STANAG 4178 Ed. 2 – A New, Internationally Accepted Standard For Testing of Nitrocellulose

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RHEINMETALL NITROCHEMIE

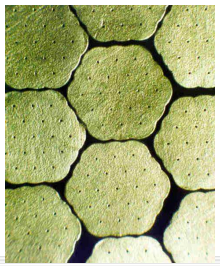
Contents

Why do we still use Nitrocellulose?

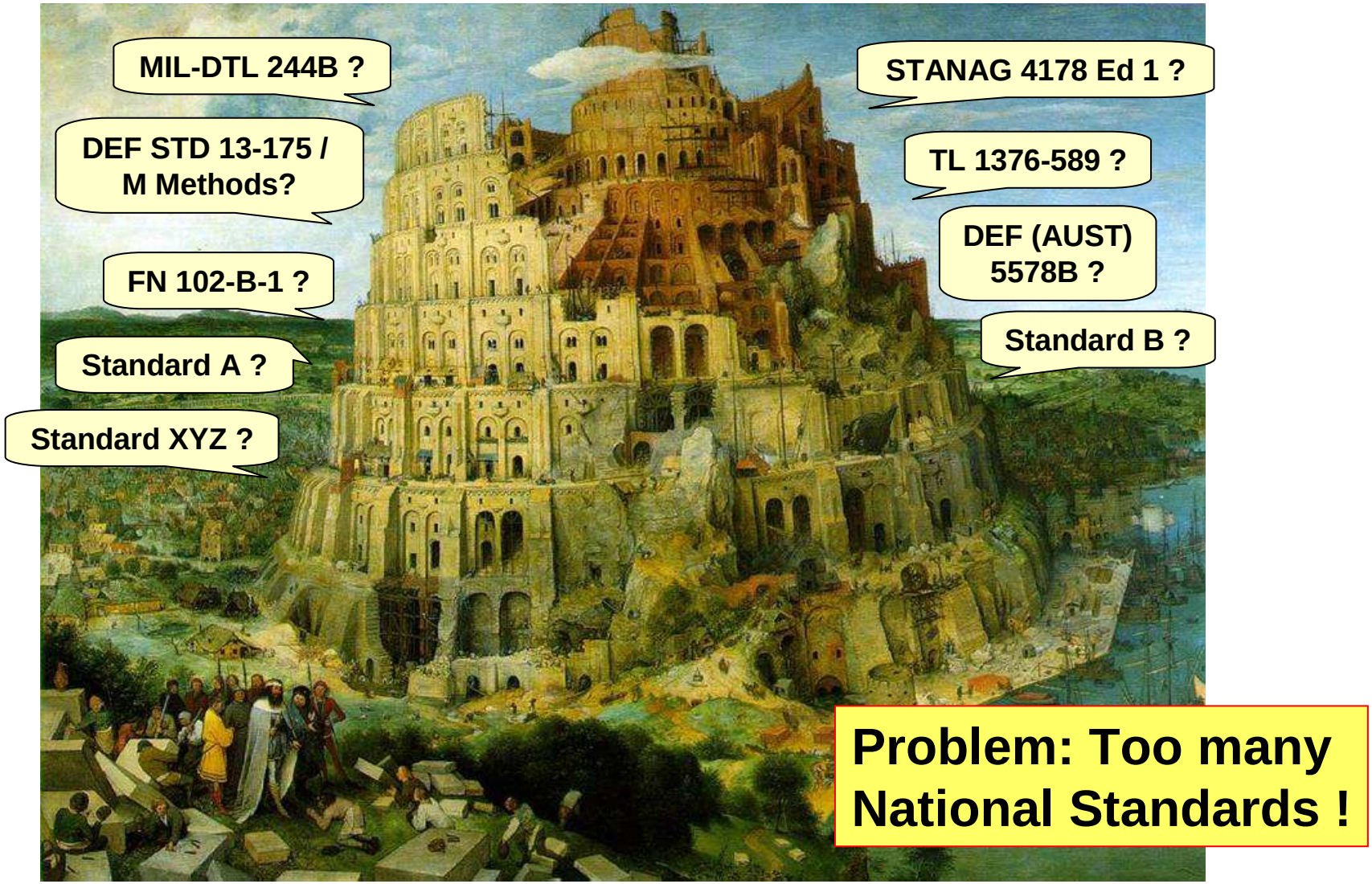
- **Nitrocellulose (NC) is still used as main ingredient in >99% of all gun propellants (and in many other explosives) because:**
  - **it is <u>energetic material + binder</u>**
  - **it is <u>cheap</u> (≈ 5 US$ / kg)**
  - **it is <u>easy to process</u> in many different ways (solvent and solvent-less extrusion, ….)**
  - **<u>more than 110 years of experience</u>**
  - **products show <u>good-natured burning behaviour</u> (low pressure-exponent → <u>save and reproducible interior ballistics</u>)**
  - **<u>excellent IM properties achievable</u> (with new formulation and processing methods)**

Example: R-type propellant for 155mm Modular Charge System MCS DM72 / DM92

Bullet Impact BI	**⇨ Type V**
SCJI	**⇨ Type IV (RPG7) – V (Bomblet)**
Slow / Fast Heating SH / FH	**⇨ Type V**
Sympathetic Reaction (SCJI)	**⇨ No Reaction**



The Problem

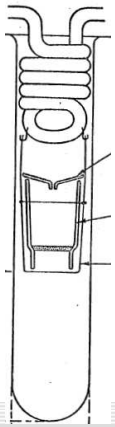


Problem: Too many National Standards !

The Problem

- **Too many national Standards**
  - **<u>MIL-DTL-244</u> is the most modern and most widely accepted Standard (also outside the USA)**
  - **<u>STANAG 4178 Ed. 1</u> was never recognized as International Standard**

- **Many of the test methods of STANAG 4178 Ed. 1 did no longer fit into today's production / quality management / working safety environment as they are:**
  - **too dangerous (e.g. Nitrometer Method – uses Mercury!)**
  - **too complicated (e.g. Devarda's Alloy Method)**
  - **too costly**
  - **too time consuming (e.g. WILEY Extraction – requires ≈ 3 days!)**



The Goal

The Strategy

- **To provide <u>test procedures for all physical-chemical properties of the NC</u> that are regarded as important by the NATO Participating Nations**
- **If test procedure for a certain property is contained in MIL-DTL-244C (Draft): To adopt this test procedure in STANAG 4178 Ed. 2**
  - **Tests in MIL-DTL-244C and STANAG 4178 Ed. 2 will be identical**
  - **MIL-DTL-244 has just been updated by the US Industrial Product Team (→ all these test procedures are already "up to date")**
- **If no MIL-DTL-244C test procedure for a certain property is available:**
  - **To use best suited test procedure(s) from other national standards**
  - **To improve these test procedures if necessary / possible**
  - **Or to develop new test procedure(s)**
- **To ensure that the test procedures are also applicable to "chalked NC" (as used in the UK and Australia): Alterations / corrections if necessary**
- **To include Quality Management Requirements, Safety Precaution Notes, and Typical Ranges of Test Results**

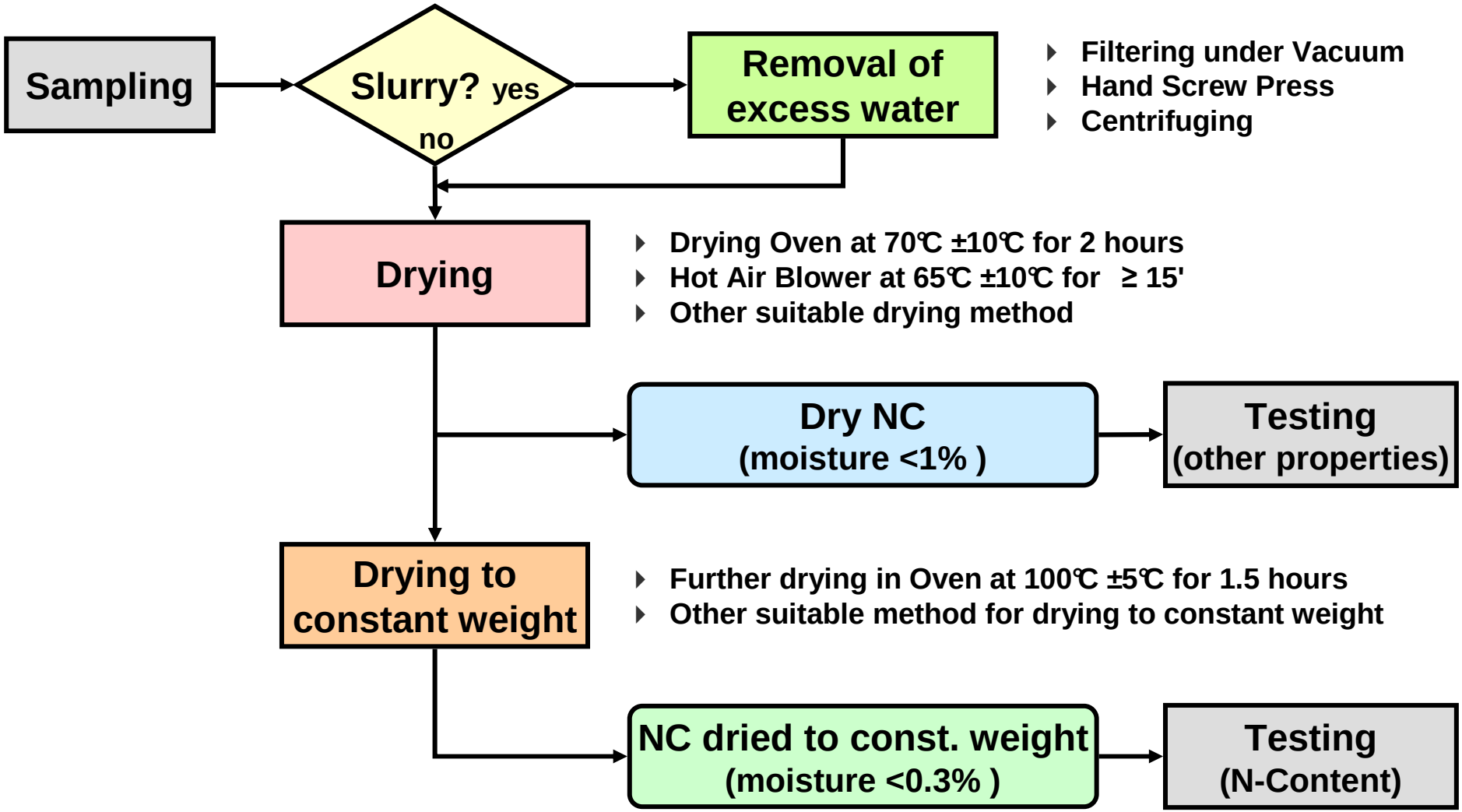
The Team

NATO/PfP AC/326 SG/1 CNG
+ US Industrial Product Team (IPT)
+ international NC experts

- **55 Persons**
- **36 MoD/DoD Agencies / Institutes / Companies**
- **17 Nations**

Australia:	- Thales Australia, Mulwala
Austria:	- Bowas-Induplan Chemie GmbH
Belgium:	- PB Clermont S.A.
Canada:	- DRDC-RDDC, Valcartier
	- General Dynamics OTS, Valleyfield
Croatia:	- Brodarski Institut
Czech Republic:	- Explosia a.s.
	- Synthesia a.s.
Denmark:	- Danish Defence, Acquisition and Log. Org. (DALO)
Finland:	- PVTT, Lakiala
	- EURENCO, Vihtavuori Oy
France:	- ETBS Bourges
	- SNPE / MANUCO, Bergerac
	- Eurenco France
Germany:	- WIWEB, Swisttal
	- Fraunhofer ICT
Italy:	- CSSN Italian Navy
	- Stabilimento Militare Propellenti
	- Explosives Company SEI
Netherlands:	- TNO-Defence, Security and Safety
Singapore:	- Defence Science & Technology Agency
South Africa:	- Rheinmetall Denel Muntions RDM
Switzerland:	- armasuisse, Federal Department of Defence
	- Nitrochemie Wimmis AG
United Kingdom:	- Defence Science & Techn. Lab. (DSTL), Fort Halstead
	- Defence Ordnance Safety Group, MOD, Abbey Wood
	- QinetiQ, Ardeer
	- Cranfield University
	- AWE Plc, Aldermaston
	- BAE Systems
	- Roxel UK, Kidderminster
USA:	- Naval Surface Warfare Center, Indian Head
	- ARDEC, Picatinny
	- ATK, Radford
	- Esterline Defense Group, Coachella
	- GD-OTS, St. Marks Powder

The Sample Preparation



The Test Procedures – Overview

Basic Characterisation

- **Nitrogen Content**
  - Ferrous Ion Titration
  - Nitrogen Analyzer
  - Combustion Calorimetry
  - (Devarda's Alloy Method)
  - (Schulze-Tiemann)
  - (Nitrometer)
- **Ether-Alcohol Solubles**
  - Filtration Method
  - Evaporation Method
- **Acetone Insolubles**

Stability

- **132°C Stability Test**
  - Bergmann-Junk
  - Bergmann-Junk-Siebert
- **134.5°C Heat Test (MV)**

Purity

- **Visual Purity Test**
- **Ash**
- **Grit**
- **Ionic Impurities**
  - Ion Chromatography
  - Sulphate Content
  - Residual Acidity
  - Alkalinity
  - Calcium by Spectroscopy
- **Oil and Grease Content**
- **(Abel-Type Heat Tests)**

Fibre Quality

- **Fineness**
- **Fibre Length Distribution**
- **Water Retention Value**
- **Drainability**
- **Agglomerates**

Polymeric Properties

- **Viscosity**
- **Molecular Mass Distribut.**

Water / Alcohol Cont.

- **Total Volatile Content**
  - Oven Method
  - Moisture Analyzer
- **Water Content**
  - Karl-Fischer Titration
  - Karl-Fischer Oven
- **Alcohol and / or Water**
  - Gas Chromatography
  - NIR Spectroscopy

Other Properties

- **Temperature of Ignition**
- **Heat of Explosion**

The Test Procedures – Mandatory Tests

Basic Characterisation

- **Nitrogen Content**
  - Ferrous Ion Titration
  - Nitrogen Analyzer
  - Combustion Calorimetry
  - (Devarda's Alloy Method)
  - (Schulze-Tiemann)
  - (Nitrometer)
- **Ether-Alcohol Solubles**
  - Filtration Method
  - Evaporation Method
- **Acetone Insolubles**

Stability

- **132°C Stability Test**
  - Bergmann-Junk
  - Bergmann-Junk-Siebert
- **134.5°C Heat Test (MV)**

- **Testing of the following properties is mandatory**
  - Nitrogen Content *)
  - Ether-Alcohol Solubles *)
  - Acetone Insolubles
  - Stability *)

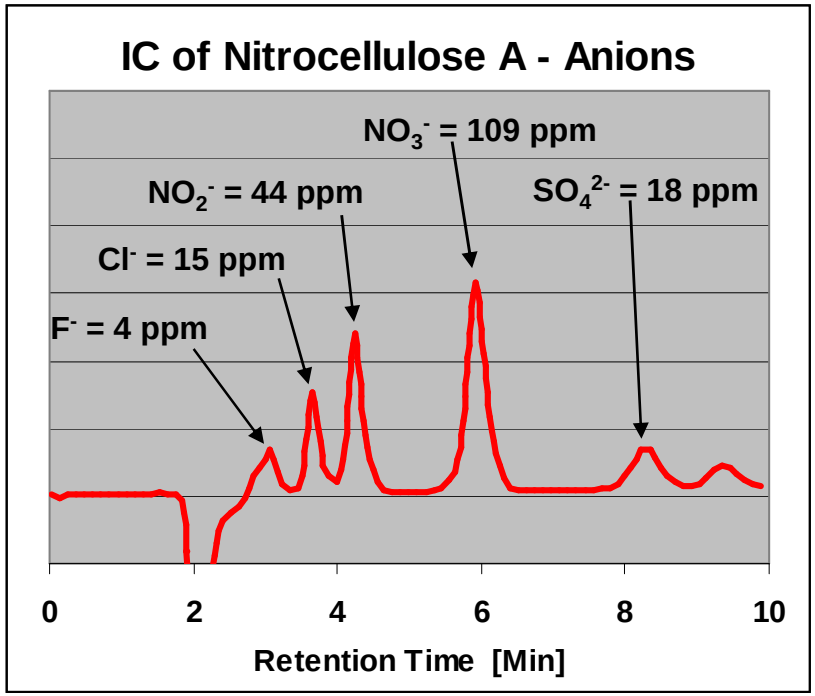
*) Different test methods available

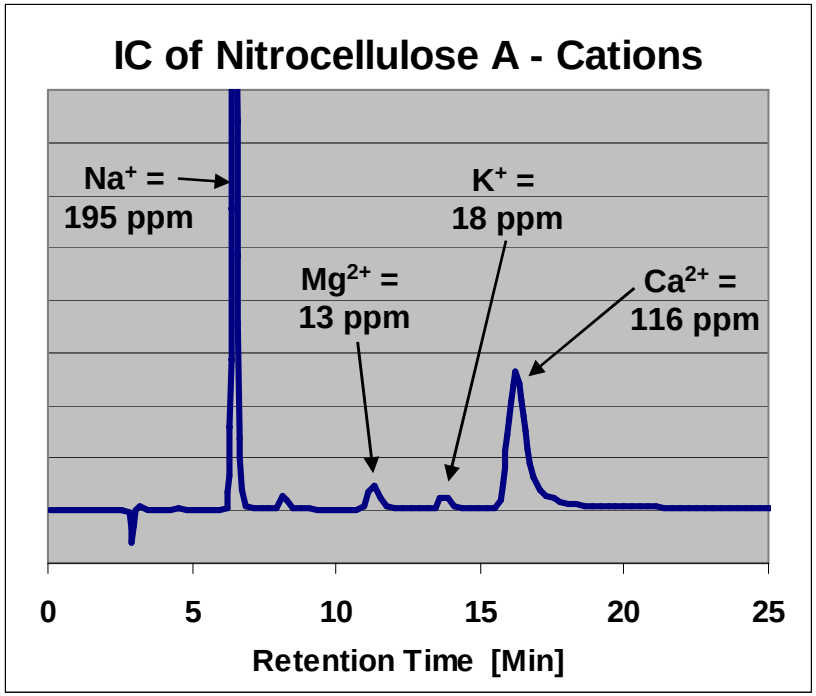
- **All other tests are not mandatory and should only be performed if regarded as necessary or if requested by contract or customer**

The Test Procedures – Analysis of Ionic Impurities

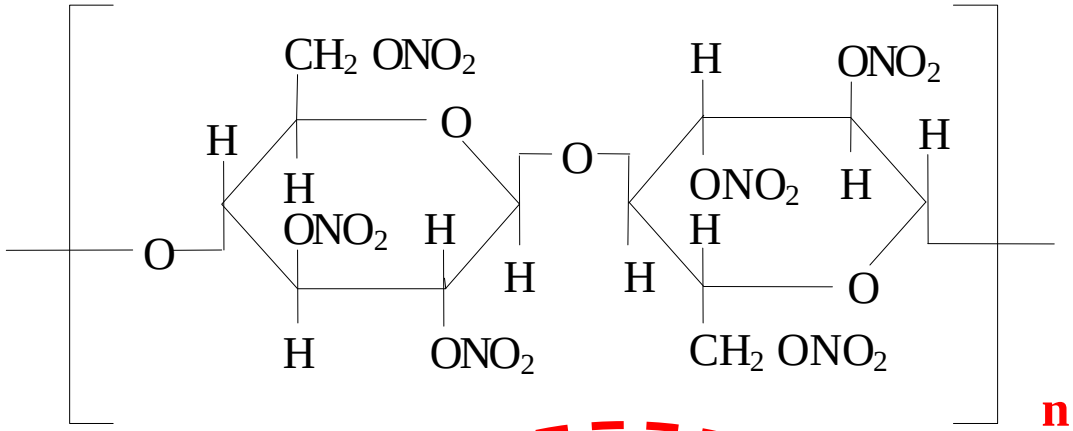
- **Ion Chromatography Method:**
  - **New method; procedure has been supplied by USA**
  - **Can be used to assess numerous different ionic impurities**
  - **Principle: Extraction of the ions from the NC with boiling water, followed by analysis with ion chromatography**
  - **Recommended method in Ed. 2 !**

Chromatograms provided by





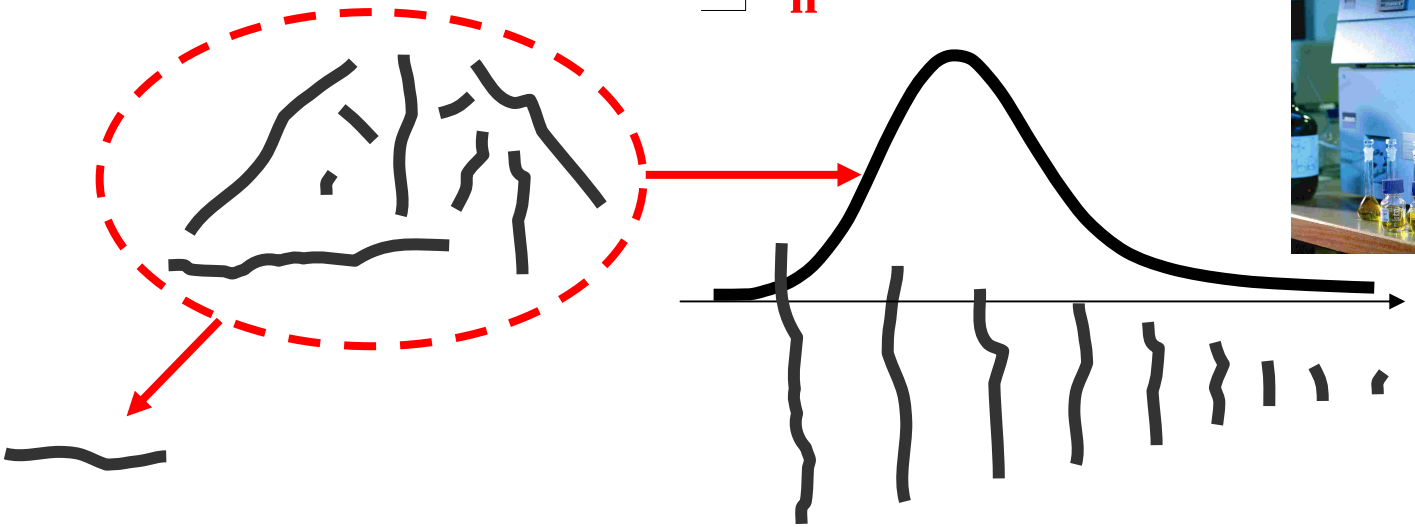
The Test Procedures – Polymeric Properties



Degree of Polymerisation = Length of NC Polymer Chain

Typical Value: n = 500 – 1'400

(= Molecular Mass of 300'000 – 800'000 Daltons; may be lower or higher for specific NC qualities)



- **Viscosity → Average Degree of Polymerisation**
- **GPC → Distribution of Degree of Polymerisation / Molecular Mass**

The Test Procedures – Fibre Length Determination

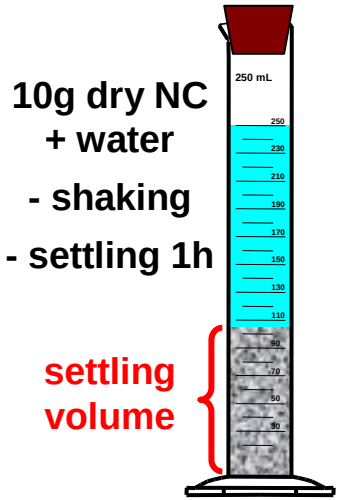
The Fibre Length of NC is a process relevant property as it reveals the amount of cutting / grinding / refining the NC had undergone during manufacture

Indirect Methods

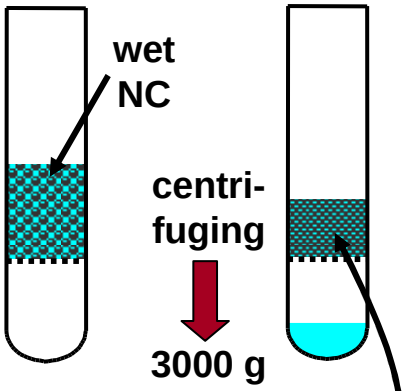
- **Fineness**
- **Water Retention Value WRV**
- **Drainability**

Direct Method

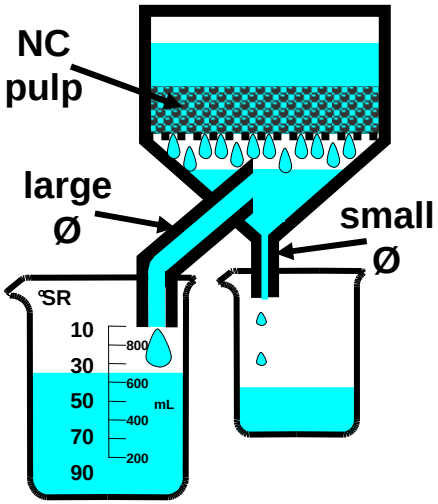
- **Fibre Length / Shape Analyzer**



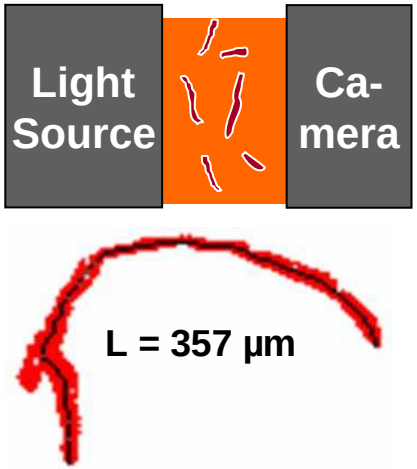
→ NC settling volume



→ Water retention value = water content of centrifuged NC

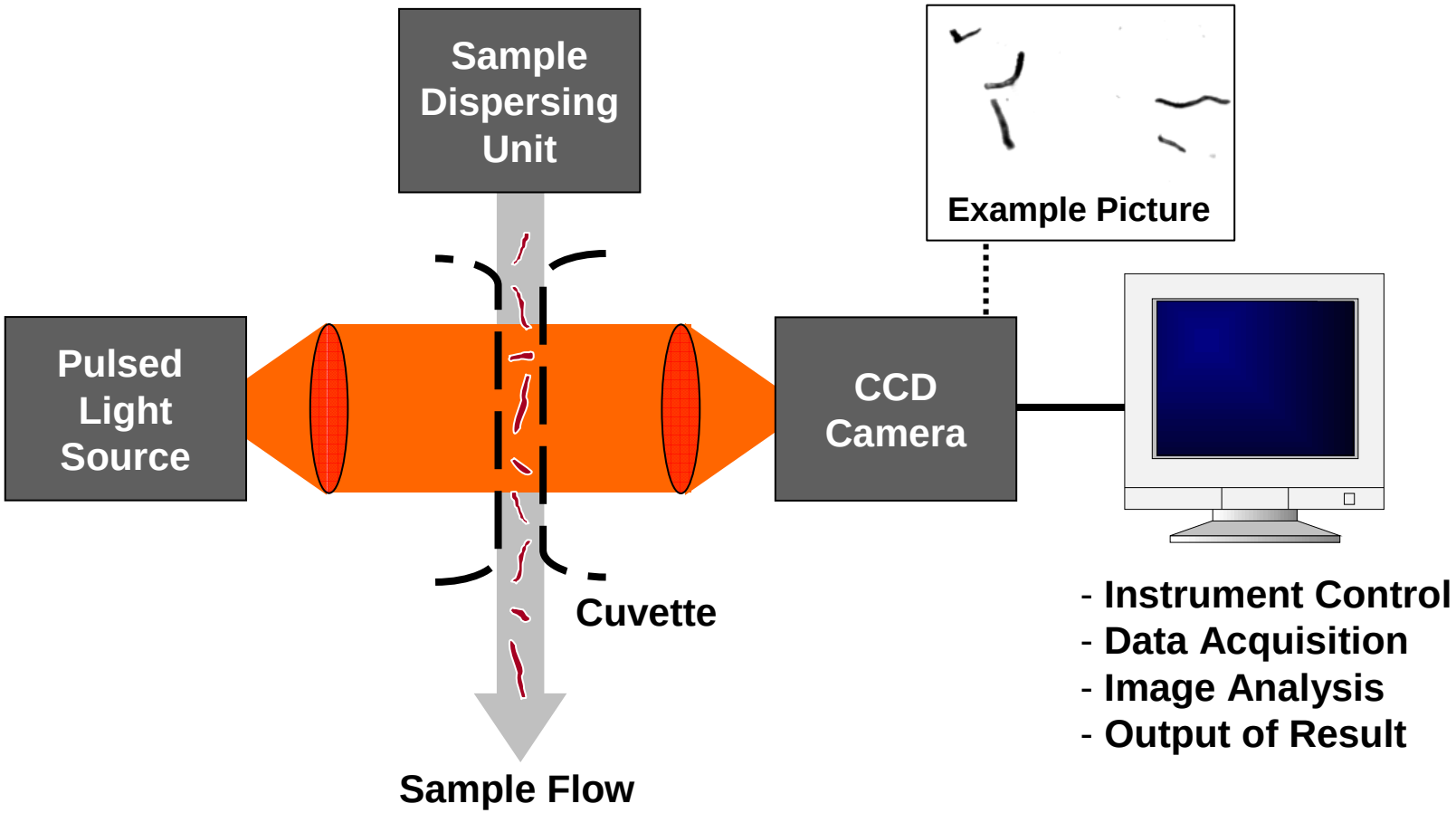


→ Drainability = rate of dewatering of pulp



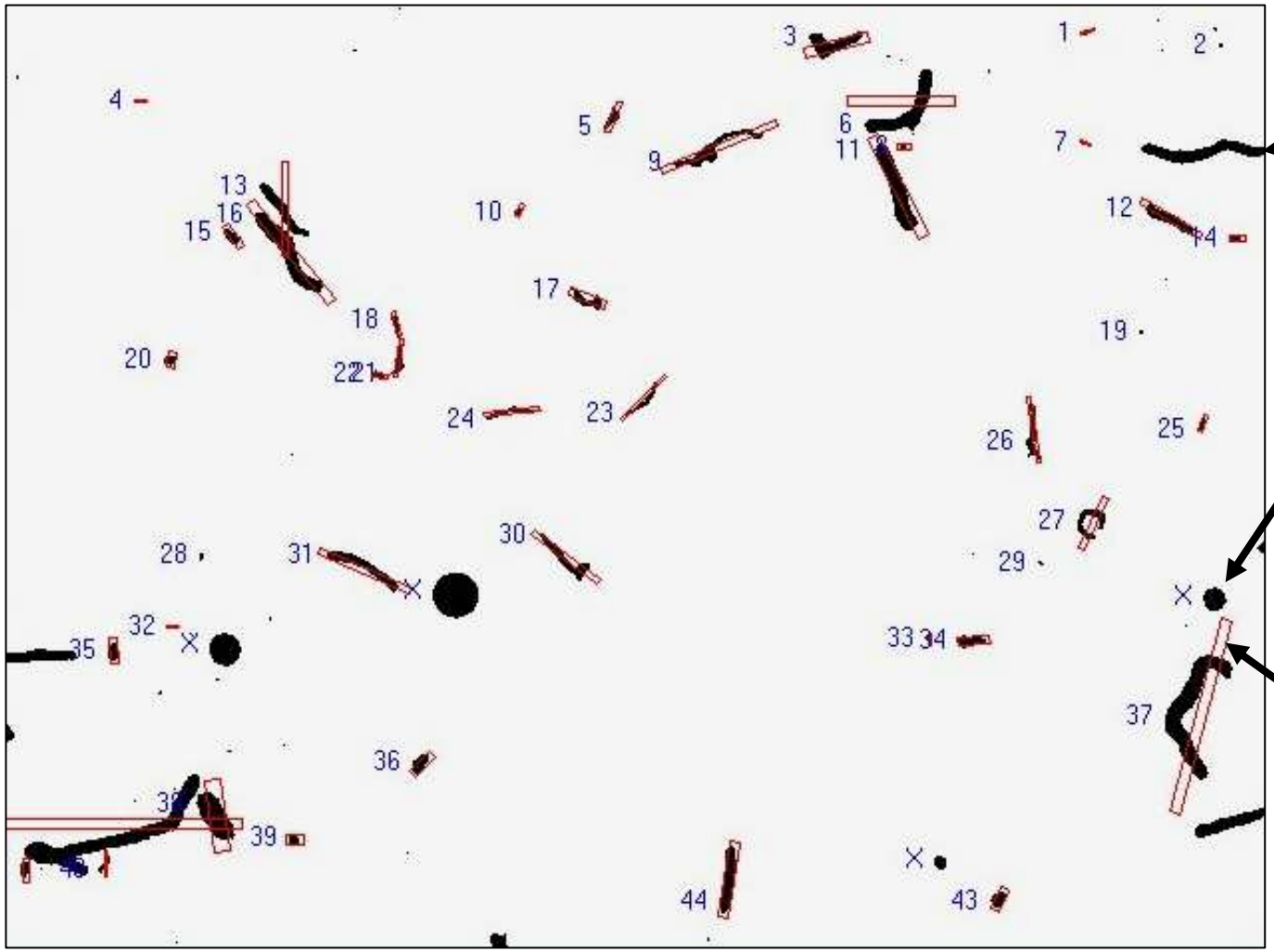
→ NC average fibre length and distribution

Fibre Quality Analyzers – Principle of Operation



Fibre Quality Analyzers – Picture Frame with Image Analysis

Example: Beckman-Coulter RapidVUE



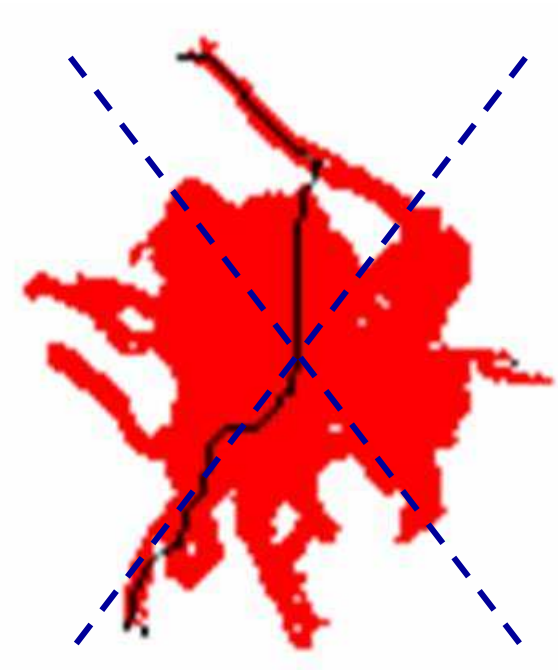
Fibre touches frame → excluded from further analysis

Round particle (air bubble) identified by shape factor or sphericity factor → excluded from further analysis

Red rectangle represents flattened fibre (with calculated equivalent length and width)

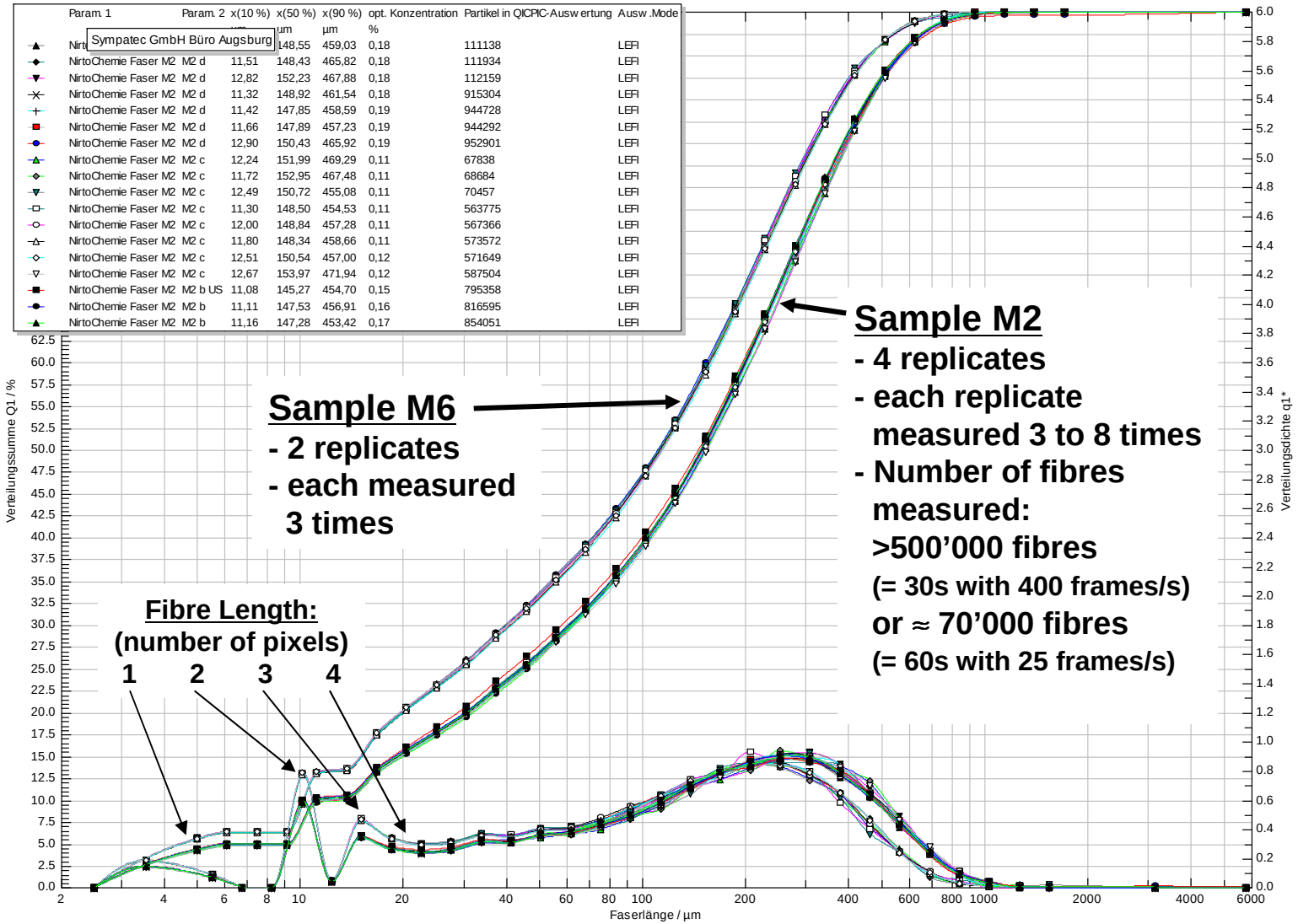
Fibre Quality Analyzers – Image Analysis / Skeletonizing Method

Example: Sympatec QICPIC



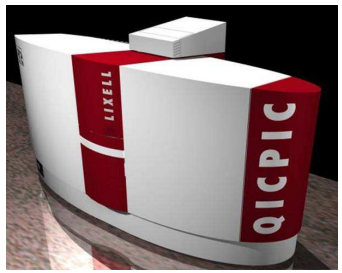
Agglomerates can be eliminated from analysis

Fibre Quality Analyzers – Repeatability of Results



Param. 1	Param. 2	x(10 %)	x(50 %)	x(90 %)	opt. Konzentration	Partikel in QICPIC-Auswertung	Ausw.Mode
		µm	µm	µm	%		
Nirt			148,55	459,03	0,18	111138	LEFI
NirtoChemie Faser M2	M2 d	11,51	148,43	465,82	0,18	111934	LEFI
NirtoChemie Faser M2	M2 d	12,82	152,23	467,88	0,18	112159	LEFI
NirtoChemie Faser M2	M2 d	11,32	148,92	461,54	0,18	915304	LEFI
NirtoChemie Faser M2	M2 d	11,42	147,85	458,59	0,19	944728	LEFI
NirtoChemie Faser M2	M2 d	11,66	147,89	457,23	0,19	944292	LEFI
NirtoChemie Faser M2	M2 d	12,90	150,43	465,92	0,19	952901	LEFI
NirtoChemie Faser M2	M2 c	12,24	151,99	469,29	0,11	67838	LEFI
NirtoChemie Faser M2	M2 c	11,72	152,95	467,48	0,11	68684	LEFI
NirtoChemie Faser M2	M2 c	12,49	150,72	455,08	0,11	70457	LEFI
NirtoChemie Faser M2	M2 c	11,30	148,50	454,53	0,11	563775	LEFI
NirtoChemie Faser M2	M2 c	12,00	148,84	457,28	0,11	567366	LEFI
NirtoChemie Faser M2	M2 c	11,80	148,34	458,66	0,11	573572	LEFI
NirtoChemie Faser M2	M2 c	12,51	150,54	457,00	0,12	571649	LEFI
NirtoChemie Faser M2	M2 c	12,67	153,97	471,94	0,12	587504	LEFI
NirtoChemie Faser M2	M2 b US	11,08	145,27	454,70	0,15	795358	LEFI
NirtoChemie Faser M2	M2 b	11,11	147,53	456,91	0,16	816595	LEFI
NirtoChemie Faser M2	M2 b	11,16	147,28	453,42	0,17	854051	LEFI

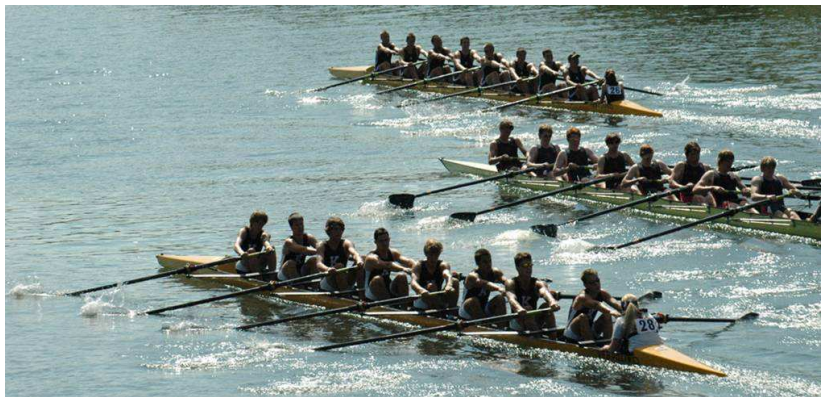
- **Excellent Repeatability (replicate analysis)**
- **Significant difference between samples M2 and M6 found (as expected)**



The Summary and Conclusions

STANAG 4178 Ed. 2

- **Was prepared in the timeframe 2007 – 2009 in a joint effort of about 55 persons from 17 nations**
- **Is based on the internationally accepted MIL-Standard, but contains additional test methods from Ed. 1 and from other sources**
- **Incorporates improvements to make tests more accurate and reliable, faster, safer and cheaper**
- **STANAG 4178 Ed. 2 is currently in NATO ratification (7 nations have already ratified; a total of 13 needed)**



The Acknowledgments

- All contributors (NATO/PfP AC/326 SG/1 CNG + US Industrial Product Team + international NC experts)
- Nitrochemie Laboratory Team (Ruth Sopranetti, Marc Müller; Melanie Wolf, Dominik Werfl, …)
- Patrick Folly / armasuisse for support and funding (Project LFP R-3210-042-67)

Australia:	- Thales Australia, Mulwala
Austria:	- Bowas-Induplan Chemie GmbH
Belgium:	- PB Clermont S.A.
Canada:	- DRDC-RDDC, Valcartier
	- General Dynamics OTS, Valleyfield
Croatia:	- Brodarski Institut
Czech Republic:	- Explosia a.s.
	- Synthesia a.s.
Denmark:	- Danish Defence, Acquisition and Log. Org. (DALO)
Finland:	- PVTT, Lakiala
	- EURENCO, Vihtavuori Oy
France:	- ETBS Bourges
	- SNPE / MANUCO, Bergerac
	- Eurenco France
Germany:	- WIWEB, Swisttal
	- Fraunhofer ICT
Italy:	- CSSN Italian Navy
	- Stabilimento Militare Propellenti
	- Explosives Company SEI
Netherlands:	- TNO-Defence, Security and Safety
Singapore:	- Defence Science & Technology Agency
South Africa:	- Rheinmetall Denel Muntions RDM
Switzerland:	- armasuisse, Federal Department of Defence
	- Nitrochemie Wimmis AG
United Kingdom:	- Defence Science & Techn. Lab. (DSTL), Fort Halstead
	- Defence Ordnance Safety Group, MOD, Abbey Wood
	- QinetiQ, Ardeer
	- Cranfield University
	- AWE Plc, Aldermaston
	- BAE Systems
	- Roxel UK, Kidderminster
USA:	- Naval Surface Warfare Center, Indian Head
	- ARDEC, Picatinny
	- ATK, Radford
	- Esterline Defense Group, Coachella
	- GD-OTS, St. Marks Powder