U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 1579
Powdered Lead Based Paint

This Standard Reference Material is intended for use in the calibration of apparatus and methods used in the determination of lead in paint removed from the interior surfaces of old housing. The certified value is based on at least a 100 milligram sample of the as-received, total material.

Lead Content ..... 11.87 ± 0.04 Weight Percent

The certified value of 11.87 percent lead is the weighted average value determined by a statistical analysis of the results of 32 determinations by atomic absorption spectrometry (average 11.84 percent lead, s = 0.13 percent lead), and 16 determinations by polarography (average 11.93 percent lead, s = 0.13 percent lead). The standard error of the weighted average is 0.02 percent lead, and the half-width of the 95 percent confidence interval is taken to include ± 0.04 percent lead by weight.

X-ray fluorescence spectrometry showed the bottle-to-bottle inhomogeneity of the material with respect to lead content to be no greater than 0.02 percent lead; no within-bottle inhomogeneity was detected.

Analyses for lead and determinations of homogeneity were carried out in the NBS Analytical Chemistry Division by the following persons:

X-ray Fluorescence: S. D. Rasberry
Atomic Absorption Spectrometry: T. C. Rains and T. A. Rush
Polarography: E. J. Maienthal

Statistical calculations were carried out by J. Mandel of the NBS Institute for Materials Research.

The overall direction and coordination of the technical measurements leading to this certificate were performed under the chairmanship of B. Greifer.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
January 23, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

Preparation, Testing, and Analysis

Collection

The paint for this Standard Reference Material was collected by the staff of the Philadelphia Department of Public Health from the interior surfaces of dwellings undergoing renovation. The paint was softened with a hand torch, scraped from the plaster and wood substrates, and collected in plastic bags as a heterogeneous mixture of many different kinds of paints. In the laboratory, non-paint matter such as bits of metal, plastic, glass, and wood were removed and the paint mixture was ground in a disk mill to produce a material suitable for feeding into a jet mill. The paint was comminuted in a jet mill operating at 100 psig air pressure, then sieved through a 100-mesh vibrating screen to remove the coarse, non-grindable fraction. Two additional passes through the jet mill at 97 to 107 psig gave a fine powder with 99.31 weight percent passing through a 325 mesh sieve.

Homogeneity

Sample homogeneity was ascertained by x-ray fluorescence analysis for lead content on 17 samples chosen at random from the total lot. A statistical analysis of the data from 136 observations showed the bottle-to-bottle variability among the samples to be no greater than 0.02 percent lead. No within-bottle variation with respect to lead was detected.

Dissolution

A procedure used to dissolve the sample is summarized briefly: dry ash the weighed paint for 2 hours at 450 ° C, digest with 2:5 HCl - HNO_3 containing HF, evaporate to dryness; treat with HNO_3, evaporate to dryness; treat twice with HCl and evaporate to dryness each time. Extract the solids twice with portions of acetic acid - ammonium acetate solution, heating for several hours just below boiling. Combine the extracts and heat the mixture (including solids) for one hour, just below boiling. Cool the mixture and determine lead in solution. (The solids need not be removed for polarographic analysis.)

An alternate procedure for sample dissolution is: dry ash the weighed paint for 6 hours at 500 ° C, cool, then digest for 2 hours in 1:1 HCl - HNO_3. Separate the insoluble solids from the solution by centrifuging, and wash 3 times with 1:10 HNO_3 combining the rinsings with the principal solution. Determine lead in solution.

Details of the dissolution procedures, the analytical procedures, and results will be published in the 260 series of NBS Special Publications.