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## Applications of NIR Spectroscopy for Fast Quantitative Analysis in the Explosives Industry

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## Summary

The modern explosives industry is facing numerous analytical challenges, including the need for very precise analysis of non-explosive raw materials, time-critical in-process testing of intermediate components, and extensive analysis of final explosive products. Traditionally, volumetric, gravimetric and chromatographic techniques have been employed for these tasks, despite being time-consuming and relatively expensive. Adoption of fast and more economical spectroscopic and spectrometric methods (e.g. infrared IR, near-infrared NIR and ultraviolet/visible UV/VIS) was slow due to the perception of inadequate specificity and accuracy.

However, recent improvements to both spectrometer hardware and chemometric data analysis software have enabled NIR methods to overcome these hurdles, with the added benefit of little or no sample preparation. This paper discusses applications of NIR technology in explosives industry, including measurements relevant to nitrocellulose and nitroglycerine production (i.e., analysis of sulphuric acid/nitric acid/nitrous acid/water/nitroglycerine contents in nitrating acids, and of nitrogen/water/solvent contents in nitrocellulose), as well as the analysis of nitroglycerine and oligomeric

plasticizer content in gun propellants. The speed and reproducibility of NIR methods can be used toward process control and increased process safety, in addition to cost-savings. Future projects include (a) the transfer of NIR laboratory methods to direct, in-process analysis in the nitrocellulose production plant, and (b) the evaluation of NIR applications to replace additional conventional analytical methods.

## 1. Nitrocellulose Production Process

### 1.1 Overview of Applications

Nitrocellulose (NC) is produced by a reaction of cellulose (e.g. cotton linters or wood pulp) with nitrating acid (i.e. a mixture of nitric and sulphuric acids). The reaction step is followed by a stabilisation phase, including boiling and refining, then blending of multiple batches. Finally, NC is extracted from the blend NC pulp by centrifugal action. For some NC grades, residual water in the NC fibres is replaced by a solvent (usually ethyl alcohol) in a pusher centrifuge. The NC production process requires extensive analysis of raw materials, NC batches and final NC blends properties.

Fig. 1 shows a simplified flowchart of the NC produc-

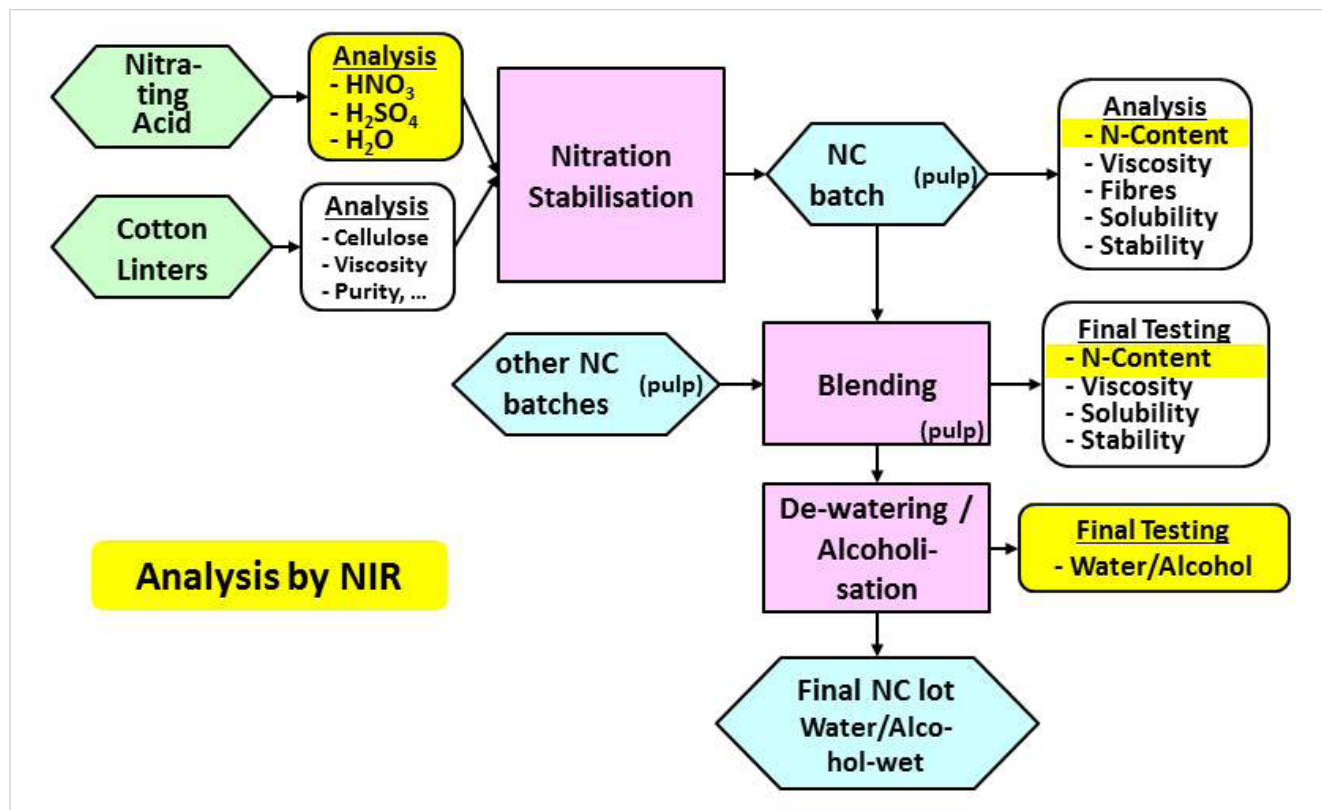


Fig. 1: Flowchart of nitrocellulose production process including testing.

tion process, including a testing scheme. Tests that may be performed using NIR technology are highlighted in yellow.

## 1.2 Nitrating acids

The degree of nitration determines the solubility and energetic content of the final NC product, and is dependent on the composition of the nitrating acids used in production. Specifications for total nitrogen content are in the range of 0.1 to 0.2%, yet an increase in water content by as little as 0.3% will reduce nitrogen content in the final NC by 0.1%. Thus, accurate and reproducible measurement of the nitrating acid content is essential. Furthermore, in accordance with the principles of Total Quality Management and Measurement System Analysis, the analytical error should not exceed 30% of the specified tolerance range (i.e. precision/tolerance-ratio  $\leq 0.3$ ); therefore, the reproducibility of nitrating acid analysis needs to be within 0.1 to 0.2% water content. This is a challenging task using conventional methods.

Traditional nitrating acid analysis is based on (a) duplicate measurements of total acid content by potentiometric acid-base titration, (b) duplicate measurements of sulphuric acid content by evaporation of nitric acid in a sand bath, followed by potentiometric acid-base titration, (c) calculation of nitric acid from the above results and then calculation of water content as difference to 100%. This method is time-consuming (approx. 2 hours) and prone to experimental errors. Pre-



Fig. 2: Acid analysis set-up with all-quartz transmission probe and sample.

cision in water content determinations may be on the order of 0.1 % in a best-case scenario, but more likely in the range of 0.2 to 0.3%.

Alternatively, a redox titration (with ferrous ammonium sulphate in concentrated sulphuric acid) may be used to determine the nitric acid content, in addition to the measurement of total acid. While this method is faster, it requires additional titration equipment and is still prone to imprecision due to experimental error.

Faster and more direct analytical techniques for nitrating acid analysis are desired. Attempts to provide simultaneous determination of both sulphuric and nitric acid content by either non-aqueous titration or by ion chromatography could not meet the precision requirements. Furthermore, direct Karl-Fischer titration of nitrating acids produces systematic errors of up to 6% for water values due to the esterification of the Karl-Fischer solvent with sulphuric acid for which water is a by-product.

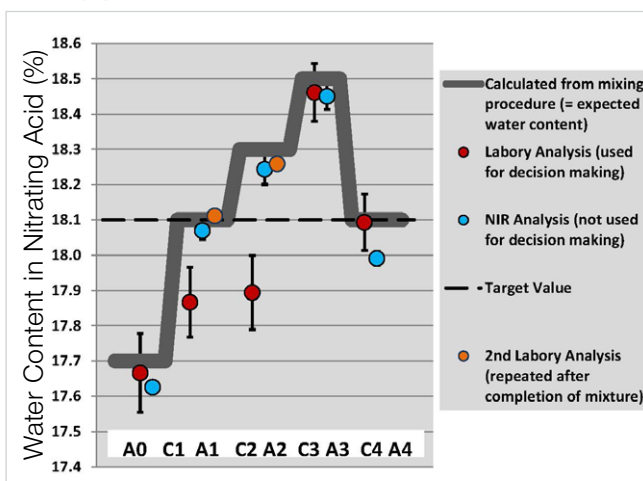


Fig. 3: Real example showing superiority of NIR method to assist preparation of a nitrating acid with target value 18.1 % water. Points along the horizontal axis are preceded by an "A" if they represent measurement points in the analysis, and "C" if they represent points at which additional components were added to achieve the desired water content.

In this example, the laboratory analysis (classic titration) of the pre-mixed acid (A0) indicated a water content of only 17.7%. In response, 0.4% water was added (point C1) to reach the target value. However, laboratory analysis revealed that this corrected sample (A1) only yielded 17.9% water. Based on the measured A1 value, additional water was added (point C2) to reach the 18.1% target. Despite this second step to correct the water content, the laboratory analysis still measured 17.9% water content. A third attempt to reach 18.1% water (point C3) resulted in a laboratory analysis value of 18.5% water. From there, oleum and pure nitric acid was added (point C4) to reduce the water content and achieve the target value, which was verified by titration (point A4). A repeat analysis by a different operator revealed that the initial laboratory values for analyses A1 and A2 were incorrect, resulting in additional and unnecessary correction steps, despite the fact that the target water content was actually achieved after the first correction (C1). However, the NIR analysis which was done in parallel with the classical titration was found to be both more accurate and precise. Using NIR, re-processing and re-testing of the nitrating acids could have been avoided.

A NIR method for fast and simultaneous analysis of all three components in nitrating acid has been developed. The method is based on measurement of ac-

ids using a BUCHI NIRFlex N-500 SMA Cell equipped with a fibre optic Hellma all-quartz transmission probe (1 mm optical path length). A ventilated box was constructed which allowed for fast measurement of acids and then easy cleaning of the probe (Fig. 2).

Over a period of 3 months, all nitrating acids were measured by both the classic titration method (to produce a reference value) and NIR. Spectra were collected over the range of 10'000 to 4'000  $\text{cm}^{-1}$ . Partial least-squares (PLS) analysis was performed within NIRCAl chemometric software package using a reduced variable range (9'000 to 5'000  $\text{cm}^{-1}$ ). Data analysis showed that the absorption band at 6897  $\text{cm}^{-1}$  (i.e. -OH stretching 1<sup>st</sup> overtone) had the greatest contribution to the model, while the -OH band at 5155  $\text{cm}^{-1}$  was non-informative

due to saturated absorbance at that wavenumber. Initial calibration efforts (N = 54 samples) identified systematic errors which occurred sometimes with the reference method (Fig. 3). The final model (N = 205) was calculated based on nitrating acids which spanned 38 to 65% sulphuric acid and 9 to 37% water (with most samples between 10 and 18%), and approximately 25% nitric acid content. The concentration of nitric acid was calculated based on closure to 100%, but was not included in the calibration model.

The NIR calibration model for water content in nitrating acids was characterized by a regression coefficient ( $R^2$ ) of 0.999, and a standard error of calibration (SEC) and standard error of prediction (SEP) both of 0.09%, as shown in (Fig. 4).

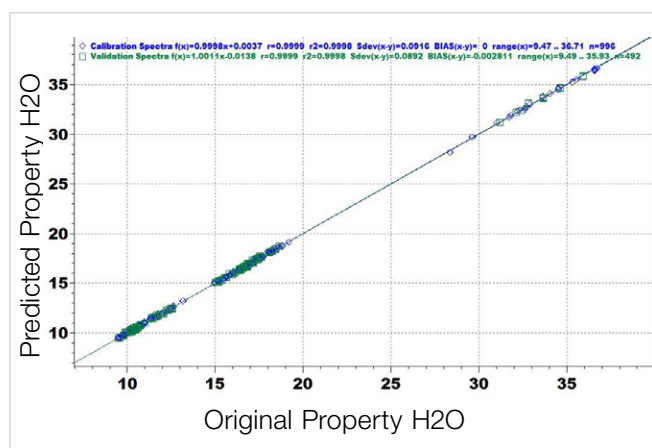


Fig. 4: Calibration for water in nitrating acids.

In practice, the reproducibility of water content measurements was 3 times better with NIR relative to the conventional method (Fig. 5). A quality control protocol consisting of at least weekly parallel analysis of several nitrating acids by NIR and classic titration method ensured constant NIR performance. The NIR acid analysis method, which has been implemented for over 9 months, has resulted in a significant reduction of workload for laboratory technicians, shorter response times, and more consistent and reproducible results, further enabling better process and product control in the nitration plant.

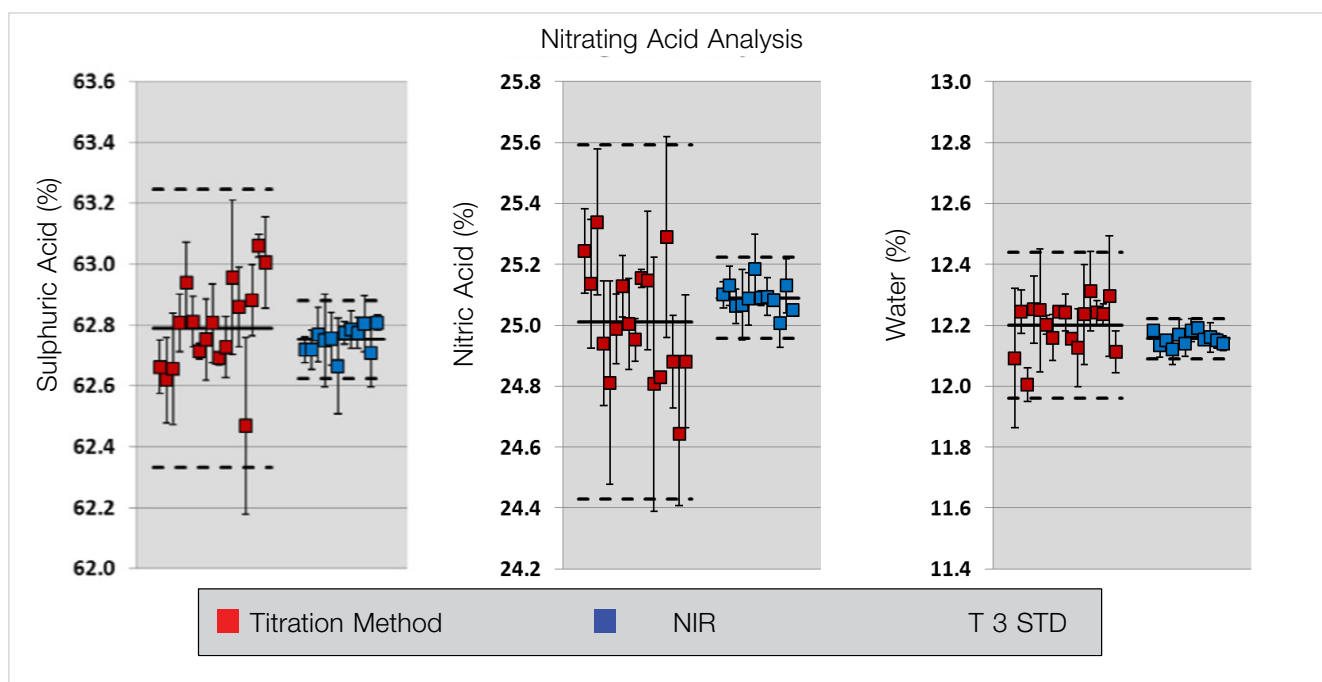


Fig. 5: Contents of sulphuric acid, nitric acid and water in nitrating acid; analysed 17 times by 8 different operators using the conventional method, and 12 times within one week by 6 different operators using NIR. Each analysis was performed in duplicate; the bars indicate three standard deviations. The markedly better reproducibility of the NIR method is evident.



### 1.3 Nitrogen Content of Nitrocellulose

In the case of NC, the degree of nitrogen substitution at hydroxyl groups of cellulose is indicated directly by nitrogen content (in relation to dry substances). Military and industrial applications require NC with nitrogen content ranging from 10.2 to 13.6%, equivalent to 72 to 96% hydroxyl group substitution. As nitrogen content rises, so too does the energy content of the NC.

Nitrogen content is an intrinsic property of the NC and can be determined in different ways. According to NATO standard STANAG 4178 Ed. 2, the ferrous ion titration methods (ferrous sulphate FS or ferrous ammonium sulphate FAS) are preferred and should be employed as reference methods, but nitrogen analyzer, Devarda's alloy method, Schulze-Tiemann, nitrometer, as well as the indirect combustion calorimeter methods are also allowed.

Unfortunately, all of these methods are complex, time-consuming, expensive and error-prone (at least at the required accuracy/precision level of below 0.02% absolute or 0.16% RSD). In fact, significant variation in obtained nitrogen content values often appear (i) between different methods, (ii) between different laboratories, and (iii) even with reference FAS method when the experimental conditions are varied within the range allowed by the standardized procedure.



Fig. 7: Sample preparation: approx. 10 g of dried NC is filled into the BUCHI NIRFlex-Solids-Cell; the NC is then compressed by inserting a plunger (approx. 1.5 kg).

NIR spectroscopy is an alternative method for NC nitrogen content determination, as the spectrum changes significantly with degree of hydroxyl group substitution (Fig. 6).

The suitability of NIR was investigated using a BUCHI NIRFlex N-500 NIR spectrometer. Preliminary tests included the measurement of dry NC with a NIRFlex Solids Cell using the high performance sample cup (Fig. 7) as well as recording the spectra of NC solutions (2% NC in acetone) with a fibre optic transmission probe. Measurement of dry NC was found to be much easier and yielded better results.

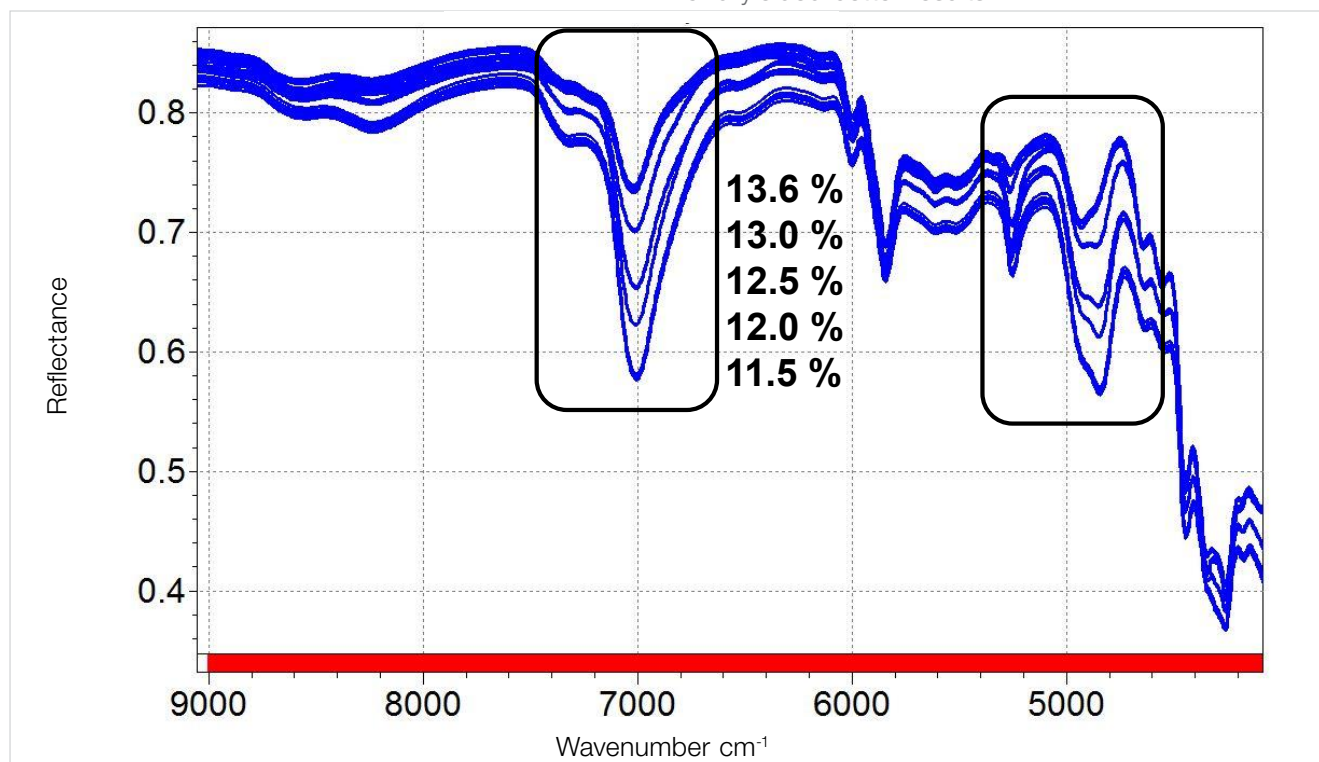


Fig. 6: NIR spectra of different NC grades. In particular the absorption bands between 4'500 and 5'300  $\text{cm}^{-1}$  (R-OH combination bands, -C-O- stretching 2<sup>nd</sup> overtone) as well as around 7'000  $\text{cm}^{-1}$  (R-OH 1<sup>st</sup> overtone) change when the hydroxide group in the cellulose is replaced with nitrate groups. While nitrate bands may also contribute to the spectrum, e.g. -O-NO<sub>2</sub> 2<sup>nd</sup> overtone between 4'000 and 4'149  $\text{cm}^{-1}$  and 3<sup>rd</sup> overtone between 6'667 and 6'944  $\text{cm}^{-1}$ , these absorptions are weak.

A NIR calibration model based on 251 different NC samples was then established, where nitrogen content ranged from 11.4 to 13.6%. Of the 251 unique NC samples, 113 were prepared at different water content levels (0.2 to 1.0%) in order to eliminate the influence of moisture on nitrogen content measurements. The main obstacle to NIR calibration was the definition of reference nitrogen content values of calibration samples due to the uncertainty of the conventional methods (as discussed above) and the absence of reference NC material with certified nitrogen content.

Most calibration samples originated from recent production and were analysed by FAS titration (N = 83) or combustion calorimeter (N = 154).

Using only the FAS samples gave a significantly better model (initial models including 61 FAS samples had an SEC and SEP of 0.009 and 0.010%, respectively); however, inclusion of the less accurate combustion calorimeter samples turned out to be necessary in order to obtain a more robust model, where the FAS method was inconsistent for two grades of NC. An additional set of 14 NC samples produced in-house and by other suppliers were added to the calibration, with reference analysis provided by FAS titration, Devarda's alloy method, Schulze-Tiemann and nitrogen analyzer (BUCHI DuMaster); these samples turned out to be essential in order to further improve and verify robustness of the model. Quality parameters of the final NIR model for nitrogen content in NC included an  $R^2$  of 0.9996, and SEC and SEP of 0.017, and 0.018%, respectively (Fig. 8). It was then demonstrated that this NIR method is capable of reliably determining nitrogen content

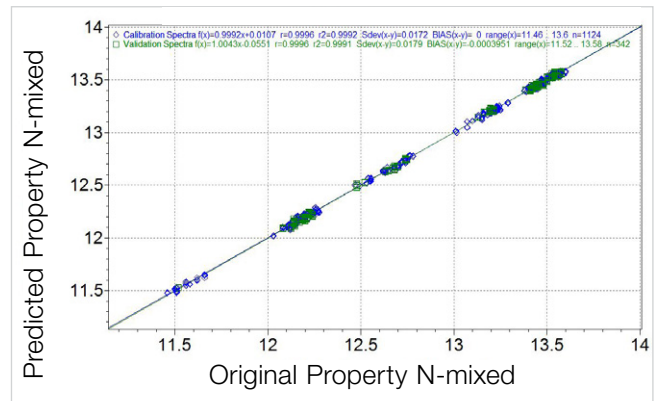


Fig. 8: Calibration for nitrogen content in nitrocellulose.

values also for new NC samples within the calibrated range. An independent validation set of 8 samples with corresponding reference values produced deviations below 0.03%.

Main advantages of the NIR method are:

- Much shorter analysis time (10 minutes for duplicate analysis as compared to at least 2 hours with most of the other methods) and thus markedly reduced cost.
- Larger sample mass (approx. 10 g as compared to 0.2 to 1 g with most of the other methods) provides a more representative sample.
- High precision ( $s_{z_i}$ =0.007% absolute or 0.06% RSD; laboratory precision by measuring the same sample by different persons 65 times over a period of several months; Fig. 9); this is only slightly inferior to the FAS method ( $s_{z_i}$ =0.05% RSD) but considerably better than for nitrogen analyzer, Schulze-Tiemann or combustion calorimetry, all of which give  $s_{z_i}$ -values of ap-

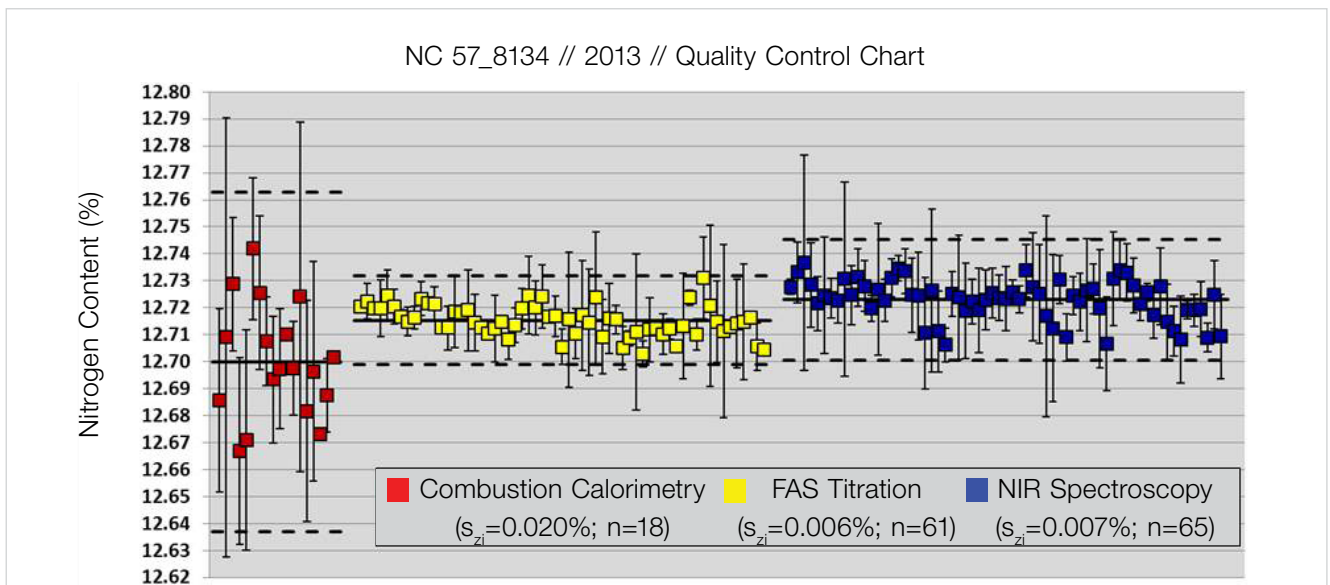


Fig. 9: Quality control chart of regularly measured NC sample. It is evident that laboratory precision  $s_{z_i}$  of the new routine method NIR is almost as good as for the FAS reference method and by a factor of 3 better than for the previous routine method combustion calorimeter.

prox. 0.15% RSD. According to STANAG 4178 Ed. 2, a repeatability standard deviation  $s_r$  of 0.08% RSD is to be regarded as excellent, of 0.12% RSD as acceptable, and of 0.16% RSD as marginal.

- Unlike conventional methods, the NIR method accuracy is unaffected by changing moisture levels, so long as they fall within calibrated moisture-range.
- Only one short drying step at 60°C is required in order to prepare the NC sample for NIR analysis. All other methods require a second drying step at 100°C; however, the very dry NC will quickly take up moisture, particularly in humid conditions, which will adversely affect the test result unless moisture uptake is analysed and corrected.
- Superior robustness of the NIR method relative to conventional methods, with insensitivity to small variations in test procedures and loss of NC fibres or presence of agglomerates.
- No reagents, waste-management or pollution.

Despite its advantages, the NIR is an indirect method; thus, it requires a reference analysis for calibration development and validation, the problems of which have been described. Also, it must be noted that the physical characteristics, such as fibre texture or surface morphology of the NC may influence the resulting NIR spectrum. Therefore, it is important to include these sources of variability within the calibration model, so as not to affect the NIR-measured nitrogen content.

Several quality control steps were implemented in order to verify NIR method performance.

- Prior to each series of measurements, a known (reference) NC sample was measured; the nitrogen content of this sample was monitored by means of a quality chart.
- Weekly, at least 2 NC samples from production were subjected to parallel analysis by NIR and a primary method (here FAS titration).
- Each NC type (i.e. those originating from a new grade or lot of cellulose, or for which there was an alteration in the production process) was analysed by both NIR and the primary method; representative samples from these types were added to the NIR model before the model was used for routine analysis.

Introduction of the NIR method significantly reduced the laboratory technician workload and response times. Additionally, due to the improved reproducibility of nitrogen measurements, confidence in the results was increased and unnecessary retesting could be avoided.

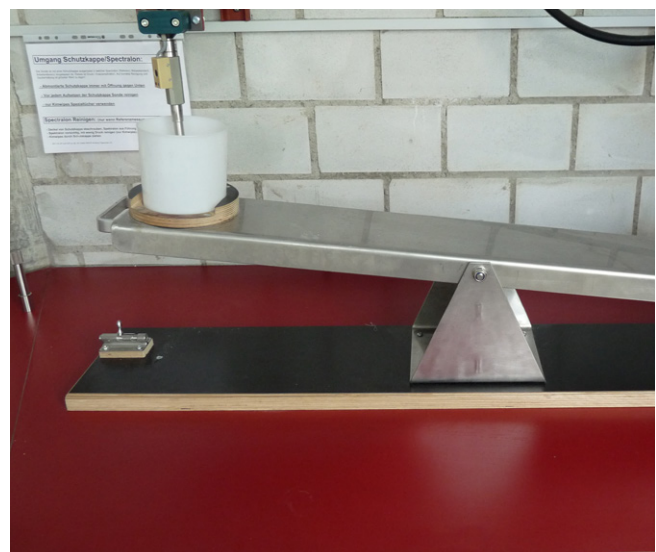
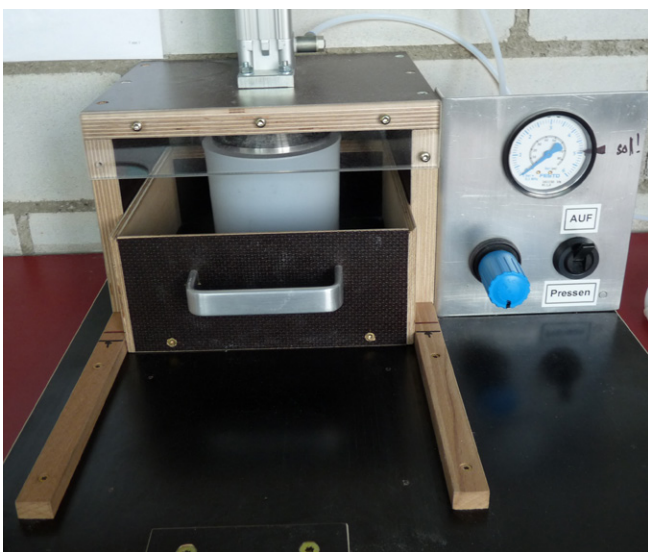


Fig. 10.1 / 10.2 Pneumatic press for preparation of NC sample for testing, and equipment to ensure that sample is pressed to the probe with constant force.



#### 1.4 Water and Alcohol Content in Wet Nitrocellulose

For safety purposes, NC needs to be stored and shipped wet with either water or a mixture of alcohol and water used as the liquid. This may be accomplished by processing the NC slurry through a pusher centrifuge. Depending on the application, total volatile content of between 20 and 35% is usually required.

In water-wet NC, water content can easily be analysed using a moisture analyser. In NC wetted with a mixture of alcohol and water, both solvents need to be assessed. Traditionally, this is performed by analysis of water content by Karl-Fischer titration in combination with gravimetric determination of total volatile content, where NC samples are held at 60°C and measured until a constant weight is maintained. This procedure requires more than 5 hours, and test results are obtained well after an entire lot of NC was processed and packed. If analysis reveals that alcohol or water contents are outside specifications, the entire lot has to be unpacked and reprocessed. Moreover, several dozen samples must be analysed for each batch, which is very time-consuming with the conventional analysis.

An NIR method for fast determination of the contents of water, alcohol and total volatiles in NC was developed in 2008. This method is also incorporated in NATO standard STANAG 4178 Ed. 2. The samples were prepared by pressing the voluminous NC using an air driven pneumatic press. The NIR measurement was performed using a BUCHI NIRFlex N-400 equipped with a fibre optic reflectance probe. In order to obtain reproducible results, it was ensured that the fibre optic cable did not move and that the sample was pressed to the probe with constant force (Fig. 10.1 / 10.2).

Over a period of 10 months, NIR data of NC samples were collected together with classic Karl-Fischer titration and gravimetric methods ("reference values"). NIR spectra were recorded in the range of 10'000 to 4'000  $\text{cm}^{-1}$ .

Following NIR model development, the spectroscopic method was implemented for routine use; only 3 samples per batch were checked by classical methods, and the model was updated annually. The quality control protocol requires that, for any batch, the difference between the mean values obtained by NIR and classic method do not exceed 0.3% in water content and 0.5 % in total volatiles (Fig. 11). The actual model is based on 1200 samples in the concentration range of 2.0 to

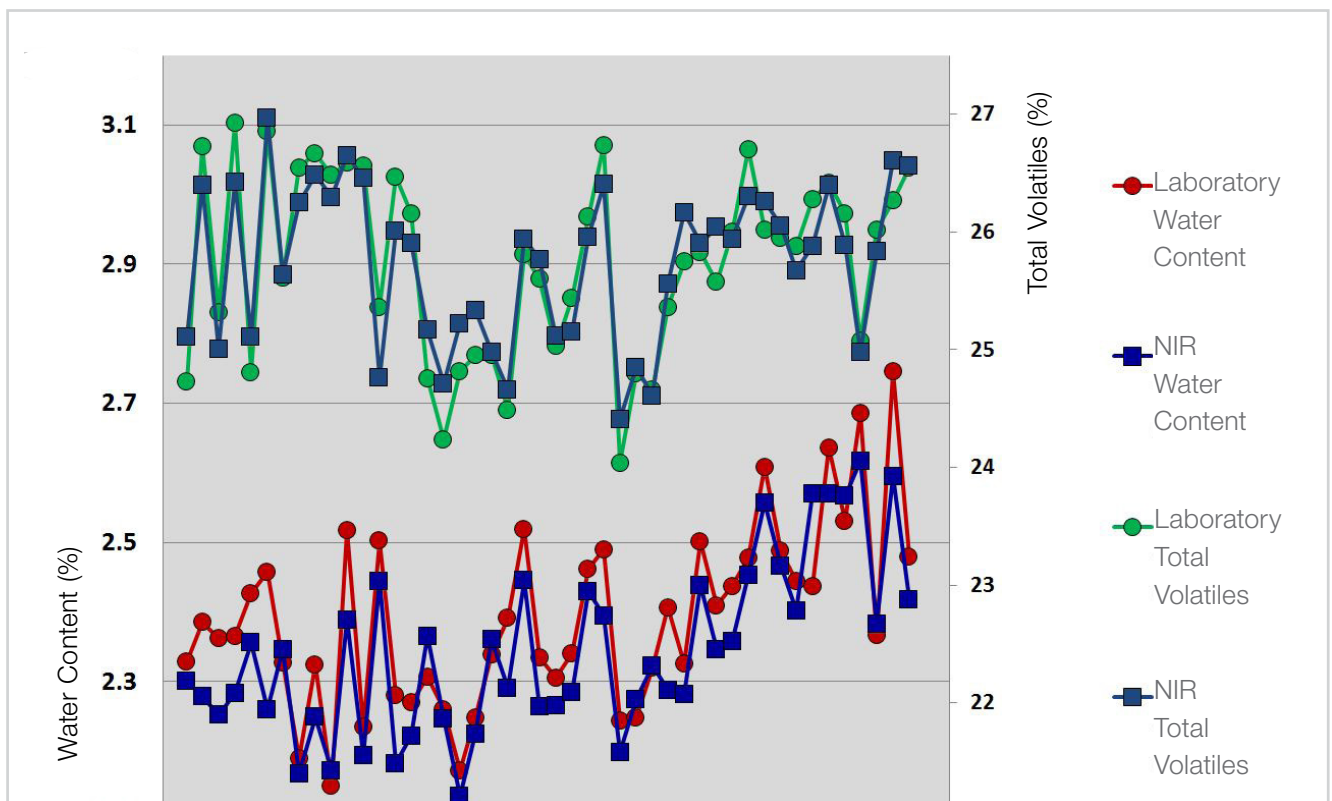


Fig. 11: Comparison of results obtained with NIR and conventional laboratory methods. Mean values generated from the analysis of 46 NC batches analysed between December 2010 and August 2011. Differences between NIR and laboratory methods are generally below 0.3% for water content and below 0.5% for total volatiles.



4.0% water and 23 to 29% total volatiles. The obtained quality parameters were, for water content:  $R^2$  0.92, SEC 0.11%, SEP 0.11%; and for total volatiles:  $R^2$  0.88, SEC 0.47%, SEP 0.51%.

The NIR method for analysis of water, alcohol and total volatiles has been successfully implemented for over 6 years. Testing can easily be performed by production staff; this in contrast to the classic analysis, which required qualified laboratory personnel.

Moreover, the potential for much shorter response times enables immediate action if the product parameters approach or exceed the specified limits, in addition to reducing the workload of laboratory personnel.

## 2. Nitroglycerine Production Process

In the production of nitroglycerine and other similar liquid nitrate esters such as diethyleneglycol dinitrate, the raw material (glycerine or other poly-alcohol) is first nitrated by nitrating acids, followed by stabilisation (neutralising/washing). While there are similarities to NC production, the acids analysis is much more critical to the production of nitroglycerine. Spent acids (production by-products) contain up to 10% nitrate esters; the stability of the acids is dependent on the ratio of acids, water and nitrate ester content. Instable spent acids risk explosion within hours, and must be pre-stabilised by the addition of sulphuric acid until the composition is outside of the critical range. Spent acids are then stabilised by destroying nitrate esters at elevated temperature.

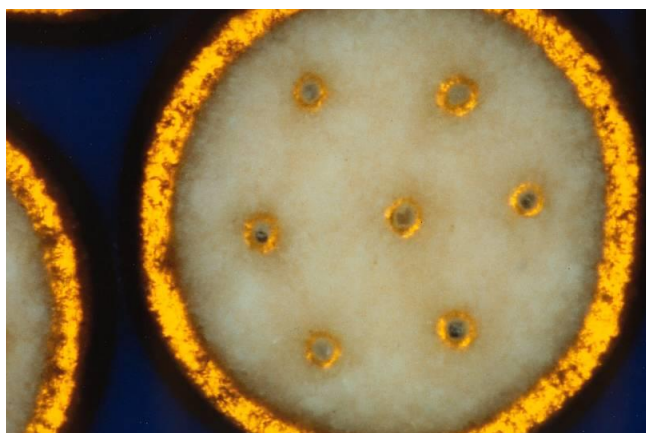


Fig. 12: Microscopic photograph of cross-section of cylindrical propellant grain with 7 perforations. Main body of grains consists mainly of NC (greyish colour). The grains are surface coated with nitroglycerine and oligomer plasticizer, forming layers that are visible as yellow rings at the outside and around the perforations of the grains.

These stabilised acids contain up to 10% nitrous acid, which must be removed before the waste acid can be recycled by distillation to pure sulphuric and nitric acid. NIR analysis of such acids is performed as was described in section 1.2 (NC nitrating acids), using an extended calibration that (a) accounts for the differing concentration ranges and (b) includes nitrous acid as well as the respective nitrate ester (nitroglycerine or diethyleneglycol dinitrate).

## 3. Quantitative Analysis of Gun Propellants

Gun propellants usually are granules consisting of nitrocellulose and several organic and inorganic additives (Fig. 12). Propellants are used in ammunition where, after ignition, they burn in a controlled way, producing large amounts of combustion gases capable of accelerating the projectile in the gun.

Organic components in propellants include stabilisers (e.g. diphenylamine), plasticizers (often monomeric or oligomer phthalate/adipate esters) and energetic components (e.g. nitroglycerine).

Analysis of these organic components is usually performed by either extraction or digestion of the sample, followed by gas chromatography (GC) or liquid chromatography (HPLC).

Although these testing methods are straightforward for most organic components, they are not well-suited for oligomer plasticizers. Such plasticizers consist of a large number of very similar but nevertheless different compounds with molecular mass between 1'000 and 20'000 D (which is too high for GC analysis). HPLC analysis of these oligomers usually gives something between a single, very broad peak and numerous peaks which overlap with one or several of the other organic compounds that are also present in the sample. Whereas oligomer phthalate esters can be easily monitored using standard UV HPLC detectors, adipate esters give very weak UV signals.

The only practical chromatographic analysis for such oligomers is based on HPLC with an evaporative light scattering ELS detector. This detector is much more sensitive to, and has higher specificity for oligomers than to the other monomeric compounds (which allows oligomer content to be assessed, even if the HPLC method does not properly separate the oligomer from the other compounds).

However, the HPLC/ELS method suffers from poor linearity of ELS detector (lead to reduced accuracy), considerable consumption of both tetrahydrofuran solvent and nitrogen gas to run the detector, and the long time required to equilibrate and run the system, which consequently increases both the cost and workload associated with the analysis. These factors were drivers for an alternative method for determination of oligomer plasticizers in propellant.

Since oligomer plasticizers are used in a specific propellant which contains no other carbonyl-containing compounds, Fourier Transform Infrared FTIR or NIR spectroscopy are potential options. A preliminary study demonstrated the potential to analyse oligomer plasticizers in the presence of organic additives by FTIR spectroscopy. These analyses, carried out using either a KBr transmission cell or attenuated total reflection ATR cell, identified that the strong and specific carbonyl absorption at  $1740\text{ cm}^{-1}$  could be used to calibrate for plasticizer content, and at significant cost and time savings relative to the HPLC/ELS method, with comparable precision and accuracy.

Analysis with NIR delivered similar results with even more time- and cost-savings, and with more convenient sampling. The NIR method uses the same sample preparation of the conventional method.

First, the organic additives were extracted from the propellant grains (approx. 5 g) using dichloromethane. The residual remaining after evaporation of the dichloromethane was dissolved in solvent (typically 10 mL). Dichloroethane was the preferred solvent, as its spectrum has less interference with that of the plasticizer than tetrahydrofuran, and less toxicity than the other alternative solvent, trichloromethane. The solution was measured using a BUCHI NIRFlex N-500 SMA Cell equipped with a fibre optic Hellma all-quartz transmission probe with 1 mm optical path length. The calibration was performed using NIRCAl chemometric software.

The spectral regions between  $6'000 - 4'000\text{ cm}^{-1}$ , including  $5'100\text{ cm}^{-1}$  and  $5'300\text{ cm}^{-1}$  ( $\text{C}=\text{O}$  2<sup>nd</sup> overtone) contribute most to the model. The obtained quality parameters for oligomer plasticizer content determination in the concentration range of 0 to 5% including an  $R^2$  of 0.97, and an SEC and SEP of 0.12%.

Excluding sample preparation, the total time associated with the NIR analysis, including the measurement, data analysis, and cleaning the probe was approximately 10 minutes, compared to approximately 30 minutes for FTIR and several hours for HPLC/ELS. The precision and accuracy of the NIR method were found to be around 4% RSD; this is not excellent, but sufficient for this application and in the same range as with FTIR or HPLC/ELS (Fig 13).

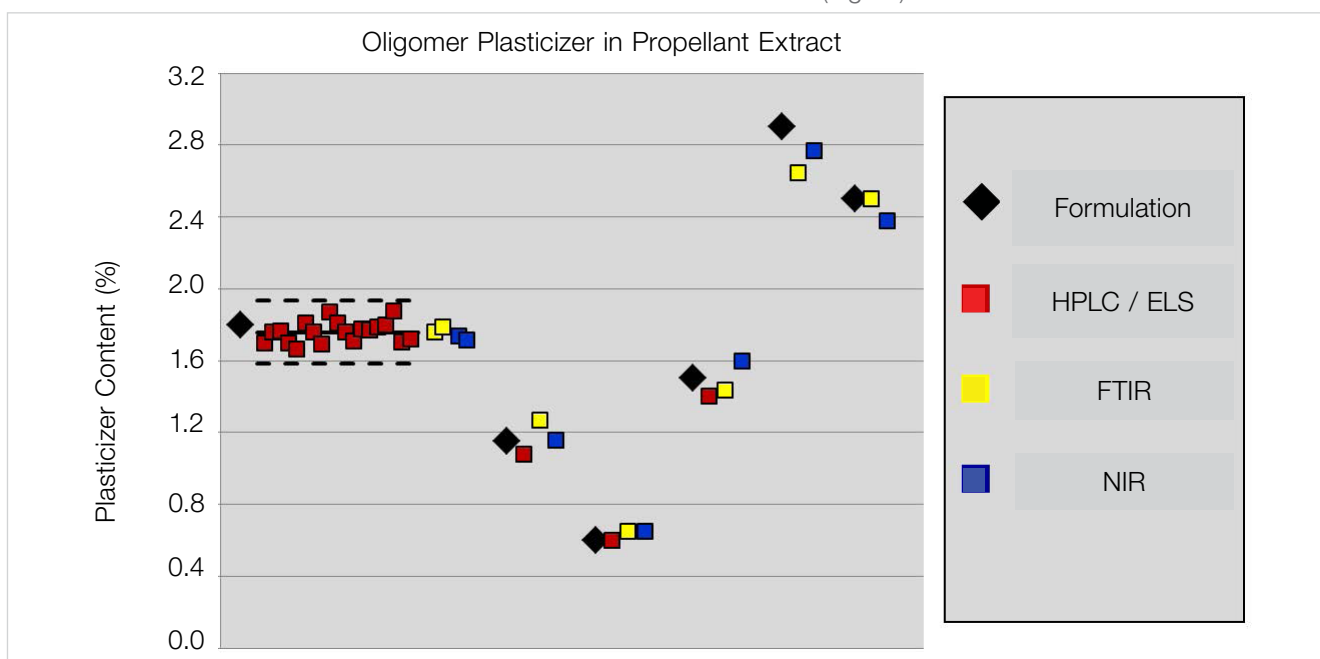


Fig. 13: Content of oligomer plasticizer from propellant formulation (calculated value) and from analysis by HPLC/ELS, FTIR and NIR spectroscopy for reference propellant (including quality control chart of previous HPLC/ELS results) and for 5 other propellants. All results are plausible.

The relatively high RSD of the method was attributed mainly to the small concentration of oligomer in the solution used for NIR (and FTIR) analysis, combined with potential errors related to sample preparation. While measuring the much more concentrated evaporation residue directly, (e.g. in a Hellma reflexion cuvette) might improve precision, it was considered too dangerous. The residue may contain up to 80% pure nitroglycerine, which could detonate if friction is applied whilst preparing the cuvette.

Introduction of the NIR method for oligomer plasticizer analysis in propellant avoided the otherwise necessary replacement of the old broken HPLC/ELS system, reduced consumption of solvents for this analysis by more than a factor of 2, and minimised both analysis time and workload. It should be noted that other organic components of the propellant formulation, such as nitroglycerine and stabilisers, can also be analysed with the NIR method.

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