Reduced Sensitivity RDX Round Robin Program

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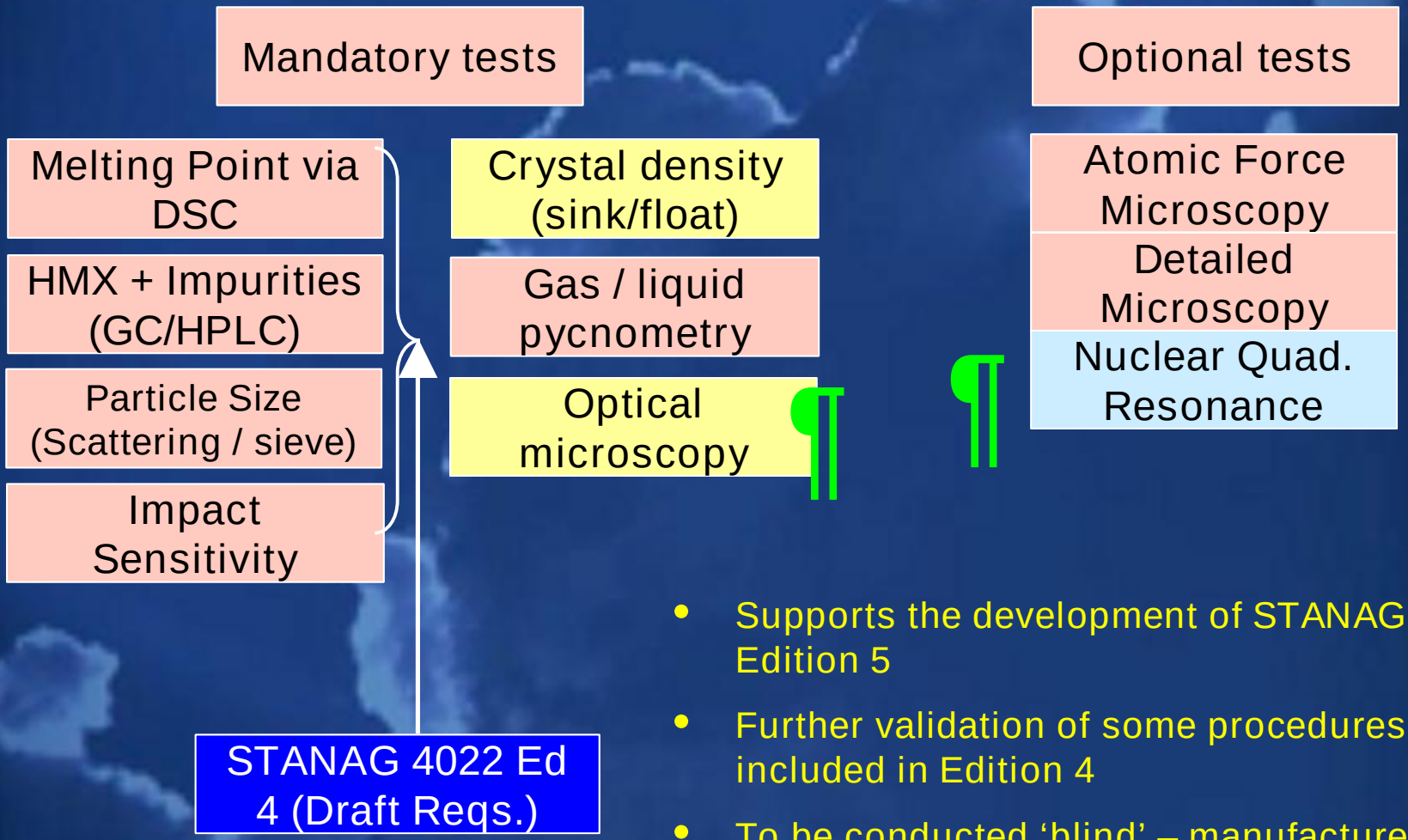
NDIA IMEMTS
25 – 28 April 2006
Bristol, UK

R^4 Program - Participating Laboratories

- Australia – DSTO
- Canada – DRDC/Valcartier
- France
  - ETBS
  - ISL
- Germany
  - WIWEB
  - ICT
  - WTD 91
- Italy – Mariperman
- Netherlands – TNO
- Switzerland - Armasuisse
- UK – DSTL et al.
- US
  - AFRL/MNME
  - US Army TACOM ARDEC
  - US Army AMRDEC
  - NSWC/Indian Head Division
  - NAVAIR/Weapons Division

Analytical tests – R^4 Program



- Supports the development of STANAG 4022 Edition 5
- Further validation of some procedures included in Edition 4
- To be conducted ‘blind’ – manufacturer of individual samples not known to testing laboratory

R^4 Materials

All samples will be commercially available RDX meeting US MIL-DTL-398D Class 1 granulation requirement (same requirements specified in STANAG 4022 Ed 3 Draft)

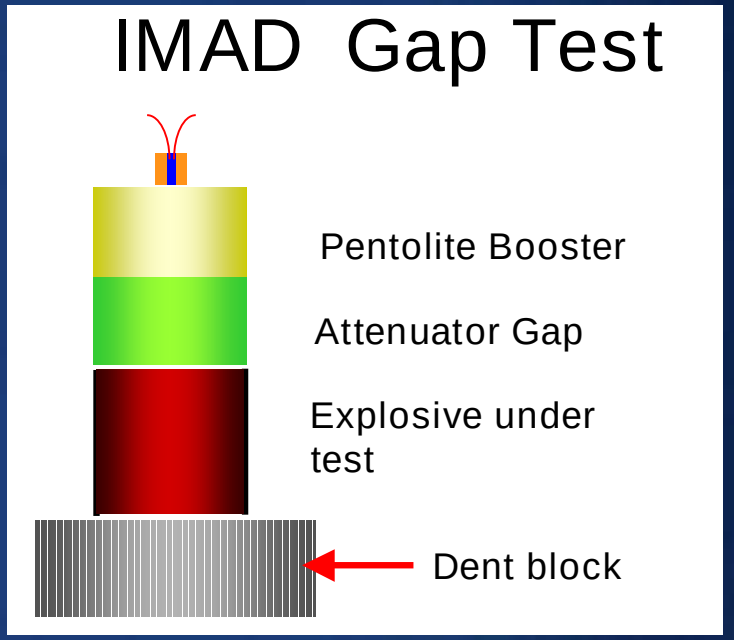
Source	Type	Process		HMX Content		Quality	
		Bachman	Woolwich	> 5%	< 0.5%	RS-RDX	non-RS-RDX
OSI/Holston	Type II	X		X			X
Dyno	Type II	X		X			X
	RS-RDX	X			X	X	
ADI	Grade A		X		X	X	
SME	IRDX		X		X	X	
	MI-RDX		X		X		X
RO/Bridgwater	Type I		X		X	?	

R^4 Program – Shock Sensitivity

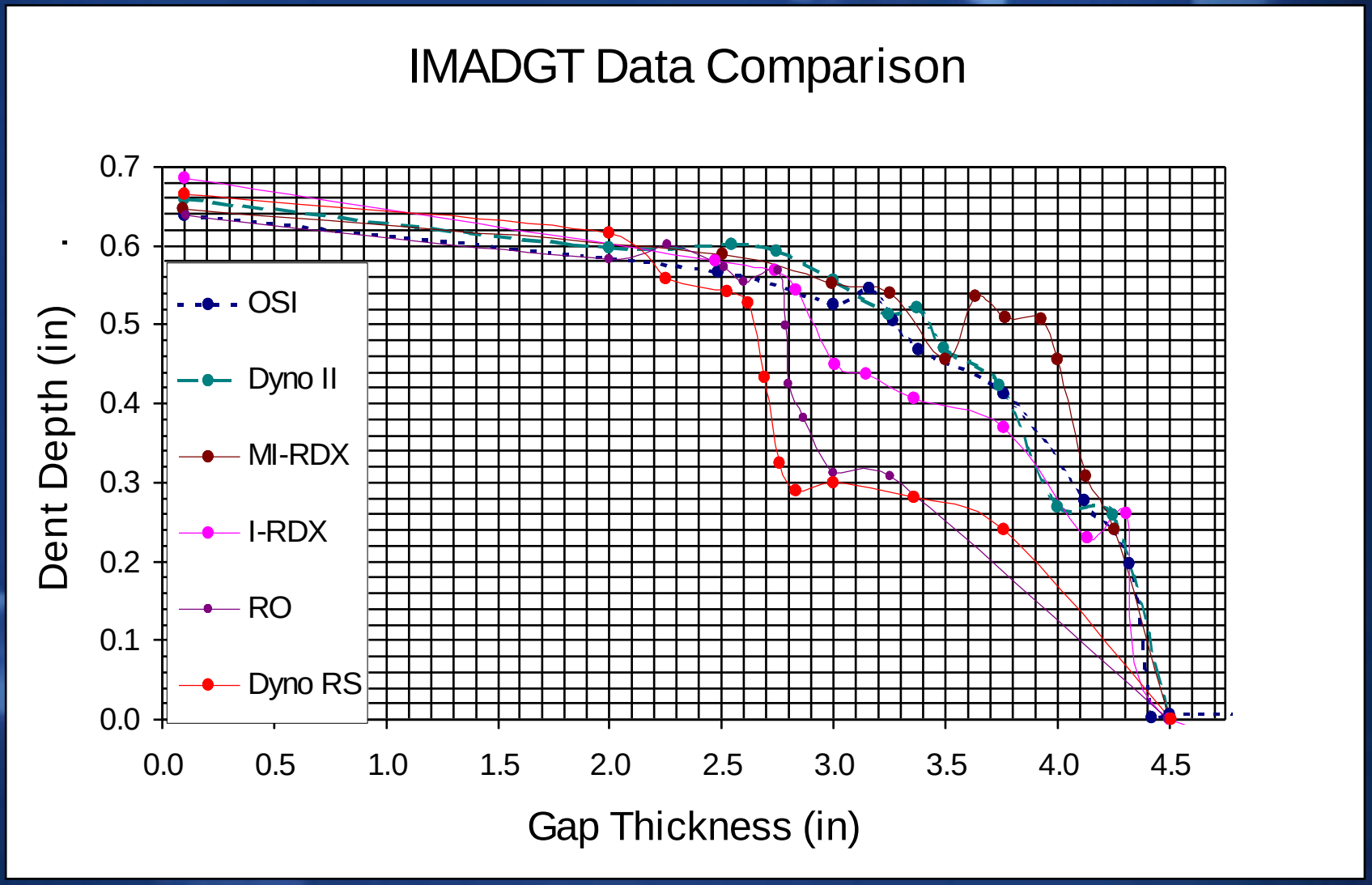
- Companion program to analytical tests performed on seven lots of RDX
- IMAD Gap Test used
- Performed only at NSWCIHDIV
- Currently a set of LSGT series are being fired to link the IMAD GT results to literature data.

R^4 Shock Sensitivity Study

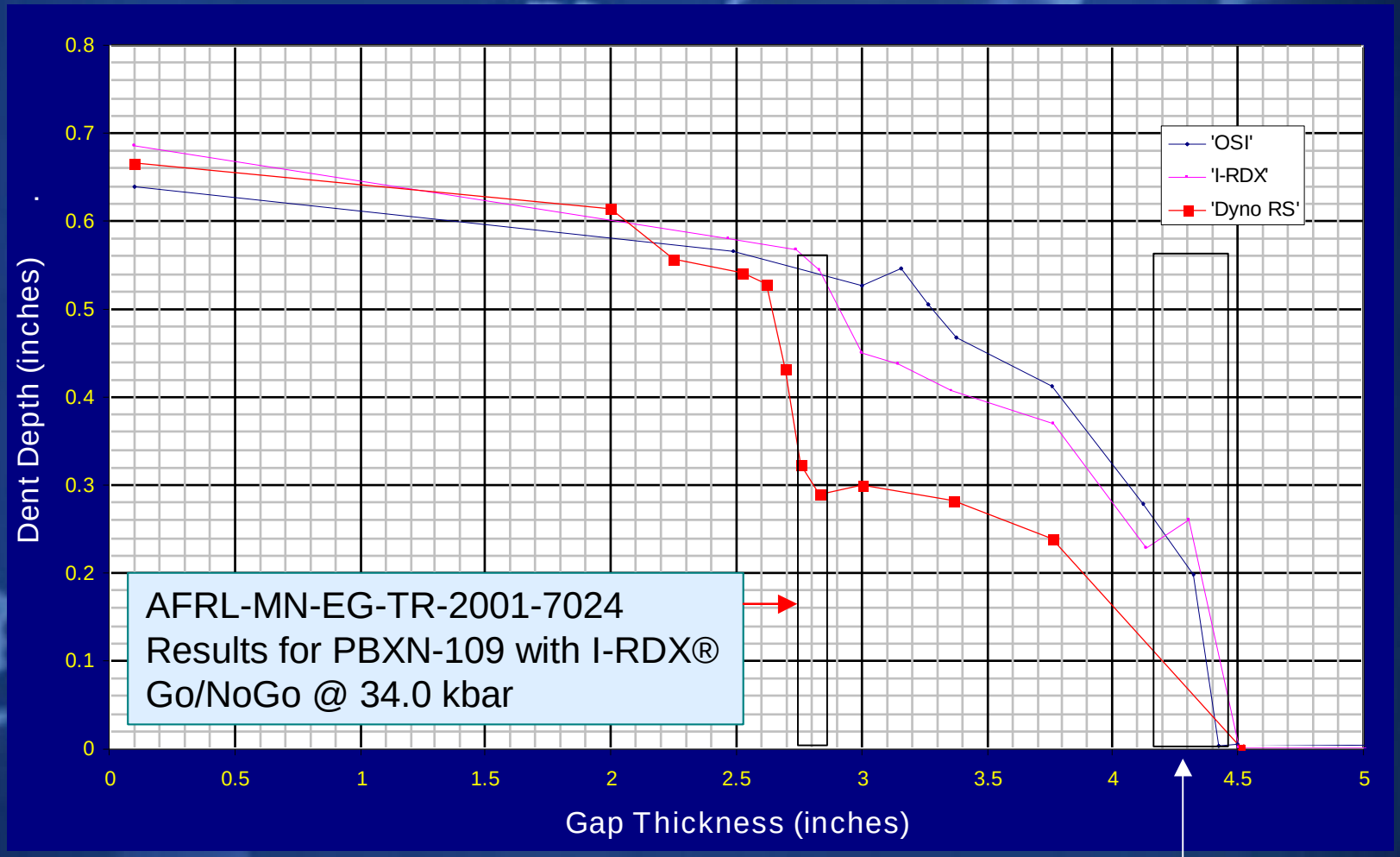
- Necessary to link crystal properties with observed sensitivity of a formulation
- Formulation:PBXN-109 (RDX / Al / HTPB-based binder)
  - Monomodal (Class 1) RDX used in all formulations
- IMAD Gap Test
  - Same booster system as Expanded Large Scale Gap Test (ELSGT)
  - Same test charge diameter as ELSGT, but shorter length
  - Dent block in place of witness plate
  - 12 shots fired in each series



Overall Gap Test Results



IMADGT Results



Observations on IMADGT Results

- Sensitivities can be grouped in three categories
  - High sensitivity: Type II materials (OSI and Dyno), MI-RDX
  - Intermediate sensitivity: I-RDX
  - Low sensitivity: Dyno RS-RDX and RO
- Point at which sharp change in dent depth occurs corresponds approximately to observed values from ELSGT results.
- Unexpected plateau for lower sensitivity variants was observed.
  - Complicates identification of pressure comparable to critical pressure in LSGT or similar tests.
  - May suggest two mechanisms contributing to the initiation of PBXN-109.
- 12 additional tubes of each of the batches used in the IMADGT series are available for further testing.
- LSGT tests will be performed on all seven varieties of RDX; some series already completed.

Analytical tests – R^4 Program – NQR

Background

- NQR is a technique for determining the relative defect densities of explosives compounds and formulations.
- The line width of the NQR resonance bands are directly proportional to the number of disordered regions within the RDX crystal lattice, including dislocations and defects.

- Part of Optional section of R4
- General procedure and recommendations included in "Testing Methods for RS-RDX Round Robin (R4) Program"
- R4 specifies five trials for each RDX analysis

NSWC-IHD | DSTO

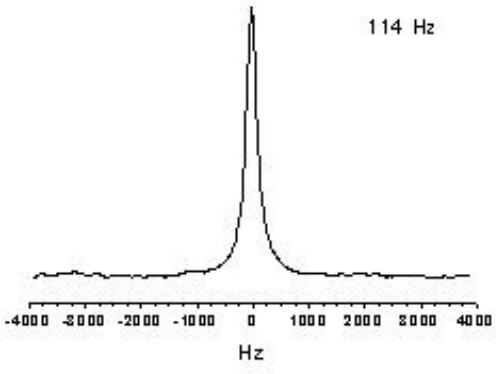
NQR – Comparison of Methods

- NSWC-IHD used a custom built NQR pulsed spectrometer that was designed and constructed at the Naval Research Laboratory.
- DSTO used a spectrometer employing a TECMAG “Apollo” console for pulse generation and data collection, a power amplifier (Model A150), a preamplifier Miteq (Model AU-2A-0150-BNC) and a home-made probe.
- The software used to manipulate the raw data and calculate the line width is unique to each instrument.
- NSWC used a sample size of approximately 10 grams. DSTO used a sample size of approximately 100 grams.
- ISL reported at R4 Workshop on Monday, 24 April, that they have built a spectrometer in collaboration with the University of Nancy. That instrument requires only 3 g of sample.

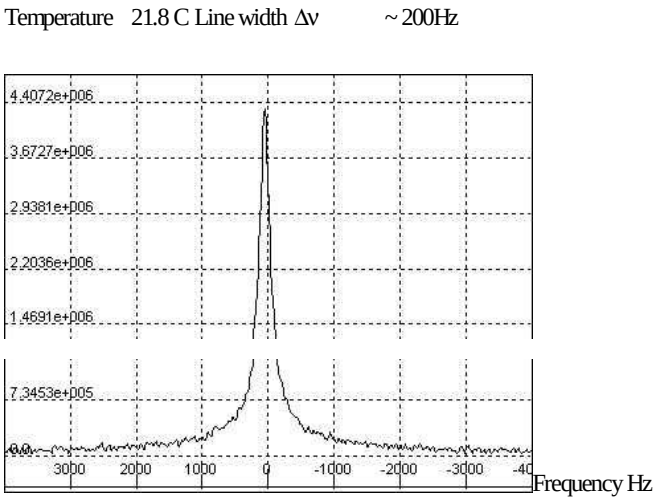
NQR – Results

- Example of Reported Spectra

NQR Spectra of BAE Systems Royal Ordnance RDX



NSWC IHD



DSTO

Dr. S.M. Caulder
NSWCIHDIV

Dr. T. N. Rudakov
QR Sciences

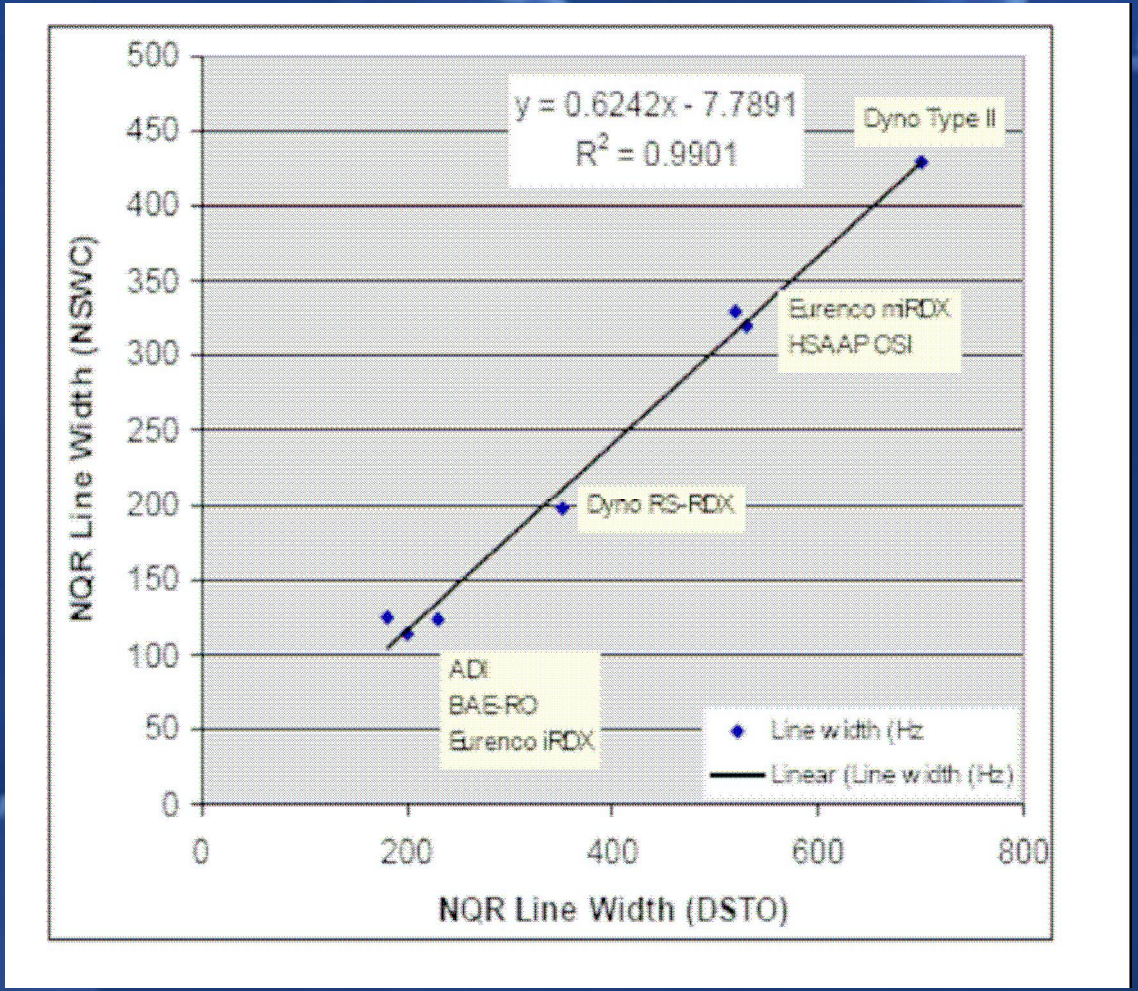
NQR – Results

- Reported Results

Sample	DSTO		NSWCIHD	
	Line width (Hz)	Std Deviation (%)	Line width (Hz)	Std Deviation (%)
ADI Grade A RDX	180	6.1	125	
BAE Royal Ordnance RDX	200	7.0	114	
Eurenco IRDX	230	3.3	123	10
DYNO RS-RDX	350	4.6	197	
BAE OSI	530	3.0	320	
Eurenco MIRDX	520	1.9	329	
DYNO Type II RDX	700	3.7	429	

NQR – Results

- Correlation of Results



Optical Microscopy

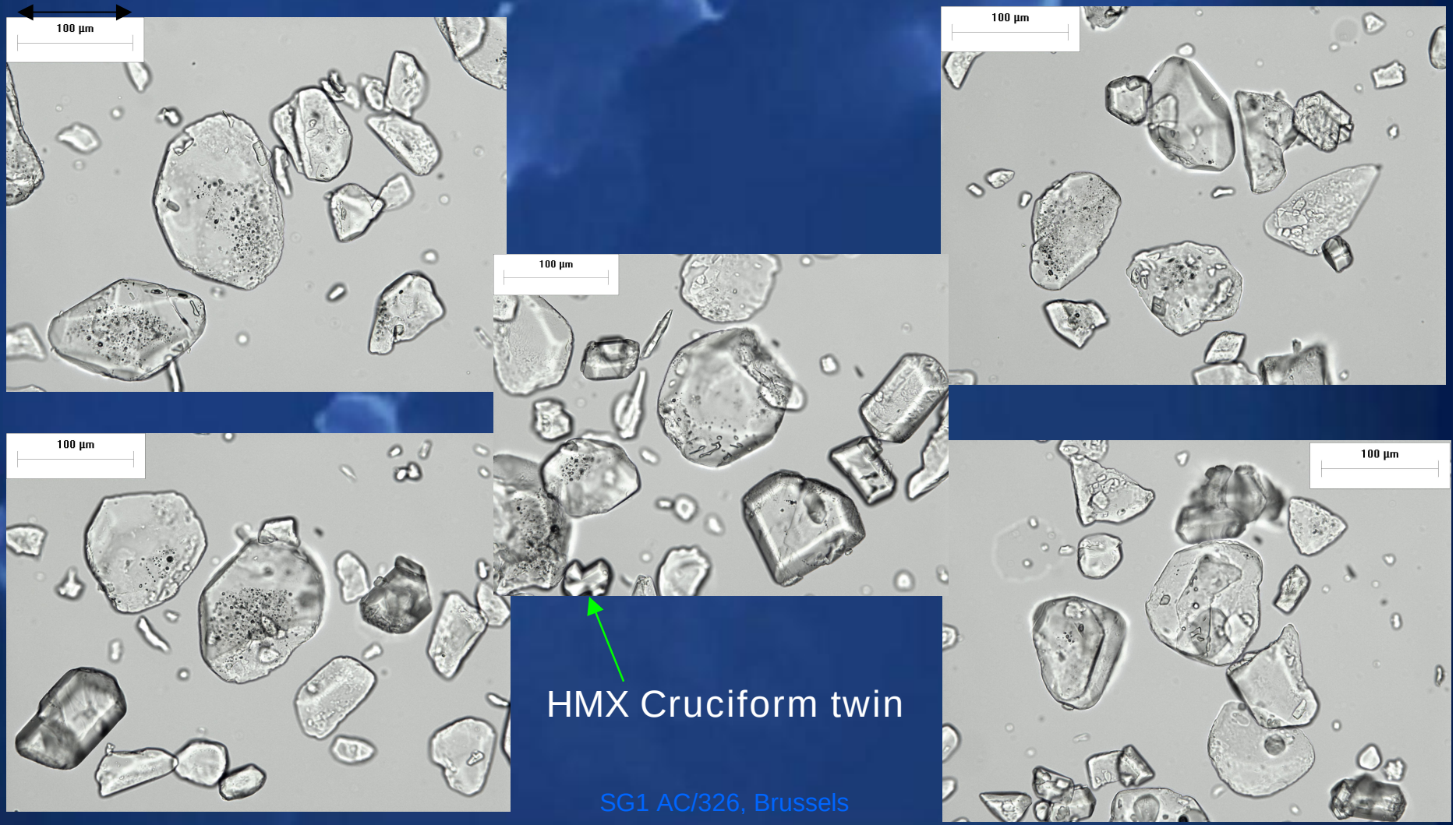
- Part of Mandatory section of R^4
- Method defined in R^4 method manual distributed with the samples
  - Calls for a specific number of photographs to be recorded at different magnifications.
  - Calls for analysis in fluids with
    - Contrasting refractive index
    - Matching refractive
- Requested information
  - Digitized photomicrographs
  - Count of internal defects

Participants	
Contrasting	Matching
AFRL	AFRL
ARDEC	ARDEC
Armasuisse	Armasuisse
DRDC	DRDC
DSTO	DSTO
ICT	ICT
NSWCIHD	NSWCIHD
WTD-91	TNO
	WTD-91

Selected Images

Photomicrographs from DRDC – contrasting refractive index RDX from OSI

100 µm

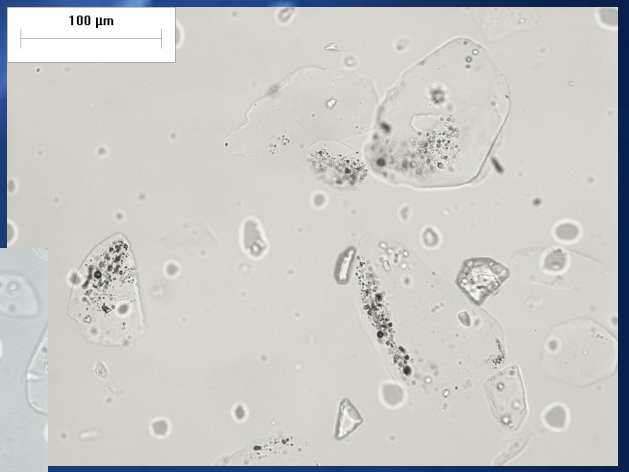


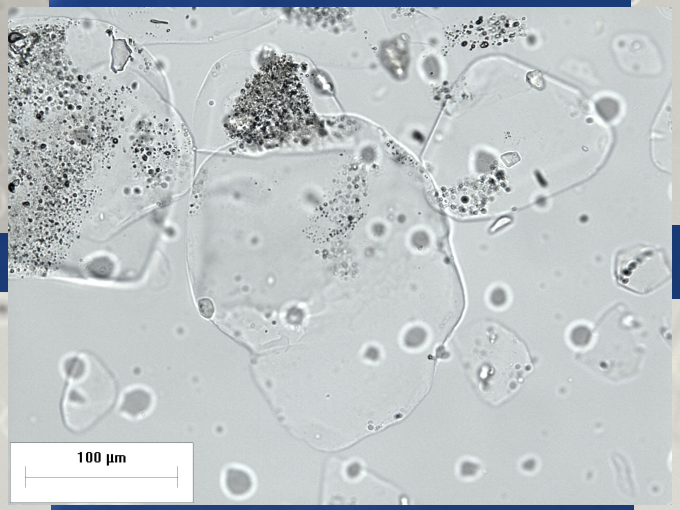
SG1 AC/326, Brussels

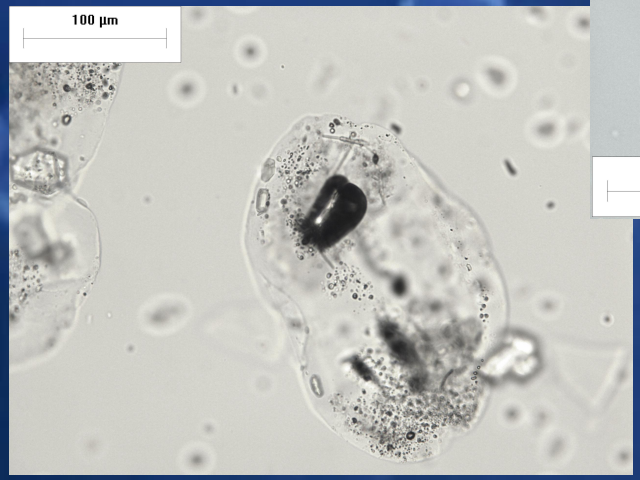
Selected Images

Photomicrographs from DRDC – matching refractive index RDX from OSI

100 µm



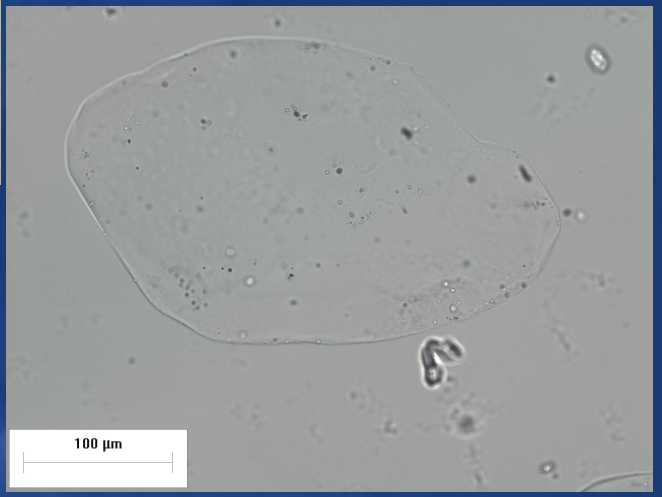


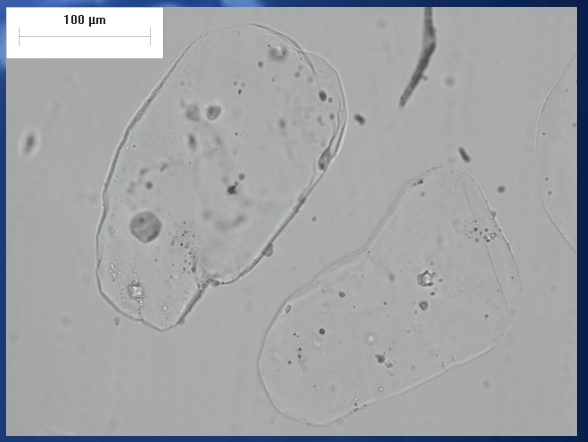


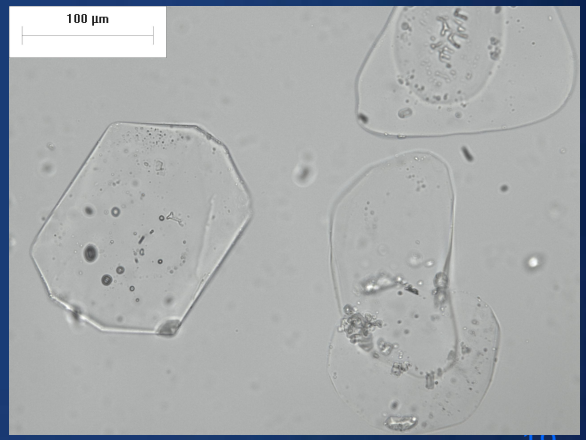
Selected Images

Photomicrographs from DRDC – matching refractive index RS-RDX from Dyno

100 µm

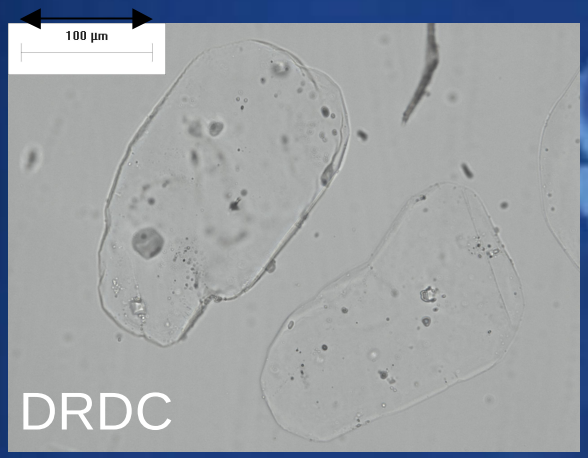


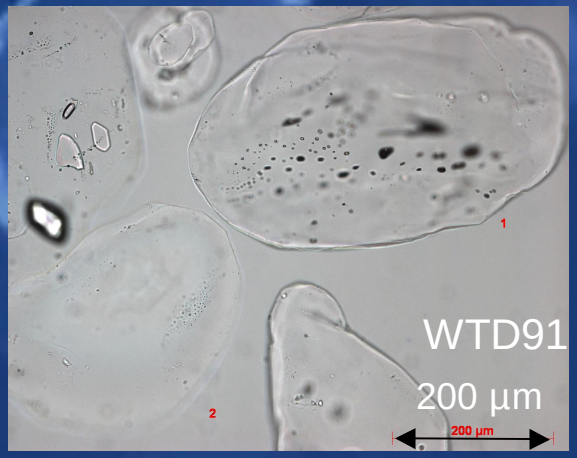


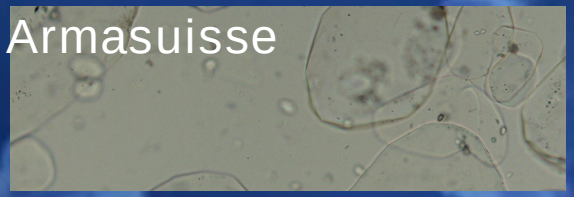


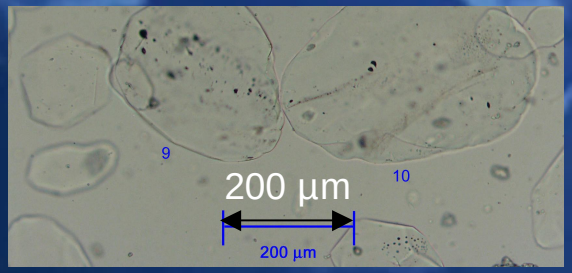
Same RDX, Different Labs

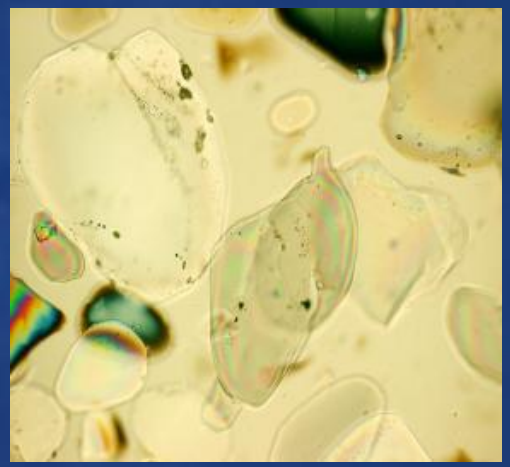
Photomicrographs, matching refractive index RS-RDX from Dyno

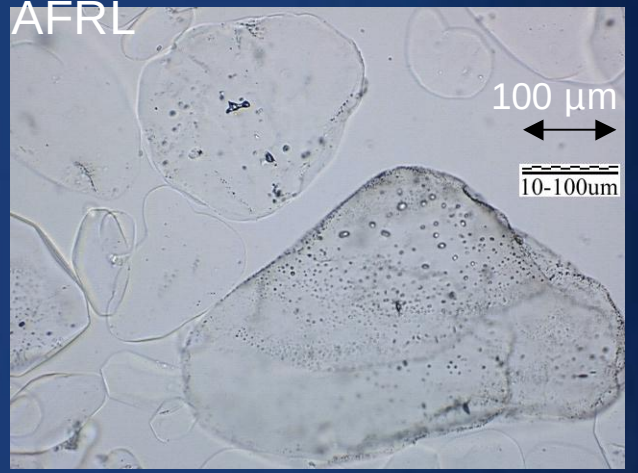












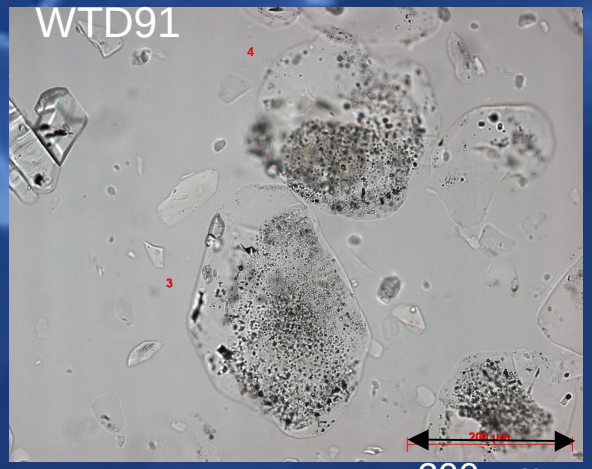
Same RDX, Different Labs

Photomicrographs, matching refractive index

OSI RDX

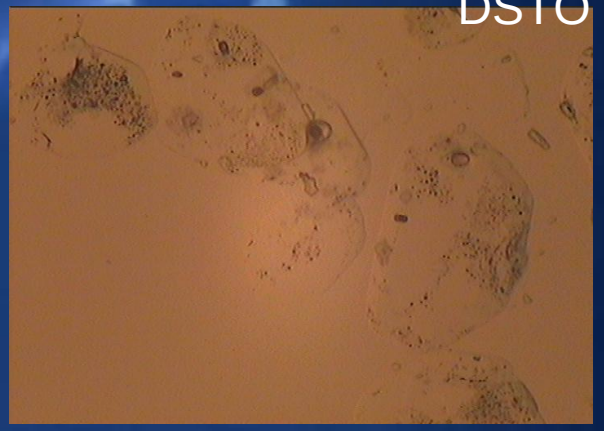
100 µm

WTD91

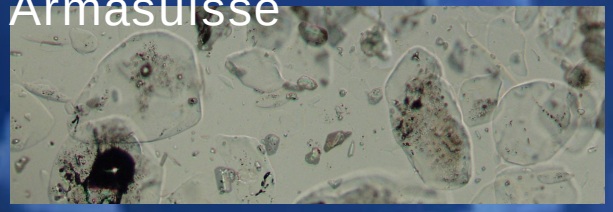


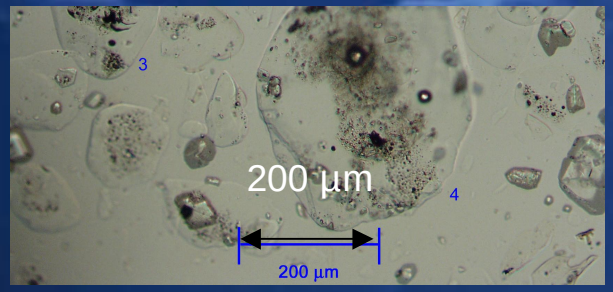
200 µm

DSTO

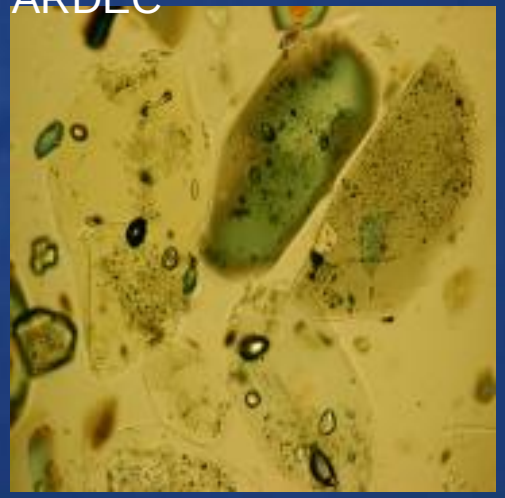


Armasuisse

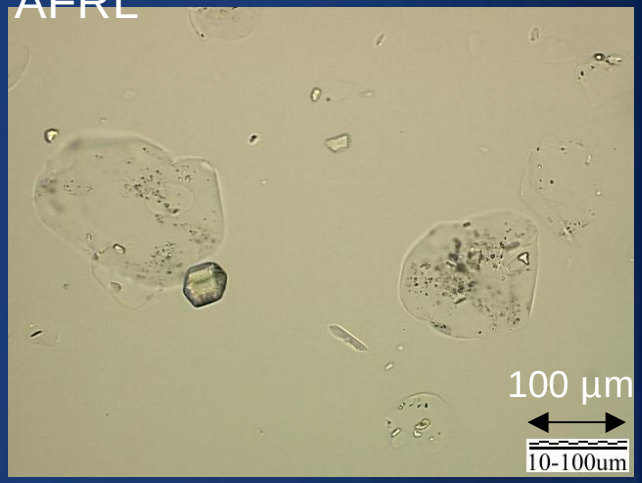




ARDEC



AFRL



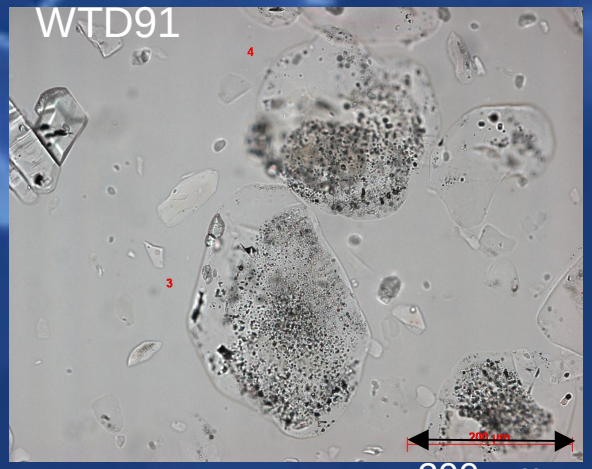
Same RDX, Different Labs

Photomicrographs, matching refractive index

OSI RDX

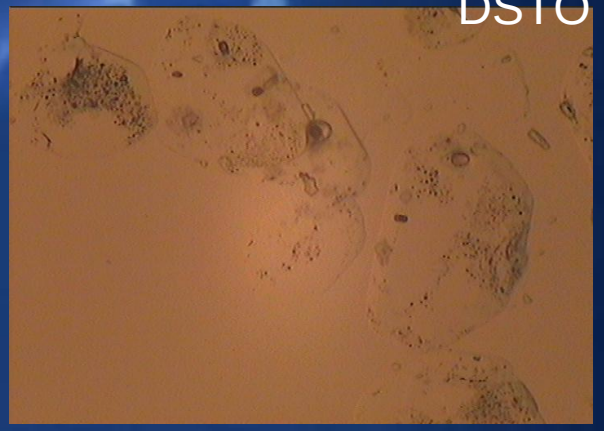
100 µm

WTD91



200 µm

DSTO



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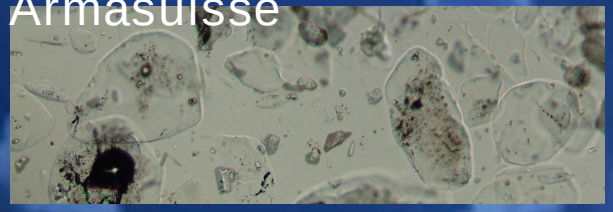

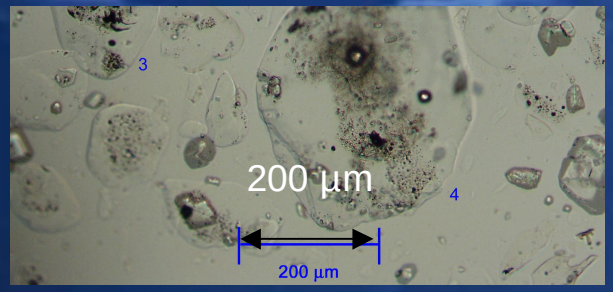

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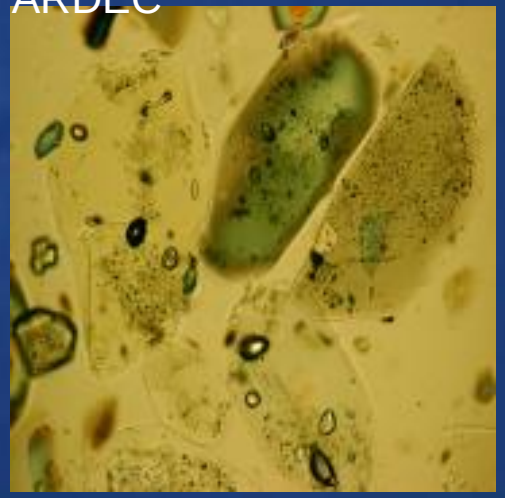

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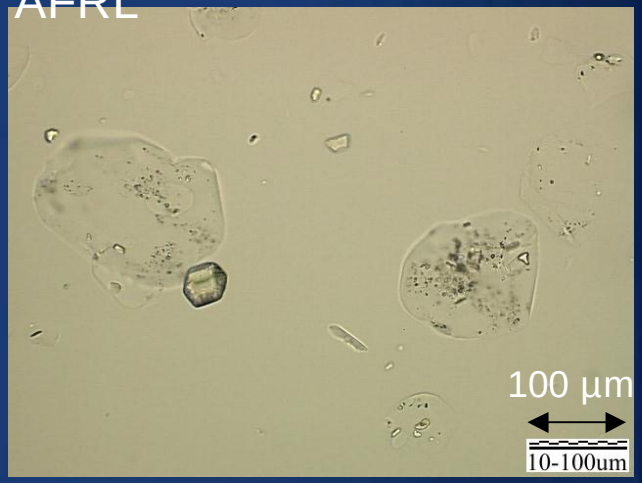
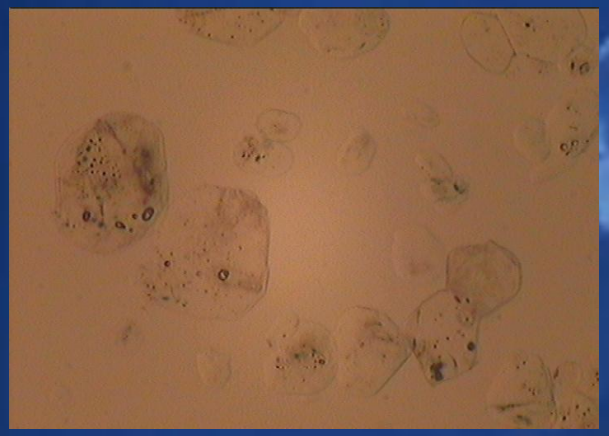
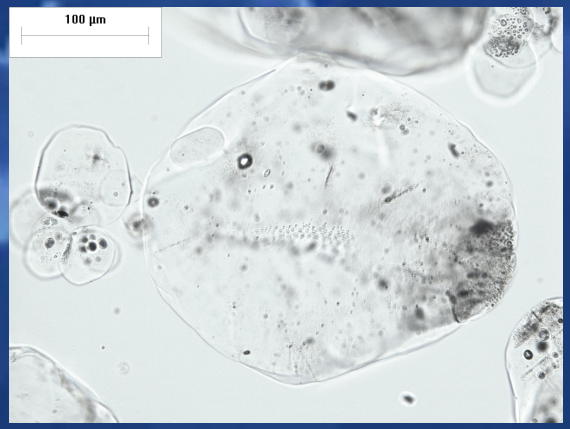
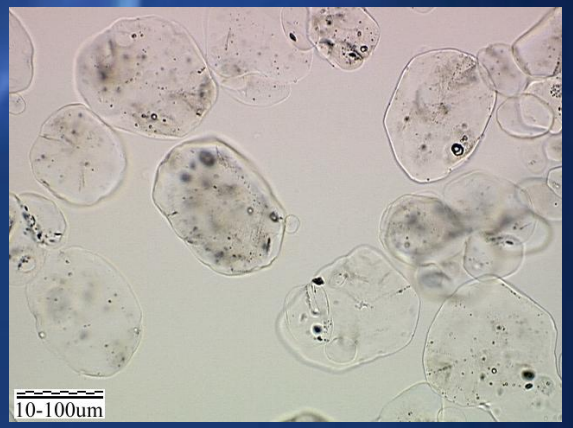


Other Types of non-RS-RDX

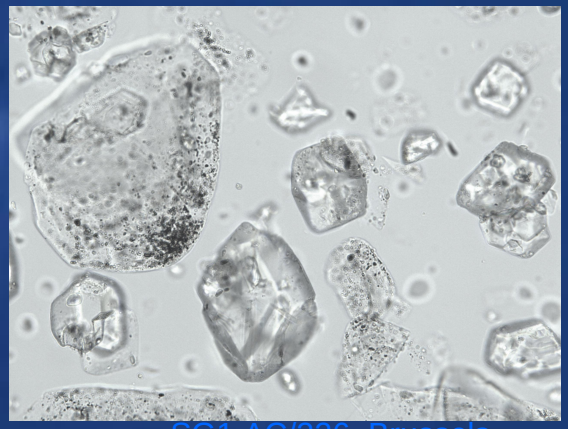
MI-RDX

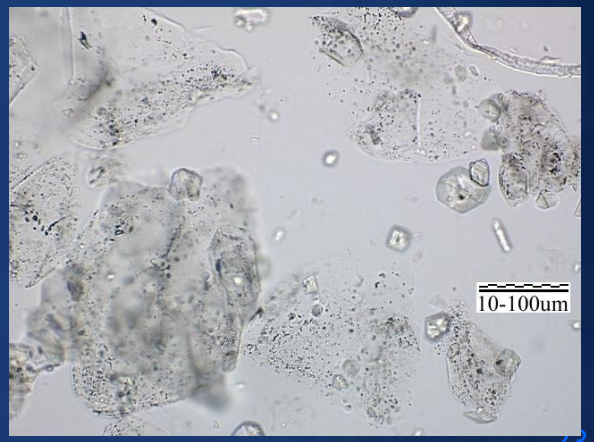






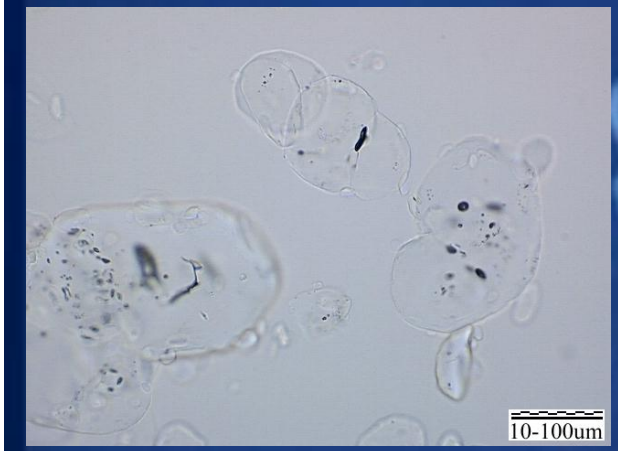
Dyno Type II

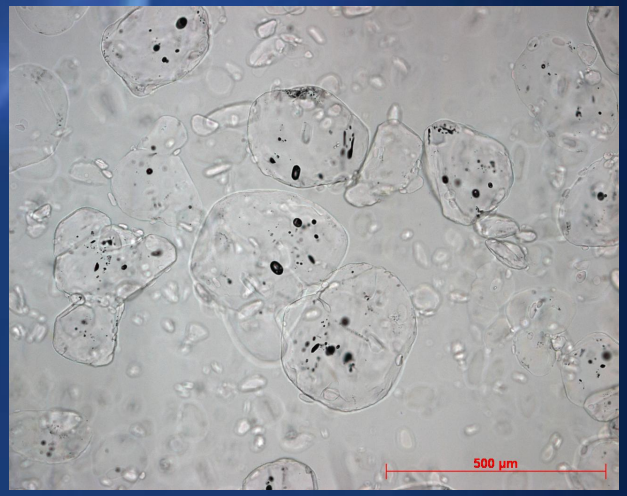




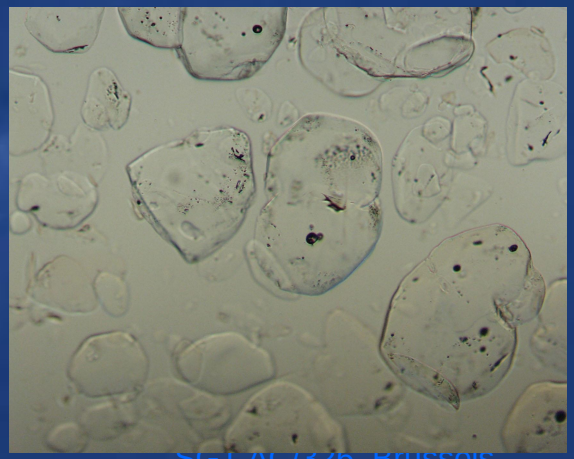
Other Types of RS-RDX

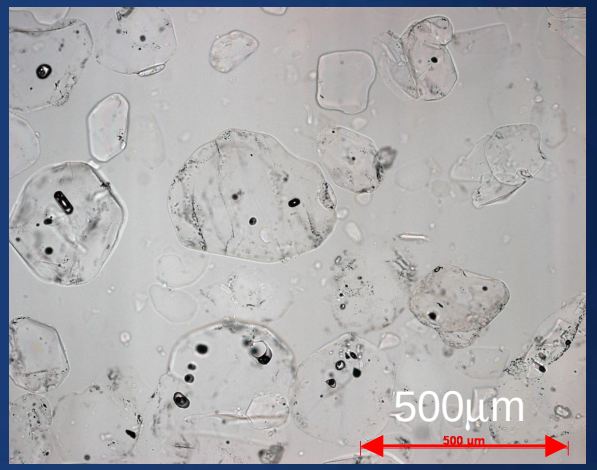
I-RDX





RO





Conclusions: Optical Microscopy

- There are qualitative differences in the types of imperfections seen in RS- and non-RS-RDX, but the attempt to quantify the differences did not provide useful information.
- Pervasive “pinhole” defects may be more important in determining sensitivity than a few discrete holes of larger size.
- Automation of the process for analyzing images may be a way to take the subjectivity out of the analysis.

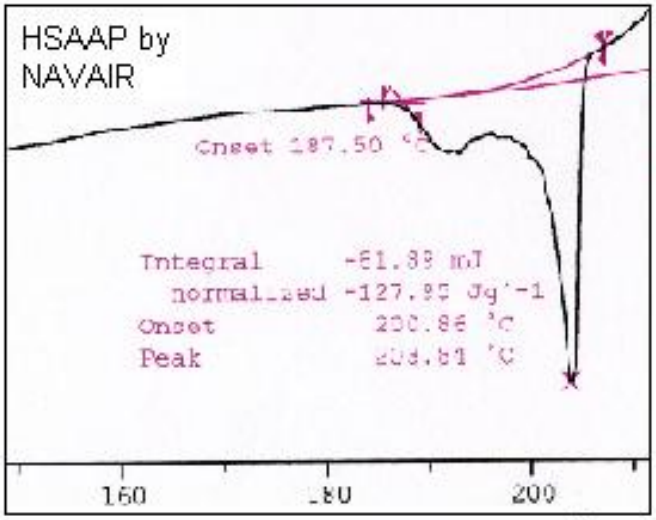
Mandatory Test Results

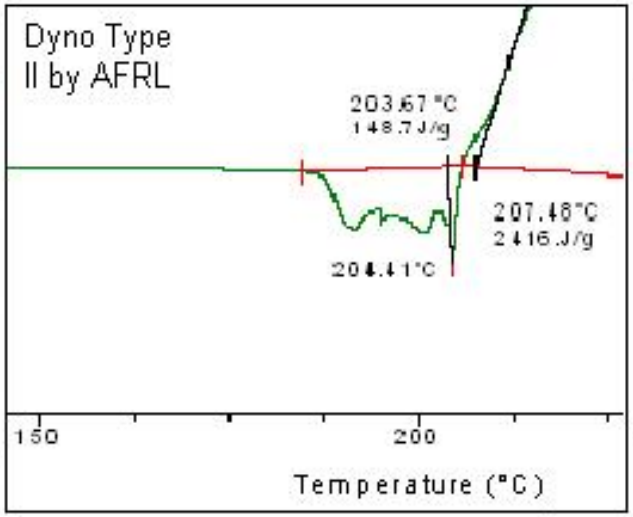
Headline Summary

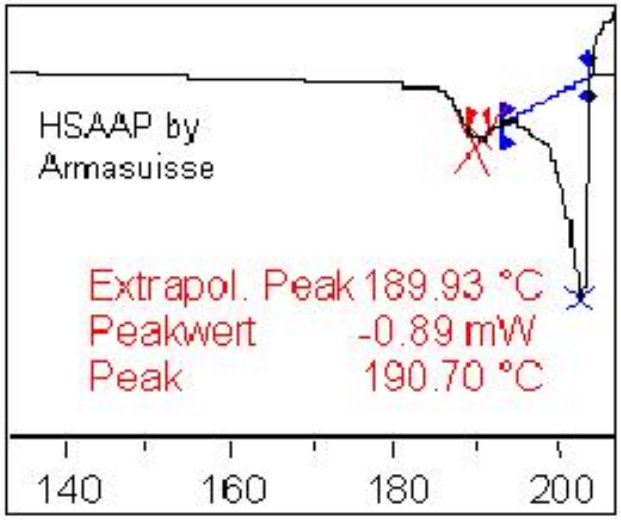
Mandatory Tests (STANAG)

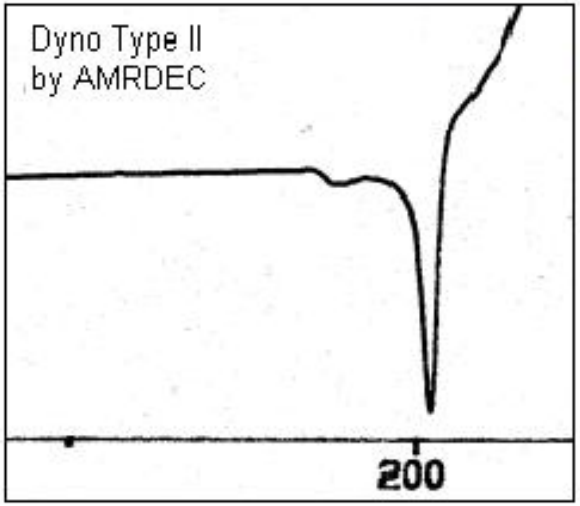
- DSC/melting point:
  - The thermograms of RDX samples containing substantial amounts of HMX are very complex
    - Phase change of HMX above 180°C
    - Melting point depression by presence of HMX in the RDX, formation of a eutectic
    - Decomposition of RDX in liquid phase
- HPLC:
  - The precision of the method is good for a given sample.
  - Type II RDX is intrinsically heterogeneous, and so great care must be taken to get a sample that is representative of the whole lot.
- GC/MS
  - Cyclohexanone was found in all samples at low levels.
  - Acetone determination was problematic due to co-elution of acetonitrile and acetone in many cases. When it was measured and reported, the levels were very low. [N.B. This method is not included in STANAG 4022 Edition 4.]

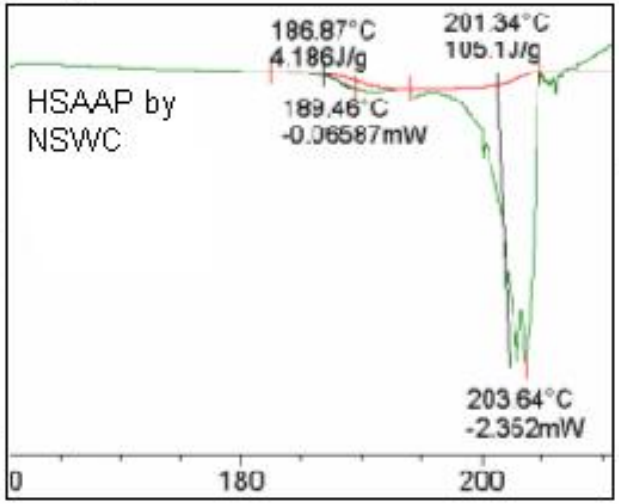
Issues with Interpreting Melting Point via DSC (for Type II Materials)

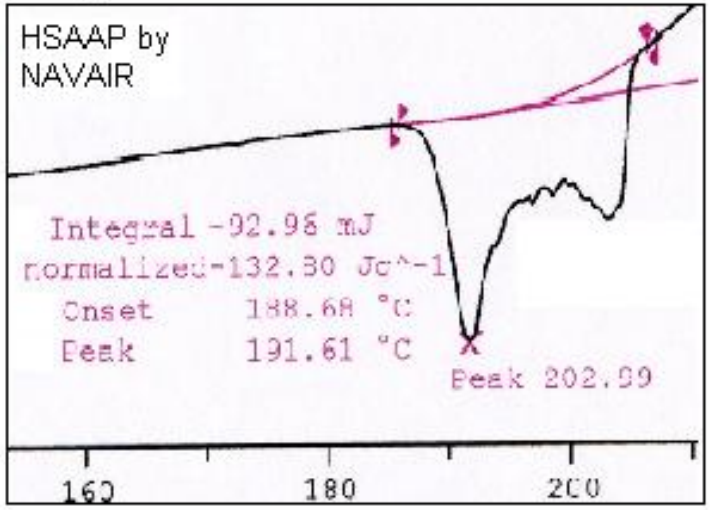












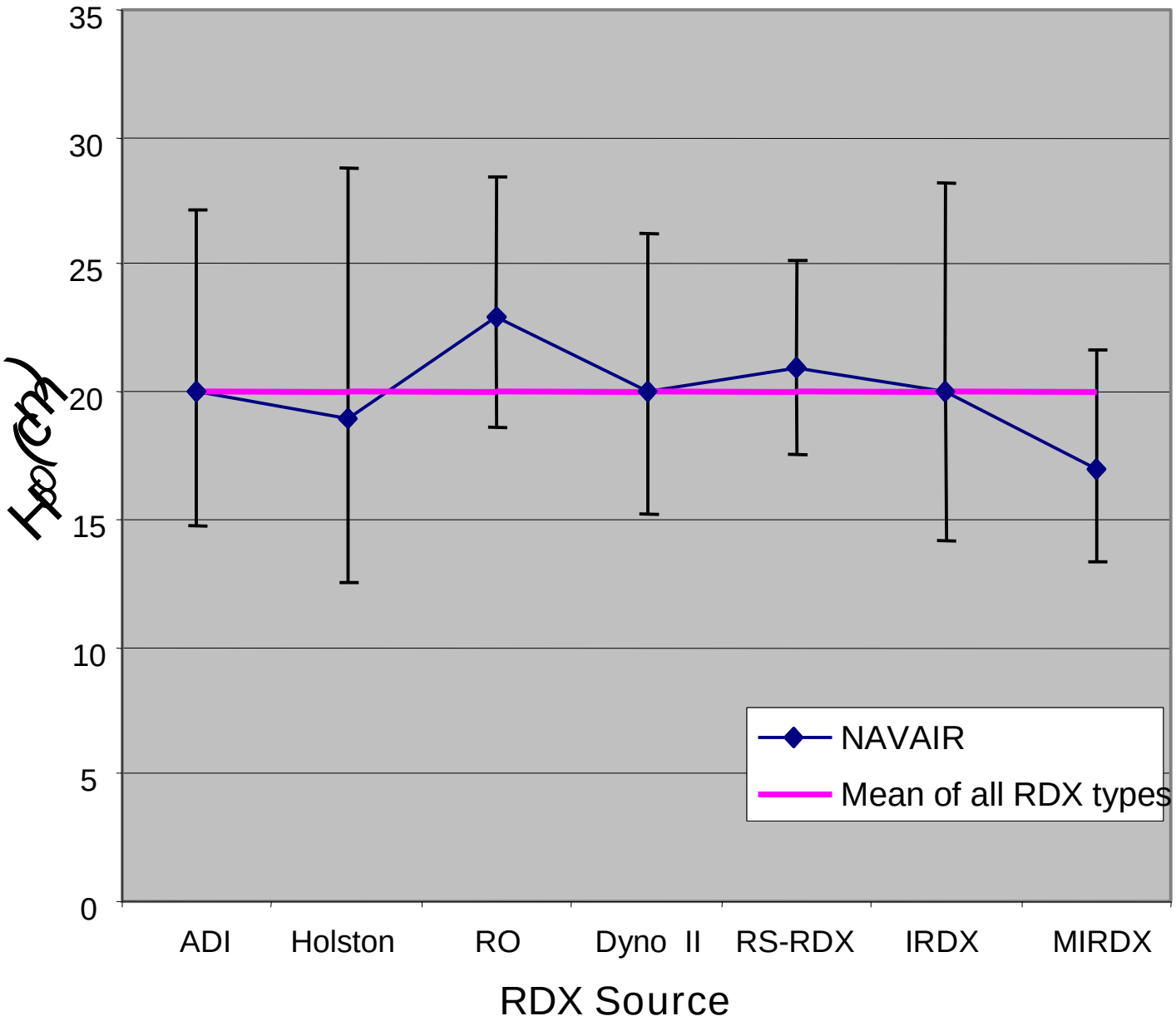
Mandatory Tests (STANAG)

- Particle size determination
  - The assessment of whether a lot of RDX meets the specification for Class 1 RDX, in accordance with STANAG 4022 Edition 4 (Draft), does not always agree with that of the manufacturer.
  - Some of the RDX lots had a distinctly bimodal character, with a small peak at small particle sizes, as determined by LALLS.
- Impact sensitivity:
  - There is no perceptible difference in the impact sensitivity of RS-RDXs and non-RS-RDXs.
  - Determination of impact sensitivity by a Bruceton protocol is a time-honored technique, but not necessarily the best one.

Impact Sensitivity

- Mainly two methods reported
  - BAM Impact Test (5 labs)
  - ERL/BOE variation (3 labs)
- STANAG 4489 also includes the Rotter Impact Test, which will be reported by DSTO and dstl.
- Results
  - BAM: poor consistency
    - BAM has been considered to be more reproducible, lab-to-lab, than ERL
  - ERL: high level of consistency between labs

ERL Impact Results from NAVAIR



Mandatory Tests (Non-STANAG)

- Crystal density by sink-float method
  - The R4 version of this test showed too much scatter in the data for it to be useful in distinguishing RS- from non-RS-RDX.
  - Single-run data from ISL showed well-formed curves, but did not distinguish RS- from non-RS-RDX.
- Pycnometric density
  - Values from liquid pycnometry were slightly higher than from gas pycnometry.
  - Results for Type II RDX are confounded by the presence of HMX crystals, which have a higher TMD than RDX.
  - The span of the measurements is narrow, so it is difficult to correct for the presence of HMX crystals.

Summary

- The R^4 program has resulted in a unique data set that will help to identify the features leading to lowered sensitivity in some forms of RDX.
- Shock sensitivity differences for a wide variety of types of RDX have been measured.
- NQR and microscopic examination appear to be the post promising tools to discriminate between RS- and non-RS-RDX.
- Additional analysis of the data from this program is continuing and some new techniques may be applied to samples that remain at the participating laboratories.

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  - Those who contributed to the methods
  - Those who performed the analyses
  - Those who undertook additional studies
- And also sponsors who funded the efforts.