STANAG 4178 Ed. 2
Testing of Nitrocellulose

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RHEINMETALL NITROCHEMIE

Contents

The Problem

MIL-DTL 244B ?

DEF STD 13-175 / M Methods?

FN 102-B-1 ?

Standard A ?

Standard XYZ ?

STANAG 4178 Ed 1 ?

TL 1376-589 ?

DEF (AUST) 5578B ?

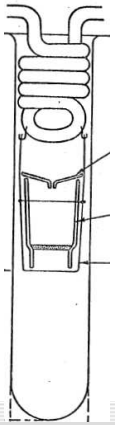
Standard B ?

Problem: Too many National Standards !

The Problem

- **Too many national Standards**
  - **MIL-DTL-244 is the most modern and most widely accepted Standard (also outside the USA)**
  - **STANAG 4178 Ed. 1 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

Goal: Internationally accepted and used Standard !

STANAG 4178 Ed 2 !

STANAG 4178 Ed 2 !

STANAG 4178 Ed 2 !

The Strategy

- **To provide test procedures for all physical-chemical properties of the NC 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 History (of STANAG 4178 Ed. 2)

- May 2007: Decision to revise STANAG 4178 by NATO/PfP AC/326 SG/1 CNG
- August 2007: Custodianship passed over from UK to Switzerland
- Different Meetings of NATO/PfP AC/326 SG/1 CNG + US IPT-Team + International NC Experts took place:
  - 2007 May 3, at NSWC, Indian Head, USA (Topic: NC Testing; Kick-off meeting)
  - 2008 April 8, at DSTL, Fort Halstead, UK (Topic: Testing of Chalked NC; UK and custodian)
  - 2008 May 14-15, at Spiez, Switzerland (Topic: NC Testing; first review of Draft STANAG)
  - 2009 May 7-8, at ARDEC, Picatinny, USA (Topic: NC Testing; second review of Draft)
  - 2008/2009 several meetings of GPC Method sub-team in different countries
- August 2009: Draft of STANAG 4178 Ed. 2 was forwarded to NATO/PfP AC/326 SG/1 by the custodian nation Switzerland.
- September 2009: Final draft of STANAG 4178 Ed. 2 accepted by NATO/PfP AC/326 SG/1; this was confirmed by NATO/PfP AC/326 Main Group in December 2009
- October 2009: STANAG 4178 Ed. 2 issued for NATO ratification
- April 2010: STANAG 4178 Ed. 2 has already been ratified by 5 nations

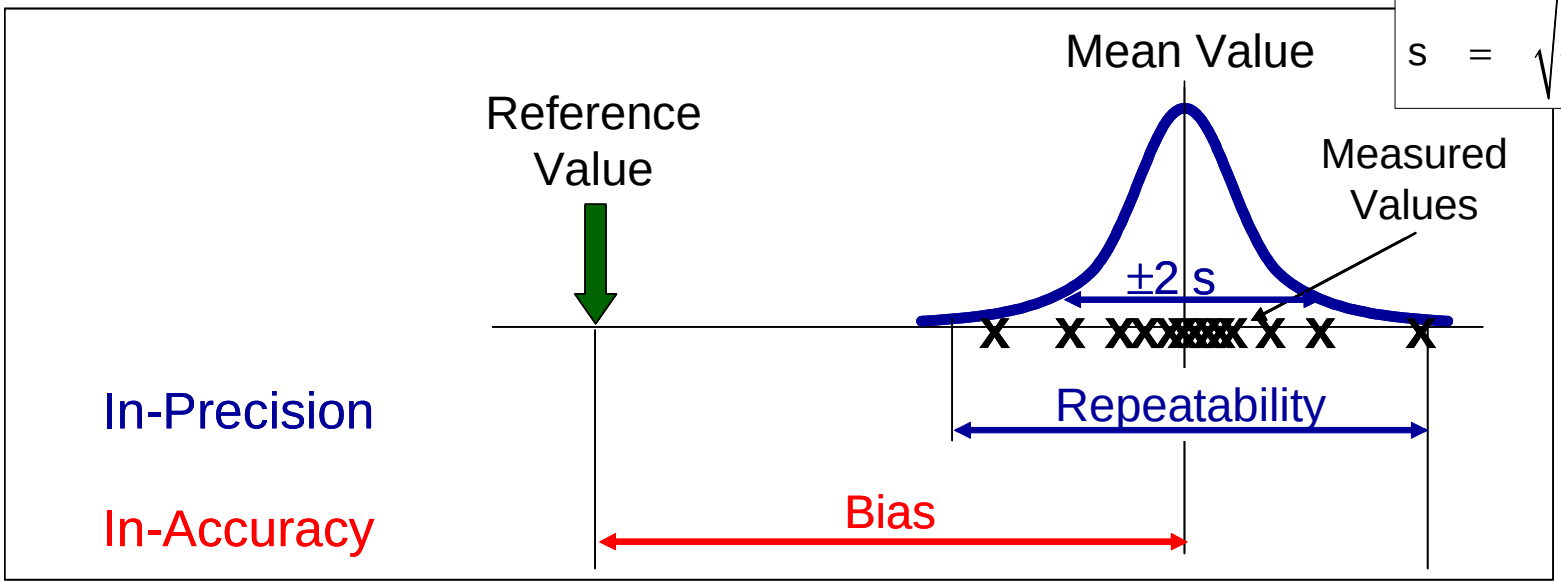
The Aim and Scope (of STANAG 4178 Ed. 2)

- Covers procedures for nitrocellulose sample preparation and testing
- Contains mandatory and optional test procedures:
  - Mandatory test methods must be performed on each nitrocellulose lot
  - Optional test methods give additional information → Choice of optional test methods must be agreed between purchaser and manufacturer (and stated in the contract or order)
- Classifications and specifications of nitrocellulose types:
  - Are not contained in STANAG 4178 Ed. 2
  - Must be agreed between purchaser and manufacturer (and stated in the contract or order)
  - Can be based on national standards (e.g. → MIL-DTL-244B/C, ….)
  - But should refer to properties / testing procedures of this STANAG
- STANAG 4178 Ed. 2 does neither cover raw material testing (e.g. linters) nor specify production processes (e.g. specific stabilisation processes; chalking of nitrocellulose, …)

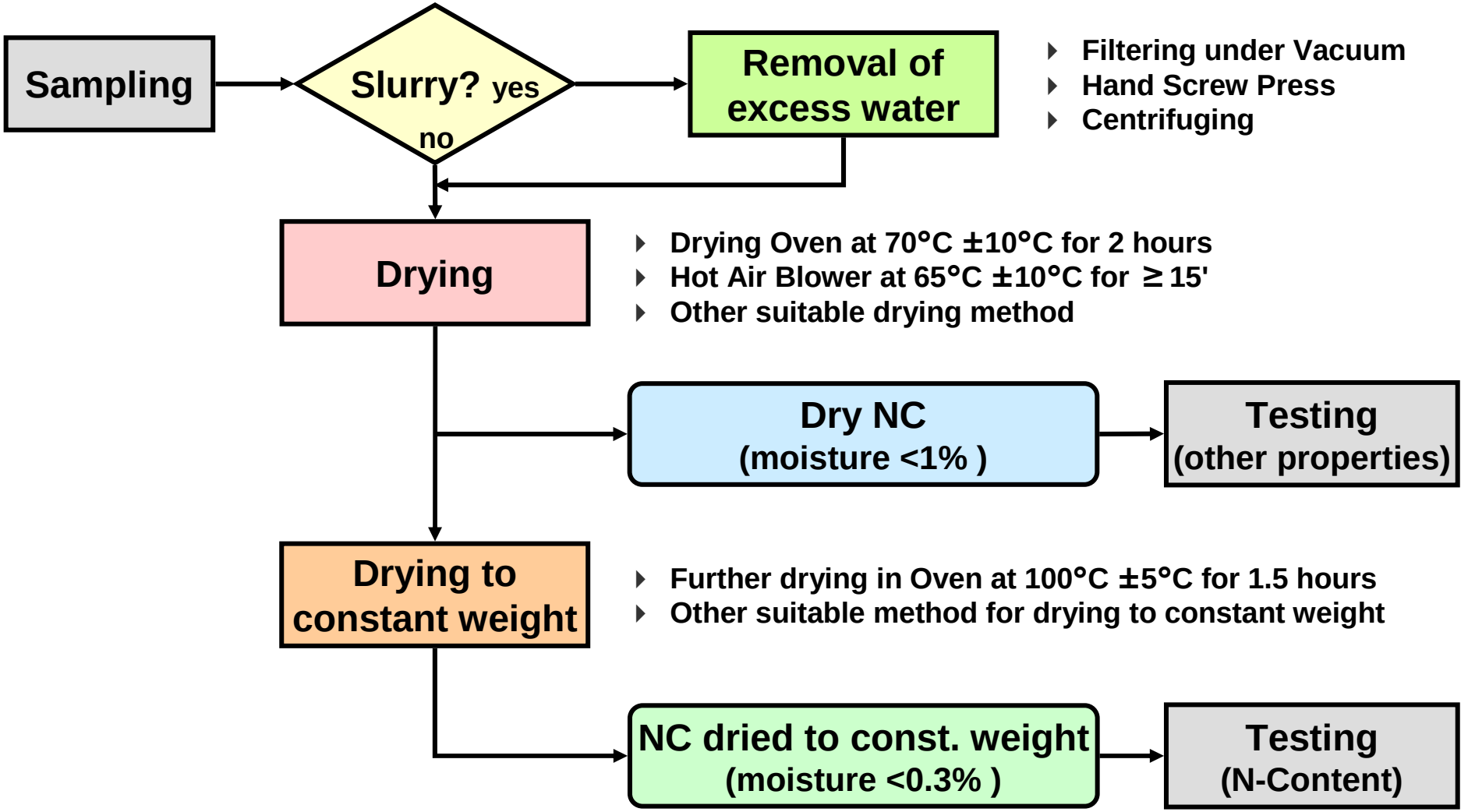
The Quality Control

- **STANAG 4178 sets quality control requirements:**
  - **Laboratory shall operate a quality system**
  - **Each test procedure has to be validated regarding:**
    - **Precision: "Repeatability Standard Deviation" s_r as determined from at least 12-fold analysis must not exceed given limit**
    - **Accuracy: "Bias" = deviation of main value of validated method from reference value (e.g. result of ref. method) must not exceed given limit**
  - **Results of validation have to be documented**

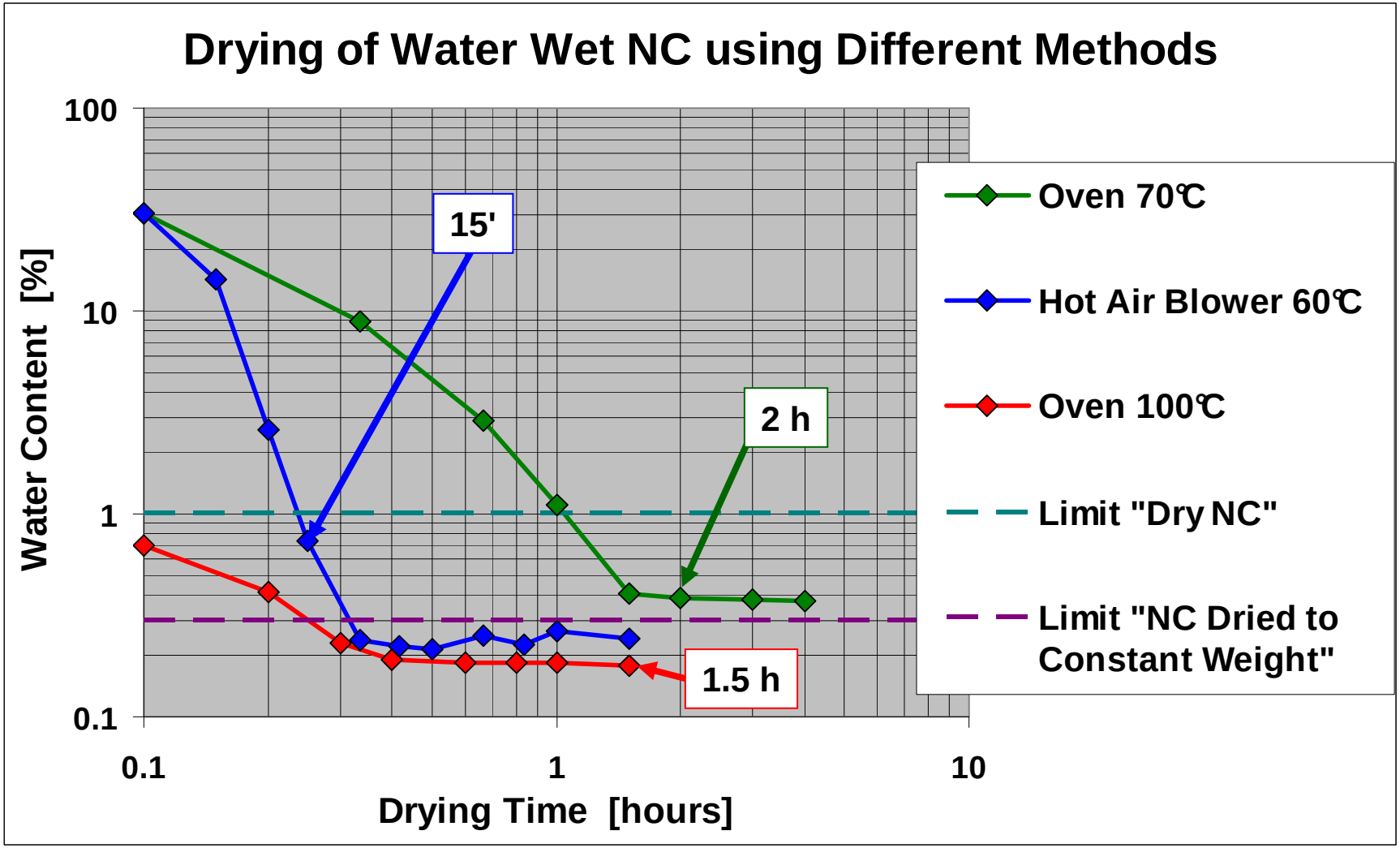
$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$



The Sample Preparation



The Sample Preparation – Drying Methods



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

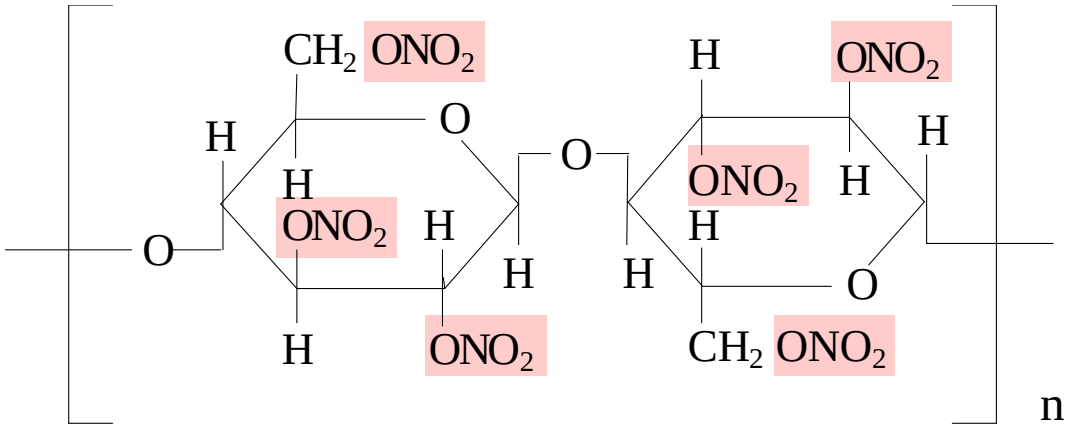
- All other tests are not mandatory and should only be performed if regarded as necessary or if requested by contract or customer

The Test Procedures

Basic Characterisation Tests

- Nitrogen Content
- Ether-Alcohol Solubles
- Acetone Insolubles

The Test Procedures – Nitrogen Content / Distribution



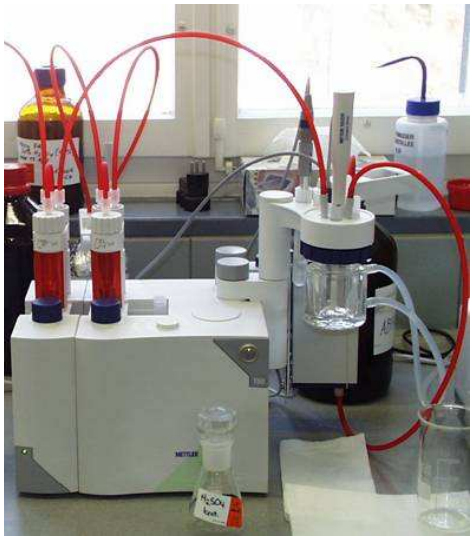
x	N-Content	NC Type
6	14.15%	--- (not achievable)
5.5	13.45%	Guncotton
5	12.75%	Pyrocellulose
4.5	11.96%	Lacquer Grade NC
4	11.11%	
3.5	10.16%	

x = number of nitrated hydroxyl groups per structure unit

- **Nitrogen Content (degree of nitration) is the most important attribute of NC; it determines most physical and chemical properties (e.g. energy content, flame temperature, solubility, density, reactivity, ….)**
- **Nitrogen Content Tests determine the average Nitrogen Content of the NC**
- **Ether-Alcohol Solubles and Acetone Insolubles give some information regarding Distribution of Nitrogen Content (→ quality of nitration)**

The Test Procedures – Average Nitrogen Content

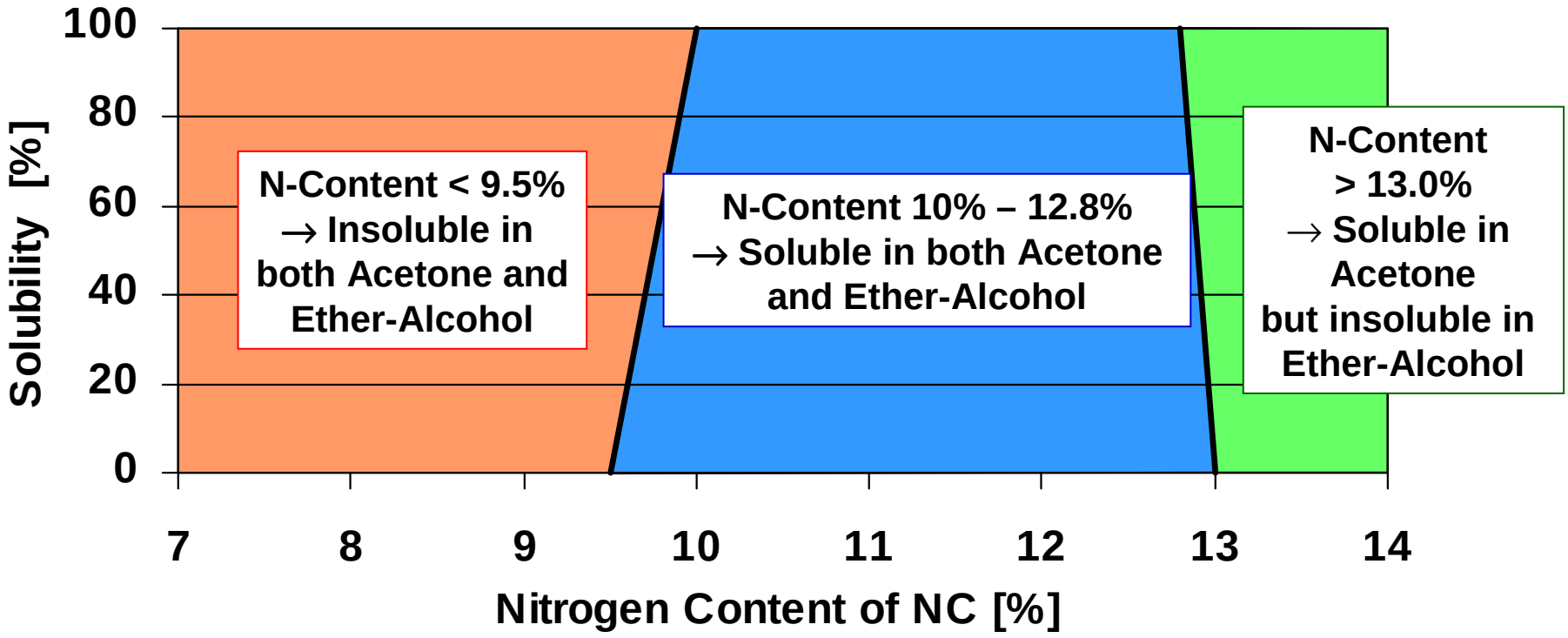
- **Nitrogen Content is a mandatory test – it determines the average degree of nitrate ester substitution and thus the energy content of the NC**
- **As an intrinsic property of the NC, it can be determined in different ways – the following methods are considered to be equivalent and can be used:**
  - **Ferrous Ion Titration Methods FS/FAS (reference method; direct method)**
  - **Nitrogen Analyzer Method (alternative direct method)**
  - **Combustion Calorimetry Method (indirect method; allows 10x larger samples)**



- **Also accepted are the 3 traditional methods:**
  - **Devarda's Alloy Method**
  - **Schulze-Tiemann Method**
  - **Nitrometer Method (not recommended method; uses mercury!)**
  - **These 3 methods have to be performed following approved national standards (or STANAG 4178 Ed. 1) and must fulfill quality requirements**

The Test Procedures – Solubility of NC in Solvents

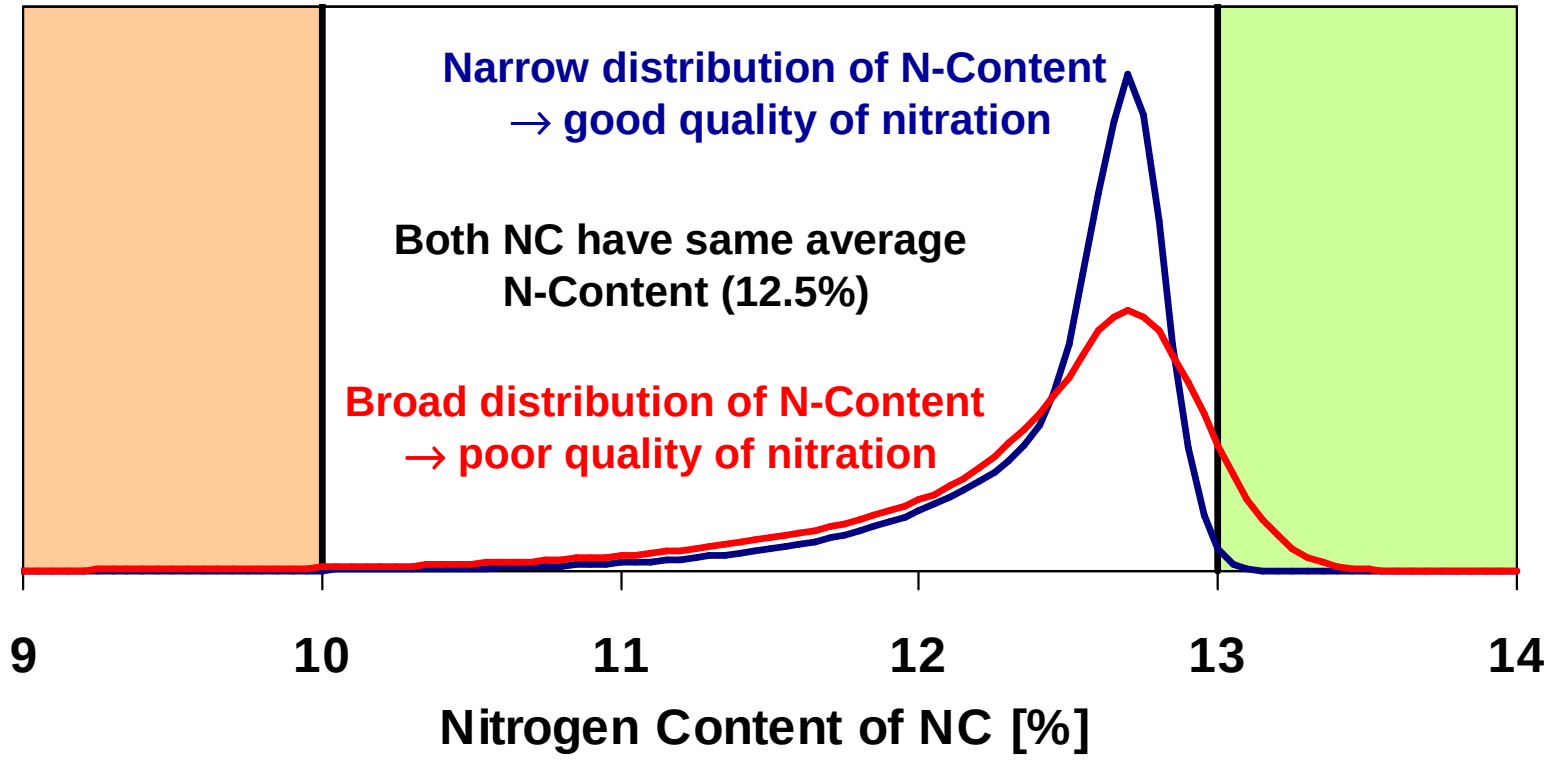
- The solubility in Ether-Alcohol (diethyl ether / ethyl alcohol) and Acetone depends on the Nitrogen Content of the NC
- Ether-Alcohol Solubles and Acetone Insolubles thus give some information regarding Distribution of Nitrogen Content (→ quality of nitration)



The Test Procedures – Solubility of Pyrocellulose

Acetone Insolubles
<0.20% = "excellent"
0.20% – 0.40% = "good"
>0.40% = "marginal"

Ether-Alcohol Insolubles
<0.7% = "excellent"
0.7% – 1.5% = "good"
>1.5% = "marginal"



The Test Procedures – Solubility of Pyrocellulose

Acetone Insolubles
<0.20% = "excellent"
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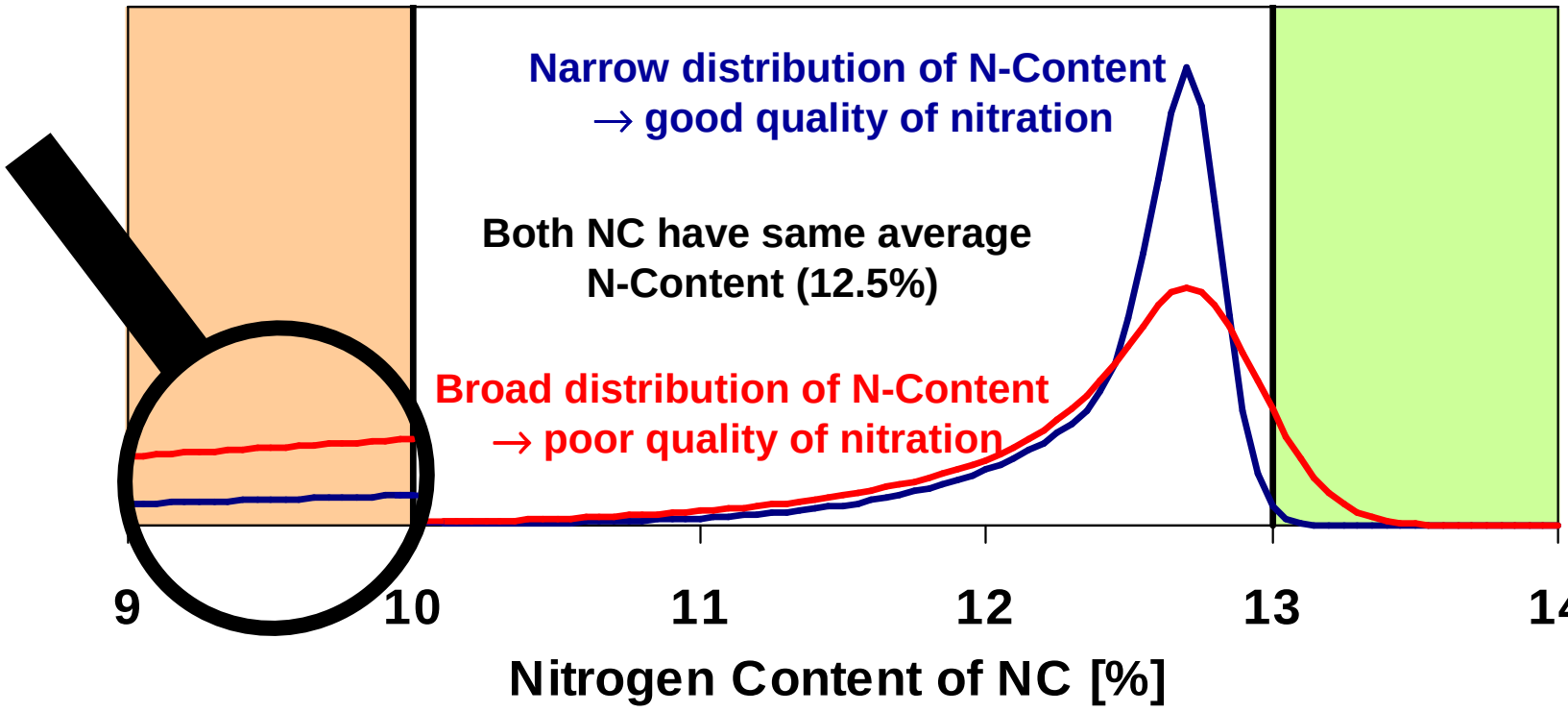
Ether-Alcohol Insolubles
<0.7% = "excellent"
0.7% – 1.5% = "good"
>1.5% = "marginal"



The Test Procedures – Solubility of Guncotton

Acetone Insolubles

<0.05% = "excellent"

0.05% – 0.20% = "good"

>0.20% = "marginal"

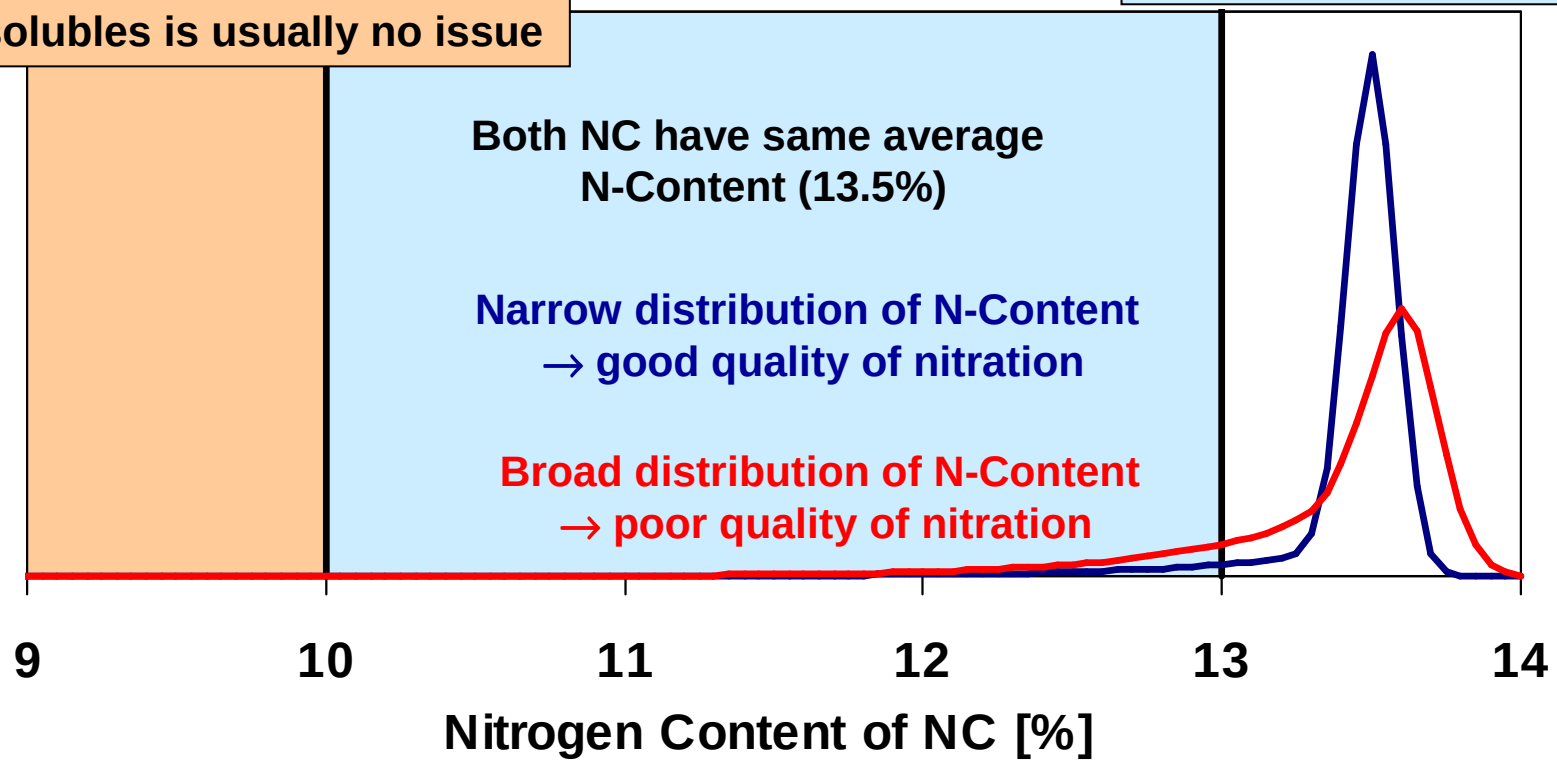
→ for guncotton; Acetone Insolubles is usually no issue

Ether-Alcohol Solubles

<5% = "excellent"

5% – 7% = "good"

>7% = "marginal"



The Test Procedures – Solubility of NC Blends

Acetone Insolubles

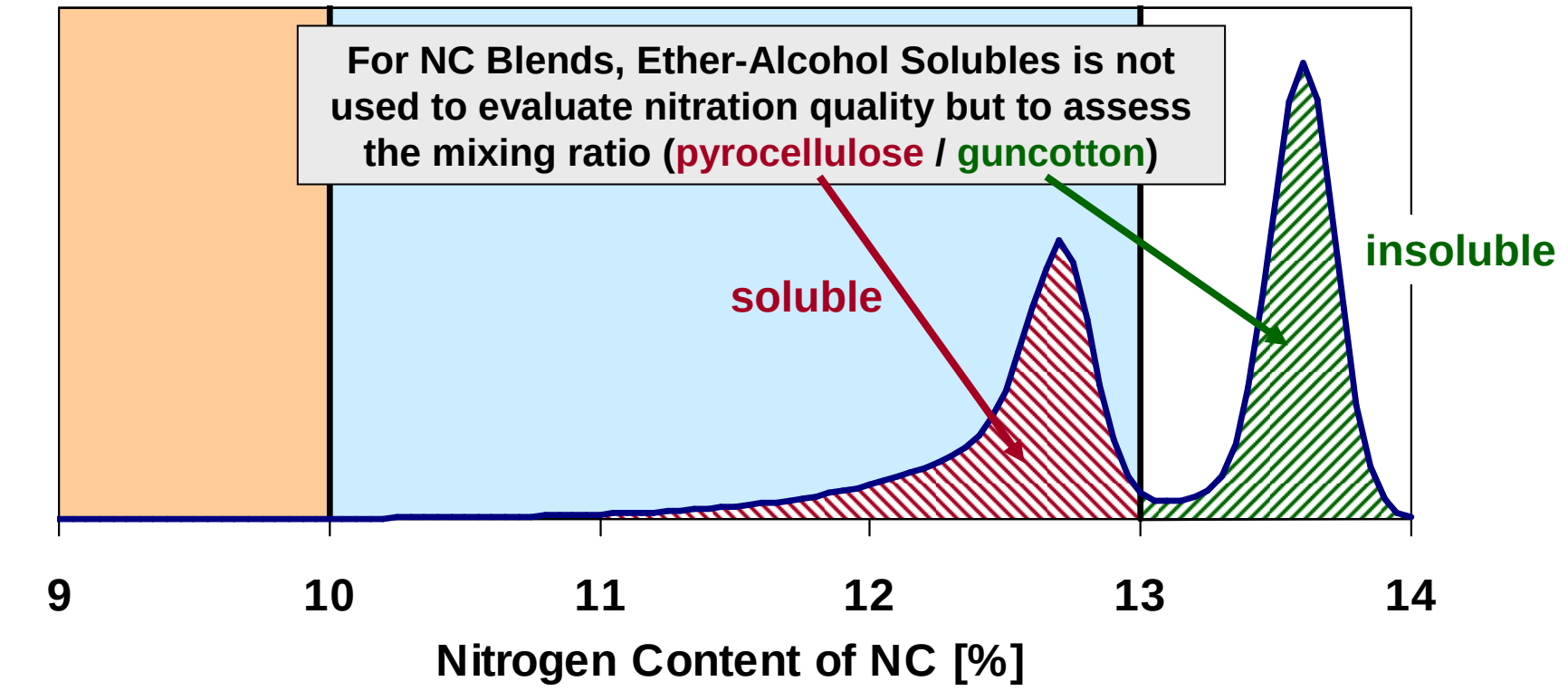
<0.15% = "excellent"

0.15% – 0.30% = "good"

>0.30% = "marginal"

Ether-Alcohol Solubles

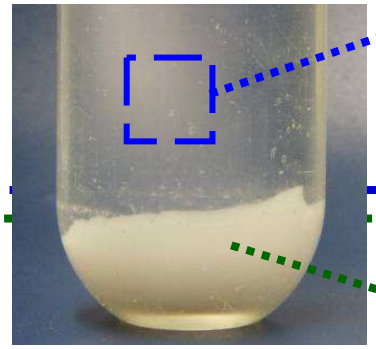
typical 20% – 50%



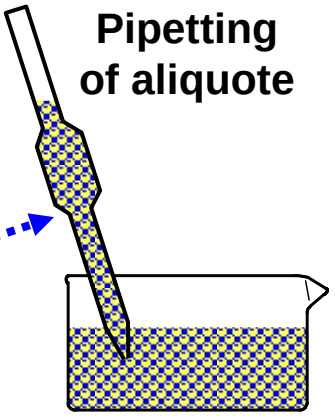
The Test Procedures – Solubility Tests: Experimental

- **Solubility of NC in given solvents (e.g. Ether-Alcohol or Acetone) can be determined in 2 different ways:**

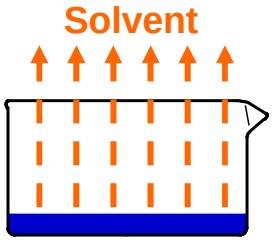
- **By assessing the part of the NC which is dissolved ("Evaporation Method")**



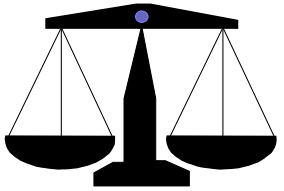
Pipetting of aliquote



Evaporation of solvent / drying

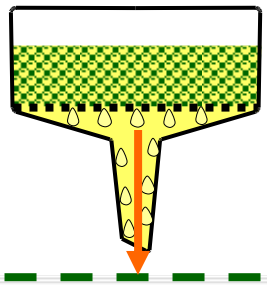


Weighing of dry residue

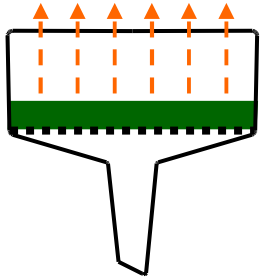


- **By assessing the part of the NC which remains undissolved ("Filtration Method")**

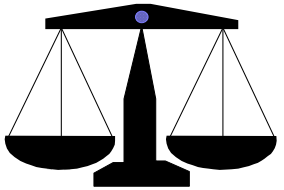
Filtration



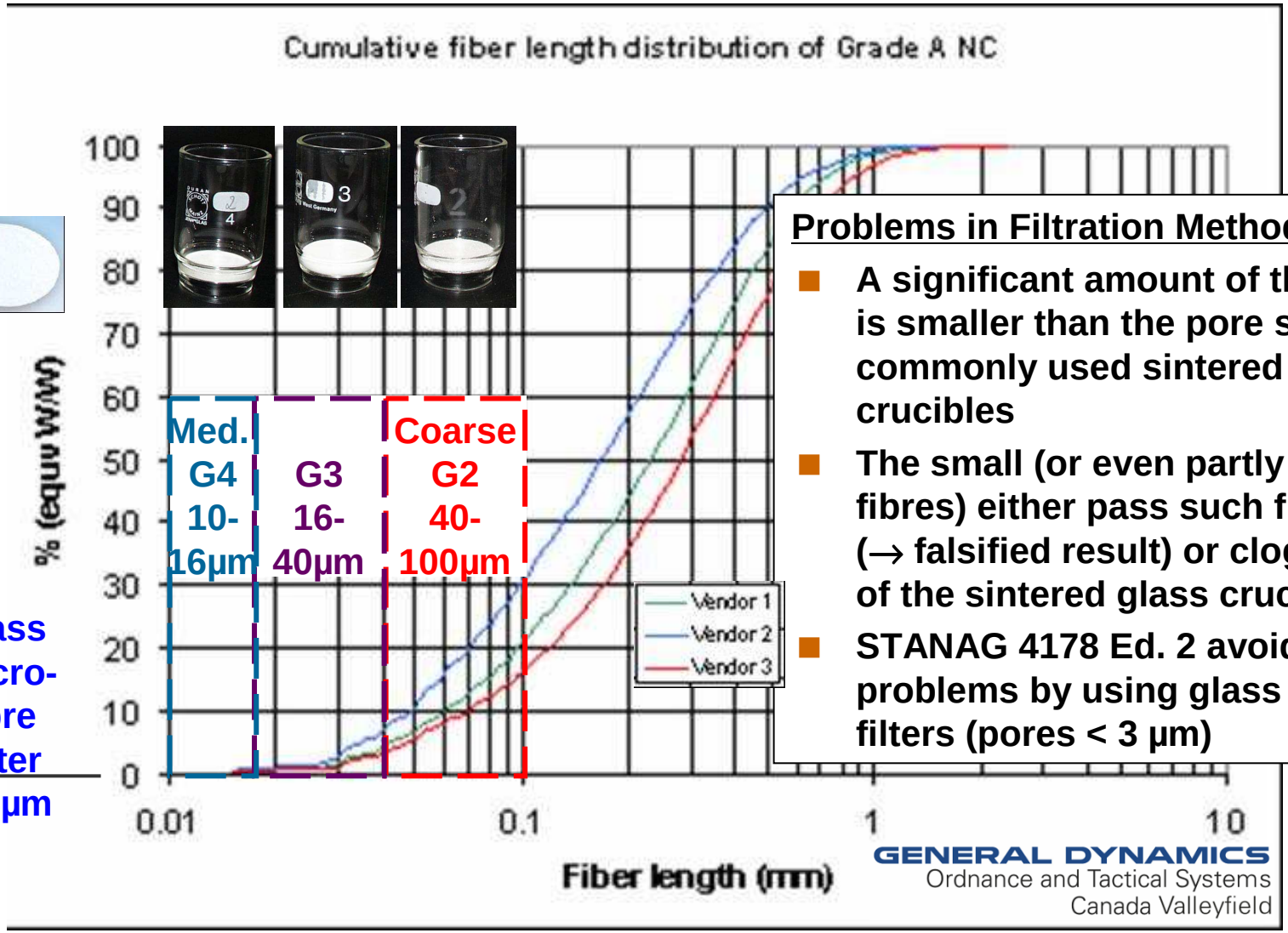
Drying of filtration residue



Weighing of dry residue



The Test Procedures – Solubility Tests: Filtration Method



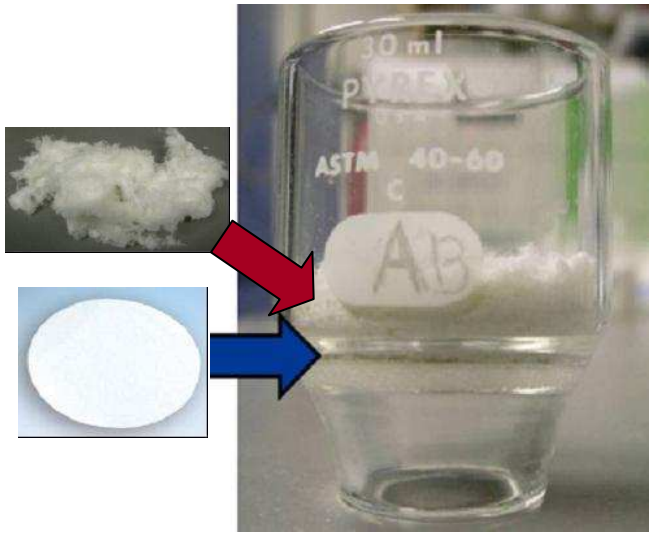
Problems in Filtration Method:

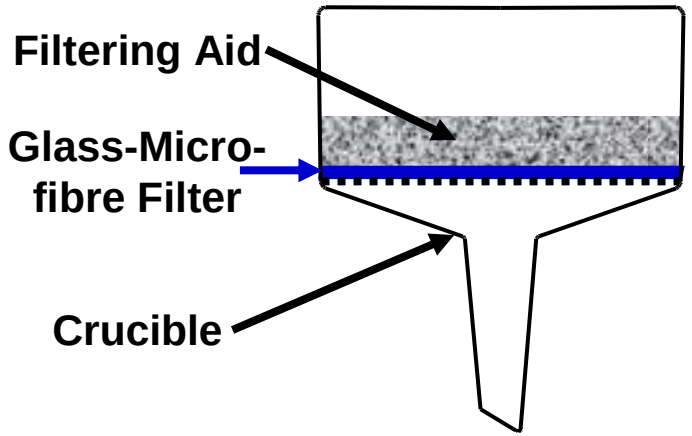
- **A significant amount of the NC fibres is smaller than the pore size of the commonly used sintered glass crucibles**
- **The small (or even partly colloidal fibres) either pass such filters (→ falsified result) or clog the pores of the sintered glass crucibles**
- **STANAG 4178 Ed. 2 avoids these problems by using glass micro-fibre filters (pores < 3 µm)**

The Test Procedures – Solubility Tests: Filtration Method

Filter Set-Up:

- **Gooch or sintered glass/silica crucible**
  - Only needed to support / hold the glass-microfibre filter
- **Glass-microfibre filter <3 µm**
  - E.g. Whatman 934/AH
- **Filtering Aids:**
  - Only if necessary (slow filtration)
  - Effective filtration aids are:
    - Aluminium Silicate Hydrate: Kaowool (kaolin-based refractory fibre)
    - Aluminium Silicate Fibres for Gooch Crucibles, e.g. Sigma-Aldrich 06416
    - Aluminium Oxide Fibres for Gooch Crucibles, e.g. Merck 1.15754
    - Glass Wool





The Test Procedures

Stability Tests

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

The Test Procedures – Chemical Stability

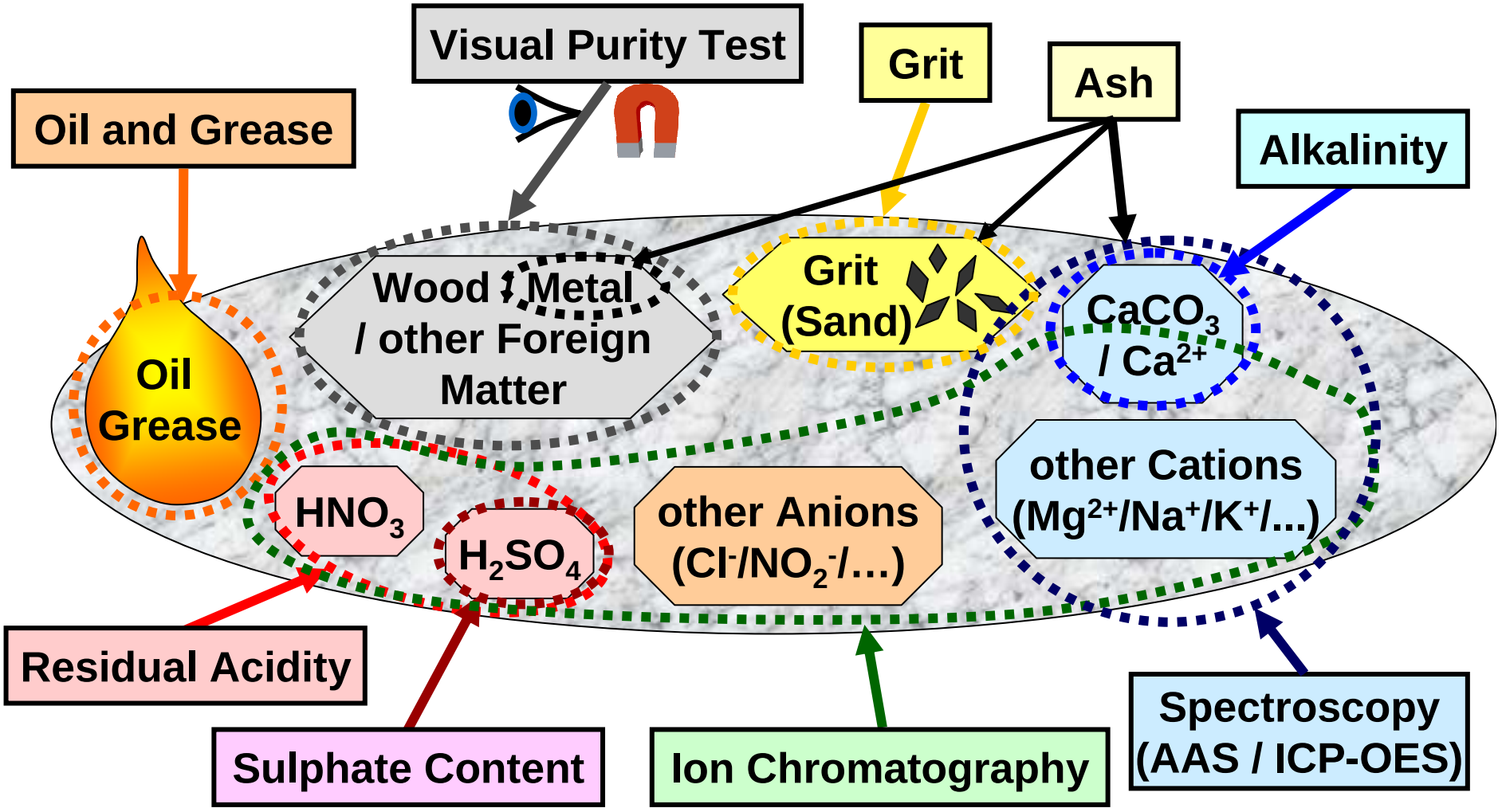
- **Chemical Stability is the most important safety-relevant property – it reveals whether a nitrocellulose lot can be safely stored and transported or not**
- **Testing of Chemical Stability is mandatory**
- **Chemical Stability can be determined by**
  - **132°C Stability Test ("Bergmann-Junk-Type Tests"); preferred method**
  - **134.5°C Heat Test ("Methyl Violet Paper Test")**
- **132°C Stability Test:**
  - **Is based on test described in STANAG 4178 Ed. 1 (Section 9)**
  - **Preferred method (since quantitative and more reliable that Heat Test)**
  - **More expensive and time consuming than 134.5°C Heat Test**
  - **Test 5A: Direct Titration Method; easier and more precise variant, not applicable to chalked nitrocellulose**
  - **Test 5B: Back Titration Method; more elaborate variant, corrects for influence of chalk; thus applicable to all types of nitrocellulose**
  - **Test 5C: Bergmann-Junk-Siebert Test variant; similar to Test 5A**
- **134.5°C Heat Test ("Methyl-Violet Test"):**
  - **From MIL-DTL-244C**
  - **Easy to perform and fast**
  - **Test result strongly depends on quality of heat test paper**

The Test Procedures

Purity Tests

- Visual Purity Test
- Ash
- Grit
- Ionic Impurities
- Oil and Grease Content
- (Abel-Type Heat Tests)

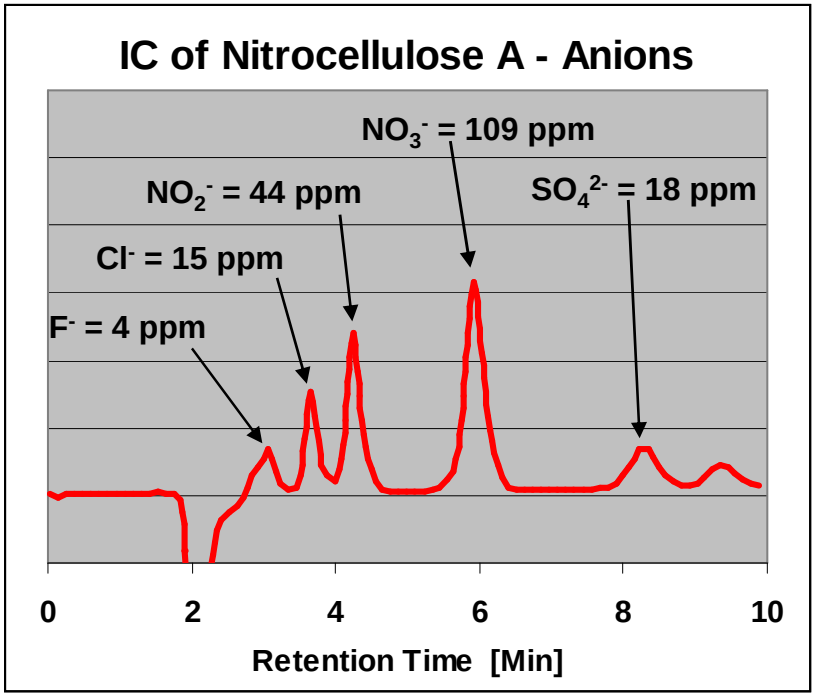
The Test Procedures – Purity Tests (Overview)

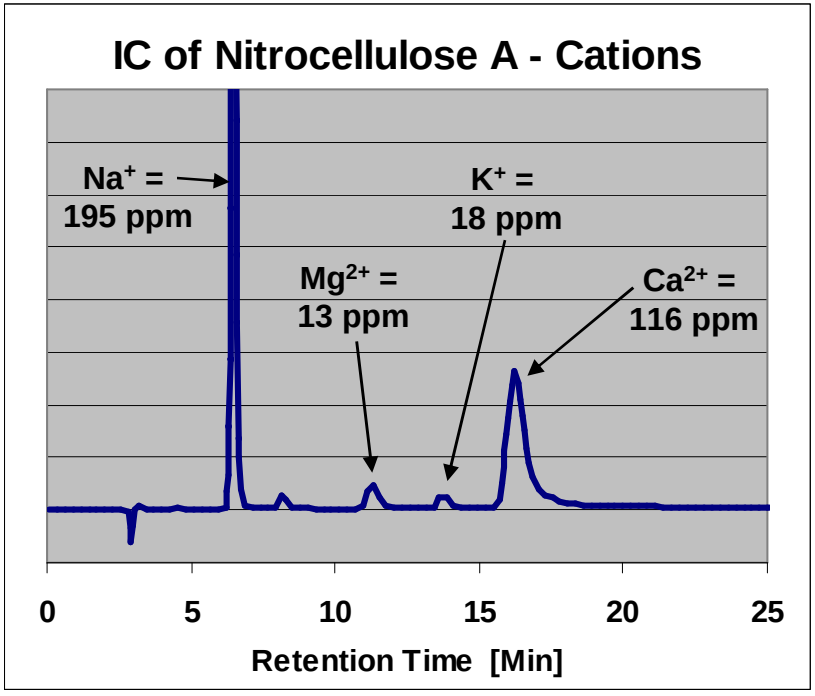


The Test Procedures – Ion Chromatography Method

- **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 SMP



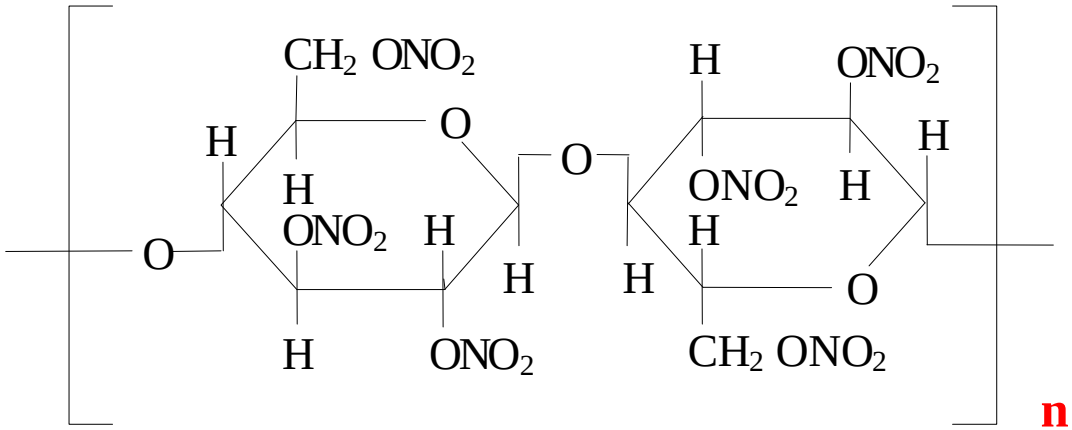


The Test Procedures

Test for Polymeric Properties

- **Viscosity**
- **Molecular Mass Distribution**

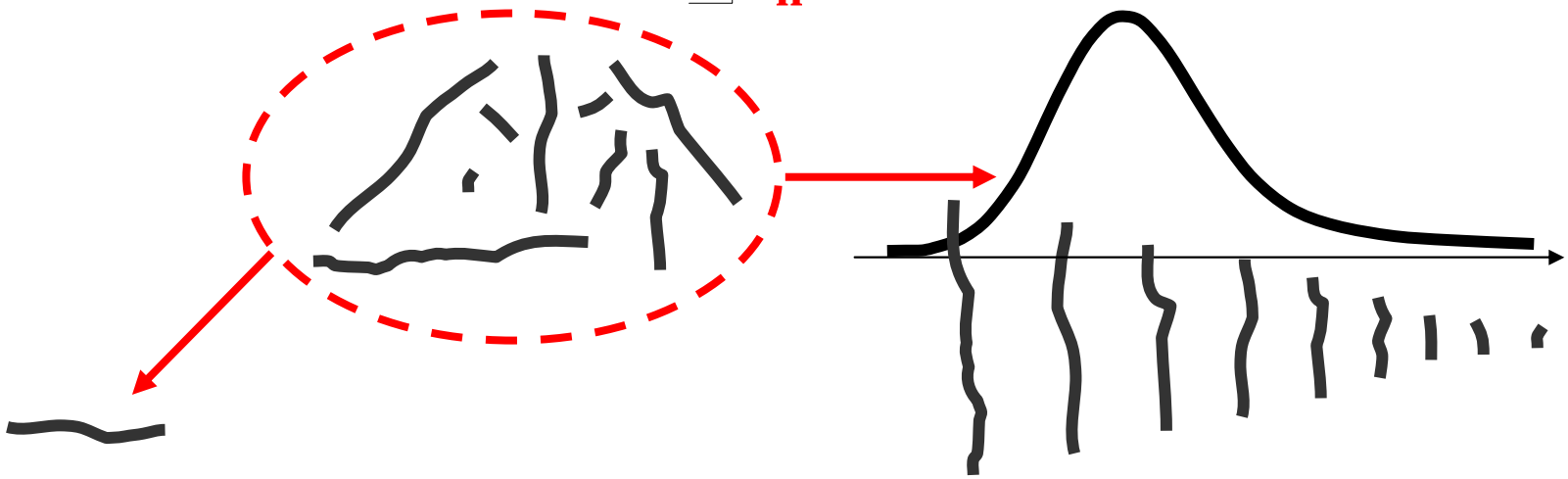
The Test Procedures – Polymeric Properties



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**

Additional Slide with more detailed Information

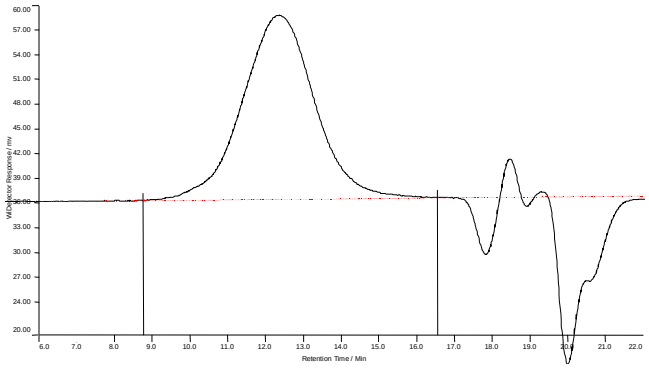
The Test Procedures – Viscosity

- The Viscosity
  - is an indirect measure for the degree of polymerisation which itself can be regarded as being the most important polymeric property of the NC
  - correlates at least to some extent with the processability of the NC
- Testing of Viscosity is not mandatory
- Testing Method:
  - From MIL-DTL-244C
  - Principle: The Viscosity is determined by measuring the time duration required for medium sized steel balls to vertically fall for a defined distance through a NC solution of defined concentration and temperature (falling sphere viscometer)
- Comments:
  - At the time being, numerous different methods are used to assess viscosity of NC
    - Falling sphere viscometers (MIL / UK M-Methods) and Höppler viscometer
    - Tube viscometers (Ubbelohde / Baume BNC) → intrinsic viscosity
    - Rotation viscometers (Brookfield in UK AWRE Specification HR 1843)
  - Results on the different tests cannot be converted into each other !
  - The MIL-DTL-244C falling sphere method might not be best suited from scientific point of view, but it this method is very simple and most widespread
  - Problem: Falling time is too short (and thus precision of viscosity measurement insufficient) for certain low viscosity NC grades → Hoeppler Method recommended for these NC types

Additional Slide with more detailed Information

The Test Procedures – Molecular Mass Distribution

- **The Molecular Mass Distribution (or distribution of degree of polymerisation) is the most important polymeric property of the NC**
- **Testing of Molecular Mass Distribution is not mandatory**
- **Testing Method:**
  - **Method is based on collaborate work undertaken at the AWE UK, Cranfield University UK, Fraunhofer ICT Germany, TNO Netherlands and others**
  - **Principle: Gel Permeation Chromatographic (GPC) analysis of a solution of the NC using a concentration sensitive detector (refractive index RI or UV/VIS); measurement relative to a given standard (e.g. polystyrene)**
  - **Test is more costly and time-consuming than the simple viscosity test**
  - **GPC analysis provides additional structural information that can be related to the physical properties of NC**



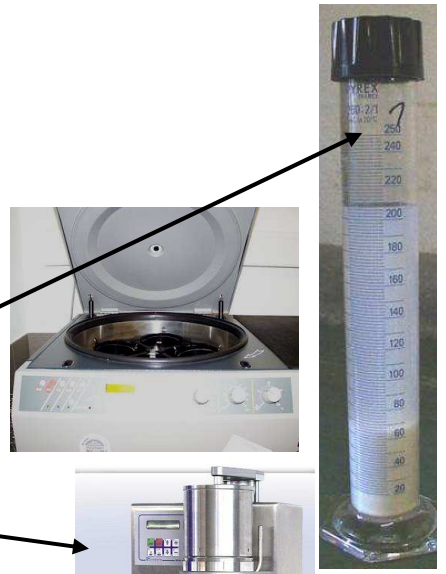
The Test Procedures

Tests for Fibre Quality

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

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**
- **Until recently, only indirect methods were available:**
  - **Fineness / Settling Volume (STANAG 4178 Ed2 Test 14)**
  - **Liquid Retention / Water Retention Value (STANAG 4178 Ed2 Test 16A)**
  - **Drainability (Schopper Riegler / Canadian Standard Freeness; STANAG 4178 Ed2 Test 16B)**
- **It is well known that the indirect methods do not always correlate with the amount of grinding applied – in particular the Fineness versus cutting curve levels off at a certain stage from which on further cutting does no longer reduce Fineness despite Fibre Length further decreases**
- **Nowadays, the Fibre Length can be directly determined using sophisticated Fibre Quality Analyzers (STANAG 4178 Ed2 Test 16C)**



The Test Procedures – Fibre Length Determination

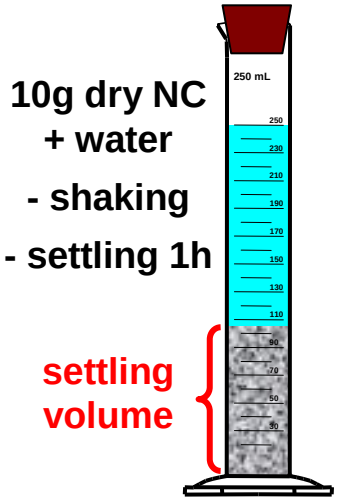
The Fibre Length of NC is a process relevant property as it reveals the amount of cutting / grinding / refining the NC had undergone during manufacture

Indirect Methods

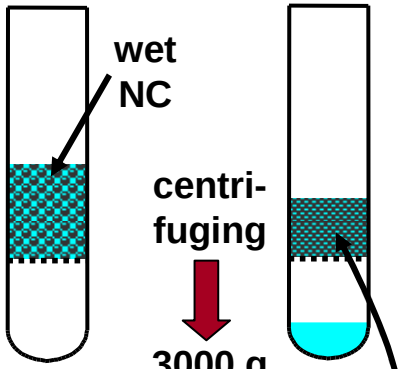
- Fineness
- Water Retention Value WRV
- Drainability

Direct Method

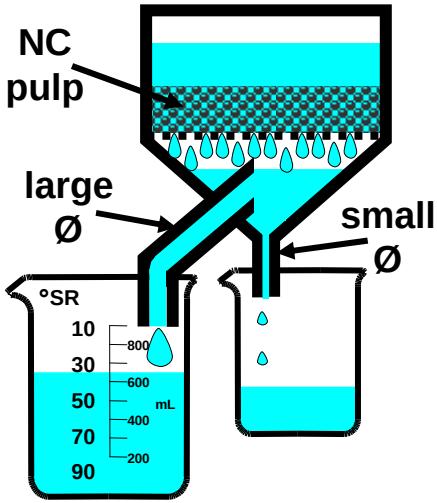
- Fibre Length / Shape Analyzer



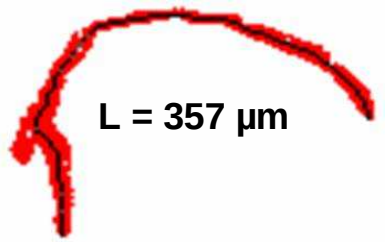
→ NC settling volume



→ Water retention value = water content of centrifuged NC



→ Drainability = rate of dewatering of pulp

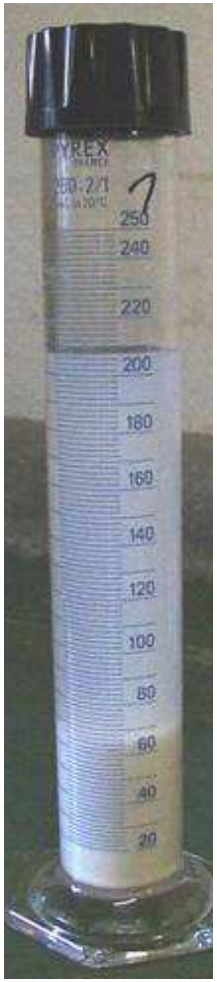


→ NC average fibre length and distribution

Additional Slide with more detailed Information

The Test Procedures – Fineness

- **The Fineness Test (Settling Test)**
  - **is an indirect measure for the fibre length of the NC**
  - **correlates at least to some extent with the processability of the NC**
- **Testing of Fineness is not mandatory**
- **Testing Method:**
  - **From MIL-DTL-244C**
  - **Principle: The Fineness test is based on making aqueous NC slurry, followed by settling of fibres in a graduated cylinder and recording of the volume occupied by the NC fibres after a specified settling time**
  - **Test method is fast and easy to perform and thus well suited for routine testing of NC**



Additional Slide with more detailed Information

The Test Procedures – Water Retention Value WRV

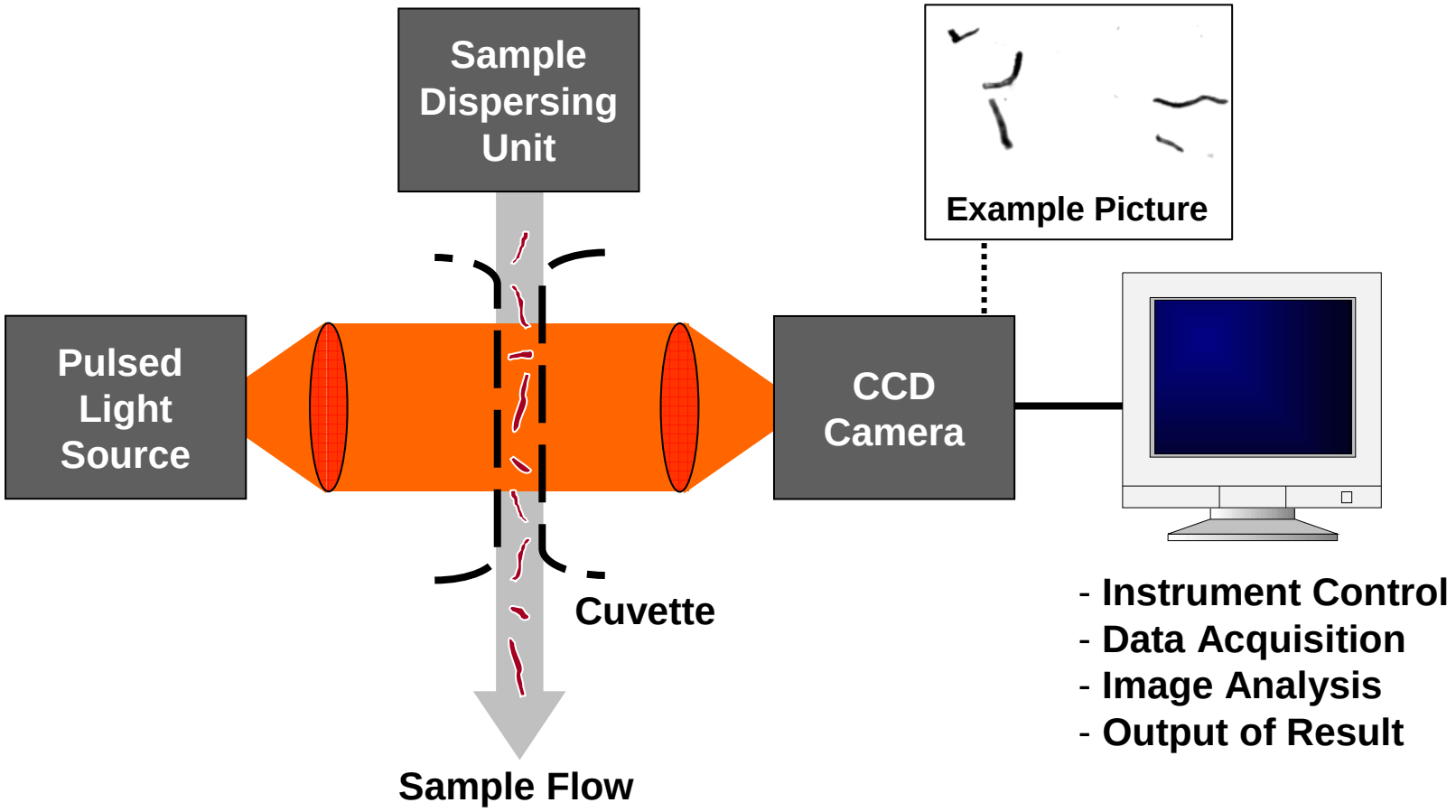
- **The Water Retention Value Test checks the ability of the NC fibres to retain solvents – in particular NC made from new raw materials may differ in this property**
- **Testing of Water Retention Value is not mandatory**
- **Testing Method:**
  - **Test to be performed according to ISO 23714:2007 'Pulps – Determination of Water Retention Value'**
  - **Principle: The NC is wetted with water and centrifuged in a cylindrical container which is screened at the bottom side – the weight fraction of water which is retained by the NC is assessed by weighing**

Additional Slide with more detailed Information

The Test Procedures – Drainability

- **The Drainability Test provides a measure of the rate at which a dilute suspension of pulp may be dewatered – it has been shown that the Drainability is related to the surface conditions and swelling of the fibres, and constitutes a useful index of the amount of mechanical treatment (grinding) to which the fibres have been subjected**
- **Test is widely used in paper industry**
- **Testing of Drainability is not mandatory**
- **Testing Method:**
  - **Test to be performed according to ISO 5267 'Pulps – Determination of Drainability:**
    - **Part 1: Schopper-Riegler Method, or**
    - **Part 2: Canadian Standard Freeness Method'**
  - **Principle: Suspension of fibres is put on a sieve, and kinetics of dewatering is measured by continuous collection and weighing of the released water**

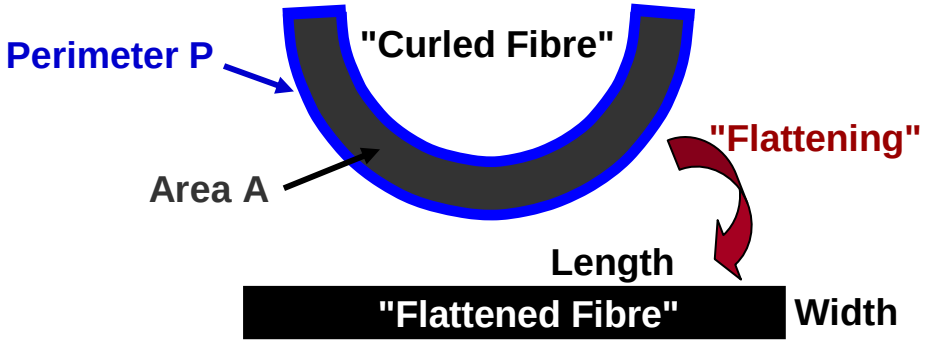
Fibre Quality Analyzers – Principle of Operation



- Instrument Control
- Data Acquisition
- Image Analysis
- Output of Result

Fibre Quality Analyzers – Image Analysis of Fibres

Fibre Model (Geodesic Length)



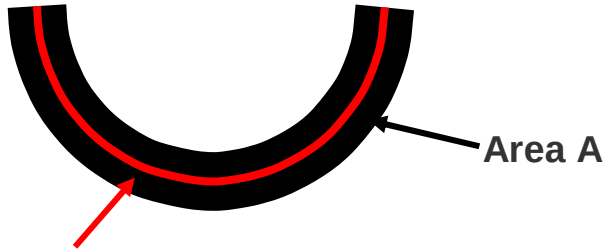
Fibre Length L and Width W are calculated from obtained Perimeter P and Area A

$$L = \frac{1}{4}\left(P + \sqrt{P^2 - 16A}\right)$$

$$W = \frac{A}{L}$$

- Easy / fast to calculate
- O.k. for simple fibre shapes
- Fails if shape becomes more complex → complex fibres must be excluded

Skeletonizing Method

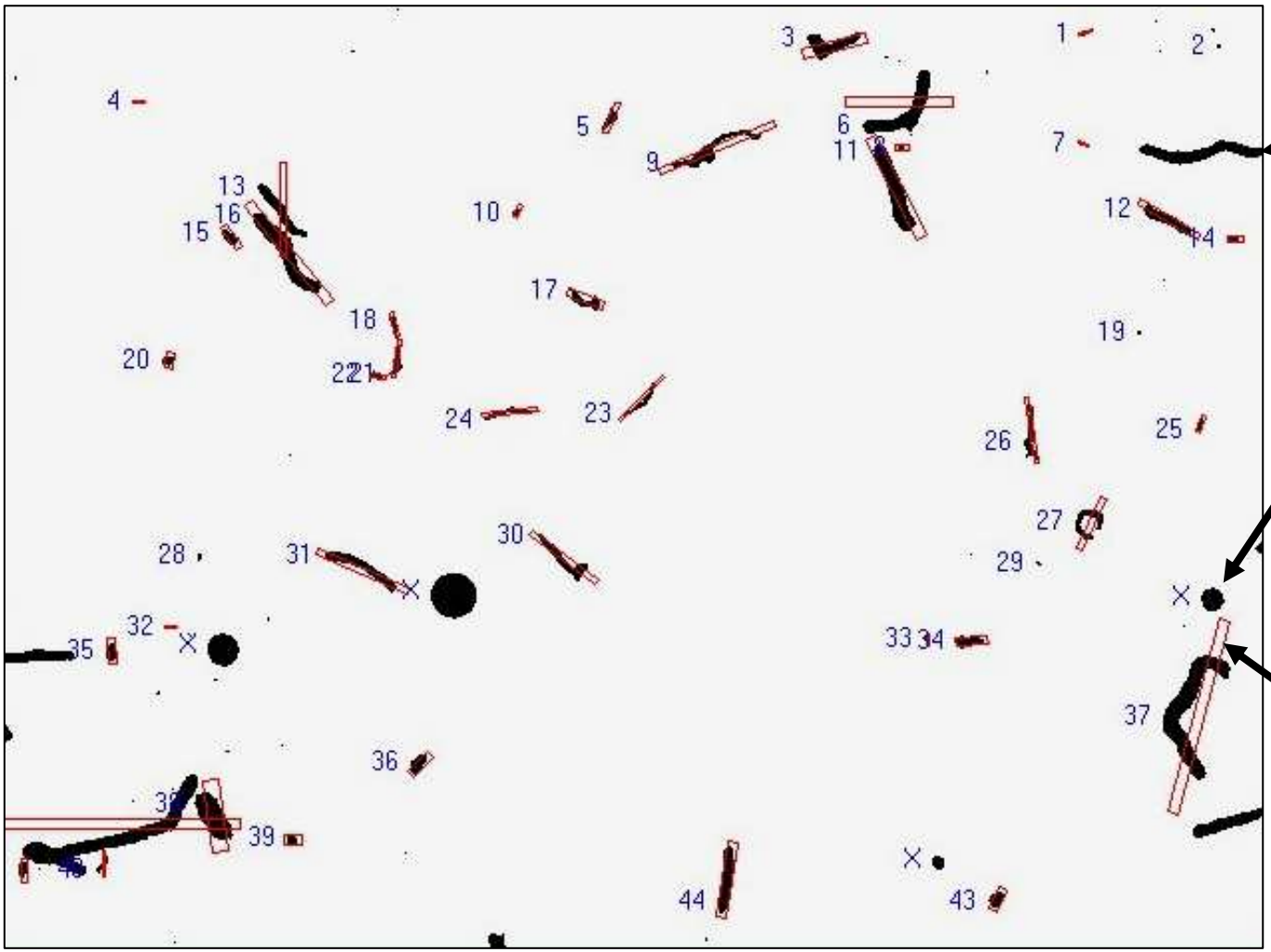


Fibre Length L is determined as longest direct way between two endpoints of the particle (fibre)

- Much more time-consuming to calculate
- Works well for both simple and complex fibre shapes → almost all particles can be included in the final assessment

Fibre Quality Analyzers – Picture Frame with Image Analysis

Example: Beckman-Coulter RapidVUE



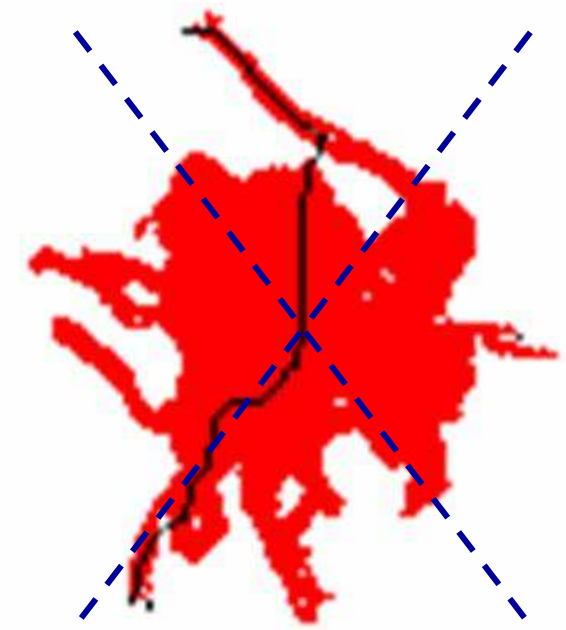
Fibre touches frame → excluded from further analysis

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

Red rectangle repre-sents flattened fibre (with calculated equivalent length and width)

Fibre Quality Analyzers – Image Analysis / Skeletonizing Method

Example: Sympatec QICPIC (Sample M2)



Agglomerates can be eliminated from analysis

Calculation of Average Fibre Length

- **As in any other particle measurement, it is possible to calculate several kinds of average (or mean) fibre lengths**
- **The most popular ones are:**
  - **Numerical Average L_n**
    - Strongly dependent on fine content

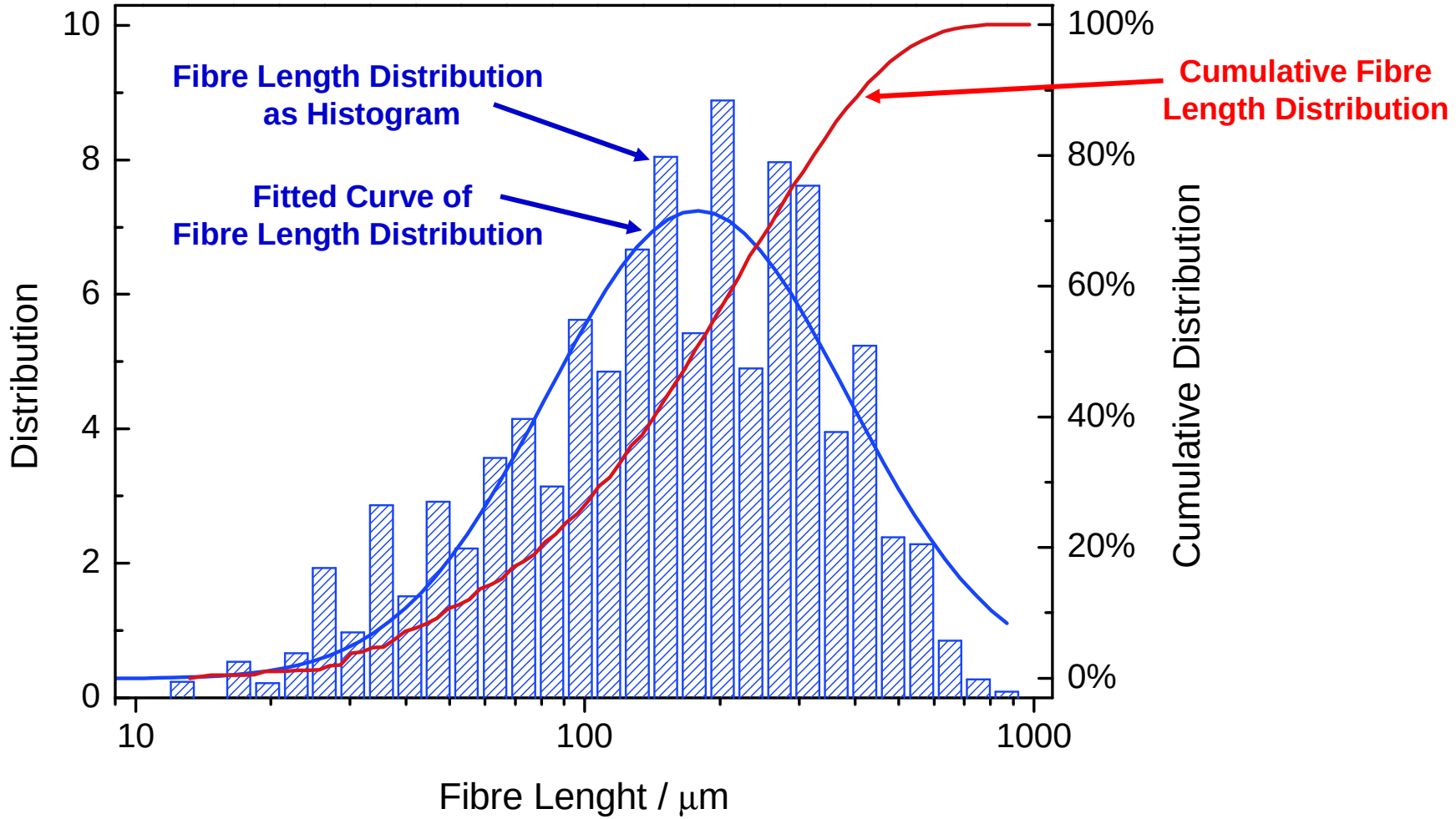
    $$L_n = \sum n_i \cdot l_i \;/\; \sum n_i$$
  - **Length-Weighted Average L_l**
    - Most often used; less dependent on fine content

    $$L_l = \sum n_i \cdot l_i^2 \;/\; \sum n_i \cdot l_i$$
  - **Area-Weighted Average L_A**
    - Less often used

    $$L_A = \sum n_i \cdot A_i \cdot l_i \;/\; \sum n_i \cdot A_i$$
  - **Volume-Weighted Average L_V**
    - Often used; represents the real volume- or weight-fractions of the respective length ranges

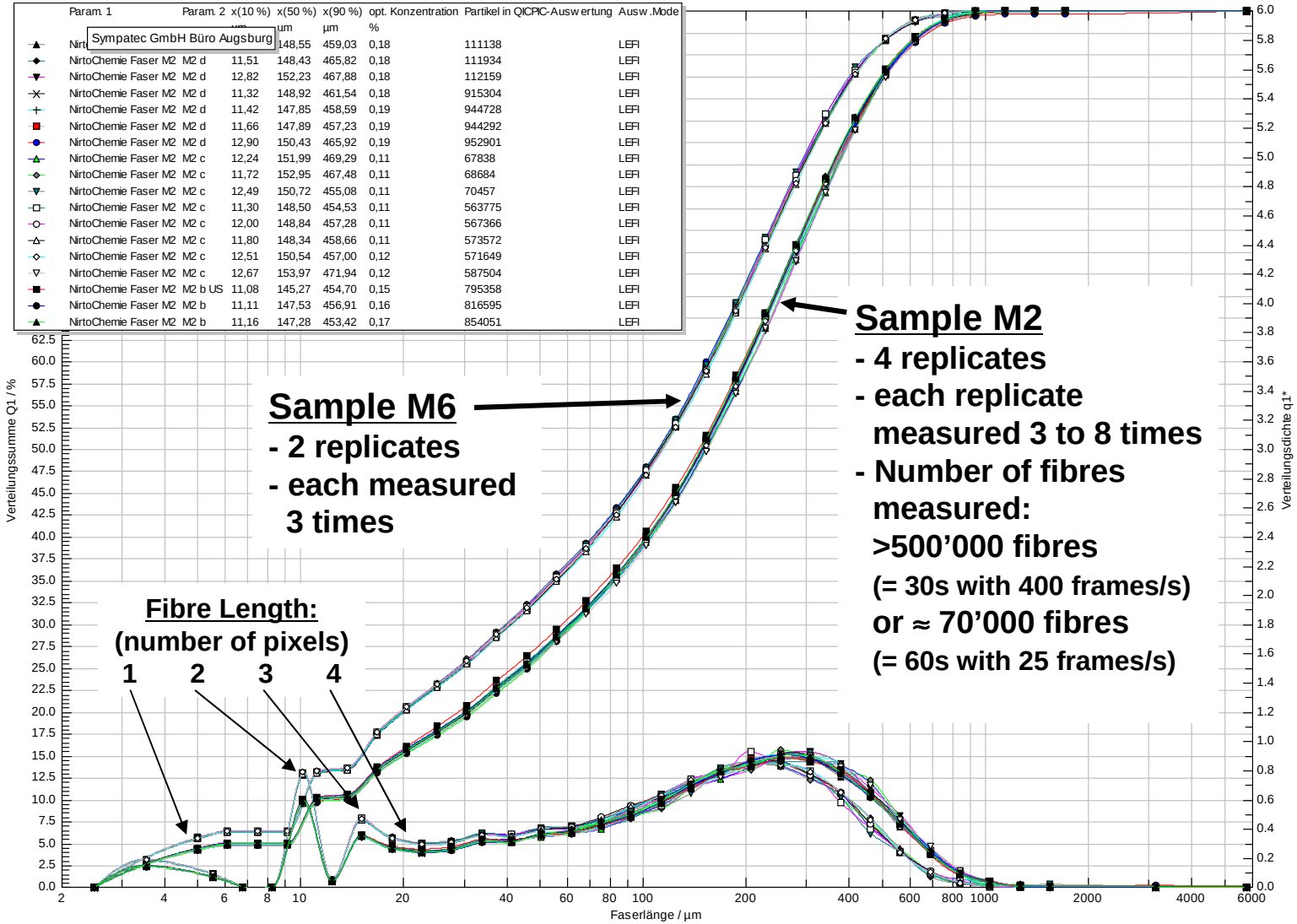
    $$L_V = \sum n_i \cdot V_i \cdot l_i \;/\; \sum n_i \cdot V_i$$
- **For cylindrical fibres which have uniform width (diameter), length-weighted, area-weighted and volume-weighted average become identical (since then, area and volume become proportional to the length)**

Fibre Quality Analyzers – Display of Results



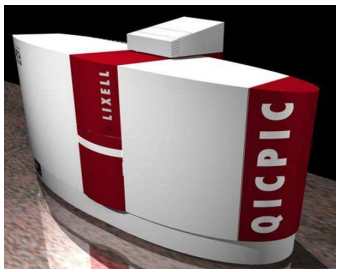
Fibre Quality Analyzers – Repeatability of Results

Param. 1	Param. 2	x(10 %) µm	x(50 %) µm	x(90 %) µm	opt. Konzentration %	Partikel in QICPIC-Auswertung	Ausw.Mode
Sympatec GmbH Büro Augsburg			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



Sympatec QICPIC

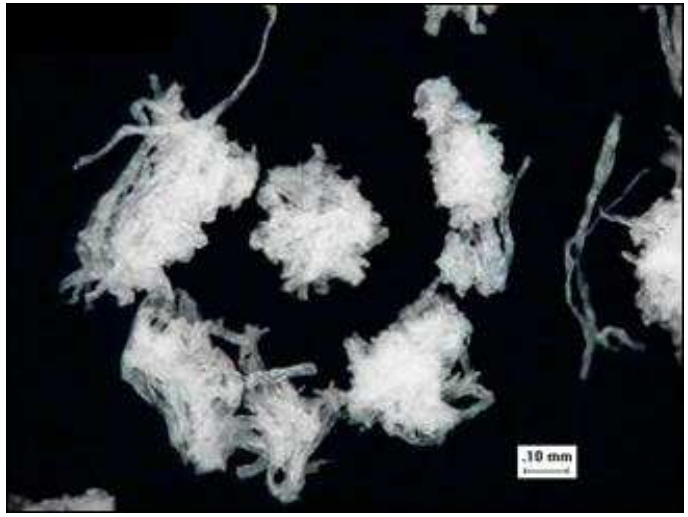
- **Excellent Repeatability (replicate analysis)**
- **Significant difference between samples M2 and M6 found (as expected)**



Additional Slide with more detailed Information

The Test Procedures – Agglomerates

- The fraction of the NC which is present as agglomerates can be determined with the Agglomerates test
- Testing of Agglomerates is not mandatory (but recommended in particular for NC produced from sheeted linters / cellulose)
- Testing Method:
  - From MIL-DTL-244C
  - Principle: The NC is shaken with water, followed by sieving, washing, drying and weighing of the fibre agglomerates
  - Test method is easy to perform and thus well suited for routine testing of NC



The Test Procedures

Tests for Water / Alcohol Content

- **Total Volatile Content**
- **Water Content**
- **Alcohol and / or Water Content**

The Test Procedures – Volatile Content

- **Due to safety reasons, NC must be transported and stored in wetted form**
- **Assessment of the contents of Total Volatiles and of the Volatile Components is:**
  - **important to know the total NC content in the wet sample**
  - **significant because the presence of excessive moisture or organic solvents can impede the gelatinization process**
- **Testing of Volatile Content is not mandatory**
- **The content of Total Volatiles or of the individual components can be determined by a variety of methods:**
  - **Methods that determine Total Volatile Content**
    - **Oven Method (at 100°C; Reference Method for Total Volatile Content)**
    - **Moisture Analyzer Method**
  - **Methods that determine Water Content**
    - **Karl Fischer Titration Method (Reference Method for Water Content)**
    - **Karl Fischer Oven Method**
  - **Methods that determine Alcohol and/or Water Content**
    - **Gas Chromatography Method**
    - **NIR Spectroscopy Method**

The Adaptations for Chalked Nitrocellulose

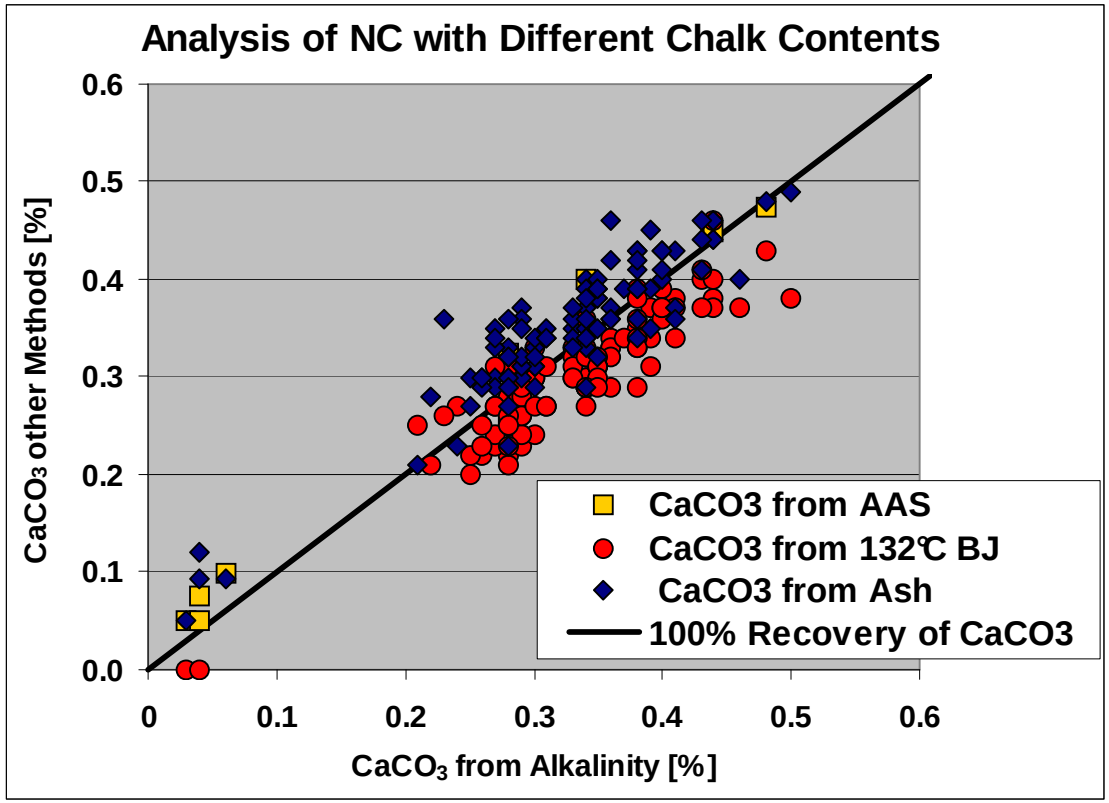
Adaptations for Chalked Nitrocellulose

The Treatment of Chalked Nitrocellulose

- Some NATO / PfP nations require that chalk ($CaCO_3$) is added to certain NC types during manufacture
- These deliberately chalked NC types contain much higher amounts of calcium carbonate (typically between 0.2% and 0.5%) which can no longer be neglected but require additional tests, alterations or corrections:
  - Calcium carbonate content: Must be additionally determined; e.g. by Alkalinity Method or Spectroscopy Method
  - Nitrogen Content Test: Correction of the result by the amount of $CaCO_3$ present
  - Ash Content: Application of higher limits due to the higher $CaCO_3$ content
  - Ether-Alcohol Solubles / Acetone Insolubles Tests: Corrections for chalk content or alteration of test procedure in Ether-Alcohol Solubles (only Filtration Method) and Acetone Insolubles are needed, as filtration is performed with glass micro-fibre filters which retain the chalk
  - 132°C Bergmann-Junk Stability Test: Use of the alternative, more elaborate and time-consuming method 5B which corrects effects arising from $CaCO_3$ content (Tests 5A and 5C are not applicable as they do not correct for chalk content)

Chalked Nitrocellulose – Calcium Carbonate Content

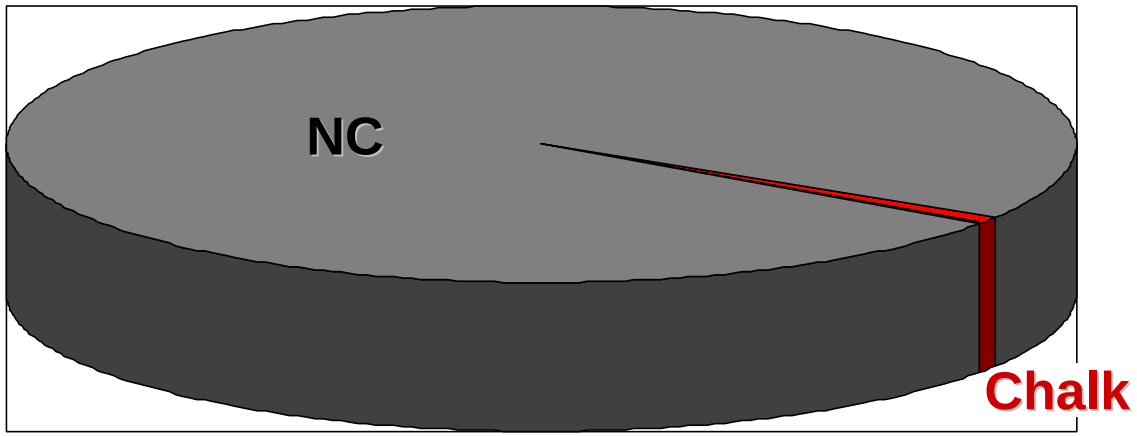
- The $CaCO_3$ content of chalked NC must be determined in order to:
  - check whether the chalk content lies within the specified range
  - as basis for the correction of the result of the nitrogen content test
- The Calcium Carbonate Content can be determined as follows:
  - **Alkalinity Method** (determination of alkaline content by acid-base titration)
  - **Spectroscopy Method** (AAS; determination of Calcium)
  - **Ash Content** (for chalked NC, Ash Content also correlates well with $CaCO_3$ content; this since ash mainly consists of CaO)
  - **Blank determination of the 132°C Stability Test** (this titration of the not heated blank NC is equivalent to the Alkalinity Method)



Chalked Nitrocellulose – Nitrogen Content

- **All test methods for Nitrogen Content determine the average N-content value of the entire sample (here of NC + $CaCO_3$)**
- **In order to obtain the Nitrogen Content of the NC-part of the sample solely, presence of calcium carbonate must be considered by the following calculation:**

$$\%\,\text{Nitrogen} = \frac{\%\,\text{Nitrogen}_{\text{uncorrected}}}{\left(1 - \frac{\%\,\text{Chalk}}{100}\right)}$$



Chalked Nitrocellulose – Ash Test

"Chalked NC"
NC + $CaCO_3$

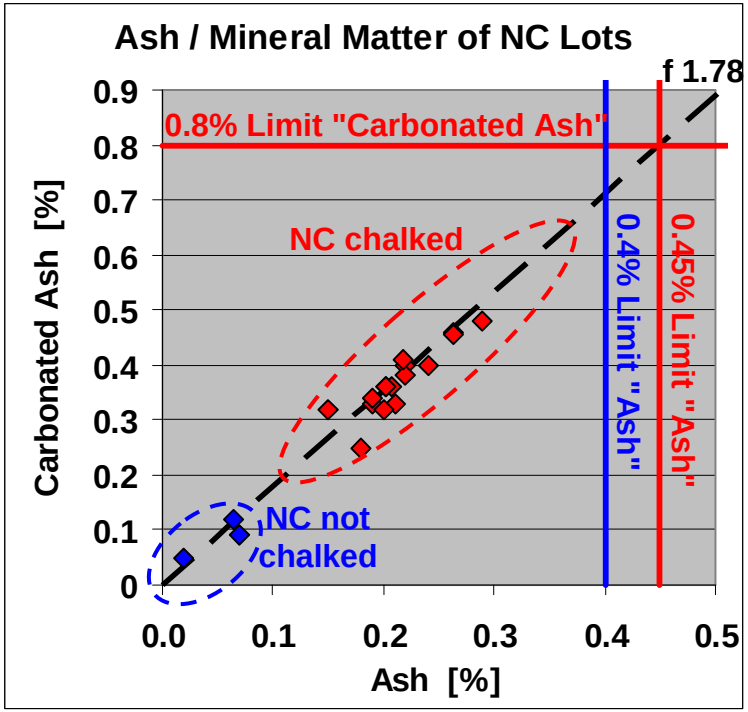
→ Ashing (Ed. 1 + 2)

"Ash"
mainly CaO

→ + $(NH_4)_2CO_3$ Carbonation (only Ed. 1)

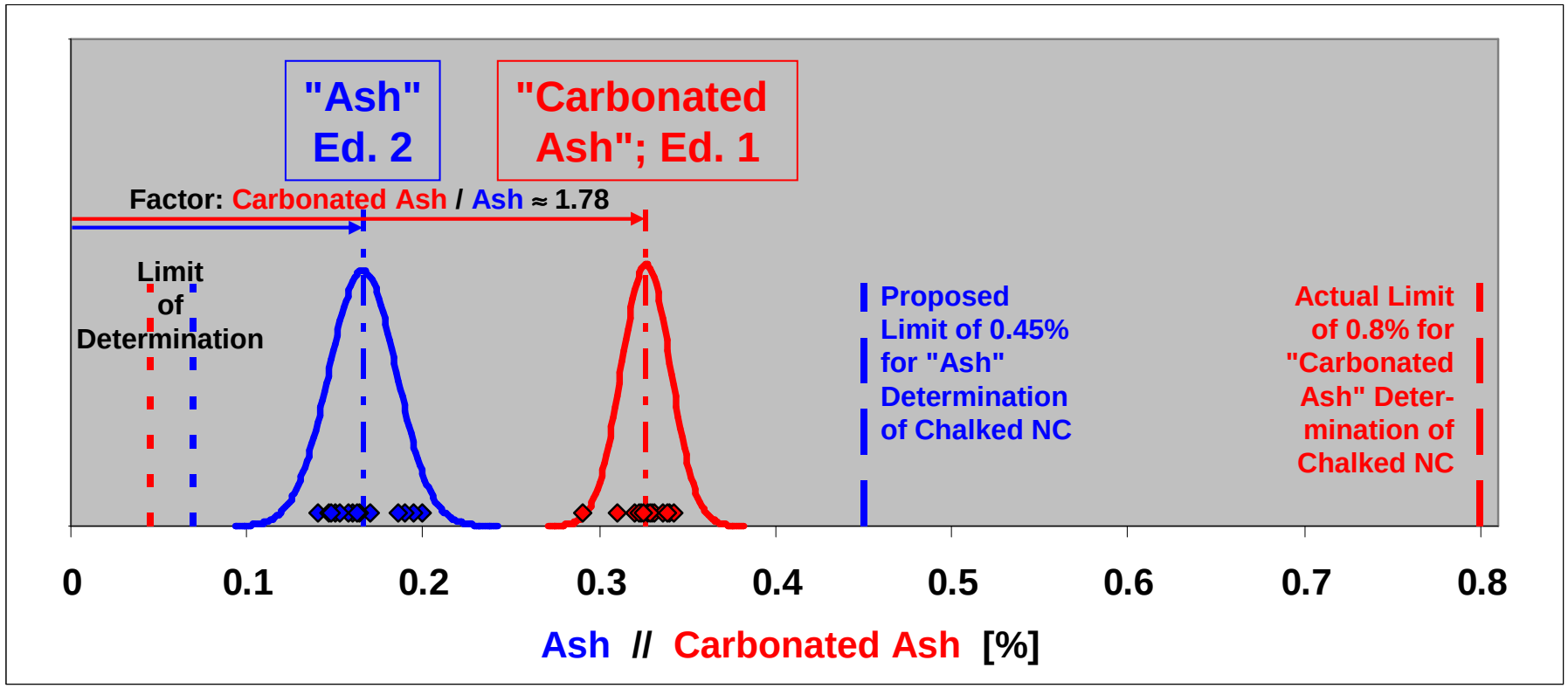
"Carbonated Ash"
mainly $CaCO_3$

- "Ash Test" of STANAG 4178 Ed. 2 finishes after the ashing / calcination step; the sub-sequent carbonation step which was optional in Ed. 1 has been deleted in Ed. 2
  - The "Carbonated Ash" value correlates with "Ash" (with stoichiometric factor 1.78 = $CaCO_3$ / CaO; this proofs that "Ash" consists mainly of CaO)
  - For not chalked NC, limit remains 0.4%
  - For chalked NC, a slightly higher "Ash" limit of 0.45% is suggested, as this is equivalent to the Ed. 1 / UK-Method's limit of 0.8% for "Carbonated Ash" (0.45% CaO is equivalent to 0.8% $CaCO_3$)



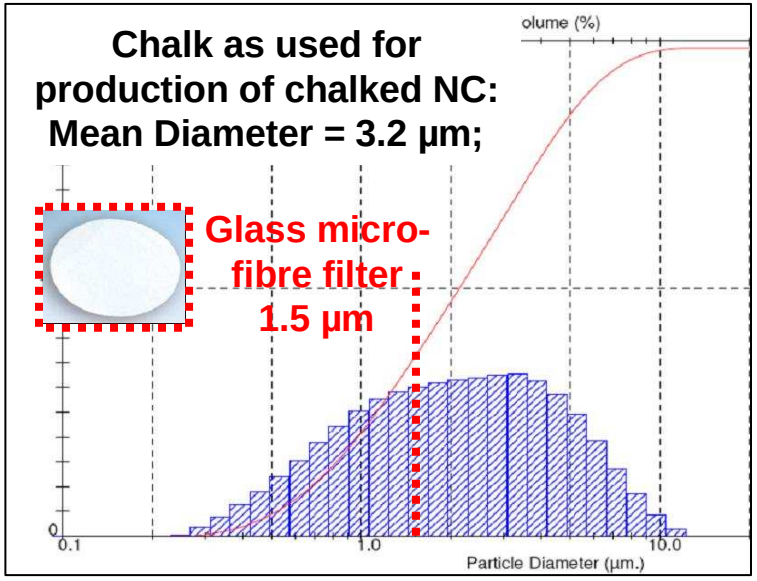
Chalked Nitrocellulose – Ash Test

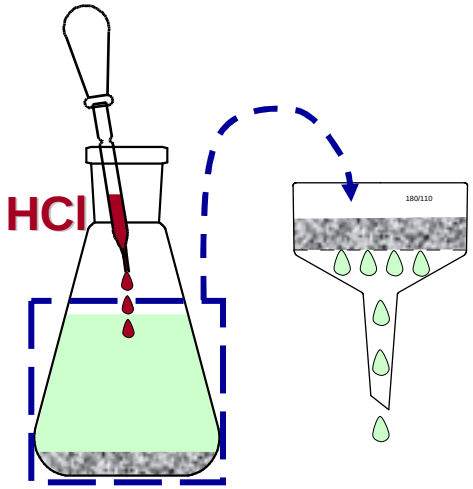
- **Result of 14-fold analysis of one chalked NC for "Ash" and "Carbonated Ash"**
  - **"Ash" and "Carbonated Ash" determination yield equivalent results; with the difference of the shift by stoichiometric factor**



Chalked NC – Ether-Alcohol Solubles / Acetone Insolubles

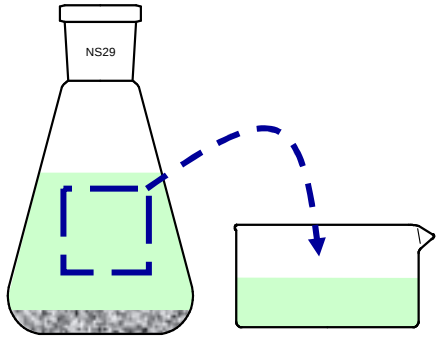
- In the tests for Ether-Alcohol Solubles and Acetone Insolubles based on the filtration method, the micro-fibre filter retains not only undissolved NC fibres but also chalk
  - This influence of chalk can be eliminated by adding a few drops (approx. 0.1 ml) of concentrated hydrochloric acid to the ether-alcohol or acetone solvent in order to dissolve the chalk directly after adding the solvent to the NC
- The evaporation method for Ether-Alcohol Solubles determination can be applied without alterations (due to complete sedimentation of chalk before pipetting)





Filtration Method:

Chalk dissolved by a few drops of HCl

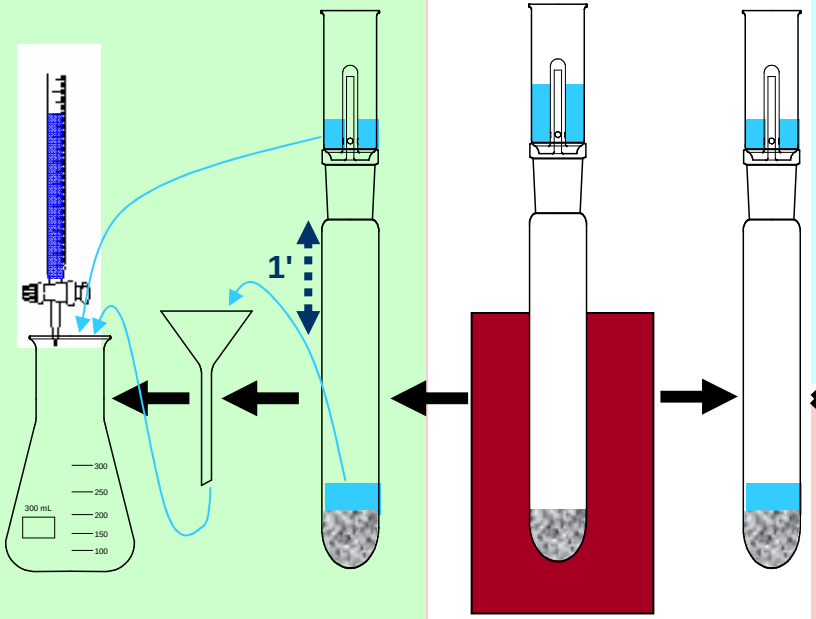


Evaporation Method:

Chalk completely settled by centrifuging

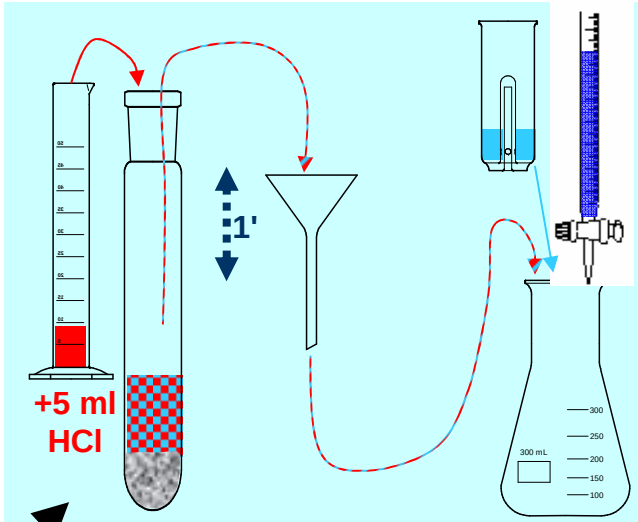
Chalked Nitrocellulose – 132°C Bergmann-Junk Stability Test

Not applicable for chalked NC



Test 5A

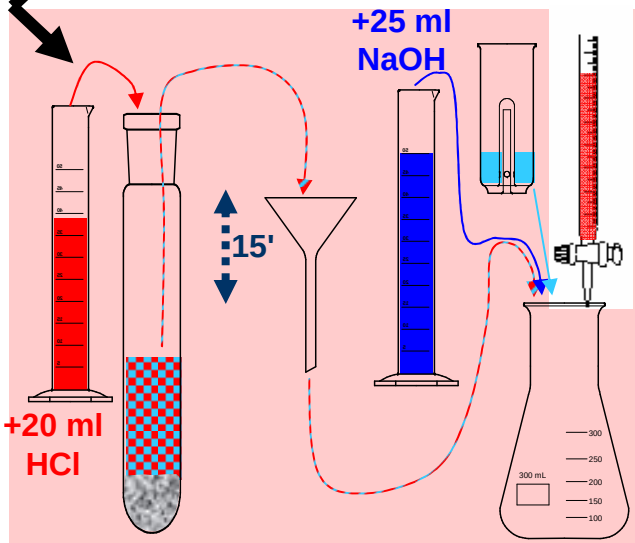
- Shaking 1'
- Filtration
- Direct Titration with 0.1 n NaOH
- No Blank



Test 5B

- + 5 ml HCl 0.1n
- Shaking 1'
- Filtration
- Back Titration with 0.1 n NaOH
- Repeat w. Blank

(simplified Back Titration Method)



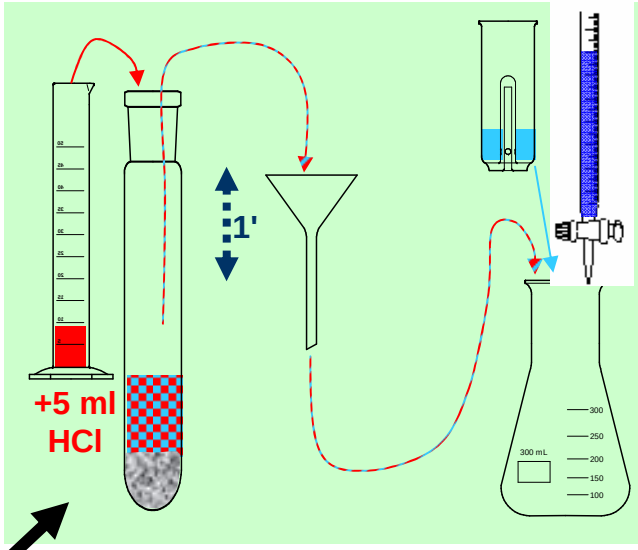
Test Ed. 1

- + 20 ml HCl 0.1n
- Shaking 15'
- Filtration
- + 25 ml 0.1 n NaOH
- Back Titration with 0.1 n HCl
- Repeat w. Blank

(Original Back Titration Method)

Chalked Nitrocellulose – 132°C Bergmann-Junk Stability Test

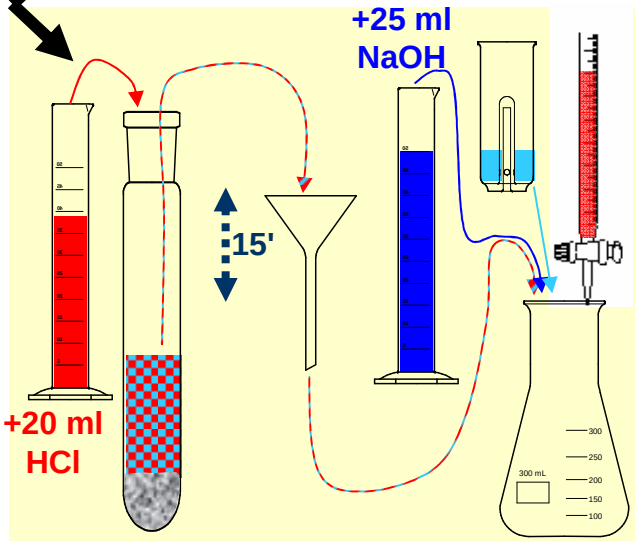
- **Comment: Test 5B is a simplified version of the 132°C Heat Test of STANAG 4178 Ed. 1 / UK M23 which gives equal results**
- **Simplifications in Test 5B are:**
  - **Excess of HCl which is added to dissolve chalk is reduced from 20 ml to 5 ml (not chalked NC consumes no HCl, chalked NC typically 1 to 1.5 ml of HCl)**
  - **Shaking time is reduced from 15' to 1' (since already 10 s are sufficient to dissolve all chalk)**
  - **Back titration directly with NaOH instead of adding first excess of NaOH and then back titration with HCl – this titration scheme is also used in the UK M22/87 "Alkalinity Test" which has the same task (dissolution / determination of chalk content)**



Test 5B

- + 5 ml HCl 0.1n
- Shaking 1'
- Filtration
- Back Titration with 0.1 n NaOH
- Repeat w. Blank

(simplified Back Titration Method)



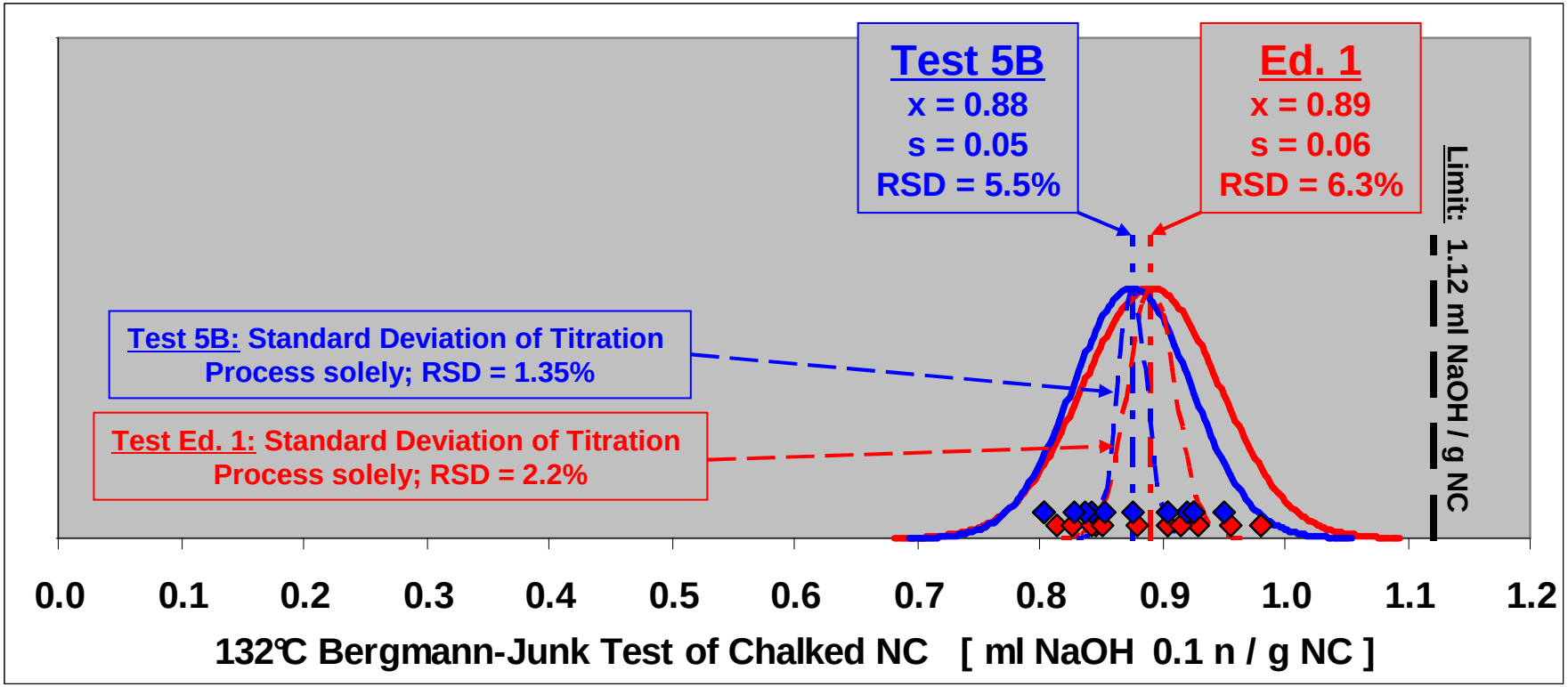
Test Ed. 1

- + 20 ml HCl 0.1n
- Shaking 15'
- Filtration
- + 25 ml 0.1 n NaOH
- Back Titration with 0.1 n HCl
- Repeat w. Blank

(Original Back Titration Method)

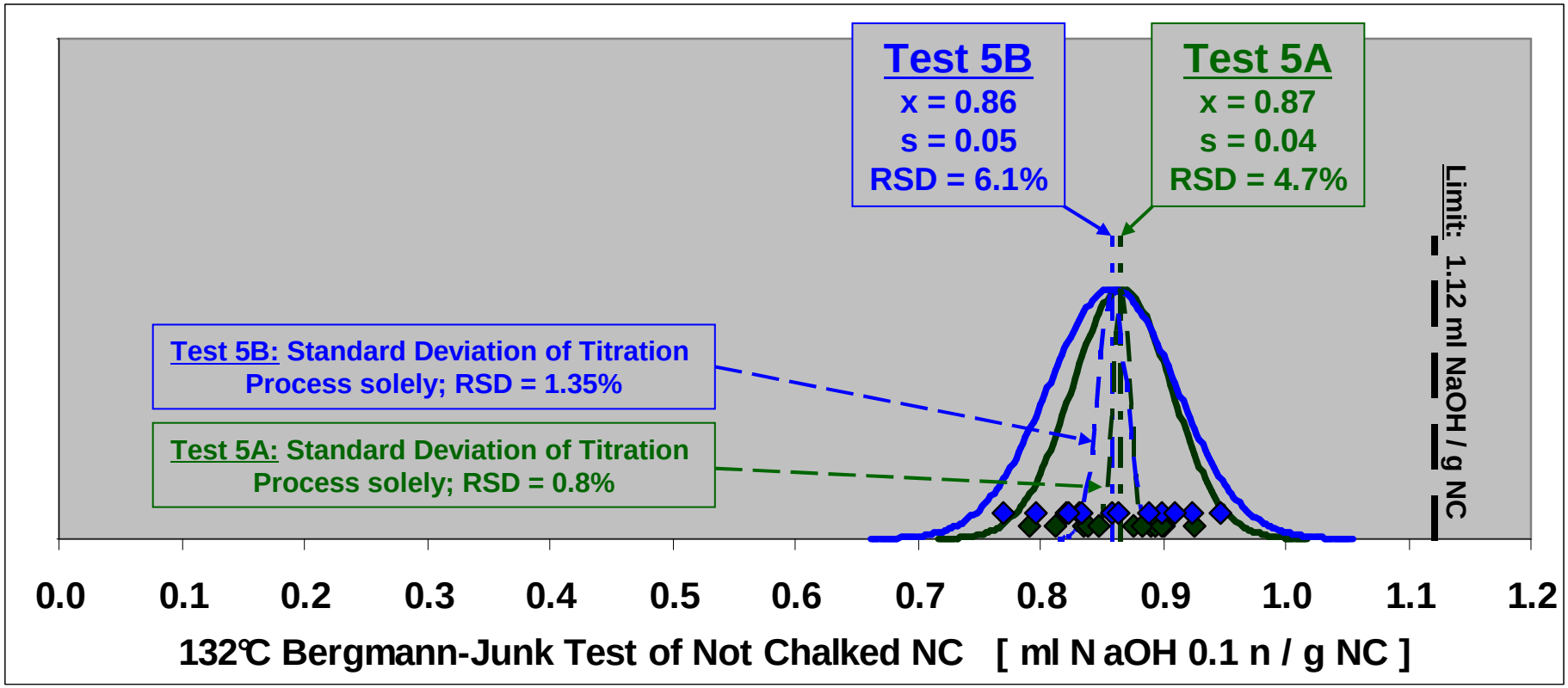
Chalked Nitrocellulose – 132°C Bergmann-Junk Stability Test

- **Bergmann-Junk Test 5B versus Test from Ed. 1; Testing of <u>chalked nitrocellulose</u>**
  - For testing of chalked NC, Test 5B gives equal result as the more complicated test described in Ed. 1 of STANAG 4178
  - Test 5B with simplified titration procedure can thus be used for chalked NC (as postulated in Ed. 2)



132°C Bergmann-Junk Stability Test – Test 5A vs Test 5B

- **Bergmann-Junk Test 5A versus Test 5B; Testing of <u>not chalked</u> nitrocellulose**
  - Test 5B and Test 5B give equal result if applied for testing of not chalked NC
  - Both test versions can thus be used for not chalked NC (as mentioned in Ed. 2)
  - Precision of more complicated Test 5B is lower than precision of Test 5A



The Last Few Slides

- **The Summary**
- **The Way Ahead**
- **The Acknowledgments**

The Summary

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**
- **Contains additional test methods from Ed. 1 and from other sources:**
  - **To allow the use of alternative test procedures which give equal results**
  - **To include test procedures for additional important properties of NC**
- **Incorporates improvements to make tests more accurate and reliable, faster, safer and cheaper**
- **STANAG 4178 Ed. 2 is currently in NATO ratification (5 nations have already ratified; a total of 13 needed)**

The Way Ahead

- **STANAG 4178 Ed. 2 needs to be ratified by another 8 NATO Nations**
- **Test methods need to be implemented**
- **International Round Robin Test will be organised (by ARDEC USA)**
- **Tests which are new in Ed. 2 should be carefully examined and, if necessary, further improved for Ed. 3 (e.g. Ion Chromatography and Fibre Length Distribution)**
- **New / improved test methods should be developed in the near future (for Ed. 3 of this STANAG):**
  - **Test methods that better characterise the Processability of NC**
  - **Better test methods for Nitrogen Content Distribution to replace Ether-Alcohol and Acetone Solubility Tests (e.g. chromatographic method)**
  - **…**

The Acknowledgments

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- Nitrochemie Laboratory Team (Ruth Sopranetti, Marc Müller; Melanie Wolf, Dominik Werfl, …)
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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
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	- Cranfield University
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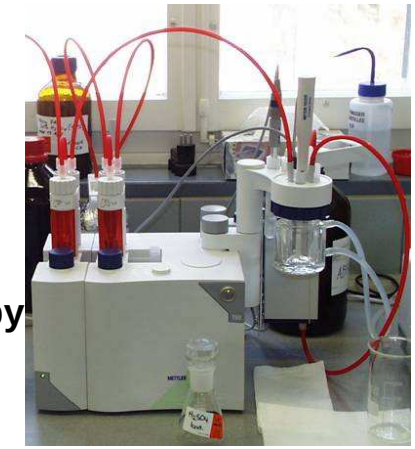
	- Esterline Defense Group, Coachella
	- GD-OTS, St. Marks Powder

Additional Files

Additional Slide with more detailed Information

The Test Procedures – Average Nitrogen Content (1)

- Nitrogen Content is a mandatory test – it determines the average degree of nitrate ester substitution and thus the energy content of the NC
- As an intrinsic property of the NC, it can be determined in different ways – the following methods are considered to be equivalent and can be used:
  - Ferrous Ion Titration Methods
  - Nitrogen Analyzer Method
  - Combustion Calorimetry Method



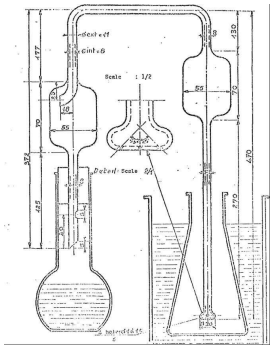
- Ferrous Ion Titration Methods (FS/FAS):
  - From MIL-DTL-244C
  - Fast and accurate but uses only small sample mass, requires cooling
  - Principle: Acidic hydrolysis of the NC to nitrate ion (NO3-), followed by redox titration of the nitrate using either Ferrous Sulphate (FS) or Ferrous Ammonium Sulphate (FAS) as titrant
  - Reference Method
- Nitrogen Analyzer Method:
  - New method; procedure has been supplied by Australia
  - Highly automated method; fast and accurate if properly calibrated / operated
  - Principle: NC is combusted in commercially available Nitrogen Analyser (Combustion Elemental Analyser) to nitrogen oxides; after reduction of nitrogen oxides and removal of the other product gases, the amount of nitrogen gas is determined by a thermal conductivity detector

Additional Slide with more detailed Information

The Test Procedures – Average Nitrogen Content (2)

- **Combustion Calorimetry Method:**
  - New method; has been supplied by Switzerland
  - Uses about ten times larger sample amounts than all other methods (more representative sample)
  - Principle: Indirect method; heat of combustion which is strongly correlated to nitrogen content is determined in combustion calorimeter; must be calibrated by a direct method (preferably FS/FAS)

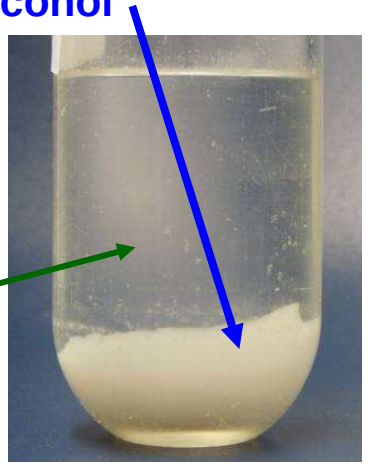
- **Other accepted Methods:**
  - **Devarda's Alloy Method:** Nitrogen content is determined by alkaline hydrolysis of NC to produce nitrate ion, which is then reduced by Devarda's alloy to ammonia; the later is determined by titration
  - **Schulze-Tiemann Method:** NC is first digested in iron (II) chloride and hydrochloric acid, followed by determination of the volume of evolved nitrogen oxide
  - **Nitrometer Method:** NC is decomposed by concentrated sulphuric acid in the presence of mercury, followed by volumetric determination of the gas evolved - use of this method is strongly discouraged due to worker safety concerns (large amount of mercury)
  - These 3 methods have been the main methods for Nitrogen Content determination in the past – they have proven to be reliable and accurate – thus they can still be used, provided that an approved national standard is followed, and that the quality requirements are fulfilled



Additional Slide with more detailed Information

The Test Procedures – Ether-Alcohol Solubles

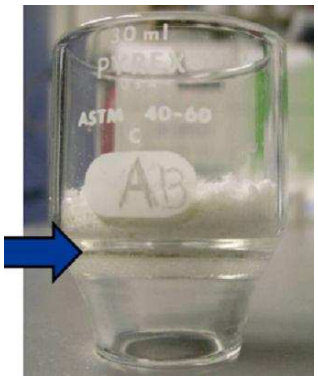
- The Ether-Alcohol Solubles is a **mandatory** test – it is used to check:
  - The mixing ratio (pyrocellulose / guncotton) of nitrocellulose blends
  - The purity and nitration quality of pyrocellulose and guncotton
- The following methods can be used:
  - Filtration Method – preferred method
  - Evaporation Method – accepted for NC with N-contents ≥12.75%
- **Filtration Method:**
  - From MIL-DTL-244C; similar to STANAG 4178 Ed. 1 Meth. B
  - Assesses part of the NC which remains undissolved in ether-alcohol
  - Easier to perform; yields higher precision
  - New: Glass-microfibre filters <3 µm have to be used !
- **Evaporation Method:**
  - From MIL-DTL-244C; similar to STANAG 4178 Ed. 1 Section 4.2
  - Assesses part of the NC which dissolves in ether-alcohol
  - More time-consuming



Additional Slide with more detailed Information

The Test Procedures – Acetone Insolubles

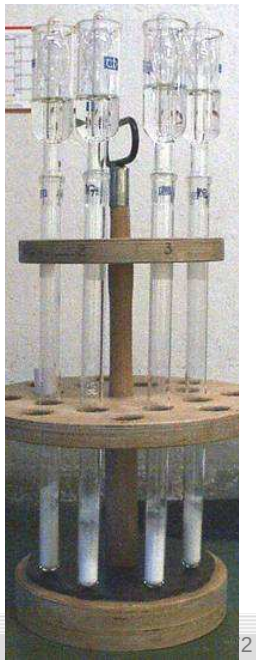
- **The Acetone Insolubles is a mandatory quality and purity test:**
  - **It measures the amount of NC with low nitrogen content ("unnitrated NC")**
  - **Other insoluble matter (e.g. inorganic impurities) may also add to the Acetone-Insolubles value**
- **The Acetone Insolubles value is determined by the Filtration Method:**
- **Filtration Method:**
  - **From MIL-DTL-244C; similar to STANAG 4178 Ed. 1 Meth. B**
  - **Principle: Dissolution of the NC in acetone, followed by assessing of insoluble matter by filtration / drying / weighing of the remainder**
  - **New: Glass-microfibre filters <3 µm have to be used !**



Additional Slide with more detailed Information

132°C Bergmann-Junk Stability Test – Overview

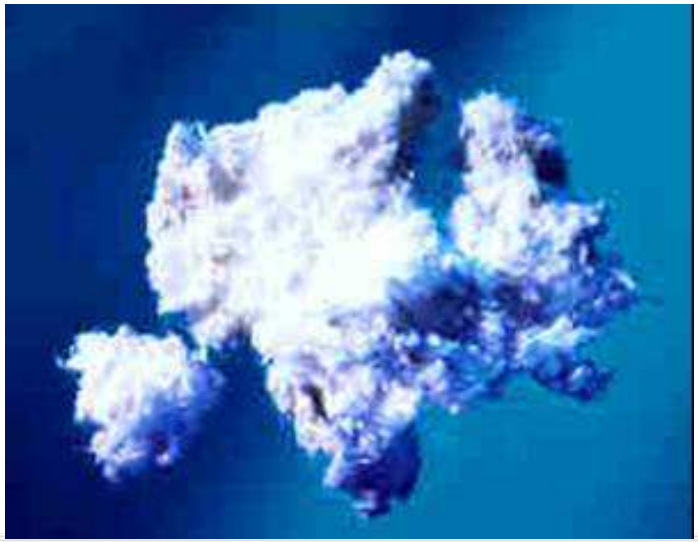
- In the 132°C Bergmann-Junk Stability Test, the sample is heated for 2 hours at 132°C, followed by assessment of the evolved and collected nitrogen oxides by acid/base titration
- For not chalked NC, direct titration with sodium hydroxide (Test 5A) is chosen as this method is easy, fast and delivers reliable results
- In case of chalked NC, the calcium carbonate partly reacts with the nitrogen oxides (which are dissolved in the solution which was sucked back into the test tube during cooling down) and thus reduces the amount of sodium hydroxide consumed in the "standard direct titration procedure" – thus NC samples with high calcium carbonate content appear to be more stable than they are
- Therefore, a more complicated back titration procedure (Test 5B), with first adding hydrochloric acid to neutralize the chalk, followed by back titration with sodium hydroxide), in combination with blank determination (same titration procedure with not heated NC sample) has to be used for chalked NC in order to correct for the influence of the calcium carbonate

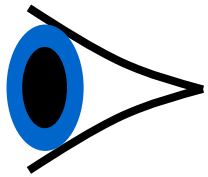


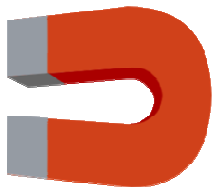
Additional Slide with more detailed Information

The Test Procedures – Visual Inspection

- The Visual Inspection checks for foreign matter, for impurities that lead to discoloration, or for other visually observable anomalies
- For that, the NC is spread as thinly as possible on a clean table, followed by:
  - Visually examination for any foreign matter (metal, wood, ….)
  - If foreign matter found: sampling (with tweezers or magnet) and weighing
  - Checking of general appearance and colour of the sample
- If visual inspection or other information indicates possibility of contamination with oil or grease: Assessment of oil and grease content
- Visual Inspection of NC is not mandatory







Additional Slide with more detailed Information

The Test Procedures – Ash

- The determination of Ash is a purity test:
  - It accounts for non-combustible impurities
- Necessity of this test is questioned (no failures have been reported for many years)
- Testing of Ash is thus not mandatory
- Testing Method:
  - From STANAG 4178 Ed. 1, procedure 2.2.a; includes procedure of MIL-DTL-244C
  - Only one method (but with several possible ways of gelatinization / digestion and burning / ashing / calcination of the nitrocellulose) is described for the determination of Ash
  - Principle: First gelatinization or digestion of the nitrocellulose, followed by burning and ashing / calcination at higher temperatures, and finally by weighing of the residue as Ash

Ignition / burning of NC saturated with liquid paraffin

Ashing / calcination in muffle furnace at 800°C

Additional Slide with more detailed Information

The Test Procedures – Grit

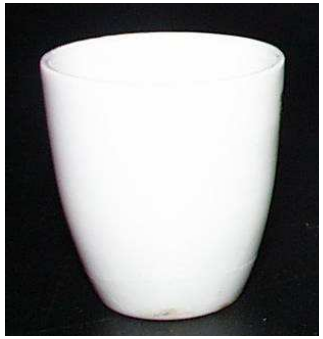
- **The determination of Grit is a purity and safety test:**
  - **Grit is defined as the amount of mineral matter, insoluble in both hot concentrated hydrochloric acid and hot concentrated sodium hydroxide, which will not pass a defined sieve**
  - **Grit is determined in the residue of the Ash test**
- **Necessity of this test is questioned (no failures have been reported for many years)**
- **Testing of Grit is thus not mandatory**
- **Testing Method:**
  - **From STANAG 4178 Ed. 1, procedure 3**
  - **Principle: The residue of the ash test is first treated with hydrochloric acid, filtered and ignited, treated with aqueous sodium hydroxide, filtered and ignited again, and finally sieved to determine size, number and nature of the particles retained**

Additional Slide with more detailed Information

The Test Procedures – Ionic Impurities (1)

- **All test methods for Ionic Impurities can be regarded as purity tests**
- **Testing of Ionic Impurities is not mandatory**
- **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 !**

- **Sulphate Content Method:**
  - **Checks for sulphuric acid / sulphuric esters (which would affect the results of the stability tests)**
  - **From UK M-Method M22/87 Method 14 (Procedure A)**
  - **Principle: Digestion of the NC in nitric acid, oxidation of sulphur components into sulphate, precipitation of the sulphates with barium chloride, and filtration / drying / igniting / weighing of the formed barium sulphate**
  - **Method is elaborate and time-consuming**



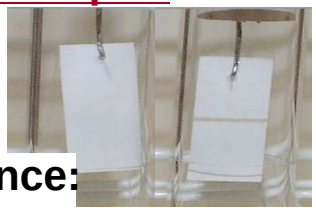
Additional Slide with more detailed Information

The Test Procedures – Ionic Impurities (2)

- **Residual Acidity Method:**
  - Checks for total residual acids (which would affect the results of the stability tests)
  - Method adopted from US Federal Specifications TT-N-350B
  - Principle: Dissolution of the NC in acetone in order to release the residual fibre acidity, followed by adding of water, and finally titration with NaOH
- **Alkalinity Method:**
  - Checks for total residual alkalis; can be used to determine the level of calcium carbonate in chalked nitrocellulose
  - From UK M-Method M22/87 Method 11
  - Principle: Extraction of the alkalis from the NC with HCl, followed by back titration with NaOH
- **Spectroscopy Method:**
  - Assesses numerous different anoinic impurities (incl. level of calcium carbonate in chalked nitrocellulose)
  - New method; procedure has been supplied by Switzerland
  - Principle: Digestion of the NC in nitric acid, followed by dilution and determination of calcium (and, if required, of other elements) by Atomic Absorption Spectroscopy (AAS); or by equivalent spectroscopic methods such as Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)

Additional Slide with more detailed Information

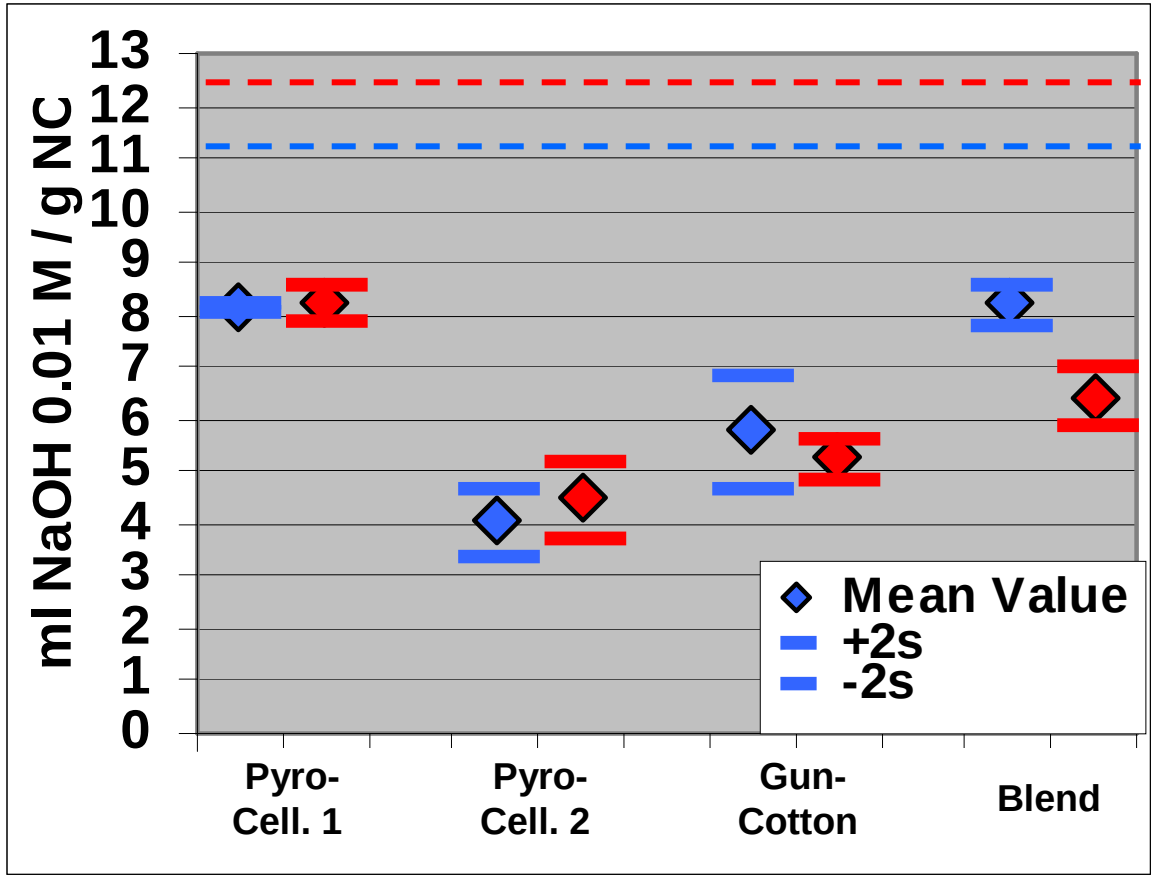
The Test Procedures – Detection of Impurities by Heat Tests



- **Heat Tests based on the reaction of nitrogen oxides with KI/starch Test Paper:**
  - **65.5°C Heat Test; 35' at 65.5°C; MIL-DTL-244B**
  - **76.6°C Abel Heat Test; 10' at 76.6°C; STANAG 4178 Ed. 1, Test 10**
- **65.5°C and 76.6°C Heat Tests can not be regarded as stability tests since:**
  - **Ageing conditions in these tests are much too weak to obtain a sufficient horizon of stability prognosis (weaker by factor 1000 than in 132°C / 134.5°C Heat Tests)**
  - **These tests do not primarily examine the main ageing reaction of the NC (thermolysis of nitric esters) but are extremely sensitive in detecting traces of nitrogen oxides that are released during heating up to test temperature (often by other processes than ageing)**
  - **Thus these two tests often give contradictory results to the stability tests (132°C / 134.5°C Test) – e.g. slightly aged but demonstrably stable NC often fails 65.5°C / 76.6°C Heat Tests**
- **Both tests, however, can be used as indirect impurity tests in freshly produced NC (e.g. for presence of neutralization salts which have not been properly removed during production) – these impurities lead to the evolution of nitrogen oxides**
- **Testing of Impurities by Heat Tests is not mandatory**
- **The significance of test results as obtained with these two heat tests is questioned**
  - **The two tests have been maintained mainly from historical reasons**
  - **The use of the other purity tests as described in this STANAG is to be preferred**

Additional Slide with more detailed Information

132°C Bergmann-Junk / BJ-Siebert Stability Tests

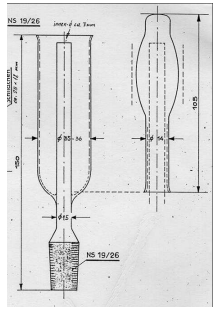


Experimental conditions according to original test procedure; potentiometric titration:

Bergmann-Junk:

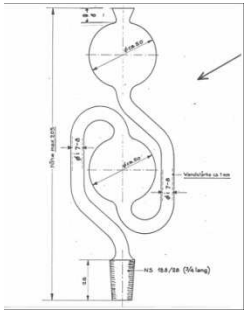
Cups filled with 25 ml water

15 mm depth of immersion



Bergmann-Junk-Siebert:

Globes filled 50 ml H_2O_2 3%
20 mm depth of immersion



- **Bergmann-Junk-Siebert Test (20 mm immersion; globes + H_2O_2) gives similar result to Bergmann-Junk (15 mm immersion; cups + water)**