HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

CAT. NO. CRM-S-D

Certificate of Analysis

HPS Certified Reference Material

Metals in Domestic Sludge

Lot No. 729010

This Certified Reference Material is an industrial sludge obtained from the Greenville, South Carolina, domestic waste sludge facilities. The certified values are based on at least two different analytical techniques for major, minor and trace elements after a total digestion.

The material was collected from the Greenville dry waste facilities, stored in polyethylene bags, and transported to the laboratory. The sludge was transferred onto a 2 ft x 4 ft polyethylene-lined drying tray and air dried for five days with frequent mixing in direct sunlight. The sludge was ground in a pulverizer and sieved. Coarse particles were removed and only particles that pass a 100 mesh sieve and collected on a 300 mesh sieve were retained. The coarse particles were reground and sieved. The fraction that passed a 100 mesh sieve was combined in a 55-gallon polyethylene mixing-drum and blended for several hours to provide a homogeneous material. The material was packaged in a plastic bag in a 12 x 12 x 16 inch cardboard cartons and shipped to Neutron Products, Inc., Dickerson, Maryland. It was then sterilized with 25 kGy of gamma radiation. The material was returned to High-Purity Standards and bottled into 50-g units under ultraviolet radiation. Random selected bottles were taken for the final homogeneity testing.

Total Digestion Certified Values

Instructions for drying: Sample should be dried for 2 h at 110°C. Volatile elements (i.e., Hg) should be determined on samples as received. Separate samples should be dried as previously described to obtain a correction factor for moisture. (CAUTION: Sample used for Hg analyses should not be stored in a desiccator as this may lead to contamination.)

Total Digestion Method: Transfer 1.000 g of the dried material to a clean 100 mL Telfon beaker. Add 5 mL of high-purity HNO_3 and 10 mL of HF, cover beaker with a Teflon lid and digest on a hot plate at 120°C for 6-8 h. Remove the lid and add 10 mL of $HClO_4$. *(NOTE: If the sample has gone dry or contains less than 5 mL of acid, add an additional 5 mL of HNO_3). Continue the digestion on a hot plate at 160°C or strong fumes of perchlorate until sample is dry. Rinse down the sides of the beaker with water and take sample to dryness.

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Continue to heat until all signs of acid fumes are removed. Add 5 mL of HNO_3 and 20 mL of water and heat to dissolve the residue. Transfer the solution to a volumetric flask and dilute to calibrate volume with water.
***CAUTION** - $HClO_4$ should be handled with care.

If the sample contains any undissolved solids, filter off the insoluble residue using a quantitative filter paper. Save the filtrate and return the filter paper with the residue to the beaker. Add 10 mL of HNO_3 to the beaker and heat to destroy the filter paper. Evaporate the solution to a low volume and add 10 mL of HCl and 5 mL of HNO_3. Evaporate the solution to approximately 5 mL. Add 10 mL of HF, and cover beaker with a Teflon lid. Heat the solution for 1 hour at a temperature of 120°C. Then remove the Teflon lid, rinse down the side of the beaker, and add 10 mL of $HClO_4$. Heat to strong fumes of perchlorate. Continue to heat until sample is dry. Rinse down the sides of the beaker and continue to heat until all signs of acids fumes are removed. Add 5 mL of HNO_3 and 15 mL of water and heat to dissolve residue. (NOTE: The solution should be clear at this stage.) Combine this solution with the filtrate and dilute to a calibrated volume with water and proceed with the analysis of the analytes be Flame AAS, GFAAS, and ICP.

Certified Values:

The certified values are based on the results of 5 to 30 determinations by two different analytical techniques. The estimated uncertainties at the 95-percent confidence limits include those due to sample variations, possible method differences and errors of measurement.

Certified Values for Domestic Sludge
Major Constituents

Element	Conc., mg/g
Al	139 ± 18
Ca	1.54 ± 0.15
Fe	30.8 ± 3.0
K	4.65 ± 0.51
Mg	1.37 ± 0.18
Mn	2.21 ± 0.12
Na	0.69 ± 0.15
P	(1.9)
S	(3.2)
Ti	(2.5)

Certified Values for Domestic Sludge
Trace Constituents

Element	Conc., µg/g	Element	Conc., µg/g
Ag	(0.06)	Mo	(11)
As	17.8 ± 3.0	Ni	20.8 ± 2.7
Ba	198 ± 15	Pb	22.3 ± 2.4
Be	1.37 ± 0.18	Sb	<0.005
Cd	(0.1)	Se	<0.005
Co	(9.6)	Sr	38.7 ± 5.7
Cr	108 ± 12	Tl	<0.005
Cu	40.9 ± 3.2	V	104 ± 12
Li	(24)	Zn	96 ± 18

Values in parenthesis are given for information purposes only.

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EPA Method 3050B Certified Values

Instructions for preparation: Proceed as described in EPA Method 3050B.

Certified Values: The certified values are based on the results of 5 to 30 determinations by two different analytical techniques. The estimated uncertainties at the 95-percent confidence limits include those due to sample variations, possible method differences and errors of measurement.

Certified Values for Domestic Sludge Major Constituents	
Element	**Conc. (mg/g)**
Al	120 ± 10
Ca	1.3 ± 0.1
Fe	29 ± 3
K	0.79 ± 0.08
Mg	0.97 ± 0.15
Mn	2.1 ± 0.2
Na	0.077 ± 0.007
Ti	0.60 ± 0.06
P	(2)
S	(3.3)

Certified Values for Domestic Sludge Trace Constituents	
Element	**Conc. (*u*g/g)**
Ag	(0.05)
As	17 ± 3
Ba	92 ± 6
Be	1.1 ± 0.2
Cd	(0.12)
Co	9.3 ± 0.7
Cr	100 ± 15
Cu	39 ± 2
Li	20 ± 3
Mo	(9)
Ni	18 ± 2
Pb	19 ± 3
Sb	< 0.005
Se	< 0.005
Sr	14 ± 2
Tl	< 0.005
V	(90)

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Zn	94 ± 7

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Theodore C. Rains, Ph.D.
President