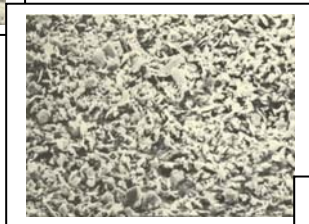


Molysulfide[®]

Particle Size Analysis



INTRODUCTION

Molysulfide[®] is the Climax Molybdenum Company trade name for lubricant grade molybdenum disulfide (MoS₂). The mineral designation for MoS₂ is Molybdenite which is the naturally occurring form of molybdenum. When the mineral is mined, processed and purified by Climax, it becomes Molysulfide[®].

It is used by itself as a lubricant and as an additive to greases, fluids, bonded coatings, and plastic and metallic composites. Its function is to reduce friction and wear as well as to increase load-carrying capability.

Molysulfide[®] is available commercially in three grades, Technical, Technical Fine and Super Fine, and are mainly differentiated by particle size, Technical Grade being the coarsest and Super Fine the finest. Molysulfide[®] consists of irregularly shaped platelets having a large diameter to thickness ratio which accounts in part for the fact that particle size and distribution values will vary depending on the method used to measure it.

Randomly selected multiple lots of each particle size grade (Technical, Technical Fine and Super Fine) were used throughout this study. These lots are typical of standard production material produced in Climax Molybdenum facilities in the USA and the UK.

PARTICLE SIZE ANALYSIS

Particle size analysis can be conducted using a diverse number of instruments and techniques. These include the traditional optical and electron microscopy tools, and sieve analysis. Instrumental techniques include the rapid and simple Fisher Number (which measures an average particle size), and more sophisticated methods such as Microtrac[®] and Malvern Mastersizer, that utilize laser light scattering principles and self-contained software to obtain particle size distribution data.

The particle size of three grades of Molysulfide[®] produced by Climax Molybdenum, Technical, Technical Fine and Super Fine was compared using these methods. Table 1 presents an overview of the Molysulfide[®] particle size information obtained.



Figure 1 Technical Grade Molysulfide[®]

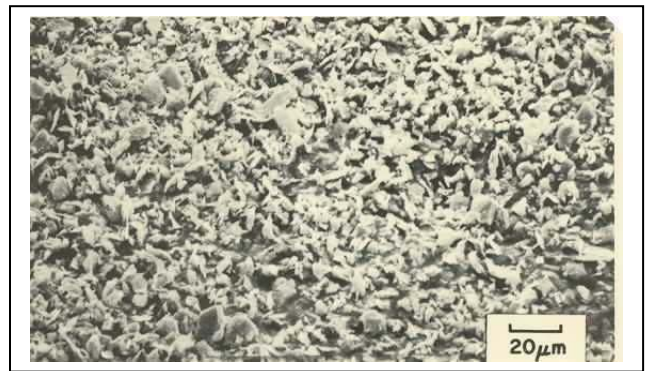


Figure 2 Technical Fine Grade Molysulfide[®]



Figure 3 Super Fine Grade Molysulfide[®]

Specific information on each method is detailed in the following sections.

Fisher Number - This number is an average particle size obtained using the Fisher Sub-Sieve Sizer manufactured by Fisher Scientific, Pittsburgh, PA. The instrument uses an air permeability principle where the pressure drop of air flowing through a packed column of the material is measured. The

change in pressure is related to *average* particle size in μm using a special empirically developed chart. The average particle size so obtained is not intended to be absolute and, in fact is not numerically similar to average particle size obtained by other particle size analyzers. Nevertheless, the Fisher Number has been found to be useful in MoS_2 production for lot-to-lot comparison as a quality control measure. It does not, however reveal any information regarding particle size distribution in a test sample.

As shown in Table 1, Technical Grade Molysulfide[®] typically gives a Fisher Number of 3 to 4 μm , Technical Fine Grade gives a Fisher Number of 0.65 to 0.8 μm , while Super Fine ranges from 0.4 to 0.5 μm .

Sieve Analysis - This technique is perhaps the oldest and most widely used method of particle size analysis and gives data in terms of weight (mass) fraction versus sieve mesh size (aperture). The sieves used in this analysis comply with the International Sieve Series (ISO 565), U.S. Sieve Series specification (ASTM E11) or one of the European standards, e.g., British Standard 410. The different European Sieve Series vary somewhat from each other in the larger sieves but are essentially identical at apertures less than 1000 μm .

The test is conducted with a nested stack of sieves, each one having smaller openings than the one above it. A typical stack of sieves may consist of four elements such as 80, 100, 200, and 325 mesh sieves and the collection pan below the lowest sieve. The mass % retained or passing through specific sieves is reported relative to mesh size or sieve opening in μm . Standard sieve analyses are usually not conducted on dry powders using sieves smaller than 325 mesh (45 μm). Technical Grade Molysulfide[®] has a typical sieve analysis as shown in Table 1. Technical Fine and Super Fine can not be analyzed using sieves. These grades are too fine for meaningful sieve analysis due to the tendency of particles to cling to small sieve openings causing erroneous results. In addition, due to the presence of a small amount (~0.3%) of a highly refined oil, these grades contain loosely held agglomerates. The oil is purposely added to minimize surface

oxidation of the MoS_2 during micronization and to enhance subsequent long-term storage properties.

Care should be taken when comparing sieve results from different sources as the sieving time and techniques, such as brushing to break agglomerates, significantly affect results.

SEM Analysis - Microscopy of various types, optical, transmission electron and scanning electron (SEM), can be used to obtain visual images of particle size and shape of solids. SEM images can be processed with computer programs to obtain particle size distribution data but this technique is not suitable for quality or production control. The shortcoming of microscopy as a particle size tool is unrepresentative sampling. Only a small number of particles are viewed at one time, and to get true statistical sampling, several thousand images should be examined, each one requiring special sample preparation. However, SEM is valuable for presenting comparative visual photomicrographs of the three grades of Molysulfide[®]. SEM photomicrographs, shown in Figures 1-3, were selected from numerous fields of each grade and are considered representative. The particle size range observed for each grade in photomicrographs is shown in Table 1.

Laser Diffraction - This type of particle size analysis, now preferred in many industries, uses light scattering (diffraction angle) and Fraunhofer diffraction theory to obtain particle size distribution, median and average particle size. The instruments use microprocessors and appropriate programmable software to obtain such data. Particle shape, density and refractive index values are chosen in the software menu to match the solid material being tested as closely as possible. In North America, the most widely used instrument of the laser diffraction type is Microtrac[®], a division of Nikkiso, Ltd., whereas in Europe, other instruments such as Malvern (Malvern Instruments, Ltd.) and Sympatec-Helos are more common. Other manufacturers of laser diffraction instruments include Beckman-Coulter and Horiba.

Typically, the sample of the solid being measured is dispersed (with the assistance of ultrasonic energy)

in water (and a wetting agent) or a polar organic solvent (e.g., 2-propanol). The sample dispersion, in a special cell, is circulated through the detector section of the instrument. The data generated may be presented as a histogram showing the % of particles falling within specified size ranges, or 90%, 50% and 10% of particles smaller than a specific particle size. Table 1 shows the typical particle size range reported by Microtrac[®] and Malvern Mastersizer[®] in Climax Technical, Technical Fine and Super Fine grades.

Other Instrumental Techniques - Other methods used in some industries to measure particle size distribution include sedimentation and electrozone sensing.

Sedimentation methods rely on Stokes' Law, which relates particle size (equivalent spherical diameter) to the rate of settling in a fluid of known viscosity.

Electrozone sensing is based on the principle of electrical resistance change of an electrolyte containing the dispersed solid circulating through an "electrozone." Voltage pulses caused by individual particles are accumulated by a microprocessor to produce a particle size distribution.

No data on Climax Molybdenum Sulfide[®] was obtained using these methods, but experience has shown that these instruments give smaller particle size values than the laser techniques.

SUMMARY

This bulletin has attempted to acquaint the reader with the various particle size measurement principles and devices used by Climax to describe its products. The particle size and/or distribution of the three commercially available grades of Climax Molybdenum Sulfide[®], Technical, Technical Fine and Super Fine, were measured using the most common methods found in industry today. These methods include Fisher, Sieve Analysis, SEM, Microtrac[®],

and Malvern MasterSizer[®]. One of the most important things to keep in mind is that, for a given sample of MoS₂, numerical values of particle size vary according to the instrument used to measure them. Thus, a particle size number listed in a specification should always be accompanied by a reference to the relevant test method.

GENERAL REFERENCES

1. A. Rawle, Basic Principles of Particle Size Analysis, Malvern Instruments Ltd., UK, 2001
2. H. G. Barth, ed., "Modern Methods of Particle Size Analysis", John Wiley & Sons, New York, 1984.

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TABLE 1
PARTICLE SIZE OF MOLYSULFIDE[®] BY VARIOUS METHODS

Molysulfide [®] Grade	Fisher Number, μm	Sieve Analysis (U.S. Sieve Series) Mass % retained	Particle Size Range by SEM, μm	Particle Size Distribution by Laser Instrument, μm	
				Microtrac X100 ²	Malvern Mastersizer ³
Technical	3 to 4	0% on 100 mesh (150 μm) 3% on 200 mesh (75 μm) 16% on 325 mesh (45 μm) 81% through 325 mesh	<1 to 100	99% <123 50% <26.2 10% <7.05	90% <78.38 50% <31.59 10% <9.44
Technical Fine	0.65 to 0.80	Not applicable ¹	<0.5 to 20	99% <18.51 50% <4.29 10% <1.20	90% <11.82 50% <5.22 10% <1.97
Super Fine	0.40 to 0.45	Not applicable ¹	<0.5 to 8	99% <6.5 50% <1.55 10% <0.86	90% <4.38 50% <2.05 10% <1.00

Notes:

1. Technical Fine and Super Fine contain ~0.3% oil that creates loosely held agglomerates; fine particles also tend to blind sieve openings.
2. Microtrac Model X100; MoS₂ is dispersed in DI water + wetting agent
3. Malvern Mastersizer 2000; MoS₂ is dispersed in 2-propanol