BRITISH STANDARD

BS EN
13652:2001

Soil improvers and growing media — Extraction of water soluble nutrients and elements

The European Standard EN 13652:2001 has the status of a British Standard

ICS 65.080

National foreword

This British Standard is the official English language version of EN 13652:2001.

The UK participation in its preparation was entrusted to Technical Committee AW/20, Top soil and other growing media, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Soil improvers and growing media - Extraction of water soluble nutrients and elements

Amendements du sol et supports de culture - Extraction des éléments nutritifs solubles dans l'eau

Bodenverbesserungsmittel und Kultursubstrate - Extraktion wasserlöslicher Nährstoffe und Elemente

This European Standard was approved by CEN on 11 August 2001.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 13652:2001 E

Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 223 "Soil improvers and growing media", the secretariat of which is held by BSI.

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 March 2002, and conflicting national standards shall be withdrawn at the latest by March 2002.

The annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

SAFETY PRECAUTIONS — Care should be taken when handling samples that may contain sharps or is of a dusty nature.

1 Scope

This European Standard specifies a method for the routine extraction of water-soluble extractable nutrients and elements (as listed in annex B) in soil improvers or growing media.

The method is not applicable to liming materials and preformed materials such as mineral wool slabs and foam slabs.

NOTE The requirements of the standard may differ from the national legal requirements for the declaration of the products concerned.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

EN 13039, *Soil improvers and growing media – Determination of organic matter content and ash.*

EN 13040:1999, *Soil improvers and growing media - Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density*.

3 Terms and definitions

For the purposes of this standard the terms and definitions given in EN 13039 and EN 13040 apply.

4 Principle

A sample is extracted with water at 22 °C ± 3 °C in an extraction volume ratio of 1 + 5. The extracted nutrients are determined by various methods as appropriate.

5 Reagents

5.1 General

All reagents used shall be of recognised analytical quality and water of grade 2 according to EN ISO 3696.

5.2 Nitric acid, $c(HNO_3)$ = 15 mol/l; $\rho \approx 1{,}42$ g/ml; not less than 65 % mass/volume.

5.3 Nitric acid, $c(HNO_3)$ = 0,5 mol/l, 35 ml nitric acid (**5.2**) dilute required volume of nitric acid (**5.2**) with water.

6 Apparatus

6.1 General

NOTE It has been found convenient to keep separate sets of glassware for the determinations given in annex B, in order to reduce the possibility of within-laboratory contamination.

All glass apparatus and plastic vessels used in the procedure should be subject to an appropriate preparation procedure in order to keep the risk of contamination to a minimum. It is recommended that all vessels (glass and plastic) are cleansed by carefully immersing in warm nitric acid (**5.3**) for a minimum of 6 h and then rinsed in water.

Borosilicate glassware shall not be used if boron is to be determined.

Rubber stoppers, which may contain traces of metals shall not be used. It is recommended to use plastic or any other stopper free of all substances to be analyzed.

The apparatus consists of the usual laboratory apparatus, and in particular the following:

6.2 Analytical balance with an accuracy of 10 mg.

6.3 Plastic bottles or containers, sufficiently large (500 ml to 1500 ml) with screw cap to accommodate the volume of the sample, extractant and at least 10 % air volume.

6.4 Shaking or mixing machine, capable of holding the plastic bottles or containers (**6.3**) and maintaining the sample in suspension without damaging the structure of the sample. The use of a horizontal table shaker is recommended.

6.5 Filter paper, cellulose-based ashless types, with a medium pore size of approximately 8 μm and diameter of 150 mm.

NOTE Centrifugation is an acceptable alternative.

7 Test sample

Prepare the laboratory sample in accordance with EN 13040:1999, clause 8, and determine the laboratory compacted bulk density in accordance with EN 13040:1999, annex A.

8 Procedure

8.1 Extraction

8.1.1 Test samples passing through a 20 mm sieve

Take a weight equivalent to 60 ml of the sample (EN 13040:1999, clause 8.5) volume to the nearest 1 g and transfer to the container (**6.3**). Add 300 ml of water, secure the cap and shake for 1 h on the shaking machine (**6.4**) at 22 °C ± 3 °C.

8.1.2 Test samples passing through a 40 mm sieve

Take a weight equivalent to 250 ml of the sample (EN 13040: 1999, clause 8.3) volume to the nearest 1 g and transfer to the container (**6.3**). Add 1250 ml of water, secure the cap and shake for 1 h on the shaking machine (**6.4**) at 22 °C ± 3 °C.

8.1.3 Filtration

Filter through filter paper (**6.5**) discarding at least the first 10 ml. In some cases paper filtration is too slow or even impossible. In such cases alternative procedures for obtaining a clear supernatant are acceptable and the technique used shall be reported.

The filtered extract is stable for three days in a hermetically closed polyethylene bottle if stored in a refrigerator at 0 °C to 5 °C. The filtrate can be stored for longer periods in a deep freezer at about - 18 °C.

Before using a solution that has been frozen, the thawed solution shall be thoroughly mixed to eliminate gradient separation that occurs on freezing and subsequent thawing.

8.2 Blank

The reagent blank test shall be carried out in parallel with the determination, by the same procedure as outlined in **8.1.1 or 8.1.2 and 8.1.3**, using the same quantities of all the reagents as in the determination but omitting the test portion.

NOTE The measurement of a blank is introduced to determine the contribution of the extracting solution, glassware and filter paper used.

9 Determination of extracted nutrients and elements.

See annex B.

10 Expression of results

Subtract the values determined for reagent blank from those obtained for the samples. All results shall be calculated using the determined compacted laboratory bulk density and expressed in mg/l substrate as received basis.

11 Precision

The repeatability and reproducibility of the water soluble nutrient or element content in separately prepared samples should be in accordance with Tables A.1 to A.6.

A summary of the results of an interlaboratory trial to determine the precision of the method, in accordance with ISO 5725 [2], is given in annex A.

NOTE The values derived from this interlaboratory trial may not be applicable to concentrations and matrices other than those tested.

12 Test report

The test report shall contain the following information:

a) a reference to this European Standard;

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

c) all the analytical methods used;

d) the results of the determination, expressed as mg/l fresh sample;

e) details of any operations not specified in the European Standard or regarded as optional, as well as any factor, which may have affected the results;

f) the compacted laboratory bulk density result.

Annex A
(informative)

Results of an interlaboratory trial to determine water soluble nutrients and elements

An interlaboratory trial was organised in 1997 under the auspices of the European Committee for Standardization, to test the procedures specified in this European Standard.

In this trial the number of laboratories given in the Tables A.1 to A.6 determined the water soluble nutrient content in six sample types.

All results are reported on a fresh basis.

A summary of the results of the interlaboratory trial for the determination of water soluble nutrients and elements in six sample types is given in Tables A.1 to A.6.

Table A.1 - Composted bark

Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value mg/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2{,}8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2{,}8\ s_R$
NH_4-N	22	0	764,7	23,21	8,50	65,00	260,94	95,55	730,63
NO_3-N	18	2	25,1	1,50	16,68	4,19	45,54	507,93	127,50
P	18	3	236,2	6,53	7,74	18,28	67,73	80,28	189,65
K	23	0	1910,0	44,16	6,47	123,65	300,48	44,05	841,36
Ca	20	2	59,0	4,24	20,11	11,87	19,75	93,75	55,31
Mg	21	1	23,8	1,03	12,17	2,89	3,86	45,46	10,82
SO_4-S	13	0	109,4	7,01	17,94	19,62	53,73	137,57	150,43
Na	19	1	275,1	9,32	9,49	26,11	22,35	22,75	62,59
B	13	1	1,4	0,10	20,40	0,28	0,26	53,10	0,72
Cu	19	0	1,3	0,07	15,86	0,20	0,49	108,66	1,37
Fe	20	0	10,4	0,61	16,35	1,69	3,87	104,62	10,84
Mn	20	0	1,3	0,10	21,29	0,27	0,44	96,17	1,22
Mo	8	1	0,2	0,02	23,58	0,05	0,02	33,83	0,07
Zn	20	0	2,6	0,18	19,14	0,49	0,54	58,06	1,50
Cl	16	0	898,3	35,77	11,15	100,15	131,76	41,07	368,93
F	7	1	1,0	0,36	102,93	1,01	0,91	258,07	2,54
Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value g/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2{,}8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2{,}8\ s_R$
CLBD[a]	14	1	392,8	8,3	5,9	23,3	22,1	15,8	61,9

[a] CLBD - Compacted laboratory bulk density

Table A.2 - Biowaste

Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value mg/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2{,}8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2{,}8\ s_R$
NH_4-N	20	1	115,4	2,45	5,94	6,85	60,70	147,25	169,97
NO_3-N	20	0	76,7	10,21	37,26	28,58	108,59	396,44	304,06
P	16	3	38,8	1,89	13,65	5,30	7,82	56,35	21,88
K	22	0	1430,8	50,35	9,85	140,97	267,66	52,38	749,45
Ca	20	1	101,2	8,34	23,08	23,36	37,37	103,34	104,62
Mg	20	1	31,6	2,77	24,58	7,76	14,44	128,01	40,43
SO_4-S	11	1	56,9	2,23	10,97	6,24	15,28	75,26	42,79
Na	17	2	138,7	4,24	8,56	11,86	15,29	30,87	42,81
B	13	0	1,8	0,14	21,37	0,39	0,59	90,69	1,64
Cu	18	0	0,4	0,05	29,59	0,13	0,20	124,71	0,56
Fe	19	0	43,0	3,62	23,58	10,13	34,15	222,57	95,61
Mn	17	1	1,3	0,09	20,45	0,26	0,86	187,18	2,42
Mo	9	0	0,2	0,01	19,66	0,04	0,05	72,83	0,13
Zn	18	0	1,1	0,20	51,32	0,55	0,58	150,97	1,61
Cl	14	1	578,3	10,75	5,20	30,09	48,79	23,62	136,62
F	8	1	1,1	0,15	37,02	0,41	0,39	99,64	1,10
Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value g/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2{,}8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2{,}8\ s_R$
CLBD[a]	15	1	649,5	3,5	1,5	9,7	22,7	9,8	63,6

[a] CLBD - Compacted laboratory bulk density

Table A.3 - Clay peat (fertilized)

Element	No, of labs after Eliminating outliers	No, of outliers (labs)	mean value mg/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
NH_4-N	18	4	69,7	3,09	12,41	8,65	8,96	35,96	25,08
NO_3-N	23	0	82,7	7,69	26,05	21,55	27,39	92,70	76,68
P	20	1	30,8	1,16	10,54	3,25	4,09	37,15	11,44
K	23	0	113,9	2,52	6,20	7,06	13,72	33,73	38,42
Ca	20	2	135,6	3,19	6,59	8,93	14,04	29,00	39,32
Mg	22	0	16,8	1,67	27,72	4,67	2,85	47,48	7,99
SO_4-S	13	1	109,4	4,78	12,23	13,37	9,64	24,68	26,99
Na	18	2	25,5	0,76	8,30	2,12	5,40	59,27	15,12
B	9	1	0,1	0,03	78,49	0,09	0,07	168,69	0,19
Cu	11	0	0,0	0,01	47,50	0,02	0,03	170,07	0,07
Fe	19	1	2,1	0,63	83,22	1,76	1,15	151,77	3,22
Mn	19	0	0,3	0,05	44,83	0,13	0,10	100,87	0,29
Mo	10	0	0,3	0,03	30,25	0,08	0,09	96,65	0,25
Zn	16	3	0,2	0,01	6,87	0,01	0,05	71,13	0,15
Cl	14	0	14,3	1,27	24,90	3,56	7,79	152,51	21,82
F	9	0	1,2	0,12	29,04	0,33	0,35	85,37	0,98
Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value g/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
CLBD[a]	16	0	349,2	11,0	8,8	30,8	35,5	28,5	99,4

[a] CLBD - Compacted laboratory bulk density

Table A.4 - Coarse peat (fertilized)

Element	No, of labs after Eliminating outliers	No, of outliers (labs)	mean value mg/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
NH_4-N	23	0	76,7	6,35	23,19	17,79	19,66	71,79	55,06
NO_3-N	22	2	57,0	5,88	28,90	16,48	9,17	45,05	25,69
P	20	2	88,9	4,78	15,06	13,39	21,20	66,78	59,35
K	24	0	100,9	6,71	18,63	18,80	17,96	49,86	50,30
Ca	22	1	51,9	6,24	33,68	17,48	14,39	77,64	40,30
Mg	23	0	37,4	3,62	27,09	10,14	12,00	89,83	33,61
SO_4-S	14	0	106,1	8,06	21,27	22,58	15,79	41,67	44,22
Na	20	1	31,1	2,37	21,31	6,63	4,99	44,90	13,97
B	10	3	0,1	0,01	29,71	0,04	0,03	64,25	0,08
Cu	18	0	0,1	0,01	40,40	0,04	0,04	111,18	0,11
Fe	21	0	1,0	0,15	43,32	0,42	0,38	110,73	1,07
Mn	19	0	0,2	0,03	41,62	0,08	0,07	99,84	0,18
Mo	9	0	0,2	0,02	23,70	0,04	0,07	110,97	0,20
Zn	19	1	0,3	0,04	42,80	0,12	0,14	136,63	0,39
Cl	14	0	18,9	3,22	47,88	9,03	8,41	124,92	23,56
F	9	0	0,5	0,05	28,36	0,15	0,18	97,53	0,51
Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value g/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
CLBD[a]	16	0	224,8	7,6	9,5	21,4	19,5	24,3	54,6

[a] CLBD - Compacted laboratory bulk density

Table A.5 - Composted sludge

Element	No, of labs after Eliminating outliers	No, of outliers (labs)	mean value mg/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
NH_4-N	20	2	341,0	6,35	5,21	17,77	125,57	103,11	351,59
NO_3-N	22	1	244,3	9,67	11,09	27,08	97,85	112,17	273,97
P	18	1	94,3	4,85	14,41	13,59	66,90	198,63	187,31
K	17	2	1755,3	25,17	4,01	70,46	289,69	46,21	811,12
Ca	20	1	105,3	4,60	12,23	12,87	28,81	76,64	80,67
Mg	22	0	27,2	2,58	26,59	7,23	12,18	125,40	34,09
SO_4-S	12	0	203,7	5,07	6,98	14,21	25,17	34,61	70,49
Na	19	1	174,0	4,10	6,59	11,48	17,53	28,20	49,07
B	12	1	2,3	0,12	14,47	0,33	0,32	39,70	0,90
Cu	17	2	0,6	0,02	8,96	0,05	0,37	170,25	1,03
Fe	20	0	9,0	0,95	29,59	2,66	7,13	222,25	19,98
Mn	19	0	0,8	0,11	37,97	0,30	0,68	238,98	1,90
Mo	7	2	0,3	0,01	14,30	0,04	0,03	32,08	0,09
Zn	20	0	0,8	0,06	20,77	0,17	0,48	159,10	1,33
Cl	17	0	606,8	31,31	14,44	87,65	65,81	30,37	184,27
F	6	2	0,5	0,06	36,86	0,17	0,26	163,80	0,74
Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value g/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
CLBD[a]	14	1	535,5	12,7	6,6	35,4	26,1	13,6	73,0

[a] CLBD - Compacted laboratory bulk density

Table A.6 - Composted wood fibre

Element	No, of labs after Eliminating outliers	No, of outliers (labs)	mean value Mg/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
NH_4-N	22	1	57,9	3,19	15,43	8,94	25,87	125,12	72,45
NO_3-N	22	2	66,6	3,08	12,97	8,64	66,60	280,00	186,49
P	21	1	48,5	2,49	14,37	6,98	8,09	46,68	22,66
K	23	1	125,6	3,13	6,98	8,76	16,11	35,93	45,12
Ca	23	0	110,2	6,79	17,26	19,02	21,12	53,66	59,15
Mg	22	0	14,8	0,85	16,13	2,39	2,65	50,05	7,42
SO_4-S	14	0	88,0	3,89	12,38	10,89	8,41	26,76	23,56
Na	20	1	22,9	1,56	19,08	4,36	3,50	42,93	9,81
B	12	1	0,2	0,04	63,61	0,12	0,10	150,75	0,27
Cu	14	1	0,0	0,01	34,14	0,02	0,02	116,37	0,06
Fe	21	0	1,3	0,16	35,89	0,45	0,55	123,08	1,54
Mn	18	2	0,4	0,03	23,06	0,09	0,08	56,13	0,21
Mo	10	0	0,2	0,03	33,25	0,07	0,11	142,51	0,32
Zn	17	2	0,1	0,02	38,15	0,05	0,05	111,56	0,13
Cl	15	1	17,9	2,07	32,38	5,80	8,09	126,43	22,64
F	8	1	0,8	0,10	38,38	0,29	0,21	78,61	0,60
Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value g/l	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8\ s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8\ s_R$
CLBD[a]	15	0	263,5	13,5	14,3	37,7	30,1	32,0	84,3

[a] CLBD - Compacted laboratory bulk density

Annex B
(informative)

Methods of analysis used in the interlaboratory trial

ELEMENT	METHOD
Chloride	4, 8, 14
Ammonium-N	4, 6, 7, 8, 10
Nitrate-N	4, 6, 8, 9, 10
Phosphorus	1, 4, 10, 11
Potassium	1, 2, 3
Calcium	1, 2, 3
Magnesium	1, 2
Sulfate S	1, 4
Sodium	1, 2, 3
Boron	1, 5, 12
Copper	1, 2
Iron	1, 2
Manganese	1, 2
Molybdenum	1, 2
Zinc	1, 2
Fluoride	4, 13

Method	
1	ISO 11885: 1998 Inductively coupled plasma – atomic emission spectrometry [12]
2	ISO 11047: 1998 Flame or furnace atomic absorption spectrometry [10]
3	ISO 9964-3:1993 Flame emission spectrometry [7]
4	ISO 10304-2: 1995 Ion chromatography [8]
5	Hoffmann 1997: Dianthrimide method [14]
6	ISO 5664: 1984 Distillation [1]
7	ISO 7150-1: 1984 Manual spectrophotometric [3]
8	ISO 11732: 1997 Flow spectrophotometric [11]
9	ISO 7890-3: 1988 Spectrometric - sulphosalic acid [5]
10	ISO 7150-2: 1986 Automated spectrophotometric method [4]
11	Hoffmann 1966 Ammonium molybdate ascorbic acid/stannous chloride reduction [16]
12	John M, K, et al, 1975, azomethine-H [15]
13	ISO 10359-1: 1992 Fluoride - electrochemical probe (specific ion electrode) [9]
14	ISO 9297: 1989 Silver nitrate titration, (Mohr's method) [6]

Alternative methods may be suitable for the concentration range and extract used. The user is to confirm that the method chosen gives results equivalent to those obtained by the methods listed above.

Bibliography

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[2] ISO 5725: 1994, *Accuracy (trueness and precision) of measurement methods and results.*

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