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Client:	Wodgina Lithium	**Date Received:**	22/07/2024
Client address:	20 Walters DriveOSBORNE PARK WA 6017	**Date Analysed:**	05/08/2024
Job number:	24_1255	**Date Reported:**	08/08/2024
Lab ID:	24_1255_001		
Client ID:	Spodumene Concentrate SC6.0		
Analysis :	Respirable (PM4) silica concentration analysis by X-ray diffraction (XRD) and scanning electron microscopy (SEM) using the modified SWeRF (EN17289-3) method[1]		
Revision number:	0		
Comments:	None		

Sample preparation

The sample was supplied to Microanalysis Australia as a bulk material. The sample was tested as received.

A representative sub-sample of was wet sieved at 150 µm, and the < 150 µm fraction (suspension) was thoroughly homogenized and sized by laser diffraction reporting size between 150 µm and 20 nm.

The respirable fraction was abstracted 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 by SEM, the abstracted fraction was analysed qualitatively and quantitatively by X-ray diffraction to assess the crystalline silica concentration.

Analysis

The wet sieving was conducted using a light-flow (approximately 1 L /min) water spray jet on a 150 µm stainless steel Endecotts sieve. The < 150 µm fraction was collected in a bucket for laser diffraction analysis. Each size fraction was then oven dried at 105°C. The dried weights of each of the fractions were noted and the fraction percentage calculated based on the original dried starting weight.

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 ISO13320-1:2020.

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 of 2.65 g/cc were used.

The electron microscope used was a Carl Zeiss EVO50 equipped with an Oxford Instruments INCA energy dispersive spectrometer (EDS). All images were acquired using backscatter electrons, unless otherwise specified to highlight particle composition. The contrast in backscatter electron images is proportional to average elemental composition i.e. the brighter the particle the higher the atomic number. Some images with contrasting brightness particles were examined by EDS for elemental composition.

The extracted fraction was deposited on a filter membrane for XRD analysis. Quantification was by the RIR method. Only crystalline material present in the sample will give peaks in the XRD scan. Amorphous (non-crystalline) material will add to the background but is estimated by the software. The search match software used was EVA (Bruker). The ICDD card set was ICDD PDF4+ 2021. The X-ray source was cobalt radiation. ICCD 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 (as defined in ISO 7708) silica concentration of the bulk was calculated by multiplying the volume percent of the respirable-only (PM4) fraction by the α-quartz, cristobalite and tridymite concentrations of the respirable only fraction.

Summary

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

Client ID	Size fraction (by aerodynamic diameter) volume percent			
	Non-inhalable	Inhalable, PM100	Thoracic, PM10	Respirable, PM4
Spodumene	51.78	48.22	4.70	1.85

Assuming all mineral phases occur at the same relative concentrations across all size intervals, a volume percent distribution equates to a mass distribution. The respirable fraction, PM4 is therefore 1.85 wt %.

The normalised, interpreted semi-quantitative mineralogy by X-Ray diffraction of the abstracted PM4 fraction is shown below:

Crystalline mineral phase	Concentration (wt %) of PM4 only	ICDD match probability
Albite (Na(AlSi3O8))	37	High
Quartz, syn (SiO2)	15	High
Spodumene (LiAlSi2O6)	12	High
Clinochlore-IIb-4 ((Mg11.06Fe0.94)((Si5.22Al2.78)O20(OH)16))	12	Medium
Microcline (K(AlSi3O8))	12	Medium
Muscovite-2M1 (KAl2((Si3Al)O10(OH)2))	7	Low
Montmorillonite-18A (Na0.3(AlMg)2Si4O10OH2·6H2O)	5	Low

The XRD interpretation determined The PM4 fraction to be approximately 25 wt % amorphous. The above percentages represent only the crystalline fraction

The respirable (PM4) crystalline silica concentrations with respect to the bulk sample are shown below:

Lab ID	Client ID	Respirable (PM4) wt % of the bulk material for mineral phase		
		α-Quartz	Cristobalite	Tridymite
24_1255_001	Spodumene Concentrate SC6.0	0.214	<0.001	<0.001

Note: Three polymorphs of crystalline silica are scheduled as Group 1 carcinogens by IARC – α-quartz, cristobalite and tridymite[2].

Analyst: [redacted] B.Sc.(Chemistry)
Reported: B.Sc.(Chemistry)
Approved: Sc.(Multidisciplinary)

[1] https://www.ncbi.nlm.nih.gov/pmc/articles/PMC3979281/

[2] https://monographs.iarc.fr/ENG/Monographs/vol100C/mono100C-14.pdf