Walsroder® Nitrocellulose

Essential for an Extra-Special Finish
Walsroder®
Nitrocellulose

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Dow Wolff Cellulosics

A brand name for cellulose derivatives

The core area of expertise of Dow Wolff Cellulosics is the development, production and sales of cellulose derivatives. Nitrocellulose (NC) is a raw material for printing inks and varnishes. Walocel methylcellulose is a highly effective additive for building materials. Methylcellulose regulates the consistency and hardening of mineral based building materials such as plaster, fillers and tile adhesives as well as dispersion paints. The high-purity cellulose derivatives carboxymethylcellulose (CMC) and hydroxypropylmethylcellulose (HPMC) have applications in food, cosmetics and pharmaceutical products. At its modern pilot plant Dow Wolff Cellulosics develops new products for new technologies for the production and processing of the sustainable raw material cellulose and its derivatives.

Continual investment in plants, products and processes put Dow Wolff Cellulosics among the most modern facilities in the world. This investment assures a reliable supply of products with consistently high quality.

Intensive research and development enables Dow Wolff Cellulosics to offer customers modern products with expert technical advice.

Our sales and technical marketing help customers to expand their business success through personal contacts.

Dow Wolff Cellulosics offers its customers worldwide reliable and flexible supplies through its global logistic network.

At Dow Wolff Cellulosics quality management is a daily practice. Our own quality control laboratories monitor the production processes 24 hours a day. Together with the production workers they ensure an optimal quality product.

Dow Wolff Cellulosics fulfills the standards ISO 9000 and ISO 14000.
Structure and
The most important raw material of nitrocellulose is chemical pulp, i.e. cellulose. It is obtained from wood or cotton. Both of these sustainable raw materials are available in large quantities now and will be in the longterm future. The sustainable quantities of cellulose far exceeds that used by man. The high-purity chemical pulp for nitrocellulose is produced from the long fiber woods of conifers. These conifers as a rule originate in the immediate vicinity of the paper mills. Cotton linters are the short fibers on the seed capsules and not the long fibers of the white tufts. Cotton Linters used in the production of cellulose derivatives are normally blends from various fields and commonly from various countries. Nitrocellulose is produced through a reaction of cellulose with nitrating acid (a mixture of nitric and sulfuric acid). Chemically the reaction itself is an esterification and not a nitration. An esterification is an equilibrium reaction. The degree of esterification can be controlled by the concentration of water in the reacting mixture. The higher the water content, the less the degree of esterification. Nitrocellulose with a very high degree of esterification (nitrogen content higher than 12.6 %) is used as gun cotton. Dow Wolff Cellulosics produces only industrial nitrocellulose with a nitrogen content of between 10.7 % and 12.3 %. This is used as a binder in printing inks and coatings.
Characterization

Walsroder Nitrocellulose and Walsroder NC-Chips are characterized by:

- the nitrogen content (degree of substitution)
- the viscosity (molecular weight)
- the phlegmatizer (damping agent or plasticizer respectively)
- phlegmatizer content

The product name contains all four characteristics. They entail:

**Walsroder Nitrocellulose E 560 Isopropanol 30 %**

- E: Substitution range (= nitrogen content)
- 560: Viscosity number
- Isopropanol: Damping agent
- 30 %: Content of damping agent

**Nitrogen Content**

In contrast to other products based on cellulose the degree of substitution of nitrocellulose is given indirectly by means of the nitrogen content (in reference to the substance in the dry state). A nitrogen content of 14.14 % is theoretically possible, all three positions of an anhydroglucose unit are substituted. In practice only a nitrogen content of 13.6 % can be reached. The nitrogen content of Walsroder Nitrocellulose and Walsroder NC-Chips for coatings and printing inks lies between 10.7 % and 12.3 %. Nitrocellulose with a nitrogen content above 12.6 % is classified as an explosive.

The degree of substitution (maximum value: 3) can be calculated from the nitrogen content by the following formula.

\[
\text{Degree of substitution} = \frac{3.6 \cdot \text{nitrogen content} \%}{31.13 - \text{nitrogen content} \%}
\]
Characterization and Properties
## Grades

### A-grades

**Nitrogen content:** 10.7 % – 11.3 %  
**Degree of substitution:** 1.89 – 2.05

Walsroder NC with a nitrogen content of 10.7 % to 11.3 % has the designation A, as this nitrocellulose is soluble in ethanol (= Alcohol).

A-grades are particularly characterized by their thermoplastic behavior. This is important for heat-sealing foils and/or films. A-grades are preferred in the production of printing inks because of their solubility in alcohol.

Varnish films of A-grades display a poor resistance to ethanol with the result that they are rarely used in wood coatings.

**Properties of A-grades**

- Formation of films with thermoplastic properties (heat-usable, e.g. for coating of aluminum foils)
- Fast solvent evaporation
- Good blending properties with aromatic hydrocarbons
- Good mechanical properties
- Solve specific coating related problems, such as:
  - Coatings dilutable with any amount of ethanol (wood polish)
  - Low odor coatings (printing inks)
  - Gel-type dip coatings
  - Heat-usable coatings (cellulose film and aluminum foil coatings)

### AM-grades

**Nitrogen content:** 11.3 % – 11.8 %  
**Degree of substitution:** 2.05 – 2.20

Walsroder NC with a nitrogen content of 11.3 % to 11.8 % has the designation AM, as this nitrocellulose is partially soluble in ethanol (=Alcohol Medium-soluble).

**Properties of AM-grades**

AM-grades lie in their behavior between the A and the E-grades. They are used only in a few special areas, e.g. in the coating of cellulose films or staple coatings (which are actually glues to keep the staples together).

### E-grades

**Nitrogen content:** 11.8 % – 12.3 %  
**Degree of substitution:** 2.20 – 2.35

Walsroder NC with a nitrogen content of 11.8 % to 12.3 % has the designation E, as this nitrocellulose is soluble in Esters (the A and AM-grades are also soluble in esters of course). E-grades are the standard grades for wood and leather coatings. In comparison to the A and AM-grades they are more resistant to alcohol.

**Properties of E-grades**

- Form hard films
- Very fast solvent evaporation
- Easy to dilute with alcohols, aliphatic and aromatic hydrocarbons
- Attain very good mechanical properties (cold-check, elongation, hardness, tear resistance)
The viscosity of a solution of Walsroder Nitrocellulose and Walsroder NC-Chips is influenced by the molecular weight of the nitrocellulose. The determination of the molecular weight is far more complex than the determination of the viscosity. The viscosity of a nitrocellulose solution is very important, therefore a viscosity index is provided (measured in a specified solvent mixture). Fikentscher’s K-value (see page 51) was used as the viscosity index for describing Walsroder Nitrocellulose and the Walsroder NC-Chips. This appears after the degree of substitution (A, AM or E). A low K-value indicates a low viscosity, i.e. low molecular weight. A high K-value indicates a high viscosity, i.e. high molecular weight.

The viscosity of the individual nitrocellulose from Dow Wolff Cellulosics is specified by the Cochius method (see page 44). To compare the viscosity of nitrocellulose from various suppliers, ISO 14446 is a useful standard to follow (see page 47).


Viscosity

Low Viscosity Nitrocellulose

The low viscosity grades of nitrocellulose include the viscosity levels up to A 400, AM 330 and E 375/E 380 (in accordance with ISO 14 446 this corresponds to levels from and including 30 A, 30 M, 30 E). Low viscosity nitrocellulose can be used for formulating high solids coatings and printing inks. Highly pigmented coatings and inks can also be produced using low viscosity nitrocellulose. The low viscosity grades are used predominantly in printing inks or overprint varnish for plastic films or aluminum foils in food packaging applications. They are also used in primers for wood coatings.

Medium Viscosity Nitrocellulose

The medium viscosity grades of nitrocellulose include the viscosity levels A 500, AM 500, E 400 up to E 620 (in accordance with ISO 14446 this corresponds to: 18 E – 29 E, 18 M – 29 M, 18 A – 29 A). The medium viscosity grades are the most frequently used grades and are used above all in printing inks for packaging (mainly A 500), in wood lacquers and varnishes, automotive repair paints and nail varnishes.

High Viscosity Nitrocellulose

The high viscosity grades of nitrocellulose include the viscosity levels A 700, E 840 and above (corresponding to the ISO 14446 grades: 17 E, 17 M, 17 A and below). The high viscosity grades provide very flexible and low-gauge films. Applications include leather coatings, metal coatings (zapon lacquers) and highly specialized uses such as the production of membrane filters. In many cases high viscosity nitrocellulose is used for improving the viscosity of coating systems.
Industrial nitrocellulose is required by law to contain at least 25% damping agent (e.g., alcohol, water) or 18% plasticizer. The purpose of damping agents or plasticizers is to phlegmatize the nitrocellulose in order to deactivate the hazardous properties of dry nitrocellulose (high flammability, high burning rates). Nitrocellulose with a damping agent content below 25% or respectively a plasticizer content of under 18% is classified as an explosive without regard of its nitrogen content. The nitrocellulose grades offered by Dow Wolff Cellulosics are damped with at least 30% alcohol or water, or in the case of NC-Chips phlegmatized with 20% plasticizer. The difference between Walsroder Nitrocellulose and Walsroder NC-Chips is the type of phlegmatizer used (alcohol or plasticizer). The noticeable difference is only their physical form and the method of handling them. Within the mean viscosity indicated, the nitrocellulose in both Walsroder Nitrocellulose and Walsroder NC-Chips is the same.

Phlegmatizer and plasticizer

Dow Wolff Cellulosics are damped with at least 30% alcohol or water, or in the case of NC-Chips phlegmatized with 20% plasticizer. The difference between Walsroder Nitrocellulose and Walsroder NC-Chips is the type of phlegmatizer used (alcohol or plasticizer). The noticeable difference is only their physical form and the method of handling them. Within the mean viscosity indicated, the nitrocellulose in both Walsroder Nitrocellulose and Walsroder NC-Chips is the same.

Walsroder® Nitrocellulose

Walsroder Nitrocellulose is most frequently phlegmatized by damping it with Isopropanol. It is used for almost all kinds of coating systems, including wood coatings, industrial coatings, nail varnishes and automotive repair paints. In some regions, nitrocellulose damped with isopropanol is also used for manufacturing printing inks.

Nitrocellulose damped with ethanol is mainly used for manufacturing printing inks, which are usually ethanol based (with a minor amount of ethyl acetate). No other solvent is required. In some regions nitrocellulose damped with ethanol is also used for manufacturing wood coatings.

Nitrocellulose damped with water is used to produce color preparations (color chips) and leather coatings: the so-called NC emulsions.
Walsroder® NC-Chips

Walsroder NC-Chips are a specific form of nitrocellulose containing a plasticizer as their phlegmatizing agent. They are used where Walsroder Nitrocellulose cannot meet certain technical requirements. This includes:

- Production of printing inks where only ethyl acetate is to be the solvent used
- Production of coatings for electrostatic spraying techniques. By using NC-Chips, solvents with a high flash point can be selected (ethanol and isopropanol have a flash point of around 12 °C)
- Production of coatings that have a flash point above 55 °C and are therefore not classed as flammable and do not require to be labeled as such. This is possible by selecting suitable solvents with a high flash point
- Use in two-pack polyurethane coatings. The expensive isocyanate component would react with the alcohol and the water in nitrocellulose damped with alcohol. By using NC-Chips it is possible to reduce the amount of isocyanate to obtain a certain cross-linking

- If the package is opened and not properly re-closed, the phlegmatizing agent cannot evaporate in the case of NC-Chips
- Walsroder NC-Chips are highly suitable for producing coating systems where only one solvent is to be used
- Walsroder NC-Chips with ESO (epoxidized soybean oil) as their plasticizer are mainly used for wood and industrial coatings. A major application is two-pack polyurethane coatings. Walsroder NC-Chips with ATBC (acetyl tributyl citrate) are used mainly in the production of printing inks which contain only ethyl acetate as the solvent

DBP (dibutyl phthalate), which was in widespread use in the past, is no longer offered as it is not considered safe due to a possible hormonal effect. The European Union has issued new classification and labeling requirements for DBP. Coatings produced with more than 0.5 percent DBP, for example, are required to be labeled with the skull and crossbones sign as from July 30, 2002 (28th adaptation of Directive 67/548/EEC).
Industrial Production

Raw Materials
The raw material used for producing Walsroder Nitrocellulose and Walsroder NC-Chips is carefully selected and well-characterized types of cellulose, the exact specifications having been agreed with the cellulose suppliers. Compliance with these specifications is regularly checked during incoming goods inspections. This also applies to all other raw materials, thus assuring the consistent quality of Walsroder Nitrocellulose and Walsroder NC-Chips.

Nitration (Control of nitrogen content)
The cellulose is separated into fibers to obtain a large surface for the chemical reaction (esterification). In the next step the cellulose fibers are mixed with the nitrating acid, a mixture of nitric and sulfuric acid, and is caused to react with the acid. By varying the water content of the nitrating acid, the nitrogen content of the nitrocellulose is controlled (esterification is an equilibrium reaction). The nitration plant was brought into operation in 1996. It is a state-of-the-art production unit incorporating more than 100 years of nitrocellulose manufacturing experience and engineering technology.

Acid Separation
After nitration, the nitrocellulose is separated from the spent acid in the centrifuges, suspended in water and as a suspension is transported to the next part of the plant.

Pressure Boiling (Control of viscosity)
The viscosity of nitrocellulose is adjusted by heating the nitrocellulose water suspension under pressure to temperatures above 100 °C. In this step the nitrocellulose molecule is thermally reduced. To obtain a nitrocellulose with a high molecular weight (high viscosity) it is only heated briefly; however, to gain a low molecular weight (lower viscosity) it is heated longer.

Post-Stabilization
The pressure boiling is followed by the post-stabilization. The nitrocellulose is stabilized by washing it with water and heating it from 70 ° to 90 °C. The sulfuric esters which are less stable than those of nitric acid are split in this way. Following this the nitrocellulose is washed neutral.

Dehydration and Alcoholization (Control of damping agent and water content)
The nitrocellulose suspension is dehydrated on centrifuges to a water content of 35 %. This water-damped nitrocellulose can be packed directly or used for the production of NC-Chips. For the production of alcohol-damped nitrocellulose water is washed out in a further centrifuge by the appropriate damping alcohol and then the alcohol-damped nitrocellulose is filled into drums or cartons. The resulting diluted alcohol is transferred to the alcohol distillation plant.

Chips Production (Control of plasticizer and water content)
For the production of Walsroder NC-Chips the water-damped nitrocellulose is mixed with the appropriate plasticizer, dried and filled into drums.

Acid Recovery
The spent acid is partly regenerated by fresh acid and used again directly in nitration. The other part of the spent acid is distilled into nitric acid and sulfuric acid and stored for the meantime in acid tanks.

Alcohol Distillation
The diluted alcohol is distilled and the separated alcohol goes back into the process.
Nitrocellulose is produced by causing cellulose to react with nitrating acid (a mixture of nitric acid and sulfuric acid). Following complex washing and stabilizing stages, damping agents (alcohols or water) or plasticizers are added to the nitrocellulose which is then marketed as Walsroder Nitrocellulose or Walsroder NC-Chips. The schematic flow chart gives an outline of the individual stages of the process.
Environmental Protection

The raw materials used are treated, as far as possible, and fed back into production. This is clearly shown in the example of the acid recovery plant and in the alcohol distillation in which the diluted acids and alcohols from the production process are re-concentrated.

A further aspect of environmental protection is the exhaust air cleaning. Exhaust air from the nitration that is contaminated with nitrous gases is washed in a column. From this a diluted nitric acid is obtained. This acid is fed into the acid recovery plant and will be used in the nitration process again. The concentration of nitrous gases in the exhaust air is far below the values stipulated in the regulations.

Quality Control

Testing procedures are at hand for all raw materials, half-finished goods and end products. The integrated quality control system automatically generates checking tickets and prints out directions to be checked at the appropriate places. A further processing of the materials is only then possible when all features to be tested within the specifications are available.

This is the case with all raw materials, half-finished goods and end products. Through these comprehensive controls, before, during and after the production process, all relevant features of quality are apparent. In this way a constantly high-quality level of Walsroder Nitrocellulose and Walsroder NC-Chips is guaranteed.
Walsroder® Nitrocellulose and Walsroder® NC-Chips are predominantly used as binders in printing inks and wood coatings. Their unique properties are the reason for their widespread use and large variety of applications:

### Printing Inks

Walsroder Nitrocellulose is used in liquid printing inks, for instance for printing plastic films and aluminum foils intended mainly for food packaging. The printing methods involved are rotogravure and flexographic printing. Printing inks containing nitrocellulose result in graphics with high brilliance and high resolution. The rapid and complete solvent evaporation obtainable with nitrocellulose-based printing inks allows high printing speeds to be achieved on modern high speed printing machines.

A crucial factor for food packaging is the fact that nitrocellulose is toxicologically harmless. As nitrocellulose is produced from a natural macromolecule (cellulose) it does not contain any toxic monomers.

- wide compatibility with many other binders and plasticizers
- solubility in and compatibility with many organic solvents
- transparent clear film
- odorless
- non-toxic
- free of monomers
- robust and easy-to-use systems
- rapid evaporation of solvents (rapid drying)
- extremely low solvent retention
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Recommended NC grades for printing inks
Applications
A further wide area where Walsroder Nitrocellulose is used is wood coatings. Nitrocellulose is contained in primers, intermediate coats and top coats. Nitrocellulose-based lacquers and varnishes used as wood coatings have a unique quality that no other coating binder achieves: they accentuate wood grain particularly well. Nitrocellulose is primarily used in combination with alkyd resins (nitrocellulose combination lacquers). It is also contained in acid curing lacquers and two component (two-pack) polyurethane coatings in order to accelerate solvent evaporation and to provide a harder coating film. NC coatings are very easy to apply. They give a smooth and decorative surface much more readily than water-based coating systems do. Another benefit: NC coatings dry much faster.

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Recommended NC grades for wood coatings
Other Applications

Beside printing inks and wood coatings there are numerous other applications in which Walsroder Nitrocellulose is used, including:

- Metal coatings
- Paper coatings
- Leather coatings
- Effect finishes
- Nail varnishes

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- **Metal coatings**
- **Zapon lacquers**
- **Coatings for staples**
- **Primers**
- **Fillers**
- **Automotive (repair) coatings**
- **Paper coatings**
- **Coatings for paper**
- **Coatings for calendering**
- **Leather coatings**
- **Protective coatings**
- **Pigment base coats**
- **Split hide coatings**
- **Base coatings**
- **Aqueous emulsions**
- **Effect finishes**
- **Multicolor coatings**
- **Hammer finishes**
- **Cackle finishes**
- **Others**
- **Nail varnishes**
- **Adhesives**
- **Electric bulb lacquers**
- **Glass coatings**

*Recommended NC grades for different applications*
ons
The degree of substitution determines the solubility of nitrocellulose in organic solvents. Walsroder Nitrocellulose and Walsroder NC-Chips are divided according to their solubility.

### A-grades
Nitrogen content: 10.7 % – 11.3 %
Soluble in: alcohols, esters, ketones and glycol ethers.

### AM-grades
Nitrogen content: 11.3 % – 11.8 %
Soluble in: esters, ketones and glycol ethers, with good blending capability and compatibility with alcohol.

### E-grades
Nitrogen content: 11.8 % – 12.3 %
Soluble in esters, ketones and glycol ethers. Blending capability with alcohol.

The solvents used for dissolving nitrocellulose are divided into 3 groups in accordance with their dissolving capabilities:

#### Active or true solvents
These solvents are capable of completely dissolving nitrocellulose at room temperature.

- Ketones such as acetone, methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK)
- Esters such as ethyl acetate, butyl acetate, methoxy propyl acetate
- Glycol ethers such as methyl glycol ether, ethyl glycol ether, isopropyl glycol ether
- Alcohols, but only methanol, ethanol with A-grades

#### Latent solvents
These solvents are not capable of dissolving nitrocellulose on their own at room temperature. They are “activated” by adding true solvents or certain non-solvents, thus virtually becoming a solvent.

- Alcohols such as ethanol, isopropanol, butanol
- Ether, such as diethyl ether

#### Non-solvents
These substances are incapable of dissolving nitrocellulose either directly or indirectly. They can be used in combination with true solvents as extenders or diluents.

- Aliphatic and aromatic hydrocarbons such as benzene, toluene, xylenes
Nitrocellulose in Solution

The dissolving capacity (solvency) decreases within a class of substances in proportion to the increase in relative molecular weight. This results from the increasing hydrocarbon content, which is particularly evident with alcohols:

Methanol is a true solvent, ethanol is a true solvent for the A grades and a latent solvent for the E grades; n-decyl alcohol is a non-solvent both for the E grades and the A grades. Similar behavior can be observed with ketones and esters.

A mixture of a true solvent and a latent solvent can have the same or a partly better solvency than the true solvent. This is frequently observed with mixtures of some true solvents with for instance ethanol, isopropanol or butanol.

Sometimes a mixture of two latent solvents may have the dissolving capability of a true solvent. An example of this is when ethanol and diethyl ether are mixed.

Non-solvents are generally added to the coating to reduce the cost of material. Another function of non-solvents is their effect on the coating’s evaporating behavior.

The viscosity of a nitrocellulose solution depends not only on the molecular weight of the nitrocellulose grade, but also on the solvent or solvent mixture used. This behavior is caused by the varying structure of the individual solvents, which can be described by, among other things, the solubility parameter, dipole moment and the capability of forming hydrogen bonds. In general the following rules apply:

- Within a given class of solvent, the higher the relative molecular weight of the solvent, the higher the viscosity of the nitrocellulose solution (the dissolving capability of the solvent decreases equally in relation to the increasing molar weight). Hence, the viscosity of a nitrocellulose solution increases when using acetates in the sequence ethyl acetate, butyl acetate, amyl acetate.

- Latent solvents can increase the solvency and cause a reduction in the viscosity of the nitrocellulose solution.

- The use of non-solvents (diluents), which have no dissolving capability, causes the viscosity of the nitrocellulose solution to increase. Excessive addition of non-solvents may cause gelling and flocculation.
By adding ethanol as a latent solvent for nitrocellulose to a 10 % solution of E 510 in butyl acetate, the viscosity of the solution will decrease. The viscosity reaches a minimum at around 30 % ethanol and 70% butyl acetate and then increases again.

Increasing the content of non-solvent (toluene) increases the viscosity of the nitrocellulose solution. This is shown for a 10 % solution of E 510 in butyl acetate. At a content of 10 to 30 % by weight of toluene in the solution a kind of plateau is reached where the viscosity remains virtually constant. If the amount of toluene is further increased, the viscosity of the solution will rise sharply until flocculation finally occurs.

The viscosity of a nitrocellulose solution is dependent on the nitrocellulose concentration and the grade of nitrocellulose. The viscosity will increase if you increase the concentration or use a nitrocellulose with a higher viscosity. The figure shows the viscosity of different nitrocellulose grades at different concentrations, measured in the Höppler viscometer at 20 °C with ethylacetate as solvent.
**Technical Properties**

**Nitrocellulose coatings**
The excellent film-forming properties of the physically drying nitrocellulose characterizes nitrocellulose coatings. Besides this nitrocellulose is also compatible with many other raw materials for coatings, and can be combined with resins, plasticizers, pigments and additives for varying effects. Film formation is dependent on the content of non-volatile coating components and on the composition of the solvent mixture (active and latent solvents and non-solvents).

**Resins**
The incorporation of synthetic resins into nitrocellulose coatings such as: alkyd resins, maleic resins, ketone resins, urea resins, polyurethane resins, polycrylates, polyester and polycrylate resins containing hydroxyl groups brings the following benefits:

- an increase in solids content
- an improvement in adhesive strength and gloss
- improved resistance to heat, light, alcohol and water

The use of natural resins (such as dammar, shellac, and colophonium) in nitrocellulose coatings has largely been replaced by synthetic resins named above. The selection criteria for the appropriate resins are: color, effect on solvent release, gloss, hardness, sandability, resistance to light and durability of the coating.

**Plasticizers**
Plasticizers which are compatible with nitrocellulose have a positive influence on the coating:

- improve adhesion and gloss
- improvement in mechanical properties such as elongation, flexing fastness, resistance to folding and creasing and thermo-formability
- increasing the resistance to light, heat, low temperatures, and changes in temperature (cold-check-test)

Plasticizers are classified as solvent (gelatinizing) and non-solvent for NC. Nitrocellulose is soluble, for example in:

- diisobutyl phthalate (DIBP),
- dicyclohexyl phthalate (DCHP),
- epoxidized soya oil (ESO),
- acetyl tributyl citrate (ATBC),
- octyl diphenyl phosphate (ODDP),
- triphenyl phosphate

Plasticizers in which nitrocellulose is insoluble, are:

- raw and blown vegetable oils,
- stearates, oleates

All classes of substances with long aliphatic substituents (phthalates or adipates for example) such as:

- disononyl phthalate (DINP)
- dioctyl adipate (DOA)

are non-solvents for nitrocellulose.

The use of solvent and non-solvent plasticizers or a combination of both depends on the application.

A characteristic of non-solvent plasticizer is that they exude when the coating film is heated above a certain temperature. This behaviour is undesirable. By combining non-solvent plasticizers with solvent plasticizers this temperature could be raised significantly.

This is important with leather coatings, as they contain a high amount of plasticizers. Leather coatings therefore mostly contain raw or blown castor oil (non-solvent plasticizer) in combination with a solvent plasticizer such as diisobutyl phthalate.

Non-solvent plasticizers such as stearates give off solvents quickly. For this reason they are used in rapid-sanding primers for wood. To increase adhesion often plasticizers which are a solvent for nitrocellulose are added; there are only a few non-solvent plasticizers which improve adhesion. In practice often a combination of plasticizers are used to meet the requirements as good as possible.
ties
Lacquers
Influence of light, acids and alkalis

Exposure to sunlight or UV radiation will reduce properties of the nitrocellulose films causing yellowing and brittleness. Solvents, plasticizers and resins can reduce or accelerate the tendencies of yellowing. Coating additives and solvents should react as neutrally as possible. Acids affect a viscosity reduction of nitrocellulose coatings. Weak alkalis lower the viscosity of nitrocellulose coatings analogously to acids. Strong alkalis (some amines for example) lead to discoloration of the nitrocellulose solution and films. Exceptions are urea derivatives and urethanes. The coating system should be tested beforehand.

Combination Lacquers

The mechanical properties of combination lacquers consisting of nitrocellulose, alkyd resins and isocyanates lie between those of pure polyurethane coatings and nitrocellulose-alkyd resin coatings. An effective ratio has proven to be 1 part nitrocellulose: 1 part alkyd resin: 0.5 parts polyisocyanates (100 %). Nitrocellulose combination lacquers of this type are distinguished by:

- excellent flow and rapid drying
- good abrasion resistance and solvent stability
- very good matting properties

The following properties can be affected by using different combinations of nitrocellulose and polyisocyanates (such as Desmodur® products):

- viscosity, pot life, sandability, flexibility, hardening

The addition of zinc octoate (0.2 – 0.3 % relative to nitrocellulose) accelerates the hardening of the film, but reduces the pot life of the lacquer. Polyacrylates containing OH groups (e.g. the range Desmophen®) are preferably converted with aliphatic isocyanates, and then to add up to 50 % nitrocellulose (relative to the polyacrylate content).

The addition of nitrocellulose accelerates solvent release and improves physical pre-drying. The use of nitrocellulose is also advantageous to coatings based on unsaturated polyester, acid-curing coatings and UV-curing coatings. Particularly suitable for combination lacquers containing isocyanates are the plasticized ester soluble nitrocellulose chips of low or medium viscosity. These grades have particularly beneficial effect on the hardness and sandability of the coating.
Nitrocellulose falls under numerous laws and regulations in different countries. All users are obliged to acquaint themselves with legal requirements and local regulations governing the handling and storage of nitrocellulose in their country. In what follows as an example some German and European regulations are highlighted without being exhaustive.

**Legal Regulations**

**The German Explosives Act**
Industrial nitrocellulose, because of its chemical similarity to gun cotton, falls under the EC “Council Directive 93/15/EEC of 5 April 1993 on the harmonization of the provisions relating to the placing on the market and supervision of explosives for civil uses”.

This was implemented into German law in the amendment to the law on explosive substances and other regulations (SprengAndG. from 1997, published on June 23, 1998 in the Bundesgesetzblatt 1998, part 1, number 39, issued on June 29, 1998).

**Regulations on dangerous substances**
As a dangerous substance (flammable) Walsroder Nitrocellulose falls under the EU directive 67/54/EEC and the amendments. In Germany this directive has been implemented by the “Gefahrstoffverordnung” (Regulation on dangerous substances).

Questions on the handling and storage of nitrocellulose are covered in our booklet “Handling and Storage of Nitrocellulose”

For download and ordering:
[www.dowwolffcellulosics.com](http://www.dowwolffcellulosics.com)
**Transport regulations**

According to the international regulations on the transport of dangerous goods Walsroder Nitrocellulose is a substance of the class 4.1 (flammable solid)

UN 2555 Nitrocellulose with water
UN 2556 Nitrocellulose with alcohols
UN 2557 nitrocellulose, mixture, with plasticizer

Different rules apply to various carriers: rail (RID), road (ADR), water (IMDG code), air (ICAO, IATA, DGR regulations).

**Packaging**

- Walsroder Nitrocellulose is packed in fiber drums of various sizes (110 l, 200 l) and in cardboard boxes.
- Walsroder NC-Chips are packed in 110 liter fiber drums.

The packaging of Walsroder Nitrocellulose and Walsroder NC-Chips is in accordance with the legal requirements for transport over land and by sea and in line with the recommendations of the UN and with the national and international rules derived from them.

**Food contact**

Nitrocellulose is approved for use as a coating or printing ink for food packaging. Direct contact with the food is permitted in accordance with:

**European Union:**
2002/72/EG, 93/10/EWG

**FDA regulations:**
21 CFR 175.105, 21 CFR 175.300
21 CFR 176.170, 21 CFR 177.1200
Blending Charts

By mixing two types of nitrocellulose of different viscosity’s an intermediate viscosity could be reached. The ratio of the two nitrocellulose grades can be obtained from the blending charts. Actually some small deviations in the ratio compared with the ratio from the blending chart can occur. Reasons for this are that the actual viscosity of the individual grades fluctuates within the viscosity specification and that also the actual solid content of the nitrocellulose fluctuates within the specification.

Viscosity measured in seconds according to Cochius in butanol / ethyl glycol / toluene / ethanol (1:2:3:4)
How to use the blending chart?

For example:

By mixing A 400 and A 700 an A 500 viscosity should be produced. What ratio of A 400 to A 700 is to be used?

Procedure:

(1) Enter the value for the viscosity of the A 400 on the left-hand side of the blending chart and the value of the A 700 on the right-hand side (vertical scale). Draw a straight line through the points.

(2) Mark the point this straight line crosses with the required viscosity and draw through this cross point a perpendicular line downwards.

(3) The cross point of this line with the horizontal scale is the mixing ratio you seek. The upper scale gives the share of type entered on the left (here: A 400), and the bottom scale the share of the type entered on the right (here: A 700).

The mixing ratio is:
87 parts A 400 and 13 parts A 700.
Methods of Analysis

Viscosity by Cochius

The viscosity of Walsroder Nitrocellulose and Walsroder NC-Chips is determined exclusively by the Cochius method for grading purposes. To test the viscosity, dried nitrocellulose is dissolved in a solvent mixture in concentrations appropriate to the viscosity category. In a Cochius tube (temperature controllable vertical glass tube with a diameter of 7 mm) at 18 °C the time required for a rising air bubble of around 0.5 to 2.0 cm³ to cover the distance of 500 mm between two marks indicated on the viscometer is measured in seconds.

The Cochius viscosity of the individual grades is measured in the following solvent mixtures:

- **A-grades**: butanol : ethyl glycol : toluene : ethanol = 1 : 2 : 3 : 4
- **AM-grades**: butanol : ethyl glycol : toluene : ethanol = 1 : 2 : 3 : 4
- **E-grades**: butanol : butyl acetate (98/100) : toluene = 3 : 4 : 5

<table>
<thead>
<tr>
<th>Grade</th>
<th>Cochius Viscosity</th>
<th>Concentration (ato)</th>
<th>Solvent mixture</th>
<th>Nitrogen content</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 400</td>
<td>20s – 23s</td>
<td>12 %</td>
<td>1:2:3:4</td>
<td>10.7 % - 11.3 %</td>
</tr>
<tr>
<td>A 500</td>
<td>30s – 34s</td>
<td>6 %</td>
<td></td>
<td>11.3 % - 11.8 %</td>
</tr>
<tr>
<td>A 700</td>
<td>24s – 28s</td>
<td>15 %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>AM 330</td>
<td>24s – 26s</td>
<td>12 %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>AM 500</td>
<td>34s – 37s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 330/E 360</td>
<td>12.5s – 14.5s</td>
<td>12 %</td>
<td>3:4:5</td>
<td>11.8 % - 12.3 %</td>
</tr>
<tr>
<td>E 375/E 380</td>
<td>16s – 18s</td>
<td>10 %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 400</td>
<td>15s – 18s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 510</td>
<td>24s – 27s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 560</td>
<td>34s – 37s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 620</td>
<td>40s – 44s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 840</td>
<td>33s – 38s</td>
<td>5 %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 950</td>
<td>100s – 120s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E 1160</td>
<td>50s – 75s</td>
<td>3 %</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The advantage of this method is that it gives repetitive results very quickly. This is important for process control. Other methods of viscosity determination need more time and do not allow a fast response closed loop to control the production process.
Comparison between the Dow Wolff Cellulosics products, ISO 14446 and ASTM 1343:

<table>
<thead>
<tr>
<th>Description</th>
<th>ISO 14446</th>
<th>ASTMD 1343</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 400</td>
<td>30A</td>
<td>30 – 35 cps</td>
</tr>
<tr>
<td>A 500</td>
<td>27A</td>
<td>1/4 sec</td>
</tr>
<tr>
<td>A 700</td>
<td>15A</td>
<td>3 – 4 sec</td>
</tr>
<tr>
<td>AM 330</td>
<td>34M</td>
<td>18 – 25 cps</td>
</tr>
<tr>
<td>AM 500</td>
<td>27M</td>
<td>1/4 sec</td>
</tr>
<tr>
<td>E 330/E 360</td>
<td>34E</td>
<td>18 – 25 cps</td>
</tr>
<tr>
<td>E 375/E 380</td>
<td>31E</td>
<td>30 – 35 cps</td>
</tr>
<tr>
<td>E 400</td>
<td>28E</td>
<td>1/4 sec</td>
</tr>
<tr>
<td>E 510</td>
<td>24E</td>
<td>3/8 sec</td>
</tr>
<tr>
<td>E 560</td>
<td>23E</td>
<td>1/2 sec</td>
</tr>
<tr>
<td>E 620</td>
<td>22E</td>
<td>-</td>
</tr>
<tr>
<td>E 840</td>
<td>12E</td>
<td>5 – 6 sec</td>
</tr>
<tr>
<td>E 950</td>
<td>9E</td>
<td>15 – 20 sec</td>
</tr>
<tr>
<td>E 1160</td>
<td>7E</td>
<td>60 – 80 sec</td>
</tr>
</tbody>
</table>
Viscosity by ISO 14446

This method of determining the viscosity can be used alternatively to the Cochius method. The viscosity is measured at 20 °C in a Höppler falling ball viscosimeter. The result of the method is the concentration of dried nitrocellulose in acetone, to which 5 % water has been added, that gives an apparent viscosity of 400 ± 25mPas in the falling ball viscosimeter. Sometimes referred to as standard viscosity. The ISO 14446 viscosity is stated as a numerical value identical to the nitrocellulose concentration.

If this numerical value is an integer, the grade is a so called standard grade. Depending on its solubility in ethanol, the numerical value is prefixed with an A (= A-grade), M (= AM-grade) or E (= E-grade).

Using the standard viscosity it is possible to compare nitrocellulose grades supplied by different manufacturers.

Viscosity by ASTM D 1343

The viscosity of a nitrocellulose (solution) of medium and high viscous types according to ASTM D 1343 is determined in a special falling ball viscosimeter, low viscosity grades are measured in a capillary viscosimeter (Ubbelohde).

The solvent used is a mixture of 25 % ethanol, 20 % ethyl acetate, and 55 % toluene, temperature is 25 °C. The falling ball viscosimeter has an internal diameter of 63.5 mm (2.5 inch), the distance between the two marks is 50.8 mm (2 inch), the steel ball has a diameter of 2.38 mm (3/32 inch). With high viscosity grades (higher than E 620) the nitrocellulose concentration is 12.2 % (dry NC), at 1/2 sec grades (E 560) 20 % and at 1/4 sec grades (E 400) 25 %. For measurements in the capillary viscometer (viscosity less than E 400) a concentration of 12.2 % is used.

The ASTM method is very widespread in the Americas. In Asia differing methods are used that correspond to the ASTM nomenclature, with their results being very similar, although the geometry of the Asian methods deviate from the ASTM method.

Two examples are: KSM 3814 (Korea), identical with JIS K 6703 (Japan).
The deflagration temperature (ignition point) is determined in accordance with the transport regulations for dangerous substances (ADR). 0.2 g of dried nitrocellulose is put into a 125 mm tall test tube with an inside diameter of 15 mm and a wall thickness of 0.5 mm, which is placed in a Wood’s metal bath that has been heated to 100 °C. The test tube must be immersed exactly 20 mm into the metal bath. The centre of the calibrated thermometer must be level with the bottom of the test tube. The temperature of the metal bath is incremented by 5 °C every minute. As soon as the nitrocellulose self-ignites (as can be heard from the deflagration that occurs), the temperature (i.e. deflagration temperature or ignition point) is read off. The deflagration temperature of Walsroder Nitrocellulose is above 180 °C in compliance with regulations, and that of Walsroder NC-Chips is above 170 °C.
In special apparatus for splitting off gases which is kept at a constant temperature of 132 °C, the tubes into which 2 g of dry Walsroder Nitrocellulose has been filled are heated for two hours. The amount of nitrous gases split off after reduction to nitrogen monoxide is then measured. The nitrocellulose is considered stable if no more than 2.5 ml of nitrogen monoxide is split off per gram of nitrocellulose.

K-Value according to Fikentscher

As a measure of the mean molecular weight of a polymeric substance, Fikentscher derived the k value from measurements of the relative viscosity of solutions of polymer solutions. The k value is calculated by applying the following equation:

\[
\log \frac{\eta_c}{\eta_0} = \left( \frac{75 \cdot k^2}{1 + 1.5 k \cdot c} + k \right) \cdot c
\]

It signifies:
where \( c \) = concentration in g/100 ml; \( \eta_c \) = viscosity of the solution; \( \eta_0 \) = viscosity of the solvent; \( k \) = value acc. to Fikentscher (\( K = 1000 \cdot k \))

For Walsroder Nitrocellulose and Walsroder NC-Chips the k value calculated from Fikentscher’s equation is multiplied by 1000 and stated as the K value.

To determine the k value, dissolve 2 g of dried nitrocellulose in 100 ml acetone (with high-viscosity grades an appropriately smaller quantity), and determine the viscosity of the solution at 25 °C, using for example a Höppler falling-ball viscosimeter. Then determine the viscosity of the pure solvent in the same viscosimeter. Insert the viscosity readings and the nitrocellulose concentrations into the above equation and calculate the k value. The K value is obtained by multiplying the k value by 1000.

Literature: Fikentscher, Cellulosechemie 13, (1932) 58
To determine the nitrogen content by the Schlösing-Schulze-Tiemann method, a precision-weighed amount of nitrocellulose is heated in the presence of iron(II) chloride and hydrochloric acid. Under these conditions nitrate is reduced to NO, which can be measured volumetrically and is proportional to the nitrogen content of the nitrocellulose.

Comparison tests applying automated methods for determining the nitrogen content have repeatedly showed that the Schlösing-Schulze-Tiemann method was the most reproducible method, showing the least variations of individual readings from the mean value.

Further developments in instrumental chemical analytics made it possible to reduce the time for analysis drastically. A new method of analysis was successfully introduced, the near-infrared spectroscopy (NIR). This method is used today for production control. As the NIR is not an absolute method and requires calibration, the Schlösing-Schulze-Tiemann method is used parallel to it in order to check the calibration of the NIR method.
Nitrogen Content
by Schlösing-Schulze-Tiemann
Nitrogen Content by NIR
Glossary

Walsroder NC
All the grades of nitrocellulose damped with alcohol and water (Walsroder Nitrocellulose) and all nitrocellulose grades plasticized with plasticizers (Walsroder NC-Chips)

Walsroder Nitrocellulose
All grades of nitrocellulose damped with water and alcohol

Walsroder NC-Chips
All grades of nitrocellulose plasticized with plasticizers

Industrial Nitrocellulose
Nitrocellulose with a nitrogen content below 12.6% which are mainly used as raw materials for coatings and printing inks

Gun cotton
Nitrocellulose which is used as a propellant in ammunitions and in explosives

Phlegmatizer
In order to reduce the flammability and burning speed of dry nitrocellulose a phlegmatizer (alcohol, water or plasticizer) is added to industrial nitrocellulose in accordance with the legal requirements.

Cotton Linters
The small hairs present on the outside of the cotton seeds. They are produced as a by-product of the production of cotton seed oil.

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• downturns in the business cycle of the industries in which we compete;
• new regulations, or changes to existing regulations, that increase our operating costs or otherwise reduce our profitability;
• increases in the price of our raw materials, especially if we are unable to pass these costs along to customers;
• loss or reduction of patent protection of our products;
• liabilities, especially those incurred as a result of environmental laws or product liability litigation;
• fluctuation in international currency exchange rates as well as changes in the general economic climate;
• and any other factors identified in this brochure.
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When it comes to coatings and printing inks, Walsroder Nitrocellulose is the prime choice. These tried-and-tested products are finest brand quality with characteristics that are second-to-none. Safe, reliable and economically efficient.

Walsroder Nitrocellulose stands for quality from product development all along the line to the finished product. Our in-house test laboratories monitor the production process 24/7. This total quality management gives our customers additional security. More than other suppliers we are committed to close collaboration, supporting our customers by providing training and knowledgeable advice on all issues relating to using and handling nitrocellulose. Our focus is always on customer benefits.
Product Range

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