PILOT OF AN EXTERNAL QUALITY ASSURANCE PROGRAMME FOR CRYSTALLINE SILICA

MJ Shai, National Institute for Occupational Health

ABSTRACT

The mining industry has committed itself to reducing work-related silicosis. As part of this commitment thousands of personal air samples are being collected and analysed every year to establish the exposure of miners to respirable crystalline silica (RCS) dust. There is however no independently established scheme to assess the accuracy of the results obtained and the costly silicosis reduction programme may be being compromised by doubts about the accuracy of silica exposure measurements.

Although there are a few expensive international schemes available, there is no national external quality assurance programme for the analysis of respirable crystalline silica dust currently in South Africa. A number of laboratories analysing silica are certified by the South African National Accreditation System (SANAS) but this only confirms that they have adequate management systems in place.

An external quality assurance programme, or proficiency testing scheme, is a system for objectively checking laboratory results by means of an external agency. Such a programme for respirable crystalline silica dust in occupational hygiene air samples would provide the mining industry with a considerable degree of assurance that the results being reported by laboratories can be relied on to provide an accurate estimate of exposure.

The National Institute for Occupational Health (NIOH) has taken the initiative and piloted an external quality assurance programme for the analysis of respirable crystalline silica dust samples using modern X-Ray diffraction or infra-red methods. The external quality assurance programme is based on the internationally recognised UK Health and Safety Laboratory Workplace Analysis Scheme for Proficiency (WASP) programme.

Six laboratories agreed to participate in a six-month pilot scheme. These laboratories initially produced results with poor accuracy, but this improved in later rounds when support and advice to resolve problems was provided by the NIOH.

The initial results show that, with this challenging analysis, there is a high potential for error and the production of inaccurate results. With technical support and assistance however, the results can rapidly be improved to acceptable levels. This paper will present the results of the pilot scheme.

BACKGROUND

An external quality assurance (EQA) programme, or proficiency testing scheme, is a system for objectively checking laboratory results by means of an external agency[1]. In the South African mining industry, thousands of personal air samples are being collected and analysed every year to establish the exposure of miners to respirable crystalline silica (RCS) dust. An EQA programme provides an external and independent quality assurance service for laboratories carrying out the analysis of RCS dust in occupational hygiene air samples taken in the workplace environment.

There is currently no local external quality assurance (EQA) programme for the analysis of respirable crystalline silica dust in South Africa. Although international quality assurance schemes are available, these are expensive, turnaround times are normally long and inconvenient.

The lack of participation in EQA and dependence on internal quality assurance programmes means that there is no simple way of confirming the quality of the results being produced by laboratories carrying out this analysis. This has a deleterious effect on the “Elimination of Silicosis” programmes put in place by the Department of Minerals and Energy and the Department of Labour.

The Occupational Hygiene Section of the National Institute for Occupational Health (NIOH) undertook to develop and pilot an EQA programme for respirable crystalline silica analysis. This is based on similar international models.

Participation in this programme will instil confidence in clients and the regulatory authorities that laboratories are reporting RCS results that are correct and reliable. Other benefits of a national EQA system include low cost relative to international schemes, shorter turnaround times, and easy access to advice and support.

It should be noted that the EQA programme does not replace the routine internal quality control system to ensure that they are routinely producing accurate and reliable results. Nor does it replace the South African National Accreditation System (SANAS) for evaluating the management and administrative procedures of the laboratory.

METHODS

Preparation of standard RCS filters

Filters with a known mass of respirable crystalline silica deposited on them were prepared[2]. This was carried out using a glass flask into which a small amount of respirable crystalline silica dust was placed. The dust was aerosolised with a puff of air and some of that air is then drawn through a pre-weighed filter using a personal dust sampling pump and a dust cyclone. The mass of RCS dust collected on the filter was then accurately determined from the increase in weight of the filter.

Confirmation of amount of RCS deposited on standard filters

There are several techniques used for determining the amount of RCS on filters. These methods include Infra Red (IR), Fourier Transform Infrared (FTIR) and X-ray diffraction (XRD) spectroscopy. The filter sample is traditionally dry ashed then mixed with KBr and pressed to make a pellet which is then analysed by infrared spectroscopy (IR KBr[3]), however more modern methods allow a direct-on-filter analysis followed by FTIR or XRD (XRD DOF or XRD DOF[2]). The direct-on filter method is non-destructive. There is no sample preparation which reduces the likelihood of sample losses and, since the filters are analysed directly, they can be retained for quality control purposes.

The mass of RCS dust collected on the standard filters was confirmed by analysing them using both (XRD DOF) & Fourier Transform Infrared (FTIR DOF) spectroscopy.

Participation in the EQA pilot scheme

Seven laboratories were invited to participate in the NIOH RCS EQA pilot scheme but only four sent in results.

The four laboratories, identified as A through D, participated in the pilot study. One of these laboratories used the traditional ashing and KBr pellet method followed by infrared spectroscopy to analyse the RCS dust. One other laboratory analysed the filters using direct-on-filter with both XRD and FTIR spectroscopy.

Table 1: Laboratory code and method used for analysis

Laboratory code	Method used
A	XRD DOF[2]
B	XRD DOF[2]
C	IR KBr[3]
D_1	XRD DOF[2]
D_2	FTIR DOF[2]

Most of the participating laboratories initially struggled to achieve accurate results and to return the results within the agreed timeframe. However, as the pilot programme progressed both these aspects improved considerably.

Analysis of results

Two indicators were used to look at the results statistically:

– Firstly, a percentage difference from the target value is calculated for each sample analysed at each participating laboratory.

  A result within the range of ±15% from the target value was regarded as an indicator of acceptable performance during the pilot stage of the NIOH EQA programme. This is the same as is used in the UK EQA scheme[4].

– Secondly, an indicator known as the Z–score was also used[5]. This is a value that standardises the results of the EQA programme and allows comparisons within and between laboratories in different rounds.

  The Z-score for each sample analyzed corresponds to a point in a normal distribution. It is sometimes called a 'normal deviate' since it describes how much a point deviates from a mean or specified point.

  In this case, the true value of the crystalline silica result is the specified point and the Z–score describes how far from this the participating laboratory's value lies. The Z-score is measured in standard deviations.

  The Z-score is calculated as indicated below:

$$\text{Z-score} = \frac{(\text{Laboratory result} - \text{target value})}{(\text{SD})}$$

  The SD is the standard deviation of all the gravimetric and analysis results from the NIOH and the submitting laboratory for each standard filter.

  Z-scores of up to ±2 are acceptable they contain 95% of values expected in a normal distribution. A Z-score of between ± 2 to 3 provides a warning and outside ±3 is regarded as unacceptable as the value is outside 99.7% of expected values in a normal distribution.

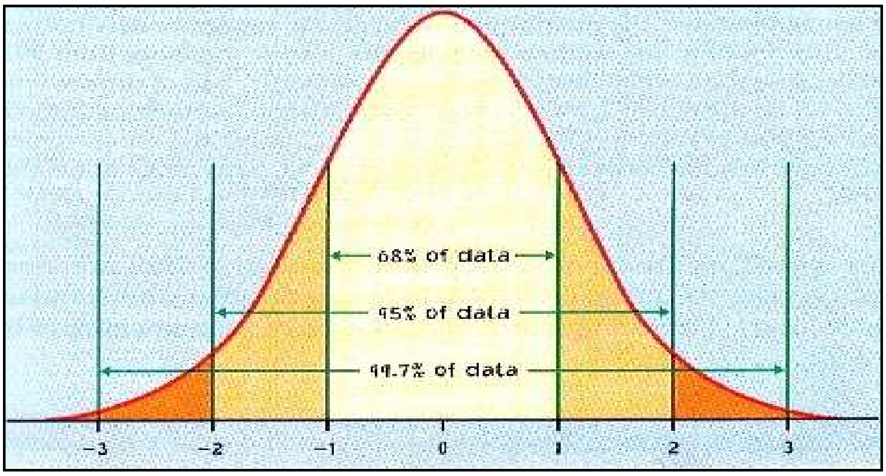

Figure 1: Chart of Z-scores indication

The Z-score is the Performance Index and is used to allocate the laboratory to a Performance Category of 1 – acceptable, 2 - warning or 3 - unacceptable.
The single worst result is ignored to make some limited allowance for one-off problems i.e. with analytical equipment.

Support

As an integral part of the pilot programme, ongoing support was provided to the analysts in the participating laboratories. This mostly involved providing copies of analytical methods, sets of calibration standards, and advice on techniques and the interpretation of data.

RESULTS

Table 2: Differences of laboratory results from the target values

	% Difference from known mass of RCS																			
Laboratory	**Round 1**				**Round 2**				**Round 3**				**Round 4**				**Round 5**			
A	+7	+7	-	-	-2	+19	+12	+27	3	-5	-2	+6	0	-1	-13	-10	-8	-13	-10	-16
B	+84	+69	-	-	0	+1	+6	+1	2	+5	+9	+2	-5	+1	-2	-4	-3	+3	-4	-7
C	NR	NR	-	-	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	+77	+91	-8	-79
D_1	NR	NR	-	-	+56	+55	+33	+59	110	-36	+12	-10	+4	+10	+11	-7	+6	+22	+6	+13
D_2									146	-44	+22	-8								

Table 3: Z-scores

	Z-Scores*																			
Laboratory	**Round 1**				**Round 2**				**Round 3**				**Round 4**				**Round 5**			
A	+1	+1	-	-	0	+2	+2	+4	0	-1	0	+1	0	0	-2	-1	-1	-2	-1	-2
B	+11	+9	-	-	0	0	+1	0	0	+1	+1	0	-1	0	0	-1	0	0	-1	-1
C	NR	NR	-	-	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	+10	+12	-1	-11
D_1	NR	NR	-	-	+7	+7	+4	+8	+15	-5	+2	-1	+1	+1	+1	-1	+1	+3	+1	+2
D_2									+19	-6	+3	-1								

NR = No return

DISCUSSION AND CONCLUSION

In general, most laboratories initially produced results with poor accuracy but this rapidly improved in later rounds. Support and advice to resolve problems was provided by the NIOH. The most common sources of error were the unavailability of high quality reference material and the inability to accurately prepare suitable calibration filters.

In some cases, where the participating laboratory obtained very poor results, the samples were returned to the NIOH for re-analysis. Similar results to the target value were again obtained. This suggests that the source of the inaccuracy is at the participating laboratory.

These initial results show that, with this challenging analysis, there is a high potential for error and the production of inaccurate results. With technical support and assistance however, the results can rapidly be improved to acceptable levels.

REFERENCES

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