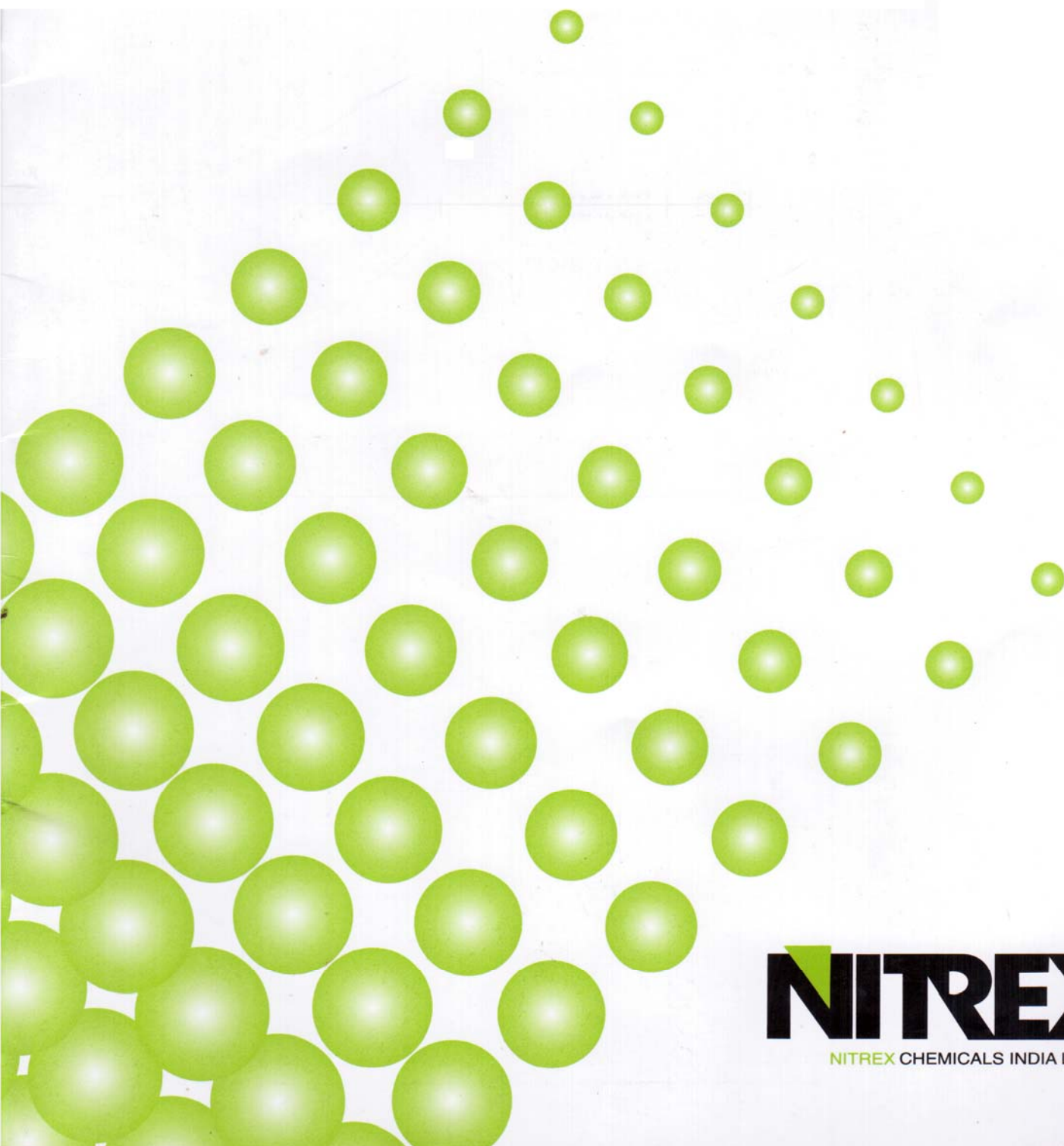


INDUSTRIAL NITROCELLULOSE



NITREX
NITREX CHEMICALS INDIA LIMITED

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Nitrex Chemicals India Limited is the largest manufacturer of industrial grade nitrocellulose in the country. We service our customers from our factory at Valsad, Gujarat, India, both directly and through a network of agents. Over the years it has been our constant endeavour to supply the highest quality product and services to our customers, while maintaining our commitment to safety, health and environment. We have the capability to meet all requirements of nitrocellulose users through a comprehensive product range that covers both solvent damped and plasticized products and can also offer customized grades.

Nitrex Chemicals India Limited is an ISO:9001 and ISO:14001 certified Company.

INDUSTRIAL NITROCELLULOSE

Industrial nitrocellulose is known to be one of the oldest film formers and is today a familiar part of the coatings industry.

Nitrocellulose is a derivative of cellulose and is manufactured from purified cellulose either in the form of cotton linters or woodpulp. The manufacturing process involves nitration of purified cellulose with nitric acid and sulphuric acid under controlled conditions followed by stabilization and washing.

Nitrex Chemicals manufactures nitrocellulose from cellulose with nitrogen content in the range of 10.7% to 12.2%. The packaging and transportation of nitrocellulose conform to international standards as also to country specific standards.

Nitrocellulose, also called cellulose nitrate, is classified for industrial use provided

- Its nitrogen content is less than 12.3%, and
 - it is uniformly mixed with not less than one third of its weight of a specified dewatering agent (e.g. water, isopropanol, n butanol, ethanol, etc.)
- or
- It is plasticized with a minimum of 18 parts of plasticizer to 82 parts of nitrocellulose
- or
- It is in a solution.

Product Range

Nitrex Chemicals classifies its range of industrial nitrocellulose on the basis of :

I. NITROGEN CONTENT

Nitrogen Class	Nitrogen Content	Typical Application
High(H)	11.7% to 12.2%	Auto Refinish, Wood Lacquers
Medium (M)	11.2% to 11.7%	Printing Inks, Film Coatings
Low(L)	10.7% to 11.2%	Printing Inks

Nitrogen content influences the solubility and thermoplasticity of nitrocellulose. Low nitrogen grades of nitrocellulose have a higher solubility in alcohols and higher thermoplasticity. The selection of proper grade of nitrocellulose will depend on the solvent system and the thermoplasticity requirement.

2. VISCOSITY RANGE

Viscosity Class	Measurement
Medium (M)	10 gms in 100 ml of 95% v/v aqueous acetone
Low (L)	20 gms in 100 ml of 95% v/v aqueous acetone
Extra Low(X)	40 gms in 100 ml of 95% v/v aqueous acetone

The viscosity of the grade determines the strength and durability of the nitrocellulose film. Low viscosity grades are suitable for applications where high solids and gloss are primary requirements as in case of printing inks. The high viscosity grades provide better durability and resistance to films as desired in auto refinish paints.

3. DAMPING MEDIA

Including but not limited to :

Solvents	Plasticizers	
Butyl Alcohol	Di-isobutyl phthalate	Epoxidised soyabean oil
Isopropyl Alcohol	Acetyl tri-butyl citrate	Di-isononyl phthalate
Ethyl Alcohol	Di-butyl phthalate	
Water wet	Octyl di-phenyl phosphate	

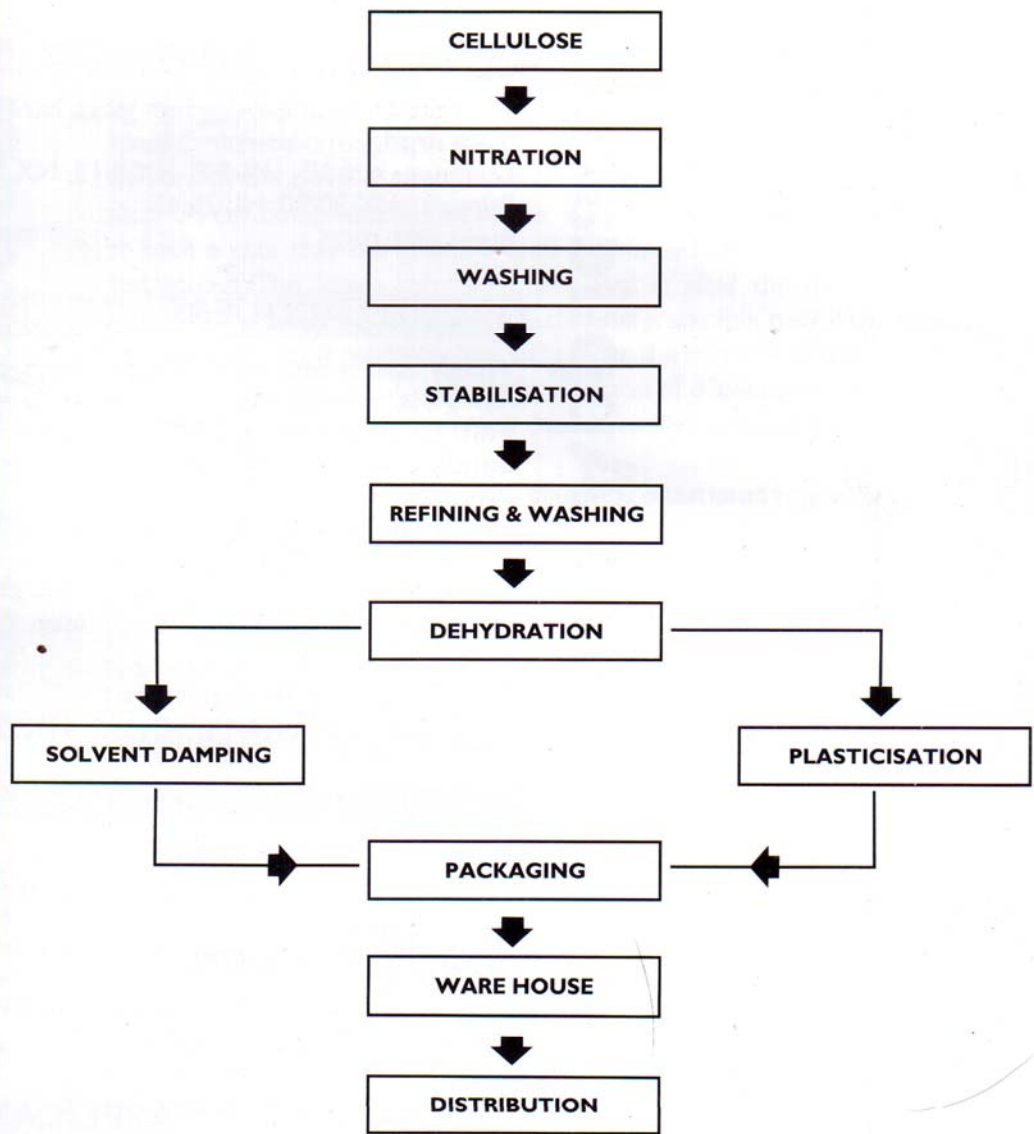
The damping medium in the nitrocellulose is the wetting agent that acts as a desensitizer.

Manufacture

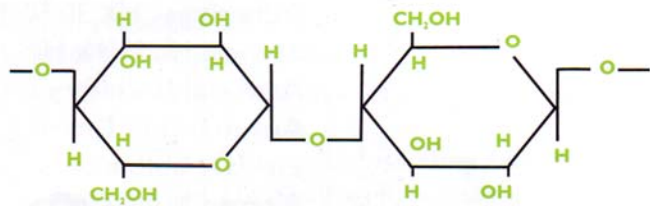
Teased cotton linters or shredded wood pulp are reacted with mixed nitric and sulphuric acid under carefully controlled conditions. During nitration, acid concentration, temperature and immersion time are varied depending on the nitrogen content required. Excess acid is removed by centrifuge and residual acid by washing with water to render maximum stability which is determined by the Bergmann and Junk test for the end product.

The raw cellulose has a high viscosity which is passed on to the nitrated product. The viscosity of nitrocellulose is reduced to the required level by heating with water in specially designed equipment. This process stabilizes the nitrocellulose. After stabilization nitrocellulose is washed successively with demineralised water. Nitrocellulose of known viscosity passes to the finishing plant as a slurry in water. This product is dehydrated and water replaced by damping medium. The final product is tested for solution properties before being packed in drums.

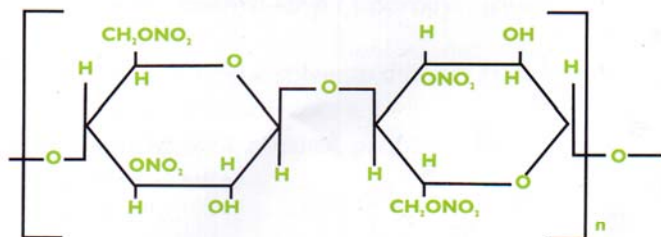
Schematic Flowchart Of Manufacturing Process



Chemical Structure of Cellulose



Chemical Structure of Nitrocellulose



Applications

Industrial Nitrocellulose finds application in :

AUTO-REFINISH PAINTS

Well known for imparting high gloss, hard film, ease of application and quick drying.

Coatings : HX 3/5, HX 5/8, HX 8/13, NX 2Q/30 HX 30/50

Primers : HX 30/50, HL 25/45

Putty :HM 10/25

WOOD LACQUERS

Widely used as base coat and top coat on wooden furniture and articles for high gloss, washability and amenability to spray painting.

Film Coating : HX 3/5, HX 5/8, HX 8/13, HX 30/50

Sanding Sealer : HX 30/50, HL 25/45, HM 10/25

PRINTING INKS

Provides gloss, fast drying capability and good adhesion to many substrates. Most gravure and flexographic inks for the packaging industry use nitrocellulose.

Flexographic & Rotogravure :

LX3/5, LX5/8, LX8/13, HX3/5, HX5/8, HX8/13, MX 3/5

LEATHER FINISHING LACQUERS

Preferred on leather goods both for protection and high quality finish
HX 30/50, HL 25/45, HL 120/170, HM 10/25

NAIL VARNISHES

Imparts gloss, quick drying capability to nail varnishes.

HX 8/13, HX 20/30, HX 30/50

MISCELLANEOUS APPLICATIONS

Finds applications in sealants and primers that are used in painting industry
Also used in adhesives, coatings, artificial jewellery etc.

Adhesives : HX 30/50, MM 10/25, HM 40/60

Stencil: HL 25/45, HM 10/15

Artificial Jewellery : HX 8/13, HX 30/50

Ammunition Coating : HL 120/170

Electric bulb Lacquers : HM 10/25, HM 40/60

Picture Tube Coatings : HH 2/4

Nitrocellulose Lacquers

PREPARATION OF NITROCELLULOSE SOLUTION

Ideally, nitrocellulose lacquers are prepared with modern high speed stirrers which are first charged with non-active solvents (or diluents) and alcohols (or co-solvents). The stirrer is activated and nitrocellulose added at such a rate that the grains are all maintained in suspension and none settles out. The active solvent is then run in after the nitrocellulose is completely added. In practice this technique is suitable only if the resulting solution viscosity is relatively low, otherwise the stirrer will begin to labour. A too rapid reduction of viscosity by addition of diluents can result in local precipitation of gelled pieces which do not readily re-dissolve.

In practice a premix of solvent and diluent is often used to give satisfactory results.

Solvent systems containing high proportions of diluents take longer dissolution time. It is preferred that a portion of the diluent be held back for later addition after the nitrocellulose has gone into solution.

For nitrocellulose chips higher speed stirrers are preferred.

SOLVENT TYPES & RELATION TO SYSTEM SELECTION

The solvent system used for preparation of the solution has a very important role in the final property of the solution including its viscosity.

The choice of the solvent depends upon:

- Viscosity
- Solids content that gives a particular viscosity
- Speed of evaporation
- Cost

Therefore the properties of solvent of relevance to the nitrocellulose solution are :

- Solubility of nitrocellulose in the given solvent
- Boiling point and evaporation rate of the solvent.
- The solvents are categorized into the following types:

Based on solubility of nitrocellulose they are categorized as :

A. **True Solvents** that can dissolve nitrocellulose on their own e.g.

Ketones : acetone, methyl ethyl ketone, methyl isobutyl ketone

Esters : ethyl acetate, propyl acetate, butyl acetate, amyl acetate

Glycol Ethers: methyl/ ethyl / isopropyl / glycol ethers

B. **Co-Solvents** or latent solvents that act as solvents but only in the presence of true solvents e.g.

Alcohols: methanol, ethanol, propanol, butanol, etc.

Ethers: diethyl ether

Nitrocellulose Lacquers

- C. **Non-Solvents** or diluents or extenders that are used primarily on account of low cost but in which nitrocellulose is not soluble e.g. aliphatic & aromatic hydrocarbons e.g. benzene, xylene, toluene, white spirit, etc.

Based on their boiling ranges they are categorized as follows:

- A. **Low Boilers** with boiling point range below 100°C e.g. ethyl acetate, methylethyl ketone, acetone, isopropyl alcohol, industrial methylated spirit and toluene.
- B. **Medium Boilers** with boiling point ranging between 100°C & 150°C e.g. butyl acetate, MIBK, ethylene glycol, xylene, etc
- C. **High Boilers** with boiling point above 150°C which are slow evaporators.

Low boilers yield solutions of low viscosity and quick evaporation. The medium boilers check the rate of evaporation, impart good flow properties and reduce the defects caused by too rapid an evaporation. High boilers also act similarly and impart high gloss and brushing properties. Evaporation rates of pure solvents and their mixtures give only an approximate guide to the rate of evaporation from lacquer film

Taking the evaporation of butyl acetate equal to one as standard, the following are the relative rates of evaporation at 50% loss of some of the common solvents used in lacquers.

Ethyl Alcohol	2.1	MIBK	1.8
Isopropyl Alcohol	2.1	Aliphatic Diluent	2.6
Butyl Alcohol	0.4	Toluene	2.6
Ethyl Acetate	8.9	Xylene	0.7
Acetone	20.0	Butyl Acetate	1.0
Diacetone Alcohol	0.09		

COMMON PROBLEMS ENCOUNTERED WITH IMPROPER SOLVENT SELECTION

It is necessary to control evaporation of various volatile components of the lacquer in order to prevent 'Chilling', 'Gum Blush' and 'Cotton Blush'. These features are described below:

'Chiling' refers to the formation of an opaque film and is attributable to the lowering of the temperature of the film and air near the film to below the dew point. It is caused by the high rate of evaporation of a highly volatile solvent from the solution and problem can be remedied by increasing the proportion of the medium boiler in the solvent mix and butanol is particularly useful for this.

'Gum Blush' and 'Cotton Blush' occur either when resins or nitrocellulose respectively are precipitated from the lacquer 'Gum Blush' is caused by too rapid an evaporation of solvent for the resin which eventually is precipitated out and similarly 'Cotton Blush' is caused if at any point during evaporation of solvents the dilution ratio is exceeded. Blushing can be avoided by using a small proportion of high boiling solvent which dissolves both nitrocellulose and resin.

Modifiers

PLASTICIZERS

Plasticizers are substances of low volatility which are added to lacquers to improve flexibility tensile strength, gloss adhesion, thermoplasticity, evaporation of volatile solvents and the susceptibility of 'Gum Blush' and 'Cotton Blush'. These are classified as follows:

- Solvent types e.g. di-butyl phthalate, di-isooctyl phthalate and tricresyl phosphate
- Non-solvent plasticizers like castor oil and blown castor oil
- Sebacates and adipates
- Polymeric plasticizers

Choice of plasticizer type depends on characteristics desired in the film i.e. durability, colour retention, inflammability and temperature flexibility.

RESINS

Solutions of nitrocellulose require resins which enable formulators to increase solids content significantly without any increase in viscosity. In this way thickness of film is increased in addition to gloss, adhesion, chemical resistance and durability. Common resins are alkyds, ketonic, acrylic, polyvinylacetate and sulphonamide types.

PIGMENTS

Most colored lacquer films are pigmented although for some purposes, such as coatings on aluminum foil, dyes are used.

Selection Of Nitrocellulose Type

Choice of the correct grade of nitrocellulose for formulating lacquers is governed mainly by solution characteristics, and also the properties on the final coating. A knowledge of the factors that govern the solubility of nitrocellulose, the viscosity of solution and the properties of lacquers and films made from it is therefore of considerable practical value to lacquer manufacture.

SOLUBILITY

The solubility of nitrocellulose depends on the nitrogen content and to a lesser extent on viscosity. All industrial nitrocellulose grades with a nitrogen content of 10.7% to 12.2% are soluble in esters, ketones, glycol ethers (active solvents) or in suitably formulated mixtures of these with hydrocarbons and / or alcohols of low molecular weight. Low nitrogen grades are used where high alcohol or spirit solubility is desired.

Selection Of Nitrocellulose Type

HYDROCARBON TOLERANCE : DILUTION RATIO

A hydrocarbon non-solvent is added normally to a solution of nitrocellulose for achieving economy and dilution. In this addition a point is eventually reached when the nitrocellulose no longer remains in solution and is precipitated out

The volume ratio of hydrocarbon non-solvent or diluent to solvent this point is called dilution ratio.

Hydrocarbon tolerance of any given nitrocellulose varies with the solvent used and the type of diluent. For a given solvent mixture it depends on

1. The grade of nitrocellulose
2. Purity of solvents
3. Ratio of active solvents to alcohol
4. Concentration of nitrocellulose (the dilution ratio diminishes with increasing concentration)
5. Temperature (the dilution ratio diminishes with increasing temperature)

Variation of dilution ratio with type of solvent and dilution is illustrated in Table I . In this table the value of dilution ratio is shown to vary with type of solvent i.e. butylacetate or MIBK or ethyl alcohol and the type of diluent i.e. toluene, SBP petroleum solvents and n-butanol for the same concentration and grade of HX 30/50. In Table 2 the value of dilution ratio has been shown to be varying for different grades of nitrocellulose in the Same solvent.

Active Solvent	Dilution Ratio (HX 30/50)		
	Toluene/Xylene	SBP/Petroleum Solvents	N-Butanol
BUTY LACETATE	2.7	1.5	10
MIBK	3.6	0.8	-
ETHYLENE GLYCOL	4.7	1.1	

Table I : Dilution ratio of common solvents (nitrocellulose conce. 8%)

Nitrocellulose Grade	Dilution Ratio	Final Concentration of Nitrocellulose
HX	2.7	8.0
HL	3.0	3.5
LX	2.0	10.0

Table 2 : Variation of toluene dilution ratio grade of industrial nitrocellulose (Solvent butyl acetate)

Dilution ratio is useful as a guide to the quantity of one diluent that may be used as a substitute for another in an established formulation. The actual ratio of diluent/hydrocarbon non-solvent to active solvent in practice should always be appreciably less than quoted dilution ratio for two reasons:

Selection Of Nitrocellulose Type

- Dilution ratios are referred to at 8% concentration while in most formulations nitrocellulose concentrations are higher than 8%.
- Most lacquers contain resins and plasticizers which adversely effect dilution ratios i.e. they reduce hydrocarbon tolerance.

EFFECT OF SOLVENT COMPOSITION TYPE ON NITROCELLULOSE LACQUER VISCOSITY

The viscosity of a solution containing a given concentration of nitrocellulose, depends on the chemical type of the solvent i.e. whether esters, ketones, etc. Within a particular chemical type, the solvent with the lowest boiling point gives the lowest viscosity than esters at the same level of concentration. Of the ketones, acetone gives the lowest viscosity.

In mixed solvents moderate amount of alcohols reduce viscosity. Minor amounts of hydrocarbon diluents have little effect, but as the proportion of hydrocarbon non-solvent increases, the viscosity rises until finally the nitrocellulose becomes insoluble.

AGEING EFFECTS

The viscosity of nitrocellulose solutions drops some what on storage. This fall is marked during first week and varies according to composition and storage temperature. The effect is attributable entirely to solvation and implies no chemical degradation of the nitrocellulose. There is no known way of preventing it and users are not recommended to try additives as a short-cut to attaining equilibrium.

FILM PROPERTIES

As with all polymers, the strength and durability of nitrocellulose lacquer films depends on the molecular weight, which in turn varies with viscosity. Thus the lowest viscosity grades e.g. HX 8/13 which have low molecular weight would give the weakest, least durable film, These are recommended for high solid lacquers and inks where high gloss and build up are more important than durability and high film strength

The latter properties improve as we move up the viscosity scale and HX 30/50 represents the best compromise between good durability and reasonable solids. Where more flexibility is needed as in leather lacquers, HL 25/45 is recommended. Grades with viscosity higher than this offer no significant improvement in film properties and are used only in specialized outlets e.g. where a minimum of film former is required or for blending with lower viscosity grades to make lacquers with a particular combination of solids and viscosity.

STABILITY

All industrial nitrocellulose supplied passes the Bergmann and Junk stability test. However the following points should be noted:

1. The addition of alkaline and strongly acidic material is harmful.
2. Certain amines (diethylamine, monoethanolamine, morpholine, etc.)

if allowed to come in contact with industrial nitrocellulose can cause spontaneous ignition or charring. There is a risk of fire particularly with dry nitrocellulose. Any spillages should be cleaned up at once.

These amines also cause severe discoloration of nitrocellulose films and solution. The use of alcohol denatured with pyridine should be rigorously avoided.

Avoid storing nitrocellulose or nitrocellulose lacquers at high temperature or indirect sunlight.

Avoid using pigments which give an alkaline reaction.

Blending

With the wide range of viscosity grades supplied, it is not normally necessary to blend different grades of nitrocellulose. Occasionally, however, stocks of a particular grade may be exhausted or there may be a special requirement that cannot be met by available materials. In such cases mixtures of suitable grades will give the desired viscosity and the Blending Chart can be used to determine quickly the proportions required the viscosity equivalent of a known mixture.

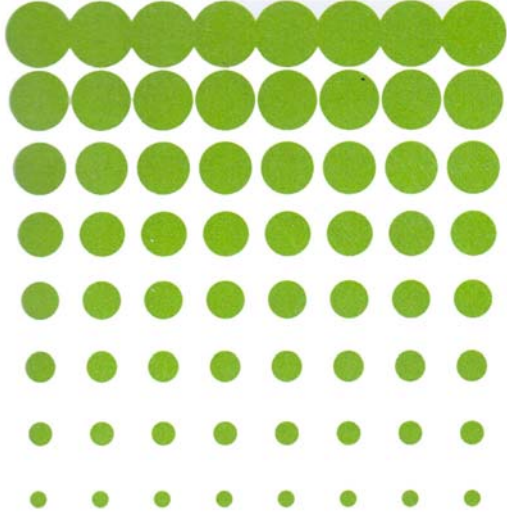
BLENDING LIMITATIONS

It is not advisable to blend different nitrogen grades or any grades which are far apart in viscosity since either procedure can cause rough, granular solutions or resin in compatibility problems.

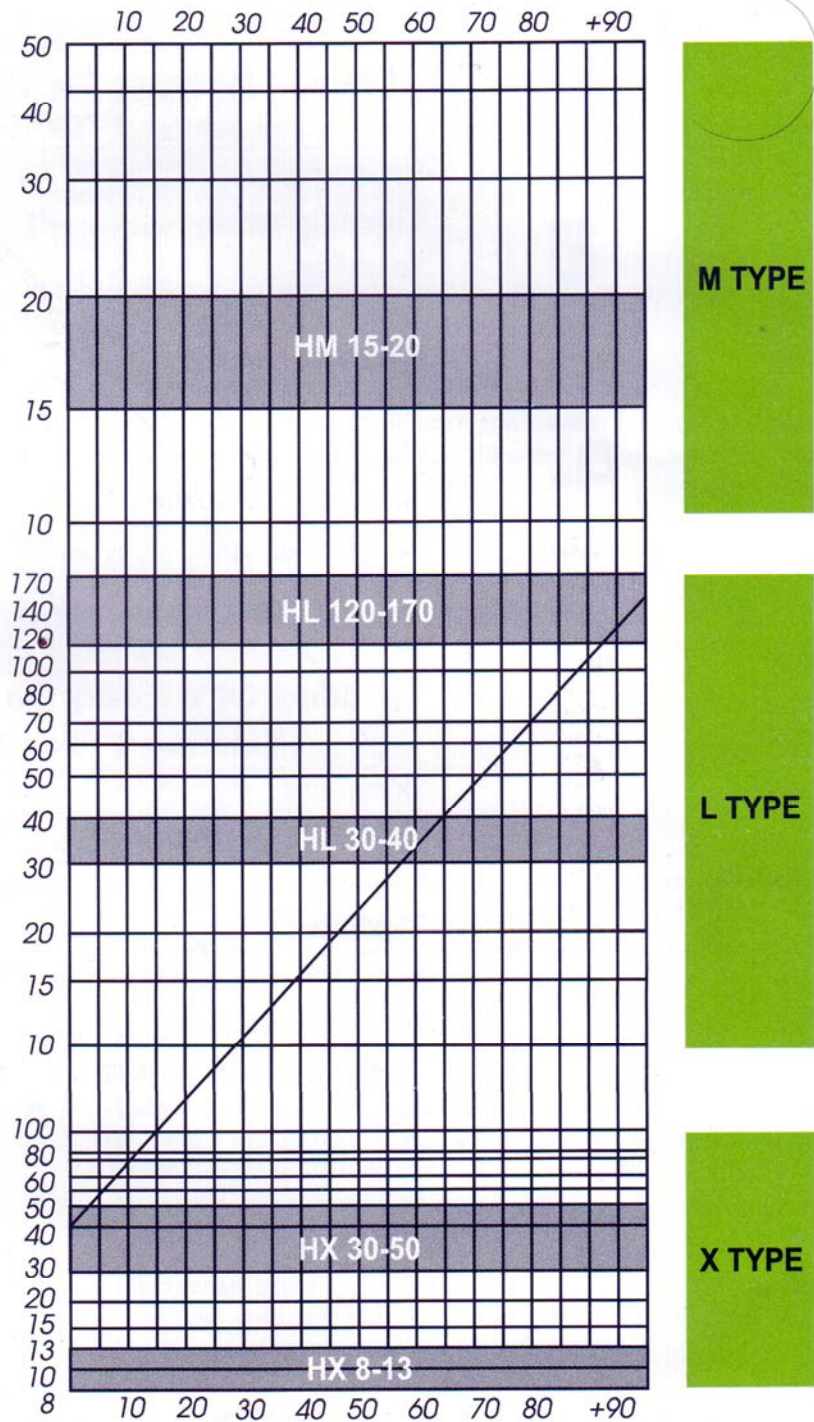
USING THE BLENDING CHART

Pick out the points to the two vertical axes corresponding to the two grades you want to blend. Join them with a straight edge. Where the drawn line crosses the desired viscosity line read off the percentage on the horizontal scale. This will be the percentage of the nitrocellulose represented by the point on the right-hand axis which you should use in the blend.

The dotted line on the chart illustrates the blending of HX 30/50 and HL 120/170. In each case the midpoint of the range is taken.



Viscosity Chart



Suppose a viscosity equivalent to HL 25/45 is required. Since the dotted line crosses viscosity L35 at about 60% the required blend will be (60% HL 120/170 with 40% HX 30/50. Conversely, a blend of 75%HL 120/170 with 25% HX 30/50 will have a viscosity equivalent to L 60. Once the blend is established, the two grades are dissolved together in a given ratio. The solution is often smoother if the high viscosity nitrocellulose is dissolved first. Alternatively, solutions of known nitrocellulose content can be mixed in the appropriate proportions.



Properties

Effect of Nitrogen Content on Nitrocellulose Properties

Property	As % of Nitrogen Increases
Thermoplasticity	Decreases ↓
Solubility	Decreases ↓
Water Resistance	Increases ↑
Alcohol Solubility	Decreases ↓
Resin Comparability	Increases ↑
Hardness	Increases ↑
Aromatic & Aliphatic	Increases ↑
Solvent Toierance	Increases ↑

Effect Of Viscosity On Nitrocellulose Solution & Film Properties

Property	As Viscosity Decreases
Flexibility & Elongation	Decreases ↓
Hardness	Increases ↑
Thermoplasticity	Decreases ↓
Solubility	Increases ↑
Solution Viscosity	Decreases ↓
Solids at Equal Viscosity	Increases ↑
Tendency for Solution Discoloration	Increases ↑

Handling Of Industrial Nitrocellulose

NITREX

1.0 HAZARDS

1.1 LOSS OF DAMPING MEDIUM

Industrial nitrocellulose containing less than 30% water or other solvent damping medium is more susceptible to ignition. When the water / solvent content of nitrocellulose decreases its burning rate increases rapidly.

Dry nitrocellulose burns fiercely.

Nitrocellulose must not be allowed to dry out at any stage of processing, handling and storage.

1.2 HEAT AND IGNITION SOURCE

Decomposition of nitrocellulose and ignition can result if it is stored for a long period near any heat source. Rapid overheating caused by friction can also result in ignition of nitrocellulose.

Smoking and use of naked light/fire to be strictly prohibited.

1.3 REACTIVE CHEMICALS

Contact with strong alkaline, acidic materials or with oxidizing agent can lead to decomposition and ignition.

Never allow concentrated acids, alkalis or amines to come into contact with nitrocellulose.

1.4 EFFECT ON HEALTH

1.4.1 Hazard data should be available wherever nitrocellulose is handled or stored. The user should read and understand MSDS, which gives detailed chemical and toxicological properties, before using the product.

1.4.2 Clothing contaminated by nitrocellulose and lacquers is a fire hazard and should be changed at the earliest.

1.4.3 Butanol/ethanol/isopropanol damped nitrocellulose is not dangerous to health but nitrous fumes from burning nitrocellulose are a lung irritant with delayed action.

1.4.4 Symptoms (in case of exposure to high concentration of nitrous fumes) :

Distressed breathing, tightness in the chest, coughing, headache, vomiting, cold, perspiration, anxious look, protrusion of eye balls, blueness of the skin, fainting and collapse.

2.0 HANDLING

2.1 HANDLING OF NITROCELLULOSE

2.1.1 Nitrocellulose must not be allowed to become dry in any circumstances.

2.1.1 Any waste nitrocellulose should be collected at once, full/ wetted with water and kept inside a closed container in a designated place for disposal.

2.1.3 Before transferring nitrocellulose drums to the warehouse it is to be ensured that drums are properly packed and no loose nitrocellulose

Handling Of Industrial Nitrocellulose

is sticking anywhere in the drums. While transferring drums in trailers, the vehicle should be driven very slowly

- 2.1.4 It is preferable to consume an entire drum at one time by the user for any preparations. Part filled nitrocellulose drums should not be kept in the warehouse. If this cannot be avoided, the drums should be properly sealed and used at the next opportunity.
- 2.1.5 Partially used drums should have proper damping content. In case nitrocellulose is partially dry, add required quantity of damping medium. Keep the mouth of the polythene liner properly tied. Leftover nitrocellulose from a particular grade should be used immediately with the next lot.
- 2.1.6 Solvent wet nitrocellulose exhibits the same degree of handling hazard as do flammable liquids. Care should be taken to eliminate the possibility of ignition from struck sparks, frictional heat, flame or static electricity while handling solvent damped nitrocellulose.
- 2.1.7 Solvent wet nitrocellulose is a free flowing material that can generate static electricity while being poured from the drum. All equipment shall be earthed suitably.

2.2 HANDLING OF NITROCELLULOSE DRUMS

- 2.2.1 Nitrocellulose drums should not be rolled or dragged on concrete, steel or any other hard floor/surface. Hand trolleys should be used for shifting drums from one place to another. While handling, drums should not be allowed to bump into one another.
- 2.2.2 Drums containing nitrocellulose must not be allowed to fall during loading /unloading from vehicles. The preferred method of loading drums onto and from a truck is when the loading /unloading platform is at level with the truck floor.
- 2.2.3 Drums should never be transported in horizontal position. It should be transported as they are stored i.e. vertically with lids up.
- 2.2.4 Non-ferrous tools should be used while opening nitrocellulose drums.
- 2.2.5 Covers of nitrocellulose should fit tightly and should always be kept in place to prevent drying due to evaporation of solvent.

2.3 SAMPLING

- 2.3.1 Ensure drums have been reversed adequately before sampling.
- 2.3.2 Use a proper sample which will collect sample from the entire section of the drum length to get a representative sample. Collect samples from different places and make a composite sample.
- 2.3.3 Do not collect samples from top only as this will give misleading result.

Handling Of Industrial Nitrocellulose

2.4 WASTE NITROCELLULOSE HANDLING

- 2.4.1 Wet down and sweep up all spilled nitrocellulose promptly
- 2.4.2 Place wet sweepings in a covered metal waste container and keep wet with water. Do not accumulate waste nitrocellulose. Try to destroy it daily.
- 2.4.3 If daily destruction is not possible and waste nitrocellulose has to be stored periodically check the contents to ensure it is fully wet. If necessary, add water to the contents.

3.0 STORAGE

- 3.1 Nitrocellulose drums should be stored in a cool area. Smoking and use of naked light/fire to be strictly prohibited.
- 3.2 Only flameproof electric light/fittings shall be used in the solvent damped nitrocellulose handling and storage area.
- 3.3 The building where nitrocellulose is stored should have adequate ventilation. Nitrocellulose drums should always be kept under shed. The drums shall not be subjected to heat, e.g. direct sunlight, impact and friction, as these can lead to fire and explosion.
- 3.4 The storage shall always be kept clean, tidy and free from any combustible items. There should always be adequate passage all around the wall of the building and storage space for easy access and exit.
- 3.5 Adequate supply of water shall be made available near the storage for extinguishing fire. Preferably automatic water sprinklers should be provided in the storage area.
- 3.6 Nitrocellulose drums shall not be opened in the storage area. If needed drums should be brought back to processing area for necessary action.
- 3.7 Nitrocellulose drums shall not be stored for very long time. Nitrocellulose drums shall be preferably issued on a first-in-first-out basis as much as possible. It is desirable to maintain a minimum stock of nitrocellulose drums in the store with proper supply and dispatch planning. However, if the drums are required to be stored longer, those should be reversed at regular intervals of 2 to 3 weeks to allow reverse migration of damping medium from top to bottom of the drums.
- 3.8 Nitrocellulose drums should be preferably stored only in single tier in an upright position. It must be ensured that the drums are sealed properly and the lids/ clamping arrangements are intact to prevent loss of damping medium by evaporation.
- 3.9 Protect all nitrocellulose processing and packing equipment, in solvent/ manufacturing area against static electricity by adequate grounding of metal parts.
- 3,10 The walls and ceiling of the storage and processing building should be washed down periodically to remove dry nitrocellulose dust.

- 3.11 Adequate supply of water should be available for extinguishing fire, flooding unburnt material and cooling adjacent drums.
- 3.12 Drums should have proper marking of lot numbers, grade of nitrocellulose, date of manufacture, etc.
- 3.13 Trucks coming to the storage area for loading drums should have a flame arrester at the exhaust.
- 3.14 Nitrocellulose warehouse or handling room must have lighting conductors.

4.0 DISPOSAL OF EMPTY CONTAINERS

- 4.1 Ensure drums are emptied out fully after use. The drums must be cleaned thoroughly (inside & Outside) with a cloth damped with water. Ensure the drum is properly decontaminated from the residual nitrocellulose before disposing off. Cleaning rags must be disposed off in the same manner as in the case of waste nitrocellulose.
- 4.2 The drum lid should then be put on the drum and the sealing ring tightened.
- 4.3 Keep the empty drums in a designated place with proper marking/label. Disposal should be in conformance with local regulatory laws.

5.0 DISPOSAL OF WASTE NITROCELLULOSE

- 5.1 All nitrocellulose spillage should be collected immediately. Any small spillage that is unsuitable for use should be completely wetted with water and transferred to metal container with a suitable cover and removed to a safe place.
- 5.2 Only wet nitrocellulose should be taken for destruction. Dry or partially dried material shall never be used.
- 5.3 Waste nitrocellulose may be destroyed by burning in small lots in a safe and isolated location. Complete burning of the waste nitrocellulose has to be ensured as partially burnt nitrocellulose is very dangerous. Nitrocellulose should not be burnt in an enclosed place/fire e.g. boiler, incinerator or other confined equipment.
- 5.4 Nitrocellulose also may be destroyed chemically in small amounts by treating it with dilute caustic soda solution using a proper vessel in a well ventilated location. The slurry should be stirred until the nitrocellulose has been completely digested i.e. all of the nitrocellulose particles have been dissolved. The digested dark brown liquor can then be disposed off in accordance with local regulations.

6.0 FIRE FIGHTING

- 6.1 In case of fire in nitrocellulose warehouse or handling area, immediate action must be taken to fight the fire as any nitrocellulose fire which remains uncontrolled for even short time could destroy the premises.

- 6.2 Water is the most effective fire extinguishing medium for nitrocellulose and should be used in large quantities.
- 6.3 Nitrocellulose burns fiercely when ignited releasing poisonous nitrous fumes and excessive heat. High temperature inside the drums of nitrocellulose will increase the pressure inside and may throw out the drum lid. Fire fighting shall be done from a safe distance.
- 6.4 As burning of nitrocellulose gives off highly poisonous nitrons fumes, fire personnel must be warned to wear proper breathing apparatus.
- 6.5 Never attempt to fight a nitrocellulose fire which is at an advanced stage. Only professional fire fighters with adequate protective devices and proper fire lighting appliances shall be deployed for advanced nitrocellulose fire.
- 6.6 Keep fire fighting equipment in good condition. Ensure that all concerned are familiar with the available fire fighting equipment.

- The test methods used for some of the important characteristics of nitrocellulose are described in this section.

1.0 SOILIDS CONTENT IN DEHYDRATED NITROCELLULOSE

1.1 PREPARATION OF SAMPLE

A representative sample of nitrocellulose is spread out on an aluminum tray and any aggregates are broken by rubbing the sample gently between the hands. The speed of handling is essential to avoid evaporation of the damping medium.

1.2 PROCEDURE

Thirty to forty gm of the prepared sample is weighed into a tared aluminum dish with close fitting lid. The lid is removed and the dish is placed in a water jacketed oven maintained at 75°-80°C After 4 hours the dish is cooled in a dessicator, the lid fitted and the whole is weighed.

The heating cycle is continued until constant weight is attained.

$$\% \text{ solids (dry weight)} = \frac{\text{Loss in weighy} \times 100}{\text{Weight of sample taken}}$$

The dry nitrocellulose after test should be *immediately wetted with water and destroyed.*

2.0 WATER IN DEHYDRATED NITROCELLULOSE

2.1 PROCEDURE

In a dry tared weighing bottle 1.0-1.5gm of prepared sample is taken and weighed accurately. 5 ml of anhydrous methanol is added using a 5 ml pipette. The bottle is fitted in the Karl Fisher titration assembly and the mix is titrated with a KF reagent, A blank titration is also

carried out with 5 ml anhydrous methanol without the sample.

$$\% \text{ water in sample} = \frac{(\text{Titre-Blank}) \times f}{10 \times \text{weight of sample}}$$

where f is the factor for the KF reagent.

2.2 DETERMINATION OF FACTOR FOR KF REAGENT

In a tared weighing bottle 0.10-0.15 gms of sodium acetate is taken and weighed accurately. Titrate it with KF reagent until a first permanent reddish tinge is observed.

$$\text{Factor, } f \text{ (mg water/ml)} = \frac{\text{Weight of acetate} \times 0.3971 \times 1000}{\text{ml of KF reagent}}$$

3.0 ACIDITY IN NITROCELLULOSE

3.1 PROCEDURE

2 gm of dried nitrocellulose (following the procedure given in 1.2) are weighed accurately and taken in a clean, dry 250 ml stoppered conical flask. To this, 90 ml of acetone is added, the stopper returned and the flask is agitated till nitrocellulose dissolves, 10 ml of distilled water is added and the flask is shaken to dissolve any precipitated nitrocellulose. To the solution, 0.5 ml of mixed indicator (neutral red and bromocresol green) is added and the solution is titrated with 0.01 N NaOH until a green colour appears. A blank titration is carried out using 90 ml of acetone, 10 ml of distilled water and 0.5 ml of the indicator.

$$\text{Acidity as } \% \text{ H}_2\text{SO}_4 = \frac{(\text{Titre-Blank}) \times 0.049 \times f}{\text{Weight of sample taken}}$$

where f = factor of 0.01 N NaOH used.

4.0 NITROGEN CONTENT IN NITROCELLULOSE

4.1 APPARATUS

There are several methods for the determination of the nitrogen content, but it is usually done using Lunge Nitrometer. For details of the apparatus refer to IS:1091-1976, 'Specification for Nitrocellulose'.

4.2 PROCEDURE

About 0.56-0.62 gm of the dried sample (following the procedure given in 1.2) is taken in a tared weighing bottle. The sample is further dried for 4 hours in a water oven maintained at 75°-80°C. The weighing bottle with the sample is then cooled in a desiccator over calcium chloride and weighed accurately. The difference gives the mass of the material. 5 ml of analar sulphuric acid (94.5%) is added into the weighing bottle and the mass is made into a slurry with a glass rod. The slurry is transferred into the cup of the Nitrometer

and drawn into the Nitrometer by operating the stopcock taking care to avoid sucking air into the reaction tube. The weighing bottle and the cup of the Nitrometer are rinsed several times with 94.5% sulphuric acid using a total of 15 ml for dissolving and rinsing. The reaction mixture is allowed to stand in the Nitrometer arm for 15 minutes and then shaken vigorously for 2 to 3 minutes. The gas generated is allowed to cool to room temperature and the volume is measured under atmospheric pressure by adjusting the leveling tube.

$$\% \text{ Nitrogen} = \frac{V \times P \times 0.02246}{(t + 273) \times W}$$

where V = volume of gas in ml
 P = corrected barometric pressure in mm of mercury
 t = room temperature in °C
 W = weight of the dried material taken

5. BERGMANN AND JUNK TEST FOR STABILITY OF NITROCELLULOSE

5.1 APPARATUS

For details of B & J bath and reaction tube assembly refer to IS: 109-1976, 'Specification for Nitrocellulose'.

5.2 PROCEDURE

The bath is filled with glycerine water mixture (adjusted to a boiling point of $132 \pm 0.2^\circ\text{C}$) and the copper tubes with liquid paraffin to the level of 50 mm below the top of the cover plate when reaction tubes are inserted to a depth of 1.5 cm. The bath is heated and maintained at $132 \pm 0.2^\circ\text{C}$.

About 10 gm of prepared sample (as given in I. 1) is dried for 4 hours at 65°C - 70°C in a water oven and cooled in a dessicator over calcium chloride. 2 gm each of dried sample are weighed accurately in two portions and transferred into the reaction tubes, brushing down any material adhering to the sides. The samples are compacted gently by pressing to 5 cm height. A third reaction tube is set apart as blank. In each of the head-pieces, 1.5 ml of water is filled and these are fitted to the reaction tubes using 3-in-1 oil at the joints. The reaction tubes are then lowered into the bath with suitable holders. After two hours the tubes are raised up and allowed to cool for about one hour. Water in each cup is transferred into a conical flask then rinsed with 25 ml distilled water & rinsing added to the flask. 20 ml of standard 0.1N hydrochloric acid is added to each reaction tube and to the blank. The tubes are stoppered and shaken vigorously to form a uniform slurry. The contents of the tubes are transferred to the corresponding flask with the rinsings. To each of the conical flasks 25ml of 0.1 N sodium hydroxide is added and the excess alkali is titrated with 0.1 NHCl using methyl red methylene blue mixed indicator

Mg of Nitrogen/gm. of dry Nitrocellulose = (Blank-Titre) $\times 0.7 \times f$
 where f = factor of 0.1 NHCL

6.0 VISCOSITY OF SOLUTIONS OF NITROCELLULOSE IN 95% V/V AQUEOUS ACTONE

The viscosity of Nitrocellulose solution is determined by the falling-sphere method using a steel ball of 1.58 ± 0.01 mm diameter.

6.1 APPARATUS

The details of the viscometer have been given in IS:1091 - 1976, 'Specification for Nitrocellulose'.

6.2 PROCEDURE

A solution of the requisite concentration is prepared by adding a fixed weight of dried nitrocellulose (see Table 3) in 100 ml of 95% v/v aqueous acetone (relative density 0.8097 at 20°C) taken in a glass tube 45 cm long with a 2.5 cm internal diameter. The tube is securely stoppered, shaken by hand to prevent the caking of the nitrocellulose into hard mass, and then rotated on a wheel until the mixture is homogeneous and free from any lump or gel pip. The solution is transferred to the viscometer, which is stoppered and clamped in a vertical position in a thermostat maintained at 20 ± 0.1 °C. The solution is allowed to stand until free from air bubbles and it has attained the temperature of 20 ± 0.1 °C. The stopper of the viscometer is then replaced by the one carrying the release tube and the still ball introduced in to the solution through this device.

The time of fall of the ball through 15 cm is noted. Similar determination is made using castor oil of known viscosity in place of nitrocellulose solution.

6.3 CALCULATION

$$\text{Viscosity, in poise} = \frac{T(S-S)}{T_1(S-S)} \times \text{viscosity of castor oil in poise}$$

where T = Time of fall (in seconds) in the nitrocellulose solution

T₁ = Time of fall in the castor oil

S = Density of steel ball

S₁ = Density of nitrocellulose solution at 20°C

S₂ = Density of castor oil (Viscosity of castor oil at 20°C = 9.65 poise)

Type of Nitrocellulose	Weight of Dry Sample (GM)
HM	10
HL, ML	20
HX, LX	40

Table 3 : sample size for falling sphere method viscosity determination.

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Viscosity Comparison Table *

HIGH NITROGEN GRADE (% N = 11.7% To 12.2%)

Euro Norms	Nitrex (India)	Bergerac (France)	Wolff (Germany)	Hercules (USA)	Green Tree (USA)
3E		E 250	E 1440	RS 1000-1500 sec	RS 1300 sec
4E	HH 2/4	E 250		RS 600-1000sec	
5E		E 150			RS 300
6E				RS 125-175 sec	RS 150 sec
7E	HM 40/60	E 140	E 1160	RS 60-80 sec	RS 75 sec
8E	HM 10/25	E 130		RS 30-40 sec	RS 35 sec
9E	HL 120/170	E 110	E 950		
10E		E 90		RS 15-20 sec	RS 15 sec
11E		E 80			
12E	HL 25/45		E 840	RS 5-6	
13E					RS 5 sec
14E					
15E		E 60	E 730		
19E		E 40			
20E	HX 70/90			RS 3/4 sec	
21E	HX 40/70				
22E		E 35	E 620		
23E	HX 30/50		E 560	RS 1/2 sec	RS 1/2 sec
24E	HX 20/30	E 32		RS 3/8 sec	
25E			E 510		
27E		E 27	E 400		
28E	HX 8/13			RS 1/4 sec	RS 1/4 sec
30E		E 24		RS 30-35 cp	RS 30 cp
31E	HX 5/8				
32E			E 375		
33E		E 20	E 360	RS 18-25 cp	
34E	HX 3/5	E 19	E 330	RS 1/8 sec	RS 10 cp

MEDIUM NITROGEN GRDE (% N = 11.2 To 11.7%)

14M	ML 18/25		AM 760	AS 3-6	AS 3-6
17M			AM 700		
27M			AM 500		
28M	MX 8/13				
34M	MX 3/5		AM 330		

LOW NITROGEN GRADE (% N = 10.7% To 11.2%)

23A	LX 30/50	A 27	A 600		
24A				SSI/2"	SSI/2"
27A			A 500		
28A	LX8/13	A 20		SSI/4"	SSI/4"
30A		A 15	A 400		
31A	LX 5/8				
34A	LX 3/5			SSI/8"	SSI/8"

Note : *An approximate equivalent for the grades produced by different manufactures.

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