

ANNEX 1 DIGESTION PROCEDURES

Procedures of Aqua regia digestion

A.1.1 Thermal heating digestion with Aqua regia:

Digestions performed at NUA:

Procedure:

The procedure of method A of the draft standard was followed.

Mass of test portion: about 3g of sample
moistened with 0,5ml H₂O

7ml HNO₃, Suprapur, VWR art.:1.00318

21ml HCL, Suprapur, VWR art.:1.00441

The absorptions vessel was filled with 6ml HCL, Suprapur, VWR art.:1.00441 and 2ml HNO₃, Suprapur, VWR art.:1.00318.

Programme: 2h under reflux conditions with condensation zone 1/3 the height of the condenser

Cool down

Addition of the content from the absorption vessel via the condenser

Both the absorptions vessel and the condenser were rinsed with doubled deionised water. The digest was transferred to a volumetric flask and filled up to 200ml with doubled deionised water. Before measurement the sample was allowed to settle down.

Measurement:

Instrument: ICP-OES. Optima 3000XL, Perkin Elmer

Sample Preparation: sample dilution with aqua regia in following Steps 10,100, 1000
Internal Standardisation with Ytterbium
calibration in aqua regia matrix

Apparatus for digestion: composed glassware and heating devices by NUA-UA GmbH

Location: NUA-Umweltanalytik GMBH

Südstadtzentrum 4

2344 Maria Enzersdorf

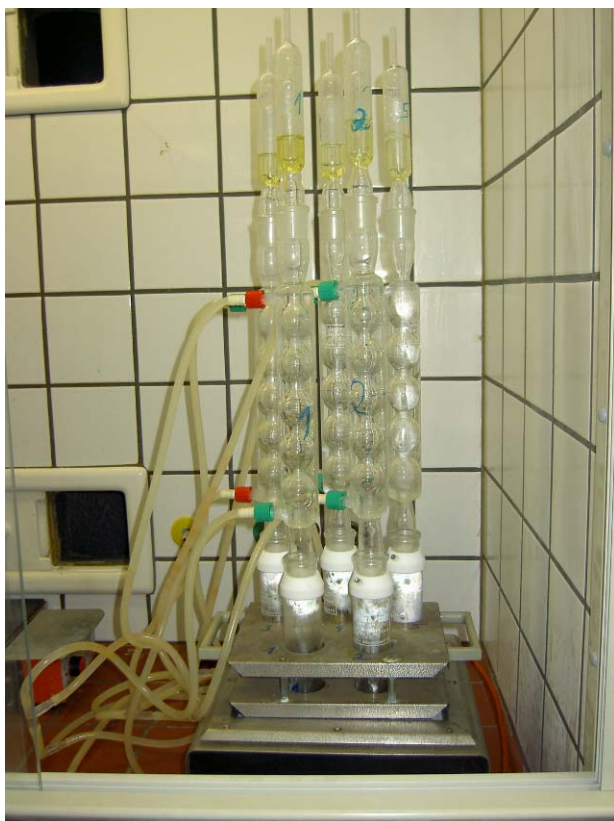


Figure A 1.1 Apparatus for thermal heating digestion

Digestions performed at LHL in Kassel:

Procedure:

Mass of test portion: about 3g of sample for soil samples, about 1,5 g of sample for compost and sludge

moistened with 0,5 ml deionised H₂O

7 ml HNO₃, p.A.,

21 ml HCL, Suprapur,

Used vessels: conventional glassware, cleaning in the washer, rinsing with deionised water

The absorptions vessel was filled with 2M HNO₃ solution.

Programme: let stand overnight, cooking 2h under reflux conditions with condensation zone 1/3 the height of the condenser

Cool down

Addition of the content from the absorption vessel via the condenser

Both the absorptions vessel and the condenser were rinsed with doubled deionised water. The digest was transferred to a volumetric flask and filled up to 100ml with deionised water.

Before measurement the sample was allowed to settle down.

A.1.2 Closed Microwave digestion with Aqua regia:

Digestions performed at NUA:

Procedure:

The procedure of method B of the horizontal draft standard was followed.

Mass of test portion: about 0,5g of sample

Added reagents:

2ml HNO₃, Suprapur, VWR art.:1.00318

6ml HCL, Suprapur, VWR art.:1.00441

Quartz vessels were used and cleaned by cooking with aqua regia

Programme for a batch of 6 samples:

Table 1.1 Programme for closed microwave oven

Time (min)	Power(W)
2	250
2	0
5	250
5	400
5	500

After digestion the vessels were allowed to cool down, transferred quantitatively to a volumetric flask and filled to 50ml with doubled deionised water. Before measurement the sample was allowed to settle down. Recording of temperature and pressure was done during digestion.

Measurement:

Instrument: ICP-OES. Optima 3000XL, Perkin Elmer

Sample Preparation: sample dilution with aqua regia in following Steps 10,100, 1000

Internal Standardisation with Ytterbium

Calibration in aqua regia matrix

Apparatus for digestion: Anton Paar, Multiwave

Location: NUA-Umweltanalytik GMBH

Südstadtzentrum 4

2344 Maria Enzerdorf



Figure A 1.2 Apparatus for closed microwave digestion

Digestions performed at Eurofins:

Procedure:

The procedure of method B of the horizontal draft standard was followed.

Mass of test portion: about 0,25g – 0,4 g of sample

Added reagents:

2ml HNO₃,

6ml HCL,

Programme for a batch of 6 samples:

Table A.2 Programme for closed microwave oven

Time (min)	Power(W)
2	250
2	0
5	250
5	400
5	500

After digestion the vessels were allowed to cool down, transferred quantitatively to a volumetric flask and filled up.

Apparatus for digestion:

Location: Eurofins, Denmark

Digestions performed at LHL in Kassel:

Procedure:

Mass of test portion: about 0,4g of sample for soil samples, about 0,2 g for sludge and compost samples

Added reagents:

1 ml HNO₃, p.A:

3 ml HCL, Suprapur,

Teflon vessels were used and cleaned by rinsing the vessels three times with diluted nitric acid

Microwave oven used: MLS 1200

Programme for a batch of 6 samples:

Table A 1.3 Programme for closed microwave oven at LHL Kassel

Time (min)	Power(W)
4	250
2	0
1,3	250
2	450
5	850
1	250

After digestion the vessels were allowed to cool down, transferred quantitatively to a volumetric flask and filled to 25ml with doubled deionised water. Before measurement the sample was allowed to settle down.

Digestions performed at UBA Wien:

Procedure 1:

The procedure of method B of the horizontal draft standard was followed.

Mass of test portion: about 0,3 respectively 0,5g of sample

Added reagents:

2ml HNO₃, Suprapur,

6ml HCL, Suprapur,

Quartz vessels were used and cleaned by cooking with aqua regia

Programme for a batch of 6 samples:

Table A 1.4 Programme for closed microwave oven

Time (min)	Power(W)
2	250
2	0
5	250
5	400
5	500

After digestion the vessels were allowed to cool down, transferred quantitatively to a volumetric flask and filled to 50ml with doubled deionised water. Then the samples were filtrated through a folded filter (Macherey-Nagel MN280 ¼, 150 mm diameter).

Measurement:

Instrument: ICP-OES. Optima 3000DV, Perkin Elmer

Calibration in aqua regia matrix

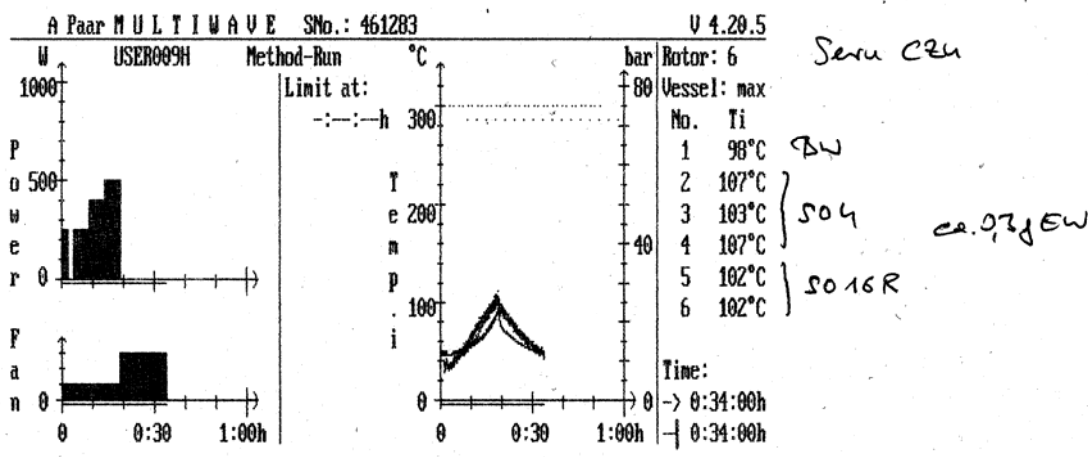
Analysis of quality control solutions after calibration and at the end

Analysis of samples, spiked with known amounts of Al, B, Cd, Co, Cr, Cu, Fe, Ni, Pb and Zn standards

ICP-OES according to ON EN 11885 (modified)

Apparatus for digestion: Anton Paar, Multiwave

Location: Umweltbundesamt Wien



Transmissivity: 0.316

Method: USER009H

Note:

Sample (USER):

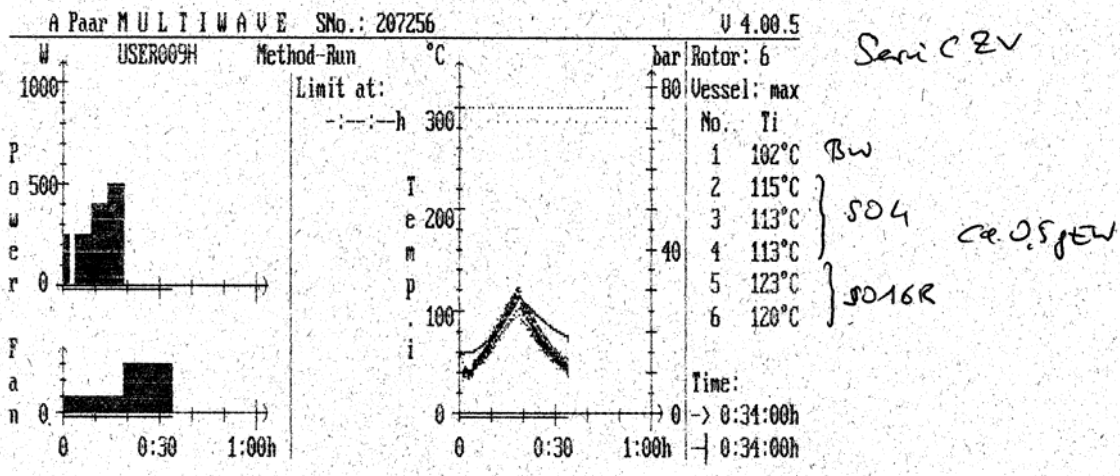
Note:

Ph	Power W	Time mm:ss	Power W	Fan
1	250	2:00	250	1
2	0	2:00	0	1
3	250	5:00	250	1
4	400	5:00	400	1
5	500	5:00	500	1
6	0	15:00	0	3
7	0	0:00	0	0
8	0	0:00	0	0

Ues	Weight max	Reag 1	Reag 2	Reag 3	Remark
	g	ml	ml	ml	
1					
2					
3					
4					
5					
6					

Run started on 00-10-07 at 22:54

Figure A 1.3 Temperature and Power Programme closed microwave UBA Wien for procedure 1 with mass of test portion of 0,3 g



Method: USER009H
Note:
Abfall-CEN mit KW + HF

Sample (USER):
Note:

Ph	Power W	Time mm:ss	Power W	Fan
1	250	2:00	250	1
2	0	2:00	0	1
3	250	5:00	250	1
4	400	5:00	400	1
5	500	5:00	500	1
6	0	15:00	0	3
7	0	0:00	0	0
8	0	0:00	0	0

Ves	Weight max	Reag 1 ml	Reag 2 ml	Reag 3 ml	Remark
1					
2					
3					
4					
5					
6					

Run started on 05-11-22 at 07:55

Figure A 1.4 Temperature and Power Programme closed microwave UBA Wien for procedure 1 with mass of test portion of 0,5 g

UBA Procedure 2:

Mass of test portion: about 0,3 respectively 0,5g of sample

Added reagents:

2ml HNO₃, p.A, subboiled

6ml HCL, p.A, subboiled

Quartz vessels were used and cleaned by cooking with aqua regia

Programme for a batch of 6 samples:

Table A 1.5 Programme for closed microwave oven

Time (min)	Power(W)
5	200
25	900
15	0

After digestion the vessels were allowed to cool down, transferred quantitatively to a volumetric flask and filled to 50ml with doubled deionised water. Then the samples were filtrated through a folded filter (Macherey-Nagel MN280 ¼, 150 mm diameter).

Measurement:

Instrument: ICP-OES. Optima 3000DV, Perkin Elmer

Calibration in aqua regia matrix

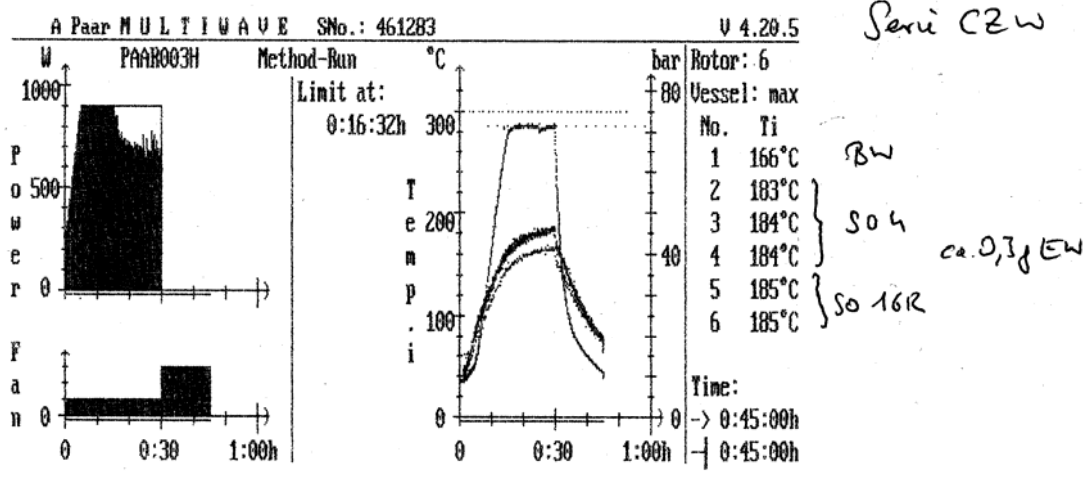
Analysis of quality control solutions after calibration and at the end

Analysis of samples, spiked with known amounts of Al, B, Cd, Co, Cr, Cu, Fe, Ni, Pb and Zn standards

ICP-OES according to ON EN 11885 (modified)

Apparatus for digestion: Anton Paar, Multiwave

Location: Umweltbundesamt Wien



Transmissivity: 0.316

Method: PAAR003H

Note:

Sample (USER):

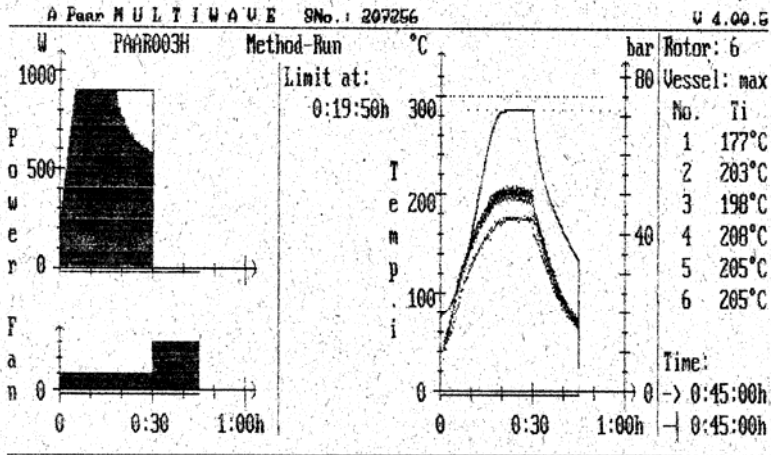
Note:

Ph	Power W	Time mm:ss	Power W	Fan
1	200	5:00	900	1
2	900	25:00	900	1
3	0	15:00	0	3
4	0	0:00	0	0
5	0	0:00	0	0
6	0	0:00	0	0
7	0	0:00	0	0
8	0	0:00	0	0

Ves	Weight max	Reag 1	Reag 2	Reag 3	Remark
	g	ml	ml	ml	
1					
2					
3					
4					
5					
6					

Run started on 00-10-07 at 23:59

Figure A 1.5 Temperature and Power Programme closed microwave UBA Procedure 2 - High Temperature Power programme, mass of test portion of 0,3 g



Pen C2x
BW
SO41
ca. 0.5 g EW
SO16R

Method: PAAR003H
Note:

Sample (USER):
Note:

Ph	Power W	Time mm:ss	Power W	Fan
1	200	5:00	900	1
2	900	25:00	900	1
3	0	15:00	0	3
4	0	0:00	0	0
5	0	0:00	0	0
6	0	0:00	0	0
7	0	0:00	0	0
8	0	0:00	0	0

Ves	Weight max g	Reag 1 ml	Reag 2 ml	Reag 3 ml	Remark
1					
2					
3					
4					
5					
6					

Run started on 05-11-22 at 08:45

Figure A 1.6 Temperature and Power Programme closed microwave UBA Procedure 2 - High Temperature Power programme, mass of test portion of 0,3 g

A.1.3 Open Microwave digestion with Aqua regia:

Procedure:

The method C described in the first version (2004) of the horizontal draft standard was followed.

Mass of test portion: about. 0,5g of sample

6ml HNO₃, Suprapur, VWR art.:1.00318

18ml HCL, Suprapur, VWR art.:1.00441

Instrument: CEM Star System

Location: Fernwärme Wien

Simmeringer Haide

11. Haidequerstrasse 6

1110 Wien



Figure A 1.7 Apparatus for open microwave digestion

Programme for a batch of 2 samples:

Table A 1.6 Programme for open microwave digestion

Time (min)	Temperature(°C)
3	103
12	103
3	103
12	103

After digestion the vessels were allowed to cool down, transferred quantitatively to a volumetric flask and filled to 50ml with doubled deionised water. Before measurement the sample was allowed to settle down.