QUARTZ ANALYSIS IN GRAVIMETRIC SAMPLING

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An overview of the methods employed in the assessment of quartz exposure is provided. The principles and some of the problems associated with each method is discussed. The methods reviewed include wet chemical methods, X-ray diffraction and infrared absorption of which the latter two methods are deemed appropriate for analysing quartz on personal gravimetric collected samples. The implications of combining area samples collected over a six month period, and performing only a single quartz analysis rather than separate analyses, are considered. Finally, various options open to mines with regard to their involvement with quartz analysis are also briefly discussed.

Introduction

General

In the mining industry a fundamental problem from a health point of view is the production of airborne dust in the working environment. With the introduction of personal gravimetric sampling as an alternative to konimeter assessment of dust exposure, the need arose for analyzing dust samples for one of its detrimental constituents, namely quartz. The various analytical procedures for the analysis of quartz in environmental samples all suffer serious deficiencies, to the extent that there is no reliable method. Each sample represents "a major research effort"[1].

It is the objective of this paper to provide a simple overview of the principles involved in the different analytical methods of quartz analysis, and to describe some of the problems associated with each method. Since mines will be responsible for submitting a result on quartz content, it is anticipated that this paper would provide some guidelines in planning a strategy for quartz assessment. The choice of the mine is simply whether to perform the analysis in-house, or whether to despatch the samples to an accredited authority for analysis.

The methods most commonly used in quartz analysis are the wet chemical methods, X-ray diffraction and infrared absorbtion. Before describing these methods it is necessary to briefly mention some of the properties of silica and the health effects related to exposure to free crystalline silica.

Silicon and its derivatives

Apart from oxygen, silicon is the most abundant element in the earth's surface constituting 27,6 per cent of the earth's crust[2]. Being a tetravalent element like carbon, it crystallizes in the diamond lattice, which is the basis for forming chains of polymers with alternating atoms of silicon and oxygen. There are three classes of silicon-containing substances: i) alloys and silicides; ii) inorganic compounds occurring as the oxide (SiO_2) and as silicates as found in asbestos, cement, mica and soapstone amongst others; and iii) organic compounds such as silicon esters[3]. Of these forms, we are mostly concerned with the dioxide form of silica. Silicon dioxide can exist in two varieties, amorphous and crystalline. Amorphous SiO_2 such as diatomaceous earth (kieselguhr) appears to have a low risk to health. On the other hand, the free and uncombined cryscrystalline forms do present a health risk. At atmospheric pressures these can exist as quartz, tridymite and cristobalite. Quartz is the most common form in nature, but if free silica is heated between 860 and 1 470°C it is converted to tridymite and above 1 470°C it becomes cristobalite. These temperatures are often encountered in calcining or sintering processes, and since tridymite and cristobalite are more harmful than quartz, extra precautions need to be taken at such operations[4].

Health effect of free crystalline silica

In order to exert a health effect on man the free crystalline silica should be present in the respirable fraction of the dust inhaled (< 7 μm particle size). The respirable fraction could be described as the portion of dust that would be able to penetrate the deepest areas of the lung, these being the alveoli (minute sacks in the lung where gas exchange takes place). The determination of exposure levels for quartz in mines would therefore be restricted to the respirable fraction of dust. With the exposure of the alveoli to free crystalline silica a fibrogenic type lung disease termed silicosis may develop[5]. Through this disease irreversible changes in the lung occur and thereby the functional capacity of the lung is reduced.

Methods of analysis

Wet chemical methods

One of the first methods to be described was the method whereby quartz was separated from impurities by phosphoric acid digestion[6]. A considerable amount of time and effort went into improving this method of analysis. A method frequently used nowadays is the method as described by Sweet *et al.* [7]. In this method the dust sample is placed in a beaker whereafter phosphoric acid is added and the mixture heated to a temperature of 220°C. At this temperature phosphoric acid dehydrates and is converted to pyrophosphoric acid which solubilizes most silicates while reacting only minimally with free crystalline silica. After cooling down, the mixture is filtered through a membrane filter and the liquid phase containing the contaminant silicates is discarded. The free crystalline silica on the membrane filter is brought into solution with hydrofluoric and boric acids whereafter it is reacted with an indicator reagent to form a coloured complex. The intensity of the colour will depend on the amount of free crystalline silica that was initially present in the sample. This intensity could be accurately measured in a spectrophotometer by measuring the absorption of light at a specified wavelength. For instance, if 1-amino-2 naphtol-4-sulfonic acid was used as indicator reagent, a blue coloured compound will be formed in the presence of silica and the intensity of this colour would be measured as the absorption at a wavelength of 820 nanometers (nm). To be able to relate this absorption to the amount of quartz present, use is made of a standard curve. Different amounts of pure quartz are weighed out and treated in exactly the same way as the sample. The absorption for each standard is obtained at 820 nm and a regression line is fitted through the points in the standard curve. A hypothetical standard curve is illustrated in **Figure 1**.

By fitting the measured absorption of the sample to the standard curve, the amount of quartz present could be read off. From this data, and from the weight of dust and volume of air sampled, the average quartz concentration at the position of sampling could be calculated and expressed in mg quartz/m^3 air.

Although the method is fairly sensitive and accurate, there are a number of disadvantages using this method for environmental quartz assessment. These include:

a) the method is time consuming and skilled laboratory personnel are required to perform the analysis;

b) potentially dangerous chemicals such as hydrofluoric acid are used in the procedure;

c) the sample is destroyed and cannot be used for any further analysis that may be required;

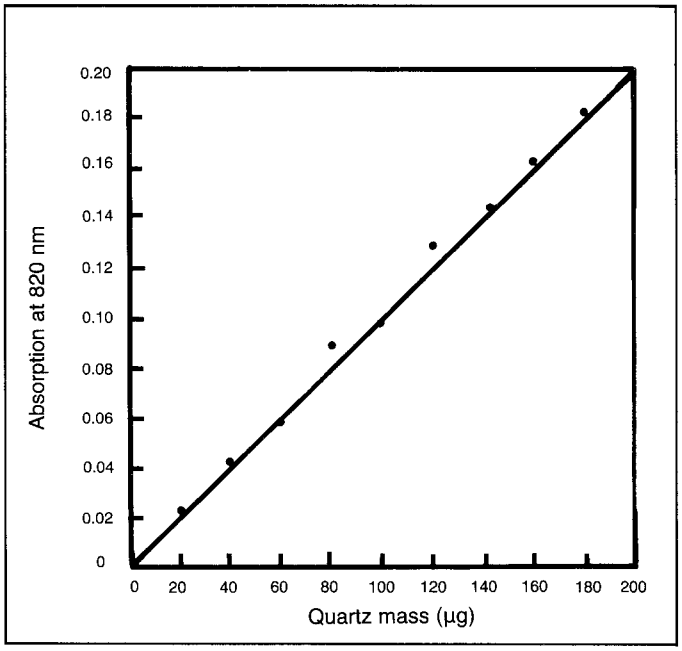

Figure 1 ***Standard curve for the spectrophotometric determination of quartz***

d) quartz, especially in the respirable size range, may be lost by its dissolution during the time dependent phosphoric acid digestion stage. Also, it is conceivable that some silicates may not be solubilized in the allotted time and, therefore may cause an erroneously high free silica result[8];

e) the method does not distinguish between the different polymorphs of free crystalline silica namely quartz, tridymite and cristobalite. Should the last two forms be present in the sample, an overestimation of quartz would be made.

X-ray diffraction

The X-ray diffraction (XRD) method of quartz analysis is perhaps the most extensively used of all methods. This method is attractive because it uses a small sample, is nondestructive, rapid, and all the individual mineral phases in a sample are potentially identifiable[1].

X-rays are produced whenever fast-moving electrons strike a substance. In XRD this stream of electrons is usually generated in a so called Coolidge tube (**Figure 2**).

When the filament (**Figure 2**) is heated to incandescence by means of a current supplied, electrons are emitted by the filament. These electrons are accelerated to the target by a difference of potential maintained between them. The target of general-purpose X-ray tubes are usually made of tungsten or molybdenum, but in the analysis of quartz a copper target is usually preferred. Copper radiation greatly improves resolution and sensitivity for quartz analysis[10]. The intensity of the X-rays coming from any tube depends

upon the element used as the target, the power supplied to the tube, and the difference of potential between the target and cathode. The X-ray diffraction instrument is furthermore so designed to allow only a single wavelength of X-rays to strike the sample. In the case of using a copper target, the wavelength produced is 1,5405 Angström (Å). When this beam of monochromatic X-rays strikes a sample of crystals (e.g. quartz), the X-rays will be diffracted (bent) at certain angles, the angle depending on the properties of the crystal. This concept is illustrated in **Figure 3**.

The angle of diffraction depends on the Bragg equation

$$\sin \theta = \frac{n\lambda}{2d}$$

where θ = angle of diffraction

n = an integer (1, 2, 3 – – –) known as the order number of the diffracted beam

λ = wavelength of the X-rays (1,5405 Å when a copper target is used in the X-ray tube)

d = distance of interplanar spacings in a crystal (d-spacings).

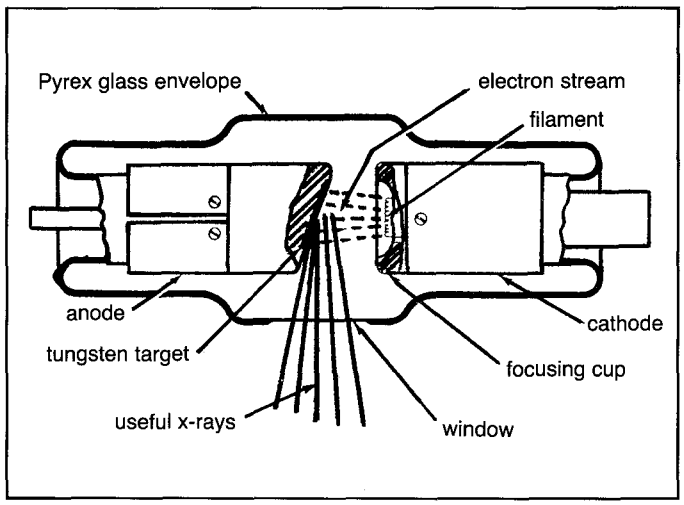

Figure 2 *The Coolidge type of X-ray tube*[9]

An example of a crystal being irradiated with X-rays is illustrated in **Figure 4**.

For quartz, the different d-spacings is known, and therefore the angles of diffraction (θ) can be calculated using the specific wavelength of the copper target X-ray tube. Therefore, should the detector be rotated around the sample, diffracted X-rays would be detected at different angles. The four most intense diffraction lines for quartz are at the d-spacings of 4,26, 3,34, 1,82 and 1,51 Å. Of these the 3,34 Å (θ = 13,3 Å for the copper target) is the line most often used for quantification of quartz. Should there be interference at this line from other elements, any

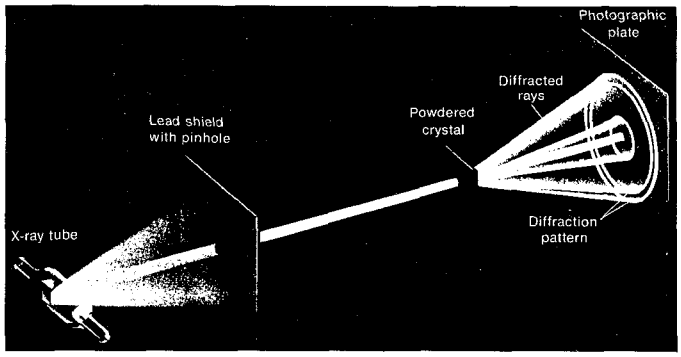

Figure 3 *The diffraction of X-rays by crystals*[11]

of the alternate lines (although less sensitive) can be used for the quantification of quartz.

One of the advantages of employing the XRD technique for quartz analysis is that direct on-filter analysis is possible. This provides a saving in sample preparation time and thereby reduces cost of analysis. However, this advantage will be negated should it be required to pool dust samples collected over a period of time for a certain area in the underground workings. Under these circumstances five dust samples would be collected over a period of six months, whereafter the samples would be pooled, ashed and redeposited on another filter for quartz analysis. The advantages and disadvantages of combining samples will be discussed in a later section of this paper.

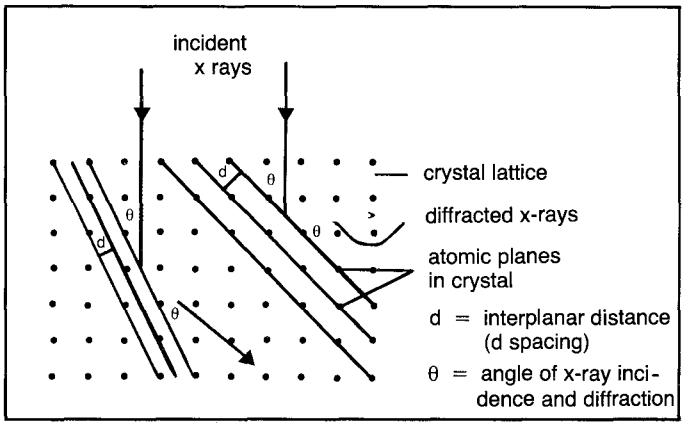

Figure 4 *Reflection of X-rays from sets of atomic planes within a crystal*

This principle of quantification of quartz is similar to the wet chemical methods, in that a standard curve needs to be determined, from which the amount of quartz present on the filter could be calculated (see **Figure 1**). The only difference is that the intensity of diffraction for a specific d-spacing is determined instead of absorption. To set up the standard curve, increasing amounts of pure quartz standard are deposited onto membrane filters, whereafter diffraction intensities are measured at the specific

diffraction angles (2θ). A typical scan of a dust sample obtained underground is illustrated in **Figure 5**.

From the graph the two main quartz peaks can be distinguished (d = 3,34 Å and d = 4,26 Å). By measuring the peak height (or peak area) the amount of quartz present on the filter could be calculated from the standard curves. (Individual standard curves need to be set up for each diffraction angle employed). The additional peaks observed in the figure are due to other minerals that are present on the filter.

Some of the problems encountered in analysing for quartz with the XRD method are as follows.

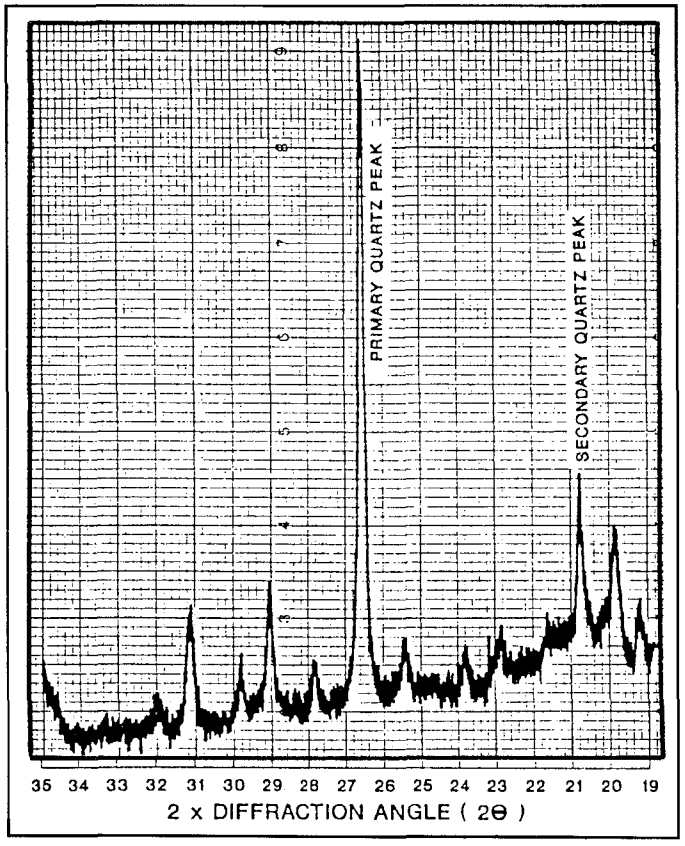

Figure 5 *A typical XRD scan indicating the 2 main quartz peaks*

Amorphous silica

Crystalline quartz particles contain an amorphous silica surface layer approximately 0,03 μm thick. As amorphous silica does not have an X-ray diffraction pattern, it can shield off diffraction from the crystalline particles, especially in the smaller particles (smaller than 1 μm diameter). Therefore, if the size distribution of the dust sample is too fine, underestimation of quartz is probable[12].

The effect of mass absorption

The intensity measurement of quartz in a very thin coating of dust is practically proportional to its weight because of the low X-ray absorption by the sample matrix, thus there is a linear relationship between quartz mass and diffraction intensity[13]. If this layer of dust becomes too thick, increasing absorption of X-rays by the sample matrix takes place and the response becomes non-linear. This problem could be overcome by applying correction factors (to be determined by every laboratory) or by making use of internal standards or a metal substrate standard[14].

Interference

A major limitation in the application of XRD analysis for quartz in environmental samples is the overlap of the quartz diffraction lines by diffraction lines from other components in dust. Such overlaps may result in either erroneously high or low results depending on whether the measured quartz line is completely or partly overlapped by a line from another phase present in the sample[15]. It is unknown what the extent of this problem will be in the South African mining industry, but Bradley[16] stated that the only mineral in samples from the South African gold mines likely to cause interference is muscovite. This however, needs to be confirmed by more detailed studies. Some other materials that will cause interference with quartz are mica, sillimanite, graphite and agonite. The interference due to graphite can be reduced by ashing of the filters[10]. If interference is a problem it can be overcome by using an alternative diffraction line of quartz, but with a decreased sensitivity. It is always necessary to cross-check using two or more quartz diffraction lines to verify that the peak detected is indeed a quartz peak[17].

In addition to line overlapping, the presence of specific elements in the sample (iron in particular) can result in appreciable X-ray fluoresence leading to high background intensity. This would adversely effect the detection limit for quartz determination, for the quartz peak may not be distinguishable from the high background noise. One would, in such a case, first have to dispose of the interfering element by acid digestion, thereby adding additional steps to the analysis and consequently adding to the time and expense of analysis.

Particle size

It is known that different diffraction intensities are obtained for different particle sizes of quartz crystals. Particles of 1 to 3 μm in size have the strongest diffraction intensity[12]. Diffraction intensity greatly decreases in coarse particles of more than 10 μm and also in very fine particles[14]. Because we only collect dust with a particle size distribution

in the respirable range (< 7 μm diameter) we are not concerned with the effect of larger particle size. It is, however, essential that the particle size distribution of the sample and the standards used be similar. Because the size distribution over the filter surface may also vary (larger particles tend to settle in the middle) it is necessary to irradiate as much as possible of the filter with the incident X-rays. To average out the effect of particle size distribution on the filter, use is made of a spinning sample holder[18].

Preferred orientation

A thin layer of quartz crystals on a smooth surface may exhibit a preferred orientation favouring the 3,34 Å line. As the deposition depth increases or the filter material becomes more porous, the orientation approaches random[19]. Rotation of the sample is helpful to even out the preferred orientation and non-uniform distribution, but their effects cannot be totally compensated for in this way[12].

Filter choice

The choice of filter material and pore size is a compromise between X-ray and dust sampling requirements. For X-ray analysis the requirements are low backgrounds under the quartz peaks, a linear calibration and dust deposit of high surface density: for dust sampling, high collection efficiency, low air resistance and moisture absorption, ease of handling, and strength[20]. The filter most commonly used for sample collection and direct on-filter quartz analysis is the silver membrane filter. The mass of silver filters is very stable and this assists in the accurate determination of total mass of sample collected. The silver filter also gives the least background and highest signal-to-noise ratio for the 3,34 Å diffraction line of quartz[21]. However, because of a large resistance over the silver membrane filter, one has to make use of a filter with large pore size (e.g. 5 μm).

In such an event, only 55 to 60 per cent of the estimated mass of quartz is presented to the X-ray beam. The balance of the quartz either penetrates through the membrane or is deposited in the filter matrix where the silver screens it from the X-rays. For these reasons it is often preferred to collect samples on a small pore size organic filter (e.g. 0,8 μm Cellulose nitrate), which has a tolerable filter resistance even with such a small pore size. The use of 0,8 μm pore size filters will ensure collection of all particles of size greater than 0,4 μm as well as the majority of particles of size 0,2 - 0,4 μm[22]. As the dust deposit builds up the efficiency of collection improves, and eventually all particles (even those < 2 μm) will be collected. One of the main disadvantages of using organic filters is the high background intensities at the primary quartz diffraction line. Removal of dust from the collection filter (e.g. by ashing of the filter) and redepositing on a small pore size silver membrane filter would optimize conditions for XRD analysis of quartz.

Keeping all of the above points in mind, and performing analyses meticulously, extremely accurate and sensitive measurements of environmental quartz content could be made by means of X-ray diffraction methods. The addition of computer control in analysis will further improve the method by reducing the manual effort required to check for and avoid interference. It will also reduce counting time per line per sample and improve throughput on routine mine dust samples and so reduce technician skill and professional supervision[23].

Infra-red (IR) spectroscopy

All matter continuously emits and absorbs electromagnetic radiation. This emission is a consequence of the continual motion of the elementary charged particles within the substance[24]. Infra-red spectrometers operate in a manner similar to that of visible ultraviolet spectrometers. A beam of infra-red radiation is passed through the sample and this beam is constantly compared with a reference beam as the frequency of the incident beam is varied. The spectrometer plots the results as a graph showing absorption versus frequency, wavelength or most commonly, wavenumber. The wavenumber is the number of cycles of the wave in each centimeter along the lightbeam as is measured in reciprocal centimeters (cm^{-1}). In their vibration, covalent bonds (e.g. the Si-0 bond in quartz) behave as if they were tiny springs connecting the atoms. When the atoms vibrate they can do so only at certain frequencies, as if the bonds were "tuned". Because of this, covalently bonded atoms have only particular vibrational energy levels. The excitation of a molecule from one vibrational energy level to another occurs only when the compound absorbs infra-red radiation of a particular frequency[25]. The infra-red spectra of even relatively simple compounds contain many absorption peaks and because of this, the possibility that two compounds will have the same infra-red spectrum is exceedingly small. An infra-red spectrum can be thought of as the fingerprint of a molecule.

When the sample to be analyzed is placed in front of the beam of infra-red rays a certain absorption pattern (infra-red spectrum) will be obtained. The infra-red spectrum for quartz is typically as is illustrated in **Figure 6**.

From the spectrum the three main quartz absorption bands can easily be identified. These are the bands at 800 cm^{-1} (12,5 μm), 780 cm^{-1} (12,8 μm) and 694 cm^{1} (14,4 μm). The other bands in the spectrum are due to matrix absorption by the potassium bromide, in which the

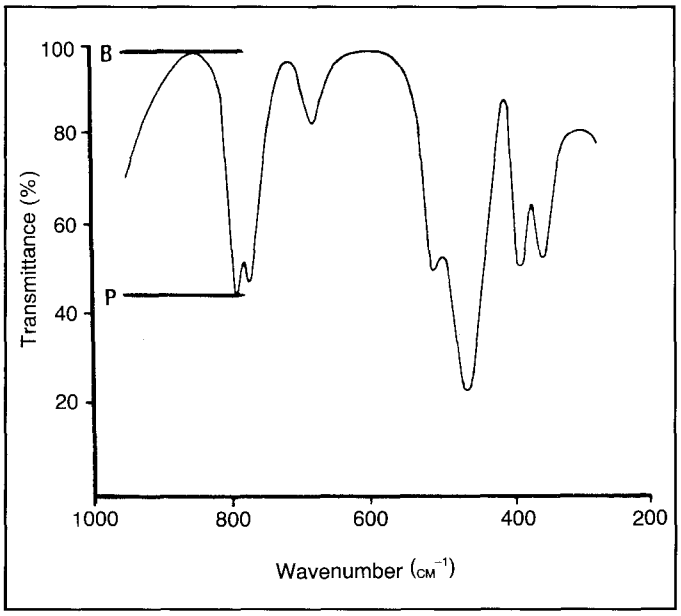

Figure 6 *IR spectrum of quartz in a potassium bromide matrix* [26]

quartz particles are suspended. The band at 800 cm^{-1} is the band of choice for quantification of quartz for it is the most intense of the three bands. It can be seen from the spectrum that use is made of percentage transmittance of the beam at the different frequencies of infra-red radiation. In order to perform quantitative analysis, percentage absorption needs to be converted to absorption by means of a simple calculation

$$A = \log \frac{Io}{I}$$

where A = absorbance

Io = value of baseline transmittance (line B in **Figure 6**)

I = value of peak transmittance (line P in **Figure 6**)

A standard curve, using varying amounts of pure quartz can be obtained (similar to **Figure 1**) from which the quartz content of the sample can be calculated.

There are two established infra-red methods, one based on the direct analysis of the membrane filter with the collected sample and the other, an indirect method involving the preparation of potassium bromide disks.

The filter choice is extremely important when using the direct method. Polyvinylchloride (PVC) filters are preferred for direct measurement because this material has a region of infra-red transparency in the area of the 800 cm^{-1}/ 780 cm^{-1} absorption bands[27]. To average out uneven distribution of dust on the filter it is suggested that a sample spinner be employed. It is also common practice to redeposit the dust obtained from a field sample onto a smaller diameter PVC filter. This will also ensure an even distribution. In addition, when dust is redeposited from a 37 mm diameter collection filter (such as used in the South African coal mines) onto a 10,4 mm diameter filter, a concentration effect of about 13 times is achieved[28]. This provides adequate sensitivity for analaysis with very small amounts of dust collected on single filter samples. In using the direct method of analysis one has to be careful when analyzing dark-coloured samples (e.g. those containing graphite or magnetite). These samples can act as black body radiators, resulting in up to 75 per cent attenuation of the quartz absorbances at 800 and 780 cm^{-1} [29].

The preferred method of analysis is often the KBr disc method. In this method the collection filter containing the sample is ashed in a low temperature radio frequency asher to destroy the organic material, whereafter the residue is mixed with KBr and the mixture is pelletized in a die with a 30 ton laboratory press. The infra-red spectrum of the pellet (thickness is about 0,85 mm) is then determined, whereafter the quartz content can be calculated as before[30]. These KBr pellets can be permanently retained. Potassium bromide is chosen as matrix because of its infra-red transparency in the area of quartz absorption bands. The method offers good sensitivity. The major disadvantage is that the procedures are time consuming and there is a possibility of sample loss during the isolation stages[27].

In general, some of the most common problems associated with the infra-red analysis of quartz are as follows.

Interference. It is important to note the spectral interferences on the quartz absorption bands due to the presence of other molecular species. Other crystalline polymorphs of silica such as cristobalite and tridimite, interfere with the infra-red determination of quartz[28]. These substances are unlikely to occur in coal or metalliferous mine dust, but are likely to be present in foundry operations. Quartz or amorphous silica may undergo conversion to cristobalite (and possibly tridymite) when exposed to excessive temperatures (as in foundry operations)[26]. Crystobalite appears to crystallize in the solidification of silicon melts in the range 1 000 – 1 100°C[31]. In case of interference from other crystalline silica variants, the 694 cm^{-1} peak can be used for quartz analysis. This peak is not subject to any interferences from other crystalline silica variants but is unfortunately less intense than the other absorption bands[26]. Organic materials in dust (such as diesel soot and coal) and several clay minerals such as illite, kaolinite and muscovite interfere with infra-red analysis. Much of this interference appears to be eliminated by ashing the sample[28]. A list of the most common interfering minerals is presented in **Table 1**.

Table 1 *Minerals interfering with IR quartz analysis*[32]

Mineral	Interfering peaks (cm^{-1})	Identifying peaks (cm^{-1})
Cristobalite	800	630, 500
Kaolinite	790, 750	910, 540, 470, 430
Muscovite	740	540, 480
Albite	780, 760, 740	780, 760, 740, 720, 650, 590, 530
Anorthite	780, 760, 740	780, 760, 740, 720
Orthoclase	760, 730	640, 590, 540

Particle size. If the particle size is equal to or greater than the wavelength at which the absorption is to be measured, the intensity of the peaks will decrease due to "non-wavelength" selective reflection and diffraction[33]. Therefore, for the analysis of quartz at 800 cm^{-1} (12,5 μm) the particles should not be larger than 12,5 μm. If only the respirable fraction of dust is to be collected, the effect of large particle size is not a matter of concern. It is, however, important that the size distributions of the samples and the standards be similar.

Amount of dust. As the amount of dust collected increases, broad band spectral differences begin to show in the 850 cm^{-1} region. With the amount of dust large enough, the 800 cm^{-1} peak will eventually be masked by these interferences. It is suggested that the optimum amount of dust on the filter be between 0,5 to 1 milligram[34]. These limits are easy to determine by individual laboratories performing quartz analysis.

A uniform path length (distance the beam travels through the sample) should be kept in infra-red analysis. Since this is not always possible, use can be made of an internal standard to illuminate this variable. If an internal standard is mixed with the sample prior to analysis (only if sample is to be redeposited or a KBr pellet is prepared), both will have the same path length. Therefore, if a ratio of the absorptions of the internal standard peak and the sample peak is used, the path length variable will cancel out[33]. Ideally, the internal standard would be a compound which would not be found in the dust sample, and which would have a sharp absorption band occurring at a point where the sample has a flat region of high transmission, and at a wavelength close to that used for measurement of quartz.

Although there are some difficulties associated with the technique, overall infra-red methods provide a powerful and sensitive technique for environmental quartz analysis. Computer processing gives provision for extensive scale expansion, elimination of the spectral components from an interfering matrix by background subtraction, and the removal of instrumental noise in trace level measurements. These all lead to an improvement in sensitivity. Furthermore with the introduction of Fourier transform infra-red spectroscopy, the method of quartz analysis by infra-red technique could be further optimized by improving on scan time, wavelength accuracy, sensitivity and limit of detection[35].

Advantages and disadvantages of sample combination

It has been proposed that five personal gravimetric samples be collected per area over a period of six months in the South African mining industry. These samples will need to be analysed for quartz content by either combining the five samples and performing only a single quartz analysis, or alternatively, each sample will have to analyzed individually. There are a few advantages and also some disadvantages for combining samples. These are summarized as follows.

Advantages

a) Combination will increase the effective sample size used for analysis therefore improving on sensitivity.

b) A chemical rather than mathematical average of quartz concentration is obtained.

c) By including an ashing step, interfering substances such as diesel soot and coal dust will be removed.

d) After combination and ashing of samples a choice can be made on the matrix used for redepositing (e.g. using a silver membrane filter with its low background noise for X-ray diffraction or using a KBr method for indirect IR analysis or a PVC filter for direct IR analysis after redeposition).

e) Should the avenue of sample combination be followed, the bottleneck of sample analysis would not be with the apparatus but with sample preparation. The implication of this is that only a single instrument (X-ray or IR) is required to perform analysis.

f) Redepositing of KBr disk preparation will most likely provide a more even sample distribution. (During field sampling the large particles tend to accumulate in the middle of the filter.)

g) It will undoubtably be cheaper to perform one combined analysis rather than five individual analyses.

h) Should interferences by other minerals occur on the main peaks, a larger sample size (as obtained by combining samples) will improve the resolution of the secondary peaks (in X-ray and IR methods) thereby

ensuring that an accurate result could always be obtained. Combination of samples also allows for extra analytical steps to be included in the analysis where necessary, e.g. where excessive iron has to be removed by acid digestion. This step could be performed directly after ashing and before redepositing or KBr disk preparation.

i) It allows for the utilization of an internal standard which will improve accuracy.

Disadvantages

a) If there was a sample in a series that differed totally from other samples (e.g. as a consequence of sampling error) this would not be recognized when all samples are combined.

b) An increased number of analytical steps in a procedure could possibly lead to increased operator error. This could, however, be minimized by painstaking attention to detail by the laboratory technician involved.

c) The method is destructive and individual filters could not be analyzed for other substances should the need arise.

d) The effectiveness of redepositing is questionable. However, using an internal standard will correct for any sample that may have been lost during the procedure.

e) A large number of skilled laboratory personnel have to be employed. This fact has to be weighed against additional instrumentation which would require a large initial capital outlay.

Possible scenarios regarding the analysis of mine samples

The responsibility of obtaining underground personal gravimetric samples lies solely with each individual mine. In the analysis of these samples there are several options. The first option is to perform all analysis on the mine. In such an event a fully equipped laboratory has to be established. Depending on whether X-ray or infra-red methods will be employed, the appropriate apparatus needs to be acquired. It may be a proposition for a number of mines in close proximity to one another to open a centralized laboratory. The economic viability of such a venture will, however, be totally dependent on the number of samples that need to be analyzed daily. The greater the number of samples analyzed, the more economically viable the proposition will become. Because of the expense of the apparatus required for quartz analysis, it is inadvisable that small concerns (individual mines) establish their own laboratory.

The second possibility is that sample preparation be performed at the mine and that actual quartz analysis be performed by an accredited laboratory. If the method of sample combination is preferred, the required input from the mine would be weighing of the samples after sample collection, ashing, combining and redeposition or KBr disk preparation. All that is required for this, is a laboratory equipped with fairly simple apparatus and task orientated, skilled laboratory technicians. The most expensive equipment needed is a balance (five decimal points of gram weight) and either a muffle furnace or a low temperature radiofrequency asher. Its own sample preparation would greatly reduce the cost to the mine for quartz analysis performed by an accredited laboratory.

The last possibility is that samples be directly dispatched to the accredited laboratory for sample preparation and analysis. This will substantially increase the analysis charge. However, in the case of small concerns with a small sample output this may be the choice that is economically the most viable.

Conclusions

It can be concluded that the analysis of quartz requires a fair amount of skill and some rather expensive equipment. Although all methods for quartz assessment suffer some deficiencies, there are currently two acceptable methods for quartz analysis on filter samples namely, X-ray diffraction and infra-red absorption. Because of the time of analysis and the probability of error, spectrophotometric methods involving colour complex formation are not suggested. When performing quartz analysis by X-ray diffraction, care should be taken in avoiding problems such as the effect of amorphous silica, mass absorption, interference from other minerals, particle size distribution and preferred orientation. Infra-red methods share the X-ray problems of mass absorption, interference and particle size distribution. Most of these problems could be overcome and then provide an accurate assessment of quartz. Computer processing will further assist in automating analysis and improving on sample throughput.

There are several advantages and disadvantages in combining samples from the same area taken over a six month period for quartz analysis. The advantages outweigh the disadvantages and it is therefore suggested that samples be combined before performing quartz analysis.

There are three options concerning the location of quartz analysis for the mining industry. These include a centralized laboratory for a group of mines to perform sample preparation and quartz analysis; the mine to perform sample preparation and an accredited laboratory to perform quartz analysis; or an accredited laboratory to perform

sample preparation and quartz analysis. There would be merit in investigating the extent to which Industry consensus could be obtained, if at all, on the various options available.

Acknowledgement

This paper is published with the permission of the Chamber of Mines Research Organization (COMRO).

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Diary of events

24/8/90	Indoor Radon, NACA.
10/9/90-14/9/90	MINESAFE INTERNATIONAL 1990, Perth.
10/9/90	Seventh Annual International Pittsburgh Coal Conference. Clean Coal Technology: Meeting Energy and Environmental Needs.
17/9/90-21/9/90	International Deep Mining Conference, Jhb, SAIMM.
24/10/90-26/10/90	1st IUAPPA Regional Conference on Air Pollution. CSIR Conference Centre. Tel: (012) 841-3816.
12/12/90-15/12/90	Safety Management, Amsterdam.
25/2/91-28/2/91	SME (U.S.A.) 120th Annual Meeting and Exhibit. Theme – Mining is alive – with emphasis on environmental management in the 1990's. Fax: (303) 973-3461.
26/5/91-30/5/91	International Symposium on Dust Explosions, Mine Fires and Mine Rescue hosted by Brunswick Mining and Smelting (Canada).