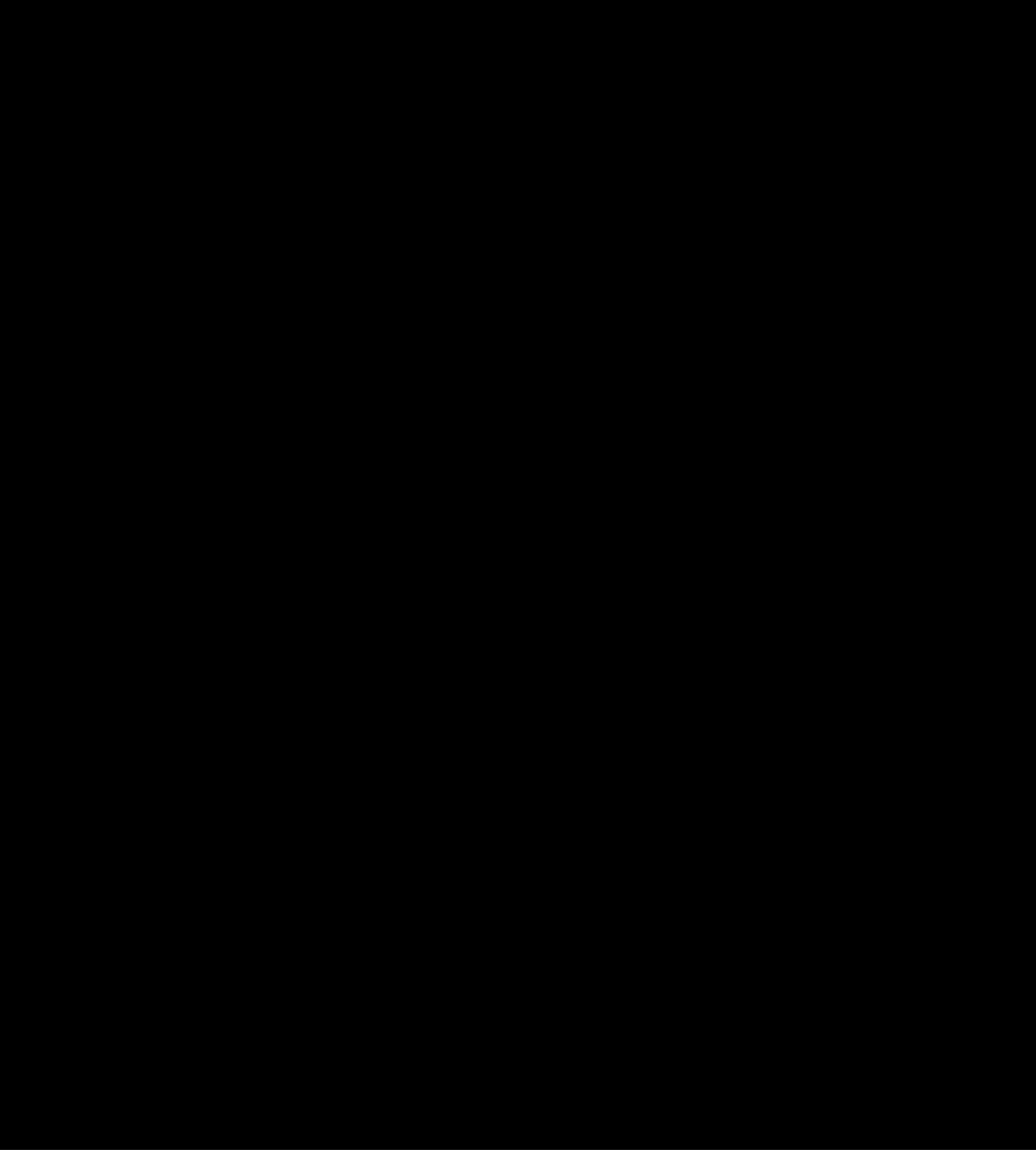




The Determination of Lead in Paint Films with a Portable Isotope Fluorescence Analyser

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Although there are numerous environmental sources of lead of significance for children (1) the major lead hazard in most urban populations is domestic paint (2). Conventional lead screening programs have previously been focussed on the child rather than his environment and so detect only those children who have accumulated a sufficient body burden of lead to render the tests positive. Although such programs have shown an annual poisoning rate of 0.5% among children at risk (3) they fail to identify those dwellings that are potential hazards for children not yet exposed. Chemical analysis of paint from surfaces accessible to children have shown that a wide range of lead contents may be present in homes of varying age and social status (4). Paint sampling in this way has disadvantages in that the subsequent analysis is relatively time consuming and costly and that the painted surface must be damaged so that a representative full-thickness sample may be obtained.

The portable isotope fluorescence analyser has long been used by geologists for the determination of the mineral content of ores but has not previously found application in this field. The apparatus is based on the principle of x-ray fluorescence and has the advantages that it is portable and that it can be applied repeatedly to any surface without damage. Preliminary studies have suggested that the device might find application in the determination of lead in domestic paint films in situ. In this paper the apparatus and its application to the analysis of paint films both in the laboratory and in the domestic environment are described.

Apparatus

The device employed was the Portable Isotope Fluorescence Analyser manufactured by Rank Precision Instruments Ltd., London, England. It comprises a portable battery-powered ratemeter which is conn-

ected by means of a flexible cable for application to the test surface. The weight of the assembly is 7.5 lb and contains a radioactive source (60Co) sealed in an aluminium container shielded when not in use by a lead shutter which is displaced on being applied to the test surface. The source is simultaneously operated. The instrument when not in use emits 1000 rad/h and thus presents no hazard. The radiations emanating from the source excite a fluorescent x-ray emission from the test material, are uranium or thorium, which penetrate to an extent depending on its composition, but seldom more than 1 cm. The fluorescent emission arising from the test material enters the probe unit through an aperture surrounding the source and is detected through one of a pair of beta-ray detectors and measured by means of a sodium iodide crystal and photomultiplier housed in the probe. The beta-ray detectors are made of gallium or germanium and are cut off emissions above and below the lead emission at 1.175 Å. The effective lead content of the test material is determined from the difference in count rates obtained when the filter is removed and when it is in use.

Standardisation

The observed difference in count rates for a given element will vary according to the thickness of the element in which the element is dispersed. A standard reading can be obtained with a known thickness of element of sufficient thickness to give a reading with lesser concentrations with a similar absorption of the excitation. Ideally a series of standards of similar composition would have to be used in the test situation so that a standard curve could be drawn in count rates against lead content. While this is impossible for a single element it is simply achieved for a compound by the addition of a lead compound to a sample from which uniform films can be prepared to give a standard series.

In this study a commercial undercoat domestic paint containing 1% lead was used as the standard.

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of the paint, increasing weights of lead oxide (Pb_3O_4) were added and thoroughly mixed such that a series of paint films of lead content in the range 1-31% were obtained. Subtraction of the reading due to the lead content of the base paint allowed the construction of a calibration curve for the range 0-30%, the maximum reading was obtained by substitution of lead foil for paint to give 100% lead (Fig. 1). The readings obtained allowed

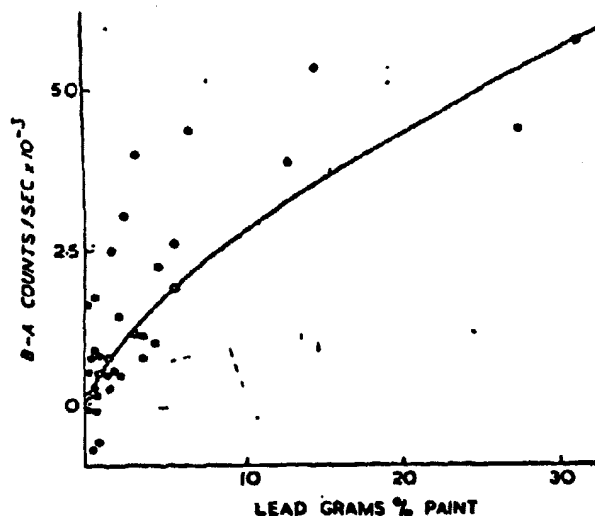


Figure 1. Count rate differences for paints of varying lead content a) laboratory prepared standards (open circles and curve) and b) domestic paint films in situ (closed circles).

determination of the lead content of a given film in the range 0-5% to an accuracy better than $\pm 0.2\%$. It was found that foils of $0.002''$ thickness gave readings that did not differ from those obtained from the surface of a lead brick, so that the effective thickness for the pure element was probably less than this. Standard paint films were obtained by pouring equal weights of the prepared paint stan-

dards (6.5 g.) into Petri dishes of uniform area (64 cm^2) supported on a top-loading balance. The paint was allowed to cover the whole of the surface such that a relatively thick film of depth was obtained. The standards were dried thoroughly and reweighed before use in order to determine the lead content per gram of dried paint film.

NON-HOMOGENEITY

Non-homogeneity of the test paint film may introduce difficulties in interpretation of the readings obtained and in some cases render it impossible to obtain a representative figure. In some situations this results from the successive application of films of different lead or matrix content to a given surface. In its simplest form this comprises a double layered film but in many field situations more complex films with multiple layers are encountered. A double layered film may produce anomalous results in two ways depending on whether the layer is superficial with regard to the analytical probe. A surface layer of high lead content will thus register a falsely high lead content for the whole film, conversely if the lead containing layer is deep, the overlying film of low lead content will impair the efficiency of detection. The usual situation in the field involves the second of these two situations in which the deepest layer of the film has the highest lead content.

In order to assess the effect of overlying layers a series of standard paint films was prepared in Petri dishes as described above to contain 31% lead. After drying each film was covered with successive films of increasing thickness of the base paint of low lead content (1.0% lead) such that each film weighed 8 mg/cm^2 when dry. Successive observations showed a progressive decrease in the apparent lead concentration until this approached zero with the covering paint film of 77 mg/cm^2 equivalent to a thinly brushed layer (Fig. 2).

FIELD TESTING

Thirty-four homes inhabited by families with children in the 1-5 age group were selected from a group of families previously studied (4). The selection was arranged such that there was a wide scatter of lead concentrations in the domestic environment to which the children had access. In each home a reading was obtained from a painted surface, usually a window sill or door frame, in a room usually occupied by the children, in order to determine this was a window sill or door frame. The

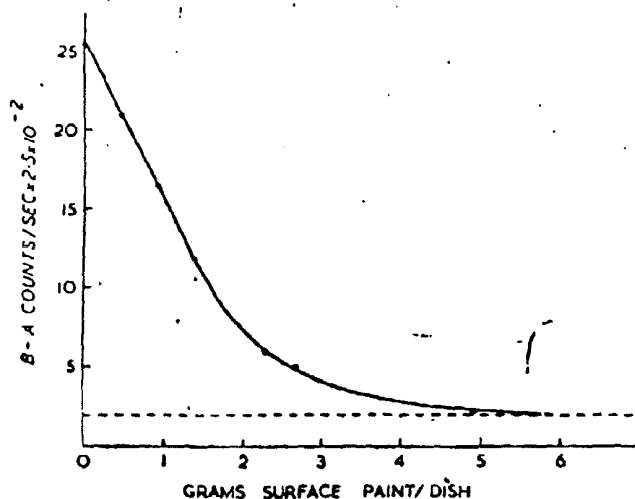


Figure 2. Graph showing the decrease in count rate difference for a paint containing 31 g% lead with successive applications of a second paint of low lead content (1.0 g%) in a dish of 64 cm². The interrupted line indicates the value obtained with the 1.0% lead containing paint alone.

fluorescence analyser was standardised in each home by means of a standard paint film previously prepared in the laboratory but in practice no appreciable change in machine performance occurred between home visits. Several readings were obtained from each surface to ensure that local variations in paint film thickness or lead content did not bias the results. After each series of readings a full thickness specimen of the paint film weighing about 50-100 mg. was obtained from the surface with the aid of a stainless steel scalpel blade. Chemical analysis of each paint specimen was made after wet digestion by means of a semiautomated alkaline dithizone method (6) and the results compared with the analyser readings.

FINDINGS

In general there was good correlation between the chemical and x-ray methods and the results are given in Figure 1. which also part of the standard curve derived in the Curve fitting of the data from the domestic together with statistical analysis was used by means of a least squares program with 1950E computer and the derived curve together with 95% confidence limits are reproduced (Figure 3). It is evident that the scatter of results was such that an apparent zero lead content could be obtained by the analyser in a paint film with an actual lead content of 5% and that conversely a count rate as low as 2.5×10^3 cps could be obtained with very high lead concentrations. Similarly the wide separation of the 95% confidence limits requires that a count rate of 3.0×10^3 cps before it could be inferred that degree of certainty that the lead content of the paint film exceeded 1%, conversely a count rate could equally indicate a lead content of 8%.

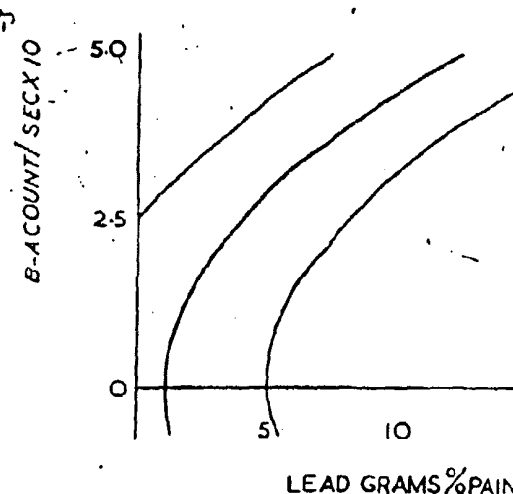


Figure 3. Best fit curve and 95% confidence limits for domestic paint film data with lead content in the range 0-15%.

In its present form it is unlikely that this type of apparatus could find application in lead screening programs since it is incapable of discriminating between lead contents of domestic paint films in the 0-6% range. These findings are in marked contrast to the laboratory results for a series of standard paints and almost certainly result from the non-homogeneity and the variable thickness of paint films encountered in the field.

SUMMARY

The application of x-ray fluorescence to the determination of the lead content of paint films was evaluated using a portable isotope fluorescence analyser. Accurate determinations were achieved in the laboratory using prepared films in which the lead was dispersed through matrices of constant composition in a single layer. The presence of lead could be masked by overlying paint films of low lead content and was almost obscured by an overlying paint film of 77 mg/cm². The results for domestic paint films correlated with chemical analyses but the confidence limits were too widely separated to allow application of this technique to lead screening programs. The field results probably reflect non-homogeneity of domestic paint films.

Acknowledgements

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