Insensitive Octogen

Peter Gerber

Fraunhofer Institut für Chemische Technologie, Joseph-von-Fraunhofer-Straße 7, 76327 Pfinztal, Germany, e-mail: Peter.Gerber@ict.fhg.de

ABSTRACT

The sensitivity of explosives charges against shock is linked to the properties of the crystal structure of the energetic filler. To understand the insensitivity of a charge it is necessary to characterize the used Nitramine crystals. In this paper conventional Octogen is compared with insensitive HMX. First the crystallization process of insensitive HMX is described. All particles are characterized with many different analysis methods.

The morphology is described by microscopic and scanning electron microscope (SEM) pictures. Determining the physical properties of the particles, the particle size and the particle size distribution was measured by laser light scattering. The density of the particles was measured with a fluid pycnometer. To find out whether there are any differences in the content of RDX in the Octogen crystal, High performance liquid chromatography (HPLC) was used. With an Accelerating Rate Calorimeter (ARC), from Columbia Scientific Industries (CSI), USA, measurements were made to detect differences in the self heating rate of the samples.

Formulation work was done to find out differences in shock sensitivity. PBXN-109 analogue charges were made with conventional and insensitive Octogen. During the manufacturing process the viscosity of the uncured PBX slurry was determined. A 50 mm GAP – Test with PMMA was performed on cured samples.

1 INTRODUCTION

To compare different Octogen crystal qualities, conventional HMX was bought, manufactured by Dyno Nobel ASA, Norway. At Fraunhofer ICT NSI-1-295 was used to recrystallize Octogen in the solvents γ-Butyrolactone (γ-Bl) and Propylencarbonate (PC), to achieve insensitive Octogen. Table 1 summarizes the different charges of HMX, which were studied.

Table 1: Different types of Octogen

product	Lot-#
HMX, Type B, Class 1	DDP04D0049E
HMX, Type B, 10-15 µm	NSI 00 E 000 E 004
HMX for recrystallization	NSI -1 - 295
HMX recrystallized in PC	100110-1
HMX recrystallized in γ-Bl	100110-2

2 Crystallization Process

To get a controllable crystal quality, it is necessary to know the solubility of Octogen in the used solvents. Figure 1 shows the solubility of HMX in γ-Butyrolactone and Propylencarbonate as a function of temperature. The solubility of HMX in γ-Bl in the studied temperature range between 20°C and 80°C is more than two times higher compared to PC.

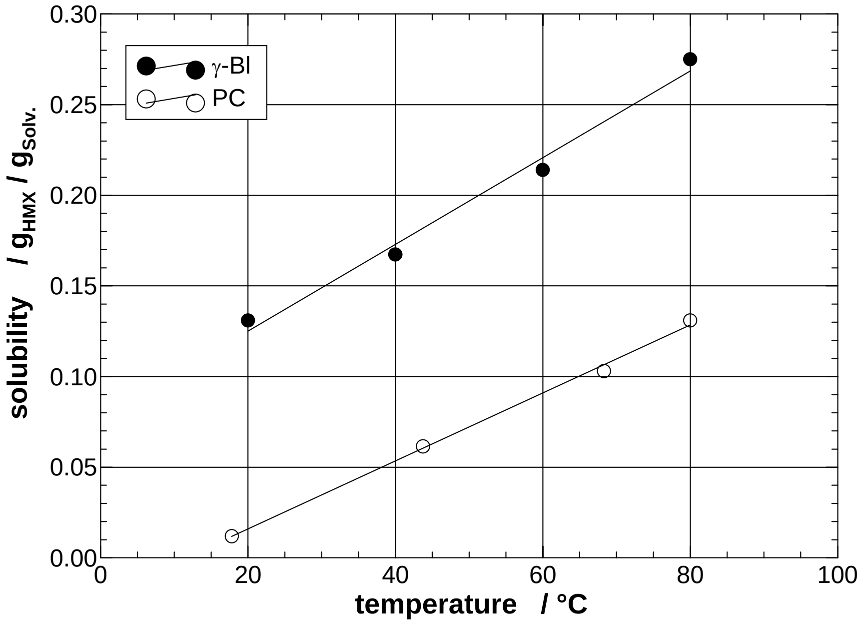


Figure 1: Solubility of HMX in γ-Butyrolactone or Propylencarbonate

The temperature control of the crystallization process is necessary to get a reproducible crystal quality. After heating the solvent up to 85°C to dissolve all crystals, the solution is cooled to generate supersaturation and crystal growth.

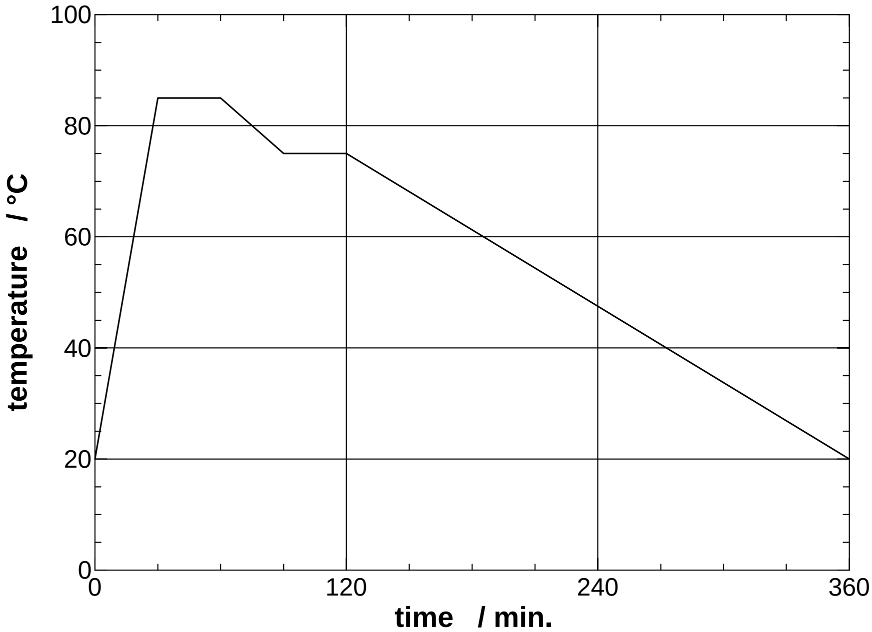
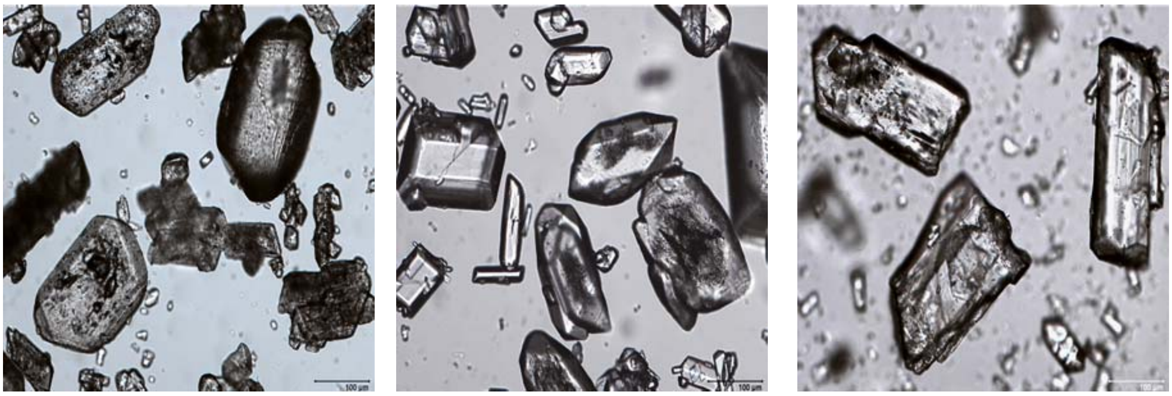


Figure 2: March of temperature during HMX – recrystallization process

3 Particle Characterisation

The produced particles by recrystallization process and the conventional available Octogen were characterized with different methods. To get an impression of the morphology, microscopic pictures were made. Figure 3 shows the microscopic pictures of crystals with approximately the same particle size.



HMX, Grade B, Class 1, Lot. Nr.: DDP04D0049E

HMX, recrystallized in PC, Lot-Nr.: ICT 100110-1

HMX, recrystallized in γ-Bl Lot-Nr.: ICT 100110-2

Figure 3: Microscopic picture of different crystal types

The particle size and the particle size distribution were measured by laser light scattering with a Malvern Mastersizer. As it is shown in Table 2, the mean particle size of HMX recrystallized in PC is smaller compared to HMX, Type B, Class 1. The largest particles were obtained by recrystallization in γ-Bl.

Table 2: Particle size and Particle sizes distribution

	$x_{50.3}$	Span
Product	µm	-
HMX, Type B, Class 1	161.6	0.640
HMX, Type B, 10-15 µm	15.5	0.411
HMX, recrystallized in PC	103.9	0.927
HMX, recrystallized in γ-Bl	285.9	0.832

To characterize the particle size distribution, the Span

$$\frac{x_{84.3} - x_{16.3}}{2 \cdot x_{50.3}}$$

of each HMX fraction was determined. With a Span greater than 0.8, both recrystallized HMX types have a wider particle size distribution compared to conventional HMX, Type B, Class 1.

The density of the particles was measured with a fluid pycnometer. As you can see in Table 3 the recrystallized Octogen has a significant higher density, compared to conventional Octogen.

Table 3: Density of different crystal types

	density
Product	g/cm³
HMX, Type B, Class 1	1.8806 (0.0005)
HMX, Type B, 10-15 µm	1.8891 (0.0009)
HMX, recrystallized in PC	1.8974 (0.0010)
HMX, recrystallized in γ-Bl	1.9016 (0.0003)

Standard deviation in bracket

To find out whether there are any differences in the content of RDX in Octogen, High performance liquid chromatography (HPLC) was used. As a consequence of the results in Table 3 and 4, the recrystallization process reduces the content of Hexogen in the Octogen crystals, and enhances the density of the crystals. This manifest the improvement of crystal quality compared to conventional HMX.

Table 4: Percentage of RDX in HMX

	ICT	specification	manufactor
Product	%	%	%
HMX, Type B, Class 1	0.67	< 2.0	0.7
HMX, Type B, 10-15 µm	0.22	< 2.0	0.6
HMX, to recrystallize	0.37	< 2.0	1.2
HMX, recrystallized in PC	0.04	–	–
HMX, recrystallized in γ-Bl	0.02	–	–

With an Accelerating Rate Calorimeter (ARC), from Columbia Scientific Industries (CSI), USA, measurements were made to detect differences in the self heating rate of the samples. As it is shown in Figure 4, at constant temperature the recrystallized material has a lower self heating rate compared to conventional HMX.

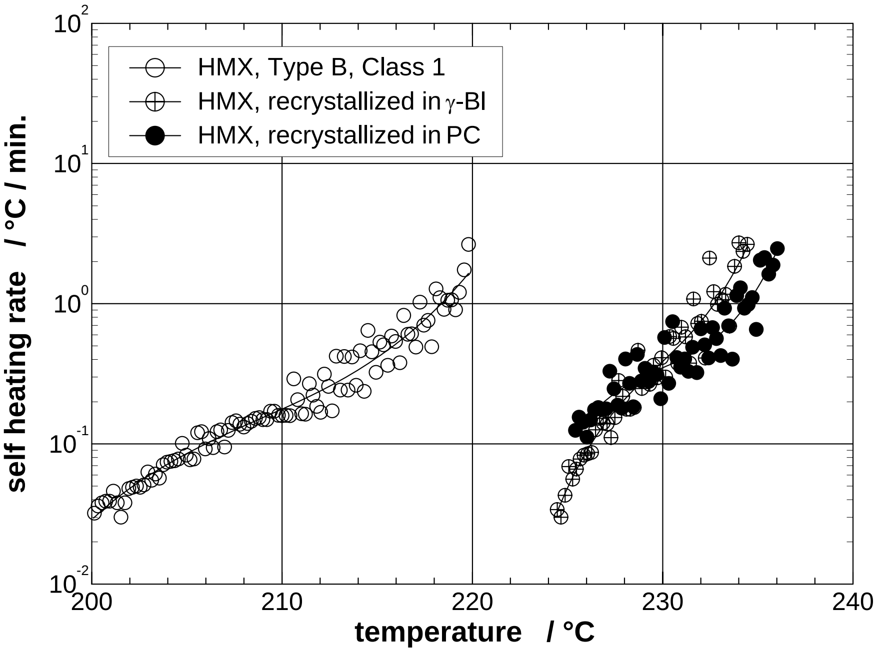


Figure 4: Adiabatic self heating of different HMX types

4 Formulation work

Formulation work was done to find out differences in shock sensitivity. PBXN-109 analogue charges with 16 % HTPB binder (HTPB-IPDI/DOA : 50/50), 20 % Aluminium and 64% HMX were made. After adding all ingredients in the mixer, the viscosity of the castable formulation was measured with a Brookfield viscosimeter. The results are listed in Table 5. The lowest viscosity was obtained with Octogen recrystallized in PC. Possibly the shape of the particle influences the viscosity in a favourable way.

Table 5: viscosity

		viscosity
HXA	Nitramin-Type	Pa•s
195	HMX, Typ II, Class 1 / Class 5	109
201	HMC, recrystallized in γ-Bl	78
202	HMC, recrystallized in PC	69

After curing the samples the density was measured. The highest density was obtained with charges, containing HMX recrystallized in PC.

Table 6: Density of the charges

		density	density
HXA	Nitramine-Type	g/cm³	%
195	HMX, Typ I, Class 1 / Class 5	1.708	98.9
201	HMX, recrystallized in γ-Bl	1.722	99.6
202	HMX, recrystallized in PC	1.725	99.8

5 GAP – Test

With the three formulations mentioned in Table 6, a 50 mm GAP – Test was carried out. Figure 5 shows some details of the test setup.

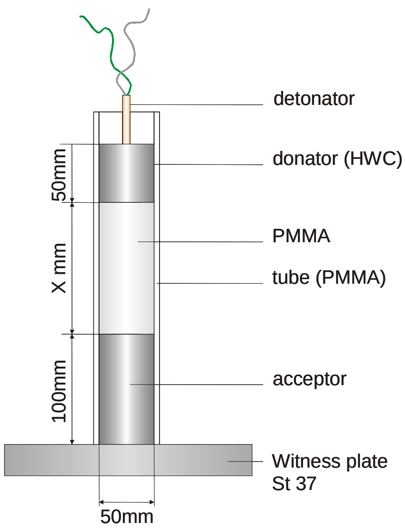


Figure 5: Diagrammatic view of the GAP-Test

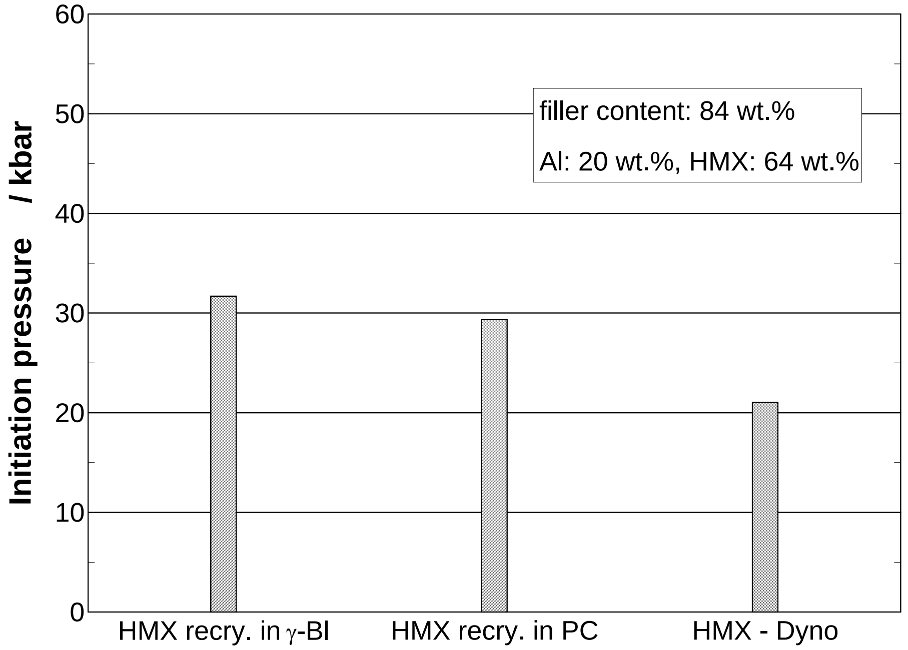


Figure 6: Comparison of recrystallized HMX and conventional HMX (Dyno)

The highest initiation pressure is reached with charges, containing HMX recrystallized in γ-Bl. The initiation pressure of this formulation is more than 10 kbar higher compared to charges containing conventional HMX.

To compare the achieved results with charges containing Hexogen, the Nitramine – Type was changed from Octogen to insensitive Hexogen. Everything else, the binder, aluminium type, mixing and charge diameter was kept constant. With insensitive RDX an initiation pressure of more than 50 kbar was reached.

6 Summary

The described recrystallization process of Octogen in the solvents γ-Butyrolactone and Propylencarbonate, is used to receive a high crystal quality of Octogen. The crystals show reduced content of Hexogen. The density of the recrystallized particles is higher compared to conventional Octogen. The self heating rate of the recrystallized material is more advantageous compared to conventional HMX. Because of the shape of the particles and the particle size distribution, the viscosity of the castable formulation is less compared to conventional Octogen.

In a 50 mm GAP – Test a higher initiation pressure in determined with recrystallized Octogen. HMX recrystallized in the solvents γ-Butyrolactone and Propylencarbonate show insensitive properties.

In total the results display an improvement in crystal quality of HMX. Future work will concentrate on the improvement of the crystal quality of HMX by changing the crystallization process.