NBSIR 78-1466

Evaluation of New Portable X-ray Fluorescent Lead Analyzers for Measuring Lead in Paint

A. Philip Cramp
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Center for Building Technology
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Washington, D.C. 20234

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U.S. DEPARTMENT OF COMMERCE, Juanita M. Kreps, ***Secretary***

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SI Conversion Units

The conversion factors and units contained in this report are in accordance with the International System of Units (Abbreviated SI for Systeme International d'Unites). The SI was defined and given official status by the 11th General Conference on Weights and Measures which met in Paris in October 1960. For assistance in converting U.S. customary units to SI units, see ASTM E 380, ASTM Standard Metric Practice Guide, available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA. 19103. The conversion factors for the units found in this standard are as follows:

Length

1 in - 0.0254* meter

1 ft = 0.3048* meter

1 mil = 0.001* in

Area

1 in^2 = 6.4516* x 10^{-4} $meter^2$

1 ft^2 = 0.9290* $meter^2$

Volume

1 in^3 = 1.638 x 10^{-5} $meter^3$

1 liter = 1.000* x 10^{-3} meter ^{3}

Mass

1 grain = 6.479 x 10^{-5} kilogram

1 ounce-mass (avoirdupois) = 2.834 x 10^{-2} kilogram

1 pound-mass (avoirdupois) = 0.4535 kilogram

Pressure or Stress (Force/Area)

1 inch of mercury (60°F) = 3376 newton/$meter^2$

1 pound-force/$inch^2$ (psi) = 6894 newton/$meter^2$

---

*Exactly

Energy

1 inch-pound-force (in-lbf) = 0.1130 joule

Plane Angle

1 degree (angle) = 1.745×10^{2} radian

Power

1 watt = $1.000^{*} \times 10^{7}$ erg/second

Temperature

°C = 5/9 (Temperature °F - 32)

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*Exactly

Evaluation of New Portable
X-Ray Fluorescent Lead Analyzers for
Measuring Lead in Paint

A. Philip Cramp and Harvey W. Berger

Abstract

Portable X-ray fluorescent lead analyzers offer the most cost-effective and adaptable means for the non-destructive detection and measurement of lead in paint in housing. However, commercially available portable lead analyzers have had poor accuracy and precision below lead levels of about 3.0 milligrams of lead per square centimeter of surface area. This is particularly serious because the current operational criteria for lead paint hazard abatement, 1.5 or 2.0 mg lead/cm^2 maximum (used in many communities), is in this range. They have also performed relatively unsatisfactorily with regard to serviceability and maintenance.

Two new portable lead analyzers based on x-ray fluorescence developed elsewhere under HUD contracts have been evaluated. One of the new devices was found to be unacceptable for field use because the prototypes of it were all inaccurate and imprecise, difficult to use and maintain. They were also very bulky and heavy. Although prototypes of the other lead analyzer did not meet all the requirements of the HUD contract, they showed considerable improvement over all previously available instruments in terms of accuracy, precision, ease of operation, and portability. They did, however, have some deficiencies which are discussed in the report.

Evaluation of New Portable X-Ray Fluorescent Lead Analyzers for Measuring Lead in Paint

1. INTRODUCTION

1.1 NEED FOR NEW INSTRUMENTS

At the present time, portable X-ray fluorescent lead detectors offer the best and most rapid method of insitu, non-destructive lead analysis of paint films on housing surfaces. Two models of this type of instrument, made by different manufacturers, have been widely used by housing inspectors as part of lead poisoning control programs throughout the country. These instruments are the Princeton Gamma Tech (PGT) Model XK-2*, and the Nuclear Chicago (NC) Model PB-3.* These particular instruments are fairly easily operated by semi-skilled personnel and have rapid analysis rates. Although they have performed fairly well in screening and survey activities, they have some serious deficiencies.

The most serious deficiency of these instruments is that they are inaccurate in determining lead levels in paint in the range of zero to 3.0 mg/cm^2. The low accuracy in this range is particularly serious because the current operational criteria for lead paint hazard abatement, 1.5 mg/cm^2 and 2.0 mg lead/cm^2 maximum, (used in many communities) is in this range.

Another serious deficiency of the instruments is that they are easily damaged when they are transported. Also, the instruments consist of two rather large components which make them awkward to handle and tiresome when in use a full working day.

1.2 INITIATION OF NEW INSTRUMENT DEVELOPMENT

In 1972 Rasberry [1][1] evaluated four different types of commercial portable X-ray lead analyzers all being used in the field for lead paint analysis at that time. They were all made by different companies. He found them all to have, at best, only fairly good accuracy and precision. Because of an agreement with the manufacturers, their names were not devulged in the report.

In May 1974, HUD issued a Request for Proposal (RFP) for the development of new lead analyzers which would meet the attributes described in Task II of the RFP. The specifications contained in the RFP require the new instruments to be much more accurate and precise than the PGT XK-2 and NC PB-3, easier to calibrate, much

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*In no case does such identification imply recommendation or endorsement by the National Bureau of Standards (NBS), nor does it imply that these instruments are necessarily the best available for the purpose.

[1] Numbers in brackets refer to the references in section 11.

lighter in weight and much more compact. Other properties such as operational safety, speed of analysis, length of continuous operation without battery charge (if a battery is used), retention of accuracy and precision under abnormal environmental conditions, durability, and cost per test were required to be at least equal to those of the two existing XRF instruments.

The recipient of a HUD contract was required to submit 3 prototypes of the new instrument to HUD for testing. NBS in turn was assigned the responsibility, by HUD, for evaluating the prototype in accordance with a testing and evaluation program devised by NBS for this purpose.

Of all the proposals submitted to HUD, those which seemed to have the best potential were from Columbia Scientific Industries Corp. (CSI), Austin, Texas and Princeton Gamma Tech Inc. (PGT), Princeton, New Jersey. Both firms proposed developing portable X-ray fluorescent analyzers of new types.

HUD Contract Nos. H-2191R and H-2192R were issued to CSI and PGT respectively. Subsequently, each of these firms delveloped new XRF lead analyzers, and submitted 3 prototypes of them to HUD, who in turn delivered them to NBS for evaluation. The new CSI lead analyzer is called CSI model 750, Portable Lead Wallpaint Analyzer; the new PGT instrument is called PGT XK-3 Lead Analyzer.

2. REPORT OBJECTIVES

The principal objectives of this report are:

1. To evaluate the physical properties and performance characteristics of three prototypes of the two new lead analyzers compared to the required and optimum performance levels required in the contracts under which they were developed.

2. To compare the physical properties and performances of the above lead analyzers to those of the most widely used existing portable XRF lead analyzers; the Princeton Gamma Tech XK-2 and the Nuclear Chicago PB-3.

3. DESCRIPTIONS OF THE NEW LEAD ANALYZERS

3.1 COLUMBIA SCIENTIFIC INDUSTRIES MODEL 750, LEAD ANALYZER

The CSI Model 750 lead analyzer consists of 3 components, namely a measurement probe, electronics unit and a calibrate module. The following is a brief description of these components, their function, and their most important parts.

3.1.1 The Probe

The probe is a cylindrical unit 11 in (27.94 cm) long, with a diameter of 2-3/4 in (6.99 cm) and weighing 3.0 lbs. (1.36 kg). It contains a 25 milliCurie, cadmium 109 Cd K hard X-ray source with a half life of 470 days. It has an "X-ray window" at the forward end with a trigger operated fail safe shutter. It contains two detector tubes (scintillation counters) fitted with appropriate X-ray filters for isolating and measuring the radiation given off by the lead contained in the sample being analyzed. The probe is connected to the electronics unit by an armored cable which transmits the electronic signals from the detectors to the electronics unit for processing. The minimum sample size required by the CSI Model 750 is about 1-1/2 in x 2-1/2 in (3.8 cm x 6.4 cm).

3.1.2 Electronics Unit

The electronics unit is 6 in x 9 in x 11 in (15.2 cm x 22.9 cm x 27.9 cm) and weighs about 11 lbs (4.99 Kg). It contains:

°two analog preamplifiers for processing the signals from the detector tubes;

°a microprocessor for collecting and storing data;

°a panel visual display on which is displayed the results of determinations in mg/cm^2 of lead;

°a battery with enough capacity for at least 8 hours of operation, assuming a 50% duty cycle of measurement time to idle time;

°signal tones for indicating the operational status of the lead analyzer, if it is idle, measuring or malfunctioning, or if the battery is approaching exhaustion.

The probe and electronics unit together constitute the lead analyzer. The combination weighs 14 lbs (6.35 Kg). Figure 1 shows the combination ready for use.

3.1.3 Calibration Module

This component (see Figure 2) is used only when the lead detector (the probe and electronics units combination) is being calibrated and the electronics unit's battery is being charged. It would not normally be taken into the field. Consequently, its dimensions and weight are not relevant to the performance and user characteristics of the lead detector during field use.

The calibration module consists of the following principal components:

°a keyboard for entering data into the instrument, and a clearing button for removing the data when the instrument is being calibrated;

°indicator lights which lead the operator through the required calibration sequence;

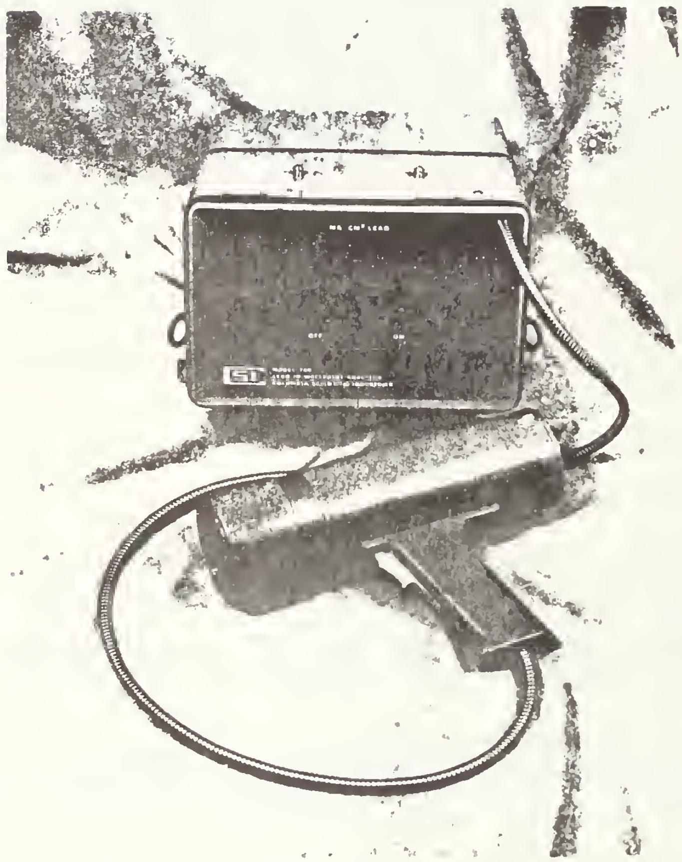

Figure 1 - CSI Model 750 Lead Analyzer (Probe and Electronic Unit) Ready for Use

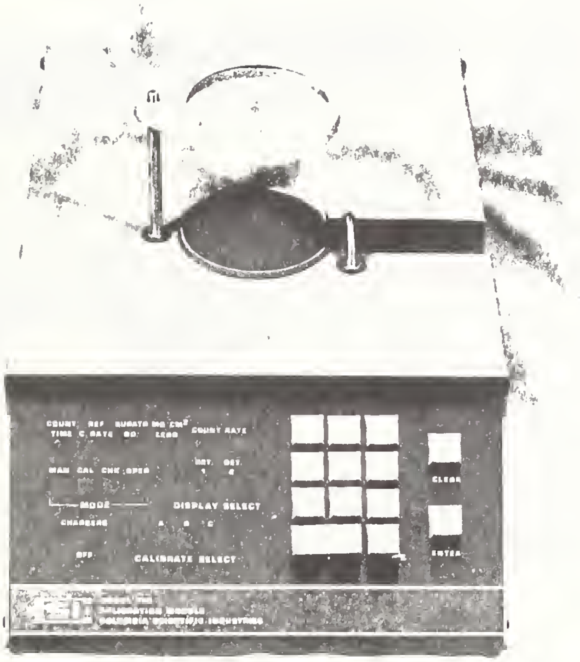

Figure 2 - CSI Model 750 Calibrate Module

°three independent battery chargers to which three instruments can be connected for simultaneous charging;

°a probe holder in which the probe is placed when the instrument is being calibrated;

°a set of calibration films with lead contents from zero to about 10 mg/cm^2, and a lead plate about 1/16 inch (1.6 mm) thick. The calibration films are thin polyethylene films intended to simulate paint films. They contain paint pigments such as ZnO, $CaCo_3$, TiO_2, and PbO;

°a set of reference substrates for use in calibrating the instrument. They include wood varying from 1/4 in (6.3 mm), to 4 in (101.6 mm) thick, 1/2 in (12.7 mm) gypsum wall board, 1/8 in (3.18 mm) masonite, 1 in (25.4 mm) plaster, and a clay building brick 2-1/4 in (57.0 mm) thick. These materials are intended to represent the full range of painted substrates likely to be encountered in the field. A 1/16 in (1.6 mm) thick lead plate is also included to provide a common calibration point for the matrices developed in the instrument when it is calibrated.

°an operation manual.

3.1.4 Calibration of CSI Model 750

The lead analyzer must be calibrated before use. The manufacturer states that this is necessary every six months. Calibration is a relatively long and complicated procedure. Figure 3 shows the lead analyzer being calibrated (the probe is clamped in position in the calibrate module).

Basically, calibration requires that a series of mathematical matrices be developed in the instrument representing the reflected fluorescent X-rays (photons) received by the instrument from a series of films varying in lead content, placed on a wide variety of substrates.

The calibration films and reference substrates described above are used for this purpose. Sets of the films are placed successively on the desired substrates and, by following the calibration procedure described in the operational manual, the emitted X-rays received from them by the probe are processed in the probe and electronics unit to generate the required matrices. According to the manufacturer, after calibration the instrument should be ready to measure accurately the lead content of any paint film on substrates not only for which the instrument is calibrated but also on any of the infinite number of substrates that yield data within the range covered by the matrices stored in the memory of the instrument.

3.1.5 Method of Operation of CSI, Model 750

For determining the lead content of paint applications, the lead analyzer is detached from the calibration unit. The lead analyzer can be carried conveniently by the operator during field use by means of a harness provided for this purpose (see Figure 4).

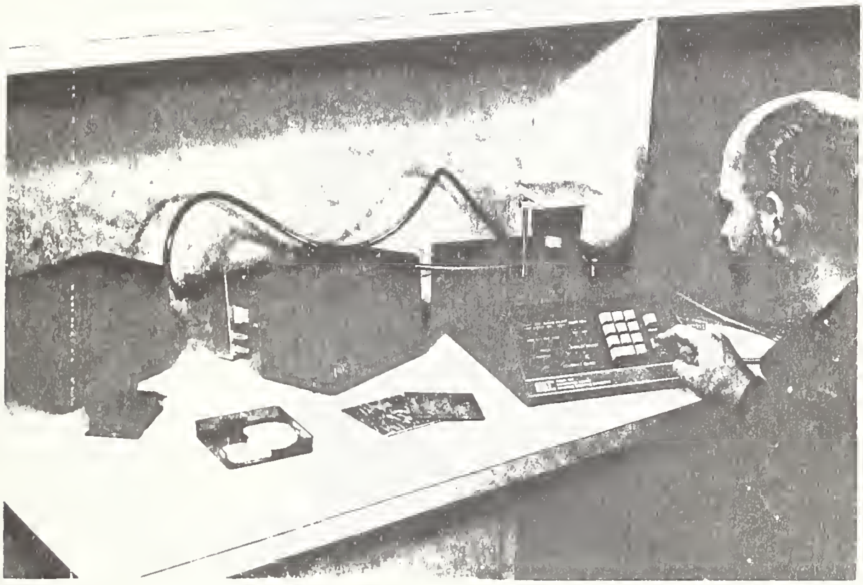

Figure 3 - CSI Model 750 Lead Analyzer Being Calibrated

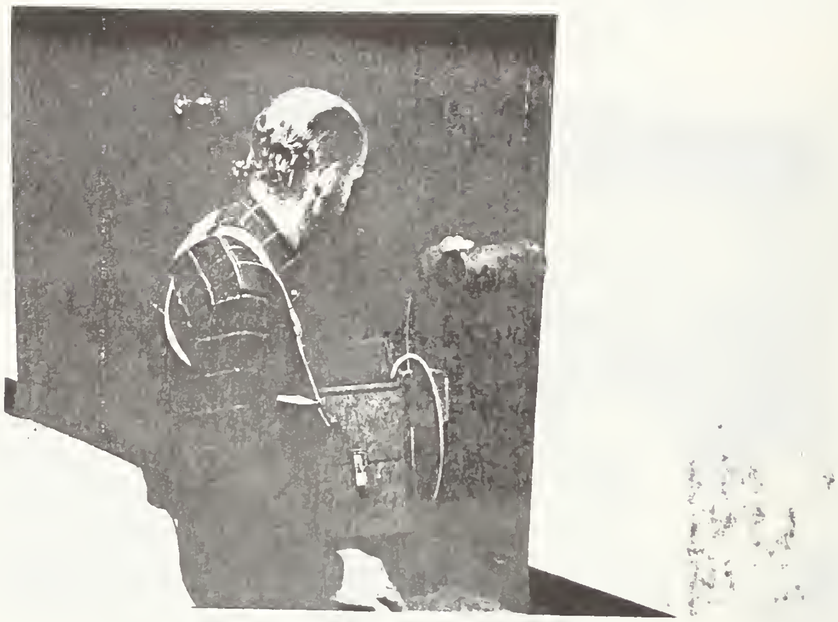

Figure 4 - CSI Model 750 Lead Analyzer in Operation

The procedure to be followed in determining the lead content of a paint film is briefly as follows:

The probe head is placed against the painted surface. The probe trigger switch is depressed and a "ready to measure" tone is heard. The probe head is then pushed in by pressing it against the surface being analyzed until it contacts an internal microswitch. This initiates the "measuring period" and a "measuring tone" sounds until this period is complete. When the "measurement complete" tone sounds, the trigger is released and the measurement head is removed from the analyzed surface. The result is then displayed in about 5 seconds.

3.2 PRINCETON GAMMA TECH INC., XK-3 LEAD PAINT ANALYZER

All the working parts of the PGT XK-3 lead analyzer are in a single case. The power for most of its electronic system is provided by a nine volt rechargeable nickel cadmium (NiCd) power pack in a separate case except for a small current required to maintain instrumental calibration. The power pack is connected to the lead analyzer by means of a detachable cable. Figure 5 illustrates the lead analyzer attached to the power pack, ready for operation. Its carrying case is in the background.

The dimensions of the lead analyzer are 9-1/2 in long, 7 in high, and 3-3/4 in wide (24.1 cm x 17.8 cm x 9.5 cm). It weighs 5.4 lbs (2.45 Kg). The power pack is 2-1/4 in x 3-1/4 in x 6 in (5.7 cm x 9.5 cm x 15.2 cm), and weighs 1.45 lbs. (0.66 Kg). The total weight of the power pack and connecting cord is 7.1 lbs (3.22 Kg).

The principal components of the Model XK-3 lead analyzer are:

°a 10 milliCurie Cobalt 57 Co K hard X-ray source. The source has a half life of about nine months;

°a proportional counter tube which receives the reflected photons from the material being analyzed and converts their energy into an ionic current;

°a trigger operated shutter through which the X-rays pass;

°a preamplifier for converting ionic current into voltage;

°a linear amplifier for both shaping the current from the preamplifier and and increasing its power;

°a liquid crystal display (read out) system on which the lead analysis results appear;

°a small rechargeable NiCd battery for supplying an extremely small current for maintaining the instrumental calibration;

°an electrical cord for use in charging the batteries;

The XK-3 has a CMOS (complementary metallic oxide semiconductor) circuit which requires an extremely small electric current to operate it. This current is about 1/100 that required to operate the PGT XK-2 commercial XRF lead analyzer.

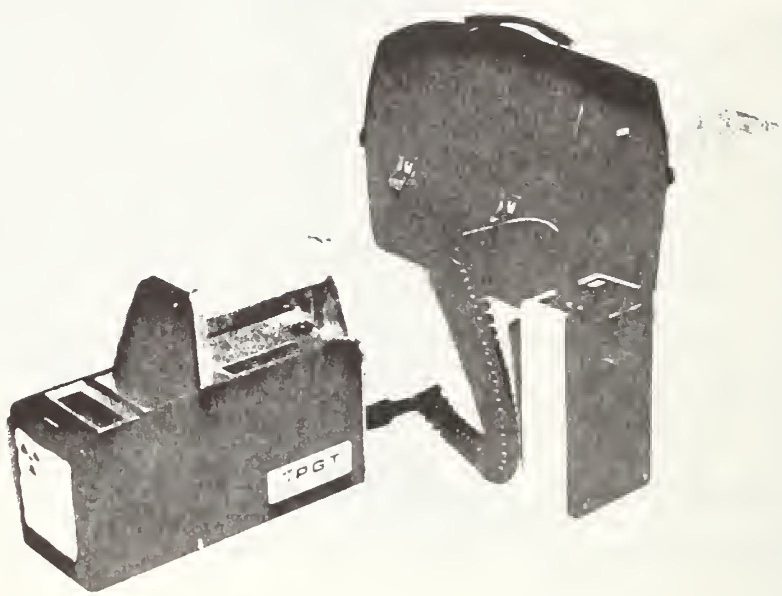

Figure 5 - PGT XK-3 Lead Analyzer Attached to Power
Pack Ready for Use

The minimum sample size required for analysis by the XK-3 is about 2 in x 2 in (50.8 mm x 50.8 mm).

3.2.1 Calibration of PGT XK-3

Calibration is necessary before the XK-3 can be used. This must be done by the manufacturer because it involves electronic adjustments requiring special expertise.

Calibrations are made against NBS reference films containing about 0.6 mg/cm^2 1.5 mg/cm^2, 3.0 mg/cm^2, and 5.0 mg/cm^2 of lead, placed successively on wood, gypsum wallboard and steel substrate. These substrates were chosen to represent low to high ranges in density, which might be found as painted substrates in housing.

3.2.2 Method of Operation of PGT XK-3

The operation of the XK-3 is very simple. First, the batteries should be charged for about 8 hours. The battery pack is then attached to the lead analyzer, and the instrument is ready for use. Attached to the battery pack is a metal clamp for fastening it to the belt of the operator. A lock switch on the lead analyzer is released to allow the instrument switch to be turned on. The lead analyzer is then placed on the material to be analyzed and the handle depressed fully to set the instrument in operation. The lead analysis takes about 30 seconds, and the results appear on the liquid crystal display. If the battery pack power approaches exhaustion, a warning signal of three dots appears on the display.

The highest reading this lead analyzer can indicate is 10.0 mg/cm^2. Any lead content higher than this amount is recorded on the display as 10.0 mg/cm^2. The inability of the instrument to indicate lead contents higher than 10 mg/cm^2 is not serious because any lead level over 2.0 mg/cm^2 is generally regarded to be hazardous. Figure 6 shows the XK-3 in operation.

4. PERFORMANCE REQUIREMENTS OF THE INSTRUMENT

The HUD contracts for both the Columbia Scientific and Princeton Gamma Tech experimental XRF lead analyzers presented two sets of design specifications. These are identical in each contract. The first set, as described in Task IIA of the contracts are minimum performance levels that the instruments <u>must</u> meet, which are as follows:

1) <u>Safety</u> - if the detector system involves the use of radioactive materials, it shall be fully shielded so as to protect occupants and/or operator.

2) <u>Sensitivity and Precision</u> - Minimum sensitivity 1.0 mg lead/cm^2. Precision $\pm$ 0.2 mg lead/cm^2 at 1.0 mg/cm^2.

3) <u>Adaptability</u> - shall measure 50% of the total lead through 20 coats of paint on planar or curved surfaces.

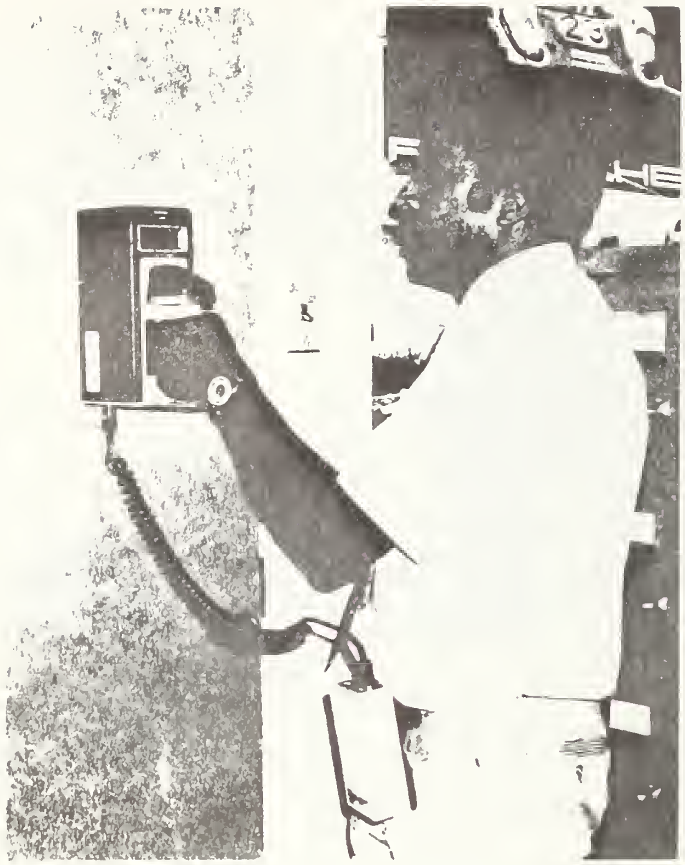

Figure 6 - PGT XK-3 Lead Analyzer in Operation

4) Use - shall be readily operated by semiskilled personnel.

5) Cost per Detection - shall not cost more than $2.00 per analysis.

The second set of requirements, as described in Task IIB, are optimum performance levels which the contractor is encouraged to use his best efforts to achieve. Those optimum performance levels are as follows:

1) Sensitivity, Accuracy and Precision - the system shall be capable of quantitative determinations to a lower limit of 0.5 mg lead/cm^2. For the range of 0.5 to 2.5 mg/cm^2, the system shall measure lead within $\pm$ 0.1 mg/cm^2 for 9 out of 10 measurements and within $\pm$ 0.2 mg/cm^2 for 10 out of 10 measurements. In the range of 2.5 to 10 mg/cm^2 the system shall be capable of measuring lead levels to within $\pm$10% for 9 out of 10 measurements and within $\pm$20% for 10 out of 10 measurements. In the range of 0.1 to 0.4 mg/cm^2 the system shall be able to detect the presence of lead in 9 out of 10 measurements with no specific accuracy.

2) Adaptability - the system shall be capable of detecting at least 80% of the total lead present in a coating of at least 20 layers of paint.

The contract described 10 additional attributes for which the prototype lead detectors are intended to achieve specific performance levels. They are:

3) Service Life

4) Durability

5) Maintenance and Use

6) Sensitivity to Temperature

7) Sensitivity to Humidity

8) Cost per Detection and Measurement

9) Sample Requirement

10) Portability

11) Speed of Analysis

12) Aesthetics

The procedures used for evaluating the CSI and PGT prototypes are contained in an NBS manual entitled "Manual for Evaluating Prototype X-Ray Fluorescent Lead Detection Instruments for the Lead Paint Poisoning Project."

This report presents data and analyses resulting from an evaluation of the CSI Model 750 and the PGT XK-3 prototype lead detectors relevant to both the minimum performance levels and optimum performance levels, in the areas of sensitivity, accuracy, precision, adaptability, and safety. It also includes evaluation of the prototypes for optimum performance requirements selected by the HUD Lead Paint Poisoning Project Manager from the above Task IIB list of requirements namely:

1) Temperature

2) Sample Requirement

3) Portability

4) Speed of Analysis

5) Aesthetics

4.1 DEFINITIONS OF TERMS

For the purpose of this report the following definitions will be used:

a. Sensitivity - a positive instrumental response to the presence of a predetermined level of lead. The response need not be reproducible nor accurate. It need only be a positive indication of the presence of lead.

b. Minimum Sensitivity - the limit of detection, or the lowest lead content which will elicit a positive instrumental response to the presence of lead.

c. Accuracy - the degree to which the instrument measures a known amount of lead in a reference material or other standard. The accuracy of an instrument may be determined as a function of either a single measurement or as a function of the average of replicate measurements.

d. Precision - the degree of repeatability of replicate measurements on the same test sample. An instrument which has a high degree of repeatability does not necessarily have good accuracy.

5. EVALUATIONS

5.1 CSI MODEL 750

5.1.1 Preliminary Evaluation

One of the 3 prototypes (No. 106) and the calibration module fabricated under the HUD contract were first received at NBS in June 1976.

Firstly, the prototype was tested for radiation safety. The radiation levels emitted by it were determined by the staff of the NBS Health Physics Laboratory.

Their findings were that the prototype exhibited "no significant" radiation emission or radioactive contamination when exposed to both the Radiation Survey Meter Test, and the Radiation Smear Test.

Attempts at NBS to calibrate instrument No. 106 failed and it was returned to CSI along with the calibration module for some electronic modifications. Following this, CSI calibrated it and the other two prototypes, No.'s 107 and 108, and sent all three to NBS in October 1976 for evaluation.

During the period of October 7 to December 10, 1976, NBS evaluated the performance of the three prototypes. Instruments 106 and 107 were very inaccurate and imprecise while No. 108 was inoperable and had to be returned to CSI for repairs.

5.1.2 Final Evaluation of CSI Model 750 - Accuracy and Precision

During the period of December 15 to 17, 1976, 106 and 107 were evaluated at NBS by NBS personnel assisted by a member of CSI's technical staff, after their radiation safety had been assured.

No. 106 still had not been repaired at that time, so it was not evaluated. Further evaluations of No.'s 106 and 107 were made by NBS personnel in January and February, 1977.

5.1.3 Procedure for Determining the Accuracy and Precision of CSI Model 750's

The accuracy and precision of CSI lead analyzers No.'s 106 and 107 were determined, using three series of reference materials of known lead levels: CSI references, NBS reference coatings, and paper backed lead paint references (prepared for HUD's use) on different substrates. The temperature of the test area was 73° F ± 4°F, and the relative himidity 50% ± 5%.

The following is a detailed description of the series of references and substrates used in the evaluations of the CSI analyzers No.'s 106 and 107.

1) CSI References, lead content mg/cm^2

    zero

    0.69

    1.55

    3.11

    6.69

As recommended by CSI, each reference used had a total thickness equivalent to about 8 layers of paint. The references were made up of films containing lead contents of the desired amount, and additional non-leaded films, as necessary, placed on top of the leaded film to give the desired thickness. This supposedly eliminated any effect reference thickness variation might have on instrumental accuracy or precision.

Each of the above references was placed on the following substrates for the evaluations:

1/8" plywood (unsealed)

1/2" gypsum wallboard

0.014" Mylar sheet

2) NBS References

Lead Content (mg/cm^2)	Substrate
1.05	1/8" (3.18 mm) plywood (unsealed)
1.06	1/8" (3.18 mm) plywood (sealed)*
0.70	1/2" (1.27 mm) gypsum wallboard
0.82	0.014" (0.355 mm) Mylar sheet
0.88	0.035" (0.89 mm) steel sheet

The NBS references were used to determine not only how well the CSI analyzers performed, but also whether or not they met the HUD contract requirement that they be capable of measuring within ± 0.2 mg lead/cm^2 of a nominal 1.0 mg/cm^2 standard.

3) Paper backed lead paint references for field use, mg lead/cm^2

0.60

1.50

3.0

Each of the above references was placed on the following substrates for the determinations:

1/8" (3.18 mm) plywood (unsealed)

1/2" (12.7 mm) gypsum wallboard

0.014" (0.356 mm) Mylar sheet

Differences in the material composition of the table tops on which the determinations might be carried out were found to cause appreciable differences in the results.

*Sealed with a varnish primer

To eliminate this variable, all determinations were conducted on a 3/4" (19.1 mm) thick wood pull-out writing board of a desk.

Ten replicate measurements of each CSI, NBS, and painted paper backed references, on the substrates indicated, were made with each instrument. The results appear in Tables 1 and 2.

The relative accuracy of the instruments is determined from the Difference (Col. 4) between the Mean (Col. 2) and Standard Value (Col. 1) (D=M-SV). The precision is indicated by the standard deviation (Col. 3).

Column 5 lists the number of measurements departing from the mean (not the standard value) by more than ± 0.2 mg/cm. These data (in Col. 5) also give an indication of the precision of the instruments and their conformance to the HUD contract specification.

5.1.4 Comments on CSI Model 750 Performance

CSI No. 106

This analyzer generally exhibited poor accuracy and precision. For example, the means of the determinations on all reference films and coatings with plywood as substrate were significantly lower than the standard values of the references and coatings. This was also true for the results with Mylar as the substrate, except for one case where the result was considerably higher than the standard value. The means of the determination with gypsum wallboard as the substrates were too high except for two instances where the means were too low. The determinations with steel as substrate were much too high.

CSI No. 107

This analyzer had the same shortcomings as No. 106 and was also generally inaccurate and imprecise. The results from the determinations with steel and gypsum as substrates resembled those from No. 106 in that they were generally higher than the values of the references. With Mylar as substrate the results were too high. This was a decided contrast to results from No. 106 which were too low. The results with plywood as substrate ranged from lower to much higher than the standard values fo the references. There were no definite error trends in the results from either of the analyzers.

5.1.5 Compliance with the HUD contract

5.1.5.1 User Qualities - Portability, Ease of Operation and Aesthetics

(a) Portability

The CSI XRF Model 750 lead analyzer consists of an electronic unit weighing 11.5 lbs. (5.23 kg) and a probe weighing 2.9 lbs. (1.31 kg) connected by an electrical cable. Thus, it weighs more than the 10 lb (4.5 kg) maximum weight

Table I Performance Evaluation Results
of CSI XRF Lead Analyzer No. 106

Response Time: 18 secs.

(1) Standard Value (SV) mg/cm^2 lead	(2) Mean (M) mg/cm^2	(3) Standard Deviation (SD) mg/cm^2	(4) Difference (D=M-SV) mg/cm^2	(5) Determination* > ± 0.2 mg/cm^2 lead
CSI Reference films				
1/8" plywood unsealed				
0	0	0	0	0
0.69	0.56	0.22	-0.13	2
1.55	1.43	0.20	-0.12	3
3.11	2.44	0.31	-0.67	6
6.69	5.54	0.31	-1.15	5
1/2" Gypsum wallboard				
0	0.21	0.21	0.21	4
0.69	1.06	0.28	0.37	4
1.55	2.24	0.35	0.68	5
3.11	3.65	0.38	0.54	7
6.69	6.87	0.43	0.18	6
0.014" Mylar sheet				
0	0	0	0	0
0.69	0.08	0.11	-0.61	1
1.55	0.66	0.25	-0.89	3
3.11	2.26	0.27	-0.85	2
6.69	5.45	0.15	-1.24	1
NBS References				
1/8" plywood (unsealed)				
1.05	0.27	0.22	-0.78	4
1/8" plywood (sealed)				
1.06	0.26	0.23	-0.80	3
1/2" Gypsum wallboard				
0.70	1.17	0.29	0.47	4
0.014" Mylar sheet				
0.82	1.48	0.28	0.66	5
0.035" Steel sheet				
0.88	2.85	0.21	1.97	3
Paper backed lead paint reference films				
1/8" plywood (unsealed)				
0.60	0.16	0.18	-0.44	2
1.50	1.13	0.26	-0.37	5
3.00	2.59	0.34	-0.41	5
1/2" Gypsum wallboard				
0.60	0.37	0.21	-0.23	3
1.50	1.55	0.36	0.05	6
3.00	2.87	0.38	-0.13	4
0.014" Mylar sheet				
0.60	0.16	0.21	-0.44	2
1.50	1.38	0.35	-0.12	5
3.00	2.40	0.22	-0.60	4

*No. of determinations exceeding M ± 0.2

1 inch = 25.4 mm

Table 2 Performance Evaluation Results of CSI, XRF Lead Analyzer No. 107

Response Time 20 secs.

(1) Standard Value (SV) mg/cm^2 lead	(2) Mean (M) mg/cm^2	(3) Standard Deviation (SD) mg/cm^2	(4) Difference (D=M-SV) mg/cm^2	(5) Determinations* > ± 0.2 mg/cm^2 lead
CSI Reference films				
1/8" plywood (unsealed)				
0	0	0	0	0
0.69	0.52	0.59	-0.17	9
1.55	1.23	0.44	-0.32	5
3.11	3.37	0.35	0.26	5
6.69	7.59	0.20	0.90	2
1/2" Gypsum wallboard				
0	0.48	0.24	0.48	3
0.69	1.16	0.56	0.64	5
1.55	2.02	0.39	0.47	5
3.11	3.44	1.29	0.33	7
6.69	7.63	0.58	0.94	8
0.014" Mylar sheet				
0	0.32	0.31	0.32	4
0.69	0.76	0.35	0.07	4
1.55	1.44	0.35	-0.11	7
3.11	3.52	0.32	0.41	6
6.69	6.95	0.32	0.26	5
NBS References				
1/8" Plywood (unsealed)				
1.05	0.67	0.33	-0.38	8
1/8" Plywood (sealed)				
1.06	1.07	0.31	0.01	6
1/2" Gypsum wallboard				
0.70	1.78	0.24	1.08	3
0.014" Mylar sheet				
0.82	1.37	0.29	0.55	3
0.035" Steel sheet				
0.88	2.86	0.28	1.98	6
Paper backed lead paint references				
1/8" plywood (unsealed)				
0.60	1.76	0.34	1.16	4
1.50	2.15	0.40	0.65	8
3.00	3.82	0.28	0.82	7
1/2" Gypsum wallboard				
0.60	1.08	0.28	0.48	4
1.50	2.15	0.40	0.65	8
3.00	3.82	0.28	0.82	6
0.014" Mylar sheet				
0.60	0.85	0.39	0.25	5
1.50	2.20	0.18	0.70	3
3.00	4.26	0.31	1.26	4

*No. of determinations exceeding M ± 0.2

1 inch = 25.4 mm

criterion in the HUD contract. In the opinion of a number of operators, the analyzer is excessively heavy and bulky which makes it awkward to carry and operate, especially for long periods of time.

Although the probe is rather light and easy to carry, a pushing force of about 8 lbs (3.63 kg) is required to depress its spring-loaded measurement head to initiate measurement. The measurement head must be held in the depressed position during the measurement period of about 20 seconds. This is fatiguing to the operator, especially if the measurement head binds when it is being depressed, which occasionally happens.

(b) Ease of Operation

The operational procedure of the CSI lead analyzer is relatively easy. Consequently, it meets the minimum performance level requirement of the HUD specification which specifies that the instrument "shall be readily operated by semiskilled personnel." However, the calibration procedure for this instrument is complex and too difficult to be entrusted to semiskilled personnel. It would be necessary to send the lead analyzer to CSI for this purpose.

(c) Aesthetics

The HUD contract requires that the instrument shall not emit "noxious fumes" or loud noise, produce black marks, gouges or excessive holes on the surface being examined.

The CSI lead analyzer does not emit fumes, but the tonal signal system (a "beeper") which indicates by means of pulsating tones what its operational status of action is (i.e., measuring, turned on but not measuring, etc.) can be annoying to the operator or other people in the vicinity.

The facing of the probe is steel and contains some recessed screws. The facing frequently leaves grey-black marks on the surfaces being analyzed, and the screw heads occasionally scratch them.

5.1.5.2 Sensitivity and Precision

The HUD contract requires the following performance of newly developed X-ray Fluorescent lead detectors .

°Minimum sensitivity 1.0 mg lead/cm^2

°Precision $\pm$ 0.2 milligrams lead/cm^2 at 1.0 mg/cm^2

With regard to the requirement on precision, some interpretation is required. If the word precision was intended in the original contract to be synonymous with accuracy with which the instrument measure the true lead content at a nominal 1 mg lead/cm^2, with the deviation of $\pm$ 0.2 mg/cm^2 from the true value, reference to Table 3 (an abstract from Tables 1 and 2) shows that in only 5 out of 22 cases did the means of 10 repetitive measurements meet that requirement. Even in those 5 cases, the number of single determinations that deviated from the

Table 3 Results From CSI Lead Analyzers on References with Lead Contents of 0.70 mg/cm^2 to 1.55 mg/cm

Standard Value (SV) mg/cm^2 lead	CSI XRF Instrument No. 106 Response Time: 18 secs.				CSI XRF Instrument No. 107 Response Time: 20 secs.			
	Mean (M) mg/cm^2	Standard Deviation (SD) mg/cm^2	Difference (D=M-SV) mg/cm^2	Determinations* ± 0.2 mg/cm^2 lead	Mean (M) mg/cm^2	Standard Deviation (SD) mg/cm^2	Difference (D=M-SV) mg/cm^2	Determinations* ± 0.2 mg/cm^2 lead
CSI Reference films								
1/8" plywood (unsealed) 1.55	1.43	0.20	-0.12	3	1.23	0.44	-0.32	5
1/2" Gypsum wallboard 1.55	2.24	0.35	0.68	5	2.02	0.39	0.47	5
0.014" Mylar sheet 1.55	0.66	0.25	-0.89	3	1.44	0.35	-0.11	7
NBS References								
1/8" plywood (unsealed) 1.05	0.27	0.22	-0.78	4	0.67	0.33	-0.38	8
1/8" plywood (sealed) 1.06	0.26	0.23	-0.80	3	1.07	0.31	0.01	6
1/2" Gypsum wallboard 0.70	1.17	0.29	0.47	4	1.78	0.24	1.08	3
0.014" Mylar sheet 0.82	1.48	0.28	0.66	5	1.37	0.29	0.55	3
0.035" Steel sheet 0.88	2.85	0.21	1.97	3	2.86	0.28	1.98	6
Paper Backed lead paint references								
1/8" plywood (unsealed) 1.50	1.13	0.26	-0.37	5	2.84	0.34	1.34	5
1/2" Gypsum wallboard 1.50	1.55	0.36	0.05	6	2.15	0.40	0.65	8
0.014" Mylar sheet 1.50	1.38	0.35	-0.12	5	2.20	0.18	0.70	3

*No. of determinations exceeding M ±0.2

1 inch = 25.4 mm.

mean by more than $\pm$ 0.2 mg/cm^2 of the mean, then the standard deviations shown in Table 4 indicate that in all cases but 2, the requirement was not met.

In short, by any reasonable definition or interpretation the performance of the CSI instruments fail to meet the letter, or intent of the HUD requirements as expressed in the contract document.

In addition to their failure to accurately measure the lead contents of the three series of reference materials, the two CSI instruments indicate severe limitations with regard to consistency between themselves. For example, reference to Table 3 shows comparisons between instruments numbers 106 and 107 in measuring the paper backed lead paint references. The differences ranged as high as about 1.8 mg/cm^2 on identical reference panels.

Because the CSI Model 750 prototypes failed to meet the accuracy and precision specifications of the HUD contract, and lesser requirements such as portability and asesthetics, further testing as required by the HUD contract was abandoned for these instruments.

5.1.6 Conclusions

As far as can be determined there are no set patterns that indicate that mere recalibrations will resolve the basic accuracy and precision limitations of these instruments. There is no consistency between the two instruments with regard to preferential accuracy or precision on any particular substrate. We are not in a position to comment on retention of the calibration of the instruments although previous experience seems to indicate that that will be a rather frequent problem.

HUD should consider the inaccuracy of the instruments, their generally unsatisfactory user characteristics, the need for a calibration module and the apparent need for continued developmental work, in making any further decisions with regard to these instruments.

5.1.7 Comments by NBS Analytical Chemists

Three analytical chemists from the Analytical Chemistry Division who have expertise in X-ray fluorescent analyzers were asked for a critique of the CSI analyzers. All believe that the principles by which the analyzers operate are technically sound. However, it appeared to them that a considerable amount of research and redesign on the analyzers might be necessary to bring their accuracy and precision to an acceptable level.

5.2 PGT XK-3

5.2.1 Preliminary Evaluations

In all, three preliminary evaluations of the three PGT XK-3 prototypes, No.'s 101, 102, and 103 were conducted for radiation safety, and accuracy and precision by NBS during the period of July 15, 1976 to January 31, 1977. Standard paint films of precisely known lead contents on various substrates were used for these evaluations.

The first preliminary evaluation revealed that not only were all the prototypes very inaccurate, but also the two small 9 volt alkaline nonrechargeable batteries located in the instruments became exhausted after only 3 to 4 hours of use. Obviously, this cannot be tolerated in field service.

Consequently, PGT not only recalibrated the instruments, but also equipped them each with a 12 volt rechargeable nickel cadmium (NiCd) power pack connected to the XK-3 with a cable. One of the small 9 volt batteries was retained to supply the minute amount of current required to maintain instrumental calibration.

The second evaluation revealed that the accuracy and precision of No. 101 were quite good, but were poor for the other two. It was decided by PGT that the poor accuracy and precision may have been caused by the 12 volt power pack not having sufficient storage capacity for use with the XK-3. Consequently, this power pack was replaced by a 9 volt type with a much higher storage capacity. The prototypes were then recalibrated and submitted to NBS for reevaluation.

5.2.2 Final Evaluation

In the final evaluation the prototypes were first submitted to appropriate test methods in the "Manual for Evaluating Prototype X-ray Fluorescent Lead Detection Instruments for the Lead Paint Poisoning Project" to determine whether they met the requirements in Task IIA and B discussed in Section 4.0 of this report. The following is a discussion of the results.

5.2.2.1 Minimum Requirements Task IIA

1. Safety

   The NBS Health Physics Laboratory found that the XK-3 prototypes exhibited "no significant" radiation emission or radioactive contamination when exposed to both the Radiation Survey Meter Test, and the Radiation Smear Test.

2. Minimum Sensitivity

   The minimum sensitivity required by the HUD contract is 1.0 mg lead/cm^2. The PGT XK-3 prototypes consistently gave a positive indication of the presence of lead at a level of 1.0 mg/cm^2, on a variety of substrates such as wood, gypsum board, Mylar and steel.

3. Precision

The HUD specification requires a minimum precision of $\pm$0.2 mg lead/cm^2 at 1.0 mg/cm^2. It is our interpretation that the word precision, as it is used in the HUD specification, refers to what we have defined as accuracy, (see section 4.1). Therefore, the following data is directed at presenting the evaluation of the XK-3's with regard to their ability to accurately measure lead in paint at or about 1.0 mg/cm^2. If our interpretation is in error, the following data is still appropriate for evaluating the repeatability of the instruments in measuring lead at a nominal 1.0 mg/cm^2 level.

The data in Table 4 serve for several analyses. For example, column 3 shows the difference between the known lead content of the reference materials prepared by NBS and the averages of ten replicate measurements made with each of the three prototype instruments on five different substrates. Of 15 series of measurements, all the measurements but 5 exceeded a difference of $\pm$ 0.2 mg lead/cm^2 from the actual lead content. Furthermore, out of a total of 150 replicate measurements with the three detectors, 100 measurements exceeded $\pm$ 0.2 mg/cm^2 of the actual lead contents of the reference materials. Using the averages of the observed replicate measurements, 41 out of a total 150 measurements exceeded $\pm$ 0.2 mg/cm^2 of the observed average readings as listed in column 2 of Table 1. In summary, therefore, if the required precision statement is taken to mean accuracy, only 50 of 150 measurements, on reference materials having a nominal value of approximately 1 mg/cm^2, were found to be within $\pm$ mg/cm^2 of the actual lead value.

The XK-3 lead detectors cannot be calibrated by the operator: the calibration is accomplished by the manufacturer. As part of the calibration process, the detectors are tuned to a particular substrate. X-ray fluorescent lead detectors are supposedly sensitive to variation in substrates both with regard to the nature of the substrate and substrate thickness. Table 5 presents a compilation of the data in Table 4 in a manner intended to highlight the effects of the substrate on the measurement capability of the XK-3 lead detectors.

The results for instrument No. 102 are clearly different from those of the other two instruments. We believe that recalibration of instrument No. 102 would lead to a substantial improvement in its accuracy, at least insofar as the average of replicate measurements is concerned.

NBS was informed by Princeton Gamma Tech, Inc. that the limit of accuracy to be expected of the XK-3 instrument is $\pm$ 0.3 mg lead/cm^2. Table 7 shows that 54 out of 150 measurements made with the three prototypes on the 5 substrates shown in Table 4 exceed $\pm$ 0.3 mg/cm^2 of the lead content of those reference materials.

Table 4 Accuracy of PGT XK-3's at about the 1 mg/cm^2 Lead Level

Substrate	(1) Standard Value (SV) mg/cm^2 lead[(a)]	(2) Mean (M) mg/cm^2 [(b)]	(3) Difference (D=M-SV) mg/cm^2	(4) Determinations > $\pm$ 0.2 mg/cm^2 of (1)	(5) Determinations > $\pm$ 0.2 mg/cm^2 of (2)
<u>XK-3 No. 101</u>					
Unsealed wood	1.05	1.07	+0.02	6	6
Sealed wood	1.06	0.93	-0.14	5	5
Gypsum board	0.70	0.75	+0.05	3	0
Mylar	0.82	1.17	+0.35	9	1
Steel	0.88	0.91	+0.03	3	3
<u>XK-3 No. 102</u>					
Unsealed wood	1.05	0.84	-0.21	7	2
Sealed wood	1.06	0.58	-0.48	10	2
Gypsum board	0.70	0.38	-0.32	10	0
Mylar	0.82	0.62	-0.20	6	5
Steel	0.88	0.55	-0.33	7	3
<u>XK-3 No. 103</u>					
Unsealed wood	1.05	1.00	-0.05	7	3
Sealed wood	1.06	0.64	-0.42	9	1
Gypsum board	0.70	0.85	+0.15	4	3
Mylar	0.82	1.01	+0.19	7	4
Steel	0.88	1.08	+0.20	7	3

[(a)] The lead contents of the reference materials used by NBS were determined by chemical analysis using Atomic Absorption Spectrometry.

[(b)] The average of 10 replicate measurements. The instruments read to 0.1 mg/cm^2: an additional calculated significant figure is used here for convenience.

Table 5 Substrate Effects (Combined data for 3 instruments)

Substrate	Lead Content (a)	Mg Lead/cm² Gross Average (b)[1]	Average Difference (a-b)
Unsealed wood	1.05	0.97	-0.08
Sealed wood	1.06	0.72	-0.34
Gypsum board	0.70	0.66	-0.04
Mylar	0.82	0.93	+0.11
Steel	0.88	0.85	-0.03

[1]Average of 3 averages in Column (2) Table 4.

The second column in Table 5 presents the gross averages of readings for all 3 lead detectors for each particular substrate. The average difference between the actual lead content and the average of 30 replicate measurements is shown in the third column of the table. The greatest difference is obtained for measurements on sealed wood. There is no obvious reason for this effect especially since the average difference obtained for measurements on unsealed wood is comparable to the averages for gypsum wallboard, Mylar and steel. The data in Table 5 leads us to conclude that the XK-3 lead detectors do not show extreme trends for particular substrates. This conclusion is supported by the data in Table 6 (based on all of the data in Table 4) which shows general trends in the measurement response of the three instruments.

Table 6 Instrument Trends

Instrument	Mg Lead/cm² Arithmetic Sum of Difference[1]	Mg Lead/cm² Algebraic Sum of Differences[1]
No. 101	0.6	+0.4
No. 102	1.5	-1.5
No. 103	1.1	+0.1

[1]Differences in Column (3) of Table 4.

Table 7 Measurements Exceeding Reference Values by More than $\pm$ 0.3 mg/cm^2*.

Substrate	XK-3 Prototype 101	102	103
Unsealed wood	1	3	1
Sealed wood	3	9	7
Sypsum board	0	5	2
Mylar	6	4	3
Steel	3	4	3

Sum exceeding $\pm$ 0.3 mg lead/cm^2, 54 (out of 150 measurements)

*10 replicate measurements on each substrate using each instrument.

The distribution of differences between known lead values and observed averages leads us to believe that instruments No. 101 and 103 cannot be significantly improved by recalibration. Although we believe that instrument No. 102 can be improved, the overall accuracy or ability to read within $\pm$ 0.2 mg of a known value is not expected to increase significantly.

4. Adaptability

The performance requirements for adaptability relate to two essentially different parameters. One is the ability to detect lead paint on non-planar surfaces, such as ornate wood trim, and the second is the ability to detect lead paint which has been covered by as many as 20 layers of non-leaded paint. The minimum performance requirement as stated in the HUD contract is interpreted as requiring an instrumental capability to detect 50% of the total lead present in both multi-layer paints and in coatings on non-planar surfaces. Table 8 indicates the degree to which the XK-3 prototypes meet the requirements relating to measurement capability on non-planar surfaces.

Table 8 Measurement of Lead on Non-Planar (Trim) Surfaces*

Trim Specimen	Position	Measurement Direction[1]	Lead Content (mg/cm^2)	Observed Average (mg/cm^2)	% Lead Detected
1	a	T	1.1	0.3	27
2	a	T	3.0	3.8	> 100
2	b	L	3.0	3.3	> 100
2	c	L	3.0	1.4	47
2	c	L	1.5	0.6	40
3	a	T	3.0	2.6	87
3	b	L	3.0	2.4	80
3	a	T	1.5	1.4	93
3	b	L	1.5	1.6	> 100
4	a	T	17.2	10.0	58

[1]Transverse to trim (T), Longitudinal to trim (L)
*Instrument No. 101

Measurements were made on ornate wood trim samples which are shown in cross-sections in Figure 7. The placement of the lead detector obviously has a bearing on the observed measurement. Surfaces which are parallel to the plane of the instrument's window seem to produce more accurate measurements than skewed surfaces even when they are displaced from the instrument by an air gap. The instrument has a circular window 1.75 inches (4.45 cm) in diameter and is therefore bridging a complex surface in most of the measurements on ornate wood trim. In the case of the wood trim containing 17.2 mg lead/cm^2 the instrument satisfactorily detected the lead present by indicating 10.0 mg/cm^2 (which is the highest reading the instrument can make).

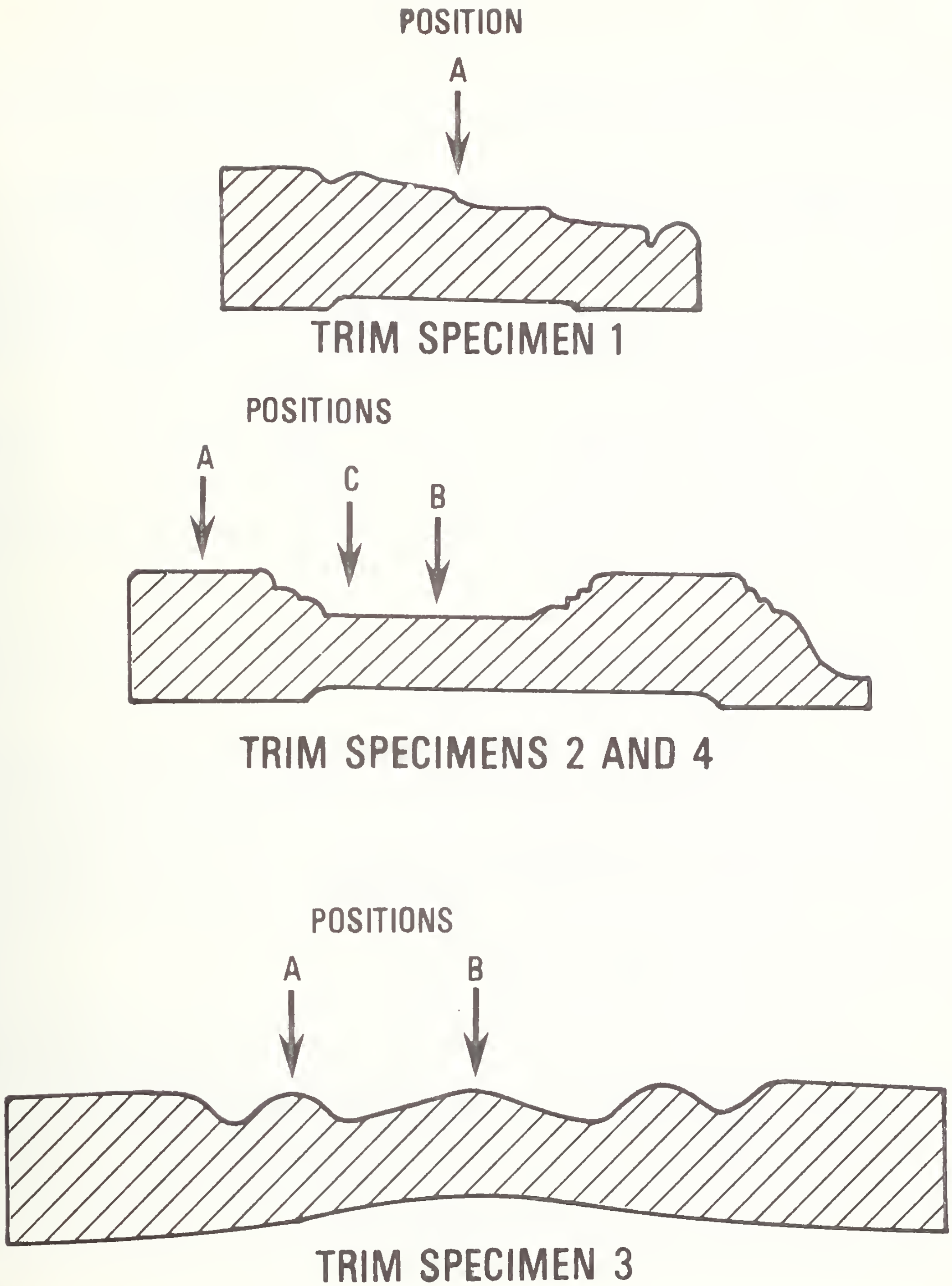

Figure 7 - Measurement Positions of the PGT XK-3 on Ornate Wood Trim Coated With Lead Paint

The performance of the XK-3 prototype in measuring and detecting lead in multi-layer paint films is shown in Table 9 below.

Table 9 Measurement of Lead in Multi-Layered Paint* on 1/2 in Gypsum Wallboard

Number of Layers	Lead Content (mg/cm^2)	Average Instrument Reading (10 replicates)	Number of Readings < 50%
4	0.17	0.24	3
8	1.31	1.45	0
15	2.03	2.14	0
20	2.97	2.71	0

*Instrument No. 101

5.2.2.2 Optimum Performance Levels Task IIB

1. Accuracy

The HUD Contract defines optimum performance levels for accuracy as follows:

For the range 0.5 to 2.5 mg lead/cm^2
$\pm$ 0.1 mg/cm^2 for 9 out of 10 replicate measurements
$\pm$ 0.2 mg/cm^2 for 10 out of 10 replicate measurements

For the range 2.5 to 10 mg/cm^2
$\pm$ 10% for the 9 out of 10 replicate measurements
$\pm$ 20% for 10 out of 10 replicate measurements

Table 10 presents the results of the evaluation of the XK-3 prototypes relative to the accuracy requirements stated above. Reference materials containing 0.6, 1.5 and 2.1 mg lead/cm^2 placed on wood, gypsum wallboard, and steel substrates were used for this purpose. Ten replicate measurements were made for each instrument using each of the three reference materials on each of the three substrates. The data in Table 10 indicate that in no case did the XK-3 prototype instruments meet the stated requirements for accuracy in the range 0.5 to 2.5 mg/cm^2. An inspection of the data further indicates that instrument No. 102 is not operating as accurately as instruments No. 101 and 103, as previously stated.

The results for instrument No. 101 seem to indicate a substrate effect such that measurement accuracy on the steel substrate is not as high as for the wood and gypsum wallboard substrates. However, the results for instrument No. 103 are contradictory and seem to indicate that the accuracy on the steel substrate is better than that for the gypsum wallboard and wood. The overall conclusion that can be reached is that individual instruments may be substrate sensitive due to their calibration or tuning.

Table 10 Accuracy of XK-3 Lead Detector in the Range 0.5 mg lead/cm^2 to 2.5 mg/cm^2

Instrument	Reference Material Lead Content	Number of Measurements* within $\pm$ 0.1 mg/cm^2			Number of Measurements within $\pm$ 0.2 mg/cm^2		
		Wood	Gypsum board	Steel	Wood	Gypsum board	Steel
101	0.6	5	2	3	6	6	5
	1.5	4	5	2	8	7	3
	2.1	4	3	1	6	5	1
102	0.6	5	3	1	8	3	3
	1.5	5	6	0	5	7	0
	2.1	0	2	0	0	6	0
103	0.6	1	5	2	2	6	3
	1.5	3	5	4	3	8	6
	2.1	1	0	5	4	5	5

*10 replicate measurements for each reference material and substrate

Table 11 presents data directed at the requirement for accuracy in the range above 2.5 mg/cm^2. In no case did the instruments meet the requirements of falling within $\pm$ 10% of the known value 9 out of 10 times when a reference material containing 3.0 mg/cm^2 was used. Using the second criterion of $\pm$ 20%, instrument No. 103 satisfactorily met that requirement for both the wood and gypsum board substrates.

Table 11 Accuracy of XK-3 Lead Detector Above 2.5 mg lead/cm^2

Instrument	Number of Measurements* within $\pm$ 10% of 3.0 mg/cm^2			Number of Measurements* within $\pm$ 20% of 3.0 mg/cm^2		
	Wood	Gypsum board	Steel	Wood	Gypsum board	Steel
101	4	3	1	9	8	8
102	0	0	0	0	7	0
103	6	8	6	10	10	9

*10 replicate measurements for each reference material and substrate

2. Sensitivity

The minimum performance requirement for sensitivity states that the lead detectors shall be capable of lead detection in the range of 0.1 to 0.4 mg lead/cm^2 with no particular degree of accuracy. On two series of measurements using 1/2 in gypsum wallboard reference material containing 0.25 mg lead/cm^2, instrument No 101* failed to give a positive indication of the presence of lead in 20% of the measurements.

3. Adaptability

The minimum performance requirement for adaptability, states that the lead detectors shall be capable of detecting 80% of the total lead present in a coating containing 20-layers of paint. In an evaluation of instrument No. 101,* using the reference materials listed in Table 9, it was found that the instrument failed to detect 80% of the lead present in the 4-layer material 3 out of 10 times, in the 8-layer material, in 1 of 10 measurements; and successfully detected at least 80% of the lead in all cases for both the 15-layer and 20-layer reference materials. Thus, the detection adaptability rating was the same for the 80% level as for the 50% level.

5.2.2.3 Detection of Lead On Thin Substrates

Although no consistent trend was observed for substrate effects due to the use of one type of substrate material or another, consistent substrate effects were observed relative to the thickness of the substrate being measured. These substrate effects were apparently due to x-ray backscattering phenomena. Prototype No. 101 was used for their evaluation.

The data in Table 12 indicates what might occur in a practical field situation, such as the measurement of lead on a gypsum wallboard or plaster on lath substrate. If, for example, the measurement is made in a location between the supporting 2 in by 4 in (5.1 cm x 10.2 cm) wood studs, the measurement is likely to be lower than one made in a location directly in front of such a supporting member. Table 12 shows that, at lower lead levels, the observed readings with the XK-3 are consistently lower for thinner substrates. The results obtained with the gypsum wallboard substrate placed on a 3-1/2 in (8.9 cm) thick wood backing (which is comparable to either gypsum board or plaster on a wood stud) results in measurements that are essentially satisfactory. Results obtained with the use of gypsum board reference materials containing 1.83 mg lead/cm^2 indicate that the thin substrate effects do not occur at higher lead contents.

*Instrument No. 101 only was evaluated for sensitivity and adaptability.

Table 12 Lead in Paint Measurements on Thick Substrates

Lead Content (mg/cm^2) of Reference	Average Measurement (mg lead/cm^2) Gypsum Board (1/2")	Gypsum Board + 3/8" plywood	Gypsum Board + 3-1/2" wood
0	-0.25	-0.05	0.01
0.25	0.14	0.16	0.56
0.70	0.40	0.68	0.75
1.33	0.86	1.21	1.28
1.83	1.86	1.71	1.80

5.2.2.4 Temperature Effects

An operational temperature susceptibility test was conducted in an environmental chamber at temperatures between 20°F and 140°F (-6.7 °C and 60 °C) in increments of 10°F (5.5 °C). The XK-3 was stored for 1 hour at each temperature, then removed from the chamber briefly for testing. The operational characteristics were tested by determining the lead content of reference materials of known lead content. The XK-3 was found to be inoperable after storage at 25°F (-4 °C) and lower, and 135°F (57°C) and above but operated normally between 30°F (-1°C) and 130°F (54.5°C).

5.2.3 Field Performance

Periodically, HUD loaned the XK-3 prototypes to agencies conducting surveys of housing for lead paint. This is presently being done both to field test the prototypes and to assist agencies in their surveys.

5.2.4 Compliance With The HUD Contract

5.2.4.1 Minimum Requirements, Task IIA

The XK-3 prototypes meet the safety and use requirements in general but not the sensitivity and precision specifications. No. 101 meets the adaptability specification except for multiple paint layers over a paint containing about 0.2 mg lead/cm^2 or less. (No. 101 was the only prototype tested for adaptability).

5.2.4.2 Optimum Performance Levels, Task IIB

The XK-3 meets the following requirements:

1. Sensitivity (i.e., can detect the presence of lead at levels of 0.1-0.4 mg/cm^2 in 9 out of 10 cases with no specific accuracy)

2. Service life

3. Maintenance and Use

4. Temperature

5. Cost per detection and measurement (this was not calculated but the XK-3 should meet this easily)

6. Sample requirement

7. Portability

8. Speed of analysis

9. Aesthetics

The XK-3 does <u>not</u> met the accuracy and precision requirements.

It does not meet the non-planar surface adaptability requirements, but does meet those for multi-paint layers over lead paint.

6. DESCRIPTION OF THE ESTABLISHED COMMERCIAL XRF LEAD ANALYZERS

6.1 PRINCETON GAMMA TECH. XK-2

The PGT XK-2 consists of 2 units; a measuring head, and a control module fitted with a shoulder sling. (See Figure 8).

The measuring head is rectangular and weighs 5.09 lbs (2.31 Kg). Its dimensions are 3-3/4 in x 9-7/8 in x 5-1/8 in, (9.525 cm x 25.08 cm x 20.32 cm).

The chief components of the measuring head are:

°10 milliCurie cobalt 57 Co K hard X-ray source, having a half life of about 9 months (same source as that of the XK-3).

°a proportional counter tube

°a trigger operated shutter for passage of X-rays

°a "shutter open" indicator light

The measuring head is connected to the control module by a power cable through which the current from the proportional counter passes to be processed in the control module.

Control Module

The control module contains:

°Electronic equipment for processing the current from the proportional counter including analog and digital circuitry;

°Calibration controls;

°Modular rechargeable battery pack with battery charger;

°Single line lighted digital numerical display;

°Three indicator lights for indicating on/off, measure, and battery charging modes of the instrument;

°On/off switch;

°A battery charging cord.

Two standard references are provided with the XK-2 for use in calibrating it. They are both 1/2 in (12.7 mm) thick wooden blocks. One of these is a zero lead reference while the other, which has a thin lead plate attached to it, has a precisely known lead content rating of about 75 mg/cm^2. They appear in the foreground of Figure 8.

Operation of PGT XK-2

Firstly, the battery pack must be charged. This requires about 8 hours. Secondly, the XK-2 must be calibrated. This is relatively easy and can be done by the operator, by following instruction in the operations manual. However, it is a fairly lengthy operation and can take as much as 45 minutes.

To measure the lead content of a surface, the measurement head is placed against it and the trigger is squeezed to open the shutter. The trigger is held open until the result, in mg lead/cm^2 appears in the lighted digital numerical display. This takes 10-30 seconds, depending upon the age of X-ray source. Figure 9 shows the XK-2 in field use.

6.2 NUCLEAR CHICAGO PB-3

The Nuclear Chicago (NC) PB-3 lead analyzer consists of two units, a hand held probe and a data processor - display unit. (See Figure 10).

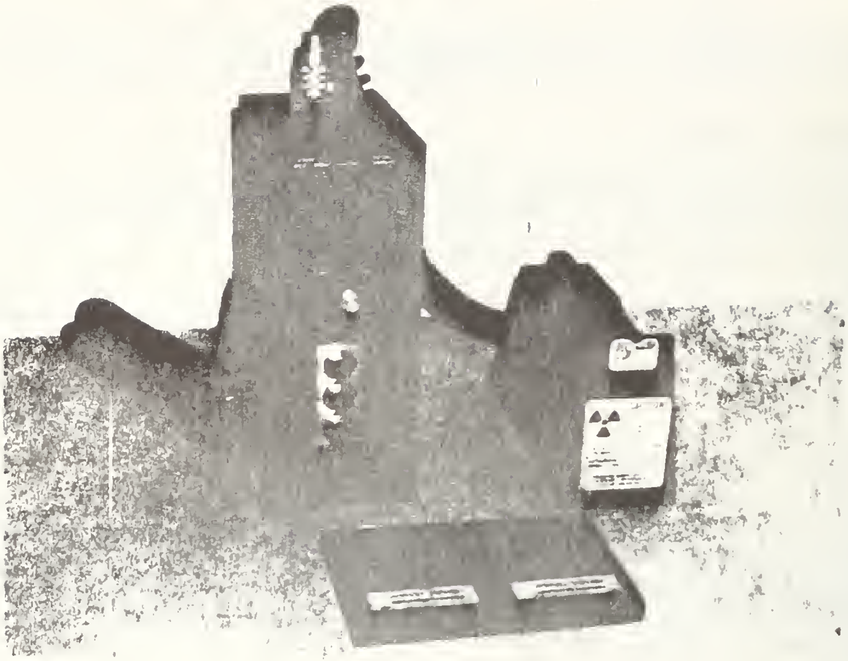

Figure 8 - PGT XK-2 Lead Analyzer Ready for Use

Figure 9 - PGT XK-2 Lead Analyzer in Operation

6.2.1 Probe

The probe is cylindrical and has a pistol grip type of handle. It is 9 in (22.8 cm) long, 2-1/2 in (7.35 cm) in diameter and weighs 3 lbs (1.362 Kg). The handle is 5-1/2 in (13.97 cm) long. The probe's principal components are a Cobalt 57, Co K hard 2 milliCurie X-ray source, an X-ray detector system, and a shutter controlled by a trigger recessed in the handle.

6.2.2 Data Processor

The data processor is rectangular and measures 6 in (15.2 cm) x 8 in (20.3 cm) x 4 in (27.9 cm). It weighs 12 lbs (5.45 Kg) and it contains the following important components:

°analog and digital circuitry to normalize the data from the probe;

°rechargeable battery which is the source of power for both units, and battery charger;

°lighted digital numerical display, showing lead content in mg/cm^2;

°an on/off switch;

°battery condition indicator;

°cord for charging the battery;

°detachable carrying harness.

The instrument is provided with an operation manual and reference sample for checking the calibration. It is calibrated by the manufacturer and must be returned to him for recalibration. The battery must be charged prior to operation.

6.2.3 Operation

In brief, lead analysis is achieved by placing the probe against the painted surface and depressing the shutter trigger (see Figure 11). At the end of the analysis period, an audible signal is sounded and the result in mg/cm^2 is displayed in the data window. The analysis takes 10 to 20 seconds. The manufacturer states that substrate effects are compensated for the instrument.

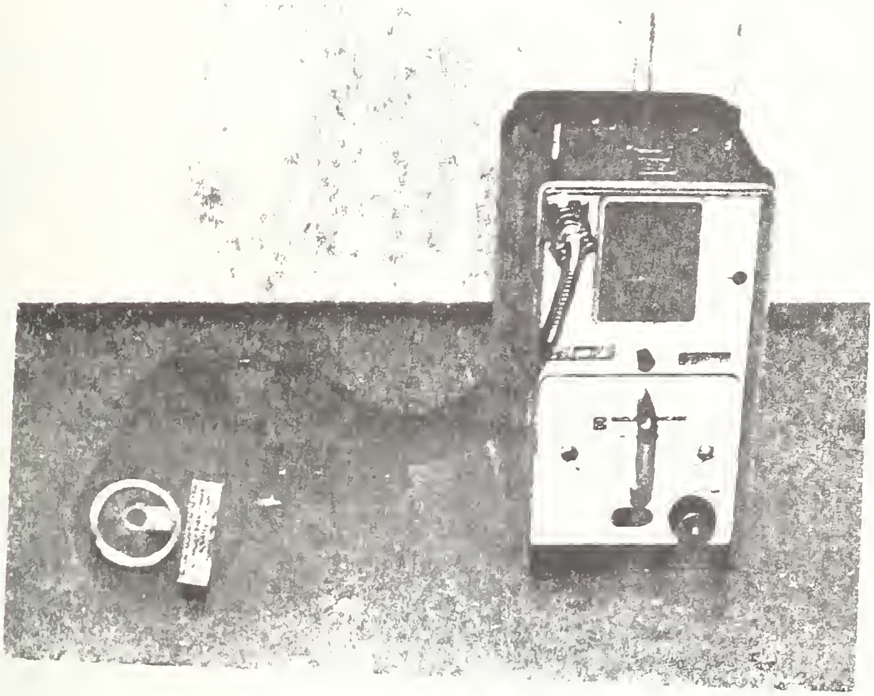

Figure 10 - Nuclear Chicago PB-3 Lead Analyzer Ready for Use

Figure 11 - NC, PB-3 in Operation

7. COMPARISON OF THE PGT XK-3 PROTOTYPES WITH THE COMMERCIAL LEAD ANALYZERS

7.1 PGT - XK2

7.1.2 Portability

The XK-3 which weighs a total of only 7.1 lbs (3.22 kg) (lead analyzer plus power pack) is easier to carry in the field than the PGT XK-2 which is very bulky and weighs 12.7 lbs (5.67 kg).

7.1.3 Calibration

The XK-3 is factory calibrated and, barring damage, needs recalibration only when the X-ray source is replaced (each 9-12 month period). By contrast the XK-2 needs recalibration each time it is used. This requires more time than the accuracy and precision check for the XK-3.

7.1.4 Speed of Analysis

The speed of analysis of the XK-3 prototypes at 35 to 40 seconds is slower than the XK-2 speed of 10 to 30 seconds. However, a new commercial version of the XK-3 has just been developed which has an analysis time of 10 to 15 seconds.

7.1.5 Evaluation

In order to perform a measurement, the XK-3 must be held against the test surface and the handle/trigger mechanism is pressed toward the machine to actuate the trigger mechanism. A force of about 23-1/2 lbs (10.7 kg) is required to depress the handle of the instrument and we have found this level of pressure to be quite fatiguing, especially when making a large number of replicate measurements. By contrast, the small gun type trigger of the XK-2 requires very little effort to operate. The XK-3 has a liquid crystal readout system which has permitted a significant weight reduction due to its much lower power requirement. However, the liquid crystal readout cannot be seen in low light level situations. This is a serious deficiency in field use since inspections are made quite frequently in unlit houses.

7.1.6 Accuracy and Precision

The XK-3 prototypes generally exhibited a better accuracy than the XK-2, especially for the range of 0 to 1.0 mg lead/cm^2. However, we have found that XK-2 instrument C141 has the best accuracy and precision of eleven of these instruments we have worked with. Therefore, we can state that if the XK-3's are properly calibrated, they should have better accuracy, consistently, than XK-2's.

The results in Table 13 show that the precision of the XK-3 prototypes is 2 to 3 times as good as that for the XK-2 in the 0.5 mg/cm^2 range (see Table 14).

The spreads for the XK-3's were obtained by determining the maximum difference from the mean for each of the 10 determinations done on each substrate in the series, and doubling them.

The data in Table 13 further substantiates the above finding that the XK-3's have better precision than the XK-2. The totals of measurements greater than ± 0.2 mg lead/cm^2 of the average determinations are all roughly half for each XK-3 than the total for the XK-2.

Figure 12 shows the XK-2 and XK-3 lead analyzers side by side to illustrate how much smaller the latter instrument is.

Table 13 Comparison of Precision between PGT XK-2 and XK-3 Prototypes at the 0.5 mg/cm^2 Lead Level

Instrument	Range from 0.5 for a single determination,* mg lead/cm^2	Spread mg/cm^2
XK-2, C141[2]	-0.5 to +1.5	2.0
XK-3 No. 101	0 to 1.0	1.0
XK-3 No. 102	0.2 to 0.8	0.6
XK-3 No. 103	0.1 to 0.9	0.8
	Average of XK-3 spreads	0.8

*The determinations selected were those farthest from the mean of 10 determinations.

7.2 NUCLEAR CHICAGO PB-2

7.2.1 Portability

The NC PB-3 weighs a total of 15 lbs (6.8 kg), is bulky and like the PGT XK-2 is much more difficult to carry in the field than the PGT XK-3.

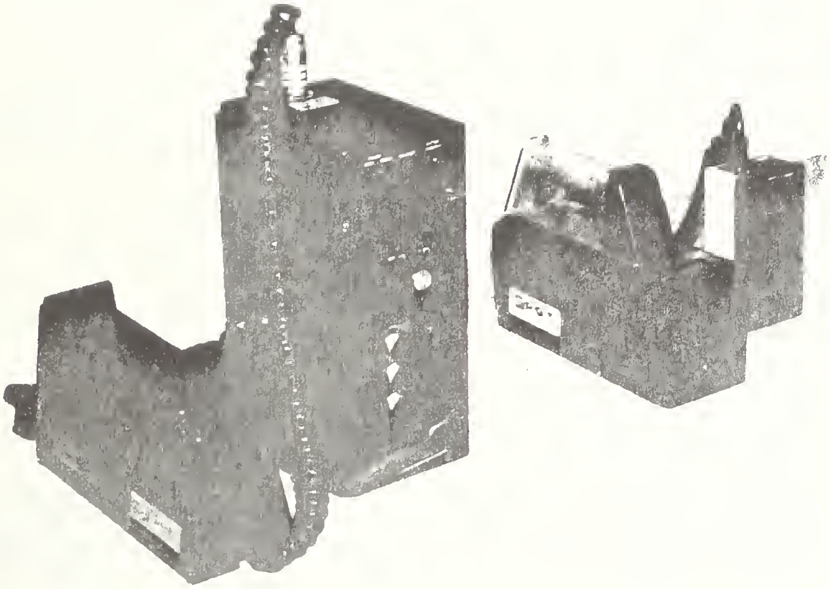

Figure 12 - PGT XK-2 and XK-3 Lead Analyzers
(Size comparison)

7.2.2 Calibration

Like the XK-3, the PB-3 is factory calibrated initially but must be recalibrated periodically by the operator with a reference sample of known lead content, and also one of zero lead content.

7.2.3 Speed of Analysis

At its normal speed of 8-10 seconds, it is faster in this respect than the XK-3.

7.2.4 Ease of Operation

It is easier to operate than the XK-2 because of its small gun trigger X-ray shutter opening system.

7.2.5 Accuracy and Precision

It is not as accurate and precise as either the XK-3 or XK-2.

8. COMMERCIAL VERSION PGT XK-3

The commercial version XK-3 is an improved version of the prototypes. Through the use of an improved preamplifier and other electronic modifications, time required for analysis in the commercial version was reduced to 10 to 15 seconds from 35 to 40 seconds for the prototypes.

Except for the above improvements, it was found that all other performance characteristics of the commercial XK-3 were the same as those of the prototypes.

Table 14 shows that the accuracy and precision of the commercial XK-3 are comparable with those of the best of the prototypes, No. 101.

Table 14 Comparison Between Lead Analyzers for Accuracy and Precision, at about 1 mg/cm^2 Lead

		PGT XK-3, No. 101			PGT, XK-3, No. 102		
	1	2	3	4	5	6	7
Substrate	Lead Content of Reference mg/cm^2 (a)	Observed Averages mg/cm^2 (b)	Difference (1) - (2)	No. of Measurements > ± 0.2 of (2)	Observed Averages mg/cm^2 (b)	Difference (1) - (5)	No. of Measurements > ± 0.2 of (5)
1/8 in Unsealed Wood	1.05	1.07	+0.02	6	0.84	-0.21	2
1/8 in Seal Wood	1.06	0.93	-0.13	5	0.58	-0.48	2
1/2 in Gypsum Board	0.70	0.75	+0.05	0	0.38	-0.32	0
0.014 in Mylar	0.82	1.17	+0.35	1	0.62	-0.20	5
0.035 in Steel	0.88	0.91	+0.03	3	0.55	-0.33	3
Total Measurements Exceeding ± 0.2 From Observed Average				15			12

Table 14 (Continued) Comparison Betweeen Lead Analzers for Accuracy and Precision, at about 1 mg/cm^2 Lead

PGT XK-3, No. 103			PGT XK-2, C141			NC PB-3		
8	9	10	11	12	13	14	15	16
Observed Averages mg/cm^2 (b)	Difference (1) - (8) mg/cm^2	No. of Measurements > ± 0.2 of (8)	Observed Averages mg/cm^2 (b)	Difference (1) - (11) mg/cm^2	No. of Measurements > ± 0.2 of (11)	Observed Averages mg/cm^2 (b)	Difference (1) - (14) mg/cm^2	No. of Measurements > ± 0.2 of (14)
1.00	-0.05	3	1.55	+0.50	5	0.52	-0.53	5
0.64	-0.41	1	1.42	+0.36	4	0.55	-0.51	6
0.85	+0.15	3	0.71	0.01	5	0.30	-0.40	10
1.01	+0.19	4	1.39	+0.57	6	0.45	-0.40	6
1.08	+0.20	3	0.84	-0.04	6	0.52	-0.36	8
		15			26			35

9. CONCLUSIONS

1. The CSI Model 750 lead analyzer is not suitable for field use because it is inaccurate and imprecise, too difficult for semi-skilled personnel to use and maintain, and is very bulky and heavy.

2. Although the XK-3 prototypes did not meet all the requirements of the HUD contract, H-2192R, the XK-3 is a much better field instrument than either the commercial lead analyzers, namely, the PGT XK-2 and the Nuclear Chicago PB-3 in these very important respects:

(a) Accuracy

(b) Precision

(c) Portability and operation

Although the XK-3 is a definite improvement over the commercial predecessors, it has several important deficiencies. The following is a discussion of them.

In order to perform a measurement, the XK-3 must be held against the test surface and the handle/trigger mechanism pressed toward the machine to actuate the trigger mechanism. A force of about 23-1/2 lbs (10.7 kg) is required to depress the handle of the instrument and we have found this level of hand applied pressure to be quite fatiguing especially when making a larger number of replicate measurements.

The XK-3 has a liquid crystal digital readout system which has permitted a significant weight reduction due to its lower power requirement. However, the liquid crystal readout cannot be seen in low light situations. This is a serious deficiency with regard to field operations since inspections are made quite frequently in unlit houses.

A further disadvantage relates to calibrations. It is, in our opinion, very unfortunate that the user of the instrument has no means for making simple calibration adjustments but must send the instrument back to the manufacturer for that purpose. In fact, the manufacturer recommends that the XK-3's be recalibrated about every 9 to 12 months, or as often as a new Colbalt 57 source is required.

A redesign of the trigger mechanism (which would allow for a lower pressure effort) would significantly enhance the usability of the instrument.

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11. REFERENCES

1. Rasberry, S.D., Investigation of Portable X-ray Fluorescence Analyzers for Determining Lead Painted Surfaces; Applied Spectroscopy, Vol 27, No 2, 1973.

2. Spurgeon, Joe C., "Response Characteristics of a Portable X-ray Fluorescence Lead Detector: Detector of Lead in Paint", National Bureau of Standards Report, NBSIR 73-231.

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15. SUPPLEMENTARY NOTES

16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

Portable X-Ray fluorescent lead analyzers offer the most cost-effective and adaptable means for the non-destructive detection and measurement of lead in paint in housing. However, commercially available portable lead analyzers have had poor accuracy and precision below lead levels of about 3.0 milligrams of lead per square centimeter of surface area. This is particularly serious because the current operational criteria for lead paint hazard abatement, 1.5 or 2.0 mg/cm of lead maximum (used in many communities) is in this range. They have also performed relatively unsatisfactorily with regard to serviceability and maintenance. Two new portable lead analyzers based on x-ray fluorescence have been developed under HUD contracts. The prototypes of one of these devices have shown considerable improvement over previously available instruments in terms of accuracy, portability, and user characteristics. This report discusses the performance and operating characteristics of the new lead analyzers.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

Accuracy; calibration; evaluation; lead; paint; poisoning; portable; precision; radiation; references; substrates; x-ray fluorescent.

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