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Standard Reference Materials:

DEVELOPMENT OF NBS STANDARD REFERENCE MATERIAL: NO. 1579 POWDERED LEAD-BASED PAINT

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Standard Reference Materials:

**Development of
NBS Standard Reference Material No. 1579
Powdered Lead-Based Paint**

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DEVELOPMENT OF NBS STANDARD REFERENCE MATERIAL: 1579

POWDERED LEAD-BASED PAINT*

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The development of NBS Standard Reference Material 1579, Powdered Lead-Base Paint is described. This SRM is intended for use in the calibration of apparatus and methods used in determining lead in paint removed from old housing.

Paints scraped from the interior surfaces of old housing were collected, blended, homogenized, and characterized for lead content. The average lead concentration was found to be 11.87 percent \pm 0.04 percent lead by weight determined by atomic absorption spectrometry and by differential cathode-ray polarography.

The analytical procedures involved sample dry ashing in a furnace at 450-550 °C followed by exhaustive extractions of the insoluble residue with acids and with ammonium acetate solution.

Key words: Analytical standard; atomic absorption spectrometry; differential cathode-ray polarography; electrodeposition; lead-based paint; neutron activation analysis; x-ray fluorescence spectrometry; Standard Reference Material.

I. INTRODUCTION

This report describes the development of Standard Reference Material 1579, Powdered Lead-Based Paint developed as part of the Lead Paint Poisoning Project by the Analytical Chemistry Division, Institute for Materials Research. The

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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. It is representative of the types of samples collected in the field, and its homogeneity and proven lead content of 11.87 percent make it useful for comparing the performance of different analytical procedures, or for evaluating the measurements of different laboratories.

II. OBJECTIVE

The objective of this project task was the development of a standard reference material of known lead content which would represent the types of paint samples collected in the field from old housing, and which could provide a calibration capability to increase the reliability of lead determinations in dry, aged paint chips and scrapings.

III. RATIONALE FOR PAINT STANDARD REFERENCE MATERIAL (SRM)

A. Form of SRM

Originally, six types of materials were considered as possible Standard Reference Materials: (1) dried paint chips finely ground and sieved, mixed thoroughly to a homogeneous state for general laboratory use; (2) liquid paints formulated specifically for the desired lead content; (3) cast films of paint on suitable substrates for subsequent removal and analysis as free paint films; (4) dry paint powders such as the electrostatic spray powder paints; (5) pure pigments diluted to desired lead concentrations; and (6) painted panels suitably protected from abrasion by sophisticated packaging, for calibration of portable x-ray fluorescence instruments used in the field.

B. Desirable Characteristics of SRM

The material chosen to be a Standard Reference Material must be a paint of certified lead content which can be used to calibrate analytical instruments and techniques in a useful range of lead concentrations. It is desirable that this material be homogeneous, stable, and representative of samples taken for analysis. These characteristics are described quantitatively as follows:

1. Homogeneity: the sample-to-sample variation (i.e., bottle-to-bottle, panel-to-panel, etc.) should not exceed one percent of the total lead content.
2. Stability: the lead content should not change significantly with storage over a one-year period.
3. Representative of Field Samples: the material should have the same chemical properties as dried, aged (40-year old) lead-based paints overlayed with younger titanium-oxide paints.
4. Calibration Within Useful Range: the SRM should permit calibration in the expected range of lead concentrations (i.e., zero to 30 weight percent lead in loose paint chips or zero to 30 mg/cm² lead in paint in situ), with special consideration given to calibration near the legally significant concentrations 1.0 weight percent lead (or 1.0 mg/cm² lead in situ).

C. Choice of SRM Materials

Freshly prepared paint materials including liquid paints, cast dried films, and pure pigments differ from aged paint chips removed from the interior walls of old housing. For example, the lead-based pigments in fresh paints are easily soluble in acids or ammonium acetate solutions and can be analyzed by many methods in the laboratory [1]. On the other hand, dried aged paints are less soluble in acids (including nitric acid + perchloric acid mixtures) and may give erroneous results due to the diffi-

culty of extracting the lead from the paint matrix. In at least this one important respect it is clear that freshly prepared paint standards do not resemble the materials they are meant to simulate.

Liquid paints are difficult to maintain in a stable state after the containers are opened. Pigments can be expected to settle, surface skins form, and volatiles evaporate over a period of time, causing changes in the analysis. Dry paint powders of the type used for electrostatic spray painting would be more stable; these could be doped with lead-based pigment to suitable concentration levels, and blended to give homogeneous mixtures. However, no dry powder paints could be found to simulate linseed oil-based paints. Available materials such as epoxy-based dry powder paints did not resemble old paint samples sufficiently to be considered as Standard Reference Materials.

Painted panels containing zero to 30.0 mg/cm^2 lead were considered as a Standard Reference Material for calibration of portable x-ray fluorescence spectrometers used for field detection of lead on painted walls. However, the preparation of uniform panels posed considerable difficulty. After conversations with specialty painting contractors and painting equipment manufacturers it was concluded that the required degree of sample-to-sample uniformity could not be achieved either by paint spraying, dipping, or draw-down with a film applicator. In addition, a survey of six principal brands of portable x-ray fluorescence instruments for lead analysis revealed that while they were able to measure zero to 30.0 mg/cm^2 lead concentrations in coatings on walls and woodwork, the measurements of low lead concentrations were not reliable; the errors were as high as 50 percent at the 1.0 mg/cm^2 lead level [2]. If uniform panels could have been prepared, they would not have been useful for calibrating portable x-ray fluorescence spectrometers at or below the 1.0 mg/cm^2 lead level. For these two reasons, consideration of painted panels was deferred.

Dried paint chips collected from the old housing had the desirable properties of stability (already being 30 to 50 years old); they were truly representative of material collected in the field, and they could be made homogeneous with careful preparation. The paint chips had the disadvantage that their lead content probably was not near the legally significant 1.0 percent lead by weight. The material could not be diluted to a lower lead value because any diluent (e.g. titanium oxide) would have to be added in such a heavy quantity (e.g. 10 times the weight of paint) that its properties would override the characteristics of the paint. The best diluent, dried paint chips from housing free of lead-based paint, was not obtainable. Nevertheless, the potential advantages of using lead-based paint from old housing were considered to be great enough to warrant accepting the ambient lead value, knowing that solutions prepared from the material could be diluted to the desired concentrations during the course of the chemical analyses.

IV. COLLECTION AND PREPARATION OF PAINT MATERIAL

The paint for this standard reference material was collected by the staff of the Philadelphia, Pennsylvania Department of Public Health from the interior surfaces of dwellings undergoing renovation. The paint was softened with a hand torch and scraped from the plaster and wood substrates of walls and woodwork, and collected in plastic bags as a heterogeneous mixture of many different kinds of paints. In the laboratory, non-paint ingredients such as metal and plastic objects, glass, and wood shavings were removed, and the mixture ground in a disk mill to produce a material suitable for feeding into a jet mill. The paint was milled in a jet mill operating at 100 psi air pressure, then it was passed through a 100-mesh vibrating screen to

remove the coarse non-grindable fraction, mostly wood shavings. Two additional passes through the jet mill at 97 to 107 psi gave a powder whose final retention on a 325 mesh screen was less than 0.69 weight percent. A total of 125 pounds was prepared.

Four hundred and seventy-five bottles, numbered one through 475 were prepared from one drum of material, loading from the top down through about 100 pounds of material. The remaining 25 pounds were reserved for future use.

V. TESTING OF PAINT MATERIAL

Sample homogeneity was investigated by x-ray fluorescence analysis (XRF) for lead content on 17 bottles chosen at random from the total lot. The bottles were numbered one through 475 with increasing numbers representing successive layers (slices) from the top to the bottom of the drum of powdered paint. The random selection chose bottles numbered 19, 39, 65, 107, 131, 172, 207, 233, 258, 271, 315, 346, 376, 419, 444, 472, and 473.

X-ray fluorescence spectrometry gives very precise results which are ideal for homogeneity testing but it cannot determine accurately the amount of lead without suitable calibration. For determining the homogeneity of a material it is common to accept the readings in uncalibrated arbitrary units since only the variation of the readings from sample to sample is of interest.

Two samples from each bottle were poured into spectro-cups which were fitted with mylar windows. Each of the 34 samples thus obtained was analyzed four times with a general-purpose single-channel x-ray fluorescence spectrometer. The results of the tests in terms of averages of four measurements are shown in Table 1. The lead content is expressed in a scale of arbitrary units which has been normalized so as to give an overall average value of 10.00 units. In this arbitrary scale the data ranged between 9.95 and 10.05 units.

Table 1. Homogeneity Testing of Powdered Paint
by X-Ray Fluorescence Spectrometry

| Bottle Order | Measured Lead Content ^a (arbitrary units) | |
|-----------------|---|--------------------|
| | Sample 1 | Sample 2 |
| 1 | 10.00 ^b | 10.02 ^b |
| 2 | 10.00 | 10.04 |
| 3 | 9.99 | 10.04 |
| 4 | 10.04 | 10.02 |
| 5 | 10.01 | 10.00 |
| 6 | 10.02 | 10.02 |
| 7 | 10.02 | 10.00 |
| 8 | 10.02 | 10.05 |
| 9 | 10.01 | 10.00 |
| 10 | 10.00 | 10.02 |
| 11 | 9.99 | 10.00 |
| 12 | 9.97 | 9.98 |
| 13 | 9.98 | 9.97 |
| 14 | 9.98 | 10.00 |
| 15 | 9.98 | 9.98 |
| 16 | 9.96 | 9.99 |
| 17 | 9.95 | 9.96 |

Average: 10.00 units

^aThe analysis measures the lead content in arbitrary units and shows the bottle-to-bottle variation among 17 randomly selected samples of dry powdered paint, in descending order from top to bottom in the bulk material.

^bValues in this column are averages of 4 measurements.

A trend may be observed, showing lower values for samples originating from the lower part of the bulk material container.*

A statistical analysis of the data calculated from all 136 measurements (rather than the 34 averages of four measurements listed in Table 1) lead to the following conclusions:

- a) The standard deviation of measurement error (i.e., of variability among replicate measurements made on the same sample) was 0.034 unit.*
- b) No evidence of heterogeneity of the material within bottles was detected.
- c) A significant amount of bottle-to-bottle variability was found, as evinced by the trend shown by the averages in Table 1. However, the magnitude of this variability was small; expressed as a standard deviation among bottles it was equal to 0.02 unit.*

The material was considered to be sufficiently homogeneous for use as a Standard Reference Material.

VI. ANALYSIS OF PAINT MATERIAL

A. Design of Experiment

The chemical analysis for lead was carried out on 16 bottles arranged in a four-by-four Latin Square design experiment in which four samples were taken at random from each of four randomly selected bottles. The four bottles for analysis were chosen by a double randomization process from the series of bottles used for homogeneity testing (the order of the 17 bottles was randomized and bottles numbered 65, 233, 376, and 444 were chosen at random from this random listing). The design of the Latin Square experiment is shown in Table 2.

*Subsequent chemical analysis showed the paint to contain 11.87 percent lead by weight. The XRF measurements, converted to percent lead by multiplying by the factor 11.87/10.00, fall between 11.81 and 11.93 percent lead; the standard deviation of measurement error is 0.04 percent lead; and the bottle-to-bottle variation is 0.02 percent lead.

Because of the great variety of paint pigments expected in the samples, some of them insoluble in acids (e.g. PbSO_4) special care was taken to insure that all the lead in the material was dissolved completely. As a special precaution the insoluble residues from both the atomic absorption spectrometry and the polarographic dissolution procedures were spot checked by emission spectrographic analysis to make sure that they did not contain a significant quantity of lead.

Table 2. Latin Square Design for Analysis of Four Bottles 65, 233, 376, 444

| Order Within Analysis | Bottle Number | | | |
|-----------------------------|---------------|---------------|---------------|---------------|
| | Analysis 1 | Analysis 2 | Analysis 3 | Analysis 4 |
| 1 | 233 | 376 | 65 | 444 |
| 2 | 65 | 444 | 233 | 376 |
| 3 | 376 | 233 | 444 | 65 |
| 4 | 444 | 65 | 376 | 233 |

B. Atomic Absorption Spectrometry (AAS)

Lead was determined by atomic absorption spectrometry in duplicate determinations on the 16 bottles, according to the experimental design shown in Table 2.

1. Analytical Procedure for AAS

Transfer 0.5 g of sample to a porcelain crucible and ignite in furnace at 550 °C for 6 hours. Cool and transfer the test portion to a 250-ml Vycor beaker. Add 10 ml of high-purity HNO_3 [3] and 10 ml of high-purity HCl [3] and digest for 2 hours, or until the sample volume is approximately 3 ml. Transfer the sample to a 50-ml centrifuge tube and centrifuge at 2500 rpm for 10 minutes. Decant the supernatant solution into a 100-ml volumetric flask. Wash the solids with .15 ml of 1:10 HNO_3 and centrifuge at 2500 rpm for 10 minutes. Decant the supernatant solution into the volumetric flask. Repeat the washings of solids two additional times. Combine the washings with the sample

solution and dilute to the calibrated volume. Determine lead by atomic absorption spectrometry at a wavelength of 283.3 nm using a slightly fuel-rich acetylene-air flame. Calibrate the instrument with a series of solutions prepared from lead of high purity (e.g. NBS SRM 49e).

2. Results of AAS Analyses

The results are shown in Table 3. The average of 32 determinations was 11.84 percent lead by weight. A statistical analysis of the data in accordance with the Latin Square design led to the following conclusions:

- a) The standard deviation of measurement error (exclusive of bottle-to-bottle variability and of systematic run-to-run shifts) was 0.13 percent lead.
- b) A small amount of bottle-to-bottle variability was noted; the standard deviation of variability among bottles was an additional 0.13 percent lead.
- c) A significant but small systematic shift was observed between the two series of AAS measurements. This shift, averaged over all determinations, was 0.009 percent lead.

In addition to the 32 analyses reported in Table 3, four additional lead determinations were carried out on samples numbered 65, 233, 376, and 444 as a part of Analysis number 4. These results, reported in Appendix A, were not a part of the Latin Square experiment and were not included in the statistical calculations leading to the certified value for lead.

C. Polarography

Lead was determined by polarographic analysis of 16 samples taken from the same bottles used for AAS analysis. The samples were analyzed singly according to the same experimental design, Table 2.

Table 3. Lead Determination by Atomic Absorption Spectrometry

| <u>Analysis Number</u> | <u>Bottle Number</u> | <u>Lead, wt. Percent</u> | |
|------------------------|----------------------|--------------------------|--------------|
| | | <u>Run 1</u> | <u>Run 2</u> |
| 1 | 233 | 11.99 | 12.12 |
| | 65 | 11.83 | 11.89 |
| | 376 | 11.66 | 11.69 |
| | 444 | 11.78 | 11.68 |
| 2 | 376 | 11.87 | 11.86 |
| | 444 | 11.76 | 11.91 |
| | 233 | 11.98 | 12.07 |
| | 65 | 11.93 | 12.14 |
| 3 | 65 | 11.79 | 11.96 |
| | 233 | 11.76 | 11.93 |
| | 444 | 11.64 | 11.70 |
| | 376 | 11.70 | 11.65 |
| 4 | 444 | 11.57 | 11.80 |
| | 376 | 11.80 | 12.13 |
| | 65 | 11.78 | 11.74 |
| | 233 | 11.93 | 11.98 |

Average of 32 determinations: 11.84% lead by weight

1. Analytical Procedure for Polarography [4]

Shake the bottle for one minute, then weigh 100 milligram portions into a Vycor crucible and ignite for 2 hours at 450 °C. Treat the ash with 2 ml high purity HCl [3], 5 ml high purity HNO_3 [3], and 5 drops high purity HF [3] and evaporate to dryness. Add 5 ml HNO_3 and evaporate to dryness. Evaporate to dryness twice more with the addition of 2 ml HCl each time. Treat the solids with 30 ml of acetic acid - ammonium acetate solution (100 ml acetic acid + 95 ml NH_4OH + 100 ml H_2O), cover, and heat for several hours just below boiling. Decant the supernatant solution into a 400-ml beaker. Add 25 ml more of the acetic acid - ammonium acetate solution to the solids in the crucible and heat again just below boiling. Combine all the crucible contents with the original solution, rinsing the crucible well with water. Heat the combined solution just below

boiling for about one hour, cool, and transfer quantitatively to a 200-ml volumetric flask. Dilute to calibrated volume.

Transfer a 5.0 ml portion to a polarographic cell, deaerate, and measure the lead reduction peak at about -0.7 volts against a mercury pool anode, with a differential cathode ray polarograph against a blank in the second cell which has been carried through the entire procedure. Standard solutions prepared from melting point lead (NBS SRM 49d) are carried through the procedure to establish a calibration curve.

2. Results of Polarographic Analyses

The results are shown in Table 4. The average of 16 determinations was 11.93 percent lead by weight. The standard deviation of measurement error, exclusive of possible bottle-to-bottle variability, was calculated to be 0.13 percent lead.

No bottle-to-bottle variation in lead content was detected by polarography.

Table 4. Lead Determination by Polarography

| <u>Analysis Number</u> | <u>Bottle Number</u> | <u>Lead Wt. Percent</u> |
|----------------------------|--------------------------|-----------------------------|
| 1 | 233 | 11.91 |
| | 65 | 11.99 |
| | 376 | 12.08 |
| | 444 | 11.93 |
| 2 | 376 | 12.00 |
| | 444 | 11.83 |
| | 233 | 11.74 |
| | 65 | 11.99 |
| 3 | 65 | 11.98 |
| | 233 | 11.65 |
| | 444 | 11.99 |
| | 376 | 12.02 |
| 4 | 444 | 12.02 |
| | 376 | 11.68 |
| | 65 | 12.00 |
| | 233 | 12.05 |

Average of 16 determinations: 11.93% lead by weight

In addition to the 16 analyses reported in Table 4, four additional lead determinations were carried out on sample number 65 before the beginning of the Latin Square experiment. These results, reported in Appendix B, were not included in the statistical calculations leading to the certified value for lead.

D. Calculation of Combined Data

The results of 32 AAS and 16 polarographic analyses are summarized in Table 5. The difference between the AAS results (11.84 percent lead) and the polarographic results (11.93 percent lead) was determined to be on the borderline of significance by a t-test calculation ($t = 2.26$), using the estimates of the standard deviations of measurement error obtained from the two Latin Square experiments.

The average value, weighted for 32 AAS determinations and 16 polarographic determinations was 11.87 percent lead by weight. The standard error of the weighted average was 0.02 percent, hence the approximate 95 percent confidence interval for the average was ± 0.04 percent lead by weight.

The average value of lead for the entire lot was taken to be:

11.87 percent \pm 0.04 percent lead by weight.

Table 5. Lead Content of Powdered Paint:
Summary of Results^a

| Bottle Number | Weight Percent Lead ^b | |
|------------------|---------------------------------------|---------------------------------------|
| | AAS ^b 32 Determinations | POL ^b 16 Determinations |
| 65 | 11.88 | 11.99 |
| 233 | 11.97 | 11.84 |
| 376 | 11.80 | 11.94 |
| <u>444</u> | <u>11.73</u> | <u>11.94</u> |
| Average | 11.84 | 11.93 |

Standard deviation
for an individual measurement:^c

| | |
|--------------------------------------|--------------------------------------|
| $\hat{\sigma} = 0.13\% \text{ lead}$ | $\hat{\sigma} = 0.13\% \text{ lead}$ |
|--------------------------------------|--------------------------------------|

Standard error of
averages:

| | |
|--------------------------|--------------------------|
| $= 0.023\% \text{ lead}$ | $= 0.032\% \text{ lead}$ |
|--------------------------|--------------------------|

t test: $t = 2.26$

Weighted average value: 11.87% lead

Standard error of
weighted average : 0.02% lead

Approximate 95 percent
confidence interval : $11.87 \pm 0.04\% \text{ lead}$

^aThese are the results of 48 lead determinations carried out according to the experimental design shown in Table 2. The results of 56 lead determinations are summarized in Appendix C.

^bSample averages calculated from data in Tables 3 and 4.

^cEstimates of standard deviations of measurement error obtained from Latin Square experiments.

VII. MISCELLANEOUS ANALYSES

The powdered lead-based paint was analyzed by electrolytic deposition and by neutron activation analysis as a further check on the lead content.

A. Electrolytic Deposition

Follow the AAS dissolution procedure to the point where the solution is ready to be measured. Take 25 ml aliquots of this solution and evaporate to dryness with HNO_3 , twice, to destroy the HCl . Dissolve the solid residue in 3 percent HNO_3 (10 ml HNO_3 + 325 ml H_2O) and electrolyze with platinum electrodes at 0.4 ampere for at least 5 hours, or for overnight if more convenient. After electrolysis remove the platinum anode, dry for one hour at 135 °C, cool and weigh. The weight increase is calculated as PbO_2 .

The results are summarized in Table 6. The average value of lead in 3 bottles was 11.92 percent by weight.

Table 6. Lead Determination by Electrolytic Deposition

| <u>Bottle Number</u> | <u>Lead, Weight Percent</u> | |
|--------------------------|--|---|
| | <u>5-Hour Electrolysis^a</u> | <u>Overnight Electrolysis^a</u> |
| 233 | 12.13 | 12.06 |
| 376 | 11.69 | 11.83 |
| 444 | 11.93 | 11.86 |

Average of all results: 11.92% lead by weight

^a0.40 ampere, platinum electrodes.

B. Neutron Activation Analysis

Neutron activation analysis was carried out on 4 samples taken from the bulk material remaining after bottling. The procedure that was used employed the $^{204}\text{Pb} (n,n') ^{204\text{m}}\text{Pb}$ reaction. Four samples, 1.6 g each and four standards, reagent grade lead acetate were irradiated for 2 hours with a 600-microgram ^{252}Cf Californium source. The gamma ray doublet of $^{204\text{m}}\text{Pb}$ (half-life 67 minutes) at 0.91 MeV was counted for 100 minutes using a 60 ml Ge(Li) detector.

The results for 4 samples were: 10.7, 10.6, 11.7 and 11.4 percent lead by weight. The average value was 11.1 percent lead.

VIII. SUMMARY AND CONCLUSIONS

The development of Standard Reference Material 1579, Powdered Lead-Based Paint is described. This SRM is intended for use in calibration of apparatus and methods used in determining lead in paint from old housing. The certificate issued with the material is reproduced in Appendix C. Paints scraped from the interior surfaces of houses undergoing renovation have been collected, blended, homogenized, and characterized for lead content. The average lead concentration is 11.87 percent \pm 0.04 percent lead by weight, as analyzed by atomic absorption spectrometry and polarography and corroborated by electrolytic deposition and by neutron activation analysis.

The uncertainty expressed in the above result is the half-width of the 95 percent confidence interval, based on a total of 48 measurements. Analysis by x-ray fluorescence spectrometry showed the homogeneity of the material with respect to lead to be within the certification limits.

This material is considered to be more representative of old, dried-out, intractable paints collected in the field than freshly prepared paint formulations containing precisely measured amounts of lead as a single pigment or a mixture of pigments. Two recommended sample dissolution procedures involve dry ashing in a furnace at 450 to 550 °C followed by digestion of the ash with HCl-HNO₃ mixtures, and exhaustive extraction of the insoluble residue with either dilute HNO₃ (AAS analysis) or with HCl and with ammonium acetate solutions (polarographic analysis).

IX. RECOMMENDATIONS FOR FUTURE WORK

The powdered lead-based paint may contain traces of toxic heavy metals other than lead. Some metals of concern which should be investigated in future work are antimony, arsenic, cadmium, mercury, selenium, and soluble barium.

The search for a suitable diluent, in the form of dried, old, lead-free paint chips should be continued with the thought of blending the as-received lead based paint to a lower lead concentration in the 1.0 percent or the 0.06 percent range. This could increase the SRM's value to the analytical chemist.

The search for a procedure to prepare uniform standard reference painted panels should be continued, against the time when portable x-ray fluorescence spectrometers may be improved to where they can be calibrated at the 0.06 to 1.0 mg/cm² lead level.

X. REFERENCES

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Sample Collection was handled by Mr. Edward Wiener, Assistant Chief, Accident Control Section, Department of Public Health, City of Philadelphia, 500 South Broad Street, Philadelphia, Pa. 19146.

Sample Grinding was carried out by Frank and James Albus, Alnort, Inc., 2430 Broadway, Camden, N. J. 08104.

Homogeneity determination was carried out by X-ray Fluorescence: S. D. Raspberry, Analytical Chemistry Division.

Analyses for lead were carried out by the following methods:

Atomic Absorption Spectrometry: T. C. Rains and T. A. Rush, Analytical Chemistry Division.

Polarography: E. J. Maienthal, Analytical Chemistry Division.

Electrolytic Deposition: R. K. Bell, Analytical Chemistry Division.

Neutron Activation: G. J. Lutz, Analytical Chemistry Division.

Statistical calculations were carried out by J. Mandel, Institute for Materials Research.

APPENDIX A.

LEAD DETERMINATION BY ATOMIC ABSORPTION SPECTROMETRY:

Results of 36 determinations

| <u>Analysis Number</u> | <u>Bottle Number</u> | <u>Lead, Wt. Percent</u> | | |
|------------------------|----------------------|--------------------------|--------------|--------------------------|
| | | <u>Run 1</u> | <u>Run 2</u> | <u>Run 3^a</u> |
| 1 | 233 | 11.99 | 12.12 | |
| | 65 | 11.83 | 11.89 | |
| | 376 | 11.66 | 11.69 | |
| | 444 | 11.78 | 11.68 | |
| 2 | 376 | 11.87 | 11.87 | |
| | 444 | 11.76 | 11.91 | |
| | 233 | 11.98 | 12.07 | |
| | 65 | 11.93 | 12.14 | |
| 3 | 65 | 11.79 | 11.96 | |
| | 233 | 11.76 | 11.93 | |
| | 444 | 11.64 | 11.70 | |
| | 376 | 11.70 | 11.65 | |
| 4 | 444 | 11.57 | 11.80 | 11.53 |
| | 376 | 11.80 | 12.13 | 11.74 |
| | 65 | 11.78 | 11.74 | 11.66 |
| | 233 | 11.93 | 11.98 | 11.77 |

Average of 36 determinations: 11.83% lead by weight

^aResults of 4 extra measurements, not a part of the Latin Square experiment, and not included in the statistical calculations leading to the certified value for lead.

APPENDIX B. LEAD DETERMINATION BY POLAROGRAPHY:

Results of 20 determinations

| <u>Analysis Number</u> | <u>Bottle Number</u> | <u>Lead, Wt. Percent</u> |
|------------------------|----------------------|--------------------------|
| -- | 65 | 12.04 ^a |
| | 65 | 12.09 ^a |
| | 65 | 11.95 ^a |
| | 65 | 11.98 ^a |
| 1 | 233 | 11.91 |
| | 65 | 11.99 |
| | 376 | 12.08 |
| | 444 | 11.93 |
| 2 | 376 | 12.00 |
| | 444 | 11.83 |
| | 233 | 11.74 |
| | 65 | 11.99 |
| 3 | 65 | 11.98 |
| | 233 | 11.65 |
| | 444 | 11.99 |
| | 376 | 12.02 |
| 4 | 444 | 12.02 |
| | 376 | 11.68 |
| | 65 | 12.00 |
| | 233 | 12.05 |

Average of 20 determinations: 11.95% lead by weight

^aResults of 4 extra measurements, not a part of the Latin Square experiment, and not included in the statistical calculations leading to the certified value for lead.



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% lead is the weighted average value determined by a statistical analysis of the results of 32 determinations by atomic absorption spectrometry (average 11.84% lead, $s = 0.13\%$ lead), and 16 determinations by polarography (average 11.93% lead, $s = 0.13\%$ lead). The standard error of the weighted average is 0.02% lead, and the half-width of the 95% confidence interval is taken to include $\pm 0.04\%$ 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% 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
September 29, 1972

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% 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-HNO₃ containing HF, evaporate to dryness; treat with HNO₃, 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-HNO₃. Separate the insoluble solids from the solution by centrifuging, and wash 3 times with 1:10 HNO₃ 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.

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