

METRIC

MIL-DTL-244C
w/AMENDMENT 1
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SUPERSEDING
MIL-DTL-244C
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DETAIL SPECIFICATION

NITROCELLULOSE

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the requirements, examinations and tests for various grades, types, and classes of nitrocellulose (NC) for use in propellant and combustible case manufacturing.

1.2 Classification. Nitrocellulose is classified into the following grades, types, and classes.

Grade	Name	Nitrogen content
A Type I	Pyrocellulose	12.60 \pm 0.10%
A Type II	Pyrocellulose	12.60 \pm 0.15%
B Type I	Guncotton	13.35% minimum
B Type II	Guncotton	13.20% minimum
B Type III	Guncotton	13.55% minimum
C Type I	Blend of pyrocellulose and guncotton	13.15 \pm 0.05%
C Type II	Blend of pyrocellulose and guncotton	13.25 \pm 0.05%
D	Pyrocellulose	12.20 \pm 0.10%
E	Pyrocellulose	12.00 \pm 0.10%
F	Pyrocellulose	11.55 \pm 0.25%

Class 1 - For combustible cartridge case (CCC) applications

Class 2 - For solvent and solventless propellant applications

Class 3 - For propellants made from lacquer applications

(Lacquer applications are propellant products that are made with completely solvated nitrocellulose from a homogenous mixture of nitrocellulose, stabilizer and solvent.)

Comments, suggestions, or questions on this document should be addressed to: Commander, U.S. Army ARDEC, ATTN: RDAR-EIQ-SE, Picatinny Arsenal, New Jersey 07806-5000, or emailed to usarmy.picatinny.ardec.list.ardec-stdzn-branch@mail.mil. Since contact information can change, you may want to verify the currency of this information using ASSIST Online database at <https://assist.dla.mil>.

AMSC N/A

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2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all requirements of documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

COMMERCIAL ITEM DESCRIPTIONS

A-A-59342	Ethyl Alcohol (for Ordnance Use)
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DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-C-206	Cellulose (Cotton)
MIL-C-216	Cellulose, Woodpulp, Sulfite
MIL-C-20330	Cellulose, Woodpulp (Sulfate)

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-1171	Acceptance and Description Sheets for propellant and explosives
MIL-STD-1916	DOD Preferred Methods for Acceptance of Product

(Copies of these documents are available online at <http://quicksearch.dla.mil> or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.3 Order of precedence. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 First article. When specified (see 6.2) a nitrocellulose sample shall be subjected to first article inspection in accordance with 4.2. Analytical results shall be in accordance with (IAW) MIL-STD-1171.

3.1.1 Product performance. The final product produced using the nitrocellulose manufactured in accordance with this specification shall meet the performance requirements specified in the final product specification (see 6.9).

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3.2 Conformance. A sample shall be subject to conformance inspection in accordance with 4.5. Analytical results shall be IAW MIL-STD-1171 (see 6.2).

3.3 Materials.

3.3.1 Alcohol. The alcohol used in the manufacture of alcohol wet nitrocellulose shall conform to the requirements for Grade 2 of A-A-59342.

3.3.2 Cellulose. The cellulose used shall be derived from cotton cellulose, woodpulp cellulose obtained by the sulfate process, or woodpulp cellulose obtained by the sulfite process, conforming to the requirements of MIL-C-206, MIL-C-20330, or MIL-C-216 respectively (see 6.14).

3.4 Nitrocellulose.

3.4.1 Nitrogen content and blending allowance. The nitrocellulose shall meet the nitrogen content requirement for the required grade and type specified in Table I. Blending may be used to achieve the applicable requirements for each particular grade in accordance with the range specified in Table I. Blending allowance is defined as the required nitrogen content obtained by blending nitrocellulose containing the percent nitrogen in the range listed. Unless otherwise specified, Grade C blends shall be manufactured with Grade B Type I and Grade A Type II. For certain Grade C blend applications, Grade B Type II or Grade F nitrocellulose components may be used when specified in the contract or order.

TABLE I. Nitrogen content and blending allowance.

Grade / Type	Nitrogen Content	Blending Allowance
A / I	12.60 ± 0.10%	12.45-12.75
A / II	12.60 ± 0.15%	12.45-12.75
B / I	13.35% minimum	
B / II	13.20% minimum	
B / III	13.55% minimum	
C / I	13.15 ± 0.05%	Grade A + B
C / II	13.25 ± 0.05%	Grade A + B
D	12.20 ± 0.10%	12.05-12.35
E	12.00 ± 0.10%	11.90-12.20
F	11.55 ± 0.25%	11.30-11.80
NOTES: Class 1 – Combustible cartridge applications. The nitrocellulose shall be of either Grade A or Grade B and shall meet the nitrogen content in Table I for the grade/type specified in the contract or purchase order. Class 2 – Solvent and solventless propellant applications. The nitrocellulose shall meet the nitrogen content in Table I for the grade/type specified in the contract or purchase order. Class 3 – Propellants made from lacquer applications. Lacquer applications are propellant products that are made with completely solvated nitrocellulose from a homogenous mixture of nitrocellulose, stabilizer and solvent. The nitrocellulose shall be Grade C and shall meet the nitrogen content in Table 1 for the grade/type specified in the contract or purchase order. It is permissible that during the lacquer manufacturing process, nitrocellulose with a nitrogen content of 12.4% or greater may be used as long as the final nitrogen meets the nitrogen requirements of Grade C nitrocellulose. Any nitrocellulose used with a nitrogen content below 12.75% shall be subject to the chemical requirements (other than nitrogen content) of Grade A nitrocellulose. Any nitrocellulose used with a nitrogen content above 12.75% shall be subject to the chemical requirements (other than nitrogen content) for Grade B nitrocellulose.		

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3.4.2 Ether-alcohol solubility. For all classes of nitrocellulose, the nitrocellulose shall meet the ether-alcohol solubility requirement for the required grade as listed in Table II.

TABLE II. Ether-alcohol solubility for all classes of nitrocellulose.

Grade	Minimum Ether-alcohol solubility (%)
A	99.0
B	None specified
C	Specified in contract or purchase order
Grade A for making Grade C	98.5 or as specified in contract or purchase order
D	97.0
E	97.0
F	95.0

3.4.3 Stability. For all classes of nitrocellulose, the nitrocellulose shall meet one of the following:

- a) maximum of 2.5 mL NO/g NC for the 132 °C Bergman-Junk test
- b) minimum of 30 minutes on the 134.5 °C methyl violet paper test

3.4.3.1 Deterioration in storage. For all classes, nitrocellulose that has not been used within 12 months of the date of stability testing by the nitrocellulose manufacturer shall meet the requirement for stability (see 3.4.3) within two (2) weeks prior to use.

3.4.4 Acetone insolubles. For all classes of nitrocellulose, the nitrocellulose shall meet the acetone insolubles requirement for the required grade as listed in Table III.

TABLE III. Acetone insolubles for all classes of nitrocellulose.

Grade	Maximum acetone insolubles (%)
A	0.60
B	0.40
C	0.40 ¹
D	2.0
E	3.0
F	5.0
<u>Notes:</u> ¹ / Grade C nitrocellulose for Class 3 applications may be 0.6% maximum acetone insolubles.	

3.4.5 Viscosity. Unless otherwise specified, for all classes of nitrocellulose, the nitrocellulose shall have a viscosity specified in the contract or purchase order.

3.4.6 Fineness. Unless otherwise specified, for all classes of nitrocellulose, the nitrocellulose shall have a fineness specified in the contract or purchase order.

3.4.7 Fiberization quality.

3.4.7.1 Class 1 - For combustible cartridge applications. Unless otherwise specified, for Class 1 Grade B nitrocellulose, the total content in sinkers shall be not greater than 0.75% and total content in agglomerates less than 0.25%. For Class 1 Grade A nitrocellulose, the total content in sinkers shall be less than 1.0% and total content in agglomerates less than 0.5% or as specified in contract or purchase order (See 6.11).

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3.4.7.2 Class 2 - For solvent and solventless propellant applications. For Class 2 nitrocellulose, the tolerance for agglomerates varies among final extruded propellant applications. For applications requiring a low agglomerate level, the nitrocellulose shall contain no more than 1.0% agglomerates. For applications requiring a moderate agglomerate level, the nitrocellulose shall contain no more than 5.0% agglomerates. For applications allowing a high agglomerate level, the nitrocellulose shall contain no more than 20% agglomerates. For applications allowing very high agglomerate levels, the nitrocellulose shall contain no greater than 50% agglomerates. The level of agglomerates (low, moderate, high, or very high) shall be specified in contract or purchase order (See 6.11).

3.4.8 Oil and grease content. For Class 1 nitrocellulose, the oil and grease content shall not exceed 0.1%.

3.4.9 Dry weight. For Class 1 nitrocellulose, at time of pack out, the dry weight of nitrocellulose in each packed transport container shall be within 1.50 weight percent of the weight specified in the contract or purchase order.

3.4.10 Total volatile content. The volatile constituent in nitrocellulose can be composed of water or ethyl alcohol or a mixture of both. Unless otherwise specified, for all classes of nitrocellulose, the nitrocellulose shall have a total volatile content of no less than 25% by weight for off-facility shipments. The maximum total volatile content shall meet the requirement specified in the contract or purchase order when required.

3.4.10.1 Preservation of total volatile content. The nitrocellulose shall be stored and transported in a manner that preserves the total volatile content.

3.4.11 Workmanship. For all classes of nitrocellulose, the nitrocellulose lot shall maintain a uniform composition, texture, and color and be free of foreign matter. The nitrocellulose shall be prepared, stored and transported in-house in a manner that prevents contamination by foreign material.

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4. VERIFICATION

TABLE IV. Requirement/verification cross reference matrix.

Method of verification		Classes of verification						
1 – Analysis 2 – Demonstration 3 – Examination 4 – Test		A – First article B – Conformance						
Section 3 Requirement	Description	Verification Methods				Verification Class		Section 4 Verification
		1	2	3	4	A	B	
3.1	First article			X	X	X		4.2
3.1.1	Product performance			X	X	X		4.5.13
3.2	Conformance			X	X		X	4.3
3.3.1	Alcohol				X	X	X	4.5.12.1
3.3.2	Cellulose				X	X	X	4.5.12.2
3.4.1	Nitrogen content				X	X	X	4.5.1
3.4.2	Ether-alcohol solubility				X	X	X	4.5.2
3.4.3	Stability				X	X	X	4.5.3
3.4.3.1	Deterioration in storage				X		X	4.5.3
3.4.4	Acetone insolubles				X	X	X	4.5.4
3.4.5	Viscosity				X	X	X	4.5.5
3.4.6	Fineness			X		X	X	4.5.6
3.4.7.1/3.4.7.2	Fiberization quality				X	X	X	4.5.7
3.4.8	Oil and grease content				X	X	X	4.5.8
3.4.9	Dry weight			X		X	X	4.5.9
3.4.10	Total volatile content				X	X	X	4.5.10
3.4.10.1	Preservation of total volatile content			X		X	X	4.5.14
3.4.11	Workmanship			X		X	X	4.5.11

4.1 Classification of inspection. The inspections requirements specified herein are classified as follows:

- a. First article inspection (see 4.2)
- b. Conformance inspection (see 4.3)

4.2 First article inspection. A nitrocellulose sample shall be subjected to first article inspection in accordance with Table V. Results shall be IAW MIL-STD-1171.

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TABLE V. First article inspection.

Examination or Test	Requirement Paragraph	Paragraph Reference/ Inspection Method	Test Measurements
<u>Raw Materials</u>			
Alcohol	3.3.1	4.5.12.1	N/A
Cellulose	3.3.2	4.5.12.2	N/A
<u>Nitrocellulose</u>			
Nitrogen content	3.4.1	4.5.1	4.4a.
Ether-alcohol solubility	3.4.2	4.5.2	4.4b.
Stability	3.4.3	4.5.3	4.4c.
Acetone insolubles	3.4.4	4.5.4	4.4d.
Viscosity	3.4.5	4.5.5	4.4e.
Fineness	3.4.6	4.5.6	4.4f.
Fiberization quality	3.4.7	4.5.7	4.4g.
Oil and grease content	3.4.8	4.5.8	4.4h.
Dry weight	3.4.9	4.5.9	4.4i.
Total volatile content	3.4.10	4.5.10	4.4j.
Preservation of total volatile content	3.4.10.1	4.5.14	100%
Workmanship	3.4.11	4.5.11	4.4k.
Performance first article	3.1.1	4.5.13	4.4.4

4.2.1 Sampling and submission. Prior to initiation of regular production, the manufacturer shall submit a first article sample and a performance first article sample for each specific Class, Grade, and Type of nitrocellulose pertaining to the contract in accordance with instruction issued by the contracting officer for evaluation. All samples submitted shall have been produced by the manufacturer using the same production process, procedures, and equipment as will be used in regular production, and shall be accompanied by certificates of analysis. Supplemental first article samples or portion thereof, as directed by the contracting officer, shall also be submitted in accordance with the contract provision. Future needs for the same product by the same procuring activity will not trigger a new First Article Test if the nitrocellulose is produced by the manufacturer using the same production process, equipment, conditions, and the same cellulose (supplier, source, form and configuration).

4.2.2 Inspection to be performed. The sample shall be subjected to all examinations and tests specified in Table V.

4.2.3 Rejection of first article samples. If any sample fails to comply with any of the applicable requirements, the first article shall be rejected.

4.3 Conformance inspection. Samples from lot shall be subjected to conformance inspection in accordance with 4.3.2. Results shall be IAW MIL-STD-1171.

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4.3.1 Classification of characteristics. The classification characteristics are provided for nitrocellulose on 4.3.2. The classification characteristics are based on the following definitions:

- a. Critical - A critical defect is a defect that is likely to result in hazardous or unsafe conditions for individuals using, maintaining, or depending upon the product, or a defect that analysis indicates is likely to prevent performance of the tactical function of a major end item such as a tank, land vehicle, missile, aircraft, artillery, or other major weapon system.
- b. Major - A major defect is a defect, other than critical, that is likely to result in failure, or to reduce materially the usability of the unit of product for its intended purpose.
- c. Minor - A minor defect is a defect that is not likely to reduce materially the usability of the unit of product for its intended purpose, or is a departure from established standards having little bearing on the effective use or operation of the unit.

4.3.2	<u>Nitrocellulose (all classes/grades/types)</u>			
Category	Examination or Test	Test Measurements	Requirement Paragraph	Paragraph Reference/ Inspection Method
<u>Critical</u>	None defined			
<u>Major</u>				
101	Nitrogen content	4.4a.	3.4.1	4.5.1
102	Stability	4.4c.	3.4.3	4.5.3
103	Deterioration in storage	4.4c.	3.4.3.1	4.5.3
104	Total volatile content	4.4j.	3.4.10	4.5.10
105	Preservation of total volatile content	100%	3.4.10.1	4.5.14
106	Alcohol	N/A	3.3.1	4.5.12.1
107	Cellulose	N/A	3.3.2	4.5.12.2
108	Viscosity	4.4e.	3.4.5	4.5.5
109	Fineness	4.4f.	3.4.6	4.5.6
110	Fiberization quality	4.4g.	3.4.7	4.5.7
111	Oil and grease	4.4h.	3.4.8	4.5.8
112	Workmanship – Foreign matter	4.4k.	3.4.11	4.5.11
<u>Minor</u>				
201	Dry weight	4.4i.	3.4.9	4.5.9
202	Workmanship – All other characteristics	4.4k.	3.4.11	4.5.11
203	Ether-alcohol solubility <u>1/</u>	4.4b.	3.4.2	4.5.2
204	Acetone insolubles <u>1/</u>	4.4d.	3.4.4	4.5.4
Notes:				
<u>1/</u> In the event that both ether-alcohol solubility and acetone insolubles do not meet the requirements of 3.4.2 and 3.4.4, it shall be classified as a major defect.				

4.3.3 Inspection lot formation. Nitrocellulose lot formation shall be in accordance with the lot formation requirements of MIL-STD-1916, Formation and identification of lots or batches. In addition, the following conditions shall apply:

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- a. The nitrocellulose grade/type shall be produced using the same process controls and the same nominal processing parameters.
- b. The nitrocellulose shall contain cellulose from not more than one source using the same process (except for pit cotton added, as allowed, see 6.10).
- c. Blends or final batches which are produced from the same materials, by the same processes (with pit cotton, if allowed, see 6.10), and meet the specification requirements may be grouped with other blends or final batches to form an inspection lot. However, identification on each drum or box of the lots shall permit traceability to various blends or final batches composing the lot.
- d. If a blend or final batch in an inspection lot fails to meet the requirements of this specification, the blend/batch shall be rejected from the lot.
- e. A nitrocellulose blend shall be defined as a homogenous collection of process batches of nitrocellulose from a single agitated tub/tank (or a multi-tank agitated and circulated system) from which a representative sample is drawn to determine conformance with applicable requirements. Typically, manufacturer formulated blends from a single tank are approximately 2,700 to 4,500 kilograms (6,000 to 10,000 pounds), but there may be times when technical needs require a larger or smaller blend.
- f. A nitrocellulose final batch shall be defined as a single fully processed batch obtained from a single agitated tub or tank from which a representative sample is drawn to determine conformance with applicable requirements. The final batch is differentiated from process batches, which are still being adjusted.
- g. The contract or purchase order can further define special blend parameters to meet specific needs.

4.3.4 Sampling.

- a. Sampling from process vessels as final product (blend or final batch). When sampling before pack-out, to certify blends or final batches of nitrocellulose, take one slurry sample from a homogeneous process vessel. To ensure homogeneity of the material, the process vessel shall be stirred or vigorously agitated for an adequate time before sampling. The samples shall be obtained from the process vessel while stirring or being agitated, by a means that ensures the sample is representative of the total contents. Minimum sample size for complete analysis is 500 g.
- b. Sampling from transport containers packed out as final product. It may be required to certify blends or final batches of nitrocellulose after pack-out. To do so, a minimum of 10 transport containers shall be selected for sampling representing the entire blend or final batch. These samples shall be mixed together to produce a composite sample for analysis of each blend or final batch. Minimum sample size for complete analysis is 500 g.
- c. Sampling for verification of previously certified blend or final batches. It may be required to verify specific criteria, such as nitrocellulose stability, deterioration in storage or volatile content, on a nitrocellulose blend or final batch after it has been certified. A minimum of 3 random transport containers shall be sampled from each blend or final batch. The sampling shall occur such that the transport containers selected for sampling represent the entire blend or final batch. These 3 samples shall be mixed together to produce a composite sample for analysis. Minimum sample size for complete analysis is 500 g.
- d. Sampling during pack-out for verification of total volatile content (water and/or ethanol) for all classes of nitrocellulose, workmanship, and fiberization quality for Class 1 nitrocellulose. At the packing facility, beginning with the first transport container, one grab sample shall be obtained every ten transport containers or less. In cases where the number of transport containers is less than 30, a minimum of three samples shall be taken representing the beginning (first transport container), the middle (a sample from the middle third of the

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batch) and the end (last third) of the transport containers packed. Minimum sample size required for analysis is 200 g.

e. Sampling for verification of oil and grease for Class 1 nitrocellulose. For Class 1 nitrocellulose, the verification of oil and grease content shall be performed on samples after pack-out. To do so, a minimum of three samples shall be taken representing the beginning (first transport container), the middle (a sample from the middle third of the batch) and the end (last third) of the transport containers packed. Each sample shall be verified individually according to the test measurements requirements of this specification. This sampling procedure may be coordinated with sampling procedure 4.3.4d. to minimize the number of samples taken.

4.4 Test measurements. When more than one measurement of any analytical test sample is required by this specification, the nitrocellulose description sheet IAW MIL-STD-1171 will contain the results of each measurement. All methods shall be conducted with the number of measurements of the composite sample required in 4.3.4 a., b. or c. for each blend or final batch as defined by the following:

a. Nitrogen content. If the precision of the test method is 0.015% nitrogen or less, two measurements of the composite sample from 4.3.4 a., b. or c. for each blend are required. If the precision of the test method is between 0.015 and 0.020% nitrogen, four measurements of the composite sample from 4.3.4 a., b. or c. for each blend is required. Precision of the test method shall not be greater than 0.020% nitrogen, unless specified in the contract or purchase order. The precision of the test method shall be the standard deviation of the same sample analyzed a minimum of twelve (12) times in a short period of time or the standard deviation of the residual from each average of 6 similar samples analyzed in duplicate. Once the precision of the test method has been established, a running precision shall be maintained based on the last 6 similar samples. Conformance with this specification will be based on the average for each blend or final batch.

b. Ether-alcohol solubles. Two measurements of the composite sample from 4.3.4 a., b. or c. for each blend shall be performed. If one measurement fails to meet the requirement, an additional three measurements of the same composite sample shall be performed. If two or more overall measurements fail the requirement, the blend or final batch shall be rejected.

c. Stability test at 132 °C or 134.5 °C. A minimum of two measurements of the composite sample from 4.3.4 a., b. or c. for each blend shall be performed. All measurements must meet the stability requirement set by this specification.

d. Acetone insolubles. Two measurements of the composite sample from 4.3.4 a., b. or c. for each blend shall be performed. If one measurement fails to meet the requirement, an additional three measurements of the same composite sample shall be performed. If two or more overall measurements fail the requirement, the blend or final batch shall be rejected.

e. Viscosity. A minimum of 3 balls shall be dropped in one prepared solution of the composite sample from 4.3.4 a., b. or c. for each blend shall be performed. Conformance with this specification will be based on the average for each blend or final batch.

f. Fineness. Conformance with this specification will be based on the average of a minimum of 1 measurement of the composite sample required in 4.3.4 a., b. or c. for each blend or final batch.

g. Fiberization quality.

i. For Class 1 nitrocellulose: Samples from 4.3.4 d. shall be analyzed with one measurement for each sample. All samples must meet the fiberization quality requirement set by this specification.

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ii. For Class 2 nitrocellulose: A duplicate analysis of the composite sample from 4.3.4 a., b. or c. for each blend shall be performed. If one measurement fails to meet the requirement, an additional three measurements of the same composite sample shall be performed. If two or more overall measurements fail the requirement, the blend or final batch shall be rejected.

h. Oil and grease content. Each sample from 4.3.4 e. shall be analyzed with one measurement for each sample.

i. Dry weight. Conformance with this specification will be based on a minimum of 1 measurement of the composite sample required in 4.3.4 b. for each blend or final batch.

j. Total volatile content (water and/or ethanol). Each sample required in 4.3.4 d. shall be analyzed with one measurement for each sample. Conformance with this specification will be based on the average for each blend or final batch with no individual sample below the required level.

k. Workmanship. Conformance with this specification will be based on a minimum of 1 inspection of the composite sample required in 4.3.4 a., b. or c. for each blend or final batch; however, any workmanship defects discovered in any other sample shall be counted with respect to conformance with this specification.

4.4.1 Drying nitrocellulose for analysis. Nitrocellulose of two different dryness levels is used in the subsequent testing methods:

- a. Dry nitrocellulose (moisture level <1.0%);
- b. Dry to constant weight nitrocellulose (moisture level <0.3%).

The specified dryness levels are based on the result of the Nitrocellulose Karl Fischer Titration Method. Dry nitrocellulose can be used for all testing methods except for nitrogen content. Dry to constant weight nitrocellulose shall be used for nitrogen content determination but can also be used in all other testing methods. If the nitrocellulose sample received is a slurry, excess water shall be removed to produce a wet nitrocellulose with a typical water content of 30-60% using any method suitable to reduce the water content prior to final drying of the sample. Proper care shall be taken to prevent contamination and maintaining the homogeneity of the nitrocellulose. The nitrocellulose shall not be washed with fresh water during this process.

4.4.2 Dry nitrocellulose for various analytical tests. Dry nitrocellulose with moisture level <1.0% can be prepared by further drying the wet nitrocellulose. Any suitable drying method can be used provided that (i) it has been previously confirmed that a moisture level of <1.0% (measured by the Nitrocellulose Karl Fischer Titration Method) is obtained for all nitrocellulose grades which are dried with this method, and that (ii) storage temperature and duration are sufficiently low to avoid significant decomposition of the nitrocellulose. A maximum temperature of 80 °C with storage time not exceeding 2 hours is regarded as acceptable. At lower storage temperatures, longer storage times are allowed, e.g., 6 hours at 70 °C or 20 hours at 60 °C. If lumps of fibers are present after drying, it is advisable to break them up. Recommended methods for further drying are the following.

a. Drying in oven at 60 to 80 °C. The wet nitrocellulose is spread on a paper or aluminum tray and dried in an oven set between 60 and 80 °C for 2 hours.

b. Drying in hot air blower at 65 °C. The wet nitrocellulose is placed in a cylindrical container, which is open or screened at one end and screened at the other. This container is set in a hot air blower through which air at a temperature of 65 ±10 °C and a pressure of 100 – 200 kPa (15 – 30 psi) is blown for not less than 15 minutes.

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The dried nitrocellulose is transferred to a tightly stoppered container for further storage. Alternatively, the dried nitrocellulose can be stored in a paper tray which is kept in an oven at 40 to 50 °C. This material is ready to be used in analytical procedures requiring dry nitrocellulose.

4.4.3 Dry to constant weight nitrocellulose for various analytical tests. The dry nitrocellulose can further be dried to constant weight. Any suitable method for further drying can be used provided that (i) it has been previously confirmed that the required moisture level of <0.3% (measured by the Nitrocellulose Karl Fischer Titration Method) is obtained for all nitrocellulose grades, and that (ii) storage temperature and duration are sufficiently low to avoid significant decomposition of the nitrocellulose. A maximum temperature of 95 to 105 °C with storage time not exceeding 2 hours is regarded as acceptable. Recommended methods for further drying are the following.

a. Drying in oven at 100 °C. The needed amount of dry nitrocellulose is transferred to a weighing bottle and placed in an oven set at 100 ± 5 °C for 1.5 hours.

b. Drying in vacuum oven at 65 °C. The needed amount of dry nitrocellulose is transferred to a weighing bottle and placed in a vacuum oven set at 65 ± 5 °C and a pressure not exceeding 5 kPa (kilo-Pascal) for 1.5 hours.

Directly after removal from the oven, the weighing bottle is tightly stoppered and transferred to a desiccator containing desiccant for cooling to room temperature. This material is ready to be used in analytical procedures requiring nitrocellulose dried to constant weight.

Note: For nitrocellulose obtained from fiberboard cellulose and containing more than 1% agglomerates: nitrocellulose may prove difficult to analyze for nitrogen content if agglomerates are not dissolved by sulfuric acid during the analysis. It may be necessary to pulverize the nitrocellulose using a wet milling grinder or any other safe methods of fiberization which are demonstrated to reduce the content in agglomerates below about 0.1% content in agglomerates.

4.4.4 First article product performance. The quantity of nitrocellulose required to test for product performance will be in accordance with the first article requirements of the product specification.

4.5 Methods of inspection. The tests in this section shall be performed using prescribed analytical procedures.

4.5.1 Nitrocellulose nitrogen content.

4.5.1.1 Titrant. Both the ferrous sulfate (FS) and ferrous ammonium sulfate (FAS) titrants can be used. The titrant solutions (FS or FAS) may be prepared in-house or commercially obtained with concentration similar to recommended value. Titrant solutions with other concentrations may be used if the titration is adjusted accordingly. The titrant temperature must either be kept constant to within ± 2 °C using thermoregulation of the buret, or of the titrator room, or the influence of temperature on titrant volume must be compensated by measuring titrant temperature during each titration and correcting the titrant volume to a reference temperature.

4.5.1.2 Electrodes. Three different electrode set-ups may be used (other electrodes that have been demonstrated to accomplish the detection may be use):

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- a. Platinum detection electrode and Ag/AgCl glass reference electrode in titration beaker.
- b. Combination electrode (platinum detection electrode and Ag/AgCl reference electrode both combined into one) in titration beaker.
- c. Separate electrodes: The gold detection electrode is put into the titration beaker, the platinum reference electrode is installed in the titrant delivery system, where the titrant works as reference with no salt bridge (the burette tip is the bridge).

4.5.1.3 Reagents. The following are the reagents used in this test method:

- a. Potassium nitrate, analytical standard (99.99% minimum purity); pulverized and dried at 135 °C for 4 hours.
- b. Sulfuric acid $\geq 95\%$, reagent or analytical grade.
- c. Distilled or deionized water.
- d. Ferrous sulfate (FS) reagent: 350 to 400 g of iron (II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) is dissolved in 800 mL of water. 1000 mL of a 1:1 volume/volume mixture of sulfuric acid and water are then added to make up to 2 liters.
- e. Ferrous ammonium sulfate (FAS) reagent: 480 to 550 g of ammonium iron (II) sulfate hexahydrate $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ are dissolved in 800 mL of 25% sulfuric acid (v/v), 1000 mL of a 25% sulfuric acid (v/v) and water are then added to make up to 2 liters.
- f. The titrant solutions (FS or FAS) shall be kept in a carboy, which is painted black, or in an amber bottle. Any air remaining in the solution shall be immediately flushed from the solution by bubbling nitrogen gas for 30 minutes. A blanket of nitrogen or a layer of toluene shall then be added to protect the solution from air oxidation.

4.5.1.4 Apparatus.

- a. An analytical balance with a minimum precision of 0.1 mg.
- b. A potentiometric titrating unit fitted with a delivery system capable of increment resolution equal or better than 1/2000 of the maximum volume of the selected burette, equipped with:
 - c. Calibrated Class A burette (see 6.12) or equivalent (25 mL maximum volume) filled with FS or FAS titrant.
 - d. Properly stirred titration cell, thermo-regulated if necessary to maintain the temperature of the titration below 40 °C, thermoregulated, as necessary. Once the titration method is established, the temperature reading device may be removed.
 - e. Detection electrode and reference electrode suitable for reduction oxidation titration detection. Electrodes shall be maintained following supplier recommendations.
 - f. Titration processor for mV end-point titration. Alternatively, a voltmeter or pH meter capable of measuring $\pm 1\text{V}$ to a resolution of 1 mV can be used for manual end point titration.

4.5.1.5 Procedure. The procedure applies both to the standardization with potassium nitrate and for nitrogen content determination of nitrocellulose. Standardization shall be performed (with a minimum of 3 measurements) within 24 hours using potassium nitrate. Each nitrocellulose sample shall be analyzed following measurement requirements (see 4.4).

To a clean, dry beaker, add a stir bar if required and a 75-150 mL portion of sulfuric acid. The beaker shall be placed in the titration unit which will enable the beaker to be stirred with a minimal vortex and thermo-regulated if necessary to prevent temperatures above 40 °C during titrant addition. A 0.25 to 0.50 g sample of dried to constant weight KNO_3 or nitrocellulose shall be weighed to the nearest 0.1 mg and added to the titration beaker. (Note:

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Sample may be added before the sulfuric acid and installation of the beaker on titration unit). Record the sample weight as W_{KNO_3} or W_{NC} . The beaker shall be stirred until the sample is entirely dissolved, but not less than 5 minutes. The titration is then started and may include the addition of a pre-dosed amount of titrant. The end-point shall be approached slowly with an increment no greater than 0.01 mL and be sustained for at least 10 seconds. The endpoint determination is dependent upon the type of electrode used and must be optimized by experimentation. The titrant volume (with temperature compensation applied if necessary) shall be recorded as $V_{\text{Titrant } KNO_3}$ or $V_{\text{Titrant } NC}$. The nitrogen equivalent factor of the solution (for KNO_3 titration) shall be calculated or the nitrogen content of the sample (for nitrocellulose sample) shall be calculated.

4.5.1.6 Calculation. The content shall be calculated using the following calculations or the equivalent. The nitrogen equivalent factor F of the solution may be calculated from the potassium nitrate (KNO_3) titration as follows:

$$F = \frac{g \text{ Nitrogen}}{mL \text{ Titrant}} = \frac{W_{KNO_3} * 0.13853}{V_{\text{Titrant } KNO_3}}$$

Where: F = equivalent factor, g nitrogen/mL titrant.
 W_{KNO_3} = weight of potassium nitrate (calibration standard), g.
 $V_{\text{Titrant } KNO_3}$ = volume of titrant for calibration, mL.
 0.13853 = Atomic Weight(N)/Molecular Weight(KNO_3) = 14.007 g/mol / 101.11 g/mol, no units.

The nitrogen content of the sample (in percentage by weight) shall be calculated from the nitrocellulose titration as follows:

$$\% \text{ Nitrogen} = 100 * \frac{V_{\text{Titrant } NC} * F}{W_{NC}}$$

Where: $V_{\text{Titrant } NC}$ = volume of titrant for NC sample, mL.
 F = equivalent factor, g nitrogen/mL titrant; see above.
 W_{NC} = weight of nitrocellulose, g.

4.5.2 Nitrocellulose ether-alcohol solubility.

4.5.2.1 Reagents. The following reagents shall be used for the filtration method of the ether-alcohol solubles test:

- a. Ethyl alcohol, absolute $\geq 99.8\%$ or alternate (see 6.6.1).
- b. Diethyl ether, reagent grade ($\geq 98\%$, with $\approx 2\%$ ethyl alcohol as stabilizer and ≈ 10 ppm of BHT inhibitor) or alternate (see 6.6.2).
- c. Wash solvent: mixture (2:1 volume) of diethyl ether and ethyl alcohol.

4.5.2.2 Apparatus. The following equipment shall be used in setting up the apparatus:

- a. Analytical balance or balances: for preparation, precision 0.01 g or better and for final weighing, precision 0.1 mg or better

- b. Container, 250 mL or 300 mL, stoppered/capped.
- c. Wrist action shaker or equivalent strong action mechanical shaker assembly.

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d. Silica sintered crucible (Porosity 60 microns or less) fitted with glass micro-fiber filter (porosity < 3 μm , Whatman 934/AH or equivalent); with pre-filtering aid (refractory ceramic filter aid Kaowool or equivalent) if required. Preparation of filtering crucible: The filtering crucible is prepared with the glass micro-fiber filter with the rough side up. If needed, pre-filtering aid is added just to cover the top of the glass micro-fiber filter. The so prepared crucible is washed with 50 mL of wash solvent and then dried in an oven at 100 ± 5 °C for a minimum of one hour or until it has reached a constant weight.

e. Filtering flask, e.g., Buchner flask, 2000 mL.

f. Wash bottle.

g. Drying oven at 100 ± 5 °C.

h. Desiccator containing desiccant (e.g., silica gel or calcium sulfate). When required for analysis, the crucible is cooled to room temperature and kept in a desiccator until used. For Grade A nitrocellulose or higher precision analysis: It is important that the time in desiccator for first weighing and final weighing be similar because of error caused by glass hydration during storage in desiccator.

4.5.2.3 Procedure. Approximately 1 g of dry nitrocellulose is weighed into the flask. The weight is recorded to the nearest 0.01 g as W_{NC} . A 75 mL portion of ethanol is added to the flask. The flask is then closed tightly with a solvent resistant stopper/cap. The flask is shaken, manually or using a mechanical shaker until the nitrocellulose is completely wet. A 150 mL portion of diethyl ether is then added to the flask. The flask is stoppered and shaken for a minimum of 60 minutes at room temperature (20 ± 5 °C) on a mechanical shaker (the presence of agglomerates may require a longer time). The flask is removed from the shaker. A dry filtering crucible is weighed using an analytical balance. The weight is recorded to the nearest 0.1 mg as W_{FC1} . The crucible is installed on the filtering flask fitted with an appropriate crucible holder. A gentle vacuum is applied. The supernatant is decanted onto the crucible. The remaining nitrocellulose is then quantitatively transferred into the crucible using the wash solvent in the wash bottle. Once the transfer is complete the crucible is washed with three or more portions of wash solvent. The filtering crucible shall be left under vacuum to evaporate most of the solvents. The crucible is then dried in the oven at 100 ± 5 °C for 60 minutes. The dried crucible is cooled to room temperature in a desiccator. The crucible is finally weighed using an analytical balance. The weight is recorded to the nearest 0.1 mg as W_{FC2} .

4.5.2.4 Calculation. The content shall be calculated using the following calculation or the equivalent. The ether-alcohol solubles of the nitrocellulose (in percentage by weight) are calculated from the increase in weight of the crucible as follows:

$$\% \text{Ether-Alcohol Solubles} = 100 * \left\{ 1 - \frac{(W_{\text{FC2}} - W_{\text{FC1}})}{W_{\text{NC}}} \right\}$$

Where: W_{FC1} = weight of empty filtering crucible, g.
 W_{FC2} = weight of filtering crucible and nitrocellulose, g.
 W_{NC} = weight of nitrocellulose sample, g.

4.5.3 Nitrocellulose stability.

4.5.3.1 Nitrocellulose stability test: 132 °C Stability test (Bergman-Junk).

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4.5.3.1.1 Reagents.

- a. Non-reactive grease; e.g., neutral petroleum jelly or grease as prepared by dissolving 35 g of paraffin wax in 65 g of warm liquid paraffin and allowing the resulting mixture to cool.
- b. Distilled or deionized water.
- c. Titration standard solution, sodium hydroxide, 0.1 M (or 0.01 M if manual titration with standard burette is performed), with factor determined to obtain exact molarity M_{NaOH} .
- d. Suitable pH indicator (if manual titration is used); e.g., methyl orange, methyl red, or Tashiro mixed indicator (methylene blue + methyl red 10:3).

4.5.3.1.2 Apparatus.

- a. Analytical balance, precision 10 mg or better.
- b. Stability bath: Oil or suitable fluid bath or metal block oven capable of maintaining the temperature of the stability tubes at 132 ± 0.2 °C. Temperature of bath has to be monitored with a calibrated thermometer or thermocouple (precision 0.1 °C) which is located in a test tube filled with inert material (e.g., sand); the test tube is placed in one of the thermowells. The inner diameter of each thermowell in the apparatus is 20.5 ± 0.5 mm. Depth of immersion of the stability test tubes is 150 mm. The bath has to be located in a safety cabinet with safety screens. Assembly of a linkage for remotely removing the tubes from the bath is recommended.
- c. Stability test tubes made of clear glass, approximately 17.5 mm inner diameter, 19.5 mm outer diameter, and 350 mm length; fitted with stoppers or caps.
- d. Powder funnel; metal funnel with long tube (to prevent electrostatic charging).
- e. Stoppers made from silicon rubber to fit into joint of stability test tubes.
- f. Glass funnel with paper filter or cotton wool.
- g. Conical titration flasks (Erlenmeyer flask), volume 100 mL or 300 mL depending on method used.
- h. Titration burette 10 – 25 mL; or automated potentiometric titration apparatus with pH-electrode and calibrated class A burette.

4.5.3.1.3 Procedure. Sample and interior of test tubes shall not be touched by bare hands. The test is to be performed in duplicate; with further repetition of test if the two results of the duplicate measurement differ by more than 0.1 mL NaOH 0.1 M / g.

Two portions of 2 ± 0.01 g each of dry nitrocellulose sample are transferred into the stability test tubes, preferably by a powder funnel. Each tube is tapped gently in order to settle the material, and any material adhering to the sides of the tubes is brushed down. If the nitrocellulose occupies a greater length than 5 cm, it has to be compressed to that length by means of a flat headed wood or glass rod.

The joints of the two tubes are lubricated with the non-reactive grease and fitted with absorption cups with caps. The cups are sealed off with 25 mL of water. The water may not enter the tube so that the contents of the stability tubes remain dry.

The two tubes are placed in the bath and maintained at a temperature of 132 ± 0.2 °C for 2 hours unless pronounced fuming is observed. In case of fuming, the test shall be stopped immediately and the duration of the heating period noted. The stability tubes are then carefully removed from the bath, preferably by a linkage, and left for at least 15 minutes to cool down behind a safety screen. Water will be drawn into the tubes during this time.

4.5.3.1.4 Standard filtering / titration procedure. The caps are removed from the tubes. The water from each cup is quantitatively transferred to a 300 mL titration flask; cap and cup are rinsed with water. Approximately 20 mL of water are added to each of the stability tubes.

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The tubes are stoppered and shaken vigorously. The tops of the stems of two 8 cm filter funnels are plugged with pads of absorbent cotton wool, followed by flushing the pads with water. The contents of the tubes are then filtered through these funnels into the appropriate titration flasks. The nitrocellulose on the filter funnels has to be washed thoroughly. The solutions in the flasks are then titrated with sodium hydroxide solution (0.1 M if automated potentiometric titration is used; 0.01 M if manual titration is performed) to the endpoint which is either detected by potentiometry (automated titration) or by color change of added pH indicator. The volume of sodium hydroxide standard solution is recorded in mL as V_{NaOH} . The number of milliliters of 0.1 M sodium hydroxide consumed per gram of nitrocellulose (volume at 0 °C and 101.3 kPa; rounded to the nearest 0.01%) is then calculated as follows:

$$\text{mL NaOH 0.1 M/g nitrocellulose} = 5 * V_{\text{NaOH}} * M_{\text{NaOH}}$$

Where: M_{NaOH} = molar concentration of sodium hydroxide solution, mol/L.
 V_{NaOH} = volume of sodium hydroxide solution consumed, mL.
 5 = 10/weight NC = 10/2 g = 5 g⁻¹; 10 is conversion factor to account for determining results in mL 0.1 M (instead of 1 M) NaOH.

The number of milliliters of nitrogen oxides (NO) evolved per gram of nitrocellulose is calculated as follows:

$$\text{mL NO/g nitrocellulose} = \text{mL NaOH 0.1 M/g nitrocellulose} * 2.24$$

Where: 2.24 = $0.1 \times 22.4 \text{ mL/mmol} = 2.24 \text{ mL/mmol}$; equation is based on the assumptions that nitrogen oxide evolves as NO and that NO is ideal gas; according to the ideal gas law, 1 mol of ideal gas occupies a volume of 22.4 l at 0 °C and standard atmosphere (101.3 kPa); 1 mL 0.1 M NaOH is equivalent to 0.1 mmol = 2.24 mL NO

4.5.3.1.5 Alternative filtering / titration procedure. (requires that test tubes have engraved marks at 50 mL): The caps are removed and rinsed with water; the rinsing water is poured into the respective cup. Then the cups are removed from the tubes, and, after removing of the grease from the joints of the tubes, the content of each cup is poured into the respective tube, followed by rinsing of the cups with water. Water is added into each tube up to the volume of exactly 50 mL. The tubes are closed with rubber plugs and shaken vigorously. The content of each tube is then poured into a funnel with paper filter. The first approximately 10 mL of the filtrate are discarded, and then 25 mL of the following clear filtrate are collected in a graduated cylinder. Exactly 25 mL of each of these filtrates are transferred from the graduated cylinder into a 100 mL titration flask and titrated with sodium hydroxide solution (0.1 M if automated potentiometric titration is used; 0.01 M if manual titration is performed) to the endpoint which is either detected by potentiometry (automated titration) or by color change of added pH indicator. The volume of sodium hydroxide standard solution is recorded in mL as V_{NaOH} . The number of milliliters of 0.1 M sodium hydroxide consumed per gram of nitrocellulose (volume at 0 °C and 101.3 kPa; rounded to the nearest 0.01%) is then calculated as follows:

$$\text{mL NaOH 0.1 M/g nitrocellulose} = 5 * M_{\text{NaOH}} * (V_S - V_B)$$

Where: M_{NaOH} = molar concentration of sodium hydroxide solution, mol/l.

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V_S	=	volume of sodium hydroxide solution for heated sample, mL.
V_B	=	volume of sodium hydroxide solution for unheated blank sample, mL.
5	=	$10/\text{weight NC} = 10/2 \text{ g} = 5 \text{ g}^{-1}$; 10 is conversion factor to account for determining results in mL 0.1 M (instead of 1 M) NaOH.

The number of millilitres of nitrogen oxides (NO) evolved per gram of nitrocellulose is calculated as follows:

$$\text{mL NO/g nitrocellulose} = \text{mL NaOH 0.1 M/g nitrocellulose} * 2.24$$

Where: 2.24 = $0.1 \times 22.4 \text{ mL/mmol} = 2.24 \text{ mL/mmol}$; equation is based on the assumptions that nitrogen oxide evolves as NO and that NO is ideal gas; according to the ideal gas law, 1 mol of ideal gas occupies a volume of 22.4 l at 0 °C and standard atmosphere (101.3 kPa); 1 mL 0.1 M NaOH is equivalent to 0.1 mmol = 2.24 mL NO

4.5.3.2 Nitrocellulose stability test: 134.5 °C Heat test (Methyl Violet Paper Test).

4.5.3.2.1 Reagents.

a. Standardized methyl violet test papers approximately 70 ± 1.0 mm long and 20 ± 0.6 mm wide (see 6.16) or methyl violet test papers prepared and tested using the following method:

i. Preparation of the indicator solution. To prepare 100 mL of indicator solution (note: if different amount of solution is required, it can be prepared while maintaining these proportions): 0.250 g of basic rosaniline (equivalent to CAS number 632-99-5) is weighed into a porcelain dish, and about 10 mL of reagent grade acetic acid is added. The dish is heated on a water bath until all excess of acid is removed. In a 100 mL graduate cylinder, 0.168 g of crystal violet (equivalent to CAS number 548-62-9) is dissolved in 30 mL of high purity water and 5.0 g (4 mL) of reagent grade glycerine is added. The content of the porcelain dish is added to the cylinder using ethanol (minimum 95% v/v) and adjusted to produce 100 mL of solution. The solution is mixed thoroughly.

ii. Preparation of the methyl violet paper. Sheets of paper are prepared by cutting filter papers (equivalent to Whatman 597, typically 580 mm x 580 mm with approximately 8.5 mg/cm^2) into square parts that will fit into a low edge dish large enough to fit the cut sheet (typically cut in 4 square parts about 290 mm by 290 mm). In a fume-hood, the methyl violet solution is poured into the low edge dish. Separately, each cut sheet of paper is dipped completely into the solution for about 30 seconds. The strip is removed from the solution and the wet sheet of paper rotated vertically until the solution stops dripping (excess alcohol will evaporate in about 1 minute). The strip is hung up overnight to dry in a room free from deleterious fumes. When dry, the strips are cut in the size of 70 ± 1.0 mm long and 20 ± 0.6 mm wide. Once certified, they are kept in tightly closed amber glass bottles or opaque plastic bottles with a maximum of 200 papers per bottle. The bottle shall be kept closed, stored at room temperature, and out of direct light at all times except to briefly extract indicator papers.

iii. Certification of the methyl violet paper. A minimum of one paper from each 200 max bottle is tested for the content in water and shall be 7.5 to 15% water content by oven drying. If required, the paper may be rehydrated by keeping the paper in a controlled humidity chamber controlled at 60 to 80% RH until the correct water content is obtained. To confirm

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that the reactivity of the methyl violet paper is acceptable, a minimum of 1 paper from each 200 max bottle shall be tested using nitrogen dioxide gas of known concentration in air between 1500 and 2500 ppm (v/v). The gas may be obtained already diluted and certified or obtained by dilution using pure nitrogen dioxide. The gas concentration shall be known with an accuracy of $\pm 2.5\%$. Based on the concentration of the nitrogen dioxide gas, the required flowrate for an end-point centered at 55 minutes is given by: $\text{Flowrate (ml/min)} = 83636 / \text{Gas concentration in ppm (v/v) of nitrogen dioxide gas}$. The flowrate shall be maintained within ± 1.5 ml/min of the calculated value during the certification of the paper. The paper is tested using the standard gas and a cylindrical flow cell of about 30 ml containing one paper (the flow cell diameter is similar to the MV paper width). The end-point is obtained when the paper is completely salmon pink after 55 ± 7 minutes. Only the batches that meet those 2 criteria (water content and reaction time) will be considered certified methyl violet paper. The paper shall be stored at room temperature and in the shade. The maximum shelf-life of the indicator papers in a sealed bottle is 5 years. Once the bottle is open, the shelf-life of the bottle's contents is reduced to 1 year. After 1 year, the water content of the paper shall be verified and adjusted, if necessary. The bottle containing the verified indicator papers shall be given another 1 year of shelf-life. Under no circumstances shall the indicator paper shelf-life be extended beyond 5 years after manufacture.

4.5.3.2.2 Apparatus. The following equipment shall be used in the apparatus for 134.5 °C Heat Test (Methyl violet paper test):

- a. Analytical balance, precision 0.01 g or better.
- b. Stability bath: Water-ethylene glycol bath, oil bath, or metal block oven capable of maintaining the temperature of the stability tubes at 134.5 ± 0.5 °C. Temperature of bath has to be monitored with a calibrated thermometer or thermocouple (precision 0.1 °C) which is located in a test tube filled with inert material (e.g., sand); the test tube is placed in one of the thermowells. The inner diameter of each thermowell in the apparatus shall be 19 ± 0.5 mm. Depth of immersion of the stability test tubes shall be such that no more than 6 to 7 mm of the tubes project above the bath.
- c. Stability test tubes made of clear glass, approximately 15 mm inner diameter; 18 mm outer diameter; and 290 mm length.
- d. Powder funnel; metal or conductive plastic funnel with a long tube (to prevent electrostatic charging).
- e. Corks, each containing one breather hole 4 mm in diameter (or notch of equivalent area).

4.5.3.2.3 Procedure. Sample and interior of test tubes shall not be touched by bare hands. The test is to be performed in duplicate; with further repetition of test if the two results of the duplicate measurement differ by more than 5 minutes.

Two portions of 2.5 ± 0.01 g each of dry nitrocellulose sample are transferred into the stability test tubes, preferably by a powder funnel. Each tube is tapped gently in order to settle the material, and any material adhering to the sides of the tubes is brushed down. If the nitrocellulose occupies a greater length than 5 cm, it has to be compressed to that length by means of a flat headed rod. Into each tube a piece of the test paper is placed vertically so that the lower end of the paper is 25 mm above the specimen. Then a cork is placed in each tube. The two tubes are placed in the bath and maintained at a temperature of 134.5 ± 0.5 °C.

In order to determine the test time, the test papers are examined after the first 20 minutes in the bath, and thereafter at 5 minute intervals. For each examination of test papers, the tubes are lifted half way out of the bath to monitor test paper color change, and

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quickly replaced. When the test paper in any tube has changed color completely to salmon pink, the test is considered complete. The test time is then recorded (for example, if the violet paper is not completely changed in 25 minutes, but is completely changed in 30 minutes, the time of the test is recorded as 30 minutes). The test is discontinued when the salmon pink end point is attained in any of the papers.

4.5.3.2.4 Calculation. The test time in minutes of stability of the nitrocellulose.

4.5.4 Nitrocellulose acetone insolubles.

4.5.4.1 Reagents.

- a. Ethyl alcohol, absolute $\geq 99.8\%$ or alternate (see 6.6.1).
- b. Acetone, reagent grade or equivalent, filtered if necessary or alternate (see 6.6.3).

4.5.4.2 Apparatus.

- a. Analytical balance or balances: for preparation, precision 0.01 g or better and for final weighing, precision 0.1 mg or better.
- b. Container, 250 mL or 300 mL, stoppered/capped.
- c. Mechanical shaker assembly.
- d. Silica sintered crucible (Porosity 60 microns or less) fitted with glass micro-fiber filter (porosity $< 3 \mu\text{m}$, Whatman 934/AH or equivalent); with pre-filtering aid (refractory ceramic filter aid Kaowool or equivalent) if required. Preparation of filtering crucible: The filtering crucible is prepared with the glass micro-fiber filter with the rough side up. If needed, pre-filtering aid is added just to cover the top of the glass micro-fiber filter. The so prepared crucible is washed with 50 mL of acetone and then dried in an oven at $100 \pm 5^\circ\text{C}$ for one hour or until it has reached a constant weight. The crucible is finally cooled down to room temperature and kept in a desiccator until used.
- e. Filtering flask, e.g., Buchner flask, 2000 mL.
- f. Drying oven at $100 \pm 5^\circ\text{C}$.
- g. Desiccator containing desiccant (e.g., silica gel, calcium chloride, or calcium sulfate).

4.5.4.3 Procedure. Approximately 2 g of dry nitrocellulose is weighed into the flask. The weight is recorded to the nearest 0.01 g or better as W_{NC} . A 10 mL portion of ethanol is added to the flask to moisten the material before adding a 150 mL portion of acetone. The flask is then closed tightly with a solvent resistant stopper/cap. The flask is shaken at room temperature ($20 \pm 5^\circ\text{C}$) until dissolution is complete (usually 2 hours of mechanical shaking).

A dry filtering crucible is weighed using an analytical balance. The weight is recorded to the nearest 0.1 mg as W_{FC1} . The crucible is installed on the filtering flask fitted with an appropriate crucible holder. A gentle vacuum is applied. The solution is poured onto the crucible. Any residue in the flask is then transferred onto the crucible by means of several small rinses of acetone. The filtering crucible is then left under vacuum to evaporate most of the solvents. The crucible is then dried in the oven at $100 \pm 5^\circ\text{C}$ for 60 minutes. The dried crucible is cooled to room temperature in a desiccator. The crucible is finally weighed using an analytical balance. The weight is recorded to the nearest 0.1 mg as W_{FC2} .

4.5.4.4 Calculation. The content shall be calculated using the following calculations or the equivalent. The acetone insolubles of the nitrocellulose (in percentage by weight) are calculated from the increase in weight of the crucible as follows:

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$$\% \text{Acetone Insolubles} = \frac{(W_{\text{FC2}} - W_{\text{FC1}})}{W_{\text{NC}}} * 100$$

Where: W_{FC1} = weight of empty filtering crucible, g.
 W_{FC2} = weight of filtering crucible and nitrocellulose, g.
 W_{NC} = weight of nitrocellulose sample, g.

4.5.5 Nitrocellulose viscosity.

4.5.5.1 Reagents.

- a. Ethyl alcohol, absolute; $\geq 98\%$ or alternate (see 6.6.1).
- b. Acetone, reagent grade or alternate (see 6.6.3).

4.5.5.2 Apparatus.

- a. Analytical balance, precision 0.01 g or better.
- b. Container 250 mL or 500 mL, with leak-proof stopper/cap.
- c. Mixing device capable of mixing so that the material will flow rather than mix with air. The optimal rate of rotation to produce a fast dissolution is approximately 12 rotations per minutes and the optimum angle of rotation for the flask is at an angle of 45° to the plane of rotation.
- d. Viscometer: Falling ball viscometer or approved equivalent. This viscometer consists of a glass tube approximately 356 mm (14 inches) in length with an internal diameter of 25 mm (1 inch). The lower end of the tube is closed by means of a tightly fitting solvent resistant stopper. Two circles are etched on the tube approximately 50 mm (2 inches) from each end making 2 marks exactly 254 mm (10 inches) apart.
- e. Spheres: The spheres used in the viscometer are ordinary 5/16 inch steel ball bearings, having a diameter of 7.93 to 7.95 mm weighing 2.025 g to 2.045 g.
- f. Thermostat and water bath capable of maintaining a temperature of $25 \pm 0.2^\circ \text{C}$ in the bath. The bath may accommodate one or several viscometers.
- g. Stopwatch or suitable time measuring device capable to 0.01 seconds.

4.5.5.3 Calibration. To calibrate the spheres, the viscometer tube is filled with reference oil or reference solution of known viscosity (sugar, glycerin, etc.) and immersed in a constant temperature bath maintained at $25 \pm 0.2^\circ \text{C}$ until its contents reach equilibrium. The spheres are then individually dropped through the tube. The time of passage between the etched circles is noted.

The tube factor K is determined as follows:

$$K = \frac{N}{t * (D - d)}$$

Where: N = viscosity of reference solution in centipoises.
t = time in seconds (0.01 seconds).
D = density of the spheres in g/mL at 25°C .
d = density of the reference solution in g/mL at 25°C .

When one sphere has been selected by careful measurement, it shall be weighed. The other spheres chosen shall be of approximately the same weight. The other spheres have to be

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calibrated and only those are retained which have the same constant within plus or minus 5% of that of the first sphere selected. Specific gravity and density are at 25 °C, referred to water at 4 °C.

4.5.5.4 Procedure. A 20 ± 0.01 g portion of dry nitrocellulose is transferred to the container. A 20 g (or 25 mL) portion of ethyl alcohol is added and the mixture shaken gently to wet the mixture as thoroughly as possible. A 160 g (or 205 mL) portion of acetone is then added and the container closed with a leak proof stopper. The container is fastened to a mixing device and mixed until solution is homogeneous by visual inspection (typical mixing time is about 4 hours when mixing is done in optimum conditions: the optimal rate of rotation to produce a fast dissolution is approximately 12 rotations per minutes and the optimum angle of rotation for the flask is at an angle of 45° to the plane of rotation.).

The lower end of the glass viscometer tube is tightly closed by means of a solvent resistant stopper. The tube is then filled with nitrocellulose solution until the liquid level is at least 25 mm above the upper circle. The top of the tube is sealed with a stopper to prevent evaporation of the acetone during equilibration. In order to reach equilibrium, the tube is then immersed in the bath maintained at 25 ± 0.2 °C for at least 1 hour.

If the bath is non-transparent: At the end of this period the tube is withdrawn from the bath, wiped dry and then supported in a vertical position by means of a clamp and ring stand. The viscosity measurement must be completed within 5 minutes of withdrawing the tube from the bath. If the bath is transparent: The test is performed with the tube inside the bath. The top stopper is removed and, if a skin or crust is observed, then a glass rod is used to remove it.

Three or more calibrated spheres are allowed to fall separately through the solution, and the time required for each sphere to fall from the upper to the lower etched circle is noted by means of a stopwatch. If necessary, the axis of the tube shall be adjusted until the spheres fall concentrically, thus showing the glass tube to be in a vertical position. The arithmetic average in seconds, of the time required for the spheres to fall from the upper to the lower etched circle, is recorded.

4.5.5.5 Calculation. The viscosity of the nitrocellulose (in centipoises) is calculated from the average time as follows:

$$(n) \text{ viscosity} = K * t * [D - d]$$

Where: n = viscosity of nitrocellulose solution in centipoises.
K = viscometer constant expressed in centipoises.
t = average falling time in seconds.
D = density of the spheres in g/mL at 25 °C.
d = density of the liquid in g/mL at 25 °C.

4.5.6 Nitrocellulose fineness.

4.5.6.1 Apparatus.

a. Fineness measuring cylinder: 250 mL graduated volume (about 34 cm total height, about 24 cm total graduation height and about 4.1 cm outside diameter), leakproof stoppered, graduated in 2 mL divisions.

b. Analytical balance, precision 0.01 g or better.

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c. Overhead shaker capable to rotate the fineness measuring cylinder end over end at a nominal rate of about 12 rotations per minute.

4.5.6.2 Procedure. A 10 ± 0.01 g portion of dry nitrocellulose is transferred to the measuring cylinder with a 100 to 150 mL portion of distilled or deionized water; this addition may be done directly into the fineness cylinder using a funnel or by pre-mixing the nitrocellulose in a beaker with some water prior to transfer. The cylinder is sealed with a leak proof stopper properly secured and then the fineness cylinder is rotated end over end at a rate of about 12 rotation/minutes for 30 minutes (alternatively, the cylinder may be shaken manually: holding the stopper, the tube in position horizontally and shaken vigorously in the long axis of the tube for 30 seconds or more at a rate of about 1 shake per second). The cylinder is removed from the overhead shaker and installed up-right on a flat surface. The stopper is removed and the stopper and sides of the cylinder are rinsed down with water until the volume of the suspension reaches 230 to 240 mL. The stopper is replaced and the cylinder shaken a few times by inverting up-side down to mix the water and nitrocellulose evenly. The cylinder is again installed up-right on a flat surface with no vibration. The stopper is removed and the inner top side of the cylinder washes with a few mL of water so the total volume inside the tube is about 250 mL and the top portion of the cylinder, above the water, is clean of nitrocellulose. The cylinder is then allowed to stand for 1 hour. At the end of this time, the volume in mL occupied by the nitrocellulose is obtained by reading the graduated scale of the cylinder and estimating the top of the nitrocellulose. The volume shall be measured to a precision of 1 mL.

4.5.6.3 Calculation. The fineness of the nitrocellulose is the volume (in mL).

4.5.7 Nitrocellulose fiberization quality.

4.5.7.1 Nitrocellulose total content in sinkers by elutriation.

4.5.7.1.1 Reagents. Tap water - the temperature of the water is critical for the elutriation to be successful. Warm water is degassed more readily when exiting the injection tube in the elutriator creating numerous micro-bubbles which stick to agglomerates affecting their buoyancy. Water temperature shall be between 10 and 25 °C. If necessary, the equipment and procedure can be verified using a control sample (with a known quantity of sinkers).

4.5.7.1.2 Apparatus.

a. Fineness cylinder (elutriator), 250 mL graduated volume (about 34 cm total height, about 24 cm total graduation height and about 4.1 cm outside diameter) and leak proof stopper.

b. Overhead shaker: equipment to rotate the fineness tube end over end at a nominal rate of about 12 rotations per minutes.

c. Beaker approximately 500 mL.

d. Stainless steel agitating rod about 30 cm in length and about 2 mm in diameter

e. Imhoff sedimentation cone of 1000 mL (similar to Brandtech Scientific catalog number 388000). The cone is approximately 470 mm high and cone opening on top is about 116 mm internal diameter.

f. Stainless steel tube approximately 48 cm in length and approximately 4.6 mm inside diameter and approximately 6.4 mm outside diameter.

g. Lab stand, clamps and ring holders to support the cone level and vertical.

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- h. Flexible tygon tubing approximately 6 mm inside diameter (approximately 2 meters) or equivalent.
- i. 1 plastic ball valve with barbs approximately 6 mm outside diameter (similar to Cole-Palmer catalog number R-01377-30).
- j. 1 plastic Y connector with barbs approximately 6 mm outside diameter (similar to Cole-Palmer catalog number ED-06296-20).
- k. One (1) connection to tap water with adapter with barb approximately 6 mm outside diameter to connect a tygon tubing or equivalent.
- l. Crucible (fritted or Gooch) about 2 cm in diameter. Crucibles are dried in an oven at about 100 °C for about 1 hour prior to use.
- m. Crucible filtration set up.
- n. Desiccator.
- o. Analytical balance, precision 0.1 mg or better.
- p. Laboratory ultrasonic bath or probe.
- q. Flowmeter with metric-reading scale about 100 to 1500 mL/min (similar to direct reading flowmeter from Cole-Palmer catalog number R-32461-40).

4.5.7.1.3 Procedure.

- a. Set up the elutriator, the lab stand, clamps and ring holders to support the cone level and vertical above a collection system to filter system or drain which can receive/dispose of water contaminated with nitrocellulose fibers.
- b. Connect the stainless steel flow tube to the tap water using the tap water adapter which enables connection of a plastic flexible tubing. The flowrate of the water coming out of the stainless tubing is monitored using a flowmeter and controlled using a plastic ball valve installed in series on the water feed line. A derivation using a Y-connector can be installed between the flowmeter and the water tap connector to prevent over pressurization of the water line.
- c. Set up the stainless steel flow tube in the center of the Imhoff sedimentation cone with water outlet of the stainless tube about 45 to 50 mm from the bottom of the cone. Ensure that the stainless tube is relatively centered by using holders to keep the tube centered.
- d. Sample preparation.
 - i. Transfer about 4g of dry nitrocellulose or the equivalent into a clean 250 mL fineness tube. Weigh and record the weight to a minimum precision to ± 0.01 g ($W_{\text{Dry NC}}$).
 - ii. Wet the nitrocellulose with 200 mL of water and seal the tube with a stopper.
 - iii. Install on a rotational shaker which rotate the fineness tube end over end for about 30 minutes at a rate of about 12 rotations/minutes.
- d. Main elutriator extraction procedure.
 - i. Install the water feed stainless steel tube into the cone. Ensure that the tube is centered.
 - ii. Transfer sample to the elutriation cone and rinse container into the cone.
 - iii. Start the water flow. Set and maintain the water flow rate to approximately 950 mL/min.
 - iv. Elutriate the nitrocellulose for 15 minutes. During the elutriation, the stainless steel rod can be used to agitate the slurry at regular interval of about 3 minutes to help break-up the flocculation, speed up the separation and clean the side of the cone. This agitation must be very slow and for short periods of time (about 3 to 6 seconds). The central tube can also be agitated to remove clinging nitrocellulose.
 - v. Once the elutriation time is completed, stop the flow of water and remove the water feed stainless steel tube from the cone.

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- e. Collecting sinkers (agglomerates and aggregates)
 - i. Using the stainless steel rod wipe the side of the cone to ensure that all sinkers are in suspension.
 - ii. Decant the solids for about 5 minutes.
 - iii. Transfer about 200 to 400 mL of the water and all the nitrocellulose sinkers into the 500 mL beaker.
- f. Beaker extraction
 - i. Using the stainless steel rod, agitate the slurry in the 500 mL beaker to suspend all the solids. Wait about 15-30 seconds so the all the heavier materials decant. Using another beaker: transfer by pouring water containing floating free fibers down to about 100 mL of water and heavier material left in the 500 mL beaker. Care shall be taken not to discard any of the heavier material (sinkers): the water transferred out can be visual inspected to ensure only free fibers were transferred out. If necessary, transfer back the water just removed and repeat the operation.
 - ii. Add about 100 to 200 mL of fresh tap water and repeat the last operation a minimum of 3 times, after which the remaining solids in the beaker shall consist essentially of sinkers with a minimal quantity of free fibers visible.
- g. Quantification by gravimetric assessment:
 - i. Obtain a crucible fitted with a filter paper that was dried to constant weight and maintained in a desiccator to room temperature. Weigh to a precision of 0.1 mg and record the weight of the crucible (W_{crucible}).
 - ii. Filter the contents of the 500 mL beaker in the prepared crucible.
 - iii. Transfer the crucible with the sinkers into a drying oven and dry to constant weight.
 - iv. Transfer the crucible with the sinkers into a desiccator for cooling.
 - v. Once at room temperature weigh and record (to a precision of 0.1mg) the weight of the crucible with the sinkers ($W_{\text{crucible+NC}}$).

Note: if required, keep the crucible and the sinkers to analyze the *nitrocellulose total content in agglomerates*.

4.5.7.1.4 Calculation.

$$\text{Content in sinkers at 10 cm/min} = \frac{W_{\text{crucible+NC}} - W_{\text{crucible}}}{W_{\text{Dry NC}}} * 100$$

4.5.7.2 Nitrocellulose total content in agglomerates by elutriation.

4.5.7.2.1 Apparatus, reagent, set-up of equipment. This method uses the same apparatus, reagents, and procedure as the nitrocellulose total content in sinkers method (see 4.5.7.1) and the crucible containing the sinkers from 4.5.7.1.3 is used at this point to quantify the total content in agglomerates.

4.5.7.2.2 Procedure.

- a. Sonication
 - i. The sinkers contained in the crucible are transferred by water wash into a 500 mL beaker with about 100 – 200 mL of tap water.
 - ii. Install the 500 mL beaker containing the sinkers into a sonication bath or install the sonication probe to sonicate the contents of the beaker. The sonication is done for about 15 minutes. This can be adjusted depending on the quality of the nitrocellulose. If using

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a sonication bath: about 2 cm of water should be present in the bath and about 100 to 200 mL of water in the beaker.

iii. The solution is now ready for the final cleaning after sonication.

b. Final cleaning after sonication

i. Using the stainless steel rod, agitate the slurry in the 500 mL beaker to suspend all the solids. Wait about 15-30 seconds so the all the heavier materials decant. Using another beaker: transfer by pouring water containing floating free fibers down to about 100 mL of water with heavier material left in the 500 mL beaker. Care shall be taken not to discard any of the heavier material (agglomerates): the water transferred out can be visually inspected to ensure only free fibers were transferred out. If necessary, transfer back the water just removed and repeat the operation.

ii. Add about 100 to 200 mL of fresh tap water and repeat the last operation a minimum of 3 times. After which, the remaining solids in the beaker shall consist essentially of agglomerates with a minimal quantity of free fibers visible.

c. Quantification by gravimetric assessment:

i. Obtain a crucible fitted with a filter paper that was dried to constant weight and maintained in a desiccator to room temperature. Weigh to a precision of 0.1 mg and record the weight of the crucible (W_{crucible}).

ii. Filter the content of the 500 mL in the prepared crucible.

iii. Transfer the crucible with the agglomerates into a drying oven and dry to constant weight.

iv. Transfer the crucible with the agglomerates into a desiccator for cooling.

v. Once at room temperature, weigh and record (to a precision of 0.1 mg) the weight of the crucible with the agglomerates ($W_{\text{crucible+agglo}}$).

4.5.7.2.3 Calculation.

$$\text{Total Content in Agglomerates} = \frac{W_{\text{crucible+agglo}} - W_{\text{crucible}}}{W_{\text{Dry NC}}} * 100$$

Where: $W_{\text{Dry NC}}$ = weight of dry nitrocellulose, g.
 W_{crucible} = weight of the crucible, g.
 $W_{\text{crucible+agglo}}$ = weight of the crucible with the agglomerates, g.

4.5.8 Nitrocellulose oil and grease content. Testing of oil and grease content of the nitrocellulose is usually performed on dewatered nitrocellulose samples with a nominal 30% water wet weight. Alternatively, the nitrocellulose can be dried at room temperature, but not at elevated temperatures to avoid loss of contaminants.

4.5.8.1. Reagents.

a. Dichloromethane, reagent grade.

4.5.8.2. Apparatus.

- a. Analytical balance, precision 0.1 mg or better.
- b. Balance, precision 0.01 g or better.
- c. Beakers, approx. 400 mL. Conditioning of beakers: The beakers are placed in the oven and kept at a minimum of 70 °C for a minimum of two hours. The beakers are then removed from the oven and placed in the desiccator until used.
- d. Glass or polyethylene containers with lids, approx. 500 mL.
- e. Volumetric dispenser or 200 mL pipette.

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- f. Mechanical shaker.
- g. Wash bottle.
- h. Glass funnels with paper filter.
- i. Sand bath or hot plate or steam chest.
- j. Drying oven at a minimum temperature of 70 °C.
- k. Desiccator containing desiccant (e.g., silica gel, calcium chloride, or calcium sulfate).

4.5.8.3 Procedure. 35 ± 1 g of nitrocellulose is weighed into a glass or polyethylene container. The sample weight is recorded as W_{NC} to the nearest 0.01 g. 200 mL of dichloromethane is added to the container using either the volumetric dispenser or the pipette. The container is then capped securely. 200 mL of dichloromethane are added to another container for a blank. The sample and blank are shaken vigorously for 30 to 35 minutes using a mechanical shaker. Vigorously shaking is a key element for ensuring proper contact between the water wet nitrocellulose and the dichloromethane. Failure to maintain vigorous shaking will result in significant variability in outcomes. After shaking, the containers are transferred to an operating hood, where the lids are opened slowly to allow pressure to be released.

The dry evaporation beakers are weighed using an analytical balance. The weights are recorded to the nearest 0.1 mg as W_{S1} (sample) and W_{B1} (blank). The beakers are then placed below glass funnels with paper filter. The contents of the containers are then quantitatively transferred into the respective funnels. The sample and blank are filtered until the filtrate stops dripping. The evaporation beakers with contents are placed on a warm surface (sand bath or hot plate) or steam chest inside an exhaust hood to evaporate to dryness. The evaporation beakers are transferred to an oven and dried for 30 minutes at a minimum temperature of 70 °C. The beakers are then cooled to room temperature in a desiccator. The evaporation beakers are finally weighed using an analytical balance. The weights are recorded to the nearest 0.1 mg as W_{S2} (samples) and W_{B2} (blank).

4.5.8.4. Calculation. The content shall be calculated using the following calculations or the equivalent. The content of oil and grease is calculated as follows:

$$\% \text{Oil \& Grease} = \frac{[(W_{S2} - W_{S1}) - (W_{B2} - W_{B1})]}{W_{NC} * [1 - (\% \text{Volatiles} / 100)]} * 100$$

Where: W_{NC} = original wet NC sample weight, g.
 W_{S1} = initial weight of sample beaker, g.
 W_{S2} = final weight of sample beaker, g.
 W_{B1} = initial weight of blank beaker, g.
 W_{B2} = final weight of blank beaker, g.
 $\% \text{ Volatiles}$ = percentage of total volatile content from 4.5.10.

4.5.9 Nitrocellulose dry weight. The dry nitrocellulose weight shall be determined by subtracting the total volatile weight of the packed nitrocellulose from the net weight of the wetted nitrocellulose in the container. The total volatile weight shall be calculated using the measurements made on those samples that best represent the total volatiles of the packed transport container.

$$\text{Nitrocellulose Dry Weight} = W_{NCwet} * \left(1 - \frac{W_{NCvolatiles}}{100}\right)$$

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Where: W_{NCwet} = net weight of wetted nitrocellulose in container, lbs.
 $W_{NCvolatiles}$ = total volatile content contained in container from 4.5.10, %

4.5.10 Nitrocellulose total volatile content.

4.5.10.1 Nitrocellulose total volatile content: oven method.

4.5.10.1.1 Apparatus.

- a. Analytical balance, precision 0.01 g or better.
- b. Dish approximately 15 cm in diameter or drying thimble.
- c. Heating cabinet (oven or steam chest) maintained at the selected target temperature ± 5 °C. The target temperature is a temperature above 65 °C and no more than 105 °C which has been demonstrated to adequately and safely dry nitrocellulose to constant weight in a reasonable time period.
- d. Desiccator containing desiccant (e.g., silica gel, calcium chloride, or calcium sulfate).

4.5.10.1.2 Procedure. Weigh a dried dish or drying thimble and record to the nearest 0.01 g as W_{DISH} . A 5 to 30 g portion of wet nitrocellulose (containing water and/or alcohol) is added into a dried dish or drying thimble. The weight of the dried dish and the wet nitrocellulose combined is recorded to the nearest 0.01 g as $W_{NC\ wet}$. The dish and sample are then dried to constant weight in a heating cabinet. The time until each nitrocellulose type reaches constant weight under the given conditions might be previously determined and kept constant. The dish/thimble and sample are then removed and cooled to room temperature in a desiccator. The dry nitrocellulose and dish/thimble is weighed and recorded to the nearest 0.01 g as $W_{NC\ dry}$.

4.5.10.1.3 Calculation. The total volatile content of the nitrocellulose (in percentage by weight) is calculated from the loss in weight as follows:

$$\% \text{ Total Volatiles} = \frac{W_{NC\ wet} - W_{NC\ dry}}{W_{NC\ wet} - W_{Dish}} * 100$$

Where: $W_{NC\ wet}$ = original weight of specimen plus drying dish, g.
 $W_{NC\ dry}$ = weight of specimen plus drying dish after drying to constant weight, g.
 W_{DISH} = weight of dried dish or drying thimble, g.

4.5.10.2 Nitrocellulose total volatile content: Moisture Analyzer Method.

4.5.10.2.1 Apparatus. A moisture analyzer using IR or halogen heat source (similar to Sartorius MA-45 moisture analyzer) shall be used.

4.5.10.2.2 Procedure.

a. Preparation of apparatus. The instrument is programmed to heat the sample chamber to a selected target temperature between 65 and 105 °C for a period of time that has been demonstrated to adequately and safely dry nitrocellulose to constant weight. The use of sodium tartrate as a traceable, analytical standard is recommended.

b. Sample analysis. The balance sample container is tare weighed and 2 to 10 g of water wet nitrocellulose are added to the sample container. After recording the weight, the

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chamber is closed and the test started. The sample is dried until no discernible weight loss is observed.

4.5.10.2.3 Calculation. The total volatile content of the nitrocellulose (in percentage by weight) is direct read out from the moisture analyzer.

4.5.10.3 Nitrocellulose water content: direct Karl Fischer titration method.

4.5.10.3.1 Reagents.

- a. KF single or double component titrant.
- b. Methanol, anhydrous or double component solvent.

4.5.10.3.2 Apparatus.

- a. Analytical balance, precision 0.1 mg or better.
- b. Titrator: Karl Fischer titrator with a Class A burette or equivalent (see 6.12) and a titration cell protected from atmospheric moisture by a drying tube or equivalent, the burette filled with KF single or double component titrant. The burette, stock vessel and their interconnections shall be dry to within detection limit of instrument. The stock vessel has to be protected from atmospheric moisture by a drying tube or equivalent.
- c. Flask 125 mL (usually an Erlenmeyer flask) with a solvent resistant stopper.
- d. Desiccator containing calcium chloride desiccant or equivalent.

4.5.10.3.3 Procedure.

- a. Pre-titration. Anhydrous methanol (or double component solvent) is added to the titration vessel. The amount of solvent added depends on the size of the titration cell. The titration cell shall be closed immediately after the addition of the solvent.
- b. Standardization. Standardization of KF titrant is to be performed by following the titrator's recommended procedure, using standard sodium tartrate or distilled water to determine the water equivalent of the reagent. The water equivalent of the reagent in mg H₂O/mL is recorded as W_E .
- c. Sample preparation. The stoppered flask as obtained from a desiccator is tared on an analytical balance. Approximately 5 g of nitrocellulose is weighed in the flask and closed tightly. The flask is weighed to the nearest mg and recorded as W_{NC} . The pipette has to be purged previously by discarding the first volume of methanol that may have absorbed some moisture from the air. The stopper is then removed and an adequate amount of anhydrous methanol for titration is added using a volumetric pipette. The flask is then closed tightly. The added methanol is weighed to the nearest mg and recorded as W_{MeOH} . A blank solution of methanol has to be prepared following the same procedure. The content is stirred at room temperature for a minimum of 45 minutes.
- d. Sample analysis. The sample solution is dispersed by shaking vigorously. A nominal 5 mL aliquot of the homogenized sample is taken using a 10 mL syringe without a needle (glass or disposable syringe). The syringe containing the sample is weighed on an analytical balance and tared. The entire sample has to be rapidly transferred into the pre-titrated solvent. The syringe is then reweighed to the nearest mg and recorded as W_{Aliq} . The solution is titrated to an appropriate end-point (typically of 20 second or more). If the titrator is equipped for drift compensation: An end-point of 10 sec with a stop drift of 10 l/min is typical. The added volume of reagent is measured to the nearest 0.01 mL and recorded as R_2 . The blank solution of methanol is analyzed following the same procedure.

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4.5.10.3.4 Calculation. The content shall be calculated using the following calculations or the equivalent.

The water content in the aliquot is calculated as follows:

$$\% \text{Water in Aliquot} = 0.1 * \frac{(R_2 * W_E)}{W_{\text{Aliq}}}$$

Where: W_E = water equivalent of the reagent, mg $\text{H}_2\text{O}/\text{mL}$.
 R_2 = volume of KF reagent, mL.
 W_{Aliq} = weight of aliquot, g.
0.1 = 100%/conversion factor $\text{g} \rightarrow \text{mg} = 100\% / 1000$.

The water content in the nitrocellulose (in percentage by weight) is calculated as follows:

$$\% \text{Water in NC} = \left\{ \% \text{H}_2\text{O}_{\text{Aliq}} * (W_{\text{NC}} + W_{\text{MeOH}}) \right\} - \frac{(\% \text{H}_2\text{O}_{\text{blank}} * W_{\text{MeOH}})}{W_{\text{NC}}}$$

Where: W_{NC} = weight of nitrocellulose in sample preparation, g.
 W_{MeOH} = weight of methanol in sample preparation, g.
 $\% \text{H}_2\text{O}_{\text{Aliq}}$ = water content in sample solution, %.
 $\% \text{H}_2\text{O}_{\text{blank}}$ = water content in blank solution (methanol), %.

4.5.10.4 Nitrocellulose water content by Karl Fischer oven method.

4.5.10.4.1 Reagents.

- a. KF single or double component titrant.
- b. Methanol, anhydrous or double component solvent.

4.5.10.4.2 Apparatus.

- a. Analytical balance, precision 0.1 mg or better.
- b. Titrator: Karl Fischer oven apparatus, with oven and titrator

4.5.10.4.3 Procedure.

a. Preparation of apparatus. The Karl Fischer oven apparatus has to be prepared following the instrument's recommended procedure. This in general includes adding / changing of solvent (methanol if single component titrant is used, otherwise Karl Fischer double component solvent), pre-titration of the solvent, and standardization of Karl Fischer titrant.

b. Preparation of sample. A clean, dry glass vial and cap are tared on an analytical balance. A suitable amount of nitrocellulose is weighed in the vial. The amount of NC used maybe dependent upon vial size, moisture content and Karl Fischer reagent strength; typically 0.5 g to 1 g. The rim of the vial is cleaned, sealed, and weighed to the nearest mg, recorded as W_{NC} .

c. Sample analysis. The sample vial is then transferred into the Karl Fischer oven, with the oven set to 135 °C. After starting of the instrument, the sample vial is automatically heated in the oven to 135 °C until the titration is deemed completed, usually 15 minutes or less. If total volatile content is also to be determined, the sample vial is allowed to cool down for several minutes before being reweighed.

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4.5.10.4.4 Calculation. The content shall be calculated using the following calculations or the equivalent. The water content in the nitrocellulose (in percentage by weight) is calculated as follows:

$$\% \text{Water} = 0.1 * \frac{(R_2 * W_E)}{W_{NC}}$$

Where: W_E = water equivalent of the reagent, mg H_2O /mL.
 R_2 = volume of Karl Fischer reagent, mL.
 W_{NC} = weight of nitrocellulose, g.
 0.1 = 100%/conversion factor g \rightarrow mg = 100%/1000.

4.5.10.5 Nitrocellulose alcohol and/or water content using alcohol content and total volatile content. The specific volatile constituents, alcohol and water content, can be determined from results of the total volatile content (4.5.10.1 or 4.5.10.2) and the water content by Karl Fischer methods (4.5.10.3 or 4.5.10.4).

$$\% \text{ Ethyl Alcohol} = \% \text{ Total Volatile} - \% \text{ Water by Karl Fischer}$$

4.5.10.6 Nitrocellulose alcohol and/or water content from gas chromatography method.

4.5.10.6.1 Reagents. The following reagents shall be used in the gas chromatography method:

- a. Isopropyl alcohol, reagent grade, dried by storing above an approximately 2 cm thick layer of fresh molecular sieves (moisture content to be checked by gas chromatography analysis to be less than 0.01%).
- b. Calibration samples of ethyl alcohol and water (and/or possibly other volatile components present) in isopropyl alcohol, covering the expected range of analyze contents; to be used within one month after preparation. The calibration samples have to be stored, tightly capped, in the refrigerator.

4.5.10.6.2 Apparatus. The following equipment shall be used in gas chromatography method:

- a. Analytical balance, precision 0.1 mg or better.
- b. Gas chromatograph (GC) with thermal conductivity detector and column, the latter suitable to separate water, ethyl alcohol and isopropyl alcohol.
- c. Suitable data accumulation and processing system.
- d. Glass vial with cap (for gas chromatograph).
- e. Flask, approx. 125 mL volume, stoppered (e.g., Erlenmeyer flask).
- f. Burette 50 mL, or volumetric dispenser.
- g. Mechanical shaker assembly.

4.5.10.6.3 Procedure.

- a. Sample preparation. 50 mL of dried isopropyl alcohol is dispensed into a flask and stoppered immediately. The flask is placed on the balance and tared. Approximately 5 g of nitrocellulose is transferred to the flask and re-stoppered immediately. The weight is recorded to the nearest mg as W_{NC} . Alternatively, the sample may be weighed in a tared weighing dish and then added to the extraction flask. The stoppered flask is shaken for 1 hour at room temperature on a mechanical shaker. The sample container is then removed from the shaker. The content of the flask is swirled several times before being allowed to settle for 30 minutes.

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Without disturbing the settled solids in the prepared sample, a portion of the liquid is transferred to an auto-sampler vial. The vial is filled approximately three-quarters full. The sample vial is sealed, e.g., by capping or crimping.

b. Calibration. The gas chromatography system is calibrated by running the calibration samples.

c. Sample analysis. The samples are analyzed by the calibrated gas chromatography system. The concentrations of water and ethyl alcohol (and possibly other volatile components) in the nitrocellulose extract are obtained as C_i in %.

4.5.10.6.4 Calculation. The alcohol and water content in the nitrocellulose (in percentage by weight) is calculated as follows:

$$\% \text{ Volatile Component}_i = \frac{C_i * V_s}{W_{NC}}$$

Where: C_i = concentrations of volatile component i in the extract, % m/v.
 V_s = volume of extracting solvent (isopropyl alcohol), mL (here: 50 mL).
 W_{NC} = weight of nitrocellulose sample, g.

4.5.10.7 Nitrocellulose alcohol and/or water content from Near Infrared (NIR) spectroscopy method.

4.5.10.7.1 Apparatus. The following equipment shall be used in NIR spectroscopy method:

- a. Near Infrared spectrometer (covering wavelength range from about 1000 to 2200 nm), e.g., Fourier Transform NIR Spectrometer, or AOTF (Acousto-Optic Tunable Filters) NIR Probe, or similar.
- b. Diffuse reflectance probe for NIR Spectrometer, laid-out for powdery samples.
- c. Data analysis system, including calibration software, preferably adapted for NIR multiple wavelength calibration, otherwise suitable statistical software (e.g., principal components analysis or partial least squares).
- d. Closable container to take up nitrocellulose sample, e.g., plastic bag, flask, bucket with cover.

4.5.10.7.2 Procedure.

a. Preparation and operation of NIR Spectrometer. The instrument is prepared and operated by following the procedure as outlined in the operator's instruction manual. Allowing the instrument to warm up for 10 to 15 minutes before taking any spectra is advisable. System suitability checks (SST) and measurement of the instrument's reference standard spectrum (cover cap of the probe closed) shall be performed every time the instrument had been switched off. During continuous operation, the reference spectrum shall be re-measured every 4 hours and the system suitability check shall be repeated every 12 hours (or as recommended by the instrument's manufacturer) to confirm proper operation of the NIR Spectrometer.

b. Sample analysis. Spectrum of nitrocellulose sample can be simply taken by pushing the NIR probe into the nitrocellulose sample in the container (or by pushing the container towards the probe if the probe is fixed) and starting the spectrum recording mode of the instrument. Duration of accumulation of spectra depends on sensitivity of instrument and the desired signal-to-noise ratio, typically are 0.5 to 2 minutes. It is advisable to take spectra from 2 to 4 different positions within the same sample container. Keeping the pressure (of NIR

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probe towards sample) as constant as possible improves reproducibility of the method. If the probe is attached to a flexible light guide, it is advisable to fixate probe and flexible light guide since movement of the light guide slightly affects the spectrum.

c. Calibration. For proper calibration, as many nitrocellulose samples as possible (at least 60 covering the expected measurement range regarding nitrocellulose types and concentration ranges of alcohol and water) have to be employed. From each sample, NIR spectrum is to be taken as described above, and total volatile content and/or water content and/or alcohol content have to be determined using a primary method. NIR spectra and associated analyzed contents of volatiles are given into the data analysis system. A calibration model is then calculated. The calculated prediction error may serve as a first indication of the quality of the model. The model might need to be improved by deleting suspicious samples (as indicated by being outliers) or by adding new calibration samples. If significant differences between different nitrocellulose types appear, it might be required to build a separate calibration models for each nitrocellulose type. The calibration model shall be finally validated by analyzing several independent samples with both NIR and primary methods. If the results coincide within the desired range, the calibration model can be approved for routine analysis. Regular parallel analysis of actual nitrocellulose samples (e.g., one or several samples per day, per shift or per nitrocellulose lot) with both NIR and primary methods shall be performed, followed by checking that the obtained values coincide within the desired range.

4.5.10.7.3 Calculation. The predicted NIR values of ethyl alcohol, water and total volatiles in the nitrocellulose are reported in percentage by weight.

4.5.11 Workmanship. Visually verify that the nitrocellulose blend or final batch maintains a uniform composition, texture, and color and be free of foreign matter (including oil and grease) by visually inspecting the composite sample.

4.5.12 Materials.

4.5.12.1 Alcohol. The alcohol used in preparation of alcohol wet nitrocellulose products shall meet all verifications listed for Grade 2 of A-A-59342.

4.5.12.2 Cellulose. The cellulose used in manufacture of nitrocellulose from cotton cellulose, woodpulp cellulose obtained by the sulfate process or woodpulp cellulose obtained by the sulfite process, shall meet all verifications listed in MIL-C-206, MIL-C-20330, or MIL-C-216 respectively.

4.5.13 Product performance. The product produced from the nitrocellulose must be evaluated and tested IAW the product performance requirement verifications as specified in the product specification or purchase order (see 6.9).

4.5.14 Preservation of total volatile content. Visually verify the preservation of total volatile content (e.g. no holes, rips or tears, broken or damaged seals, or incorrectly sealed).

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by

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DoD personnel, or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use & applications of nitrocellulose classes. Nitrocellulose is a primary ingredient of many products, such as propellants, combustible case, rocket motors, and other energetic items.

- 6.2 Acquisition requirements. Acquisition documents should specify the following:
- a. Title, number, and date of this specification.
 - b. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced.
 - c. Requirements for submission of first article sample.
 - d. Packaging requirements including dry weight for Class I nitrocellulose.
 - e. Certificate of conformance for each lot or shipment of product, including quantity of product, date submitted, and evidence that the lot complies with all of the conformance inspection provisions of this specification
 - f. Class, grade, and/or type required.
 - g. Ether-alcohol solubility limits for Grade C nitrocellulose.
 - h. Requirements for viscosity and reporting requirements (seconds and cP) .
 - i. Fineness.
 - j. The level of agglomerates (low, moderate, high or very high) for Class 2 nitrocellulose.
 - k. Combustible case or propellant manufacturer prescribed test methodology.
 - l. Drawing, specification number and date, together with identification and date of changes thereto.
 - m. Certificates of analysis on all materials used directly by the manufacturer when materials are controlled by Government specifications.
 - n. Description sheet, MIL-STD-1171 (Description Sheet for Explosives, Chemicals, Etc.) recording all results for inspections performed.
 - o. Confirmation that the test methods, chemical and physical properties including cellulose supplier/form/configuration, and equipment, process and processing conditions other than what is necessary for the maintenance of the process and the required ranges of variation necessary for normal operation of the processing unit are unchanged from the first article samples. Certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.
 - p. Maximum total volatile content.

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q. Reporting and approval of using cellulose other than those specified, cellulose and nitrocellulose process changes, and alternate conformance methods (see 3.3, 4.2.1, and 6.6).

6.3 Specification updating. There have been many advances in nitrocellulose manufacturing, testing and evaluation. This specification combines the prior versions of MIL-DTL-244 with its Amendments and Engineering Changes along with the STANAG 4178 and creates a comprehensive document with new requirements and advanced test methods to meet the future needs for military grade nitrocellulose. Incorporated into this specification is MIL-DTL-244B with outstanding ECP's R06Y2014 and R10C3001 with additional recommendations and changes. Revision C was administratively released and amended with this document to eliminate confusion with approved ECP R10C3001 which was intended to be released as base Revision C, but did not meet all of the MIL-STD-961 content requirements for official release. Therefore, this approved document is listed as Revision C Amendment 1.

6.4 Acceptable packaging requirements. The following packaging and marking requirements have been used for packaging nitrocellulose, and are found to be acceptable to the Government. These requirements should be included in the contract or order for the procurement of nitrocellulose because nitrocellulose is a hazardous energetic material. (Caution: If the following paragraphs are to be incorporated in a contract, they must be modified, using standard contract language, to make them compulsory requirements.)

6.4.1 Preservation.

6.4.1.1 Condition-1. Nitrocellulose should be uniformly wetted with not less than 25% by weight of water and has to be packed in a Department of Transportation Specification Container (see 6.4.2.1).

6.4.1.2 Condition-2. Nitrocellulose should be uniformly wetted with not less than 25% by weight of alcohol or a solvent with a flash point not lower than -3 °C (25 °F), and has to be packed in a Department of Transportation Specification Container (see 6.4.2.1).

6.4.2 Packaging/packing.

6.4.2.1 Level A. Level A packaging for condition 1 or condition 2 materials should be packed in steel drums with a nominal capacity of 208 liters (55 U.S. gallons) conforming to specification 1A2, paragraph 178.504 of Title 49, Code of Federal Regulations (49CFR). The drums should be free of any foreign materials. A bag conforming to Type-I, Class-B, Style-2, MIL-DTL-117 (an alternate bag which meets the requirements of the present bag may be used), should be placed in the drum and filled with material conforming to 6.4.1.1 or 6.4.1.2, as applicable. Close the bag and secure with tape conforming to ASTM D5486, 2.54 cm (1 inch) wide minimum, (an alternate closure of a plastic cable tie is permissible provided that no damage occurs to the bag). Close the drum and secure the cover.

6.4.3 Container weight. Gross weight of container should not exceed 490 pounds.

6.4.4 Performance oriented packaging (POP). The exterior pack cited above has to meet all of the POP requirements in accordance with 49 CFR, including testing and retesting, as necessary. POP test reports should be generated in accordance with Data Item DI-PACK-

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81059 following the tests. POP testing may be waived in accordance with 49 CFR, if an acceptable analogy can be made to another pack which has successfully completed the testing. An analogy also has to be documented IAW DI-PACK-81059. When completed, all test reports should be kept on file by the manufacturer and also should be submitted to the U.S. Army Armament Research, Development and Engineering Center, ATTN: RDAR-EIL-P, Picatinny Arsenal, N.J. 07806-5000.

6.4.5 Item hazard classification. All U.S. manufacturers should make certain that every hazardous item is tested in accordance with Part 173 of 49 CFR in order to assign proper Class and Division for the item. Registration with the Associate Administrator of Hazardous Materials Safety is required by 49 CFR so that containers may be properly marked in accordance with 49 CFR. All foreign manufacturers should make certain that the dangerous goods are tested in accordance with the United Nations Committee of Experts on the Transportation of Dangerous Goods (as published in UN document ST/SG/AC.10.11, latest revision, Recommendations on the Transportation of Dangerous Goods – Tests and Criteria) to determine the proper Class and Division. Registration for air and vessel is required with each manufacturing country's National Competent Authority. The Hazard Classification letter of Competent authority is issued in accordance with Part 2, Paragraph 1.3 of the International Civil Aviation Organization (ICAO) Technical Instructions and approves the Hazard Classification and Compatibility Group assignment and assigns the appropriate shipping name to the dangerous goods.

The proper packaging, marking, and labeling is contained in the United Nations Committee of Experts on the Transportation of Dangerous Goods (as published in UN Document ST/SG/AC.10.11 latest revision, Recommendations on the Transport of Dangerous Goods). For air transport, the dangerous goods have to comply with the provisions of the International Air Transport Association (IATA) Dangerous Goods Regulations. Vessel transport requires that dangerous goods are complied with the provisions of the Intragovernmental Maritime Organization's International Maritime dangerous Goods Code (IMDG Code). These documents should be forwarded to US Army ARDEC, ATTN: RDAR-QES-C, Picatinny Arsenal, NJ, 07806-5000

6.4.6 Referenced documents for packaging. The following list of documents referenced in 6.4 should be included in the contract or purchase order as requirement documents. Document users are cautioned that they must meet all requirements of these documents if cited in the contract or purchase order.

a. Federal and military specifications. (See 2.2.1 for issues of documents and address to obtain the documents) ASTM D5486: Packaging, Waterproof MIL-DTL-117 - Bags, Sleeves and Tubing Interior Packaging

b. Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

CODE OF FEDERAL REGULATION.

49 CFR Parts 106-180 - Transportation, Research and Special Programs,
Department of Transportation

(The Code of Federal Regulations are available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. Orders for the above publications should cite, "49 CFR 106-180 (latest revision)").

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United Nations Committee of Experts on the Transportation of Dangerous Goods, (UN Document ST/SG/AC.10.11, latest revision)

International Air Transport Association (IATA) Dangerous Good Regulations

International Civil Aviation Organization (ICAO) Technical Instructions

Intragovernmental Maritime Organization's International Maritime Dangerous Goods Code (IMDG Code)

(The UN document can be obtained from United Nations Headquarter, Publications Sale Department, New York City, N.Y. 10017. The IATA, ICAO and IMO Dangerous Goods Code/Regulations can be obtained from INTEREG, 5724 N. Pulaski Road, Chicago, Illinois 60646)

6.5 Submission of manufacturer inspection equipment designs for approval. Submit copies of designs as required to: Commander, U.S. Army ARDEC, ATTN: RDAR-QEM-E, Picatinny Arsenal, New Jersey 07806-5000. This address will be specified on the Contract Data Requirements List, DD Form 1423 in the contract.

6.6 Submission of alternative conformance provisions. Unless otherwise specified herein or provided for in the contract, alternative conformance procedures, methods, or equipment, such as statistical process control, tool control, other types of sampling procedures, etc. may be proposed by the manufacturer when they provide as a minimum the level of verification required by this document, and other documents referenced herein. Prior to applying such alternative procedures, methods, or equipment, the manufacturer should describe them in a written proposal submitted to the Government for evaluation and approval as directed by the contracting or procuring agency. When required, the manufacturer should demonstrate that the effectiveness of each proposed alternative is equal to or better than the specified quality conformance provision(s) herein. In case of dispute as to whether the manufacturer's proposed alternative(s) provides equivalent assurance, the provisions of this specification should apply. All approved alternative provisions should be specifically incorporated into the manufacturer's quality program or inspection system, as applicable. Detailed requirements for specific methods to be used should be as specified in the contract or order.

6.6.1 Ethyl-alcohol alternate grade. A-A-59342 Type 2 94.9% ethanol with denaturant (0.75% toluene). Description sheet should note use of approved alternate.

6.6.2 Diethyl ether alternate grade. Ether, MIL-E-199. Description sheet should note use of approved alternate.

6.6.3 Acetone alternate grade. Acetone 99.5%, ASTM D329. Description sheet should note use of approved alternate.

6.7 Technical agency. All correspondence to the technical agency should be submitted through the Contracting Officer to the Project Manager for Combat Ammunition System, Picatinny Arsenal, N.J. 07806-5000 who will distribute it to the applicable technical agencies.

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6.8 Configuration management. The Configuration Change Authority for this document is the Project Manager for Combat Ammunition Systems, Picatinny Arsenal, N.J. 07806-5000.

6.9 Product performance. Nitrocellulose is a primary ingredient of many products, such as, propellants, combustible case, rocket motors, and other energetic items. Changes in nitrocellulose can affect the performance of those products. Whenever a first article sample is required under this specification, an evaluation will be made to the potential impact on performance of the products in which the nitrocellulose will be used and, if determined necessary by the product technical group, a first article test of the product will be performed to determine if the nitrocellulose is acceptable for use in that product.

6.10 Use of pit cotton nitrocellulose in Grade C blends. Pit cotton nitrocellulose is defined as a collection of various grades of nitrocellulose not conforming to an individual grade/type/cellulose source, inherent to the design of the facility and means by which the NC is recovered for reuse. The contracting organization that is acquiring the nitrocellulose should decide if pit cotton nitrocellulose can be added to Grade C nitrocellulose. It should also decide the limit of acceptable quality and the maximum amount tolerated in Grade C nitrocellulose. The pit cotton nitrocellulose should be properly screened to remove any foreign matter before being transferred to poacher tubs for processing. A sample should be taken after processing for stability and any other analysis required to evaluate the quality of nitrocellulose for blending. Only material that meets minimum requirements should be added to the Grade C nitrocellulose. After any pit cotton is added, the nitrocellulose should still conform to the requirements for Grade C nitrocellulose.

6.11 Definition of agglomerates, aggregates, and sinkers.

a. *Agglomerates* are fibers bound or fused together, as a result of the nitration process, in a solid collection which cannot be fiberized without the help of very energetic process equipment like refiners or deflakers that are available only in NC manufacturing facilities. When using fiberboard cellulose, agglomerates are produced from the original cellulose either from partial fiberization of the fluff process or from the chip process (using shredders or cutters).

b. *Aggregates* are formed in the dewatering centrifuge of the NC plant, where the free fibers from the NC slurry are compressed into small clumps that are not bonded or fused together. Since the dewatering process is the last stage of the NC manufacturing process, refining equipment cannot be used to eliminate aggregates. However, certain NC user processes may be capable of reducing or eliminating aggregates.

c. *Sinkers* are all material (nitrocellulose agglomerates and aggregates) that have a terminal velocity greater than the flow velocity within the cone elutriator. Sinkers are the material that “sinks” in the cone elutriator during the test (the material that remains in the cone elutriator).

6.12 Class A burette or equivalent. The following are references for class A burette and precision volumetric equivalents:

ASTM E287-02(2012)	Standard specification for laboratory glass graduated burettes
ISO standard 8655-3:2002	Piston-operated volumetric apparatus – Part 3: Piston burette

6.13 Nitrocellulose fiberization quality test accuracy improvement recommendations. In the event the nitrocellulose fiberization quality test provides inconsistent results or an

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unexpectedly high rate of failure, it is recommended to evaluate replacing the reagent tap water source with a source supply of deionized water capable of the required 950 mL/min flow rate. This will remove errors in weight measurement caused by impurities in the water source.

6.14 Cellulose supplier, source, form, or configuration changes. All proposed changes to the cellulose supplier, source, form, or configuration will be submitted to the Government for evaluation and approval as directed by the contracting or procuring activity. Any changes to the cellulose from the configuration approved during first article testing (including cellulose supplier, source, form, or configuration) should be considered process changes. This further includes any changes in test methods, changes in cellulose chemical or physical properties, and changes to the cooking, bleaching, beating, sheeting, or other applicable processes. While the requirements in the specifications allow a range of values, any change from the values approved during first article testing should also be considered a process change. In accordance with the contract or purchase order (see 6.2), any proposed process change should be reported, should require approval prior to implementation, and may trigger a first article. This includes: proposals to use cellulose from other plant or microbial sources; the establishment or modification of the requirements for the cellulose from other plant or microbial sources; requests to use wood cellulose obtained by the wood sulfate process; and requests for modification to the identification and nominal ratio of wood species from which the paperboard wood cellulose is obtained.

a. Cellulose *source* has three main parameters: the origin of the cellulose such as type of plant, microbe, or algae; the different varieties of origins (such as tree varieties) and associated composition percentages; and the pulping process (such as the sulfite or sulfate process for woodpulp).

b. Cellulose *form* refers to the format of the raw cellulose after processing such as paperboard or baled pulp.

c. Cellulose *configuration* refers to the nominal physical dimensions including thickness, density, and weight of the cellulose form as prepared. For example, the configuration may be defined by or associated with a specific cellulose manufacturer part number, type, or grade. Changing the part number ordered would change the configuration of the cellulose and will require approval.

6.15 Nitrocellulose characterization. Nitrocellulose characterization FAT requirements have been removed from this specification. Reference to characterization test methods (including ion chromatography, residual acidity, molecular mass distribution, and fiber length distribution testing) as requested by contract or purchase order can be found in STANAG 4178.

6.16 Methy Violet Test Papers. A known source of standard methyl violet test papers is:

Naval Surface Warfare Center
Indian Head EOD Technology Division
Indian Head, MD 20640

6.17 Classification of characteristics. The classification of characteristics was added to the specification as part of Engineering Change Proposal (ECP) Y13C2001. Definitions and rationale can be found in the supporting documentation of this ECP package.

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6.18 Consideration of data requirements. The following data requirements should be considered when this specification is applied on a contract, per the Contract Data Requirements List, DD Form 1423.

Reference Paragraph	DID Number	DID Title	Suggested Tailoring
6.4.4	DI-Pack-81059	POP Test Report	none

The above DID was cleared as of the date of this specification. The current issue of DOD 5010.12-L, Acquisition Management Systems and Data Requirements Control List (AMSDL), must be researched to ensure that only current, cleared DID's are cited on the DD Form 1423.

6.19 Subject term (keyword) listing.

NC
Cellulose
Cellulose nitrate
Pyrocellulose
Guncotton

6.20 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

Custodian:
Army - AR
Navy - OS
Air Force - 99

Preparing activity:
Army-AR

(Project 1375-2014-026)

Review Activities:
Army – MI, AV
Navy –AS, SH
Air Force – 11, 70

Civil Agency:
GSA - FAS

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <https://assist.dla.mil/>.