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NATIONAL INDUSTRIAL CHEMICALS NOTIFICATION AND ASSESSMENT SCHEME

FULL PUBLIC REPORT

LITHIUM SILICOTUNGSTATE

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Director Chemicals Notification and Assessment

FULL PUBLIC REPORT

LITHIUM SILICOTUNGSTATE

1. APPLICANT

Australian Mineral Industries Research Association Limited of 9th Floor, 128 Exhibition Street, Melbourne, Victoria 3000 has submitted for standard notification with their application for an assessment certificate for Lithium Silicotungstate. The notified chemical will be used in mining laboratories for mineral separation on the basis of density.

2. IDENTITY OF THE CHEMICAL

Chemical name: Tetralithium 1.4, 1.9, 2.5, 2.6, 3.7, 3.8, 4.10,

5.10, 6.11, 7.11, 8.12, 9.12-dodeca-μ-oxoμ₁₂- (tetrasilicato O_{1.4.9}, O_{2.5.6}, O_{3.7}, O_{10.11.12})

tetrakis[tri-µ-oxo-tri-(oxotungstate)]

Chemical Abstracts Service

(CAS) Registry No.: not available

Other names: Lithium silicotungstate, Lithium

tungstosilicate

Trade name: LST

Molecular formula: Li₄[SiW₁₂O₄₀]

Structural formula: The notified chemical is a complex

inorganic substance. X-ray crystallography

has not been carried out.

Molecular weight: 2901.6

Method of detection and determination: UV/Visible, ¹⁸³W NMR (CDCl₃), IR

Spectrum

Spectral data: IR Spectrum, Major Peaks at: 1019.22,

981.11, 927.77, 832.15, 786.84, 553.43, 538.80, 475.67 and 417.22 cm⁻¹ for Na⁴⁺

salt

3. PHYSICAL AND CHEMICAL PROPERTIES

Appearance at 20°C and 101.3 kPa: yellow crystalline solid

Odour: none

Melting Point: decomposes above 500°C

Density: 3900 kg/m³

Vapour Pressure: notified chemical is an inorganic salt,

vapour pressure expected to be

negligible.

Water Solubility: 5500 g/L at 20°C

Partition Coefficient

(n-octanol/water) log K_{ow}: not determined. LST is expected to

partition solely into the (n-octanol/water)

aqueous phase.

Hydrolysis as a function of pH: data provided indicate a complex

hydrolysis as a function of pH. At pH>9,

WO₄-2 predominates.

Adsorption/Desorption: LST will dissociate to form both

cationic and anionic species. These

will bind to oppositely charged

particles in soil.

Dissociation Constant

pK_a: not determined in terms of pKa. Data

provided indicate LST dissociates into a complex series of species and it is not

possible to determine all possible

dissociation constants.

Flash Point: LST is not flammable

Flammability Limits: LST is not flammable

Decomposition Temperature: see melting point

Autoignition Temperature: substance will not ignite

Explosive Properties: LST has no explosive properties

Reactivity/Stability: LST is a weak oxidiser

Particle size distribution: range - 1 mm (crystals)

Comments on physico-chemical properties

The substance is a complex inorganic salt with mild oxidising properties. The water solubility was not determined according to internationally approved OECD methods but was acceptable to show that LST is highly soluble. The octanol-water partitioning coefficient was not determined, however, the high water solubility of the compound would likely lead to a low Kow.

4. PURITY OF THE CHEMICAL

Degree of purity: following recrystallisation > 99%

Toxic or hazardous impurity/impurities: none known

Non-hazardous impurity/impurities (> 1% by weight): oxides of silicon and

tungsten

Additives/Adjuvants: water can be added to obtain desired

density

5. INDUSTRIAL USE

LST will be manufactured as a yellow crystalline solid. It will be used in mining laboratories to produce dense aqueous solutions for mineral separation on the basis of density (flotation of higher fractions). Approximately3 tonnes/year will be manufactured for the first five years.

6. OCCUPATIONAL EXPOSURE

Manufacture is to take place in Australia in the chemistry laboratory of the University of Western Australia. The production of the notified chemical will be small scale involving 2 chemists. The manufacture of LST will involve (1) the dissolution of α -12-silicotungstic acid and lithium hydroxide in deionised water , (2) the addition of lithium hydroxide solution to a α -12-silicotungstic acid solution in a large mixing vessel, (3) filtration of the solution (4) concentration by reverse osmosis and evaporation. The resulting product (~1000 L) will be stored in plastic containers out of direct sunlight. This production technique results in a potential exposure to LST for eight hours/day, 5 to 6 days per year. The major occupational risk will be the handling of caustic and acidic precursors of LST with some risk occurring with the handling of the finished product. The production procedure will be performed in a fume hood. The chemists will be required to wear gloves, eye protection and protective clothing during synthesis of LST.

The practical applications of LST initially take place at the University of Western Australia will involve 5-10 laboratory chemists. It is perceivable that the separation process may eventually be done in mining laboratories of companies utilising LST.

In this instance the notified chemical will be stored and transported in sealed plastic containers away from direct sunlight.

Water can be added to the LST solutions to achieve a desired density required for the mineral separation. A typical mineral separation will involve the usage of 100 mL to 2 L of LST solution and mineral samples of 50 g to 2 kg. The separation process is carried out by hand by laboratory staff using beakers and separatory funnels. Typical exposure of laboratory staff from direct handling of LST will be for 8 hours/day, 100-150 days/year. Laboratory staff will be required to wear goggles, gloves and protective clothing during the separatory process.

7. PUBLIC EXPOSURE

The notified chemical will be manufactured in the chemistry laboratory at the University of Western Australia. The chemical will be stored in plastic containers and distributed to mining laboratories from the site of manufacture. No public exposure to the notified chemical is expected to occur during its distribution.

No public exposure to the notified chemical is expected to occur during its manufacture or industrial use. In mining laboratories, minimal waste is anticipated as the chemical is recycled during separation procedures. Disposal of any waste chemical will be to landfill and will be in accordance with local and state regulations. Disposal of the notified chemical is not expected to result in any significant public exposure.

8. ENVIRONMENTAL EXPOSURE

. Release

LST will be used in mining laboratories, predominantly in Western Australia, to separate minerals based on density. A typical separation involves 100 - 2000 mL of LST solution and a mineral sample of 50 - 2000 g carried out by hand using beakers and separatory funnels. Because of the high costs of the material, recycling of LST will be extensive. Separated minerals will be washed free of LST with water and the washings collected in 100 - 200 L vessels. Solutions are then concentrated through the evaporation of excess water to reconstitute an LST solution of the desired specific gravity. In large scale use, washings are collected in vessels up to 1000 - 2000 L and concentrated by reverse osmosis and boiling. Any separated minerals contaminated with LST will be disposed of in landfill as non-hazardous liquid waste according to local and state regulations. Total losses to the environment during use are estimated at < 10 kg/year.

The Material Safety Data Sheet (MSDS) gives directions for spills and disposals. If spilled, LST is not to be allowed to enter waterways. If liquid, it should be absorbed with an inert material such as dirt or saw dust and shovelled into a container for disposal according to regulations.

A total of < 13 kg/year are estimated to be released from both the manufacturing and use of LST. These wastes are to be disposed of in landfills according to regulations.

. Fate

LST dissolves readily in water to form a complex series of ionic species, the fates of which are unknown. The biochemical oxygen demand (BOD) and OECD test for ready biodegradability were not performed. This is acceptable as LST is an inorganic salt and not expected to degrade or reduce dissolved oxygen concentrations. Since it is also highly water soluble, it is not expected to bioaccumulate in aquatic organisms.

9. EVALUATION OF TOXICOLOGICAL DATA

9.1 Acute Toxicity

Table 1 Summary of the acute toxicity of Lithium Silicotungstate

Test	Species	Outcome	Reference
Acute oral toxicity	Rat	LD ₅₀ > 2000 mg/kg	(1)
Acute dermal toxicity	Rat	LD ₅₀ > 2000 mg/kg	(3)
Skin Irritation	Rabbit	non-irritant	(4)
Eye irritation	Rabbit	severe eye irritant	(6)
Skin sensitisation	Guinea-pig	non sensitiser	(7)

9.1.1 Oral Toxicity (1)

 LD_{50} : > 2000 mg/kg Species/strain: Sprague-Dawley rats

Number/sex of animals: 5 animals/sex Observation period: 14 days

Method of administration (vehicle): 50% w/w aqueous solution administered orally

by gavage at a dosage of 2000 mg/kg.

Clinical observations: No abnormal clinical signs were observed.

Morphological findings: no

abnormalities when subjected to

necropsy.

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

9.1.2 Dermal Toxicity (3)

Number/sex of animals: 5/sex Observation period: 14 days

Method of administration (vehicle): 2 g/kg of ground LST (solid) applied to shaved dorsal area and covered with a moistened gauze patch.

Clinical observations: no abnormal signs were observed.

Mortality: none Morphological findings: no abnormalities when subjected

to necropsy.

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

9.1.3 Skin Irritation (4)

Result: non-irritant

Species/strain: New
Zealand White rabbits

Number/sex of animals: 6 adult females.

Method of administration: 0.5g of LST (solid) applied to dorsal area and covered

with moistened semi-occlusive dressing.

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

Draize (5) Scores¹:

Animal		Time a	fter decontam	ination	
	60 min	1 day	2 days	3 days	7 days
ERYTHEMA					
1	0	0	0	0	0
2	0	0	0	0	0
3	0	0	0	0	0
4	0	0	0	0	0
5	0	0	0	0	0
6	0	0	0	0	0
OEDEMA					
1	0	0	0	0	0
2	0	0	0	0	0
3	0	0	0	0	0
4	0	0	0	0	0
5	0	0	0	0	0
6	0	0	0	0	0

¹ see Attachment 1

9.1.4 Eye Irritation (6)

Result: severe eye irritant, irreversible damage including corneal peeling. The severe irritant effect of tungsten metal salts is an established phenomena in literature (7)

Species/strain: New Zealand White rabbits

Number of animals: 2
males. 1 female

Method of administration: Each rabbit received 0.1g of ground test material placed in the everted lower lid of each right eye, the left serving as the untreated control. Treated eye were observed at 1, 24, 48, 72 and 96 hours and 7,14 and 21 post treatment.

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

Draize (4) Scores²

Animal	Time after instillation																	
		1 ho	ur	1 day		2	day	/S	3 days		4 days		7 days					
CORNEA:	opa	acity		opa	acity	′	opacity		opacity		opacity		opacity					
	are	ea		are	a		are	a		are	ea		are	ea		are	ea	
1	4		1	4		1	4		1	4		1	4		1	3		1
2	4		2	4		1	4		1	4		1	4		1	4		1
3	4		1	4		1	4		1	4		1	3		1	3		1
IRIS																		
1		1			1			1			1			1			1	
2		1			1			1			1			1			1	
3		1			1			1			1			1			0	
CONJUNCTIVA	ra	Cp	dc	ra	Cp	dc	ra	Cp	dc	ra	Cp	dc	ra	Cp	dc	ra	Cp	dc
1	2	3	2	2	3	3	2	3	2	3	3	2	3	2	2	3	2	2
2	2	3	2	2	4	3	2	3	3	3	3	1	3	3	1	3	3	2
3	2	2	2	2	2	2	2	2	2	3	2	1	3	2	0	2	2	1

² see Attachment 1

9.1.5 Skin Sensitisation (8)

Result: No irritating effects observed, LST is a non sensitiser.

Species/strain: Dunkin Hartley Guinea-Pig

Number of animals: 51

(39 males, 12 females)

Induction: A dose of 0.5 g of ground LST moistened with 200-250 µl water was applied epicutaneously on a patch and occluded for six hours. Procedure done twice a week for three consecutive weeks, a total of six applications made. Skin reactions measured 24 and 48 hours after patch removal.

^a redness ^b chemosis ^c discharge

Challenge: An epidermal application of 0.5g of ground LST moistened and applied for 6 hours under occlusion was made 13 days after the last induction.

Results: scored to Bühlers method.

Challenge	24	hrs	48hrs			
Concentration	test	control	test	control		
0.5 g	0	0.1	0	0.1		

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

9.2 Repeated Dose Toxicity (9)

Result: No toxic effects of LST (solids) over 14 days of repeat dose testing were observed at 200 mg/kg.

Species/strain: Sprague Dawley rats Number/sex: 25/sex

Method of administration (vehicle): LST in aqueous solution delivered by gavage

Dose/ Duration of administration:

high dose group: 1000 mg/kg body weight medium dose group: 500 mg/kg body weight

low dose: 200 mg/kg body weight

control groups (2): 2000 mg/kg body weight of water

All rats dosed daily for 6 out of 7 days for 14 days.

Significant Observations:

1. Clinical

At 1000 mg/kg some rats showed signs of piloerection between days 5-14, hunching between days 6-14 ten minutes after dosing and abdominal distension on days 8-12 and 14. On rat (female) died on day 9. Loose faeces were also noticed in a couple of rats on days 4-6.

At 500 mg/kg, all rats were hunched after dosing for 10 minutes on day 9 to 12 and 14. A few rats also experienced loose faeces on days 2-3.

No abnormal signs were observed at 200 mg/kg.

2. Clinical Chemistry/Haematology

White cell counts (males, males + females), lymphocytes (males, males + females), and eosinophils (males) were significantly higher for the 1000 mg/kg group than control group 1, however the values were still within the normal range given in literature. There was no significant difference in the percentages of each type of white cell between the treated and control groups.

There were no clinically significant differences in biochemistry parameters.

3. Necropsy Findings/ Histopathology

At 1000 mg/kg, one rat was found to have cellular proliferation in the liver and several foci of hepatocellular carcinoma, however this is not thought to be due to the treatment as 14 days not long enough for tumour initiation or growth. Changes in other rats include eosinophilic gastritis, lymphoid hyperplasmia and renal cortical mineralisation which were all observed in the Control group1 and not thought to be due to treatment.

There were no other pathologically significant observations

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

9.3 Genotoxicity

9.3.1 Salmonella typhimurium Reverse Mutation Assay (10)

Result: LST was not found to be mutagenic.

Strains: Salmonella typhimurium TA 98, 100, 102, 1537.

Concentration range: 0.25, 1.00, 2.50, 3.75, 5.00 mg/plate with and without S9 rat liver metabolic activation. The positive control in the absence of metabolic activation was 0.1 mg/mL 2,4-dinitrophenylhydrazine, apart from S.typhimurium TA 100, in which case 0.1 mg/mL sodium azide was used. The positive control in the presence of metabolic activation was 0.1 mg/mL 2-acetamidofluorene.

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

9.3.2 Micronucleus Assay in the Bone Marrow Cells of the Mouse (11)

Result: LST did not produce micronuclei in the polychromatic erythrocytes 24,48 or 72 hours after intraperitoneal injection.

Species/strain: B6C3F1 Grey Mice

Method of administration (vehicle): LST dissolved in PBS (phosphate buffered saline) was injected intraperitoneally

Test Method: according to OECD Guidelines for the Testing of Chemicals (2)

9.4 Overall Assessment of Toxicological Data

The notified chemical exhibited low acute oral and dermal toxicity in rats with LD50 values for both routes of administration greater than 2000 mg/kg. LST was not found to be a skin irritant but was severely irritating to the eyes of rabbits. It was not a skin sensitiser in guinea-pigs. In a 14 day repeat dose toxicity study one female given 1000 mg/kg died on day 9. Another rat given 1000 mg/kg was reported to have moderate bilary hyperplasia, peritportal cellular proliferation in the liver and several foci of hepatocellular carcinoma, the duration of treatment was not considered sufficient for initiation of tumour promotion or growth. Given that hepatocellular carcinoma can occur in untreated Sprague-Dawley rats, this finding is not considered to be related to treatment. Clinical observations from 500 mg/kg included loose faeces and hunched postures, and at 1000 mg/kg abdominal distension and pilioerection occurred. In the presence and absence of metabolic activation, the notified chemical was not mutagenic in strains of *S. typhimurium* and did not produce chromosomal aberrations in a mouse micronucleus study.

On the basis of eye irritancy, the notified chemical is classed as hazardous according to Worksafe Australia Approved Criteria for Classifying Hazardous Substances (12).

10. ASSESSMENT OF ENVIRONMENTAL EFFECTS

The following ecotoxicology studies have been provided.

Test	Species	Result
Acute Toxicity	Fathead minnow	62.5 mg/L < 96-h LC50 < 125 mg/L
		NOEC = 62.5 mg/L
Acute Toxicity	Daphnia magna	48h EC50 = 162.3 (135.3, 188.2) mg/L
-		NOEC = 100 mg/L, LOEC = 200 mg/L
Chronic Toxicity	Daphnia magna	14d reproduction EC50=1.20 (0.84,1.72) mg/L
_		NOEC = 0 mg/L, LOEC = 0.625 mg/L

The toxicity of LST to fathead minnows (*Pimephales promelas*) was investigated by Woodward-Clyde (13) in a 96-h static experiment. Ten fish (1.8 - 2.4 cm in length) were placed in each of six aquaria containing 2 L of LST solution at concentrations ranging from 0 to 500 mg/L. At the end of 96 h, all fish in treatments up to 62.5 mg/L were normal in appearance and behaviour. At 125 mg/L 90% mortality was observed and in the two higher treatments, all fish died. As only one treatment with partial mortality was recorded, there was insufficient data to estimate the LC50 by probit

analysis. The LC50 is likely between 62.5 and 125 mg/L, the concentrations causing 0 and 90% mortality, respectively.

Woodward-Clyde (14) studied the acute and chronic toxicity of LST to *Daphnia magna*. For the acute testing, 20 daphnid neonates ≤ 24 h old (5 per replicate, 4 replicates) were placed in each of six treatments containing LST at nominal concentrations ranging from 0 to 500 mg/L. After 48 h, the organisms were assessed for immobilisation. The EC50 was 162.3 (135.3, 188.2) mg/L with a NOEC and LOEC of 100 and 200 mg/L, respectively.

In the chronic experiment, 40 neonates were placed in six nominal LST concentrations ranging from 0 to 10 mg/L (10 daphnids per replicate, 4 replicates per treatment). Observations on survival and reproduction were made daily for 14 d, however, the frequency of solution renewal was not specified. Results showed a 14-d LC50 of 2.47 (1.89, 3.06) mg/L and inhibited reproduction at all test concentrations. The LOEC was the lowest concentration tested of 0.625 mg/L with an EC50 for reproduction of 1.20 (0.84, 1.72) mg/L.

The notifier has requested that the requirement of an algal growth inhibition test be waived on the grounds that LST will not likely be released to the environment in significant amounts. The rationale for a data waiver is acceptable.

LST would be classified as slightly to practically non-toxic to fathead minnows, and practically non-toxic to daphnids on an acute basis. However, daphnids chronically exposed to low concentrations of LST may be adversely affected.

11. ASSESSMENT OF ENVIRONMENTAL HAZARD

The chronic toxicity of LST to *D. magna* is 135 times higher than the acute toxicity, and is therefore a potential hazard at concentrations of approximately 0.12 mg/L, which is 1/10 the 14-d EC50 for reproductive effects. However, any chemical wasted in association with contaminated minerals (estimated < 13 kg/year) is to be disposed of in approved landfills. The environmental releases of LST to natural aquatic and terrestrial ecosystems are expected to be low, and the use of LST is unlikely to be a hazard to the environment.

12. ASSESSMENT OF PUBLIC AND OCCUPATIONAL HEALTH AND SAFETY EFFECTS

Based on the data given for LST, there is low potential for acute and oral toxicity and skin irritancy. There is however the potential for irreversible eye damage due to the severe eye irritancy of LST. There is little potential for skin sensitisation, repeated dose toxicity, mutation or chromosomal damage.

The manufacture and usage of LST will involve the possible exposure of personnel within chemical laboratories. There will be no other sites of manufacture or usage. There exists some potential risk of dermal or oral exposure from spillage or splashing to the caustic and acidic precursors of LST, as well as the final product.

While skin contact is not expected to result in toxicity or irritancy, any eye contact is likely to cause permanent damage. To reduce the risk of exposure, the manufacturing process will take place within a fume hood. To further reduce the risk of exposure, personal protective equipment such as gloves, goggles and protective clothing are essential.

The practical application of LST will involve manual handling of the notified chemical in laboratory glass ware during the separation process. The potential also exists here for exposure via the dermal or oral routes from spillage or splashing. Again, personal protective equipment such as gloves, goggles for eye contact and protective clothing are necessary.

No public exposure to the notified chemical is expected to occur.

LST poses a significant risk during its manufacture and usage due to the severe eye irritancy properties. However, lack of public exposure as well as the stringent personal protection and engineering controls employed during laboratory production and application of the notified chemical should minimise this risk.

13. RECOMMENDATIONS

To minimise occupational exposure to Lithium Silicotungstate the following guidelines and precautions should be observed:

during manufacture fume hoods and exhaust ventilation should be utilised where possible.

eye protection should be selected and fitted in accordance to AS 1336 (15) and used in accordance to AS/NZS 1337 (16).

industrial clothing must conform to the specifications detailed in AS 2919 (17) and AS 3765.1 (18).

industrial gloves should conform to the standards detailed in AS 2161 (19) and AS 3765.1 (18).

all occupational footwear should conform to the standards detailed in AS/NZS 2210 (20).

particular care should be taken to avoid spillage or splashing of the notified chemical.

good personal hygiene should be practised to minimise the potential for ingestion.

a copy of the Material Safety Data Sheet should be easily accessible to employees.

14. MATERIAL SAFETY DATA SHEET

The Material Safety Data Sheet (MSDS) for Lithium Silicotungstate was provided in an acceptable format (21).

This MSDS was provided by Australian Mineral Industries Research Association Limited as part of their notification statement. The accuracy of this information remains the responsibility of Australian Mineral Industries Research Association Limited.

15. REQUIREMENTS FOR SECONDARY NOTIFICATION

Under the *Industrial Chemicals* (*Notification and Assessment*) Act 1989, secondary notification of Lithium Silicotungstate shall be required if any of the circumstances stipulated under subsection 64(2) of the Act arise. No other specific conditions are prescribed.

16. REFERENCES

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Attachment 1

¹ The Draize Scale for evaluation of skin reactions is as follows:

Erythema Formation	rating	Oedema Formation	rating
No erythema	0	No oedema	0
Very slight erythema (barely perceptible)	1	Very slight oedema (barely perceptible)	1
Well-defined erythema	2	Slight oedema (edges of area well- defined by definite raising	2
Moderate to severe erythema	3	Moderate oedema (raised approx. 1mm)	3
Severe erythema (beet redness)	4	Severe oedema (raised more than 1 mm and extending beyond area of exposure)	4

² The Draize scale for evaluation of eye reactions is as follows:

² The Draize scale for	evaluation (of eye reactions is as fo	ollows:				
CORNEA							
Opacity		rating	Area of Cor	rating			
No opacity		0 none	25% or less (r	not zero)	1		
Diffuse area, details of iris clearly visible		1 slight	25% to 50%		2		
Easily visible translu cent areas, details of iris slightly obscure		2 mild	50% to 75%		3		
Opalescent areas, no details of iris visible, size of pupil barely discernible		3 moderate	Greater than 75%		4		
Opaque, iris invisible		4 severe					
CONJUNCTIVAE							
Redness	rating	Chemosis	rating	Discharge	rating		
Vessels normal	0 none	No swelling	0 none	No discharge	0 none		
Vessels definitely injected above normal	1 slight	Any swelling above normal	1 slight	Any amount different from normal	1 slight		
More diffuse, deeper crimson red with individual vessels not easily discernible	2 mod.	Obvious swelling with partial eversion of lids		Discharge with moistening of lids and adjacent hairs	2 mod.		
Diffuse beefy red	3 severe	Swelling with lids half- closed	· 3 mod.	Disharge with moistening of lids and hairs and considerable area around eye	3 severe		
		Swelling with lids half- closed to completely closed	- 4 severe				
IRIS							
Values					rating		
Normal					0 none		
Folds above normal, congestion, swelling, circumcorneal injection, iris reacts to light							
No reaction to light, haer	morrhage, gro	oss destruction			2 severe		