How to interpret sample volume limitations stated in reference methodologies published for air sampling for Occupational Hygiene purposes

At a recent OH AIA Association meeting, I was asked to explain the concept of "prescribed" minimum sampling duration of the various active sampling techniques we apply.

Firstly, one must ask the very basic question of sampling – namely, what is a sample?:

- In very basic terms it is a portion of the whole that is representative of the whole.

That brings us to the second question – what is meant by "representative" if we talk about a sample of a person's exposure to an airborne HCS?

- The sample must contain portions (defined quantities) of the HCS the person is exposed to, continuously collected over a period, that represents the entire duration (and thus time variations) of the person's exposure.

So, in terms of duration of sampling, the most representative sample, will cover the entire shift of the workday, as it will be 100% representative of all variations in exposure that occurred during that day, for that particular person. Because it is impractical to commence with sampling, at the exact moment that the shift starts, and terminate sampling at the exact moment that the shift ends, it is generally considered acceptable (or required by DoL) to cover at least 80% of whatever the duration of the shift is – as 80% is considered to be a "good enough" statistical representative portion of a shift. In OESSM, there is a lot written about the effect of the duration of the sample, on the "statistical representation" of the sample – as duration of sampling is directly linked to the precision (standard deviation) of the sample (see OESSM Chapter 4 and Appendices D and E – where it becomes clear that a single sample that covers less than 75% of the exposure duration (shift) cannot be used to determine compliance/non-compliance due its excessive Coefficient of Variance, that increases the Standard Deviation of the sample.

For example (to illustrate the concept discussed in detail in OESSM; Chapter 4 and Appendix E), the stated precision of a particular NIOSH method is a standard deviation of 20%. If sampling only covered 80% of the shift, the standard deviation could increase to 30%, and if sampling only covered 60% of the shift, the standard deviation increases to 50%.....what is a sample with a standard deviation of 50% really worth? You could probably have guessed a better result, without going through the effort and expense of taking a sample……

So, there is nothing more important in sampling, than to collect a sample that is representative of the exposure, in terms of the total, actual sample duration (or shift duration)….either through a single sample covering at least 75% - as prescribed by OESSM

(or the DoL prescribed 80%), or several shorter consecutive samples covering the entire shift….

The second most important aspect that determines how representative a sample is of a person's exposure, is the accuracy and precision of the sampling technique and analytical method (and the associated sampling and analytical procedures aimed at preventing and minimising systematic and random errors).

To prevent systematic errors, we use "recognised" sampling methods (some are even validated), such as NIOSH, OSHA, MDHS, methods, etc.

The important aspects of these methods (active sampling methods in particular) to take note of are:

- Prescribed sampling media (the importance of that is quite obvious and needs no further explanation)
- Prescribed sampling flowrate – as every type of sampling media, is designed to function optimally at a specific sampling flowrate (sampling at a rate too fast or too slow, the sample collected will no longer be representative of the person's actual exposure, which is why one cannot deviate from that – else you will incur a systematic error in your measurement result)
- The next aspect that must be considered is the sample mass limitations, as you can collect too much sample mass (over sample), or you can collect so little sample mass that it cannot be detected or quantified by the analyses technique
    - This latter aspect, is absolutely independent from sample size in terms of sample volume – therefore, one cannot be limited by a "prescribed" minimum or maximum sample volume – as it is totally dependent on the prevailing contaminant concentration.
    - The sample volumes indicated in these reference methods (e.g. NIOSH methods), are based on a specific, very narrow range of contaminant concentrations – therefore, if the actual contaminant concentration differs from that indicated in the NIOSH method, then clearly (considering the most basic scientific principles), that narrow range of sample volumes also cannot apply.
    - Therefore, cannot look at the maximum and minimum sample volumes "prescribed" in these methods as absolute limits, but look at it in context of the detection and quantification limits (LODs and LOQs respectively) of the sampling and analytical technique concerned

Look at the very well-known NIOSH Method 7602: SILICA, CRYSTALLINE by IR (KBr pellet) 7602, where the following sampling information is provided:

SAMPLING

SAMPLER:	CYCLONE + FILTER (10-mm nylon or Higgins-Dewell (HD), and PVC filter, 37-mm, 5-µm) *see sampling section
FLOW RATE:	Nylon cyclone: 1.7L/min HD cyclone: 2.2 L/min
VOL-MIN: **-MAX:**	400 L 800 L

Logic dictates that at a sampling rate of 2.2 L/min, one "may" only sample for roughly 6 hours and 4 minutes, (which is far less than 80% of an 8 hour shift and thus not a statistical representative sample) and you will reach the "max" volume of 800L.

So the question/s is, is there really a reason for limiting the sample to just 800 L (or 6 hrs and 4 minutes), especially if it is known that it will not return an accurate result (because the sample is not statistically representative of the person's exposure) – what will really happen if you sample for longer – what will the effect actually be – can you really be found to be non-compliant to the method?

The analytical procedure for crystalline Silica does not analyse the amount of air that was drawn through the filter – so how can there be a limit on the amount of air that was drawn through your filter? We are only interested in the amount of dust that was collected on the filter, and the crystalline silica content thereof. Therefore the volume of air is immaterial, as it is the concentration of dust suspended in the air that is important – for example:

- I collected 2mg of dust on each of two samples (filters; sample A and B) – to achieve that, I had to draw a 1,000 L of air through sample A (because the actual dust concentration was 2mg/m^3), and 500 L of air through sample B (because the actual dust concentration was 4mg/m^3)

Both samples collected 2mg of dust – so why must sample A be considered "invalid", just because a volume of air that is more than the "proposed Max" was sampled? There is absolutely no justification for that.

To fully understand (and be able to interpret and apply reference methods correctly) these reference methods, one must unfortunately read in full – see the following extract from the same method; NIOSH 7602:

APPLICABILITY: The working range is 0.025 to 0.4 mg/m^3 for a 400-L air sample.

All this means is that for a 400L sample, one should be able to quantify crystalline Silica concentrations as low as 0.025mg/m^3 and as high as 0.4mg/m^3 - so what does this imply (the hidden, or not so hidden message)?

To answer that, you must also read and understand the following:

MEASUREMENT

TECHNIQUE:	INFRARED ABSORPTION SPECTROPHOTOMETRY
ANALYTE:	Quartz
ASH:	Muffle furnace of RF plasma asher
PELLET:	Mix residue with KBr; press 13-mm pellet
IR:	Scan absorbance from 1000 to 600 cm^{-1}
CALIBRATION:	NIST SRM 1878a quartz, NIST SRM 1879a cristobalite, USGS 210-75-0043 tridymite diluted in KBr
RANGE:	10 to 160 µg quartz
ESTIMATED LOD:	5 µg quartz

And further on in the method it is also stated that (and I silently wonder how many OH practitioners that worry about sample volumes have picked this up):

Do not exceed 2 mg total dust loading on the filter.

The above implies, that the instrument they used, when they developed the method (today there are many other instruments that are far better), could repeatedly, accurately quantify crystalline Silica masses deposited on PVC filters, between 10 and 160µg – so, converting these masses to concentrations, using a sample volume of 400L gives one the above-mentioned "as low as 0.025mg/m^3 and as high as 0.4mg/m^3 ". One must understand, that "400 L" is an arbitrary number – it is not meant as any limitation on application of the method. They could just as well as have stated it as: "the working range is 0.01 to 0.16mg/m^3 for a 1000 L air sample".

So, it is not the volume of air that is important (or a limitation), or the concentration of dust, but only the amount of crystalline Silica in the sample …..it is only when the sample contained more than 160 µg or less than 10 µg (LOQ - Range) of crystalline Silica (or a total dust load of >2 mg), that the sample does not comply with the limitation of the reference method.

However – the analytical facility that you use, may have completely different LODs and LOQs (Range) than what is stated above.

- So, make sure that the laboratory you use is SANAS accredited (that implies that there methods are validated, and that they comply with the prescribed quality standards and that their LODs and LOQs are known), and that they are accredited for that specific NIOSH method (or in-house method based on the NIOSH method).
- Then, enquire what the laboratory's LOD and LOQs (Range) are for crystalline Silica on filters – you may be surprised - it may be less than the stated NIOSH method, in which case even if you comply with the published NIOSH method (Range), your sample may still be discarded because your crystalline Silica load is less than or exceeds the limits (Range) applicable to the specific laboratory…..

So, the sample volumes stated in reference methods are not absolute limits that must be complied with.

To collect a representative airborne HCS sample accurately, you need to do the following (or if you audit compliance, you must look at the following):

- Conduct a risk assessment, and try and establish the approximate airborne HCS concentration
- Research the most appropriate sampling and analysis technique
- Consult with your laboratory, on whether they can perform/are accredited for this method, and their LOD and LOQ for the HCS
- Obtain the sampling media prescribed by the reference method, and observe the prescribed flowrate for that media (as per the prescripts of the reference method – this you may not deviate from)
- From the knowledge of the estimated HCS concentration, prescribed flowrate and the Upper and Lower LOQs of your laboratory, you can calculate your specific minimum and maximum sample volumes to collect (to ensure you do not collect HCS sample masses outside the upper and lower LOQ of your Lab)
- Find out how long the shift duration of the person to be sampled is – if the calculated duration for collecting the sample volume that was determined in the previous step comfortably covers the entire shift duration, your sampling strategy will be a full shift (or >80% of shift) single sample. If the sample volume that was determined in the previous step will only cover a period that is <80% of the shift duration, you will have to follow a partial period consecutive sampling strategy.

- If the laboratory returns your sample as being “over-sampled”, then unfortunately, you will have to repeat sampling, but this time, collect “smaller” volume consecutive samples to cover the entire shift (or >80% of the shift).

I hope this explains – you may direct any additional questions to me.

Kind Regards

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