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**IDENTIFICATION OF PROBLEMATIC DRYWALL:  
SOURCE MARKERS AND DETECTION METHODS**

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## **4.0 SOURCE MARKER ANALYSIS—ORTHORHOMBIC SULFUR**

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### **4.1 INTRODUCTION**

Similar to the investigation of strontium as a source marker, there were two specific aims in evaluating orthorhombic sulfur as a marker of problematic drywall. The first aim was to evaluate the precision and accuracy of orthorhombic sulfur measurements. The second aim was to evaluate the sensitivity and specificity of orthorhombic sulfur as a marker of problematic drywall. This section focuses on the first aim—testing done to compare laboratory methods and evaluate the precision of orthorhombic sulfur measurements. The analysis of orthorhombic sulfur as a sensitive and specific marker of problematic drywall through comparison to gas emissions and corrosion rates is described in Section 6.

Elemental sulfur exists as three allotropes with the most stable and common being S<sub>8</sub>. S<sub>8</sub> has been proposed as a marker of suspect drywall by the EPA and other governmental and non-governmental groups.

EH&E conducted an evaluation of S<sub>8</sub> that included the following objectives:

#### *Objective 1*

Compare three analytical methods (EPA Environmental Response Team [ERT]/REAC SOP 1805, GC/MS [toluene-extraction] and GC/ECD) for analyzing orthorhombic sulfur in a subset of drywall samples from the CPSC 'catalog' drywall samples in this study

#### *Objective 2*

Assess intra-board variability to determine representativeness of using a single 'core' sample to characterize larger pieces of drywall

#### *Objective 3*

Determine orthorhombic sulfur concentrations in catalog drywall samples and archived drywall samples from the 51-home study

# DRAFT

## 4.2 METHODS

### 4.2.1 Study Design

#### 4.2.1.1 Laboratory Method Comparison—Catalog Samples

To evaluate agreement among the three analytic methods, a subset (n=13) of the catalog samples were analyzed using each of the three methods discussed previously (Table 4.1). A 15g sample was removed from each board, homogenized, and divided into three equivalent subsamples to ensure sample uniformity across methods.

<b>Table 4.1</b> Overview of Analytic Methods Used to Determine Orthorhombic Sulfur Concentrations		
<b>Analytic Method</b>	<b>Description</b>	<b>Number of Primary Samples</b>
EPA ERT/REAC SOP 1805	<ul style="list-style-type: none"><li>• Soxhlet extraction (dichloromethane:acetone)</li><li>• GC/MS</li></ul>	13
GC/MS (toluene extraction)	<ul style="list-style-type: none"><li>• Solvent extraction (toluene)</li><li>• GC/MS</li></ul>	13
GC/ECD	<ul style="list-style-type: none"><li>• Solvent extraction (toluene)</li><li>• GC/ECD</li></ul>	35
EPA	U.S. Environmental Protection Agency	
ERT/REAC	Environmental Response Team Response Engineering and Analytical Contract	
SOP	standard operating procedure	
GC/MS	gas chromatography mass spectrometry	
GC/ECD	gas chromatography electron capture detector	

Thirteen drywall samples were selected from the catalog samples for analysis by EPA's ERT/REAC SOP 1805 ('REAC SOP 1805') and a similar laboratory method that also uses GC/MS but with a toluene based extraction ('GC/MS (toluene extraction)'). While both methods rely on quantification of S<sub>8</sub> using GC/MS, the methods differ in the solvent extraction method and chromatographic column used to separate constituents of the sample. A complete description of the laboratory methods can be found in the Section 4.2.2.

In addition to the thirteen samples analyzed by both EPA REAC SOP 1805 and the GC/MS (toluene extraction) method, all of the catalog samples (n = 35) were analyzed for S<sub>8</sub> using a third method—GC/ECD. This method has been proposed as equivalent to

## DRAFT

the two methods that use GC/MS for determining  $S_8$  concentrations (Singhvi et al. 2009). Duplicate samples were prepared from six of the catalog boards and analyzed by GC/ECD to evaluate method precision.

### 4.2.1.2 Intra-Board Variability

Intra-board variability of  $S_8$  was assessed by analyzing multiple samples (n=3 to 12) from eight separate catalog samples (Table 4.2). The boards were marked in evenly distributed sections and samples were taken from the mid-point of each location. Before sending the samples for  $S_8$  analysis, the samples were measured for strontium concentration by XRF.

<b>Table 4.2</b> Description of Samples Used for Intra-board Variability Tests		
<b>Catalog ID</b>	<b>Original Board Size</b>	<b>Number of Samples</b>
CPSC 3	1' x 1'	12
CPSC 10	9" x 6"	3
CPSC 14	9" x 6"	12
CPSC 15	1' x 1'	3
CPSC 25	9.5" x 16"	3
CPSC 32	1' x 1'	12
CPSC 34	1' x 1'	5
CPSC 35	1' x 1'	5

CPSC U.S. Consumer Product Safety Commission

### 4.2.1.3 Archived In-Home Samples

In addition to the catalog samples, archived samples from EH&E's 51-home study were analyzed for  $S_8$  concentrations by GC/ECD. During the 51-home study, samples were cored from the wallboard around electrical outlets in most rooms. These samples were typically <0.5 grams per core sample. Additionally, a larger piece (~1'x1') of drywall was obtained from each home and archived. The cored samples, not originally collected for this purpose, did not provide sufficient mass to provide detection limits in the 5 – 10 mg/kg range and therefore be comparable to the sensitivity anticipated for categorizing problematic drywall. Therefore, cored samples from the same room were composited to provide sufficient mass for analysis and provide a limit of detection (LOD) in the 5 mg/kg range. For these core composite samples, an equal mass from each of

## DRAFT

the individual core samples was combined to evenly represent the different wall boards sampled from a room. For the large wallboard piece, a 2 – 5 gram sample was removed and sent for analysis. The result was that each home had up to three total samples analyzed for S<sub>8</sub> – two composite samples (one from each of two rooms), and one non-composite sample (n=153).

### 4.2.2 Laboratory Methods

Orthorhombic sulfur analysis was conducted by Columbia Analytical Services (Simi, California). The laboratory was provided an EH&E sample ID but were blinded to the origin of all samples. Three methods were used: EPA REAC SOP 1805, GC/MS (toluene extraction) and GC/ECD. Drywall samples were crushed by EH&E, homogenized, and divided into three equal subsamples to ensure uniformity. A negative control sample (known unaffected drywall) was prepared by Columbia Analytical Services and analyzed along with the various subsamples as an added check for quality control. A summary of each method was provided by Columbia Analytical Services and each is presented in the following sections.

#### 4.2.2.1 EPA REAC SOP 1805

Subsamples were extracted using EPA Method SW846 3541, “Automated Soxhlet Extraction” (Soxtherm). Samples were aliquoted, mixed with sodium sulfate to remove moisture, and surrogates (2-fluorophenol; phenol-d6; nitrobenzene-d5; 2-fluorobiphenyl; 2,4,6-tribromophenol; and terphenyl-d14) were added to evaluate extraction efficiency. Samples were then extracted per the method using a 4:1 mixture of dichloromethane:acetone. The samples were concentrated to a final volume of 1 mL and solvent exchanged into dichloromethane on an N-EVAP evaporator unit under nitrogen.

All samples contained sediment and therefore were filtered using a 0.45 micrometer (µm) polytetrafluoroethylene (PTFE) syringe tip filter. The extracted samples were colored, which ranged from light yellow to dark brown. The darkest extract was analyzed at a 10 fold dilution.

A four point calibration was performed for the surrogate compounds. An EPA Method SW846 8270 internal standard mixture was added to an aliquot of the extracts. Sample

## DRAFT

extracts were analyzed on a GC/MS utilizing a DB-5 column (30m x 0.25mm x 0.25 $\mu$ m film thickness). Helium was used as the carrier gas in the analytical system. S<sub>8</sub> allotropes were identified based on the spectral match, comparing the mass spectra of the sample peak with mass spectra in a comprehensive mass spectral library. The concentration of S<sub>8</sub> was estimated using a response factor of one and the response of the associated internal standard (phenanthrene-d10). The results are reported as S<sub>8</sub> in mg/kg.

### 4.2.2.2 GC/MS (Toluene Extraction)

One gram (1g) aliquots of each subsample was solvent extracted with agitation for two minutes using toluene (5 mL). A 1.0 microliter ( $\mu$ L) aliquot of the sample extract is injected into the gas chromatograph by splitless injection where a fused silica capillary column separates S<sub>8</sub> from other species in the sample and a mass selective detector operated in the SCAN mode detected the S<sub>8</sub>. Helium was used as the carrier gas in the analytical system. The retention time and select characteristic ions of S<sub>8</sub> were used for identification. Quantitative analysis was performed by using an internal standard calibration procedure, which involves the comparison of instrument responses from the target compounds in the sample to the response of the internal standard that is added to the sample prior to analysis. The ratio of the peak area of the target compound in the sample to the peak area of the internal standard in the sample was compared to a similar ratio derived for each calibration standard.

Additional instrument quality control checks included daily tuning of the mass spectrometer using decafluorotriphenylphosphine (DFTPP), a five point calibration for S<sub>8</sub>, initial calibration verification standard analysis, and evaluation of extraction surrogate recovery.

The concentration of S<sub>8</sub> in the sample was reported in mg/kg, and was calculated using the analytical result, the sample weight and the final extract volume.

### 4.2.2.3 GC/ECD

One gram (1g) aliquots of the subsamples were solvent extracted with agitation for two minutes using toluene (5 mL). A 1.0  $\mu$ L aliquot of the sample extract was injected into

## DRAFT

the gas chromatograph by splitless injection where a fused silica capillary column separates  $S_8$  from other species and an ECD detects the  $S_8$ . Helium was used as the carrier gas in the analytical system. The identification of  $S_8$  was performed by comparing the retention time of  $S_8$  with the respective retention time of an authentic standard. Quantitative analysis was performed by using an internal standard calibration procedure, which involves the comparison of instrument responses from the target compounds in the sample to the response of the internal standard that is added to the sample prior to analysis. The ratio of the peak area of the target compound in the sample to the peak area of the internal standard in the sample was compared to a similar ratio derived for each calibration standard.

Additional instrument quality control checks included a five point calibration for  $S_8$ , initial calibration verification standard analysis, and evaluation of extraction surrogate recovery. The concentration of  $S_8$  in the sample was reported in mg/kg, and was calculated using the analytical result, the sample weight and the final extract volume.

### 4.3 RESULTS

#### 4.3.1 Objective 1—Analytic Method Comparison

Results of the  $S_8$  measurements of the catalog samples by three different methods are presented in Table 4.3.

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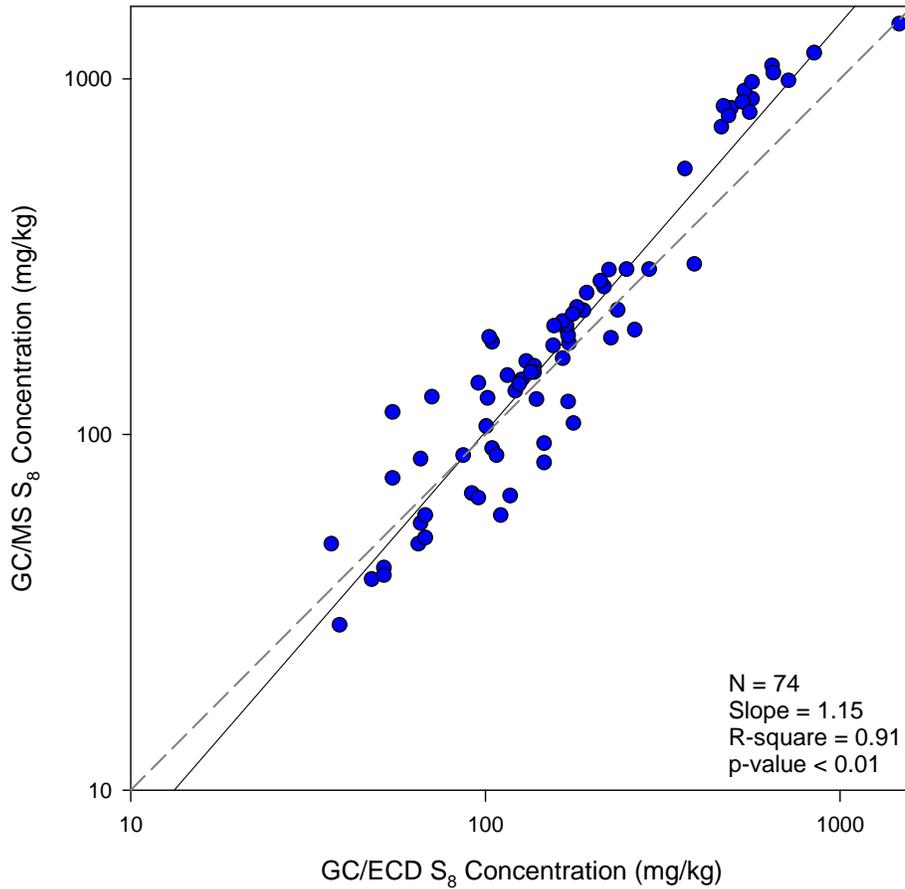
<b>Table 4.3</b> Comparison of Orthorhombic Sulfur Concentrations (mg/kg) Measured Using Three Different Methods			
<b>Catalog ID</b>	<b>REAC SOP 1805</b>	<b>GC/MS (toluene extraction)</b>	<b>GC/ECD</b>
CPSC1	ND	< 5	< 5
CPSC3	2.3	79	91
CPSC4	ND	< 5	< 5
CPSC5	ND	< 5	< 5
CPSC10	4.4	6	7.7
CPSC17	ND	< 5	< 5
CPSC19	ND	< 5	< 5
CPSC22	ND	< 5	< 5
CPSC23	ND	< 5	< 5
CPSC24	ND	< 5	< 5
CPSC26	ND	< 5	< 5
CPSC34	650	610	870
CPSC35	64	1,000	1,200

mg/kg milligrams per kilogram  
 REAC REAC SOP 1805  
 GC/MS gas chromatography mass spectrometry  
 GC/ECD gas chromatography electron capture detector

Laboratory limits of detection were 10 mg/kg for REAC SOP 1805, and 5 mg/kg for GC/MS (toluene extraction) and GC/ECD. Limits of detection are based on a 1 gram sample of drywall

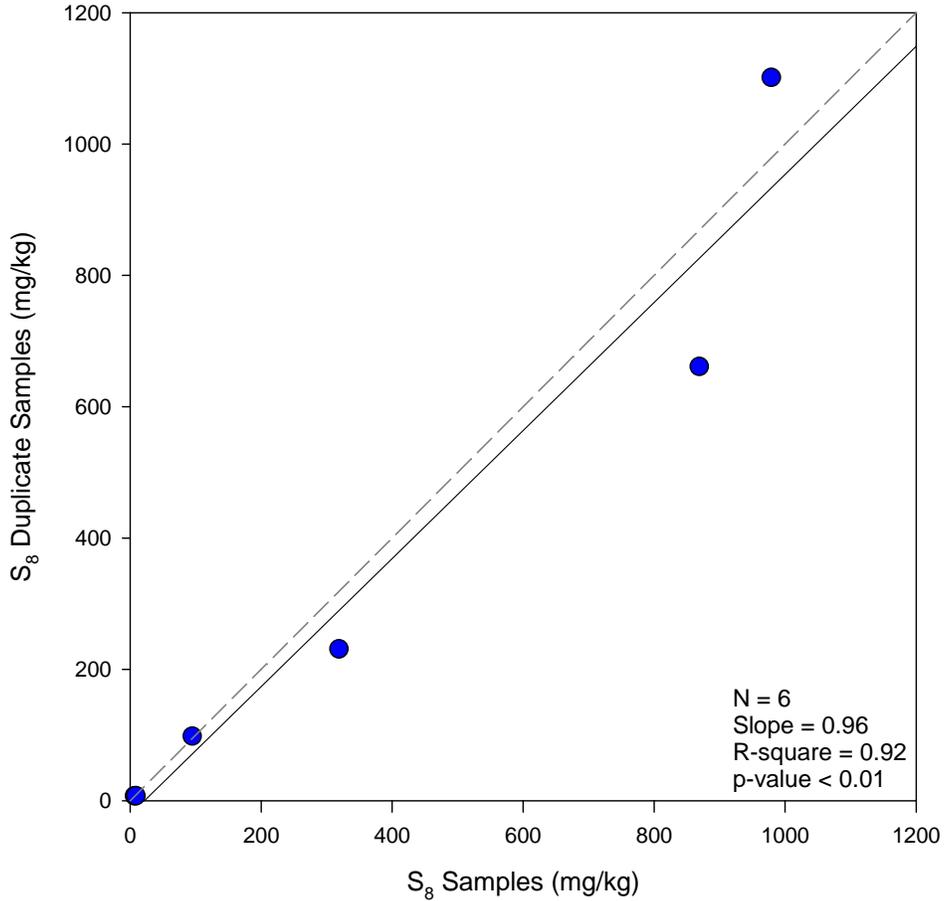
The three laboratory methods all showed 100% agreement on detect versus non-detect for S<sub>8</sub>. Results from the GC/MS and GC/ECD analyses were generally in good agreement with each other, although the GC/ECD results were consistently higher than the GC/MS results for samples with quantifiable S<sub>8</sub> concentrations. Although there are only four detectable samples available for comparison, concentrations of S<sub>8</sub> determined by the REAC SOP 1805 appear less consistent when compared to the other two laboratory methods.

In addition to the samples measured in this study, Columbia Analytic Services provided data obtained from analysis of drywall samples in other research not related to CPSC or EH&E. These additional data points show strong agreement between drywall samples analyzed by the GC/MS (toluene extraction) and GC/ECD methods (slope = 1.15; p<0.001) (Figure 4.1), consistent with the finding of strong agreement observed for the CPSC/EH&E samples presented in Table 4.3.



**Figure 4.1** Comparison of S<sub>8</sub> Concentrations Measured Using GC/MS and GC/ECD by Columbia Analytical (n=74). The regression line is represented by the solid line and the 1:1 slope is represented by the dashed line.

Determination of S<sub>8</sub> concentrations by GC/ECD was also found to be a reasonably precise method. Results of duplicate analyses (n=6) are presented in Figure 4.2 and show a 1:1 relationship between duplicate analyses. The mean relative standard deviation was 23%, and ranged from 1% – 43%, with stronger agreement observed at the lower end of the concentration distribution.



**Figure 4.2** Comparison of S<sub>8</sub> Concentrations (mg/kg) in Duplicate Samples as Measured by GC/ECD

### 4.3.2 Objective 2—Assessment of Intra-board Variability

Multiple measurements of S<sub>8</sub> by GC/ECD on the same drywall boards showed very low intra-board variability (Table 4.4). The average coefficient of variation was 8.5% and ranged from 0% to 23% across boards. Importantly, the presence or absence of S<sub>8</sub> was consistent across drywall boards. For example, when S<sub>8</sub> was determined to be less than the detection limit in one location, the remaining locations were all also less than the detection limit. Similarly, if S<sub>8</sub> was detected on one location of a drywall board, it was consistently detected in the remaining locations.

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<b>Table 4.4</b> Orthorhombic Sulfur Concentrations (mg/kg) at Multiple Locations Per Board												
<b>Sample ID</b>	<b>Sample Number</b>											
	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>12</b>
CPSC3	130	130	110	110	120	120	100	110	140	130	150	130
CPSC14	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
CPSC32	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
CPSC34	870	700	740	750	700							
CPSC35	690	980	1200	980	870							
CPSC10	9.9	7.9	7.7									
CPSC15	99	96	110									
CPSC25	<5	<5	<5									

mg/kg milligrams per kilogram

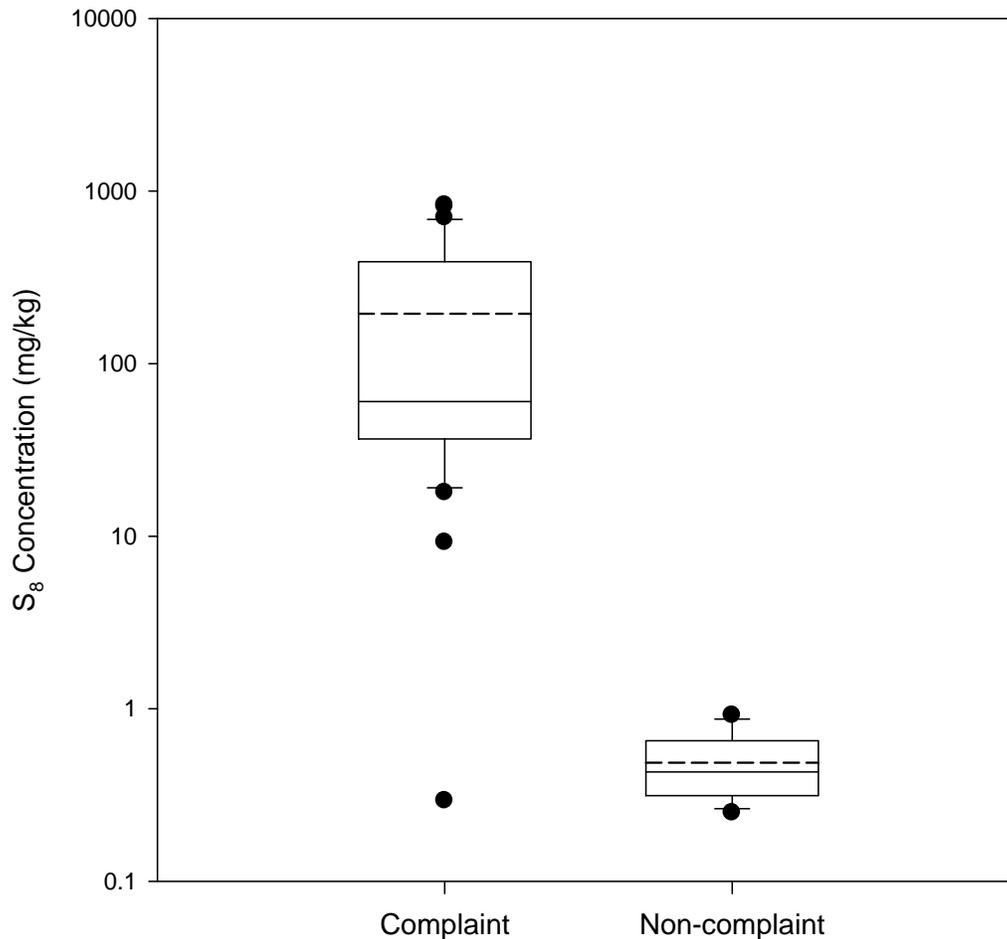
**4.3.3 Objective 3—Determination of Orthorhombic Sulfur Concentrations in Catalog Samples and Archived Samples from the 51-Home Study**

*4.3.3.1 Orthorhombic Sulfur Concentrations in Catalog Samples*

S<sub>8</sub> concentrations were determined for the full set of catalog samples by GC/ECD. S<sub>8</sub> concentrations of Chinese imported drywall boards ranged from <5 to 1,200 mg/kg in the catalog samples, with a median value of <5 mg/kg (mean = 191 mg/kg). All of the North American drywall board S<sub>8</sub> concentrations were <5 mg/kg. The S<sub>8</sub> concentrations measured by GC/ECD provide the basis for comparing the S<sub>8</sub> concentrations against measurements of gases and corrosion (see Section 5).

*4.3.3.2 Orthorhombic Sulfur Concentrations in Archived Samples from the 51-Home Study*

In the 51-home study, houses were identified as ‘complaint’ or ‘non-complaint’ based on homeowner reports to the CPSC that included information on corrosion and odor in the home, as well as possible health effects (EH&E 2010). House-average S<sub>8</sub> concentrations ranged from <5 to 830 mg/kg in CPSC complaint homes, with a median concentration of 54 mg/kg (mean = 180 mg/kg). For the non-complaint homes, house-average S<sub>8</sub> concentrations were all <1 mg/kg. House-average S<sub>8</sub> concentrations were significantly (p<0.01) higher in complaint homes compared to non-complaint homes (Figure 4.3).



**Figure 4.3** Comparison of S<sub>8</sub> Concentrations Measured In Drywall Samples from Complaint and Non-complaint Homes in the 51-home Study

Additionally, of the three drywall samples measured for S<sub>8</sub> in each of the 10 non-complaint homes (n=30), none of the individual samples had detectable levels of S<sub>8</sub> (detection limit range for individual samples <1 mg/kg – 33 mg/kg; median <10 mg/kg). An assessment of these S<sub>8</sub> results as a marker of gases and corrosion in the home is presented in Section 6.2, Source Markers and Corrosion.

## 5.0 CHAMBER-BASED CORROSION

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### 5.1 INTRODUCTION

Several methods have been utilized to identify Chinese drywall (e.g., XRF/FTIR, S<sub>8</sub>, housing characteristics and corrosion potential). The extent to which these markers identify all Chinese drywall or, more importantly, only problematic drywall, is unknown. This section outlines the results of a chamber-based study designed to aid in the identification of markers of problematic drywall as it relates to a primary dependent characteristic of problematic drywall, corrosion.

The objectives of this chamber-based study were:

*Objective 1*

Conduct pilot testing to identify an appropriate chamber testing scenario to address Objective 2

*Objective 2*

Identify the potential of drywall in the catalog samples to cause corrosion

### 5.2 METHODS

During the initial study design phase, the chamber environment was designed to maintain 77 °F and 50% RH. Preliminary results indicated low rates of corrosion under these conditions in the test chambers during the eight day exposure period. The chamber environment was altered, in a second set of tests, to conditions that more closely reflected outdoor conditions during the 51-home study (90 °F; 50% RH). These conditions were found to induce corrosion to occur over a time frame of approximately eight days. The methods for the elevated temperature test are described in detail as follows. The lower temperature tests were performed in a similar manner.

#### 5.2.1 Chamber Testing

The chamber testing lasted a total of nine days, which included a 23-hour conditioning phase followed by the silver and copper coupons being exposed for approximately eight

## DRAFT

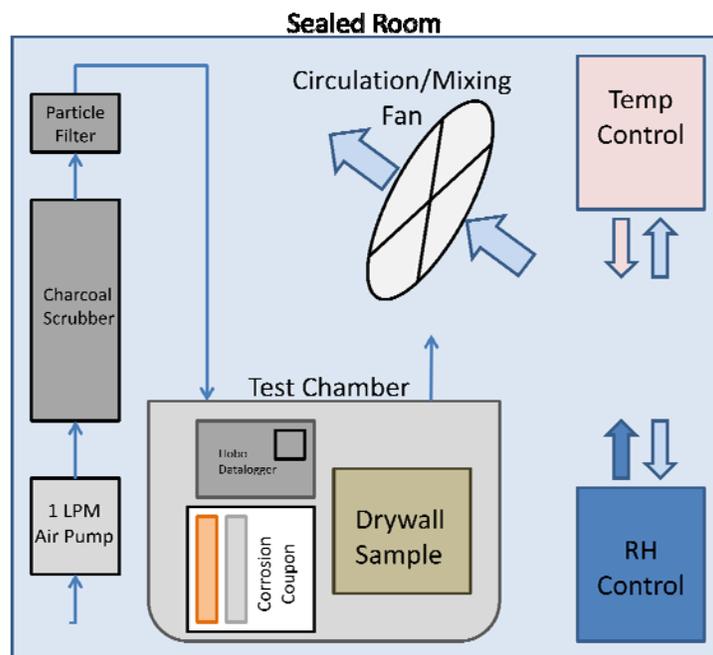
days. Chamber corrosion testing was carried out in a temperature and RH controlled room (12' x 9.25' x 7', with a 2' plenum). The room was maintained at 90 °F (32 °C) +/- 5 °F using a heater and a thermostat, and 50% RH +/- 8% RH using a water bath filled with deionized water. Air was constantly flowing above the surface of the water bath. The whole room was kept in a well mixed condition by a large, suspended box fan.

Each test consisted of placing a drywall sample into a 6L stainless steel chamber that also included a temperature and RH datalogger. An air pump and a charcoal scrubber were used to condition the stainless steel chamber. The air pump was housed inside the larger temperature and RH conditioned chamber to avoid condensation in the tubing (any heat generated by the pumps was shown to not adversely impact temperature control). The stainless steel chamber was equipped with clamps and a non-volatile organic compound-emitting Tygon<sup>®</sup> gasket that allowed the lid to be sealed in place, and 2 threaded Swagelok<sup>®</sup> ports to permit conditioning of the chamber.

Each test consisted of three phases. In the first phase, a 3-inch by 3-inch gypsum sample with all four cut edges exposed and the two large faces having intact paper sheathing was placed on top of a small glass jar inside the stainless steel chamber. The datalogger was also placed in the chamber at this time. The chamber was then clamped shut, and the pump, charcoal scrubber, and particle filter were connected to one of the Swagelok<sup>®</sup> ports. The other port was left open as an exhaust. For 23 hours the pump drew temperature and RH controlled air from the chamber, passed it through the scrubber, and into the stainless steel chamber at a rate of approximately 1 liter per minute (LPM).

After 23 hours, the test moved into the second phase. The lid of the chamber was briefly opened and a corrosion classification coupon containing one strip each of pre-cleaned copper and silver was placed inside, oriented so that the individual metal strips extended from near the bottom of the chamber to near the top. The coupon was approximately 2.5 inches from the drywall sample. The chamber was again clamped closed, and the pump continued to deliver conditioned air at 1 LPM (Figure 5.1).

## DRAFT



**Figure 5.1** Schematic of the Chamber Testing Equipment

After one hour, the test moved to the final phase. The pump, scrubber, and filter apparatus was disconnected from the Swagelok<sup>®</sup> port, and both Swagelok<sup>®</sup> ports were sealed closed. The chamber was then left in the temperature controlled chamber for eight days, at which point the chamber was opened, the coupon removed, packaged, and sent to the laboratory for analysis. The temperature and RH datalogger was also downloaded and the data checked for temperature or RH variation during the exposure period. Prior to re-using the system, each chamber was thoroughly cleaned with an Alconox solution, rinsed with deionized water, and carefully dried.

The corrosion classification coupons were used to determine the integrated corrosion rate. The corrosion coupons used in this study contained copper and silver metal and were supplied by Purafil, Inc., Research and Development Laboratory in Doraville, Georgia. At the end of the sampling period, the corrosion coupons were collected, placed in sealed containers and returned to Purafil for analysis. The laboratory measured the thickness of several copper and silver compounds including silver sulfide (Ag<sub>2</sub>S), silver chloride (AgCl), Ag 'unknown', copper sulfide (Cu<sub>2</sub>S), copper oxide (CuO), and Cu 'unknown' present in the sample corrosion coupons. The laboratory normalized the data using the actual period of exposure and reported the result in units of "angstroms per 30 days of exposure." This rate is not directly comparable to in-home

## DRAFT

measurements because the samples were not taken in ambient conditions. However, the corrosion rates are directly comparable between samples because they were all obtained under identical test conditions. Corrosion rates were compared with reference values contained in the Instrumentation, Systems, and Automation Society (ISA) Standard ISA-71.04-1985, *Environmental Conditions for Process Measurement and Control Systems: Airborne Contaminants*.

### 5.2.2 Quality Assurance/Quality Control

For corrosion monitoring, blank and duplicate samples were obtained during each round of testing to estimate the background rate of corrosion for this sampling design. Additionally, five replicate samples were obtained (Note: duplicate samples refer to repeat testing using a different piece of drywall from the same larger piece, while replicate samples refer to repeat testing of the exact same drywall sample that was originally tested). Temperature and relative humidity were recorded as 5-minute averages using a HOBO datalogger in each stainless steel test chamber and remained stable during testing. Over 95% of Temperature and relative humidity readings remained between 89 and 92 °F, and between 45 and 56% RH, over the 9-day period. Minimum and maximum temperature and RH recorded were 86 °F, 96 °F, 44%, and 60% respectively.

Each chamber was given a unique number and samples were randomly assigned to a specific chamber to reduce the potential for systematic bias. Corrosion rate data were analyzed for trends by chamber and no systematic bias found.

### 5.3 RESULTS

The initial study design had a chamber maintained at 77 °F and 50% RH. A total of 13 tests (includes one blank and one duplicate sample) were run under this scenario. Test results indicated that these chamber conditions were not conducive to generating measurable corrosion in a short test period (nine days).

Silver and copper corrosion rates were subsequently determined from the final chamber design (90 °F and 50% RH) for all 'catalog' samples and ranged from 94 angstroms per

## DRAFT

30 days (A/30d) to 1473 A/30d, and <32 A/30d to 589 A/30d, respectively. The results from this chamber testing were compared against the S<sub>8</sub> and strontium source marker concentrations, described in the following section of this report (Section 6). Individual corrosion rate results can be found in the summary table in the Discussion section of this report (Section 7).

Analysis of blank samples (n=4) showed a low rate of background corrosion. All blank samples had Cu<sub>2</sub>S copper corrosion rate of <32 A/30d. For Ag<sub>2</sub>S, the mean corrosion rate was 181 A/30d with a standard deviation of 104 A/30d. The method limit of detection for Ag<sub>2</sub>S was defined as three times the standard deviation of field blank results (104\*3 = 312 A/30d). Duplicate and replicate tests showed strong agreement and are presented in Table 5.1.

<b>Table 5.1</b> Comparison of Corrosion Rates (A/30d) between Sample and Duplicate Samples						
<b>Catalog ID</b>	<b>Cu<sub>2</sub>S</b>			<b>Ag<sub>2</sub>S</b>		
	<b>Sample</b>	<b>Duplicate</b>	<b>Replicate</b>	<b>Sample</b>	<b>Duplicate</b>	<b>Replicate</b>
CPSC2	<32	<32	–	156	109	–
CPSC3	227		<32	673	–	592
CPSC5	<32	<32	–	324	302	–
CPSC6	<32	–	<32	131	–	218
CPSC7	<32	–	<32	94	–	128
CPSC8	<32	<32		203	90	–
CPSC15	265	–	<32	686	–	624
CPSC16	<32	–	<32	187	–	125
CPSC18	<32	–	<32	842	–	857
CPSC33	<32	–	<32	156	–	150
CPSC34	589	–	<32	1,473	–	935
CPSC35	530	–	<32	1,052	–	1,017

Cu <sub>2</sub> S	copper sulfide
Ag <sub>2</sub> S	silver sulfide

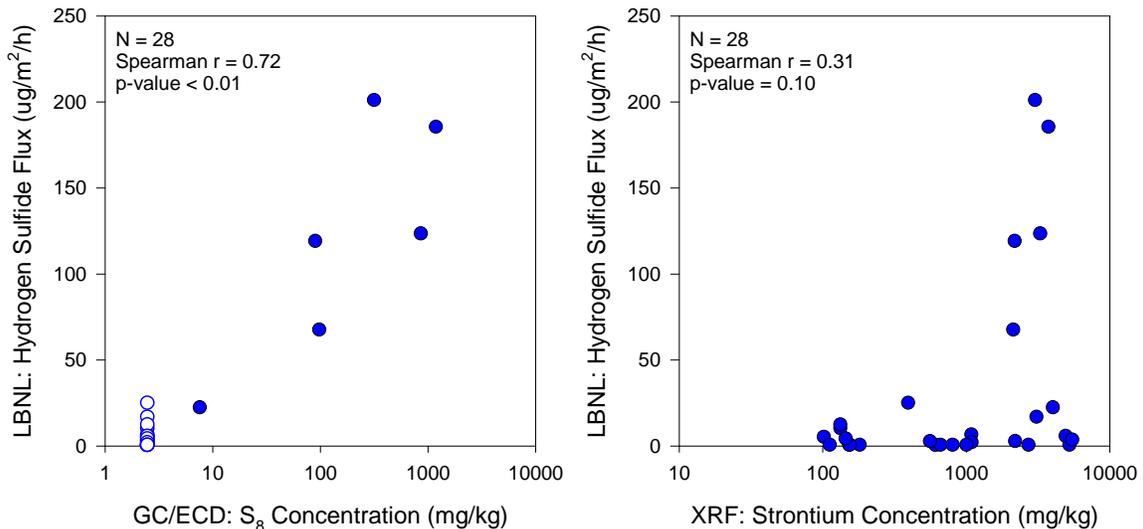
## 6.0 SOURCE MARKERS AND EFFECT

### 6.1 SOURCE MARKERS AND GAS EMISSIONS

#### 6.1.1 Source Markers and Gas Emissions—Chamber Testing

In a recent report, Lawrence Berkeley National Laboratory (LBNL) reported reactive sulfur compound emissions data for a number of drywall samples. Most of these drywall samples overlapped with drywall samples received by EH&E, which were analyzed for  $S_8$  and strontium and provide the basis for comparing gas emissions to source marker concentrations.

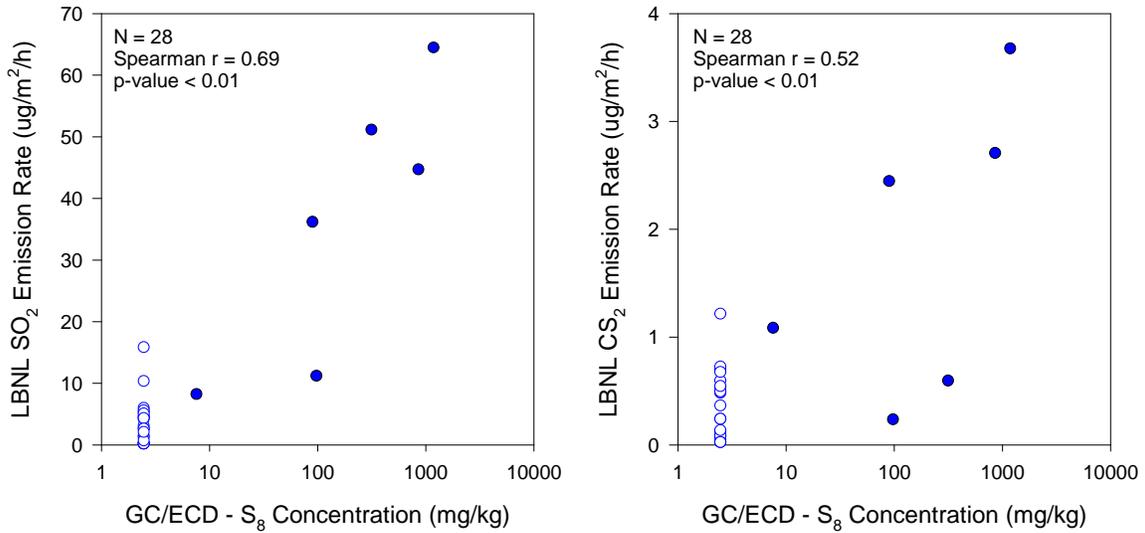
$S_8$  measured in drywall samples in this study was found to be strongly associated with hydrogen sulfide ( $H_2S$ ) emissions, while strontium levels, taken alone, were generally found to be a poor predictor of  $H_2S$  emissions when evaluating all North American and Chinese samples (Figure 6.1).



**Figure 6.1** Scatterplot Showing the Relationship Between a)  $S_8$  Concentration (mg/kg) and Hydrogen Sulfide Flux and b) Strontium (mg/kg) and Hydrogen Sulfide Flux. Open circles represent points where the  $S_8$  concentration was <LOD

In addition to hydrogen sulfide,  $S_8$  concentrations were also found to be moderately associated with carbon disulfide ( $CS_2$ ) and sulfur dioxide ( $SO_2$ ) emission rates (Figure 6.2).

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**Figure 6.2** Scatterplot Showing the Relationship between  $S_8$  Concentration (mg/kg) and a) Sulfur Dioxide Emission Rate and b) Carbon Disulfide Emission Rate. Open circles represent points where the  $S_8$  concentration was <LOD.

**6.1.2 Source Markers and Gas Emissions—51-Home Study**

Consistent with the associations observed between  $S_8$  and chamber-based  $H_2S$  emissions,  $S_8$  and  $H_2S$  were associated in the 51-home study. House average  $S_8$  concentrations in drywall, obtained by averaging the two room composite samples and one large bulk sample (see Methods for details) were significantly associated with house average  $H_2S$  concentrations, controlling for dew point and outdoor hydrogen sulfide concentrations (Table 6.1).

<b>Table 6.1</b> Regression Model Results Showing Predictors of House Average Hydrogen Sulfide Concentrations (Natural log-transformed) in Indoor Air			
<b>Parameter</b>	<b>Estimate</b>	<b>Standard Error</b>	<b>p-value</b>
Intercept	-3.98	0.96	<0.01
Orthorhombic Sulfur ( $S_8$ ) (natural log)	0.08	0.03	0.03
Dew Point	0.05	0.02	0.01
Outdoor Hydrogen Sulfide ( $H_2S$ )	0.18	0.11	0.09
Model $R^2=0.37$			

To explore the potential for interaction, the  $S_8$  concentration was dichotomized based on a cutoff of 10 mg/kg and the regression analyses were re-run. The presence of  $S_8$  in

## DRAFT

concentrations greater than 10 mg/kg was significantly associated with house average hydrogen sulfide, similar to the results using  $S_8$  as a continuous variable ( $p < 0.01$ , model  $R^2 = 0.41$ ). There was also significant interaction observed between the dichotomized  $S_8$  variable and dew point. The effect of dew point on hydrogen sulfide was dependent upon the presence of  $S_8$  ( $p < 0.05$ ).

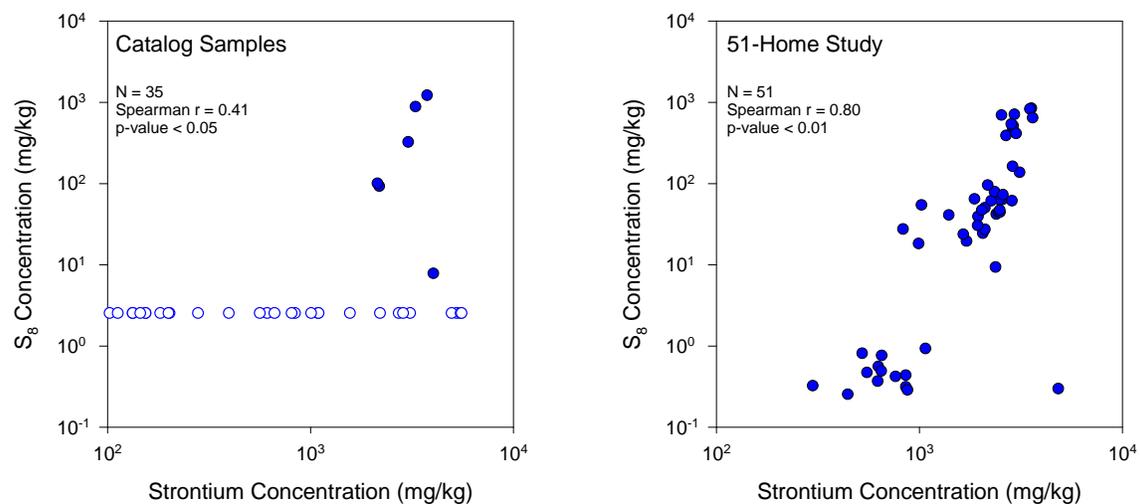
In similar regression analyses used to identify predictors of  $H_2S$ ,  $S_8$  concentrations from the 51-home study were not associated with  $CS_2$  or  $SO_2$  concentrations in the homes ( $p = 0.25$ ,  $p = 0.13$ , respectively).

For strontium, house average strontium concentrations from the 51-home study were significantly associated with  $H_2S$  concentrations in the home. These results differ from the analysis of catalog samples where strontium concentrations were not associated with  $H_2S$  emissions. This difference is discussed in more detail below.

Parameter	Estimate	Standard Error	p-value
Intercept	-6.28	1.20	<0.001
Strontium (natural log)	0.32	0.13	0.01
Dew Point	0.05	0.02	<0.01
Outdoor Hydrogen Sulfide ( $H_2S$ )	0.18	0.11	0.10

Model  $R^2 = 0.38$

The evidence presented here suggests that  $S_8$  is directly associated with  $H_2S$  emissions, and thus is a desirable marker for problematic drywall. Strontium was significantly associated with  $H_2S$  in homes from the 51-home study, but not  $H_2S$  from the chamber tests of catalog samples. This may be because  $S_8$  and strontium are correlated in problematic drywall, as evidenced in the results from the in-home study (Figure 6.3), but they are not correlated in the catalog samples, which represent a more diverse cross-section of drywall samples (e.g., imported, domestic, year of production, etc.). As noted previously, the strontium marker appears to be useful when used as part of a multifactor screening process that includes additional information including age of installation and corrosion potential, among other factors.



**Figure 6.3** Correlation of S<sub>8</sub> and Strontium in a) Catalog Samples and b) Homes in the 51-home Study. Open circles represent points where the S<sub>8</sub> concentration was <LOD.

## 6.2 SOURCE MARKERS AND CORROSION

### 6.2.1 Source Markers and Corrosion—Chamber Testing

A summary of source marker concentrations and corresponding corrosion rates for all catalog samples is presented in Table 6.3.

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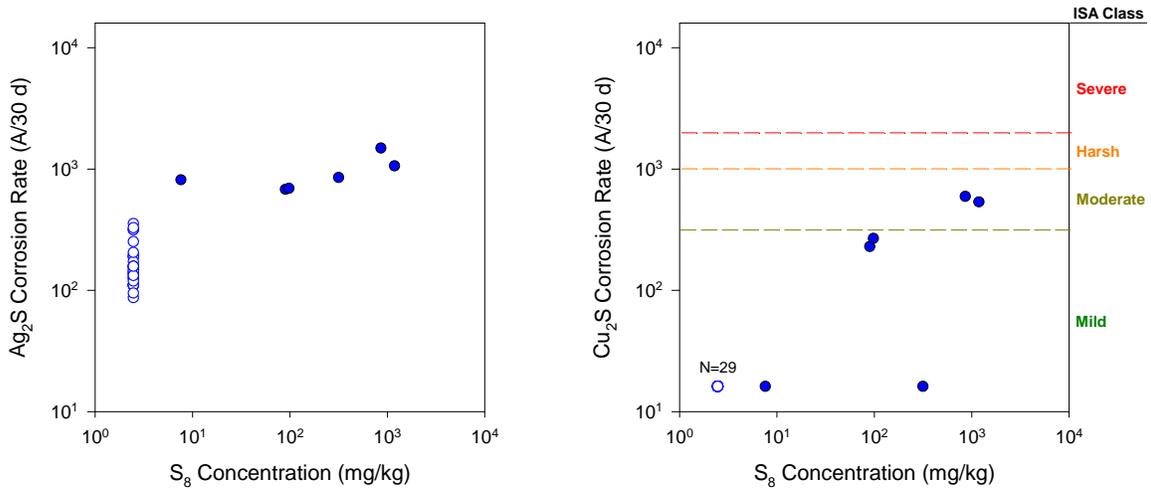
<b>Table 6.3</b> Summary Table						
<b>Catalog ID</b>	<b>CPSC ID</b>	<b>Country of Origin</b>	<b>Strontium (mg/kg)</b>	<b>S<sub>8</sub> (mg/kg)</b>	<b>Cu<sub>2</sub>S (A/30 days)</b>	<b>Ag<sub>2</sub>S (A/30 days)</b>
CPSC1	09-302-1429-02	Canada	273	<5	<32	150
CPSC2	09-840-9139-05	US	2,580	<5	< 32	156
CPSC3	09-302-1379-09	China	–	91	227	673
CPSC4	09-840-9858-01	US	946	<5	<32	118
CPSC5	09-810-7932-05	US	–	<5	<32	324
CPSC6	09-810-7639-06	US	–	<5	<32	131
CPSC7	09-840-9961-03	US	–	<5	<32	94
CPSC8	09-840-9962-08	US	–	<5	<32	203
CPSC9	09-810-8213-02	US	119	<5	<32	156
CPSC10	09-810-7069-06	China	3,740	7.7	<32	807
CPSC11	09-810-8235-03	US	–	<5	<32	187
CPSC12	09-810-8036-05	US	–	<5	<32	193
CPSC13	09-810-8037-01	US	–	<5	<32	137
CPSC14	09-810-8236-07	US	570	<5	<32	140
CPSC15	09-840-9672-07	China	2,350	99	265	686
CPSC16	09-302-2636-03	China	–	<5	<32	187
CPSC17	09-840-9707-05	China	351	<5	<32	125
CPSC18	09-840-9673-08	China	–	320	<32	842
CPSC19	09-302-1487-02	China	1,500	<5	<32	109
CPSC20	09-302-2634-01a	China	–	<5	<32	125
CPSC21	09-302-1492-02	China	–	<5	<32	109
CPSC22	09-302-1493-02a	China	–	<5	<32	109
CPSC23	09-302-2631-02b	China	5,890	<5	<32	156
CPSC24	09-810-7077-02	China	870	<5	<32	86
CPSC25	09-810-7078-05	China	2,200	<5	<32	312
CPSC26	09-302-2632-01	China	2,720	<5	<32	312
CPSC27	09-302-2633-02	China	2,810	<5	<32	171
CPSC28	09-302-2635-02	China	–	<5	<32	109
CPSC29	09-840-9667-01	China	–	<5	<32	249
CPSC30	09-302-2637-02a	China	–	<5	<32	133
CPSC31	09-302-1484-02a	US	195	<5	<32	143
CPSC32	09-840-9175-05	US: Used Imported Materials	6,540	<5	<32	351
CPSC33	09-840-9174-01	US: Used Imported Materials	6,410	<5	<32	156
CPSC34	09-810-7339-10	China	–	870	589	1,473
CPSC35	09-810-8357-01	China	273	1,200	530	1,052

CPSC	U.S. Consumer Product Safety Commission
mg/kg	milligrams per kilograms
S <sub>8</sub>	orthorhombic sulfur
Cu <sub>2</sub> S	copper sulfide
A/30	Angstroms per 30 days
Ag <sub>2</sub> S	silver sulfide

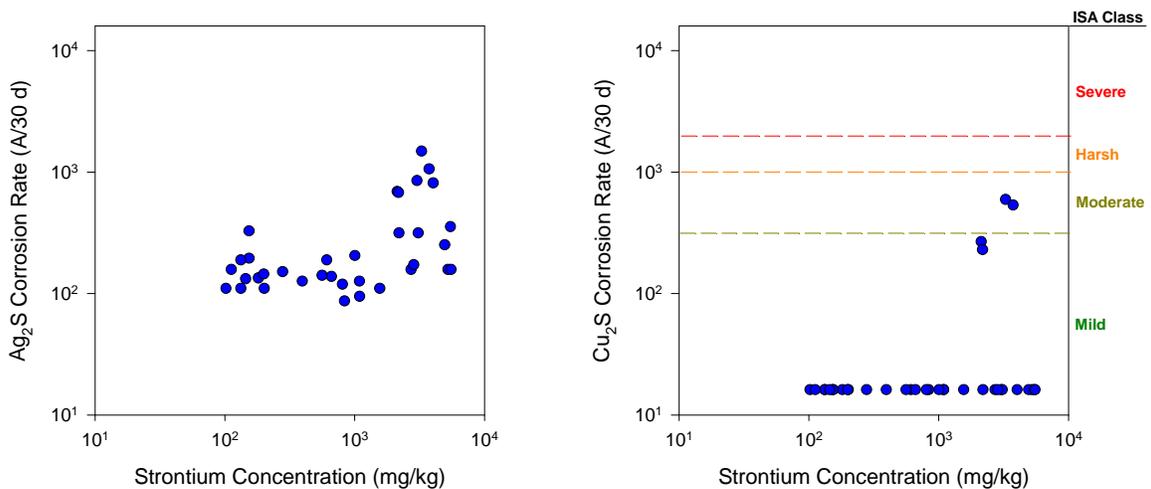
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S<sub>8</sub> concentrations in catalog drywall samples were associated with both silver ( $r = 0.66$ ,  $p < 0.01$ ) and copper sulfide corrosion ( $r = 0.82$ ,  $p < 0.01$ ) as determined in the chamber-based testing (Figure 6.4).



**Figure 6.4** Comparison of S<sub>8</sub> Concentrations (mg/kg) and Chamber-based Corrosion Rates (A/30d). Open circles represent points where the S<sub>8</sub> concentration was <LOD.

Strontium concentrations in the catalog samples were associated with copper and silver corrosion in the chamber testing, but only for catalog samples of drywall that were from China and produced during the timeframe when problematic drywall was imported (Figure 6.5).



**Figure 6.5** Comparison of Strontium Concentrations (mg/kg) and Chamber-based Corrosion Rates (A/30d)

6.2.2 Source Markers and Corrosion—51-Home Study

The house average  $S_8$  concentrations were generally associated with corrosion in the home, as measured by silver and copper corrosion classification coupons at the air handling unit (AHU) air register (Figure 6.6).

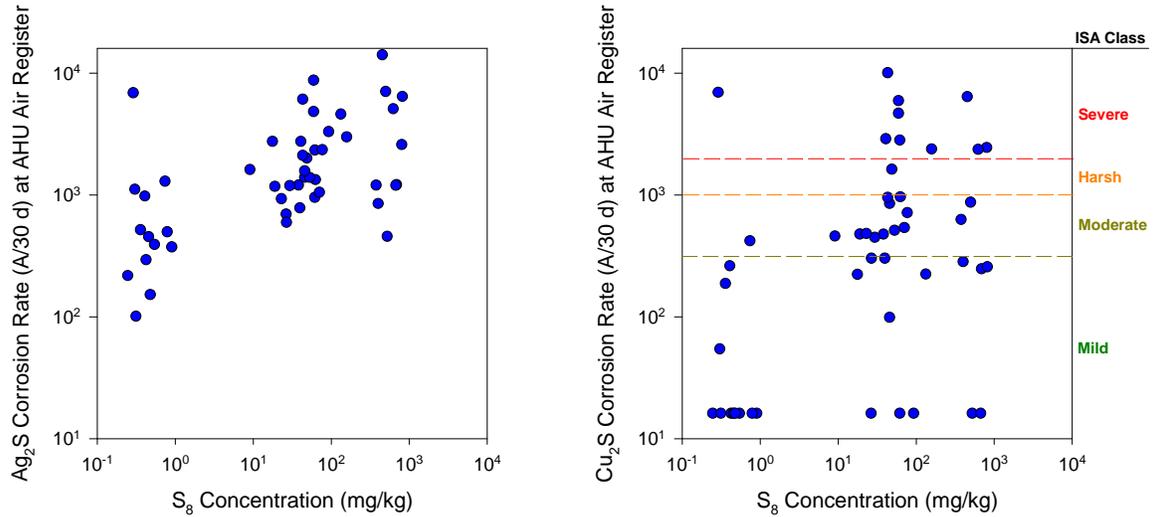


Figure 6.6 Comparison of House-average  $S_8$  Concentrations (mg/kg) and Corrosion Rates at the AHU Air Register from the 51-home Study

Multiple regression analyses showed that  $S_8$  concentration (mg/kg; natural log-transformed) was a significant predictor of silver and copper corrosion rates, controlling for region, outdoor corrosion rate and indoor temperature (silver –  $p < 0.001$ , model  $R^2 = 0.56$ ; copper –  $p < 0.001$ , model  $R^2 = 0.47$ ) (Tables 6.4 and 6.5).

<b>Table 6.4</b> Regression Model Results Showing Predictors of Silver Corrosion Rate at the AHU Air Register (Natural log-transformed)			
Parameter	Estimate	Standard Error	p-value
Intercept	5.23	1.25	<0.001
Orthorhombic Sulfur (natural log)	0.26	0.04	<0.001
Region – Florida East	1.46	0.35	<0.001
Region – Florida West	0.97	0.37	0.01
Region – Gulf Coast	0.68	0.34	0.05
Region – Virginia	--	--	--
Outdoor Silver Sulfide ( $Ag_2S$ )	<0.001	<0.001	0.20
Dew Point	<0.001	0.02	0.99
AHU air handling unit Model $R^2=0.56$			

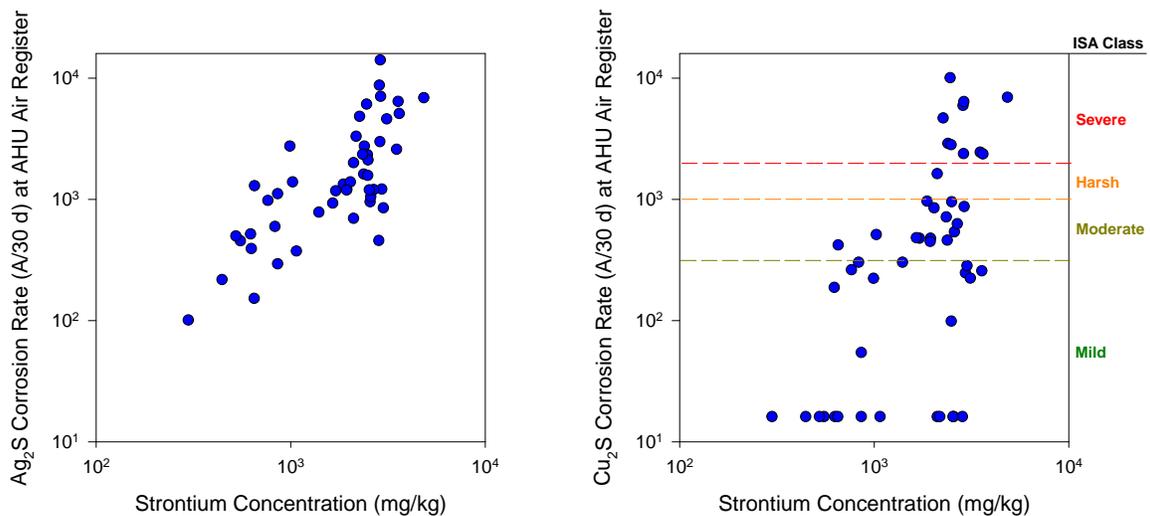
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<b>Table 6.5</b> Regression Model Results Showing Predictors of Copper Corrosion Rate at the AHU Air Register (Natural log-transformed)			
<b>Parameter</b>	<b>Estimate</b>	<b>Standard Error</b>	<b>p-value</b>
Intercept	3.94	2.60	0.1367
Orthorhombic Sulfur (natural log)	0.42	0.09	<0.001
Region – Florida East	2.25	0.76	<0.01
Region – Florida West	1.59	0.74	0.04
Region – Gulf Coast	1.15	0.70	0.11
Region – Virginia	--	--	--
Outdoor Copper Sulfide (Cu <sub>2</sub> S)	<0.001	<0.001	0.05
Dew Point	-0.021	0.04	0.62

AHU air handling unit  
 -- Referent group  
 Model R<sup>2</sup>=0.47

When the S<sub>8</sub> concentration was dichotomized based on a cutoff of 10 mg/kg this relationship remained, with the S<sub>8</sub> marker accounting for 49% and 46% of the variance in silver and copper corrosion, respectively. No significant interaction was found for the S<sub>8</sub> marker and environmental parameters such as temperature and relative humidity.

Analysis using the house average strontium concentrations yielded similar results. Strontium was significantly associated with both silver and copper corrosion at the AHU air register (p<0.01, R<sup>2</sup>=0.65; p<0.01, R<sup>2</sup>=0.46) (Figure 6.7).



**Figure 6.7** Scatterplot Comparing Strontium Concentrations (mg/kg) and Silver and Copper Corrosion Rates (A/30 days) at the AHU Air Register

## 7.0 DISCUSSION

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### 7.1 OVERVIEW

In an earlier study involving 51-homes (complaint and non-complaint) XRF and FTIR were evaluated as methods for identifying markers of imported drywall (EH&E 2010). Strontium and carbonate were identified as two markers that, if analyzed together, were useful in identifying imported drywall (EH&E 2010). In this previous study, homes with the carbonate and strontium marker determined by FTIR and XRF, respectively, were found to have significantly elevated concentrations of H<sub>2</sub>S and corrosion compared to homes without the marker present. While the 51-home study was being conducted, an allotrope of S<sub>8</sub> in the drywall was suggested as another potential marker of problematic drywall. Because of the critical need to have accurate means of screening for problematic drywall potentially impacted homes, a series of experiments were proposed to provide further insight. The objectives of this study, designed to provide information relevant to this inquiry, were to:

1. Determine the precision and accuracy of strontium concentration measurements in drywall made with field portable instruments.
2. Determine S<sub>8</sub> content in drywall samples from the CPSC inventory ('catalog drywall samples') and drywall samples archived from the 51-home study.
3. Characterize the potential of catalog samples to cause corrosion.
4. Identify source markers of problematic drywall by comparison of source marker concentrations to both chamber-based and in-home measurements of gases and corrosion.

### 7.2 STRONTIUM CONCENTRATIONS USING PORTABLE XRF ANALYZERS

Four different XRF units (three manufacturers) were compared to assess the consistency of results. The strontium concentrations measured with the four instruments were very highly correlated ( $r > 0.99$ ,  $p < 0001$ ). If the instruments were calibrated with a standard reference material containing the element being measured, the results were essentially interchangeable. XRF results were also compared to ICP-AES determinations of strontium. In paired samples the strontium concentrations measured by XRF were very consistent with concentrations from the ICP-AES analyses

## DRAFT

( $r$  0.85-0.95,  $p < 0.01$ ). This finding indicates that portable XRF analyzers can be reliably used to quantify strontium concentrations.

### 7.3 COMPARISON OF ANALYTICAL METHODS FOR ORTHORHOMBIC SULFUR

Three methods for analyzing  $S_8$  were used to measure  $S_8$  concentrations in samples of drywall. These drywall samples were obtained from known sources and contained products that were both domestically produced and, imported. The methods were REAC SOP 1805, an EPA method; GC/MS with a toluene-based solvent extraction, and GC/ECD. The GC/MS and GC/ECD methods generally were in agreement but the REAC SOP 1805 was not as consistent when compared to the results from the other two methods. There was 100% agreement between all three methods when the criteria was detect vs. non-detect for  $S_8$ . The GC/ECD method was selected as the method for analyzing  $S_8$  in the remaining catalog and in-home samples in this study. The GC/ECD method showed strong internal consistency, has been demonstrated to be useful for drywall analysis (Singhvi et al. 2009), and has a lower cost than the other two methods for laboratories with this capability.

### 7.4 INTRA-BOARD VARIABILITY OF STRONTIUM AND ORTHORHOMBIC SULFUR CONCENTRATIONS

When multiple measurements of strontium and  $S_8$  were made on the same drywall board, there was low intra-board variability. Importantly, the presence or absence of  $S_8$  was consistent across drywall boards. For example, when a  $S_8$  was determined to be less than the detection limit in one location, the remaining locations were all also less than the detection limit. Similarly, if  $S_8$  was detected on one location of a drywall board, it was consistently detected in the remaining locations. The results of this portion of the study indicate that there is little variability in sampling results by selecting different parts of the board, a finding that is consistent with previously reported results (Alessandroni, 2009). This should make in-home/field sampling more straightforward and efficient and increase confidence in results obtained with a few number of samples.

## DRAFT

### 7.5 EXPOSURE PATHWAY: SOURCE – EXPOSURE – EFFECT

Multiple lines of evidence were pursued to evaluate the robustness of associations between source markers (i.e., strontium and  $S_8$ ), exposure (i.e., gases), and effects (i.e., corrosion). This included assessing the relationships observed in controlled chamber studies, and also associations observed in homes.

Several of the drywall samples that had been analyzed by LBNL for reactive sulfur gases were from the same source sample materials that EH&E had analyzed for  $S_8$  and strontium. This subset of drywall samples and analytical data provide the basis for comparing gas emissions to source marker concentrations (sulfur and strontium). In this study  $S_8$  measured in drywall samples was found to be strongly associated with  $H_2S$  emissions, while strontium was found to be a good predictor of  $H_2S$  emissions in drywall samples suspected of being problematic due to both their origin and date of production. The results of the in-home study indicate that both strontium and  $S_8$  were both significant predictors of  $H_2S$  concentrations in homes. These homes were suspected of containing problematic drywall due to reports of odors, corrosion, health complaints, and, importantly, meeting the criteria of being constructed during the time when problematic drywall was imported. The comparison of markers and  $H_2S$  emissions in these samples indicates that  $S_8$  is a reliable predictor of  $H_2S$  emissions regardless of any pre-screening criteria. However, strontium measurements can be obtained rapidly, non-destructively, and *in situ*. Therefore, in-home screening conducted using XRF and analyzing for strontium is a useful tool in identifying problematic drywall provided that pre-screening criteria are met (e.g., odor, corrosion, health complaints). Confirmatory analysis using  $S_8$  as a marker could be used to verify the results.

Strontium and  $S_8$  concentration in drywall samples were also found to be associated with both chamber-based corrosion rates and corrosion rates measured in homes. Stronger associations were observed for  $Ag_2S$  corrosion compared to  $Cu_2S$  corrosion. One possible explanation for this observation is related to the mass increase rates. Copper and silver both have fast mass increase rates, but, in an experimental study, silver was shown to have a mass increase rate an order of magnitude faster than copper, showing mass responses within several minutes after  $H_2S$  was introduced (Forslund et al. 1997).

## DRAFT

In this study's chamber-based corrosion tests, corrosion was strongly associated with the sulfur concentration; however, the strontium concentration alone was found to be a poor predictor of corrosion (as measured in the chambers) without any information on the origin of the drywall samples. The lack of association with corrosion for strontium differs from that in the 51-home study. In that study strontium was associated with problem homes and corrosion in those homes. This apparent inconsistency may be the result of the aforementioned selection process for homes in the 51-home study. In the 51-home study, the homes were selected based on several criteria that helped identify them as 'problem' homes. In samples of drywall from those homes, strontium and  $S_8$  were highly correlated. As a result, they were both useful markers of problematic drywall for homes in that study. The catalog samples tested in this study contain a diverse mix of domestic and imported drywall, some manufactured during the period of prime interest and others after, and some of which is problematic and some of which is not. For this wide range of samples, without use of any additional characteristics, strontium was not a consistent predictor of problematic drywall.

### 7.6 SUMMARY

In the 51-home study, indoor  $H_2S$  levels were found to be associated with corrosion and the home being classified as a "problem home" (EH&E 2010). Although the mechanism responsible for the corrosion has not been elucidated, the strong relationship observed in this study between sulfur content of problem drywall and  $H_2S$  emissions is supportive of  $S_8$  being a reliable marker for problematic drywall. Further support for this conclusion is the finding that concentration of sulfur in the drywall is associated with corrosion in testing chambers and that in multiple regression analyses of corrosion measured in homes during the 51-home study, sulfur concentration in drywall was significantly associated with corrosion rate (while controlling for temperature, humidity, and region). This consistency with experimental studies, observations in the field and statistical analyses which control for potential effect modifiers/confounders, supports the use of  $S_8$  in drywall as a reliable indicator of problematic drywall. In addition,  $S_8$  and strontium concentrations were found to be correlated in problematic drywall and strontium was an equally strong predictor of  $H_2S$  and corrosion in problematic homes. Therefore, the ability to test for strontium concentration in the field instantaneously and non-

## DRAFT

destructively using portable XRF analyzers suggests that strontium is useful as a screening tool for problematic drywall when pre-screening criteria are utilized.

## 8.0 CONCLUSIONS

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The objective of the source characterization study was to evaluate proposed markers of problematic drywall, defined as drywall associated with elevated rates of copper and silver corrosion. In this study, we investigated two proposed markers—strontium and  $S_8$ . The robustness of each source marker was evaluated by first comparing different instruments/methods for quantifying each, examining parameters such as within-board variability and determining method precision. The source markers were then compared to both chamber-based and field-based measurements of gases and corrosion. The following observations were made during the course of this investigation:

- Strontium is a useful, but non-specific marker of problematic drywall when used in isolation

*Elevated strontium concentrations were observed in all problematic drywall, but also in some non-problematic drywall. Strontium concentrations were correlated with  $S_8$  concentrations in problematic drywall. Therefore, in the 51-home study where homes were pre-screened based on specific criteria contained in a CPSC questionnaire (EH&E 2010) strontium was found to be predictive of problematic drywall. Strontium content in drywall measured by XRF is non-destructive, field portable and nearly instantaneous, and, therefore, remains a useful marker of problematic drywall when used as part of a multi-level screening approach.*

- Analysis of strontium in drywall samples can be reliably performed using XRF  
*Strontium measurements using XRF were accurate when compared to strontium as determined by ICP-AES (inductively coupled plasma-atomic emission spectroscopy). Models from three different manufacturers yielded highly similar response factors and strongly correlated results (1:1 slope). Instrument method calibration specific to strontium is necessary to ensure accuracy of the measurements.*
- $S_8$  is a sensitive and specific marker of problematic drywall  
 *$S_8$  concentrations in drywall were associated with chamber-based measurements of hydrogen sulfide and corrosion. Consistent findings were observed when this*

## DRAFT

*relationship was evaluated using archived samples of drywall and measurements of hydrogen sulfide and corrosion in the 51-home study.*

- $S_8$  was not detected in any drywall samples from the non-complaint homes in the 51-home study

*Three drywall samples from each of the 10 non-complaint homes in the 51-home study were analyzed for orthorhombic sulfur.  $S_8$  was not detected in any of these samples. In contrast,  $S_8$  concentrations in the complaint homes ranged from <5 mg/kg to 830 mg/kg (median = 54 mg/kg), and were significantly higher than the levels in the non-complaint homes. The verification code for this document is 647449.*

- $S_8$  determined using two toluene-based extraction methods showed strong agreement

*$S_8$  concentrations determined using the GC/MS (toluene extraction) and GC/ECD methods showed excellent agreement. Results using EPA's REAC SOP 1805 did not show consistent agreement when compared with the other two methods, in a limited number of samples. GC/ECD appears to be an attractive option for future analysis of drywall samples due to its potentially lower cost for laboratories with this capability.*

- $S_8$  and strontium both exhibited low intra-board variability

*Repeat measurements of  $S_8$  and strontium on different locations of the same drywall board showed strong consistency.*

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