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Office of Healthy Homes and Lead Hazard Control

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ABSTRACT

A laboratory study was conducted to evaluate the reliability of commercial, field-portable ultrasonic extraction-anodic stripping voltammetry (UE/ASV) for determining the lead levels of laboratory-prepared paint films when tests were performed by certified lead inspectors trained to conduct UE/ASV testing. Two factory-calibrated UE/ASV apparatuses from the same supplier were purchased and used to conduct an experiment investigating the effects of lead level, apparatus, lead pigment type, operator, paint-film substrate, and overlayer applied to the lead-based paint film. Test panels, with either white lead (i.e., basic lead carbonate) or lead chromate pigments, had 10 lead levels ranging from 0 mg/cm^2 to 3.5 mg/cm^2. The lead-based paint films were adhered to steel or plaster substrates, which were considered for experimental design purposes to be difficult or easy to sample, respectively. The overlayers were either a thickly applied oil-based paint (about 0.75 mm to 1.4 mm) or a thinly applied latex paint (about 0.13 mm to 0.28 mm). The five operators were trained by a UE/ASV supplier's representative to conduct the tests using a written protocol developed from the supplier's instructions. The study showed that one of the two ASV electrochemical instruments was in calibration, whereas the response of the second ASV instrument was low at the lower lead concentrations used to check calibration. Consequently, the data were analyzed both as "unadjusted for calibration" and "adjusted for calibration." Lead levels determined by the UE/ASV tests were often considerably less than the lead levels in the test panels. Depending on the combination of five experimental factors—apparatus, operator, lead pigment type, substrate type, and overlayer—the recovered lead for the data adjusted for calibration ranged from 28 % to 94 %, with the median recovery being 63 %. These findings are in sharp contrast with previously published results of an UE/ASV field study in which lead recoveries generally ranged from 75 % to more than 100 %. In the present study, ASV measurement error did not appear to play a role in the low lead recoveries based on quality assurance measures. A key contributor appeared to be incomplete lead solubilization during paint specimen sonication. The major experimental factor affecting UE/ASV response was overlayer, with test panels having thick-oil overlayers yielding lower lead recoveries than those with thin-latex overlayers. It may have been that thick-oil overlayers were more difficult to sonicate, and/or grind before sonication, than thin-latex overlayers. Effects of the other experimental factors on UE/ASV response were considered primarily for the calibration-adjusted data. Operator and substrate factors were found to have a significant effect; whereas no effects were found for lead pigment type or apparatus.

Key Words: analysis; anodic stripping voltammetry (ASV); building technology; lead-based paint; lead recovery; overlayer effect; testing; ultrasonic extraction (UE)

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

1. INTRODUCTION

1.1 Background

As defined in Public Law 102-550, Residential Lead-Based Paint Hazard Reduction Act of 1992, the term lead-based paint means paint or other surface coatings that contain lead at contents that "equal or exceed a level of 1.0 milligram per centimeter squared or 0.5 percent by weight" [1]. The accurate and efficient identification of lead-based paint in housing is important to the Federal government. For example, in most cases where the presence of lead-based paint has been identified in pre-1978 "target" housing,* that information is disclosed to the owner, prospective purchasers, or tenants (42 U.S.C. 4852d, 24 CFR 35.80-98). Also, in certain target housing receiving financial assistance from the U.S. Department of Housing and Urban Development (HUD), or being sold by the Federal government, identification of lead-based paint results in requirements for lead-based paint hazard evaluation and/or control (42 U.S.C. 4822, 24 CFR 35.1-1355). The extensiveness of lead-based paint in housing is illustrated in a recent report from the President's Task Force on Environmental Health Risks and Safety Risks to Children in which it was estimated that, in 1999, approximately 24 million U.S. dwellings were at risk for lead-based paint hazards [3].

The National Institute of Standards and Technology (NIST) has been providing technical assistance to the HUD Office of Healthy Homes and Lead Hazard Control on measurement of lead-based paint in housing. For example, NIST recently completed a HUD-sponsored study [4] on the reliability of spot tests, which are qualitative, for detecting the presence of lead in household paints when tests were conducted in the laboratory by certified lead inspectors or risk assessors. This report presents the results of a HUD-sponsored study on the use of field-portable ultrasonic extraction/anodic stripping voltammetry (UE/ASV) for detecting the presence of lead-based paint in housing. This field method, in contrast to spot test kits, provides a quantitative estimate of the amount of lead in the specimen. Presently, one firm located in the United States supplies field-portable instrumentation for conducting on-site UE/ASV measurements of lead in paint. The basic procedure for using this instrumentation includes two main steps: (1) sample preparation and (2) electrochemical analysis. First, a paint sample is removed from its substrate, manually ground, and subjected to ultrasonic extraction (UE) with nitric acid. The concentration of lead in the resulting solution is then analyzed by ASV. The acronym for ultrasonic extraction followed by anodic stripping voltammetry measurement is UE/ASV, which is used in this report.

ASTM Standard Guide E 1775, "Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead" [5], provides a short description of the principle of ASV analysis of an analyte metal species dissolved in solution (e.g., Pb^{+2} ion):

> "The analyte is first deposited (preconcentrated) electrochemically by reducing the dissolved ion in solution to immobilized metal species at a mercury electrode surface. The metal is deposited in the form of an amalgam (with Hg) at an applied potential (voltage) which is negative of the standard oxidation potential for the metal/ion redox couple. After deposition, the preconcentrated metal species is then "stripped" from the mercury electrode by applying a positive potential sweep, which causes anodic oxidation of the analyte metal

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* The definition of target housing is: "Any residential unit constructed before 1978, except dwellings that do not contain bedrooms or dwellings that were developed specifically for the elderly or persons with disabilities—unless a child younger than 6 resides or is expected to reside in the dwelling. In the case of jurisdictions that banned the sale or use of lead-based paint before 1978, the Secretary of HUD may designate an earlier date for defining target housing" [1,2].

> species to dissolved ion. The current associated with this reoxidation is measured. The peak current is proportional to the original concentration of dissolved analyte species over a wide range of concentrations."

In addition to the ASTM Standard Guide, a NIOSH[*] analytical method for determining lead by UE/ASV is also available [6]. For detailed discussions of the principles, instrumentation, and applications of ASV and related techniques, the reader is referred to Wang [7,8].

Since the mid-1990s, the use of field-portable, quantitative UE/ASV as a measurement method for lead in paint and other environmental samples has received scrutiny [9-14]. These studies have concluded that UE/ASV may be suitable for conducting extensive quantitative on-site testing of lead in household paint due to relative ease of operation, rapid on-site response, and potentially acceptable cost.

In a 1995 laboratory investigation, Ashley [9] conducted 10 % nitric acid ultrasonic extraction followed by ASV analyses on a number of lead-containing airborne particulate samples and on NIST Standard Reference Materials (SRMs) including SRM 1579-Lead in Paint (11.87 % ± 0.04 % by mass fraction). He found that the recoveries of lead from the SRMs using UE/ASV were statistically equivalent to those obtained by NIOSH hotplate digestion methods with concentrated acid solutions. In the case of SRM 1579, the average recovery was about 95 % ± 6 %. Later, in a 1998 follow-up study, Ashley, Mapp, and Millson [13] reported that SRM 1579 showed an average recovery of about 91 % ± 10 %. The conclusions of both studies [9,13] were that UE/ASV appeared to be viable for the on-site determination of lead, and that UE might be superior to hotplate or microwave methods for specimen digestion.

In a 1996 laboratory study, Williams, Van Hise, and Gutknecht [10] examined the performance of selected solution-based methods for lead analyses of environmental samples. UE/ASV analyses included seven lead-containing paint specimens and NIST SRM 1579. The average lead recoveries ranged[†] from 80 % to 93 %, with SRM 1579 being 84 %. Similar to the earlier Ashley study [9], Williams et al. concluded that UE/ASV offered promise for the measurement of lead in environmental samples and that the specific ASV instrument appeared to have promise for field applications.

In a 1998 field study, Ashley, Hunter, Tait, Dozier, Seaman, and Berry [11] included UE/ASV analyses in their investigation of three on-site techniques used for determining lead in paint films. The results of UE/ASV analyses of 165 paint samples taken from plaster, metal, wood, and brick substrates were compared to those obtained from analyses using laboratory hotplate digestion-atomic absorption spectrometry (AAS). The authors indicated that the data were reasonably well correlated ($R^2 = 0.814$ for all data), but that better correlation had been obtained in laboratory analyses on specimens such as NIST SRMs. They concluded that the on-site testing with UE/ASV was found to meet analytical performance guidelines promulgated by EPA and ASTM.

One limitation to adopting field-portable UE/ASV for determining lead in paint in the field is that the effect of the operator (i.e., analyst) on the reliability of the analysis has not been demonstrated.

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[*] National Institute for Occupational Safety and Health.

[†] Williams et al. [11] generally reported percent lead recoveries as measured for the two sensitivity settings available on the ASV instrument. The range of values referenced above is for the low (or more sensitive) range; NIST SRM 1579 was analyzed using the high range.

Laboratory technicians or chemists have conducted measurements made in previous studies evaluating UE/ASV. Potential users of UE/ASV in practice may be certified lead-based paint inspectors or others, whose skill in conducting chemical analyses may arguably be less than that of laboratory technicians or chemists. Other factors affecting UE/ASV reliability may include the degree of difficulty in removing the paint sample from its substrate and the ease of dissolution of the lead pigment. The present study provides further evaluation of UE/ASV performance using well-trained certified lead inspectors as operators. The results should help to support future decisions regarding the use of UE/ASV in Federal programs.

1.2 Objective and Scope of the Study

The objective of the study was to evaluate the reliability of commercial, field-portable ultrasonic extraction-anodic stripping voltammetry (UE/ASV) for quantitatively determining the lead level of laboratory-prepared paint films using certified lead inspectors trained to conduct UE/ASV testing. Six factors were examined: lead level[*], operator, lead pigment type, paint-film substrate, overlayer, and UE/ASV apparatus. Table 1 provides a comment as to why each factor was included.

The study design recognized that, in practice, the amount of lead in paints varies over a wide range and that operator skill is variable. Consequently, 10 lead levels varying from 0 mg/cm^2 to 3.5 mg/cm^2 were incorporated[†] and five operators were included in the study. For the remaining four factors, i.e., lead pigment type, paint-film substrate, overlayer type, and UE/ASV apparatus, two levels were chosen for each. In these cases, the levels were generally selected to be representative of practice—for example, latex and oil-based paint overlayers—or to bracket the extremes of what is likely to be encountered in practice—for example, the solubility of the lead pigment. Steel and plaster panels were selected to represent substrates from which it might be relatively difficult and easy, respectively, to obtain paint specimens (Table 1). Neither substrate type was expected to interfere in general with the ASV analysis; for example, the field-portable ASV electrochemical instrument uses a disposable electrode (for a single measurement) that is designed to be specific for lead analysis [13]. Finally, two UE/ASV apparatuses were included as a preliminary examination of instrument effect.

2. EXPERIMENTAL

2.1 Design and Quality Control

A full factorial experiment was designed to investigate the effects of the six factors—lead level, operator, lead pigment type, paint-film substrate, overlayer, and apparatus—on UE/ASV performance. Before the test program began, all operators attended a one-day training session that was conducted at NIST by a UE/ASV supplier's representative[‡]. They received a certificate of successful course completion.

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[*] HUD prefers determining the amount of lead in a paint film on the basis of area content (i.e., mg/cm^2) as opposed to mass concentration. Hence, the experimental design of this study was based on area content, which is referred to as "lead level" in this report.

[†] A lead level of 0 mg/cm^2 is the designation assigned to test panels for which lead was not added to the paint films. Measurements using inductively coupled plasma (ICP) emission spectrometry showed that the lead levels of these panels were < 0.009 mg/cm^2 [4].

[‡] This representative did not work directly for the ASV supplier, but was authorized by the supplier to work with NIST during the study.

Table 1. Factors varied during the UE/ASV study[a]

Factor	Description	Comment
Lead Level	Ten lead levels were selected for each lead pigment type, and had the following targeted values (in mg/cm^2): white lead • 0 • 0.1 • 0.2 • 0.3 • 0.4 • 0.5 • 0.7 • 1.0 • 1.6 • 3.5 lead chromate • 0 • 0.5 • 0.7 • 1.0 • 1.2 • 1.6 • 1.8 • 2.0 • 2.5 • 3.5	Lead in paint films in existing houses varies from none (i.e., lead was not purposely added to the paint) to substantial, as measured in field studies [15,16]. The range of lead level in the present study was consistent with those found in the field. Note: A lead level of 0 mg/cm^2 is the designation assigned to test panels for which lead was not added to the paint films. Measurements using inductively coupled plasma (ICP) emission spectrometry showed that the lead levels of these panels were < 0.009 mg/cm^2 [4].
Operator	Five operators were included: • Operator 1 (Op1) • Operator 2 (Op2) • Operator 3 (Op3) • Operator 4 (Op4) • Operator 5 (Op5)	Many people having variable skills will perform UE/ASV field tests. The operator factor addressed the effect of "the human element" on UE/ASV response. Initial planning for the experimental program considered selecting four operators (Op1 through Op4), who were either certified lead inspectors or risk assessors. A fifth operator (Op5), a laboratory technician from NIST, was added. Op5 provided a comparison between testing conducted by a laboratory technician and that performed by certified lead inspectors. All five operators participated in a one-day training course conducted at NIST by a trainer approved by the UE/ASV manufacturer. Each operator received a certificate of successful course completion.
Lead Pigment Type	Two lead pigment types were included: • relatively soluble (white lead) • relatively insoluble (lead chromate)	A number of lead-based pigments have historically been used in paint production. These include basic lead carbonate, basic lead sulfate, lead silicate, chrome yellows and oranges (lead chromate combined with lead sulfate, lead carbonate, and lead phosphate to obtain different hues), chrome greens (chrome yellow and iron blue), molybdate orange (lead molybdate and lead sulfate) and red lead (Pb_3O_4) [17]. The most common pigment was basic lead carbonate, whose composition is approximately $2PbCO_3$—$Pb(OH)_2$ [18-20]. This pigment is often referred to as "white lead." Although used considerably less than white lead, lead chromate was commonly used as a basic pigment for some green, red, orange, and yellow house paints through the mid-1960s [20].

[a] Some information in this table was repeated from NIST Report 6893, "Spot Test Kits For Detecting Lead in Household Paint: A Laboratory Evaluation" [4], because the test panels used in the present UE/ASV study were prepared during the spot test kit study.

Table 1. Factors varied during the UE/ASV study (cont.)

Factor	Description	Comment
Substrate	Two types of substrates were selected: • steel (difficult-to-sample) • plaster (easy-to-sample)	Lead-based paint has been applied over many different substrates. In conducting an UE/ASV analysis, the initial step is to inscribe a circle through the paint film using a cork borer. The paint film within the inscribed circle is then removed from the substrate and transferred to a sonication tube. When the results of an UE/ASV analysis is reported in mg/cm^2 (as in the present study), the fraction of the sample removed from the inscribed circle and placed in the sonication tube determines, in principle, the upper bound of the recovery. Preliminary sampling of selected test panels indicated that paint specimens might be more completely obtained from plaster substrates than from steel substrates when using the cork borer procedure. In the case of plaster, the paint film could generally be taken intact by cutting through the paint and slightly into the plaster surface, because the film within the inscribed area was captured in the cork borer. In the case of steel, after cutting into the paint with the cork borer, the paint film within the inscribed area generally remained on the substrate and had to be removed by scraping. Thus, steel and plaster substrates were incorporated in the experimental design, and designated "difficult-to-sample" and "easy-to-sample," respectively.
Overlayer	Two overlayers covered the lead-containing film in the test panels: • latex paint applied relatively thin, about 0.13 mm to 0.28 mm • oil-based paint applied relatively thick, about 0.75 mm to 1.4 mm	Lead-based paint is quite likely to be covered with additional layers of paint. These overlayers may be latex or oil paint. Additionally, paint-film thickness in the field varies; for example, Reames et al. [16] have reported thickness values ranging from about 0.1 mm to 1.5 mm. Overlayers on the test panels reflected field experience. The overlayer factor investigates the effect of whether the overlayer on the lead-containing paint film affects UE/ASV response without distinguishing between its type and thickness. The test panels used in the UE/ASV study had been previously prepared for use in the NIST spot test kit study [4], and panels having thick-latex paint and thin-oil paint overlayers were not prepared on steel and plaster substrates.
Apparatus	Two apparatuses were included: • apparatus 1 • apparatus 2	The apparatus factor was included as a preliminary examination of instrument effect on UE/ASV performance, when investigated using the "as received" instrumentation. At the time of this study, UE/ASV apparatus for conducting on-site measurements of lead in paint was commercially available from one supplier. This firm sold a field-portable kit which included a sonicator, ASV electrochemical instrument with disposal electrodes, carrying case, and laboratory accessories necessary to conduct lead analyses. Two of these kits (with the same model number) were purchased, and designated "apparatus 1" and "apparatus 2."

The number of UE/ASV analyses in a series conducted by one operator using one apparatus was 93. Each series was comprised of 80 randomly sequenced test panels*, seven control specimens and six method blanks. The controls were pre-cut paint films, 8 mm in diameter by about 0.08 mm thick, that contained white lead and that had a mean† lead level of 1 mg/cm^2. A paint-film control was used instead of a standardized lead solution because the film provided a check on both the sonication and analysis steps of the test protocol. The controls were supplied to the operators in a specimen preparation tube and subsequently ground, sonicated, and analyzed according to the steps in the UE/ASV study protocol. Method blanks consisted of performing the UE/ASV procedure without a specimen. They were used to monitor background levels and check for contamination.

A set of seven specimens could be simultaneously sonicated for lead extraction using the sonicator supplied with the ASV. For a single test series, every set of seven specimens subjected to simultaneous sonication included either a 1 mg/cm^2 control specimen or a method blank, with the first sonication set having a control. If the result of a UE/ASV measurement of a control was outside the range of 0.75 mg/cm^2 to 1.25 mg/cm^2, then the performance of the sonicator was checked according to the UE/ASV supplier's instructions and the ASV response was checked using a standardized lead solution. This standardized solution contained lead in 2.5 % nitric acid (volume fraction) at a concentration (Pb content of 50 mg/L) that corresponded to a lead level of 5 mg/cm^2 for the UE/ASV procedure. If these two checks provided acceptable results, the operator continued with the test series.

Finally, as a quality control procedure, at the beginning and end of each day of tests, the operator performed ASV analyses of the standardized lead solution (Pb content of 50 mg/L) and also of a blank solution (i.e., no lead added) of 2.5 % nitric acid. Testing of the specimen series was not to begin unless the results were within the established criteria. The repeated measurements at the end of the day were intended to provide a measure of instrument drift over the course of the day.

2.2 UE/ASV Apparatus

Two field-portable UE/ASV apparatuses‡ were purchased from the same supplier. The two apparatuses had the same model number and were designated Apparatus 1 and Apparatus 2. The sonicators were specified as having an average power of 45 W. For sonication, the paint specimens were covered with 5 mL of 25 % nitric acid (volume fraction) and sonicated for 30 min. The temperature of the tap water placed in the sonicator bath was approximately 45° C. These sonication parameters were the same as those recommended by Grohse, Luk, Hodson, Wilson, Gutknecht, Harper, Beard, Lim, and Breen [21], who investigated the suitability of various sonication conditions for extraction of lead from paint, such as nitric acid concentration, time, and sonicator power.

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[*] 10 lead levels x 1 operator x 2 lead pigments x 2 substrates x 2 overlayers x 1 apparatus.

[†] One hundred four controls were sampled from a lead-containing paint film prepared for the spot test kit study [4]. Lead levels of twelve randomly selected controls were measured by a National Lead Laboratory Accreditation Program (NLLAP) accredited laboratory. Lead extraction was by sonication digestion and lead analysis was by flame atomic absorption spectrometry (FAAS). The coefficient of variation (CoV) of the mean lead level for the 12 controls analyzed was 2.5 %.

[‡] In this report, the term, "apparatus," refers to the combination of UE sonicator and ASV electrochemical instrument that was purchased as part of a field-portable kit. For the individual pieces of equipment, the terms, "[UE] sonicator" and "[ASV] instrument" are used.

The ASV instruments were factory-calibrated. The limit of detection for lead reported in the supplier's instruction booklet was 0.04 mg/cm^2. Factory calibration of each ASV instrument was checked in the laboratory using six standard solutions of lead nitrate in 2.5 % nitric acid (volume fraction). Because the results of the lead analyses of the test panels were recorded in "mg/cm^2," the calibration checks were conducted in units of mg/cm^2. The six standard solutions (Pb contents ranging from 1 mg/L to 50 mg/L) covered a concentration range that corresponded to an ASV range from 0.1 mg/cm^2 to 5 mg/cm^2, which bracketed the lead levels in the test panels. In performing the calibration checks, four analyses of each of the six standard solutions were made using the ASV instruments of both Apparatus 1 and Apparatus 2.

2.3 Test Panels and Specimen Size

Tables 2A and 2B describe the test panels having white lead and lead chromate pigments, respectively. These test panels had been previously prepared for use in the HUD-sponsored spot test study [4]. The amount of lead in the panel paint-films was determined by a NLLAP accredited laboratory using inductively coupled plasma (ICP) emission spectrometry [4]. The phrase, "lead level," is the expression used in this report when referring to the amount of lead in the test panels. Use of the test panels from the spot test study precluded independent investigation of the effect of overlayer type and overlayer thickness on UE/ASV response. Note in Table 1 that latex paint overlayers were thin; whereas oil paint overlayers were thick.

A grid of 25 mm by 25 mm squares was indelibly marked on the surface of each panel [4]. Each square represented the location where the operator randomly sampled the paint film. Paint-film specimens were sampled from the test panels using a 7.9 mm (inside diameter) No. 4 cork borer and, thus, had areas of 49 mm^2. As an estimate of maximum specimen mass, specimens sampled from 10 ten randomly selected plaster panels having thick-oil overlayers were found to have masses ranging from 50 mg to 170 mg with a median mass of 110 mg. That is, for these 10 determinations, mass was not greater than 200 mg, which is the maximum specimen mass stated in the UE/ASV supplier's instructions*.

2.4 Testing and Data Recording

The operators conducted the UE/ASV tests according to a written protocol that is summarized in Table 3. The protocol was reviewed prior to initiating testing by the UE/ASV supplier's representative to assure that the steps were consistent with the supplier's instructions. Some additional procedures were included for completeness. For example, the supplier's instructions were not specific regarding steps to be taken in cleaning the paint surfaces of laboratory-prepared test panels, and did not address formats for recording data. Because all testing was conducted at a single laboratory workstation, the cleaning procedures were important to avoid cross-contamination of the specimens.

A deviation from the supplier's instructions was that the electrolyte tablet (see Table 3, section 7, step 1) was added to the analysis vial before addition of the sonication extract solution. This was done because of the convenience of crushing the tablet in the empty vial. Another deviation from the supplier's instructions was that, prior to grinding, all specimens were cooled using dry ice to

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* The limitation on specimen mass (i.e., 200 mg) is only given in the ASV supplier's instructions for analyses for which results are to be reported as percent lead by mass fraction. These instructions indicate that, for results to be reported in mg/cm^2, the sample collected is to be sonicated.

Table 2A. Test panels having white lead pigment

Test Panel ID Code	Panel Description			
	Substrate Type	Overlayer Type	Overlayer Thickness	Lead Level[a,b,c] mg/cm^2
107	Plaster	Latex	Thin	< 0.009
313	Plaster	Oil	Thick	< 0.009
188	Plaster	Latex	Thin	0.10
285	Plaster	Oil	Thick	0.09
234	Plaster	Latex	Thin	0.19
237	Plaster	Oil	Thick	0.19
138	Plaster	Oil	Thick	0.31
165	Plaster	Latex	Thin	0.28
129	Plaster	Oil	Thick	0.43
262	Plaster	Latex	Thin	0.39
206	Plaster	Latex	Thin	0.48
281	Plaster	Oil	Thick	0.50
179	Plaster	Latex	Thin	0.70
292	Plaster	Oil	Thick	0.66
102	Plaster	Oil	Thick	0.96
256	Plaster	Latex	Thin	1.04
251	Plaster	Latex	Thin	1.53
279	Plaster	Oil	Thick	1.54
127	Plaster	Oil	Thick	3.88
177	Plaster	Latex	Thin	3.53
101	Steel	Oil	Thick	< 0.009
322	Steel	Latex	Thin	< 0.009
168	Steel	Oil	Thick	0.09
221	Steel	Latex	Thin	0.09
180	Steel	Oil	Thick	0.18
269	Steel	Latex	Thin	0.17
135	Steel	Oil	Thick	0.23
203	Steel	Latex	Thin	0.23
189	Steel	Oil	Thick	0.29
222	Steel	Latex	Thin	0.32
130	Steel	Oil	Thick	0.36
326	Steel	Latex	Thin	0.44
224	Steel	Latex	Thin	0.58
226	Steel	Oil	Thick	0.54
246	Steel	Oil	Thick	0.86
278	Steel	Latex	Thin	0.78
123	Steel	Oil	Thick	1.46
284	Steel	Latex	Thin	1.39
145	Steel	Oil	Thick	3.39
215	Steel	Latex	Thin	3.20

[a] A lead level of 0 mg/cm^2 is the designation assigned to test panels for which lead was not added to the paint films. Measurements using ICP showed that the lead level for these panels was < 0.009 mg/cm^2.

[b] A distinction between white lead and lead chromate for specimens having a 0 mg/cm^2 lead level is artificial because such specimens did not have added lead. Nevertheless, the distinction is maintained to balance the experimental design.

[c] The measurement process used in assigning the values is given in Ref. [4].

Table 2B. Test panels having lead chromate pigment

Test Panel ID Code	Panel Description			
	Substrate Type	Overlayer		Lead Level[a,b,c] mg/cm^2
		Type	Thickness	
175	Plaster	Latex	Thin	< 0.009
202	Plaster	Oil	Thick	< 0.009
140	Plaster	Oil	Thick	0.51
250	Plaster	Latex	Thin	0.47
163	Plaster	Oil	Thick	0.74
274	Plaster	Latex	Thin	0.69
297	Plaster	Oil	Thick	0.94
323	Plaster	Latex	Thin	0.94
126	Plaster	Latex	Thin	1.09
178	Plaster	Oil	Thick	1.20
148	Plaster	Latex	Thin	1.46
183	Plaster	Oil	Thick	1.44
249	Plaster	Latex	Thin	1.71
253	Plaster	Oil	Thick	1.62
139	Plaster	Latex	Thin	1.98
141	Plaster	Oil	Thick	1.86
330	Plaster	Oil	Thick	2.30
333	Plaster	Latex	Thin	2.51
167	Plaster	Latex	Thin	3.29
205	Plaster	Oil	Thick	3.64
303	Steel	Latex	Thin	< 0.009
319	Steel	Oil	Thick	< 0.009
223	Steel	Latex	Thin	0.49
308	Steel	Oil	Thick	0.43
289	Steel	Latex	Thin	0.57
310	Steel	Oil	Thick	0.52
216	Steel	Latex	Thin	0.74
302	Steel	Oil	Thick	0.75
113	Steel	Oil	Thick	0.92
231	Steel	Latex	Thin	0.98
124	Steel	Oil	Thick	1.48
158	Steel	Latex	Thin	1.34
187	Steel	Latex	Thin	1.55
315	Steel	Oil	Thick	1.57
263	Steel	Latex	Thin	1.56
294	Steel	Oil	Thick	1.89
329	Steel	Latex	Thin	1.72
331	Steel	Oil	Thick	2.25
142	Steel	Oil	Thick	2.81
199	Steel	Latex	Thin	3.09

[a] A lead level of 0 mg/cm^2 is the designation assigned to test panels for which lead was not added to the paint films. Measurements using ICP showed that the lead level for these panels was < 0.009 mg/cm^2.

[b] A distinction between white lead and lead chromate for specimens having a 0 mg/cm^2 lead level is artificial because such specimens did not have added lead. Nevertheless, the distinction is maintained to balance the experimental design.

[c] The measurement process used in assigning the measured values is given in Ref. [4].

Table 3. Summary of the UE/ASV protocol

Section	Summary of the Steps in the Protocol
1. Cleaning the Panel Surface and Labeling the Sonicator Tube	• Take a test panel and place a checkmark on the designated test square. • Wipe the surface of this test square with a wet wipe and then with a dry wipe. • Write the test panel ID number on a new 50 mL sonicator tube using an indelible pen.
2. Sampling the Test Panel	• Fold a sheet of sample collection paper in half such that the adhesive strip is at the top outside edge of the folded sheet. • Using the coring tool (i.e., cork borer), inscribed a circle cut into the paint-film surface to the substrate. • Brush away any paint chips on the outside of the coring tool. • Place the folded sample collection paper adjacent to the coring tool; remove the coring tool from the paint-film surface allowing chips from the inside to fall onto the paper. • Dislodge any remaining chips of paint in the coring tool onto the sample collection paper using a plastic paint-crushing rod. • Scrape any paint remaining inside the inscribed circle onto the sample collection paper using the tip of a utility-knife blade or the sharp edge of the coring tool.
3. Specimen Transfer and Grinding	• Transfer the specimen to the ID-marked 50 mL sonicator tube; assure that the entire specimen is transferred into the tube from the sample collection paper. • Place the sonicator tube in dry ice for a few minutes to embrittle the specimen. • After removal from the dry ice, grind the specimen using a plastic paint-crushing rod until all large particles have been broken down. • Leave the paint-crushing rod in the sonicator tube.
4. Specimen Acidification	• Fill the 5 mL pipette with 25 % nitric acid (volume fraction). • Hold the paint-crushing rod about 25 mm above the ground paint specimen. • Dispense the 5 mL of 25 % nitric acid from the pipette directly onto the paint-crushing rod so that it rinses any residual particles from the rod. • Do not agitate the specimen in the sonicator tube by shaking, stirring, or swirling the tube as such actions can cause the particles to be left on the tube walls above the acid; cap the sonicator tube.
5. Cleaning of the Test Panel and Accessories Used in Sampling	• Wipe the surface of the sampled test square with a wet wipe and then with a dry wipe. • Cut a section of "blue" masking tape just big enough to cover the test square area and place it on the sampled surface. • Clean the accessory items such as knife blades and coring tools.
6. Specimen Sonication (normally performed simultaneously on seven specimens)	• Add warm tap water (≈ 50 °C) to the ridge in the sonicator bath to assure that the water level is at least 15 mm above the level of the acid in the sonicator tube. • Position the 7-hole cover on the sonicator bath, and place the specimen-containing sonicator tube(s) into the bath such that the cap rests on the bath cover. • Place a weight on top of the sonicator tube(s) to prevent floating. • Sonicate the specimen(s) for 30 minutes.
7. Specimen Analysis	• Add an electrolyte tablet into a 5 mL analysis vial; crush the tablet with a tablet-crushing rod. • Take the sonicated specimen/tube and, after removing the cap, carefully add distilled water to the 50 mL mark; do not fill over this mark during dilution. • Replace the cap on the sonicator tube and shake the tube gently to mix. • Take the 5 mL analysis vial containing the crushed tablet and mark it with the specimen ID number using an indelible marking pencil. • Carefully pour the diluted extract solution from the sonicator tube into the analysis vial, filling to the 5 mL mark. • Cap and shake the analysis vial to dissolve the crushed electrolyte tablet. • Place a disposable electrode into the electrode connector. • Perform the lead analysis according to the manufacturer's instructions for operation of the ASV electrochemical instrument. • Record the lead result (in mg/cm^2) on the NIST-provided data form along with the analysis ID number assigned by the ASV instrument, the instrument sensitivity range for the analysis, and a check mark indicating whether the paint specimen was difficult to grind.

embrittle them. The use of dry ice cooling of all specimens was initiated in consultation with the supplier's representative. Dry ice cooling had been previously employed by Ashley et al. [11], and is an alternative step in the ASTM E 1979 procedure for grinding paint film samples before ultrasonic extraction prior to lead analysis [22].

During testing, operators used data forms developed for the study to record the results of the lead analysis in mg/cm^2. Operators noted on the form whether the specimen was considered to be difficult to grind, and also recorded a code that was pre-marked on each test panel. This code was used by NIST research staff as a check that each analysis was performed on the correct panel. No errors were found for any operator.

3. RESULTS, ANALYSIS, AND DISCUSSION

3.1 Quality Assurance Measurements

3.1.1 Calibration Check. In checking the ASV instrument calibration, the data suggested that the measurement uncertainty for the ASV response was proportional to the true concentration. Consequently, analysis of calibration relations was performed on a log scale. This analysis showed (Figure 1) that, for the ASV instruments of both Apparatus 1 and Apparatus 2, the relation between ASV measured result and level lead of the calibration solution was linear (i.e., the quadratic term of a polynomial was not statistically significant at the 0.05 level). The calibration line for Apparatus 1 was virtually indistinguishable from a straight line having a slope of one and having an intercept of zero, indicating that the ASV instrument of Apparatus 1 was in calibration. In contrast, the calibration line for the ASV instrument of Apparatus 2 was below that for Apparatus 1 at low concentrations (Figure 1). In this case, the slope of the calibration line was significantly different from 1 and the intercept was significantly different from 0. Although some responses for the ASV instrument of Apparatus 2 were less than the established concentrations, it was used in the study because the experimental design (Table 1) included examination of instrument effect on UE/ASV performance, when investigated using "as received" instrumentation. Because of the low responses of the ASV instrument of Apparatus 2 at some lead concentrations, the data were analyzed (Section 3.2) both as "unadjusted for calibration" and "adjusted for calibration."

3.1.2 Paint-Film Controls. Seven paint-film controls having a lead level of 1 mg/cm^2 were included in each series of analyses to provide the operators with a periodic check that their analyses remained within acceptable calibration bounds. Table 4 summarizes the paint-film control data unadjusted for calibration. Figure 2 is a plot of the paint-film control results, unadjusted for calibration, in the sequence in which the analyses where conducted by each operator using each apparatus. The line in this figure represents the lower limit (i.e., 0.75 mg/cm^2 lead level) of the guideline range for acceptance of the control data without checking the sonicator and ASV instrument performance. In general, the results were within the guidelines given to the operators, and no reading exceeded the upper limit of the guideline range. For those cases where a result was beyond the guideline range for acceptance of the data, the checks (Section 2.1) of the sonicator and ASV instrument provided no evidence that they were not operating as anticipated. Note also from Table 4 that the mean values for Apparatus 2, unadjusted for calibration, are less than those for Apparatus 1 by 12 % or more, which is consistent with the ASV instrument readings for Apparatus 2 in Figure 1.

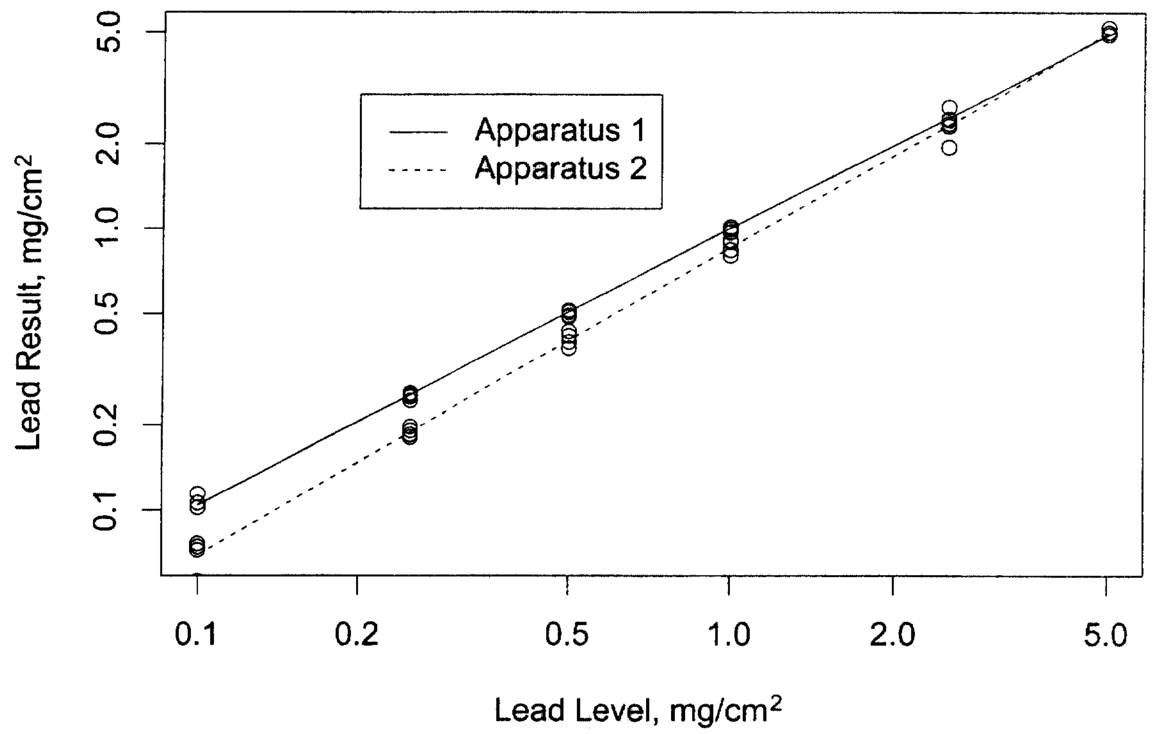

Figure 1. Calibration Checks of the ASV Instruments of Apparatus 1 and Apparatus 2.

Table 4. Results of the ASV analyses of the paint-film controls

Operator No.	Apparatus No.	Paint-Film Control Results, mg/cm^2					
		Mean[a]	Minimum	Maximum	sd[b]	CoV, %[c]	Sign. Diff.[d]
1	1	0.97	0.91	1.0	0.035	3.6	Yes
	2	0.82	0.67	0.87	0.073	8.9	
2	1	0.94	0.82	1.0	0.060	6.4	Yes
	2	0.77	0.75	0.78	0.013	1.7	
3	1	0.95	0.89	0.99	0.037	3.9	Yes
	2	0.80	0.59	0.91	0.10	12.8	
4	1	0.91	0.83	0.95	0.043	4.8	Yes
	2	0.72	0.60	0.78	0.074	10.3	
5	1	0.97	0.92	1.0	0.037	3.8	Yes
	2	0.85	0.79	0.93	0.050	5.9	
All	1	0.94	0.82	1.0	0.046	4.9	Yes
	2	0.79	0.59	0.93	0.080	10.1	

[a] Mean of seven analyses; except "all operators" for which the mean is for 35 analyses.
[b] sd indicates standard deviation.
[c] CoV indicates coefficient of variation.
[d] Sign. Diff. indicates whether the means are statistically different at the 0.05 significance level.

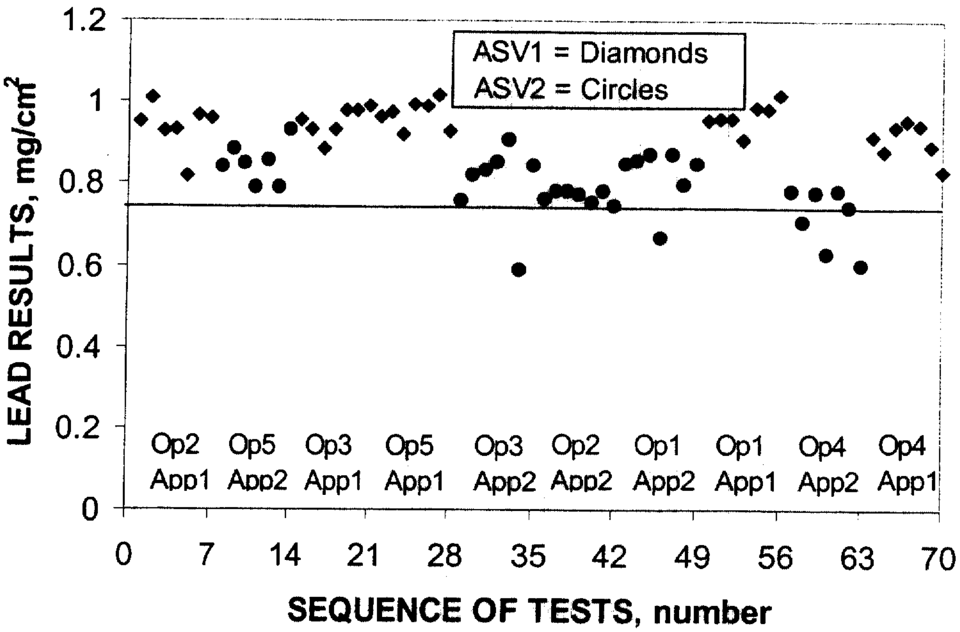

Figure 2. Results of the UE/ASV Analyses of the Paint-Film Controls Having a Lead Level of 1 mg/cm^2. The Line Represents the Measured Lead Result Below Which the Operators Were to Check the Sonicator and the Response of the ASV Instrument.

3.1.3 Method Blanks. The analyses of all method blanks gave results recorded as "below the detection limit," indicating that measurable contamination had not occurred with the method blanks. Consistent with this finding, no evidence was obtained during the test program that contamination was an issue. Observations of operators during the testing indicated that they were following the written protocol regarding test panel handling, specimen preparation, and workstation clean up.

3.1.4 Drift. Measurements made at the beginning and end of each test day on the standard solution of lead (Pb content of 50 mg/L) in 2.5 % nitric acid (volume fraction) showed no indications of instrument drift.

3.1.5 Paint-Film Matrix Effect. Standard addition analyses [23] conducted to investigate a paint-film matrix effect indicated that none was present. Two plaster test panels with thick-oil overlayers and a lead level of 0 mg/cm^2 were used in this experiment. Without addition of a standardized lead solution (i.e., no spiking), the results of the UE/ASV analyses of specimens from these panels were "below the detection limit" (i.e., < 0.04 mg/cm^2) when using either Apparatus 1 or Apparatus 2. Duplicate standard addition analyses of specimens from each panel were then performed. After sonication, 10 mL of the diluting solution (See Table 3, section 7, step 2) was replaced with 10 mL of a standard lead nitrate solution. The concentration of this standard solution (Pb content of 50 mg/L) was selected such that the ASV response would be 1 mg/cm^2 without a paint-film matrix effect. For Apparatus 1 and Apparatus 2, respectively, the mean lead levels of the spiked specimens were 0.99 mg/cm^2 (CoV of 1 %) and 0.90 mg/cm^2 (CoV of 14 %). In both cases, these mean values were not statistically different from 1 mg/cm^2.

3.2 ASV Result Versus Test Panel Lead Level

Figures 3A through 3E are plots of the measured lead results versus lead level in the test panels for each of the five operators for the two UE/ASV apparatuses. Figure 3F is a similar plot containing all data. These figures also include a solid line representing the ideal case in which all the lead is hypothetically recovered. For plotting purposes, the test specimens yielding results of "below the detection limit" were assigned a value of 0.02 mg/cm^2 or one half the detection limit of the ASV instruments.

Four key points should be noted from the data sets for Figures 3A through 3E:

- Most of the UE/ASV lead results appeared to be proportional to the lead level of the test panel.
- All responses for the 80 specimens taken from test panels having a 0 mg/cm^2 lead level were "below the detection limit."
- For all five operators, the overwhelming majority of ASV measured lead results fell below the ideal line. In other words, the UE/ASV procedure conducted under the conditions of this controlled laboratory study produced lead recoveries less than the lead levels of the test panels.
- Eighteen UE/ASV analyses of specimens from test panels had an ASV instrument response of "below the detection limit" when, in fact, the lead level of the test panel was greater than the instrument detection limit (i.e., 0.04 mg/cm^2). In the extreme, one of the "below the detection limit" measurements was recorded for a specimen sampled from a test panel having a lead level of 3.2 mg/cm^2. These 18 analyses comprised 2.5 % of the number[*] of analyses conducted on panels having lead levels ≥ 0.1 mg/cm^2.

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[*] The total was 720: 9 lead levels x 5 operators x 2 lead pigments x 2 substrates x 2 overlayers x 2 apparatuses.

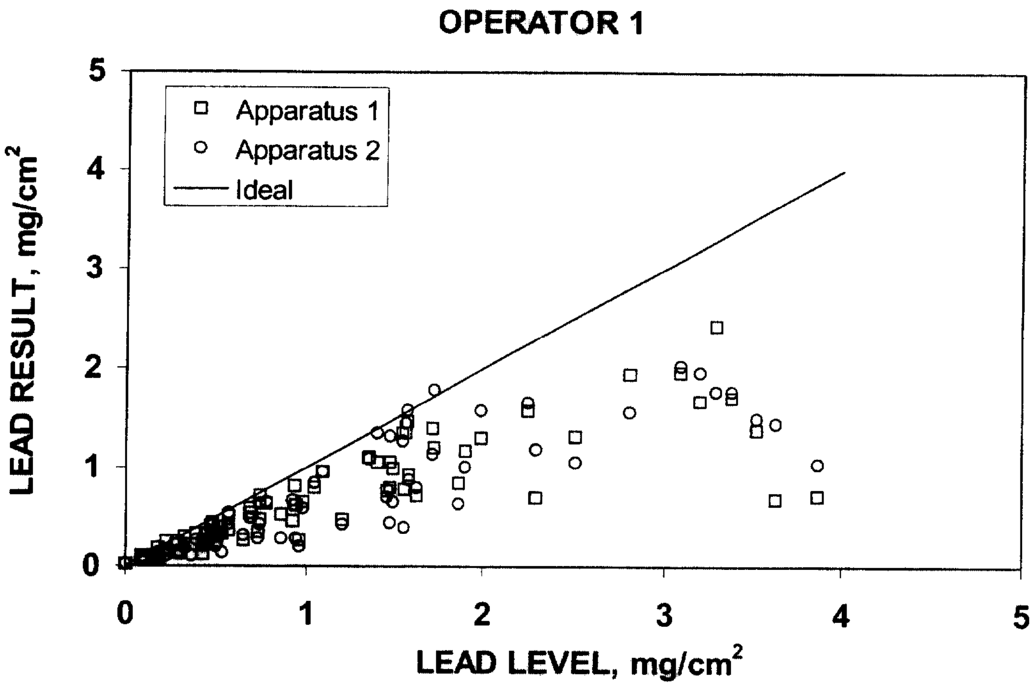

Figure 3A. UE/ASV Lead Result Measured by Operator 1 Versus Lead Level. In the Ideal Case, Lead is Completely Recovered During the Analysis.

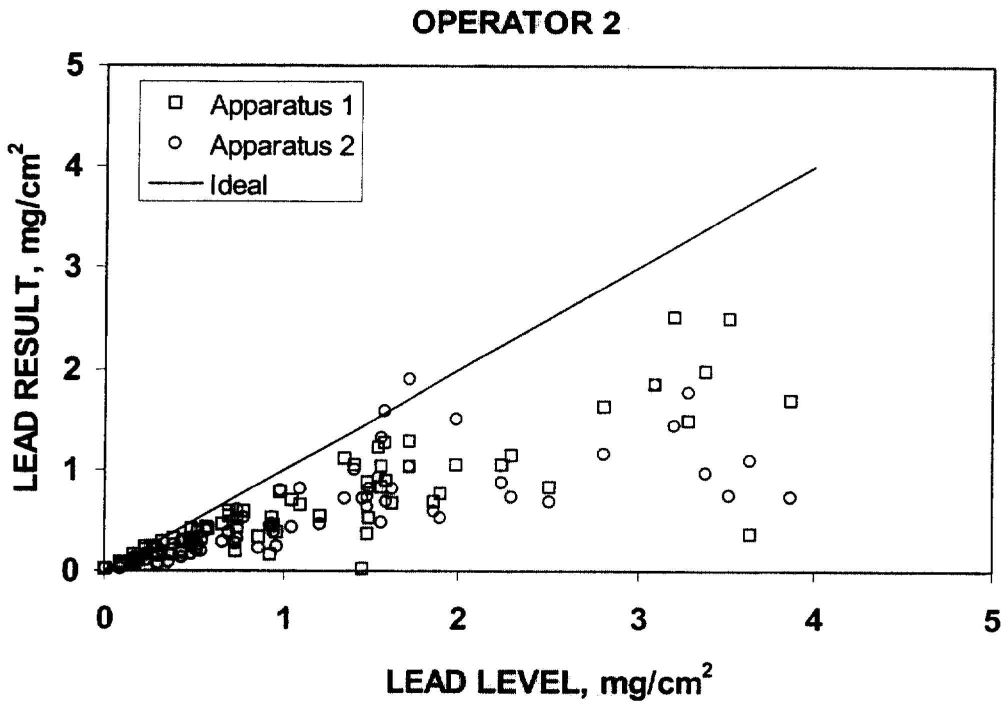

Figure 3B. UE/ASV Lead Result Measured by Operator 2 Versus Lead Level. In the Ideal Case, Lead is Completely Recovered During the Analysis.

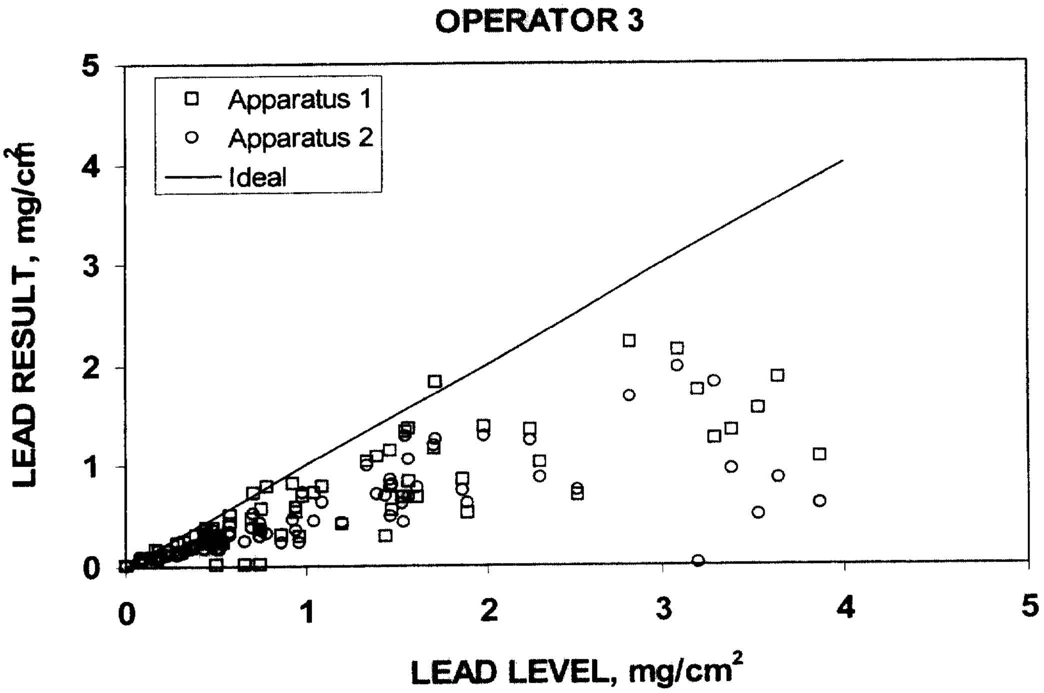

Figure 3C. UE/ASV Lead Result Measured by Operator 3 Versus Lead Level. In the Ideal Case, Lead is Completely Recovered During the Analysis.

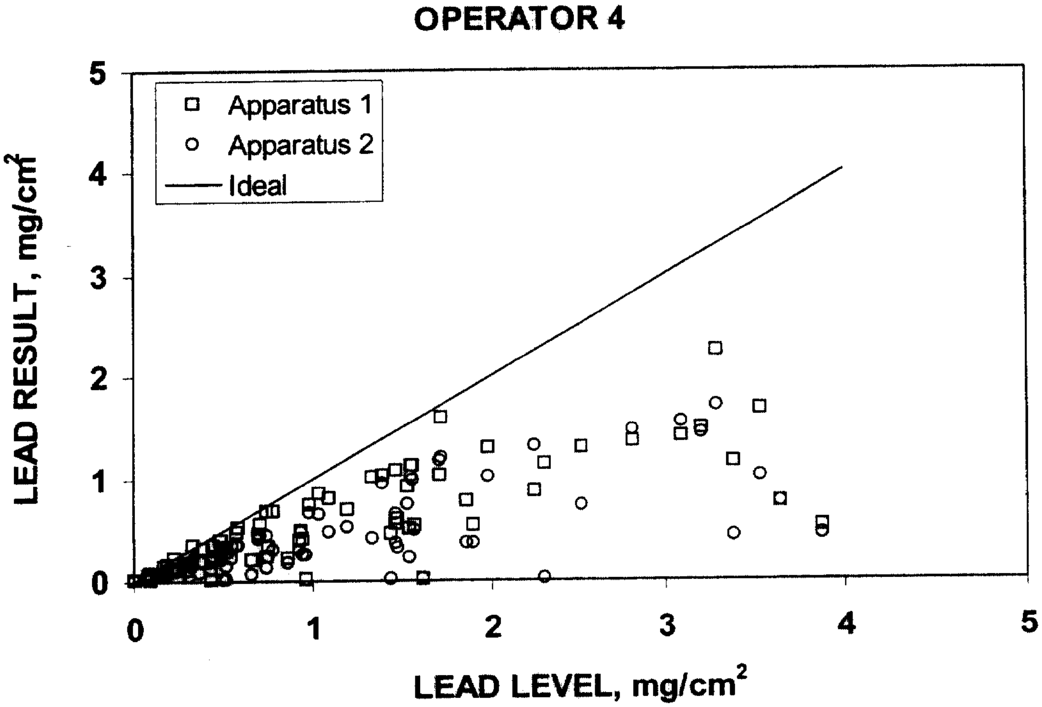

Figure 3D. UE/ASV Lead Result Measured by Operator 4 Versus Lead Level. In the Ideal Case, Lead is Completely Recovered During the Analysis.

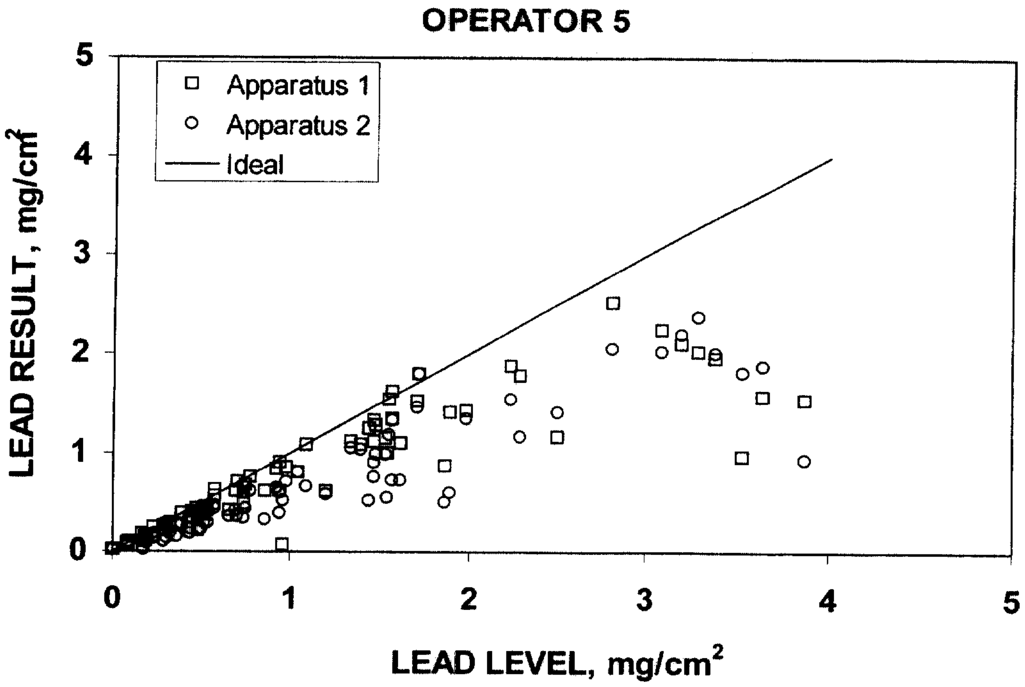

Figure 3E. UE/ASV Lead Result Measured by Operator 5 Versus Lead Level. In the Ideal Case, Lead is Completely Recovered During the Analysis.

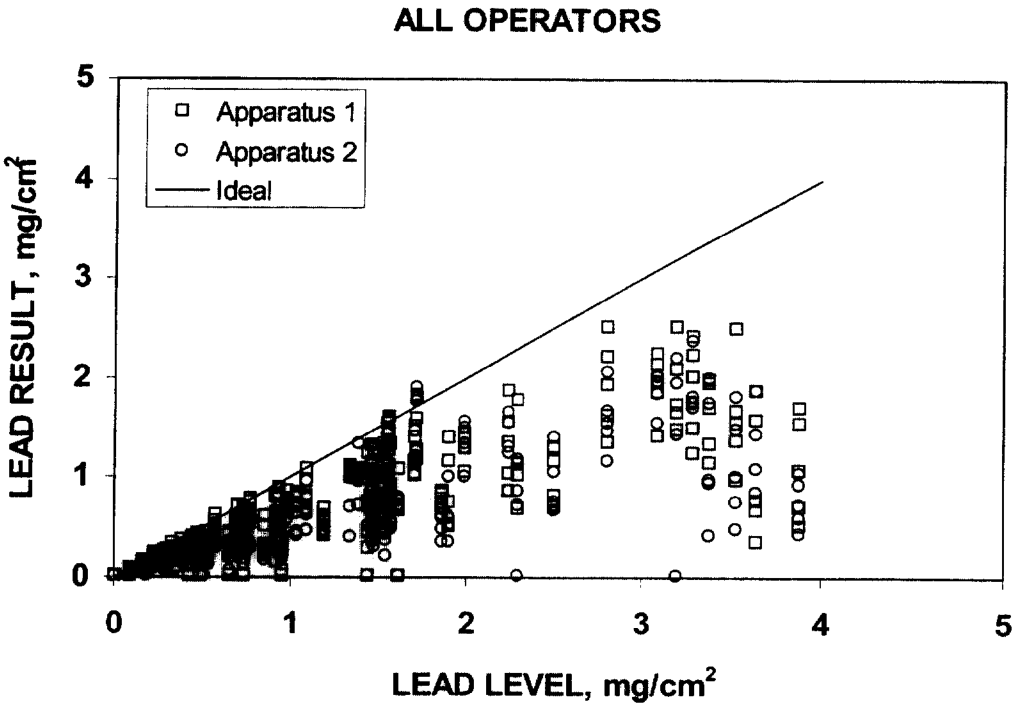

Figure 3F. UE/ASV Lead Result Measured by All Operators Versus Lead Level. In the Ideal Case, Lead is Completely Recovered During the Analysis.

In analyzing the UE/ASV results, the data were transformed to a log scale to make the variability less dependent on lead level. That is, taking logs makes the scatter about the mean lead result for any given combination of experimental variables more constant, thereby, resulting in data which are more amenable to standard statistical procedures. Figure 4 is a log-log plot of the ASV data, unadjusted for calibration, as a function of test panel lead level, and includes a line representing ideal performance (i.e., 100 % recovery) of the UE/ASV method. Again, for plotting purposes, the test specimens yielding results of "below the detection limit" were assigned a value of 0.02 mg/cm^2. From Figure 4, it appeared that the data for panels having lead fell into two categories: (1) those points for which the lead results appear to be proportional to the test panel lead level, and (2) those points for which specimens gave an instrument response of "below the detection limit" when the test panels contained lead. For the data analysis, a model (discussed below) relating measured result to test panel lead level was chosen that was based on the assumption of proportionality. In addition to estimating lead recovery, the model can be used to examine the effects of experimental factors (Table 1) incorporated in the study. Most of the data analyses were performed excluding the "below the detection limit" points (i.e., 18 specimens from panels having lead and 80 specimens from panels having 0 mg/cm^2 lead levels). In the case of the 18 points, it was considered that the responses arose due to some unknown mechanism(s) influenced by factors that were likely not among those under experimental control such as mishandled specimens or faulty apparatus performance*. The points for the 80 specimens from panels having 0 mg/cm^2 lead levels were excluded due to the log transformation of the data. However, some data analyses (Section 3.2.1) were also conducted with all points included to compare the effect of the "below the detection limit" points on the conclusion reached regarding lead recovery.

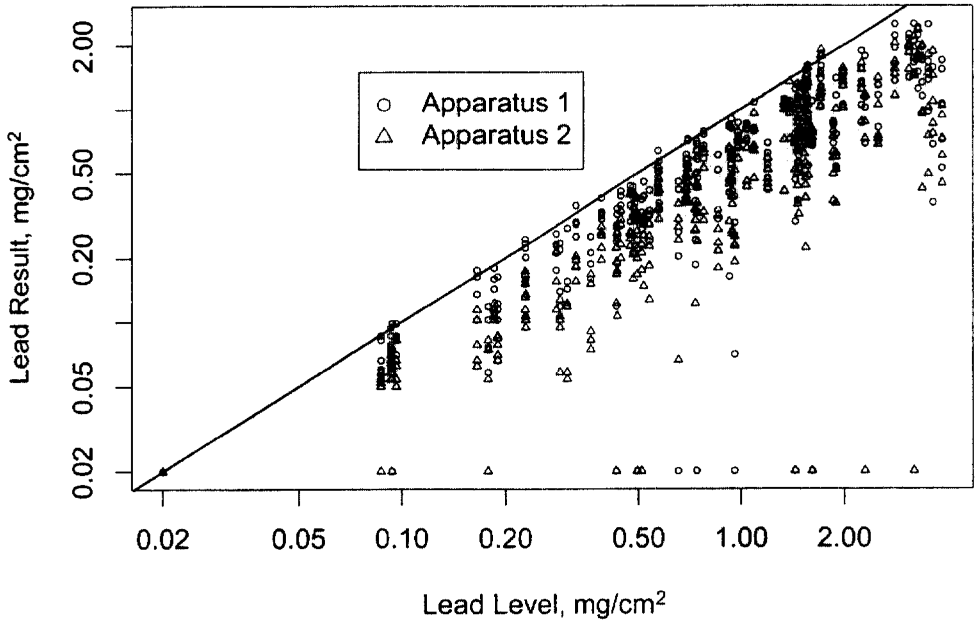

Figure 4. log(UE/ASV Lead Result) Versus log(Test Panel Lead Level) for All Operators. The Line Represents the Ideal Case of Complete Lead Recovery.

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* Although the quality control measures and operator training were intended to avoid such occurrences, it cannot be ruled out that such events did not occasionally occur.

The proportionality model, based on Figure 4, linearly relates the logarithm of UE/ASV response (i.e., measured lead result) to the logarithm of the test panel lead level:

$$\text{Log(ASV Test Result)} = m \times \log(\text{Panel Lead Level}) + d, \qquad \text{(Eq 1)}$$

where m and d are constants for the slope and intercept, respectively.

Using the model, for each combination of five experimental factors, operator, apparatus, lead pigment type, substrate type, and overlayer, a log-log plot of UE/ASV measured result versus test panel lead level was prepared. Figures 5 and 6 are examples of such plots. Figure 5 is for the case of Operator 5 using Apparatus 1 to analyze steel panels having lead chromate pigment and a thin-latex overlayer. Figure 6 shows the results of Operator 4 using Apparatus 2 to analyze those plaster panels having white lead pigment and a thick-oil overlayer. Plots for the 80 combinations* of these five experimental factors are given in Appendix A for the data unadjusted for calibration.

The key features in Figures 5 and 6 are:

- The *plot characters*, representing the individual UE/ASV results for the given combination of experimental factors.
- A *solid bold line*, which represents ideal UE/ASV performance (i.e., complete lead recovery). Ideal performance is modeled by Eq 1 with a slope of one and an intercept of zero:

$$\log(\text{ASV Test Result}) = \log(\text{Panel Lead Level}), \qquad \text{(Eq 2)}$$

or

$$\text{ASV Test Result} = \text{Panel Lead Level}. \qquad \text{(Eq 3)}$$

- A *solid thin line*, representing a linear regression analysis of the data points in which the slope of the regression line was fixed at one, but the intercept is estimated from the data:

$$\log(\text{ASV Test Result}) = d + \log(\text{Panel Lead Level}) \qquad \text{(Eq 4)}$$

or

$$\text{ASV Test Result} = 10^{d} \times (\text{Panel Lead Level}) \qquad \text{(Eq 5)}$$

Because the UE/ASV test results tended to be less than the panel lead levels, the value of 10^d is typically between zero and one. Note in Figures 5 and 6 that the solid thin line is parallel to the bold line. The vertical distance separating these two lines is d, which is, thus, a measure of the lead recovery of the UE/ASV analysis for the given combination of experimental factors. For example, the closer the two solid lines, the greater is the lead recovery. In comparing Figures 5 and 6, it is evident that lead recovery for the combination of experimental factors represented in Figure 5 was much greater than that in Figure 6.

- A *dashed line*, representing a linear regression in which neither the slope nor the intercept were fixed. The extent to which the dashed line overlaps the solid thin line is a measure of the appropriateness of the assumption that the UE/ASV response is linearly related on the log scale to panel lead level for the combination of experimental factors. Again, in comparing Figures 5 and 6, the dashed line in Figure 5 is seen to be closer to overlapping the thin solid line than in Figure 6.

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* 5 operators x 2 apparatus x 2 lead pigment types x 2 substrate types, and 2 overlayers.

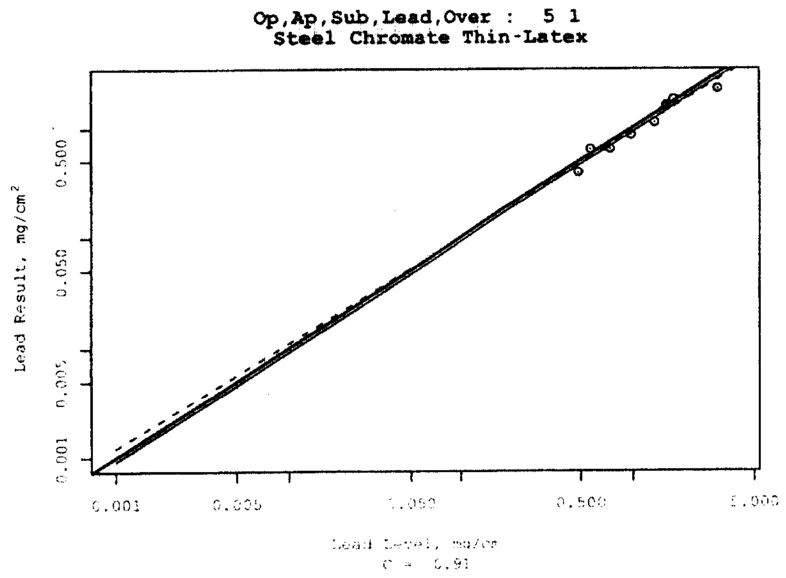

Figure 5. log(UE/ASV Lead Result Versus log(Test Panel Lead Level) for the Following Combination of the Experimental Variables: Operator 5, Apparatus 1, Steel Substrate, Lead Chromate Pigment, and Thin-Latex Overlayer. The Solid Bold Line Represents Ideal Performance, i.e., Complete Lead Recovery. The Thin Solid Line is From Linear Regression Analysis in Which the Slope of the Line was Fixed at One, but the Intercept is Estimated from the Data. The Dashed Line is From Linear Regression in Which the Slope of the Line was not Fixed.

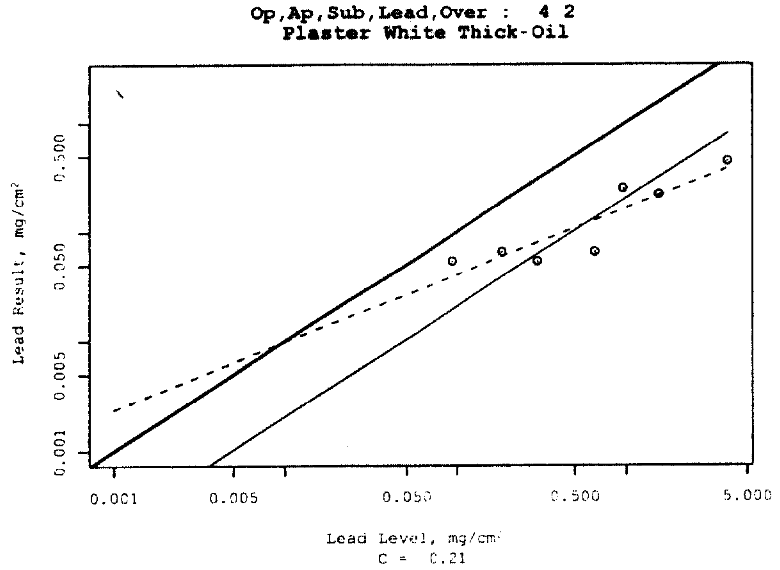

Figure 6. log(UE/ASV Lead Result Versus log(Test Panel Lead Level) for the Following Combination of the Experimental Variables: Operator 4, Apparatus 2, Plaster Substrate, White Lead Pigment, and Thick-Oil Overlayer. The Solid Bold Line Represents Ideal Performance, i.e., Complete Lead Recovery. The Thin Solid Line is From Linear Regression Analysis in Which the Slope of the Line was Fixed at One, but the Intercept is Estimated from the Data. The Dashed Line is From Linear Regression in Which the Slope of the Line was not Fixed.

- A *constant,* $C = 10^d$ (given below the x-axis label). C is an estimate of the fraction of lead recovery for the given combination of five experimental factors*. It is called the Recovery Constant and is the basis of further discussion and analyses of the data. Appendix B has tables of Recovery Constants calculated for data adjusted and unadjusted for calibration.

The Recovery Constants, ordered in increasing extent of recovery, are displayed in Figure 7A along with the corresponding combinations of five experimental factors associated with each of the constants for the data adjusted for calibration. For example, the lowest Recovery Constant (i.e., left most data point on the plot) corresponds to the following combination of experimental factors: *Overlayer Type* - thick-oil, *Apparatus No.* - 2, *Operator No.* - 4, *Lead Pigment Type* - Lead Chromate, and *Substrate Type* - Steel. The purpose of Figure 7A is to visualize the range of Recovery Constants for all combinations of the five experimental factors and to illustrate qualitatively the relative importance of these factors on lead recovery.

From Figure 7A, it is evident that, depending on the combination of the five experimental factors, the proportion of recovered lead (in percent) ranged from 28 % to 94 % with a median value of 63 %. For only about 28 % of the combinations of experimental variables were the proportions of recovered lead within 25 % of the test panel lead level. That is, in this controlled laboratory study using trained operators, lead recoveries determined according to the study protocol were often considerably less than the lead levels in the test panels. This was in sharp contrast to the previous studies [9-11,13] reported on the UE/ASV analyses of lead in paint, for which the recoveries were at least 75 %, and usually more. For example, analyses of NIST SRM 1579 yielded recoveries from 84 % to 95 % [10,11,13]. Additionally, depending on the paint-film substrate, the lead-recoveries determined by Ashley et al. [11] in their field study were generally about 76 % to 128 %.

In examining Figure 7A for qualitative effects of the five experimental factors on the UE/ASV analyses, it is apparent that the most dramatic effect was due to overlayer type. Note the preponderance of thick-oil overlayers in cases where the recovery was relatively low; whereas thin-latex overlayers were predominant in cases where the recoveries were relatively high. Figure 7A also shows evidence for operator and substrate effects. Operator 5 was included in many combinations having high recoveries; whereas Operator 4 tended to appear among many of the combinations having low recoveries. Additionally, plaster and steel substrates were among many of the high and low recovery combinations, respectively. Effects due to lead pigment type and apparatus do not appear to be present.

Figure 7B is a plot of the Recovery Constants in increasing extent of recovery for the 80 combinations of experimental variables for the data unadjusted for calibration. A notable difference between Figures 7A and 7B is that the percent recoveries now range from about 21 % to 91 %, with a median value of 55 %. The decrease in range and median values is a result of not adjusting for the low responses of the ASV instrument of Apparatus 2 at some lead concentrations. Observe that Figure 7B shows a moderate apparatus effect for the unadjusted data, as Apparatus 1 and Apparatus 2 tended towards yielding high and low recoveries, respectively. Note also in Figure 7B that the overlayer, operator, and substrate effects are still apparent.

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[*] To be precise, the least squares estimate of d is the difference between the mean log(ASV Test Result) and the mean log(Panel Lead Level). C is 10^d or, equivalently, the geometric mean of the ratios of these two quantities.

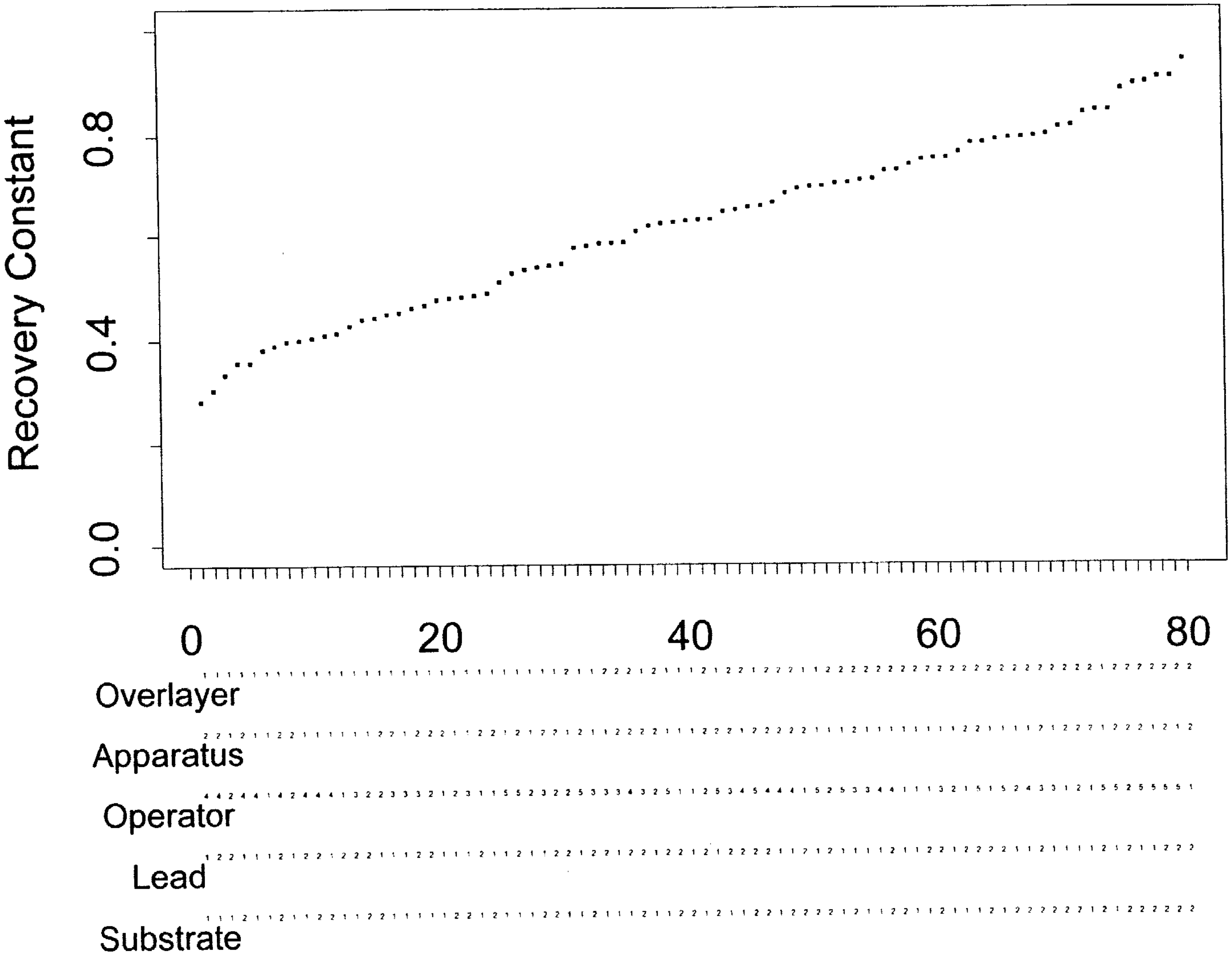

Figure 7A. Recovery Constant Versus Combination of Experimental Variables for the Data Adjusted for Calibration. (Overlayer 1 & 2 = Thick-Oil and Thin-Latex; Lead 1 & 2 = White Lead and Lead Chromate; Substrate 1 & 2 = Plaster and Steel)

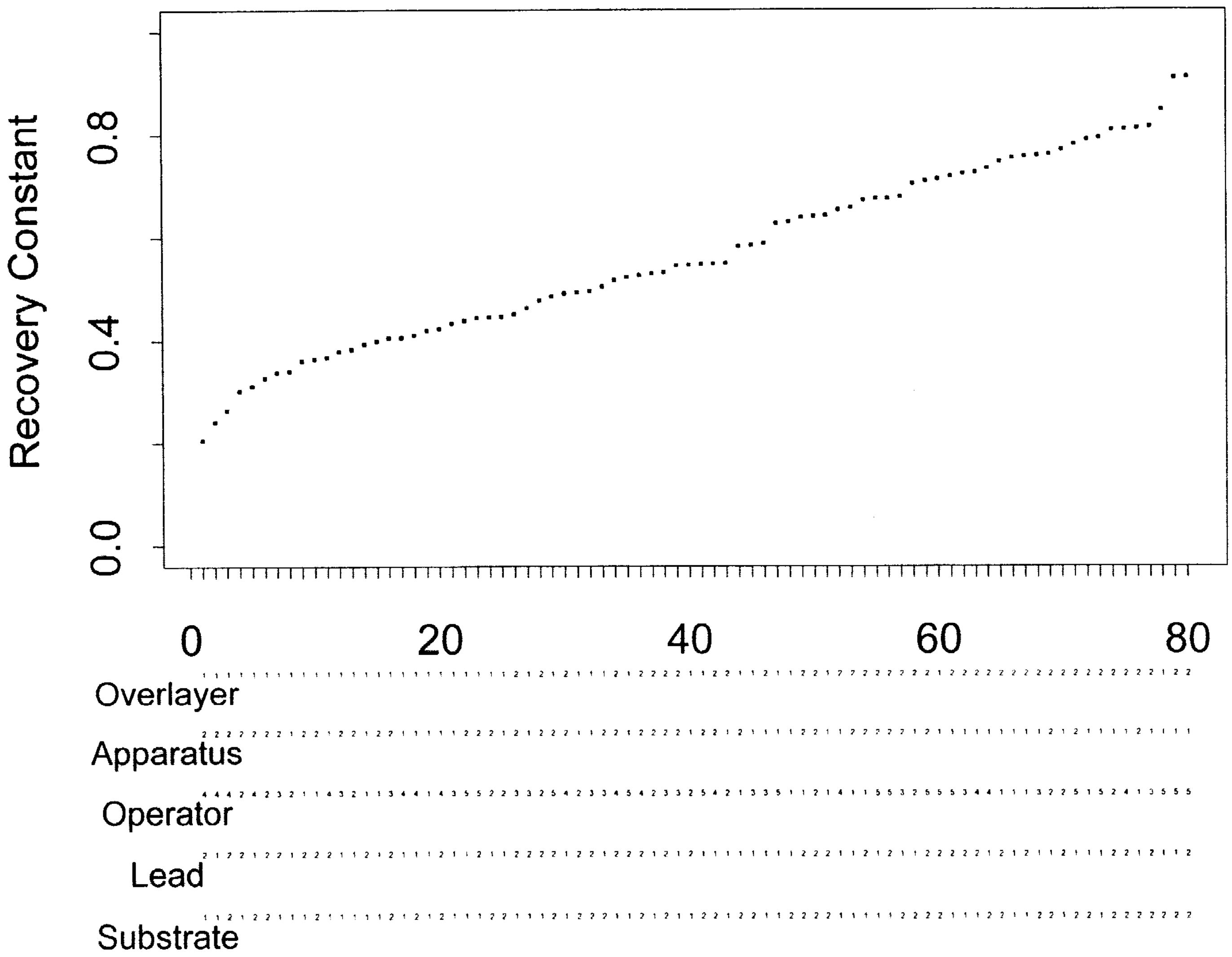

Figure 7B. Recovery Constant Versus Combination of Experimental Variables for the Data Unadjusted for Calibration. (Overlayer 1 & 2 = Thick-Oil and Thin-Latex; Lead 1 & 2 = White Lead and Lead Chromate; Substrate 1 & 2 = Plaster and Steel)

Analysis of variance was performed on the Recovery Constants to quantify the effects observed qualitatively in Figures 7A and 7B. As previously indicated, the overlayer effect evident from these figures was very large. This observation was confirmed from the further analysis. Consequently, separate analyses of variance were performed for the two overlayer types—thick-oil and thin-latex. For the data unadjusted for calibration, a large apparatus effect was observed, both as a main effect and as a part of several two-factor interactions. In contrast, the analysis of variance for the calibration-adjusted data indicated neither a significant apparatus effect nor any significant interactions. Further discussion below is restricted to calibration-adjusted data.

The results of analysis of variance models, which include all main effects and two-factor interactions, are summarized for "thick-oil" and "thin-latex" calibration-adjusted data in Tables 5A and 5B, respectively, using F-statistics and P-values. F-statistics substantially exceeding 1 tend to indicate that an effect is not due to chance. A measure of how unlikely that an observed effect is due to chance is provided by the P-value. For purposes of the present report, the P-value is the probability of observing an F-statistic as large or larger than the one obtained, if the effect were not present. Conventionally, effects which have P-values less than 0.05 are referred to as being statistically significant. From Tables 5A and 5B, it is seen that no interactions are statistically significant, and the only significant main effects are due to substrate and operator. However, these two effects are extremely significant, as indicated by the very small P-values. Regarding the substrate effect, further analysis indicated that steel substrates had higher lead recoveries than plaster substrates. This finding did not support a study premise that steel panels might be more difficult to sample than plaster panels. Moreover, it was in contrast to the field-study findings of Ashley et al. [11] who reported generally higher recoveries for plaster substrates than for metal substrates. Reasons why steel substrates had higher recoveries than plaster substrates in the present study were not investigated.

3.2.1 Additional Analysis. As indicated previously, data analysis using the proportionality model was also conducted for the data adjusted for calibration without excluding points recorded as "below the detection limit" to investigate the effect of these points on the calculated Recovery Constants. Because the model was on a log scale, for purposes of the analysis, lead levels of test panels having lead levels of 0 mg/cm^2 were assigned a lead level of 0.001 mg/cm^2. Similarly, results recorded as "below the detection limit" were set at a value of 0.001 mg/cm^2. This analysis found that the Recovery Constants calculated using all points were, in most cases, lower than those obtained when the "below the detection limit" points were excluded from analysis. That is, with all data points included, UE/ASV performance was seen to be generally less indicative of lead level in the specimens. This finding is illustrated graphically in Figure 8. In this figure, the values of the Recovery Constants with and without the "below the detection limit" points are given on the horizontal and vertical axes, respectively. The line corresponds to no difference between the Recovery Constants calculated with and without these points. Hence, the vertical distance between the line and the plotted point is the difference between the two analyses. Data points plotted above the line indicate that the Recovery Constant for a given combination of the five experimental factors was less when the "below the detection limit" points were included than when they were excluded.

Table 5A. Analysis of variance on the Recovery Constants for the calibration-adjusted data set for panels having thick-oil overlayers

Main Effects and Interactions	Analysis Result	
	F-Statistic	P-value
Operator	21.66	$1.7 * 10^{-6}$
Apparatus	0.444	0.514
Substrate	30.02	$4.1 * 10^{-5}$
Lead Pigment Type	2.94	0.105
Operator—Apparatus	1.33	0.298
Operator—Substrate	1.73	0.189
Operator—Lead Pigment Type	0.59	0.677
Apparatus—Substrate	0.40	0.533
Apparatus—Lead Pigment Type	0.76	0.395
Substrate—Lead Pigment Type	2.54	0.129

Table 5B. Analysis of variance on the Recovery Constants for the calibration-adjusted data set for panels having thin-latex overlayers

Main Effects and Interactions	Analysis Result	
	F-Statistic	P-value
Operator	12.33	$6.9 * 10^{-5}$
Apparatus	0.48	0.498
Substrate	35.98	$1.4 * 10^{-5}$
Lead Pigment Type	0.84	0.374
Operator—Apparatus	2.68	0.067
Operator—Substrate	1.64	0.209
Operator—Lead Pigment Type	1.05	0.412
Apparatus—Substrate	0.29	0.596
Apparatus—Lead Pigment Type	2.71	0.118
Substrate—Lead Pigment Type	2.77	0.115

In Figure 8, the numerical plot character indicates, for a given combination of the five experimental factors, the number of UE/ASV analyses for which the result was recorded as "below the detection limit" when the test panel lead level exceeded the detection limit of the ASV instruments (0.04 mg/cm^2). For example, note the many points having a "zero" plot character which is consistent with the UE/ASV findings (see Figure 4) that, for most analyses of specimens from test panels having lead, the result was quantitative (i.e., not recorded as "below the detection limit"). Note also that majority of the "zero" plot characters fall above the line, indicating that inclusion of the "below the detection limit" data points lowered the Recovery Constants.

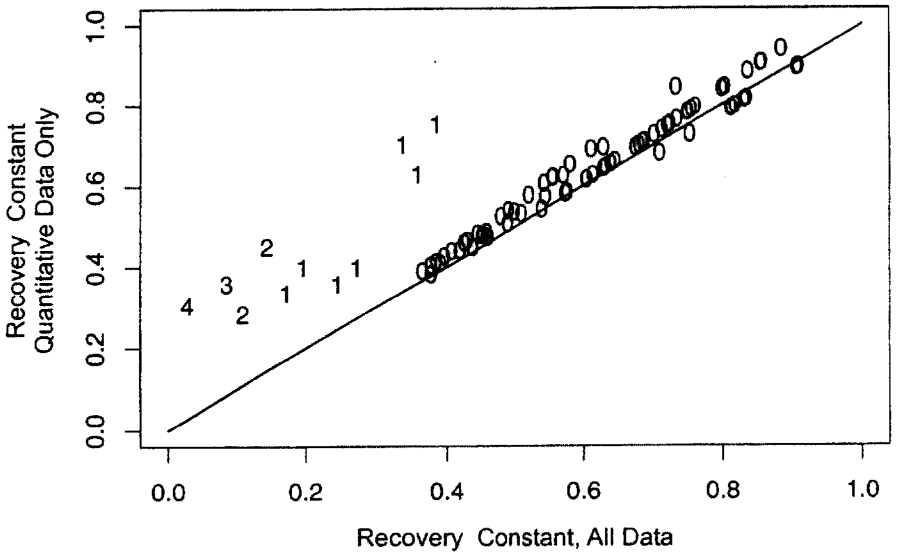

Figure 8. Comparison of Recovery Constants for Data Sets With and Without the "Below the Detection Limit" Points. The Plot Character Indicates the Number of Results Recorded as "Below the Detection Limit" for Cases in Which the Panel Lead Level was Below 0.04 mg/cm^2. The Line Corresponds to No Difference Between Recovery Constants Calculated With and Without the "Below the Detection Limit" Points.

The 18 data points for specimens from panels having lead, but for which the results were reported as "below the detection limit" are represented by the "non-zero" plot characters (i.e., 1, 2, 3 & 4) towards the left of the plot and above the line. Their positions, being above the line, indicate that the Recovery Constants always decreased when these 18 points were included in the data analysis. In some cases, the decrease was relatively large, as noted by the distance that these points lie above the line.

3.2.2 Discussions on Low Lead Recovery. The scope of the UE/ASV study did not provide for extensive investigation as to why the lead recoveries were, in many cases, considerably less than the lead levels of the test panels. It may be broadly hypothesized that the low lead recoveries were possibly associated with one or more of the following steps of the UE/ASV protocol of the study:

- Paint film removal from the substrate including:
  - incomplete removal of the lead-containing paint film from the substrate, and
  - loss of paint-film specimen during transfer from the substrate to the sonication tube.
- Grinding of the paint-film specimen in the sonication tube including:
  - loss of paint-film specimen because it was embrittled (due to dry ice cooling) and tended to "shatter" in the sonication tube during grinding. Such action might cause some loss of specimen from the tube, or deposition on the upper wall of the tube where it might not be covered with, or washed into, the acid solution during sonication.
- Sonication of the paint specimen in that:
  - the lead was incompletely solubilized during this step.

- ASV measurement of lead:
  - an error in the measurement including a paint-film matrix effect.

In considering, in turn, the potential contribution from each of these factors to the low lead recovery, the following comments and data are given:

- Incomplete removal of the lead-containing paint film from the substrate. When the substrate was plaster, it did not appear that incomplete removal was a contributing factor. Normally, the cork borer (See Table 3, section 2, step 2) penetrated through the paint film and into the plaster. A specimen "plug," held within the cork borer, was removed from the panel and normally consisted of paint film and some plaster. Alternatively, when the substrate was steel, the cork borer inscribed a circle in the paint film, which generally remained adhered to the steel substrate. The paint film within the inscribed circle was then scraped from the steel substrate. If scraping were incomplete, some residual paint film would be left on the steel substrate. Thus, in considering this factor, selected plaster and steel panels were examined after the operators had completed their analyses. No evidence that the paint films were not completely removed from the plaster substrates was found. However, for the steel panels, some specimen-sampling locations were seen to have some residual paint on the substrate, although the majority of the sampling locations examined were considered to be free of residual paint.

  As a measure of the effect of incomplete paint removal from steel substrates, residual paint was scraped from two previously sampled locations on each of three steel panels having lead levels of about 3 mg/cm^2. This residual paint was subjected to UE/ASV analysis. The results (Table 6) were that lead in the residual paint specimens ranged from about 3 % to 10 % of the lead levels of the paint films. The evidence from this limited experiment is that incomplete removal of the paint film from the steel substrate can occur and that, when it happens, it can account for some percentage (although perhaps small) of the unrecovered lead. This finding supported the study premise that steel panels may be difficult to sample. However, this is an apparent contradiction to the statistical analysis result discussed above that showed a significant substrate effect in which steel test panels had greater lead recovery than did plaster panels. Seemingly, whatever mechanism(s) contributed to the lower lead recovery for the plaster panels overrode the effects of instances of incomplete paint removal from steel panels.

Table 6. Results of UE/ASV analyses of residual paint film scraped from steel test panels

Test Panel ID No.	Test Panel Lead Level mg/cm^2	ASV Lead Analysis of Residual Paint Scraped from Previously Sampled Test Locations	
		Measured Results, mg/cm^2	Lead Recovery, %[a]
145	3.39	0.090	2.7
		0.201	5.9
215	3.20	0.340	10.6
		0.245	7.7
142	2.81	0.241	8.6
		0.173	6.2

[a] The percent is based on the lead level of the test panel.

- Loss of paint during transfer from the substrate to the sonication tube. No testing was conducted to examine this factor, so comments on its role are not presented.
- Loss of specimen during grinding. As with the previous factor, no testing was performed to examine the role of loss of specimen during grinding. The operators empirically learned that the dry-ice cooled, embrittled paint specimens tended to "shatter" in the sonication tubes during grinding, and they were cautious to prevent specimen loss from the tube. Moreover, the operators were trained that the entire ground paint specimen was to be covered with acid solution prior to sonication.
- Incomplete lead solubilization during paint specimen sonication. The contribution of incomplete lead solubilization during sonication was examined in a limited experiment. Specimens from six paint test panels having lead and one paint-film control (i.e., lead level of 1 mg/cm^2) were subjected to the UE/ASV procedure. The selected panels had plaster substrates and white lead pigments so that potential for loss of specimen during sampling would be minimized and the potential for solubilizing the lead during sonication would be maximized. After sonication, the sonication tubes were centrifuged and the solutions above the residues in the bottom of the tubes were decanted. The tubes were allowed to remain at ambient laboratory conditions until no liquid was apparent. The tubes with the sonication residues were then sent to a commercial NLLAP laboratory, and the residues analyzed for the presence of lead. The results are given in Table 7, where it is evident that lead in the residues ranged from 5 % to 58 % of the level levels of the test panels. Also, it is seen that specimens from test panels having thick-oil overlayers had greater percents of lead in the sonication residue than those having thin-latex overlayers. This observation was consistent with a strong overlayer effect (Figures 7A and 7B). It may have been that the thick-oil overlayers were more difficult to sonicate, or to grind before sonication, than the thin-latex overlayers. In this regard, it is noted that the UE/ASV measurements on paint-film controls, which were thin (about 0.08 mm) and had no overlayers, generally provided mean recoveries within 10 % of the lead level (for data that were adjusted for calibration).

In further regard to specimen thickness, Grohse et al. [21] recommended that the mass of a paint specimen for sonication should be 100 mg. The recommendation was based on the finding that some paint samples showed a decline in recovery using ultrasonic extraction when specimen mass was more than 100 mg, although their data seemed to show little difference in recovery between specimens (from the same paint sample) having masses of 100 mg and 250 mg. The 100 mg specimen mass recommended by Grohse et al. is less than that of some representative thick-oil specimens for which mass measurements were performed (Section 2.3).

Table 7. Results of lead analysis of residue in sonication tubes

Test Panel ID No.	Substrate Type	Lead Pigment Type	Overlayer Type	Test Panel Lead Level	Lead Analysis of Residue After Sonication	
				mg/cm^2	Results, mg/cm^2	Lead recovery, %[a]
102	Plaster	White	Thick – Oil	0.96	0.41	43
279	Plaster	White	Thick – Oil	1.54	0.73	48
127	Plaster	White	Thick – Oil	3.88	2.24	58
256	Plaster	White	Thin – Latex	1.04	0.05	5
251	Plaster	White	Thin – Latex	1.53	0.29	19
177	Plaster	White	Thin – Latex	3.53	1.22	35
Paint-Film Control	---	---	---	1	0.04	4

[a]The percent is based on the lead level of the test panel.

Regarding grinding, based on comments by Ashley et al. [11], it might be asked whether the grinding procedure in the UE/ASV protocol used in the present study was adequate. In the present study, grinding of the dry-ice cooled paint-film specimens was performed according to the method that was taught in the UE/ASV training session, and was generally carried out for about 30 s to 2 min or 3 min. These times were consistent with the supplier's instructions that direct grinding to be performed for "... about 15 s. Repeat the process as necessary until all large particles are broken down." In contrast to the present study, Ashley et al. [11] indicated that, in their field study, "typical grinding times ranged from 10 min to more than 30 min." Moreover, in discussing the importance of proper grinding, they also commented that: "The consistency of the grinding process for different types and ages of paints may present problems when one is using this [UE/ASV] method ..." They also indicated that samples, considered to be inadequately ground, resulted in lower lead recoveries using field ultrasonic extraction than those obtained in the laboratory with hot-plate digestion.

- Measurement error including a paint-film matrix effect. Measurement error including a paint-film matrix effect was not considered to be a major contributor to the low lead recoveries from the test panels. As discussed in Section 4.1, the quality assurance procedures taken during the study indicated that instrument drift was not an issue, and a paint-film matrix effect was not found. Moreover, the low readings of the ASV instrument for Apparatus 2 at some lead concentrations (Figure 2) can be adequately resolved by calibration adjustment.

3.3 Factors Associated with "Below the Detection Limit" Results

Table 8 provides a summary of the experimental factors associated with the 18 analyses for which specimens were sampled from test panels having lead, but for which the operators reported the result as "below the detection limit." Review of the table suggests that these "below the detection limit" findings may be related to differences in technique among operators. Operator 4 accounted

Table 8. Summary of the test variables for panels having lead, but for which the UE/ASV results were recorded as "below the detection limit"

Test Panel Lead Level mg/cm^2	Test Panel ID No.	Operator No.	Apparatus No.	Difficult To Grind	Overlayer Type	Lead Pigment Type	Substrate Type
1.44	183	2	1	No	Thick – Oil	Lead Chromate	Plaster
0.09	285	2	2	Yes	Thick – Oil	White Lead	Plaster
0.50	281	3	1	Yes	Thick – Oil	White Lead	Plaster
0.66	292	3	1	No	Thick – Oil	White Lead	Plaster
0.74	216	3	1	No	Thin – Latex	Lead Chromate	Steel
3.20	215	3	2	No	Thin – Latex	White Lead	Steel
0.09	285	4	1	No	Thick – Oil	White Lead	Plaster
0.43	129	4	1	No	Thick – Oil	White Lead	Plaster
0.96	102	4	1	No	Thick – Oil	White Lead	Plaster
1.62	253	4	1	No	Thick – Oil	Lead Chromate	Plaster
0.09	168	4	2	Yes	Thick – Oil	White Lead	Steel
0.43	129	4	2	No	Thick – Oil	White Lead	Plaster
0.50	281	4	2	No	Thick – Oil	White Lead	Plaster
0.51	140	4	2	No	Thick – Oil	Lead Chromate	Plaster
1.44	183	4	2	Yes	Thick – Oil	Lead Chromate	Plaster
1.62	253	4	2	Yes	Thick – Oil	Lead Chromate	Plaster
2.30	330	4	2	No	Thick – Oil	Lead Chromate	Plaster
0.18	180	5	2	No	Thick – Oil	White Lead	Steel

for 11 of the 18 analyses (i.e., 61 %). Moreover, of these 11 analyses, 10 cases were for plaster test panels having thick-oil overlayers. Investigation of the cause(s) for these 18 "below the detection limit" results was beyond the scope of the study.

3.4 Specimens Noted as Difficult to Grind

During the course of the study, the operators noted that specimens from 109 test panels were difficult to grind. Although this was a subjective judgment, it was of interest to know the characteristics of the test panels for which such a judgment was made. The distributions according to operator and test panel variables were as follows:

- Operator: Op1 – 26 %; Op2 – 11 %; Op3 – 21 %; Op4 – 23 %; Op5 – 19 %.
- Overlayer: Thick/Oil – 95 %; Thin/Latex – 5 %.
- Lead Pigment: White Lead – 50 %; Lead Chromate – 50 %.
- Substrate: Plaster – 54 %; Steel – 46 %.

As seen above, 95 % of the 109 specimens considered to be difficult to grind had thick–oil overlayers. This observation was consistent with the analysis of the main data set showing a strong overlayer effect in which the thick-oil panels provided lower lead recoveries than the thin-latex panels.

4. SUMMARY AND CONCLUSIONS

A laboratory study was conducted to evaluate the reliability of commercial, field-portable ultrasonic extraction-anodic stripping voltammetry (UE/ASV) for determining the lead levels of laboratory-prepared paint films when tests were conducted by certified lead inspectors trained to conduct UE/ASV testing. Two UE/ASV apparatuses—both obtained from the same supplier—were used to conduct more than 900 tests in a full factorial experiment investigating the effects of lead level, apparatus, lead pigment type, operator, paint-film substrate, and the overlayer film applied to the lead-based paint film. The test panels included lead-based paint films prepared with either white lead or lead chromate pigments mixed in an oil-based paint. For each pigment type, these panels had 10 lead levels ranging from 0 mg/cm^2 to 3.5 mg/cm^2. The paint films were adhered to steel or plaster substrates, which were a priori considered to be difficult or easy to sample, respectively. For the steel panels, the paint film was generally scraped from the substrate; whereas, for the plaster panels, it was removed by boring. The overlayers covering the lead-based films were either a thickly applied oil-based paint (about 0.75 mm to 1.4 mm), or a thinly applied latex paint (about 0.13 mm to 0.28 mm). Five operators, four of whom were certified lead inspectors or risk assessors, participated in the testing. The fifth operator was a NIST laboratory technician. All operators were trained by a representative of the UE/ASV supplier to conduct the tests using a written protocol developed from the supplier's instructions, and all received a certificate of successful course completion.

The analyses and discussions of the results addressed: (1) UE/ASV response as a function of the lead level of the test panel, and (2) effects of the experimental variables, or interactions among them, on UE/ASV response. Preliminary graphical analyses suggested a reasonably constant proportionality between the UE/ASV response and the lead level of the test panels. The constant of proportionality in these relationships appeared to be less than one indicating less than complete lead recovery from the paint films. Consequently, detailed analyses of the data focused on determining these constants of proportionality, defined as Recovery Constants, and on establishing how these constants depended on the experimental factors.

Formal statistical analysis was performed in three stages. First, separate log-log plots of UE/ASV response versus panel lead level were prepared for each of the 80 combinations of experimental factors. A Recovery Constant was then determined for each combination of experimental factors. Next, ordered plots of the Recovery Constants were examined to ascertain the qualitative importance of each of the experimental factors. Finally, this qualitative analysis of the importance of the experimental factors was confirmed by analysis of variance on the Recovery Constants.

The main conclusions of the study are:

- As purchased, one of the two commercial ASV instruments was out-of-calibration. Both instruments were used in the study, because the experimental design included a preliminary examination of instrument effect on UE/ASV response, when investigated using "as received" instrumentation. The data were analyzed both as "unadjusted for calibration" and "adjusted for calibration."
- UE/ASV results for the 80 specimens sampled from test panels having a 0 mg/cm^2 lead level were always "below the detection limit," which was 0.04 mg/cm^2.
- Lead levels determined according to the UE/ASV study protocol were often considerably less than the test panel lead levels. Depending on the combination of experimental factors—apparatus, operator, lead pigment type, substrate type, and overlayer—the proportion of recovered lead for the data set adjusted for calibration ranged from 28 % to 94 %, with a median of 63 %. These findings are in sharp contrast with previously published results of an UE/ASV field study in which lead recoveries generally ranged from 75 % to more than 100 %.
- Eighteen analyses of test panels having lead levels of ≥ 0.1 mg/cm^2 were "below the detection limit." These 18 analyses were 2.5 % of those performed on specimens from panels having lead.
- ASV measurement error did not appear to play a role in the low lead recoveries based on quality assurance measures. The low response of the ASV instrument of Apparatus 2 at the lower lead concentrations used to check the instrument calibration could be accounted for by a calibration adjustment.
- A key contributor to the low lead recoveries appeared to be incomplete lead solubilization during paint specimen sonication. Analysis of a limited number of paint-film residues remaining in the sonicator tubes after sonication found lead levels ranging from about 5 % to 58 % of the lead levels of the test panels. Reasons for these findings were not investigated and it is not known whether specimen sonication, specimen grinding before sonication, or interaction between these two factors played a role in the incomplete lead solubilization. Based on discussions in the literature, questions were raised regarding the grinding and sonication procedures performed using the protocol in the present study.
- Another contributor to the low lead recoveries, in the case of steel panels, appeared to be incomplete removal of the paint-film specimen from the substrate. Analysis of a limited number of operator-sampled test locations (where some paint-film residue was observed) found lead levels ranging from about 3 % to 10 % of the lead levels of the test panels. Paint-film residue was not found on the plaster samples at locations sampled by the operators.
- The major experimental variable affecting UE/ASV response was overlayer with test panels having thick-oil overlayers yielding lower lead recoveries than those with thin-latex overlayers. It may have been that thick-oil overlayers were more difficult to sonicate, or grind before sonication, than were thin-latex overlayers. The individual effects of overlayer type and overlayer thickness were not examined, because test panels for such an examination were not available for the study.
- Effects of the other experimental variables incorporated in the study—apparatus, lead pigment type, operator, and substrate—on UE/ASV response were considered primarily for the

calibration-adjusted data. Operator and substrate were found to be significant. Regarding the substrate effect, steel panels had greater lead recoveries than plaster panels, which did not support a study premise that steel panels might cause lower recoveries because they might be more difficult to sample than plaster panels. This substrate effect was seemingly in contrast with the observation that, in some cases, operators did not remove all the paint film that was adhered to the steel panel. Reasons for the observed substrate effect were not investigated.

- No effects were found for lead pigment type or apparatus for the calibration-adjusted data. However, an apparatus effect was present for the data unadjusted for calibration.

5. RECOMMENDATIONS

It is recommended that the results of this study be used to design experiments to investigate the sensitivity of the UE/ASV procedure to changes in the test conditions. The purpose would be to ascertain the variables that strongly influence the measurements provided by the experimental procedure and to determine how closely these variables need to be controlled. Based on the findings of the present study, two issues are of concern, namely: the generally low lead recovery (i.e., a median value of about 60 %) and the failure to detect lead in specimens from panels having lead levels of ≥ 0.1 mg/cm^2. These findings are, as was discussed previously, in contrast with previously published results showing lead recoveries of 75 % and above. Sensitivity testing is suggested because a key contributor to low lead recovery appeared to be associated with the steps used in preparing specimens for ASV measurements, for example, inadequate lead grinding before and/or incomplete solubilization during sonication, and not with the ASV measurements themselves. Hence, sensitivity experiments to examine controllable variables associated with grinding such as the degree (i.e., resultant particle size) and time, and with sonication such as time, temperature, and power are recommended.

6. ACKNOWLEDGMENTS

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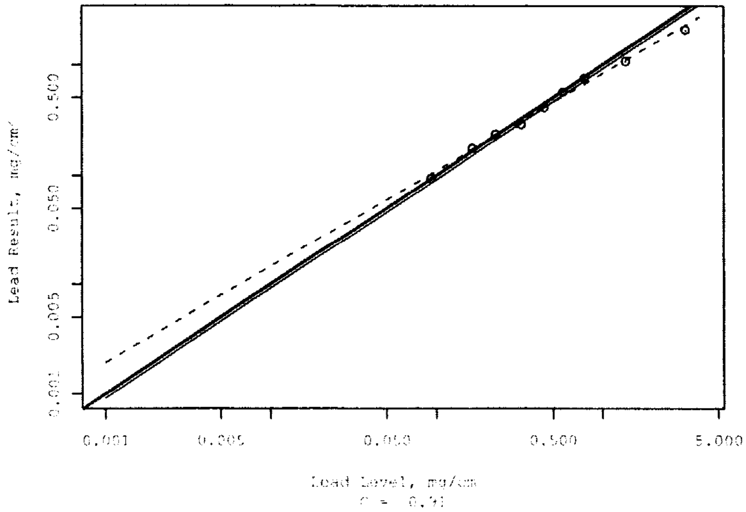
APPENDIX A. PLOTS OF UE/ASV MEASURED RESULT VERSUS PANEL LEAD LEVEL FOR COMBINATIONS OF EXPERIMENTAL FACTORS

This Appendix provides log-log plots of UE/ASV measured result, unadjusted for calibration, versus test panel lead level for each of the 80 combinations of five experimental factors—operator, apparatus, substrate, lead pigment, and overlayer—included in the test program. The unadjusted data are given because they are the data measured by the operators. A discussion of the features of these plots is given in Section 3.2 of the Main Text. The identification of the specific experimental factors is noted at the top of each plot and has the following key:

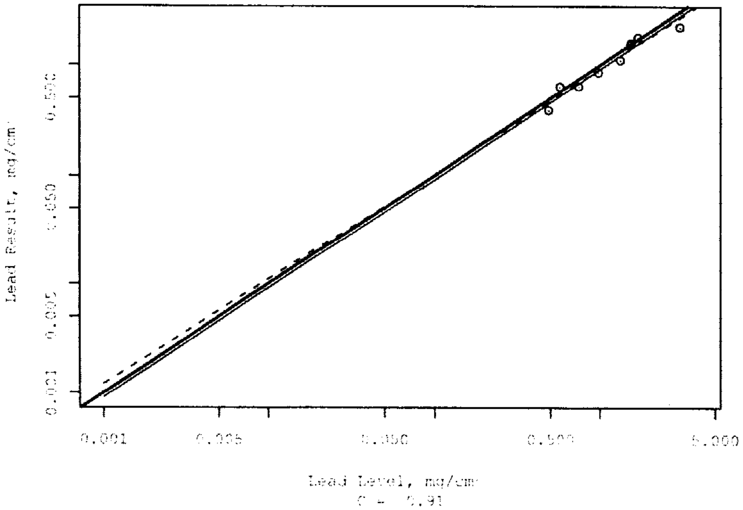
Experimental Factor	Descriptor
Operator (Op)	1 through 5
Apparatus (Ap)	1 and 2
Substrate (Sub)	Steel and Plaster
Lead Pigment (Lead)	White (for White Lead) Chromate (for Lead Chromate)
Overlayer (Over)	Thick-Oil and Thin-Latex

The plots are presented in decreasing order of lead recovery, as indicated by the Recovery Constant, C, given on each plot below the x-axis label.

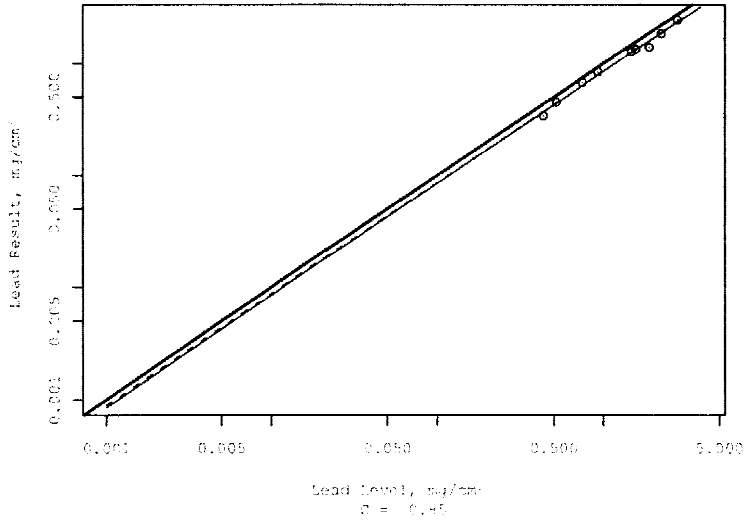
Op,Ap,Sub,Lead,Over : 5 1
Steel White Thin-Latex



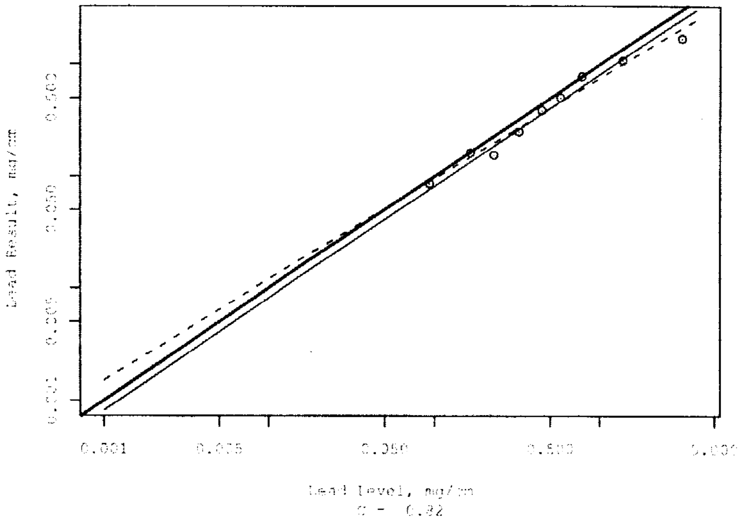
Op,Ap,Sub,Lead,Over : 5 1
Steel Chromate Thin-Latex



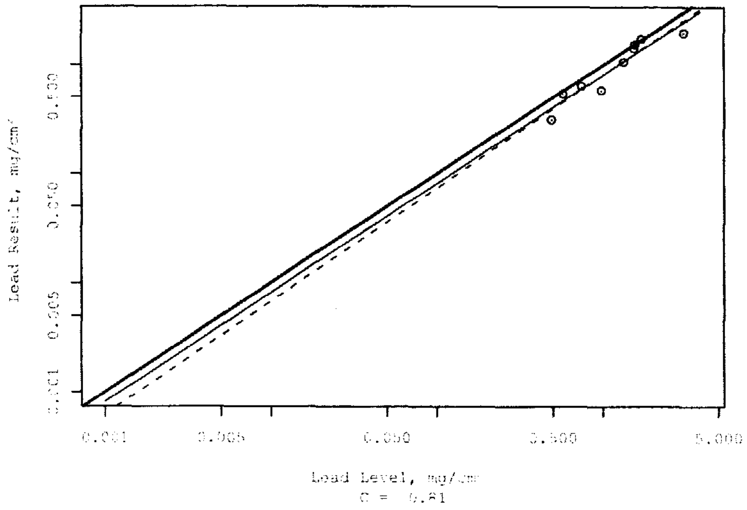
Op,Ap,Sub,Lead,Over : 5 1
Steel Chromate Thick-Oil



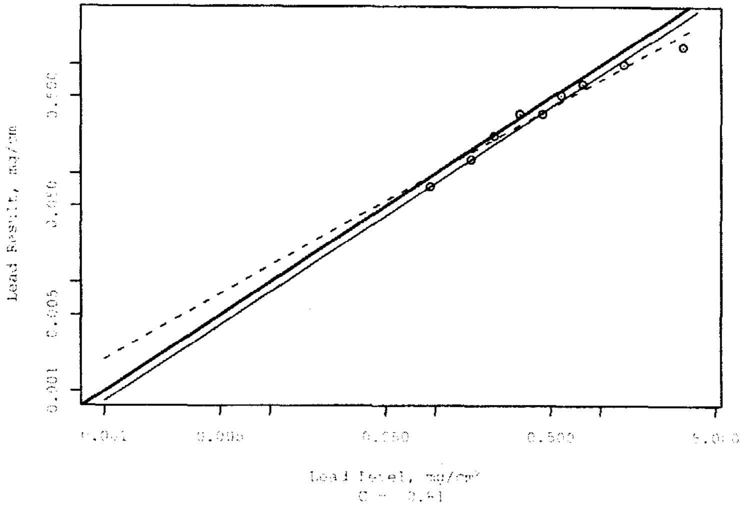
Op,Ap,Sub,Lead,Over : 3 1
Steel White Thin-Latex



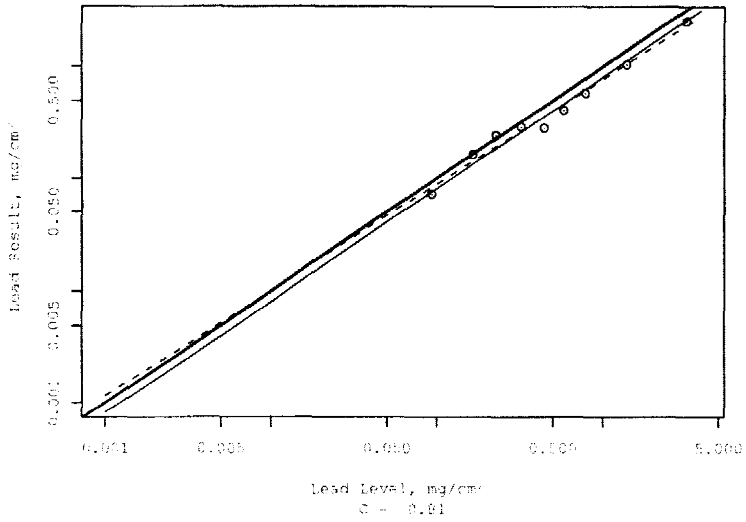
Op,Ap,Sub,Lead,Over : 1 2
Steel Chromate Thin-Latex



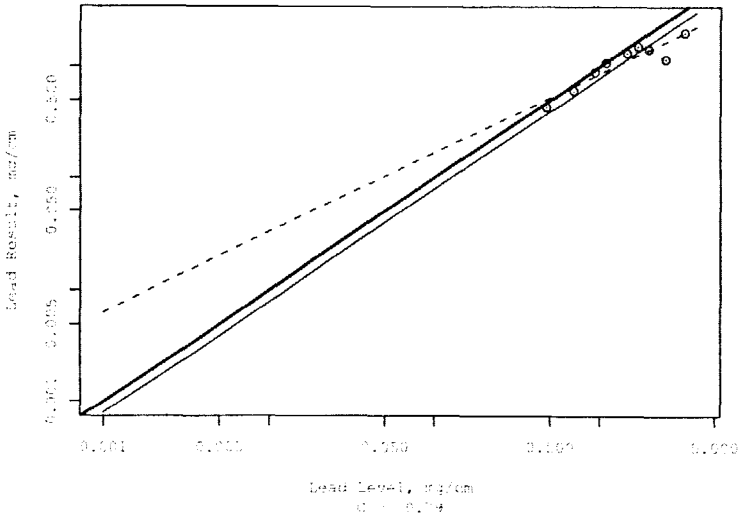
Op,Ap,Sub,Lead,Over : 4 1
Steel White Thin-Latex



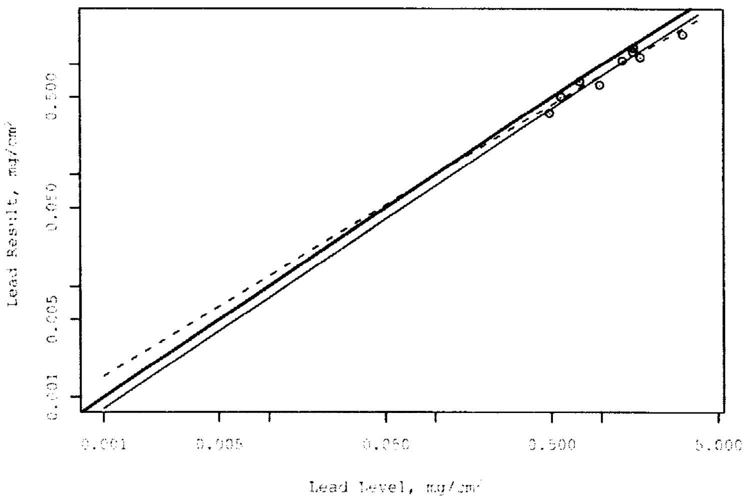
Op,Ap,Sub,Lead,Over : 2 1
Steel White Thin-Latex



Op,Ap,Sub,Lead,Over : 5 1
Plaster Chromate Thin-Latex

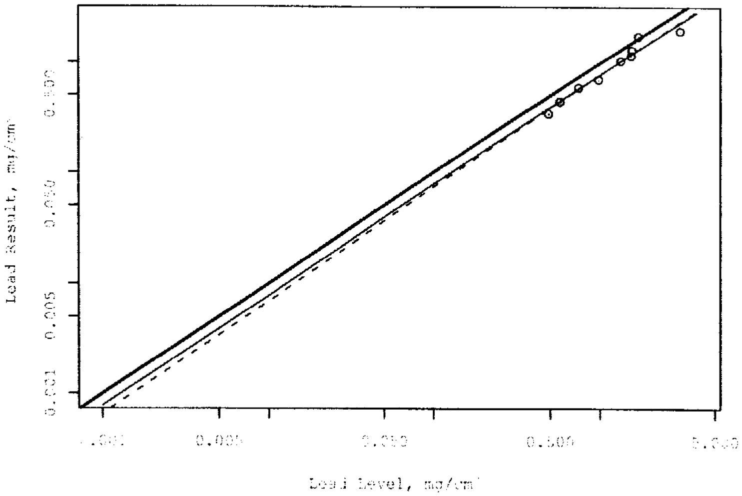


Op,Ap,Sub,Lead,Over : 1 1
Steel Chromate Thin-Latex



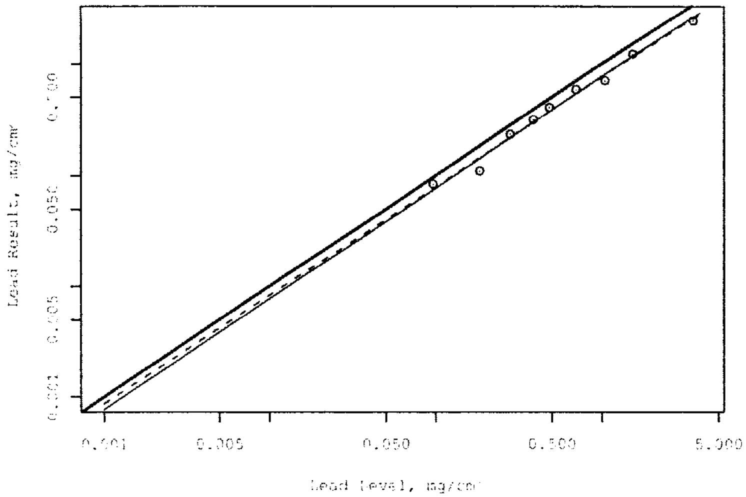
C = 0.79

Op,Ap,Sub,Lead,Over : 5 2
Steel Chromate Thin-Latex



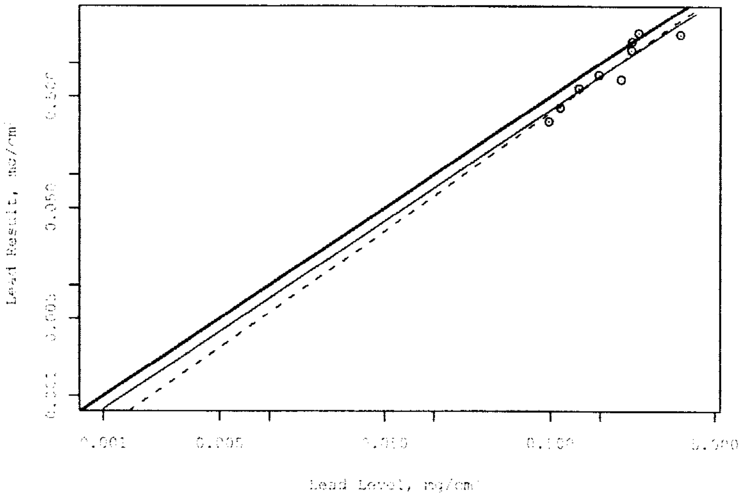
C = 0.78

Op,Ap,Sub,Lead,Over : 2 1
Plaster White Thin-Latex



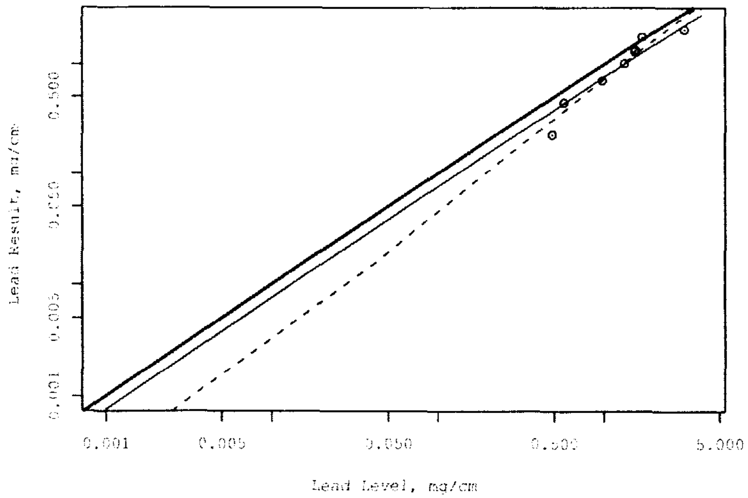
C = 0.77

Op,Ap,Sub,Lead,Over : 2 2
Steel Chromate Thin-Latex



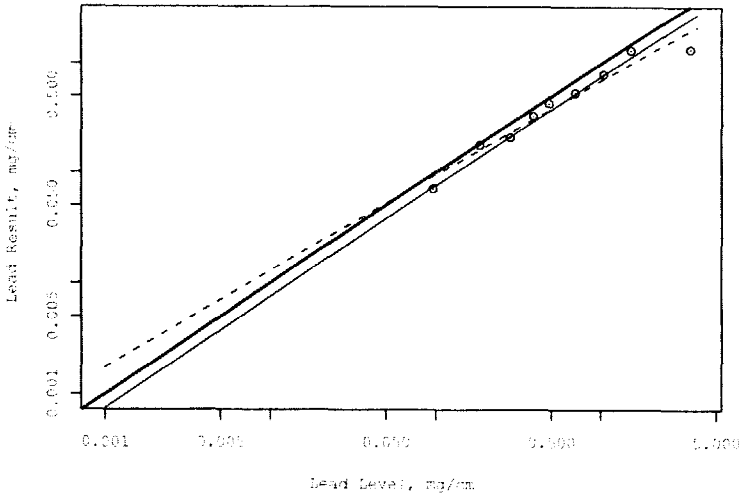
C = 0.76

Op,Ap,Sub,Lead,Over : 3 1
Steel Chromate Thin-Latex



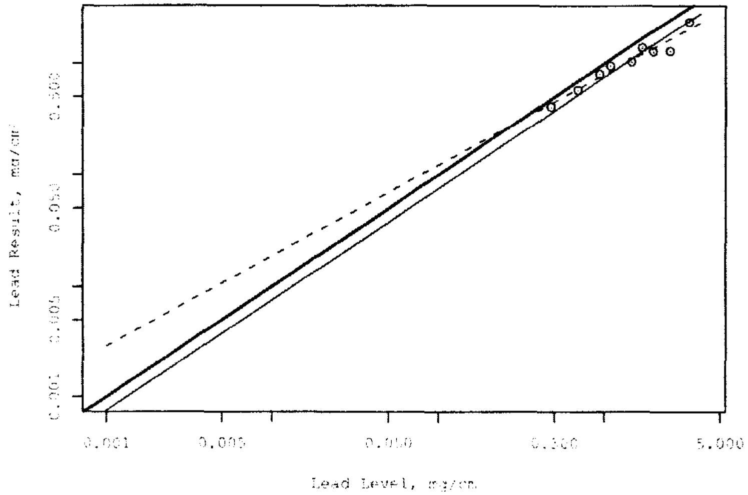
C = 0.76

Op,Ap,Sub,Lead,Over : 1 1
Plaster White Thin-Latex



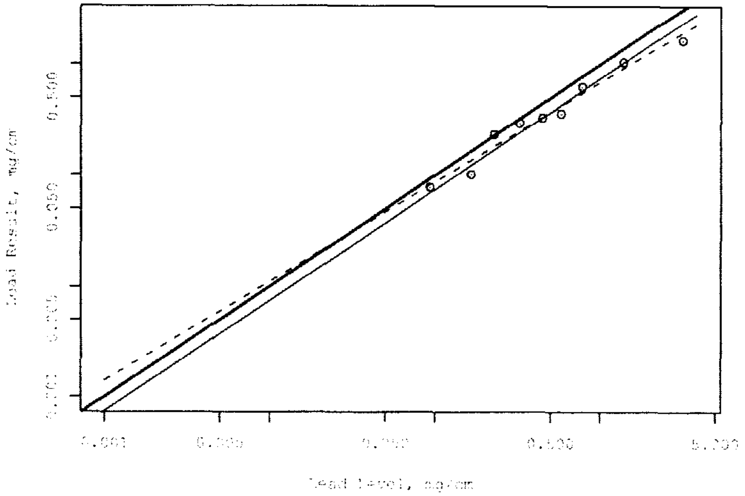
C = 0.75

Op,Ap,Sub,Lead,Over : 1 1
Plaster Chromate Thin-Latex



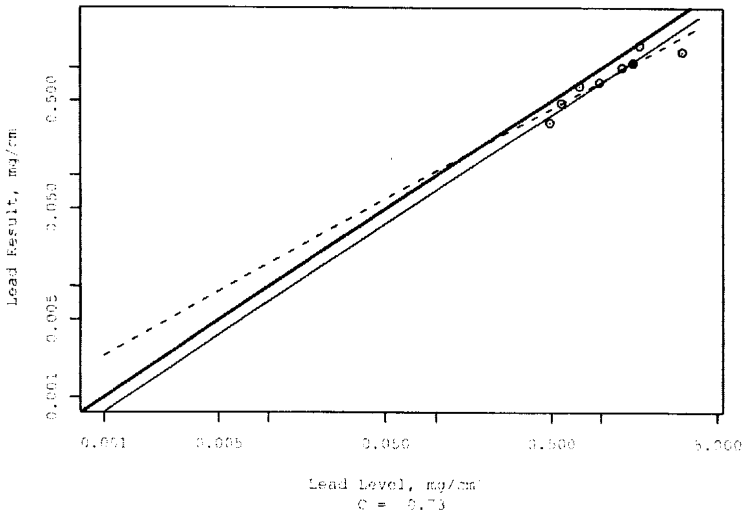
C = 0.75

Op,Ap,Sub,Lead,Over : 1 1
Steel White Thin-Latex

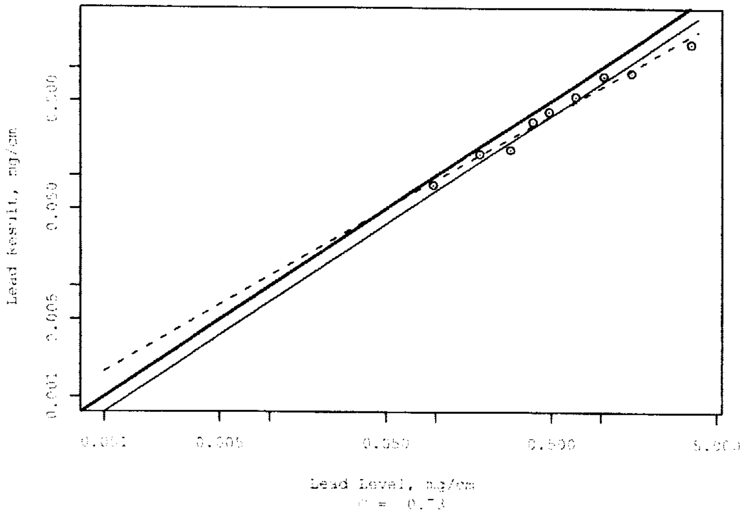


C = 0.74

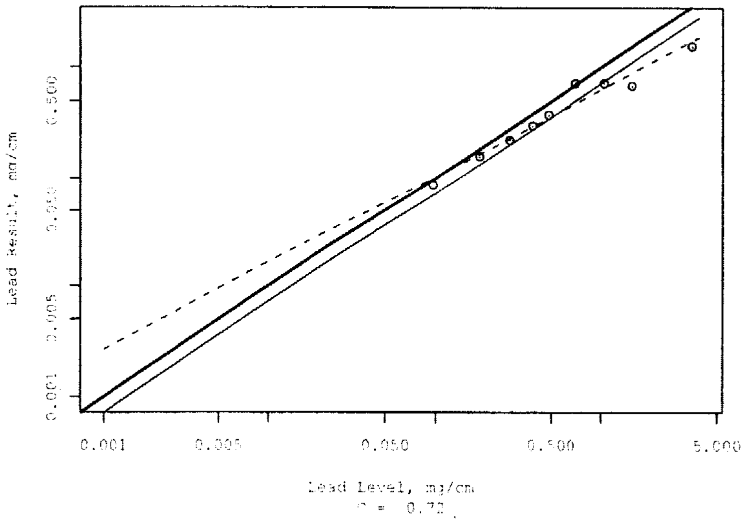
Op,Ap,Sub,Lead,Over : 4 1
Steel Chromate Thin-Latex



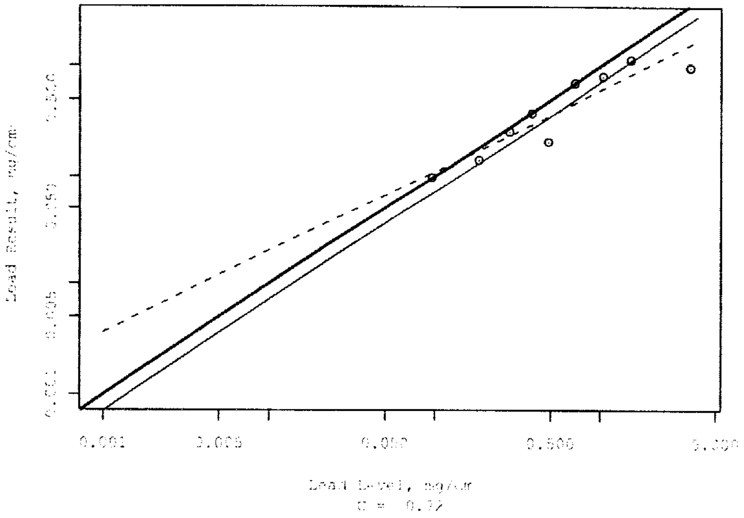
Op,Ap,Sub,Lead,Over : 4 1
Plaster White Thin-Latex

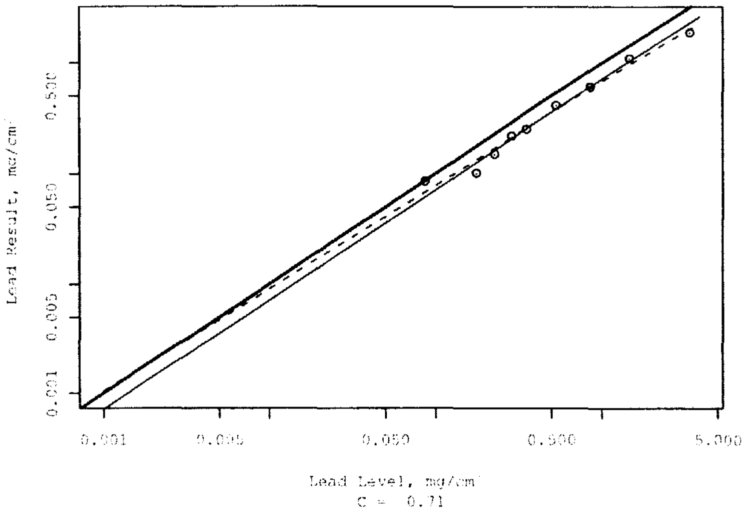


Op,Ap,Sub,Lead,Over : 3 1
Plaster White Thin-Latex

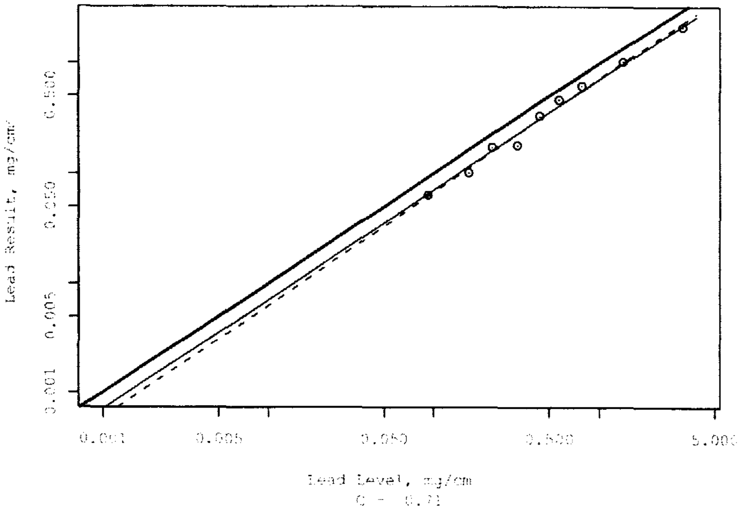


Op,Ap,Sub,Lead,Over : 5 1
Plaster White Thin-Latex

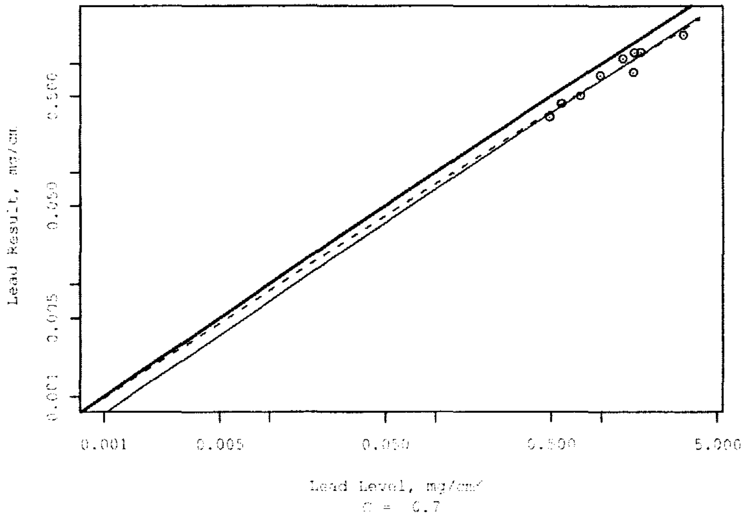




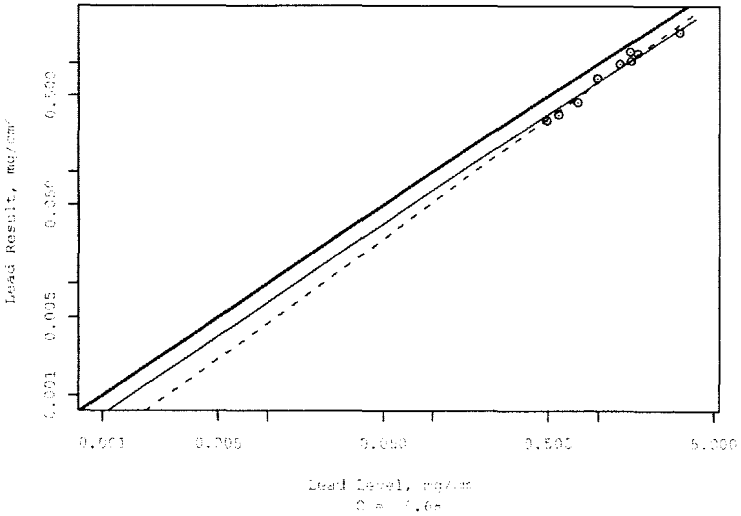
Op,Ap,Sub,Lead,Over : 5 1
Steel White Thick-Oil



Op,Ap,Sub,Lead,Over : 5 2
Steel White Thin-Latex

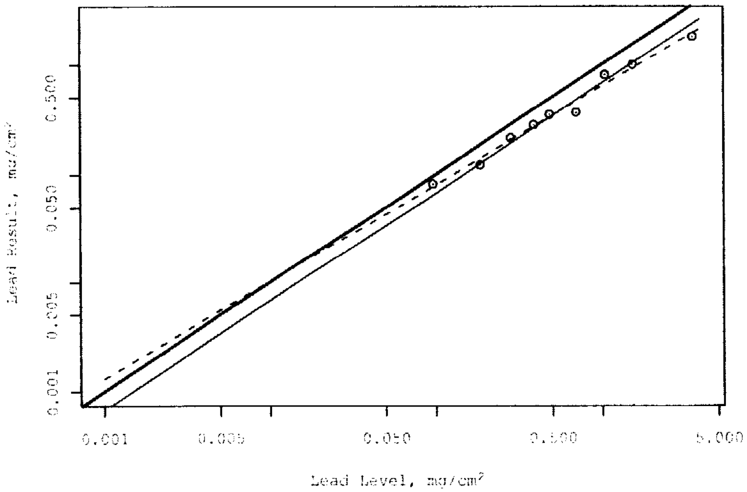


Op,Ap,Sub,Lead,Over : 2 1
Steel Chromate Thin-Latex



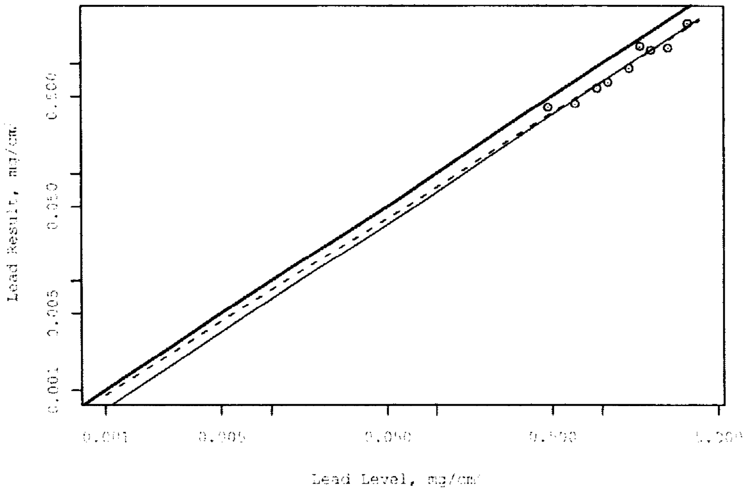
Op,Ap,Sub,Lead,Over : 3 2
Steel Chromate Thin-Latex

Op,Ap,Sub,Lead,Over : 5 2
Plaster White Thin-Latex



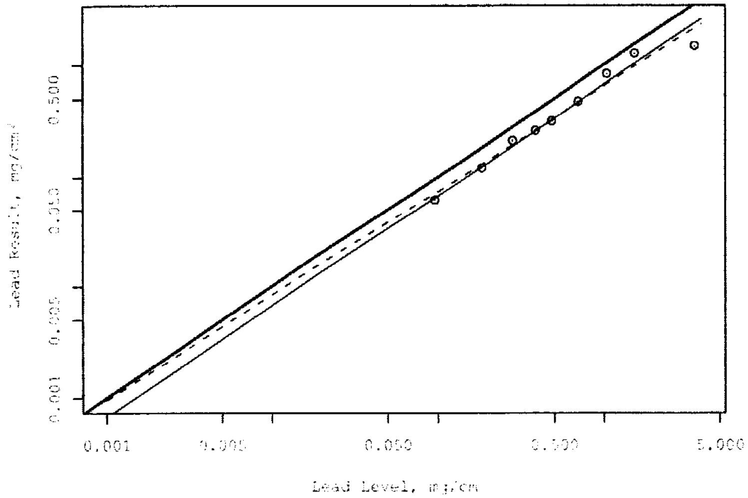
C = 0.67

Op,Ap,Sub,Lead,Over : 5 2
Plaster Chromate Thin-Latex



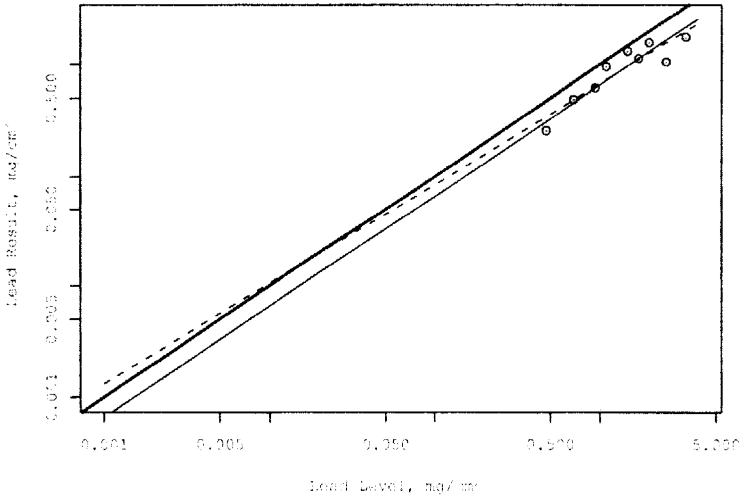
C = 0.67

Op,Ap,Sub,Lead,Over : 1 2
Plaster White Thin-Latex

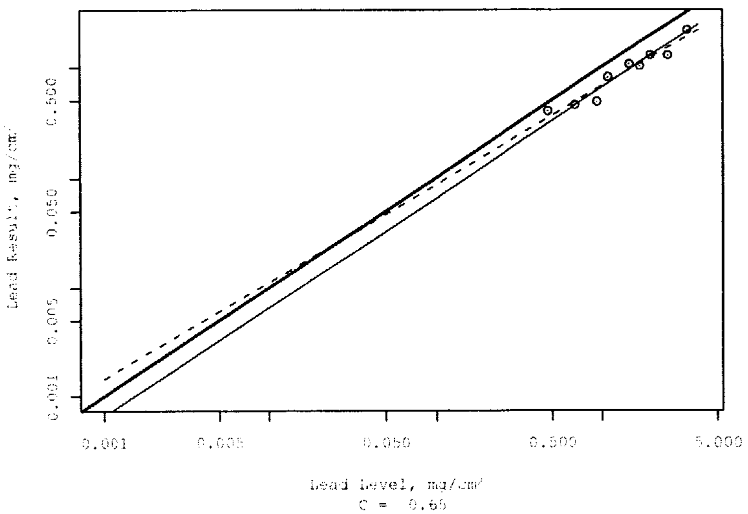


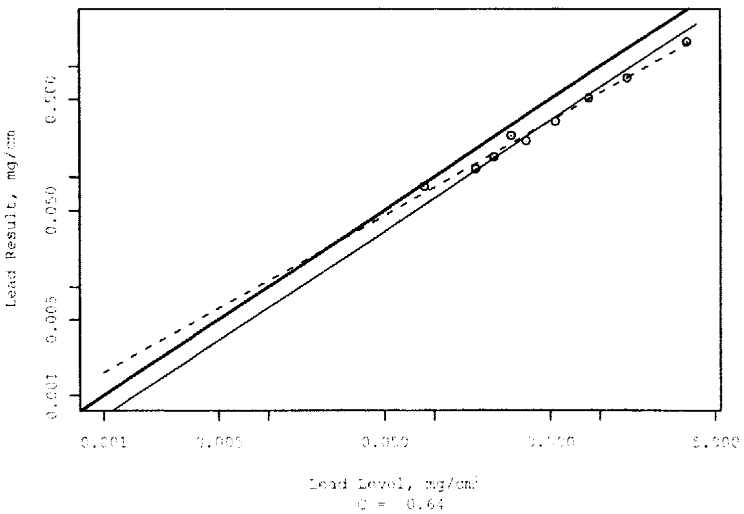
C = 0.67

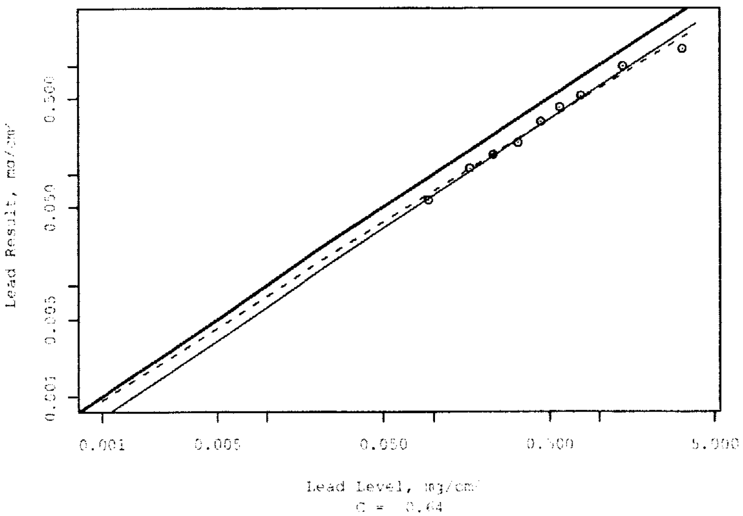
Op,Ap,Sub,Lead,Over : 1 2
Plaster Chromate Thin-Latex

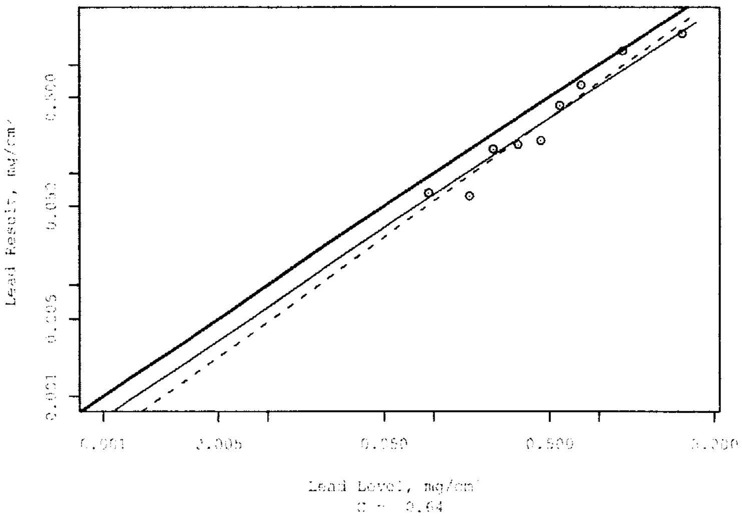


C = 0.66

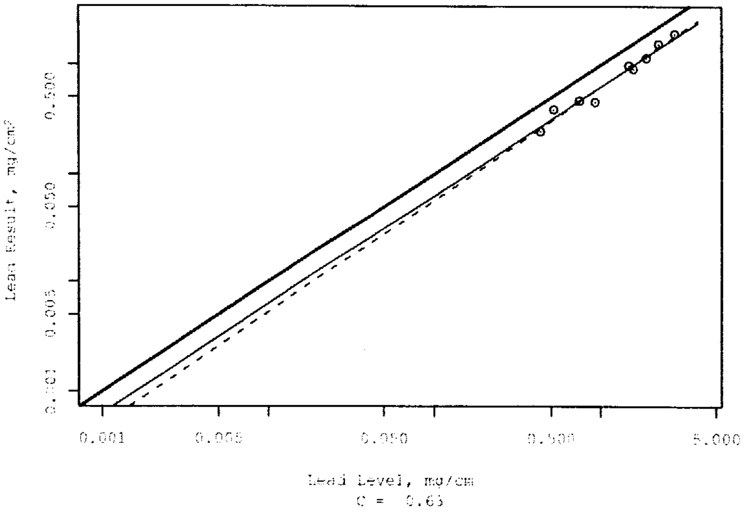

Op,Ap,Sub,Lead,Over : 4 1
Plaster Chromate Thin-Latex


Op,Ap,Sub,Lead,Over : 1 1
Steel White Thick-Oil

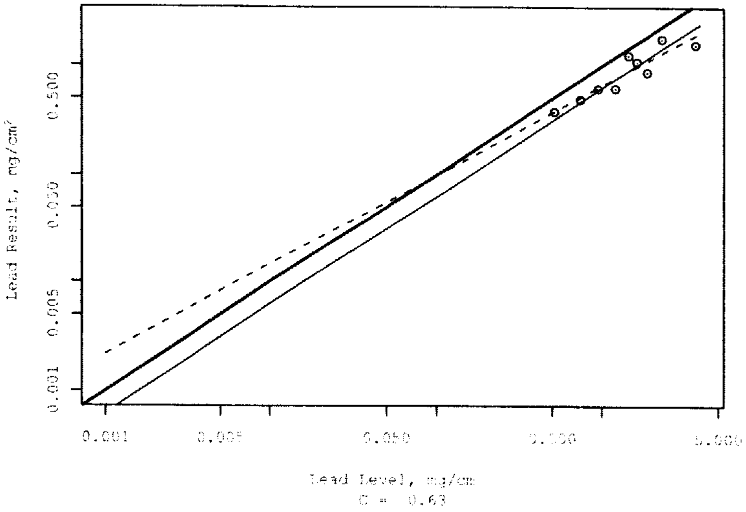

Op,Ap,Sub,Lead,Over : 2 2
Steel White Thin-Latex


Op,Ap,Sub,Lead,Over : 1 2
Steel White Thin-Latex

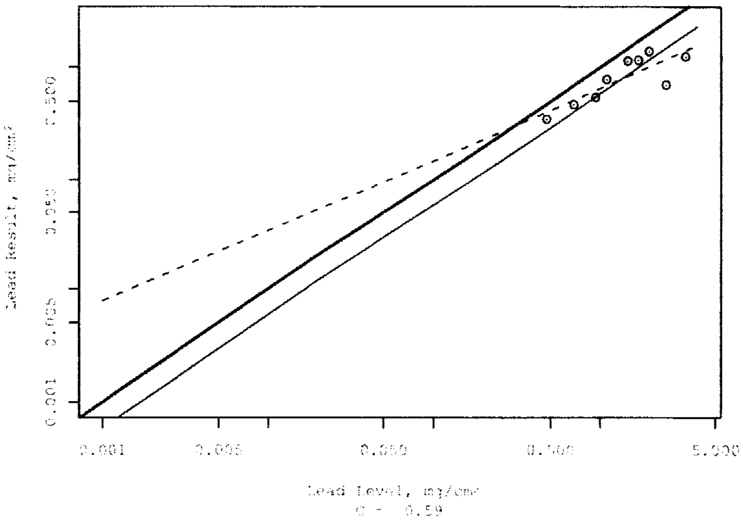
Op,Ap,Sub,Lead,Over : 1 1
Steel Chromate Thick-Oil



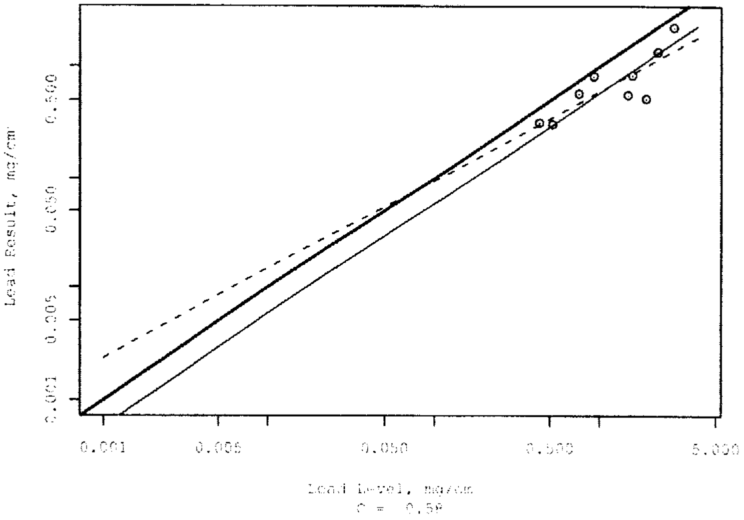
Op,Ap,Sub,Lead,Over : 5 1
Plaster Chromate Thick-Oil

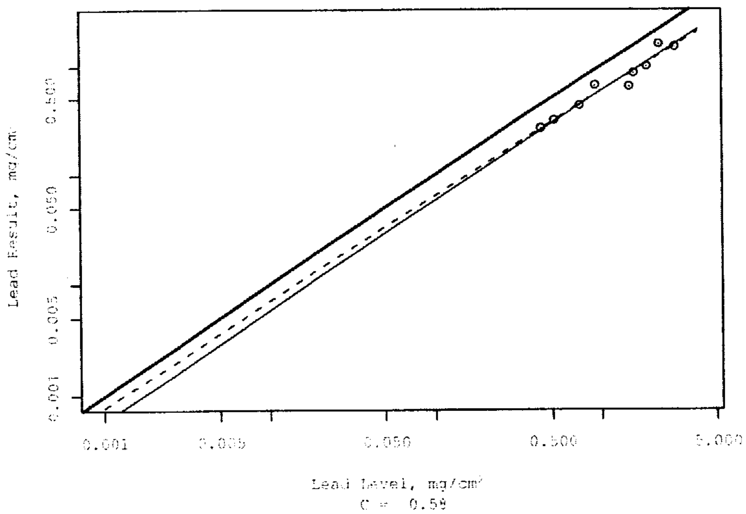


Op,Ap,Sub,Lead,Over : 3 1
Plaster Chromate Thin-Latex

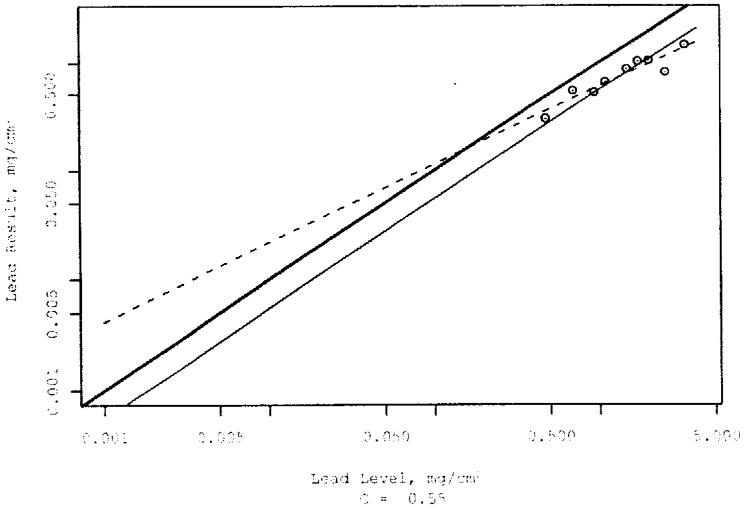


Op,Ap,Sub,Lead,Over : 3 1
Steel Chromate Thick-Oil





Op,Ap,Sub,Lead,Over : 1 2
Steel Chromate Thick-Oil



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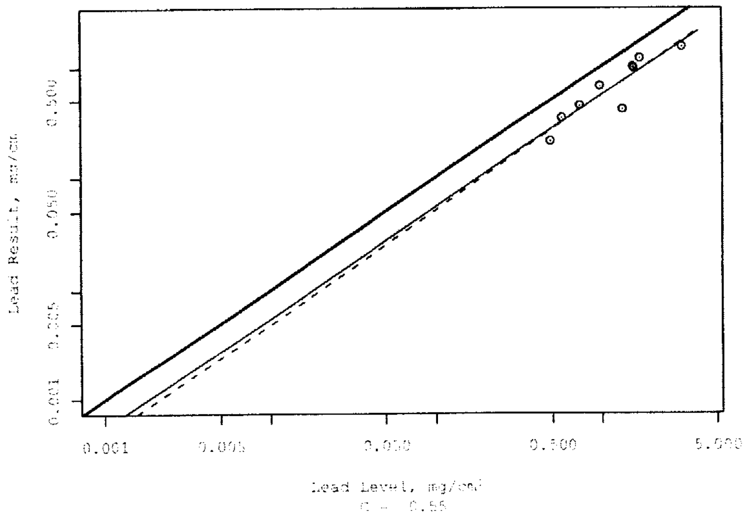
Plaster Chromate Thin-Latex



Op,Ap,Sub,Lead,Over : 4 2
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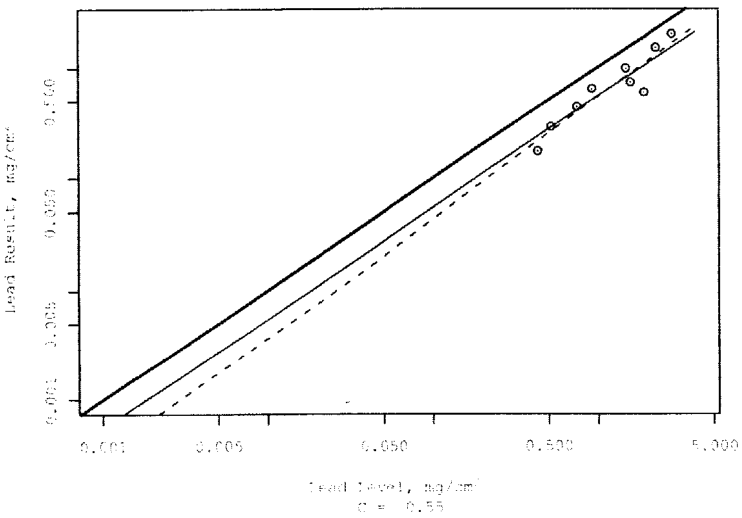
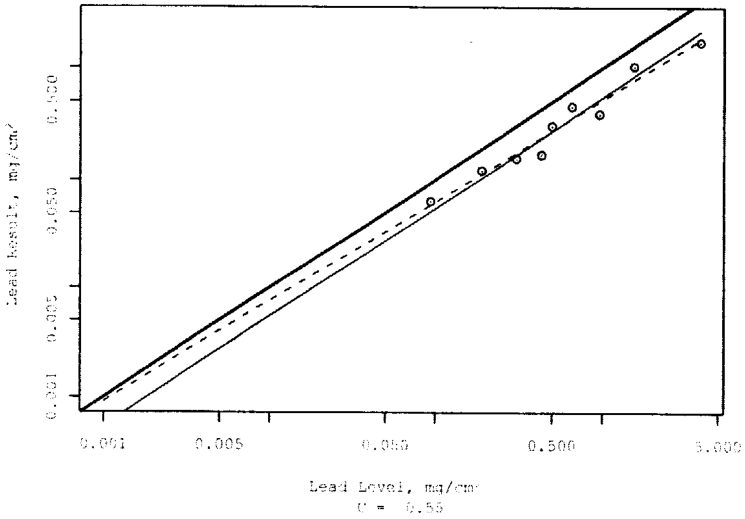
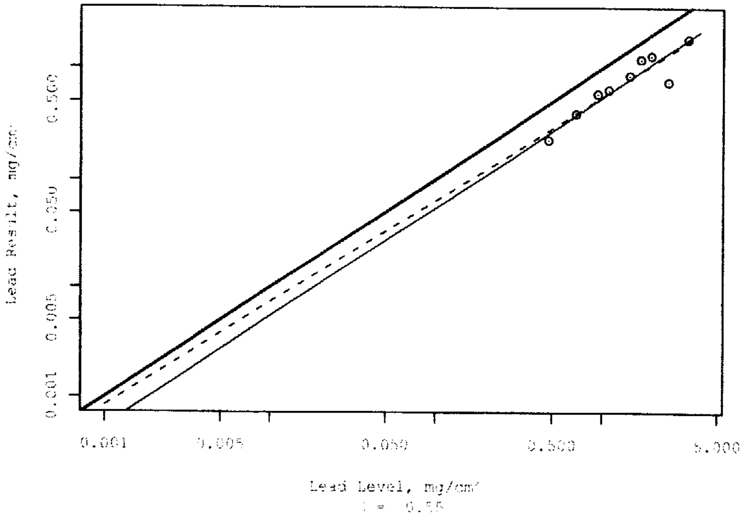


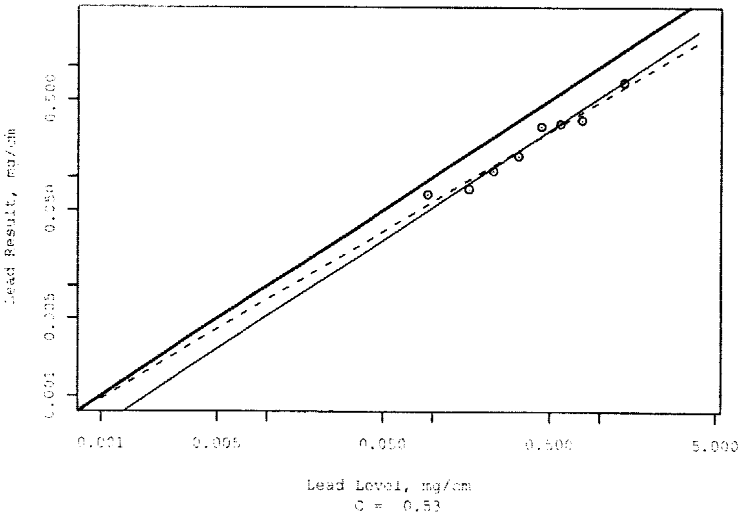
Op,Ap,Sub,Lead,Over : 5 2
Steel Chromate Thick-Oil



Op,Ap,Sub,Lead,Over : 2 1
Plaster White Thick-Oil



Op,Ap,Sub,Lead,Over : 3 2
Plaster Chromate Thin-Latex



Op,Ap,Sub,Lead,Over : 3 2
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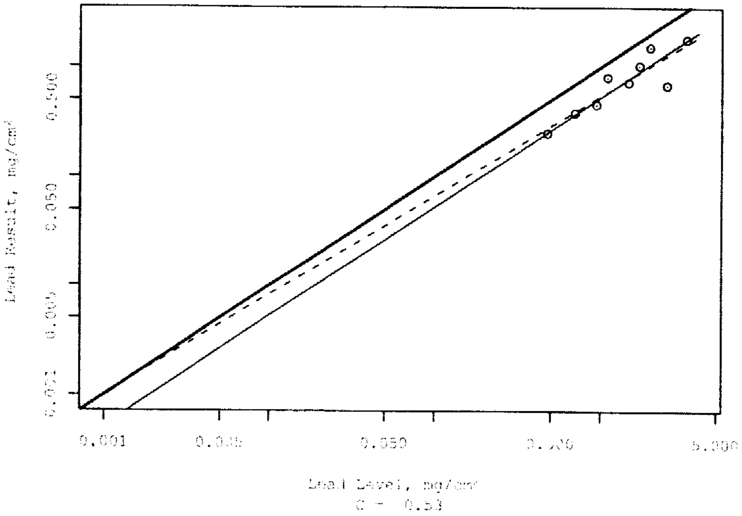
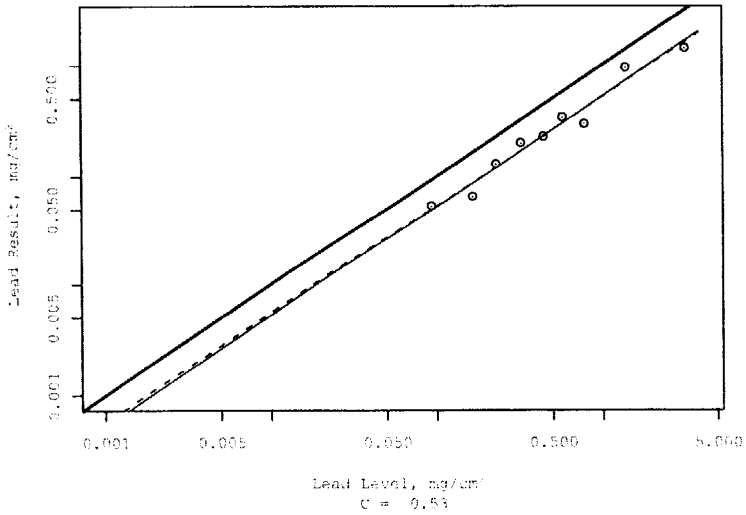


Op,Ap,Sub,Lead,Over : 2 2
Plaster Chromate Thin-Latex


Op,Ap,Sub,Lead,Over : 4 2
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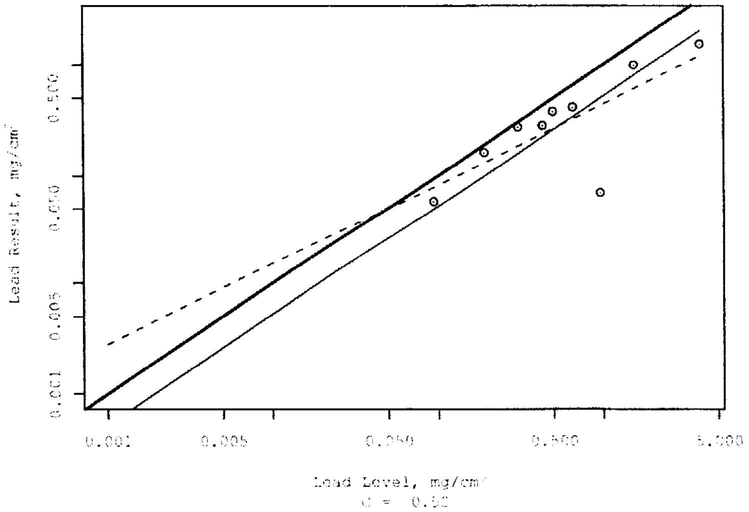
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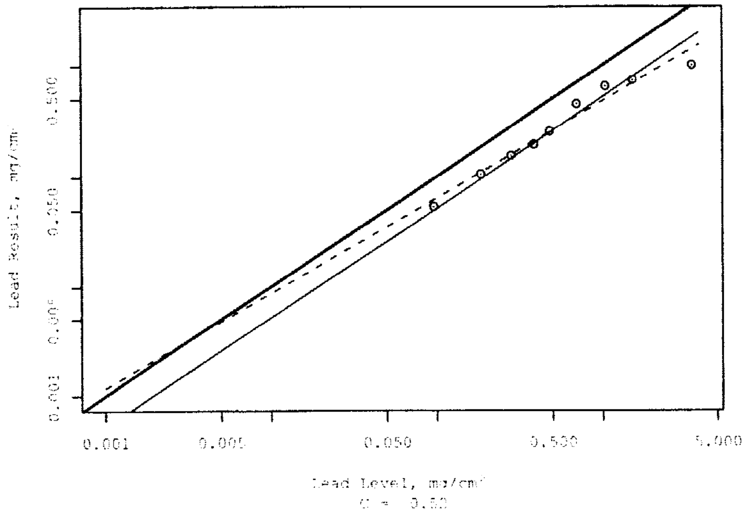
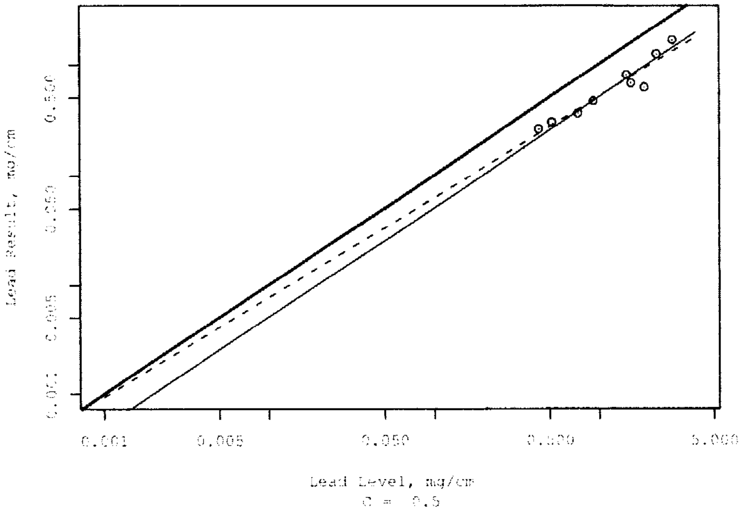
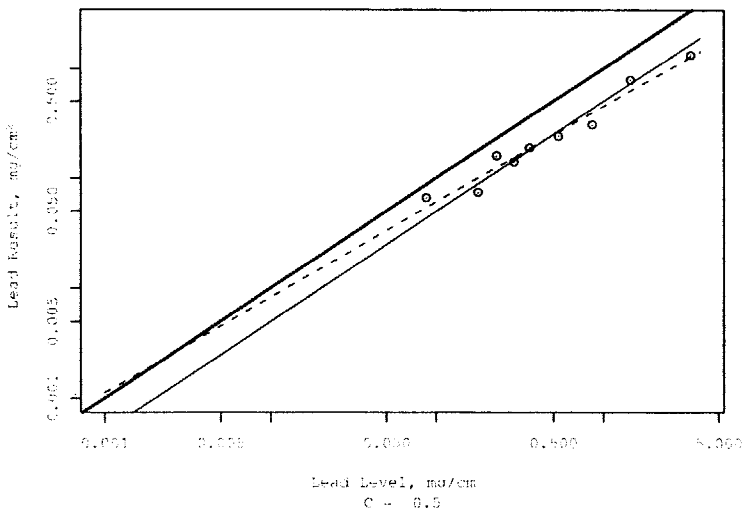
Plaster White Thick-Oil

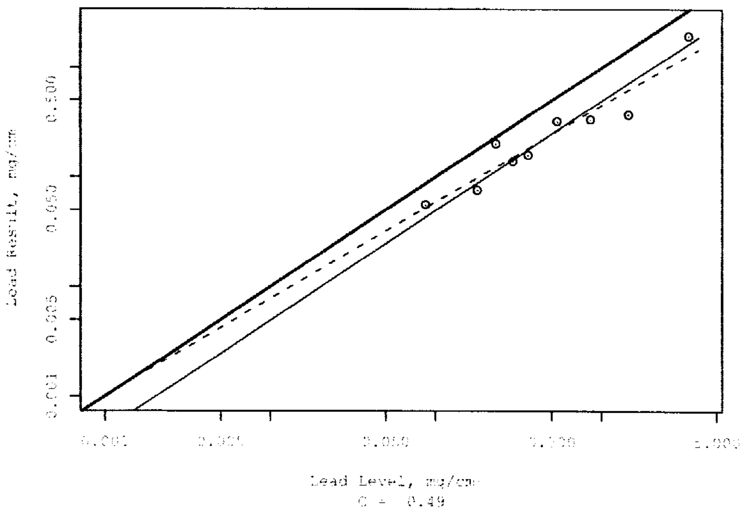

Op,Ap,Sub,Lead,Over : 4 2
Plaster White Thin-Latex


Op,Ap,Sub,Lead,Over : 3 2
Steel Chromate Thick-Oil

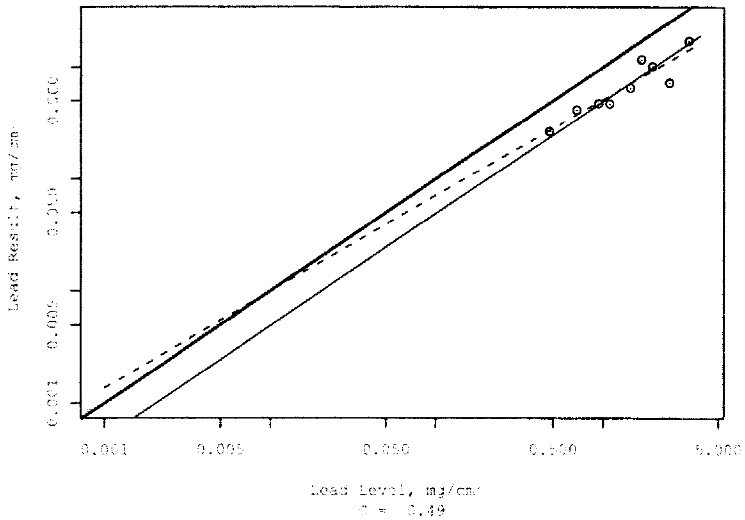
Op,Ap,Sub,Lead,Over : 3 1
Steel White Thick-Oil



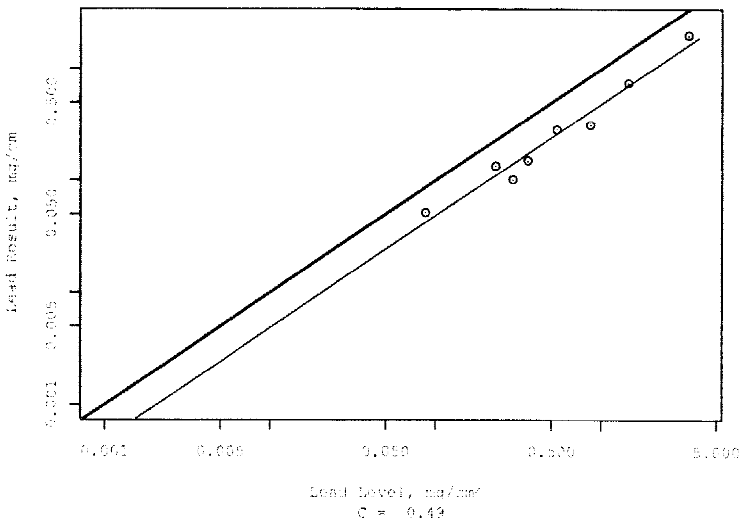
Op,Ap,Sub,Lead,Over : 2 1
Steel White Thick-Oil



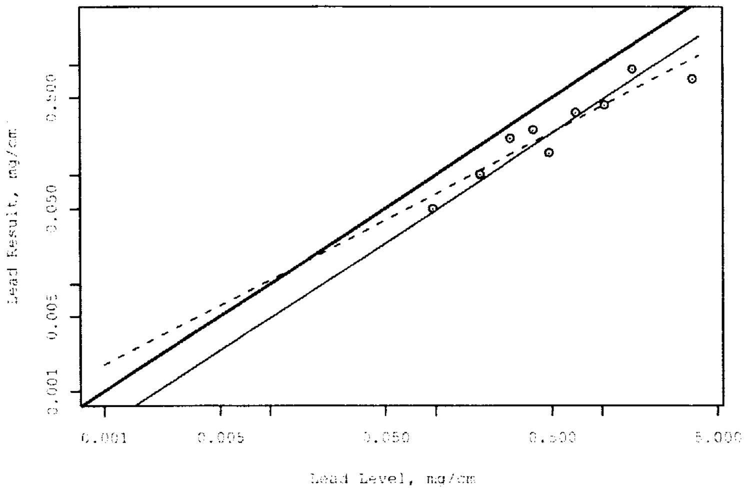
Op,Ap,Sub,Lead,Over : 4 2
Plaster Chromate Thin-Latex



Op,Ap,Sub,Lead,Over : 5 2
Steel White Thick-Oil

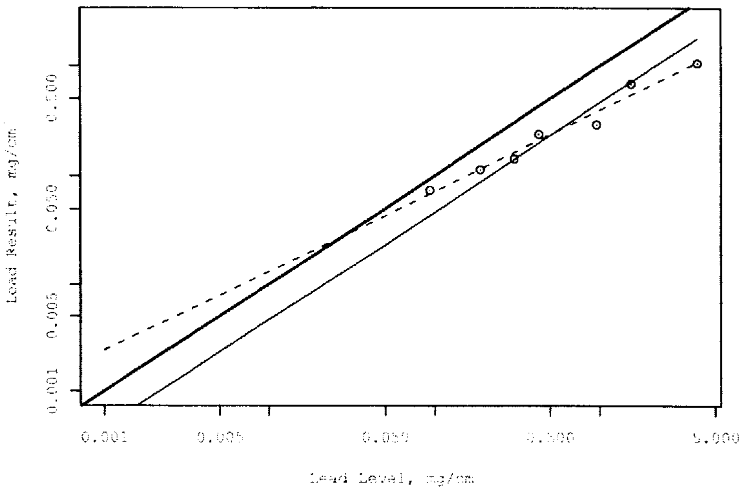


Op,Ap,Sub,Lead,Over : 2 2
Plaster White Thin-Latex



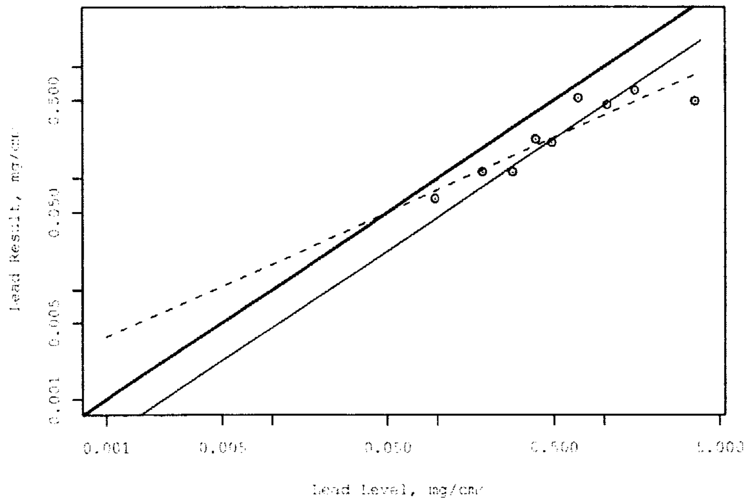
C = 0.49

Op,Ap,Sub,Lead,Over : 3 1
Plaster White Thick-Oil



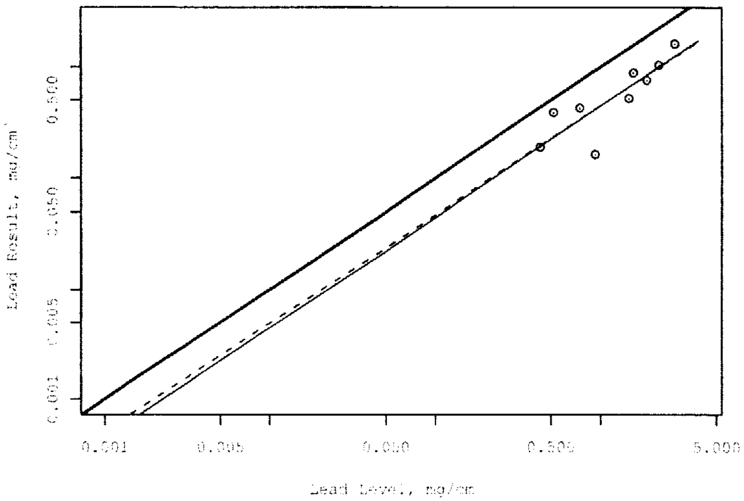
C = 0.46

Op,Ap,Sub,Lead,Over : 3 2
Plaster White Thin-Latex



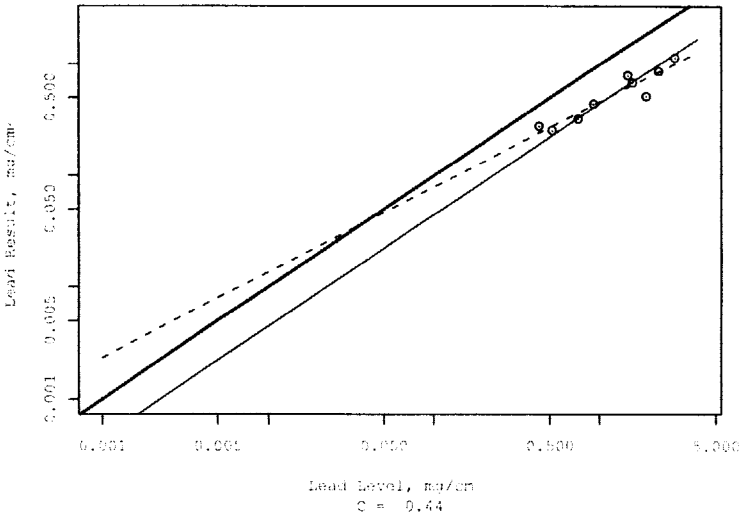
C = 0.45

Op,Ap,Sub,Lead,Over : 2 1
Steel Chromate Thick-Oil

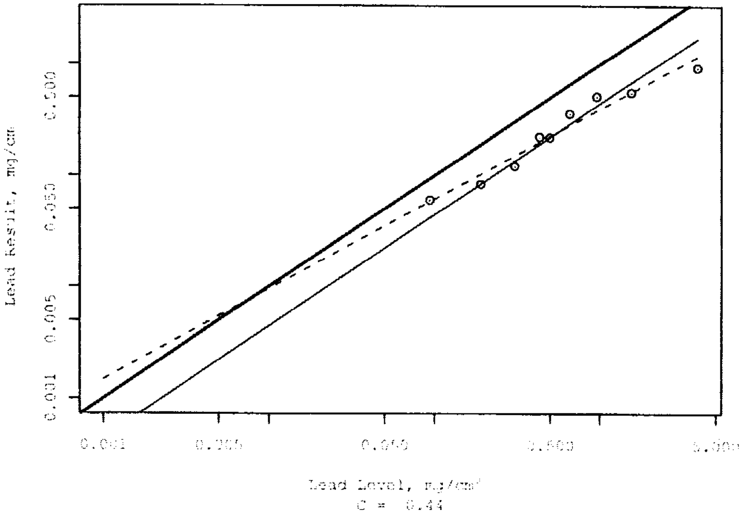


C = 0.15

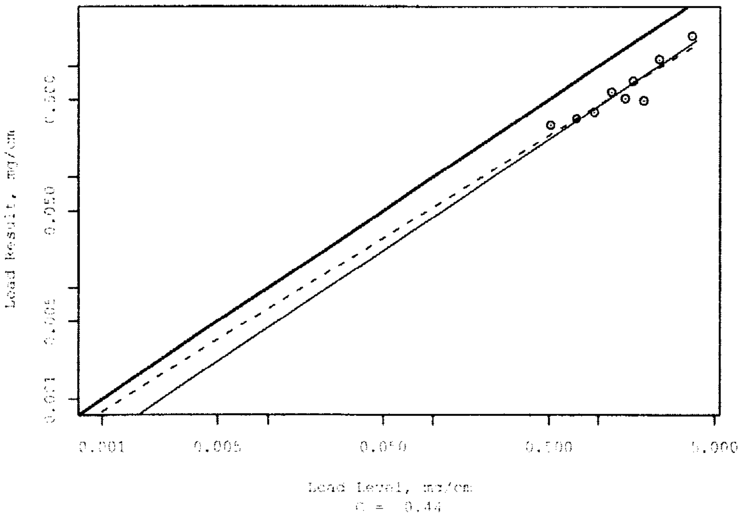
Op,Ap,Sub,Lead,Over : 2 2
Steel Chromate Thick-Oil



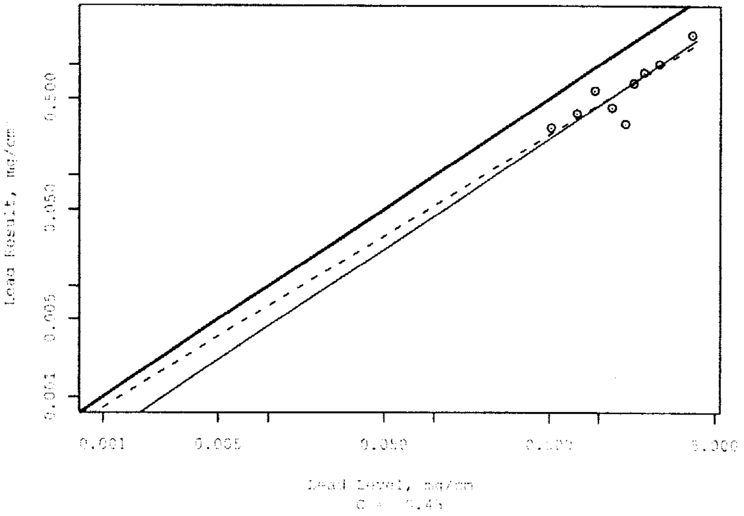
Op,Ap,Sub,Lead,Over : 5 2
Plaster White Thick-Oil

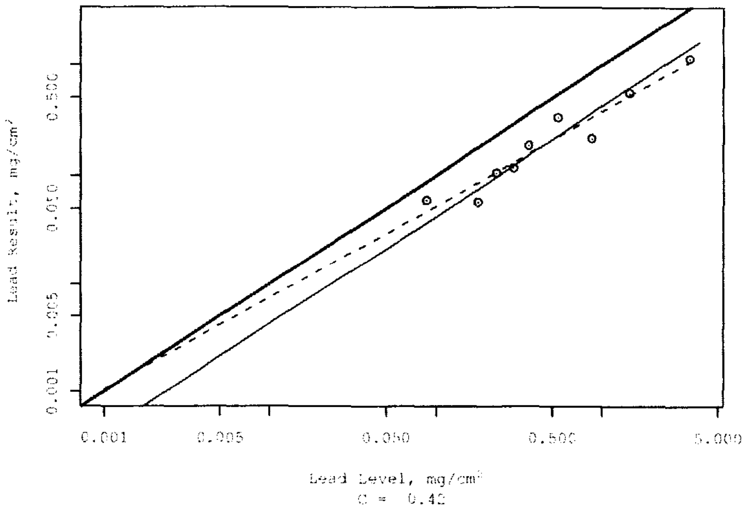


Op,Ap,Sub,Lead,Over : 5 2
Plaster Chromate Thick-Oil

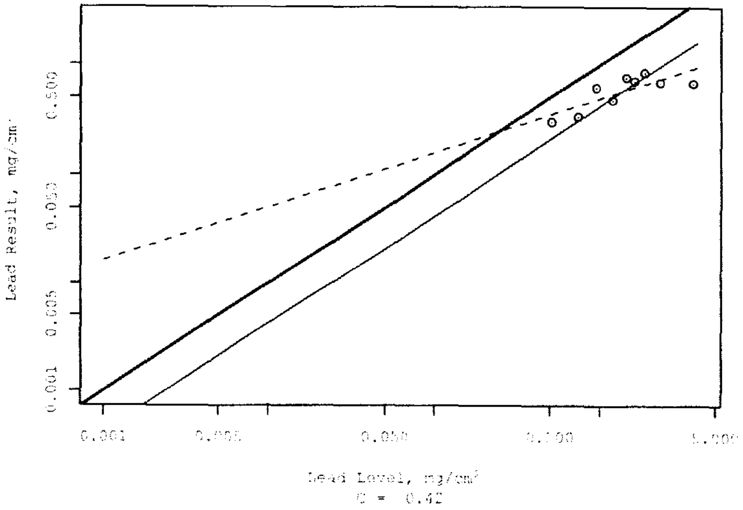


Op,Ap,Sub,Lead,Over : 3 1
Plaster Chromate Thick-Oil

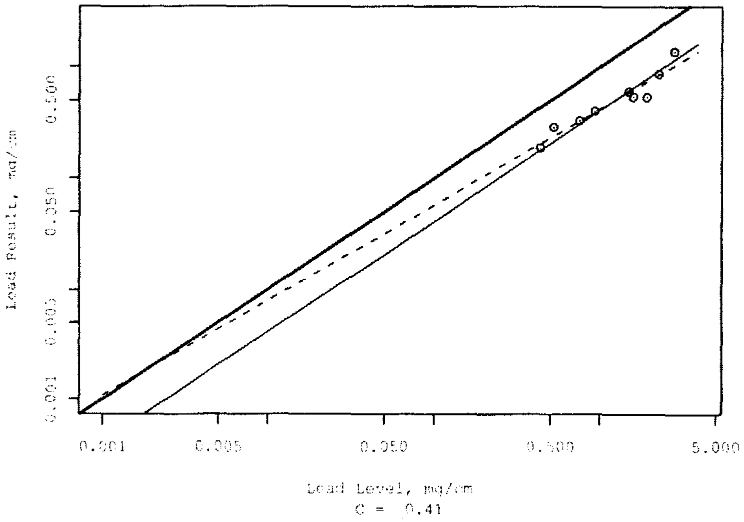




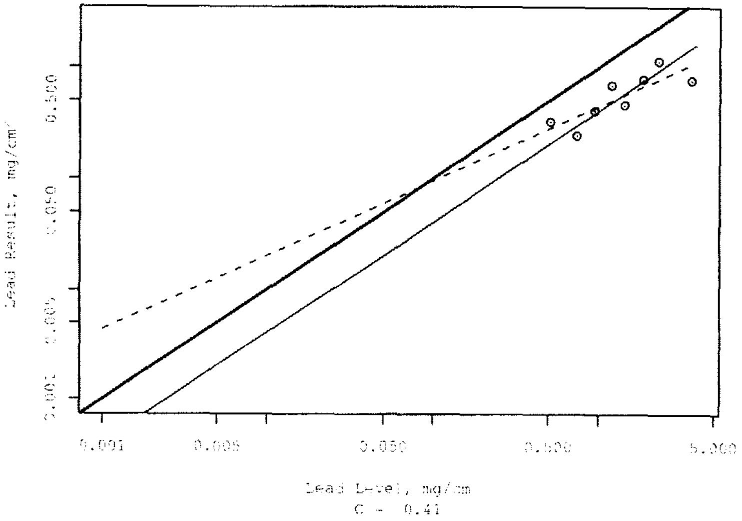
Op,Ap,Sub,Lead,Over : 4 1
Steel White Thick-Oil



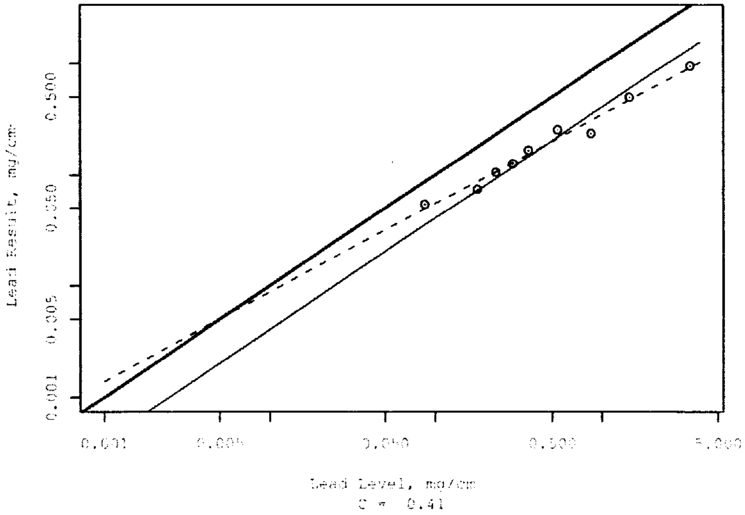
Op,Ap,Sub,Lead,Over : 1 1
Plaster Chromate Thick-Oil



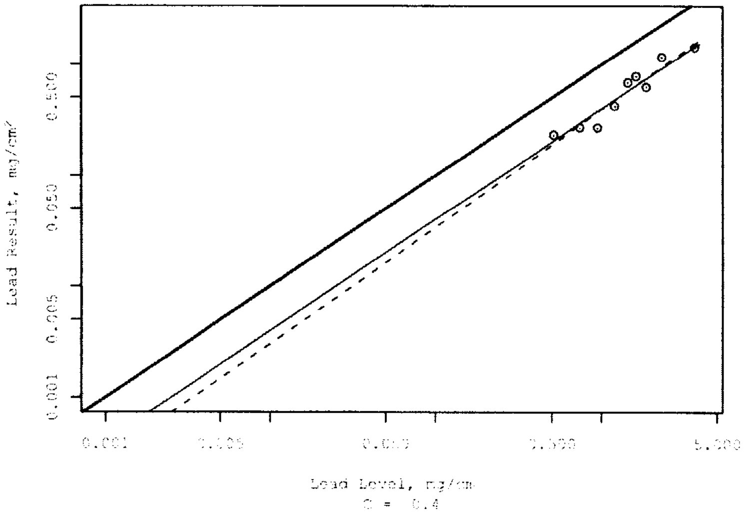
Op,Ap,Sub,Lead,Over : 4 1
Steel Chromate Thick-Oil



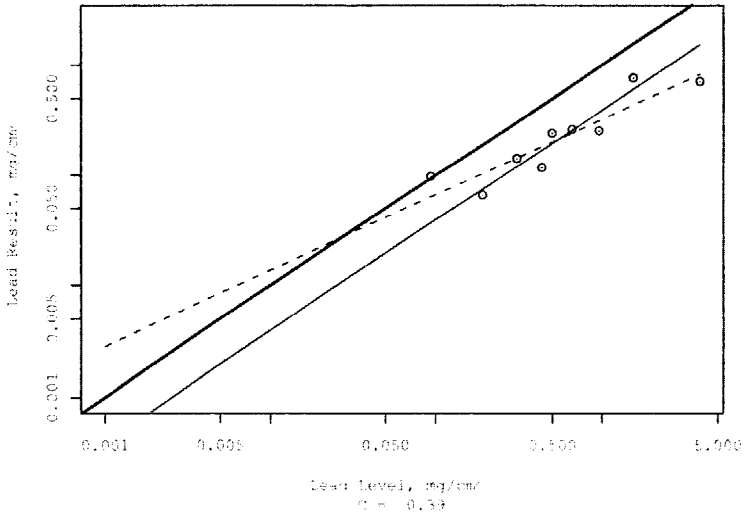
Op,Ap,Sub,Lead,Over : 4 1
Plaster Chromate Thick-Oil



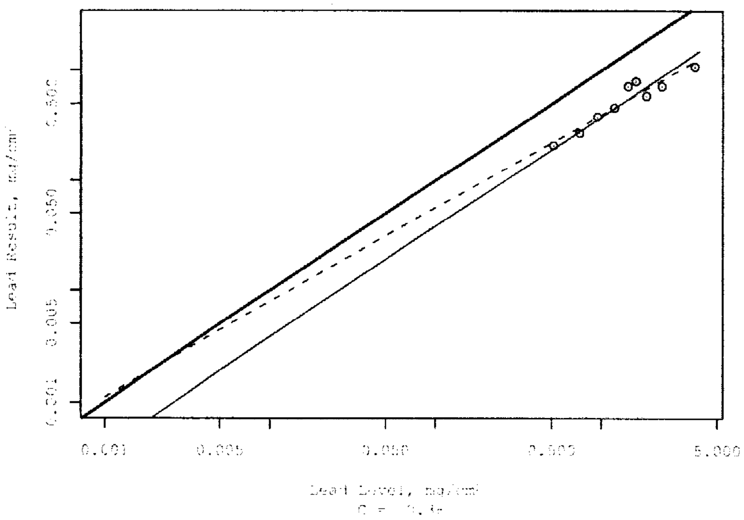
Op,Ap,Sub,Lead,Over : 3 2
Steel White Thick-Oil



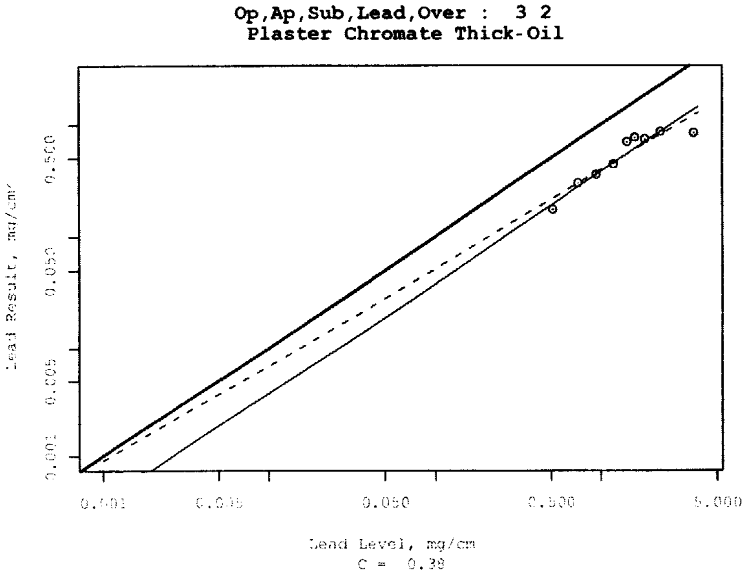
Op,Ap,Sub,Lead,Over : 1 2
Plaster Chromate Thick-Oil



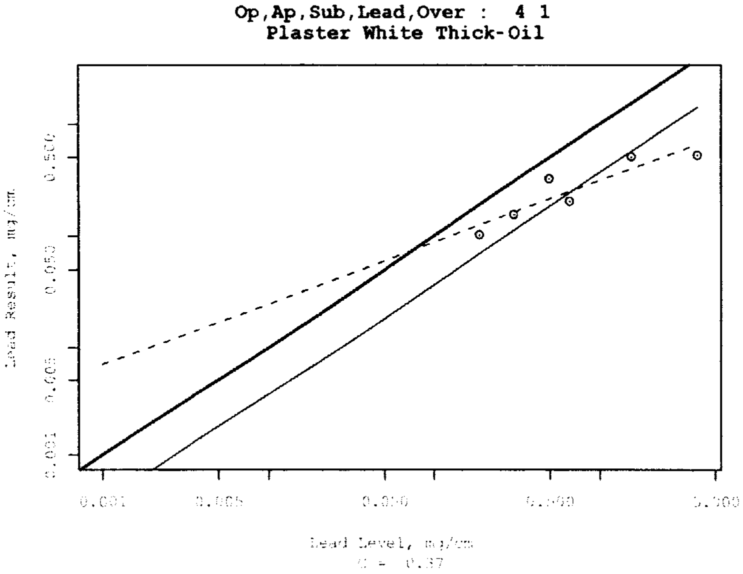
Op,Ap,Sub,Lead,Over : 1 1
Plaster White Thick-Oil



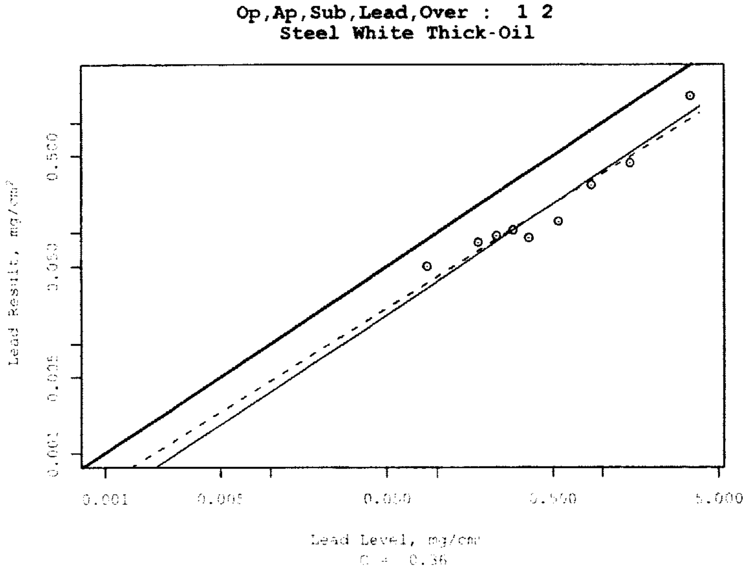
Op,Ap,Sub,Lead,Over : 2 2
Plaster Chromate Thick-Oil



Op,Ap,Sub,Lead,Over : 3 2
Plaster Chromate Thick-Oil



Op,Ap,Sub,Lead,Over : 4 1
Plaster White Thick-Oil



Op,Ap,Sub,Lead,Over : 1 2
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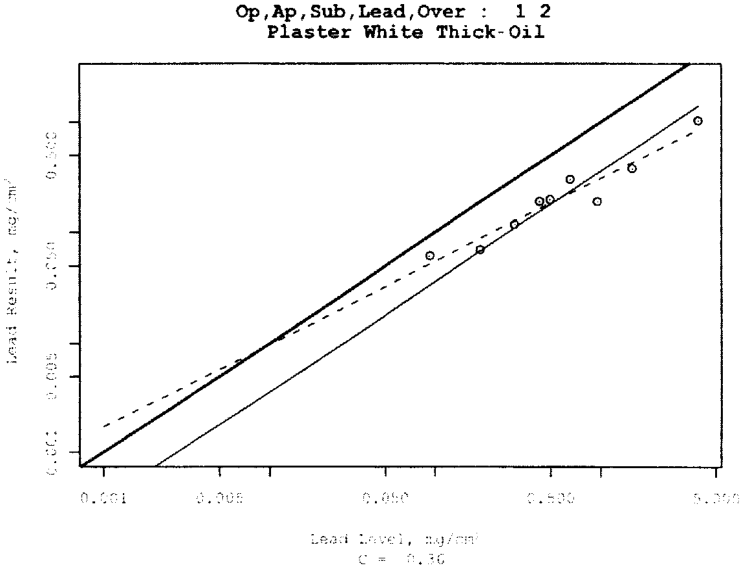

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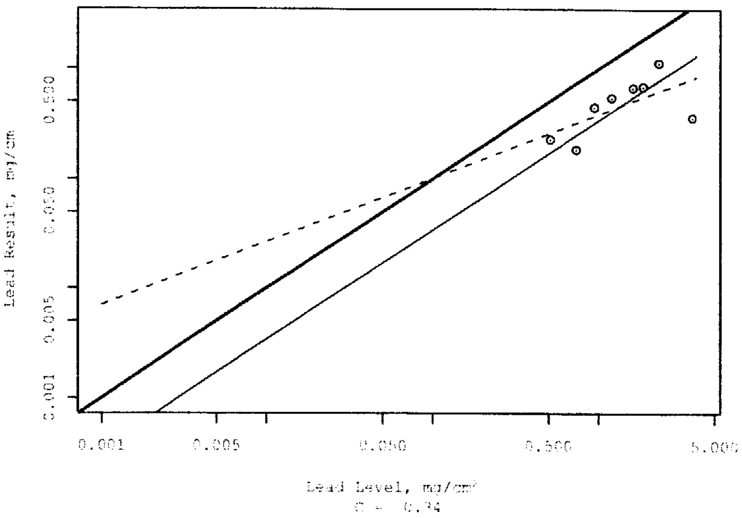
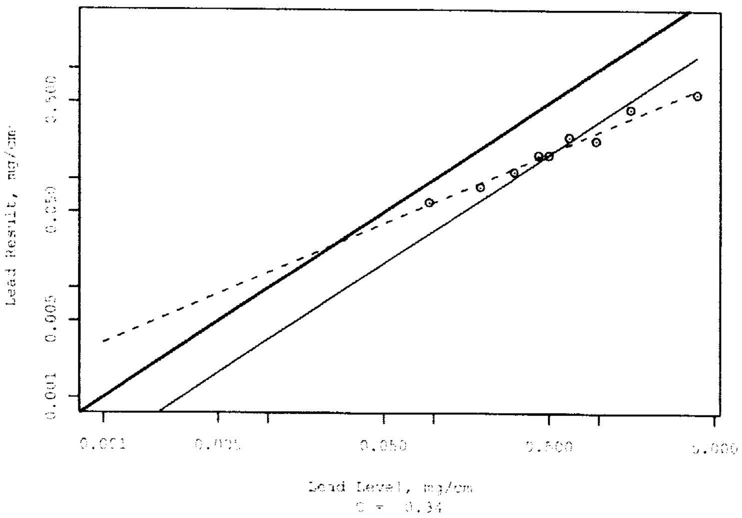
Plaster Chromate Thick-Oil



Op,Ap,Sub,Lead,Over : 3 2
Plaster White Thick-Oil



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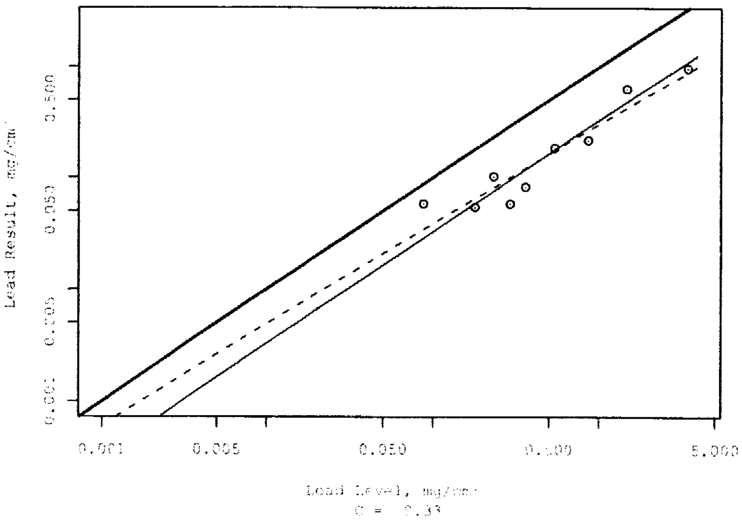

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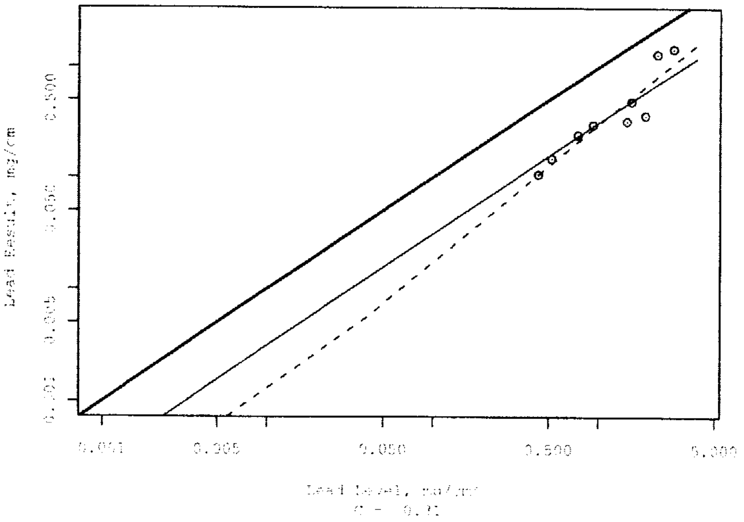
Steel Chromate Thick-Oil



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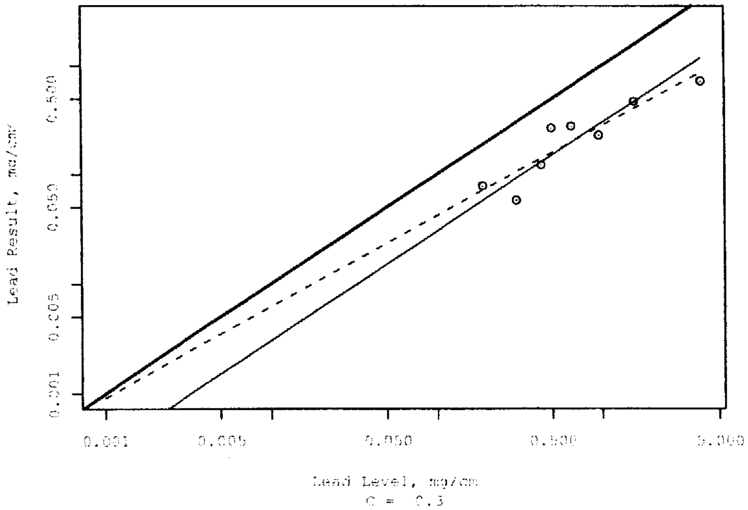
Plaster White Thick-Oil



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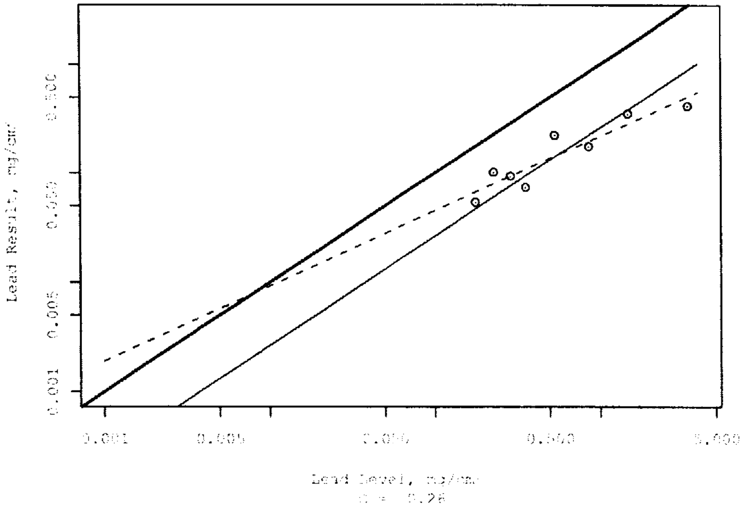
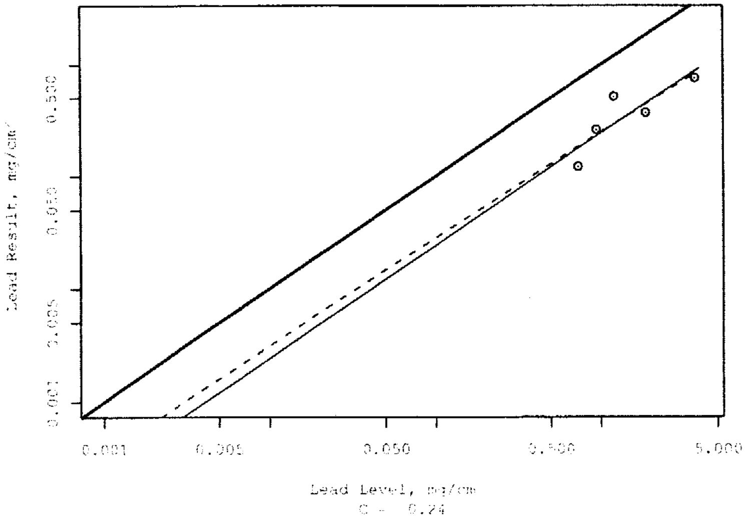
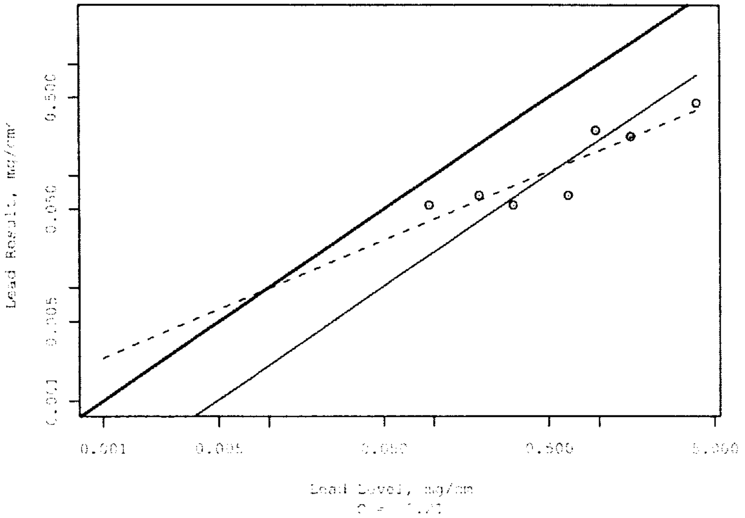
Steel White Thick-Oil



Op,Ap,Sub,Lead,Over : 4 2
Plaster Chromate Thick-Oil



Op,Ap,Sub,Lead,Over : 4 2
Plaster White Thick-Oil



APPENDIX B. RECOVERY CONSTANTS

This Appendix tabulates Recovery Constants for the data adjusted and unadjusted for calibration in decreasing order of lead recovery. In addition, 95 % confidence intervals for these values are provided. These confidence intervals were calculated on a log scale and are, thus, not symmetrical. The Recovery Constants were used to prepare Figures 7A and 7B of the Main Text.

Table B1. Recovery Constants for the data adjusted for calibration

Combination of Experimental Factors					Recovery Constant		
					Estimated	95 % Conf. Intvl.	
Operator No.	Apparatus No.	Substrate Type	Lead Pigment Type	Overlayer Type	Value	Lower	Upper
1	2	Steel	Lead Chromate	Thin-Latex	0.94	0.81	1.09
5	1	Steel	Lead Chromate	Thin-Latex	0.91	0.81	1.01
5	2	Steel	Lead Chromate	Thin-Latex	0.91	0.82	1.00
5	1	Steel	White Lead	Thin-Latex	0.90	0.80	1.00
5	2	Steel	White Lead	Thin-Latex	0.90	0.81	0.98
2	2	Steel	Lead Chromate	Thin-Latex	0.88	0.75	1.05
5	2	Plaster	White Lead	Thin-Latex	0.85	0.71	1.01
5	1	Steel	Lead Chromate	Thick-Oil	0.85	0.80	0.89
1	2	Plaster	White Lead	Thin-Latex	0.84	0.71	1.00
2	2	Steel	White Lead	Thin-Latex	0.82	0.70	0.95
1	2	Steel	White Lead	Thin-Latex	0.81	0.67	0.99
3	1	Steel	White Lead	Thin-Latex	0.80	0.69	0.93
3	2	Steel	Lead Chromate	Thin-Latex	0.80	0.74	0.86
4	1	Steel	White Lead	Thin-Latex	0.79	0.66	0.95
2	1	Steel	White Lead	Thin-Latex	0.79	0.70	0.90
5	1	Plaster	Lead Chromate	Thin-Latex	0.79	0.65	0.96
1	1	Steel	Lead Chromate	Thin-Latex	0.78	0.70	0.88
5	2	Plaster	Lead Chromate	Thin-Latex	0.78	0.70	0.88
1	2	Plaster	Lead Chromate	Thin-Latex	0.77	0.63	0.92
2	1	Plaster	White Lead	Thin-Latex	0.75	0.68	0.83
3	1	Steel	Lead Chromate	Thin-Latex	0.75	0.62	0.91
1	1	Plaster	Lead Chromate	Thin-Latex	0.75	0.66	0.85
1	1	Plaster	White Lead	Thin-Latex	0.74	0.61	0.90
1	1	Steel	White Lead	Thin-Latex	0.73	0.62	0.86
4	1	Steel	Lead Chromate	Thin-Latex	0.73	0.62	0.86
4	1	Plaster	White Lead	Thin-Latex	0.71	0.61	0.83
3	1	Plaster	White Lead	Thin-Latex	0.71	0.57	0.88
3	2	Steel	White Lead	Thin-Latex	0.70	0.60	0.83
5	1	Plaster	White Lead	Thin-Latex	0.70	0.50	0.99
2	1	Steel	Lead Chromate	Thin-Latex	0.70	0.62	0.78
5	1	Steel	White Lead	Thick-Oil	0.70	0.61	0.79
1	2	Steel	Lead Chromate	Thick-Oil	0.69	0.61	0.78
4	2	Steel	White Lead	Thin-Latex	0.68	0.58	0.80
4	2	Plaster	White Lead	Thin-Latex	0.66	0.53	0.82
4	2	Steel	Lead Chromate	Thin-Latex	0.66	0.54	0.79
5	2	Steel	Lead Chromate	Thick-Oil	0.65	0.54	0.80
4	1	Plaster	Lead Chromate	Thin-Latex	0.65	0.57	0.73
3	2	Plaster	Lead Chromate	Thin-Latex	0.64	0.53	0.78

Table B1. Recovery Constants for the data adjusted for calibration (cont.)

Combination of Experimental Factors					Recovery Constant		
Operator No.	Apparatus No.	Substrate Type	Lead Pigment Type	Overlayer Type	Estimated Value	95 % Conf. Intvl.	
						Lower	Upper
5	2	Steel	White Lead	Thick-Oil	0.63	0.53	0.75
2	2	Plaster	Lead Chromate	Thin-Latex	0.63	0.50	0.78
1	1	Steel	White Lead	Thick-Oil	0.63	0.54	0.72
1	1	Steel	Lead Chromate	Thick-Oil	0.62	0.56	0.69
5	1	Plaster	Lead Chromate	Thick-Oil	0.62	0.52	0.74
2	2	Plaster	White Lead	Thin-Latex	0.62	0.45	0.85
3	2	Steel	Lead Chromate	Thick-Oil	0.61	0.52	0.71
4	2	Plaster	Lead Chromate	Thin-Latex	0.59	0.48	0.71
3	2	Plaster	White Lead	Thin-Latex	0.58	0.39	0.88
3	1	Plaster	Lead Chromate	Thin-Latex	0.58	0.44	0.76
3	1	Steel	Lead Chromate	Thick-Oil	0.58	0.43	0.78
5	2	Plaster	White Lead	Thick-Oil	0.58	0.44	0.75
2	1	Plaster	Lead Chromate	Thin-Latex	0.54	0.45	0.66
2	2	Steel	Lead Chromate	Thick-Oil	0.54	0.44	0.66
3	2	Steel	White Lead	Thick-Oil	0.54	0.42	0.69
2	1	Plaster	White Lead	Thick-Oil	0.53	0.45	0.64
5	2	Plaster	Lead Chromate	Thick-Oil	0.53	0.44	0.63
5	1	Plaster	White Lead	Thick-Oil	0.51	0.28	0.93
1	2	Steel	White Lead	Thick-Oil	0.49	0.38	0.63
1	2	Plaster	Lead Chromate	Thick-Oil	0.48	0.42	0.56
3	1	Steel	White Lead	Thick-Oil	0.48	0.40	0.58
2	1	Steel	White Lead	Thick-Oil	0.48	0.37	0.63
1	2	Plaster	White Lead	Thick-Oil	0.48	0.35	0.65
2	2	Plaster	Lead Chromate	Thick-Oil	0.47	0.40	0.54
3	2	Plaster	Lead Chromate	Thick-Oil	0.46	0.39	0.55
3	1	Plaster	White Lead	Thick-Oil	0.45	0.34	0.60
3	2	Plaster	White Lead	Thick-Oil	0.45	0.31	0.64
2	2	Steel	White Lead	Thick-Oil	0.44	0.33	0.60
2	1	Steel	Lead Chromate	Thick-Oil	0.44	0.32	0.61
3	1	Plaster	Lead Chromate	Thick-Oil	0.43	0.33	0.55
1	1	Plaster	Lead Chromate	Thick-Oil	0.41	0.31	0.55
4	1	Steel	White Lead	Thick-Oil	0.41	0.32	0.52
4	1	Steel	Lead Chromate	Thick-Oil	0.40	0.35	0.47
4	1	Plaster	Lead Chromate	Thick-Oil	0.40	0.30	0.53
2	2	Plaster	White Lead	Thick-Oil	0.40	0.29	0.54
4	2	Steel	Lead Chromate	Thick-Oil	0.39	0.31	0.50
1	1	Plaster	White Lead	Thick-Oil	0.38	0.26	0.56
4	1	Plaster	White Lead	Thick-Oil	0.36	0.23	0.55
4	2	Steel	White Lead	Thick-Oil	0.36	0.25	0.51
2	1	Plaster	Lead Chromate	Thick-Oil	0.34	0.22	0.51
4	2	Plaster	Lead Chromate	Thick-Oil	0.30	0.23	0.40
4	2	Plaster	White Lead	Thick-Oil	0.28	0.17	0.47

Table B2. Recovery Constants for the data unadjusted for calibration

Combination of Experimental Factors					Recovery Constant		
Operator No.	Apparatus No.	Substrate Type	Lead Pigment Type	Overlayer Type	Estimated Value	95 % Conf. Intvl.	
						Lower	Upper
5	1	Steel	White Lead	Thin-Latex	0.91	0.81	1.03
5	1	Steel	Lead Chromate	Thin-Latex	0.91	0.82	1.02
5	1	Steel	Lead Chromate	Thick-Oil	0.85	0.80	0.90
3	1	Steel	White Lead	Thin-Latex	0.82	0.70	0.95
1	2	Steel	Lead Chromate	Thin-Latex	0.81	0.69	0.95
4	1	Steel	White Lead	Thin-Latex	0.81	0.67	0.97
2	1	Steel	White Lead	Thin-Latex	0.81	0.71	0.92
5	1	Plaster	Lead Chromate	Thin-Latex	0.79	0.65	0.96
1	1	Steel	Lead Chromate	Thin-Latex	0.79	0.70	0.89
5	2	Steel	Lead Chromate	Thin-Latex	0.78	0.70	0.87
2	1	Plaster	White Lead	Thin-Latex	0.77	0.70	0.85
2	2	Steel	Lead Chromate	Thin-Latex	0.76	0.63	0.92
3	1	Steel	Lead Chromate	Thin-Latex	0.76	0.62	0.92
1	1	Plaster	White Lead	Thin-Latex	0.75	0.61	0.93
1	1	Plaster	Lead Chromate	Thin-Latex	0.75	0.66	0.86
1	1	Steel	White Lead	Thin-Latex	0.74	0.63	0.88
4	1	Steel	Lead Chromate	Thin-Latex	0.73	0.62	0.87
4	1	Plaster	White Lead	Thin-Latex	0.73	0.62	0.85
3	1	Plaster	White Lead	Thin-Latex	0.72	0.57	0.91
5	1	Plaster	White Lead	Thin-Latex	0.72	0.51	1.01
5	1	Steel	White Lead	Thick-Oil	0.71	0.63	0.81
5	2	Steel	White Lead	Thin-Latex	0.71	0.64	0.78
2	1	Steel	Lead Chromate	Thin-Latex	0.70	0.63	0.79
3	2	Steel	Lead Chromate	Thin-Latex	0.68	0.62	0.74
5	2	Plaster	White Lead	Thin-Latex	0.67	0.59	0.77
5	2	Plaster	Lead Chromate	Thin-Latex	0.67	0.60	0.75
1	2	Plaster	White Lead	Thin-Latex	0.67	0.58	0.78
1	2	Plaster	Lead Chromate	Thin-Latex	0.66	0.54	0.80
4	1	Plaster	Lead Chromate	Thin-Latex	0.65	0.57	0.74
1	1	Steel	White Lead	Thick-Oil	0.64	0.55	0.75
2	2	Steel	White Lead	Thin-Latex	0.64	0.57	0.72
1	2	Steel	White Lead	Thin-Latex	0.64	0.51	0.80
1	1	Steel	Lead Chromate	Thick-Oil	0.63	0.57	0.69
5	1	Plaster	Lead Chromate	Thick-Oil	0.63	0.52	0.75
3	1	Plaster	Lead Chromate	Thin-Latex	0.59	0.45	0.77
3	1	Steel	Lead Chromate	Thick-Oil	0.58	0.44	0.78
1	2	Steel	Lead Chromate	Thick-Oil	0.58	0.52	0.66
2	1	Plaster	Lead Chromate	Thin-Latex	0.55	0.46	0.66
4	2	Steel	Lead Chromate	Thin-Latex	0.55	0.45	0.67
5	2	Steel	Lead Chromate	Thick-Oil	0.55	0.44	0.68
2	1	Plaster	White Lead	Thick-Oil	0.55	0.46	0.65
3	2	Plaster	Lead Chromate	Thin-Latex	0.55	0.45	0.66
3	2	Steel	White Lead	Thin-Latex	0.53	0.45	0.62
2	2	Plaster	Lead Chromate	Thin-Latex	0.53	0.42	0.67
4	2	Steel	White Lead	Thin-Latex	0.53	0.45	0.62
5	1	Plaster	White Lead	Thick-Oil	0.52	0.29	0.95
4	2	Plaster	White Lead	Thin-Latex	0.52	0.43	0.62
3	2	Steel	Lead Chromate	Thick-Oil	0.50	0.43	0.59

Table B2. Recovery Constants for the data unadjusted for calibration (cont.)

Combination of Experimental Factors					Recovery Constant		
						95 % Conf. Intvl.	
Operator No.	Apparatus No.	Substrate Type	Lead Pigment Type	Overlayer Type	Estimated Value	Lower	Upper
3	1	Steel	White Lead	Thick-Oil	0.50	0.41	0.60
2	1	Steel	White Lead	Thick-Oil	0.49	0.38	0.65
4	2	Plaster	Lead Chromate	Thin-Latex	0.49	0.41	0.59
5	2	Steel	White Lead	Thick-Oil	0.49	0.40	0.58
2	2	Plaster	White Lead	Thin-Latex	0.48	0.35	0.65
3	1	Plaster	White Lead	Thick-Oil	0.46	0.33	0.65
3	2	Plaster	White Lead	Thin-Latex	0.45	0.31	0.66
2	1	Steel	Lead Chromate	Thick-Oil	0.45	0.32	0.61
2	2	Steel	Lead Chromate	Thick-Oil	0.44	0.37	0.53
5	2	Plaster	White Lead	Thick-Oil	0.44	0.35	0.55
5	2	Plaster	Lead Chromate	Thick-Oil	0.44	0.37	0.52
3	1	Plaster	Lead Chromate	Thick-Oil	0.43	0.34	0.55
4	1	Steel	White Lead	Thick-Oil	0.42	0.33	0.54
1	1	Plaster	Lead Chromate	Thick-Oil	0.42	0.31	0.56
4	1	Steel	Lead Chromate	Thick-Oil	0.41	0.35	0.48
4	1	Plaster	Lead Chromate	Thick-Oil	0.41	0.30	0.55
3	2	Steel	White Lead	Thick-Oil	0.41	0.33	0.50
1	2	Plaster	Lead Chromate	Thick-Oil	0.40	0.34	0.47
1	1	Plaster	White Lead	Thick-Oil	0.39	0.27	0.58
2	2	Plaster	Lead Chromate	Thick-Oil	0.38	0.33	0.44
3	2	Plaster	Lead Chromate	Thick-Oil	0.38	0.32	0.45
4	1	Plaster	White Lead	Thick-Oil	0.37	0.20	0.67
1	2	Steel	White Lead	Thick-Oil	0.36	0.29	0.46
1	2	Plaster	White Lead	Thick-Oil	0.36	0.27	0.48
2	1	Plaster	Lead Chromate	Thick-Oil	0.34	0.22	0.54
3	2	Plaster	White Lead	Thick-Oil	0.34	0.25	0.46
2	2	Steel	White Lead	Thick-Oil	0.33	0.24	0.44
4	2	Steel	Lead Chromate	Thick-Oil	0.31	0.23	0.41
2	2	Plaster	White Lead	Thick-Oil	0.30	0.22	0.42
4	2	Steel	White Lead	Thick-Oil	0.26	0.19	0.38
4	2	Plaster	Lead Chromate	Thick-Oil	0.24	0.15	0.38
4	2	Plaster	White Lead	Thick-Oil	0.21	0.11	0.37