SCAQMD METHOD 311-91

**ANALYSIS OF PERCENT METAL IN METALLIC COATINGS
BY SPECTROGRAPHIC METHOD**

1. **Principle**

    A mixture of "wet ashed" (with sulfuric acid) samples and a portion of spectrographically pure graphite is excited by a controlled D.C. arc at 13 amperes and 300 volts (short circuit). Light from the sample excitation, attenuated to give an exposure with suitable background, is integrated on a photographic film. The concentration of metals in sample are determined from the densities of the spectrum lines produced by the samples compared to known standards.

2. **Equipment**

    2.1 Applied Research Labs. 1 1/2 meter grating spectrograph; dispersion 6.9 angstroms per mm; resolution .35 angstroms with a 50 micron primary slit.

    2.2 Applied Research Labs. "Multisource" unit used as a source of controlled D.C. arc current for sample excitation at the spectrograph.

    2.3 Applied Research Labs. film developing machine with controlled time, temperature and agitation.

    2.4 Applied Research Labs densitometer with Sola voltage regulator.

    2.5 Thermolyne electric furnaces with temperature controllers.

    2.6 Analytical balance, capable of weighing accurately to 10 mg.

    2.7 Ainsworth microanalytical balance.

    2.8 Miscellaneous equipment and fixtures for handling samples, ashing and loading electrodes.

3. **Reagents**

    3.1 Sulfuric acid, concentrated, spectrographically pure.

    3.2 Graphite, Spectrographically pure.

3.3   Photographic film, Eastman spectrum #3.

3.4   Solutions, film developing .

3.5   Standard (a mixture of elements).

4.   **Analytical Procedure**

4.1   Preparation of Sample Spectra

   4.1.1   Weigh 10 to 20 grams of sample on an analytical balance. Sample size must be large enough to yield at least 3 milligrams of ash.

   4.1.2   Sample is "Wet ashed" with sulfuric acid in an electrical furnace at 500°C until all carbon is removed.

   4.1.3   Crucible and ash is reweighed and the loss on ignition is calculated.

   4.1.4   A 3 milligram portion of the ash of each sample is weighed on a microbalance and mixed with a 10 milligram portion of spectrographically pure graphite.

   4.1.5   Quantitatively transfer the mixture to the crater of a perforated graphite electrode.

   4.1.6   The sample electrode and a matching counter electrode are placed with clips on the arc-spark stand of the spectrograph. The electrodes are spaced 9 millimeters apart with center of the gap at the focal point of the instrument.

   4.1.7   Sample is excited and completely consumed by a controlled D.C. arc at 13 amperes and 300 volts (short circuit).

   4.1.8   Light from the sample excitation, attenuated to give an exposure with suitable background, is integrated on Eastman spectrum #3 photographic film.

4.1.9 Film strip is processed in a developing machine under controlled conditions with solutions prepared and stored in such a manner that reproducible processing obtained.

4.1.10 Densities of the spectrum lines produced by each sample are determined by reading the light transmission of the lines on a densitometer. The transmission of the line and the background adjacent to the lines are read in one sweep of the pickup head of the densitometer.

4.2 Determination of Calibration/Sensitivity Factor

4.2.1 Determine the light response of each emulsion lot (background) by using identical conditions as the sample.

4.2.2 Determine the sensitivity factor of each metal by using standards containing all the metals in the samples. The concentration ranges of the standards must cover the concentration of the sample run under the same controlled conditions.

5. Calculation

5.1 Use the calibration curve of the emulsion to correct for background. A line-to-background ratio is calculated.

5.2 The concentration of each element in the ash is determined by multiplying these ratios by the sensitivity factors which have been determined for each spectrum line used.

5.3 The results obtained at this point are on the basis of the ashed samples. Previously determined loss on ignition figures are used to convert results to the "as received" or original samples basis.

5.4   Calculate gram metals per liter of coating by the following formulas:

$$\text{Vol \% of coating in paint} = 100 \; - \; \frac{(W)(Dp)}{Dw} \; - \; \frac{(E)(Dp)}{De}$$

$$\text{Metal, g/L} = (Dp \times 10) \sum_{i=1} M_i$$
(of paint)

$$\text{Metal, g/L} = (\text{g metal/L paint}) \quad \times \quad \frac{100 \text{ L paint}}{\text{Vol \% Coating}}$$
(of coating)

5.5   Definition of variables

W = Amount of water in paint, weight percent
E = Amount of exempt compounds in paint, weight percent
M_i = Amount of each metal component in paint, weight percent
Dp = Density of paint, g/mL
Dw = Density of water, g/mL
De = Density of exempt compound, g/mL

6.   **Reference**

6.1   Spectrographic Method for the Analysis of Carbon Dust and Carbon Laminates, Used by Pacific Spectrochemical Laboratory, Inc.

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The Method for the "Analysis of Percent Metal in Metallic Coatings by Spectrographic Method" is applicable in the analysis of coating samples relating to Regulation XI.

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SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

APPLIED SCIENCE & TECHNOLOGY DIVISION

LABORATORY SERVICES BRANCH

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