SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

APPLIED SCIENCE & TECHNOLOGY DIVISION

LABORATORY SERVICES BRANCH

SCAQMD METHOD 301-91

IDENTIFICATION OF PARTICLES BY MICROSCOPY

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This method applies to the identification of particles in samples as regulated by Rule 402.

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IDENTIFICATION OF PARTICLES BY MICROSCOPY

1.0 Principle

1.1 This method is intended to analyze a wide variety of particles. All techniques to determine the optical properties cannot be documented in this method. Instead, several reference sources are suggested in this method that document the properties of common particles observed in ambient air particulate samples and explain in detail the techniques necessary to determine the optical properties. For this reason, only analysts who have satisfied the criteria in Section 6.1 should conduct this method of analysis.

1.2 This method is for the qualitative identification of particles. The particles are analyzed under a low power stereo binocular microscope (SBM) and a polarized light microscope (PLM) with transmitted and/or reflected light. Identification is made by comparison of morphology and optical properties with published properties in reference sources and/or comparison with reference samples or standards.

2.0 Equipment

2.1 Hood equipped with HEPA filter (optional).

2.2 Low power stereo binocular microscope (SBM), 10X - 30X magnification.

2.3 Polarizing light microscope (PLM) with 360^{o} rotating stage equipped with transmitted and reflected light capabilities.

2.4 Dispersion staining objective, central stop, 10X.

2.5 Compensator plate, 530 nm retardation.

2.6 Objective lenses, 10X, 20X and 40X.

2.7 Forceps and dissecting needles.

2.8 Microscope slides, standard, and coverslips type no. 1.

2.9 Petri dishes.

2.10 Reference particle standards, bulk or slides, of known particles which can be purchased through microscope and accessory supply companies.

2.11 Razor blades

2.12 Droppers

2.13 Alcohol lamp

3.0 Reagents

3.1 Refractive index liquids, 1.400 - 1.700, in increments of 0.004.

3.2 Solvents, reagents and acids needed to test samples (reagent grade or as specified in reference).

4.0 Analytical Procedure

4.1 Reference Samples (Reference samples are typically submitted as bulk materials or as liquid paints.)

4.1.1 If reference samples from the suspected source are submitted, transfer a homogeneous portion to a glass slide. Reference samples must be dry before starting analysis. If necessary, expose material to ambient conditions to dry. If a reference paint sample from the suspected source is submitted, spray it on to a slide and allow to dry under ambient conditions before analysis.

4.1.2 Examine the reference sample using the SBM. If a higher magnification is necessary, scan the reference sample using the PLM at the necessary magnification and lighting conditions. Record the morphological properties such as shape, color, texture, size, size distribution, inclusions and transparency of the particles of interest.

4.1.3 If additional information is necessary for identification, the determination of isotropism/aniostropism and refractive index/indices (> or < 1.66) can be made by examining the particles in 1.66 Refractive Index liquid.

4.1.4 Solubility properties can be observed by mounting the particles in refractive index liquid, water, acetone or other solvents.

4.1.5 Document all properties observed and compare with the published properties listed in *The Particle Atlas*, W.C. McCrone and J. G. Delly and/or compare with standard reference materials.

4.2 Unknown samples (Unknown samples are typically collected on glass plates, as scrapings sampled from surfaces or as bulk samples.)

4.2.1 Unknown samples must be dry before starting analysis. If necessary, expose material to ambient conditions to dry.

4.2.1 If the unknown sample is collected in a jar, transfer a homogeneous portion to a petri dish or slide for examination. Examine glass plates "as is". Make a preliminary scan of the unknown samples using the SBM. If higher magnification is necessary, scan the sample using the PLM at the necessary magnification and lighting conditions. Record morphological properties such as shape, color, texture, size, size distribution, inclusions and transparency of any observed particles of interest.

4.2.2 If additional information is necessary for identification or for comparison with reference samples and/or reference standards, the particles of interest in the unknown sample can be separated and removed with forceps/needles and mounted on a slide for further analysis. A representative portion of unknown sample can be removed or scraped from the glass plate with razor blades and mounted on a slide for further analysis.

4.2.3 The determination of isotropism/aniostropism and refractive index/indices (> or < 1.66) can be made by examining the particles in 1.66 Refractive Index liquid.

4.2.4 Solubility properties can be observed by mounting the particles in refractive index liquid, water, acetone or other solvents.

4.2.5 Document all properties of the particles of interest observed and compare with the published properties listed in *The Particle Atlas*, W. C. McCrone and J. G. Delly or compare with standard reference materials or reference samples analyzed in Section 4.1.2. Record conclusions.

4.3 If the identification of the particles of interest in the unknown sample cannot be confirmed or a match with reference/standard reference samples cannot be determined, the samples may be analyzed by one or more of the following additional analytical techniques which will not be addressed in detail in this method.

4.3.1 Microchemical tests on the particles of interest can be conducted as described in Volume I and II of *The Handbook of Chemical Microscopy*, E. M.Chamot and C.W. Mason

4.3.2 Fourier-Transform Infrared (FTIR) spectroscopy can be used on powder coatings and paint particles for chemical comparison or identity confirmation.

4.3.3 X-ray Diffraction

4.3.4 Scanning Electron Microscopy

4.3.5 Electron Microprobe Analysis

5.0 Documentation

5.1 In the analyst's notebook, document sample source, lab number, date sample received, date analysis started, date analysis completed, type of analysis requested and sample description.

5.2 Document all properties determined on the particles of interest and reference sources used as described in section 4.0. Record any conclusions which can be made and supported by published reference sources from the analysis conducted .

6.0 Quality Assurance/Quality Control

6.1 Analysts using this method must have demonstrated training and proficiency in fiber identification by satisfying at least one of the following criteria.

6.1.1 Successful completion of two courses in the identification of particles and fibers by polarized light microscopy.

6.1.2 In-house training and one year supervised experience in the identification of particles.

6.2 A second analyst must review the results and sign and date the notebook.

6.3 The laboratory must have available and within access of the analyst, reference manuals and reference materials necessary for identification.

6.4 The samples must be stored in a locked room or a locked cabinet.

7.0 References

1) McCrone,W.C., Delly, J.G., *The Particle Atlas, Edition II.*

2) Chamot, E.M. and Mason, C.W., Volume I of *The Handbook of Chemical Microscopy*

3) Chamot, E.M. and Mason C.W., Volume II of *The Handbook of Chemical Microscopy*

4) *SCAQMD Quality Assurance Manual for the Analysis of Asbestos in Bulk Materials by Polarized Light Microscopy*

5) McCrone,W.C., McCrone,L.B., Delly, J.G., *Polarized Light Microscopy*