

Fax (843) 767-7906

## **Certificate of Analysis** HPS Certified Reference Material Loam B Lot No. 691029

This Certified Reference Material is loam soil obtained from Shelby County, Kentucky. The certified values are based on at least two independent analytical techniques for major, minor, and trace elements after a total and EPA-3050 digestion procedure.

The material was collected from the field and stored in polyethylene bags in cardboard boxes (12 in. x 12 in. x 18 in.) and transported to the laboratory. The soil was transferred onto a 2 ft. x 4 ft. polyethylene-lined drying tray and air dried for three days with frequent mixing in direct sunlight during the month of July. During the drying period all foreign objects were removed by hand. The soil was then dried under infrared heat lamps, ground in a ballmill, and sieved. Coarse particles were removed and only particles that pass a 100 mesh sieve were saved. The coarse particles were reground and sieved. The saved material was transferred to a 55-gal polyethylene mixing-drum and blended for several hours. Then the material was bottled into 50-g units. Randomly selected bottles were taken for the final homogeneity testing.

<u>Instructions for drying</u>: 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. The weight loss on drying was determined to be in the range of 0.5 to 1.0 percent. (CAUTION: Samples used for Hg analyses should not be stored in a desiccator as this may lead to contamination.)

## Preparation of Sample for Analysis:

(A) Total Digestion Method: Transfer 2.000 g of the dried material to a clean 100 mL Teflon beaker. Add 5 mL of high-purity HNO<sub>3</sub> 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<sub>4</sub>. (NOTE: If the sample has gone dry or contains less than 5 mL of acid, add an additional 5 mL of HNO<sub>3</sub>). 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. Continue to heat until all signs of acid fumes are removed. Add 5 mL of HNO<sub>3</sub> and 20 mL of water and heat to dissolve the residue. Filter off the insoluble residue using a quantitative filter paper. Save the filtrate and return the filter paper with residue to the beaker. Add 10 mL of HNO<sub>3</sub> 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<sub>3</sub>. Evaporate the solution to approximately 5 mL. Add



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10 mL of HF, and cover beaker with a Teflon lid. Heat the solution for 1 hour at a temperature of  $120^{\circ}$ C. Then remove the Teflon lid, rinse down the side of the beaker, and add 10 mL of HClO<sub>4</sub>. 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 acid fumes are removed. Add 5 mL of HNO<sub>3</sub> and 15 mL of water and heat to dissolve residue. (NOTE: The solution should be clear at this stage.) If so desired, the analyst can ignore any residue or repeat the addition of HF and HClO<sub>4</sub>. Combine this solution with the filtration and dilute to a calibrated volume with water and proceed with the analysis of the analytes by AAS, ICP, or ICP/MS.

(B) EPA 3050A Digestion Method: Transfer 2.000 g of the dried material to a clean Teflon beaker and proceed as described in the EPA procedure.

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

Certified	Values for Loam Soil B Total Digestion
Element	Conc., mg/g
Al	$51.4 \pm 1.0$
Ca	$3.39 \pm 0.30$
Fe	$27.4 \pm 0.9$
Κ	$15.5 \pm 0.6$
Mg	$4.04 \pm 0.08$
Mn	$1.59 \pm 0.04$
Na	$4.10 \pm 0.03$
Р	(1.3)
Ti	(5.7)
	Conc., $\mu g/g$
Ag	$21 \pm 1$
As	$53 \pm 6$
В	(58)
Ba	$440 \pm 40$
Be	$9.6 \pm 0.6$
Cd	$92 \pm 7$
Со	$53 \pm 3$
Cr	$80 \pm 4$
Cu	$54 \pm 3$
Li	(50)
Мо	$35 \pm 4$
Ni	$57 \pm 4$
Pb	$105 \pm 6$
Sb	43 ± 5
Sr	$142 \pm 11$
Tl	47 ± 5
V	$102 \pm 6$
Zn	$227 \pm 20$



P.O. Box 41727 Charleston, SC 29423

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## **Carbon Analysis**

<u>Reference Value</u>: The reference value is based on the results of 20 determinations by two different CNS Analyzers. The estimated uncertainties at the 95-percent confidence limits include those due to sample variation, possible method differences and errors of measurement.

<b>Reference Value for Loam Soil B</b>	
Element	Concentration (mg/g)
Carbon	7.7 ± 0.5

Theodore C Rains

Theodore C. Rains, Ph. D. President