
Soil improvers and growing media — Extraction of aqua regia soluble elements

The European Standard EN 13650:2001 has the status of a
British Standard

ICS 65.080

National foreword

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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;
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Amendements du sol et supports de culture - Extraction
d'éléments solubles dans l'eau régale

Bodenverbesserungsmittel und Kultursubstrate - Extraktion
von in Königswasser löslichen Elementen

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

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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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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of an identical text or by endorsement, at the latest by March 2002, and conflicting national standards shall be withdrawn at the latest by March 2002.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 are of a dusty nature.

1 Scope

This European Standard specifies a method for the routine extraction of aqua regia soluble elements (as listed in annex B) from soil improvers or growing media. Materials containing more than about 28 % (m/m) organic matter will require treatment with additional nitric acid. With high solute concentrations in extract solutions, spectral interference's and background enhancement should be expected.

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

NOTE 1 Aqua regia will not totally dissolve most soil improvers or growing media, and the efficiency of extraction for particular elements differs from element to element. Such efficiency might also differ for the same element in different matrices. Elements extractable in aqua regia cannot therefore, be described as "Totals"; conversely they cannot be regarded as the "bio-available" fraction, as the extraction procedure is too vigorous to represent any biological process.

NOTE 2 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

The dried sample is finely ground and extracted with a hydrochloric/nitric acid mixture by standing for 16 h at room temperature, followed by boiling under reflux for 2 h. The extract is clarified and the extracted elements determined.

The test sample shall be ground to less than 500 µ prior to aqua regia digestion. Such grinding is designed to

- a) give a more homogeneous sample from which a sub-sample is taken;
- b) increase the efficiency of acid attack by increasing the surface area of the particles.

5 Reagents

5.1 General

All reagents used shall be of recognized analytical quality. Use water of grade 2 complying with EN ISO 3696.

Deionized water may be used, providing that it meets the requirements given above. It is recommended that the same batch of water be used throughout a given batch of determinations and that blank determinations are carried out.

5.2 Hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$; 1,18 g/ml; 37 % mass/volume.

5.3 Nitric acid, $c(\text{HNO}_3) = 15 \text{ mol/l}$; 1,42 g/ml; not less than 65 % mass/volume.

5.4 Nitric acid, $c(\text{HNO}_3) = 0,5 \text{ mol/l}$, 35 ml nitric acid (5.3) is diluted to one litre of water.

6 Apparatus

6.1 General

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

Clean all new glassware by carefully immersing in warm nitric acid (5.4) for a minimum of 6 h and then rinse in water.

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

6.2 Grinding mill, capable of grinding dried samples to a size less than 500 μm without contamination by the elements to be determined.

A mill causing little or no contamination is essential, as is adequate cleaning between samples to eliminate memory effects. Both agate and zirconium oxide mills have been found suitable. [5]

6.3 Test sieve, of aperture size 500 μm , e.g. test sieve with gauze preferably made from plastic materials, e.g. nylon.

6.4 Desiccator, of nominal volume 2 l.

6.5 Reaction vessel, of nominal volume of not less than 250 ml.

NOTE 1 It has been found convenient to use for mixing a two-neck Erlenmeyer type reaction vessel of nominal value 500 ml with ground-glass joints and a drip funnel of nominal value 100 ml with conical ground-glass joints and stopper.

NOTE 2 A larger reaction vessel may be required for samples when a sample weight exceeding 1 g is taken for analyses or excessive frothing is experienced.

6.6 Reflux condenser, straight-through type, with conical ground-glass joints.

NOTE Water-cooled condensers with a minimum effective length of at least 200 mm have been found suitable. The effective length is the internal surface, which is in contact with the cooling water. The overall external length of such condensers is usually at least 363 mm.

6.7 Roughened glass beads, diameter 2 mm to 3 mm (or anti-bumping granules).

6.8 Temperature-controlled heating apparatus, capable of heating the contents of the reaction vessel to reflux temperature.

6.9 Funnel, of diameter approximately 100 mm.

6.10 Volumetric flask, of nominal capacity 100 ml.

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6.11 Filter papers, cellulose-based ashless types, with a medium pore size of approximately 8 μ m and diameter of 150 mm.

6.12 Analytical balance, capable of weighing accurately to 1,0 mg.

7 Test sample passing through a 500 μ m sieve

Take approximately 5 g of the sample prepared in accordance with EN 13040:1999, clause 9, and grind the sub-sample using a mill (6.2) until all the sub sample has passed through the sieve (6.3).

8 Procedure

8.1 WARNING — Digestions with hydrochloric and nitric acid are potentially hazardous and laboratory coats, gloves and safety spectacles or goggles shall be used.

The digestion shall be carried out in a well-ventilated fume cupboard with the reflux digestion on a temperature - controlled heating apparatus. It is essential to add antibumping granules (or roughened glass beads) both to the blank and the samples to prevent bumping and loss of solution. It is important to maintain gentle reflux, both of the blank and the test samples, to avoid temperature fluctuations, which could cause local superheating.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound needs to be treated as a potential health hazard. From this viewpoint, reduce exposure to these chemicals to the lowest possible level by whatever means available.

Toxic fumes are evolved by nitric acid. Always use the concentrated acid in a fume cupboard.

8.2 Digestion

Weigh approximately 1 g to 3 g, to the nearest 0,001 g, of the finely ground test sample (**clause 7**) into the reaction vessel (6.5). Moisten with about 0,5 ml to 1,0 ml water. Add while mixing, 21 ml of hydrochloric acid (5.2) followed by 7 ml of nitric acid (5.3), drop by drop if necessary to reduce foaming. Allow to stand for 16 h at room temperature to allow for slow oxidation of the organic matter in the sample.

NOTE 1 It is common practice to add hydrochloric acid directly to the reaction vessel, followed by the nitric acid, and to assume that the aqua regia forms in the reaction vessel. However, problems have been encountered with materials rich in carbonates, where a substantial portion of the hydrochloric acid has been consumed before the nitric acid can be added. Under such circumstances, the correct formation of aqua regia is doubtful.

The amount of aqua regia is sufficient only for oxidation of about 0,85 g of organic matter. If there is any doubt about the amount of organic matter present, carry out a determination according to EN 13039. If there is more than 0,85 g of organic matter (corresponding to about 0,5 g of organic carbon) in the test sample reduce the quantity of sample analyzed or proceed as follows.

Allow the first reaction with aqua regia to subside. Then add an extra 1 ml of nitric acid (5.3) only to every 0,17 g of organic matter above 0,85 g. Do not add more than 10 ml of nitric acid at any time, and allow any reaction to subside before proceeding further.

Add a few roughened glass beads (6.7) and place the reaction vessel on a cool heating apparatus (6.8). Raise the temperature of the reaction mixture slowly until reflux conditions are reached and maintain for 2 h ensuring that the condensation zone is lower than 1/3 of the height of the condenser, then allow to cool.

Transfer the contents of the reaction vessel to a 100 ml volumetric flask (6.10). Wash the reaction vessel at least three times with water decanting each washing into the volumetric flask before adding the next. Dilute to the mark with water and mix. Allow to stand so that most of any insoluble residue settles out of suspension. Decant the relatively sediment-free supernatant onto a filter paper (6.11), discarding at least the first 10 ml of filtrate.

NOTE 2 The flask containing the extract could require the addition of releasing agents depending on the element(s) of interest, and the spectroscopic method chosen.

8.3 Reagent blank

The reagent blank test shall be carried out in parallel with the determination, by the same procedure, 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.

8.4 Residual moisture

The moisture content of the sample is determined in accordance with EN 13040:1999, clause 10, using 10 g of the finely ground sample (clause 7).

8.5 Laboratory compacted bulk density

The laboratory compacted bulk density shall be determined in accordance with EN 13040:1999, annex A.

9 Determination of extracted elements

See annex B.

10 Expression of results

Subtract values determined for reagent blanks from those obtained for the samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion. Results shall be reported on a mass/mass basis.

11 Precision

The repeatability and reproducibility of the aqua regia soluble 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 [1], 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 can be prepared separately or in conjunction with the test report of the subsequent analytical method.

The test report shall include the following information:

- a) a reference to the European Standard;
- b) a complete identification of the sample;
- c) all the analytical methods used;
- d) the results of the determination expressed as mass/mass on dry matter basis;
- e) the laboratory compacted bulk density;
- f) moisture content;

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g) any details not specified in the European Standard, or which are optional, as well as any other factor which may have affected the results.

A summary of the results of the interlaboratory trial for the determination of aqua regia soluble elements 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/kg dry matter basis	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8 s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8 s_R$
P	16	0	10469,4	414,33	11,08	1160,12	1117,25	29,88	3128,29
K	18	1	14888,4	277,06	5,21	775,77	1402,03	26,37	3925,67
Ca	19	0	23873,1	674,87	7,92	1889,63	2372,26	27,82	6642,33
Mg	18	1	4566,0	161,99	9,93	453,58	404,83	24,83	1133,53
Na	18	1	2279,2	56,76	6,97	158,92	311,90	38,32	873,33
B	10	0	27,2	2,19	22,54	6,14	6,14	63,20	17,20
Cu	21	0	59,7	2,42	11,35	6,77	6,37	29,91	17,85
Fe	19	0	2929,6	238,76	22,82	668,53	603,87	57,72	1690,85
Mn	19	1	287,5	9,45	9,20	26,45	25,80	25,13	72,24
Mo	9	0	2,2	0,19	24,67	0,54	1,34	171,54	3,75
Zn	21	0	197,6	8,99	12,74	25,17	23,17	32,84	64,88
S	9	1	3958,2	77,56	5,49	217,17	362,24	25,62	1014,29
Cd	16	1	0,4	0,12	83,67	0,33	0,20	144,81	0,57
Pb	18	0	8,9	0,76	23,90	2,13	9,55	299,54	26,74
Cr	19	0	89,3	7,07	22,17	19,80	35,81	112,30	100,27
Ni	15	4	4,6	0,30	18,41	0,85	1,26	76,27	3,53

Table A.2 - Biowaste

Element	No. of labs after eliminating outliers	No. of outliers (labs)	Mean value mg/kg	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8 s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility Limit $R = 2,8 s_R$
P	15	1	2603,8	71,02	7,64	198,85	92,15	9,91	258,02
K	19	0	10045,2	230,06	6,41	644,17	949,92	26,48	2659,79
Ca	18	1	60816,6	2052,65	9,45	5747,42	4911,55	22,61	13752,35
Mg	19	0	13762,6	501,80	10,21	1405,03	998,33	20,31	2795,32
Na	19	0	769,5	27,95	10,17	78,27	129,68	47,19	363,11
B	9	1	32,9	4,08	34,71	11,42	5,58	47,53	15,63
Cu	20	1	51,6	2,14	11,61	5,99	4,71	25,61	13,20
Fe	20	0	15120,3	461,90	8,55	1293,33	1596,46	29,56	4470,10
Mn	19	0	497,8	18,62	10,47	52,13	47,54	26,74	133,11
Mo	9	1	1,7	0,23	37,75	0,63	1,02	169,50	2,84
Zn	20	1	226,0	7,20	8,92	20,16	23,30	28,86	65,23
S	10	0	2074,5	50,12	6,76	140,33	285,91	38,59	800,55
Cd	14	3	0,4	0,03	19,75	0,09	0,19	118,74	0,53
Pb	19	1	59,3	3,80	17,94	10,64	8,87	41,90	24,85
Cr	16	3	27,5	2,11	21,55	5,92	7,19	73,30	20,12
Ni	19	0	21,9	1,13	14,51	3,17	5,75	73,66	16,10

Table A.3 Clay peat (fertilized)

Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value mg/kg	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
P	15	1	975,7	45,60	13,09	127,69	100,99	28,98	282,78
K	19	0	3821,1	149,71	10,97	419,19	796,32	58,35	2229,70
Ca	19	0	20856,4	450,41	6,05	1261,14	2117,64	28,43	5929,39
Mg	19	0	3147,9	85,95	7,65	240,66	317,02	28,20	887,65
Na	19	0	293,1	24,16	23,08	67,65	57,06	54,51	159,76
B	8	1	20,2	1,83	25,30	5,11	2,33	32,34	6,53
Cu	21	0	20,2	0,73	10,07	2,03	2,55	35,30	7,13
Fe	18	1	11996,5	417,54	9,75	1169,10	1872,94	43,71	5244,23
Mn	20	0	218,4	11,86	15,21	33,22	31,63	40,56	88,57
Mo	13	0	16,4	0,66	11,26	1,85	7,24	123,29	20,27
Zn	21	0	43,4	4,02	25,95	11,26	8,98	57,96	25,14
S	10	0	2175,2	83,47	10,74	233,72	238,53	30,70	667,89
Cd	14	0	0,3	0,03	27,79	0,08	0,18	180,35	0,51
Pb	19	1	14,7	1,15	22,01	3,23	5,33	101,69	14,92
Cr	15	3	15,8	1,00	17,67	2,79	2,06	36,53	5,77
Ni	17	1	11,4	0,84	20,64	2,35	1,99	49,07	5,58

Table A.4 - Coarse peat (fertilized)

Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value mg/kg	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8 s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8 s_R$
P	17	0	1065,7	35,16	9,24	98,45	190,80	50,13	534,23
K	20	0	1436,4	51,37	10,01	143,83	202,19	39,41	566,13
Ca	19	1	10526,4	445,03	11,84	1246,08	1647,46	43,82	4612,90
Mg	20	0	4473,4	187,27	11,72	524,35	597,38	37,39	1672,65
Na	18	2	367,5	10,94	8,33	30,62	41,52	31,64	116,27
B	9	0	13,7	4,82	98,59	13,49	5,88	120,35	16,47
Cu	22	0	17,7	1,47	23,25	4,12	7,73	122,27	21,64
Fe	16	3	2525,2	358,88	39,79	1004,86	1378,15	152,81	3858,81
Mn	17	3	45,3	2,01	12,43	5,63	9,85	60,93	27,58
Mo	11	0	26,0	1,01	10,93	2,84	12,58	135,69	35,21
Zn	20	0	13,4	1,55	32,44	4,34	6,16	128,79	17,24
S	11	0	2978,5	98,73	9,28	276,44	315,14	29,63	882,40
Cd	14	0	0,3	0,03	28,03	0,08	0,27	277,84	0,77
Pb	17	0	4,8	0,66	38,66	1,86	4,08	237,12	11,42
Cr	13	0	4,0	0,86	59,99	2,40	1,79	124,99	5,00
Ni	17	0	5,5	0,92	46,97	2,57	7,03	359,76	19,68

Table A.5 - Composted sludge

Element	No. of labs after eliminating outliers	No. of outliers (labs)	mean value mg/kg	Repeatability s_r	Repeatability Std Dev %	Repeatability limit $r = 2,8 s_r$	Reproducibility s_R	Reproducibility Std Dev %	Reproducibility limit $R = 2,8 s_R$
P	16	0	9580,5	207,10	6,05	579,88	496,17	14,50	1389,28
K	19	0	12732,0	327,89	7,21	918,10	1984,36	43,64	5556,20
Ca	19	0	28454,9	531,02	5,23	1486,85	2820,92	27,76	7898,57
Mg	17	2	4432,9	110,58	6,98	309,62	191,64	12,10	536,60
Na	19	0	1115,9	45,10	11,32	126,28	169,79	42,60	475,40
B	9	1	44,8	4,26	26,62	11,93	9,94	62,09	27,83
Cu	19	2	131,8	3,05	6,49	8,55	8,50	18,06	23,81
Fe	17	3	8658,8	438,89	14,19	1228,90	916,03	29,62	2564,88
Mn	19	1	386,3	6,43	4,66	17,99	26,16	18,97	73,26
Mo	7	2	3,3	0,22	18,96	0,62	0,69	58,96	1,93
Zn	21	0	290,3	9,29	8,96	26,02	33,22	32,04	93,02
S	10	0	4114,9	101,69	6,92	284,73	385,95	26,26	1080,65
Cd	17	1	1,4	0,06	11,85	0,16	0,43	86,53	1,19
Pb	19	1	61,0	3,16	14,53	8,86	8,68	39,88	24,31
Cr	9	4	21,4	2,49	32,64	6,99	6,79	88,79	19,00
Ni	11	4	14,7	0,85	16,30	2,39	1,70	32,38	4,76

Table A.6 - Composted wood fibre

Element	No. of labs	No. of outliers	Mean value	Repeatability	Repeatability	Repeatability	Reproducibility	Reproducibility	Reproducibility
	after			s_r	Std Dev %	limit	s_R	Std Dev %	limit
	eliminating	(labs)	Mg/kg			$r = 2,8 s_r$			$R = 2,8 s_R$
	outliers								
P	16	1	1150,7	40,53	9,86	113,49	125,36	30,50	351,00
K	20	0	2096,6	71,72	9,58	200,80	213,28	28,48	597,17
Ca	19	1	17901,0	684,46	10,71	1916,49	1662,22	26,00	4654,22
Mg	20	0	1123,4	36,03	8,98	100,87	87,05	21,70	243,73
Na	20	0	298,9	27,55	25,81	77,13	57,27	53,66	160,37
B	8	1	13,7	0,74	15,13	2,08	7,12	145,12	19,94
Cu	20	2	19,7	0,81	11,49	2,26	2,85	40,48	7,97
Fe	21	0	1839,9	69,04	10,51	193,32	152,38	23,19	426,66
Mn	20	1	108,1	4,68	12,11	13,09	11,74	30,39	32,86
Mo	11	1	18,3	1,09	16,75	3,06	2,87	43,92	8,03
Zn	18	2	25,6	1,04	11,35	2,90	5,53	60,53	15,50
S	11	0	2606,5	89,59	9,62	250,85	267,32	28,72	748,49
Cd	16	2	0,3	0,07	70,33	0,21	0,13	122,97	0,36
Pb	20	0	12,8	0,43	9,31	1,19	3,61	79,11	10,12
Cr	17	1	6,0	0,81	38,17	2,27	1,90	89,33	5,32
Ni	13	3	2,7	0,23	23,18	0,64	1,12	114,35	3,14

Annex B (informative)

Methods of analysis used in the interlaboratory trial

ELEMENT	METHOD
Phosphorus	1, 5
Potassium	1, 2, 3
Calcium	1, 2
Magnesium	1, 2
Sodium	1, 2, 3
Boron	1, 4
Copper	1, 2
Iron	1, 2
Manganese	1, 2
Molybdenum	1, 2
Zinc	1, 2
Sulfur	1
Cadmium	1, 2
Lead	1, 2
Chromium	1, 2
Nickel	1, 2

Methods

- | | |
|---|---|
| 1 | ISO 11885: 1998 Inductively coupled plasma – atomic emission spectrometry [4] |
| 2 | ISO 11047: 1998 Flame or furnace atomic absorption spectrometry [3] |
| 3 | ISO 9964-3: 1993 Flame emission spectrometry [2] |
| 4 | Dianthrimide method : Hoffmann 1997 [5] |
| 5 | Ammonium molybdate ascorbic acid/stannous chloride reduction: Hoffmann 1966 [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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