NEW NTO WORKSHOP AND ASSOCIATED PRODUCT CHARACTERIZATIONS

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ABSTRACT

NTO was produced for more than 25 years at the EURENCO Sorgues plant. However in 2016, the existing workshop was redesigned and rebuilt, to allow a continuous way of production, increase control on the process safety and improve the final product quality.
This paper will firstly describe the different steps of the NTO manufacturing showing some specific process improvements put in place in the workshop. Then will be presented the results of the characterizations of the different grades of NTO (Class II, III and IV) and the specific quality NTO CF which has a granular size distribution and a high bulk density designed for IM melt cast applications.
All the results obtained during this study allow our product to be qualified by the French MoD.
The new NTO has then been also tested and checked in two IM compositions, one cast PBX B2214B and one for a pressed application P16945. The results obtained confirm that the main characteristics in performance, safety and vulnerability of this two IM products are kept with using the new NTO.

1- INTRODUCTION

3-Nitro-1,2,4-triazol-5-one (NTO) is mainly used by EURENCO's customers in Melt-Cast compositions for Insensitive Munitions (NTO CF), very Insensitive Cast-PBX formulations (B2214B, B2268A) as well as moulding powders for Pressed-PBX formulations (P16945). Their applications include Mortar and Artillery ammunition as well as munitions used from naval platforms such as aircraft carriers (bombs & penetrators).

NTO explosives production traces its roots back to the 1980s when EURENCO (at the time SNPE) patented NTO as an explosive for military use. After more than 25 years of production, it has been decided to redesign and rebuild the existing workshop to allow a continuous way of production, increase control on the process safety, reduce the environmental impacts of this type of production (waste acid, air) and improve the final product quality.

Different grades of NTO are produced in this new workshop, Class II, III and IV for cast-PBX formulations and also a specific quality NTO CF which has a granular size distribution and a high bulk density designed for IM melt-cast applications.

The challenge was to improve the process (robustness & reproducible) and to keep exactly the same qualities of NTO as those produced in the old workshop (already qualified for our customer's applications) without changing the technical specifications.

2- WHERE WE WERE…

Some pictures of the old workshop are gathered in the figure 1. This workshop was built in 1972, and before the NTO production which started in 1987, it was dedicated for PETN manufacturing. No specific instrumentation was automated and the control station and command control wasn't deported. Moreover, it was impossible to reach the required level regarding safety and environmental regulation.

Outside view

Synthesis

Crystallization

Sieving

Figure 1: Old workshop views

3- WHERE WE ARE

EURENCO has commissioned in May 2016 the new workshop, figure 2, dedicated for the production of NTO insensitive explosives as part of its investment strategy in Sorgues (France) to modernize explosive production lines and answer current and future customer's needs. This new workshop is able to produce more than 200T/year.

Even if the production process has been kept (chemical process), each step has been improved, from the raw material supply (NA, TO) to the drying phase and the packaging cell. All these steps are completely instrumented which allow us to a fully masterized production.

Outside view

Command Control room

Figure 2: New workshop

3.1- Production process

The production process includes different steps as shown in figure 3:

- Synthesis step: nitration of TO by nitric acid
- Dilution Step: to eliminate the impurities
- Crystallization: to purify the product & to determine the final quality of NTO
- Filtration using a new technology (banding filter)
- Drying using a new technology (fluidising drying)

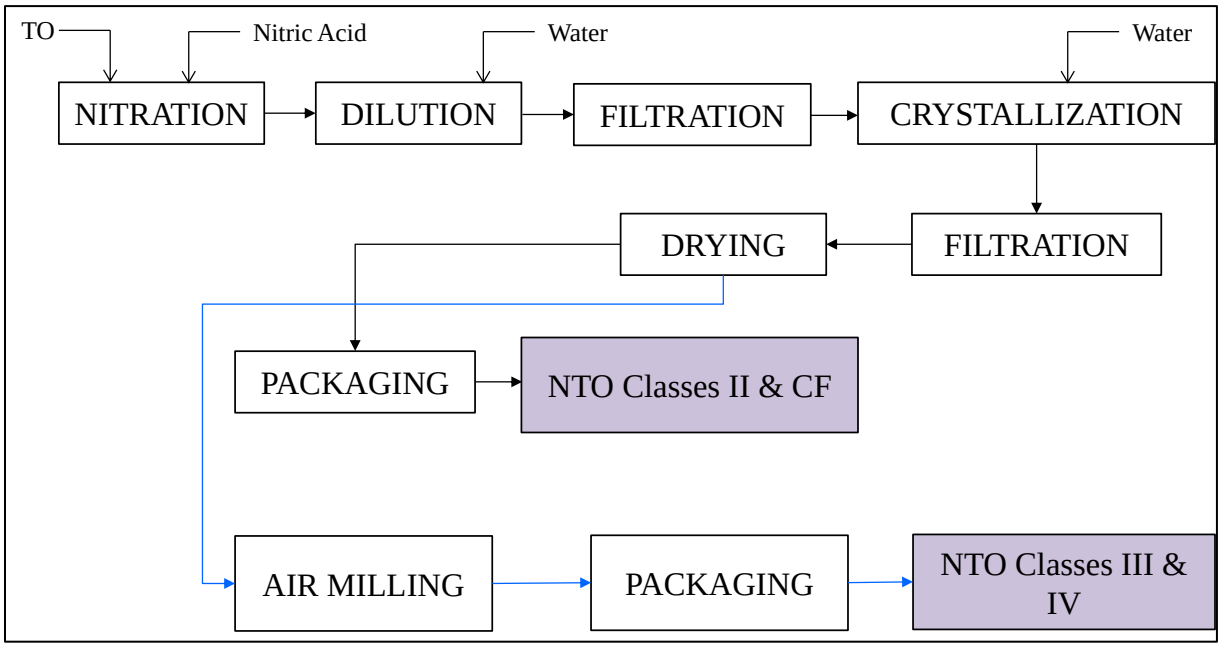


Figure 3: NTO production Flow-sheet

3.2- Synthesis & filtration cell

Figure 4: Synthesis cell

The raw materials are introduced in a continuous way with a controlled rate of flow, the synthesis phase, figure 4, has been optimized by adding instrumentation allowing the control station to be deported in a specific building outside the workshop.

The main improvement consists of the use of a dynamic band filter for the cleaning phase of the NTO (separation between acid and NTO) and which allows the continuous production. A perfect cleaning phase is important to ensure the NTO quality and to master the final crystallized product.

3.3- Crystallization

Before to be crystallized, the ratio NTO/water is exactly measured using a pycnometer. Through this step, we can choose the final quality of the NTO (NTO class II & CF) which depends on the accurate quantity of NTO in the crystallizer and a specific process (temperature, ramp...).

Pycnometers

Crystallizers

Figure 5: Crystallization cell

3.4- Drying & packaging cell,

The main improvement at this step is the use of the fluidizing technology, figure 6, a compress air is used to dry the NTO particles in movement inside the vessel, and this allows a continuous drying and effectively a continuous production.

Before to be automatically packed, detectors are used to ensure the no-presence of foreign matter or metallic particles in the product.

Drying

Packaging

Figure 6: Drying & packaging cell

4- QUALIFICATION PROGRAM

In order to qualify the new plant of NTO, a characterization program was established and validated by the French MoD. It includes a characterization of the granular explosive (NTO Class II, III, IV & CF), Cast-PBX formulations (B2214B) and Pressed-PBX formulations (P16945).

4.1- Granular explosive

The characterization includes:

- Definition File Test (Technical Specifications)
- Safety & vulnerability test
- Others (Microscope, SEM…)

Two levels of characterization were selected: Level 1 corresponding to the products that will undergo a complete characterization and level 2 which corresponds to a limited characterization as shown in the table 1 below. It was selected to place at level 1: NTO class II, IV and CF and Level 2 would apply to Class III.
The results obtained will be compared with the reference (old process), specifications imposed by the definition files and the values from the control charts.

Analysis		NTO Class II	NTO Class III	NTO Class IV	NTO CF
	Humidity	x	x	x	x
	Purity	x	x	x	x
	Acidity (HNO_3)	x	x	x	x
	TO Content	x	x	x	x
	Chlorine content	x	x	x	x
	Volatile matter	x	x	x	x
	Granulation	x	x	x	x
	Bulk density	x	x	x	x
Safety	Friction sensitivity (BAM)	x	x	x	x
	Impact sensitivity	x	x	x	x
	Progressive heating	x	x	x	x
	DSC	x	x	x	x
	Vacuum stability	x		x	x
	Gutter combustion	x		x	x
	Electric spark	x		x	x
Others	Optical microscope	x	x	x	x
	SEM	x		x	x
	Density	x		x	x
	Melting point	x		x	x

Table 1: Characterization program of the Granular Explosive (NTO)

4.2- Cast-PBX formulations

A B2214B composition (NTO, HMX, binder) was produced using the new NTO, the aim is to characterize this composition at t0 & t6month (table 2) in order to prove that there is no difference compared to the reference (B2214B using old NTO).

Analyses	**B2214B**
Bulk density	x
Constituent content	x
Mechanical properties at 20 °C	x
Impact sensitivity (t0 & t6month)	x
Friction sensitivity (t0 & t6month)	x
Hammer 30 Kg	x
Progressive heating	x
Gutter combustion	x
Electric spark	x
Vacuum stability	x
Card Gap test (t0 & t6 month)	x
Critical diameter (t0 & t6month)	x
DSC (t0 & t6month)	x
Friability	x
Velocity of detonation	x
Critical temperature for thermoinitiation	x

Table 2: Characterization program of the B2214B

4.3- Pressed-PBX formulations

A granular composition P16945 (NTO, RDX, binder & graphite) was produced using the new NTO, and characterized as shown in the table below. A comparison will be done with the reference (P16945 using old NTO).

Analyses	P16945
Bulk density	x
Constituent content	x
Volatile matter	x
Impact sensitivity	x
Friction sensitivity	x
Granulation	x
Progressive heating	x
Electric spark (ESD)	x
Vacuum stability	x
Card Gap test	x
Critical diameter	x
DSC	x
Friability	x
Velocity of detonation	x

Table 3: Characterization program of the P16945

5- TECHNICAL RESULTS

5.1. Granular explosive (NTO Class II, III, IV & CF)

In order to demonstrate the robustness of the new production line as well as the conformity of the NTO product, several batch's (> 10) have been analyzed according to definition file (technical specifications). Then, one operation was chosen for the series of tests of the qualification program.

5.1.1. NTO class II

The NTO class II was compared to the reference as shown in table 4. We note that the physicochemical & pyrotechnical results are similar to the reference. The NTO class 2 is compliant to the specifications.

Analyses	NTO Class II (New line)	NTO Class II (reference)	Definition File (Specifications)
Purity (%)	99.2	99.9	≥ 99
Acidity (HNO_3) (%)	0.00	0.03	≤ 0.05
TO Content (%)	0.03	0.13	≤ 0.2
Chlorine content (%)	0.00	0.00	≤ 0.02
Volatile matter (%)	0.02	0.00	≤ 0.1
Bulk density (kg/m^3)	994	814	-
Friction sensitivity (BAM) (N)	5 + at 353 N	4 + at 353 N	-
Impact sensitivity (J)	18	19	-
Progressive heating (°C)	266	265	-
DSC (Onset point °C)	270.5	271	-
Vacuum stability (cm^3/g)	0.38	0.4	-
Gutter combustion	No propagation	No propagation	-

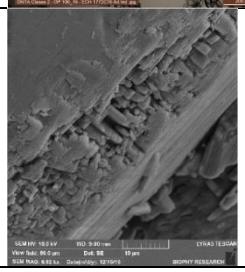
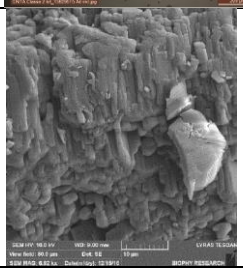
Electric spark (mJ)	> 792	> 792	-
Optical microscope			-
SEM			-
Melting point (°C)	Decomposition at 250°C	Decomposition at 250°C	-
Granulation (% retained on)			
> 0.800 mm	2	0	≤ 4
> 0.500 mm	18	5	≤ 20
> 0.315 mm	53	45	30/60
> 0.200 mm	88	88	≥ 85

Table 4: Characterization results of NTO Class II

5.1.2. NTO class III

The NTO class III was compared to the reference (table 5). We note that the physicochemical & pyrotechnical results are nearly identical. The NTO class III is compliant to the specifications.

Analyses	NTO Class III (New line)	NTO Class III (Reference)	Definition File (Specifications)
Humidity (%)	0.01	0.01	-
Purity (%)	99.1	99.3	≥ 99
Acidity (HNO_3) (%)	0.03	0.03	≤ 0.05
TO Content (%)	0.03	0.07	≤ 0.2
Chlorine content (%)	0.00	0.00	≤ 0.02
Volatile matter (%)	0.02	0.02	≤ 0.1
Laser granulometry (µm)	55.2	56.5	50 ± 10
Bulk density (Kg/m^3)	560	510	-
Friction sensitivity (BAM) (N)	9 + at 353 N	8 + at 353 N	-
Impact sensitivity (J)	9.1	11	-
Progressive heating (°C)	267	266	-
DSC (onset point °C)	270.4	270.5	-

Table 5: Characterization results of NTO Class III

5.1.3. NTO class IV

Table 6 represents a comparison between the NTO class IV and the reference. We note that the physicochemical & pyrotechnical results are similar. The NTO class IV is compliant to the specifications.

Analyses	NTO Class IV (New line)	NTO Class IV (reference)	Definition File (Specifications)
Humidity (%)	0.01	0.02	-
Purity (%)	99.7	99.3	≥ 99
Acidity (HNO_3) (%)	0.01	0.01	≤ 0.05
TO Content (%)	0.02	0.04	≤ 0.2
Chlorine content (%)	0.01	0.00	≤ 0.02
Volatile matter (%)	0.01	0.01-0.02	≤ 0.1
Laser Granulometry (µm)	12.5	11.7	12 ± 3
Friction sensitivity (BAM) (N)	3 + at 353 N	0 + at 353 N	-
Impact sensitivity (J)	13	8.1	-
Progressive heating (°C)	269	269	-
DSC (Onset point °C)	272.4	271.6	-
Vacuum stability (cm^3/g)	0.16	0.26	-
Gutter combustion	No propagation	No propagation	
Electric spark (mJ)	> 792	> 792	-

Table 6: Characterization results of NTO Class IV

5.1.4. NTO CF

The NTO CF was compared to the reference as shown in table 7. We note that the physicochemical & pyrotechnical results are nearly identical. The NTO CF is compliant to the specifications.

Analyses	NTO CF (New line)	NTO CF (reference)	Definition File (Specifications)
Humidity (%)	0.03	0.02	-
Purity (%)	100	99.7	≥ 99
Acidity (HNO_3) (%)	0	0	≤ 0.05
TO Content (%)	0	0.001	≤ 0.2
Chlorine content (%)	0	0	≤ 0.02
Volatile matter (%)	0.01	0	≤ 0.1
Bulk density (Kg/m^3)	927	939	> 900
Friction sensitivity (BAM) (N)	0 + at 353 N	0 + at 353 N	-
Impact sensitivity (J)	16	13	-
Progressive heating (°C)	266	265	-
DSC (Onset point °C)	272.4	271.6	-
Vacuum stability (cm^3/g)	0.12	0.16	-
Gutter combustion	No propagation	No propagation	
Electric spark (mJ)	> 792	> 792	-

Table 7: Characterization results of NTO CF

5.2. Cast-PBX formulations

After analyzing the 3 grades of NTO (II, III & IV) a B2214B composition was produced and analyzed. The results are represented in table 8. We note that the B2214B is compliant to the specifications and there is no-difference between new B2214B (New NTO) and old B2214B (old NTO).

Analyses	B2214B (NTO New line)	Reference	Specifications
density (kg/m^3)	1630	1643	1605-1645
Constituent content (NTO, HMX, binder) (%)	72.2/12.5/15.3	72.2/11.6/16.2	
Mechanical properties at 20 °C (Stress Mpa)	0.89	0.75	0.7±0.3
Impact sensitivity (J)	31	27	-
Friction sensitivity (N)	15 + at 353N	4 + at 353 N	-
Progressive heating (°C)	234	232	233
Gutter combustion	No propagation	No propagation	-
Electric spark (mJ)	> 792	> 792	
Card Gap test (phi 75 mm)	35 mm	35mm	-
Velocity of detonation (m/s)	7414	7482	-

Table 8: Characterization results of B2214B

5.3. Pressed-PBX formulations

The NTO class II was produced and analyzed for the production of the P16945 (NTO, RDX, Binder & Graphite). The P16945 was analyzed as shown in table 9. We note that the P16945 is compliant to the specifications and there is no-difference between new P16945 (New NTO) and old P16945 (old NTO).

Analyses	P16945 (NTO New line)	Reference	Specifications
Bulk density (kg/m^3)	693	721	-
Constituent content (NTO, RDX, Binder & graphite) (%)	75.2/19.3/5/0.5	75.3/19.6/4.5/0.6	75±3/20±2/5±0.5/0.5±0.3
Impact sensitivity (J)	9.7	10	-
Friction sensitivity (N)	3 + at 353N	2+ at 353N	-
Granulation (% retained on)			
> 0.800 mm	43	49	-
> 0.500 mm	63	73	> 50
> 0.315 mm	81	88	> 80
> 0.040 mm	100	100	> 98
Progressive heating (°C)	210	209	-
Electric spark (mJ)	> 792	> 792	-
Vacuum stability (cm^3/g)	0.14	0.31	-
Card Gap test (phi 40 mm)	200	190	-
DSC (Onset point °C)	202	205	-
Velocity of detonation	In progress	7893	-

Table 9: Characterization results of P16945

6- CONCLUSION

After more than 25 years, the workshop used for the NTO production has been designed and rebuilt, the new one is now able to more than 200 T per year. Different qualities of NTO are produced, class II, III, IV & CF for Cast-PBX IM applications but also a specific quality designed for IM Melt-Cast formulations.

The challenge was to demonstrate that with this new production line and with this optimized process, the quality of our NTO was the same of that produced before and already qualified for our customers.

The qualification program was established and validated by the French MoD. The results confirm that all grades of NTO (NTO class II, III, IV & CF) are compliant to the specifications and identical to the references. The new NTO has then been also tested and checked in two IM compositions, one plastic bonded explosive B2214B and one for a pressed application P16945.

The line is fully qualified and commissioned.