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STUDY ON FURTHER APPLICATION OF MOLYBDENUM DISULFIDE EXTRACTED FROM ERDENET MININGS AS A LUBRICANT

Bachelor Thesis

by

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I did not use any sources other than those stated. In case that the work is additionally submitted on a data medium, I declare that the written and the electronic form are completely identical. The work was not submitted in the same or similar form to any examination authority.

UB/Nalaikh, 05/16/2022
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Signature

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I would like to express my deepest gratitude to every faculty member and helpful staffs of the German - Mongolian Institute for Resources and Technology. It was a great experience in collaborating with and learning from hard-working fellow students at the German - Mongolian Institute for Resources and Technology.

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List of Abbreviations

CV	Certified Value
UCRM	Extended Uncertainty of Measurement
N	Number of independent results
IV	Information value
SEM	Scanning Electron Microscope
HV	Vickers Diamond Hardness

Abstract

Purpose of the study is to investigate molybdenum disulfide extracted from the Erdenet Cu-Mo deposit as a solid lubricant and its further application.

This paper consists of a literature review of molybdenum disulfide's applications, physical and chemical properties, crystal structure and behavior under different conditions, and the lubricating mechanism. The scope of the research was molybdenum disulfide-free powder rather than other forms of molybdenum disulfide-based lubricants. Also, a lubricating condition in a regular atmosphere is considered rather than in a vacuum environment.

The main methods of the investigation are molybdenite concentrate analysis and observation of scanning electron micrographs of molybdenum disulfide particles. An experiment with a laboratory benchtop ring mill machine was done to observe the lubricating behavior.

As a result, molybdenum disulfide produced at the Erdenet Mining has the potential to be used as a solid lubricant even though the application of molybdenum disulfide as a free powder for lubrication is limited. Further purification and refining procedures are crucial to produce molybdenum disulfide with higher quality and to make it applicable for tribological tests. The powder with fine grade and better quality will be able to exported at a higher price.

1. Introduction

Liquid lubricants such as oils and greases are conventionally used in many mechanical systems because of satisfactory reduction in friction, wear rate, and straightforward application. However, there are several drawbacks of liquid lubricant. It decomposes and oxidizes at high temperature, solidifies or becomes too viscous at low temperature, has low load-carrying capacity in high pressure, picks up dust easily, is heavy for transportation, and degrades over time. Solid lubricants overcome many of these issues. [11].

Common solid lubricants are graphite, molybdenum disulfide, tungsten disulfide, and polytetrafluorethylene. Of all the solid lubricants, molybdenum disulfide is inferior due to its ability to perform in a wide range of temperatures, high loading capacity, and unchanged properties in high pressure and extreme conditions. Due to its excellent properties in lubrication, it is utilized in more applications than any other solid lubricant. [11].

Mongolia is one of the top 10 molybdenite producers in the world. Erdenet Mining Corporation produces 4700 thousand tons of molybdenum concentrate each year with 48-50% molybdenum concentration, and exports to China, Korea, and Vietnam. Molybdenum disulfide is the primary raw material for molybdenum metal production [17].

According to the bachelor thesis "Molybdenum disulfide based lubricant and its market in Mongolia" [2] by Uurintuya Unenbat, German-Mongolian Institute for Resources and Technology, molybdenum disulfide based lubricants are all imported and its estimated market is high in next 5 years. However, there aren't any lubricant producers in Mongolia. Based on her research and critical study, possible lubricant products to be manufactured were 100% molybdenum disulfide powder lubricant because molybdenum disulfide-based greases and pastes have production complexity and the required binders and matrices need to be imported [2].

The aim of this thesis is to determine if it is possible to use the molybdenum disulfide produced at Erdenet Mining as a free solid lubricant and its further application.

2. State of Art

2.1. Brief History

Lubrication is one of the oldest technologies that has been used since the invention of early machines and mechanisms. Under rapid wear and friction, the advanced machine technology would not be able to develop successfully until today without lubrication. There are various types of lubricant materials used between the contacting surfaces to reduce friction and wear.

Lubricious solids such as ice, loose sand, and powdered snow were used for centuries. The invention of the common dry lubricants in modern days, such as graphite and molybdenum disulfide date back to the Greek and Roman civilization and the use of molybdenum disulfide in lubrication has recorded in the early seventeenth century. The lubricating properties of molybdenum disulfide had taken the interest of material scientists and engineers, but its application was still not common during the industrial revolution because of other effective lubricants' availability and cheapness [1].

In the late 19th century, the extraction of molybdenite and the satisfactory purification of molybdenum disulfide were developed but its interest in the technical application was still slow to develop until the investigation of the potential value of its crystal structure. This has led to further technical inspections resulting in its superior lubrication properties, stability under extreme contact pressures in vacuum environments, and more. The use of molybdenum disulfide expanded extensively since 1940's and it's inferior lubricating property in space attracted the attention of technical industries including the National Aeronautics and Space Administration which started in-depth investigations of its use in vacuum [5].

Now, there are extensive applications of molybdenum disulfide based lubricants are utilized both in space and biosphere for various industries as of its excellent lubricating properties [1].

2.2. Range of Applications

Molybdenum disulfide lubricants have excellent lubricating properties in extreme conditions of vacuum and space, but the majority of its application is utilized in the automotive field followed by the aviation industry [1]. The technological applications of molybdenum disulfide extend from manufacturing, metalworking, and production processes to the food industry, and medical tools.

Depending on the molybdenum content and other constituents, molybdenum disulfide based lubricants have several different forms suited for specific applications. Molybdenum disulfide is used as in free powder form by burnishing it on the surfaces with or without binders, used as a constituent in dry lubricants, and additive to greases, pastes and coatings [5]. A variety types of molybdenum disulfide lubrication forms and their uses are listed in Table 1. In this paper, more focus is given to the free powder form of the molybdenum disulfide.

Table 1. Some applications of molybdenum disulfide

Molybdenum disulfide formulations		
Mo Content (%)	Product Type	Uses
1 – 20	Greases – for manufacturing, mining and transportation	Ball and roller bearings, splines, chassis, conveyors
20 – 60	Pastes – mineral or synthetic base	Assembly of machinery, splines, gears, universal joints, metal forming
0.5 – 5	Industrial and Motor Oils or Synthetic fluids	All automotive and industrial gears, reducers, cams, etc
1 – 20	Water Suspensions	Metalworking and process lubrication, threads, slices, packaging, die casting
Up to 85	Bonded Coatings – air or heat cured, organic, inorganic	Threads, tools, switches, locks, valves, slide,

		process lubrication, metalworking
1 – 40	Metal working compounds, soaps, powders	Extrusion, cold forming, wire drawing, deep drawing
10 – 100	Pure or Mixed Powders	Punch line, stamping, forming, relays, switches, packing
Composites		
1 – 10	Friction products, sintered Cu brakes, Semi-metallic and Non-asbestos pads	Aircraft, automotive and rail brake pads and linings
1 – 30	Plastic, rubber and metal composites	Gears, slides, bearings, thrust washers, O-rings

2.5. Using Molybdenum Disulfide as a Free Powder

Molybdenum disulfide powder is mostly used with other materials to improve the lubricating properties for specific applications. There are several techniques of using the powder alone for lubrication listed in Table 2, but other substances are used in all these techniques in a very small amount. Since, the adding substances don't really affect the properties of the free powder and occur in a small amount compared to mixing with binders, it can still be considered as lubricating with molybdenum disulfide alone. This study is focused on the techniques that require less additional materials and complexity.

Table 2. Using Molybdenum Disulfide as free powder

Process	Advantages	Disadvantages	Applications
Free powder	Simplicity	Messy, not very effective	Open gears, screw threads
Dispersion in water	Convenience, fire resistance	Limited storage life	Metalworking

Dispersion in volatile liquid	Rapid evaporation of carrier	Limited storage life	Anti-seize, assembly
Dispersion in gas	Continuous feed	Complicated supply	Rolling bearings
Burnished film	High quality film and good load-carrying capacity	Laborious, limited to simple shapes	Shafts, bushes, slideways
Sputtered film	High quality film and good load-carrying capacity	Complex equipment required	Vacuum
In situ film	High quality film and good load-carrying capacity	Complex process	Complex shapes
Compact	Mouldable to required shape	Low structural strength	Test work, Transfer lubrication
Transfer film	Simple, possible to supply continuously	Quality control difficult	Bearings or gears

Although using the molybdenum disulfide in a free powder is the simplest technique of all, there are several drawbacks. The utmost concern is the free powder doesn’t distribute on the surface evenly which results low lubrication on the insufficient area and causes jamming on the excess points. It easily adheres to the surrounding and the black colored particles remain dirty mark on the work space.

In this technique the purity and the fine particle size of the powder as well as creating strong and uniform film is crucial. Vaisfeld [1] has experimented the free powder in some cases but the lubrication was not very effective in rolling contacts because loose molybdenum disulfide wasn’t very adhesive to the surface, the burnished films easily scraped from the contact area. Also, it is perfectly lubricated in the beginning of the operation but friction reduction declines as it is operated without lubricant. [1].

In conclusion, this technique is more suitable by forming burnished films for simple mechanisms such as open gears and screw threads that the geometry of contacts is able to keep the free powder. Also, the operation should be aware of the powder jamming.

2.3. Characteristics of Molybdenum Disulfide

2.3.1. Impurities and Particle Size

Impurities

In the mineral processing of molybdenite, the first stage of refining produces 85-90% molybdenum disulfide concentrate. Using molybdenum disulfide at this grade in lubrication results high wear rates. The concentrate undergoes further processes including grinding and acid washing for lubrication application. After these processes, other residual impurities such as silica, iron, copper compound, and other materials may be present [1]. The flowchart of molybdenite processing is shown in Figure1.

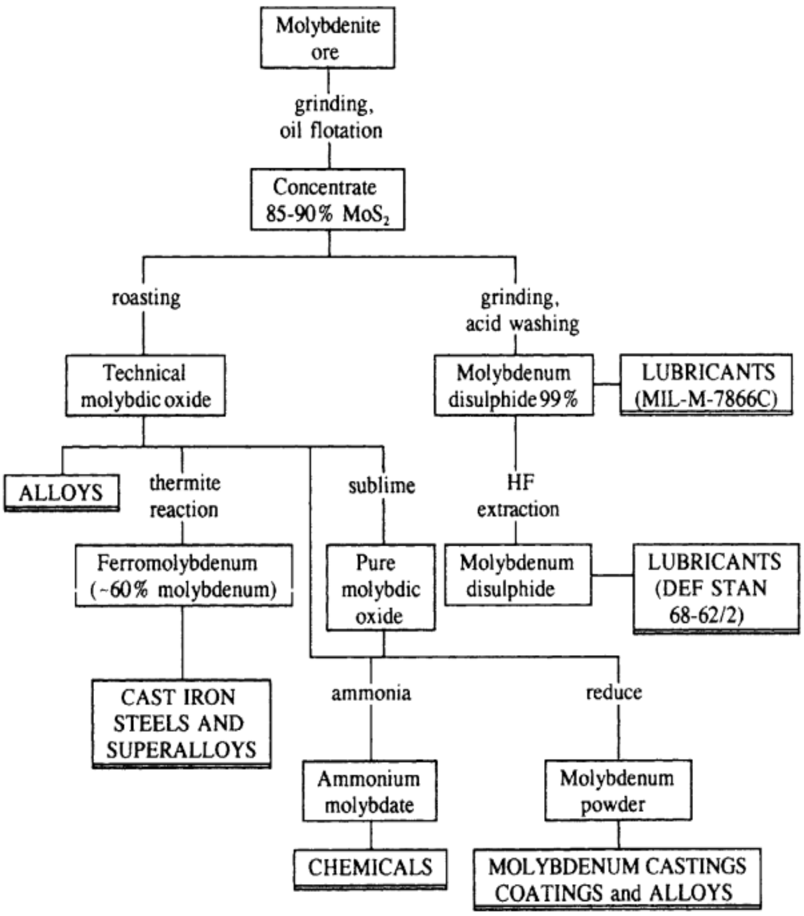


Figure 1. Flow Chart for Molybdenite Processing

The purified powder of molybdenum disulfide with less than 2% impurities has been available since the 1950s, and the main effect of impurities in solid lubrication is abrasion. The only method to distinguish the abrasion of molybdenum disulfide with different qualities is the direct abrasion test. In the United States specification MIL-M-7866B and British specification DEF-234 of molybdenum disulfide technical lubrication grade, the impurity limit is up to 1%, and the amount of silica limit to 0.02% accordingly. In order to use the molybdenum disulfide as a free powder in lubrication alone, the purity must be at least 98% to be applicable. Table 3 shows the standard grades of the molybdenum disulfide powder depending on the impurities [1].

Table 3. Chemical Composition of Molybdenum Disulfide Powder Standard

Grade		Standard 1	Standard 2	Standard 3	Standard 4
MoS2	≥	99	98.5	98	96
Total Impurities	≤	0.5	0.5	0.65	2.5
Fe		0.15	0.15	0.3	0.7
Pb		0.02	0.02	0.02	0.02
MoO3		0.2	0.2	0.2	0.2
SiO3		0.1	0.1	0.2	-
H2O		0.2	0.2	0.2	0.2
Acid No. (KOH mg/g)		0.5	0.5	0.5	1
Oil (Acetone Extraction)		0.5	0.5	0.5	0.5

Particle Size

The molybdenum disulfide has standard 4 powder grades listed in the Table 4.

Table 4. Molybdenum Disulfide Commercial Lubricant Grade

Particle Size	0	1	2	3	4
D50/u	≤ 1.0um	1.0 – 1.5 um	1.5 – 5 um	5 – 10 um	≥ 10 um

2.3.2. Physical Properties

Molybdenum disulfide is a dark blue-gray to black colored solid compound depending on its particle size [3]. It feels slick and smooth to the touch and easily transfers to other surfaces. It occurs in nature in rhombohedral and mostly in hexagonal crystalline forms. The most important physical properties of it are listed in Table 5 [1].

Table 5. Physical Properties of Molybdenum Disulfide

Property	Value
Melting Point	1185 decomposes
Lubrication Temperature Range	Ambient: from -185 °C to 350 °C Vacuum: from -185 °C to 1100 °C
Molecular Weight	160.08
Density	5060 $\frac{kg}{m^3}$
Crystal form	Hexagonal, rhombohedral
Hardness (basal planes)	1.0-1.5 Mohs scale
Hardness (crystal edges)	7-8 Mohs scale
Color	Blue-gray to black
Magnetic Properties	Diamagnetic
Electrical conductivity	Low
Sublimation temperature	1050°C in high vacuum
Dissociation temperature	1370°C +
Friction Coefficient	0.02 – 0.05

Load Bearing Capacity	1723 MPa

2.3.3. Chemical Properties

Molybdenum disulfide is non-toxic chemically very inert and insoluble in water. It is resistant to the most acid attack except for aqua regia, hot concentrated sulfuric, nitric, hydrochloric, and dilute acids. The product of the acid attack is molybdenum trioxide. The reaction with the hydrofluoric acid is gradual, and it doesn't react with dry hydrogen fluoride but is attacked by fluorine. Some changes occur in the chemical composition and crystalline structure parameters when heated with mixtures of graphite and PTFE [1].

Molybdenum disulfide produces embrittlement of stainless steel at temperatures above 300 ºC and reacts with iron at 700 ºC and copper at 500 ºC [1].

2.3.4. Crystal Structure

Molybdenum disulfide is classified into the thin-layered transitional-metal dichalcogenides group of 2D materials. In this thesis paper, hexagonal structure, the form of natural molybdenum disulfide is studied. The crystal structure of hexagonal form has six-fold symmetry with two molecules per unit cell. Each sulfur atom is located from three molybdenum atoms at an equal distance, and each molybdenum atom is equally distanced from six sulfur atoms. In other words, every molybdenum atom is at the center top of the triangular prism and the sulfur atoms are at the bottom corners. The unit cells are in a laminar arrangement [3] as illustrated in Figure 2.

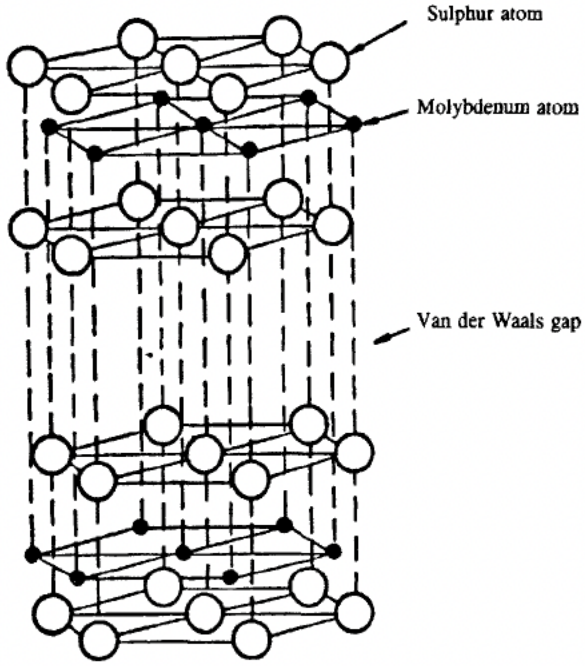


Figure 2. Crystal Structure of Molybdenum Disulfide

The interatomic distance is 2.41±0.06 Å, the height of the prism is 3.17±0.1 Å, and the triangular edge is 3.15±0.02 Å. The lattice parameters are a = 3.15 Å and b = 12.39 Å. The spacing between the sulfur layers is 3.49 Å [3]. The bonding between the molybdenum and sulfur are strong covalent bonds, and the lattice layers are bonded or the sulfur atoms are attracted by weak Van der Waals force [1].

2.4. Lubricating Mechanism of Molybdenum Disulfide

Friction between the surfaces

On a microscopic scale, the contact between objects is supported on the extremely small area of the tiny peak points of the rough surface. Since the area of contact is small, the load generated by the contact is relatively high which exceeds the yield stress of the materials. When the contact load exceeds yield stress, elastic and plastic deformation occurs at the contact zone which increases the contact area. When the sliding occurs between the surfaces, lateral force adding to the relatively high contact load causes adhesion. The energy required in deformation and adhesion resistance adds up and contributes to the total friction.

Mechanism of friction in molybdenum disulfide

The lubricating behavior of molybdenum disulfide can be explained by (1) Intrinsic cleavage mechanism, (2) Adsorption mechanism theory, and (3) Intracrystalline slip mechanism. Currently, the first mechanism has the most experimental support and simply explains the frictional behavior of molybdenum disulfide.

Intrinsic cleavage mechanism

Crystal structure configuration and the weak bonding between the laminas result lubrication effect. As discussed in 2.3.4, the distance between the sulfur atoms is far and bonded with weak Van der Waals forces. The distance between the sulfur atoms is greater than the thickness of the lamina Figure 2, which results easy slippage across the laminas [3].

In other words, in its crystal structure, the molybdenum atoms in each layer don’t reside above or below each other but the sulfur atoms reside directly above and below of sulfur atoms in other layers. This causes high spacing and low attraction between the layers. This is the only interlaminar attractive force that can be interpreted as the compressive force which is perpendicular to the laminas Figure 3. The compressive force acts like the adhesive force between the surfaces as mentioned previously. When sliding occurs, the shearing force F between the laminas is relatively large to overcome the compressive force.

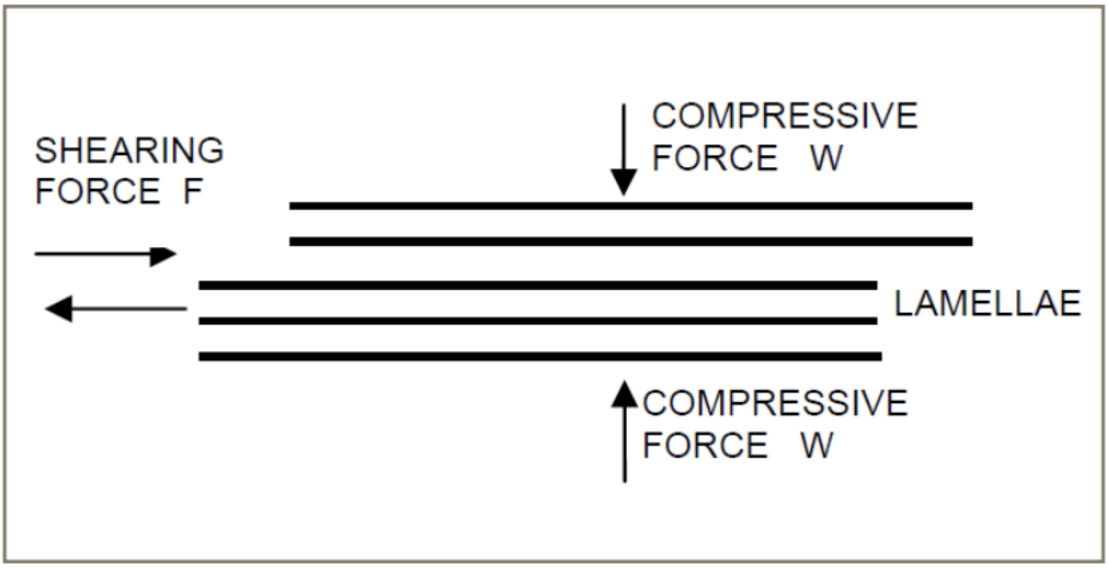


Figure 3. Lubricationing Mechanism of Molybdenum Disulfide

The coefficient of friction for molybdenum disulfide crystals is approximately 0.025, which is one of the lowest coefficients of friction of solid lubricants.

2.6. Behavior of molybdenum disulfide as a free powder

In this chapter, the various influences to the lubrication of molybdenum disulfide free powder is discussed.

Effect of moisture

The effect of moisture on molybdenum disulfide lubrication is important because it is used in wide range of applications under various climate changes. Water vapors have significant influence on the frictional behavior of molybdenum disulfide [5]. According to the investigation by Marshall B and Robert L [6], the coefficient of friction of molybdenum disulfide is greater at high humidity than in dry air. The free powder did not adhere to steel surfaces at high humidity and the contact between the surfaces was greater which resulted increment in friction.

Moreover, due to the poor adherence to the substrate surface, the area of wear on the surface was larger than tested in the dry environment. Adding on, corrosion formed between the unlubricated metallic surfaces [6].

However, there are more factors adds to the moisture effect which create different results. Barry and Binkelman [12] investigated these effects by considering varying speed, loads, relative humidity and substrate including metals with hardness from 99 to 882 HV. Their investigation resulted the molybdenum disulfide friction decrease with both increasing load and speed when moisture present. The relationship is shown in Figure 4 and Figure 5.

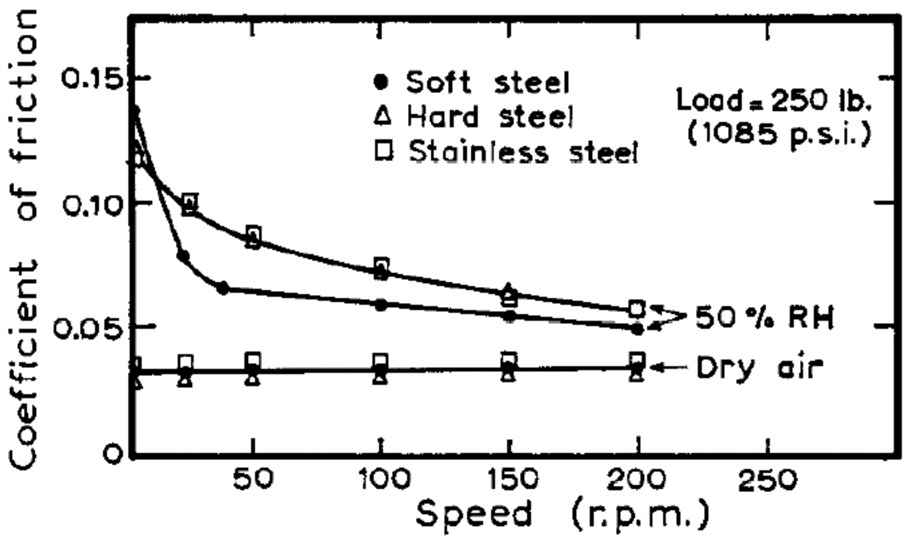


Figure 4. Coefficient of friction VS Speed

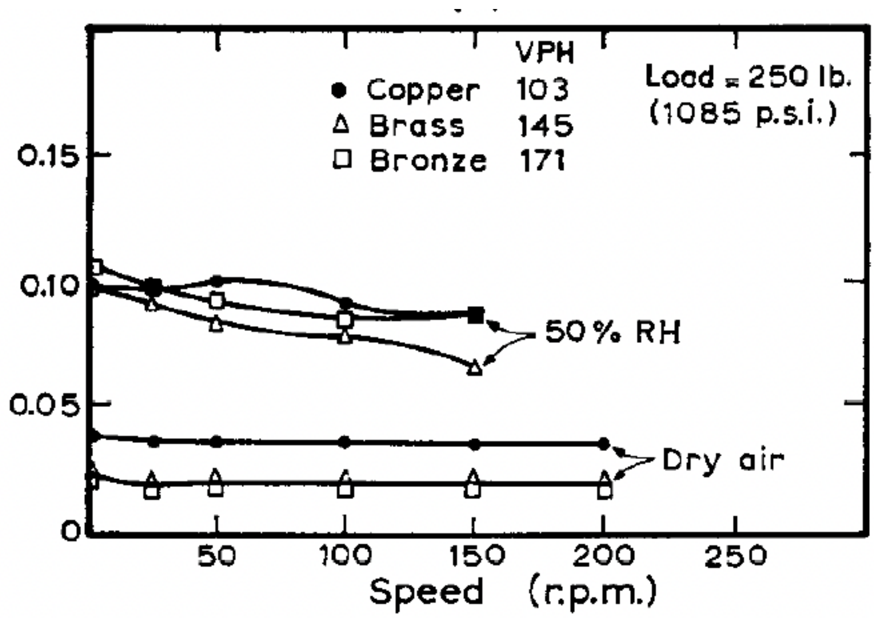


Figure 5. Coefficient of friction VS Speed

Effect of pressure and sliding speed

As mentioned above, the effect of the load has a direct relationship with the operating environment humidity. The change in friction under high load and speed is due to the change in moisture content of the molybdenum disulfide film. In the figure, the coefficient of friction is constant under various loads, speed, and substrate hardness in dry environment. According to Body and Robertson [13], the coefficient of friction is decreased down to 0.032 with increasing load up to 600,000 psi which was the lowest coefficient of friction of any material [5].

The effect of sliding speed has similar influence as pressure, because both correlated with moisture change between the surfaces. In the absence of moisture, the friction is constant at varying speed, and in a humid environment, the friction variation depends on the moisture level [14].

Effect of substrate

The effect of substrate depends on the surface finish, hardness, and chemistry. Surface finish with 2 um gives an optimum wear life for molybdenum disulfide film. Surface finish with 0.3-0.4 µm has an average wear life of 13 hours, and with 0.004 µm the wear life is only a few minutes [15].

The surface hardness and surface chemistry effect on molybdenum disulfide are not well understood because various studies conflict depending on the experiment conditions, which makes it impossible to compare the results.

Effects of particle size

The particle size effect on wear performance is very small, but under high load and speed, there is considerable difference in wear. Coarser particles results high wear, and finer particles result low wear. Moreover this relation extend to surface roughness. Finer particles have better performance for coarser surface than smaller particles [8].

Effect of impurities

The major impurity occurs in molybdenite is silica. Silica content of 0.5% does not affect the molybdenum disulfide frictional characteristic but effects the wear. Commercially pure molybdenum disulfide contains 0.7 to 1 % carbon, and its the influence has not understood fully, but according to [16], carbon reduces the life of burnished molybdenum disulfide films in great amount.

3. Materials and Methods

In this chapter, the molybdenum concentrate analysis and scanning electron microscopy are discussed to investigate the possibility of using the molybdenum disulfide produced at Erdenet Mining for lubrication.

Also, to mimic the 4-ball wear test, experiment on bench top mill machine is included. There were other experiments to test the lubricating behavior, but the procedure and the results were not suitable for the coarse and impure sample.

During the test processes, the sample material is sealed and stored at room temperature to prevent the ingress of chemicals and moisture.

3.1. Molybdenite Concentrate Analysis

One of the most important factor of molybdenum disulfide lubrication effectiveness is its grade, and the purity. The molybdenite sample is tested at the Central Geological Laboratory of Mongolia.

Validity of the certification

The certified reference material is found in Appendix. This certificate is updated in 2017 in accordance to the requirements of ISO Guide 31 and based on the Geological Survey Laboratory Scientific and Technical Council meeting decision on May 5, 2016. Based on the results of the re-certification, the validity of the CRM certificate was extended until 2027 [16].

Sample description

Central Geological Laboratory has obtained the primary samples of molybdenum concentrate for CRM from the Erdenet, a Mongolian-Soviet concentrator in Orkhon province. Sample processing and packaging was carried out in 1986 by the Central Geological Laboratory [16].

Sample processing

Sample processing, homogeneous analysis and research performed by the Central Geological Laboratory, Sverdlovsk branch of All-Union Metrology Research Institute in 1986. The pulverized sample was homogenized using a mixing device and 95.0% of the pulverized sample was less than 0.071 mm [16].

Attestation process

The molybdenum concentrate attestation analysis was performed by 12 competent laboratories, and their results determined the attested values. In order to ensure the reliability of the measurement results, the "Nested Design" model was selected for the certification analysis. The unit transfer is determined by the SRM 333a Molybdenum Concentrate CRM produced in "National Institute of Standards and Technology, (NIST) " USA. Science and Technology Council of Central Geological Laboratory reviewed and certified that the statistical analysis of results was performed in accordance with international standard and guideline. The certified and informative values were determined by analysis of samples dried at 105ºC [16].

Method used for attestation analysis

Analytical methods used in the concentration analysis is listed in the Table 6.

Table 6. Analytical Methods for attestation analysis

№	Analytical Method	Element compound
1	Gravimetry	Mo, SiO2
2	Titrimetry	Cu
3	Photometer	Cu, Re, SiO2, P
4	Atomic absorption spectrometry	Cu
5	spectrometry	Cu, Mo, P, SiO2
6	Neutron activation analysis	Cu, Re
7	XRF	Mo, Cu, P, SiO2
8	ICP-MS	Re
9	ICP-OES	Cu, Re, SiO2
10	Other	Cu, Re

3.2. Scanning Electron Microscopy

In this part, the lattice structure, hexagonal form, and particle size of molybdenum disulfide were observed and examined from the scanning electron micrographs of the sample. The SEM images were provided by the thesis supervisor. To examine the lattice structure, the micrographs are compared and contrasted with the micrographs of molybdenum disulfide in the literature [4].

In the literature, the investigation of lubricating effectiveness and mechanism of coarse and fine molybdenum disulfide powder, and suspensions of these powders have been carried out by Bartz W.J and Müller K [4].

The mean and maximum values of the dry powder particle sizes as well as the particle size distribution of suspensions of this study that were used for the comparison are listed in Table 7 and Table 8.

Table 7. Particle Size of the Dry MoS2 Powders

Powder	**Particle size (µm)**	
	1	2
Maximum	150-200	5
Mean	3-6	0.2-0.5

Table 8. Particle size distribution of the MoS2 suspensions

Suspension	**Particle Size (µm)**	
	1	2
Maximum	40	5
Some	80	10
Mean	1	2

Most	1-5	1-5

In the molybdenite concentrate analysis of the Central Geological Laboratory, 95 % of the pulverized sample was less than 0.071 mm or 71 µm which can be assumed as the maximum particle size of the sample produced at Erdenet Mining is 71 µm. Although, this amount lies in between the maximum and mean size of the dry powder of the literature, and close to the some of the MoS_2 suspensions particle size in the literature, all the micrographs were comparable.

3.3. Bench Top Mill experiment

The experiment is done to examine the lubrication of molybdenum disulfide. Rocklabs Laboratory Bench Top Mill, Figure 6 is used for a testing machine that pulverizes 1 – 100 gram samples such as rocks, soil, cement, coal, and other materials. Material is ground inside the cup due to the rotation and vibration of the plates in the container Figure 7.

Figure 6. Rocklabs Bench Top Mill

Figure 7. Mill plates and container

The grinding plates' temperature increases up to 70 C due to the friction after grinding small particles for 10 minutes.

This experiment hypothesizes that if molybdenum disulfide has a lubricating behavior, the temperature of the plates should not increase as much as the temperature increases after grinding other materials.

50 grams of molybdenum disulfide is placed in the milling cup and between the plates. The grinding procedure has taken two times for 13 minutes each. The initial temperature of the plates was 21 C as shown in Figure 8 , a thermographic camera is used to capture the temperature.

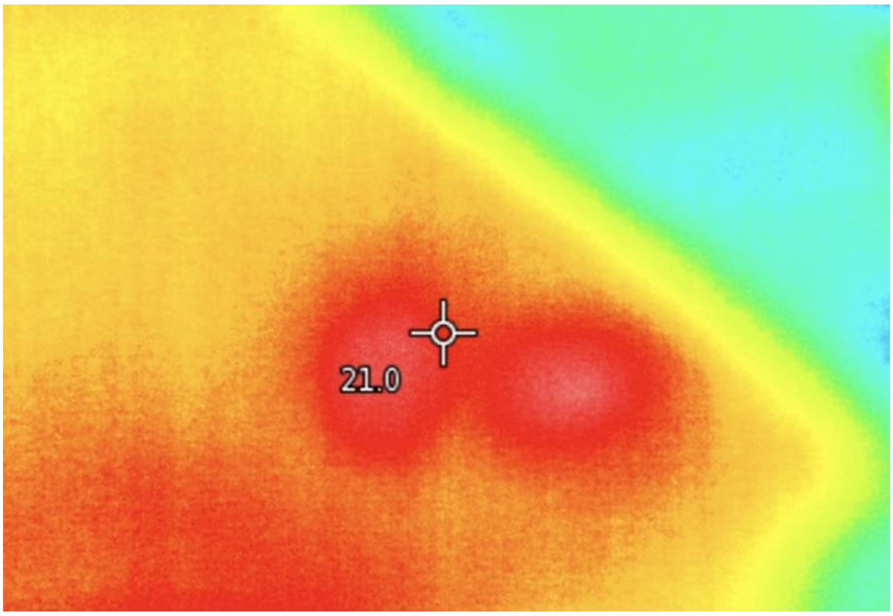


Figure 8. Temperature of the plates before the test

4. Results and Discussion

4.1. Molybdenite Concentrate Analysis

The components of the molybdenite from the concentrate analysis results are listed in Table 9 and Table 10.

Table 9. Molybdenum Concentrate

№	Element	Unit of Measurement	CV^4	$U_{CRM}{}^2$	N^3
1	Mo	%	51.5	0.367	10

1 Certified Value (CV) - value obtained by analyzing 10 and more independent results from two or more analytical methods

2 Extended Uncertainty of Measurement (UCRM) - value obtained by calculating with coverage factor (k) of 2 with a 95% probability according to the ISO / IEC GUIDE 98-3: 2008 "Uncertainty of measurement-Part 3: Guide to the Expression of Uncertainty in Measurement (GUM: 1995) "

3 Number of independent results (N)

Table 10. Other Elements in Molybdenite

№	Element	Unit of Measurement	IV^4	N^3
1	Cu	%	1.34	9
2	SiO2	%	4.50	13
3	Fetotal	%	1.44	7
4	Stotal	%	35.66	9
5	Re	%	0.05	8
6	P	%	0.014	12
7	As	mg/kg	278	5
8	Pb	mg/kg	160	6
9	Ce	mg/kg	16.56	4
10	La	mg/kg	8.7	4
11	Y	mg/kg	1.96	4

4 Information value (IV) - value that does not meet the certification requirements

According to the molybdenite concentrate analysis, the amount of Mo in the molybdenite concentrate is 51.5%, the amount of total S is 35.66% and the remaining impurities are mostly silica.

Molybdenum disulfide concentrate can be calculated by assuming the elements 7-11 in Table 10 are negligible and taking the rest of the elements as 100%. By subtracting the elements 1,2,3,5,6 from 100%, the remaining amount is the molybdenum disulfide concentrate which is 92.656%.

This agrees with the literature [1], and according to Figure 1, the molybdenite concentrate is at the first stage of processing. Using molybdenum disulfide at this stage as a lubricant will produce a high amount of wear [1].

To use it for lubrication, the concentrate has to undergo a further grinding process, acid treatment, and milling and has to be dried and graded. Other remaining impurities after these stages are usually silica, iron, and copper compounds, and other materials may present. If the refining process is done well, the purified powder can reach less than 2% impurities.

4.2. Scanning Electron Microscopy

In Figure 9, micrographs of the coarse MoS_2 powder in the literature have even shape and size distribution. In Figure 10, micrographs of the MoS_2 sample have uneven shapes and sizes, which are not very consistent. This might be due to the impurity of the molybdenite concentrate as discussed in chapter 4.1.

Figure 9. Scanning electron micrograph of the coarse MoS2 powder 1

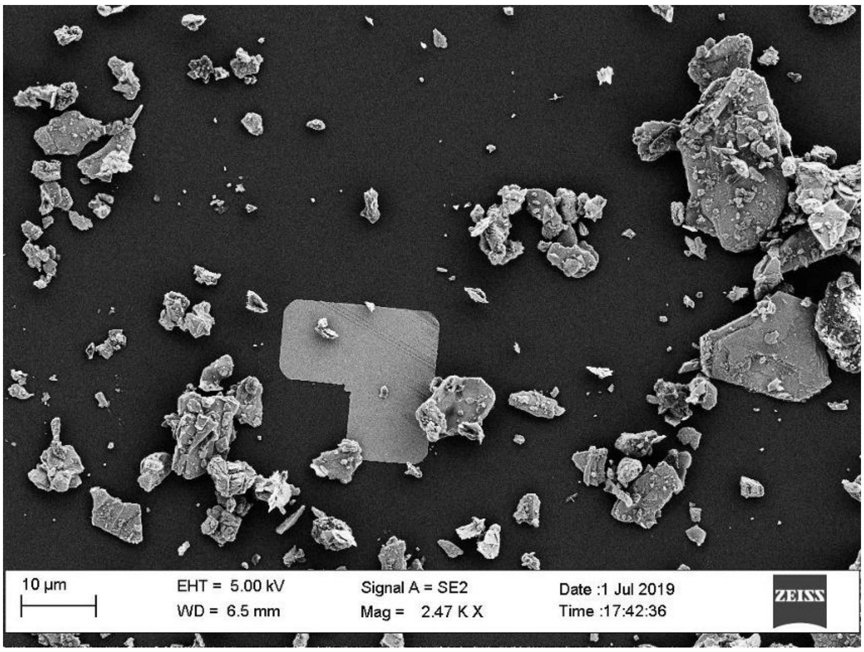


Figure 10. Scanning Electron Micrograph of the sample

In Figure 11, the coarse particle of MoS_2, has almost perfectly shaped typical hexagonal form of molybdenum disulfide. In the micrograph of the MoS_2 sample in Figure 12, hexagonal shape is noticeable, but not as symmetrical as in Figure 11.

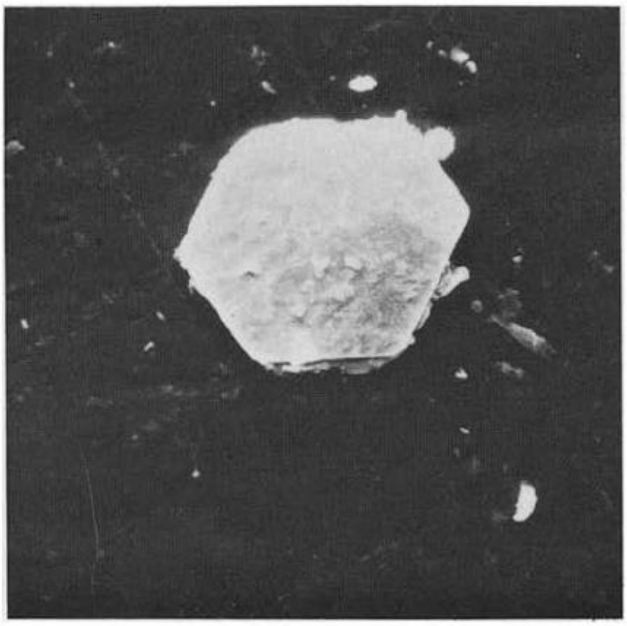


Figure 11. Scanning Electron Micrograph of a Hexagonal MoS2 Particle of the MoS2 Powder 1

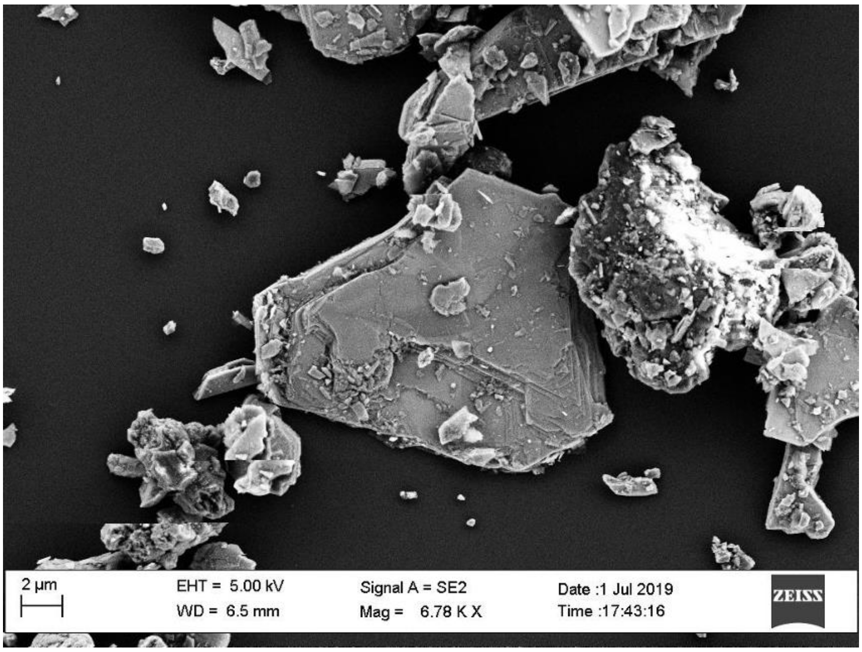


Figure 12. Scanning Electron Micrograph of MoS2 Sample

After the suspension process, the majority of the particles were broken down into 1-5 μm [4] which got closer to the particle size of the study samples. From its micrograph in Figure 13, the larger particle's hexagonal shape is now almost as identical to the MoS_2, particle of the study. The observation confirms that the sample from the Erdenet Mining has the naturally occurring hexagonal crystal form.

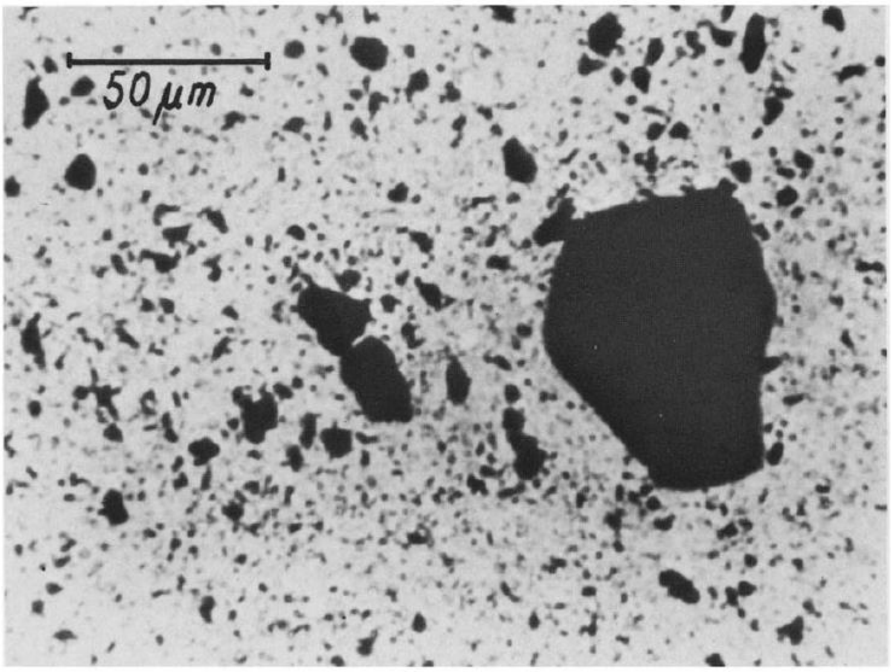


Figure 13. Scanning Electron Micrograph of MoS2 suspension 1

In Figure 14, the layered structure of the sample is observable comparing it with Figure 15 which is found in the literature, it clarifies that the MoS_2, sample from Erdenet Mining has the lattice layers.

Figure 14. Lattice Structure of the MoS2 suspension 2

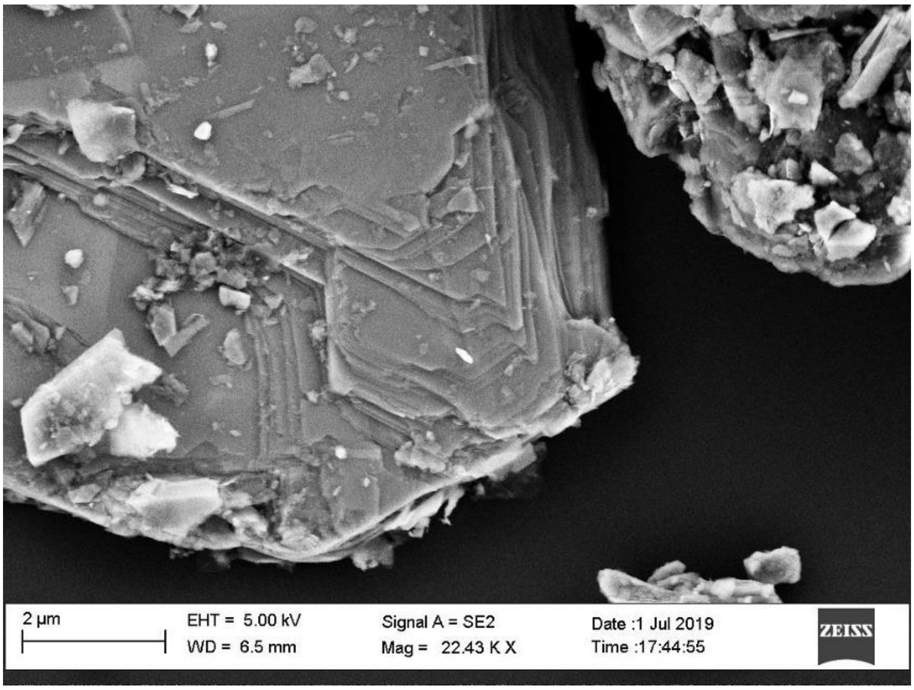


Figure 15. Lattice Structure of the MoS2 Sample

4.3. Bench Top Mill Experiment

Final temperatures of the mill plates after each test are shown in Figure 16 and Figure 17.

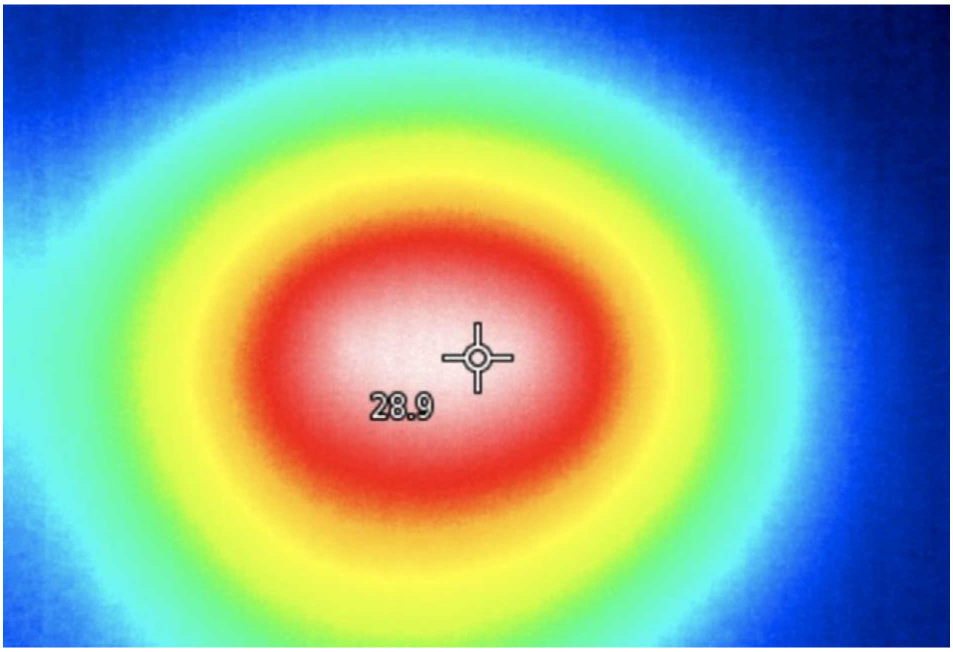


Figure 16. Temperature after the test 1

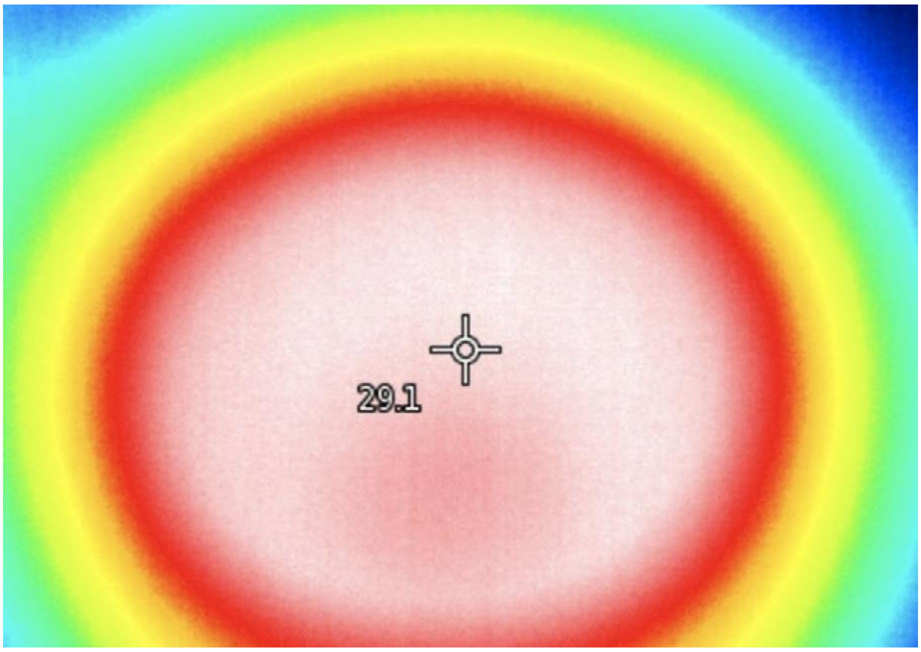


Figure 17. Temperature after the test 2

As discussed in 2.4. , when the sliding occurs between the surfaces, lateral force adding to the relatively high contact load causes adhesion. The energy required in deformation and adhesion resistance adds up and contributes to the total friction. The heat generated to deform the material surfaces in micro scale is transferred into the mill plates. Since the friction and the generated heat is directly proportional, the lower the generated heat, lower the frictional force.

From the resulting temperature increase, it indicates that molybdenum disulfide sample has much lower friction and do have a lubricating behavior compared to other materials.

5. Case Study

Tribological Testing by 4 Ball Method

Wear track and surface examination can be done for the refined molybdenum disulfide powder with high purity which is applicable for the lubricant application. The tribological properties including coefficient of friction, wear and lubrication are tested by the 4-Ball Wear tester. In this part, a conference paper “Spectrum Loading in Milled Greases Using Four-Ball Wear Test” by Gabi Nehme, University of Balamand is reviewed to perceive the further possible tribological test of the molybdenum disulfide produced at the Erdenet Mining LLC.

According to the ASTM 2266 standard, the 4-Ball Wear test is extensively conducted to examine the tribology behavior of the lubricants. The load on the four-ball test machine simulates the rotational speed of the real condition with a chromium steel ball. A series of a test is done on each condition. The grease has to be heated up to 75 ºC, and the speed and the load applied to the wear tester are adjusted by the software application of the testing machine. The different loadings and speed rate is analyzed by the Design of Experiments (DOE). After the testing procedure, scanning electron microscopy of the steel ball surface wear is paired with EDS (energy dispersive spectroscopy) responses to analyze the steel balls’ surface wear scar diameter.

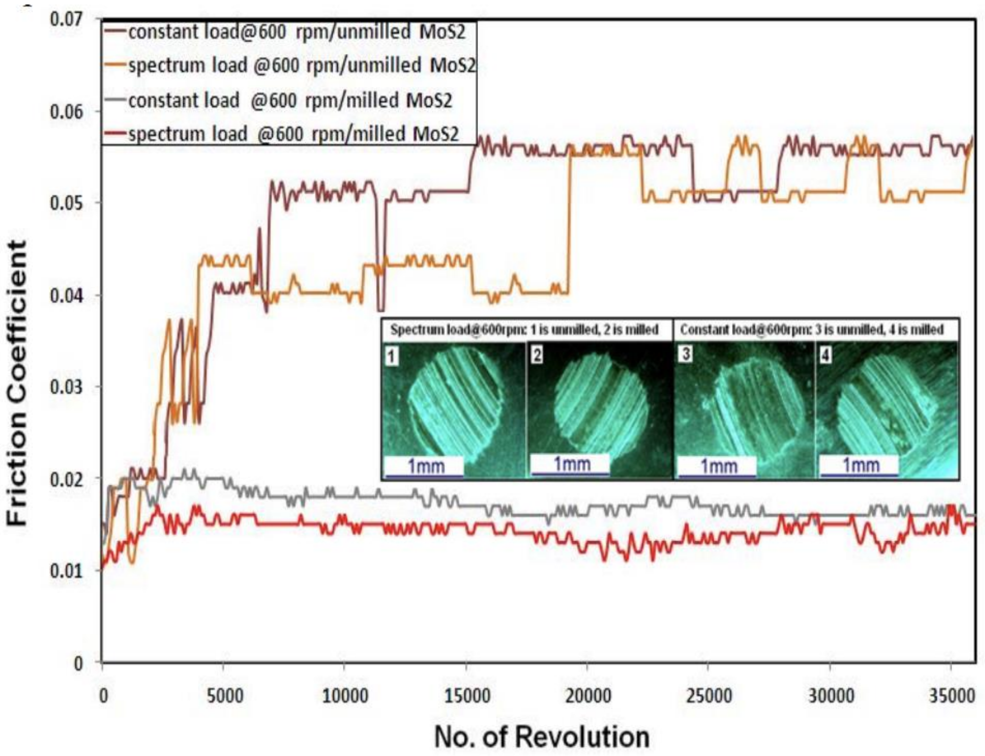


Figure 18. Friction Coefficient vs. Number of Revolutions and Wear Scar at 600 rpm

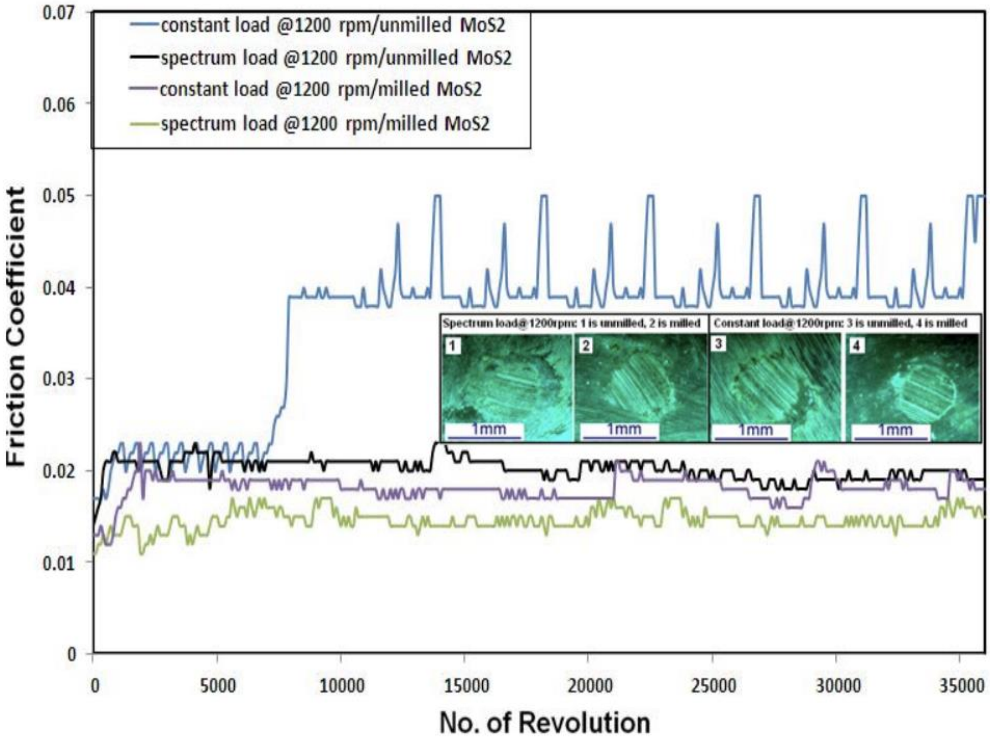


Figure 19. Friction Coefficient vs. Number of Revolutions and Wear Scar at 1200 rpm

In the Figure 18 and Figure 19, the friction coefficients at different loads at the number of revolutions and the wear scar micrograph of the test are shown.

In the further investigation of the molybdenum disulfide produced in Erdenet Mining, it is possible to test the sample alone or adding other additives to compare the effectiveness of the lubrication. The results will indicate the improvements of the solid lubricant if other additives are added.

5. Conclusion

According to the element analysis and SEM results, the molybdenite produced at the Erdenet Mining is a potential solid lubricant. In the concentrate analysis, the amount of molybdenum disulfide in molybdenite has a high concentration but not enough to use as a dry lubricant. Further purification processes including grinding, acid treatment, milling, and grading are needed to be applicable for dry lubrication and meet the technical standard of purity.

The scanning electron micrographs clarified molybdenum disulfide sample has the typical hexagonal form and layered lattice structure. The layered structure is one of the most important factors in lubricating mechanism of molybdenum disulfide. Also, by observing the micrographs, the particle size distributed unevenly which shows the impurities. The particle size was coarse compared to the fine powders of molybdenum disulfide used in the lubrication market. Further grinding is needed to obtain lubricant grade molybdenum disulfide.

Following the purification and size reduction, the effectiveness of the molybdenum disulfide can be tested by the 4-ball test method. Its tribological properties can be compared with high-quality molybdenum disulfide powders on the market to obtain a fine grade. If molybdenum disulfide with finer grade and higher quality is obtained, it will increase the export price.

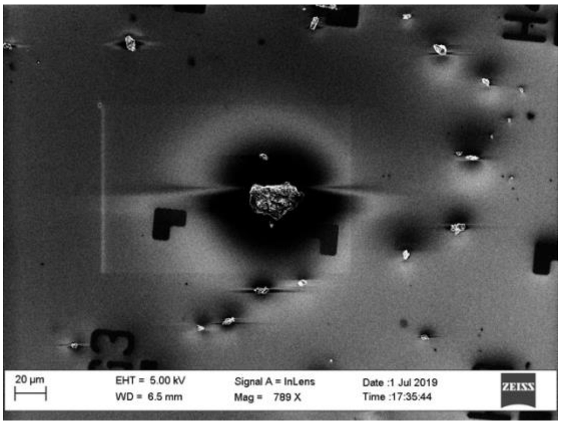
The purpose of this study was to investigate the molybdenum disulfide extracted from Erdenet Mining as solid lubrication alone. However, using molybdenum disulfide as a free powder for lubrication has limited application and effectiveness for lubrication when used alone. It can only be used with simple mechanisms and low technology.

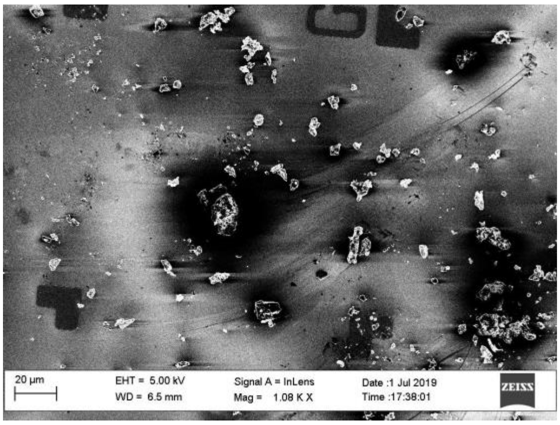
The behavior of molybdenum disulfide is confusing, for the direct application it is not possible to determine the optimum operating temperature range, humidity, particle size, and substrate types. All these effects must be taken into account to transfer individual results under one condition.

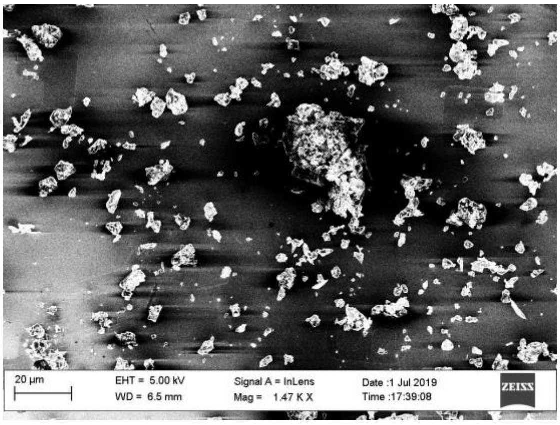
In conclusion, the molybdenite produced at Erdenet Mining has a potential to be used in a solid lubrication. It can be exported at higher price if undergoes further refining process. Based on the research, it is improbable to use 100% molybdenum disulfide lubricant in majority of the application without other constituents.

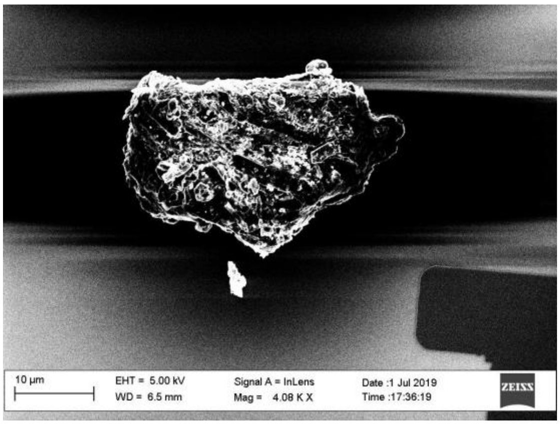
Appendix

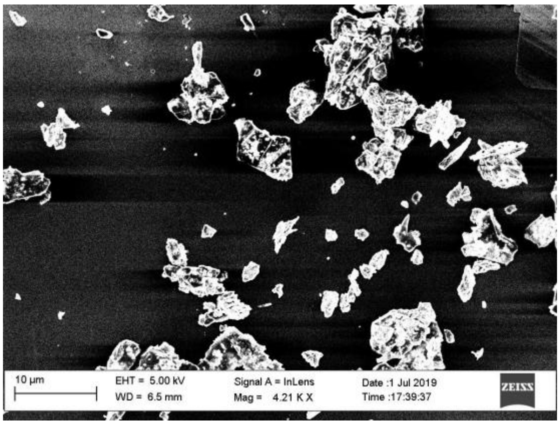
Scanning Electron Micrographs







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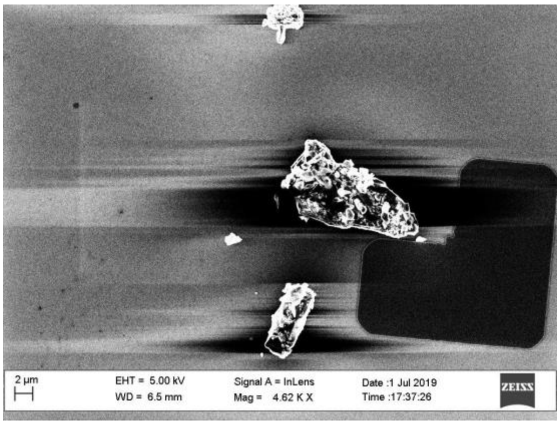
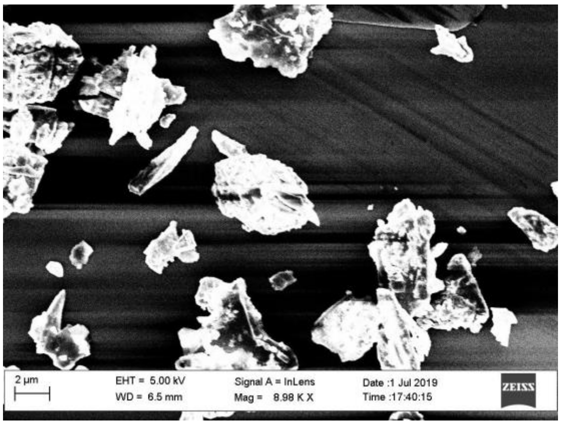
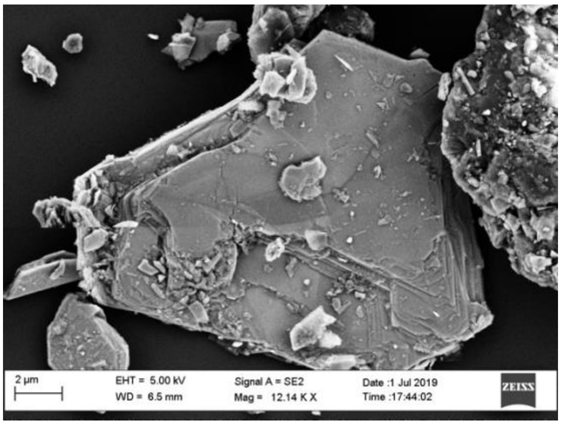
EHT = 5.00 kV
WD = 6.5 mm
Signal A = InLens
Mag = 4.21 K X
Date :1 Jul 2019
Time :17:39:37
ZEISS


2 µm
EHT = 5.00 kV
WD = 6.5 mm
Signal A = InLens
Mag = 4.62 K X
Date :1 Jul 2019
Time :17:37:26
ZEISS


2 µm
EHT = 5.00 kV
WD = 6.5 mm
Signal A = InLens
Mag = 8.98 K X
Date :1 Jul 2019
Time :17:40:15
ZEISS


2 µm
EHT = 5.00 kV
WD = 6.5 mm
Signal A = SE2
Mag = 12.14 K X
Date :1 Jul 2019
Time :17:44:02
ZEISS

МОНГОЛ УЛС
ГЕОЛОГИЙН ТӨВ ЛАБОРАТОРИ

СТАНДАРТЧИЛСАН ЗАГВАРЫН ГЭРЧИЛГЭЭ

(Аттестатчилсан стандартчилсан загвар)

CGL 202
MoB (МОЛИБДЕНИЙ БАЯЖМАЛ)

Аттестатчилсан утга

№	Элемент нэгдэл	Хэмжих нэгж	CV[1]	U_{CRM}[2]	N[3]
1.	Mo	%	51.5	0.367	10

[1]*Аттестатчилсан утга (CV) - хоёр болон түүнээс дээш шинжилгээний арга хэрэглэн, 10 болон түүнээс дээш бие даасан үр дүнгээр статистик боловсруулалт хийж гарган авсан утга*
[2]*Хэмжлийн өргөтгөсөн эргэлзээ (U_{CRM}) -ISO/IEC GUIDE 98-3:2008 "Uncertainty of measurement-Part 3: Guide to the Expression of Uncertainty in Measurement (GUM: 1995)" стандартын дагуу 95%-ийн үнэмшилт магадлалтай, хамруулах коэффициент (k)-ыг 2-оор авч тооцоолон гаргаж авсан утга*
[3]*Бие даасан үр дүнгийн тоо (N)*

2016 онд зохион байгуулсан дахин аттестатчиллаар элементүүдийн аттестатчилсан утга, тэдгээрийн хэмжлийн эргэлзээг шинээр тогтоож дахин баталгаажуулсан болно.

Нэмэлт мэдээлэл

№	Элемент	Хэмжих нэгж	IV[4]	N[3]
1	Cu	%	1.34	9
2	SiO_2	%	4.50	13
3	$Fe_{нийт}$	%	1.44	7
4	$S_{нийт}$	%	35.66	9
5	Re	%	0.05	8
6	P	%	0.014	12
7	As	мг/кг	278	5
8	Pb	мг/кг	160	6
9	Ce	мг/кг	16.56	4
10	La	мг/кг	8.7	4
11	Y	мг/кг	1.96	4

[4]*Мэдээллийн утга (IV) – аттестатчилах шаардлага хангаагүй утга*

Зориулалт

Молибдений баяжмалын Аттестатчилсан Стандартчилсан Загвар (АСЗ)-ыг түүний хэмжилзүйн (аттестатчилсан утгын хэмжилзүйн нэгж дамжуулалт, хэмжлийн эргэлзээ) болон физик шинж чанар (нэгэн төрөл, ширхэглэгийн хэмжээ)-ын үзүүлэлтүүдэд үндэслэн ижил матрицтай дээжийн шинжилгээний чанарын хяналт, чанар хангалт, арга баталгаажуулалт, багаж тоног төхөөрөмжийн калибровкад хэрэглэнэ.

Дээжийн тодорхойлолт

Молибдений баяжмалын АСЗ-т зориулсан анхдагч дээжийг Геологийн Төв Лаборатори Орхон аймаг дахь Монгол-Зөвлөлтийн уулын баяжуулах Эрдэнэт үйлдвэрээсавсан болно.
Дээжийн боловсруулалт, савлалтын ажлыг 1986 онд Геологийн Төв Лаборатори гүйцэтгэсэн.

Дээж боловсруулалт

Дээжийн боловсруулалт, нэгэн төрлийн шинжилгээ, судалгааг Геологийн Төв Лаборатори, Бүх холбоотын хэмжилзүйн эрдэм шинжилгээний институтийн Свердловскийн салбар 1986 онд гүйцэтгэв.

Нунтаглагдсан бөөн дээжийг холигч төхөөрөмжөөр нэгэн төрлийн болгон хольсон бөгөөд ширхэглэгийн шинжилгээгээр нунтаглагдсан дээжийн 95.0 % нь 0.071 мм-ээс бага хэмжээтэй болох нь тогтоогдсон.

Баталгаажуулалт

2016 оны дахин аттестатчилал

ГТЛ–ийн Шинжлэх ухаан, Техникийн зөвлөлийн хурлын СЗ-ыг дахин аттестатчилах тухай № 14/11-3 тоот шийдвэрийн дагуу CGL 202 Молибдений баяжмалын СЗ-ыг дахин аттестатчилах ажлыг 2014-2016 онд зохион байгуулсан.

Молибдений баяжмалын аттестатчиллын шинжилгээнд ур чадвартай нь тогтоогдсон 12 лаборатори оролцож, тэдгээрээс ирүүлсэн үр дүнгээр аттестатчилсан утгыг тогтоосон. Хэмжлийн үр дүнгийн найдвартай байдлыг хангахын тулд аттестатчиллын шинжилгээнд "Nested design" загварыг сонгон хэрэглэсэн.

Нэгж дамжуулалтыг АНУ-ын "Стандарт болон Технологийн Үндэсний Хүрээлэн, (NIST)"–д үйлдвэрлэсэн SRM 333a Молибдений баяжмалын АСЗ-аар тогтоосон.

Дахин аттестатчилал, шинжилгээний үр дүнгийн статистик боловсруулалтыг олон улсын стандарт, удирдамжийн дагуу гүйцэтгэсэн болохыг ГТЛ-ийн Шинжлэх Ухаан Техникийн Зөвлөл хянаж, баталгаажуулав.

1986 оны аттестатчилал

Аттестатчилах утгыг найдвартай тогтоохын тулд лаборатори хоорондын аттестатчиллын шинжилгээнд 38 лаборатори сонгон оролцуулав.

АСЗ-ын зохион бүтээлт, шинжилгээний үр дүнгийн статистик боловсруулалтыг СТ СЭВ 5892-87 стандартын дагуу гүйцэтгэж БНМАУ-ын Үнэ, Стандартын

Улсын хорооны Улсын хэмжилзүйн алба 1988 онд, ЗСБНХУ-ын Стандартын хорооны Бүх холбоотын Хэмжилзүйн эрдэм шинжилгээний институтийн Свердловскийн салбарт 1986 онд хэмжил зүйн экспертиз хийлгэсэн болно.

Тус АСЗ нь БНМАУ-ын Үнэ, Стандартын Улсын хорооноос УСЗ 5-88 дугаар, ЗХУ-ын Стандартын хороогоор батлагдан ГСО 3587-86 дугаараар бүртгэгдсэн болно.

Хэрэглэх заавар, хадгалалт

Химийн бодис, агаарын чийг нэвтрэхээс хамгаалж битүүмжлэн, тасалгааны температурт хадгална. Бүх төрлийн тээврийн хэрэгслээр тээвэрлэж болно.

Аттестатчилсан болон мэдээллийн утгуудыг 105^{0}С-д хатаасан дээжинд хийсэн шинжилгээгээр тогтоосон болно.

АСЗ-ыг бохирдохоос сэргийлэхийн тулд тасалж авсан хэсгийг саванд нь буцааж хийхийг хориглоно.

АСЗ-ын төлөөлж чадах хамгийн бага масс 100 мг байна. Хэрэв шинжилгээний аргад 100 мг–аас бага масстай хэрэглэхээр заагдсан тохиолдолд шаардлагатай массыг жигнэхийн өмнө төлөөлж чадах хамгийн бага массаас багагүйг (>100 мг) тасланавч, гартаам нухуурт дахин нунтаглах шаардлагатай.

Материалын аюулгүй ажиллагааны заавар энэхүү гэрчилгээнд дагалдана.

Гэрчилгээний хүчинтэй хугацаа

Дахин аттестатчиллын үр дүнг үндэслэн тус АСЗ-ын гэрчилгээний хүчинтэй хугацааг 2027 он хүртэл сунгав. Хүчинтэй хугацааг дуустал материалын тогтвортой байдалд тогтмол хяналт хийгдэнэ.

Үйлдвэрлэгчийн хаяг

Энэхүү молибдений баяжмалын АСЗ-ыг **CGL 202** дугаараар дараах хаягаар авах боломжтой.

Геологийн Төв Лаборатори
Монгол улс
18080 Улаанбаатар хот
Шуудангийн хайрцаг-437
Сонгинохайрхан дүүрэг
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Вэб: www.cengeolab.com

Хэрэглэгчийн эргэх холбоо

Хэрэглэгч нь Геологийн Төв Лабораторийн бүртгэлд орсноор тухайн АСЗ-тай холбоотой шинэ мэдээллийг хүлээн авах боломжтой. Энэхүү гэрчилгээнд өгөгдсөн мэдээллийн талаарх санал хүсэлтээ ирүүлбэл талархах болно.

Аттестатчиллын шинжилгээнд хэрэглэсэн арга

№	Шинжилгээний арга	Элемент нэгдэл
1.	Жингийн арга (Gravimetry)	Mo, SiO_2
2.	Эзлэхүүний арга (Titrimetry)	Cu
3.	Фотометр (Photometer)	Cu, Re, SiO_2, P
4.	Атом шингээлтийн спектрометр (Atomic absorption spectrometry)	Cu
5.	Спектрометр (Spectrometry)	Cu, Mo, P, SiO_2
6.	Нейтрон идэвхжилийн шинжилгээ (Neutron activation analysis)	Cu, Re
7.	Рентгенфлуоресценцийн спектрометрийн арга (XRF)	Mo, Cu, P, SiO_2
8.	Индукцийн холбоост плазм-масс спектрометрийн арга (ICP-MS)	Re
9.	Индукцийн холбоост плазм-оптик спектрометрийн арга (ICP-OES)	Cu, Re, SiO_2
10.	Бусад	Cu, Re

1986 онд аттестатчиллын шинжилгээнд оролцсон байгууллага, улс

1. АК-Тюзскийн хүдэр боловсруулалтын удирдах газар, ЗХУ (АК-Тюзское рудоуправление, СССР)
2. Акчатаускийн уулын баяжуулах үйлдвэр, ЗХУ (Акчатауский комбинат, СССР)
3. Бүх холбоотын Тугоплавкийн металл болон хатуу хайлш дах шинжлэх ухааны судалгаа шинжилгээний институт /ВНИИТС/, Москва, ЗХУ (Институт ВНИИТС, СССР)
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Тайлбар

Энэхүү гэрчилгээ нь ISO Guide 31-ийн шаардлага болон Геологийн Төв Лабораторийн Шинжлэх Ухаан Техникийн Зөвлөлийн 2016 оны 5-р сарын 05-ны өдрийн хурлын шийдвэрийг үндэслэн 2017 онд шинэчлэл хийгдсэн хувь болно.

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Геологийн Төв Лабораторийн захирал **Ө.Оюунбаатар**

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