

X-RAY POWDER DIFFRACTOMETRY
PART II. MICROANALYSIS OF
CRYSTALLINE SILICA.

by

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1. INTRODUCTION

A major part of the analytical monitoring of occupational exposure to dust is devoted to establishing concentrations of crystalline silica (usually α -quartz) in workplace atmospheres. The methods most widely used in characterising and analysing silica are infrared spectroscopy and X-ray diffraction. Although X-ray diffraction, as an analytical technique is well established, analyses of dust samples collected by filtering workplaces atmospheres presents a special problem; namely, the carrying out of quantitative analyses on small samples (often < 1 mg).

Therefore, the objective of the project described in this report was to develop an analytical procedure for routinely analysing such small quantities. The project was divided into three parts:

- (a) sample preparation
- (b) X-ray diffractometry
- (c) data processing

2. REQUIREMENTS OF THE TECHNIQUE

Detection limit

In order to attain optimum accuracy it is clearly desirable to reach detection limits as low as the technique will permit. In any case, the method must satisfy a minimum requirement with respect to a quartz detection limit. Thus, the minimum amount of quartz associated with an 8 hours exposure may be calculated by defining a minimum % quartz (Q) content in respirable dust and substituting this value in the threshold limit value (TLV):

$$\text{norm}_L (\text{mg}/\text{m}^3) = \frac{25}{Q + 5}, \text{ maximum } 5 \text{ mg}/\text{m}^3$$

This formula is specific to the sedimentation process undergone by the sample which selects a fine fraction $\leq 5 \mu\text{m}$ in average diameter. If one

chooses 0.5% quartz as the minimum then the (norm)_L TLV value is 4.5 mg/m³. The amount of quartz collected by a Casella Dust Sampler over 8 hours in a dust atmosphere at this TLV is 20 µg, assuming an airflow of 1.9 l/min. Setting the minimum % quartz value as low as 0.5% gives a margin of safety. The minimum requirement is therefore a detection limit of 10 - 20 µg quartz based on the most intense quartz reflection ((101) line).

3. QUANTITATIVE X-RAY POWDER DIFFRACTOMETRY

The accuracy of quantitative X-ray powder diffractometry depends on the following:

- (a) particle size distribution in the sample;
- (b) random orientation of the crystallites comprising the sample;
- (c) mass absorption (attenuation by the sample of the reflected X-ray beam);
- (d) intensity of the primary X-ray beam and its focal area on the sample;
- (e) time taken to scan over the reflection of interest.

(a) Particle size

As pointed out in Part I the sizes of the individual reflecting crystals play an important rôle. Thus, when the diffracting crystal is large it contains a great many parallel planes and it is essential that the Bragg condition be precisely satisfied if the X-rays reflected from different points on these planes are to reach the detector in phase. If the Bragg condition is fulfilled the diffraction peaks are sharp. However, as the crystallites decrease in size the resulting small number of reflecting

planes relaxes this condition. A progressive decrease in crystallite size manifests itself in a concomitant broadening of the diffraction peak, until, in the limit, the peak has broadened so much that it merges into the background. It is important to realise that the area under the peak remains constant for a given amount of sample, and that it is the peak height which is particle size dependent. For this reason it is correct to use peak areas rather than peak heights in the analyses.

An ideal sample for X-ray powder diffraction is completely homogeneous over an area of 1 μm with constant particle size between 1 and 50 μm and which does not exhibit preferred orientation or tension. Samples of interest for occupational hygiene contain particles $\leq 5 \mu\text{m}$ in average diameter.

(b) Preferred orientation of crystallites

If the crystallites of a powder are not randomly oriented and possess a needle or plate-like morphology there is a tendency for them to lie such that the plate is perpendicular to the rotation axis of the sample. This results in a larger number of crystal planes parallel to the plate and a corresponding increase in intensity of the reflections from these planes relative to others. α -Quartz exhibits a tendency to cleave parallel to the (101) planes. Consequently, there is a preference for such crystallites to lie with their (101) planes parallel to the supporting surface if the latter is flat and smooth. This effect should be borne in mind since it results in an increase in intensity of the (101) reflection relative to other reflections; for example, (100) which will be correspondingly weakened.

(c) Mass absorption

The amount of radiation passing through a specimen decreases as the specimen's thickness increases. The intensity of the transmitted beam (I) is related to the intensity of the incident beam (I_0) by

$$I = I_0 e^{-\mu t}$$

where t = sample thickness (cm) and μ = linear absorption coefficient (cm^{-1}). The linear absorption coefficient can be calculated from a knowledge of the chemical composition of the powder, its density and a table of mass absorption coefficients (μ_m). These coefficients are constant for a given element and X-ray wavelength.

$$\mu_m = \frac{\mu}{\rho}$$

where ρ = density of the element. The general expression for the absorption coefficient of a matrix is:

$$\mu = \sum_i (\mu_i w_i)$$

where μ_i and w_i are individual mass absorption coefficients (μ_m) and weight fractions. In the case of quartz (SiO_2) the atomic weights of Si and O are 28 and 16 respectively giving a molecular weight of 60.

$$w_O = 2 \times 16/60 = 0.533$$

$$w_{\text{Si}} = 28/60 = 0.467$$

The mass absorption coefficients for $\text{CuK}\alpha$ radiation are:

$$\mu_m(\text{Si}) = 63; \mu_m(\text{O}) = 12$$

Therefore,

$$\begin{aligned} \mu(\text{SiO}_2) &= (63 \times 0.46) + (12 \times 0.533) \\ &= 29.4 \quad + \quad 6.4 \\ &= \underline{35.8} \end{aligned}$$

The absorption problem may be tackled by either ensuring that the specimen is thin enough so that absorption may be ignored, or corrected

for by introducing an internal standard in order to measure the alteration. The latter method enables correction for absorption to be made without the need to calculate the absorption coefficient.

Example:

To determine the weight fraction of α -quartz (SiO_2) in a specimen. To correct for the unknown absorption in this specimen, 200 mg KCl are added to 1000 mg of the sample.

| | <u>pure</u> <u>material</u> | <u>sample</u> <u>+ KCl</u> |
|---|--------------------------------|-------------------------------|
| intensity of α -quartz line (counts) | 150 000 | 4 720 |
| " " reference | 100 000 | 20 000 |

The values of the mass absorption coefficients for CuK α can be taken as:

$$K = 150: \text{Cl} = 106: \text{Si} = 63: \text{O} = 12$$

$$\frac{I_m^a}{I_o^a} = \frac{C_a \mu_o^a}{\mu_m}$$

where I_m^a and I_o^a are intensities measured in the sample and pure material respectively, C_a is the weight fraction of compound a in the sample, μ_o and μ_m are mass absorption coefficients for Cu K for the pure substance a and the sample.

A similar equation can be set up for the reference line:

$$\frac{I_m^b}{I_o^b} = \frac{C_b \mu_o^b}{\mu_m}$$

If this is applied to sample after adding KCl, μ_m can be given the following equation:

$$C_a^1 = C_b \frac{I_o^b}{I_o^a} \frac{\mu_o^b}{\mu_o^a} \frac{I_m^a}{I_m^b}$$

where C_a^1 applies to the concentration after adding KCl. Thus, in the original sample the concentration was:

$$C_a = \frac{1200}{1000} \cdot \frac{200}{1200} \cdot \frac{100\ 000}{150\ 000} \cdot \frac{4\ 720}{20\ 000} \cdot \frac{\mu_o^b}{\mu_o^a} = 0.0315 \frac{\mu_o^b}{\mu_o^a}$$

for quartz $\mu_o^a = 36$ and for KCl $\mu_o^b = 129.0$

Therefore $C_a = 1.117 = 11.7\%$

The experimental procedure used in the above example presupposes that enough sample is available for the mixing and grinding steps.

For dust collected on filters, only a limited amount of sample is available and this procedure is not practicable. An alternative method is that proposed by Leroux. This is the silver filter method. The filter is both support and internal standard for the correction of absorption. The procedure adopted at this Institute is described further on.

(d) Intensity of primary beam

The production of X-radiation is a random process the distribution of which follows a Poisson profile, which can conveniently be approximated to a Gaussian distribution provided that the number of counts (intensity) is sufficiently large. The distribution is therefore symmetrically arranged about a mean value N_0 and the standard deviation

given by $\sqrt{N_0}$, of approximately by \sqrt{N} . Clearly, the precision of a measurement increases with increase in intensity. In other words, sensitivity is directly related to the output from the X-ray tube. Output is restricted by characteristics of the tube. For given type of tube, operation at maximum output restricts its life thereby increasing the cost per analysis. Accordingly, a compromise between cost and sensitivity is chosen. Another factor related to the tube type is the sample area irradiated by the primary beam. The tube used in this work is of the Cu-anode type. There are three types of Cu tube currently available. These are:

- (i) fine focus, 0.4 x 8 mm² focal spot, maximum power rating 1500 watt,
- (ii) normal focus, 1x 10 mm² focal spot, maximum power rating 2000 watt,
- (iii) broad focus, 2x 12 mm² focal spot, maximum power rating 2700 watt.

The maximum energies per square millimetre irradiated sample area are 469, 240 and 112 watt/mm² for fine, normal and broad focus respectively. The tube chosen in this work is the broad focus type because it permits analysis of large sample areas, i.e. increases the number of crystallites in a position to diffract. A disadvantage, which however is usually not serious for powder diffractometry, is that resolution decreases on going from fine to normal to broad focus.

The broad focus tube is not operated at its maximum power rating (2700 watt), but is run at 50 kv; 30 mA (1500 watts). The disadvantage with the lower rating is the corresponding reduction in intensity by the ratio $15/27 = 0.55$, and sensitivity by $\sqrt{0.55} = 0.74$.

(e) Scanning speed

An important factor, especially in connection with quantitative analysis, is the time taken to scan over the peak of interest. For

example, the area of the peak (counts) is doubled on halving the scanning speed, (Table 3, p. 23 in Part I) with a corresponding increase in sensitivity of $\sqrt{2} = 1.44$. Hence the lower the scanning speed the more accurate the measurement. The minimum scanning speed attainable is $1/8^\circ 2\theta/\text{min}$. but that used in all the analyses is $1/2^\circ 2\theta/\text{min}$. so as to reduce the time per analysis.

4. X-RAY ANALYSIS

From the foregoing discussion it is shown that the accuracy of quantitative X-ray diffractometry mainly depends on the mass absorption coefficient of the sample, particle size distribution, and random orientation of the crystallites, in addition to instrumental factors. The problem as to particle size is reduced when examining sedimented samples ($\ll 5\mu\text{m}$). Therefore, mass absorption is the most significant source of error although the problem may be reduced or avoided by using only small amounts of sample (in our case $< 500 \mu\text{g}$). However, provided that absorption effects can be corrected for, sensitivity is increased by using larger amounts (e.g. 1.0. mg) of sample. One way is to deposit the sample on a silver filter and correct for absorption by means of a theoretical equation using the attenuation of the silver reflections. This method has disadvantages. The filter has to be handled several times because the silver (111) or (200) reflections must be registered before and after sample deposition, thereby increasing the risk of damage. Silver filters are inelastic and are therefore particularly vulnerable to damage and the attendant errors associated with the intensity measurements. A number of different filter types have been investigated with the hope of being able to solve this problem. These filters were studied in combination with a silver filter as standard in order to make use of the attenuation of the silver intensities as already described. Of the various filter types, the Nuclepore filters were found satisfactory. Advantages of this procedure are:

- (i) Time saving - only a single measurement of Ag (111) or (200) reflection is necessary for each sample because the same silver filter is used in all analyses.
- (ii) Nuclepore filters are both elastic and mechanically strong with small risk of damage through repeated handling. This is confirmed by the consistent measurements obtained from filters which have been handled repeatedly.
- (iii) The diffraction backgrounds and absorption arising from the filters are low, (although not as low as that from the silver filters), because Nuclepore filters are only 10 μm thick.
- (iv) Nuclepore filters are cheaper than silver filters. Reduction in cost per analysis in Nkr 4 - 5.0.
- (v) Silver filters have an open structure which results in small particles being shaded from X-radiation and hence reduced intensity. This effect is absent in Nuclepore filters because of their even surface. Tables 1 and 2 show that the effects of preferred orientation are not observed for the amounts of dust examined.

The current procedure is to sediment dust, select the fraction $\leq 5 \mu\text{m}$, suspend in water and filter aliquots of suspension (so as to deposit 0.4 - 2.0 mg dust) through a Nuclepore filter (diameter 25 mm, pore size 0.8 μm). This part of the procedure is explained in detail further on. after drying, the filter is placed on a 25 mm silver filter used as a standard, (or, alternatively a silver plated sample holder), put into the diffractometer and the relevant reflections measured. In order to reduce the effects of preferred orientation and to increase the number of crystallites brought into reflecting positions the sample is rotated.

It is important that the error (standard deviation, σ) connected with placing the sample holder into the diffractometer is as low as possible so that consistent measurements may be made. The precision in our procedure has been significantly improved (from $\sigma = 1.2$ to 0.3%) by using a sample holder developed at this Institute. Silver plating the sample holder so that the silver filter can be dispersed with would be an additional improvement. In this connection it is important to note that the height of the dust sample surface with respect to the X-ray focus is important. That is, the dimensions of the sample holder are critical. If the dimensions are not optimised two effects are observed:

- (i) reflection peaks are shifted from the correct 2θ value, being either too low or too high depending on whether the sample is too low or too high respectively.
- (ii) a concomitant decrease in intensity.

Thus, maximum intensity coincides with the correct 2θ value which in turn is critically dependent on the dimensions of the sample holder which govern its height. In fact differences as small as 50 - 100 μm produce observable effects.

Apparatus

Nuclepore filters (25 mm diameter, 0.8 μm pore size), 15 ml funnel with sintered glass base (Millipore xx.10.125.14) with filtering flask xx.10.025.50. Sedimentation apparatus (Jahr). Philips X-ray powder diffractometer with vertical goniometer, sample spinner, proportional counter and graphite crystal monochromator. A broad focus copper anode X-ray tube (PW 2253/20) was used because, as previously mentioned, it irradiates an area of 24 mm^2 for a normal focus tube. At 1500 W (15 kW: 30 mA), with a large amount of sample (1.0, 2.0 mg) and correction for mass absorption, one can detect 0.5 (1.0 mg sample) - 0,3% (2,0 mg sample) quartz (based on (101) peak) in mixtures depending on the various components present. Divergence slits of 1° ($20 - 40^\circ 2\theta$) and 2° ($40 - 60^\circ 2\theta$) together with a 1° receiving slit were used.

Sample preparation and calibration

The X-ray diffractometer is first calibrated for quartz analyses by measuring the intensities of reflections from a series of filters containing known quantities of a standard quartz sample. The standard is designated Fyle quartz (from Sweden) and is used in Scandinavia. The standard is sedimented and the fine fraction $\leq 5 \mu\text{m}$ average diameter selected. A standard aqueous stock suspension is prepared by taking 100 mg Fyle quartz and making up to 500 ml. This suspension is shaken vigorously and a 5 ml aliquot withdrawn and diluted to 250 ml. The new suspension, which contains 4 μg quartz/ml, is shaken and a range of different aliquots withdrawn. Each aliquot is transferred to the filtration apparatus (a few ml water being first added to the funnel) and the solution filtered through the Nuclepore filter by applying suction.

The volumes selected were:

| <u>Aliquots (ml)</u> | <u>α-SiO₂ (μg)</u> |
|----------------------|---|
| 2.5 | 10 |
| 6.0 | 24 |
| 9.0 | 36 |
| 12.0 | 48 |
| 25.0 | 100 |
| 30.0 | 120 |

The filters were dried overnight in an oven maintained at 80°. Figure 1 shows a calibration curve based on 28 filters (each point represents the average of 4 filters).

Routine dust samples

Two procedures have been used to analyse routine samples. The first method is in conjunction with the infrared analysis of quartz and the second is similar to the procedure for preparing calibration samples.

(101) - reflection

$\times 10^3$ counts

75

50

25

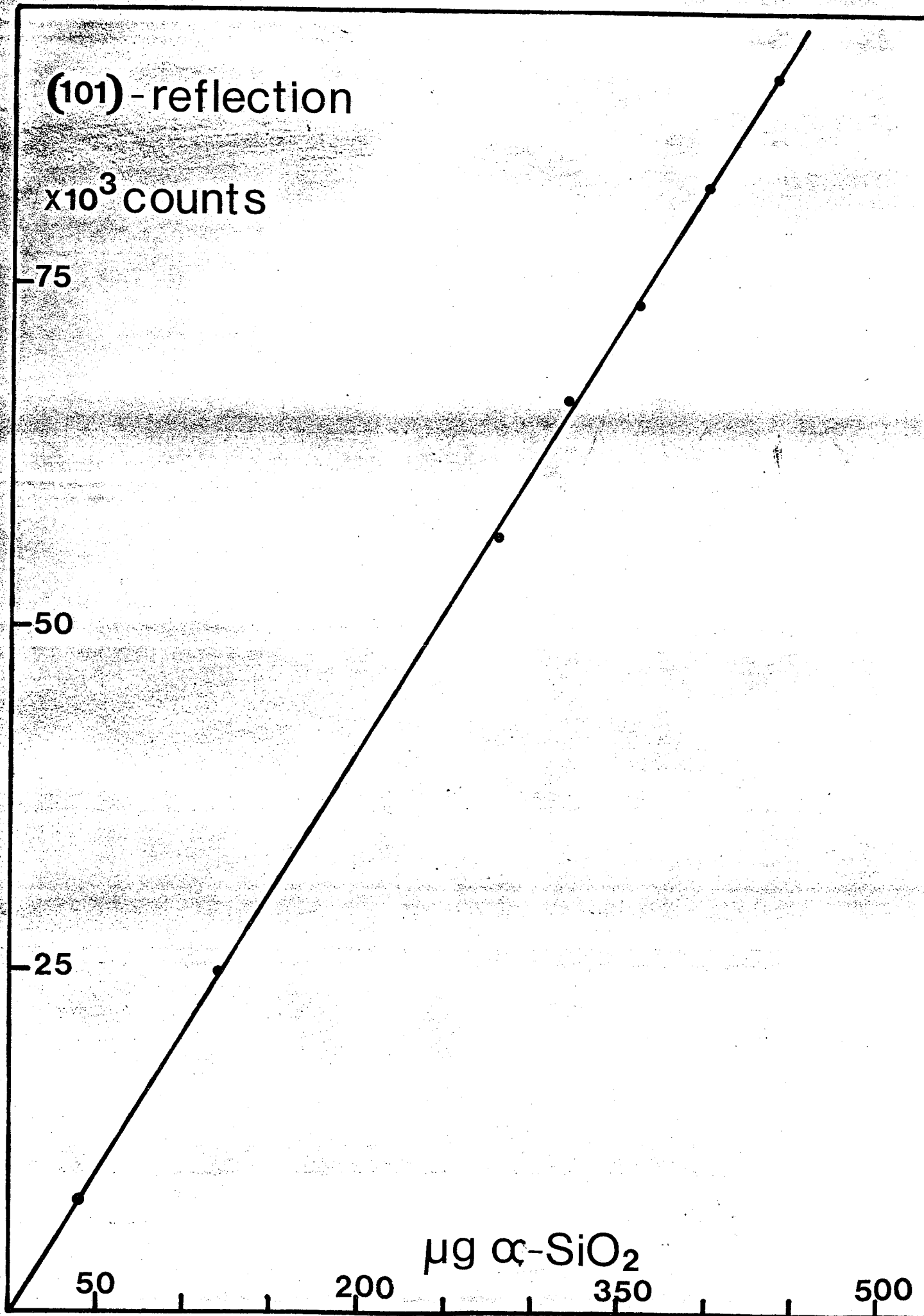
50

200

350

500

$\mu\text{g } \alpha\text{-SiO}_2$



The latter procedure is the more accurate because of the larger amounts of dust samples initially weighed out. However, the first method does have the advantage of allowing both IR and X-ray analyses to be made on an identical specimen of a given sample.

(1) Combined Infrared and X-ray analysis

The sample is sedimented (if > 40 mg) and the fine fraction with particle size $\leq 5 \mu\text{m}$ taken for analysis. Of this, 1.2 - 1.4 mg are mixed with potassium bromide in the ratio 1:300 and a KBr-disc prepared (See Report for details of IR-procedure). After the IR analysis the KBr discs (which contain 1.0 mg dust) are decomposed in water (15 ml) thereby releasing the dust samples. These are then filtered as described above and the filter analysed for quartz by X-ray diffraction.

(ii) Samples solely for X-ray analysis

Dust (10) mg is suspended in 100 ml water and aliquots (e.g. 10, 20 ml) transferred to filters, as described above, giving 1.0 and 2.0 mg deposits respectively.

X-ray analysis

- (i) Instrumental drift is corrected for by counting over a Ag (111) or (200) reflection without a Nuclepore filter.
- (ii) Quartz (100), (101), (112) and Ag (111) or (200) intensities are measured by scanning and counting over the peaks at a constant angular velocity of $\frac{1}{2}^\circ 2\theta/\text{min}$. Backgrounds are counted before and after the peaks.
- (iii) The quartz and Ag-intensities (corrected for instrumental drift) are fed into a computer program which corrects for mass absorption by means of the theoretical equation:

TABLE 1

Check of calibration contained in absorption correction programme. X-ray samples (1 - 5) prepared by dissolving IR - KBr discs in water and filtering released dust through Nuclepore filters. This permits direct comparison of IR vs X-ray.

| Nominal % α -SiO ₂ in SiO ₂ /Al ₂ O ₃ mixtures | % α -SiO ₂ IR | % α -SiO ₂ (σ) X-ray (not corrected for absorption) | % α -SiO ₂ (σ) X-ray (cor- rected for absorption) |
|---|------------------------------------|---|---|
| 5 (1.0 mg) | (9.3) | 4.1(1) | 4.98(1) |
| 15 (1.0 mg) | 17.4 | 12.36(6) | 15.15(7) |
| 30 (1.0 mg) | 29.8 | 25.8(6) | 31.7(7) |
| 50 (1.0 mg) | 47.0 | 41.03(2) | 49.65(2) |
| 75 (1.0 mg) | 70.3 | 60.67(4) | 74.17(5) |
| 100 (1.0 mg) | -- | 77.08(3) | 92.70(5) |
| 100 (1.0 mg) | -- | 78.71(5) | 98.80(5) |
| 100 (2.0 mg) | -- | 77.8(5) | 102.8(6) |
| 100 (2.0 mg) | -- | 76.9(4) | 101.8(6) |

$$I_{ij} = \frac{2k_{ij}}{A L \sin \theta_{Ag}} \cdot 1 - \exp(-L \sin \theta_{Ag} \operatorname{cosec} \theta_{ij} M_j)$$

where: -

I_{ij} = intensity of diffraction line i of phase j .

M_j = weight of phase j on the filter.

k_{ij} = constant - dependent on conditions of analysis and diffraction line i .

A = irradiated sample area.

θ_{Ag} = angle of incidence of X-ray beam associated with Ag diffraction line.

θ_{ij} = angle of incidence of X-ray beam associated with phase j diffraction line.

$$L = \ln \frac{I_{Ag}^0}{I_{Ag}}$$

where I_{Ag}^0 and I_{Ag} are diffraction intensities for the Ag line before and after a Nuclepore filter is placed on the Ag-filter.

5. COMPARISON BETWEEN X-RAY AND INFRARED

The physics underlying infrared spectroscopy and X-ray diffraction are so different that it is hardly surprising that the two methods, under certain circumstances are not always in accord. It is important to bear in mind that these techniques respond differently to particle size. Thus, infrared spectroscopy is increasingly sensitive to decreasing

particle size - the upper size limit being about 15 μm ; particle size dependency of X-ray diffraction goes in the opposite direction and relative line intensities increase with increasing particle size (range 1 - 50 μm).

Hence, if an X-ray analysis result is larger than the infrared result it may well be that the particle size distribution within the sample does not match that of the standards used to calibrate the two methods. It is possible to obtain results of 0% (infrared) and $> 100\%$ (X-ray) for unsedimented samples. However, for sedimented samples good agreement is invariably obtained because the particle size distribution ($\leq 5 \mu\text{m}$) then approaches that of the standard.

To a certain extent, use may be made of the differing size selectivities of the two methods. A combined infrared and X-ray analysis of an unsedimented sample provides information on (a) total α -quartz content (X-ray and (b) respirable α -quartz fraction (infrared). For such samples, analyses in reasonable agreement could indicate that most of the quartz present matches closely that in the standards ($\leq 5 \mu\text{m}$).

6. INTERFERING PHASES

Removal of interfering phases using orthophosphoric acid and a decomposition bomb

In common with many other analytical techniques the lines produced in infrared spectrascopy and X-ray diffraction are sometimes subjected to interference from other materials. In the case of X-ray diffraction several minerals give diffraction peaks which overlap the major quartz (101) peak. These minerals include micas (biotite, muscovite), potash, feldspars (microcline, plagioclase), sillimanite, mullite, graphite, iron carbide and zircon. In order to check for the presence of interfering lines the alternative quartz (100) and (112) peaks should be measured and their intensity ratios with the major peak compared with those of the standard.

A method which we have found very useful in many cases is to decompose non-quartz phases in a teflon-lined bomb using orthophosphoric acid at 250°. A standard decomposition time of 30 minutes is used.

7. INTERLABORATORY CALIBRATION

One question which arises during development of an analytical procedure is: how reliable are the analyses? A way of obtaining an objective evaluation is to participate in interlaboratory calibrations. Such intercalibrations are particularly useful because they enable each individual laboratory to compare and contrast analyses and thereby provide information on individual accuracies and precisions.

Immediately after X-ray diffraction procedure described in this report was developed, the Institute took part in a Nordic intercalibration of crystalline silica analyses.

The results of this intercalibration, summarised in Table 2, confirm that the procedure adopted here is capable of yielding satisfactory results.

8. COMPUTER PROGRAMME FOR ABSORPTION CORRECTION

In order to avoid errors associated with routine manual reduction of primary data and to save time, the absorption correction equation was stored in the Institutes computer system. This programme is listed on page 18.

Tabell 2: Results from Intercalibration of Quartz Analyses
(%-Quartz).

| LAB \ DUST | QUARRY | QUARRY + FYLE KVARTS | FOUNDRY | FOUNDRY + FYLE KVARTS | MINE | METHOD* |
|------------------|------------|-------------------------|-------------|--------------------------|------------|---------|
| A | 4 | 23 | 12 | 23 | 6 | R |
| B | 6 | 24 | 15 | 24 | 8 | R |
| C | 9 | 22 | 10 | 19 | 4 | IR |
| D | 6 | 22 | 12 | 21 | 5 | R |
| E | 9 | 17 | 14 | 23 | 8 | R |
| F | 14 | 19 | 8 | 15 | 5 | IR |
| G ₁ | 6 | 22 | 13 | 20 | 6 | R |
| G ₂ | 6 | 22 | 11 | 17 | 2 | IR |
| H | 7 | 22 | 10 | 19 | 5 | R |
| I | 5 | 19 | 10 | 19 | 6 | R |
| ASS ₁ | 7 | 23 | 10 | 19 | 6 | R |
| ASS ₂ | 7 | 21 | 8 | 16 | 5 | IR |
| mv | 7,2 2,6 | 21,3 2,0 | 11,1 2,2 | 19,6 2,8 | 5,5 1,6 | |

* IR = infrared; R = X-ray

G₁
= YHI

G₂

```

PROGRAM INTCOR
3 CHARACTER ITEST *1
4 400 INPUT (1) LINE, FIIJ, FIAG
5 FIOAG=832633
6 100 FORMAT(IX,*LINE = *,13,* KIJ = *,F6.2,* IIJ = *,F7.0,
7 1* IOAG = *,F7.0,* IAG = *,F7.0)
8 IF(LINE.EQ.112)GOTO 3
9 IF(LINE.EQ.101) GO TO 4
10 IF(LINE.EQ.100)GOTO 5
11 3 THETA=0.4373721
12 A=2.32729
13 FKIJ=0.0
14 GOTO 7
15 4THETA =0.2329975
16 FKIJ=59.02
17 A=2.2870397
18 GOTO 7
19 5THETA =0.1823838
20 A=2.152534
21 FKIJ=6.6633
22 7 FL=LOG(FIOAG/FIAG)
23 WRITE (1,100) LINE, FKIJ, FIIJ, FIOAG, FIAG
24 CSX=1.0/SIN(THETA)
25 T=FIIJ*A*FL*0.326888
26 B=2*FKIJ*(1-EXP(-FL*0.326888*CSX))
27 FM=T/B
28 WRITE (1,101) FM
29 101 FORMAT (IX,*MIKROGRAM, KVARTS =*,F8.2)
30 WRITE (1,200) FL
31 WRITE (1,200) CSX
32 WRITE (1,200) T
33 WRITE (1,200) B
34 WRITE (1,200) THETA
35 200 FORMAT (IX, F10.5)
36 WRITE (1,103)
37 103 FORMAT (/,IX,* HVIS SLUTT ANGI S ELLERS CR :*,/)
38 READ (1,104) ITEST
39 104 FORMAT (A1)
40 IF (ITEST.EQ.'S') GO TO 300
41 GO TO 400
42 300 END
43 EOF
44
45
46
47

```