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by

J.F. Prescott*, P. Van Bostelen* and A.E. George**

ABSTRACT

A semi-continuous bench scale plant was constructed and commissioned. Samples of high volatile bituminous C coal, subbituminous coal and lignite were extracted with supercritical toluene at 400°C and 1500 psig. The yields of extracted liquids were 17.1%, 13.7% and 15.4% respectively on a dry weight basis. The non-caking free flowing chars remaining after extraction had higher calorific values on a dry basis than the coals from which they were derived. The calorific values of the chars from subbituminous coal were enhanced by 10.4% while that of the chars from lignite were enhanced by 15.7%.

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I. INTRODUCTION

The Department of Supply and Services awarded a contract to Raylo to investigate the feasibility of supercritical gas extraction of Canadian Coals.

A gas can be liquefied by increasing pressure if the temperature is below the critical temperature of the fluid. Liquefaction cannot take place if the temperature is above critical and the fluid is referred to as "supercritical gas". The gas extraction technique exploits the capability of a compressed supercritical gas to enhance the volatility of organic compounds. The technique is similar to both solvent extraction and distillation and could be considered as a combination of the two processes. The National Coal Board in Britain pioneered the application of the technique to the extraction of coal (1,2,3).

Supercritical gas extraction is particularly suitable for the recovery of liquids formed when coal is heated to about 400°C. The liquids formed are not sufficiently volatile to distil at this temperature. If the temperature is increased, these heavy liquids degrade by polymerization and by pyrolysis and only a small amount of tar can be distilled from the decomposing material. Supercritical gas extraction offers a means of recovering these liquids as they are formed without extensive thermal degradation. Gases which can be used as solvents should possess critical temperatures near the decomposition temperature of coal. Results obtained at the National Coal Board in Britain indicate that toluene was the solvent of choice for laboratory

investigation of supercritical gas extraction although less expensive solvents would be needed for commercial exploitation of the method.

Coal pyrolysis at about 400°C almost certainly leads to the formation of free radical species (4,5), which can undergo the normal free radical reactions such as hydrogen abstraction, disproportionation and coupling. In addition, coal contains resonance stabilized free radicals (6) which could initiate these radical reactions. Hydrogen abstraction will predominate in the presence of an excess of hydrogen transfer agent. Toluene can act as such an agent resulting in the formation of bibenzyl as a by-product. Using toluene to extract a South African coal at 450°C and 2465 psig (17 MPa), Kershaw reported that bibenzyl represented up to 31% of the total extract (7). However, he postulated that bibenzyl was formed by thermolysis of toluene via the benzyl free radical, implying that bibenzyl was formed by a process independent of coal pyrolysis. Bibenzyl formation was observed when toluene was heated to 350 or 400° at 2465 psig (8).

Bibenzyl has not been reported as a major product in other supercritical gas extractions. Bartle and co-workers reported it to be a minor component amounting to less than 1% of the total extract (3). Turkish workers subjecting two lignites to supercritical toluene extraction observed traces of bibenzyl in one instance but not in the other (9).

The wide range of bibenzyl formation (0 to 31% of the total extract) may be due to variability of the number of reactive free radicals formed during pyrolysis of the coal. The number of free radicals formed will probably be a function of a particular coal.

Bibenzyl production is probably an indication that benzyl groups from the solvent are also being incorporated into the liquids derived from the coal and possibly into the chars left after the extraction. The possibility does not seem to have been investigated but may be of economic importance. Other solvents, for example, tetralin, are known to be partially incorporated into the liquid fractions when coal is extracted under non-critical conditions. It is possible that the production of liquid products from coal involves some chemical reaction between the coal and the extracting solvent and this possibility will be borne in mind when assessing the experimental results.

This report summarizes the work carried out at Raylo covering the period from November 1979 to March 1980. The work was originally reported in Raylo Progress Reports numbers I through V.

III. WORK PLAN

The following work plan was discussed with Dr. A.E. George when he visited Raylo on November 15, 1979.

The objectives of the work plan are:

1. Design and construct a simple semi-continuous extraction system.
2. Extract samples of a specified coal and evaluate the effects of temperature, pressure, solvent and gas flow rate on the efficiency of extraction.
3. Define a standard set of parameters for extraction based on the experimental results.

4. Apply the procedure for screening various coals.

The literature found by a computer search is generally lacking in description of equipment and experimental details. Toluene seems to be the solvent of choice. Temperatures for extraction range from 350 to 400°C and are limited by the stability of coal. Pressures range from 100 to 200 atmospheres, well above the critical pressure of toluene (41.6 atm). Reasons for choosing these high pressure conditions are not expressly stated.

The plan for reaching the objectives is outlined below.

1. Figure 1 shows a block diagram of the process.
2. Experimental criteria:
 - a) Determine the mass balance.
 - b) Determine the carbon balance if necessary.
 - c) Define the degree of extraction.
3. Analysis:
 - a) Coal - proximate and ultimate analysis.
 - b) Solvent - determine purity by gas chromatography.
 - c) Recovered solvent - analyze by gas chromatography.
 - d) Off-gases - measure the volume and possibly determine the composition by gas chromatography.
 - e) Char - proximate analysis and pyridine solubility.
 - f) Extract - ultimate analysis, nmr and gel permeation chromatography.

4. Materials:

- a) All coals should be beneficiated. Subsequently, sources at Manalta Coal Ltd. and Luscar Coal Ltd., stated that low rank coals are not normally beneficiated. Raylo felt that it would be preferable to use coal samples which represent the actual output of the relevant mines for this experimental work. This was approved by Dr. George.
- b) Coals with high ash content are preferred as the ash may catalyze the pyrolysis.
- c) Order of preference of coals established with Dr. George.
 - i. A lignite.
 - ii. A subbituminous coal.
 - iii. A high volatile bituminous coal.

Samples of these coals were obtained from Luscar Coal Ltd. Typical proximate analyses are shown in Table I and the proximate analyses, calorific values and ultimate analyses of the actual samples are tabulated in Tables II and III. The analyses of the high volatile bituminous C coal and the lignite showed good agreement with the typical analyses while that of the subbituminous sample showed a lower ash content than the typical analysis.

- d) Order of preference for solvents established with Dr. George.
 - i. General Solvents:
 - Toluene for it is recognized as the most generally effective solvent.

ii. Special Purpose Solvents:

- Tetralin and decalin for their hydrogen donor properties. The critical temperature of tetralin (516°C) precludes its use by itself but it may be useful in conjunction with another solvent.
- Methyleneethyl ketone or methylisobutyl ketone for their excellent solvent properties.
- Isopropylalcohol for its polar nature (10).
- 90% p-cresol/10% water to be considered for later work for the good solvent capabilities of p-cresol.

Table IV lists the critical constants of some of these solvents.

5. Figure 2 shows the schematic of equipment.
6. Determination of optimum extraction conditions for a given solvent:
 - a) Initial conditions:
 - i. Dry heating of coal to extraction temperature.
 - ii. Heating in the presence of a small amount of solvent which will not reach critical pressure at the extraction temperature.
 - iii. Heating in the presence of sufficient solvent to give the desired operating pressure (above critical) when at the extraction temperature.

The second method (sub-critical) seems the most appropriate but comparison of the results obtained by the different methods could be useful.

b) Temperature:

The temperature can be varied from the critical temperature of the solvent to an upper limit of about 450°C with the pressure held constant slightly above the critical pressure.

c) Pressure:

The pressure can be varied from the critical pressure of the solvent up to about 2000 psi with the temperature held constant slightly above the critical temperature.

d) Flow Rate:

It is not known whether the rate of extraction of the solute from the coal is faster than or comparable with the rate of removal of the solute from the autoclave by the solvent flow. If the rate of extraction is fast, then the flow rate can be chosen to give effective removal of the solute in a convenient time. If the rate of extraction is slow, the quantity of solvent required will depend on the flow rate and there will be an optimum flow rate. For the purpose of this plan we have assumed a high rate of extraction.

7. Assessment of Results:

A considerable random error is expected to be present in the estimation of the variation of the degree of extraction with temperature, pressure and flow. It will be necessary to use

statistical methods to determine the interactions between the variables.

IV. CONSTRUCTION & COMMISSIONING OF THE EXPERIMENTAL APPARATUS

Figure 2 shows a schematic representation of the experimental apparatus. The solvent reservoir is a glass four litre vessel. The pump is a Bran and Lubbe Model NP-31 stainless steel metering pump with a maximum capacity of 7.08 litres per hour at 2680psig. The reaction vessel consists of a one litre Parr 316 stainless steel autoclave which has a working pressure of 7000 psig at 400°C. The catchpots are one litre 316 stainless steel jacketed autoclaves. In the actual experimental situation, it was found that only one catchpot was practical since adiabatic expansion of supercritical toluene (100 atmospheres to one atmosphere) resulted in enough cooling to condense the toluene.

The demister consists of a one foot by three-quarter inch O.D. stainless steel tube packed with stainless steel wire mesh. The condenser and recovered solvent reservoir are standard laboratory glassware items.

Other items such as valves (Sno-Trik - 40,000 psi service), fittings (Swagelok, Cajon and Sno-Trik) and pressure gauges are all 316 stainless steel. No provision has been made to measure the volume of gas evolved.

The temperature is controlled by a Thermoelectric on-off time-proportioning temperature controller operating through a solid

state relay. The temperature is monitored at various sites on the apparatus by a Thermoelectric 24 point temperature recorder.

Commissioning of the apparatus proceeded by testing the major components on an individual basis. The system was tested for leaks by pumping toluene through it at 25°C and 2000 psig. The rate of heating and the accuracy of the temperature readout was tested by heating a volume of degassed toluene to 350°C. Correlation of the internal temperature derived from the vapour pressure of toluene with the indicated temperature monitored at an external point on the autoclave revealed the necessity of measuring the internal autoclave temperature. Dr. J.F. Prescott machined a new autoclave head, from four inch 316 stainless steel bar, which has provision for a thermowell dipping into the interior of the autoclave. Two thermocouples can be accommodated in the thermowell so that the internal temperature can be accurately controlled and monitored. Excellent agreement was obtained between the measured internal temperature and the temperature calculated from the vapour pressure of degassed toluene. The system was then heated with toluene to 350°C at 2000 psig and to 400°C at 2000 psig. The flow rates of toluene at various micrometer settings of the feed pump were established at these temperatures and at pressures ranging from 1500 psig to 2000 psig. Flow rate was found to be independent of pressure and temperature.

V. EXTRACTION OF COAL

A. Coal Comminution

The coal samples provided by Luscar Coal Ltd., consisted of lumps of coal varying in size from pea size to ten inch lumps. The large lumps were manually broken down to two inch lumps. The individual samples were fed into a Fitzmill Model D Comminutor equipped with 16 hardened stainless steel knives rotating at 1460 rpm in an eight inch chamber. The exit from the chamber was fitted with an 0.125 inch screen ensuring that the maximum particle size was that dimension. About 50 kilograms of coal was comminuted in about 30 minutes. The particle size distribution for each coal appears in Table V.

B. Experimental Method

The autoclave is charged with 400 grams of 12-24 mesh coal. The apparatus is assembled and evacuated for 15 minutes using a water aspirator. Then 300 to 350 grams of toluene are introduced into the autoclave, the autoclave is isolated from the rest of the system by closing the inlet and control valve and the heater is turned on. As the autoclave heats up, the pressure increases at a faster rate than predicted from the increase in toluene vapour pressure. The pressure is allowed to increase to 2000 psig at which point it is controlled by operating the manual control valve. When the temperature reaches the operating temperature (350 or 400°C) the pump is started and the pressure is maintained at 1500 psig. The solvent enters the hot reactor by means of a dip tube reaching to the bottom of the autoclave.

This ensures that the entire coal bed is flushed with the supercritical vapour. The extraction is terminated when a fixed amount of toluene has passed through the coal bed. The amounts of char, water and organic extract are measured (Table VI).

C. Observations

Rapid pressure increases took place as the temperature passed 250°C in all the extractions. Venting was required to maintain the pressure near 1500 psig. The rapid increase is apparently due to the formation of gaseous material and dewatering of the coal. Odorous sulfur containing gases and water appear as products in the initial venting of the apparatus with water being the major component.

Extraction of high volatile bituminous C coal at 350°C led to the isolation of a large amount of solids which were collected by filtration (37% of the total organic extract). This material deposited in the tubing past the control valve indicating that it is soluble in supercritical toluene but not in hot toluene. The amount of solid isolated was much less when this coal was extracted at 400°C possibly indicating a different course of pyrolysis at the higher temperature. The amounts of solids isolated from each of the three samples of coal extracted at 400°C amounted to about 6% of the total organic extracts.

The bituminous coal was extracted at 350°C with interrupted toluene pumping during the extraction (Table VII and run 2, Table VI). The rate of extraction expressed in milligrams coal liquids per gram of toluene remained fairly constant for the first 1700 grams (3.7 litres

of supercritical toluene) passed through the coal bed. The extraction rate drops rapidly after that. About 80% of the total amount of organic material isolated was extracted by the first 3.7 litres of supercritical toluene. The total amount of liquids extracted increased significantly when compared with extraction under similar conditions of temperature and pressure but under different toluene flow conditions (compare runs 1 and 2, Table VI). The results probably indicate that coal pyrolysis is the limiting factor in the rate of extraction. Pyrolysis would be more extensive in run 2 because the coal was subjected to elevated temperatures for a longer time. The amount of liquids isolated in run 2 is the same as the amount of liquids isolated when the extraction was carried out at 400°C (run 3, Table VI).

D. Products

1) Volatile products - A number of gaseous products may be expected such as carbon monoxide and hydrogen sulfide (distinctive odour). Extraction of subbituminous coal led to the isolation of methyl sulfide in a cold trap. This material was identified by nuclear magnetic resonance spectroscopy (one peak at δ 2.15 - identical to that of the authentic material). No reliable estimate is available for the amount of volatile material released during extraction.

2) Water - The amount of water isolated closely approximates the amount of water present as indicated by proximate analysis.

3) Char - The proximate analysis and calorific value and the ultimate analysis of three chars derived from supercritical toluene extraction of the corresponding coals are tabulated in Tables VIII and

IX. The char obtained from high volatile bituminous C coal extracted at 350°C shows a decrease in moisture content, a proportionate increase in ash content, an increase in carbon content and a decrease in oxygen content compared with the original coal sample. All other parameters, including the percent volatile material, calorific value and hydrogen content, remained essentially the same.

The cumulative weight loss curves for the high volatile bituminous C coal and the char remaining after the supercritical toluene extraction at 350°C are shown in Figures 3 and 4. The coal lost 36.4% of its weight over the temperature range of 50 to 900°C. This figure compares well with the per cent volatile matter (35.6%) obtained from the proximate analysis for the same coal. The char lost 28.4% of its weight over the same temperature range. The proximate analysis is not available for this char. Maximum rate of weight loss occurred between 400 and 550°C for both samples with the coal losing 17.3% and the char losing 10.3% of its weight in this temperature range.

The chars remaining after extraction of lignite at 400°C had 43% less volatile matter compared with the starting sample and a 15.7% increase in calorific value (compare dry basis analysis). The char derived from subbituminous coal had 43% less volatile matter and a 10.4% increase in calorific value compared with the starting coal.

The ultimate analysis showed that there is an increase in the carbon content, decrease in hydrogen, sulfur and oxygen contents and no change in the nitrogen content of the chars derived from the lignite

compared with the initial sample. There are increases in carbon and nitrogen content and decreases in the hydrogen, sulfur and oxygen contents of the char derived from subbituminous coal compared with the starting sample.

The analyzed chars were derived from supercritical extraction carried out at 400°C for the lignite and subbituminous coal and from the extraction at 350°C for the high volatile bituminous coal. The latter temperature is below the temperature at which active thermal degradation occurs for this particular coal (Figure 3). This difference in extraction conditions may account for the observed differences in the analyses.

4) Extracted Products - Extraction of all the coal samples at 400°C and 1500 psig led to small amounts of solid materials which could be collected by filtration. The total yield of organic extracts obtained under the above conditions amounted to 17.1% for high volatile bituminous C, 15.4% for lignite and 13.7% for subbituminous coal on a dry weight basis (Table VI). This compares well with the results obtained by the British workers (1).

The liquids obtained have not been extensively analyzed. The nuclear magnetic resonance spectra were similar for all extracts with each showing aliphatic and aromatic protons in a ratio of three to one. Each had a sharp peak at δ 1.25 possibly indicative of methyl groups attached to tertiary or secondary carbons.

The nmr spectrum of the solids obtained from the extraction of high volatile bituminous C coal at 350°C showed a ratio of two to one

for aliphatic to aromatic protons.

VI. CONCLUSIONS

The results to date have been encouraging. Extraction with supercritical toluene has resulted in the conversion of up to 17% coal to liquids. In addition the low rank, low value coals were converted to water free chars exhibiting improved calorific values. The chars were non-caking, free flowing solids similar in appearance to the starting coal.

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TABLE I

Typical proximate analysis and calorific value for Luscar coal samples

Coal (a) Type	Coal (b) Prep	% Ash	% Moisture	% Volatile Matter	% Fixed Carbon	% Sulfur	Gross (c) Cal. Value
A	1	9.0	9.0	34.0	48.0	0.27	10,800
B	2	11.0	26.0	28.0	35.0	0.25	8,000
C	2	7.0	34.0	26.0	33.0	0.32	7,100

- a) A - Luscar Sterco, Coal Valley, Alberta - high volatile bituminous C:
 B - Diplomat, Forestburg, Alberta - subbituminous:
 C - Bienfait Mine, Bienfait, Saskatchewan - lignite.

- b) 1 - heavy media plant preparation - clean product:
 2 - screening tipple - no coal preparation.

c) Btu/lb.

TABLE II

Proximate analysis and calorific value for Luscar coal samples as determined by local laboratory.

Coal (a) Type	Determined (b) as	% Ash	% Moisture	% Volatile Matter	% Fixed Carbon	Calorific (c) Value
A	1	9.0	14.6	30.8	45.1	10,456
	2	11.2	8.3	32.6	47.9	11,020
	3	12.2	--	35.6	52.2	12,015
B	1	3.2	24.9	34.5	37.4	9,104
	2	3.6	13.3	39.8	43.3	10,510
	3	4.2	--	45.9	49.9	12,122
C	1	7.5	34.4	26.4	31.7	7,123
	2	9.5	17.6	33.1	39.8	8,947
	3	11.5	--	40.2	48.3	10,858

a) As Table I

b) 1 - sample as received: 2 - air dried sample: 3 - dry basis.

c) Btu/lb

TABLE III

Ultimate analysis of Luscar coal samples as determined by local laboratory

Coal (a) Type	Determined (b) as	% Moisture	% Ash	% C	% H	% N	% S	% O
A	1	14.62	8.94	59.77	3.86	0.88	0.38	11.55
	2	(8.28)	11.21	63.71	5.00	0.86	0.29	18.93
	3	--	12.22	69.46	4.44	0.94	0.32	12.62
	4	--	--	79.13	5.06	1.07	0.36	14.38
B	1	24.90	3.12	53.75	3.51	1.14	0.60	12.98
	2	(13.25)	3.60	62.09	5.54	1.32	0.69	26.76
	3	--	4.15	71.57	4.68	1.52	0.80	17.28
	4	--	--	74.67	4.88	1.59	0.83	18.03
C	1	34.39	7.58	42.87	2.65	0.83	0.52	11.16
	2	(17.5)	9.52	53.86	5.30	1.05	0.66	29.61
	3	--	11.55	65.34	4.04	1.27	0.80	17.00
	4	--	--	73.87	4.57	1.44	0.90	19.22

a) As Table I.

- b) 1 - sample as received, H and O do not include sample moisture (ASTM D-3180, Nov. 1974):
 2 - air dried sample:
 3 - dry basis, H and O do not include sample moisture:
 4 - dry ash free basis.

TABLE IV

Critical temperature and pressures of some compounds a)

Compound	Critical Temp ($^{\circ}\text{C}$)	Critical Pressure (atm)
<u>n</u> -Hexane	234.2	29.9
Isopropyl alcohol	235	47
Methyl ethyl ketone	262	41
<u>n</u> -Heptane	267.1	27
Cyclohexane	280.4	40
<u>n</u> -Octane	296	24.8
Toluene	320.6	41.6
<u>n</u> -Nonane	321	22.5
<u>n</u> -Decane	344.4	20.8
Water	374.1	218.3
<u>n</u> -Dodecane	386	17.9
Decalin (trans)	408	28.7
<u>p</u> -Cresol	431.4	50.8
Tetralin	516 b)	--

a) Values obtained from CRC Handbook of Chemistry and Physics, 55th Edition.

b) Dictionary of Organic Compounds, Eyre and Spottiswoode, London, 4th edition, Vol. 5, .2989.

TABLE V

Percentage size distribution of comminuted coal samples

Coal type (a)	Canadian Standard Mesh			
	+12	12 - 24	24 - 60	-60
A	7.5	39.0	38.8	14.7
B	22.8	44.5	24.9	7.8
C	25.9	39.1	22.5	12.5

a) As Table I.

TABLE VI

Supercritical toluene extraction of various coal samples.

Run	Extraction Conditions a)	Coal			Products			% Organic Extract			Material Balance (%)
		Type b)	Sample Wt (g)	Wt. Loss (g)	Char (g)	H ₂ O (g)	Organics (g)	As is Basis	dry Basis	daf Basis	
1	I	A	373	74	299	31 c)	40.6	10.9	11.9 c)	13.2 c)	96.8
2	II	A	400	98	302	33 c)	63.8	16.0	17.4 c)	19.3 c)	99.7
3	III	A	400	103	297	33 c)	62.9	15.7	17.1 c)	19.0 c)	98.2
4	III	B	400	209	191	135 d)	40.8	10.2	15.4 d)	17.4 c,d)	91.7
5	III	C	400	171	229	110 d)	39.7	9.9	13.7 d)	16.8 c,d)	94.7

a) I - heated to 350°C in presence of 390 g toluene, 36 g/min toluene flow rate, 3150 g toluene passed, 1500 psig.

II - heated to 350°C in presence of 350 g toluene, 36 g/min flow rate, 5740 g passed, 1500 psig flow intermitently interrupted (Table II).

III - heated to 400°C in presence of 350 g toluene, 36 g/min flow rate, 3150 g toluene passed, 1500 psig.

b) A - High volatile bituminous C; B - lignite; C - subbituminous.

c) Calculated from proximate analyses of coal sample used.

d) Measured moisture.

TABLE VII

Interrupted supercritical toluene extraction of high volatile bituminous C coal at 350°C and 1500 psig.

Fraction	Toluene Passed		Material extracted			Interval (min) d)
	g	l a)	g	rate b)	% c)	
1	704	1.5	24.1e)	34.2e)	37.8e)	96f)
2	595	1.3	12.3	20.7	19.5	11.5
3	433	0.9	13.9	32.1	21.8	28.5
4	866	1.8	5.5	6.4	8.6	30
5	866	1.8	3.3	3.8	6.0	30
6	1406	3.0	2.5	1.8	3.9	27
7	866	1.8	2.2	2.5	3.4	27
Totals	5736	12.1	63.8	--	100.8	--

a) Volume of toluene estimated from compressibility factors.

b) Mg extract/g toluene

c) % of total coal derived liquids.

d) Interval between pumping

e) Includes material collected in initial venting.

f) Time required to increase temperature from 20 to 350°C.

TABLE VIII

Proximate analyses and calorific value of chars remaining after supercritical toluene extraction.

Char from	Deter'd as a)	Moisture (%) e)	Ash (%)	Volatile Matter (%)	Fixed Carbon (%)	Calorific Value b)
High volatile c) Bituminous	1	1.7	13.8	33.6	50.9	11,876
	2	---	14.0	34.2	51.8	12,081
Subbituminous d)	1	1.3	6.8	25.9	66.0	13,206
	2	---	6.9	26.2	66.9	13,380
Lignite d)	1	3.7	14.5	22.1	59.7	12,092
	2	---	15.1	22.9	62.0	12,559

a) 1 - Air dried sample; 2 - dry basis.

b) Btu/lb.

c) Extracted at 350°C and 1500 psig.

d) Extracted at 400°C and 1500 psig.

e) "Moisture Analysis" is determined by sample weight loss on drying and may not represent moisture in these analyses.

TABLE IX

Ultimate analyses of chars remaining after supercritical toluene extraction.

Char from	Deter'd as a)	Moisture (%)	Ash (%)	C (%)	H (%)	N (%)	S (%)	O (%)
High volatile b) Bituminous	1	(1.70)	13.82	69.40	4.30	0.91	0.24	11.33
	2	--	14.06	70.60	4.18	0.93	0.24	9.99
	3	--	--	82.19	4.87	1.08	0.31	11.63
Subbituminous c)	1	(1.30)	6.81	78.45	4.03	1.67	0.56	8.48
	2	--	6.90	79.48	3.93	1.69	0.57	7.43
	3	--	--	85.37	4.22	1.82	0.61	7.98
Lignite c)	1	(3.75)	14.52	72.43	3.77	1.20	0.54	7.54
	2	--	15.08	75.23	3.48	1.25	0.56	4.40
	3	--	--	88.59	4.10	1.47	0.66	5.18

a) 1 - Air dried sample; 2 - Dry basis; H and O do not include sample moisture (ASTM D-3180, Nov. 1974); 3 - Dry ash free basis.

b) Extracted at 350°C and 1500 psig.

c) Extracted at 400°C and 1500 psig.

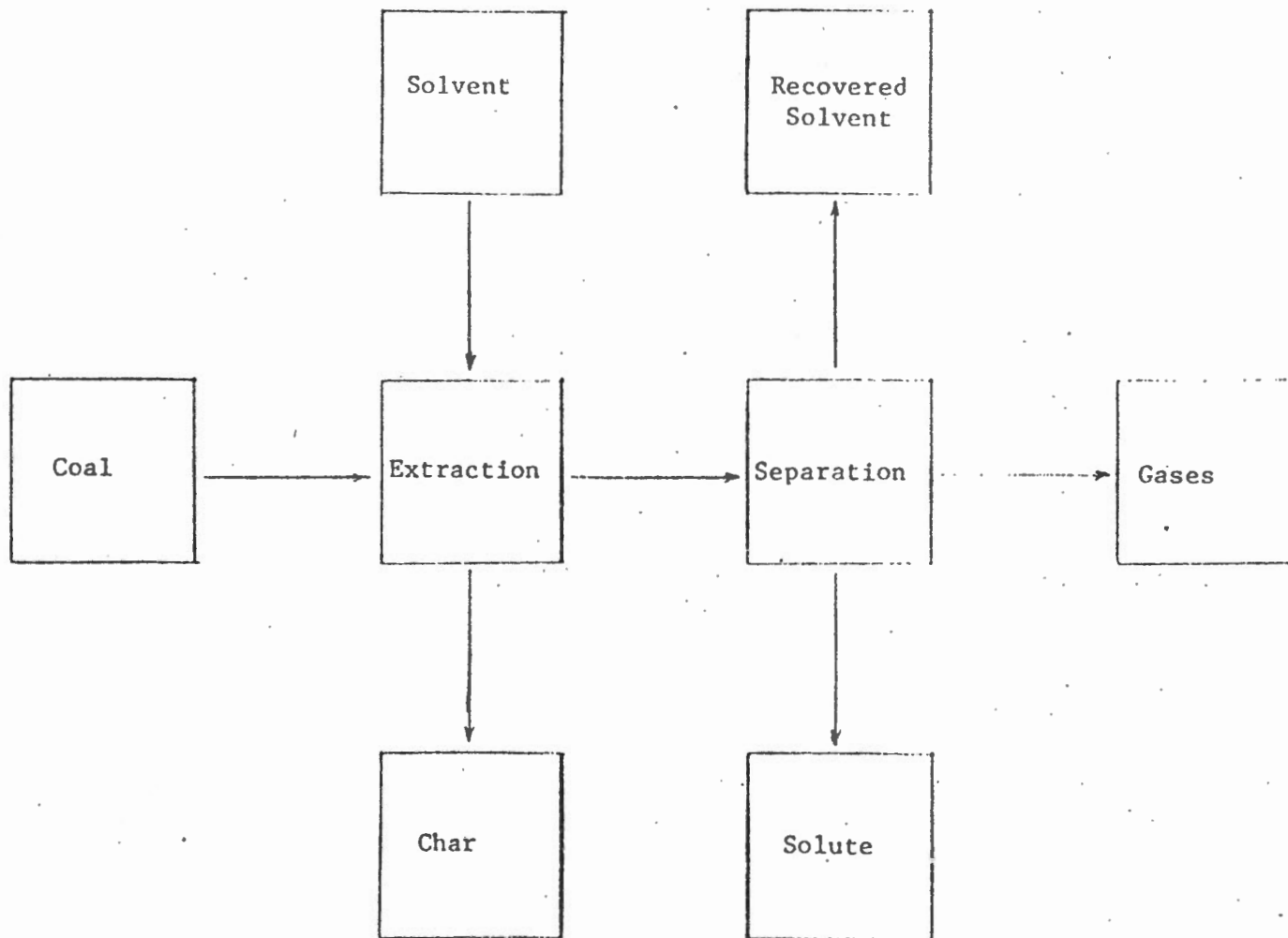


Figure 1: Process flow sheet for supercritical gas extraction of coal.

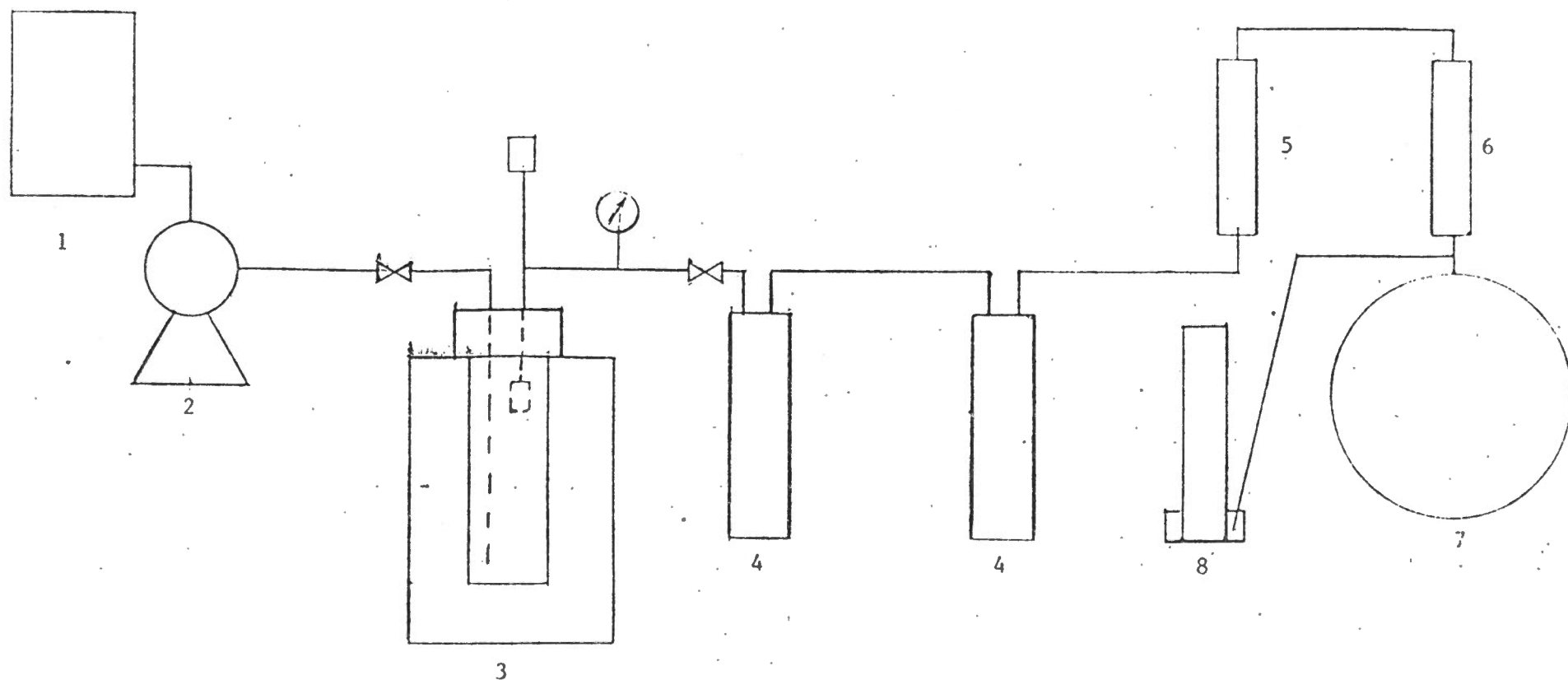


Figure 2: Equipment flow sheet for supercritical gas extraction of coal.
 1. Solvent reservoir; 2. Pump; 3. Autoclave and heater; 4. Catch pots; 5. Demister;
 6. Condenser; 7. Recovered solvent; 8. Gas meter.

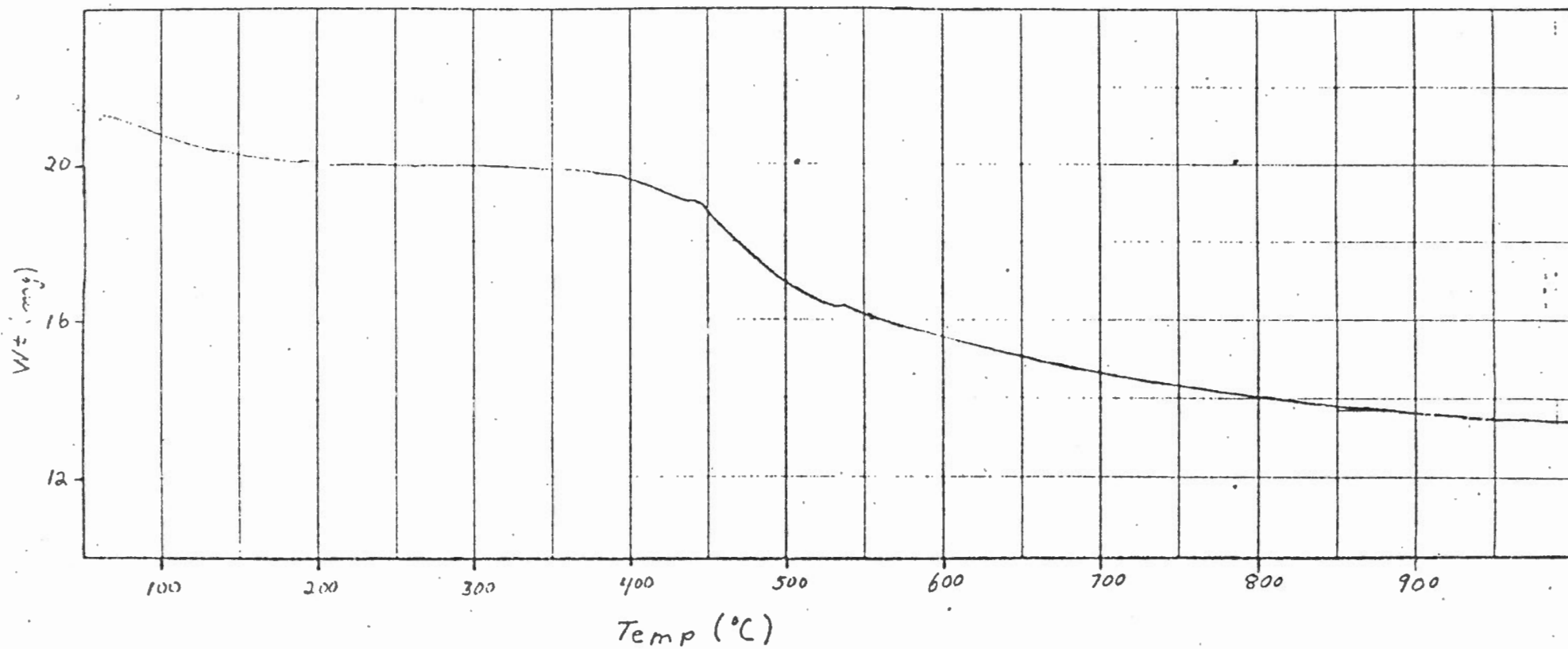


Figure 3: Cumulative weight loss curve for high volatile bituminous C coal. Heating rate 10°C/min.

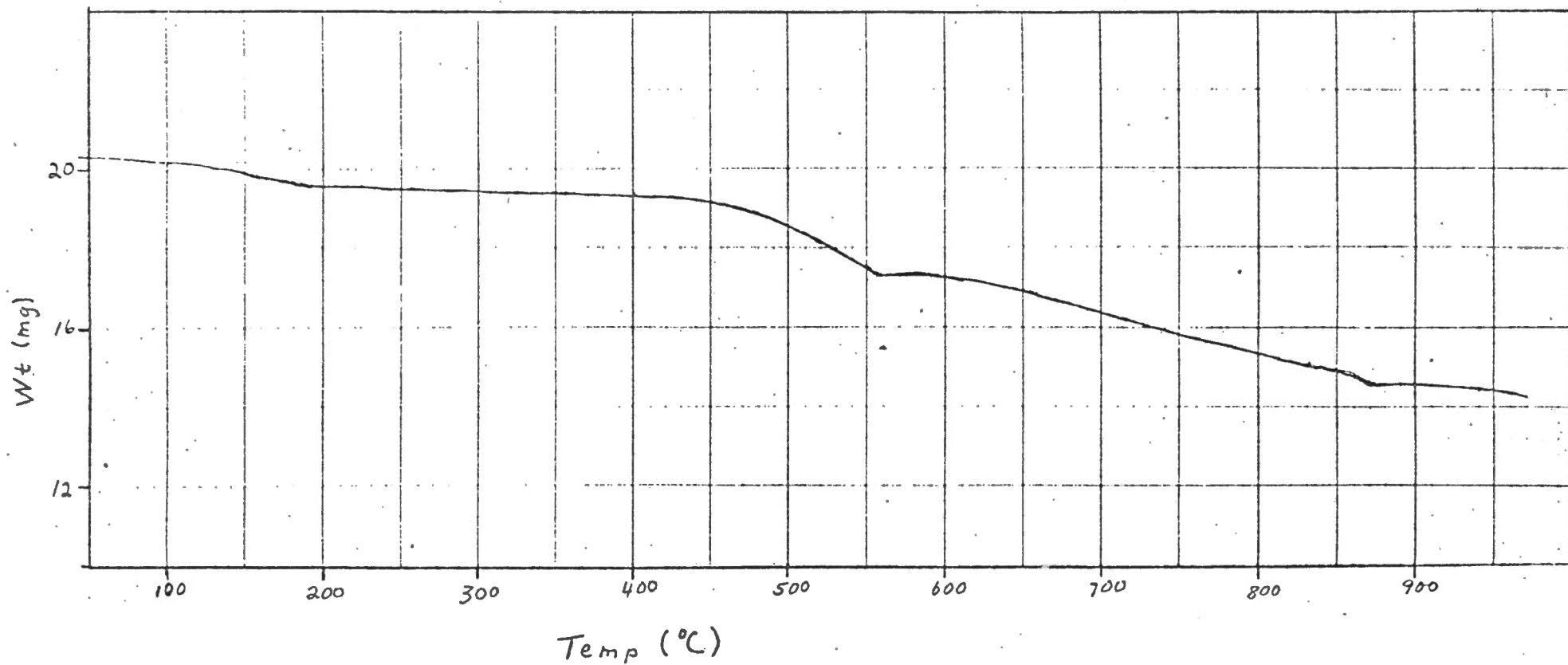


Figure 4: Cumulative weight loss curve for char remaining after supercritical toluene extraction of high volatile bituminous C coal. Heating rate 5°C/min.

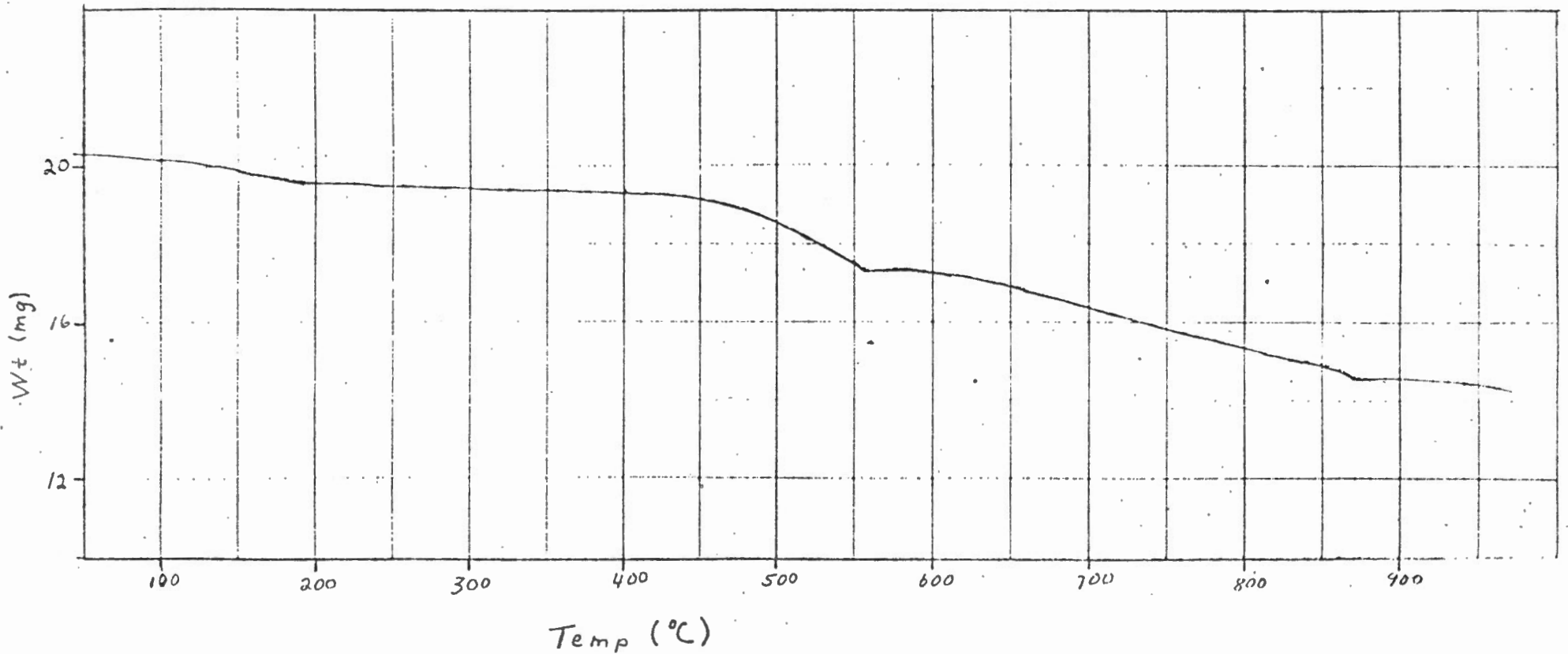


Figure 4: Cumulative weight loss curve for char remaining after supercritical toluene extraction of high volatile bituminous C coal. Heating rate 50°C/min.