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NORTHERN COAL MINES LIMITED SUMMARY REPORT

by

L. S. Trenholme

February, 1973

Vancouver, B.C.

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NORTHERN COAL MINES LIMITED SUMMARY REPORT

February, 1973

General Statement:

The purpose of this report is to summarize the results of work done on the company's property near Prince George, B.C., and to provide some indication of the commercial possibilities of the coal deposits thereon.

Property:

The company holds 19 contiguous coal licences, of approximately 1 square mile each, situated 35 miles east of Prince George, B.C., and accessible by gravel road, south of Highway No. 16.

Work to Date:

At least 42 holes were drilled prior to 1971, mainly near the southwest margin of the coal basin. These indicated continuity of the coal measures for a strike length of 2 miles. During this time, several hundred feet of underground entries indicated thicknesses of 7 to 11 feet of fairly clean coal dipping about 45° to the northeast.

In 1971, Bethlehem Copper Corporation Limited drilled 5 diamond drill holes (1-7/8" core) to a maximum depth of 2,200 feet, for a total of 7,474 feet. This drilling confirmed the continuation of the coal measures northeasterly dip into the basin and established the approximate northeasterly limit of the basin.

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Coal Reserves:

In 1967, Black (Ref. 3) estimated indicated and probable reserves at 20,185,000 tons of coal in place.

Work to date, plus geological interpretation, allows a rough estimate of the potential coal reserves underlying the company's licences.

a) In the known and probable extent of the coal measures partly tested by drilling: 5-1/2 million tons per foot of seam thickness. Assuming an average thickness of 9 feet in each of two seams, the calculation is 99 million long tons of coal in place.

b) In adjoining areas possibly underlain by the coal measures, but untested by drilling, the potential is 5 million tons per foot of seam thickness.

It is emphasized that the foregoing estimates (a) and (b) are possibilities only; not calculations based on precise data. No meaningful calculation of total recoverable reserves can be made at this time.

Coal Quality:

The rank is 'High Volatile B Bituminous' (Ref. 6). This coal is indicated to be good quality thermal coal (subject to some sulphur removal) and might be suitable for blending with other coals to produce a good quality metallurgical coke (Ref. 6).

COAL ANALYSIS - AIR DRY BASIS

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	Moisture	Ash	<u>v.u.</u>	F.C.	B.T.U.	<u>s.</u>	F.S.I.	Ref. No.
	%	%	%	%		%		
a) Near South Edge of Basin								
Underground								
Middle Seam 11 feet								
Top of Slope	5.50	2.80	39.7	52.0		1.20	1.0	10
400' Down Slope	4.92	2.77	36.5	55.8	12,550	0.85	2.5	10
Lower Seam								
At rock slope	14.10	2.60	28.6	54.7	12,470	1.50	2.5	10
13 Drill Cores: (Average)								5
Raw Coal		7.96				1.29		5
Float (1.4) 86%		4.66				1.22		5
Sink (1.4) 14%		22.86				1.79		5
b) Basin Interior								
Bethlehem Drill Cores								9
Average 19 Samples		94 69	0.6 50	40.0	11 000	1 00	0	0
in 4 Drill Holes		24.63	36.73	42.O	11,000	1.39	2	9

ESTIMATE OF DRILL-INDICATED COAL RESERVES

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Per J. M. Black

September, 1967

BLOCK	THICKNESS	SHORT TONS
1 2 3 4 .5 6	21' 29' 6.5' 8' 14' 9'	2,626,000 2,213,000 444,000 291,000 246,000 129,000
7	19'	<u>6,211,000</u> 12,160,000

Additional Estimate by L. S. Trenholme

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December, 1972, based on revised interpretation of fault location.

	1,512,000
TOTAL DRILL INDICATED	13,672,000
Less: 10% Pinch-outs	1,367,000
NET INDICATED	12,305,000

This estimate does not include "Probable Reserves" in adjoining areas of widely-spaced drilling.

Comment

The higher ash content reported by Bethlehem is difficult to explain unless more shale was included than was reported in the samples or, it may be due to an increase of shaly material toward the centre of the basin.

It is also noted that no swelling tests were made by Bethlehem to indicate coking characteristics and that no sink-float tests were made to indicate recovery and ash content of clean coal.

Washability

A bulk sample tested in Japan for Nissho-Iwai Company indicated a recovery of 85.6% clean coal with ash content of 7.3% which is considered to be quite satisfactory (Ref. 8).

Resin Content

Dr. J. M. Black (Ref. 3) reports that bulk sampling indicated a soluble resin content of about 4% and a similar amount of visible insoluble resin. The Battelle Memorial Institute (Ref. 7) describes detailed tests of a highly technical nature which suggest that these resins could be valuable products. A recent letter (November 29, 1972) from Battelle confirms their interest in continuing research.

A summary of their findings is as follows:

- 1. A distinction is made between two types of resin occurring in the coal.
 - a) Dark soluble "Refined Resin."
 - b) Visible insoluble "Amber Resin."

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- 2. All testing was done on the 'Refined Resin' which was found comparable to Congo Resin for certain uses and which was priced at \$0.21 to \$0.28 per 1b. in 1967 (\$0.24 to \$0.33 in 1972).
- 3. It was indicated that the "Amber Resin" might be the more valuable of the two, but how much more valuable is not estimated.
- 4. Further research is required to determine the best extraction methods for each of these types as well as their suitability for various industrial applications.

If it can be assumed that the total resin content of the coal is 8% (160 lb. per ton) and that an average price of \$0.30 per lb. would be obtained, then the gross value of the washed coal may be calculated as follows:

160 lb. Resins @ \$0.30	\$48.00 per tor
Coal Residue (assumed)	12.00 per tor
Total Gross Value	\$60.00 per tor

Mining Conditions, General

A considerable amount of surface drilling would be required to establish adequate continuity of individual coal seams to justify a large tonnage operation.

The known dips of $20^{\circ} - 45^{\circ}$ would make mechanized mining by current methods costly. It is reported that Kaiser Resources Limited are having considerable success with hydraulic mining of steeply pitching seams, so this method should be kept in mind for possible use in the Northern Coal deposit.

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Small Scale Mining Approach

Assuming favourable results from technological and market research, consideration should be given to mining on a small scale, say 300 to 500 tons per day.

This would involve relatively low initial capital costs for the mining plant because it would employ conventional underground mining equipment rather than the very expensive coal mining units and their supporting equipment.

Also, coal sufficient for several years' operations could be obtained from the immediate vicinity of the existing workings without extensive new development.

Future Possibilities

With the expected future shortage of cheap energy sources, consideration could be given to a larger operation, probably using hydraulic mining. Additional surface drilling would be required to establish reserves sufficient to justify the installation of a thermal power plant or coal gasification plant at the site.

Respectfully submitted,

1. 3 Vicentolm

L. S. Trenholme

February 26, 1973 Vancouver, B. C.

Maps and Attachments

- 1. General Location Map, 1'' = 50 miles.
- 2. Map of Drill Holes and Drill-Indicated Reserves, 1'' = 400'.
- 3. Battelle Memorial Institute, Reports and Correspondence.
- 4. Geological Survey of Canada, Technical Report 93-H-13W-1, 1972.

Available References

- 1. J. M. Black, Report January 31, 1967.
- 2. Chapman, Wood and Griswold Limited, Progress Report June 30, 1967.
- 3. J. M. Black, Report September 8, 1967.
- 4. J. M. Black, Report to Accompany Prospectus, August, 1965.
- 5. J. M. Black, Report on Cleaning Tests, June 4, 1969.
- 6. J. R. Donaldson, Geological Survey of Canada, Petrographical Report, 1972.
- 7. Battelle Memorial Institute
 - a) Reports on Resin Content, 1966 and 1967.
 - b) Letter of December 6, 1972.
- 8. Tagawa Machinery Manufacturing Company Limited for Nissho-Iwai Company Limited; Report on Washing Tests, April, 1969.
- 9. Richard E. Kucera, Geological Report for Bethlehem Copper Corporation Limited, November 4, 1971.
- 10. Robert B. Bonar, Report dated February 2, 1970.
- 11. Underhill and Underhill, Surveys of Drill Holes and Mine Workings.
- 12. Coal Testing, Laboratory Reports

Other Available Documentation

- 1. List of Principal Shareholders
- 2. List of Accounts Payable
- 3. Audited Financial Statement, September 30, 1971
- 4. Minutes of Annual Meeting, March 30, 1972
- 5. Unaudited Financial Statement, September 30, 1972

L. S. Trenholme - Statement of Qualifications

This will certify:

- That I am a graduate of the University of Saskatchewan, B.Sc., 1. (Geology) 1936, and of McGill University, M.Sc., (Geology) 1939,
- 2. That since graduation I have continuously (except 1942-1945) practised my profession in mining and exploration geology,
- That I have been in the employ of Brameda Resources Limited since 3. May, 1968, as exploration manager and staff geologist,
- That my experience in coal exploration has been obtained through 4. 3 years close association with the Sukunka Coal Project of Brameda Resources Limited,
- 5. That I have made one visit to the property of Northern Coal Mines Limited and have studied all significant reports relating to it and have been one of its directors (unpaid) since 1971 and,
- 6. That I do not own any shares or other interest in Northern Coal Mines Limited or its properties, other than an indirect interest through ownership of 1750 escrowed shares of Brameda Resources Limited, which company owns 840,000 shares of Northern Coal Mines Limited, nor do I expect to receive any further interest.

(Signed) <u>L. 5. Trenholme</u>

February 26, 1973 6746 Marguerite Street Vancouver 14, B. C.

PEC 4 - 1972



Columbus Laboratories 5.65 King Avenue Columbus – Olico 47(3) Telephone (614) 299 3151 Teles 24~5454

November 29, 1972

Mr. L. S. Trenholme, Director
Northern Coal Mines, Ltd.
7th Floor, Board of Trade Tower
1177 W. Hastings Street
Vancouver 1, B. C., CANADA

Dear Mr. Trenholme:

As you know, your letter of November 3, 1972, addressed to Battelle Memorial Institute was given to Mr. Neher because of his past research connections with your company. Mr. Neher's letter of November 13, 1972, gave brief reply and indicated that answers to your questions would be forthcoming. Since then, we have discussed your past research program, and how we might proceed in the future.

Research on your resin in coatings, rubbers, and plastics applications would now fall in the Division of Polymer and Paper Technology, of which I am a member. Therefore, I have taken the liberty of replying to your letter.

The minimum weight of material desired for a new research program is estimated as that quantity necessary to produce about 10 pounds of resin. If you know the percent resin in the coal, you can readily calculate the quantity of coal needed. If the research is at all extensive, 25 pounds of resin would be a more reasonable amount to work with.

At this time we cannot give an estimate of cost of a new research program. It will be necessary to plan the program carefully, and prepare a research proposal before we can give a cost estimate. In this respect, it would be very helpful to us to have you visit our Battelle-Columbus Laboratories to discuss possible research approaches.

There is an outstanding indebtedness of \$3,943.75 for prior research carried out for Northern Coal Mines, Limited. This research terminated in 1967. In our last attempt to submit an invoice for this indebtedness it was returned to us because address of company was unknown.

Enclosed is a Xerox copy of a page from a recent issue of <u>American Paint</u> <u>Journal</u>, which gives prices for some of the natural resins. However, synthetic resins might provide more competition for your resins than would be experienced from the natural resins. The paint industry went through a period many years ago when synthetics largely replaced the natural resins. More recently, alkyds, and then the water-based resins replaced most of the oleoresinous varnish in paints. Enclosed is a selected tabulation of imports of some natural resins, which you should find very interesting. Mr. L. S. Trenholme,

November 29, 1972

Please let us know if you have any additional questions.

Very truly yours,

Louis J. Nowacki VUUX Polymer and Paper Technology Division

LJN:gm

Enclosures

Markets

Barrels one cent more.

Orange, 4-lb, eutgal.		—		
5-lb. cutgal.	••		••	
White, 4 lb. cutgal.	••			
5-1b, cutib.		—	••	
iF.o.b. New York.				

F.o.b. New York and Chicago for 50-gallon F.o.b. New York and Chicago for 50-gallon barrels. 4 bbls. or more 5 cents less.

Varnish Gums and Resins

Natural Resins

Asphaltum-			
Gilsonite, selects 100-lb, bags.			
F.o.b. Craig, Coloton \$61	. 60		••
Ex-Warehouse, bags N.Y.			
or N.Jton #14.	5-16	5	••
*Congo			بد ا
No. 1lb.	••]
No. 2lb.	.29	-	.33 [
No. 3Ib.	.24		.27
-7 •Damar			
Singapore No. I BagsIb.	.29		.33
Singapore No. 2lb.	.27		.30
Singapore DustIb.	.17	~~~	.21
*East India			
Pale Nubs	.22		.25
Pale Chipsib.	.16		.19
Black Bold Scraped	.17		.20
Black Nubs and Chips lb.	.13	—	.16
Black Unscrapedlb.	.14		.17
Batu Bold	.16	—	.19
Batu Nubs and Chipslb.	.11		.14
Pontlansk-			
Nubs	.35		.38 🛬
Chipslb.	.22		,25
*Manila-			
Lobs C	.30		.35
DBBlb.	.23		.26
LAS	.21	-	.24
MA	.19		.22
Philippine Pale Bold lb.	.34		.37
Philippine Pale ChipsIb	.24		.27
Philippine Sorts	.12	_	.22
Elemiib.	••		
Yaccalb.	.12		.15
*Ex-warehouse or dock New Yor!	ĸ.		

Synthetic Resins

Chlorinated Rubber f.o.b. Parlin and Bayonne, N.J. 20 cps. t.J.lb. .614-

ZD Eps., a.J	-0.74	••
125 cps., t.Ilb.	.71%-	
Epoxy, Liquid, 100%		
solids basis, tanks, deliv 1b.	.41	
Ester Oums-Ordinary-delivered.		
C.I., drumslb.	.221/2	
Lel drums	26	

f.o.b. customer's sidinglb227	
L.c.l., drumslb24	
Styrene Butadiene Copolymer Resins.	
tob Akron O. or Gary, Ind., min, frt, pr	ođ.

f.o.b. Akton, O., or Gary, Ind., min. frt. ppd. --100-240 secs, 33%% in xylol. T.l.lb. .49 -- ..

Organic Acids

Fumaric Acid Delivered, E. of Rockies.	
C.l., bags, 60 lbslb21	
C.1., drums, 250 lbslb22	••
L.c.]. bags, 50 lbslb221/2	
Le.L. drums, 250 lbslb231/2-	
Isophthalic Acid, f.o.b. shipping point, fr	cight
equalized with Richmond, Calif .; Jollet,	111.,
and Port Hewark, N.J.	
C.1. & t.1., bags, 60 lbs., 1b, .1615-	
L.c.L. bags	••
Maleie Anhydride Delivered, E. of Rockles	i.
Tanks	
C.L. drums, 250 lbslb211/2	••
C.I., bags, 50 lbslb201/2-	• •
L.c.l., bags, 50 lbslb22	••
Phthalle Anhydride, f.o.b.	
Tanks	.08%
C.1. or t.1., bags, 50 lbs,lb	.11%
L.c.l., bags, 80 lbslb	.121/4

Polyhydric Alcohols

Olycerine, tanks divd.		
High Oravity, naturallb.	.211/2	••
CP USP sac Naturallb.	.21	
Synthetic sphydrous 99.5% lb.	.22	
All hulk natural has one		
per cent trade allowance		
states of the state of the Argo		
Methyl Chucoside, 1.0.0. http://	20	
III., CBFIORGS, 100-10. Dugs .10.	E 0 20	
Pentaerythritol, technical, divu.,	E. 01 10	
C.]		••
L.c.1	-20	• •
Mono grade		
C1lb.	•••	
L.c.llb.		
Sorbital Bould 7%, tanks		
fob	·· ~	
Deale prove pallate 1005.	••	•••
resh grade, princia, ico,		
\$01108, C.J., druins, J.Q.D.	9534	
WEB.		
Trimethyiolethane, technical, 1.0	U. NOFES-	
C.l., bagslb.	2192	••
L.c.1	.28 /2	••
Trimethyloluropane, delly., c.l.,		
2 tl. Zone 1lb.	.275 —	••

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Latex Paint Vehicles

		Resins
	Wet	118.518
Acrylic Resin Emulsion-	Basis	Bollds.
Interior Grade		
4416 56 solids tanks	.13½—	
Exterior Grade		
the self sector in	1774-	
\$0% BOIIGS, CHIKE	101/	••
50% solids, tanks	.10%-	•••
65% solids, tankslb.	.21 _	• ••
Wet Weight Ifin, frt. alld.		
Add OLIDER IN W of Rockles		
Add VI/Dry ID. W. OI ROUGICS.		
Butadiene Styrene-		~ *
48% solids, tanks10.	.1210-	25
Dry Basis, Min. 1rt. alld.		
Add 01/Dry In. W. of Rockles.		
Polystowi Acatate Homopolymer		
The state of the s		
Entratsion		
55% solids, tanks	.10	
Wet Weicht Min. fri. alld.		
Emulsion-		
ssot solids tonks	.16	
for solids, tanket 1b	1814_	_ **
65% 801103, tRN46,		- ••
Wet Weight Min. frt. alld.		

American Paint Journal

Neventer (2) 1972

Dry

	Total Imp C	orted from Ind: ounty, pounds	icated	
Year	Malaysia	Indonesia	Congo	Total Import, pounds
1970	292,320	377,960	365,388	1,035,688
1971	464,494	196,580	285,370	946,444
1972 ^(b)	259,359	82,635	~	341,994

IMPORT DATA FOR NATURAL RESIN^(a)

(a) These resins are listed under U.S. Imports for Consumption under the heading "Gum, Damar, Copal, Kauri, Sandarac, Varnish Gums, and Congo.

(b) The figures are for the first six months only.

Battelle Elemorial Institute - COLUMNIE FALORATORIES

505 KING AVENUE COLUMBUS, CHID: 4320F ALLA CODU 540 TULLPUON COU PRESSUL A SUSSE ALLA MARI

February 28, 1967

Mr. David A. Ross Northern Coal Mines, Ltd. 1296 Kings Avenue West Vancouver, British Columbia Canada

Dear Mr. Ross:

We have now completed the work so far agreed upon in our investigation of "Refined Canadian Resin in Coatings and in Rubber Compositions". The enclosed report covers this phase of the work.

The results indicate promise for the refined resin, both in oleoresinous varnishes and as a low-cost extender in certain rubber compositions, but further work will be required in both applications. At least some of the additional work should be deferred until a product more representative of the ultimate commercial product is available.

However, I believe that you should give serious consideration to the separation process. We have shown that the solvent extraction process leaves the "Amber" resin behind, and we believe that this may be the most valuable part. We would certainly like to get enough to evaluate it in coatings.

If you can arrange to visit Battelle in the next month or so, I believe that it would be worthwhile to discuss the results obtained so far and to decide the course of future work.

Sincerely yours,

The grant of File.

Maynańd B. Neher Associate Chief Structural Organic Chemistry

MBN:ja

Enc.

SUMMARY REPORT

on

STUDY OF REFINED CANADIAN RESIN IN COATING AND RUBBER COMPOSITIONS

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NORTHERN COAL MINES, LIMITED

February 28, 1967

by

M. B. Neher, G. F. Cremeans, and W. J. Mueller

BATTELLE MEMORIAL INSTITUTE Columbus Laboratories 505 King Avenue Columbus, Ohio 43201

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STUDY OF REFINED CANADIAN RESIN IN COATING AND RUBBER COMPOSITIONS

M. B. Neher, G. F. Cremeans, and W. J. Mueller

INTRODUCTION

During 1966, a preliminary study of the chemical and physical properties was undertaken of a fossil-type resin found in the Sponsor's coal deposit. This evaluation included a cursory study of potential applications of the resin in coatings and in rubber compositions.

This preliminary study indicated potential markets in both the coating and rubber fields. These results, along with the results of the chemical characterizations, were summarized in a letter report from M. B. Neher, dated November 30, 1966.

All of the work described in the present report was performed using the resin obtained by solvent extracting the fraction of coal that floats on a $CCl_4/Varsol$ mixture with a specific gravity of 1.25. The sample was supplied by Dr. Visman. This fraction has been described as "Refined Resin", and is referred to as such throughout this report.

SUMMARY AND CONCLUSIONS*

The Refined Resin was evaluated in rubber compositions for use as (1) a filler and extender, (2) as an ingredient to improve abrasion resistance, and (3) as an antioxidant. The results indicate some potential for use as a low-cost extender, but little or no potential in the other areas.

*The work reported here is recorded in Battelle Laboratory Record Books #23043, pp. 1-11; #24497, pp. 10-30. The Refined Resin was evaluated in oleoresinous varnishes of the type that normally employ fossil resins (e.g., Con_io resin). The Refined Resin appears to have potential application in these coatings. The experimental coatings made from Refined Resin are harder but darker than those made from Congo resin. However, the "Amber" resin picked out of the coal by hand appears potentially more valuable than the Refined Resin.

EVALUATION OF REFINED RESIN AS A COMPOUNDING INGREDIENT IN RUBBER

Refined Resin was evaluated as a compounding ingredient selected rubbers in three areas. These were (1) filler and extender, (2) antioxidant, and (3) ingredient to increase abrasion resistance. These areas were selected on the basis of preliminary evaluations in rubber, and the chemical properties which have been established for Refined Resin.

Filler and Extender

Large quantities of many materials are used as fillers in rubber compounding. These materials are normally added to improve certain properties, and they range over a wide area with respect to particle size and cost, depending upon the requirements of the products. Extenders are often added to supplement the fillers. These are usually low-cost materials which have little effect on properties, either good or bad, and are used primarily to increase the compound volume and decrease its cost.

The results of a preliminary examination of the Refined Resin in Neoprene W are shown in Table 1. SRF Black was used as the control since large quantities are used in this polymer for general-purpose applications. The SRF Black increased the modulus and tensile strength, while decreasing the elongation. The SRF Black also increased the minimum viscosity and decreased both the scorch (45) and cure (435) times. The major effect of using the Refined Resin as a filler (Composition 12) was a large decrease in tensile strength and an increase in elongation and minimum viscosity. The effects on other properties were not large. The effect of supplementing SRF black with a small amount of Refined Resin (Composition 13) was a further increase in the stiffness of the composition as shown by the increases in modulus and minimum viscosity; also, decreases were noted in the scorch and cure time at 250 F.

Comparable data obtained with nitrile rubber are shown in Table 2. With this polymer, the control filler was NT black, which can often be used where high product quality is not required. The use of the Refined Resin as the filler resulted in a vulcanizate with very low tensile strength. On the other hand, the compositions (16 and 17) in which the Refined Resin was used as an extender for the MT black did show the most promising results. The effect on the minimum viscosity was much less than was observed with the Neoprene W composition, possibly indicating greater compatibility of the resin with the nitrile rubber. When used as an extender, the Refined Resin increased the scorch time (A5) at 250 F, but had little effect on the cure time or rate at 302 F.

The results of this study with the two polymers suggest little possibility for the use of Refined Resin as a filler, but indicate that

		(<u>Composit</u> i	ion Numbe	er E
		10	11	12	15
Mooney Scorch and Cure Data	at 250 F				
Minimum Viscosity		32	73	6 6	90
∆5, min		8.4	6.7	9.7	6.0
∆35, min		15.2	11.9	16.1	10.5
Δ35-Δ5, min		6.8	5.2	6.4	4.5
Mooney Scorch and Cure Data	at 307 F				
Minimum Viscosity		34	85	59	101
$\Delta 5, \min$		3.1	2.0	2.9	2.6
$\Delta 35$, min		5.0	3.4	5.1	3.7
$\Delta 35-\Delta 5$, min		1.9	1.4	2.2	1.1
Cı	ure, min.				
100% Nodulus psi	10	NH 1	660	120	250
100% Hodulus, psi	20	50	030	220	900
	30	Nil	620	220	900
300% Modulus, psi	10	130	2840	370	2020
	20	210	-	380	-
~	30	220	-	400	-
Tensile Strength, psi	10	1990	3910	1040	2820
	20	2430	2920	900	2700
	30	2600	• 3000	1000	2920
Elongation, percent	10	3 50	· 320	700	300
	20	730	240	620	230
	30	760	280	640	270
Hardness, Shore A-2	10	75	01	72	75
	20	72	75	75	76
	30	76	80	77	30
Ingredients:					
Neoprene W		100	100	100	100
SRF Black		-	50	-	50
Refined Resin		-	-	50	5
Zinc Oxide		5	5	5	č
Nagnesium Oxide		۷,	4	ζ;	4
Stearic Acid		1	1]	1
Phenyl- β -naphthylamine		?	2	2	2
Z-Mercaptoimidazoline		0.5	0.5	0.5	0.5

TABLE 1.REFINED RESIN AS A FILLER AND
EXTENDER IN NEOPRENE W

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		Composition Number			
		14	15	17	18
Mooney Scorch and Cure Data	at 250 F				
Minimum Viscosity		37	47	33	43
Δ5, min		49.5	5 72.0	62.0	57.0
Δ35, min		57.5	5 88.0	71.0	66.0
Δ35-Δ5, min		8.0) 16.0	9.0	9.0
Mooney Scorch and Cure Data	at 302 F ·				
Minimum Viscosity	Cure, min.	26	22	22	21
Δ5, min		3.8	12.4	9.2	8.7
Δ35, min		10.3	14.3	10.5	10.2
Δ35-Δ5, min		1.5	1.9	1.3	1.5
	<u>at 302 F</u>				
100 percent Nodulus, psi	10	220	Nil	200	240
	20	290	200	290	300
	30	300	220	300	350
300 percent Modulus, psi	10	600	Nil	520	420
	20	1010	250	970	840
	30	1100	300	1040	920
Tensile Strength, psi	10	2080	320	1900	1510
	20	1730	800	1720	1570
	30	1780	620	1660	1380
Elongation, percent	10	720	>1000	780	780
	20	500 -	710	510	540
	30	490	560	480	430
Hardness, Shore A-2	10	62	60	65	69
	20	65	66	64	69
	30	66	70	66	70
Ingredients:					
Hycar 1041 MT Black Refined Resin Zinc Oxide Stearic Acid Sulfur Benzothiazyldisulfide	· · · ·	100 50 - 5 1 2 1.5	100 - 50 5 1 2 1.5	100 50 5 1 2 1.5	100 50 20 5 1 2 1.5

TABLE 2.REFINED RESIN AS A FILLER AND EXTENDER IN A
BUTADENE-ACRYLONITRILE COPOLYMER (HYCAR 1041)

it may have some merit as a low-cost extender. Further work would be required to establish this point. At the same time, we would need to have a fairly accurate estimate of the planned selling price for the resin, so that materials could be selected as controls against which it would probably be competing.

Improvement of Abrasion Resistance

Earlier studies at Battelle have shown that the abrasion resistance of some nitrile rubber compositions can be significantly improved by the addition of a phenolic resin. The nitrile compositions which are so affected are those which contain a low-cost carbon black which provides a low degree of reinforcement. To determine whether the Refined Resin was able to function similarly, the compositions shown in Table 3 were evaluated. Composition 24, containing a phenolic resin, Durez 12687, showed only 14 percent as much abrasion loss as Composition 23 (no additive); whereas Composition 25, containing Refined Resin, showed three times as much abrasion loss as the additive-free composition.

These results clearly show that the Refined Resin does not provide the abrasion resistance afforded by the phenolic resin.

Antioxidant

The characterization study on the Refined Resin reported in a letter of November 30, 1966, by M. B. Ncher, indicated that the resin had hydroxyl funtionality, although it was not established whether this was aliphatic or phenolic in nature. Presently, several hindered phenols and diamines are used as rubber antioxidants. The phenols are used where

	Composition Number					
	23	24	25			
100 percent Modulus, psi	400	1180	460			
300 percent Modulus, psi	1880	2980	1230			
Tensile strength, psi	2640	2980	1880			
Elongation, percent	410	300	450			
Hardness, Shore A-2	70	86	30			
Pico Abrasion Loss, cc	0.022	0.003	0.063			
Ingredients:						
Hycar 1041 HMF Black Refined Resin Durez 12687 Zinc Oxide Stearic Acid Sulfur Benzothiazyl disulfide	100 40 - 5 1 2 1.5	100 40 - 30 5 1 2 1.5	100 40 30 - 5 1 2 1.5			

TABLE 3.REFINED RESIN AS AN ADDITIVE FOR
IMPROVING ABRASION RESISTANCE

	Composition Number					
	18	19	20	21	22	
Unaged Properties						
100 percent Modulus, psi	200	140	200	190	170	
300 percent Modulus, psi	1560	. 1480	1460	1400	1210	
Tensile strength, psi	2930	3100	2720	2550	2220	
Elongation, percent	480	500	460	450	440	
Hardness, Shore A-2	56	56	54	56	55	
Properties After Aging 4 Days at 212 F	·.					
100 percent Modulus, psi	370	400	350	360	3 50	
Tensile strength, psi	1520	2280	1180	1230	1170	
Elongation, percent	230	310	210	210	220	
Hardness, Shore A-2	61	64	59	62	63	
Ingredients:						
Smoked Sheet	100	100	100	100	100	
SRF Black	45	45	45	45	45	
Zinc Oxide	5	5	5	5	5	
Sulfur -	2.5	2.5	2.5	2.5	2.5	
Stearic Acid	1	1	. 1	1	1	
Benzothiazyl Disulfide	1	1	1	1	1	
Phenyl-β-naphthyl Amine	-	2		-	-	
Refined Resin	-		• 2	5	10	

TABLE 4. REFINED RESIN AS AN ANTIOXIDANT IN NATURAL RUBBER

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staining and discoloration cannot be tolerated, while the diamines are used in other applications because of their better performance.

To determine whether the Refined Resin has any antioxidant activity, a study was made with natural rubber, using phenyl-g-naphthylamine (PBNA) as a control antioxidant. Property determinations were made on unaged compositions and on compositions aged for four days at 212 F, following standardized aging procedures. Antioxidant activity was judged by the differences in tensile strength and elongation between the unaged and aged compositions.

Table 4 shows that Composition 19 containing the PBNA had a much higher aged tensile strength and elongation than any other composition. The three compositions comtaining the Refined Resin were no better, and possibly a little poorer, than the Composition 18 containing no added antioxidant. Thus, the results clearly show no antioxidant activity for the Refined Resin.

EVALUATION OF REFINED RESIN IN COATINGS

In general, the physical and chemical properties of oils and resins used in the preparation of a varnish carry over into the finished vehicle. The primary reasons for adding hard resins to drying oils are:

- (1) To increase the hardness of the film
- (2) To improve water and chemical resistance
- (3) To improve the gloss
- (4) To shorten the drying time.

These improvements are normally attributed to the fact that the hard resin ' added is usually hard, glossy, and inert to acid, bases, and water.

The trend in the past twenty years has been to use synthetic "hard" resins in the varnish-making trade to replace the fossil resins that were once used almost exclusively. The reasons for this are primarily: (1) uniformity of product, (2) increased production capacities for synthetics, and (3) synthetic resins are competitive with fossil resins in price. However, fossil resins are still being used to prepare nominal amounts of oleoresinous varnishes, with Congo resin being one of the most popular. This material sells for \$0.21 to \$0.28 per pound, depending on quality and type.

It was initially decided to compare Refined Resin directly with Congo resin in similar coatings formulations. This involved a thermal degradation of the resin until it became soluble in a fatty acid triglyceride. For this program, linseed oil was chosen. In addition, other type formulations (resin-esters) were tried and other means of incorporating the Refined Resin into polymeric vehicles were used. The solubility of the Refined Resin in rosin derivatives was also established.

The degree of difference between a linseed oil film and a modified linseed oil film naturally depends on the hard resin content of the coating. The varnish trade refers to short-, medium-, and long-oil varnishes. This terminology indicates the gallons of oil used per 100 pounds of resin and the following definitions are used:

(1) Short-oil, 6-18 gallons oil/100 pounds resin

(2) Medium-oil, 18-30 gallons oil/100 pounds resin

(3) Long-oil, 30-60 gallons oil/100 pounds resin.

In general, hardness and chemical resistance decrease as the oil length increases, but flexibility of the film is proportional to

the oil content. For the purpose of this study, varnishes having oillength values of 10, 20, and 40 were used.

Oleoresinous Varnishes

The normal procedure for incorporating Congo or other insoluble hard fossil resins is to heat the resin at 330 to 345 C until oil solubility is attained. Congo resin is usually brought to 335 C in successive heats, each requiring about 1.5 hours. During this time, a 20 percent weight loss occurs, with the acid number of the resin decreasing from 112 to about 85.

The degree of heating required to degrade the Refined Resin was determined before any effort was made to incorporate it into a coating. To do this, 200 grams of Refined Resin was heated in a stainless steel beaker under a carbon dioxide spurge. The temperature of the resin was raised to 330 C and small samples removed at 30-minute intervals and tested for oil solubility. In addition, weight-loss data were obtained. The results of this experiment are summarized in Table 5. These data show that considerable degradation has to occur before the resin becomes readily soluble in linseed oil. Nevertheless, it was found that if the resin is heated to 327 C and held for 30 minutes (in a nitrogen atmosphere), the resulting processed resin can be dissolved in linseed oil by heating the oil and the resin for about three hours at 230 C. Congo resin, when thermally processed until a 20 percent weight loss occurs, will dissolve in less than one-half hour under similar conditions. However, since the thermal processing of Congo resin rarely results in a loss greater than 20 percent weight, it was decided to use

	Weight Loss,	Solubility in Linseed Oil				
Processing Time under CO ₂ at 327 C, Minutes	percent of Initial Resin	100 C for 5 Min	Heating at 230 C Until Solubility is Attained			
0.0 (no thermal processing)	0.0	Insoluble	Insoluble after 5 hr			
1.0 ⁽¹⁾	•	Insoluble	Insoluble after 5 hr			
30.0	20	Insoluble	Soluble after 3 hr			
60.0	29	Insoluble	Soluble after 2 hr			
90	- _	Insoluble	Soluble after 1 hr			
120	40	Soluble ⁽²⁾	Soluble in less than 1 $hr^{(2)}$			
			· · · · · · · · · · · · · · · · · · ·			

TABLE 5.THERMAL PROCESSING OF CANADIAN AMEER RESINNECESSARY TO OBTAIN AN OIL SOLUBLE PRODUCT

(1) A 30-minute heating time was used to raise the temperature of the resin from 25 C to 327 C.

(2) The resin at this point consisted of two materials--a carbonacious foam on top and a liquid resinous mass in the bottom of the reactor. The solubility of the resinous mass is reported.

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Refined Resin also thermally decomposed until a 20 percent weight loss was observed. It is recognized that many economic factors will determine whether or not the Refined Resin will compete with Congo resin or similar resins to the varnish industry. The cost criterion that will be used by a manufacturer of varnishes is the production cost of the final coating. This will include pounds of faw resin required to produce one gallon of varnish, as well as the kettle time required to produce the final product.

Accordingly, three basic linseed varnish formulations were prepared having oil lengths of 10, 20, and 40, using the thermally processed Refined Resin and Congo resin (supplied by 0. G. Innes Corporation). Coatings were deposited on steel and tinplate panels and cured at either ambient temperature (25 C for 1 week) or elevated temperature (30 minutes at 177 C). The Refined Resin coatings were compared with the control coatings, Congo-linseed varnishes, and linseed oil coatings for resistance for 40 percent nitric acid. In addition, hardness and flexibility measurements were made on all of the coatings.

The evaluation data are shown in Tables 6 and 7. All of the baked coatings had excellent resistance to methyl ethyl ketone, while the coatings cured at ambient temperature were dissolved in one to two minutes. As shown in Tables 6 and 7, the varnishes prepared using the Refined Resin compare favorably with the Congo controls. The Refined Resin imparts slightly more hardness to the linseed oil varnish while not harming its flexibility. However, the coatings formulated using the Refined Resin are considerably <u>darker</u> than the coatings prepared using the Congo resin.

Resin	Oil Length,(1) gal	Film Proper Flexibility ⁽³⁾	ties(2) Sward Hardness ⁽⁴⁾	Resistance to 40 Percent Nitric Acid, hr until failure
Congo	10	Failed 1 in.	60	5.5
Canadian	10	11	69	5.0
Congo	20	Passed 1/8 in.	44	2.3
Canadian	20	**	49	3.5
Congo	40	u .	12	1.3
Canadian	- 40		21	t.3
Linseed Oil Control		11	4	0.5

COMPARISON OF LINSEED OIL VARNISH FILMS PREPARED TABLE 6. USING CONGO AND REFINED RESIM AND CURED BY BAKING AT 177 C FOR 30 MINUTES

(1) Number of gallons of linseed oil per 100 pounds of resin.

All of the films were about 0.8 mil thick 1: 0.1 mil. (2)

Figure represents the smallest diameter of a rod that the coating on tin-(3) plate can be bent over through 180 degrees without breaking the coating.

(4) Compared to plate glass having a hardness of 100.

	0í1 (1)	Film Proper	Resistance to 40 Percent Nitric Acid,	
Resin	Length, ⁽¹⁾ gal	Flexibility ⁽³⁾	Sward Hardness(4)	hr until failure
Congo	10	Failed 1 in.	32	5.5
Canadian	10	17	65	5.5
Congo	20	Passed 1/8 in.	21	3.5
Canadian	20	11	37	2.3
Congo	40	н	7	1.3
Canadian	~ 40	11	11	1.3
Linseed Oil Control		"	. 2	0.5

TABLE 7.COMPARISON OF LINSEED OIL VARNISH FILMS
PREPARED USING CONGO AND REFINED RESIN
AND CURED AT 25 C FOR ONE WEEK

(1) Number of gallons of linseed oil per 100 pounds of resin.

(2) All of the films were about 0.8 mil thick \pm 0.1 mil.

(3) Figure represents the smallest diameter of a rod that the coating on tinplate can be bent over through 180 degrees without breaking the coating.

(4) Compared to plate glass having a hardness of 100.

<u>Attempts to Use Refined Resin in</u> <u>Coatings Without Thermal Degradation</u>

Early in this program it was observed that the unprocessed Refined Resin had an acid number of about 55 and is soluble in linseed fatty acids and insoluble in linseed oil. It was felt that perhaps on heating in the presence of litharge, an ester interchange would occur between the Refined Resin and linseed oil. An experiment was carried out by heating 12.0 g of Refined Resin with 20.0 g of linseed oil containing 0.3 g of litharge at 230 C. This mixture jelled after 3 hr of heating and never became homogeneous.

Next, 10.0 g of Refined Resin was added to 16.0 g of linseed fatty acids, and the whole mixture reacted with 1.3 g of pentaerythritol at 232 C for about 3 hr. The product was a dark resinous material soluble in toluene. Coatings prepared from this material gave air-dry and baked coatings with Sward hardness values of 20 and 30 and having excellent flexibility and adhesion.

In addition, an attempt was made to degrade the Refined Resin by adding it to a refluxing 5 percent solution of sodium hydroxide. The resin was kept in contact with the refluxing basic solution for six hours and then acidified with hydrochloric acid. The product was still insoluble in drying oils and could not be used to prepare an olcoresinous varnish without further processing.

The Refined Resin appears to be comparable to Congo resin for use as the hard resin component in varnishes. The varnishes made with Refined Resin give films that are darker than those prepared with Congo resin, but have slightly higher hardnesses with no apparent sacrifice in flexibility. The results also indicate that film-forming materials

can be prepared if Refined Resin-linseed oil mixtures are esterified with a polyol such as pentaerythritol. Otherwise, it appears that the Refined Resin will have to be thermally degraded prior to using it in the preparation of oleoresinous varnishes.

RECOMMENDATIONS FOR FUTURE WORK

This study indicates that the Refined Resin may find application as a low-cost extender in rubber. Further work will be required to establish this point.

This study also indicated that the Refined Resin gives eleoresinous coatings with physical properties equal or superior to coatings made with Congo resin. This study should be expanded to include a more comprehensive evaluation of co-esters of the Refined Resin with various polyols and drying oil fatty acids. In addition, a brief study should be made to establish the parameters of processing variables (both thermal degradation and varnish preparation) required with the Refined Resin.

However, the "Amber" resin which is not being extracted is potentially even more useful than the Refined Resin which was used in the current study, because of its lighter color and lower ash content. Any future work should include study of this material.

Thus, serious consideration should be given to the process used to separate "resin" from "coal". As recorded in the last report (November 30, 1966), the Refined Resin is in reality a soluble fraction of the coal, and the "Amber" resin remains in the extraction residue.

It is believed that this "Amber" resin probably represents a more valuable product than the Refined Resin obtained by the present extraction process.

Further, we understand that the material which will be produced in the ultimate plant will be obtained by an air classification process. In light of our observation that the "Amber" resin is completely unaffected by the solvent extraction, it is probable that the commercial product will be different. Future work plans should include study of a product more representative of the ultimate commercial material than the Refined Resin.

Battelle has had considerable experience in ore and mineral extraction, and we would be able to bring this background to studying separation of the "Amber" resin from raw coal. The sample of extraction residue currently on hand could be a starting point.

Battelle Memorial Institute · COLUMBUS LABORATORIES

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November 30, 1966

Mr. David A. Ross Northern Coal Mines Ltd. 1296 Kings Avenue West Vancouver, British Columbia Canada

Dear Mr. Ross:

This letter summarizes the work done on our study of the composition of Northern Coal Mines' resin from Coal from June 1, 1966, until November 1, 1966. Since the ten-pound sample of Refined Resin arrived only-recently, the evaluation studies in rubber and in coatings have just been started and will be reported upon at a later date.

Characterization of Refined Resin

Chromatographic and spectral data indicate that the Refined Resin is a complex mixture of some 40 - 50 components having hydroxyl, carboxylic, and ester functionality. In my letter of June 15, 1966, it was indicated that no aromatic components were present in the Refined Resin. More recent studies now indicate that the Refined Resin is predominantly a mixture of condensed ring aromatics with aliphatic substitution on the rings.

The Refined Resin is very resistant to hydrolysis. The resin could not be completely hydrolyzed by treatment for a period of 2 - 3 days with a 20 percent solution of sodium hydroxide in either water or a water-ethylene glycol mixture at temperatures up to 200°C. Only a small amount of hydrolytic products was obtained from these treatments of the resin. These products appeared to be a more complex mixture of materials than the original resin and no attempt was made to characterize them.

More informative data were obtained by solvent fractionation of the Refined Resin according to the scheme shown below and examination of the resulting fractions.



The infrared spectra of the various fraction were similar to one other and to the original resin. These data indicate that: (1) the resin is probably • a mixture of materials of a single class or family of chemical compounds rather than a mixture of different chemical classes, and (2) the various fractions obtained by the solvent fractionation represent different molecular weight species present in the resin.

The diethyl ether soluble fraction (D), 1.96 g, from the above fractionation was partially hydrolyzed by treatment for 16 hours with a 3.2 percent solution of sodium hydroxide in a 10:10:1 mixture of dimethyl sulfoxide, ethyl alcohol, and water. An acidic fraction was obtained which contained some ester carbonyl as shown by infrared examination. However, no neutral alcoholic fraction, i.e. material containing hydroxyl, but no carboxylic acid functionality, was obtained.

The acidic fraction of D was esterified with diazomethane and then fractionated by thin-layer chromatography (TLC) on Silica Gel G using a 10 percent v/v solution of acetone in diethyl ether as developing solvent. Five fractions of materials were obtained. Infrared examination indicated that each fraction consisted of a mixture of materials. The infrared spectra of four of the fractions having R₁ values[#] in the range 0.03 - 0.95 were similar and showed hydroxyl and ester carbonyl functionality. The fifth fraction, having an R₁ range of 0.95 -1.00, was a neutral material, i.e., contained no hydroxyl or ester functionality, and was aromatic in nature.

The fact that no neutral material containing hydroxyl functionality was recovered from the hydrolysis of fraction D suggests that the precursors of the resin are hydroxy acids. Hence, the ester carbonyl of the resin may arise from, (1) intraesterification to produce a lactone, or (2) interesterification to produce a normal ester. Some evidence in support of these hypotheses is found in the nuclear magnetic resonance spectrum of the resin which shows two types of esters to be present in the resin.

" The R_c value of a component is the distance that component has moved on the TLC plate divided by the distance which the solvent front has moved.

Mr. David A. Ross

A sample of the Refined Resin was reduced to hydrocarbons by distillation with zine dust. Examination of the reduced product by infrared, nuclear magnetic resonance spectroscopy and gas liquid chromatography showed it to be a complex mixture of hydrocarbons consisting predominantly of aromatic or condensed-ring aromatics with alkyl substitution on the rings. The infrared spectrum of the reduced material resembles the spectra of coals and asphalts, especially in the aromatic substitution region of 950 - 650 cm⁻¹.

On the basis of the above date, it is believed that the Refined Resin is a mixture of condensation products of a complex aromatic hydroxy acid highly substituted on the ring(s) with alkyl groups. Because of the complexity of the resin no attempt was made to determine whether the hydroxylic functionality was aliphatic or phenolic in nature, or a mixture of both types.

Because of the complexity of the resin, a considerable amount of work would be required to do a complete elucidation of its structure, and at present, further effort in this direction does not seem to be warranted.

Comparison of Amber Regin and Refined Regin

Very early in this program some incongruities in the properties of Amber Resin and Refined Resin were observed. Amber Resin is the amber-colored clear material which can be picked out of the coal by hand. Refined Resin is the dark-colored material obtained by the extraction process which was employed to obtain the ten-pound batch of resin recently received.

As reported in my letter to Mr. Ross of June 15, 1966, the following differences were observed.

- Infrared spectra indicate that the Amber Resin is principally ester, whereas Refined Resin is a mixture of ester and carboxylic acid. The composition of Refined Resin seems to lie between that of Amber Resin and the original coal.
- Amber Resin is, for all practical purposes, completely insoluble in chloroform, benzene, or pyridine. By contrast, Refined Resin is completely soluble in pyridine and soluble in varying extents in a number of other solvents.
- 3. Refined Resin softens at ~ 200 C. Amber Resin does not soften at temperatures even at 400 C. A differential thermal analysis (DTA) in nitrogen, indicates that Amber Resin does not melt until ~ 450 C, at which temperature it is volatilized (probably with decomposition).

Further consideration of these points has led to speculation about the separation being achieved by the solvent extraction process. Accordingly, samples of the various fractions involved in the separation were obtained from Northern Coal Mines Ltd. and analyzed by infrared spectrometry and examined microscopically.

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Mr. David A. Ross

The microscopic studies together with the infrared spectra showed conclusively that the Amber Resin is not being removed by the extraction process. It remains in the extraction residue. Figure 1 is a photomicrograph of Amber Resin picked out of coal by hand. The sharp edges and conchoidal fracture pattern are especially important. (The black material is coal which remained stuck to the resin.) Figure 2 is a photomicrograph of Refined Resin. These particles are of an entirely different character than the fragments of Amber Resin. None are transparent, and even those that are translucent are reddish brown rather than light amber as in the case of Amber Resin. Figure 3 is a photomicrograph of the residue left after the extraction process by which the Refined Resin is obtained. There are many clear, light-amber particles which are obviously the original Amber Resin, along with some coal residue. These clear particles still have the conchoidal fracture pattern, which indicates that they have not been affected by the solvent extraction.

I understand that the commercial plant to make resin will use an air classification process. Our work demonstrates rather conclusively that the extraction process simply dissolves the more soluble coal fractions. Ey contrast, the air separation process will function on an entirely different basis -relative particle densities. The air separation process thus may give a product rich in Amber Resin, and if so it would be vastly different from the present Refined Resin.

We are currently attempting to isolate as much of the Amber Resin as possible to study its properties further. Included will be an attempt to use air classification on the five pounds of extraction residue we received some time ago.

It has been some time since you visited Battelle, and I believe another visit in the near future to discuss the current status of the project would be helpful.

Sincerely yours,

greenet Polichia.

Mayhard B. Neher Associate Chief Structural Organic Chemistry

MBN: KR

Enc. (3)







Low-Btu gasification process will use coal to run teams of turbines

First, take a system that is quickly capturing the interest of power generating companies throughout the world-the combined-cycle turbine system.

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The combined cycle teams a gas turbine together with a steam turbine (top drawing, right). Gas-or oil-is fed to the gas turbine where it burns as fuel with incoming air. The hot, expanding gases of combustion drive the turbine, which in turn runs a generator to produce electricity.

But instead of exhausting the hot gases into the atmosphere as lost heat, as is done in conventional turbines, the gases in the combinedcycle system are shunted through a heat-recovery boiler containing water pipes to turn the water to steam. The steam then drives a typical steam-turbine system. Since the electric power from the steamdriven generator can be added to that from the gas-driven generator, the resulting power generation is more efficient than if only a conventional gas turbine or steam turbine is used.

All this, however, is not new. Although the combined-cycle concept is only recently catching on, it has been around since 1961 when Oklahoma Gas and Electric Co. picked it for one of their power plants.

What is new is a low-cost method by Westinghouse Electric Co. for taking coal and converting it directly into a gas (second drawing), without any of the enrichment processes normally employed in coal conversion systems. To be sure, such unenriched gas has a low-Btu heat content—it may have only about 150 to 200 Btu's per cu ft, as compared to the 900 to 1,000 Btu's per cu ft obtained from the high-Btu gas-conversion systems—and thus would not be suitable for long-distance piping.

But if this new low-Btu process is hooked up directly to feed one or two gas turbines in a combinedcycle plant (bottom drawing)-why, the benefits are many: The low-Btu system is inexpensive-you're taking coal and using it directly as fuel, not really trying to produce a marketable gas. And the pollution control aspects are favorable--sulfur from high-sulfur coal of the Appalachian and Midwestern reserves is easily removed during the conversion process; and so are the tars and particulate matter.

According to Bruce Morrison, general manager of Westinghouse's Heat Transfer Div., the new process can be tied to the Westinghouse PACE power plant, a combinedcycle system. The PACE system is being sold already as a packaged unit; and when used with the low-Btu process, Morrison predicts, the total system will permit generation of electricity at significantly lower cost than using today's conventional coal-burning plants with stack gascleanup systems.

The multimillion-dollar Westinghouse development program for the process has, as its objective, the goal of having a complete power plant operating by 1980.

Partners in the Westinghouse project include Public Service of Indiana, which will operate a prototype coal gasification plant as part of its generating system; AMAX Coal Company, a subsidiary of American Metal Climax, Inc.; and Bechtel Corp. This team is seeking financial support for the program from the federal government. In addition. Northern Indiana Public Service, the Tennessee Valley Authority and Consumers Power Co. have agreed to support the team effort as associate members in advancing this necessary technology.

Fluidized-bed gasifier

Westinghouse will be using a

Nicholas P. Chironis Senior Editor, Coal Age series of fluidized beds in their coal gasification process. The fluidized bed can be thought of as a grate of continually changing configuration which supports the coal particles and brings them into contact with the air and steam while preventing agglomeration and clinkering through its turbulent motion. Such a device would not be sensitive to small coal particles and could handle the fine particles, which represent some 20-30% of today's mechanically mined coal.

Segregation of particles in a fluidized bed by their relative densities allows a continuous ash stream to be tapped off at the bottom and permits chemical absorption of sulfur by a stream of limestone particles flowing through the bed. The stream of limestone particles can then diffuse through the bed, reacting with sulfur molecules as they are released from the burning coal, and can be tapped off as a stream of calcium sulfide particles. The low-Btu fucl gas coming off the top of such a bed would contain very little sulfur and require cleaning only to remove particulates.

The final system requires a series of beds to induce countercurrent flow of gases and solids and to maximize the removal of sulfur by lime injection. Each bed must operate at temperatures appropriate to the chemical reactions taking place in it-2,100F in the agglomerating chamber. 1.800F in the total gasifier, and 1.400F in the devolitilizer.

In commenting about the future of such coal-fired combined-cycle plants, a Westinghouse spokesman noted that even if the main generating plant for an area employs nuclear energy, the new system can take on the job of providing generation for intermediate loading. The base plant--the nuclear plant-would operate all the time, while the intermediate plant would start up, say, around 7 AM and shut down at 6 PM, thus taking up the excess power demands during the day.

COAL AGE · May, 1972



COAL AGE . May, 1972

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Western Miner September, 1972.

REMOVING SULPHUR FROM COAL

Under the direction of Dr. James H. Gary, head of the Chemical and Petroleum-Refining Department at the Cherado School of Mines, a unique process of removing sulfur from bituminous and sub-bituminous coal is developing.

The project, awarded by the U.S. Department of the Interior, has been funded \$78,675 for the first research year.

Presently, the research team is working with coal ores which in their natural state exceed present air-pollution standards of more than one per cent sulfur by weight. Thus the coal presently cannot be used as an energy fuel for power plants without sulfur reduction to permissible levels.

"By the hydrogen induction during the coal treatment, approximately 90 per cent of the sulfur has been removed." Dr Gary commented.

The coal is crushed and mixed with an organic solvent during the first step of the process. Once the coal is in solution the liquid is placed in an autoclave which attains a temperature of 750° F and 1.000 pounds-per-square-inch pressure. Under these conditions, the solution is agitated with the hydrogen introduced; after seven and one-half to fifteen minutes, the sulfur has been removed. The unwanted sulfur ieaves the autoclave as hydrogen sulfide in a gas form.

The final step is to take the desulfurized solution and remove the organic solvent. Removal allows the recycle of the solvent for future coal treatments,

On a large-scale basis, it is anticipated that the cost of desulfurization will be less than that of other techniques." Dr. Gary stated, "with the process being performed even at the mine provided an adequate source of hydrogen gas was available."

Several large deposits of bituminous and sub-bituminous coal ores would benefit from this treatment. Coal in the Appalachians and some coal in both Colorado and Montana fall in this category. They are low in ash content and suitable for an energy fuel but presently exceed pollution standards due to a one to four per cent sulfur content.

Once optimum conditions are established by the research team, an economic evaluation will be made to determine the desulfurization costs.

A. J. LAMB, P.Ag. CONSULTING AGROLOGIST Open Pit Reclamation and Revegetation Hydro-Seeding and Reforestation INTERIOR REFORESTATION CO. LTD.

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GEOLOGICAL SURVEY OF CANADA TECHNICAL REPORT NO. 93-H-13W-1

PETROGRAPHY OF THE COAL FROM THE GARROWAY MINE IN THE BOWRON RIVER COAL AREA, BRITISH COLUMBIA

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ABSTRACT

A petrographic study was carried out on coal from the Garroway Mine in the Bowron River-Coal area in British Columbia.

The seam studied is of Tertiary age and the examination was part of a larger investigation on Canadian Coals of Tertiary Age.

Though the coal is reported as being a coking coal, stability predictions based on the chemical composition as determined by proximate analyses, reflectance (R_0) indices and maceral composition indicated otherwise. Results showed that the reactive-inert imbalance predominantly favoured the reactive components. These constituents contribute good swelling characteristics to a coke but lack of an optimum amount of the inert or dull coal components result in a coke that is weak.

The coal is reported to be high in percentage of the maceral resinite. Visible reain nodules were observed in the coal during sampling. The subsequent microscopic analysis did not substantiate this observation because this type was not observed during the analysis. Resin of the invisible type, due to vitrinization, could be treated only as a vitrinoid.

Though this coal would not produce a coke of good quality by itself it could be used possibly as a blend to mix with a coal which has good strength contributing properties and a lack of good swelling characteristics. INTRODUCTION

The existence of coal on the Bowron River in British Columbia has been known for the last 100 years. Dawson noted in 1871 that coal was reported by Dewdney on the banks of the Bear River - later known as the Bowron River.

The scam sampled is from the Northern Coal Mines Ltd., Garroway Mine (see Fig. 1) which is located on the west bank of the Bowron, about 35 miles east of Prince George - a rapidly growing rail and industrial center. The mine is only five miles south of a newly paved highway that has recently been constructed from Prince George.

Due to the increased interest in B.C. coking coals, considerable development work has been done in the Bowron River area. Petrographic studies have been undertaken to determine the nature of the coal and its suitability for coking. The coal is reported to have a high resin content and the present company set out to mine the coal solely for the resin and have the coke as a low priced by-product. Favourable indications from swelling tests coupled with the mine's favourable location in respect to the Japanese market reversed this policy.

GENERAL GEOLOGY

The Bowron River valley is almost entirely covered with overburden and therefore the coal seam outcrops can only be observed along the banks of the river and along the creeks.

The rising ground on the valley-sides and a few points along the river itself are underlain by a group of largely green-coloured volcanic rocks and include fine tuffs, breccias and lavas. The only intrusives seen are several small dykes which intrude dark argillite near the north of Purden Creek (Holland 1948).



FIGURE 1. SAMPLE LOCATION MAP, BOWRON RIVER, B.C.

The coal is found in a series of sandstones, shales and conglomerates that outcrop along the river. It is possible that the coal-bearing series may outcrop farther to the northwest and southeast but the formation has not been traced farther in those directions.

The coal formation comprises grey and buff sandstones inter-bedded with light to dark soft shales and sandy shales, some of which are carboniferous as well as several beds of conglomerate. The conglomerate contains cobbles of as much as green volcanic rock / 10 inches in diameter proving that the volcanics are the older. Several beds, / 15 feet thick, of grey conglomerate outcrop along the river. The distribution of coal-bearing outcrops suggests that the formation underlies the Bowron River valley forming a belt 1¹/₂ to 2 miles wide and about 7 miles long.

Nolland (1948) reported that a collection of fossilized plant remains was submitted to the late Dr. W.A. Bell for identification who reported, "the fossil plants from the Bowron River area are poor indeed....but....I consider the age to be Tertiary". In 1968 Dr. Bell, in an unpublished report stated, "the evidence, unsatisfactory as it is, favours a time interval in the Upper Cretaceous, within the limits of Campanian-Maestrichtian and particularly a Campanian one".

From the foregoing it is obvious that dating the Bowron River measures is very difficult, the problem being compounded by the lack of good fossil material.

MacKay (1946) noted that the coals of the Bowron area are of Tertiary age, and at least 3 of the coal seams are of commercial interest. He also states that the coals have been classed by the Provincial Government as bituminous in rank.

In 1969, Dr. G.E. Rouse of the University of British Columbia, Dept. of Botany, reported, "I am reasonably sure that the Bowron suite is Tertiary in age; most likely between iddle Paleocene and mid Eocene, with a chance of Though the age of the measures is in doubt due to the lack of good fossil evidence, it is generally considered that the coals found in the Bowron River area are of Tertiary Age.

Sample Preparation and Megascopic Examination

A column sample was taken from the upper seam of the productive part of the coal measures from the Garroway Mine, stated to be 10 ft. thick.

The sample represents 7 feet of coal exposed at face of prospect slope. The column was taken to the laboratory cut into smaller, more manageable blocks and mounted in Paraplex - a plastis mounting medium.

After mounting, these blocks were labelled as to their relative position in the column and oriented with respect to top and bottom.

The blocks were then polished on a Buehler polishing lap using a standard acceptable procedure. They were then examined megascopically using a hand lens and a very low power stereoscopic microscope (X5).

In general appearance, the coal examined is a normal banded coal composed almost entirely of the bright components vitrain and clarain. Subordinate amounts of shaly coal, coaly shale and shale also occur. The coal has a high lustre and is hard and dense.

The megascopic profile of the seam is reproduced in figure 2, which shows that the seam is split by four distinct partings. The pure coal, contained between these partings was divided into nine petrographic intervals based on the relative proportions of the entities present.

The resulting breakdown showed that intervals I and V are composed mainly of the shale-rich entities while the remaining. 7 are of clean coal which differs only in the vitrain to clarain ratio. Vitrain exceeds clarain in interval II, IV and IX while the reverse is true in intervals III, VI, VII and VIII. Throughout these bright intervals the shaly impurities occur in minor percentages.

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FIGURE 2. PETROGRAPHIC COMPOSITION AND R. INDICES OF THE UPPER SEAM, BOWRON RIVER, B.C.

Microscopic Examination

Coal from the megascopically determined intervals was then prepared for microscopic examination.

The coal representing each interval was crushed to -20 mesh and riffled to yield a representative sample. A grain mount was prepared from each interval and polished on a Buehler Automet polishing lap. The polished grain mounts were then used for reflectance measurements and maceral determination. Reflectance measurements are normally made on vitrinite, the most abundant constituent in most coals. Measurement of this parameter is a petrographic way of expressing rank. An increase in rank is accompanied by an increase in reflectance and this can be measured quite precisely. Such measureusually ments on a given coal are '/ reported in two ways:

- An average of a number of readings may be calculated and the rank of the coal expressed by a single reflectance index.
- 2. The reflectance data also may be used to subdivide the vitrinite into so-called "V" types. These are designated by number as V6, V7, etc. These numbers signify a reflectance range. Thus V6 means vitrinite with a reflectance range between 0.60 and 0.69% in oil, while V7 has a range between 0.70 and 0.79%.

Reflective indices were determined with a Leitz microscope fitted with a photometer. This is a similar arrangement to that which is generally accepted and used by other petrology laboratories. The instrumentation and its standardizations are described in the "Third Draft, Equipment and Procedures for Determining Microscopically the Maximum and Mean Minimum Reflectance of the Organic Components in Polished Specimens of Coal, ASTM Designation D5 Subcommittee XVIII Petrographic analysis of Coal" by J.L. Bayer. It is described also by Schapiro and Gray (1960).

The results of the reflectance study are shown on figure 2 in the right hand column headed R_0 . These show that the mean maximum reflectance of the vitrinite component is almost constant from the top to the bottom of the seam.

- 6 -

That there is little variation in the consistency of the vitrinite is shown by the fact that the reflective index of the various intervals only fluctuates from 0.64 to 0.65 which is within the range of the statistical error encountered in the work. The average reflectance for the entire seam is calculated to be 0.65.

A further indication as to the lack of variety in the vitrinite is shown by the fact that there were only 4 vitrinite types in evidence in the entire nine intervals which ranged from V4 to V7. The vitrinite type V6 acper cent counted for 66/ of these reflectance readings.

The rank of the coal was determined chemically using the method outlined in the A.S.T.M. standards on coal and coke (1962). The coal was found to be of high volatile B bituminous rank. Rank as determined by reflectance was in accord with the A.S.T.M. method.

The grain mounts used in the above R_o study were repolished and re-examined to determine the maceral content of the coal. Macerals are the basic constituents of coal and are analagous to the minerals of inorganic rocks. The ones identified in the present study are vitrinite, exinite, resinite, micrinite, semifusinite and fusinite. Mineral matter also was determined. Definitions of the macerals as well as the method followed in determining their quantity conform to those suggested in the International Handbook of Coal Petrology (1963). The results of this examination are shown graphically in the center column of figure 2 and numerically in table I. During the analysis the macerals semifusinite and fusinite were considered separately but these later combined to produce the column in figure 2 because they appear to represent stages in a genetically related group. The fact that the combined total of these two macerals per cent does not exceed 5/ in any interval facilitated plotting.

As previously stated the seam in the Garroway Mine is very bright, hard and has a high lustre. The maceral analyses of the various petrographic intervals substantiates this initial impression.

- 7 -

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Interval	Height Inches	Vitrinite %	Exinite %	Resinite	Micrinite	Semifusinite	Fusinite	Mineral Matter %	Total
. IX	6. 6	84	7		1	4	1	3	100
VIII	11.3	85	6		1	1		7	100
VII	11.4	85	2	1	1			11	100
VI	7.4	88	4	<u>. </u>	2	<u> </u>		6	100
v	4.4	61	4		1	3	1	30	100
IV	5.7	90	3	2			_	5	100
III	9.2	73	3		1		•	23	100
II	4.9	9 0	2	1.	1			6	100
I	8.8	65	1		1	2	1	30	100
Seam Average	69.7	80.2	3.6	0.4	1.0	1.1	0.3	13.4	100

Maceral Content of the IX Petrographic Intervals of the Upper Seam, Bowron River Area

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Table I

The seam is composed almost entirely of the bright coal component vitrinite. This maceral is the major entity of all nine intervals reaching per cent a high of 90/ in interval IV, the brightest unit and dropping to a low of per cent 61/ in interval V, which is the dullest interval. The monotony of the column is broken by the three intervals I, III, and V which have higher amounts of mineral impurities. The mineral pyrite and various shale minerals account for the mineral matter. The other maceral components are present only in minor percentages.

The seam under study has been noted for its relatively high resin content. Black reports on two types of resin, namely soluble or "Canadian" resin and insoluble resin. The former is reported to compare favourably with "Congo" resin which has a well-established place in world markets. This resin was named "Canadian" by the Batelle Institute (Black 1967). This "Canaper cent dian" or soluble resin was reported to amount to 4/ in this coal. In contrast megascopically to the above "soluble resin" which is invisible,/the insoluble resin is visible and is said to be present in the coal in an amount equal to the "soluble resin" (approximately 4%).

During the visit to the mine to collect the sample studied, the visible resin was observed in the coal. The resin appeared as small (±1 inch), elongated,amber-yellow nodules randomly distributed throughout the seam.

The invisible, soluble resin was assumed to be so only in a megascopic examination, but would be visible during the microscopic phase. The resulting low percentages actually observed during the microscopic examinations therefore were disappointing.

A fragment of the visible resin, hand-picked from the seam, was examined in oil under reflected light and showed a distinct brownish redorange colour and therefore appeared as a readily identifiable component. During the subsequent analysis, none of this type was observed in the coal. The small percentages of resinite evident ranged from light to dark grey in colour.

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The discrepency between the high resin values mentioned in the report by Black and the paucity of resin found by the present analysis could be attributed to "vitrinization". Stach (1968) reports that the elliptical resin bodies can lose their resinous characters by polymerisation and may be transformed into grey vitrinite. Such resinous material in a petrological examination would have to be assessed as vitrinite or, more strictly, as semi-vitrinite. Thus it would appear that through this process of polymerisation the resinite loses its original optical properties and assumed the same colour as vitrinite. It is conceivable that in this process the resin loses some of its "exinitic" properties and takes on more vitrinitic characteristics.

COKE STABILITY

The present mine operators found that the coal near the top of the slope produced a coke button with a swelling index of 1 and that it increased as the mining proceeded down the dip of the seam.

per cent

Coke stability can be predicted with an accuracy of 94 / (Berry, et al.,1967) if the maceral content and the reflectance data of the seam are known. The method followed in the present study was that described by Schapiro, Gray and Eusner (1961) and the calculated stabilities for the petrographic intervals and a composite of the entire seam were compiled. The method considers the macerals as belonging to two groups, namely those which are reactive (vitrinite, exinite, resinite, 1/3 semifusinite) and those which are inert (fusinite, micrinite, 2/3's semifusinite, mineral matter). The method further assumes that for maximum coke strength there is an optimum ratio of reactives to inerts and that this ratio varies with rank.

The results of the stability determinations for the Bowron coal are shown in Table II.

Table II

Interval	I	II	III	'IV	v	VI	VII	VIII	IX	Seam Average
Stability	6	0	0	0	23	0	0	. 0	0	9

Predicted Coke Stabilities of the Petrographic Intervals and Seam Average of Upper Seam, Bowron River Area. These results show that all intervals with the exception of intervals I and V have a calculated predicted stability of 0. The remaining intervals, I and V have stabilities of 6 and 23 respectively. The predicted stability for the entire seam was 9 as compared with a stability of 50-60 for known coking coals.

The resulting low stability factor can be almost solely attributed to the lack of the so-called inert macerals such as the semifusinite, fusinite and micrinite.

per cent

As can be seen from figure 2, the seam is composed of almost 80/ of the reactive components vitrinite and exinite. These are the macerals that contribute the swelling properties to the coke. As previously stated, resinite behaves in a manner similar to exinite and this adds considerably to the swellper cent ing characteristics. Coal that contains 5/ resinite is considered a high resinper cent ous coal and therefore the 8/ reported by Black puts the seam under study in this category. This overabundance of swelling reactives would yield a favourable coke button but not necessarily a strong coke.

On the other hand, a coal that would be considered a good coking coal would have a predicted coke stability of from 50 to 60. The reason that this coal has such poor predicted stability is due to the paucity of the inert macerals which impart strength to the coke. The calculation of stabilities is based on the ratio of reactive entities which in general produce the swelling characteristics to the inert components which give the coke its strength. It is, therefore, unlikely that this coal would produce a good coke. The coal could, however, be used as a blend to mix with a low volatile coal that has a high into determine ert maceral content. Further study would be required / the suitability of this coal for blending.

SUMMARY AND CONCLUSIONS

The upper seam of the productive part of the coal measures in the Eowron River area is composed of a clean-looking coal with a very high lustre. The coal is dense and hard and contains small amber-yellow resin nodules dispersed through it.

- 11 -

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REPLACEMENT of a conventional screening step with a solids classifier using air as the separating medium results in savings of from 15 to 20¢ per ton in processing lignite at the tone, Calif., plant of American Lignite Products Co., Inc. This California lignite, much younger in geologic terms than that of the Dakotas or other interior fields, is a source of montan way, which is recovered from the lignite by solvent extraction.

Montan wax is the basic material for the production of a number of waxes, and is a substitute for carnauba and beeswax in making paper sizings and in polishes, paints, candles, soaps, pastes and so on. It is used also in production of plastics and resins, inks, waterproofing materials and as a binder in carbonpaper coatings having non-smudge characteristics.

The air classifier is a 3-ft Sturtevant separator. Its purpose in the ALPCO circuit is to isolate an δx (*Continued on p* $\delta 6$)

SIMPLE adjustment of external controls results in precise classification of fines at selected limits.

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The coal is a high volatile bituminous coal by A.S.T.M. standards and this was borne out by the mean maximum reflectance index of 0.65.

The coal has yielded a coke button with a free swelling index of 1 near the top of the slope and 3 at the face. The operators predict a button of 5 as they proceed down dip. This is not over-optimistic as the coal is composed mainly of those macerals that contribute volatiles and hence swelling of the coke. The lower indices of 1 and 3 obtained on the samples mentioned above may be due to oxidation of coal collected relatively near the outcrop.

This abundance of reactives is detrimental to the process of coke making with this coal since the inert macerals which give strength to coke are present in very small proportions. It is the lack of this inert component that gives a predicted coke stability of only 9 for the seam even though one of the established petrographic intervals gave a figure of 23. This was the highest figure obtained and, when weighed against the 50-60 stability index produced by good coking coals, indicates a coke of inferior quality. The coal possibly could be used as a blend with a coal which is low in reactive macerals.

There was little petrographic indication of the high (8%) resin content attributed to this coal. This lack could be attributed to the amount of bitrinization undergone by the resin or by the superiority of a chemical analysis to determine the resin content of this coal.



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076-1 Bethlehem DIAMOND DRILL HOLES, 1971

ESTIMATE OF DRILL-INDICATED COAL RESERVES

Per J. M. Black September, 1967

THICKNESS SHORT TONS 21' 2,626,000 29' 2,213,000 444,000 6.5' - 81 291,000 14' 246,000 129,000 19' 6,211,000 12,160,000

Additional Estin December, 1972,	nate by L. S. Tre based on revised	nholme interp	retation of fault	location	
8	•	9'		,512,000	н. Т
		TOTAL 1	ORILL INDICATED	13,672,000	
		Less:	10% Pinch-outs	1,367,000	
		NET INI	DICATED	12,305,000	:

This estimate does not include "Probable Reserves" in adjoining areas of widely-spaced drilling.

LEGEND

BY

BLOCK

Bearings Astronomic and derived from Stellar Observations at Station 7465

Co-ordinates are referred to Iron Post at S.E. Corner D.L. 9592 whose co-ordinates are 10,000 N; 10,000 E.

Elevations to Assumed Datum

Bench Mark : Bench Mark No. 1 Elev. 2400' Assumed Iron Post set shown thus in Traverse Hub Bench Mark (6"Spike intree carved (am.1). Diamond Drill Hole

River plotted from Interim Map

J-3959 FB 623

NORTHERN COAL MINES LTD. INTERCOAL RESOURCES LTD. GARRAWAY MINE PLAN OF DIAMOND DRILL HOLES . . . AND MINE WORKINGS SCALE: I INCH- 400 FEET DATE JUNE 21/67 DRAWN BY S.R.T. the second second UNDERHILL & UNDERHILL PROFESSIONAL ENGINEERS AND B.C. LAND SURVEYORS DRAWING NUMBER

1139-1