



U.S. CONSUMER PRODUCT SAFETY COMMISSION
4330 EAST WEST HIGHWAY
BETHESDA, MD 20814

Rohit Khanna
Fire Program Area Team Leader
Office of Hazard Identification and Reduction

Tel: (301) 987-2508
Fax: (301) 869-0292
Email: rkhanna@cpsc.gov

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Mr. Dick C. Engbrecht
738 South Patton Circle
Arlington Heights, IL 60005
Phone: 847-392-1542
E-mail: engbrecht@comcast.net

Subject: Proposal to ASTM C11 - WK33245 New Test Methods for the Analysis of Gypsum and Gypsum Panel Products for Impurities

Dear Mr. Engbrecht:

By this letter, the U.S. Consumer Product Safety Commission (CPSC) staff¹ is proposing new test methods for incorporation into ASTM C1396 / C1396M – 11, *Standard Specification for Gypsum Board*, or appropriate standard. The test methods would identify drywall fabricated with base materials that can result in corrosive gas emissions under certain environmental conditions when installed in buildings. As part of the CPSC problem drywall investigation, staff sponsored studies² and issued guidance³ to identify source markers that serve as reliable indicators of problem drywall. Testing revealed the following:

- 1) Elemental sulfur (S₈) was highly correlated with problem drywall;
- 2) A strong association was found between the presence of problem drywall and metal corrosion in homes; and
- 3) S₈ concentrations in drywall were associated with gaseous emissions of corrosive reduced sulfur compounds measured in chambers.

The studies also included the development of a reliable test method that could be performed by drywall manufacturers to certify that their product is free of those markers below the threshold

¹ This letter was prepared by the CPSC staff; it has not been reviewed or approved by and may not necessarily reflect the views of the Commission.

² <http://www.cpsc.gov/library/foia/foia10/os/EHESourceMarkers.pdf>.

³ <http://www.cpsc.gov/info/drywall/IDguidance031811.pdf>.

limit. CPSC staff believes that a test method to detect the elements that have been identified as reliable markers for problem boards should be adopted in the standard.

From these findings, we propose a test method based on extracting the sulfur from a drywall sample using a toluene solvent to the ASTM standard. Using a Gas Chromatograph with either a Mass Spectrometer or an Electron Capture Detector, as developed by ALS Environmental (formerly Columbia Analytical), the solvent is evaluated to identify the concentration of elemental sulfur of the species, S₈. When tested in accordance with the method detailed in Attachment 1, the measured quantity of S₈ of any gypsum drywall panel should not exceed **5 mg/kg** for any one of the three samples tested. Please consider the Attachment 1 test method and the 5 mg/kg limit for S₈ for incorporation into ASTM C1396 / C1396M – 11, *Standard Specification for Gypsum Board*, or appropriate standard.

If you have any questions or comments with regard to this request, please contact me. We look forward to working with ASTM C.11 on these proposed revisions to the ASTM drywall standards.

Sincerely,

Rohit Khanna

Attachment 1

cc: Mr. Colin Church, CPSC
Mr. Tom O'Toole, ASTM International

ATTACHMENT 1

1. Significance and Use

1.1 This test method covers the determination of sulfur (S_8) in drywall by a liquid extraction/gas chromatography method.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. Apparatus

2.1 Gas chromatograph (GC) equipped with *either* an electron capture detector (ECD) or a mass spectrometer (MS).

2.1.1 Software must be capable of integrating peak areas.

3. Reagents and Materials

3.1 Carrier gas Helium, chromatographic grade.

3.2 Toluene, spectral quality or chromatographic grade.

3.3 4,4'-dibromobiphenyl, to be used as an internal standard (ISTD).

3.4 Hexabromobenzene, to be used as surrogate spike standard.

3.5 Sulfur (S_8), to be used as a standard.

4. Sample Preparation

4.1.1 Three subsamples of drywall should be available for testing. Remove backing paper and grind a piece of each subsample with a mortar and pestle to a powder.

4.1.2 Collect 1.0 g of each ground subsample and transfer to a separate, sealable 10 mL vial.

4.2.1 Spike each sample with 50 μ L of surrogate spike standard (2.5 mg/mL hexabromobenzene in xylene).

4.2.2 Add 5 mL of toluene to each vial and shake vigorously (or otherwise agitate) sample for a minimum of 2 minutes.

4.3 For each subsample, transfer a 1.0 mL aliquot of toluene solution to a GC vial. Add 20 μ L of internal standard (5 μ g/mL 4,4'-dibromobiphenyl) to each vial.

4.4.1 Prepare at least 5 calibration standards of S_8 in toluene in the range of 1 to 200 μ g/mL.

4.4.2 Transfer 1 mL of each standard into a GC vial, and add 20 μ L of ISTD.

5. Preparation for Gas Chromatography with Mass Spectrometry

Instrument should be tuned daily using decafluorotriphenylphosphine (DFTPP).

5.1 Column: Fused silica capillary column, 30 m x 0.32 mm; 0.5 μ m film thickness

5.2 Carrier Gas: Helium

5.3 Injection Port: 250°C; Splitless

5.4 Temperature Program: 120°C for 1 min; 9°C/min to 285°C; hold for 10 min

5.5 Mass Spectrometer: Full scan (30-600 m/z range)

5.6 Sample Injection Amount: 1.0 μ L

ATTACHMENT 1

6. Preparation for Gas Chromatography with an Electron Capture Detector

6.1 Column: Fused silica capillary column, 30 m x 0.32 mm; 0.5 μ m film thickness

6.2 Carrier Gas: Helium

6.3 Injection Port: 250°C; Splitless

6.4 Temperature Program: 120°C for 1 min; 9°C/min to 285°; hold for 10 min

6.5 Sample Injection Amount: 1.0 μ L

7. Identification

7.1 S₈ is identified by mass spectrum (64 m/z will be the most abundant ion). Confirm by retention time matching to a calibration standard. The internal standard will yield ions at 152 and 312 m/z . The surrogate's most abundant ions will be found at 552 m/z .

7.2 If electron capture is used, identification can only be done by retention time matching to a standard.

8. Calculation

8.1 Integrate the S₈ and internal standard peak areas for each calibration standard. Calculate the peak area ratio by dividing the S₈ peak area by the internal standard peak area. Build a calibration curve based on the peak area ratios for each standard.

8.2 Calculate the peak area ratio for each subsample. Use the calibration curve to calculate the S₈ concentration in solution.

8.3 Calculate the concentration of S₈ in the original sample in mg/kg using the sample weight, final extract volume, and concentration value obtained in the prior step.

8.4 Calculate the percent recovery of the surrogate compound to evaluate the extraction efficiency.