

May 1, 2012

AB36-COMM-50-13

Mr. Jackson Morrill  
Director, Chemical Products & Technology Division  
American Chemistry Council  
700 Second Street NE  
Washington, DC 20002

RE: Generation of Silica Round Robin Samples  
RJ Lee Group Project No.: TLH202429

Dear Mr. Morrill:

In accordance with American Chemistry Council Contact #5288, RJ Lee Group has generated a series of round robin samples to test the precision and accuracy of x-ray diffraction (XRD) analysis of airborne silica samples. The generated filters were shipped on Friday, April 27, 2012 by UPS (tracking no. 1Z2644030395059479) to Dr. Anish Ranpuria at Sandler Occupational Medicine.

#### *Source Mineral Samples*

Three powdered mineral samples were selected for use in this project: Min-U-Sil 5, kaolin, and soda-feldspar from NIST (SRM 99a). To ensure that the minerals were of a respirable size, the oversize material in each sample was removed using sedimentation. A portion of the mineral was suspended in pH neutral, distilled water, agitated, and poured into a volumetric cylinder (similar to an Andreasen pipette). The suspension was allowed to settle over a distance of 10 cm for 75 minutes before the remaining supernatant was siphoned and filtered. Each material was dried in a low-temperature oven overnight before use in preparing the test filters.

#### *Filter Preparation*

Polyvinyl chloride (PVC) filters with a pore size of 0.8  $\mu\text{m}$  were used in this project. Each filter was assigned a random number for tracking purposes. Each filter was dried in a low temperature oven (30  $^{\circ}\text{C}$ ) for 3 days before being placed in a desiccator for a minimum of 2 days. At the end of this time, the filters were weighed.

The test materials were: 1) pure silica (Min-U-Sil; 2) kaolin; and 3) soda-feldspar. Each material was dried and desiccated prior to filter preparation. The materials to be deposited onto the filters are pure silica, and 50:50 (by weight) mixtures of silica and either kaolin or soda-feldspar.

To prepare the filters, a portion of the test material was weighed on a Mettler X5 analytical balance to the target weight. For this program, the target weights were 0.020 mg (25  $\mu\text{g}/\text{m}^3$ , 800 L sample), 0.040 mg (50  $\mu\text{g}/\text{m}^3$ , 800 L sample), and 0.080 mg (100  $\mu\text{g}/\text{m}^3$ , 800 L sample). The weighed portion was suspended in 25 mL of 2-propanol, sonicated and filtered onto the PVC filter that had been placed into a vacuum filtration holder. The filtered sample was placed into a desiccator for 2 days before weighing.

The weights and dates for this project are shown in Table 1. A spreadsheet of this Table is available.

The balance used in this project (Mettler MX5) is calibrated daily using 100 mg and 20 mg Class 1 weights. Since the start of 2012, the average reading for each calibration standard has been: (200 mg) 199.995 and (20 mg) 20.001. The standard errors for these readings are: (200 mg) 0.00023 and (20 mg) 0.00012. This suggests that, for inert materials, the observed weights are both precise ( $\pm 0.0005$  mg) and accurate.

#### *Sample Packaging*

Each filter was packaged into new, 2-piece 37-mm cassettes. A generic label was affixed to the outside of the cassette to facilitate sample tracking. The label contains no corporate markings.

The prepared cassettes were placed into zip-lock plastic bags, grouping similar matrices and loadings. For example, all of the  $25 \mu\text{g}/\text{m}^3$  silica-only samples were placed in one bag, all of the  $50 \mu\text{g}/\text{m}^3$  silica-only samples placed in another bag, etc.

The samples were boxed and shipped via UPS to Sandler Occupational Medicine.

If you have any questions, please feel free to contact me.

Sincerely,

Drew R. Van Orden, PE  
Senior Consulting Scientist  
[drew@rjlg.com](mailto:drew@rjlg.com)

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Table 1. Weights (mg) of Reference Minerals and PVC Filters Used to Generate Test Cassettes.

Sample #	Filter Weight	Date Weighed	Min-U-Sil		Kaolin		Soda-Feldspar		Total Deposit	Total Weight	Date Deposited	Post Weight	Date Post Weighed
			Target	Actual	Target	Actual	Target	Actual					
184	32.860	3/23/12	0.020	0.021	0	0	0	0	0.021	32.881	3/23/12	32.811	3/28/12
191	40.080	3/23/12	0.020	0.020	0	0	0	0	0.020	40.100	3/23/12	40.049	3/28/12
195	39.064	3/23/12	0.020	0.021	0	0	0	0	0.021	39.085	3/23/12	39.060	3/28/12
201	33.851	3/23/12	0.020	0.020	0	0	0	0	0.020	33.871	3/23/12	33.846	3/28/12
204	34.387	3/23/12	0.020	0.020	0	0	0	0	0.020	34.407	3/23/12	34.332	3/28/12
212	36.651	3/23/12	0.020	0.020	0	0	0	0	0.020	36.671	3/23/12	36.567	3/28/12
215	35.895	3/23/12	0.020	0.020	0	0	0	0	0.020	35.915	3/23/12	35.915	3/28/12
221	36.983	3/23/12	0.020	0.020	0	0	0	0	0.020	37.003	3/23/12	36.917	3/28/12
229	33.680	3/23/12	0.020	0.020	0	0	0	0	0.020	33.700	3/23/12	33.623	3/28/12
251	37.006	3/23/12	0.020	0.020	0	0	0	0	0.020	37.026	3/23/12	36.973	3/28/12
252	39.861	3/23/12	0.020	0.019	0	0	0	0	0.019	39.880	3/23/12	39.792	3/28/12
261	36.511	3/23/12	0.020	0.021	0	0	0	0	0.021	36.532	3/23/12	36.471	3/28/12
263	34.241	3/23/12	0.020	0.020	0	0	0	0	0.020	34.261	3/23/12	34.138	3/28/12
281	37.790	3/23/12	0.020	0.020	0	0	0	0	0.020	37.810	3/23/12	37.751	3/28/12
285	36.798	3/23/12	0.020	0.021	0	0	0	0	0.021	36.819	3/23/12	36.603	3/28/12
182	35.583	3/23/12	0.040	0.040	0	0	0	0	0.040	35.623	3/23/12	35.558	3/28/12
187	34.671	3/23/12	0.040	0.040	0	0	0	0	0.040	34.711	3/23/12	34.662	3/28/12
188	35.853	3/23/12	0.040	0.040	0	0	0	0	0.040	35.893	3/23/12	35.833	3/28/12
189	36.465	3/23/12	0.040	0.041	0	0	0	0	0.041	36.506	3/23/12	36.418	3/28/12
203	33.209	3/23/12	0.040	0.040	0	0	0	0	0.040	33.249	3/23/12	33.226	3/28/12
211	36.983	3/23/12	0.040	0.039	0	0	0	0	0.039	37.022	3/23/12	36.962	3/28/12
224	33.324	3/23/12	0.040	0.040	0	0	0	0	0.040	33.364	3/23/12	33.312	3/28/12
225	37.170	3/23/12	0.040	0.040	0	0	0	0	0.040	37.210	3/23/12	37.126	3/28/12
231	35.045	3/23/12	0.040	0.039	0	0	0	0	0.039	35.084	3/23/12	35.046	3/28/12
269	35.106	3/23/12	0.040	0.039	0	0	0	0	0.039	35.145	3/23/12	35.083	3/28/12
271	33.505	3/23/12	0.040	0.039	0	0	0	0	0.039	33.544	3/23/12	33.505	3/28/12
291	34.378	3/23/12	0.040	0.041	0	0	0	0	0.041	34.419	3/23/12	34.364	3/28/12
292	34.994	3/23/12	0.040	0.039	0	0	0	0	0.039	35.033	3/23/12	34.978	3/28/12
297	37.164	3/23/12	0.040	0.041	0	0	0	0	0.041	37.205	3/26/12	37.120	3/28/12

Sample #	Filter Weight	Date Weighed	Min-U-Sil		Kaolin		Soda-Feldspar		Total Deposit	Total Weight	Date Deposited	Post Weight	Date Post Weighed
			Target	Actual	Target	Actual	Target	Actual					
302	33.829	3/23/12	0.040	0.040	0	0	0	0	0.040	33.869	3/23/12	33.823	3/28/12
181	35.530	3/23/12	0.080	0.081	0	0	0	0	0.081	35.611	3/23/12	35.564	3/28/12
190	36.516	3/23/12	0.080	0.081	0	0	0	0	0.081	36.597	3/23/12	36.546	3/28/12
192	36.303	3/23/12	0.080	0.079	0	0	0	0	0.079	36.382	3/23/12	36.339	3/28/12
202	36.599	3/23/12	0.080	0.081	0	0	0	0	0.081	36.680	3/23/12	36.758	3/28/12
206	37.262	3/23/12	0.080	0.079	0	0	0	0	0.079	37.341	3/26/12	37.311	3/28/12
253	38.916	3/23/12	0.080	0.079	0	0	0	0	0.079	38.995	3/23/12	38.946	3/28/12
262	35.740	3/23/12	0.080	0.080	0	0	0	0	0.080	35.820	3/23/12	35.775	3/28/12
266	36.292	3/23/12	0.080	0.080	0	0	0	0	0.080	36.372	3/23/12	36.279	3/28/12
273	32.988	3/23/12	0.080	0.079	0	0	0	0	0.079	33.067	3/26/12	32.940	4/9/12
282	36.308	3/23/12	0.080	0.081	0	0	0	0	0.081	36.389	3/23/12	36.336	3/28/12
293	37.267	3/23/12	0.080	0.080	0	0	0	0	0.080	37.347	3/26/12	37.296	3/28/12
301	37.290	3/23/12	0.080	0.079	0	0	0	0	0.079	37.369	3/23/12	37.318	3/28/12
303	37.436	3/23/12	0.080	0.080	0	0	0	0	0.080	37.516	3/26/12	37.429	3/28/12
306	35.480	3/23/12	0.080	0.081	0	0	0	0	0.081	35.561	3/23/12	35.489	3/28/12
320	37.668	3/26/12	0.080	0.079	0	0	0	0	0.079	37.747	3/26/12	37.721	3/28/12
199	36.707	3/23/12	0.010	0.010	0	0	0.010	0.011	0.021	36.728	3/29/12	36.676	3/30/12
234	39.545	3/23/12	0.010	0.009	0	0	0.010	0.009	0.018	39.563	3/29/12	39.470	3/30/12
239	34.868	3/23/12	0.010	0.009	0	0	0.010	0.010	0.019	34.887	3/26/12	34.828	3/30/12
250	39.184	3/23/12	0.010	0.011	0	0	0.010	0.011	0.022	39.206	3/26/12	39.064	4/9/12
257	33.248	3/23/12	0.010	0.010	0	0	0.010	0.010	0.020	33.268	3/29/12	33.146	4/9/12
275	36.691	3/23/12	0.010	0.009	0	0	0.010	0.009	0.018	36.709	3/26/12	36.550	4/9/12
277	35.429	3/23/12	0.010	0.009	0	0	0.010	0.009	0.018	35.447	3/29/12	35.303	4/9/12
279	35.678	3/23/12	0.010	0.011	0	0	0.010	0.011	0.022	35.700	3/29/12	35.540	4/9/12
280	36.526	3/23/12	0.010	0.009	0	0	0.010	0.011	0.020	36.546	3/29/12	36.385	4/9/12
288	35.141	3/23/12	0.010	0.009	0	0	0.010	0.009	0.018	35.159	3/29/12	35.001	4/9/12
289	34.845	3/23/12	0.010	0.009	0	0	0.010	0.009	0.018	34.863	3/26/12	34.742	4/9/12
290	33.875	3/23/12	0.010	0.010	0	0	0.010	0.010	0.020	33.895	3/29/12	33.830	4/9/12
295	34.384	3/23/12	0.010	0.009	0	0	0.010	0.009	0.018	34.402	3/26/12	34.240	4/9/12
308	35.672	3/23/12	0.010	0.011	0	0	0.010	0.011	0.022	35.694	3/29/12	35.514	4/10/12
309	34.418	3/23/12	0.010	0.009	0	0	0.010	0.009	0.018	34.436	3/26/12	34.293	4/10/12

Sample #	Filter Weight	Date Weighed	Min-U-Sil		Kaolin		Soda-Feldspar		Total Deposit	Total Weight	Date Deposited	Post Weight	Date Post Weighed
			Target	Actual	Target	Actual	Target	Actual					
207	33.566	3/23/12	0.020	0.019	0	0	0.020	0.019	0.038	33.604	4/2/12	33.492	4/9/12
209	34.788	3/23/12	0.020	0.020	0	0	0.020	0.020	0.040	34.828	4/2/12	34.605	4/9/12
220	40.034	3/23/12	0.020	0.019	0	0	0.020	0.021	0.040	40.074	4/2/12	39.909	4/9/12
235	33.267	3/23/12	0.020	0.019	0	0	0.020	0.020	0.039	33.306	4/2/12	32.982	4/9/12
238	36.483	3/23/12	0.020	0.019	0	0	0.020	0.021	0.040	36.523	4/2/12	36.516	4/9/12
247	36.034	3/23/12	0.020	0.020	0	0	0.020	0.021	0.041	36.075	4/2/12	35.946	4/9/12
249	34.837	3/23/12	0.020	0.020	0	0	0.020	0.020	0.040	34.877	3/26/12	34.758	4/9/12
259	39.640	3/23/12	0.020	0.019	0	0	0.020	0.019	0.038	39.678	3/26/12	39.537	4/9/12
278	33.349	3/23/12	0.020	0.021	0	0	0.020	0.020	0.041	33.390	4/2/12	33.248	4/9/12
286	36.906	3/23/12	0.020	0.019	0	0	0.020	0.020	0.039	36.945	4/2/12	36.844	4/9/12
287	35.149	3/23/12	0.020	0.019	0	0	0.020	0.020	0.039	35.188	4/2/12	35.070	4/9/12
294	36.539	3/23/12	0.020	0.021	0	0	0.020	0.020	0.041	36.580	4/2/12	36.426	4/9/12
298	36.199	3/23/12	0.020	0.019	0	0	0.020	0.021	0.040	36.239	3/29/12	36.077	4/9/12
307	36.612	3/23/12	0.020	0.021	0	0	0.020	0.021	0.042	36.654	4/2/12	36.515	4/10/12
310	35.647	3/23/12	0.020	0.021	0	0	0.020	0.021	0.042	35.689	3/29/12	35.539	4/10/12
200	35.694	3/23/12	0.040	0.040	0	0	0.040	0.039	0.079	35.773	4/2/12	35.625	4/9/12
208	33.848	3/23/12	0.040	0.040	0	0	0.040	0.040	0.080	33.928	4/2/12	33.828	4/9/12
210	37.142	3/23/12	0.040	0.040	0	0	0.040	0.040	0.080	37.222	4/2/12	37.108	4/9/12
218	35.104	3/23/12	0.040	0.041	0	0	0.040	0.041	0.082	35.186	4/2/12	35.050	4/9/12
219	36.396	3/23/12	0.040	0.039	0	0	0.040	0.041	0.080	36.476	4/2/12	36.393	4/9/12
233	33.072	3/23/12	0.040	0.040	0	0	0.040	0.039	0.079	33.151	4/2/12	33.044	4/9/12
237	39.706	3/23/12	0.040	0.039	0	0	0.040	0.041	0.080	39.786	3/26/12	39.656	3/30/12
240	39.943	3/23/12	0.040	0.041	0	0	0.040	0.041	0.082	40.025	3/29/12	39.942	3/30/12
245	34.812	3/23/12	0.040	0.041	0	0	0.040	0.041	0.082	34.894	3/26/12	34.795	3/30/12
246	35.221	3/23/12	0.040	0.041	0	0	0.040	0.040	0.081	35.302	3/29/12	35.220	3/30/12
248	36.214	3/23/12	0.040	0.040	0	0	0.040	0.040	0.080	36.294	4/2/12	36.204	4/9/12
258	35.311	3/23/12	0.040	0.039	0	0	0.040	0.039	0.078	35.389	4/2/12	35.309	4/9/12
274	37.930	3/23/12	0.040	0.041	0	0	0.040	0.04	0.081	38.011	4/2/12	37.850	4/9/12
276	34.728	3/23/12	0.040	0.041	0	0	0.040	0.041	0.082	34.810	4/2/12	34.679	4/9/12
300	36.866	3/23/12	0.040	0.041	0	0	0.040	0.041	0.082	36.948	4/2/12	36.791	4/9/12
185	39.909	3/23/12	0.010	0.011	0.010	0.011	0	0	0.022	39.931	4/4/12	39.796	4/9/12

Sample #	Filter Weight	Date Weighed	Min-U-Sil		Kaolin		Soda-Feldspar		Total Deposit	Total Weight	Date Deposited	Post Weight	Date Post Weighed
			Target	Actual	Target	Actual	Target	Actual					
196	37.054	3/23/12	0.010	0.009	0.010	0.009	0	0	0.018	37.072	4/3/12	36.907	4/9/12
222	35.755	3/23/12	0.010	0.010	0.010	0.010	0	0	0.020	35.775	4/4/12	35.644	4/9/12
254	35.306	3/23/12	0.010	0.010	0.010	0.010	0	0	0.020	35.326	4/3/12	35.218	4/9/12
255	32.206	3/23/12	0.010	0.011	0.010	0.010	0	0	0.021	32.227	4/3/12	32.088	4/9/12
260	36.721	3/23/12	0.010	0.011	0.010	0.011	0	0	0.022	36.743	4/2/12	36.634	4/9/12
267	36.477	3/23/12	0.010	0.009	0.010	0.010	0	0	0.019	36.496	4/3/12	36.342	4/9/12
268	35.776	3/23/12	0.010	0.011	0.010	0.010	0	0	0.021	35.797	4/3/12	35.689	4/9/12
296	37.857	3/23/12	0.010	0.010	0.010	0.010	0	0	0.020	37.877	4/2/12	37.741	4/9/12
313	36.334	3/26/12	0.010	0.010	0.010	0.010	0	0	0.020	36.354	4/4/12	36.272	4/10/12
322	37.140	3/26/12	0.010	0.011	0.010	0.011	0	0	0.022	37.162	4/4/12	37.068	4/10/12
323	37.704	3/26/12	0.010	0.011	0.010	0.011	0	0	0.022	37.726	4/4/12	37.642	4/10/12
334	36.584	3/26/12	0.010	0.009	0.010	0.010	0	0	0.019	36.603	4/3/12	36.273	4/10/12
336	36.501	3/26/12	0.010	0.010	0.010	0.010	0	0	0.020	36.521	4/4/12	36.440	4/10/12
340	38.401	3/26/12	0.010	0.009	0.010	0.009	0	0	0.018	38.419	4/4/12	38.332	4/10/12
194	34.142	3/23/12	0.020	0.020	0.020	0.020	0	0	0.040	34.182	4/6/12	34.071	4/9/12
213	34.235	3/23/12	0.020	0.021	0.020	0.021	0	0	0.042	34.277	4/3/12	34.146	4/9/12
216	33.930	3/23/12	0.020	0.020	0.020	0.020	0	0	0.040	33.970	4/4/12	33.836	4/9/12
223	33.831	3/23/12	0.020	0.020	0.020	0.020	0	0	0.040	33.871	4/4/12	33.755	4/9/12
227	34.768	3/23/12	0.020	0.020	0.020	0.020	0	0	0.040	34.808	4/4/12	34.562	4/9/12
264	34.491	3/23/12	0.020	0.019	0.020	0.020	0	0	0.039	34.530	4/4/12	34.419	4/9/12
311	35.309	3/26/12	0.020	0.019	0.020	0.019	0	0	0.038	35.347	4/4/12	35.257	4/10/12
312	37.041	3/26/12	0.020	0.019	0.020	0.020	0	0	0.039	37.080	4/4/12	36.976	4/10/12
315	34.220	3/26/12	0.020	0.020	0.020	0.020	0	0	0.040	34.260	4/4/12	34.177	4/10/12
318	37.637	3/26/12	0.020	0.021	0.020	0.021	0	0	0.042	37.679	4/4/12	37.551	4/10/12
319	35.530	3/26/12	0.020	0.019	0.020	0.019	0	0	0.038	35.568	4/3/12	35.488	4/10/12
324	34.014	3/26/12	0.020	0.019	0.020	0.019	0	0	0.038	34.052	4/4/12	33.975	4/10/12
331	37.814	3/26/12	0.020	0.021	0.020	0.019	0	0	0.040	37.854	4/4/12	37.781	4/10/12
337	37.914	3/26/12	0.020	0.020	0.020	0.020	0	0	0.040	37.954	4/3/12	37.866	4/10/12
338	36.482	3/26/12	0.020	0.020	0.020	0.021	0	0	0.041	36.523	4/3/12	36.396	4/10/12
183	34.376	3/23/12	0.040	0.040	0.040	0.040	0	0	0.080	34.456	4/6/12	34.343	4/9/12
214	36.586	3/23/12	0.040	0.041	0.040	0.041	0	0	0.082	36.668	4/6/12	36.488	4/9/12

Sample #	Filter Weight	Date Weighed	Min-U-Sil		Kaolin		Soda-Feldspar		Total Deposit	Total Weight	Date Deposited	Post Weight	Date Post Weighed
			Target	Actual	Target	Actual	Target	Actual					
217	35.240	3/23/12	0.040	0.041	0.040	0.041	0	0	0.082	35.322	4/6/12	35.199	4/9/12
226	34.691	3/23/12	0.040	0.041	0.040	0.040	0	0	0.081	34.772	4/6/12	34.658	4/9/12
230	32.457	3/23/12	0.040	0.04	0.040	0.040	0	0	0.080	32.537	4/6/12	32.420	4/9/12
232	36.301	3/23/12	0.040	0.041	0.040	0.041	0	0	0.082	36.383	4/6/12	36.259	4/9/12
244	36.065	3/23/12	0.040	0.039	0.040	0.040	0	0	0.079	36.144	4/3/12	35.997	4/9/12
256	35.527	3/23/12	0.040	0.041	0.040	0.041	0	0	0.082	35.609	4/6/12	35.494	4/9/12
265	33.107	3/23/12	0.040	0.040	0.040	0.041	0	0	0.081	33.188	4/6/12	33.092	4/9/12
272	33.554	3/23/12	0.040	0.039	0.040	0.039	0	0	0.078	33.632	4/6/12	33.511	4/9/12
283	33.255	3/23/12	0.040	0.041	0.040	0.041	0	0	0.082	33.337	4/6/12	33.230	4/9/12
304	38.421	3/23/12	0.040	0.040	0.040	0.040	0	0	0.080	38.501	4/3/12	38.341	4/9/12
314	36.470	3/26/12	0.040	0.040	0.040	0.040	0	0	0.080	36.550	4/4/12	36.463	4/10/12
332	34.762	3/26/12	0.040	0.041	0.040	0.040	0	0	0.081	34.843	4/4/12	34.763	4/10/12
333	35.363	3/26/12	0.040	0.041	0.040	0.041	0	0	0.082	35.445	4/4/12	35.361	4/10/12
186	34.825	3/23/12	0	0	0	0	0	0	0	34.825		34.789	3/30/12
193	37.664	3/23/12	0	0	0	0	0	0	0	37.664		37.626	3/30/12
197	35.664	3/23/12	0	0	0	0	0	0	0	35.664		35.622	3/30/12
198	33.990	3/23/12	0	0	0	0	0	0	0	33.990		33.950	3/30/12
205	34.276	3/23/12	0	0	0	0	0	0	0	34.276		34.256	3/30/12
228	35.489	3/23/12	0	0	0	0	0	0	0	35.489		35.427	3/30/12
236	35.172	3/23/12	0	0	0	0	0	0	0	35.172		35.131	3/30/12
242	34.966	3/23/12	0	0	0	0	0	0	0	34.966		34.916	3/30/12
243	39.892	3/23/12	0	0	0	0	0	0	0	39.892		39.833	3/30/12
270	36.006	3/23/12	0	0	0	0	0	0	0	36.006		35.904	4/9/12
299	36.783	3/23/12	0	0	0	0	0	0	0	36.783		36.680	4/9/12
317	36.517	3/26/12	0	0	0	0	0	0	0	36.517		36.460	4/10/12
321	33.226	3/26/12	0	0	0	0	0	0	0	33.226		33.194	4/10/12
325	36.319	3/26/12	0	0	0	0	0	0	0	36.319		36.266	4/10/12
335	36.448	3/26/12	0	0	0	0	0	0	0	36.448		36.392	4/10/12