11-Oct-2011

Manuscript ID: AnnHyg-11-0178

Title: Quartz Measurement in Coal Dust with High Flow Rate Samplers: Laboratory Study

Dear Dr. Lee

We have now received the reviews of the manuscript you submitted to Annals of Occupational Hygiene, and they can be found at the foot of this e-mail. We will be pleased to publish the paper provided that it is amended satisfactorily to meet the comments, and provided no other critical problems arise at a later stage.

Please submit your revision within three months if possible, because new material is being published all the time, and reviewers’ comments go out of date. Revisions received after 6 months will normally go through peer review again.

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We are pleased to be able to publish your work, and look forward to receiving the revised version of your paper. If you decide that you will not be proceeding any further, it would be helpful if you could let us know.

Yours sincerely,

Dr. Lee Kenny

Editor for this paper

(Editor in Chief Trevor Ogden)

Here are the comments of the reviewers:

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Reviewer: 1

Comments to the Author

General Comments:The authors have performed a thorough examination of the different types of high and low volume samplers which could be used to sample respirable silica.

Major Revisions: None required.

Minor Revisions: There are numerous minor grammatical revisions required in the paper.

Industrial hygienists are looking for ways to sample at 1/10th to 1/2 of the TLV. It would be helpful to include the number of hours required to obtain a sample at 1/2 of the TLV, using a detection limit of 10 micrograms and the flow rates of the various samplers. This could be included in Table 1. The manuscript has been revised accordingly in Table 1.

Page 5, Line 54, states:"In order to destroy organic material..". The word "destroy" could be replaced with "eliminate". The manuscript has been revised accordingly in page 5.

Page 5, Line 56 and 57 states:" Two different ashing methods were employed including low temperature and muffle furnace ashing since these two methods are standard indirect methods for quartz analysis." Do the authors mean that these methods are used for quartz analysis? In other words the difference of the weights before and after ashing is taken as the weight of quartz? I think the authors mean that these are two methods of ashing employed in standard methods for quartz analysis. If this is the case, the wording should be changed to clarify the meaning of the sentence. The manuscript has been revised accordingly.

Page 6, Line 12 and 13 states:" Coal dusts from the CIP10-R sampler were extracted from the polyurethane foam by rinsing with isopropyl alcohol onto a 37 mm PVC filter and the filter was ashed by the Plasma-Prep II." I think that the extracted coal dust would be filtered onto the PVC filter to deposit the dust onto the filter. The manuscript has been revised accordingly.

Page 12, line 42. This is the only reference given in the online access format, which is incorrect. The correct web address is: doi:10,1093/annhyg/meq093. The authors can correct the web address, or use the published volume number which is Vol55, Issue 4, pp.357-368. The manuscript has been revised accordingly.

Page 14, line 23. The name of the second author is Sebestyen, not Sevestyen. The manuscript has been revised accordingly.

Reviewer: 2

Comments to the Author

1. General comment-you reference the use of the SKC sample jar when performing the flow calibrations measurements for the 10 mm nylon cyclone sampler. I hope you leak checked the jar, they are problematic. Unfortunately, we did not check the leak from the jar but when the flow rate of the pump was checked directly without jar and cyclone, the flow rate was close to with jar and cyclone. We’ll definitely check the leakage of the jar next time.

b) minor revision-the LOD of NMAM 7603 you cite on p9 was based on dual beam IR instrumentation, not FTIR, and peak height measurement. The manuscript has been revised accordingly in page 10. You later claim that your laboratory's FTIR LOQ is 5ug and reference a 1-2% relative standard deviation? Do you mean 12%? It is 1-2% not 12%. We are preparing another manuscript that is describing our laboratory LOD and LOQ for quartz using FTIR.

Reviewer: 3

Comments to the Author

This paper, although reasonable, needs clarification in some paragraphs - e.g. quartz mass with high and low flow rate samplers. The authors raise issues that are not fully thought through. It sometimes seems as if they simly thought it a good idea to mention a issue without further explianation. The paper highlights that differences between some samplers are significant and that low exposure limits for respirable crystalline silica (<0.05 mg.m-3) are not reliable with low flow rate samplers < 2l/min. It's not completely clear in the paper what flow rate is used for the high flow rate samplers.

I have the following comments

Abstract

Better analytical results – better than what? Do you mean a larger number of quantifiable results? The manuscript has been revised accordingly in abstract.

Overestimated – this word sounds a bit too harsh if you simply mean it collected more than the other sampler – were you using the nylon 10 as a reference sampler, is it a reference and if so it should be fully explained in the introduction.

The manuscript has been revised accordingly in abstract.

Introduction

Line 31 – show RSD for organics and metals – as you are comparing them and the reader doesn't have the luxuary of accessing the documents. References were added in the manuscript.

Line 35 – The lowest loading was 38 µg for silica – so what? Did you mean to add …. which obtained an RDS of xx%? More detail explanation was added in page 3.

Line 44 – leads to imprecise measurement The manuscript has been revised accordingly.

Line 52 – By whom is it not preferred – do you mean it is not common practice when measuring RCS because it covers more than one exposure reference period.

The manuscript has been revised accordingly.

Line 56 X-ray (is it not usually capital X) The manuscript has been revised accordingly.

Page 4 line 5 – do you need the phrase 'high volume samplers' twice in the same sentence? The manuscript has been revised accordingly.

A little confusion is evident in the end of this part.

The only recent work looking at differences of high flow rate samplers is Stacey and Thorpe 2009 the other article simply evaluates the analytical procedure.

Samplers were shown to collect more dust – of course they do with higher flow rates – is there a more significant conclusion? Was it worth publishing if that was the only conclusion? I thought it was that the samplers needed further optimisation of their flow rates, to more fully meet the ISO/CEN/ACIGH requirement. Is the purpose of this paper to assess the differences using the NIOSH adjusted flow rates in terms of the mass the collected from the same test material or simply to look at the differences with the low flow rate samples whn using the NIOSH adjusted flow rates. - Its simply not clear.

The last paragraph of the Introduction, on page 4, specifies that one goal of the present study is to quantify how much more quartz is collected by high flow samplers compared to low flow samplers. Of course they will collect more total dust and more quartz, but matrix effects resulting from the higher dust collection may interfere with the quartz analysis in which case it will not necessarily be seen to increase in direct proportion to the increase in total volume sampled and this effect may also differ between XRD and IR analyses. The manuscript was revised in abstract, introduction, discussion, and conclusion section. Beyond what is presented in this paper we are planning to conduct another study on adjusting flow rates in common cyclones to reduce bias against international respirable convention.

Materials

Please note that Higgins Dewell is a design type – not a sampler. I would call it the BGI4L though out the text, although in this part of the text you may wish to refer to it as reportedly based on the Higgins Dewell type design. The manuscript has been revised accordingly.

Why mention two types of flow rates for the GK2.69 and the FSP10 here, if you only use your adjusted value just report this as the value used – else readers may be searching for other results. Surely you can explain what the outcome of the previous study was in your introduction and then be consistent with flow rates in the rest of the text – else it appears you may have used the manufacturers recommendations. The manuscript has been revised accordingly in page 4.

Test Particles

What's the upper size limit for a Marple impactor and if larger sizes were present would this affect the median value ? The cut off size of the Marple impactor is 21.3 µm. The coal dust has nominal mass median aerodynamic diameters of 4 µm since the coal was ground and separated by size in accordance with previous study (Volkwein et al., 2004).

Methods

Page 5 Line 14 – did you use the same loading range? The result was from our previous study and the mass concentration range for uniformity test of the system was 30-170 mg/m3 with three different test aerosols (Lee et al., 2010).

Page 6 Did a qualitative analysis by XRD identify the presence of a clay? Coal dust was analyzed for qualitative identification of minerals present using XRD scan. Coal dust had a major quartz phase and a minor phase that is matched with kaolinite. No other mineral was present in coal dust.

Page 7 Data Analysis

Line 25 what are the authors trying to determine from the net mass ratio and quartz mass ratio – differences? Net mass ratio is for coal dust mass ratios between the samplers using gravitational analysis. Quartz mass ratio is for only quartz mass from FTIR and XRD analysis.

Line 27 – is 'actual' different from the quartz mass or do you just mean quartz mass? What do you mean in this sentence? The word ‘actual’ was removed.

Is there a simpler way of explaining what you mean by 'random blocking factor'.

The manuscript has been revised accordingly.

What is Fishers LSD? What is an interaction contrast? – plain English please.

The manuscript has been revised accordingly.

Figure 1: - If printing in black and white you had better consider the contrast between the columns, as it was impossible to distinguish on my copy. Too much text for some of the figures Figure 1-4 were revised accordingly.

Higgins Dewell – don't you mean BGI4L The manuscript has been revised accordingly.

Could the differences between nylon 10 mm Dorr Oliver and FSP 10 be due to the number of quantifiable results?

By “quantifiable” we think the reviewer is trying to point out that many of the DO results are < LOQ, so then he/she is wondering if that had an effect on the difference. The most of net mass from 10 mm Dorr Oliver cyclone showed much greater than 0.03 mg on filter that is estimated detection limit of gravimetric analysis (NIOSH method 0500 (total) and 0600 (respirable)).

Page 8 Net Mass Comparison

Surely the important information here is – is the mass collected on the filter in proportion to the increase in flow rate? I guess so, since the units that have a flow rate of 10 l/min compared with 1.7 l/min only collected 6.4 and 8 times the dust, when they should have collected about 6.6. The manuscript has been revised regarding this issue in discussion section and Figure 8 was added.

Again replace Higgins Dewell with BGI4L for consistency as Higgins Dewell is used to describe a plethora of samplers with different manufacturers. The manuscript has been revised accordingly.

Quartz concentration – It might be interesting to explain the old OSHA requirement here. Since the old OSHA requirements aren’t commonly used any more, readers might not grasp their significance here. Though interesting, adding them here might be confusing

Page 10 – It is just not clear to me – which are the results in the figures and values in the paragraph for the GK2.69 for 4.2 l/min and for 4.4 l/min. This definitely needs more clarity. Is this paper solely about the adjusted flow rates - where are the results for the unadjusted flow rates? It is described in our previous study; *Annals of Occupational Hygiene* 54: 697-709. More detail explanation was added in the manuscript in page 10.

Explain why they should become closer to unit It is explained in page 10 and more explanation was added.

Overestimated – do you really mean collect more – who is to say which sampler is more accurate – unless you can justify one of the samplers as a reference.

The manuscript has been revised accordingly.

I don't think I've seen any results comparing the different flow rates for GK2.69 and FSP10 – I could be wrong. We did not present any data with manufacturer’s flow rate but we compared in page 10 regarding mass concentration ratios between high and low flow rate sampler with two different aerosols but similar size of the particles.

Page 10 Quartz mass with high and low loadings.

I would approach your arguments from the other direction. To measure 0.025 mg.m-3 with a Dorr Oliver cyclone at 1.7 l/min an occupational hygienist would need to sample a full 8-hour shift to collect 26 µg of crystalline silica on a sample filter (Which is often not practible or possible) . This is between the reported LOQ for these techniques of 18.8 µg for NIOSH method 75000 and 33 µg for FTIR NIOSIH method 7603. The expected variability of a measurement of 0.025 mg.m-3 under these conditions is not known, but the lower 95 % confidence level is likely to be below 18 µg. Data from proficiency testing schemes estimate the precision when measuring 60 µg as ……..

The way of this discussion from the reviewer is also good to explain why high flow rate samplers would be beneficial for measuring respirable quartz compared to low flow rate samplers but it might not change significantly the subject that we’re trying to deliver to readers.

I would have though you would have some relationship between the dusts levels and that in this situation the DorrOliver and BGI4L obtained a larger number of result below the LOD and LOQ suggesting that these apparatus are unsuitable to measure 0.025 mg.m-3. With a material containing 7 % of quartz one only needs to collect 7 % of the WEL for respirable dust to meet the ACIGH guidance value.

Conclusion

Why possibly – surely they definitely would. ‘possibly’ was removed.

Are these the only conclusions you could obtain from these data – why discus different flow rates if you don't use them? Surely some samplers have sampled more or less than predicted. This study is confirming our previous work rather than comparison of the flow rates and conclusion section was revised to reduce confusion of the readers.

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