



Ageing of I-RDX® and of compositions based on I-RDX®

#### PRESENTATION OUTLINE

STATUS OF I-RDX®

AGEING OF COMPOSITIONS BASED ON I-RDX® or IH-RDX

**RDX AGEING** 

FURTHER ANALYTICAL INVESTIGATIONS

**CONCLUDING REMARKS** 



## Status of I-RDX®

- A special RDX with a lower level of shock sensitivity than regular RDX, obtained by a specific and unique recrystallization process patented by EURENCO
- This quality of RDX, has been industrially produced at Sorgues plant for more than 40 years
- Up to now, this low shock sensitivity of I-RDX<sup>®</sup> is mainly acknowledged when I-RDX<sup>®</sup> is used in Cast PBX



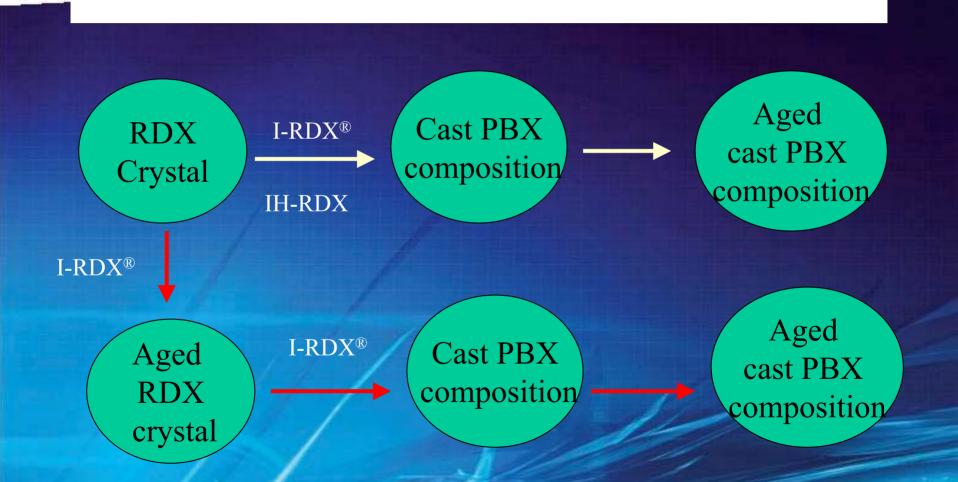
# Status of I-RDX®

# The insensitive character of I-RDX® is highly reproducible. This is proven by LSGT test results such as:

Name	composition	sampling	Barrier	Standard
			pressure	deviation
			Mean (kbars)	(kbars)
HBU88 A	$I-RDX^{\mathbb{R}} = 88 \%$	6 different lots of I-RDX <sup>®</sup>	50	3
	HTPB = 12 %	tested during 1987-95		
HBU 88 B	$I-RDX^{\text{@}} = 88 \%$	3 different lots tested	50	3
(B 2263 A)	HTPB = 12 %	during 2001-2004		
B2213	$I-RDX^{\mathbb{R}} = 64\%$	5 different lots tested	56	3
- // -	A1 = 20 %	during 1997-98		
	HTPB = 16 %			
<b>PBXN-109</b>	$I-RDX^{\mathbb{R}} = 64\%$	12 different lots of I-	54	2
14-1-1	Al = 20 %	RDX <sup>®</sup> tested during 2002-		
	HTPB = 16 %	03		



# Ageing of compositions





### Ageing of compositions

ageing of compositions prepared with « fresh » I-RDX®

Composition	RDX	Cast PBX	LSGT results (kbars)	
	nature	ageing	То	Aged
		conditions		
HBU 88 A	I-RDX <sup>®</sup>	13 years room	50	51
		temperature		
B 2263 A	I-RDX <sup>®</sup>	3 months	51	49
		60°C		
B 2213	I-RDX <sup>®</sup>	6 months	56	57
		60°C		

No significant evolution



### Ageing of compositions

ageing of compositions prepared with « fresh » IH-RDX

Composition	RDX nature	Ageing conditions	LSGT results (kbars)	
			То	Aged
B 2263 A	IH-RDX (EURENCO	3 months 60°C	44	38.5
(HBU 88 B)	recrystallization of Bachmann crystallized	9 months 60°C		33
	RDX)	9 months room temperature		37

Indication of reversion of shock sensitivity



#### Ageing of compositions

#### compositions prepared with aged I-RDX<sup>®</sup>

	I-RDX <sup>®</sup> ageing conditions	LSGT result after ageing (kbars)	LSGT reference value (kbars)
	I-RDX <sup>®</sup> aged dry 8 months at room temperature	54	$m = 54$ $\sigma = 2$
	I-RDX <sup>®</sup> aged 3 months in Isopropyl alcohol/water at room temperature	52	$m = 54$ $\sigma = 2$
B 2263A	I-RDX <sup>®</sup> / DOA aged 18 months at room temperature	47 *	$m = 50$ $\sigma = 3$

\*the initial (To) LSGT value with I-RD DOA is 50 kbars

No significant evolution



### Ageing of compositions

- ageing of compositions prepared with aged  $I\text{-}RDX^{\circledR}$ 

Composition	I-RDX <sup>®</sup> ageing conditions	Cast PBX ageing conditions	LSGT results (kbars)	
		Conditions		
			То	Aged
PBXN 109	I-RDX <sup>®</sup> aged 3	3 months	52	52
7/13 13 13 13	months in Isopropyl	60°C		
	alcohol/water at RT	Market 1		
B 2263 A	I-RDX <sup>®</sup> / DOA aged	2 months	47	50
	18 months at room	60°C		
-19	temperature			

No significant evolution



### Ageing of compositions

#### Conclusions

- With I-RDX<sup>@</sup> there is no evidence of any modification of shock sensitivity after ageing
- With IH-RDX (EURENCO recrystallization of Bachmann RDX), reversion of shock sensitivity is observed.



#### Ageing of compositions

- RDX may be obtained by two different processes:
  - Woolwich or nitric acid process (type I: almost no HMX)
  - Bachmann or aceto-nitric process (type II: HMX content usually 4-17 %)
- What is causing the initial insensitivity?
- What is the ageing affecting?



#### RDX Ageing

- Base of the study:
  - Five different qualities of RDX class 1 MIL differing by:
    - Their origin (or process of production)
    - Their HMX content
    - Their crystallization process (EURENCO or not)



#### RDX Ageing

Ageing conditions

15 % Water/Isopropyl alcohol 2/1

Carnauba wax (RDX/ Carnauba 85/15)

DOA 5%

-Room temperature ageing: 6 months

-Accelerated ageing (60°C): 30 days



#### RDX Ageing

#### Products and characterization methods

Reference	Quality	Particle	HMX
		size	content
		(µm)	(%)
I-RDX®	I-RDX <sup>®</sup>	211	0,1
A	I-RDX <sup>®</sup> + 10% HMX	206	9,0
В	Crystallized Bachmann	143	11,8
	RDX		
C	B recrystallized by	297	11,7
	EURENCO		
D	EURENCO	228	5,4
	recrystallized crude		
	Bachmann RDX		

Infra-red Spectroscopy

**HPLC** 

DSC

Particle size

Morphology



#### RDX Ageing

Conclusions

Based on the classical techniques used in this study to characterize the crystals, whatever the ageing conditions and the pature of RDX

No significant evolution

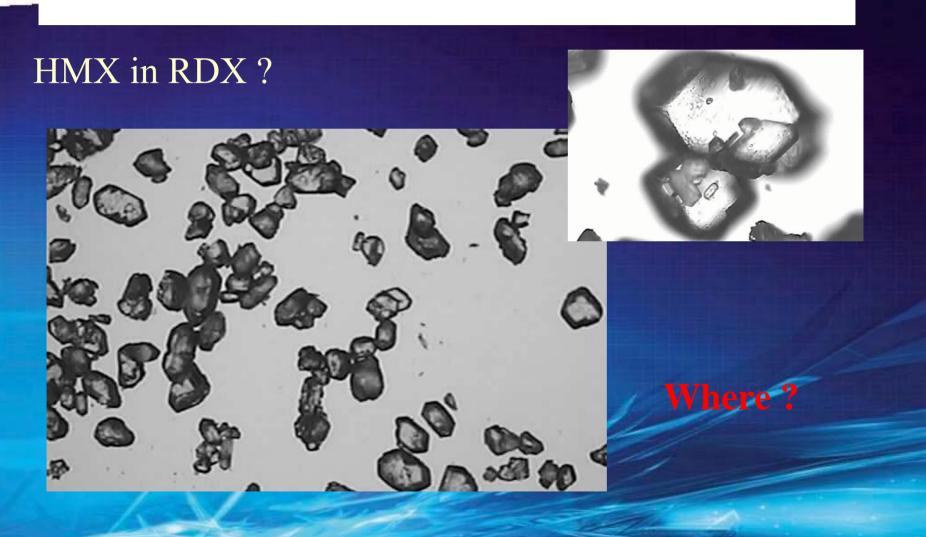


#### Further analytical investigations

- Ageing must be affecting the RDX itself
  - What may explain the difference after ageing in between I-RDX® and IH-RDX?
    - Is this difference due to the presence of HMX in RDX?
    - Do we have changes of RDX crystal in the cast PBX composition after ageing?



## Further analytical investigations





#### Further analytical investigations

• Where is the HMX?

- Based on solubility data, and HMX distribution in RDX (mostly in the fines)
  - In the Woolwich process RDX and HMX are totally separated
  - In the Bachmann process, RDX and HMX are mostly separated but may be not completely (partial co-crystallization?)





#### Further analytical investigations

• How to determine HMX in RDX?

- Flottation technique inadequate
  - 0.1 % HMX  $\rightarrow$  density increase of 0.0001
- Infra-red, DSC and HPLC for example
  - Adequate to determine the presence of HMX
  - Inadequate to say if HMX is in or out of RDX crystal

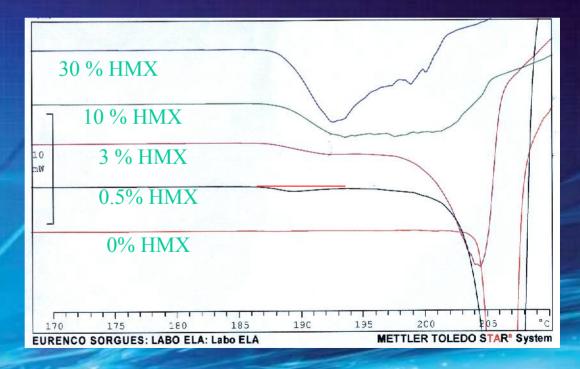


### Further analytical investigations

• HMX in RDX?

- By DSC, modification of the endotherm associated

with melting





# Further analytical investigations

- Changes of RDX crystal in cast PBX after ageing
  - NQR measurements on cast PBX compositions

Composition	RDX nature	Cast PBX Ageing conditions	NQR W ½ (Hz)	LSGT (kbars)
PBX N 109	I-RDX®	No	<b>157</b> (145-179)	54
		4 years RT	164	100
B 2263 A	I-RDX®	No	154	50
	1//	1 year RT*	207	50
1	IH-RDX	9 months RT	333	37

\*Aged (18 months) I-RDX®/DOA



# Concluding remarks

- EURENCO produce from RDX obtained by Woolwich process a unique low shock sensitivity RDX called I-RDX®
- For this I-RDX®, ageing of the raw material itself and ageing of compositions prepared with fresh or aged I-RDX® do not alter the low shock sensitivity of the cast PBX compositions



# Concluding remarks

- For IH-RDX (obtained by EURENCO recrystallisation of RDX from Bachmann process), ageing may show alteration of low shock sensitivity
- Further work is still needed to understand this evolution with time