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Visual Estimation in the Analysis of Surface Particulate by Microscopy

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KEYWORDS

Visual estimation, particulate percentages, areal estimates, ASTM Method D6602-03be1, *The Particle Atlas*, polarized light microscopy (PLM)

ABSTRACT

Visual estimation is an important instrument in determining the relative amounts of different classes of particles present in surface samples. The visual estimates are semi-quantitative determinations that are used with polarized light microscopy (PLM), and occasionally electron microscopy, for particle characterization. A number of representative comparison charts showing areas covered by different particle densities have been published and are used by analysts to calibrate their visual estimates. A test of trained analysts, who calibrated their visual estimates with representative comparison charts and inter-analyst sample studies, showed a relative standard error for the procedure of approximately 30%. This value compared well with the inter-laboratory testing of laboratories using three semi-quantitative visual estimate procedures on asbestos bulk samples.

INTRODUCTION

The semi-quantitative visual estimate of percentages of particles in a sample is a well-known technique that has been used for many years by geologists (1-4), paleontologists (5) and asbestos analysts (6). Standard methods for asbestos analysis of bulk samples describe the use of visual estimates for determining the percent of asbestos in building products (7-9). Visual estimation is used with PLM particle identification to estimate the amount of each type of component found in indoor dust or surface particulate samples (10). The use of the technique for outdoor dust samples is described in *The Particle Atlas* (11), published by McCrone Research Institute in 1972.

The "ASTM Method D6602-03be1 Standard Practice for Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both" includes the semi-quantitative determination in the ancillary method section 8.3 (12). Specifically section 8.3.3.3 in the examination by PLM section states: "Estimate the percentage of each type of component found from the list in Table 3 and record." Table 3 lists several classes of particles such as pollen, rubber dust, mold and particulate carbon. Information about the microscope analysis techniques involved in classifying various types of components found in outdoor surface samples (13) and indoor dust particle samples (10) has been published previously.

METHODOLOGY

The approximate percentages of different particle classes may be given in terms of major (>10%), minor (1% to 10%) or trace (less than 1%) designations or the classes may be estimated in terms of ranges of percentages. In some situations the middle of the range may be given as an approximate percentage with the

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Table 1. Comparison of Microscopical Visual Estimate Analysis Results*

Particle Class/Type	General Estimate	Percent Ranges	Range Mid-point
Biofilm (fungal/algal)	Major	20-40%	~30%
Soil minerals	Major	40-60%	~50%
Soot (char and aciniform)	Minor	1-5%	~3%
Plant fragments	Major	5-15%	~10%
Paint	Trace	Trace (<1%)	Trace
Rust/Metal flakes	Minor	1-5%	~3%
Insect parts	Minor	1-5%	~3%
Coal/Coke	Trace	Trace	Trace
Rubber	Trace-Minor	Trace-2%	~1%

*Analysis using American Society for Testing and Materials (ASTM) Method D6602-03be1: "Standard Practice for Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both" using stereomicroscopy, polarized light microscopy and transmission electron microscopy with energy dispersive spectroscopy X-ray analysis to confirm aciniform soot.

understanding that this represents a semi-quantitative value for the midpoint of a range. Table 1 shows a comparison of different presentations of the results of a calibrated semi-quantitative visual estimate of particle classes in a surface particulate sample.

Analysts learn to perform calibrated visual estimates by studying comparison charts where a known percentage of the particles in the chart has been filled in with dots or other dark figures. Figures 1-5 show representative comparison charts for calibrating analysts in semi-quantitative visual estimation. Samples of known composition made from known weights of various components can also used to "calibrate" an analyst. However, weight-based calibration samples are more important with asbestos analysis where percent by weight may be of interest than in surface samples where the percentage of surface covered with a darkening agent is often the critical question. Therefore, the comparison charts are the basic reference for estimating the percentage of each type of component found in surface particulate samples. Inter-analyst comparison is also an important part of the calibration process. Analysts trade samples back and forth to develop a consistency among the analyst group in performing calibrated visual estimates.

A relative standard error of 30% was found in an inter-laboratory study of semi-quantitative visual estimates of surface darkening agents (in the author's laboratory). When the estimated ranges were not exactly the same between analysts, they were close. One analyst might report 10-20% while another might report 20-30% for a component. The finding of error rate for surface dust darkening agents was consistent with the error rates for inter-laboratory tests of asbestos analysis. The error rates for semi-quantitative visual estimates for three standard methods of asbestos analysis were reported to be 32.2%, 25.7% and 26.9% (13).

DISCUSSION

Semi-quantitative visual estimation is an important tool for analysts characterizing the different types of particles present in surface samples. The approximate values of relative percentages determined by trained microscopists who have calibrated their analysis abilities using comparison charts are important in understanding the nature of particulates that cover indoor and outdoor surfaces.

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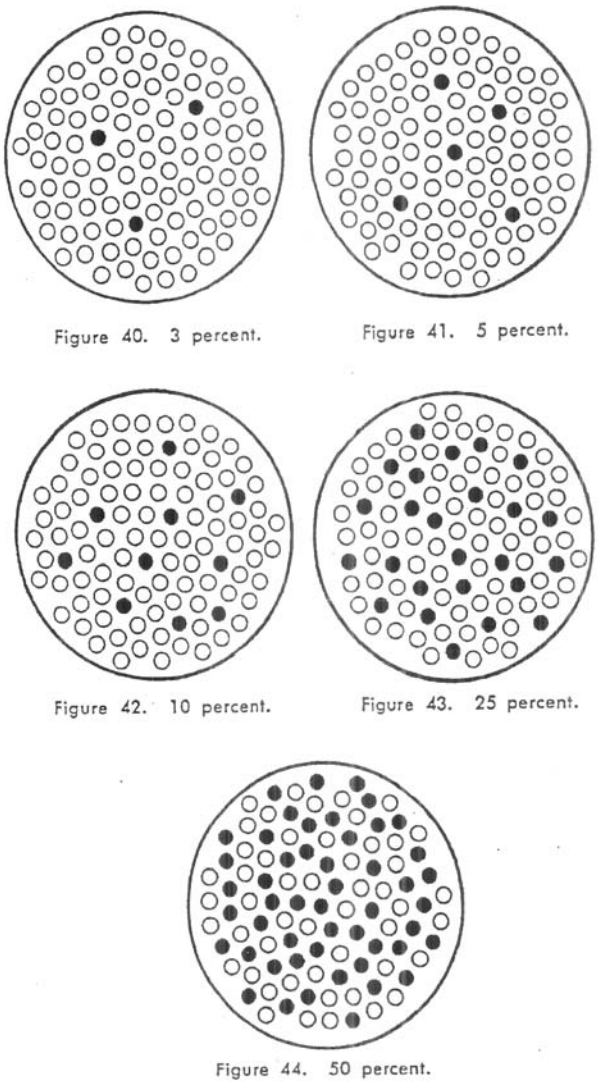

Figure 1. Visual estimate comparison chart from McCrone (6).

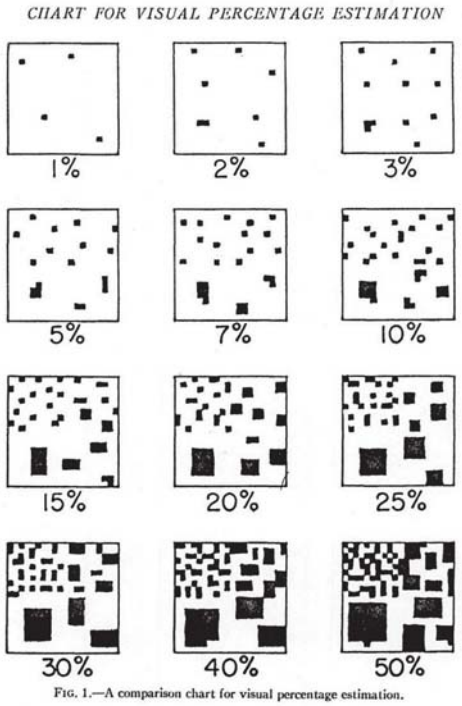

Figure 2. Visual estimate comparison chart from Folk (1).

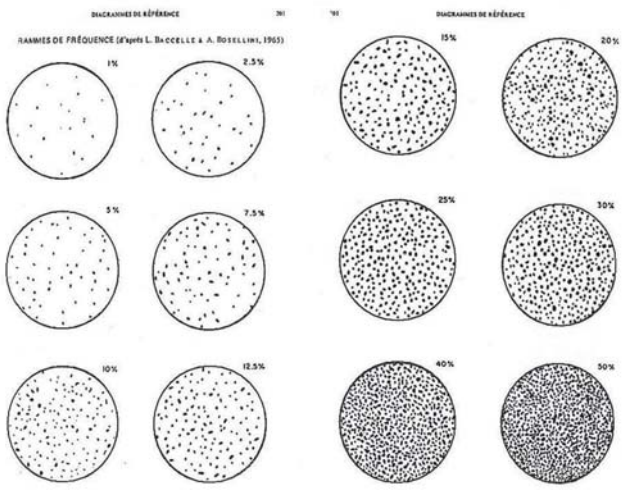

Figure 3. Visual estimate comparison chart from Baccelle and Bosellini (5).

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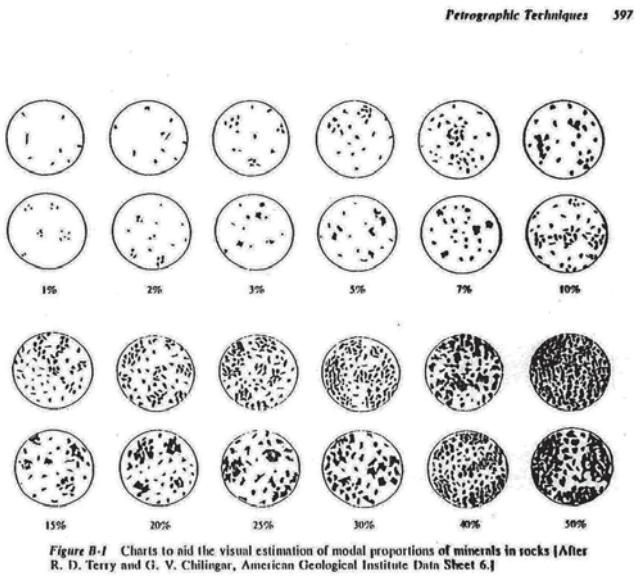

Figure 4. Visual estimate comparison chart from Terry and Chilingar (3).

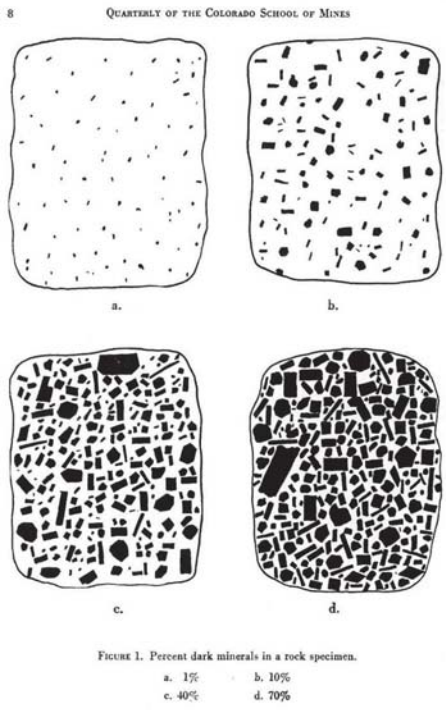

Figure 5. Visual estimate comparison chart from Travis (2).

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