Measurement of Surface Contamination

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Abstract: Methods for measuring the contamination of surfaces with mine dust are described. A combination of methods involving x-ray fluorescence, visual inspection with a hand-held microscope, and wipe tests is proposed. An evaluation of these methods is given and the ways in which they can be applied to SNO activities described. Detailed results are presented in the companion report by E. Kong, SNO-STR-92-49.

Contents

I. Introduction

The success of SNO will depend in large measure on how low a level of background radioactivity can be attained. Radioactivity is either contained in the detector materials (e.g., the U and Th in the acrylic or in the heavy water) or is located on the surfaces of the detector components. In the case of surfaces the major contaminant is expected to be mine dust, which contains U and Th at levels of typically 3-10 parts per million (ppm). The cleanliness program[1-4] is designed to keep the level of mine dust below permissible values. Such a program depends, however, on our ability to measure mine dust at these low levels. We have investigated several measurement techniques for mine dust deposited on the kinds of surfaces making up the detector. We report our results here (and in more detail in the accompanying document) and indicate how these techniques can be applied as the need arises.

II. Requirements

We would like the measurement techniques to be:

1. sensitive,
2. quantitative (or semi-quantitative),
3. analytic,
4. fast,
5. simple, and
6. inexpensive.

Of the various permissible levels of contamination, the lowest level (0.05 $\mu g/cm^2$ of mine dust) is for the interior surface of the acrylic vessel (AV). The exterior surface of the AV and the interior surfaces of the PSUP should have dust levels below 0.4 $\mu g/cm^2$, while levels an order of magnitude higher than this can be tolerated on the exterior surfaces of the PSUP. The required level of sensitivity is therefore in the range 0.05 to 5 $\mu g/cm^2$.

A quantitative measurement (as opposed to a pass-fail threshold measurement) is useful because progress toward a goal can be monitored. Not all contamination may be in the form of mine dust; thus some analytical features are desirable. Speed is important in many cases because the outcome of the measurement may influence construction activities in progress. (We cannot afford to wait three days to find out if a container that has come through the car wash is clean enough.) A simple test is one that can be applied in the field, i.e., outside a laboratory, by personnel with little training in the use of the technique. Cost, both in terms of equipment and time spent in making the measurement, is clearly important.

III. Measurement Methods

There are many methods for measuring surface contamination that can be considered. None, of course, meets all of the above criteria, but a combination of several methods may be quite satisfactory.

We propose the following combination of methods.

1. X-ray Fluorescence. Mine dust (more specifically, norite) contains 6% Fe, which is conveniently detected by x-ray fluorescence (XRF).

2. Visual inspection and comparison with "calibrated samples." The latter are specimens of the surface in question (acrylic, glass, ABS plastic, etc.) with known amounts of dust on them. Comparison can be made with a fifty power, hand-held microscope.

3. Wipe tests. A clean white cloth wrapped around the edge of an eraser is wiped along a surface, collecting and concentrating the dust, which appears as a thin dark line on the white background of the cloth. Visual comparison can be made with calibrated samples.

A. X-ray Fluorescence

The XRF spectrometer[5] at LBL that has been used in all of our measurements can detect, via the Fe component, amounts of mine dust as small as 0.15 $\mu g/cm^2$ (three sigma level) in a counting time of twenty minutes. The dust sample is removed from the surface to be tested by application of a special adhesive tape, brought to the spectrometer, and counted "in air." The surface area sampled is about three cm^2. The technique is analytical in that all elements from Ca through Sr are observed at once in the spectrum taken by the solid-state x-ray detector.

Since it is U and Th that we are ultimately concerned with, the usefulness of this method depends on mine dust (all kinds encountered in the drift to the laboratory) having the same ratio of (U/Th) to Fe. This ratio, in units of Th(ppm)/Fe(%) is about 0.8 for norite. Of six samples of mine dust, collected at the 6800 foot level by H. Evans and analyzed by R. Giauque[6], five had ratios varying from 0.75 to 0.93 (±0.2), while a sample from the end of the chiller drift had a ratio of 3.9.

The advantages of this method are:

its high sensitivity and quantitative nature,

it measures mass/area directly (rather than numbers of particles),

it samples a relatively large area, thus minimizing the effects of non-uniform dust deposition,

the level of sensitivity can be improved by a factor of 3 to 5 by repeated application of the same piece of adhesive tape to cover a larger area of the surface,

the counting time can be reduced by increasing the detector efficiency and/or the x-ray tube intensity,

the apparatus is sufficiently compact that it can be located in the mine in the SNO laboratory control room,

the operator can do something else while the spectrometer does the counting, and

liquids and gases can be examined by passing them through filters and analyzing the contaminants removed by the filter.

The disadvantages of this technique are:

cost, unless we can use borrowed equipment and can discount the labor in building a spectrometer system,

the adhesive tape works only on relatively smooth surfaces, and

training in the use of a multichannel analyzer and in x-ray safety is necessary for operation of the system.

B. Visual Inspection

Visual inspection is always the first method to be applied in assessing surface contamination. Very low levels of dust can be seen with the unaided eye, for example, on glass surfaces viewed in intense, oblique light. By definition, the method is fast, simple, and inexpensive. It can be applied in the field. The sensitivity, however, depends very much on the type of the surface. And unless the person using the method has some experience, it is not very quantitative nor analytic. The method can be made more sensitive, quantitative, and analytic by two additions that do not compromise the other advantages: i. use of a portable fifty or sixty power monocular microscope, and ii. calibrated samples having different amounts of dust. By making a direct comparison with calibrated samples, a crude (factor of 2-3) assessment of the amount of dust can be made.

The level of sensitivity of this technique, applied to smooth surfaces (glass, acrylic, black ABS) and using the portable microscope, is about 0.5 to 1 $\mu g/cm^2$. Using this magnification, which lets particles as small as about 5 microns in diameter be observed, a degree of analyticity is obtained (e.g., grains of dust can be distinguished from fibers or plastics). For best results, the monocular microscope should have good optics, and such microscopes cost about \$150-\$300.

The main disadvantage is that it is not very quantitative.

C. Wipe Tests

Wipe tests are remarkably sensitive. A six-inch long wipe of a smooth surface having about 0.7 $\mu g/cm^2$ leaves a mark visible to the naked eye. We have made a small, spring-loaded jig to hold a cloth, wrapped around the edge of an eraser, at a fixed angle and pressure while being dragged along a surface. This improves the reproducibility of the method. The sensitivity can be increased by making longer or multiple wipes of a surface. With calibrated samples, as in B. above, the method becomes semi-quantitative.

The advantages of this method are its simplicity, speed, sensitivity, and negligible cost. The nature of the test can be grasped immediately by everyone, and can be applied (roughly) by anyone carrying a clean handkerchief. Its simplicity makes it ideal for a cleanliness specification in bids and contracts. It is useful for go, no-go decisions for material entering the laboratory.

A disadvantage is that it is not applicable to rough surfaces.

IV. Results

The results of this study are given in detail in the companion report, "Contamination Control Study on Mine Dust," SNO-STR-92-49. Tables and figures referred to here are found in that report.

Our procedure has been to prepare surfaces (Figs. 2-4) with different amounts of mine dust and to study different methods of measuring this dust. Since the methods we use to characterize the dust on the surfaces are also the methods under evaluation, we look for reproducibility, consistency, and reliability as a part of the evaluation. XRF spectroscopy (Figs. 6, 11), because of the advantages mentioned above, is the most useful *laboratory* method for determining the mass/cm^2 of mine dust on a surface to be studied by another technique.

A. Evaluation of Optical Counting

Optical sizing and counting of particles has been a standard technique in clean room applications for many years, and we have used a binocular microscope with x200 and x100 magnification to characterize the dust on a surface in terms of the number of particles versus their size (Figs. 7-8, 13). The microscope was used extensively throughout our work for general inspection of surfaces and contaminants as well as for quantitative scans (Tables 1-4).

One of our results of our evaluation of optical particle counting as a method was the absence of a good correlation between mass/area and the total number of particles/area, for particles with sizes of 1 micron and larger (Fig. 21) The correlation improved if only those particles having sizes greater than 25 microns were considered (Fig. 22). This is not surprizing since, for the typical number-size distributions we encounter, most of the mass is in the larger particles.

In most clean room applications, e.g., in the electronics industry, it is those particles larger than a few tenths of a micron that do the damage, practically independent of their size or composition. Therefore, the number of particles/area is the relevant quantity and particle counting (laser based or optical, automated or manual) is a basic measurement method. For SNO, the mass/area (the mass of U/Th) is the villain and the number of particles is practically irrelevant. As our study progressed it became clear that, if we want an *accurate* measurement of the mass of mine dust present, it is better to use a technique that measures mass directly rather than some other quantity.

A serious problem with optical scanning is that it typically covers only a relatively small area of the sample. Initially, scans of a few mm^2 were made (Table 1). Later this area was increased to 29 mm^2. The non-uniformities of dust deposition on acrylic and ABS plastic are on the scale of these areas and larger. Thus, being able to cover 300 mm^2 (and in only 20 minutes) is a significant advantage of the XRF technique.

B. The Tape Lift

Adhesive tape is a convenient and practical way to bring the the dust from a surface to the measuring instrument, whether it be an XRF spectrometer or a microscope. We have measured the efficiency of the tape for removing dust and find that it is typically 95% or greater for smooth surfaces like glass, ABS plastic, acrylic, etc.

For XRF measurements of Fe, the tape should have a low content of Fe, since Fe in the tape is a background that must be subtracted, and this limits the sensitivity. After an extensive search of commercially available tapes, we located one type with a Fe content of 29 ng/cm^2.

Use of an adhesive tape can present a problem if the surface contains Fe and the tape pulls up a small portion of the surface as it is lifted. This happens with pickled stainless steel. Typically, a few hundred ng/cm^2 of Fe (and corresponding amounts of Ni and Cr) are removed by the tape, and this limits the usefulness of this technique for steel surfaces. Finally, the tape must come in contact with all of the surface over a circle about 1 inch in diameter. This restricts the use of the tape lift to smooth surfaces of this size or larger.

C. Main Results

The primary results of this study are:

1. XRF provides accurate measurements of mine dust at levels of sensitivity required for SNO.

2. Calibrated samples, consisting of known amounts of dust on different surfaces, have been produced and can be used to make semi-quantitative visual determinations of dust levels on smooth surfaces, using a fifty power, hand-held microscope. This technique is suitable for use in the field.

3. A wipe test can be used to verify that a smooth surface is clean at the level of about 0.5 $\mu g/cm^2$. The test can be made semi-quantitative by comparing with calibrated samples, which we have prepared.

D. Applications

These different methods can be expected to find application in different circumstances.

The XRF spectrometer may be used for measurements on the interior of the AV[4], in conjunction with bonding the AV panels, to monitor the deposition of dust[3] on witness plates set around the laboratory, and to provide checks, monitoring, and calibration of the visual methods and wipe tests.

The visual tests and calibration samples will be useful in cleanliness training - they give an excellent feel for the level of cleanliness required. They can be applied in the field, e.g., where components are being manufactured or assembled above ground[2]. A set of calibrated samples of dust on glass is now being used at Queens by the PMT group. Samples of dust on black ABS plastic are available to the PSUP group at LBL, and the AV group will receive samples of dust on acrylic.

Wipe tests will likely find wide application because of their simplicity and sensitivity. This test will be useful during the establishment of clean conditions before the start of clean construction[1], and contractors should use it frequently in determining the cleanliness of equipment and material they bring into the laboratory during the clean phase of construction.

V. Conclusions

Adequate methods for measuring mine dust on smooth surfaces at levels required for SNO are in hand and available for application.

An XRF spectrometer should be installed in the SNO laboratory and be available for routine analytical work during the clean phase of construction.

A binocular optical microscope (x200, x100, x60) should be available in the laboratory for general purpose use.

The measurement techniques developed here need to be assimilated by groups that will need to monitor the cleanliness of their operations and components above ground as well as underground.

Specific measurement techniques for non-flat or rough surfaces (painted shotcrete) are still being developed but likely will make use of one or more of the methods described above.

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