BRITISH STANDARD

BS EN 15705:2010

Fertilizers — Determination of urea condensates using high-performance liquid chromatography (HPLC) — Isobutylidenediurea and crotonylidenediurea (method A) and methylen-urea oligomers (method B)

ICS 65.080

BSI
British Standards

National foreword

This British Standard is the UK implementation of EN 15705:2010. It supersedes DD CEN/TS 15705:2009 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Fertilizers - Determination of urea condensates using high-performance liquid chromatography (HPLC) - Isobutylidenediurea and crotonylidenediurea (method A) and methylen-urea oligomers (method B)

Engrais - Dosage des condensats d'urée par chromatographie liquide haute performance (HPLC) - Isobutylidène diurée et crotonylidène diurée (méthode A) et oligomères de méthylène-urée (méthode B)

Düngemittel - Bestimmung von Harnstoffkondensaten mit Hochleistungs-Flüssigchromatographie (HPLC) - Isobutylidendiharnstoff und Crotonylidendiharnstoff (Verfahren A) und Methylenharnstoff-Oligomere (Verfahren B)

This European Standard was approved by CEN on 21 February 2010.

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Foreword

This document (EN 15705:2010) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.

This document supersedes CEN/TS 15705:2009.

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This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The following has been added to the former edition of the European Standard:

a) introduction;

b) information concerning the preparation of the standard substances MDU and DMTU.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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Introduction

Fertilizers containing the condensates of urea and specified aldehydes (with crotonaldehyde called crotonyliden diurea or CDU, with isobutyraldehyde called isobutylidene diurea or IBDU, with formaldehyde called urea formaldehyde or methylene urea or MU) are covered by Annex I of the Regulation (EC) 2003/2003 [1] as nitrogenous fertilizers. The methods described in this European Standard enable the quantitative determination of these condensates and the determination of the solubility of the MU-oligomers according to the Regulation.

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1 Scope

This European Standard specifies methods for the determination of isobutylidenediurea (IBDU), crotonylidenediurea (CDU) (method A) and methylene-urea oligomers (MU) (method B) in fertilizers using high-performance liquid chromatography (HPLC).

The method is applicable to all fertilizers which do not contain interfering organic compounds.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Sampling and sample preparation

Sampling is not part of the method specified in this European Standard. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2.

5 Method A: Determination of CDU and IBDU

5.1 Principle

The sample is extracted with water and, after appropriate dilution, analyzed using a suitable HPLC system.

5.2 Reagents

5.2.1 Reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696:1995).

5.2.2 **Acetonitrile,** p.a., HPLC-grade.

5.2.3 Isobutylidenediurea and crotonylidenediurea, in their pure form.

5.3 Apparatus

5.3.1 Laboratory equipment and glassware, for preparation of solutions and dilutions.

5.3.2 Analytical balance, capable of weighing to an accuracy of ± 0,1 mg.

5.3.3 HPLC-system, with UV-detector.

5.3.4 Ultrasonic bath

5.3.5 Magnetic stirrer

5.3.6 Disposable filter, 0,45 µm.

5.4 Procedure

5.4.1 System parameters of HPLC

Analytical/separating column:	silica column with C18 reverse phase [1)]
Detection wavelength:	200 nm
Eluent:	acetonitrile/water: 10/90 (volume fraction)
Flow rate:	1 ml/min
Temperature:	ambient temperature
Injection volume:	20 µl

5.4.2 Calibration

5.4.2.1 Stock solution IBDU ρ(IBDU) = 100 mg/l

Weigh 100/R mg of IBDU (5.2.3), where R is the purity of IBDU, into a 1 000 ml flask and add about 900 ml of water (5.2.1). Dissolve in an ultrasonic bath (5.3.4) for about 10 min, followed by stirring on a magnetic stirrer (5.3.5) for about 1 h. Make up to volume. Filtration is not necessary.

5.4.2.2 Stock solution CDU ρ(CDU) = 100 mg/l

Weigh 100/R mg of CDU (5.2.3) , where R is the purity of CDU, into a 1 000 ml flask and add about 900 ml of water (5.2.1). Dissolve in an ultrasonic bath (5.3.4) for about 10 min, followed by stirring on a magnetic stirrer (5.3.5) for about 1 h. Make up to volume. Filtration is not necessary.

5.4.2.3 Calibration solution

For calibration, prepare three solutions according to Table 1 using one-mark (bulb) pipettes and dilute to the mark with water (5.2.1).

[1)] LiChrosorb RP-18 7 µm 250/4 mm is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN or CENELEC of this product.

For the determination of the retention time, dilute 10 ml of the stock solution 5.4.2.1 or respectively 5.4.2.2 into two 100 ml flasks and make up to volume with water (5.2.1).

The evaluation of calibration is carried out manually or by means of a suitable PC-aided (computerized) calculation method.

Table 1 — Preparation of calibration solutions

Parameter	**Amount of stock solution IBDU/CDU** ml (to be added to the 100 ml flask)	**Content of IBDU** mg/l	**Content of CDU** mg/l
Standard 1	10	10,0	10,0
Standard 2	25	25,0	25,0
Standard 3	50	50,0	50,0

5.4.3 Preparation of the test portion

Weigh 1 g of the sample grounded to < 0,2 mm to the nearest 0,1 mg and flush into a 1 000 ml volumetric flask with water (5.2.1). Fill the flask to an amount of approximately 900 ml and treat it for 10 min in the ultrasonic bath (5.3.4). Then make up to the mark and stir for 1 h at room temperature on a magnetic stirrer (5.3.5). Dilute 10 ml of the solution in a 100 ml volumetric flask and filter into the HPLC injection vial through a disposable filter (5.3.6).

5.4.4 Measurement

Measurement is performed manually or by means of an automatic sample loading system (autosampler).

5.4.5 Important annotations

IBDU is able to form urea in aqueous solution. Therefore, the measurement of the calibration and sample solutions shall be completed within one working day.

The concentrations of CDU and IBDU in the sample solutions shall be kept within the calibration limits (5.4.2) to ensure sufficient reproducibility.

5.5 Calculation

The calculation can be performed manually or by means of a PC using the calibration parameters in respect to the amount used.

In the case of PC-aided (computerized) calculation and application of Table 1 regarding the amounts of stock solution, the content of IBDU/CDU in milligrams per litre will be calculated by the system. The calculated values are equal to the percentage mass concentration of IBDU/CDU in the analysed sample of fertilizer.

Following general rules for declaration in regulations to declare the content of the compounds as percentage mass fraction of nitrogen, calculate the contents, $w_{N(IBDU)}/w_{N(CDU)}$ in percent (g/100 g), according to the following equations:

$$w_{N(IBDU)} = w_{IBDU} \times 0{,}322 \quad (1)$$

$$w_{N(CDU)} = w_{CDU} \times 0{,}326 \quad (2)$$

where

0,322 is the conversion factor for the content of IBDU in the fertilizer into nitrogen content;

0,326 is the conversion factor for the content of CDU in the fertilizer into nitrogen content.

6 Method B: Determination of methylen-urea oligomers (MU)

NOTE By the condensation of urea and formaldehyde several oligomers like methylen–diurea (MDU), dimethylen-triurea (DMTU), trimethylen-tetraurea (TMTU) and higher oligomers are formed. These three molecules are the most soluble in water, the higher compounds are insoluble in hot water, but their nitrogen is available for plants by microbiological decomposition. Also urea is always a companion of MU – oligomers.

6.1 Principle

The sample is extracted with boiling water and analyzed using a suitable HPLC system.

The methylen-urea soluble oligomers are measured and detected by the HPLC-method.

In the HPLC-diagram methylen-urea oligomers are represented by different peaks: urea, methylen-diurea, dimethylen-triurea; trimethylen-tetraurea are, in the mean time, the most soluble and important.

6.2 Reagents

6.2.1 Reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696:1995).

6.2.2 Acetonitrile, p.a., HPLC-grade.

6.2.3 Urea, p.a., 46,6 % of total nitrogen.

6.2.4 Methylen-diurea (MDU), synthesized and purified by a special laboratory, 42,4 % of total nitrogen. [2)]

6.2.5 Dimethylen-triurea (DMTU), synthesized and purified by a special laboratory, 41,2 % of total nitrogen. [2)]

6.2.6 Trimethylen-tetraurea (TMTU), synthesized and purified by a special laboratory, 40,6 % of total nitrogen.

6.3 Apparatus

6.3.1 Laboratory equipment and glassware, for preparation of solutions and dilutions.

6.3.2 Analytical balance, capable of weighing to an accuracy of ± 0,1 mg.

6.3.3 Technical balance, capable of weighing to an accuracy of ± 0,01 g.

6.3.4 HPLC-system, equipped with an UV-detector.

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2) The standard substances MDU and DMTU can be prepared according to the method given in Official Methods of Analysis of AOAC International, AOAC Official Method 983.01, JAOAC 66, 769 (1983).

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6.3.5 Ultrasonic bath

6.3.6 Magnetic stirrer

6.3.7 Disposable filter, 0,45 µm.

6.4 Procedure

6.4.1 System parameters of HPLC

Analytical/separating column	NH_2 column, 5 µm, 250 mm × 4,6 mm [3)]
	A guard-column is recommended.
Detection wavelength	195 nm (Diode Array detector)
Eluent	acetonitrile/water 85/15 (volume fraction)
Flow rate	1 ml/min
Temperature	60 °C
Run time	30 min
Injection volume	20 µl

6.4.2 Calibration

6.4.2.1 Stock solution of urea, $\rho \approx 1\,000$ mg/kg

Weigh (6.3.2) $100/R$ mg of urea (6.2.3), where R is the purity of urea, to the nearest 0,1 mg and put into an empty and dry 100 ml volumetric flask, weighed (6.3.3) before to the nearest 0,01 g. Add 50 ml of water (6.2.1) and dissolve the urea in an ultrasonic bath (6.3.5) for about 10 min. Make up approximately to the mark with water (6.2.1) and homogenize. Weigh (6.3.3) the full flask to the nearest 0,01 g and record the net weight. Store at room temperature, well closed. This stock solution is stable for one week.

6.4.2.2 Stock solution of methylen-diurea, $\rho \approx 1\,000$ mg/kg

Weigh (6.3.2) $50/R$ mg of MDU (6.2.4), where R is the purity of MDU, to the nearest 0,1 mg and put into an empty and dry 50 ml volumetric flask, weighed (6.3.3) before to the nearest 0,01 g. Add 40 ml of water (6.2.1) and dissolve the MDU in an ultrasonic bath (6.3.5) for about 10 min (if necessary gently warm). Make up approximately to the mark with water (6.2.1) and homogenize. Weigh (6.3.3) the full flask to the nearest 0,01 g and record the net weight. Store at room temperature, well closed. This stock solution is stable for three weeks.

6.4.2.3 Stock solution of dimethylen-triurea, $\rho \approx 1\,000$ mg/kg

Weigh (6.3.2) $50/R$ mg of DMTU (6.2.5), where R is the purity of DMTU, to the nearest 0,1 mg and put into an empty and dry 50 ml volumetric flask, weighed (6.3.3) before to the nearest 0,01 g. Add 40 ml of water (6.2.1) at 60 °C and dissolve the DMTU in an ultrasonic bath (6.3.5) for about 10 min. Make up approximately to mark

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[3)] Supelcosil LC-NH_2 is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN or CENELEC of this product.

with water (6.2.1) and homogenize. Weigh (6.3.3) the full flask to the nearest 0,01 g and record the net weight. Store at room temperature, well closed. This stock solution is stable for three weeks.

6.4.2.4 Stock solution of trimethylen-tetraurea, $\rho \approx 100$ mg/kg

Weigh (6.3.2) $10/R$ mg of TMTU (6.2.6), where R is the purity of TMTU, to the nearest 0,1 mg and put into an empty and dry 100 ml volumetric flask, weighed (6.3.3)before to the nearest 0,01 g. Add 80 ml of water (6.2.1) at 60 °C and dissolve the TMTU in an ultrasonic bath (6.3.5) for about 10 min. Make up approximately to mark with water (6.2.1) at 60 °C and homogenize. Weigh (6.3.3) the full flask to the nearest 0,01 g and record the net weight. Store at room temperature, well closed. This stock solution is stable for three weeks.

6.4.2.5 Calibration solutions

For calibration, prepare three solutions according to Table 2.

Table 2 — Preparation of calibration solutions

Parameter	Urea stock solution g	MDU stock solution g	DMTU stock solution g	TMTU stock solution g
Standard 1	1	1	1	1
Standard 2	3	3	3	3
Standard 3	5	5	5	5

— Calibration solution 1: record the weight (6.3.3) of an empty and dry 100 ml volumetric flask (to the nearest 0,01 g), before transferring into 1 g (6.3.2) (to the nearest 0,1 mg) of each stock solution. Make up approximately to mark with water (6.2.1) and homogenize. Weigh (6.3.3) the full flask and record the net weight.

— Calibration solution 2: record the weight (6.3.3) of an empty and dry 100 ml volumetric flask (to the nearest 0,01 g) before transferring into 3 g (6.3.2) (to the nearest 0,1 mg) of each stock solution. Make up approximately to mark with water (6.2.1) and homogenize. Weigh (6.3.3) the full flask and record the net weight.

— Calibration solution 3: record the weight (6.3.3) of an empty and dry 100 ml volumetric flask (to the nearest 0,01 g) before transferring into 5 g (6.3.2) (to the nearest 0,1 mg) of each stock solution. Make up approximately to mark with water (6.2.1) and homogenize. Weigh (6.3.3) the full flask and record the net weight.

The content (approximate) of the methylen-urea oligomers in the three standard solutions is described in Table 3.

Table 3 — Content (approximate) of the methylen-urea oligomers

Parameter	Content of urea mg/kg	Content of MDU mg/kg	Content of DMTU mg/kg	Content of TMTU mg/kg
Standard 1	10	10	10	1
Standard 2	30	30	30	3
Standard 3	50	50	50	5

Gently warm at 60 °C the stock solutions 6.4.2.3 and 6.4.2.4 before transferring, to ensure the complete solubility of the DMTU and TMTU respectively.

All the calibration solutions shall be prepared fresh daily.

All the calibration solutions for the HPLC set up shall be brought at 60 °C before injection.

6.4.3 Preparation of the test solution

Weigh 0,5 g (6.3.2) of the sample grounded to < 0,1 mm to the nearest 0,1 mg and put it into a 2 000 ml (“dry” is unnecessary) beaker. Fill the beaker with an amount of approximately 950 ml of water (6.2.1) and some pieces of glass to help the boiling. Boil directly for 30 min.

Weigh (6.3.3) an empty and dry 1 l volumetric flask (to the nearest 0,01 g) before transferring into the content of the beaker, without the pieces. Wash well the beaker with boiling water and make up approximately to mark the flask with boiling water and homogenize. Weigh (6.3.3) the full flask and record the net weight.

If the injection will not be performed in a short time, keep the flask in a bath water at 70 °C to 80 °C.

Filter 1 ml into the HPLC injection vial and inject.

In the case where no auto sampler is available, manually inject 20 µl of this solution.

6.4.4 Measurement

Measurement is performed manually or by means of an automatic sample loading system (auto sampler).

6.4.5 Important annotations

The oligomers are analytically separated at a temperature of 60 °C of the column oven. In order to reach a quicker temperature alignment between column oven and eluents, it is recommended to adjust the eluents to 60 °C as well.

6.5 Calculations

The calculation can be performed manually or by means of a PC using the calibration parameters in respect to the amount used.

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In the case of PC-aided (computerized) calculation and application of Table 2 and Table 3 regarding the amounts of stock solution, the content of the different methylen-urea oligomers in milligrams per kilograms will be calculated by the system. The calculated values are equal to the percentage mass concentration of urea, methylen-diurea, dimethylen-triurea, trimethylen-tetraurea in the analysed sample of fertilizer.

Calculate the response factor of an oligomer, RF_O, according to the following equation:

$$RF_O = \frac{m_1}{A_1} \tag{3}$$

where

m_1 is the mass of that oligomer in 100 g of the standard solution;

A_1 is the peak area of that oligomer in that standard solution.

The response factor in percent from the three calibration solutions should be averaged using the following equation (RF_{MDU} for example):

$$RF_{MDU} = \frac{RF_{MDUO} \times A_{MDU}}{m_s} \times 100 \tag{4}$$

where

RF_{MDUO} is the response factor of the MDU-oligomer;

A_{MDU} is the peak area of the MDU in the sample;

m_s is the mass of the test portion (sample weight), in milligrams.

Following general rules for declaration in regulations to declare the content of the compounds as percentage mass fraction of nitrogen, calculate the contents, $w_{N(UREA)}$ / $w_{N(MDU)}$ / $w_{N(DMTU)}$ / $w_{N(TMTU)}$ in percent (g/100 g), according to the following equations:

$$w_{Nurea} = w_{urea} \times 0{,}466 \tag{5}$$

$$w_{NMDU} = w_{MDU} \times 0{,}424 \tag{6}$$

$$w_{NDMTU} = w_{DMTU} \times 0{,}412 \tag{7}$$

$$w_{NTMTU} = w_{TMTU} \times 0{,}406 \tag{8}$$

where

0,466 is the conversion factor for the content of urea in the fertilizer into nitrogen content;

0,424 is the conversion factor for the content of MDU in the fertilizer into nitrogen content;

0,412 is the conversion factor for the content of DMTU in the fertilizer into nitrogen content;

0,406 is the conversion factor for the content of TMTU in the fertilizer into nitrogen content.

To convert the mass fraction in mg/kg to the mass concentration in mg/l, consider the density of the water at 60 °C, which is 0,983 24 g/ml.

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7 Precision method A and method B

7.1 Inter-laboratory test

Inter-laboratory tests have been carried out in 2006 (for IBDU and CDU – method A) with 11 participating laboratories and 2008 (for methylen-urea oligomers – method B) with 10 participating laboratories and two different samples of fertilizers. Repeatability and reproducibility were calculated according to ISO 5725-1 and ISO 5725-2.

The values derived from these inter-laboratory tests may not be applicable to concentration ranges and matrices other than those given in Annex A.

7.2 Repeatability

The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of the cases exceed the values of r given in Table 4 (method A) and Table 5 (method B).

7.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of the cases exceed values of R given in Table 4 (method A) and Table 5 (method B).

Table 4 — Mean values, repeatability and reproducibility limits for method A

Sample	$\bar{x}$ %	r %	R %
Method A IBDU 1	17,838	1,312	3,222
Method A IBDU 2	35,411	1,297	3,874
Method A CDU	38,264	0,922	3,257

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Table 5 — Mean values, repeatability and reproducibility limits for method B

Sample, MU-oligomer	$\bar{x}$ %	r %	R %
Method B NPK 1, MDU	2,50	0,23	0,83
Method B NPK 1, DMTU	1,51	0,20	0,70
Method B NPK 1, TMTU	0,59	0,27	0,48
Method B NPK 2, MDU	2,29	0,52	0,98
Method B NPK 2, DMTU	2,14	0,38	0,79
Method B NPK 2, TMTU	1,14	0,42	1,11

8 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) test method used with reference to this European Standard, i.e. EN 15705;

c) test results obtained;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this European Standard, or regarded as optional, together with details of any incidents occurred when performing the method, which might have influenced the test result(s).

Annex A
(informative)

Results of the inter-laboratory tests

The precision of method A has been determined in the year 2006 in an inter-laboratory trial with 11 laboratories participating and carried out on 3 samples of fertilizer (for IBDU and CDU) and in the year 2008 for Method B with 10 participating laboratories and carried out on two samples of fertilizers (for methylen-urea). The statistical results are given in Table A.1, Table A.2 and Table A.3.

Table A.1 — Statistical results of the inter-laboratory tests (method A)

Parameter	IBDU 1	IBDU 2	CDU
Year of the test	2006	2006	2006
Number of participating laboratories	11	11	11
Number of laboratories after eliminating outliers	11	9	10
Level mean value, (g/100 g)	17,838	35,411	38,264
Repeatability standard deviation s_r, (g/100 g)	0,473	0,468	0,333
Coefficient of variation CV_r (%)	2,65	1,32	0,87
Repeatability limit r (2,77 s_r) (g/100 g)	1,312	1,297	0,922
Reproducibility standard deviation, s_R (g/100 g)	1,163	1,399	1,176
Coefficient of variation CV_R (%)	6,52	3,95	3,07
Reproducibility limit R (2,77 s_R) (g/100 g)	3,222	3,874	3,257

Table A.2 — Statistical results of the inter-laboratory tests for sample NPK 1 (method B)

Parameter	MDU	DMTU	TMTU
Year of the test	2008	2008	2008
Number of participating laboratories	10	10	10
Number of laboratories after eliminating outliers	8	9	9
Level mean value, (g/100 g)	2,50	1,51	0,59
Repeatability standard deviation s_r, (g/100 g)	0,08	0,08	0,10
Coefficient of variation CV_r (%)	3,31	4,85	16,83
Repeatability limit r (2,77 s_r) (g/100 g)	0,23	0,20	0,27
Reproducibility standard deviation, s_R (g/100 g)	0,30	0,25	0,17
Coefficient of variation CV_R (%)	11,99	16,88	29,29
Reproducibility limit R (2,77 s_R) (g/100 g)	0,83	0,70	0,48

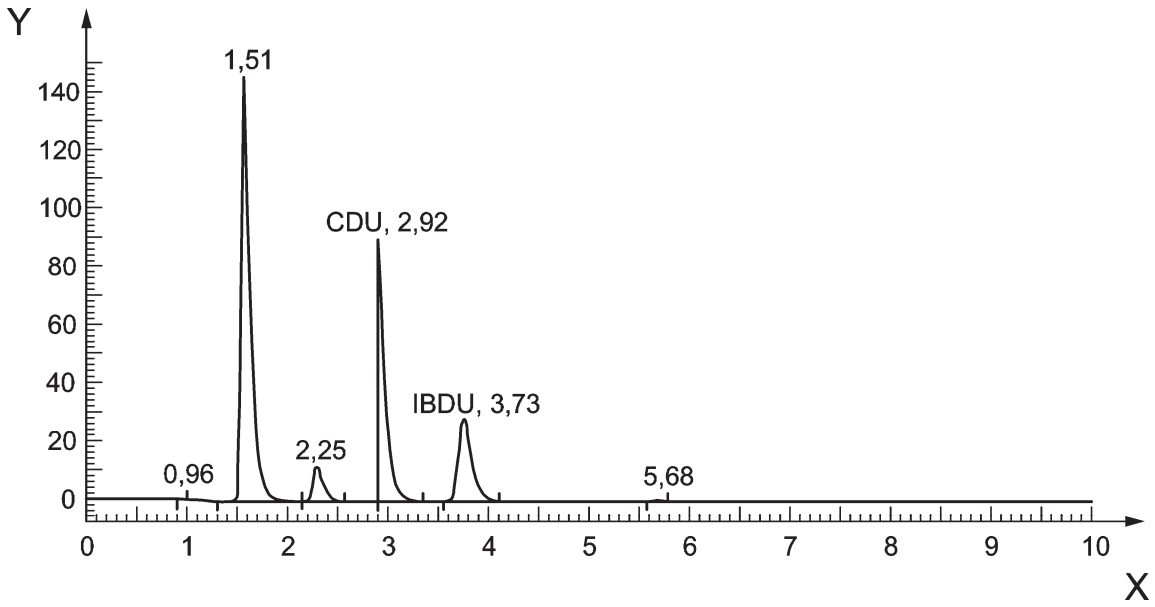
Table A.3 — Statistical results of the inter-laboratory tests for sample NPK 2 (Method B)

Parameter	MDU	DMTU	TMTU
Year of the test	2008	2008	2008
Number of participating laboratories	10	10	10
Number of laboratories after eliminating outliers	9	9	9
Level mean value, (g/100 g)	2,29	2,14	1,14
Repeatability standard deviation s_r, (g/100 g)	0,19	0,14	0,15
Coefficient of variation CV_r (%)	8,16	6,41	13,36
Repeatability limit r (2,77 s_r) (g/100 g)	0,52	0,38	0,42
Reproducibility standard deviation, s_R (g/100 g)	0,35	0,29	0,40
Coefficient of variation CV_R (%)	15,42	13,37	34,96
Reproducibility limit R (2,77 s_R) (g/100 g)	0,98	0,79	1,11

Annex B
(informative)

Chromatogram and calibration curves method A

B.1 Chromatogram

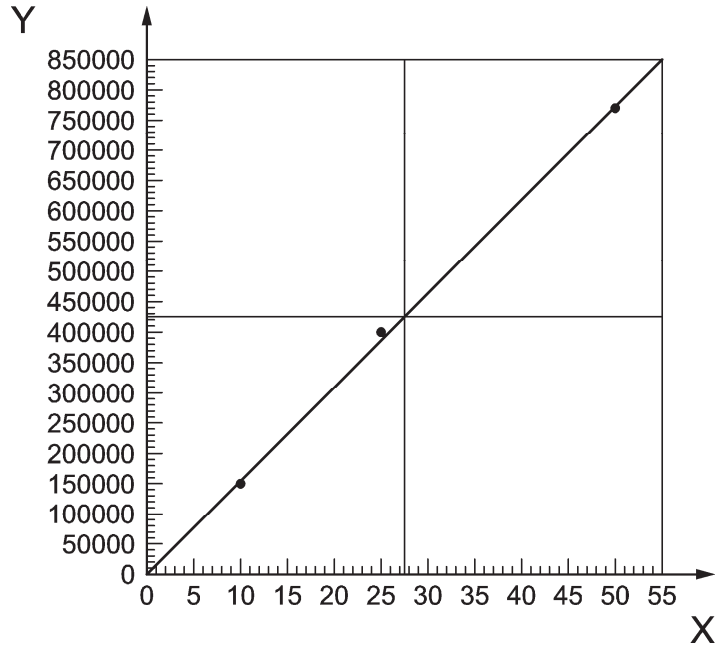


Key
X retention time (min)
Y intensity (mV)

Peak identification:	RT	Component
	0,96	Deadtime
	1,51	Inorganic components
	2,25	Urea
	2,92	Crotonylidenediurea
	3,73	Isobutylidenediurea
	5,68	Not identified peak

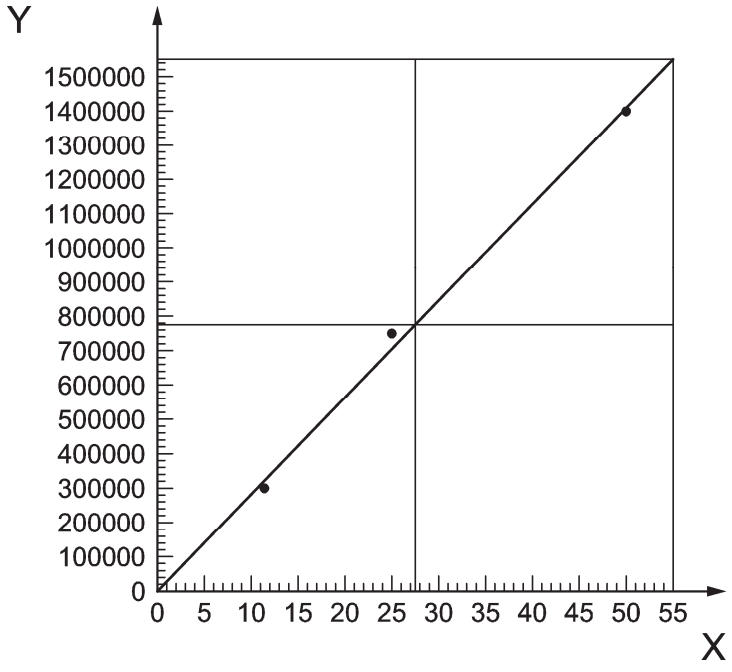
Figure B.1 — Chromatogram

B.2 Calibration curves



Key
X concentration (mg/l)
y area

Figure B.2 — Calibration curve CDU



Key
X concentration (mg/l)
y area

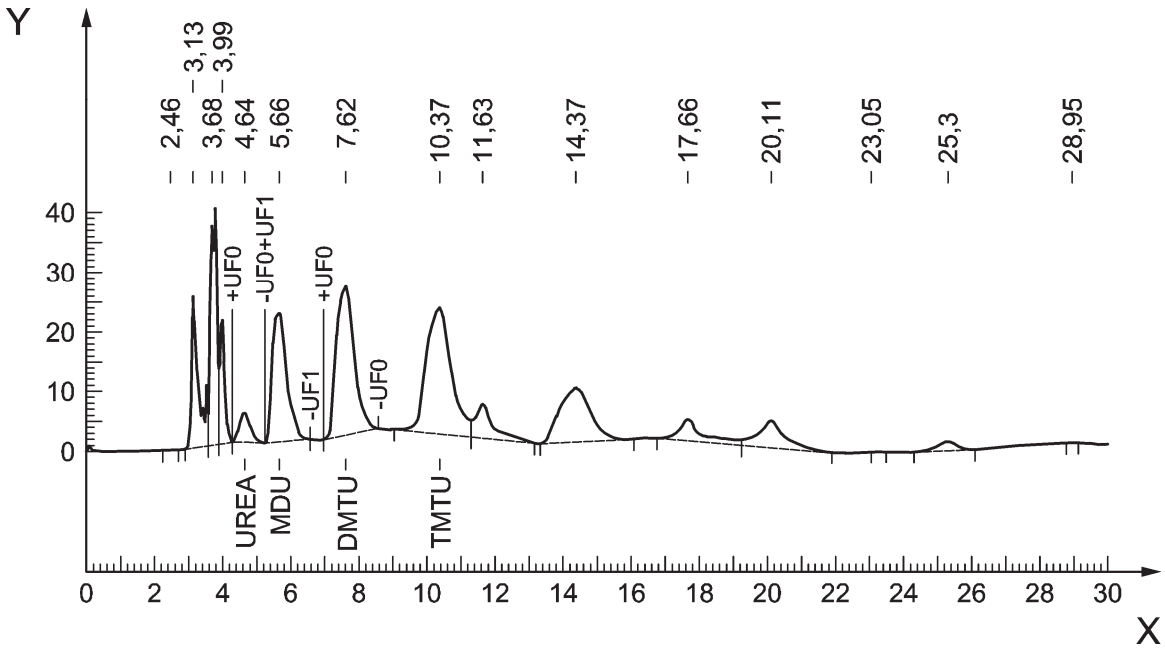
Figure B.3 — Calibration curve IBDU

Annex C
(informative)

Chromatogram and calibration curves method B

C.1 Chromatogram

Y



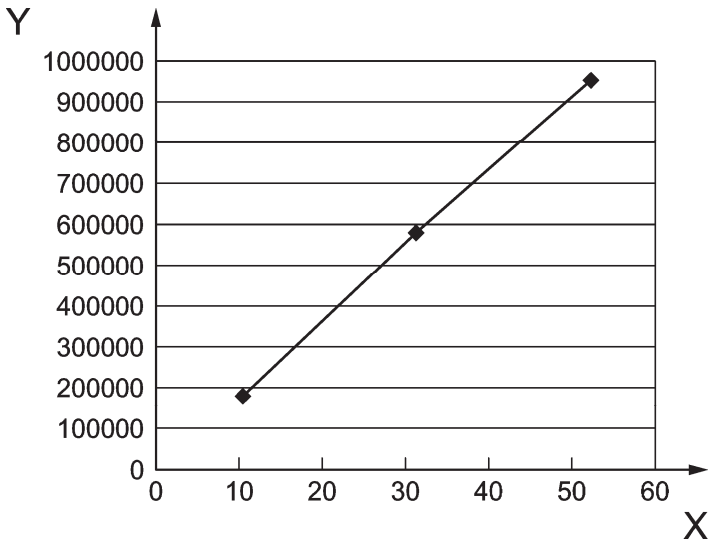
Key
X retention time (min)
Y intensity (AU)

Peak identification:	RT	Component
	4,64	Urea
	5,66	MDU
	7,62	DMTU
	10,37	TMTU

The other not identified peaks are representing components of the NPK-fertilizer

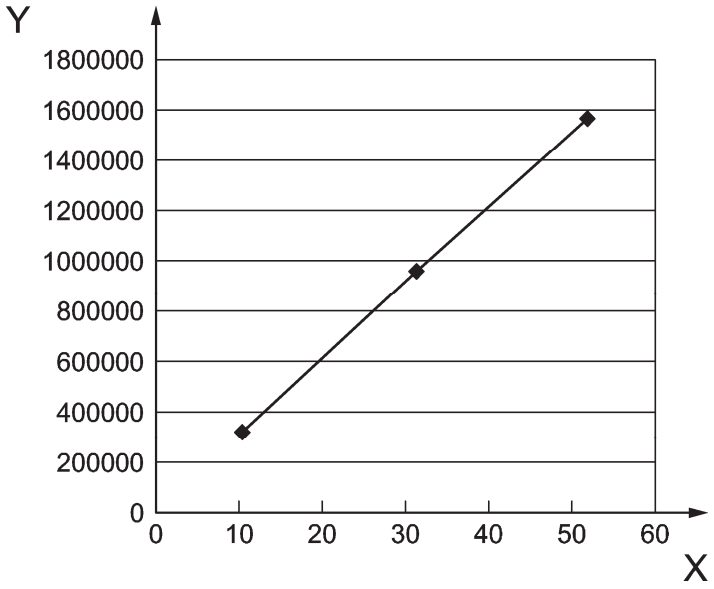
Figure C.1 — Chromatogram of the solution of a NPK – fertilizer

C.2 Calibration curves



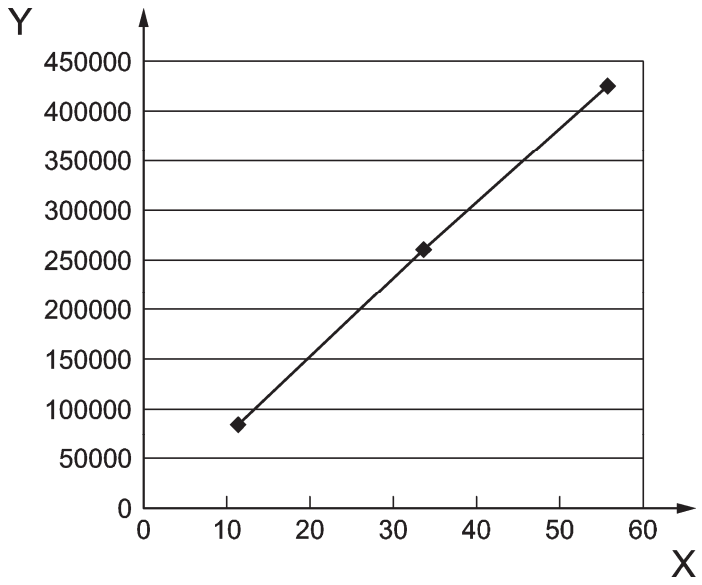
Key
X concentration (mg/kg)
y area

Figure C.2 — Calibration curve MDU



Key
X concentration (mg/kg)
y area

Figure C.3 — Calibration curve DMTU



Key
X concentration (mg/kg)
y area

Figure C.4 — Calibration curve TMTU

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