

Client: Nagrom
Job number: 17_0125
Sample: 17_0125_01
Client ID: **Tail Dam Fines Respirable Silica**
Date: 03/02/2017
Analysis: Respirable alpha-quartz concentration analysis by x-ray diffraction (XRD) using the modified SWeRF method

Sample Preparation

The sample was supplied to Microanalysis Australia as a bulk sample.

The respirable fraction was abstracted from suspension by settling and decantation, and the abstracted particle size, composition and morphology was verified by scanning electron microscope (SEM) for equivalent aerodynamic diameter (EAD).

Once the equivalent aerodynamic size was verified, the abstracted fraction was analysed qualitatively and quantitatively by x-ray diffraction to assess the alpha-quartz concentration.

Analysis

The laser diffraction size distribution analyses were conducted using a Malvern Mastersizer MS2000 calibrated using QAS3002 certified reference material and certified within specification. The analyses were conducted following ISO13320-1:1999.

For the sedimentation, the time for a specific fall height for PM4 (EAD) particles was calculated using Stokes Law. The samples were then homogenised and allowed to settle for the calculated time before the supernatant was decanted off, down to the limit of the fall height. The density and viscosity of water at 21 °C, and an assumed particle density were used.

The extracted fraction was deposited on a filter membrane for XRD analysis. A calibration suite of known alpha-quartz content, PM4 sized alpha-quartz particulate, loaded membranes were used as a calibration series against which the abstracted respirable fines on the filter membranes from the processed sample were compared. Additional scan time was undertaken to achieve better signal to noise ratios in the spectrum. Quantification was by the peak area integration method. Only crystalline material present in the sample will give peaks in the XRD scan. Amorphous (non crystalline) material will add to the background. The search match software used was EVA (Bruker). The ICDD card set was ICDD PDF4/Minerals 2015. The x-ray source was cobalt radiation. ICDD match probabilities are reported as an indication of how well the diffraction peaks of this sample compare with currently published literature on the quoted mineral. No Rietveld refinement was conducted on the acquired spectrum unless otherwise stated.

The respirable quartz concentration of the bulk was calculated by multiplying the volume percent of the respirable-only fraction by the alpha-quartz concentration of the respirable only fraction.

Summary

The size distribution of the sample by wet sieving and laser diffraction is shown below:

Sample	Size fraction (by aerodynamic diameter) volume percent			
	Non-inhalable	Inhalable, PM100	Thoracic, PM10	Respirable, PM4
Tail Dam Fines Respirable Silica	46.62	53.38	11.77	4.68

The interpreted semi-quantitative mineralogy by X-ray diffraction of the abstracted PM4 is shown below:

Mineral phase	Concentration (wt %)	ICDD match probability
Quartz, syn (Si O2)	21	good
Microcline (K Al Si3 O8)	19	good
Kaolinite (Al2 (Si2 O5) (O H)4)	16	medium
Albite, ordered (Na Al Si3 O8)	16	good
Muscovite-2M1 (K0.73 Na0.17 Ca0.01 Mg0.02 Ti0.02 Fe0.03 Al2.71 Si3.10 O10 (O H)2)	15	medium
Sepiolite (Mg4 Si6 O15 (O H)2 ·6 H2 O)	4	low
Nontronite-15A (Na0.3 Fe2 Si4 O10 (O H)2 ·4 H2 O)	3	low
Amphibole (Al3.2 Ca3.4 Fe4.0 K.6 Mg6.0 Na1.0 Si12.8 O44 (O H)4)	3	low
Hornblende (Na0.76 (Na0.46 Ca1.46 Fe1.4 Mg3.68) ((Si7.04 Al0.96) O22 (O H)2))	2	low

The respirable crystalline silica concentrations are shown below:

Lab number	Client ID	% α-quartz of bulk material	% cristobalite of bulk material	% tridymite of bulk material
17_0125_01	Tail Dam Fines Respirable Silica	0.722	<0.001	<0.001

Analysed:

[REDACTED]
[REDACTED]
[REDACTED]

Reported:

[REDACTED]

Approved:

[REDACTED]