NATIONAL BUREAU OF STANDARDS REPORT

10 623

PERFORMANCE ATTRIBUTES FOR ANALYTICAL METHODOLOGY FOR LEAD BASED PAINT

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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PERFORMANCE ATTRIBUTES FOR ANALYTICAL METHODOLOGY FOR LEAD BASED PAINT

Milestone Report (1b)

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ABSTRACT

Performance attributes have been identified for comparing and judging the acceptability of qualitative and quantitative methods for analyzing lead in paint and building materials at the 1% concentration level.

Analytical methods can be chosen on the basis of (1) scientific performance as characterized by precision, accuracy, and sensitivity; and (2) user attributes, such as cost, speed of analysis, sample pre-treatment, sample required, portability, skills required, safety considerations, and aesthetics.

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1. INTRODUCTION

1.1. Nature of the Problem

It is generally accepted that lead poisoning of children in certain susceptible age groups occurs when the children ingest lead-bearing paint, putty, or other non-food items in their environment. The National Bureau of Standards, under the sponsorship of the Department of Housing and Urban Development is concerned with determining the extent and nature of the lead paint poisoning hazard and with recommending methodologies for the elimination of this source of lead poisoning.

The localization of potentially hazardous environments may be made through an analyses of the circumstances that favor lead poisoning: (a) the presence of susceptible children who may actively seek and eat non-food items, (b) the presence of lead paint and the condition of the environment that makes it available to those children. The chemical detection of lead in paint and other building materials becomes the final step in the overall process of the lead paint hazard detection.

There are literally thousands of analytical procedures for lead, including methods for the analysis of lead in paint. Many of these techniques are used in quality control laboratories and provide reliable results. No information is presently available, however, on the relative merits of the various methods for lead analysis when applied to aged, crumbling, peeling, inhomogeneous dried paint films. Furthermore, the ordinary criteria for judging the acceptability of analytical methods, those of precision and accuracy, are not the only attributes that are applicable to the needs of this program. Not only are alternative chemical methods of analysis involved, but people carrying out the tests may be lay people of vastly differing skills, socio-economic backgrounds and education, rather than skilled scientists in well equipped laboratories. Municipalities that are faced with a pressing need for lead paint analyses often rely on advertising literature of instrument manufacturers or mount expensive evaluation programs of their own without an adequate knowledge of work that others have done. They quickly discover that analyses carried out by one city using one method may not agree with analyses done by other procedures and other people in the same or other cities. It is clear that assistance is required to establish guidelines for the evaluation and applicability of lead detection methods.

1.1.1. Definition of Problem

The definition of the problem is the first step to be taken before considering any choice of method. The analytical approaches which best suit the needs posed by the problem should be the methods of choice.

The requirements for an Analytical Methodology are:

1. Detection and measurement of lead in paint and building materials at the 1% concentration level.
2. Applicability to rapid screening of thousands of samples, with ± 50% precision.
3. Applicability to accurate analysis for the support of legal actions, with ± 10% precision.
4. Rejection of chemical interferences such as Titanium, Calcium, Barium, Antimony, and other metals commonly found in paints and building materials.
5. Ability to accomodate samples either as painted panels in situ, or paint chips mixed with wood, plaster, glass, wire, etc.
6. Operation by non-technical people (of mixed socio-economic backgrounds) after suitable training.
7. Costs within reach of municipalities having lead detection programs.

The first five parts of the problem can be solved by judging method performance in terms of precision, accuracy, sensitivity, and specificity in accordance with accepted scientific principles. The last two problem areas are special needs incurred by the nature of the lead paint poisoning problem and are discussed in more detail in a later section.

1.2. Task Objective

The objective of the overall Analytical Chemistry program is the identification, evaluation, and recommendation of methods for determining lead in paint and building materials at the one per cent level. The program is divided into three tasks. They are: Task (1) to review methods for lead and recommend the most promising procedures for experimental verification; Task (2) the experimental evaluation of laboratory and field methods for lead in paint; and Task (3) preparation of standard reference or research materials for calibrating the analytical methods.

Task (1) is further divided into three sub-tasks as follows: (1a) review the state-of-the-art of methods for the analysis of lead in paint; (1b) identify performance attributes for the evaluation and comparison of various analytical methods, and (1c) make a preliminary choice of methods(s) that warrant further experimental study based upon an evaluation in terms of the performance attributes. This report is a summary of the results of Task (1b). The results of Tasks (1a) and (1c) are reported in the respective Task reports.

1.3. Rationale for Performance Attributes

Criteria are standards on which judgments or decisions or tests of quality may be based. The qualification of criteria as "performance" simply indicates that the standards are independent of any particular alternative solution, and independent also of the particular persons making the decision.[a] Performance attributes can be used to evaluate alternatives and select a solution in the light of pre-established objectives.

The selection of performance attributes is itself governed by other performance criteria, relating to objectivity, reliability, validity, sensitivity, comparability, and utility, to assure that the selected tests of quality (1) are solution independent, (2) are usuable by the decision makers, and (3) provide a common standard for making choices from among very different alternatives.[a]

The rationale behind the choice of attributes for evaluating the acceptability of analytical methods represents a departure from usual practice. Ordinarily the most suitable methods are those which demonstrate high accuracy and precision under conditions which minimize experimental errors and operator bias. However, analyses for lead in paint are needed in such quantity that it is reasonable to expect building inspectors, health personnel, homeowners, and miscellaneous volunteers to be involved with the analyses, in addition to skilled chemists in laboratories. This is not to imply that the optimum methods may be those which give the best analyses in unskilled or semi-skilled hands, regardless of inherent accuracy and precision; but rather that socio-economic factors are involved in addition to purely scientific factors, and these should be considered.

When a municipality becomes concerned with lead analysis in paint and building materials its first attention is budgetary. Costs and personnel are budgeted, field testing is weighed against laboratory analysis, and the overall effort is developed on the basis of the best way to (1) screen homes for dangerous lead concentrations (1% or more in paint), and (2) analyze more precisely for 1% lead, to support legal actions that may result. The choice of method must be geared to the municipality's needs: some cities have active health departments and chemical instrumentation, and need purchase just one or two more instruments. Other cities have a large body of untrained labor and their needs could favor a portable instrument with admittedly narrower capability, but operable by volunteers.

Thus, the choice of method is governed, necessarily by the municipality or other user, rather than the scientist skilled in chemical analysis. User criteria can be drawn from requirements for product performance (appliances, automobiles, paints, etc.), considering the method of lead analysis to be a commodity that can be purchased by a representative of the end user.

An important consideration in the choice of analytical methodology is the method of paint sampling, and here again there are both the scientific and the consumer-oriented factors to consider. Scientific principles of sample selection must be tempered by considerations if homeowners or apartment residents who resent having holes gouged in walls and woodwork, for samples to take back to the laboratory. Here, at least, the ideal sample is none at all, and the best instrument would be one capable of reading lead "right on the walls".

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[a] David B. Hattis, "LPPP-Performance Criteria - Preliminary," August 4, 1971.

In summary, the choice of analytical methodology rests on a combination of scientific and socio-economic factors. These factors will be discussed in detail below.

2. PERFORMANCE ATTRIBUTES FOR ANALYTICAL METHODS

2.1. General

Qualitative and quantitative procedures for all chemical analysis are based on well established operations which are common to most methods. Generally, analytical procedures include (1) sampling; (2) sample preparation (ashing, digestion, dissolution); (3) isolation and purification of material of interest (rejection of interfering substances); (4) confirmation of identify (precipitation, complex formation); and (5) quantitation of the material sought (gravimetry, titrimetry, instrumental methods).

The analyst may have nothing to do with sampling, which may be carried on outside his laboratory. His responsibility may begin when he receives the sample which he reduces to a representative subsample by mixing, crushing or grinding, partitioning and subdividing until his sample is homogeneous. Functions (2) and (3) are important wet chemical operations which may or may not be a part of the analytical method. Functions (4) and (5), the detection and measuring methods, give the analytical methods their name: Atomic Absorption, for example.

The performance attributes listed in this section are divided into two groups: Scientific performance, which includes the usual evaluation criteria of method precision and accuracy, and User attributes which refers to the ability of non-scientific people to carry out the analyses.

2.2. Scientific Performance

2.2.1. Reliability: Precision and Accuracy

Testing methods are fundamental to all industrial experiments, not only those involving chemical analysis. Decisions on the reliability of alternative test methods are made daily. The usual practice is to consider the precision and accuracy of the test methods by procedures fully described in statistics textbooks[1/] and chemistry texts.[2,3/] Precision is defined as the variation among the test results themselves, and is a measure of the reproducibility of the measurements. Accuracy refers to the difference between the average test results and the true result when the latter is known or assumed.[4/]

The analytical methods must, first of all, give reproducible results; for if the analyses do not agree with each other, how can we expect them to check the true value? But the precision of any single analytical method can vary, depending upon whether the results are considered for a single observer in one laboratory on the same day, or several observers in one laboratory on the same day, or observers in different laboratories, and so on. The within-laboratory precision is pretty much the same for different laboratories because the systemic errors are comparable and affect the determinations in the same way. The results from several laboratories can differ widely because of the individual laboratory differences in location, temperatures, humidity, age of reagents, use of inferior instruments, or the way the analysts follow instructions. The ASTM Committee E-15 uses the term 'repeatability' to mean the precision of a method run by one analyst using the same apparatus in the same laboratory; and 'reproducibility' to mean the agreement attained by different laboratories in different cities.[5] It appears likely that interlaboratory tests may be required for a proper evaluation of method reproducibility.

The accuracy of the analytical methods may be ascertained by use of "knowns" or standard materials. Aliquots from the same uniform sample will reveal the performance of an instrument or procedure. The true value of lead in the standard material must be established experimentally, within very narrow limits, by a highly refined method that has an error appreciably less than any of the alternative methods being considered. Given such a standard, the errors in the less accurate methods can be evaluated.

2.2.2. Sensitivity

The sensitivity of analytical methods refers here to the limits of lead determination. No difficulty is expected with method sensitivities at the 1% lead level, except in cases where instrument sensitivities may have been reduced, in a trade-off for other parameters such as portability or ease of operation.

As the concentration of lead in the sample is reduced, the positive detection by any analytical method (signal) becomes proportionately less compared to the background noise. The lead concentration at which the signal is twice the noise level is ordinarily referred to as the limit of sensitivity. (This definition of sensitivity limit can be changed for different statistical applications). The analytical method can still be used, even at its limit, to detect lead qualitatively.

Near the detectability limit the precision too is low, and the test sensitivity may cause precision to be ± 50% or even as low as ± 100%. At the 1% lead level, readings of 0.5-1.5% lead (at ± 50% precision) can indicate the presence of lead qualitatively, subject to confirmation by an independent method. Readings of Zero to 2% lead (at ± 100% precision) are indistinguishable from Zero to some value % if no lead is present, so the lower sensitivity instrument or method is valueless.

Some workers have criticized the dependence on precision and accuracy only, and have devised other mathematical guidelines for judging the acceptability of analytical methods. McFarren, Lishka, and Parker[6] feel that these guidelines do not tell whether the results are sufficiently precise and accurate to satisfy user requirements. They propose a so-called Total Error, on the basis of which they calculate that Atomic Absorption is an unacceptable method for analyzing lead in natural water, even though this is a standard EPA method for analyzing water and wastes.[7]

In a similar vein, Mandel and Stiehler[8,9] have claimed that the two usual criteria for evaluating test methods, precision and accuracy are insufficient for a quantitative measure of the merit of test methods, and they have proposed their own concept of "Sensitivity" as a measure of analytical method performance. The relative merit of two methods suitable for determining lead would be given by the ratio of their sensitivities.

These statistical "asides" are mentioned to demonstrate some types of thinking that have gone into evaluation of test methods (1954-1970). Actually, not too much difficulty is anticipated in the management of the statistical analysis using the conventional criteria for evaluating method performance, namely precision and accuracy, as the protocol is well documented.

2.3. User Attributes

Considering the test methods now as consumer products instead of scientific experiments permits additional guidelines to be drawn up to further describe desirable performance characteristics. Basically, the user criteria for judging performance overlap the scientific criteria and encompass many more decision points. Real people, in real conditions ask not only, "How well does the method analyze for lead," but also, "For how long?" Further: "How much will it cost, now and later? Is it safe? Will it annoy the neighbors?" These basic criteria summarize the information that municipalities need, for deciding on their choice of analytical methodology. They have been used, also, in making a preliminary decision regarding which methods to evaluate further; this information is reported elsewhere.[10]

2.3.1. Costs

Performance is bound up with cost, considered as the total of the purchase price, operating cost, maintenance cost, and useful life. While there is keen price competition among manufacturers of analytical instruments, gross cost estimates for whole analytical disciplines (e.g. spectrochemical methods, electrochemical methods, wet chemistry, etc.) generally average the costs of individual instruments, and are independent of competitive interactions among manufacturers. These gross estimates give an indication of financial involvement.

Purchase price appears to be a straightforward cost; if the capital investment is too great, the availability of rental instrumentation might be considered. Similarly, useful life can be estimated either for the full instrument life (5-8 yrs.) or at two to three years, for in that time technological advances will have made the analytical instrumentation obsolete, and a reduction in costs would be achieved by the purchase of new instrumentation.

The operating costs and the maintenance costs include not only capital depreciation, but also the labor costs of the technical or other personnel for sample preparation, instrument operation, data reduction, and other necessary operations. They include also the time required to analyze a single sample, that is, the turn-around time per sample. The maintenance cost includes the down-time, that is the time the analysis is not in operation due to instrument malfunction, lack of samples, illness of personnel. The cost of expendable supplies often is the smallest part of the budget. These costs can be averaged into one overall cost to analyze a single sample.

At first glance it would appear that chemical spot tests offer the lowest cost-per-analysis of any other methodology: the instrument cost is zero, the turnaround time is short, maintenance costs are minimal, and unskilled labor may be used readily. However, detailed analysis may reveal that labor costs are high, since personal involvement is high at all steps of the analysis, from reagent preparation to actually going into dwellings to spot test the walls, doors, windows, and so on. Thus, the decision is again up to the user: does he have a cheap and plentiful but inexperienced labor supply? or is he short on personnel but have a reasonably budgeted health laboratory?

2.3.2. Speed of Analysis

Closely allied to costs and included in them is the speed of analysis or turn-around time for a sample. Depending upon the methods used, this can vary from 3 minutes to 8 hours or more. The lengthy analyses include acid digestions for solubilizing intractable paint samples, and manipulations with ion exchange columns and complexing agents to isolate lead from the other metals (e.g. titanium, calcium, barium, antimony, etc.).

The most rapid analytical methods are most suitable for screening, of course. These are the purely qualitative chemical spot tests and the semi-quantitative (almost qualitative) portable X-Ray Fluorescence instruments. They give a go/no-go indication, but no more. Further action, such as legal recourse, must be based on other tests which take longer to carry out.

The longest analyses are associated with extensive sample pre-treatment because of the need to isolate the lead as completely as possible, free of interfering metals. The actual lead determinations, whether by gravimetry, titrimetry, or instrumental techniques do not require much time. However, if enough samples are being run at one time, the effect of the long hours of wet acid digestions diminishes; for the samples can be made to come off the assembly line at the same rate that they are fed into it. The time that samples actually spend in the assembly line, up to eight hours or more, is of no consequence except during start-up. Laboratory organization is the key, here, as it is the user's choice that governs the importance of speed of analysis.

2.3.3. Sample Pre-Treatment

As pointed out in the previous section, the longest analyses are often associated with the most extensive sample pre-treatment, to assure that the quantitative determinations are as accurate as possible. The analyses requiring no sample pre-treatment, that is, spot tests and X-Ray Fluorescence, are most suitable for rapid screening tests. When lead is detected in a dwelling, these tests have to be backed up by more accurate determinations, which require that paint samples be gathered, brought to the laboratory, and ashed either wet by acid or dry in a furnace, to remove the lead from interfering organic materials.

To this date, no wet method or instrumental method other than the two mentioned above have been able to avoid sample pretreatment of one sort or another. The minimum treatment observed has been dissolution in nitric acid, a partial solvent which leaves a considerable quantity of lead behind.

There is promise that a new furnace being developed for Atomic Absorption Spectroscopy may be able to vaporize paint samples from the dry, solid state through application of a surge of electrical current (100 Amperes). Such a furnace would eliminate all sample pretreatment for this instrumental method of analysis, if it works.

2.3.4. Sample Required

This user attribute refers to the necessity of taking a paint sample away from the dwelling, rather than measuring the lead in the paint on the wall. Instruments that measure lead non-destructively may be preferred over more destructive methods that leave black stains, knife slashes, or gaping holes where paint samples were removed for laboratory testing.

2.3.5. Portability

There is a rough division between two types of analyses required: one for rapid screening, and one for accurate lead determinations that will support subsequent legal actions. Clearly, the rapid screening methods should have maximum portability, even at the expense of method precision and/or accuracy. The accurate methods do not require such portability; but it would be advantageous if one of the accurate instruments or procedures could be made portable without sacrifing other desirable characteristics. This is a research and development problem: given the extent of portability required (size, weight, electrical power requirements for hand portability, mobile van portability) it is merely a question of assurance that the optical alignment, or critical electrode spacings, or gamma ray shielding, or other parameters unique to the measuring systems under consideration do not change with vibration, temperature, humidity, operator fatigue, and other hazards encountered under field conditions. It is reasonable to expect that at least one of the purely laboratory methods of today may be designed into portable instruments some day, soon.

2.3.6. Skills Required

One of the key decisions that the municipalities will make with regard to their human resources, is the extent to which they will commit technically trained persons for lead paint analyses. The instrumental methods of analyses require technically trained people, but the techniques can be learned by high school graduates.

However, professional scientific backup should be close at hand for data interpretation or problem solving should the need arise.

Chemical spot tests are attractive because of their apparent lack of sophistication. Reagent formulation may have required considerable research to arrive at a suitable combination of complexing agents, buffers, color-formers and the like, but the application of the reagents to the walls is a simple operation. It is conceivable that an army of volunteer workers can screen many hundreds of dwellings in short order. Home-owners can use do-it-yourself kits to detect hazardous conditions in their own homes. The development of a colorimetric spot test is high on the list of priorities for lead-detection methods.

2.3.7. Safety

The evaluation of safety often is a subjective one and judgment considerations must be made. Safety must be considered from the point of view of hazard to the user, or to other individuals who may be exposed to the effect of the tests. Consider, for example, the case of X-Ray Fluorescence instruments which employ gamma radiation to excite the K-alpha or the L-alpha fluorescence of lead in the paint. Improper shielding or a malfunction in a shutter mechanism could conceivably expose individuals to atomic radiation.

A hazard of chemical spot tests might be the evolution of poisonous hydrogen sulfide gas, when reagents are washed off walls, after testing. Laboratory analyses employing wet acid digestions may be hazardous to technicians, but these hazards can be minimized by proper precautions and careful techniques.

There is an important problem in evaluating the degree of hazard: if a particular analytical method is found to produce a harmful physiological effect on two people out of 100 who use it, could a municipality assume the risk, even though the hazard is small from a statistical standpoint? This is another user judgment.

2.3.8. Aesthetics

In the design of consumer goods, aesthetic values have a positive connotation: beauty, comfort, convenience, ease. For the purpose of the lead paint application, aesthetics is more an Unpleasantness Index. Thus, analytical methods may have disagreeable side-effects that annoy people, especially residents of affected dwellings who object to noxious fumes, and black marks, gouges, or holes on walls, doors, and windows where paint was removed for testing.

Since the benefits of lead paint testing far outweigh the subjective aesthetic considerations of the affected residents, it is possible that aesthetics may be only a minor factor in any judgment of method acceptability.

2.4. Performance Verification[11/]

The useful life of the analytical method or instrumentation, and the modes in which they are liable to fail are additional, important attributes of performance. The user is interested not only in when the method fails, but how it fails. Ideally, the failure mode whould interrupt the analysis completely. Some instruments are able to withstand continuous usage for a long time, and then fall apart quickly. Others may deteriorate quickly in the first few months of use, yet do not reach a stage of obvious failure for a long time. Clearly, the first mode of failure is to be desired over the second: for, in the first case, accurate analyses are obtained until suddenly there is an obvious change in performance or complete instrument failure. In the second case, the analyses may be incorrect for a long time without any indication of poor performance.

Continuous performance verification is the only reliable protection against poor instrument performance. Standard reference paint samples must be made available to check the validity of the analytical results. These reference paints might simulate paint chips or scrapings from dwellings, yet be homogeneous, stable, and contain an accurately analyzed quantity of lead. They would be used for frequent checks of instrument calibration to guard against inaccurate results caused by instrument deterioration, operator error, reagent spoilage, and other accidents.

3. SUMMARY AND CONCLUSIONS

Performance attributes have been described for evaluating analytical methods for (1) rapidly screening, and (2) accurately quantitating lead in paint and building materials. These guidelines are classified as Method Performance which considers precision, accuracy, and reliability; and User Attributes which looks at the analyses in service, from the operational point of view.

The various analytical methods can be rated on an arbitrary scale of 1-10 for each of the performance attributes. In addition, the different attributes can be weighted to represent relative importance to the overall evaluation. The final numerical ratings can be used to classify the acceptability of the analytical methods subjectively, based on objective information. However, those who must implement lead paint programs must make the final decisions as to which methods best suit their needs.

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