



Extraction of Colstrip coal using supersolvents in conjunction with the water-gas shift reaction  
by Sylvester John Losinski

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in  
Chemical Engineering  
Montana State University  
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**Abstract:**

Coal extraction using supersolvents, tetramethylurea (TMU) and hexamethylphosphoramide (HMPA), in the presence of the water-gas shift reaction may be a viable method for hydrogenating and extracting coal products in one operation. In this investigation the effects of solvent mixture, solvent pH, temperature, and carbon monoxide pressure on coal extraction were evaluated. Experiments were conducted on Colstrip sub-bituminous coal and other coals in a magnetically stirred batch autoclave with carbon monoxide pressures ranging from 250 to 1000 psig.

Extraction of Colstrip coal to 35 percent was observed at 1607deg;C using a solvent mixture of TMU/HMPA/Tetralin/H<sub>2</sub>O with a pH of 13.12 and a carbon monoxide pressure of 500 psig.

It was found that extraction decreased with increasing carbon monoxide pressure in the range of 250 to 1000 psig, but the extraction value for any pressure was greater than when carbon monoxide was not used.

Extraction by the present procedure was not found to be temperature dependent over the range of 25 to 200°C. Possible explanations for this unexpected result have been presented.

Solvent was retained in the extraction residue in the amount of 6 to 17 percent by weight. The total extraction must be dramatically increased, or the amount of solvent retention decreased, for the process to be viable as a coal extraction method.

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MONTANA STATE UNIVERSITY  
Bozeman, Montana

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Approval

of a thesis submitted by

Sylvester John Losinski

This thesis has been read by each member of the author's graduate committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

March 5, 1986  
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## ABSTRACT

Coal extraction using supersolvents, tetramethylurea (TMU) and hexamethylphosphoramide (HMPA), in the presence of the water-gas shift reaction may be a viable method for hydrogenating and extracting coal products in one operation. In this investigation the effects of solvent mixture, solvent pH, temperature, and carbon monoxide pressure on coal extraction were evaluated. Experiments were conducted on Colstrip sub-bituminous coal and other coals in a magnetically stirred batch autoclave with carbon monoxide pressures ranging from 250 to 1000 psig.

Extraction of Colstrip coal to 35 percent was observed at 160°C using a solvent mixture of TMU/HMPA/Tetralin/H<sub>2</sub>O with a pH of 13.12 and a carbon monoxide pressure of 500 psig.

It was found that extraction decreased with increasing carbon monoxide pressure in the range of 250 to 1000 psig, but the extraction value for any pressure was greater than when carbon monoxide was not used.

Extraction by the present procedure was not found to be temperature dependent over the range of 25 to 200°C. Possible explanations for this unexpected result have been presented.

Solvent was retained in the extraction residue in the amount of 6 to 17 percent by weight. The total extraction must be dramatically increased, or the amount of solvent retention decreased, for the process to be viable as a coal extraction method.

## INTRODUCTION

Sears (1) has suggested a novel approach to the liquefaction of coal using a specific group of chemicals that he called supersolvents. In preliminary experiments, at room temperature and pressure, he was able to show that solvents of the general formula



exhibit unusually high extraction characteristics for certain types of coal. Based upon this evidence he submitted a proposal to the Department of Energy to investigate the feasibility of using a supersolvent extraction process for the liquefaction of coal. The ensuing research grant has provided the funding for this investigation and other related work.

Co-investigators T. Ward (2) and C. Ichioka (3) examined the effectiveness of tetramethylurea, hexamethylphosphoramide, and other supersolvents on the extraction of coal. Their efforts revealed that the blending of supersolvents resulted in better extraction of coal than could be obtained by any one supersolvent alone. They were also able to document that relatively large amounts of solvent were retained in the coal residue.

T. Sahin, another co-investigator, performed several experiments in a packed bed reactor with hydrogen overpressure in an effort to determine if enhanced extraction of coal could be achieved by hydrogenation of coal in the presence of supersolvents. His efforts were thwarted by materials handling and coal swelling problems, but he was able to tentatively conclude that hydrogen overpressure was ineffective in enhancing the extraction of coal.

Other investigators have documented the high reactivity of the nascent hydrogen formed in the water-gas shift reaction (4,5,6). Sears (7) postulated that enhanced extraction of coal may be possible by using the water-gas shift reaction in conjunction with supersolvent extraction. This process would have a distinct advantage over other extraction processes in that both the extraction and hydrogenation of the coal could be accomplished in one operation.

Whitehurst et al. (8) identified several factors that should be considered when evaluating a new or improved coal-liquefaction technology. The most important of these factors are:

- △ hydrogen utilization efficiency
- △ solvent overhydrogenation
- △ generation of hydrogen-rich final products
- △ ease of solids separations
- △ reduction of reaction time
- △ reduction of process temperature

Preliminary experimental work performed by Sears (1) indicates that the supersolvent coal-liquefaction process may have significant advantages

over existing coal-liquefaction processes based on the factors identified by Whitehurst.

The purpose of this investigation was to examine the extraction of coal using supersolvents in conjunction with the water-gas shift reaction. The effects of reaction parameters such as temperature, pressure, solvent, and pH of the solvent were to be characterized.

### Coal Structure

Coal deposits were formed by the action of pressure and heat on plant matter over long periods of time. During the coalification process progressive elimination of oxygen, primarily in the form of carbon dioxide and water, leads to coals of higher rank (9). On a geologic timescale plant matter is first converted to peat and then to lignite, sub-bituminous, bituminous, and finally anthracite coal. Several excellent references are available on the origin and formation of coal (9,10,11).

It has been found that the morphological nature of coal is very complex. Microscopic examination of coal reveals that it is not homogeneous, but instead is a mixture of several different identifiable macerals or lithotypes. Each lithotype exhibits different physical and chemical properties. Some of the lithotypes are primarily inert mineral matter, while others contain high quantities of extractable material.

The chemical makeup of coal has been studied by many scientists (12,13,14). Several persons have proposed structures, incorporating experimentally observed functional groups, that portray an "average" coal molecule. The structure for a bituminous coal proposed by Shinn

(15) is shown in Figure 1. As can be seen from the model structure, coal is a highly crosslinked aromatic compound with a large number of oxygen-containing functional groups. Most of the nitrogen in the coal molecule is tied up in the aromatic rings and as such is probably immune to low severity liquefaction reactions. There are several relatively weak bonds in the coal structure, primarily ester and ether bonds, and it is thought that liquefaction reactions are initiated at these sites (16).

#### Solvent Theory

Several approaches have been presented to explain the correlation between solvent characteristics and their extractive properties on coal. Most solvent theory approaches have originated from one of two dissolution models: those of Dryden and van Krevelen (17,18).

Dryden (17) asserts that coal consists of 'micelles' that can be assumed to be rigid and relatively indestructible. Coal extraction solvents act by swelling the micelle network so that smaller molecular units contained within the micelle may diffuse through the newly enlarged pores. A suitable solvent must therefore swell the coal structure and stabilize the coal fragments that are transported to the surface via osmotic diffusion.

Van Krevelen (18) contends that coal can be viewed as a cross-linked polymer and hence the principles of polymer theory apply. He believes that the amount of extractable material dissolved by a solvent is determined by that solvent's solubility parameter,  $\delta^2_0 = \delta^2_D + \delta^2_P + \delta^2_H$ , a thermodynamic property, and the solubility

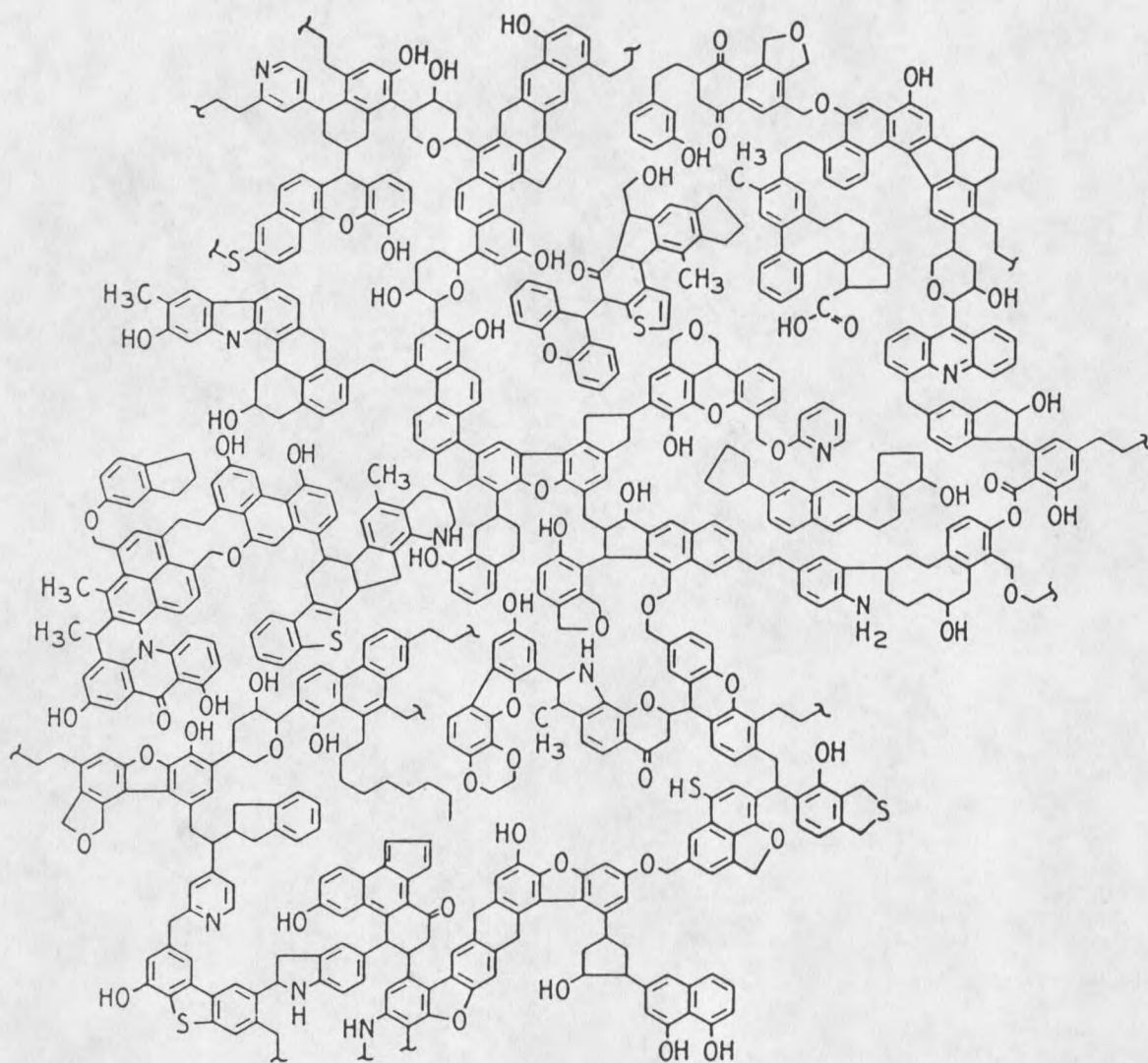


Figure 1. Shinn's Model of Bituminous Coal Structure

parameter of the coal. The overall solubility parameter is comprised of a dispersion component, a polar component, and a hydrogen bonding component. Maximum solubility of the coal is obtained when the solubility parameters of the coal and solvent are equal.

It is possible that Hombach (19) has made a significant contribution to the work of van Krevelin by suggesting that the overall solubility parameter may be adjusted by using a mixture of solvents. By using a mixture of solvents it would then be possible to tailor the solubility parameter of the solvent to that of the target coal.

Ward (2) has suggested that the overall solubility parameter may not be the ideal criteria for selection of an extraction solvent. He has presented evidence that individual solubility parameters, functions of the overall solubility parameter, should be tailored so that they match the individual solubility parameters of the target coal.

#### TMU and HMPA

Hexamethylphosphoramide, HMPA, and tetramethylurea, TMU, are the solvents of interest in this investigation. HMPA and TMU are members of a class of solvents called dipolar aprotic solvents. The structures of each are shown in Figure 2 and some of their physical properties are listed in Table 1.

HMPA is a colorless mobile liquid, which is miscible with water in all proportions. It is also miscible with many polar and nonpolar organic solvents, but not with saturated hydrocarbons (20). HMPA is prepared by reacting phosphorous oxide trichloride with an excess of



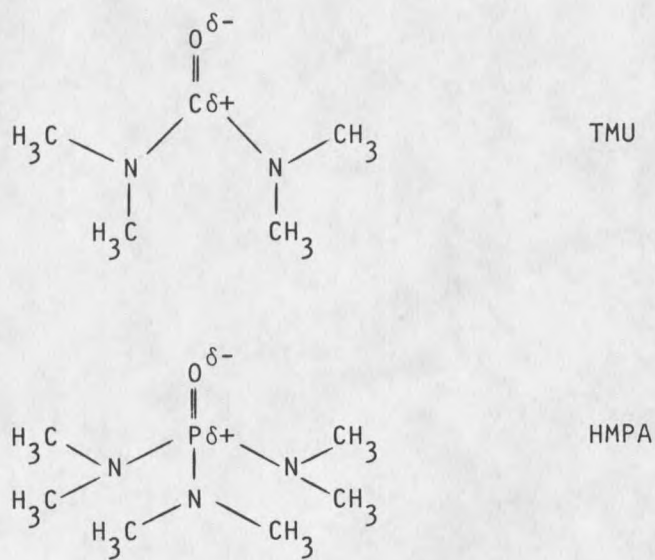
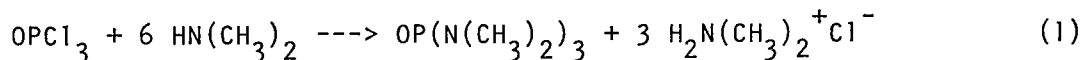


Figure 2. Structure of TMU and HMPA

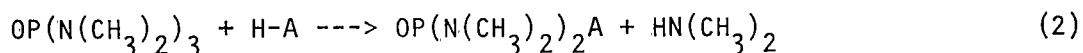
Table 1. Physical Properties of TMU and HMPA

Physical Properties	TMU	HMPA
Molecular Weight	116.16	179.20
Boiling Point ( $^{\circ}\text{C}$ )(1 atm)	176.5	235
Melting Point ( $^{\circ}\text{C}$ )	-1.2	7.20
Density (g/ml)(25 $^{\circ}\text{C}$ )	0.9619	1.0253
Dielectric Constant	23.45	30.00
Dipole Moment (debye)	3.37	5.37

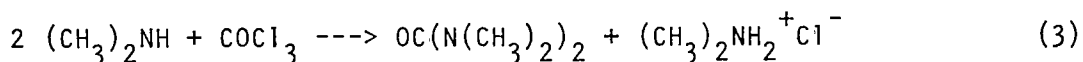
dimethylamine. The stoichiometry of the reaction is shown in Equation (1).



The chemical properties of HMPA are primarily due to the partial ionic character and polarity of the molecule. A high electron density, partial negative charge, is located around the oxygen atom, and a partial positive charge is distributed over the nitrogen and phosphorous atoms. The charge separation and unhindered electron cloud about the oxygen atom make HMPA an excellent hydrogen bond acceptor and a poor hydrogen bond donor. HMPA is very stable at temperatures below its normal boiling under basic conditions, but can readily be attacked by Lewis or Bronstead acids. The products of acidic attack are shown in Equation (2).



TMU is also a colorless liquid that is miscible with all common organic solvents as well as with water. TMU is prepared by reacting phosgene and dimethylamine as illustrated in Equation (3).



TMU is stable at temperatures below its normal boiling point under most conditions. Luttringhaus (21) has documented the protonation of TMU under severe acidic conditions, but contends that TMU is an excellent reaction medium for many conditions and is very unreactive while being an excellent solvent.

The author refers the reader to the works of Normant (20) for exhaustive information concerning the chemical properties of HMPA and to Luttringhaus (21) for the properties of TMU.

The physical and chemical properties of HMPA and TMU are similar in many ways. Some of the most notable similarities are:

- ▲ Both are miscible with water in all proportions.
- ▲ Both solubilize a wide range of organic compounds, particularly aromatic compounds.
- ▲ Both molecules have an oxygen atom that carries a negative charge and a positive charge that is delocalized by resonance over a central atom and nitrogen atoms.
- ▲ Both have high dielectric constants and large dipole moments.
- ▲ Both are liquid at room temperature and have a wide liquid-state temperature range.

A word of caution is probably in order concerning the handling of TMU and HMPA. The pathologic consequences of exposure to these chemicals have not been very well documented. It is speculated that HMPA in particular may be a skin-contact carcinogen.

#### Water-Gas Shift Reaction

Several scientists have documented the enhanced reactivity of the nascent hydrogen formed from the water-gas shift reaction over elemental hydrogen in the hydrogenation of coal and coal derived products (22,23, 24). This process has since been named 'Costeam.' The water-gas shift reaction involves the reaction of carbon monoxide and steam to produce hydrogen and carbon dioxide and derived its name from its industrial use

to increase the hydrogen content of water gas produced from the reaction of steam with hot coke or coal. The stoichiometry of the reaction is shown in Equation (4).



For the conditions involved in this experimentation, low temperature and low to moderate pressure, the thermodynamic equilibrium of this reaction lies far to the right as written in Equation (4). The reader is referred to Smith and Van Ness (25) for additional information regarding the thermodynamics of the water-gas shift reaction.

The mechanism of the water-gas shift is not very well delineated and there is some controversy over this matter. There are several catalysts, homogeneous and nonhomogeneous, that are known to catalyze the reaction. The alkali catalyzed water-gas shift mechanism proposed by Elliot (26) is shown in Figure (3). Because almost all of the experimentation of this investigation was conducted under alkaline conditions, it is believed that this mechanism may be of importance to the present investigation.

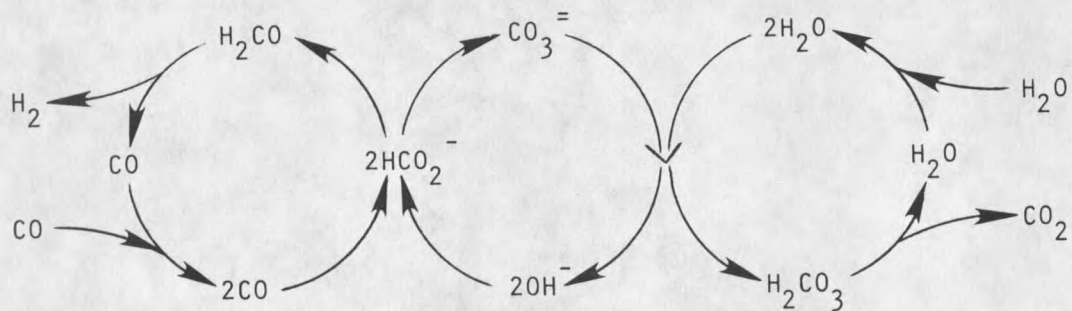


Figure 3. Proposed Mechanism of Aqueous Alkali Catalyzed Water-Gas Shift Conversion.

### Related Work

To the author's knowledge, prior to this work no one has attempted to extract coal with a strongly reactive solvent in conjunction with the water-gas shift reaction. The solubility of water in TMU and HMPA makes possible the contact of coal with both water and a good coal solvent in a single phase. A great deal of work has been performed on the solvent extraction of coal and a good deal of work has been performed on the hydrogenation of coal using the water-gas shift reaction, but the combined effects of using both at one time have not been investigated.

As discussed earlier in this thesis, Dryden (17) and van Krevelen (18) have formulated many of the currently accepted theories regarding the solvent extraction of coal. The reader is referred to their works for a detailed study of solvent extraction of coal.

Work performed by scientists subsequent to that of Dryden and van Krevelen have contributed significantly to the understanding of the solvent extraction of coal. Hombach (19) was able to demonstrate that a correlation existed for the dissolution of coal between the solubility parameter of the coal and the solubility parameter of the solvent. Using solubility parameter theory, he was able to explain the long-standing observation that bituminous coals generally exhibited the largest degree of dissolution for most of the good coal extraction solvents. Good coal extraction solvents have solubility parameters close to those of bituminous coals. Hombach was also the first person to demonstrate that a mixture of poor solvents for coal can act as a better solvent when chosen with respect to the solubility parameter.

The work of Ward (2) and Ichioka (3) substantiate that of Hombach in that they, too, were able to observe enhanced dissolution of coal by using a mixture of appropriately chosen solvents. Ward suggests that individual solubility parameters instead of the overall solubility parameter should be the criteria for engineering the ideal coal extraction solvent. He contends that individual solubility parameters of the solvent mixture should match the individual solubility parameters of the target coal.

T. Sahin, a co-worker, unsuccessfully tried to hydrogenate coal in the presence of TMU and HMPA using a packed bed reactor and hydrogen overpressure. He was thwarted by materials handling problems. Coal swelling, which Dryden (17) identified as the first step of the extraction process, caused the packed bed reactor to plug up and virtually no solvent could permeate the coal plug. Sahin determined that some other coal-solvent contact method must be incorporated if these materials-handling problems were to be eliminated.

Many scientists have studied hydrogenation of coal using the water-gas shift reaction. Fischer (27) is credited with first documenting the use of carbon monoxide and water for the hydrogenation of coal. Later, Appell (28) initiated a substantial research effort to study the solubilization of low rank coal with carbon monoxide and water. The work of Appell is often quoted in current water-gas shift reaction literature on coal hydrogenation.

The work of Appell et al. on coal hydrogenation is far too extensive to be covered in any detail in this thesis, but some observations he has made should be presented. For coal hydrogenation

using the water-gas shift reaction with carbon monoxide overpressures of 4500 psi and reaction temperatures in the range of 375-400°C, Appell has documented the following facts:

- △ Conversion of coal to solvent-soluble products is greatest for low rank coals.
- △ Conversion increases with carbon monoxide overpressure up to 1000 psi and then increases only slightly with additional pressure.
- △ Conversion increases with increasing temperature.
- △ Conversion is twice as great with carbon monoxide overpressure as with hydrogen overpressure.
- △ Exposure of coal to air causes conversion to drop.

From Appell's observations he has concluded that the water-gas shift reaction may inhibit condensation reactions that lead to insoluble products. He has also concluded that the most significant contribution to the solubilization of low rank coals is probably due to the activated hydrogen species formed by the water-gas shift reaction.

Ross et al. (23) have also performed experimentation on the hydrogenation of coal using the water-gas shift reaction. Ross's investigations have revealed some important facts not documented by Appell. The most pertinent of these facts relative to this investigation are:

- △ There is a dramatic increase in the solvent-soluble fraction of coal when the pH of the water for the water-gas shift reaction is greater than 12.6.
- △ Basic water solutions alone do not enhance solubility.

△ The use of hydrogen instead of carbon monoxide under basic conditions does not promote increased solubility.

Ross has suggested that the formate ion may act as a catalyst for the hydrogenation of coal under water-gas shift conditions.

Takemura and Ouchi (24) have investigated the catalytic liquefaction of coal using carbon monoxide and water in the presence of a cobalt-molybdenum catalyst. The results of their study roughly parallel those of Appell and Ross. Takemura and Ouchi have proposed that hydrogenolysis of the coal occurs by three routes: hydrogenolysis by the nascent hydrogen, solvolytic reaction with water, and stabilization of coal fragments in a vehicle oil.

Hydrogen can be formed in-situ by methods other than the water-gas shift reaction. Mondragon et al. (27) have studied the hydrogenation of coal using the zinc-water-coal reaction. Mondragon has documented that this method works best with low rank coals and works better at higher temperatures. These results are consistent with observations made by previously mentioned investigators working with the water-gas shift reaction and lend credible evidence to the speculation that the nascent hydrogen species is the key to enhanced coal dissolution.

Several studies have been made that attempt to characterize the reactivity of the nascent hydrogen formed in the water-gas shift reaction on coal by using model compounds (27,28,29,30). The reader is referred to these studies for information regarding the model compounds and the reaction products identified.



Research Objective

The objective of this research was to evaluate the action of the dipolar aprotic solvents TMU and HMPA under water-gas shift conditions on the extraction of coal. The total extraction of the coal observed was to be correlated with the experimental reaction variables of temperature, carbon monoxide overpressure, solvent blend, and pH of the initial solvent. Additionally, the quantity of solvent retained in the residue after extraction was to be evaluated. Designed to be a preliminary study, this research was not intended to be exhaustive in characterizing each reaction variable, but to determine the general extraction trends associated with each variable. The viability of this process and identification of promising research-work directions were to be evaluated.

## EXPERIMENTAL

Raw Materials and Chemical Reagents

Colstrip sub-bituminous coal was used almost exclusively in the work performed in this investigation. The coal sample was obtained from the crushing mill of Western Energy Corporation located in Colstrip, Montana. As received, the coal consisted of particles ranging in diameter from about one inch to approximately 60 mesh. While in transit, the coal was stored under atmospheric conditions. When received at Montana State University, the sample was placed under nitrogen to prevent any further oxidation. The time that the coal sample was exposed to the atmosphere is estimated to be about one week.

Savage lignite and Bakerstown coal were used on a limited basis in this investigation. The Savage lignite sample was obtained from a fellow student at Montana State University. It was stored under atmospheric conditions, and it is not known how much time had elapsed since the sample was mined. The Bakerstown coal was obtained directly from the mine in Preston County, West Virginia, and was stored under atmospheric conditions while in transit.

All of the coal samples were sized for experimental use using the same method. Each sample was loaded into a two-liter porcelain ball mill while under a nitrogen atmosphere. After the ball mill had operated for about 24 hours the sample was removed, while under a nitrogen atmosphere, and sized using standard Tyler screens. Only sample material

ranging in size from 60 to 100 mesh was retained. The sized sample material was stored under a nitrogen atmosphere until used for experimentation.

The manufacturer and purity of the solvents, reagents, and gases used in this investigation are shown in Table 2. No additional testing was performed to confirm the purity of these chemicals.

Table 2. Chemical Reagents Used

Chemical	Source	Purity
TMU	Aldrich Chemical	99%
HMPA	Aldrich Chemical	99%
Tetraline	Aldrich Chemical	99%
Sodium Hydroxide	Chemical Stockroom	Unknown
Hydrochloric Acid	Chemical Stockroom	Unknown
Acetone	VanWaters & Rogers	99.5%
Carbon Monoxide	Airco	99.9%
Nitrogen	Liquid Air Corp.	98.6%

#### Experimental Apparatus

The apparatus used in this experimentation was a modified magnetically stirred catalytic reactor manufactured by Autoclave Engineers. A detailed drawing of the reactor vessel is shown in Figure 4, and the pertinent mechanical characteristics are listed in Table 3. An additional agitation vane was added to the reactor to enhance the turbulence of mixing within the reactor. A flow scheme for the reactants in the apparatus is shown in Figure 5.

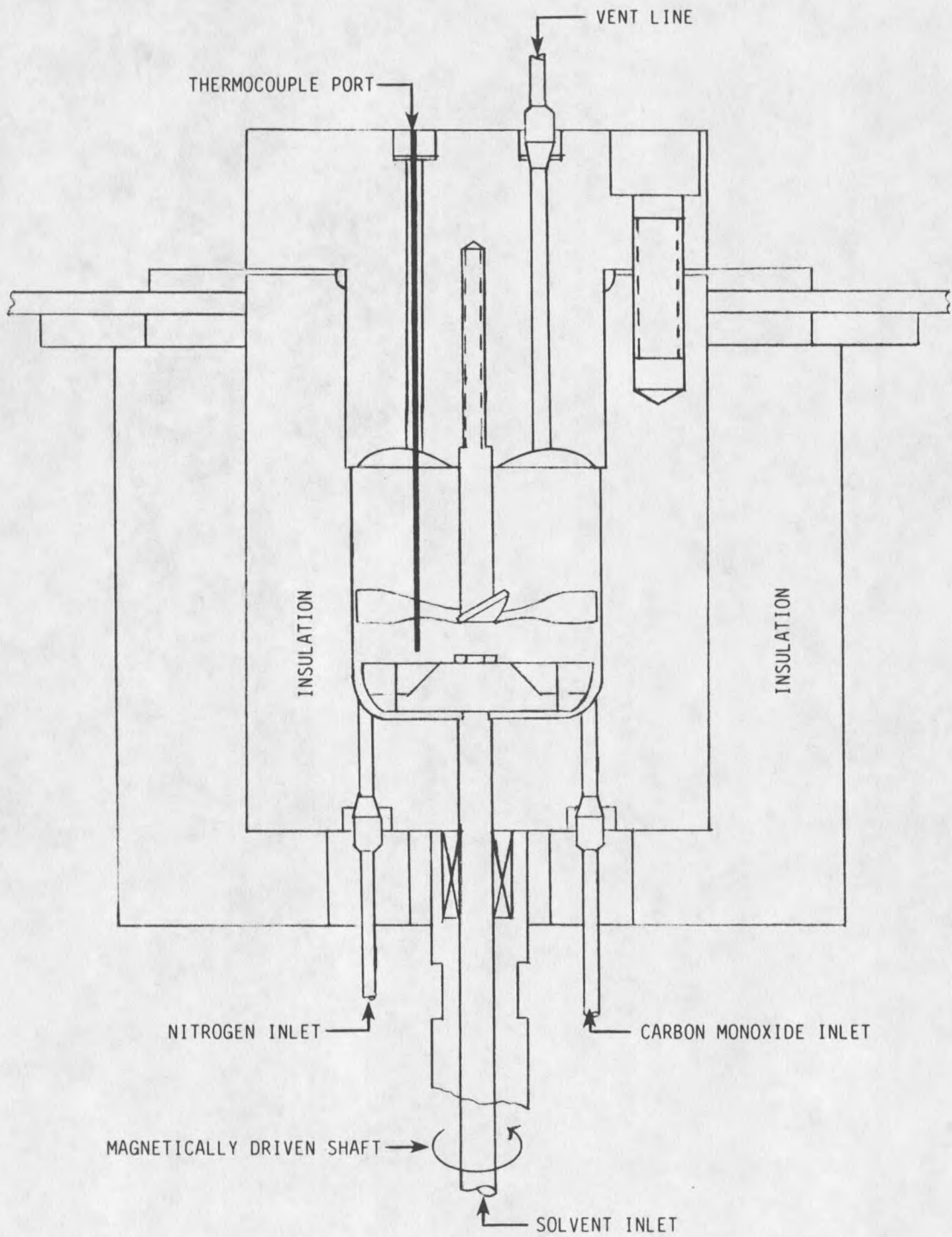


Figure 4. Magnetically Stirred Reactor.





























































































































