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Tittel: Application of X-ray Diffraction in Occupational Health

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Sammendrag:

Metodebeskrivelse for røntgen pulverdiffraktometri innen yrkes-
hygiene.

Et bidrag i:

"Users Reference Manual in X-ray Powder Diffraction" (1985),
Pilips, 7600 AA Almeto, Netherlands".

Stikkord:

Røntgendiffraksjon
Kvantitativ analyse
Støvprøver
Silika

Key words:

X-ray diffraction
Quantitative analysis
Dust samples
Silica

APPLICATION OF X-RAY POWDER DIFFRACTION
IN
OCCUPATIONAL HEALTH

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Samples

Main object is to investigate health-related problems in working atmosphere, dust particles related to lung diseases.

Samples thus include :

Raw materials
Dust on filters :
 a) stationary sampling
 b) personal sampling
Biological tissue

Sample preparation

Raw material : Grinding, if necessary sedimentation in water to obtain fraction $< 5 \mu\text{m}$ grain.

Filter : Sedimentation if enough sample ($> 20 \text{ mg}$).
 Cyclone during sampling gives fraction
 $< 7 \mu\text{m}$ grain.

Tissue sample : fraction $5-7 \mu\text{m}$ grain

Samples are filtrated with Nuclepore membrane filters (pore size $0.8 \mu\text{m}$) for quantitative measurements. A silver filter is used as an external standard, where the decrease in the intensisty of a silver diffraction line is used for matrix absorption correction.
Filter size : 25 mm; Sample size : 15 mm.

Equipment : Vertical goniometer, fixed divergence slit, graphite crystal monochromator, broad-focus Cu-tube, PW 1710/APD 1700 software system.
Specimen rotation.

Procedure

Qualitative : Sample rotation, scan range $5 - 90^\circ 2\theta$, for identification.

Quantitative : Sample rotation, determination of quartz and cristobalite.
Integrated intensities are measured for :
quartz (100) and (101); cristobalite (101);
silver (111).

Data reduction

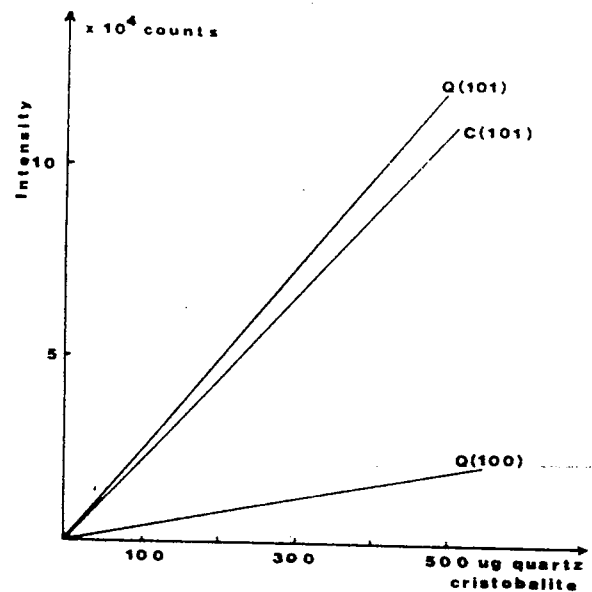
Peak	Q(100)	Q(101)	C(101)	Ag(111)
2 θ range	20.4-21.5	26.0-27.3	21.5-22.6	37.2-38.7
n, routine	3	3	3	1
n, calibration	5	5	5	10

A correction factor for day to day fluctuations is found by measuring the I_{Ag} and relate it to I_{Ag}^o (standard).

An absorption factor is found by relating the measured I_{Ag} with and without sample.

Calibration

Samples of 0.3 - 0.7 mg standard quartz (Fyle-quartz, Sweden) is deposited on Nuclepore filters. Absorption is neglected.



Future development

Include quantitation of other dust types, i.e. mica, talc, graphite.

Direct analysis on sampling filters, including absorption correction.