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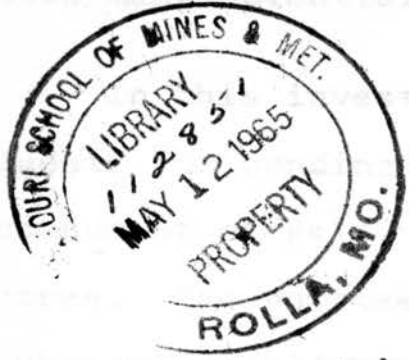
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USE OF SPENT SULPHITE LIQUOR FOR STABILIZATION
OF FRACTURED ROCK AND SEALING OFF
WATER BEARING FORMATIONS

by

LAXMAN SINGH SUNDA, 1940



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A

THESIS

submitted to the faculty of the
UNIVERSITY OF MISSOURI AT ROLLA

in partial fulfillment of the requirement for the

Degree of

MASTER OF SCIENCE IN MINING ENGINEERING

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ABSTRACT

The paper industry, in its process of manufacturing pulp from wood, produces large quantities of a substance called lignin, lignin sulphite, or lignosulphonate. This material has been disposed of as a waste product; however, it does have many potential uses.

In this investigation, lignosulfonates were used as agents for bonding rock sections together and, as the chief component of gels, for filling the pores of artificial rock cores. The purposes of these tests were to find how this substance could be used in the mining industry for stabilizing fractured ground around mine openings and for sealing off water influx into mine openings.

Strength tests conducted on cemented sections of rock and permeability tests conducted on the sealed cores indicated that lignosulfonates would make effective stabilizing and sealing compounds, when used under the proper physical conditions and when the correct proportions of chemicals were employed.

A major advantage of this substance over other cementing or sealing agents is its low cost, which in most cases would be far less than any other known substances used for these purposes.

ACKNOWLEDGMENTS

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I. INTRODUCTION

The purpose of this research project was to determine the effectiveness of spent sulphite liquor, a waste product from paper manufacturing, (1) as a cementing agent for the stabilization of fractured rock around underground openings, and (2) as a grout for reducing the permeability of rock to prevent water influx into excavations.

Several types of grouts that have been used for this purpose are: Portland cement; chemicals; or different combinations of these with bentonite, fly ash, sand, and other filling agents. Chemical stabilization is very effective in sealing off water by reducing the permeability of a treated zone of rocks in shaft sinking, railroad tunnels, oil well drilling, and other construction projects. Guniting is very popular as a procedure for stabilizing fractured ground, where roof bolting is impractical and the skin support system is very expensive.

In untreated fractured grounds, maintenance consists of repairing the loose and fractured rocks along the passage ways. This is done by removing the loose rock and providing artificial support. Unfortunately, this is not always satisfactory, because it may not prevent further ground movement. This often results in repeated scaling and re-bolting throughout the life of the opening. Stabilization of the rock by cementation can help to simplify this problem.

Ground water infiltration causes troubles in all phases of mining activities from exploratory boring to actual ore stoping. Shaft sinking through fractured and water bearing strata is very expensive. It introduces delay elements in mine development and hampers production by reducing manpower efficiency. Pumping costs are often an important part of the total mining cost. In the past, mines have been closed permanently due to severe water problems.

Water percolation in urban transportation tunnels prevents them from serving their purpose, while in railroad tunnels, water freezing on rails may produce loss of traction and possible accidents. Icing in the winter season makes shaft winding troublesome. It also reduces the cross-sectional area of a shaft by introducing frictional elements, causing a reduction in the air supply for mine ventilation.

The techniques of cement grouting are relatively simple and fairly well understood by engineers, but the cement slurries cannot effectively penetrate fine fissures and porous rocks to achieve a high degree of water shut-off. Due to the long setting time for cements and the rapid flow of water, cement could be washed out of its desired position, resulting in heavy cement loss. Chemical grouts often offer a solution to such technical problems.

The limited effectiveness of Portland cements leads to the use of inexpensive chemicals or freezing methods. The

latter methods are extremely expensive and limit the use of explosives. Chemicals like Am-9, Terra Firma, and sodium silicate have been used at several places.

A lack of knowledge of chemicals suitable for this purpose exists throughout the mining industry. The purpose of this investigation was to find an inexpensive chemical which could be used successfully in any size of construction or tunneling job and mining operations. The spent sulphite liquor was suggested by Professor C. R. Christiansen, considering Indian resources and the potential of their mining industry. Large quantities of sulphite liquor are produced by Indian paper mills, most of which is disposed of as a waste product. Thus, it may be possible for this product to be used in the mining and construction industries to solve water and stability problems inexpensively, and at the same time solve a waste disposal problem for the paper industry.

II. REVIEW OF LITERATURE

The chemical stabilization of fractured and water-bearing strata started about 100 years ago in the mining industry. According to H. Standish Ball,¹ the first application on record of the cementation process took place in the year 1864 in the Rhein-Preussen pits near Hamburg, Germany. A water opening in the brick lined shaft was treated by running milk of cement into the break by using a hand pump from the surface. Evidence is available to show that similar operations were conducted in France by M. Porter² about the same time.

L. Sarosi and A. Fitzek³ have mentioned that cement was injected behind railway tunnel walls in the 1870's to fill cavities and to prevent seepage of water. In 1884 the English engineer, Hosegood, when building a bridge in India, tried to bind the sand with some kind of chemical, the nature of which is unknown to us. The first application of the Sodium Silicate Injection Method also was made in the last Century (1887).

Numerous attempts had been made in Pas-DeCalais, a mining district of France. In 1896, M. A. Francois developed a method of drilling and grouting inside the shaft and below the permanent water level. This proved to be an effective method for the stabilization of fissured and fractured rocks. In 1904, C. Diniore⁴ described in detail an

application of the cementation process developed by E. Reaumaux that dealt with water-bearing fissures in chalk in the sinking of the No. 16 shaft at Lengs.

The other early applications to shaft sinking, which have been recorded, took place at the Franz Fredrick shaft in Mecklenburg (1901), Reimsdorf in Saxony (1903), and at Bethune No. 11 shaft in Belgium (1904).

The cementation process was known and tried in America at an early date. In a paper read by F. Donaldson⁵ in 1916 there is reference to the successful use of neat cement for displacing water by forcing it back into the rocks or ground. The Devonian Dundee formation at South Detroit, Michigan, which overlay the Silurian salt bed, was full of crevices and caves, heavily charged with strong H₂S water. In this, and most other applications, cementation operations were usually of the nature of salvage operations, after pumping had been unable to cope with the water problems at relatively shallow depths.

In the past Century, several applications have been made by drilling an arbitrary number of vertical or inclined holes spinning away from shaft peripheries. Millions of feet of holes were drilled, and thousands of tons of cement had been injected. The quantities of cement used were so enormous that fine-grained sand and slag were mixed in grouts. Their characteristics when used in a 50 per cent

mix with pure Portland cements were not dissimilar to straight Portland cement slurries. The most difficult pre-cementation job of shafts, so far, was the West Drienfontein No. 3 in South Africa. This project required 54,311 tons of cement and 38,832 tons of filler to produce tight ground. The footage drilled amounted to 155,000 feet, and the depth of the treated dolomite zone was 2,943 feet.

The freezing method was introduced by Poetsch near the end of the nineteenth Century. The brine solution, or coolant, is circulated through the complete system to produce an ice wall, and refrigeration is maintained properly during the whole excavation and concreting cycles. This is a superior procedure in controlling water, especially in alluvial grounds and quicksands. Unfortunately, this method requires high expenditures due to heavy plant installations. It prohibits the use of explosives, and a long drilling period is necessary before any excavation is done. In such cases, technical difficulties are also multiplied with an increased depth.

Antherton and Gorret⁶ have mentioned that the fine-grained cement slurries could not be forced into fissures and pores of sandstone; thus, it was quite difficult to achieve complete sealing. M. Francois conducted some research work at Sclessia and Liege University to find some inexpensive chemicals for complete solidification of porous sandstone. After much experimentation, he developed the

process of silicification. This process consisted of a simple base exchange reaction between two common chemicals, aluminum sulphate and sodium silicate, which were used in a dilute solution. The products of the reaction were sodium sulphate which remained in the solution and a precipitate of aluminum silicate. This proved to be a satisfactory substance for reducing the porosity.

In 1959, L. Sarosi and A. Fitzek⁷ concluded their investigations on bentonite clay for grouting operations for excluding edge water in mine shafts and tunnels. They also described the properties of bitumen and bitumen emulsions for this purpose, but large-scale application of these materials is expensive and troublesome. Insuring a correct temperature for bitumen could be a serious problem in mining activities. On the contrary, bentonite is inexpensive and readily available. It acts as a filler in cement grout and a lubricant in the rocks where it is placed. Bentonite forms a thixotropic grout and has the added advantage of holding cement particles in suspension. Figure 1 shows the relation of grouting pressure in atmospheres and time in minutes for the injection of equal quantities of cement, bentonite, and water.

In the 1950's, there was considerable progress in the chemical grouting field. The first was chrome lignin, which originated in the U.S. paper industry (Sulphite Pulp Manufacturers' Research League). In correct proportions a

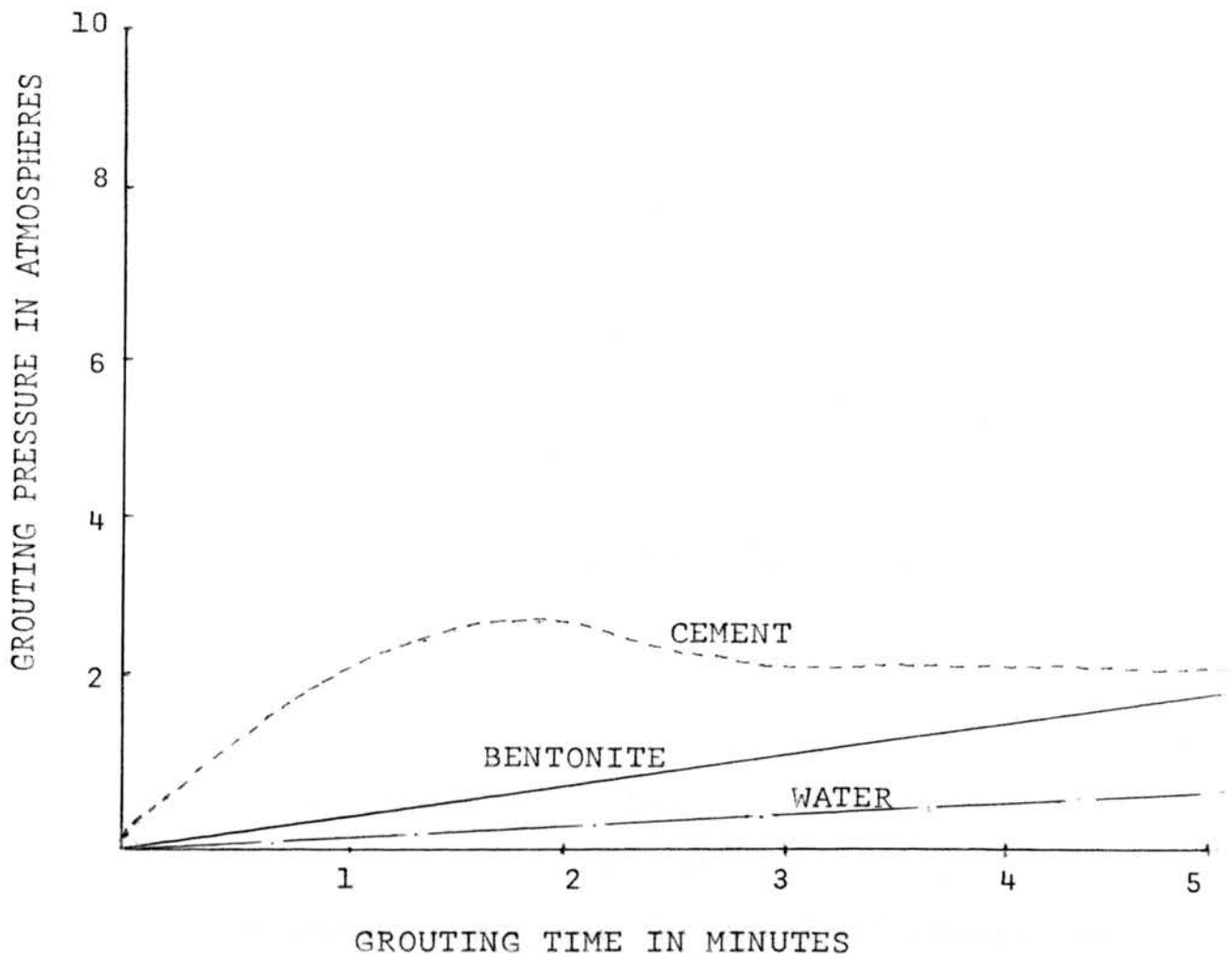


Figure 1. Results of Trial Injections in Skip Shaft at Layko⁷.

mixture of sodium dichromate and lignin sulphonate will form a water insoluble gel which is quite firm. The gel time is dependent on the concentration of solution and addition of reagents. This gel is permanent as long as the rock it impregnates remains moist.

A somewhat similar process makes the use of a substance known as Am-9, which is a mixture of two organic chemicals, acrylomide and methylenebisarcrylamide. In 1959, J. J. Reed and L. Bilheimer ^{8,9,10} mentioned that the freezing method was uneconomical and that all cement, clay, bentonite, and fly-ash grouts proved ineffective in penetrating the interstices in dolomites at the Viburnum No. 28 shaft. Am-9 grout was injected with greater ease due to its low viscosity and closed the smallest openings.

A chrome lignin-type grout was also used by St. Joseph Lead Co., in a limited way, under the trade name of Terra Firma. The gel time was controlled by the water-powder ratio. This material was effective in coarse-grained rocks and in penetrating fractures. However, it was found the product was unable to penetrate openings in fine-grained sandstone. Estimated costs for the chemical grouts used by St. Joseph were as follows:

7 1/2 per-cent Am-9,	\$0.58 per gallon;
10 per-cent Am-9,	\$0.67 per gallon; and
Terra Firma 5:1,	\$0.24 per gallon.

R. H. Oitto, Jr.¹², had used polyester resin in fractured

wall rock in the Homestake mines in South Dakota. Deformed-steel reinforcing bars were used in combination with resin to give added strength. A hardener or catalyst was used to control the setting time. After blasting these cemented rocks, all resin observed had been cured satisfactorily and was solidly attached to the rock. It had also surrounded the reinforcing bars and had bonded them firmly. At another test site, no separation was developed in wall rocks of the bonded ground over a period of three years in the same mine, while considerable failure occurred in unbonded sections in the same area.

The latest successful grouting was done at the Norad underground installation site near Colorado Springs, Colorado, by the U.S. Army Corps of Engineers.¹³ The purpose of the injection of chemical grout was to stabilize and strengthen a zone of fractured rocks. A new, specially formulated, stress-relieved epoxy-resin grout was used for this purpose. Movement gages installed in one area indicated that movement stopped after injection of epoxy resin. Visual observations indicated that other grouted areas had also been benefited.

Several lignosulfonate applications have been made for stabilizing and dust allaying roads¹⁴ in open pit mines. This reduced the haulage and water sprinkling costs and increased the speed of trucks to improve the haulage efficiency considerably. The total cost depended on the

stabilization depth, and secondary treatment was essential after every rainfall.

III. LIGNIN GEL AND CEMENT COMPONENTS

The spent sulphite liquor used for this investigation was supplied by the Sulphite Pulp Manufacturers' Research League. It was supplied in two different varieties, Ah-50v-1 and Bh-38v-2, containing 50 per cent and 38 per cent solids, respectively. Samples had been specially prepared for research and investigation purposes.

The spent sulphite liquor is a by-product of the sulphite pulping process of paper making. It is considered to be waste and is disposed of by dumping in river waters in enormous quantities.

1. Chemistry of Lignin

Lignin is a complex substance distributed throughout wood. It acts as a binding material for wood cellulose. It is associated chiefly with the middle lamella, in primary walls of fibers. Lignin content ranges from 17 per cent to 32 per cent of the weight of moisture-free wood.

Unfortunately, little is known about the physical and chemical properties of this organic substance. There are several reasons for its complexity. Lignin varies with the species of tree, locality of growth, and climatic conditions. These factors have a great influence on its properties. Another difficulty lies in the problem of isolating lignin from wood into the free state.

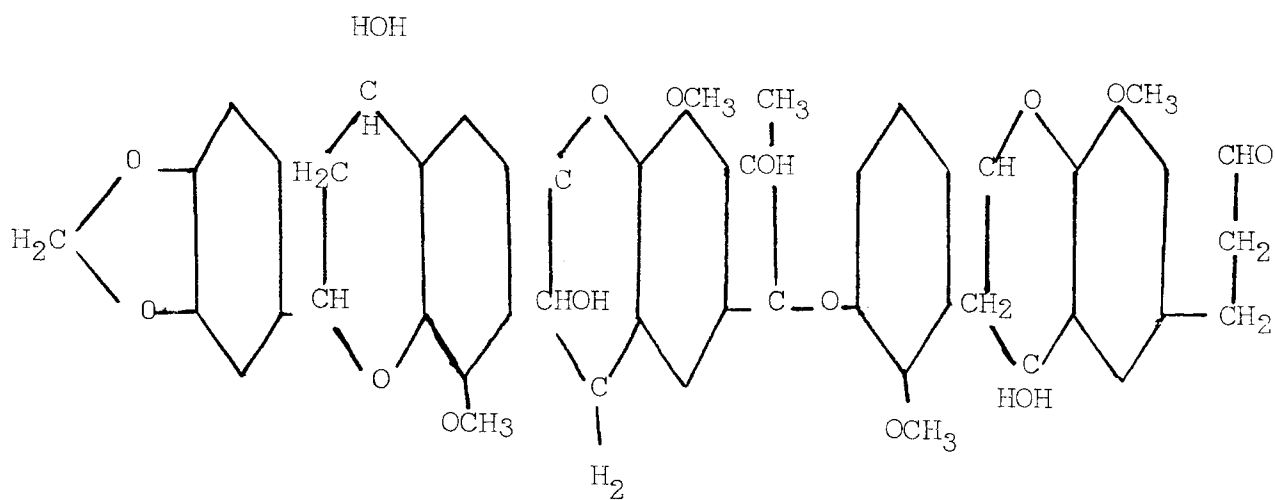


Figure 2. A Proposed Structure of Lignin Molecule¹⁵



Figure 3. Binding Units of the Lignosulphonate Molecule¹⁶.

2. Spent Sulphite Liquors

In the sulphite pulping process, the wood chips are heated under high pressure in an aqueous bi-sulphite solution containing an excess amount of sulphur dioxide. During the digestion process, cellulose fibers in the wood are freed by dissolving the lignin that binds the fibers together. During sulfonation, lignin becomes a soluble product, and about one-half of the wood is dissolved in the cooking process. After cooking, the fluid carries everything that the wood chips contained except the cellulose. This by-product is known as spent sulphite liquor.

Spent sulphite is a clear fluid that looks like freshly brewed coffee. Its pH value is in the range of 2-4. It may contain from 8% to 15% solids in solution, depending upon the nature of the cooking processes and the efficiency of liquor collection. The dissolved solids consist chiefly of lignin sulfonates and sugars.

The lignosulfonate portion of spent sulphite liquor is a mixture of polymers of differing molecular weights. The exact structure of lignosulfonic acid remains unknown, but there is good evidence that phenyl propane units are the building blocks. There are two principal types of phenyl propane units, namely; quaiacvl and syringvl.

Raw liquor contains lignosulfonates with molecular weights ranging from 1,000 to 20,000. A variety of chemical and physical properties can be obtained by processing spent

sulphite liquor to yield fractions of differing molecular weights. The soluble lignin compounds in spent liquor solids are normally present as the salts of strong organic acids. The resin and extractives in spent sulphite liquor include terpens and volatile products.

3. Gel Formation

An insoluble gel can be formed by the reaction of ligno-sulphonate with oxidizing agents such as sodium and potassium dichromates. The gel forming reaction is complex. Oxidation is not the only, or even the predominant, cause of a stable gel. The chemical reaction takes place in the presence of water, and setting time is governed by this phenomenon.

The nature of the final gel and the speed of reaction depend upon the concentration of lignosulphonates in the solution and the concentration and quantity of dichromate used. The limiting ranges of the ratio of dichromate to lignosulphonates for a successful reaction are from 0.1 to 0.5 by weight. The gel time is reduced to a fraction of a minute in the higher ranges of this ratio. Potassium dichromate causes setting 1.6 times as rapid as does sodium dichromate. The final gel strength is also a function of the percentage of dichromate in solution. Higher strength is achieved by a greater percentage of sodium dichromate.

Catalytic agents, when used, increase the reaction velocity and reduce the setting time. In general, these

agents are acidic, which in part accounts for their shortening of reaction time. The addition of aluminum sulphate increases the rate of gelling as much as five times, but the addition of trivalent ferric chloride increases it as much as 225 times, depending upon the quantity used. Thus, a very short gel time can be expected with ferric chloride.

The acidity of a solution has a profound effect on reaction velocity, but it is by no means the only factor. As the pH value decreases, setting time also decreases to a minimum with sodium dichromate at a pH of about 2.5. Alkaline solutions (pH-9) and strongly acid solutions (pH-2) do not set at all. In all cases, the pH rises as the reaction proceeds, and it continues to rise for several days after the gel has set.

Temperature has a significant influence on controlling the setting rate, which roughly doubles with every 20°F. rise in temperature.

Generally speaking, for a given ratio of dichromate and lignosulphonate, the higher the acidity of solution, the faster the reaction. At the higher acidities, a gel can be formed with less dichromate. However, the strength of gel is generally dependent upon the quantity of dichromate used.

IV. EXPERIMENTAL PROCEDURE

The primary purposes of this investigation were the determination of (1) the binding strength of a cement formed from spent sulphite liquor and (2) its effectiveness in sealing off water. Investigations were made to determine the following:

1. Density of lignin solution.
2. pH value of the lignin solution.
3. Viscosity of lignin solution.
4. Strength of cement formed from spent sulphite liquor.
5. Effectiveness of lignin gel for sealing off water and gases.

1. Density of Lignin Solution

Determination of density was essential to establish a suitable water-powder ratio, as recommended for field applications. This ratio would provide a basis for estimating the amounts and costs of required additives. A physical balance, test tubes, and other accessories were used for measuring the densities of liquors under examination. The results are reported in Tables I and II.

2. pH Value of Lignin Solutions

Determination of the pH value was important for estimating the gel time and the required amount of sodium dichromate for gel formation, prior to conducting the

Table I. Results of Density Measurement Tests for 50%
Spent Sulphite Liquor.

Sample No.	Wt. of Test Tube gm	Wt. of Test Tube & S.S.L. gm.	Wt. of S.S.L. gm.	Vol. c.c.	Density gm/c.c.
1.	24.79	30.65	5.86	4.8	1.2
2.	24.79	34.01	9.22	7.5	1.2
3.	24.79	36.35	11.56	9.4	1.2

Table II. Results of Density Measurement Tests for 38%
Spent Sulphite Liquor.

Sample No.	Wt. of Test Tube gm.	Wt. of Test Tube & S.S.L. gm.	Wt. of S.S.L. gm.	Vol. c.c.	Density gm/c.c.
1.	24.79	29.08	4.29	3.9	1.1
2.	24.79	32.71	7.92	7.05	1.1
3.	24.79	34.23	9.44	8.4	1.1

strength and sealing tests. One-inch long pH strip papers were placed on the surface of different liquors. The change in color of the upper side of the pH strip paper was matched with the standard colors provided for comparison. A pH value of four to five was recorded in 38% and 50% concentrations of the liquors received.

3. Viscosity of Lignin Solution

Fluid flow is a function of viscosity. Non-viscous fluids are accepted easily by a porous medium where water will flow, while high pressure pumping units are required to force viscous fluids through very small openings or into a low permeability zone.

The Stormer Viscosimeter shown in Plate I was used to determine the relative and absolute viscosities. Water is a common liquid for determining the relative viscosity, its viscosity at normal temperature being unity. The relative viscosity was obtained by dividing the time required for the rotor to make 100 revolutions in the liquor under examination by the time required for the rotor to make 100 revolutions in distilled water. The experiments were carried out with liquids at room temperature and constant operating weights. The experimental data and results are reported in Tables III and IV.

For determining the absolute viscosity of spent sulphite liquors the instrument was calibrated by using glycerol

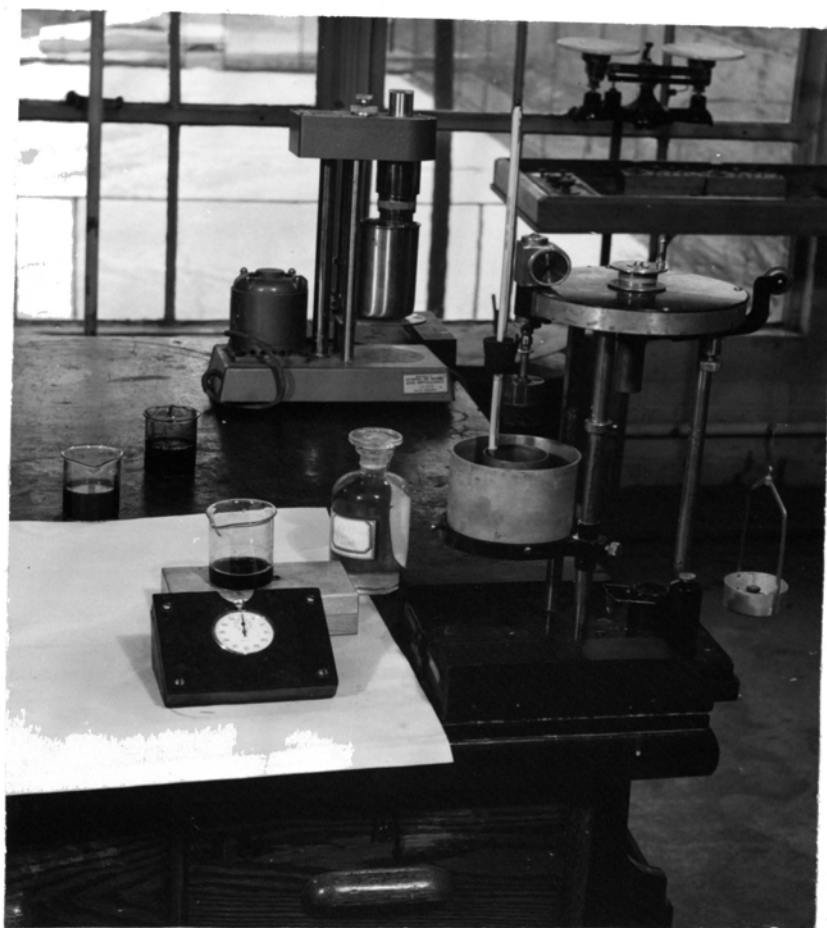


Plate I. Stormer Viscosimeter.

Table III. Results of Relative Viscosity Tests, for 50% Spent Sulphite
Liquor.

Temperature 77°F.

Sample No.	Revolutions	Driving Wt. grams	Time in seconds (lignin)	Time in seconds (water)	Viscosity in Seconds
1.	100	54.95	64.5	7.0	9.2
2.	100	54.95	64.8	7.0	9.2
3.	100	54.95	64.6	7.0	9.2
4.	100	54.95	64.5	7.0	9.2
5.	100	54.95	64.8	7.0	9.2
6.	100	54.95	64.5	7.0	9.2
7.	100	54.95	64.0	7.0	9.1
8.	100	54.95	64.5	7.0	9.2
9.	100	54.95	64.2	7.0	9.17
10.	100	54.95	64.5	7.0	9.2
11.	100	54.95	64.2	7.0	9.17
12.	100	54.95	64.8	7.0	9.2

Table IV. Results of Relative Viscosity Tests for
Spent Sulphite Liquor 38%.

Temperature 77°F.

Sample No.	Revolutions	Driving Wt. grams	Time in seconds (lignin)	Time in seconds (water)	Viscosity in seconds
1.	100	14.95	33.4	20.7	1.61
2.	100	14.95	33.4	26.7	1.61
3.	100	14.95	33.4	20.5	1.62
4.	100	14.95	33.4	20.8	1.61
5.	100	14.95	33.4	20.8	1.60
6.	100	14.95	33.4	20.7	1.61
7.	100	14.95	33.4	20.7	1.61
8.	100	14.95	33.4	20.7	1.61
9.	100	14.95	33.4	20.7	1.61
10.	100	14.95	33.4	20.7	1.61
11.	100	14.95	33.4	20.7	1.61
12.	100	14.95	33.4	20.8	1.61

as the calibrating fluid. In this case, the operating weights were adjusted to obtain 600 revolutions per minute for each set of readings. The operating weights, in grams, are the Stormer Viscosity of the calibrating fluid. The graph shown in Figure 4, or the calibrating curve for the Stormer Viscosimeter, was plotted by using the viscosity in centipoise for different percentages of glycerol in water vs. the operating weights, or Stormer Viscosity, in grams from Table V.

After calibration, the cup was filled with different liquors up to a specified height in the cylinder, as recommended by the manufacturer. The weights were adjusted again to obtain a rotation rate of 600 revolutions per minute. The absolute viscosity was read on the calibration curve in centipoise, corresponding to the operating weights. The test liquors had the following viscosities:

Ah-50-1,	2.5 cp;
Bh-38-2,	185 cp; and
Water,	1 cp.

A critical review of literature showed that liquids having a viscosity up to 7 cp. can penetrate fine grained rocks^{16, 17}. At the Norad operations an epoxy cement having a viscosity range from 12,000 to 14,800 cp. at 48° to 52°F. was injected easily into hair line cracks by using a small pumping unit¹⁷. Considering these facts, the values of relative and absolute viscosities of the liquors tested were

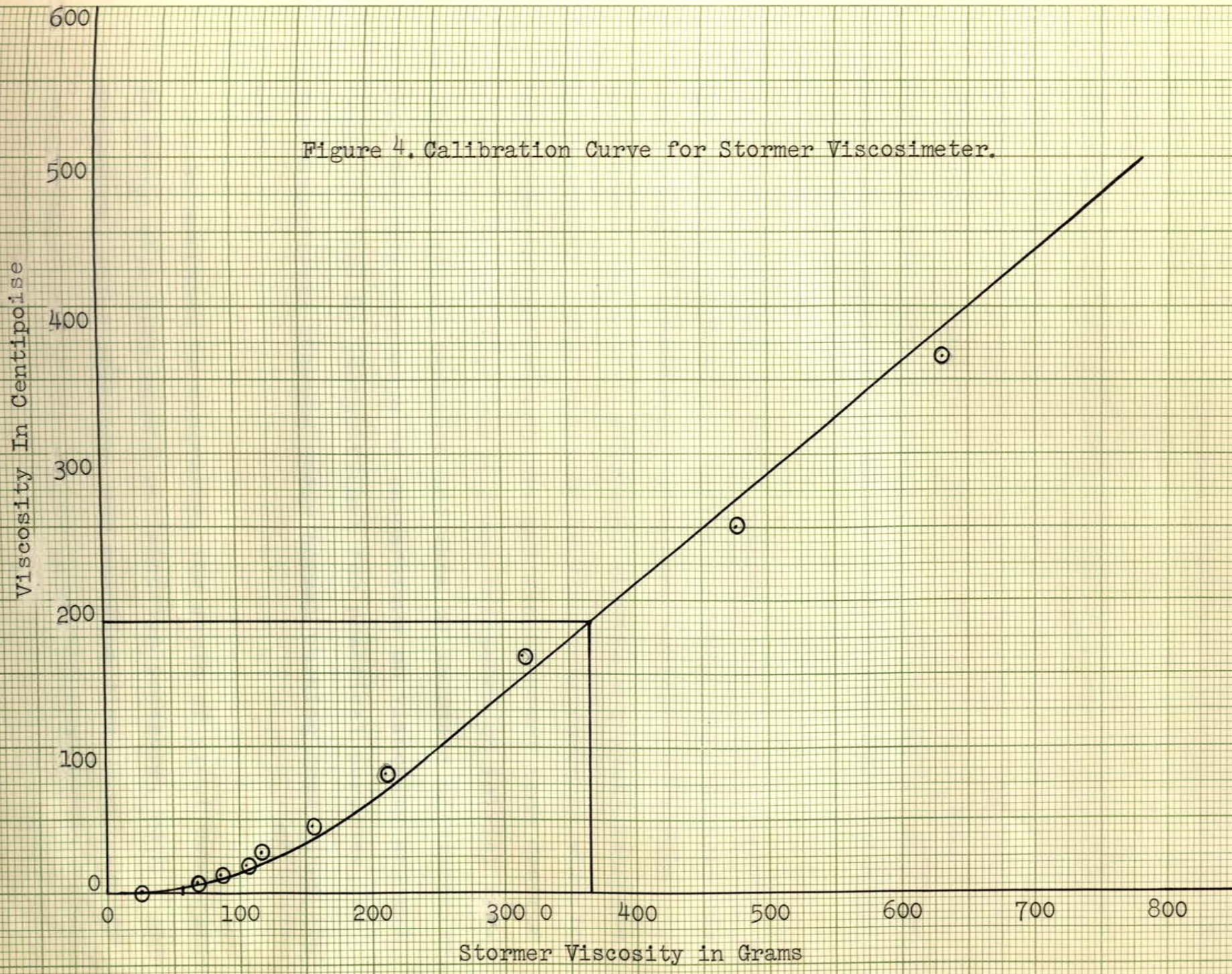
Table V. Data for Stormer Viscosimeter Calibration Curve.

Sample No.	Glycerol %	Driving Wt. at 600 r.p.m. gms	Viscosity in cp.
1.	0	39.0	0.89
2.	2	40.0	0.93
3.	10.0	42.0	1.15
4.	18.0	45.0	1.45
5.	25.0	47.0	1.81
6.	32.0	49.0	2.31
7.	36.0	51.0	2.70
8.	40.0	53.0	3.18
9.	43.0	55.0	3.62
10.	47.0	60.0	4.36
11.	50.0	63.0	5.04
12.	53.0	66.0	5.91
13.	55.0	69.0	6.58
14.	60.0	79.0	8.82
15.	65.0	85.0	12.36
16.	70.0	105.0	17.96
17.	75.0	115.0	27.73
18.	80.0	155.0	45.86
19.	85.0	210.0	81.5

Table V. (Continued)

Sample No.	Glycerol %	Driving Wt. at 600 r.p.m. gms	Viscosity in cp.
20.	90.0	315.0	163.6
21.	95.0	630.0	366.0
22.	100.0	970.0	945.0

Figure 4. Calibration Curve for Stormer Viscosimeter.



entirely satisfactory. Well-refined spent sulphite liquors would penetrate fine-grained rocks. However, the necessary precautions should be taken to prevent premature gelling, which would block the holes in fine-grained rocks before completing the injection into the desired places. This could be avoided by increasing the gel time. The molecular attraction and capillary tension would be much greater in these liquors than in water. Thus, the solution could penetrate all open spaces available even though the viscosity was higher than water.

The viscosity of spent sulphite liquor is a function of the concentration of the solids content. The chemical reaction begins immediately after the addition of sodium dichromate and ferric chloride, showing a gradual increase in viscosity. This increase in viscosity could be used to advantage in controlling the gel displacement from desired places. Temperature has a great influence on initial viscosity, chemical reaction and rate of increase of viscosity. However, the influence of temperature on viscosity and gel formation may be insignificant for practical purposes, as the temperature changes under normal working conditions are small.

In addition to viscosity, the depth of grout penetration in rocks will also depend on injection pressure, rock structure, water pressure, and reaction time.

4. Strength of Cements Formed from Spent Sulphite Liquors

In the early stage of this investigation the spent sulphite liquor was mixed with lime (CaO), epoxy cement, and sodium dichromate in different proportions. The chemical reactions were exothermic, and it was difficult to identify or analyze the final products. The strengths of the products were dependent upon the ratio of spent sulphite liquor weight to the weights of chemicals added. In the case of lime (CaO) or epoxy cement reactions, the high initial viscosity and large amounts of heat released were undesirable. Spent sulphite liquors, with no additives, proved superior and stronger for bonding.

Shear and tensile stress tests were conducted to determine the binding and adhesive properties of the liquors. These properties are important in increasing the strength of fractured rock.

a. Sample Preparation:

AX size (1.1195" diameter) diamond-drill cores were used for the strength tests. Limestone, shale, granite, fine-grained sandstone, micaceous schist, and marble cores were exposed to lignin solutions to determine if a chemical reaction occurred. Considerable difficulties were encountered in treating the micaceous schist and weak shale. They had absorbed an excessive amount of kerosene oil, which was used as a lubricant in sawing the test samples. The shale gave effervescence, probably caused by the acidity of the

solution.

Granite, limestone, and marble cores were selected for the strength tests. A diamond saw was used for slicing the cores in 2-inch and 3-inch lengths. The 3-inch cores were given a cut at an angle of 45° to find the shear stress at that plane. After slicing, all the samples were labelled 1, 2, 3, etc. The cuts and edges were practically smooth. Six samples were prepared for each test.

After coating the cut surface with spent sulphite liquor, the samples were exposed to atmospheric conditions to dry them. Within a few minutes the sulphite liquor started hardening. The surface of the samples treated with the 50 per cent liquor became gummy earlier than the others. After one-half hour, the samples having the same number were joined together. In each sample prepared for tensile strength, 2.5 ml. of spent sulphite liquor was consumed. For shear strength tests, 3 ml. of liquor was used. The samples were dried at room temperature for a period of sixteen days before testing.

A universal testing machine, shown in Plate II, was used for this investigation.

b. Shear Strength Test:

If the equation of equilibrium is applied to the free body diagram of Figure 5,

For $\sum F_t = 0,$

$$P \sin \theta = V = \tau A / \cos \theta,$$

or
$$\tau = P/A (\sin \theta \cos \theta) = (P/2A) \sin 2\theta.$$

Also, for $F_n = 0$, $P \cos \theta = N = \sigma A / \cos \theta$,

$$\sigma = (P/A) \cos^2 \theta = P/2A (1 + \cos 2\theta).$$

Example: By using the values of P and A, for Sample No. 1, we obtain:

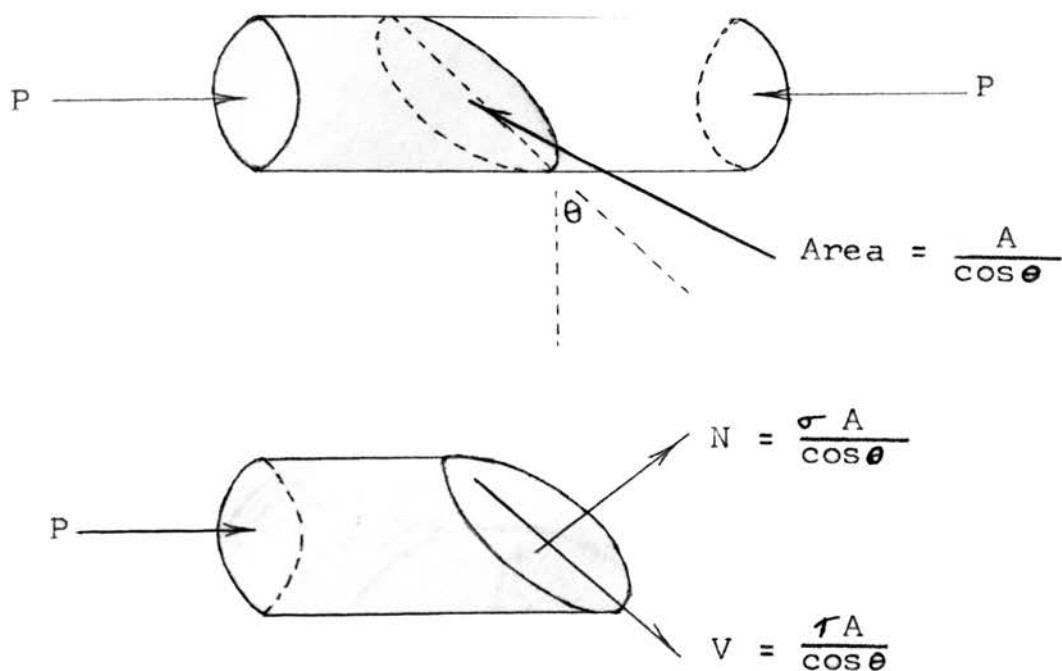
$$\tau = (250/2 \times 1.11)(\sin 2\theta)$$

$$\tau = 250/2.22 (1)$$

$$\tau = 112.5 \text{ psi.}$$

A slow loading procedure was adopted for testing. Rock specimens were tested to failure in order to study the behavior of the spent sulphite liquor cement in the plastic stage. An initial creeping occurred at smaller loads, during which readjustment of the slanted faces took place. The minimum shear stresses recorded for the initial creep in two cases were 65 psi. and 90 psi., much below the average of 116.3 psi. This was probably caused by improper cutting, which resulted in poor bonding between the rock surfaces. The highest shear stress recorded at failure was 1170 psi. In some cases, the rock samples fractured earlier than did the cement bonding. Fractures were along the loading direction, as shown in Plate IV.

These tests indicate that, if used under proper conditions, lignosulphonate could effectively strengthen the fractured ground around mine openings. If pumped under pressure into fractured ground, it would penetrate fine



A = Cross-sectional area of core (sq. in.)

θ = Angle of inclination

N = Normal force (lb)

V = Shearing force (lb)

σ = Normal stress (p.s.i.)

τ = Shearing stress (p.s.i.)

F_n = Total force acting normal to inclined surface

F_t = Total force acting parallel to inclined surface

Figure 5. Free Body Diagram for Shear Stress
Formula Derivation



Plate II. Loading Device Used for Shear Strength Test.

Table VI. Results of Shear Strength Tests with 50%
Spent Sulphite Liquor

Sample No.	Area of Core (sq. in.)	Applied Load (lb)	Load at Failure Point	Shear Stress at Initial Creeping (psi)	Shear Stress at Failure Point (psi)
1.	1.11	100	---	45	---
2.	1.11	400	---	180	---
3.	1.11	300	---	135	---
4.	1.11	300	1800	135	810
5.	1.11	800	---	360	---

Table VII. Results of Shear Stress Tests with 38%
Spent Sulphite Liquor

Sample No.	Area of Core (sq. in.)	Applied Load (lb)	Load at Failure Point	Shear Stress at Initial Creeping (psi)	Shear Stress at Failure Point (psi)
1.	1.11	250	950	112.5	428
2.	1.11	400	2400	180	1070
3.	1.11	375	2600	169	1170
4.	1.11	600	1100	270	495
5.	1.11	200	1100	90	495

crevices. After a period of drying, a strong cement would be formed, which would prevent movement of one block of rock relative to the adjacent blocks, thus preventing caving, spalling, or enlarging of existing openings. For this cement to harden and bond to the rock, the rock must be dry. For use under moist conditions, this substance would not be effective; but the addition of other chemicals could possibly overcome this difficulty.

These tests also indicated the value of this material for use as a cement in the stabilization of haulage roads for open pit mining, where small particles of weathered rock could be bound together to prevent excessive road deterioration and dust.

c. Tensile Strength Test:

Some alterations were essential in the loading unit of the machine used for tensile strength tests. Two one-inch long bolts were bonded on each side of a sample with epoxy cement. Long bolts and clamped wires were used to keep the core sample perpendicular to the horizontal axis, as shown in Plate III.

A slow loading procedure to failure was adopted for these tests. Tensile stress varied from 90 psi to 338 psi with the 50% lignosulphonate solution, and from 31.5 to 189 psi for that with a 38% solids content. Sample No. 4, a marble specimen, was bonded quite effectively, with the marble rock failing earlier than the cementing material.

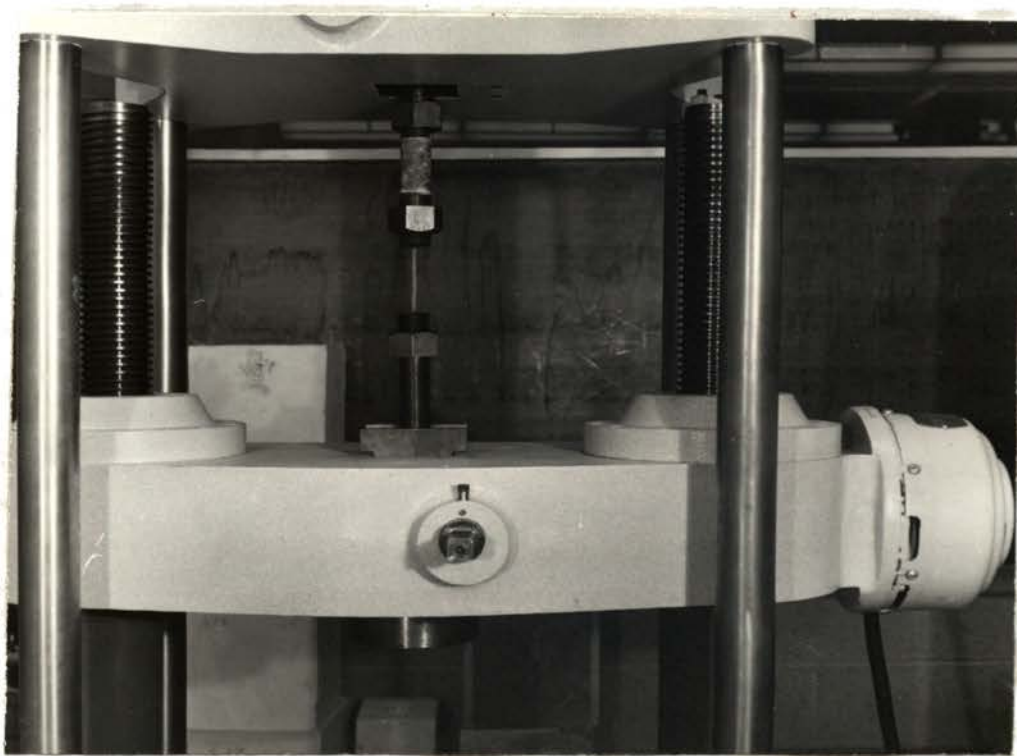


Plate III. Loading Device Used for Tensile Strength Test.

Table VIII. Results of Tensile Stress Tests with 50%
Spent Sulphite Liquor

Sample No.	Applied Load (lb)	Area of Code (sq.in.)	Amount of 50% S.S.L. (ml)	Tensile Stress (psi)
1.	180	1.11	2.5	162
2.	100	1.11	2.5	90
3.	200	1.11	2.5	180
4.	375	1.11	2.5	338

Table IX. Results of Tensile Stress Tests with 38%
Spent Sulphite Liquor

Sample No.	Applied Load (lb)	Area of Code (sq.in.)	Amount of 38% S.S.L. (ml)	Tensile Stress (psi)
1.	42	1.11	2.5	37.8
2.	106	1.11	2.5	95.5
3.	35	1.11	2.5	31.5
4.	190	1.11	2.5	171
5.	210	1.11	2.5	189



Plate IV. Effectiveness of Bonding Material
and Early Rock Failure.

Spent sulphite having 50% solids content dried quickly and proved superior in application over those with a 38% solids content.

The tensile strength of lignosulphonate cement was found to be not high enough to make this property of major importance in the stabilization of mine openings. The tests indicated that the cement's tensile strength was much lower than that of most rock in which it would be used; thus, it would be of little value in the support of bedded rock over mine openings, or in related situations. These tests may, however, have some interest for other purposes. Although the lignosulphonates are soluble in water, effects from humidity and moisture on rocks after the cement has hardened would be negligible.

5. Effectiveness of Lignin Gel for Sealing off Water and Gases

The permeability test was conducted on artificial cores with the objective of finding the sealing effectiveness of lignosulphonate gel. This property could be useful for sealing off gases and water in rock structures. The air permeameter shown in Plate V was found suitable for these investigations. It was essential to run two separate tests, before and after each gel treatment.

a. Sample Preparation:

A large number of artificial cores were molded by using a sand, cement, and water mixed in a 10:15:1 proportion. The sand used for this purpose passed a No. 8 sieve and was

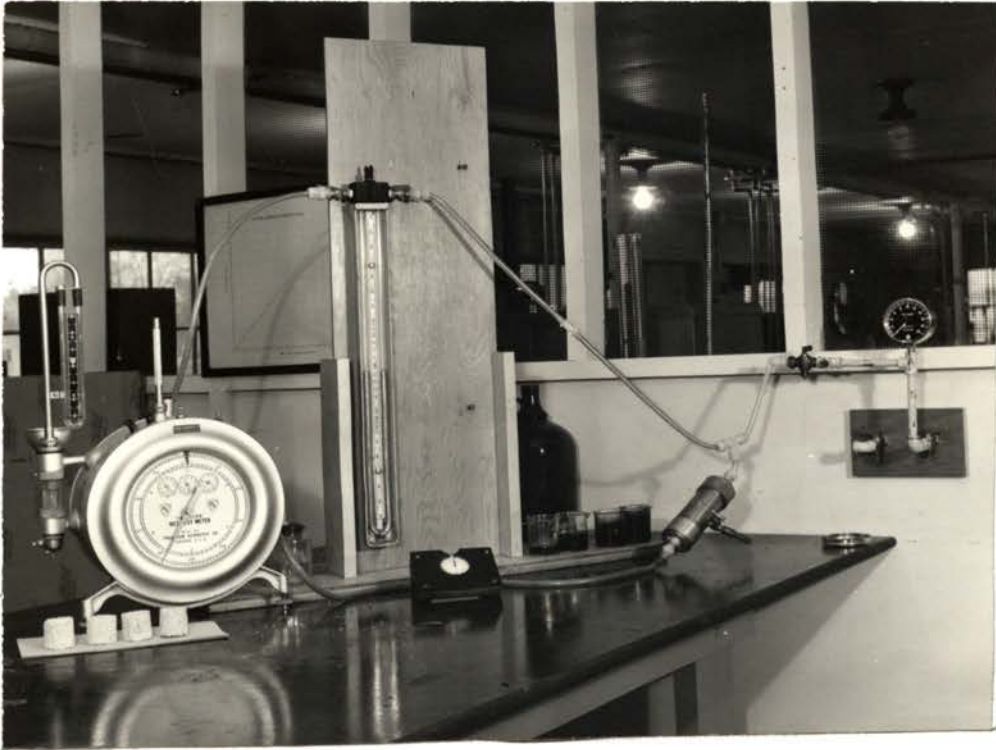


Plate V. Permeameter Setup.

retained on a No. 10 sieve. A rubber gasket (1.5" diameter and 1.5" length) was used for preparing the artificial cores. The mortar used was mixed thoroughly and was carefully packed to achieve uniform porosity in cores. After a 24-hour drying period, two curing applications were made at an interval of eight hours. The samples were prepared six weeks earlier than actual testing to permit complete drying.

A preliminary test was made to check that the inlet side valve of the core and the manometer were air tight. An oven-dried core sample was placed in a rubber gasket, plugged in from both sides by using epoxy glass connectors, then placed in the specimen chamber. The chamber was clamped on both sides with brass clamps, making sure that the sample was placed in a pressure-sealed unit. The core holder is shown in Plate VI.

The dried air was released by opening a main valve to fill the surge chamber. Then, the manometer valve was opened, and the water height was adjusted carefully. The time necessary to pass 0.1 cu. ft. of air was recorded for every pressure increment. The experimental data are shown in Table X.

b. Air Permeability Test:

Darcy's law is the simplest expression for the complex factors involved in the flow of a fluid through porous media. For a laminar and viscous flow of air, Darcy's law can be expressed mathematically as follows:

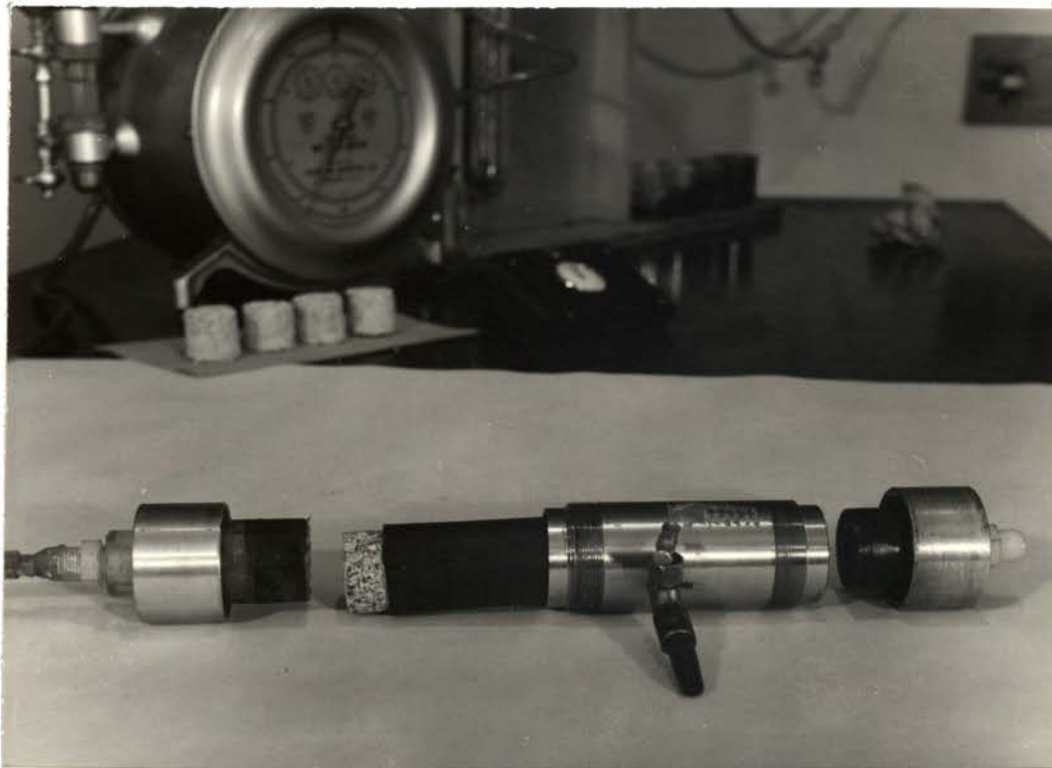


Plate VI. Exposed View of Specimen Chamber Assembly
Used for Air Permeability Test.

$$Q_b/A = K (P_1^2 - P_2^2)/2 U P_2 L,$$

where

Q_b = Volume rate of flow of air measured at P_2 and T , cc per sec,

A = Cross sectional area of sample normal to the direction of flow, cm^2 ,

K = Permeability, Darcy,

P_1 = Input pressure in atmospheres,

P_2 = Output or base pressure in atmospheres,

L = Average length of sample in centimeters,

U = Viscosity of air, Cp.

T = Atmospheric temperature, F.

The out-flow rate, $Q_b P_2/A$, was plotted against the corresponding pressure gradient,

$$P_1^2 - P_2^2/2L.$$

The result forms a straight line of constant slope, K/W , passing through the origin. The permeability calculated from this line is reported in Table X.

For example, the permeability for sample No. 1 would be as follows:

$$Q_b/A = K (P_1^2 - P_2^2)/2 U P_2 L,$$

$$18.2/11.4 = K(.9582^2 - .957^2)/2 \times .018 \times$$

$$.957 \times 3.81,$$

$$K = 182 \times 2 \times 18 \times 957 \times 381 \times 10^{-9}/19152 \times$$

$$12 \times 10^{-8},$$

$$K = 91.3 \text{ Darcy}$$

Table X. Results of Air Permeability Tests

Sample No.	Input Pressure	Output Pressure	Sample		Q_b cc/sec	$Q_b P_b / A$	$P_1^2 - P_2^2 / 2L$	K cp
	P_1 Atmos.	P_2 Atmos.	Length cms.	Area Sq.Cm.				
2	0.9582	0.957	3.81	11.4	18.2	1.53	0.301×10^{-3}	91.3
	0.9594	0.957	3.81	11.4	53.4	4.37	0.601×10^{-3}	131.0
	0.9606	0.957	3.81	11.4	69.0	5.8	0.95×10^{-3}	111.5
	0.9618	0.957	3.81	11.4	85.8	7.2	1.2×10^{-3}	109.0
3	0.9582	0.957	3.81	11.4	19.8	1.65	0.301×10^{-3}	99.0
	0.9594	0.957	3.81	11.4	49.6	4.17	0.601×10^{-3}	125.0
	0.9606	0.957	3.81	11.4	77.5	6.5	0.95×10^{-3}	124.0
	0.9618	0.957	3.81	11.4	85.5	7.17	1.2×10^{-3}	108.0
4	0.0582	0.957	3.81	11.4	36.8	3.08	0.301×10^{-3}	184.0
	0.9594	0.957	3.81	11.4	41.4	3.45	0.601×10^{-3}	104.0
	0.9606	0.957	3.81	11.4	76.5	6.4	0.95×10^{-3}	122.0
	0.9618	0.957	3.81	11.4	90.0	7.55	1.2×10^{-3}	114.0

Table X. (Continued)

Sample No.	Input Pressure P_1 Atmos.	Output Pressure P_2 Atmos.	Sample Length cms.	Sample Area Sq. Cm.	Q_b cc/sec	$Q_b P_b / A$	$P_1^2 - P_2^2 / 2L$	K cp
5	0.9582	0.957	3.81	11.4	38.9	3.26	0.201×10^{-3}	195.0
	0.9594	0.957	3.81	11.4	52.5	4.4	0.601×10^{-3}	132.0
	0.9606	0.957	3.81	11.4	68.2	5.7	0.95×10^{-3}	108.0
	0.9618	0.957	3.81	11.4	74.5	6.24	1.2×10^{-3}	100.0
6	0.9582	0.957	3.81	11.4	17.0	1.43	0.301×10^{-3}	86.0
	0.9594	0.957	3.81	11.4	57.7	4.82	0.601×10^{-3}	145.0
	0.9606	0.957	3.81	11.4	67.4	5.65	0.95×10^{-3}	108.0
	0.9618	0.957	3.81	11.4	86.7	7.27	1.2×10^{-3}	110.0
7	0.9582	0.957	3.81	11.4	15.2	1.26	0.301×10^{-3}	75.5
	0.9594	0.957	3.81	11.4	52.2	4.4	0.601×10^{-3}	132.0
	0.9606	0.957	3.81	11.4	67.4	5.6	0.95×10^{-3}	128.0
	0.9618	0.957	3.81	11.4	86.7	7.27	1.2×10^{-3}	126.0

Table X. (Continued)

Sample No.	Input Pressure	Output Pressure	Sample		Q_b cc/sec	$Q_b P_b / A$	$P_1^2 - P_2^2 / 2L$	K cp
	P_1 Atmos.	P_2 Atmos.	Length cms.	Area Sq. Cm.				
8	0.9582	0.957	3.81	11.4	21.1	1.77	0.301×10^{-3}	106.0
	0.9594	0.957	3.81	11.4	56.5	4.77	0.601×10^{-3}	140.0
	0.9606	0.957	3.81	11.4	81.0	6.7	0.95×10^{-3}	115.0
	0.9618	0.957	3.81	11.4	99.2	8.3	1.2×10^{-3}	106.0
9	0.9582	0.957	3.81	11.4	33.3	2.77	0.301×10^{-3}	166.0
	0.9594	0.957	3.81	11.4	32.0	6.88	0.601×10^{-3}	203.0
	0.9606	0.957	3.81	11.4	103.0	8.8	0.95×10^{-3}	168.0
	0.9618	0.957	3.81	11.4	123.0	10.3	1.2×10^{-3}	156.0
10	0.9582	0.957	3.81	11.4	14.3	1.2	0.301×10^{-3}	72.0
	0.9596	0.957	3.81	11.4	53.4	4.47	0.601×10^{-3}	134.0
	0.9606	0.957	3.81	11.4	71.7	6.01	0.95×10^{-3}	115.0
	0.9618	0.957	3.81	11.4	83.2	7.0	1.2×10^{-3}	105.0

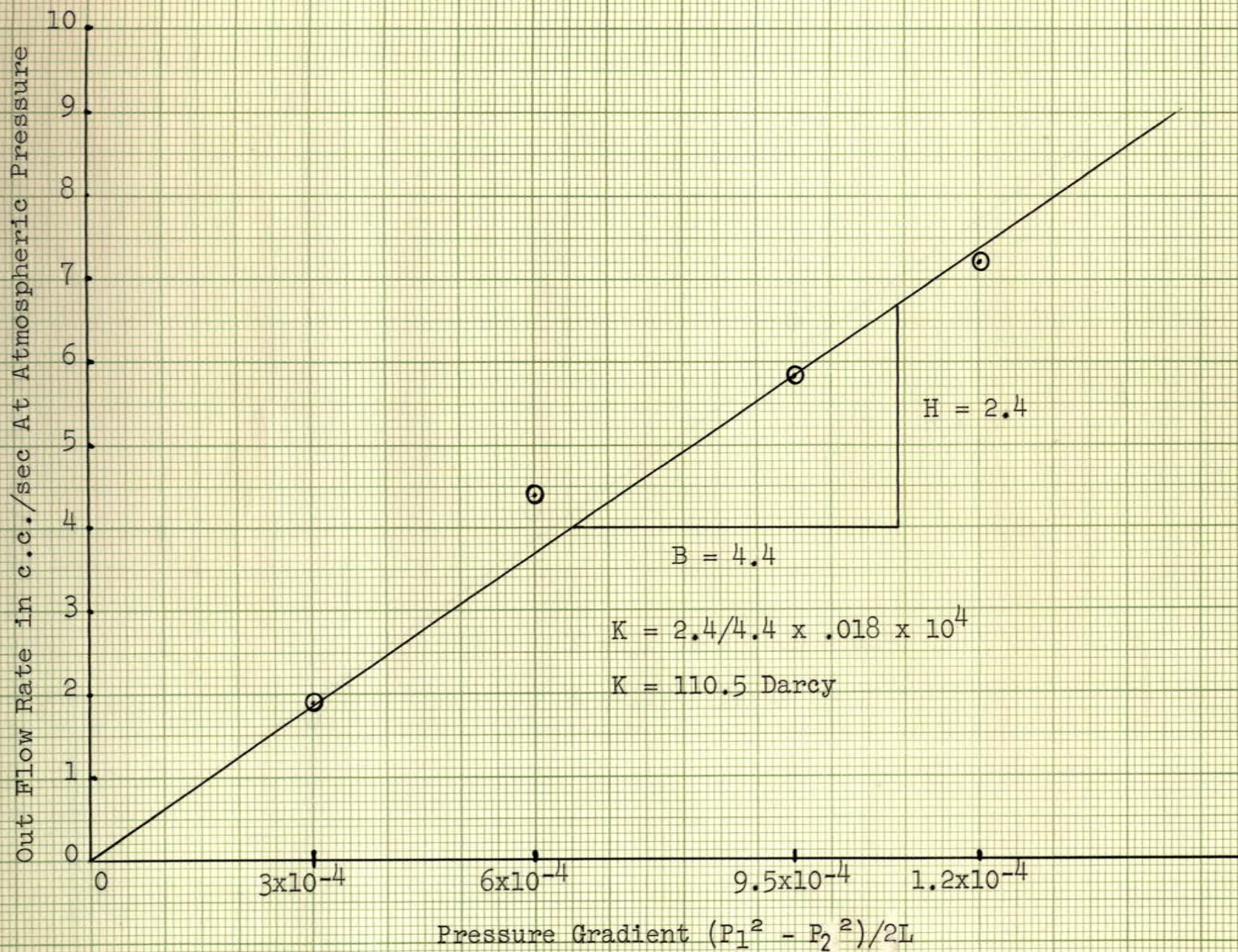


Figure 6. Graphical Solution for the Permeability of Sample No. 2

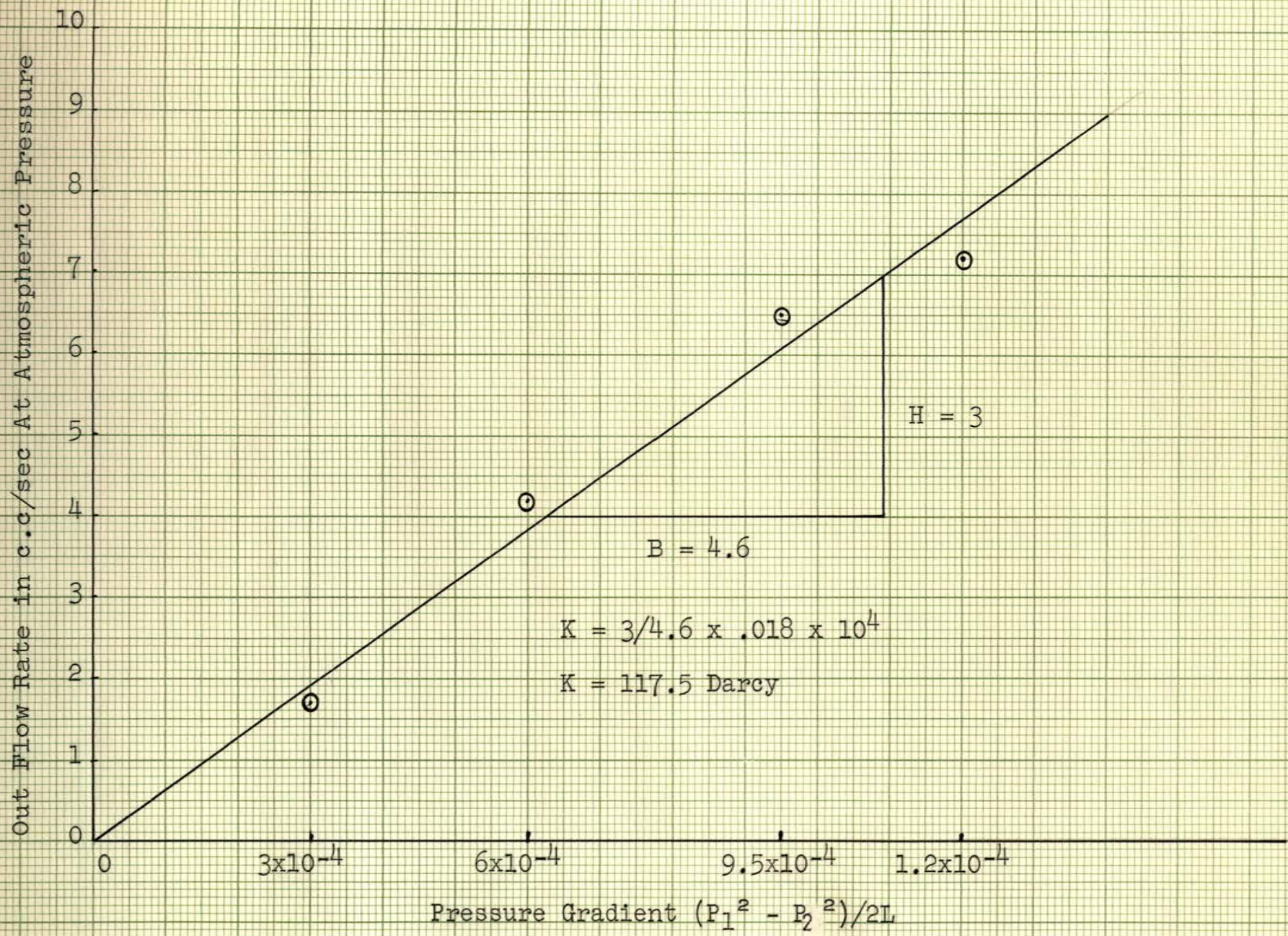


Figure 7. Graphical Solution for the Permeability of Sample No. 3

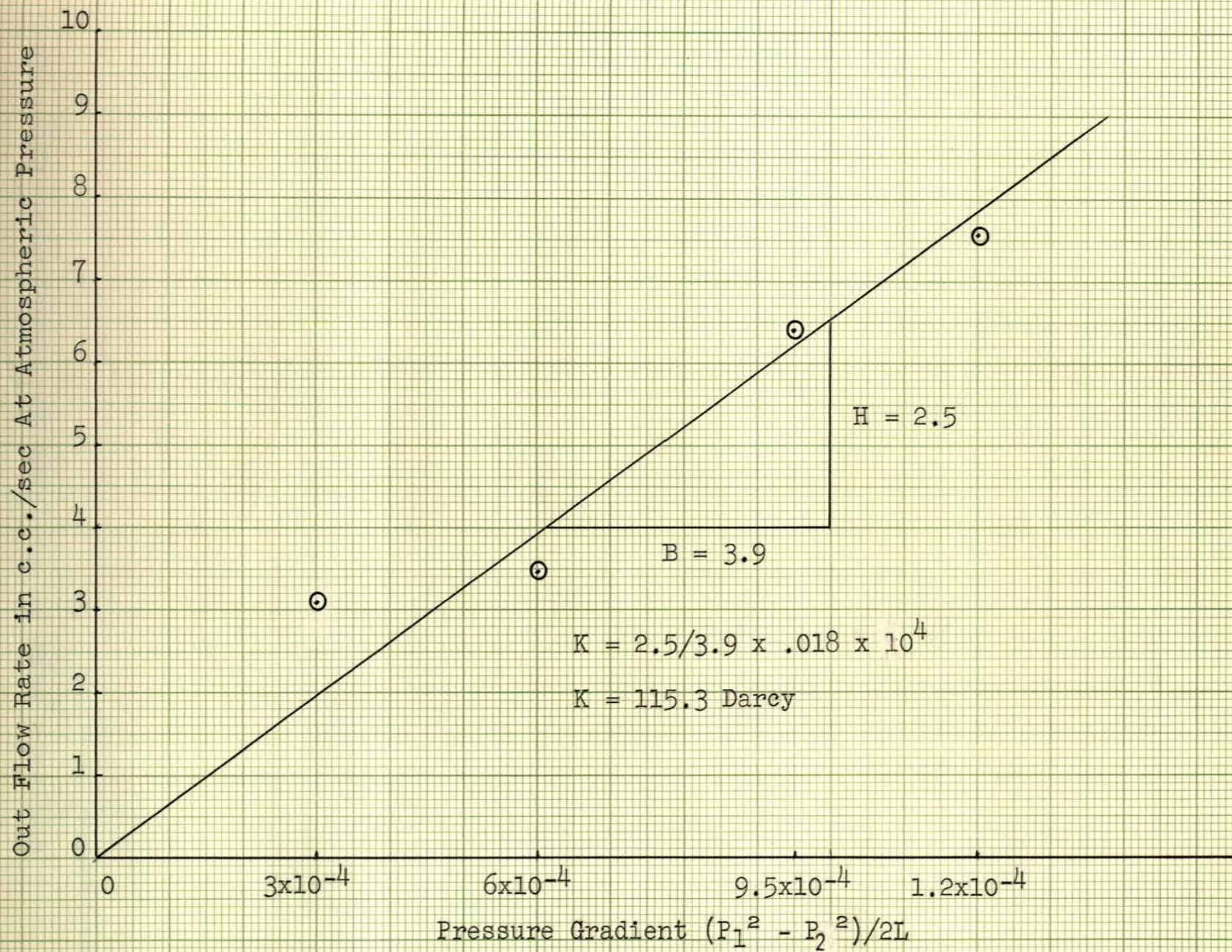


Figure 8. Graphical Solution for the Permeability of Solution No. 4

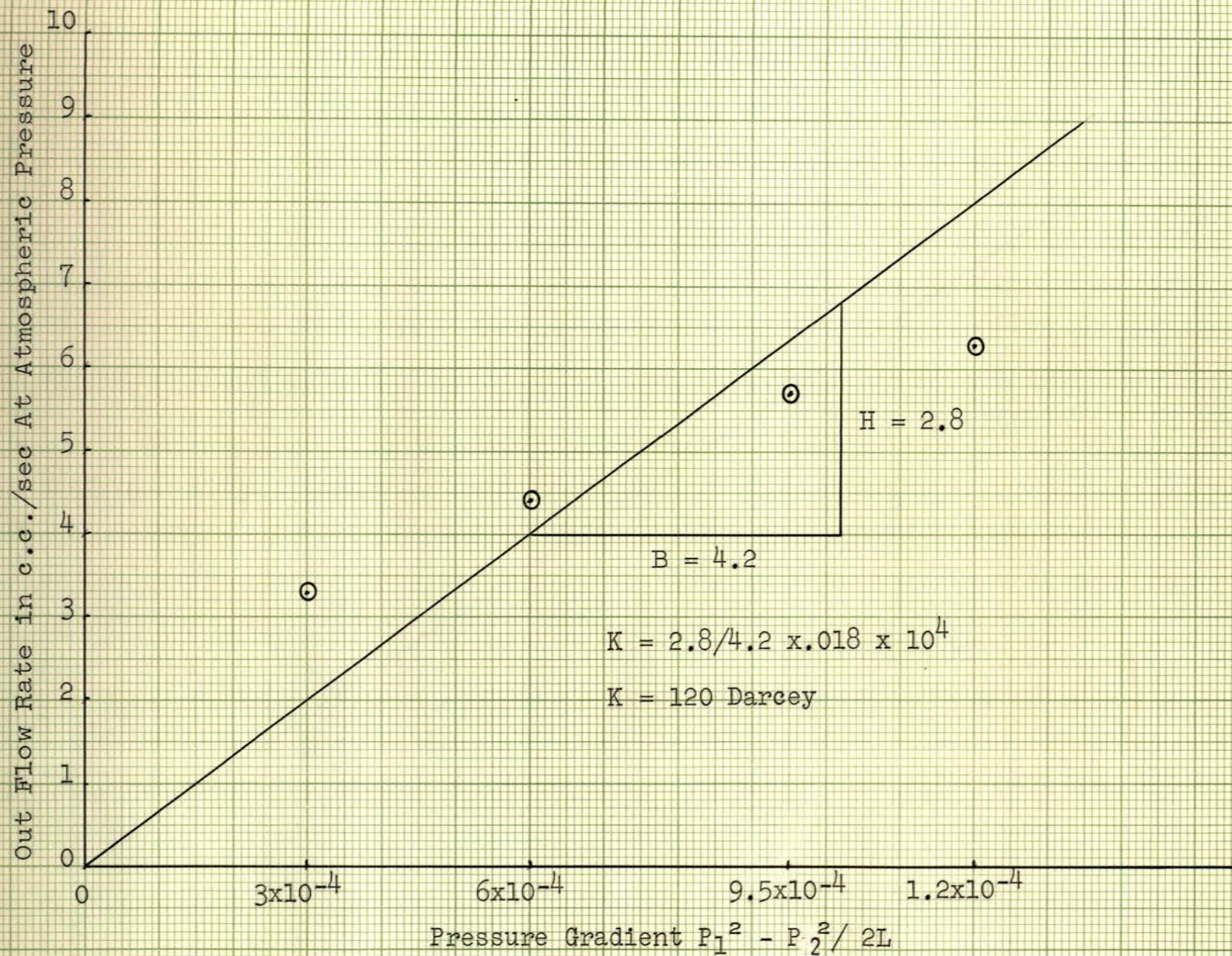


Figure 9. Graphical Solution for the Permeability of Sample No. 5

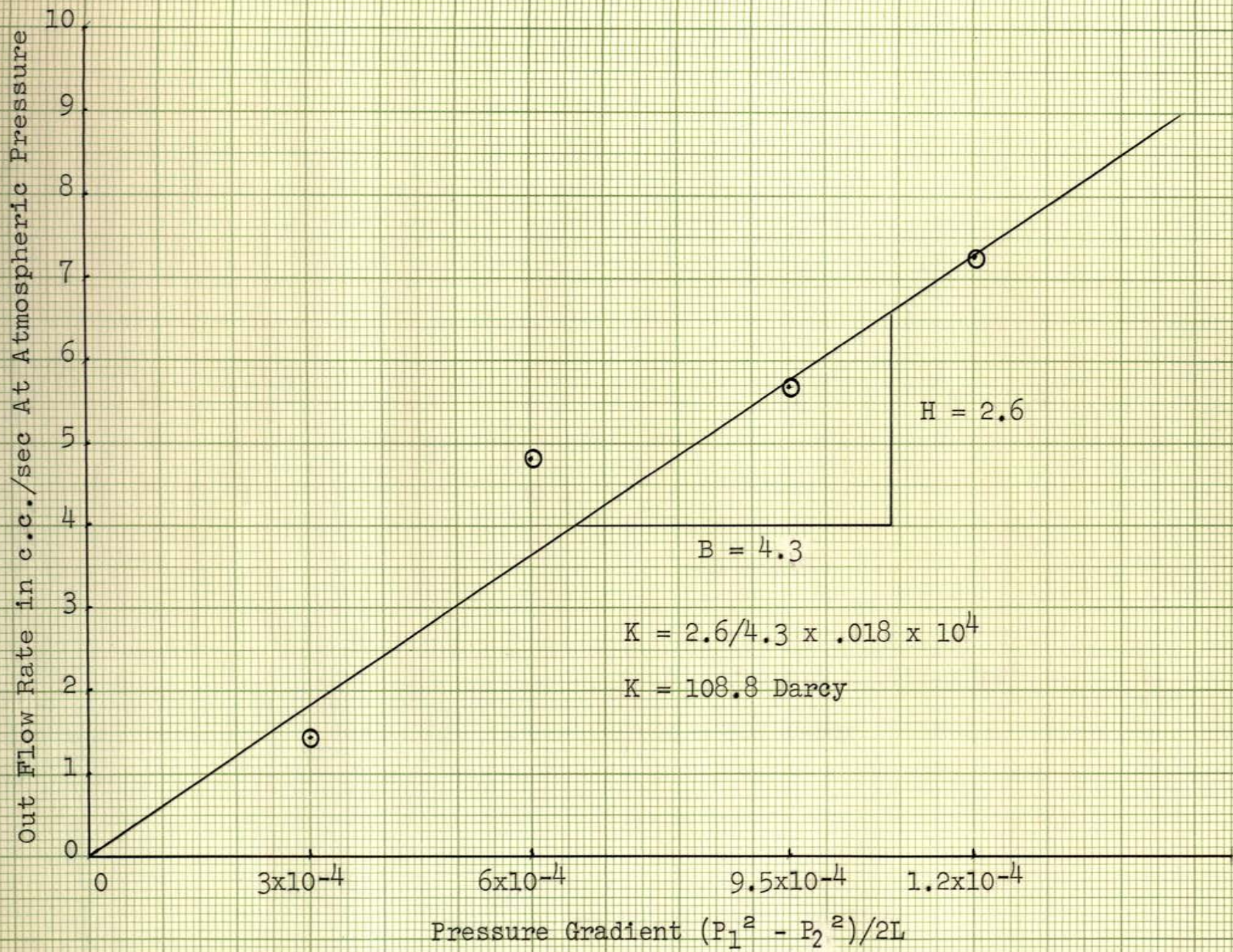


Figure 10. Graphical Solution for the Permeability of Sample No. 6

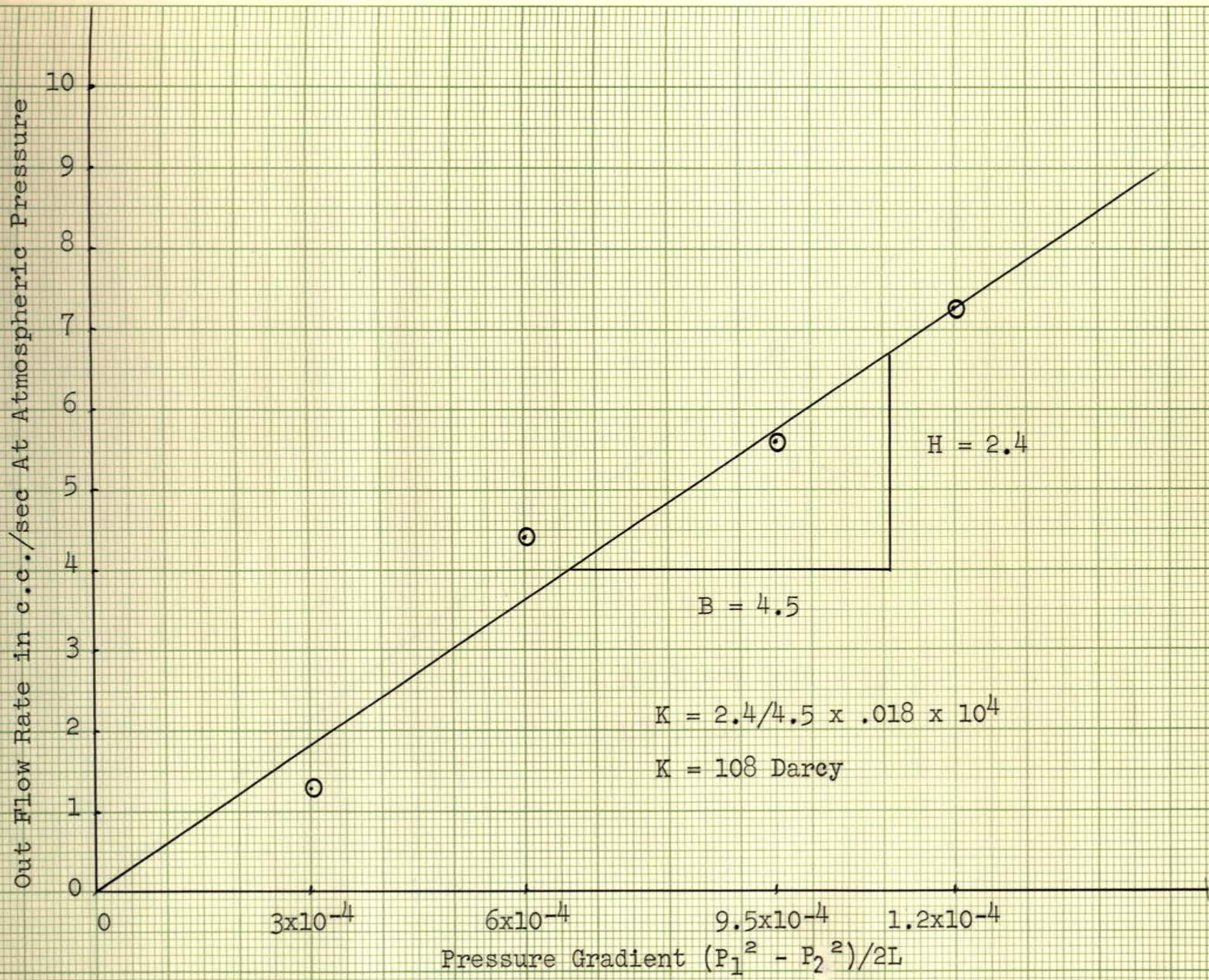


Figure 11. Graphical Solution for the Permeability of Sample No. 7

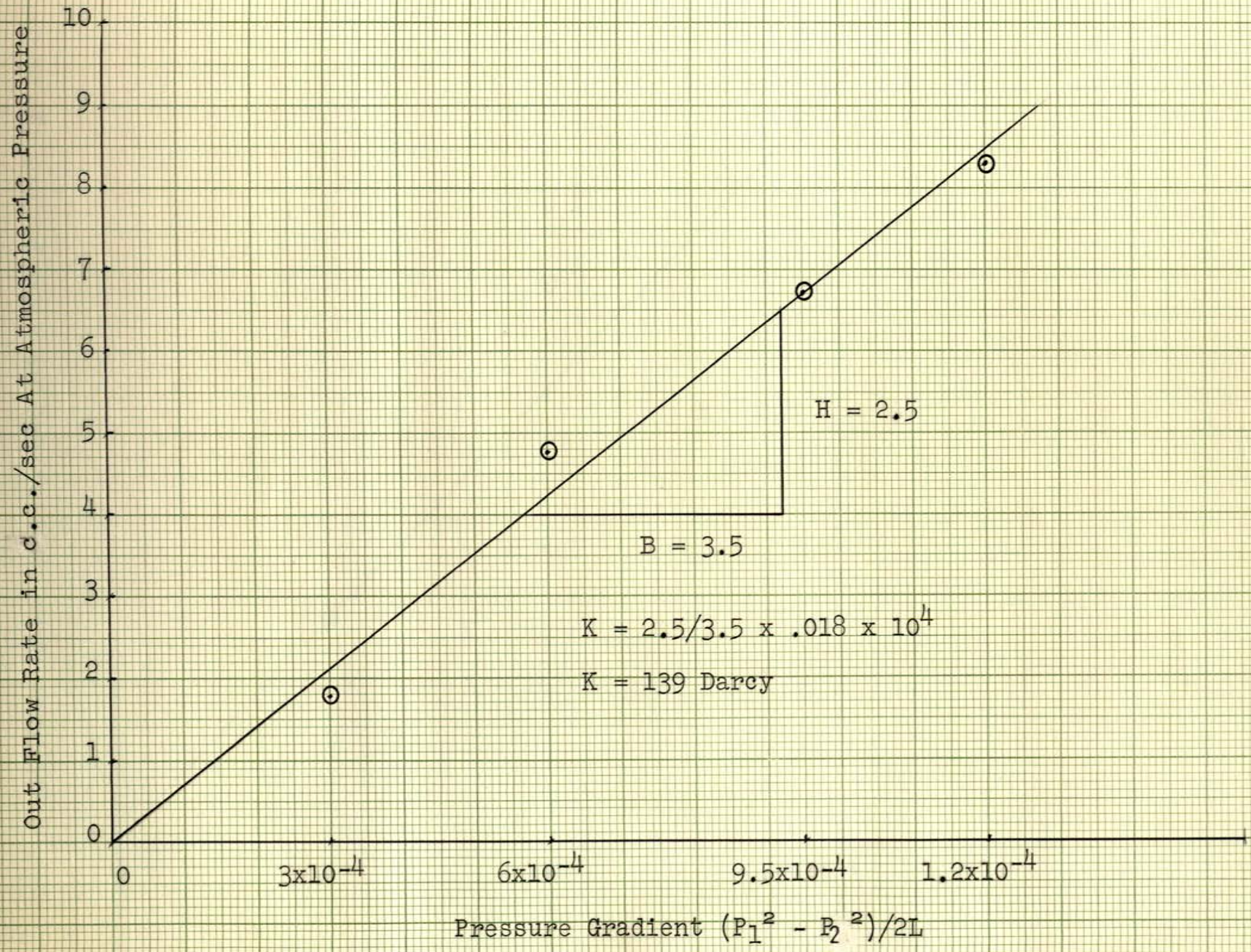


Figure 12. Graphical Solution for the Permeability of Sample No. 8

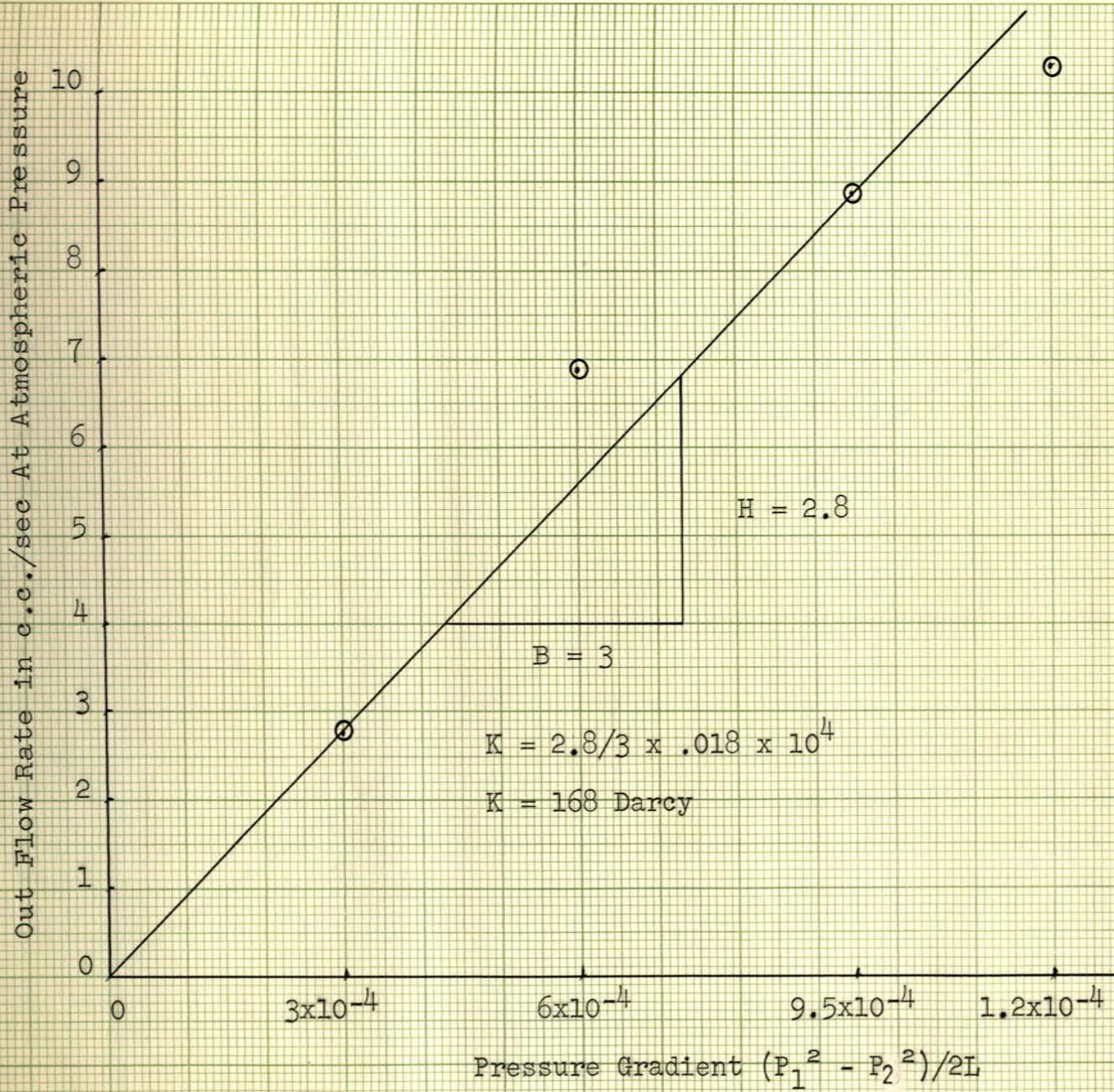


Figure 13. Graphical Solution for the Permeability of Sample No. 9

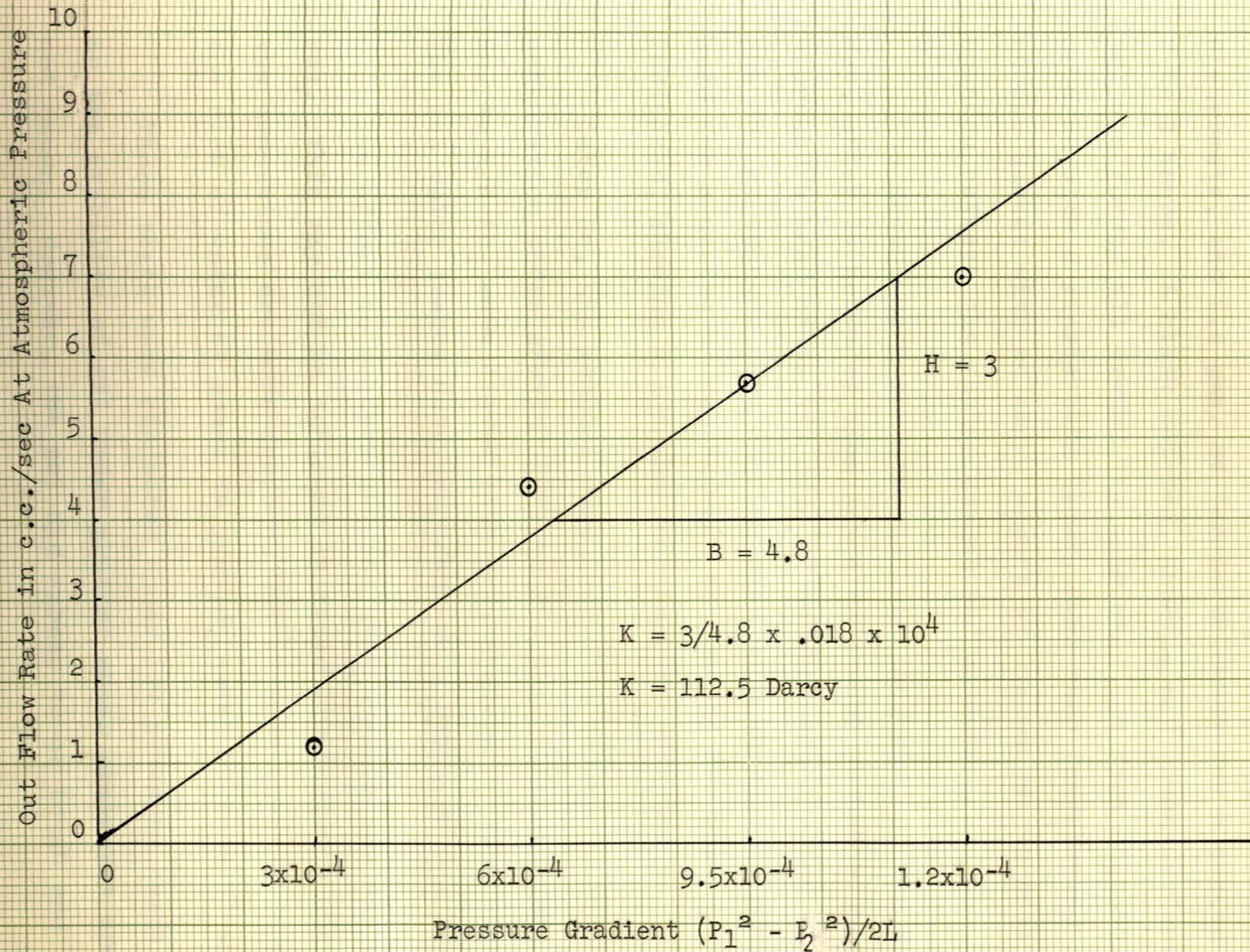


Figure 14. Graphical Solution for the Permeability of Sample No. 10

Samples No. 2 through 6 were placed in beakers containing 40 cc. of spent sulphite liquor, 6.9 gms. of sodium dichromate, and 5.8 gms. of ferric chloride for a period of 2 to 3 hours. A black colored, smooth, and clean-surfaced gel was formed all around the samples. The core samples, covered with a single thin layer, were then transferred to a rubber gasket with an extra amount of liquid gel to fill the opening between the rubber gasket and sample.

The second permeability test was conducted on the next day, after 16 hours of time lapse following actual mixing. A pressure head of 16 1/2 cms. of mercury was applied with the outlet opened. The samples were kept under this pressure head for a period of two hours. No flow of air was indicated, showing a 100 per cent sealing efficiency.

Later on, the samples were broken to determine the gel penetration under gravity influence. All openings from 1/4 in. to 1/2 in. were filled with gel, leaving no open spaces. The gel color traces were prominent to a depth of 1/2 inch.

The effort to achieve 100 per cent sealing was successful. This may not be achieved in field applications where the conditions are more complex, but it will greatly curtail the flow of water or gases to provide safer working places.

These tests were conducted under low pressure heads, so the stability of gel under greater water pressures are not known. However, the gel did appear to be tough and

permanent, so it is believed that it would perform satisfactorily under severe pressure conditions.



Plate VII. Air Dried Gel Showing the Fracture Pattern.

SUMMARY AND CONCLUSIONS

The results of this investigation indicated that ligno-sulphonate solutions may be employed to strengthen fractured rock around mine openings and to reduce the permeability of rock for prevention of water flow.

Sulphite liquor with 38% to 50% solids content was used without other additives as a cementing agent. Its low viscosity allowed it to penetrate fine crevices or pores. Under dry conditions it formed an effective and permanent cement, which possessed sufficient shear strength to stabilize jointed or faulted rock.

A tough, insoluble gel for sealing off water in porous or fractured ground was formed by combining the sulphite liquor with sodium dichromate and ferric chloride. Artificial rock cores impregnated with this gel were tested in a permeameter with good results. Under the low pressure heads used in these tests, 100% sealing was achieved. As a result of this investigation, it is believed that lignosulphonate gel will completely seal off water in the rock around mine openings under most conditions. It was shown that the viscosity and gel time could be controlled; thus, it was possible to adjust these properties of the liquid so that the gel will form at the proper time and place to assure its effectiveness.

VI. RECOMMENDATIONS

The spent sulphite liquors contain lignosulphonate, a valuable organic substance. It could be used in the mining industry for various purposes due to its low cost and adaptability to different situations.

Spent sulphite liquor is an inflammable material. When mixed with equal quantities of sodium dichromate, the mixture ignites at 80°C. This mixture is unstable and lignosulphonate acts as a reducing agent. In making further investigations for using it as an explosive ingredient, its molecular heat should be calculated by a simple calorimetric procedure.

Additional research work could be performed on the use of this substance for stabilization of water bearing and fractured rocks. Its chemical reaction with other inexpensive chemicals being used in different type of cements may result in the development of an excellent, low-cost, cementing material.

One area which should be explored is the use of this material as a stabilizer for fill-in filled stopes. In order that it might be used for this purpose, some admixture would be required which would hasten dehydration without adding greatly to the cost.

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VITA

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