

UNCLASSIFIED

PROPOSED UPGRADING OF KRAFT CELLULOSE SPECIFICATION

Kristy Klein, Lucas Lopez, Mo Elalem
U.S. Army ARDEC
Picatinny Arsenal, NJ 07806
USA

8th Nitrocellulose Symposium
5-7th JUNE 2018 – Bergerac, France

UNPARALLELED
**COMMITMENT
& SOLUTIONS**

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.

¹ Act like someone's life depends on what we do.



UNCLASSIFIED

The current revision of MIL-C-20330A for Kraft (sulfate) wood pulp, which was written in 1962, addresses cellulose purity (alpha cellulose, alkali soluble matter and pentosane), and contaminants (ether extractive matter, ash and silica and lignin), but doesn't include other known properties that may affect nitration, propellant manufacturing and performance.

Since all celluloses are not equal and their range of property variations this could have significant effects on the resulting properties of the NC and subsequently products produced.

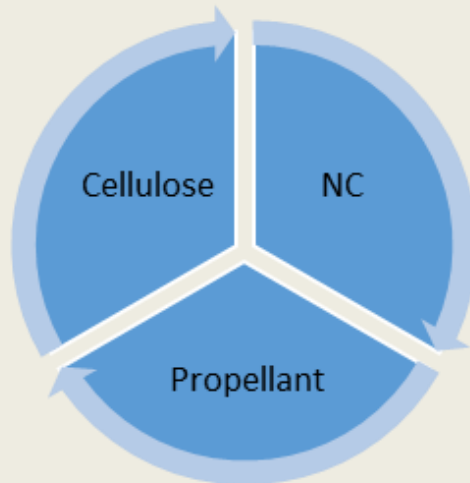
Therefore to ensure uniformity of products generated from cellulose sources a specification delineating acceptable cellulose properties is necessary.

UNCLASSIFIED

UNCLASSIFIED

In order to ensure uniformity of products generated from cellulose sources a specification delineating acceptable cellulose properties is necessary.

Raw material variability has significant impact on the processing parameters used to synthesize NC and can cause negative effects to the properties of the NC and will subsequently impact properties of the propellants being produced.



The USG is working to identify those chemical and physical properties and their acceptable ranges that will ensure the quality and consistency of cellulose used in the manufacturing of NC.

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.



U.S. ARMY
RDECOM

RATIONALE FOR PROPOSED PROPERTIES



UNCLASSIFIED

The selection of the properties identified in this paper was based on ARDEC research, and meetings with representatives from various pulp and paper mills, academia tied to the pulp and paper industry, nitrocellulose manufacturers, and other industry experts.

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.



UNCLASSIFIED

Following the format of the MIL-C-20330A specification the proposed properties are divided into two groups:

- Physical properties
- Chemical properties

Physical Properties

- **Moisture:** This is a measure of water in the Lot of cellulose. This is measured at the mills; and is related to the density of the cellulose. Any excess water has to be accounted for nitration. Eight percent does not impact the nitration process.
- **Density and Basis Weight (the weight in grams of a single sheet of area one square meter):** Higher densities and basis increase loading of nitrator pots and overall NC production throughput but may decrease acid penetration into sheet. Both refer to “bone-dry” sheet density and basis weight.
The 0.50 - 0.95 g/cc density range includes a variety of high purity grades from two suppliers we can choose from. For example, a cellulose with a density of 0.50-0.60 g/cc will work for extruded propellants. For Ball Powder we can use a more dense cellulose, say, 0.8 g/cc and increase the throughput of NC.
- **Intrinsic Viscosity and Viscosity (CED - cupriethylenediamine):** A measure of the degradation of cellulose (generation of low molecular weight carbohydrates) resulting from the pulping process. The pulping process shortens the cellulose chain but long cellulose chains make NC with better properties, therefore cellulose with high viscosity is desirable.

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.



UNCLASSIFIED

- **Coarseness:** weight per unit length of fiber. A material of higher coarseness but equal density will have the same amount of mass of cellulose per unit volume as a material of lower coarseness. But the mass will be more concentrated in fewer fibers, resulting in more inter-fiber space. Higher coarseness allows for better diffusion of acid into the cellulose matrix, resulting in more uniform nitration. The pulp mills agreed with 0.2 mg/m value as a minimum.
- **Mullen:** Relates to the resistance of the densified sheet and amount of force required to burst through the sheet. The proposed range limits the amount of fines in NC to 3-5%.
- **Fines Max:** This is the maximum amount of allowable fibers less than 0.25mm in size which will escape as loss or dust in process.
- **Fiber Length:** Weighted Average Fiber Length of pulp will determine the fiber length distribution in the final NC and the amount of refining necessary to achieve it . The fiber length distribution for both southern pine and cotton linters is between 1.75 mm to 2.00 mm

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.



UNCLASSIFIED
Chemical Properties

- **S10 (10 % sodium hydroxide solution):** A solubility test that measures the total extractable material i.e., degraded cellulose and hemicellulose content in a pulp. That is, all cellulose that cannot be nitrated. It tells the actual purity of the pulp. The lower the S10, the highest the purity of the pulp. Alpha cellulose is the cellulose in wood pulp that can be converted into NC. Alpha cellulose content is calculated by subtracting S10 from 100
 - The USG team is proposing a maximum S10 of 3.5%
 - Historically, BAE RFAAP nitrates wood pulp with S10 of 5-6% max. “In general, the purer the cellulose used, the less difficulty in nitration and subsequent elimination of the acid, the higher the yield, and the more stable the NC formed²”.
- *Note: It is not necessary to report the Alpha value because S10 tells the actual purity of the pulp*
 - There is a discrepancy on how the alpha value is calculated. For example, some mills use the formula $\text{Alpha} = 100 - (\text{S10} + \text{S18}) / 2$, others use a slide rule. So alpha value is an approximation of purity
- **S18 (18% sodium hydroxide solution):** This is a solubility test that measures the total hemicellulose content in a pulp. It is included for information only to document the hemicellulose content in the pulp

2. Chauncey Worden, Edward, (2015). *Nitrocellulose Industry, Vol. 1 of 2: A Compendium of the History, Chemistry, Manufacture Commercial Application and Analysis of Nitrates, Acetates and Nanthates of Cellulose as Applied to the Peaceful Arts, With a Chapter on Gun Cotton, Smokeless Powder.*

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.



U.S. ARMY
RDECOM

CHEMICAL PROPERTIES (2 OF 2)



UNCLASSIFIED

- **Silica (SiO₂):** Must be minimized to reduce the sand content entering into NAC/SAC. 100 PPM is the realistic lowest amount that can be achieved without going into “specialty” products
- **Ash and (Ca, Fe, Mn):** The only other impurities found in high purity Kraft products

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.



UNCLASSIFIED

Property	Range
Moisture (%)	8 Max
Density (g/cm ³)	0.65-0.75
Basis Weight (g/m ²)	650-850
Intrinsic Viscosity (dl/g)	5.7 min
Basis Wt (g/m ²)	
Viscosity 0.5% CED (cP)	9 min
Coarseness (mg/m)	0.2 min
Mullen (kpa)	550-850
Fines	5 max
Fiber Length (mm)	Information only
S18	Information only
S10	3.5 max
Silica (ppm)	100 max
Ash	0.30 max
Calcium (ppm)	100 ppm max
Iron (ppm)	6 max
Manganese (ppm)	0.30 max

TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY (TAPPI)

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.



U.S. ARMY
RDECOM

SUMMARY AND OUTLOOK



UNCLASSIFIED

The purchase specification for Kraft cellulose will serve as the basis for the revision of MIL-C-20330A specifications

A team of Government experts are currently modifying MIL-C-20330A specifications

UNCLASSIFIED

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.