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National Institute of Standards and Technology
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ABSTRACT

A pilot study was conducted for the U.S. Department of Housing and Urban Development (HUD) to measure the lead concentrations in a small sampling of new consumer paints. Although a Consumer Product Safety Commission Regulation requires that the lead concentration be no greater than 0.06 percent (600 parts per million, ppm or 600 $\mu\text{g/g}$) by mass of paint solids, the actual lead concentration is not usually measured and reported. Estimates of expected lead concentrations in new paint are needed in HUD's lead-paint abatement program. Thus, the objective of this pilot study was to determine whether the lead concentration in a small sampling of new paints tended to be near the regulatory limit. The lead concentration in each of 31 consumer paints was measured using laboratory x-ray fluorescence spectrometry. All concentration estimates were less than 100 ppm. The lead concentration of most samples was below the detection limit of the procedure used of 30 ppm.

KEYWORDS: Consumer paint; Lead concentration; X-ray fluorescence spectrometry

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1. INTRODUCTION

At the request of the U.S. Department of Housing and Urban Development, the lead concentrations in a small sampling of new consumer paints were measured. Although the Consumer Product Safety Commission (CPSC) Regulation, 16 CFR 1303 [1], requires that the lead concentration be no greater than 0.06 percent (600 parts per million) by mass of paint solids in paint, there is little information on the actual lead concentration in consumer paints. The objective of this pilot study was to determine whether the lead concentrations of new paints tended to be near the regulatory limit. This information is needed to help refine abatement procedures and recommendations. A description of the paints, the test method, and the results of the lead concentrations measurements are presented below.

2. MATERIALS

In this pilot study, a small sampling of consumer paints was selected at random for determining lead concentration. The paints were manufactured by nine major producers of paint and were obtained from local retail stores. Interior and exterior, and water-based and oil-based paints were included. Since more water-based than oil-based paints are used by consumers, the number of water-based paints selected was about twice that of the oil-based paints. Interior paints tested were white or tint bases. For the most part, exterior products were deep-tone earth colors with the colored pigments incorporated into the paints at the factory. The gloss varied from flat to high. The selection procedure was based on an incomplete random block design. The blocks or groups were oil-based paint, interior water-based paint and exterior water-based paint. This design provided for one paint of each type from each manufacturer being included in the study. Since each manufacturer makes many types of paint, further procedures for selecting a specific product were needed. It was assumed that each company had at least two quality grades and four tint levels (or colors for exterior paints) for each type of paint. Within each block (group) of paints, the grade and tint level of each paint to be purchased from a manufacturer were randomly chosen from this group of eight expected products. In addition, an additional interior water-based and exterior water-based paint were obtained from each of two larger manufacturers. For the oil-based paints, an arbitrary decision was made to sample five exterior paints and four interior paints; for a given manufacturer, the type of paint (interior or exterior) was chosen at random. Thus, the total number of paints tested was 31 (nine oil-based paints - five exterior and four interior, and 22 water-based paints - eleven exterior and eleven interior).

The merchant's recommendations were used to rank the quality of a particular manufacturer's paints and to select an appropriate tint base. In some situations, the manufacturer did not supply the complete range of paints. In these situations, the paint closest to the one described in the design was selected. For example, if the manufacturer did not have a deep-tone tint base, a medium-tone tint base was selected. Descriptions of the paints tested in this study are given in Table 1.

Table 1. Description of Paints Included in the Study

Sample Number	Generic Type	Description
1	Latex	White interior tint base
2	Latex	Green exterior, factory colored
3	Oil	Pastel interior tint base
4	Latex	White interior tint base
5	Latex	Deep-tone exterior tint base
6	Oil	Deep-tone interior tint base
7	Latex	Green exterior, factory colored
8	Latex	White interior tint base
9	Oil	Brown exterior, factory colored
10	Latex	Green exterior, factory colored
11	Latex	Pastel interior tint base
12	Latex	Intermediate interior tint base
13	Latex	Brown exterior, factory colored
14	Oil	White exterior tint base
15	Latex	Deep-tone interior tint base
16	Latex	Intermediate exterior tint base
17	Oil	White interior tint base
18	Latex	Intermediate interior tint base
19	Latex	Exterior intermediate tint base
20	Latex	Interior intermediate tint base
21	Oil	Exterior intermediate tint base
22	Latex	Deep-tone interior tint base
23	Latex	Brown exterior, factory colored
24	Oil	Green exterior, factory colored
25	Latex	Brown exterior, factory colored
26	Latex	White interior tint base
27	Oil	White interior tint base
28	Oil	Green exterior, factory colored
29	Latex	Dark exterior tint base
30	Latex	Brown exterior, factory colored
31	Latex	Pastel interior tint base

3. EXPERIMENTAL PROCEDURE

3.1 Mass Fraction of Solids

The mass fraction of solids in each of the paints was determined from the mean of triplicate specimens using ASTM D 2369 [2]. In this method, the paint is first thoroughly mixed and then a small amount placed in a tared aluminum dish. The dish with the paint is weighed and the paint is diluted with an appropriate solvent. The diluted paint is distributed over the bottom of the pan to form a smooth thin film. The specimen is baked at 110°C in an air-circulating oven for 1 hour. After cooling to room temperature in a desiccator, the sample is weighed to determine the solids content.

3.2 XRF Measurement of Lead Concentration

The lead content of each paint was measured in duplicate using a wavelength-dispersive x-ray fluorescence (XRF) spectrometer with a Mo tube operated at 60 kV, 50 mA; a LiF (200) crystal; and fine collimators. Measurements were made in helium. X-ray intensities were measured at three angles, at the lead L_{α} and on either side. Angles for each of the measurements were selected from data obtained from scans over the appropriate range of a paint sample to which lead nitrate had been added. The lead peak intensity was corrected for background by subtracting the background intensity. This background intensity was calculated from the straight line drawn between the intensities on either side of the peak and evaluated at the lead L_{α} peak position. Counting time for both background and peak intensities was 100 s.

Specimens for the XRF analyses were prepared by pouring well-mixed samples of paint into liquid XRF cups, having a depth of 20 mm. This sample depth provides a sample having essentially infinite thickness (defined in this paper as a sample that yields at least 99% of the fluorescence of an infinitely thick specimen). This thickness can be calculated from the mass attenuation coefficient, the density of the specimen and the instrumental parameters [3]. For the paints used in this study, this thickness is about 2 mm.

The spectrometer was calibrated using samples of a paint to which known masses of a 1000 parts per million (ppm or $\mu\text{g/g}$) standard lead nitrate solution had been added. Four samples were prepared; one with no added lead and three having lead concentrations of about 25, 50, and 100 ppm by mass of the liquid paint.

4. RESULTS

4.1 Calibration

The calibration curve obtained from a linear regression of XRF measurements of known masses of lead added to a paint is shown in Figure 1. Data taken on three successive days were used in the regression. The abscissa is the concentration of added lead in the liquid

paint, while the ordinate is the number of kcounts/s in the lead peak (corrected for background). The parameters of the linear regression are: slope = 14.1 kcounts/s/ppm, estimate of standard error of the slope = 0.860 kcounts/s/ppm; intercept = 58.3 kcounts/s, estimate of the standard error of the intercept = 58.3 kcounts/s; and square of correlation coefficient = 0.993.

4.2 Lead Concentration in New Paints

The lead concentrations in the paints included in this pilot study based on mass of paint solids are shown in Figure 2. The lead concentration based on mass of paint solids was determined for each paint by obtaining the lead concentrations of the duplicate liquid paint specimens from the calibration curve (Figure 1), calculating the mean and dividing it by the fraction of paint solids. The best estimates of lead concentration are shown for all samples, even though many are below the analyte detection limit, which was estimated to be about 30 ppm for the specific measurement procedures used in this study [4]. (The estimate was based upon the calibration data and the mathematical procedures described in the appendix of Currie's paper [4] using a limit of 0.05 for false-negative and false-positive decision probabilities.)

The major experimental error of this measurement method is associated with the assumption that the matrix effect of all the paints included in the study was similar to the paint used in the calibration. This error is much larger than those related to the random nature of the XRF interaction process, inhomogeneity of the paint sample, and determination of mass of solids of the paints. The matrix effect includes attenuation of primary x-rays and fluoresced lead x-rays by the matrix and depends upon the mass attenuation coefficient of the material. The mass attenuation coefficient is defined as

$$\mu(E) = \sum W_i \mu_i(E),$$

where W_i is the weight fraction of element i in the specimen, and $\mu_i(E)$ is the total mass attenuation coefficient of element i at energy E . The summation over i includes all elements in the specimen such that $\sum W_i = 1$.

To obtain an estimate of the size of the error associated with the matrix effect, it was assumed that the x-ray fluorescence intensity is inversely proportional to the mass attenuation coefficient. (This approximation is based on the assumption that the mass attenuation coefficient of the material at the energy of the Pb (lead) L_α is much greater than at the energy of the Mo (molybdenum) K_α [3]). Thus, attenuation coefficients were calculated for 10 kV, near the Pb L_α energy, for the paint used in the calibration and for each of the paints included in this study, for which the composition was described on the label. In addition, the attenuation coefficients were calculated for several raw-material suppliers suggested formulations [5, 6, 7]. Data from McMaster was used in calculating the mass attenuation coefficients [8]. A range of values of mass attenuation coefficients from 0.5 to 1.7 of the paint used in the calibration was obtained. Mass coefficients for model formulations [6,7,8]

were also in the above range for paint formulations having no barium and limited amounts of zinc. (Based upon the label information and energy dispersive x-ray analysis of the paints having no label information, none of the samples contained barium, and only two contained even a small amount of zinc.) Since the fluoresced x-ray intensity is approximately proportional to the mass attenuation coefficient, the change in the intensity due to possible matrix effects can vary by as much as a factor of 3.

This estimate of the possible error associated with the matrix effect and the uncertainty associated with the calibration curve [4] were used to estimate the 95 percent confidence intervals for the lead concentrations as shown in Figure 2. The intervals were determined by calculating the lead concentration corresponding to the greatest potential matrix affects (i.e., 0.5 and 1.7) and subtracting 20 ppm from the lower limit and adding 20 ppm to the upper one.

As a partial check of some of these values, a different paint was used for the calibration specimens, and three paints were reanalyzed. A comparison of values for lead concentration obtained for these paints is shown in Table 2. In each case the measured value fell within the confidence interval of the results shown in Figure 2.

Table 2. Comparison of Repeated Measurements of Lead Concentrations of Four Paints

Sample Number	[Pb], ppm 1st meas.	[Pb], ppm 2nd meas.
24	103	82
27	49	58
10	nd(0)	nd(7)
7	nd(0)	nd(0)

nd = not detected; the best [Pb] estimate is in parentheses

5. CONCLUSIONS

The laboratory x-ray fluorescence method is suitable for determining lead concentrations in the range of the CPSC regulatory limit in liquid paint samples. Variabilities in the results due to the matrix effect would be reduced by using an internal standard, e.g., strontium, and the use of an internal standard is recommended for further measurements of this type.

The lead concentrations in all the paints included in this pilot study were considerably less than the regulatory limit of 600 ppm (0.06%). All of the lead concentrations were less than 100 ppm and many were below the detection level, 30 ppm, of the specific method used. Further, based upon the analysis of errors, the 95 percent confidence intervals for the lead concentrations in all the paints was less than 100 ppm, except for one paint, Number 24.

The true lead concentration of this paint is likely less than the amount based on the calibration curve. This is because the mass attenuation coefficient for the paint, calculated using label information, is lower than that of the paint used in the calibration.

6. ACKNOWLEDGEMENTS

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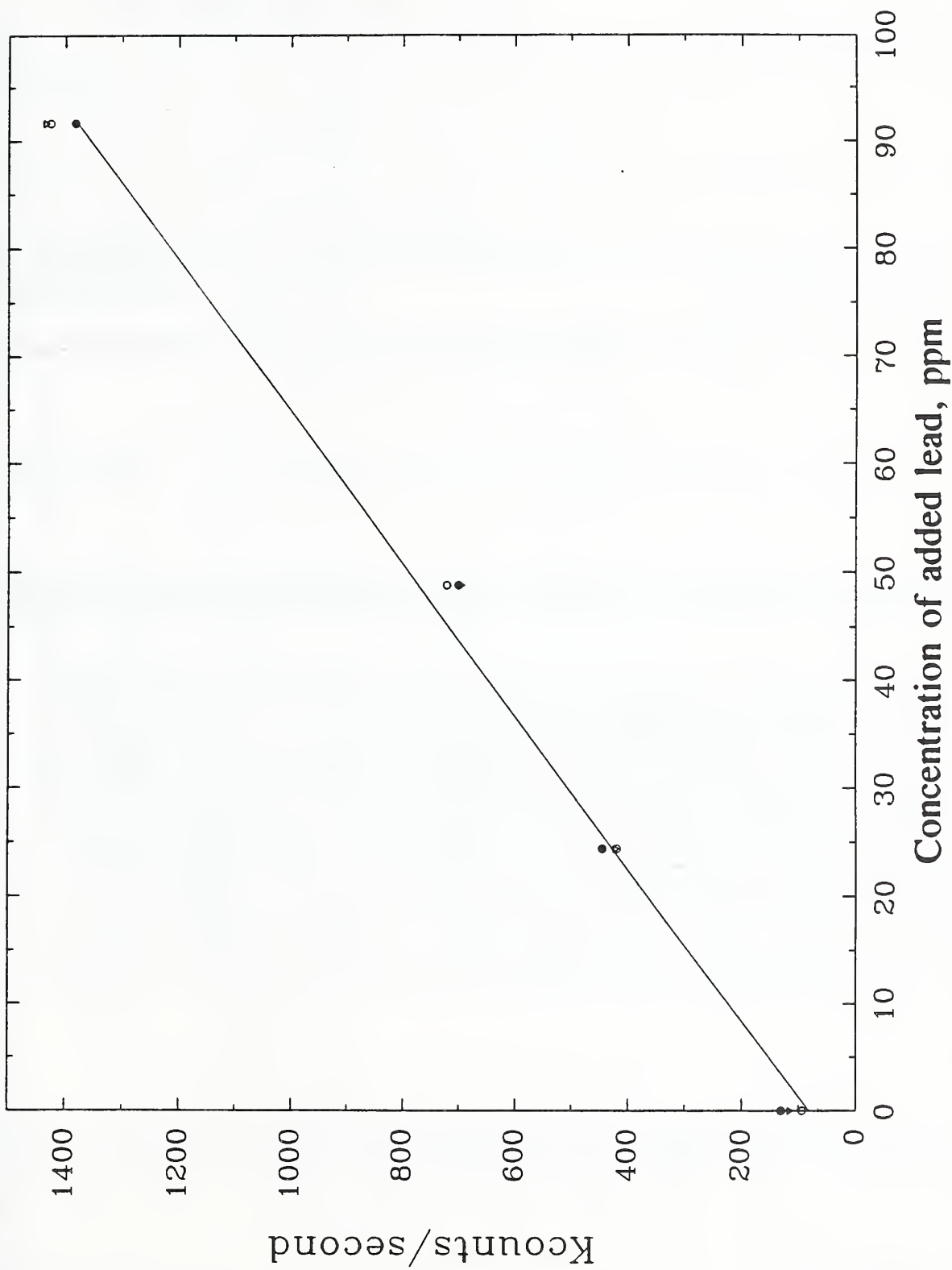


Figure 1. Concentration of added lead in parts per million (ppm or $\mu\text{g/g}$) of liquid paint vs x-ray intensity. Specimens were analyzed on each of three days, ● - Day 1, ▼ - Day 2, and ▲ - Day 3. The solid line is the linear regression of all the points.

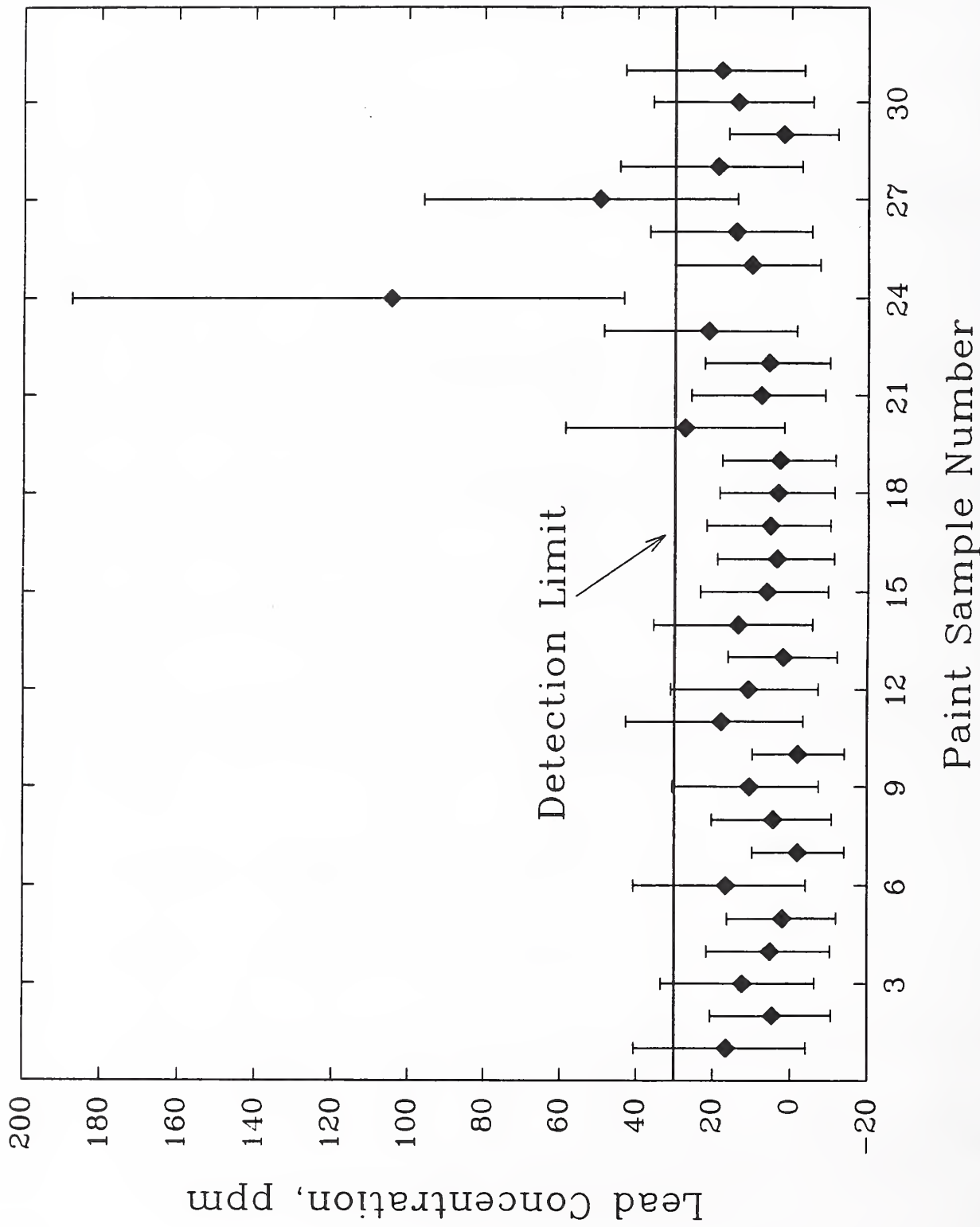


Figure 2. Lead concentration in a small sampling of consumer paints by mass of paint solids; the brackets indicate 95 percent confidence intervals.

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A pilot study was conducted for the U.S. Department of Housing and Urban Development (HUD) to measure the lead concentrations in a small sampling of new consumer paints. Although a Consumer Product Safety Commission Regulation requires that the lead concentration be no greater than 0.06 percent (600 parts per million, ppm or 600 $\mu\text{g/g}$) by mass of paint solids, the actual lead concentration is not usually measured and reported. Estimates of expected lead concentrations in new paint are needed in HUD's lead-paint abatement program. Thus, the objective of this pilot study was to determine whether the lead concentration in a small sampling of new paints tended to be near the regulatory limit. The lead concentration in each of 31 consumer paints was measured using laboratory x-ray fluorescence spectrometry. All concentration estimates were less than 100 ppm. The lead concentration of most samples was below the detection limit of the procedure used of 30 ppm.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

Consumer paint; Lead concentration; X-ray fluorescence spectrometry

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