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Analyse Service GmbH

PCS - PL-1, Sediment

Veranstalter: Polish Comittee for Standardization (PCS), Division of Physical Chemistry
Ringversuchsmaterial: PL-1, Sediment
RV geschlossen: 1995 – 1
Literatur: in Form von Analysenzertifikaten, März 1996

Hauptelemente [MA%]

	CRB	RV	1sRV	Z-Score
Na_2O	0,73	0,69		---
MgO	0,26	0,26		---
Al_2O_3	5,39	5,39		---
SiO_2	85,73	85,99		---
P_2O5	0,099	0,101		---
SO_3	0,024			---
K_2O	1,88	1,84		---
CaO	0,41	0,42		---
TiO_2	0,57	0,53		---
Fe_2O_3 tot	1,20	1,17		---
MnO	0,041	0,04		---

Spurenelemente [µg/g]

	CRB	RV	1sRV	Z-Score
Ba	351	354		---
Ce	30	40,1		---
Co	3	3,9		---
Cr	48	49,6		---
Cu	6	6,2		---
Ga	5	6		---
Hf	13	13,8		---
La	22	21,5		---
Nb	10	10		---
Ni	11	7,4		---
Pb	15	19,6		---
Rb	56	55,8		---
Sr	64	67,4		---
Th	7,5	6,3		---
U	2,2	2,2		---
V	24	24,1		---
Y	18	18,8		---
Zn	30	30		---
Zr	644	634		---

Legende

CRB: Ergebnisse CRB – **RV:** Ergebnisse Ringversuch -- **1s-RV:** Standardabweichung Ringversuch
Z-Score: Differenz des Messwertes vom Mittelwert des Ringversuchs -- ***** Wert nicht zertifiziert

CENTRAL OFFICE OF MEASURES

(GŁÓWNY URZĄD MIAR – GUM)

ul. Elektoralna 2, 00-139 Warsaw, Poland

CERTIFICATE OF ANALYSIS

CERTIFIED REFERENCE MATERIAL PL-1

Lessivé Soil

(Loess)

GENERAL INFORMATION

This Certified Reference Material (CRM) is intended for use in the analysis of soils, sediments, or other materials of a similar matrix. CRM PL–1 is an agricultural soil representing Orthic Luvisol that was dried, sieved, and blended to achieve a high degree of homogeneity. A unit of CRM PL–1 consists of 100 g of the dried and radiation–sterilized material.
The certified and informative values of component content for CRM PL–1 are given in Table 1.
The agro– and physicochemical data for the soil, and its granular composition are given in Table 2.
Preparation procedures, analytical and measurement methods used for characterization of this CRM are given in Table 3. The list of participating laboratories and analysts is given in Table 4.

SOURCE , PREPARATION AND CHARACTERIZATION OF THE SOIL

The Institute of Soil Science and Plant Cultivation (Puławy, Poland) collected the material for CRM PL-1, and performed preliminary processing. The soil was collected from a plowed field, at Parchatka, N ϕ =51°21'15", E λ =22°00'30" (Plateau of Nałęczów, Upland of Lublin). The upper layer of soil containing sticks and plant debris was removed, and the soil was collected from the 0-20 cm level below original surface of 10 m x 10 m. The material was shipped to the Institute of Glass and Ceramics (Warsaw, Poland) for grinding and sieving, and then to the Institute of Nuclear Chemistry and Technology (Warsaw, Poland) for homogenization and radiation-sterilization.
Characterization of CRM PL-1 includes certified and uncertified (informative) values of total, leach and extracted content of components , and some agro- and physicochemical properties, based on interlaboratory analyses and measurements, carried out by Polish and foreign laboratories using different preparation procedures and analytical methods.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: The certification is valid for 5 years from the date of issuance of the certificate. Should any of the certified values change before the expiration date, purchasers will be notified by GUM.

Use: A minimum representative air-dried sample of 200 mg should be used for analytical measurements to be related to the certified values on this certificate.

To reproduce the values of total content, sample preparation procedures should be designed to effect complete dissolution, or analyses should be carried out nondestructively on dry samples.
If volatile components are to be determined, precautions should be taken in the dissolution of the sample to avoid volatilization losses.

CERTIFICATION OF TOTAL CONTENT

Certified values of all constituents are the medians of data sets having no less than 10 members. The uncertainty of each certified value is reported as the assymetric bounds of the approximately 95 % confidence interval of the median. For the major constituents occuring at mass fractions > 1 %, both bounds are within ± 4 % of certified value, and for minor constituents at mass fractions from 0,1 % to 1,0 %, both bounds are within ± 10 %. For trace constituents, bounds are wider, but less then ± 20 %.

UNCERTIFIED VALUES OF TOTAL CONTENT

Median values based on fewer than 10 data points, and/or having up to a factor of two wider confidence intervals than certified values are given as informative values. In some cases the informative values are presented as a range of reported laboratory data.

LEACH AND OTHER VALUES

Medians and confidence intervals or data ranges were established as described for certified total content values. Although independent verification is in general not possible for method-specific results, the values with small uncertainty based on great number of independent laboratory means, are certified.

ADDITIONAL INFORMATION

Development of CRM PL-1 was supported by the Maria Skłodowska-Curie Joint Fund within the framework of cooperation between the National Institute of Standards and Technology (NIST, USA) and Central Office of Measures (GUM, Poland; formerly Polish Committee for Standardization, Measures and Quality Control - PKNMiJ) with participation of International Soil-Analytical Exchange (ISE) , ISO Committee on Reference Materials (ISO/REMCO), Organization of National Metrological Institutions of the States of Central and Eastern Europe (COOMET) and Geostandards Newsletter (France).
The technical and support aspects involved in the preparation, certification and issuance of this CRM were coordinated by T. Plebański (PKNMiJ), J. Lipiński and T. Stachurska (GUM).
Analytical and statistical consultations were provided by T.E. Gills and J.S. Kane of the NIST Standard Reference Materials Program, and S.B. Schiller of the NIST Statistical Engineering Division.

Warsaw, December 20, 1995

Anna Michalik, Director,
Physical Chemistry Division

Certified (figures in bold type) and informative values for CRM PL– 1

Table 1. Chemical composition

Component	Content	Value (median)	Uncertainty		Unit of measurement	No. of data
			Lower Bound	Upper Bound		
Al	**Total**	**2,85**	**–0,05**	**+0,09**	**wt. %**	**29**
Al	**Leach**	**0,64**	**–0,07**	**+0,04**	**wt. %**	**47**
As	Total	*2,0 – 12,5*			ppm	9
As	**Leach**	**1,80**	**–0,2**	**+0,1**	**ppm**	**41**
As	Extr. 4	*0,001 – 0,03*			ppm	6
B	Total	*15 – 36*			ppm	4
B	Leach	*1,8 – 7,7*			ppm	11
Ba	**Total**	**354**	**–10**	**+12**	**ppm**	**25**
Ba	**Leach**	**38,0**	**–3,5**	**+3,0**	**ppm**	**31**
Br	Total	*2,0 – 7,0*			ppm	4
C	**Leach**	**0,99**	**–0,02**	**+0,03**	**wt. %**	**47**
C	Extr. 4	*0,014 – 0,11*			ppm	13
Ca	**Total**	**0,303**	**–0,013**	**+0,007**	**wt. %**	**37**
Ca	Leach	0,090	–0,012	+0,007	wt. %	58
Ca+	Extr. 5	**343**	**–12**	**+12**	**ppm**	**30**
Ca+	Extr. 6	*1,3 – 1,6*			cmol/kg	4
Cd	Total	*0,2 – 1,4*			ppm	12
Cd	**Leach**	**0,19**	**–0,01**	**+0,02**	**ppm**	**51**
Cd	**Extr. 1**	**0,18**	**–0,01**	**+0,01**	**ppm**	**30**
Cd	Extr. 2	0,0165	–0,0006	+0,0025	ppm	13
Cd	Extr. 3	*0,10 – 0,11*			ppm	2
Cd	Extr. 4	0,053	–0,013	+0,006	ppm	11
Ce	Total	40,1	–9,9	+4,7	ppm	14
Cl	Total	*80 – 120*			ppm	2
Co	Total	3,9	–0,3	+0,8	ppm	25
Co	**Leach**	**2,92**	**–0,02**	**+0,17**	**ppm**	**48**
Co	**Extr. 1**	**2,47**	**–0,07**	**+0,09**	**ppm**	**18**
Cr	**Total (direct)**	**49,6**	**–4,7**	**+4,1**	**ppm**	**20**
Cr	Total (acid)	26,3	–6,1	+2,9	ppm	13
Cr	**Leach**	**9,46**	**–0,73**	**+0,36**	**ppm**	**82**
Cr	**Extr. 1**	**5,16**	**–0,16**	**+0,48**	**ppm**	**26**
Cr	Extr. 4	*0,007 – 0,03*			ppm	8

Cs	Total	*1,0 – 3,1*			ppm	5
Cu	**Total**	**6,2**	**–1,2**	**+0,8**	**ppm**	**26**
Cu	**Leach**	**4,04**	**–0,14**	**+0,19**	**ppm**	**103**
Cu	**Extr. 1**	**3,30**	**–0,20**	**+0,22**	**ppm**	**34**
Cu	Extr. 2	0,033	–0,011	+0,008	ppm	11
Dy	Total	2,8	–0,9	+0,5	ppm	6
Er	Total	*1,0 – 1,8*			ppm	4
Eu	Total	*0,2 – 0,6*			ppm	6
F	Total	*194 – 1850*			ppm	4
F	Water Soluble	*4,5 – 8,0*			ppm	7
F	Extr. 3	*0,5 – 8,6*			ppm	3
Fe	**Total**	**0,820**	**–0,039**	**+0,010**	**wt. %**	**35**
Fe	**Leach**	**0,600**	**–0,010**	**+0,025**	**wt. %**	**83**
Ga	**Total**	**6,0**	**–0,8**	**+1,2**	**ppm**	**13**
Gd	Total	*1,9 – 9,0*			ppm	5
Hf	Total	13,8	–3,4	+6,5	ppm	7
Hg	Total	*0,02 – 2,2*			ppm	5
Hg	Leach	*0,01 – 0,24*			ppm	36
Hg	Extr. 1	*0,01 – 0,05*			ppm	19
Hg	Extr. 4	*0,001 – 0,005*			ppm	2
Ho	Total	*0,3 – 0,6*			ppm	4
I	Total	*1,0 – 1,4*			ppm	2
K	**Total**	**1,53**	**–0,05**	**+0,01**	**wt. %**	**31**
K	**Leach**	**0,084**	**–0,006**	**+0,008**	**wt. %**	**54**
K	**Extr. 3**	**58,8**	**–3,6**	**+3,2**	**ppm**	**18**
K+	**Extr. 5**	**94,4**	**–5,4**	**+3,6**	**ppm**	**36**
K+	Extr. 6	*0,08 – 0,2*			cmol/kg	4
La	**Total**	**21,5**	**–4,0**	**+1,6**	**ppm**	**13**
Li	Total	10,7	–1,4	+0,5	ppm	6
Li	Leach	4,00	–0,81	+0,50	ppm	12
Lu	Total	*0,2 – 0,4*			ppm	5
Mg	**Total**	**0,16**	**–0,01**	**+0,01**	**wt. %**	**32**
Mg	**Leach**	**0,089**	**–0,002**	**+0,006**	**wt. %**	**67**

Mg	Extr. 3	30,2	–4,6	+1,1	ppm	17
Mg+	**Extr. 5**	**32,0**	**–1,8**	**+2,0**	**ppm**	**28**
Mg+	Extr. 6	*0,2 – 0,3*			cmol/kg	4
Mn	**Total**	**394,5**	**–7,5**	**+17,5**	**ppm**	**36**
Mn	**Leach**	**330**	**–20**	**+9**	**ppm**	**86**
Mn	Extr. 3	*42 – 72*			ppm	7
Mo	Total	*1,4 – 10,0*			ppm	6
Mo	Leach	*0,08 – 0,25*			ppm	6
Mo	Extr. 1	*0,01 – 0,2*			ppm	8
N	**Leach**	**0,098**	**–0,004**	**+0,002**	**wt. %**	**57**
N	Extr. 3	*27 – 34*			ppm	2
Na	**Total**	**0,51**	**–0,03**	**+0,02**	**wt. %**	**31**
Na	Leach	0,0061	–0,0008	+0,0016	wt. %	44
Na	Extr. 3	*4,2 – 8,1*			ppm	6
Na+	Extr. 5	8,2	–2,1	+2,2	ppm	28
Na+	Extr. 6	*0,03 – 0,06*			cmol/kg	3
Nb	Total	9,95	–2,74	+0,85	ppm	12
Nd	**Total**	**16,4**	**–2,1**	**+3,1**	**ppm**	**13**
Ni	Total	7,4	–0,7	+1,8	ppm	30
Ni	**Leach**	**5,6**	**–0,6**	**+0,4**	**ppm**	**86**
Ni	**Extr. 1**	**4,1**	**–0,2**	**+0,1**	**ppm**	**24**
Ni	Extr. 2	0,058	–0,008	+0,006	ppm	13
Ni	Extr. 4	0,209	–0,019	+0,036	ppm	11
P	**Total**	**0,044**	**–0,004**	**+0,004**	**wt. %**	**26**
P	**Leach**	**0,037**	**–0,002**	**+0,002**	**wt. %**	**57**
P	Extr. 3	*0,2 – 2,7*			ppm	11
P	Bray	53,4	–6,4	+4,2	ppm	13
P	Olsen	23,0	–3,8	+3,0	ppm	18
Pb	**Total**	**19,6**	**–3,0**	**+3,9**	**ppm**	**30**
Pb	**Leach**	**11,6**	**–0,6**	**+0,4**	**ppm**	**94**
Pb	**Extr. 1**	**10,8**	**–0,6**	**+0,4**	**ppm**	**26**
Pb	Extr. 2	*0,008 – 0,04*			ppm	7
Pb	Extr. 4	*0,05 – 0,13*			ppm	10
Pr	Total	*2,8 – 4,5*			ppm	4
Rb	**Total**	**55,8**	**–3,2**	**+1,4**	**ppm**	**20**
Rb	Leach	*3,1 – 13,8*			ppm	4
S	Total	*93 – 547*			ppm	9
S	**Leach**	**147**	**–16**	**+13**	**ppm**	**21**

Sb	Total	*0,4 – 4,5*			ppm	3
Sb	Leach	*0,07 – 0,2*			ppm	7
Sc	Total	3,8	–1,7	+1,7	ppm	10
Se	Leach	0,11	–0,02	+0,03	ppm	9
Si	**Total**	**40,2**	**–0,3**	**+0,3**	**wt. %**	**19**
Sm	Total	3,4	–1,3	+0,6	ppm	7
Sn	Total	*2,4 – 15,0*			ppm	6
Sn	Leach	*0,4 – 1,3*			ppm	5
Sr	**Total**	**67,4**	**–3,4**	**+3,2**	**ppm**	**26**
Sr	Leach	9,6	–2,9	+1,1	ppm	17
Ta	Total	*0,5 – 0,8*			ppm	3
Tb	Total	*0,4 – 0,6*			ppm	4
Th	**Total**	**6,3**	**–1,3**	**+0,5**	**ppm**	**12**
Ti	**Total**	**0,32**	**–0,03**	**+0,02**	**wt. %**	**20**
Tl	Total	*0,3 – 0,6*			ppm	2
Tl	Extr. 1	0,06	–0,02	+0,02	ppm	6
Tl	Extr. 4	*0,003 – 0,005*			ppm	3
Tm	Total	*0,2 – 0,5*			ppm	5
U	Total	2,2	–0,5	+0,5	ppm	7
V	**Total**	**24,1**	**–1,5**	**+2,9**	**ppm**	**20**
V	**Leach**	**12,0**	**–2,2**	**+1,4**	**ppm**	**17**
W	Total	*0,9 – 4,2*			ppm	3
Y	Total	18,8	–5,4	+4,5	ppm	15
Y	Leach	*3,6 – 4,3*			ppm	2
Yb	Total	2,0	–1,0	+0,6	ppm	7
Zn	**Total**	**30,0**	**–1,4**	**+4,1**	**ppm**	**33**
Zn	**Leach**	**24,4**	**–0,5**	**+0,7**	**ppm**	**103**
Zn	**Extr. 1**	**19,8**	**–0,8**	**+1,2**	**ppm**	**19**
Zn	**Extr. 2**	**0,68**	**–0,06**	**+0,06**	**ppm**	**17**
Zn	Extr. 3	*1,2 – 2,7*			ppm	7
Zn	Extr. 4	1,99	–0,09	+0,16	ppm	12
Zr	**Total**	**634,4**	**–61,6**	**+40,2**	**ppm**	**20**

Table 2. Physico– and agrochemical properties

Property or component	Content (procedure)	Value (median)	Uncertainty		Unit of measurement	No. of data
			Lower Bound	Upper Bound		
CEC	Extr. 5	5,7	–0,9	+0,9	cmol/kg	19
CEC	Extr. 6	*2,8 – 7,4*			cmol/kg	5
CaCO3		*0,1 – 0,9*			wt. %	8
EC		*0,1 – 15,0*			mS/m	18
LOI		2,68	–0,30	+0,15	wt. %	31
pH	CaCl2	4,50	–0,08	+0,10	1	28
pH	Water	5,10	–0,05	+0,07	1	57
pH	KCl	4,25	–0,05	+0,07	1	31
CN	Total complex	*0,1 – 1,8*			ppm	7
N as NH4+	Extr. 3	*4,4 – 10,7*			ppm	7
N as NO3–	Extr. 3	4,33	–0,45	+1,67	ppm	9
SO4	Extr. 3	*40 – 45*			ppm	3

Granular composition

Less than 2 micrometers	6,1	–0,6	+1,2	%	19
Less than 63 micrometers	79,3	–14,3	+14,3	%	6
More than 63 micrometers	*6,7 – 29,0*			%	2

No. of data: Number of means from independent laboratories
wt. %: centigram per gram
ppm: microgram per gram
mS/m: microsiemens per metre
cmol/kg: centimole per kilogram
Total: Real total content. Determination after total decomposition of soil (with HF) or direct determination.
Leach: So called total content ("total" content). Determination after decomposition of soil without HF.
CEC: Cations exchange capacity
LOI: Loss on ignition
EC: Electrolytic conductivity
Extr. 1 – 6: See Table 3

Table 3. Preparation procedures, analytical and measurement methods

Total content preparation procedures
Direct determination on dry sample
Determination on pressed powder pellets
Determination on fused borate discs

Lithium metaborate fusion
Sodium carbonate fusion
Sodium peroxide fusion

Mixed acid digestion with HF and final medium HCl
Mixed acid digestion with HF and final medium H_2SO_4
Mixed acid digestion with HF and final medium HNO_3
Mixed acid digestion with HF and final medium $HClO_4$

Leaching procedures
Digestion in mixture of concentrated HNO_3 and concentrated HCl
Digestion in mixture of concentrated HNO_3 and concentrated HCl using boiling under reflux
Digestion in mixture of concentrated HNO_3 and concentrated HCl using microvawe
Digestion in mixture of concentrated H_2SO_4 and concentrated HNO_3
Digestion in concentrated H_2SO_4 with catalyst(s)
Digestion in concentrated $HClO_4$
Digestion in HNO_3 and H_2O_2 (EPA Method 3050)

Extractions
Extraction 1 - with 2 M HNO_3 1:10 (*W/V*)
Extraction 2 - with 0,1 M $NaNO_3$ 1:2,5 (*W/V*)
Extraction 3 - with 0,01 M $CaCl_2$ 1:10 (*W/V*)
Extraction 4 - with 1 M NH_4NO_3 1:2,5 (*W/V*)
Extraction 5 - with 1 M $(NH)_4C_2H_3O_2$ (Exchangeable cations and CEC)
Extraction 6 - with 0,01 M $BaCl_2$ (Exchangeable cations and CEC)

Analytical and measurement methods
Instrumental neutron activation analysis (INAA)
Wavelength dispersive X-ray fluorescence spectrometry (WDXRF)
Mass spectrometry (MS)
Spectrography

Inductively coupled plasma - atomic emission spectrometry (ICP-AES)
Flame atomic emission spectrometry (FAES)
Flame atomic absorption spectrometry (FAAS)
Electrothermal atomic absorption spectrometry (ETAAS)
Hydride generation atomic absorption spectrometry (HYDAAS)
Cold vapor atomic absorption spectrometry (CVAAS)
UV-VIS and IR spectrophotometry
Colorimetry

Gas chromatography
Complexometry
Gravimetry
Titrimetry
Voltametry
Nephelometry
Ion selective electrode
Pipet and sieve (particle size)

Table 4. LIST OF PARTICIPATING LABORATORIES

1. Central Institute of Agrochemical Services for Agriculture (CINAO), Moscow, Russia (A. Pukhovsky, I. Kolokolceva, L. Pohlebkina, V. Permitin, N. Sokolova)
2. Centre for Advanced Analytical Chemistry, CSIRO – Division of Coal and Energy Technology, Lucas Heights Research Laboratory, Menai NSW, Australia (J. Fardy, O. Farrell, J. Buchanan, L. Dale)
3. CRB Analysis Service, Ltd., Hardegsen, Germany (S. Pierdzig)
4. Department of Applied Geochemistry and Economic Geology, Aachen University of Technology, Aachen, Germany (G. Deissmann)
5. Ecological Laboratories (EL), Ltd., Spišská Nová Ves, Slovakia (Ľ. Korpeľ, J. Ambruš, Ľ. Blahut).
6. Ekobinf, Sumi, Ukraine (V.P. Ivanov)
7. Forest Research Institute, Div. in Katowice, Poland (I. Matuszczyk)
8. Forest Research Institute, Div. in Sękocin, Poland (G. Szołtyk)
9. Geological Department Baden–Württemberg, Freiburg, Germany (M. Martin)
10. Geological Institute, University of Bonn, Bonn, Germany (R. Klingel)
11. IFREMER Centre (Institut français de recherche pour l'exploitation de la mer), Brest, France (J. Etoubleau)
12. Institute of Ecology, Polish Academy of Sciences, Dziekanów Leśny, Poland (P. Bieńkowski)
13. Institute of Environmental Engineering, Polish Academy of Sciences, Zabrze, Poland (I. Twardowska)
14. Institute of Environmental Protection, Warsaw, Poland (Z. Jońca)
15. Institute of Mineralogy and Mineralogical Resources, Department of Mineralogy–Geochemistry–Salt Deposits, Technical University of Clausthal, Clausthal, Germany (B.J. Knipping)
16. Institute of Nuclear Chemistry and Technology, Warsaw, Poland (J. Chwastowska, L. Pszonicki, W. Skwara)
17. Institute of Plant Nutrition and Soil Science, Braunscheweig, Germany (S. Haneklaus)
18. Institute of Soil Science and Plant Cultivation, Puławy, Poland (M. Bielawska, B. Gałczyńska, J. Gałczyńska, K. Gałczyński, K. Wiącek)
19. Institute of Theoretical Physics and Astronomy, Plasma Spectroscopy Laboratory, Vilnius, Lithuania (J. Šalkauskas, B. Miliauskaitė)
20. Mineral Processing and Analysis Department, Orléans, France (F. Augustin, A. Batel)
21. National Geophysical Research Laboratory, Hyderabad, India (V. Balaram)
22. Polish Airlines LOT, Laboratory of Chemical Analysis, Warsaw, Poland (P. Janko)
23. Polish Geological Institute, Warsaw, Poland (E. Górecka)
24. Regional Chemical–Agricultural Station, Bydgoszcz, Poland (J. Rojek)
25. Regional Research Laboratory, Bhubaneswar (Orissa), India (P. Chattopadhyay)
26. Research Institute of Vegetable Crops, Skierniewice, Poland (M. Paul)
27. Technical University, Berlin, Germany (D. Pudlo)
28. Technical University, Poznań, Poland (H. Matusiewicz)
29. University of Illinois, Department of Nuclear Engineering, Urbana, Il., USA (S. Landsberger, De Wu).
30. U.S. Geological Survey, Denver, Co., USA (S. Wilson)

and 176 laboratories of ISE (International Soil–Analytical Exchange, Wageningen Agricultural University, Wageningen, The Netherlands).