NATO STANDARDIZATION AGENCY
AGENCE OTAN DE NORMALISATION

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See CNAD AC/326 STANAG distribution

STANAG 4583 (EDITION 1) –CHEMICAL TEST PROCEDURES AND REQUIREMENTS FOR n-BUTYL 2-NITRATOETHYL NITRAMINE (n-Butyl NENA)

Reference: PFP(AC/326)D(2007)0007 dated 18 June 2007

1. The enclosed NATO Standardization Agreement, which has been ratified by nations as reflected in the NATO Standardization Document Database (NSDD), is promulgated herewith.

2. The reference listed above is to be destroyed in accordance with local document destruction procedures.

ACTION BY NATIONAL STAFFS

3. National staffs are requested to examine their ratification status of the STANAG and, if they have not already done so, advise the Defence Investment Division through their national delegation as appropriate of their intention regarding its ratification and implementation.

Cihangir AKSIT, TUR Civ
Director, NATO Standardization Agency

Enclosure:
STANAG 4583 (Edition 1)

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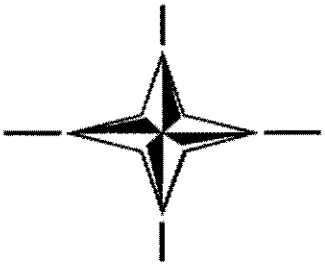
STANAG 4583
(Edition 1)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)



NATO STANDARDIZATION AGENCY
(NSA)

STANDARDIZATION AGREEMENT
(STANAG)

SUBJECT: CHEMICAL TEST PROCEDURES AND REQUIREMENTS FOR n-BUTYL 2-NITRATOETHYL NITRAMINE (n-Butyl NENA)

Promulgated on 3 August 2011

Cihangir AKSIT, TUR Civ
Director, NATO Standardization Agency

STANAG 4583
(Edition 1)

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This STANAG is promulgated by the Director NATO Standardization Agency under the authority vested in him by the NATO Standardization Organisation Charter.

2. No departure may be made from the agreement without informing the tasking authority in the form of a reservation. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.

3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

4. Ratification, implementation and reservation details are available on request or through the NSA websites (internet http://nsa.nato.int; NATO Secure WAN http://nsa.hq.nato.int).

FEEDBACK

5. Any comments concerning this publication should be directed to NATO/NSA – Bvd Leopold III - 1110 Brussels - BE.

STANAG 4583
(Edition 1)

NATO STANDARDIZATION AGREEMENT

(STANAG)

CHEMICAL TEST PROCEDURES AND REQUIREMENTS FOR n-BUTYL 2-NITRATOETHYL NITRAMINE (n-Butyl NENA).

Annexes:

A. Requirements for n-butyl 2-nitratoethyl nitramine (Bu-NENA)

B. Test procedures

Related documents:

STANAG 4582 EXPLOSIVES, NITROCELLULOSE BASED PROPELLANTS, STABILITY TEST PROCEDURE AND REQUIREMENTS USING HEAT FLOW CALORIMETRY

AIM

1. The aim of this agreement is to establish common chemical requirements, and test procedures for *n-Butyl 2-nitratoethyl nitramine (Bu-NENA)*.

2. This agreement is intended for use by the NATO Participating Nations.

AGREEMENT

3. Participating Nations agree to adopt the chemical requirements described in Annex A and the test procedures described in Annex B for Bu-NENA.

IMPLEMENTATION OF THE AGREEMENT

4. This STANAG will be considered implemented when a nation has issued the necessary orders and instruction putting the contents of this agreement into effect.

WARNING

5. This STANAG calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves the user from the statutory obligations relating to health and safety at any stage during use. Especially N-NO-MNA, the depletion product of MNA is highly carcinogenic. Refer to the information given in the safety data sheet and national regulations for each of the components used throughout this STANAG.

ANNEX A TO
STANAG 4583
(Edition 1)

Requirements for n-Butyl 2-nitratoethyl nitramine (Bu-NENA)

Parameters	(1) Quality requirements				
	Type 1	**Type 2**	**Type 3**	**Type 4**	See Para
Bu-NENA	Min 97 %	Min 91 %	Min 97 %	Min 91 %	3
Bu-NENA + Bu-AENA *	Min 99 %	Min 98 %	Min 99 %	Min 98 %	3
Stabilizer** : N-methylparanitroaniline content (MNA content)	Min 0.4 %, Max 0.6 %	Min 0.4 %, Max 0.6 %	Unstabilized	Unstabilized	3
Hydroxyl content	Max 10 μeq/g	Max 50 μeq/g	Max 10 μeq/g	Max 50 μeq/g	7
Acidity (as H_2SO_4)	Max 0.025 %				1
Alkalinity (as Na_2CO_3)	Max 0.025 %				1
Water ***	Max 0.05 %				2
Depletion product (Bu-ENA)	Max 0.10 %				3
Thermal stability at 82.2°C (Abel Test) ****	Min 4 minutes				4
Chemical stability **** (Microcalorimetry)	Max 30 J/g within 10.6 d at 80°C (or equivalent temp/time scheme, see STANAG 4582)				5
Chemical stability **** (Bu-ENA formation)	Max 0.15 %/day at 90°C				6

* Impurities can vary with synthesis route, Bu-AENA is a common impurity with some routes, and is acceptable at the level specified. Bu-ENA is another impurity and the main depletion product of Bu-NENA. For other impurities acceptable levels have to be established.

** Other stabilizers and in different contents may be used as agreed between producer and customer provided sufficient information on their use and qualification of the stabiliser and the stabilized material is documented. N-methylparanitroaniline (MNA) has been used, ethyl centralite (EC), Akardite II (AKA) and 2-nitrodiphenylamine (2-NDPA) are possible candidates.

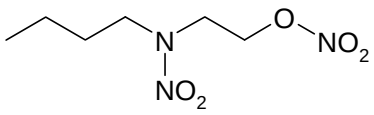
*** Bu-NENA is hygroscopic and other values for water content can be agreed upon.

**** Two out of the three methods for thermal stability have to be performed. (to be mutually agreed upon between customer and producer)

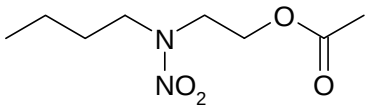
Abbreviations

Abbreviation	
Bu-NENA	n-**Bu**tyl 2-**N**itrato**e**thyl **N**itr**a**mine
Bu-AENA	2-(butylnitroamino) ethyl acetate (or Butyl Acetyl Ethyl Nitramine)
Bu-ENA	n-Butyl 2-hydroxyethyl nitramine
MNA	N-methylparanitroaniline

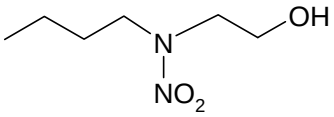
Structural formulae of Bu-NENA, Bu-AENA and Bu-ENA :



Bu-NENA



Bu-AENA



Bu-ENA

ANNEX B TO
STANAG 4583
(Edition 1)

CONTENTS OF ANNEX B

1. Acidity, alkalinity *(as according to contents above)*

Titration method

1.1. Principle

Bu-NENA is dissolved in acetone and titrated with alcoholic potassium hydroxide (KOH) with bromocresol green as indicator until the solution develops a green colour.

1.2. Materials

- Acetone (Reagent grade)
- Potassium hydroxide (KOH), 0.01N dissolved in methanol. Standardize before use against reagent grade potassium hydrogen phthalate using phenolphthalein indicator. This solution should not be stored for more than 14 days without restandardization.
- Hydrochloric acid (HCl), 0.01N dissolved in methanol, standardize before use against 0,01 N KOH
- Bromocresol green indicator (dissolved in distilled water)
- Phenolphthalein-indicator (dissolved in ethanol)

1.3. Method

Weigh approximately 10 g Bu-NENA (to nearest 0.01 g) and add 60 ml acetone. Mix well, using for example a magnetic stirrer, and add bromcresol indicator. Titrate the sample with standardized 0.01 N KOH in methanol to a visible colour change. As reference for the colour change, a reference solution containing the same concentration of coloured stabilizer in acetone can be used. Titrate a blank made by adding indicator and 0.05 g stabilizer (for Bu-NENA Type 1 and Type 2 only) to 60 ml acetone. (If the sample turns blue on adding the indicator, the sample is alkaline and should be titrated with 0.01 N HCl instead of KOH.)

1.4. Calculation

$$H_2SO_4(\%) = 4.9 * (A - B) * \frac{N}{W} \qquad Na_2CO_3(\%) = 5.3 * (A - B) * \frac{N}{W}$$

Where:A = ml used of either KOH or HCl in the titration of the sample

B = ml used of either KOH or HCl in the titration of the blank

W = weight of sample (g)

N = normality of either KOH (or HCl for alkaline samples)

2. Moisture

Karl Fischer titration method

2.1 General

Either a volumetric or coulometric method can be used. Coulometric method to be preferred

2.2. Equipment

Karl Fischer-titrator with magnetic stirrer.

2.3. Reagents

Karl Fischer reagent, methanol, HPLC quality *(for volumetric KF)*

Karl Fischer reagent for coulometric KF

Methanol, p.a. or absolute grade

2.4. Method

2.4.1 Volumetric KF

At least weekly, or when refilling the reservoir, standardize with a known amount of deionised water added under the surface of the conditioned solvent; repeat the test until checks agree within 0.03 mg/ml. Fill the sample cup with about 50 ml methanol and titrate to equilibrium. Use a disposable syringe to extract about 7 ml Bu-NENA. Weigh the syringe, and add the sample to the neutralized solution. Weigh the empty syringe, and calculate the sample weight. The sample is titrated until equilibrium is reached. The water content is calculated from the volume of reagent used and the water equivalent (WE) of the reagent:

2.4.2 Calculation

$$\% H_2O = \frac{vol * WE}{10 * M}$$

Where: vol = volume of KF reagent used in ml

WE = The water equivalent of the reagent in mg H_2O/ml

M = Weight of sample in g

2.4.3 Coulometric KF

Validation of the instrument should be performed regularly, according to the manufacturer's recommendations. Condition the instrument and add the sample (about 0.1 g) using a pre-weighed syringe. Enter the sample weight and note result. Repeat the procedure for at least three replicate samples. Report the average value.

3. Purity

Analysis by liquid chromatography

3.1. Principle

The content of n-Bu-NENA, Bu-AENA, Bu-ENA and stabilizer (if added) is determined by using HPLC.

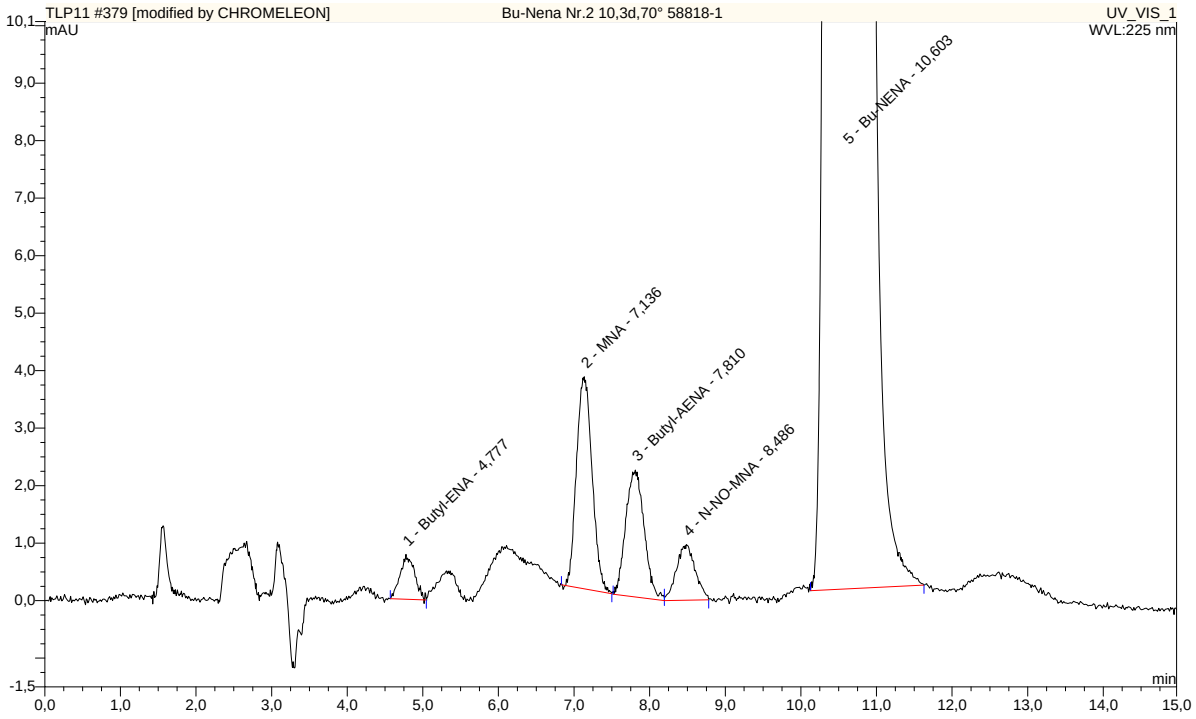


FIGURE A: Typical chromatogram of aged Bu-NENA stabilized with MNA, showing the peaks of Bu-ENA, MNA, Bu-AENA, N-NO-MNA and Bu-NENA.

3.2. Equipment

These parameters are given for information only and show one possible instrument set-up which have proven to be effective for separation of Bu-NENA, Bu-AENA, Bu-ENA, MNA and N-NO-MNA:

HPLC with RP18 column	
Mobile phase:	Acetonitrile/methanol/water, 20/40/40
Detector:	UV/DAD 225 nm
Flow rate:	0.8 ml/min
Column temperature:	15°C
Injection volume:	10 µl

3.3. Reagents

Acetonitrile, HPLC-quality

Methanol, HPLC quality

Bu-NENA (purity > 99.5%)

Bu-AENA (purity > 99.5%)

Bu-ENA (purity > 99.5 %)

Stabilizers and depletion products in high purity as reference materials

3.4. Methods

3.4.1 Standard curve calibration, absolute content

The above mentioned conditions allow a separation of Bu-ENA, Bu-AENA, Bu-NENA and of MNA and N-NO-MNA. Therefore the standard solutions can contain all these ingredients together. Alternatively two different sets of standards, one containing Bu-ENA, Bu-AENA and Bu-NENA, the second containing MNA and N-NO-MNA may be prepared. If other stabilizer(s) than MNA are used it must be proven beforehand that they and their decomposition products don't match with the Bu-ENA, Bu-AENA or Bu-NENA signals in the chromatogram.

If Bu-ENA and Bu-AENA are not available as reference materials one can work using relative HPLC procedure (see 3.4.2), which however do not have to give equivalent results with standard curve calibration method. Applied HPLC procedure has to be reported with the results.

Preparation of standards:

- Bu-NENA stock solutions (3): Accurately weigh approximate 0.85 gram, 1.0 gram and 1.1 gram of Bu-NENA with precision of ± 0.1 mg into a 100 ml volumetric flask. Dissolve, bring to volume with methanol and mix well.
- Bu-AENA stock solution: Accurately weigh approximate 0.1 grams of Bu-AENA with precision of ± 0.1 mg into a 100 ml volumetric flask. Dissolve, bring to volume with methanol and mix well.
- Bu-ENA stock solution: Accurately weigh approximate 0.1 grams of Bu-ENA with precision of ± 0.1 mg into a 100 ml volumetric flask. Dissolve, bring to volume with methanol and mix well.
- MNA stock solution: Accurately weigh approximate 0.1 grams of MNA with precision of ± 0.1 mg into a 100 ml volumetric flask. Dissolve, bring to volume with methanol and mix well.
- N-NO-MNA stock solution: Accurately weigh approximate 0.1 grams of N-NO-MNA **(caution: carcinogenic material!)** with precision of ± 0.1 mg into a 100 ml volumetric flask. Dissolve, bring to volume with methanol and mix well.

Prepare three working standards for Bu-NENA, Bu-ENA and Bu-AENA each:

- Bu-NENA working standards: Pipette 2 ml of each of the Bu-NENA stock solutions into three 100-ml volumetric flasks. Bring to volume with methanol.
- Bu-AENA working standards: Pipette 1 ml, 2 ml and 3 ml of the Bu-AENA stock solution into three 100-ml volumetric flasks. Bring to volume with methanol.
- Bu-ENA working standards: Pipette 1 ml, 2 ml and 3 ml of the Bu-ENA stock solution into three 100-ml volumetric flasks. Bring to volume with methanol.
- MNA working standards: Pipette 1 ml, 2 ml and 3 ml of the MNA stock solution into three 100-ml volumetric flasks. Bring to volume with methanol.

- N-NO-MNA working standards: Pipette 1 ml, 2 ml and 3 ml of the N-NO-MNA stock solution into three 100-ml volumetric flasks. Bring to volume with methanol.

Inject each working standard in duplicate. Use the average response (area) for each standard in the calculation. This gives you a three-point calibration curve for Bu-NENA , Bu-AENA and Bu-ENA.

Sample: Weigh approximately 100-mg sample and dissolve in 100 ml methanol. Take 20.0 ml of this solution and dilute to 100 ml with methanol. This second dilution is equivalent to 0.20 mg/ml and is used for the analysis of Bu-NENA. The first prepared solution, 100 mg/100 ml (1 mg/ml) is to be used in the analysis of MNA, N-NO-MNA, Bu-ENA and Bu-AENA. Prepare triplicates of each sample. Filter sample solutions through a 0.45-micron syringe filter before analysis.

Standards should be run before and again after samples are run. No more than five samples should be run between standards. Injections of all standards and samples are in duplicate and the duplicate injections should agree within 1% relative of each other.

The weight percent of the analyte is calculated from the calibration curve, calculated from the three standards using linear regression. The average response (area or height) of the analyte peak in the sample is used to find the concentration of analyte (mg/l) in the sample.

$$X\,(\%) = \frac{[A]}{[B]} \text{ x } 100$$

where X is weight percent analyte (MNA, N-NO-MNA, Bu-NENA, Bu-ENA or Bu-AENA) in the sample

A is mg/l X calculated from regression line

B is injected sample concentration (mg/l)

3.4.2 Relative content

If pure standards of Bu-AENA and Bu-ENA are not available an estimate of purity of water free Bu-NENA can be obtained by the following procedure :

Weigh approximately 100 mg sample to the nearest 0.1 mg in a 100 ml volumetric flask and fill with methanol. Transfer 20 ml of this solution to another 100 ml volumetric flask and fill up with methanol.

Inject sample when the chromatographic conditions are stable. Repeat the analysis three times. Use the average areas in the following calculations. Bu-ENA is eluted first, followed by MNA, then Butyl-AENA, after that N-NO-MNA and finally Bu-NENA.

Calculation of Bu-AENA, Bu-ENA and Bu-NENA:

Contents (%) = A / B x 100

Where: A = area of the respective peak

B = total area of all the peaks in the chromatogram except that from the solvent, MNA and N-NO-MNA.

Report the content of Bu-NENA, and the sum of Bu-NENA, Bu-ENA and Bu-AENA. If you work with the conditions mentioned in Para 3.3, the correction factors for Bu-ENA and Bu-AENA are as follows:

The Bu-ENA concentration is about 1.36 times higher than it would be calculated just from the peak areas. The Bu-AENA concentration is about 0.96 times as that of calculated from the peak areas.

Analysis of MNA and N-NO-MNA:

Make three different standard solutions by weighing from 5-15 mg MNA and N-NO-MNA into a 100 ml volumetric bottle, and dissolve it in acetonitrile. Dilute these three solutions 1:100 in acetonitrile, and inject each of the standards 2-3 times. Use the average areas in the following calculations.

Calculate the content of MNA and N-NO-MNA by making a standard curve using the linear regression. The area of the MNA-peak in the sample is used to find the concentration of MNA (mg/l) in the sample.

Calculation of MNA (%) = (mg/l MNA calculated from regression line) / injected sample amount x 100, and vice versa for the N-NO-MNA peak.

4. Thermal stability

Abel test method

4.0 General

Abel test method is a quick and simple test that can be used for batch control during production. Different sources of Abel test papers may give variation in results and comparison between results from different laboratories have to be verified by other methods.

Products where Abel test have been used during production have been tested with heat flow calorimeter, and the stability of the product have been found to be very good. Abel test seems to be a very sensitive test, batches failing the Abel test shows no instability in other calorimetric tests with existing criteria for pass/fail.

4.1. Principle

Bu-NENA is heated to 82.2°C, and will after a while release some nitrous gases. A stripe of 50:50-solution of glycerol/water is applied to the “Abel test paper”, and attached to the top of a test tube. The paper will react to the nitrous gases, and a brown stripe will appear between the wet and dry part of the paper. The time before the brown stripe appears gives an indication of the stability of Bu-NENA. The test must be carried out in an acid-free environment, and in an exhaust cabinet.

4.2. Equipment

Thermostated water bath with a stability of ± 1.0°C

Holder for test tubes

“Abel test paper” (operator Codite heat test papers, made in accordance with DERA Bishopton Laboratory Method 5 or similar test paper from other sources.

Test tube with approximate dimensions 13 mm (i.d.), 16 mm (o.d) and about 140 mm long

Plastic pipette

Platina hanger attached to a glass-rod through a stopper

4.3. Reagents

Glycerol (p.a.) / distilled water 50:50 (volume percent)

4.4. Method

If necessary, filter sample through 2 thickness of Whatman S&S No 604 filter paper or equivalent. Transfer 2 ml of the sample to the bottom of each of the three test tubes. The tubes must be clean, and the sample should not be spilled on the walls. Put the “Abel test paper” on a stopper covered with clean paper. Make a hole on the top of the “Abel test paper”, and add a thin stripe of the glycerol/water-solution. Secure the paper on the platina hanger attached to the glass-rod. Do not touch the “Abel test paper”, as this may interfere with the test results. Put the stopper with the glass-rod into the test tube, and make sure that the indicator stripe is placed at the same height in all test tubes. Test paper should be suspended 76 mm above the sample. Make a “blank sample” by hanging an “Abel test paper” with glycerol/water-solution in a clean tube in the same way as the sample. Place all the four test tubes simultaneously in the heating bath, which has

a temperature of 82.2 ± 1°C. The tubes should be at least 5 cm down in the bath. Start a stopwatch when the tubes are in the heating bath. Watch the test papers carefully, note the time and end the test when a brown stripe occurs between the wet and dry part of the paper. This stripe may be more visible with a white background and/or a light from a lamp shining on the paper. Strong light over a period of time may affect the result of the test.
Report the lowest value to the closest minute.

5. Chemical stability (Microcalorimetry)

5.1 General

The chemical stability is measured by microcalorimetry according to STANAG 4582 *(microcalorimetry is currently described in STANAG 4582 only. New edition of 4515 is still in the study draft version and its promulgated Edition 1 as quoted in page 1 does not contain the method yet*). The heat generation rate is recorded as a function of time for at least a period that is mentioned in STANAG 4582, table C-1.

The evaluation occurs by calculating the energy release of the decomposition reaction, which in case of Bu-NENA shall not exceed 30 J/g in the measuring period ('STANAG 4582 time').

5.2 Equipment

Microcalorimeter suitable to detect heat generation rates of 1 µW/g or less in the temperature range between 50°C and 90°C.

5.3 Procedure

Weigh approx. 3 g of the sample to the nearest 0.1 mg into a HFC ampoule and close it tightly. Place it in a microcalorimeter and measure the heat generation at 70°C, 80, or 90°C (or any temperature in-between) for the according time mentioned in STANAG 4582.

NOTE: As the loading density has no big influence on the heat flow signal it is not necessary to completely fill the ampoule. As gaseous products are formed and the volume of Bu-NENA increases with temperature it is recommended to fill the ampoule to max. 80% of its volume. Otherwise overpressure may lead to an inflation of the ampoule lid and the ampoule may get stuck in the measuring channel.

The accurate closing of the ampoule is very important.

5.4 Evaluation

Calculate the energy release of the sample within the ‘STANAG 4582’ time using the control and evaluation programme of the microcalorimeter and divide the result by the sample weight.

6. Chemical stability by Bu-ENA formation rate

6.1 General

The decomposition rate of Bu-NENA to Bu-ENA is a measure for the stability and quality of the material. Bu-NENA is aged in closed ampoules and from time to time samples are analyzed using HPLC (see Para 3).

The development of Bu-ENA during ageing is calculated by linearly regressing the data.

6.2 Equipment

Oven

Vials or microcalorimetry ampoules, which can be closed hermetically

HPLC apparatus and equipment (see Para 3)

6.3 Procedure

Fill at least 10 vials or microcalorimetry ampoules with the Bu-NENA sample (use approx. 75 % of the volume) and place them into an oven, which is adjusted to 90°C. Take out two samples at five different days within the next three weeks and analyse them by HPLC (each by double determination, see Para 3.4). (example time scheme: day 0, 4, 7, 11, 14, 18)

6.4 Analysis

Calculate the Bu-ENA content by standard HPLC evaluation procedures (see Para 3.4).

6.5 Evaluation

Plot the Bu-ENA content over time and make a linear regression, assuming a reaction of 0 order (straight line). The slope of the straight line shall not be steeper than 0.15 % a day.

If no Bu-ENA samples are available, use the relative HPLC which uses the peak area relation between Bu-ENA and Bu-NENA peak. If you measure under the conditions mentioned in Para 3.3 the concentration of Bu-ENA is 1.36 times higher than just derived from the ratio of the peaks.

7. Contents of hydroxyl

Infrared spectroscopy method

7.1. Principle

The hydroxyl content in Bu-NENA is determined by comparing the OH-peak of the sample with standards with known OH-concentration. It should be corrected for the water content, since this will affect the size of the OH-peak. The sample chamber should be flushed with dried nitrogen gas (N_2) during the whole analysis to avoid interference from moisture in the air.

7.2. Equipment

FT-IR Spectrophotometer with resolution better than 5 cm^{-1}

CaF_2-cell with 0.5 millimetres light path

7.3. Reagents

N-butanol, p.a. or purum

Standard Bu-NENA containing as low amounts as possible of water and hydroxyl, maximum 0.1%.

7.4. Method

Preparation of standards:

To a certain amount of Bu-NENA, add approximately 0.25 (weight) % water. Add three different amounts of n-butanol up to approximately 0.15 (weight) % to three other samples of the same Bu-NENA. Shake the standards for one hour. New standards should be made every 4 months.

Samples:

Install an empty CaF_2-cell in the instrument and flush the sample chamber with dried N_2-gas for a minimum of two minutes before running the background. Scan the area 3800-3200 cm^{-1}. Rinse the IR-cell twice with the sample that is to be analysed before filling and closing. Use a clean pipette for every new sample and another glass pipette to remove the solutions when rinsing.

Analyse two Bu-NENA standards with two different water levels, the three Bu-NENA standards with different mixtures of n-butanol, and finally the samples. Purge the sample chamber with N_2 for 1-2 minutes before each scan. Measure the height of the OH-peak at 3550 cm^{-1} and the water-peak at 3675 cm^{-1} in all the spectra.

The peak-height is calculated by using global base line. The base points should be set equally each time. Note base points and the corrected heights in the journal.

7.5 Calculation:

Correcting factor for water; B:

Make a curve where the absorbance values for the standard without anything added and the standard with water added are plotted. The X-values are the absorbance at 3675 cm^{-1} and the Y-values are the absorbance at 3550 cm^{-1}. Carry out a linear regression, and the slope of the line is factor *B*.

Calibrating factor for OH; A:

The net absorbance values for the standards at 3550 cm^{-1} should be corrected for water. The absorbance values for the standards are added to the formula:

$$\text{abs OH} = \text{abs } 3550 - B * \text{abs } 3675$$

Make a graph where the X-values are the values calculated above, and the Y-values are µeq OH/g Bu-NENA. The slope of the line is factor *A*.

Calculation of the hydroxyl-content:

The net absorbance at 3550 and 3675 cm^{-1} is measured for the samples. Factors A and B, which are calculated above, are used in the final calculation:

$$\text{OH}\left(\frac{\mu\text{eq}}{\text{g}}\right) = A * (\text{abs } 3550 - B * \text{abs } 3675)$$

Report instrument parameters with result.

Cleaning:

Clean the CaF_2-cell by rinsing it a couple of times with acetone. Store the cell without plugging the stoppers. This will help the cell to dry between each time it is used.

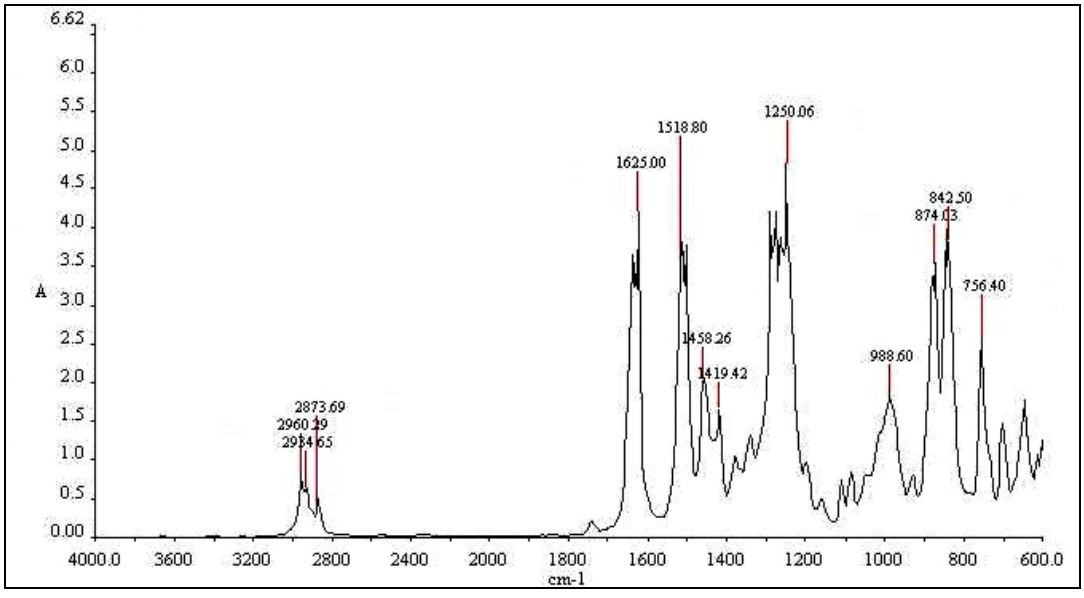


Figure C. Typical IR-spectrum of Bu-NENA