UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ENVIRONMENTAL RESPONSE TEAM
Edison, New Jersey 08837

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Ms. Lynn Wilder
Environmental Health Scientist
Agency for Toxic Substances and Disease Registry
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Subject: Drywall Sample Analysis

Dear Ms. Wilder,

The Agency for Toxic Substances and Disease Registry (ATSDR) contacted the Environmental Response Team (ERT) of the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) for analytical assistance with the Chinese-manufactured drywall used in Florida. On March 5, 2009, a teleconference was held with ERT, ATSDR and the Florida Department of Health (FDOH). The FDOH provided background information, including the work that had been previously performed by contractors from Lennar and Knauf (a German company that manufactures drywall in China). ATSDR requested that ERT conduct an independent elemental analysis of the Chinese drywall and compare it with the drywall manufactured in the U.S. With ATSDR's concurrence, six wallboard samples were selected for analysis. Two drywall samples known to have been manufactured in China were extracted by FDOH from affected homes in Florida. Four samples of U.S.-manufactured drywall were purchased from local stores in Edison, New Jersey.

Drywall Sample Analysis

ATSDR requested that the ERT analytical laboratory provide support to analyze drywall samples from China suspected of emitting rotten egg odors and causing copper corrosion (e.g., power switches, appliances) throughout the houses with complaints. The corrosion of copper containing items may lead to releases of chlorofluorocarbons (CFCs) and natural gases, depending on their construction materials. Individuals complaining about the drywall in their homes have also reported health issues such as problems with asthma, respiratory irritation, breathing difficulties, coughing, insomnia, eye irritation and headaches. At this time, FDOH has been unable to determine if these issues are directly linked to the suspect drywall. To date, a relatively low number of

samples have been analyzed, and the emission levels detected from samples tested in the laboratory are far lower than those typically associated with such symptoms.

Two Chinese painted drywall samples extracted from Florida homes by FDOH were shipped to Edison for analysis by USEPA/ERT. ERT purchased four US-manufactured drywall samples from local stores for comparison. First, the thin layer of paint was scraped off of two Chinese drywall samples for metals analysis. The top and bottom layers of paper were separated from the solid (gypsum) material of all six drywall samples and placed into separate glass jars. The paper portions of the samples were analyzed for metals, semi volatile organic compounds (SVOCs) and formaldehyde. The gypsum samples were analyzed for metals, SVOCs, volatile organic compounds (VOCs), formaldehyde, sulfide, water soluble chlorides, total organic carbon (TOC), pH and loss on ignition (LOI). Also, an optical microscopic examination was conducted to determine the presence of fly ash.

The drywall sample manufacturers and product names are as follows: US Gypsum/Hamilton (US); PROROC/Certainteed (US); National Gypsum/Gold Bond (US); GP/Tough Rock (US); Knauf/33928-20055 (China); and MIC/33966-12077 (China). The ERT/REAC analytical methods were modified to analyze these samples, as standard methods were not available in the area of sample digestion/preparation procedures.

Analytical Methods

Semi Volatile Organic Compounds: The gypsum and paper portions of the drywall samples were analyzed using ERT/REAC SOP #1805. A specific weight of sample in grams is extracted with a 1:1 methylene chloride/acetone mix in a Soxtherm extractor. The extract is concentrated, spiked with an internal standard mixture and subsequently analyzed by gas chromatography/mass spectrometry (GC/MS). Target analytes are identified by comparing the measured mass spectra and retention times with those obtained from calibration standards acquired under the same operating conditions used for the samples. Quantitation of each identified target analyte is calculated based on the internal standard method. The method was modified to determine the presence of any non-target compounds via a library search for the purpose of tentative identification. The NIST/EPA/NIH Mass Spectral Library containing more than 100,000 spectra was used. The elemental sulfur was analyzed using the sample extracts by GC/MS using an ERT/REAC modified method.

Volatile Organic Compounds: The two Chinese and one US-manufactured drywall gypsum samples were analyzed using ERT/REAC SOP #1807. A known amount of gypsum is weighed into a 40-milliliter (mL) Teflon®-lined septum vial, 5 mL of commercially available water suitable for VOC analysis is added, and the sealed vial is placed in the auto sampler. An additional 5-mL portion of VOC-free water containing surrogate/internal standards is added by the autosampler. In order to purge the compounds out of the dry wall, the samples were heated for five minutes at 75°C. These samples were then purged with helium for 20 minutes at the same temperature,

desorbed (trapped) onto the trap for four minutes and injected into the GC and detected using a 5975 MSD. The method was modified to determine the presence of any non-target compounds via a library search for the purpose of tentative identification. The NIST/EPA/NIH Mass Spectral Library containing more than 100,000 spectra was used.

Metals: The gypsum samples were first screened using a NITON x-ray fluorescence detector (XRF) to determine the presence of any metals. The XRF will help to ascertain whether additional metals that are not included in the Target Analyte List (TAL) routinely analyzed by the laboratory need to be added. The gypsum, paper and paint samples were analyzed for TAL metals using ERT/REAC SOP #1811, *Determination of Metals by Inductively Coupled Plasma (ICP) Methods,* and SOP #1832, *Determination of Mercury by Cold Vapor Atomic Absorption (CVAA).* Based on the XRF screening, strontium and sulfur were added to the list of analytes.

Formaldehyde, Sulfide, Total Organic Carbon: Analyses for these compounds were contracted to outside laboratories. Formaldehyde was analyzed by high pressure liquid chromatography (HPLC), ultraviolet detection (UV) in accordance with modified NIOSH Method 2016. For acid soluble sulfides, the gypsum samples were distilled using EPA SW-846 Method 9030B, which separates the sulfides from the matrix by adding sulfuric acid to the sample and heating to 70°C. The sulfide was quantified using an iodometric method. TOC was determined using a carbonaceous analyzer in accordance with EPA Region II SOP #C-88.

Water Soluble Chlorides: A specific weight of sample was mixed with a known volume of water prior to analysis. Samples were analyzed using a five-point calibration curve by a modified ferricyanide spectrophotometric technique, as outlined in the Standard Methods for the Examination of Water and Wastewater, Method 4500-Cl-E.

Loss on Ignition and pH: Loss on ignition data were obtained by weighing a known amount of sample into a crucible and igniting at 750°C using the modified Standard Methods for the Examination of Water and Wastewater, Method 2540G. A 5 percent weight by volume of a gypsum sample in water was prepared and mixed using a magnetic stirrer. The pH of the resulting aqueous solution was measured electrometrically using a calibrated pH meter.

Alkalinity and Sulfate: Alkalinity was performed in accordance with the Standard Methods for the Examination of Water and Wastewater, Method 2320B, that uses an acid titrant to measure the buffering capacity or ability to react with acids to a specific pH. Sulfates were determined using EPA Region II SOP #C-19.

Optical Microscopic Examination: The optical microscopic examination was performed at the ERT-Las Vegas laboratory using an Olympus optical microscope.

Discussion of the Results:

The significant differences between the Chinese drywall and the US-manufactured drywall analysis are as follows:

ERT analysis shows the presence of sulfur at 83 ppm and 119 ppm in the Chinese drywall samples and sulfur not detected in four US-manufactured drywall samples. The metal analysis shows the presence of strontium at 2,570 ppm and 2,670 ppm in the Chinese drywall samples, whereas strontium was detected in the US-manufactured drywall at 244 ppm to 1,130 ppm. The total acid soluble sulfides were not detected in any of the drywalls. Further investigation is critical to determine the presence of strontium as strontium sulfate or strontium sulfide using x-ray diffraction.

Iron concentrations of 1,390 ppm and 1,630 ppm were detected in the Chinese drywall samples and in the range of 841 ppm to 3,210 ppm for the US drywall samples. The highest concentration of iron detected in the National Gypsum/Gold Bond drywall was twice as high as the amount found in the Chinese drywall. An investigation will be done using additional drywall samples to determine whether the iron is present as oxide, sulfide or sulfate.

No evidence of fly ash in the Chinese drywall samples was noted based on the optical microscopic examination.

The ERT/REAC SVOC analysis results show the presence of two organic compounds in the Chinese drywall, as tentatively identified by the mass spectrometry library search for the Chinese drywall. The FDOH has requested that ERT further investigate these compounds. The two compounds were propanoic acid, 2-methyl-, 2,2-dimethyl-1-(2-hydroxy-1-methylethyl) propyl ester (CAS # 74367-33-2) at estimated concentrations of 58 and 92 ppm, and propanoic acid, 2-methyl-, 3-hydroxy-2,4,4-trimethylpentyl ester (CAS # 74367-34-3) at estimated concentrations of 50 and 84 ppm. These compounds were not detected in the US-manufactured drywall. ERT analyzed two samples for VOCs by GC/MS. The analyses confirm the presence of the above two compounds in the Chinese drywall, as tentatively identified by the mass spectrometry library search. ERT is in the process of obtaining standards of propanoic acid, 2-methyl-, 2,2-dimethyl-1-(2-hydroxy-1-methylethyl) propyl ester (CAS # 74367-33-2) and propanoic acid, 2-methyl-, 3-hydroxy-2,4,4-trimethylpentyl ester (CAS # 74367-34-3) to confirm the findings. The literature search reveals that these compounds are found in acrylic paints as reported in the following website:

http://www2.mst.dk/common/Udgivramme/Frame.asp?http://www2.mst.dk/udgiv/publications/2008/978-87-7052-763-7/html/kap02_eng.htm

The summary of analytical results of the six drywall (gypsum, paper, and paint) samples is presented in Summary Table 1. The semi-quantitative XRF data for gypsum

analysis are presented in Table 2. The tentatively identified compounds detected by the GC/MS library search for the SVOC analysis are presented in Table 3 for the gypsum and paper portions of the drywall samples.

Work in Progress

The additional drywall samples to be received from CPSC will be analyzed semi-quantitatively for calcium sulfate, strontium sulfide, strontium sulfate, pyrites and iron oxide by x-ray diffraction. The drywall samples from the United States and China will also be analyzed for VOCs, SVOCs, metals including strontium, sulfide, sulfite, formaldehyde, TOC and LOI. An optical microscopic examination for fly ash will also be conducted. Based on these analyses and the chamber study, ERT will conduct indoor air monitoring in Florida and Louisiana in three test houses for predetermined parameters. A QAPP is under preparation for the Technical Workgroup to review based on the available information to date, and will be modified based on any new information.

If there are any questions, please call me at 732-321-6761.

Sincerely

Raj Singhvi, Chemist

Enclosures

cc: David Krause, FDOH
Barnes Johnson, OSRTI
Arnold Layne, OSRTI/TIFSD
Jeff Heimerman, OSRTI/TIFSD
Dave Wright, ERT
Harry Compton, ERT

Table-1 Results of the Analysis for Metals in Solid Drywall Material, Paper and Paint

Sample No.		1	2	3	4	5	6
Sample ID		US Gypsum/Hamilto	Knauf/33928-20055	MIC/33966-12077	PROROC/Certainteed	ational Gypsum/Gold Bon	GP/Tough Rock
	Method	US	China	China	US	US	US
%LOI at 750C		21	22	24	21	19	24
pH of 5% slurry		7.08	7.41	7.35	7.28	7.29	7.31
Analyte		mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Aluminum	Modified REAC SOP 1811	305	1180	946	357	3570	1140
Barium	Modified REAC SOP 1811	5.14	33.3	42.8	14.2	12.8	15.0
Calcium	Modified REAC SOP 1811	278000	268000	254000	267000	245000	246000
Chromium	Modified REAC SOP 1811	1.92	5.28	3.66	2.81	4.34	1.96
Cobalt	Modified REAC SOP 1811	<0.87	<0.87	<0.93	<0.99	2.89	<0.90
Copper	Modified REAC SOP 1811	<1.52	1.79	2.80	<1.71	6.15	2.07
Iron	Modified REAC SOP 1811	841	1390	1630	1170	3210	1850
Lead	Modified REAC SOP 1811	<2.17	<2.16	<2.33	<2.44	3.48	2.61
Magnesium	Modified REAC SOP 1811	463	5020	10300	934	5250	4980
Manganese	Modified REAC SOP 1811	3.24	48.8	71.3	16.1	69.1	72.4
Mercury	Modified REAC SOP 1832	2.08	0.562	0.190	0.0668	<0.047	<0.045
Nickel	Modified REAC SOP 1811	<1.30	1.68	1.44	1.62	5.41	2.09
Potassium	Modified REAC SOP 1811	106	368	333	135	685	1490
Selenium	Modified REAC SOP 1811	8.94	2.81	<3.03	3.43	<2.87	<2.92
Sodium	Modified REAC SOP 1811	<217	428	498	<244	<220	<225
Vanadium	Modified REAC SOP 1811	<0.87	2.52	2.28	2.77	3.36	2.34
Zinc	Modified REAC SOP 1811	<6.71	<6.71	<7.24	<7.56	<6.83	10.1
Strontium (Drywall/Paper)	Modified REAC SOP 1811	244/45	2570/570	2670/636	499/110	638/19	1130/155
Strontium (Paint)	Modified REAC SOP 1811	NA	290	122	NA	NA	NA
Alkalinity (CaCO3)	SM2320B	<99	<99	970	<99	840	230
Alkalinity - Bicarbonate	SM2320B	<99	<99	970	<99	840	230
Sulfide (Lab1)	9030B	<4	<4	<4	<4	<4	12
Sulfide (Lab 2)	9030B	<10	<10	<10	<10	<10	<10
Sulfate	Region II SOP#C-19	666000	535000	507000	652000	568000	567000
Chloride (water soluble)	Modified SM 4500-Cl- E	74	250	190	36	59	143
Sulfur*	Modified REAC SOP 1805	<8.23	119	83	<8.13	<7.94	<7.94
Formaldehyde (Drywall/Paper)	Modified NIOSH 2106	ND/0.58	ND/0.44	ND/ND	ND/0.83	0.54/ND	0.24/0.67
Total Organic Carbon	Region II SOP#C-88	4300	2900	4300	2200	5500	16000
TOTAL ORGANIC COMPOUND* (Drywall/Paper)	REAC SOP 1805	7.7/75	145/125	243/246	18.3/299	31.6/70	2350/2400

* GC/MS analysis results from BNA extract including TIC'S

Raj April, 28, 2009

Table 2 Qualitative Analysis of Drywall Gypsum- XRF

Sample #	Sample ID		Ca	Fe	Sr
1	US Gypsum/Hamilton	US	222000 +/- 1200	410 +/- 90	180 +/- 10
2	Knauf/33928-20055	China	240000 +/- 1300	720 +/- 110	1970 +/- 32
2(Duplicate)	Knauf/33928-20055	China	241000 +/- 1300	730 +/- 100	1960 +/- 32
3	MIC/33966-12077	China	238000 +/- 1300	930 +/- 120	2130 +/- 34
4	Proroc/Certainyeed	US	226000 +/- 1200	990 +/- 120	370 +/- 14
5	National Gypsum/Gold Bond	US	210000 +/- 1200	2010 +/- 150	460 +/- 16
6	GP/Tough Rock	US	220000 +/- 1200	1210 +/- 130	844 +/- 21

A. Major - Calcium
Present - Iron, Strontium, Sulfur
Note: the sulfur line appears as week peak in the XRF spectrum of each sample
(sulfur cannot be quantified in these samples with Nitton XRF unit)

B. XRF Results (total concentration) in ppm +/- 1 standard deviation

Table 3 Tentatively Identified Organic Compounds, estimated concentration (mg/kg)

Sample #		1		2		3		4		5		6	
Tentatively Identified Organic Compounds		US Gypsum/Hamilton		Knauf/33928-20055		MIC/33966-12077		PROROC/Certainteed		National Gypsum/Gold Bond		GP/Tough Rock	
		US		China		China		US		US		US	
		Gypsum	Paper	Gypsum	Paper	Gypsum	Paper	Gypsum	Paper	Gypsum	Paper	Gypsum	Paper
Propylene Glycol	3.74									1.67			
Ethanol, 2-butoxy-	6.40									0.99			
Hexylene Glycol	6.60									1.98			
2-Propanol, 1-butoxy-	6.94									0.76			
Ethanol, 2,2'-oxybis-	7.24						3.62						
Hexanoic acid	7.38										1.48		
Ethanol, 2,2'-oxybis-	7.43												
2-Propanol, 1-(2-methoxy-1-methylethoxy)-	7.83			1.17									
Etane, 1,1'-oxybis[2-ethoxy-]	7.86			3.29	3.15								
2-Propanol, 1-(2-methoxypropoxy)-	8.03			2.05									
dipropylene glycol	8.52					2.56							
Hexanoic acid, 2-ethyl-	9.40									0.65			
1,3-Pentanediol, 2,2,4-trimethyl-	10.04					1.45							
Ethanol, 1-(2-butoxyethoxy)-	10.46			6.94	4.26	23.92	1.07						
Unknown	11.11					2.39							
Quinoline	11.27									0.55			
Unkown	11.45					1.52							
Unknown	11.49					1.77							
2-Propanol, 1-[2-(2-methoxy-1-methylethoxy)-1-methylethoxy]-	11.68			0.80									
2-Propanol, 1-[2-(2-methoxy-1-methylethoxy)-1-methylethoxy]-isomer	11.74			2.22									
2-Propanol, 1-[2-(2-methoxy-1-methylethoxy)-1-methylethoxy]-isomer	11.78			0.97									
Hexaethylene glycol dimethyl ether (?)	11.92			1.37									
2-Propanol, 1-(2-methoxy-1-methylethoxy)- isomer	11.96			1.48	1.69								
Cyclohexasiloxane, dodecamethyl-	11.99									0.55			
2,2,4-Trimethyl-1,3-pentanediol diisobutyrate	12.57				18.35					1.00			
Propanoic Acid, 2-methyl-,2,2-dimethyl-1-(2-hydroxy-1-methylethyl)propyl ester	12.57						2.79						
Propanoic Acid, 2-methyl-,2,2-dimethyl-1-(2-hydroxy-1-methylethyl)propyl ester+unknown	12.63			57.84		92.38							
Propanoic Acid, 2-methyl-,2,2-dimethyl-1-(2-hydroxy-1-methylethyl)propyl ester+unknown	12.83			50.45		83.57							
Vanillin	13.08				1.98				0.98	0.53	1.56		
Cyclododecane	13.75					6.24							
Phenol, 2,6-bis(1,1-dimethylethyl)-4-ethyl-	14.81				1.78								
Unknown	15.11										1.21		
Cedrol	15.47									1.26			
Benzyl Benzoate	16.84				4.24								
Homomenthyl salicylate	17.94			0.58									
n-Hexadecanoic acid	18.27		1.29		1.98	1.19			1.12	0.79	2.44		
9-Octadecenoic acid, (E)- or oleic acid	19.72		2.76					0.19			1.23		
Bis(2-ethylhexyl) maleate	19.86				7.00		1.01						
Octadecanoic acid	19.87										1.91		
C21 alkane	20.16						8.16	0.20				24.51	1.14
n-alkane	20.89	0.27	1.62	0.75	1.94			0.65		0.83		79.78	3.36
Tetracosane	21.60	0.45	3.56	1.71	3.56	2.13		1.56	1.32	1.89	1.83	198.23	7.97
morpholine, 4-phenyl-	22.26						2.13						
C25 alkane	22.27	0.62	6.61	2.16	8.57	2.57		2.76	3.29	3.03	4.96	356.70	14.26
diethyleneglycol dibenzoate isomer	22.34	0.39	6.28		18.13		7.98	0.18	2.93	0.61	8.66		
unknown	22.68						1.32	0.19					
C26 alkane	22.91	0.52	7.02	1.87	3.88	2.21		2.89	3.69	3.48	3.85	455.65	18.01
C26 alkane	23.31											18.94	0.86
C26 alkane	23.54	0.42	7.36	2.04	5.53	2.95		3.07	4.04	3.47	4.86	530.11	20.66
Alkane	23.92							0.17				32.47	1.32
Octacosane	24.15					1.12		1.37	3.05	1.54	3.58		
Alkane	23.99											20.41	
Unknown	24.44						0.91						
C28 alkane	24.14	0.29	4.47	0.92	3.07							113.86	5.60
C29 alkane	24.77	0.23	4.09	0.83	9.83	1.08	1.20	1.20	2.58	1.16	3.88	126.95	6.18
alkane	26.28		2.48		3.13	0.72	3.13	0.60	1.46		3.34	125.69	5.77
alkane	27.21		1.99		3.11		3.11	0.30	0.94		2.78	108.49	4.35
Binaphthyl sulfone isomer	27.30	0.27						0.25					
Binaphthyl sulfone isomer	28.19					0.59							
Hentriacontane	28.30		1.33										
C33 n-alkane	28.30										2.02	74.28	2.68
beta.-Sitosterol	29.51	0.17											
Tetratriacontane	29.60		0.86								1.65	50.23	1.62
Octadecanoic acid, ethenyl ester	30.82		2.07						1.43		1.13		
Alkane	30.88												0.84
C35 Alkane	31.13											28.45	
18-Pentatriacontanone	32.79		0.87										
Unknown	32.79								0.94		0.89		
Total organic		7.55	73.72	142.11	118.61	233.80	49.84	18.31	29.91	30.46	69.11	2344.74	95.80