and reflux. Taking the classical method into consideration [1] for the preparation of HMX, we decided to select 45 °C as feeding temperature, 45 °C as keeping temperature, and 108 °C as reflux temperature. In the reaction, our efforts here were directed toward the elimination of acetic anhydride and acetic acid consumed. The reaction using nitric acid alone did not give conversion to HMX even in the presence of excess acid (20 equiv.) and ammonium nitrate (4–6 equiv.). However, the use of PFOS resulted in moderate yields of HMX, which indicates that the presence of PFOS is essential for the occurrence of formation of HMX. It was found that the proper amount of metaformaldehyde could promote nitrolysis evidently, which is in accordance with the reported case [1]. Notably, the system containing 10 mol-% PFOS, 3 equiv. of ammonium nitrate, and 25 mol-% metaformaldehyde with 5 equiv. of nitric acid gave the HMX in 48.2% yield. Increase in amounts of nitric acid or PFOS did not enhance the yield of nitrolysis. Compared with the conventional process, the preparation of HMX in fluorous media produced much smaller amounts of waste acid.

We then investigated the effect of time (the feeding, keeping, and reflux time) on the outcome of the nitrolysis reaction with hexamethylenetetramine as the substrate under the conditions of entry 6 in Table 1. The results are summarized in Table 2. It was found that among the feeding times tested, 30 min was the best choice. Decrease in feeding time resulted in much lower yield of HMX due to the formation of by-product RDX which resulted from the high concentration of HNO_3 in the reaction system. In the study of keeping time, 45 min was found to be best favorable for the nitrolysis. Increase in keeping time led to the decomposition of the intermediate DPT, furthermore, the low yield
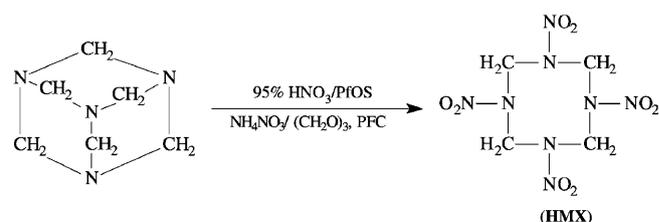
Scheme 1. Nitrolysis of hexamethylenetetramine in fluorous media.

Table 1. Nitrolysis of hexamethylenetetramine in perfluorodecalin.[a]

Entry	HNO$_3$ (equiv.)	NH$_4$NO$_3$ (equiv.)	PFOS (mol-%)	(CH$_2$O)$_3$ (mol-%)	Yield (%)[b]
1	5	–	–	–	–
2	10	4	–	–	–
3	20	6	–	–	–
4	5	2	5	–	12.3
5	5	3	10	–	36.7
6	5	3	10	25	48.2
7	5	3	10	50	40.7
8	5	3	15	25	47.6
9	8	3	10	25	38.1
10	5	5	10	25	45.4

[a] The reaction conditions: hexamethylenetetramine, 7.1 mmol; perfluorodecalin 15 mL; the feeding, keeping, and reflux time were 30, 60, and 60 min, respectively.
[b] Isolated yields calculated on the basis of one mole of HMX per mole of hexamethylenetetramine.

of HMX. As reflux time, only 20 min were required to obtain good yields of HMX. Increase in reflux time appeared not to work for the improvement of nitrolysis.

Attempts were made to recycle the catalytic system using the reaction under the conditions of entry 8 in Table 2. When the reaction was finished, the reaction mixture was filtered and the filtrate was allowed to stand for ca. 1 h at 0–5 °C. The fluorous phase with PFOS catalyst can separate from the water phase and return to the bottom layer. Use of the fluorous phase, recycled without purification was equally effective, furnishing the HMX product with 51.6, 50.8, and 49.2% yields. The problem of leaching in these catalyst recycling system was studied. Based on GC-MS data, no loss of fluorous solvent perfluorodecalin to the water phase can be detected. There is an absorbance observed for PFOS at around 230 nm on a UV–Vis apparatus that can be used as a reference signal for qualitative and quantitative measurements. According to the previous report, all compounds of the formula X(CF$_2$)$_7$CF$_3$ give essentially the same CF$_3$ NMR chemical shift [15]. Hence, the "total number of leached pony tails" can be quite sensitively determined by this method. The measurements of ^{19}F NMR and UV–Vis showed that no significant signal for PFOS leaching to the

water phase was found. Such results are in accordance with the cases of polynitration [11]. In fact, we also investigated the exact amount of PFOS in the recovered fluorous phase by ^{19}F NMR and UV–Vis, finding that 99.9% of catalyst retained in perfluorodecalin. This result suggests the robustness of the catalytic system for recycling use. However, we found that the loss of PFOS leaching to the water phase becomes obvious when the volume ratio of fluorous phase to water phase decreased. The results of ^{19}F NMR and UV–Vis showed that the loss of PFOS increased from 0.1 to 8.9% with the ratio mentioned above decreasing from 10:1 to 1:1. Thus, controlling high volume ratio of fluorous phase to water phase is an important strategy for the use of this novel catalytic system in nitrolysis.

Various fluorous solvents were investigated for the nitrolysis (Scheme 1) under the conditions of entry 8 in Table 2. As shown in Table 3, lower yields were found in perfluorooctane (C$_8$F$_{18}$) and perfluorooctyl bromide (C$_8$F$_{17}$Br). When using perfluorotoluene (C$_7$F$_8$) as a fluorous solvent, the nitrolysis also proceeded smoothly to give the desired product in 52.4% yield. However, perfluorotoluene was found to be contaminated easily by some unknown organic compounds resulting from a sidereaction in nitrolysis, leading to poor efficiency of catalyst recycling. Other commercial common fluorous solvents, such as perfluorohexane (C$_6$F$_{14}$) and perfluoromethylcyclohexane (C$_7$F$_{14}$) are not suitable for the nitrolysis because they are very volatile (perfluorohexane, bp. 57 °C; perfluoromethylcyclohexane, bp. 76 °C). Therefore, perfluorodecalin is the best fluorous solvent found for the nitrolysis.

Mechanism research for the synthesis of HMX by the nitrolysis of hexamethylenetetramine is rather complicated and challenging work. Firstly, the reasonable role for PFOS as a catalyst for PFOS–PFC catalyzed nitrolysis was discussed. Chapman et al. [16] reported that the protonitronium salt NO$_2^+$CF$_3$SO$_3^-$ derived from nitric acid and trifluoromethanesulfonic acid (CF$_3$SO$_3$H) was an efficient nitrolysis agent. With this powerful reagent, even one of the highly deactivated amides, such as nosylamide containing the most electronegative protecting group to date, can be directly nitrolyzed to the corresponding nitramine in high

Table 2. The effects of time on the nitrolysis in perfluorodecalin.[a]

Entry	Feeding (min)	Keeping (min)	Reflux (min)	Yield (%)[b]
1	15	60	60	39.7
2	30	60	60	48.2
3	45	60	60	43.1
4	60	60	60	32.6
5	30	30	60	38.4
6	30	45	60	50.9
7	30	45	40	51.4
8	30	45	20	51.6

[a] Eqivalent and mol-% correspond to reaction entry 6 in Table 1.
[b] Isolated yields calculated on the basis of one mole of HMX per mole of hexamethylenetetramine.

Table 3. Nitrolysis of hexamethylenetetramine in various fluorous solvents.[a]

| Entry	PFC	Yield (%)[b]
1	CF$_3$(CF$_2$)$_6$CF$_3$	43.9
2	CF$_3$(CF$_2$)$_6$CF$_2$Br	42.6
3		52.4
4		51.6
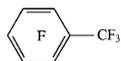
[a] Eqivalent and mol-% correspond to reaction entry 6 in Table 1, feeding keeping and reflux times to reaction entry 8 in Table 2.
[b] Isolated yields calculated on the basis of one mole of HMX per mole of hexamethylenetetramine.

yield. Thus, in our research the assumed catalytic cycle is shown in Scheme 2. A key nitrolysis agent is proposed to be nitroperflate $NO_2^+C_8F_{17}SO_3^-$, which possesses more strong nitrolysis ability because of electron drawing of fluorous tail "$CF_3(CF_2)_7$" that strengthens the electrophilicity of NO_2^+. The nitroperflate reacts with amides to afford the corresponding nitramine accompanied by the regeneration of PFOS. Secondly, the possible role of PFOS as an emulsifying agent in the microemulsion system was proposed. Microemulsion is a wellknown reaction system for the nitration because of its extremely large interfacial areas [17]. PFOS is an emulsifying agent with high surfactancy and chemical stability [18]. In the nitrolysis process there may be the formation of some kind of microemulsion derived from fluorous phase and water phase [19]. Thus, we think PFOS may act as both catalyst and surfactant for the highly efficient nitrolysis reaction. As to PFC, did it only serve as a solvent for dissolving catalyst? It is not sure! Recently, many researchers reported that fluorous solvents are suitable for those exothermic reactions because they can be used as obstructors for thermal transmission [20] which is essential in the synthesis of HMX by the nitrolysis of hexamethylenetetramine [1]. Finally, the function of metaformaldehyde and ammonium nitrate was described according to the previous report. Existence of metaformaldehyde could serve for the stabilization of the intermediate DPT. In the reaction system the intermediate DPT was stabilized by metaformaldehyde which could act as an obstructor for the decomposition of DPT [21]. Ammonium nitrate in the PFOS – PFC catalyzed process was assumed to play two important roles: contribution partly to amino nitrogen in HMX molecules and hindrance of sidereactions derived from the concentrated nitric acid.

4 Conclusion

The PFOS – PFC system can be considered as the most attractive alternative method to the existing ones for the preparation of HMX by nitric acid nitrolysis of hexamethylenetetramine. The advantages of our catalytic system over others are: The reaction can be carried out without acetic anhydride or acetic acid (i), the fluorous phase containing catalyst could be easily and efficiently recovered for reuse by simple phase separation (ii). Further investigations of more favorable synthetic conditions and mechanism for
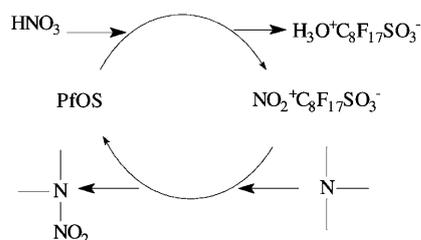
nitrolysis, as well as synthetic applications, are currently in progress and will be reported in due course.

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Scheme 2. The assumed mechanism for PFOS – PFC catalyzed nitrolysis.

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Acknowledgements

Abbreviations

HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazine
DPT	1,5-Endomethylene-3,7-dinitro-1,3,5,7-tetrazacyclooctane
TAT	1,3,5,7-Tetracetyl-1,3,5,7-tetrazacyclooctane
DADN	1,5-Diacetyl-3,7-dinitro-1,3,5,7-tetrazacyclooctane
DANNO	1,5-Diacetyl-3-nitro-5-nitroso-1,3,5,7-tetrazacyclooctane
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine
PFOS	Perfluorooctanesulfonic acid
PFC	Perfluorocarbons
DM	Dimethyl formamide