

SEMI G29-0519

TEST METHOD FOR TRACE CONTAMINANTS IN MOLDING COMPOUNDS

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1 Purpose

1.1 This Specification defines the test method for determination of extractable trace contaminants in molding compound.

2 Scope

2.1 This Test Method is suitable for all molding compound materials and may be used by supplier and customers to determine the trace contaminants in molding compound.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards and Documents

3.1 *ASTM Standards*¹

ASTM D1193 — Specification for Reagent Water

ASTM D4327 — Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Procedure

4.1 Samples of molding compound materials may be obtained from either standard molding operations, or prepared in the laboratory.

4.1.1 Samples from molding operations may include mold runners post-cured according to manufacturer's recommendations.

4.1.2 To secure a sample in the laboratory, spread a thin layer of uncured compound in a dedicated clean Teflon-coated container. Cure and post-cure the compound material according to manufacturer's recommendations.

4.2 Crush the cured material using a suitable grinding apparatus, such as Spex mixer-mill No. 8000, or equivalent.

NOTE 1: The grinder used must not generate excess localized heat, or else sample decomposition and erroneous results may be generated.

NOTE 2: Addition of liquid nitrogen is a suitable method for eliminating undesirable thermal decomposition during grinding.

4.3 Remove the ground compound and sieve it. Collect for analysis the portion which passes through a 40 mesh size, but is retained on a 100 mesh size screen. This particle size is best suited for adequate extraction of impurities.

4.4 Weigh 10 ± 0.1 grams of the powdered material and place in an extraction vessel. Parr bombs with Teflon liners are suitable extraction vessels. Add 100 mL deionized water. Prepare blank extraction vessel by processing it in the same manner as your sample. Weigh the sealed bombs and record their weights. Place the sealed bottles on their sides in an oven at $120 \pm 2^\circ\text{C}$ for 48 hours.

NOTE 3: Certain compounds may require addition of reagent grade methanol 10% (V:V) to enhance wetting.

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4.5 At the completion of 48 hours of extraction, allow the bombs to cool to room temperature and reweigh. If the weight loss exceeds 0.5 grams the sample should not be used for analysis. Portion of this extract may be used to generate pH and conductivity data. Anion analysis may be performed using ion chromatography or specific ion electrodes. Cation analysis may be conducted using atomic absorption, plasma spectrometry, or ion chromatography.

NOTE 4: It is important to avoid contamination with particulate matter in the extract used for the analysis by AA, ICAP, or IC. This procedure addresses determination of extractive species only.

5 Calculations

5.1 Ion chromatography is a form of liquid chromatography used in the separation and quantitation of ions. A filtered aliquot of sample is injected into an Ion chromatograph. The sample is pumped by the eluent stream through two different ion exchange columns: a guard column, which serves to protect the separator column from residual particulate matter and retain certain organics, and the separator column, the primary function of which is to separate analyzed ions based on their affinity for the exchange sites of the resin. Both guard column and the separator column are packed with identical low capacity anion exchanger (anion analysis) or cation exchange (cation analysis). The separated ionic species then pass to a detector module consisting of a chemical suppressor device and a conductivity cell. The suppressor device is used to reduce background conductivity of the eluent to a low, or negligible level, and convert analyte anions into their acid form or analyte cations into their hydroxide form. Thus, separated and modified ionic species are detected using an electrical conductivity cell. Anions are identified based on their retention time compared to the known standards. Quantitation is accomplished by measuring the peak height or area, and comparing it to a calibration curve generated from known standards.

NOTE 5: For recommended practice for ion chromatography analyses, see instrument manufacturer's literature and ASTM D4327.

5.2 *Method Principle* — Inductivity coupled argon plasma (ICP) uses high frequency argon plasma to excite sample constituents to 8000°K. Because the plasma ionizes most atomic species, background interferences are vastly reduced and linear response over several orders of magnitude can be observed for most elements. The sample extract is aspirated into the plasma by means of a high purity argon carrier gas. The resulting emissions are directed into the spectrometer and signal strengths are read by photomultiplier tubes placed at emission points in a focal curve. A computer is used to scan each elemental channel many times a second, and this output is sent to a printer in numerical form. The cycle or time of analysis is usually about 7 seconds. With this instrument, very little of the extracted sample is consumed.

6 Report

6.1 *Measurement of Conductivity*

6.1.1 *Apparatus* — Conductivity Meter Model RC1682 with microconductivity cell, industrial instruments, or equivalent.

6.1.2 *Measurement* — Measure conductivity of the sample and blank solutions. Calculate the specific conductance of sample using the following equation:

$$L_s = (L_e - L_b) K$$

where:

L_s = Specific conductance, $S\ cm^{-1}$

L_e = Conductance of extract, S

L_b = Conductance of the blank

K = Conductivity cell constant

6.2 *Measurement of pH*

6.2.1 *Apparatus*

6.2.1.1 pH meter (Orion, Model 601, or equivalent)

6.2.1.2 pH standard solutions