



**WAGENINGEN EVALUATING PROGRAMS  
FOR ANALYTICAL LABORATORIES**

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**Certificate of Analysis**



**International Soil-Analytical Exchange**

**REFERENCE MATERIAL**

**ISE sample 953**

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## General Information

In this report an overview is given of analytical data for this sample collected in our proficiency testing program. The consensus values are calculated using a robust statistical model. With this NDA model mean and standard deviation are calculated using all reported data when at least 8 results are left after removal of reported 'lower than' (<) and 0 (= zero) values. No outliers are removed.

This report is divided into three sections: Consensus Values, Indicative Values and Values for Information. The division is made on the reliability of the data. Consensus Values are based on at least 16 results while the coefficient of variation is smaller than 25 %. Indicative Values are based on at least 8 and less than 16 results or a coefficient of variation between 25 % and 50 %. Other values, based on more than 2 and less than 8 results or a coefficient of variation higher than 50 %, are given for information only.

In the sections with Consensus Values and Indicative Values the following parameters are given: mean, standard deviation, coefficient of variation, number of results, median and MAD (Median of Absolute Deviation) and the uncertainty in the consensus values. The confidence limits (at 95 % probability) are calculated for these determinands.

In the section with Information Values the following parameters are given: median, MAD and number of results. For determinands which have at least 5 results reported as smaller than (<) the median of these 'smaller than results' is calculated. In some cases this median of '<' values is much smaller than median and mean of the indicative values. This may be caused by a too optimistic (too low) value for the detection limit reported by a (small) majority of participating laboratories who report '<' -values.

All values, expressed on a weight basis (kg or %), are reported in oven dry (105 °C) material. Moisture is reported in the material as received.

## Sample information

WEPAL reference materials are from natural sources only. There is no spiking, mixing or other alterations of the samples. For sample preparation the ISE samples are dried at 40 °C and milled to pass a 0.5 mm sieve.

This ISE sample 953 of Heavy Clay from Czechien is prepared for the WEPAL proficiency programs. The sample is used in 1 period (or round). The results on which the values in this report are based were taken from the period given in the following table.

Year	Round	Number
1995	4	4



## Consensus Values ISE 953

### Method: Acid extractable (So-called totals)

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Ba	mg/kg	358	30.7	8.6	31	356	21.0	6.9	346	-	369
Ca	g/kg	12.8	1.17	9.1	65	12.8	0.80	0.18	12.55	-	13.13
Co	mg/kg	14.6	3.38	23.2	44	14.4	2.41	0.64	13.5	-	15.6
Cu	mg/kg	42.7	8.44	19.8	91	43.4	5.60	1.11	41.0	-	44.5
Hg	µg/kg	1760	243	13.8	57	1770	170	40	1694	-	1823
Mn	mg/kg	1350	156	11.6	70	1330	108	23	1310	-	1384
N	g/kg	0.600	0.1405	23.4	55	0.600	0.1000	0.0237	0.562	-	0.638
Na	mg/kg	289	70.2	24.3	45	278	47.0	13.1	267	-	310
Sr	mg/kg	107	10.3	9.6	22	109	7.1	2.7	103	-	112

### Method: Extraction with boiling 2M HNO3

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Co	mg/kg	9.34	0.863	9.2	32	9.37	0.565	0.191	9.03	-	9.65
Cu	mg/kg	23.2	2.76	11.9	40	23.0	1.90	0.55	22.3	-	24.1
Hg	µg/kg	1770	200	11.3	26	1730	140	49	1694	-	1855
Pb	mg/kg	9.73	1.580	16.2	40	9.87	1.085	0.312	9.23	-	10.2
Zn	mg/kg	17.3	2.92	16.9	39	17.3	1.90	0.58	16.4	-	18.3

### Method: Extraction with 0.01M CaCl2 1:10

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
K	mg/kg	43.4	3.27	7.5	19	44.0	2.20	0.94	41.8	-	44.9
Mg	mg/kg	455	52.9	11.6	18	450	37.8	15.6	429	-	481

### Method: Soil characteristics

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
pH - CaCl2	...	6.49	0.135	2.1	34	6.50	0.095	0.029	6.45	-	6.54
pH - H2O	...	6.91	0.282	4.1	73	6.90	0.190	0.041	6.85	-	6.98
pH - KCl	...	5.63	0.172	3.1	36	5.69	0.120	0.036	5.58	-	5.69
Fraction < 2 µm	%	77.3	10.11	13.1	24	79.0	6.90	2.58	73.1	-	81.6



## Indicative Values ISE 953



### Method: Real totals

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Al	g/kg	100	4.0	4.0	11	99.3	2.7	1.5	97.4	-	102.7
Ca	g/kg	15.4	1.77	11.5	15	15.4	1.21	0.57	14.4	-	16.4
Cu	mg/kg	54.7	9.28	17.0	14	52.3	6.25	3.10	49.4	-	60.1
Fe	g/kg	53.8	5.53	10.3	14	51.9	4.11	1.85	50.7	-	57.0
K	mg/kg	1170	310	26.5	14	1180	197	103	991	-	1346
Mg	mg/kg	12000	2670	22.3	15	12200	1810	860	10480	-	13420
Mn	mg/kg	1480	229	15.5	14	1450	163	77	1345	-	1607
Na	mg/kg	1510	536	35.6	14	1510	353	179	1198	-	1813
P	mg/kg	123	21.4	17.4	11	124	14.0	8.1	109	-	137
Si	g/kg	261	15.8	6.0	9	263	10.0	6.6	249	-	273
Sr	mg/kg	150	9.6	6.4	8	151	7.0	4.3	142	-	158
Zn	mg/kg	66.3	18.98	28.7	13	65.0	13.00	6.58	54.9	-	77.6

### Method: Acid extractable (So-called totals)

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Al	g/kg	71.1	18.74	26.4	47	74.0	12.00	3.42	65.6	-	76.6
Be	mg/kg	0.872	0.1932	22.2	10	0.895	0.1300	0.0764	0.736	-	1.01
Cr	mg/kg	6.12	2.077	33.9	74	6.45	1.450	0.302	5.64	-	6.60
Fe	g/kg	41.6	10.55	25.4	70	43.6	6.60	1.58	39.1	-	44.1
K	mg/kg	501	129.6	25.9	53	520	90.0	22.2	465	-	536
Li	mg/kg	7.34	1.918	26.1	9	7.80	1.300	0.799	5.89	-	8.79
Mg	mg/kg	9040	3269	36.2	64	8510	2256	511	8220	-	9850
Ni	mg/kg	3.82	1.890	49.5	64	4.05	1.360	0.295	3.35	-	4.29
Pb	mg/kg	11.7	3.18	27.2	67	12.0	2.14	0.49	10.9	-	12.4
S	mg/kg	87.0	35.78	41.1	21	93.0	27.20	9.76	70.8	-	103
V	mg/kg	87.2	28.45	32.6	23	90.0	20.00	7.42	75.0	-	99.5
Zn	mg/kg	50.8	13.26	26.1	89	52.1	8.80	1.76	48.1	-	53.6

### Method: Extraction with boiling 2M HNO3

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Cd	mg/kg	0.0309	0.0143	46.5	22	0.0300	0.0100	0.0038	0.0245	-	0.0372
Cr	mg/kg	2.02	0.726	36.0	27	2.03	0.530	0.175	1.73	-	2.30
Ni	mg/kg	1.70	0.645	38.0	24	1.76	0.455	0.164	1.43	-	1.97
Tl	mg/kg	0.122	0.0232	19.1	10	0.120	0.0150	0.0092	0.105	-	0.138



## Indicative Values ISE 953

### Method: Extraction with 0.01M CaCl2 1:10

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Mn	mg/kg	3.94	1.107	28.1	10	3.95	0.780	0.438	3.16	-	4.72
N - NH4 (as N)	mg/kg	10.6	1.80	16.9	12	10.7	1.32	0.65	9.50	-	11.8
N - NO3 (as N)	mg/kg	6.86	0.826	12.0	14	6.88	0.560	0.276	6.39	-	7.33
Na	mg/kg	79.6	8.77	11.0	14	80.3	5.38	2.93	74.6	-	84.6

### Method: Soil characteristics

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Org.matter (L.O.I.)	%	8.70	3.414	39.3	34	9.44	2.375	0.732	7.51	-	9.89
EC-SC (ISO 11265)	mS/m	12.4	5.79	46.7	29	13.0	4.10	1.34	10.2	-	14.6
Fraction < 63 µm	%	95.1	8.31	8.7	11	91.5	6.80	3.13	89.6	-	101

### Method: Fluoride (Swiss standard procedure)

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
F - Total	mg/kg	336	53.8	16.0	14	335	38.0	18.0	306	-	367

### Method: Water soluble 1:10 (w/v) (EN-12457-4)

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
F	mg/kg	16.0	0.76	4.8	13	16.0	0.60	0.26	15.6	-	16.5



## Informative Values ISE 953



### Method: Real totals

Element	Unit	Median	MAD	N
As	mg/kg	4.62	1.280	6
Ba	mg/kg	431	38.0	7
Cd	mg/kg	0.425	0.3650	4
Co	mg/kg	17.3	3.00	7
Cr	mg/kg	15.0	6.18	11
Ga	mg/kg	25.0	5.60	5
Hg	µg/kg	1770	115	6
Ni	mg/kg	8.94	4.760	9
Pb	mg/kg	18.5	4.90	7
Rb	mg/kg	16.0	1.35	6
S	mg/kg	83.5	28.50	4
Y	mg/kg	31.0	3.30	4
Zr	mg/kg	135	5.0	6

### Method: Acid extractable (So-called totals)

Element	Unit	Median	MAD	N	Results smaller than (<)	
					Median of <	N
As	mg/kg	3.78	1.280	39	5.00	15
B	mg/kg	10.00	5.050	12	1.00	12
Cd	mg/kg	0.175	0.1350	22	0.290	50
Ga	mg/kg	18.3	3.30	3		
Mo	mg/kg	0.810	0.5100	11	1.250	14
P	mg/kg	91.8	34.20	53		
Sb	mg/kg	0.565	0.5350	4	10.000	9
Se	mg/kg	0.240	0.1200	5	3.500	10
Sn	mg/kg	0.990	0.1900	5	5.000	11
Tl	mg/kg	0.180	-	4		
Zr	mg/kg	17.7	6.30	4		

### Method: Extraction with boiling 2M HNO3

Element	Unit	Median	MAD	N	Results smaller than (<)	
					Median of <	N
Mo	mg/kg	0.0800	0.0600	8	0.2500	12

### Method: Extraction with 0.1M NaNO3

Element	Unit	Median	MAD	N	Results smaller than (<)	
					Median of <	N



**Informative Values ISE 953**

Cd	µg/kg	0.790	0.3900	4	5.000	18
Cu	µg/kg	20.0	12.90	3	50.0	17
Ni	µg/kg	-	-	0	25.0	18

**Method: Extraction with 0.1M NaNO3**

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
Pb	µg/kg	-	-	0	25.0	19
Zn	µg/kg	66.0	54.00	5	50.0	15

**Method: Extraction with 0.01M CaCl2 1:10**

Element	Unit	Median	MAD	N
B	µg/kg	928	502.7	4
Cu	µg/kg	100.0	13.00	6
Fe	mg/kg	0.105	0.0700	6
N total soluble	mg/kg	17.3	2.83	3
P	mg/kg	0.350	0.3000	9
Zn	µg/kg	150	60.2	6

**Method: Soil characteristics**

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
TIC=Tot.Inorg C(as CaCO3)	%	0.600	0.4000	15	0.500	9
Fraction > 63 µm	%	2.67	0.870	5		

**Method: Other determinations**

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
CN - Total	mg/kg	-	-	0	1.00	5

**Method: Phosphorus and related analysis**

Element	Unit	Median	MAD	N
P - Bray (as P)	mg/kg	2.74	1.260	14
P - Olsen (as P)	mg/kg	3.00	1.290	21

(cont.)