

EXTRACTION AND CONVERSION OF COAL
USING SUPERCRITICAL FLUIDS (Task 4)

Background

A program for the study of supercritical fluids for extraction of coal and oil shale is being carried out. Emphasis is placed on developing a fundamental understanding of supercritical phase behavior as applied to coal conversion and for coal liquid deashing. Initial experiments⁽¹⁾ which have used supercritical water to convert coal to gases, liquids and THF Soluble products at supercritical temperature have produced high conversion with brown coal (75%) and with bituminous coal (58%). Results obtained to date have shown coal rank, coal type, extraction (reaction) time, extraction temperature and density of the supercritical phase are important variables.

It has been hypothesized that when coal is contacted with a supercritical fluid, a portion of the coal dissolves instantaneously. The amount of coal which dissolves depends on the density and the temperature of the supercritical fluid and should increase with an increase in density and temperature of the supercritical fluid. It is further hypothesized that only the part of the coal which is dissolved in the supercritical fluid undergoes liquefaction reactions which results in the formation of oils, asphaltenes, preasphaltenes, gases and char. Consequently, short contact time liquefaction of bituminous coal in a non-donor solvent like toluene is being studied to determine if these effects actually exist. This report is a thorough description of the work from September 13, 1983 through September 12, 1984.

Progress

Experiments were done with Illinois #6 coal to study the effect of coal type on conversion to gases, liquids and THF soluble products. The results

are given in Table I. It was found that the conversion with Illinois #6 coal was low (28%) compared to that obtained with Bruceton bituminous coal (58%) under identical reaction conditions. This may be attributed to the relatively high preasphaltene content in raw Bruceton coal (8-9%) as compared to 2-3% in the case of raw Illinois #6 coal. Results with Powhattan #5 coal by Towne [2] support this hypothesis. Conversion of Powhattan #5 under identical conditions was only 17%, the lowest of the three coals, and the preasphaltene content of the raw coal (1%) was also the lowest.

Experiments were done with supercritical fluid mixtures of water and a hydrogen donor (piperidine) to study the effect of the addition of a hydrogen donor to the solvent. The effects studied include the conversion of coal to gases, liquids and THF soluble products. A 20 mole % (54 wt %) piperidine water mixture was used as a supercritical fluid. The results are given in Table I. The coal conversion was lower (16%) than either water or piperidine under identical reaction conditions of temperature, reduced density, reaction time and agitation conditions. This can be attributed to the adduct-forming tendency of piperidine with coal leading to formation of THF insoluble products. The molar density of the mixture was lower due to the higher wt % (54 wt %) of a high mol. wt. substance (piperidine 85) compared to that of water (18). It was found earlier using brown coal that by increasing the density (ρ) of the supercritical water from $\rho_c/2$ to ρ_c the amount of THF solubles obtained after a fifteen minute extraction increased from 40% to 75%. Hence the lower conversion obtained using the piperidine water mixture is probably partially due to the lower molar density of the supercritical fluid mixture.

Experiments with piperidine-water mixture were conducted with coal being present during the heat-up period. The mixture composition was 20 mole %

piperidine. The results are also given in Table I. For the reaction time of one hour, the conversion obtained was 69%. This was in between the conversion obtained for water (10%) and piperidine (82%) under otherwise identical conditions. The adducts formed by piperidine and coal may be decomposing into THF soluble products as reaction time increases. Also, the presence of piperidine curbs some of the retrogressive reactions going on when coal is present during the heat-up period. Hence the conversion is higher for the piperidine-water mixture than for water alone. Piperidine is a good hydrogen donor and helps in stabilizing the products of coal decomposition during the heat-up period. Hence the conversion in the case of piperidine is greater than for either water or the piperidine-water mixture.

Experiments were done with toluene and piperidine mixtures at the mixtures critical density and temperature of 57.6°C. In the analysis of the reaction products, the presence of piperidine in the coal can affect the determination of the amount of toluene soluble. In order to eliminate this effect during extraction by toluene and THF, the slurry (piperidine + toluene + coal + products) was kept in the hood so that the piperidine and toluene could vaporize (this is Extraction Procedure A). Next it was slurried with fresh toluene and extracted with toluene and then with THF. The results are summarized in Table II.

It was found that the conversion to THF soluble products for fractions of piperidine greater than 50% were negative. It has been reported by Jolley et al. (3) that polymerization of pyridine soot during extraction of the reaction residues occurs unless an inert atmosphere is present.

An experiment was done with piperidine at 380°C, at the critical density of piperidine and for a reaction time of 30 minutes. The results are given in Table III. The piperidine was allowed to evaporate and the solid mass

extracted with toluene and then with THF. The conversion was found to be negative although the conversion at 15 minutes and 60 minutes under otherwise identical reaction conditions were 53% and 83% respectively. The only difference was in the way products were extracted. In the case with high conversions, the slurry from reactor was not dried, but rather simply diluted with toluene and filtered through an extraction thimble. (this is Extraction Procedure B).

In order to determine whether removing the piperidine from the slurry by evaporation or not made a difference in the results, an experiment was performed with piperidine at 380°C and the critical density of piperidine for a reaction time of eight minutes. The slurry taken from reactor after quenching was divided into four identical parts. Two parts were diluted with toluene and extracted (Procedure B) and two parts were dried (Procedure A) and then extracted with toluene. The results are given in Table III. The percent of the slurry which dissolved in toluene is greater in the first case. This indicates that removal of piperidine by evaporation may cause the reaction products to polymerize and/or the piperidine is extracting some of the toluene insolubles.

An experiment was carried out with high sodium lignite and water at 380°C and at 0.35 g/cc density of supercritical water for a reactor time of 30 minutes. The conversion to gases, liquids, and THF soluble products was 46%. The results are given in Table IV.

An experiment was conducted with Bruceton coal and water at 380°C and at 0.35 g/cc density of supercritical water for a reaction time of 30 minutes. The raw coal solids after filtration and the THFI of those solids were sent to PETC for microscopic analysis. The coal injected was of the order of 3g and conversion to gases, liquids and THF soluble products was 33%. The results

are given in Table IV. The microscopic analysis were done by PETC analysts who concluded that the processed coal had a higher fraction of mineral matter included in the organic matrix than the original coal which would occur if organic matter was extracted and mineral matter was not.

Experiments were carried out with Bruceton coal and toluene at different stirrer speeds to find out at which stirrer speed the mass-transfer resistances are eliminated. The results are summarized in Table V and Figures 1 and 2 show the effect of stirrer speed on Toluene Solubles and THF Solubles respectively. It can be clearly seen that the stirrer speed has very little effect. It could be attributed to the high diffusivity of the supercritical phase and long reaction time employed (30 minutes). If the experiments were carried out at low reaction time (less than 10 minutes), the usual plot of conversion increasing with stirrer speed and then flattening out with no effect of stirrer speed after a certain value of stirrer speed could be observed.

Experiments were carried out at different coal/solvent ratios by injecting different amounts of coal under identical conditions of supercritical toluene density, temperature and reaction time. The results are summarized in Table VI and Figures 3 and 4 show the effect of amount of coal injected on the Toluene/THF solubles. It is found that as the amount of coal injected decreases i.e. the solvent/coal ratio increases, the Toluene solubles/THF solubles increases. It is known from Figure 5 that the toluene solubles undergo retrogressive reactions. Hence if the solvent/coal ratio is higher the retrogressive reactions are curbed because the toluene solubles are present in a more dilute state than when a lower solvent/coal ratio is employed. This implies that retrogressive reactions follow second order kinetics.

Experiments were carried out with Bruceton coal and toluene at reduced densities of 1.0 and 1.5 and reaction time of two minutes to 60 minutes. The results are summarized in Tables VII and VIII and Figures 6 and 7 show the effect of reaction time on Toluene Solubles and THF Solubles respectively at reduced density of 1.0 and 1.5. It can be seen that the initial rate of formation of Toluene Solubles and THF Solubles is higher when the reduced density is higher. This is consistent with the hypothesis that the solubility of coal increases with an increase in the density of the supercritical fluid. Part of the coal instantaneously dissolves in the supercritical fluid due to the high diffusivity of the supercritical fluid and the high stirrer speed employed during the experiment. The coal which has dissolved instantaneously undergoes liquefaction reactions resulting in the formation of Toluene Soluble and THF Soluble products. The experimental data at higher reaction time show a lot of scatter. Amestica and Wolf⁽⁴⁾ have reported a decrease in conversion after a reaction time of 30 minutes using Illinois #6 and Toluene although their mode of contact was different from ours; in their experiments the coal was not injected into the supercritical toluene phase but was heated with toluene to supercritical conditions and the reaction time was measured from the time the slurry attained the desired temperature. They also reported an increase in conversion with an increase in density of the supercritical fluid.

A two variable regression for toluene solubles and THF solubles with density of the supercritical phase and reaction time as the two variables was tried with different kinds of functions but the data could not be smoothed as desired. Hence the raw data will be used directly for the model proposed in the 2nd Quarterly report.

Experiments were carried out with Bruceton coal and toluene at reduced densities of 1.0 and 1.5 and reaction times of two minutes to 60 minutes at 673 K. The results are summarized in Tables IX and X and Figure 5 shows the effect of reaction time on Toluene Solubles at reduced density of 1.0 and 1.5. As found at 647 K, the initial rate of formation of Toluene Solubles is higher when the reduced density is higher. This is consistent with the hypothesis that the solubility of coal increases with an increase in the density of the supercritical fluid. As seen from the experiment data, the toluene solubles decrease with reaction time after going through a maxima in toluene solubles. This is more pronounced at 673 K as expected as the retrogressive reactions become more important at higher temperature.

Model for Coal Liquefaction with a Supercritical Fluid Which Does Not React with Coal

Experimental Observations Made

1. The effect of stirrer speed did not have a very significant effect on the formation of oils and asphaltenes in the range of stirrer speed studied. Due to the high diffusivity of the supercritical fluid and the high stirrer speed employed, it can be assumed that a part of coal instantaneously dissolves on contact with the supercritical fluid.

2. The rate of formation of oils and asphaltenes is a strong function of pressure (or density) at all the temperature levels studied. The pressure was changed by changing the density of the supercritical fluid. The fluid used was toluene which does not react with coal⁽⁴⁾. The rate of formation of oils and asphaltenes increases with temperature. This effect has been reported by Amestica and Wolf⁽⁴⁾ and Solmka and Rutkowski.⁽⁵⁾

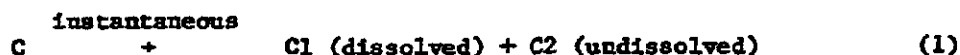
3. There is no formation of preasphaltenes during the coal liquefaction with supercritical toluene as it does not react with coal. The amount of

preasphaltenes are found to decrease from a small finite value, present in raw coal (estimated from THF solubility) to zero. This is attributed to thermal reactions of asphaltenes from preasphaltenes yielding char and oils.

4. The amount of oils plus asphaltenes initially increases with reaction time at all temperature levels but at higher temperature levels when the retrogressive reactions become significant, the oils plus asphaltenes go through a maximum and start decreasing with time. This kind of behavior has been reported by Amestica and Wolf.⁽⁴⁾

The above experimental observations have lead to the development of the following hypothesis: - when coal is contacted with a supercritical fluid in an apparatus where mass-transfer resistances have been eliminated, more non-volatile compounds in the coal dissolve, instantaneously, in the supercritical fluid than would under low pressure conditions. However, the amount of coal which dissolves depends on the temperature and density of the supercritical fluid. The solubility of any compound in a supercritical fluid has been shown by Ziger and Eckert⁽⁶⁾ to be dependent on the density of the supercritical fluid; the solubility in general, increases with an increase in the density of the fluid. Additionally, the fluid phase solubility of any compound increases with temperature due to the increase of the vapor-pressure of the compound with temperature. It is further hypothesised that only the part of the coal which is dissolved in the supercritical fluid undergoes liquefaction reactions which results in formation of oils, asphaltenes, gases and char.

The model reactions and equations are as follows:





$$dC_1/dt = -K_1 C_1 \quad (6)$$

$$dP/dt = -K_2 P - K_3 P \quad (7)$$

$$dA/dt = K_1 C_1 + K_2 P - K_4 A \quad (8)$$

$$d\text{Char}/dt = K_3 P + K_4 A \quad (9)$$

$$C = C_1 + C_2 + \text{Char} \quad (10)$$

C - coal, C₁ - coal dissolved in supercritical solvent, C₂ - coal not dissolved in supercritical solvents, A - oils, asphaltenes and gases, P - preasphaltenes, C - THF insoluble product.

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Table I
Summary of Batch Extraction/Reaction of Coal using Supercritical Fluids

Run #	33	38	44
Coal Used	BBC	BBC	Illinois #6
Procedure	A	B	A
Water, wt%	46.0	45.9	100
Piperidine, wt%	54.0	54.1	-
Fluid Density, kg/m ³	312	341	352
Coal charged $\times 10^3$, kg	11.7	60.4	14.6
Temperature, K	647.0	648.0	647.8
Pressure prior to injection, MPA	21.81	N.A.	22.57
Pressure, MPA (solvent + Argon)	24.93	23.19	24.15
Reaction time, min	15	60	60
Stirrer speed, rpm	2300	2300	2300
THFI, feed coal, %	91.3	91.3	97.2
THFI, solid product, %	16.0	31.3	72.3
Gaseous Product, %	-	-	-
Solid Product, %	98.7	-	77.1
Liquids (by diff)	-	-	-
Ash %, in THFI	5.39	11.21	17.85
p ^H of water	-	-	8.7

Table II
Summary of Batch Extraction/Reaction of Bruceton Bituminous Coal
using Supercritical Toluene-Piperidine Mixtures

Run #	39	40	41	42	43	45
Toluene, wt %	94.93	75.00	54.92	34.98	15.50	54.63
Piperidine, wt %	5.07	25.00	45.08	65.02	84.50	45.37
Fluid density, kg/m ³	292	307	295	295	298	297
Coal charged x 10 ³ kg	37.0	34.7	42.3	38.1	32.1	58.7
Temperature, K	647.6	647.6	647.6	647.6	647.6	647.6
Pressure prior to injection, MPa	7.23	6.87	6.84	6.89	7.08	7.01
Pressure, MPa (solvent + argon)	10.88	9.80	10.13	10.16	10.25	11.38
Reaction time, min.	15	15	15	15	15	15
Stirrer speed, rpm	2300	2300	2300	2300	2300	2300
Solid product, %	98.16	97.48	106.40	148.02	165.31	-
Toluene insolubles, %	93.43	97.48	82.23	130.09	144.03	86.99
THF insolubles, %	87.64	91.69	82.23	111.67	110.39	83.36
Piperidine insolubles, %	86.49	85.30	80.90	98.89	90.92	-

*The products are expressed as % of coal injected.

Table III
Summary of Batch Extraction/Reaction of Bruceton Bituminous Coal
using Supercritical Piperidine

Run #	47	57	57
Extraction Procedure	A	A	B
Piperidine Density, kg/m ³	299	305	305
Coal charged x 10 ³ kg	51.0	41.3	41.3
Temperature, K	647.6	647.6	647.6
Pressure prior to injection, MPa	7.21	7.38	7.38
Pressure, MPa (solvent + argon)	11.05	11.81	11.81
Reaction time, min.	30	8	8
Stirrer speed, rpm	2300	2300	2300
Toluene insolubles, %	150.90	107.31	52.49
THF insolubles, %	117.87	107.31	52.49
Pyridine insolubles, %	82.05	107.31	-

Table IV
Summary of Batch Extraction/Reaction of Coal/Lignite
using Supercritical Water

Run #	54	57
Solid Substrate	Brueton bituminous Coal	Hi Na Lignite
Water density, kg/m ³	352	352
Coal/Lignite charged x 10 ³ kg	3.1	11.4
Temperature, K	649.8	649.8
Pressure prior to injection, MPa	23.16	22.55
Pressure, MPa (solvent + argon)	24.28	25.46
Reaction time, min.	30	30
Stirrer speed, rpm	2300	2300
% Solid product	79.00	59.60
% Gases	1.51	7.70
% Liquids by diff.	19.49	32.70
% THFI	67.20	53.70

Table V
Summary of Batch Extraction/Reaction of Bruceton Coal
using Supercritical Toluene

Effect of Stirrer Speed on Conversion

Run #	91	70	68	69	64	71	65	49	92
Toluene Density, kg/m ³	302	304	304	304	303	301	302	301	302
Coal charged x10 ³ -kg	40.1	41.5	41.0	40.1	39.7	42.2	41.7	38.9	39.9
Temperature, K	672.6	672.5	672.6	672.5	672.3	672.4	672.4	672.4	672.6
Pressure prior to injection, MPa	7.76	7.86	7.92	7.83	7.87	7.83	7.88	7.62	7.80
Pressure, MPa (Toluene + Argon)	12.99	12.43	11.69	12.45	12.76	12.40	12.48	11.87	12.91
Reaction time, min.	30	30	30	30	30	30	30	30	30
Stirrer speed, rpm	0	508	988	988	1627	2040	2110	2300	2300
% Toluene Solubles	13.14	9.42	9.77	9.83	10.84	11.74	13.85	14.58	13.78
% THF Solubles	13.14	12.81	9.77	9.83	10.84	11.74	13.85	16.51	13.78

Table VI

Summary of Batch Extraction/Reaction of Bruceton Coal
using Supercritical Toluene

Effect of Coal/Solvent Ratio on Conversion

Run #	90	52	89	26	88
Toluene Density, kg/m ³	303	304	304	303	303
Coal charged x10 ³ kg	7.7	19.9	29.7	39.9	49.9
Temperature, K	673.0	672.4	672.3	673.3	672.4
Pressure prior to injection, MPa	7.87	7.87	7.85	7.79	7.83
Pressure, MPa (Toluene + Argon)	11.03	12.42	12.39	15.19	13.23
Reaction time, min.	15	15	15	15	15
Stirrer speed, rpm	2300	2300	2300	2300	2300
% Toluene Solubles	18.30	14.50	12.74	17.10	14.00
% THF Solubles	18.30	15.39	12.74	19.70	14.00

Table VII
Summary of Batch Extraction/Reaction Bruceton Coal
using Supercritical Toluene
Effect of Reaction Time at Reduced Density of 1.0 and Temperature of 647 K

Run #	72	76	74	67	66	25	29	46
Toluene density, kg/m ³	301	300	300	300	302	305	305	302
Coal charged x10 ³ , kg	41.1	40.2	40.0	39.0	42.7	34.9	35.6	31.7
Temperature, K	646.8	647.2	648.1	648.4	648.2	647.5	647.2	648.0
Pressure prior to injection, MPa	6.78	6.71	6.72	6.99	6.78	6.67	6.66	6.73
Pressure, MPa (Toluene + Argon)	10.93	11.12	11.33	11.45	11.54	14.74	15.45	11.09
Reaction time, min.	2	4	6	8	10	15	30	60
Stirrer speed, rpm	2300	2300	2300	2300	2300	2300	2300	2300
% Toluene Solubles	0.90	3.96	4.37	2.75	3.89	7.92	10.58	18.02
% THF Solubles	2.61	3.96	4.68	5.88	4.34	14.12	12.27	18.02

Table VIII

Summary of Batch Extraction/Reaction of Bruceton Coal
using Supercritical Toluene

Effect of Reaction Time at Reduced Density of 1.5 and Temperature of 647 K

Run #	73	75	77	28	60	97	59
Toluene Density, kg/m ³	446	447	447	438	443	444	444
Coal charged x10 ³ kg	36.8	33.9	37.1	29.0	34.9	32.9	38.3
Temperature, K	647.3	648.7	647.8	647.1	648.2	647.6	647.8
Pressure prior to injection, MPa	9.26	9.27	9.30	8.70	9.37	9.80	9.16
Pressure, MPa (Toluene + Argon)	12.77	13.48	13.96	16.92	13.78	13.42	14.15
Reaction time, min.	2	6	10	15	30	30	60
Stirrer speed, rpm	2300	2300	2300	2300	2300	2300	2300
% Toluene Solubles	4.45	7.89	8.69	19.85	7.85	10.99	13.08
% THF Solubles	5.08	7.89	9.80	22.16	11.89	10.99	13.53

Table IX

Summary of Batch Extraction/Reaction of Bruceston Coal
using Supercritical Toluene

Effect of Reaction Time at Reduced Density of 1.0 and Temperature of 673 K

Run #	56	87	86	53	85	26	92	49	48
Toluene Density, kg/m ³	303	303	300	303	300	303	302	301	298
Coal charged x10 ³ kg	39.0	38.3	39.9	48.5	40.3	39.9	39.9	38.9	39.9
Temperature, K	676.2	672.0	672.4	672.0	672.1	673.3	672.6	672.0	672.0
Pressure prior to injection, MPa	8.2	7.87	7.84	7.80	7.77	7.79	7.80	7.62	7.63
Pressure, MPa (Toluene + Argon)	12.11	12.66	12.86	11.94	12.75	15.19	12.91	11.87	11.83
Reaction time, min.	2	4	6	8	9	15	30	30	60
Stirrer speed, rpm	2300	2300	2300	2300	2300	2300	2300	2300	2300
% Toluene Solubles	7.51	5.97	11.22	11.39	9.90	17.10	13.78	14.58	15.81
% THF Solubles	9.18	5.97	11.23	11.39	9.90	19.70	13.78	16.51	16.72

Table X

Summary of Batch Extraction/Reaction of Bruceton Coal
using Supercritical Toluene

Effect of Reaction Time at Reduced Density of 1.5 and Temperature of 673 K

Run #	95	94	93	98	99	100
Toluene Density, kg/m ³	444	444	443	444	444	444
Coal charged $\times 10^3$ kg	29.1	35.9	36.4	31.1	32.9	31.4
Temperature, K	670.8	671.3	672.2	672.3	672.9	672.9
Pressure prior to injection, MPa	11.27	11.57	11.31	11.26	11.28	11.83
Pressure, MPa (Toluene + Argon)	16.03	17.47	16.78	16.49	16.17	16.96
Reaction time, min.	2	6	10	15	30	60
Stirrer speed, rpm	2300	2300	2300	2300	2300	2300
% Toluene Solubles	8.49	14.68	17.08	24.66	21.40	19.08
% THF Solubles	9.25	14.96	18.16	24.66	21.40	19.21

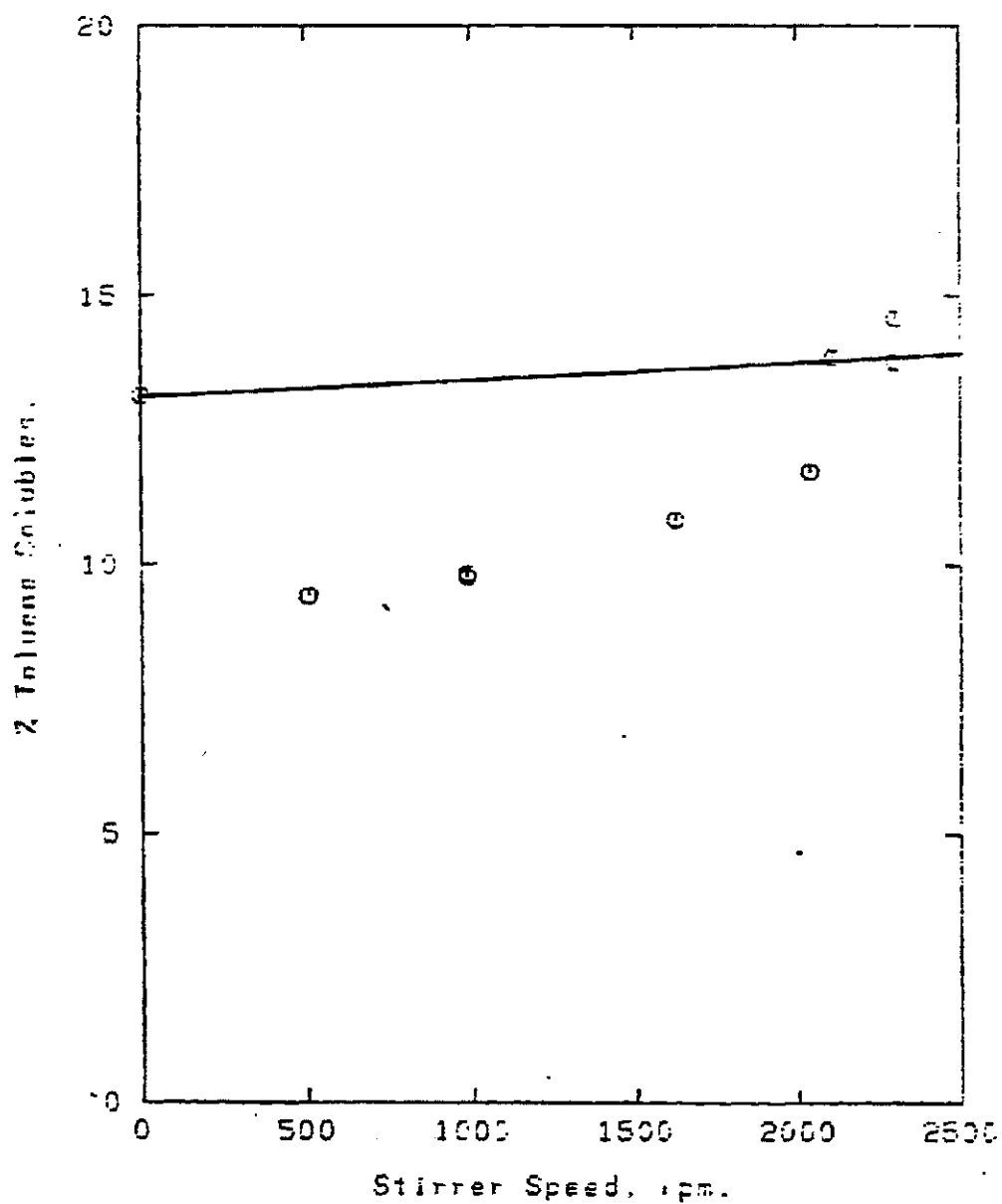


Figure 1 Effect of Stirrer Speed on Tol. Sol. @ 672.5 K.

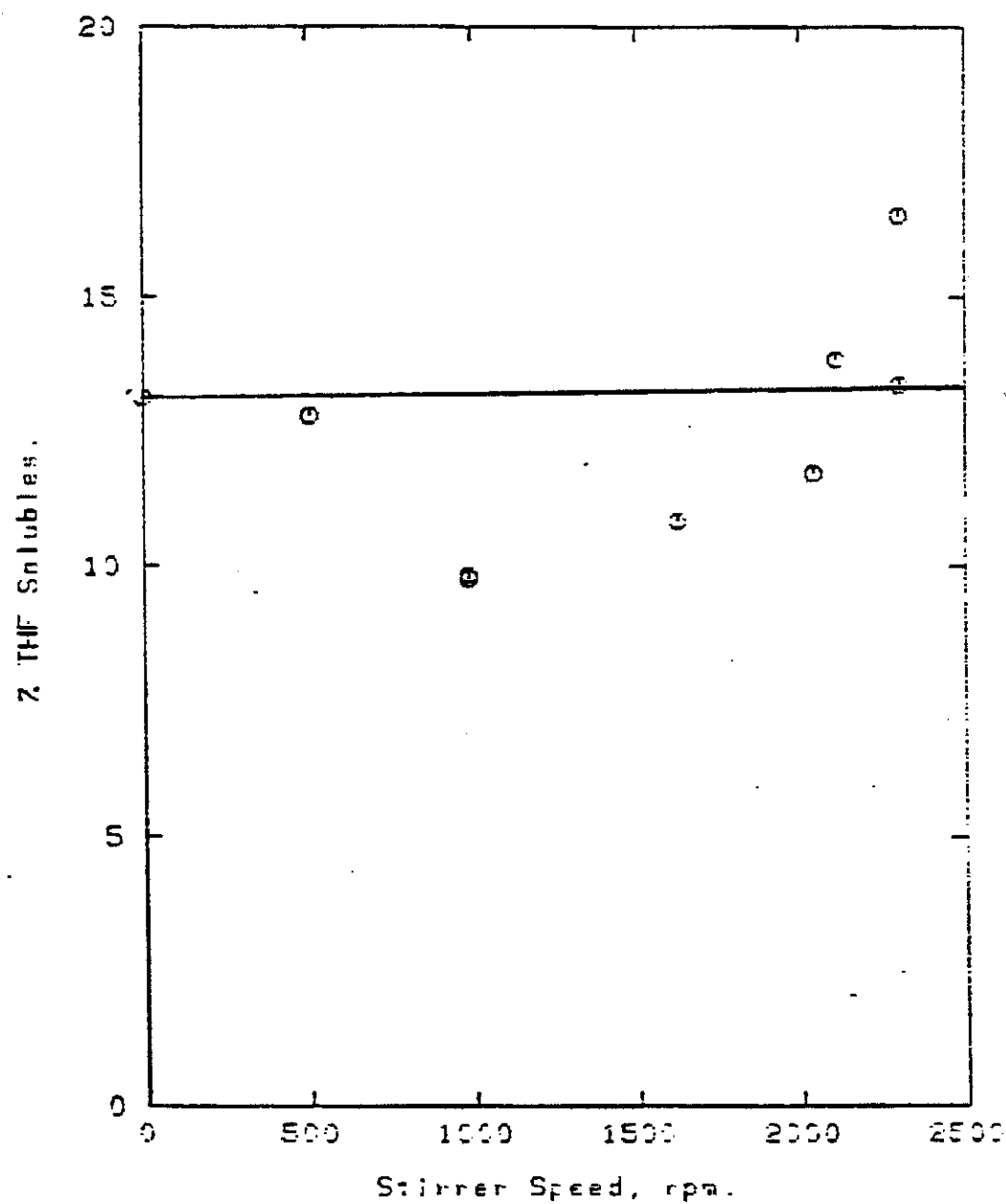


Figure 2 Effect of Stirrer Speed on THF Sol. @ 672.5 K.

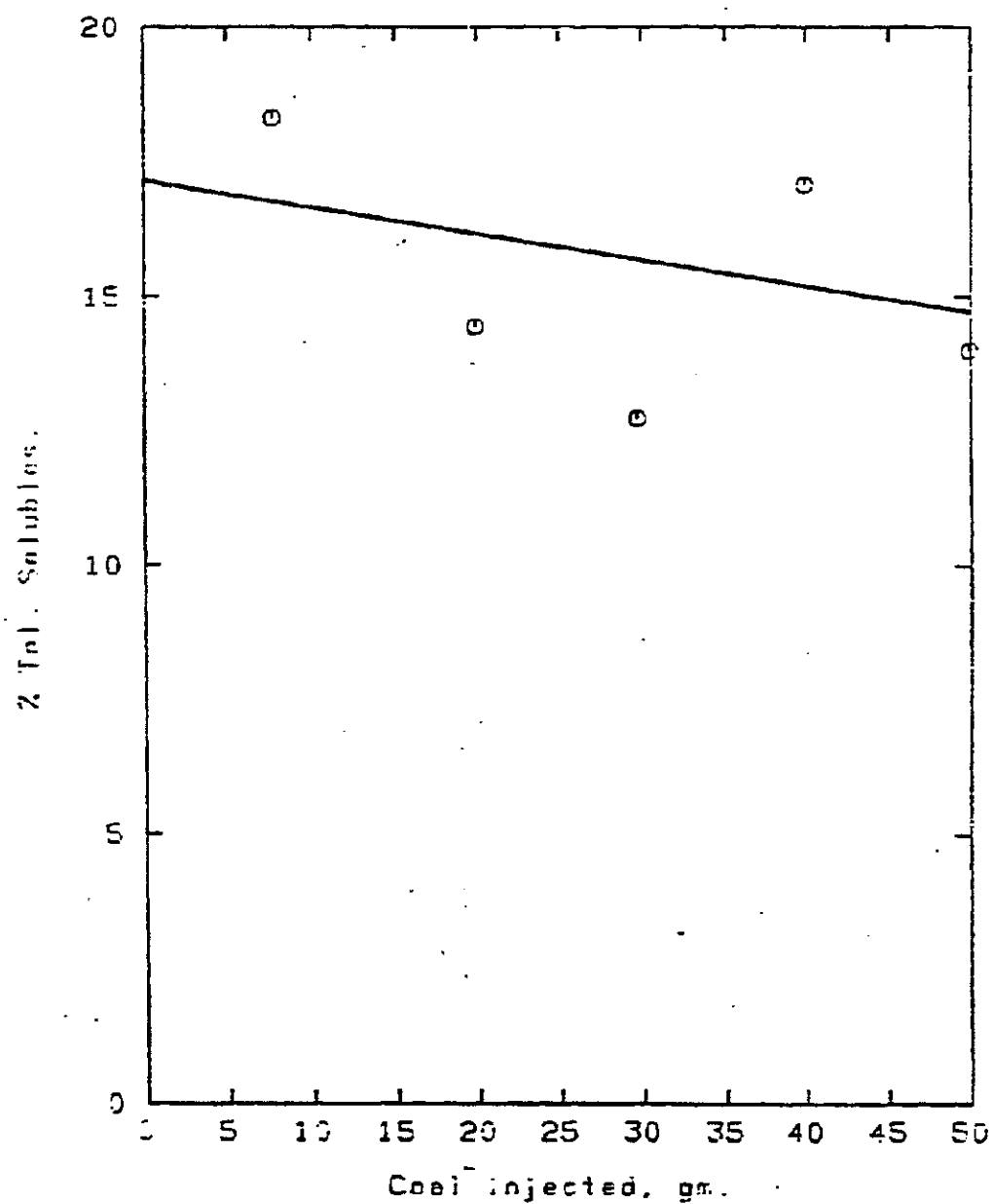


Figure 3 Effect of Amt. of Coal inj. on Tol. Sol. @ 672.5 K.

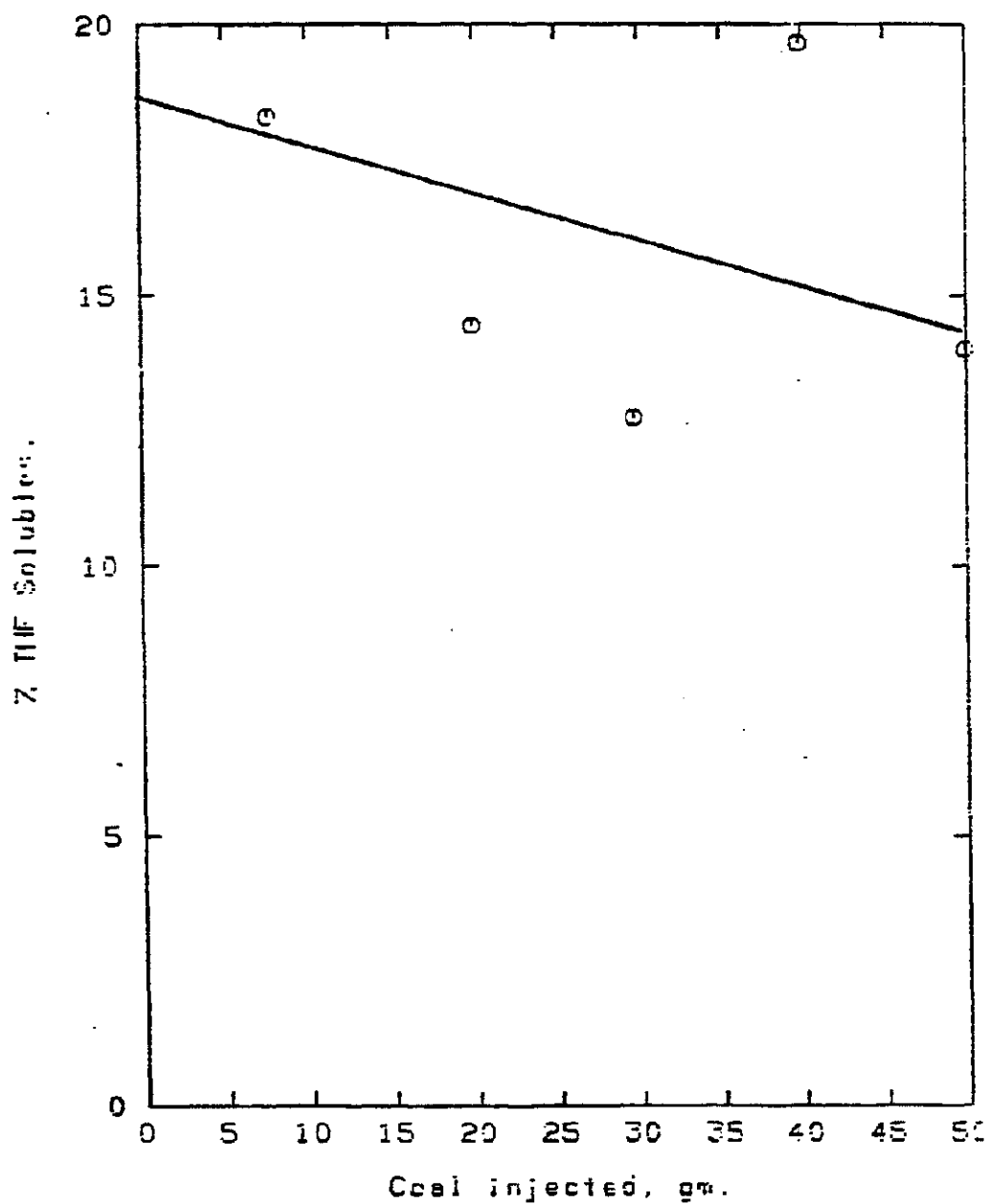


Figure 4 Effect of Amt. of Coal Inj. on THF Sol. @ 672.5 K.

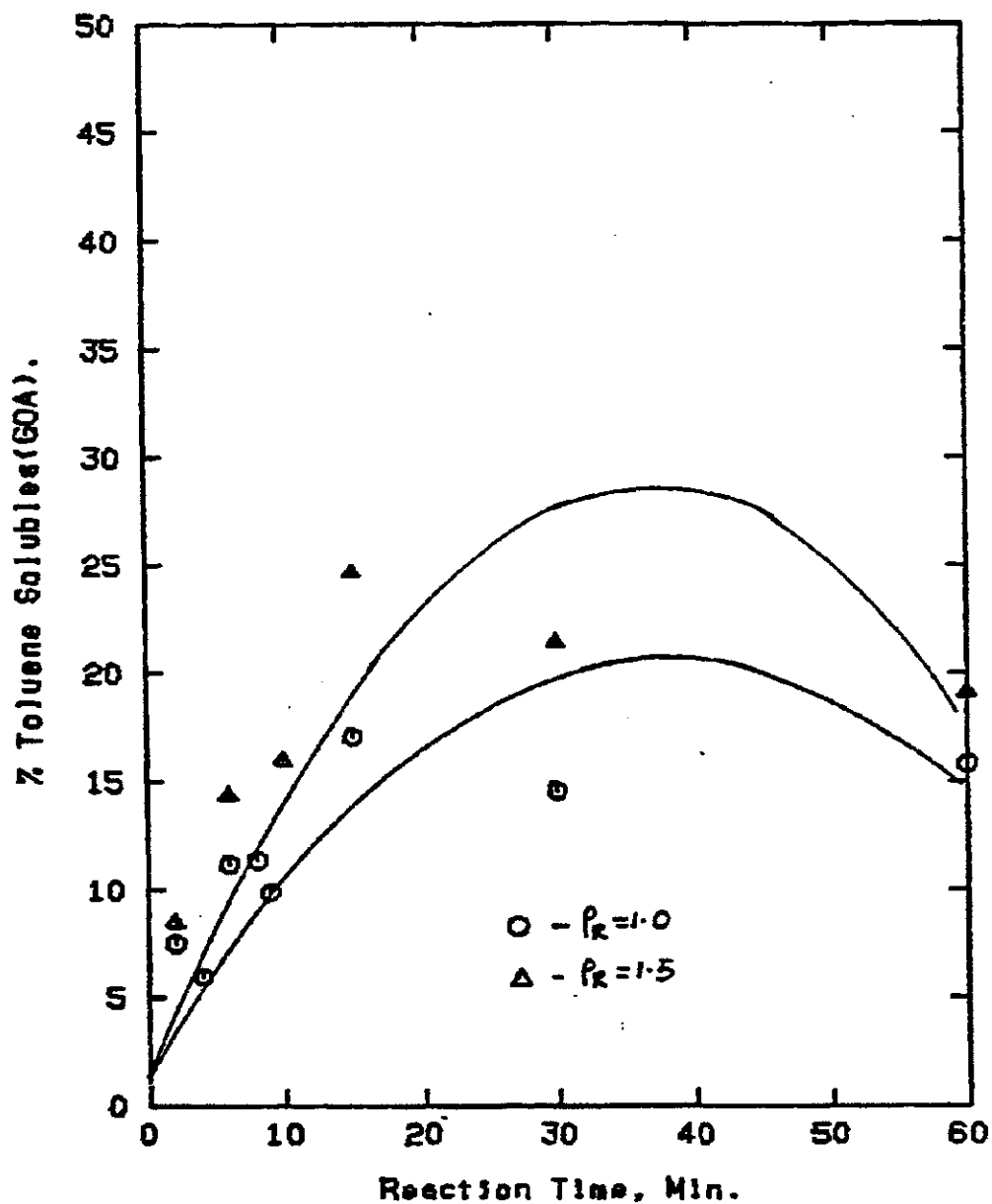


Figure 5 Effect of Reaction Time & Density on % GOA @ 750 F.

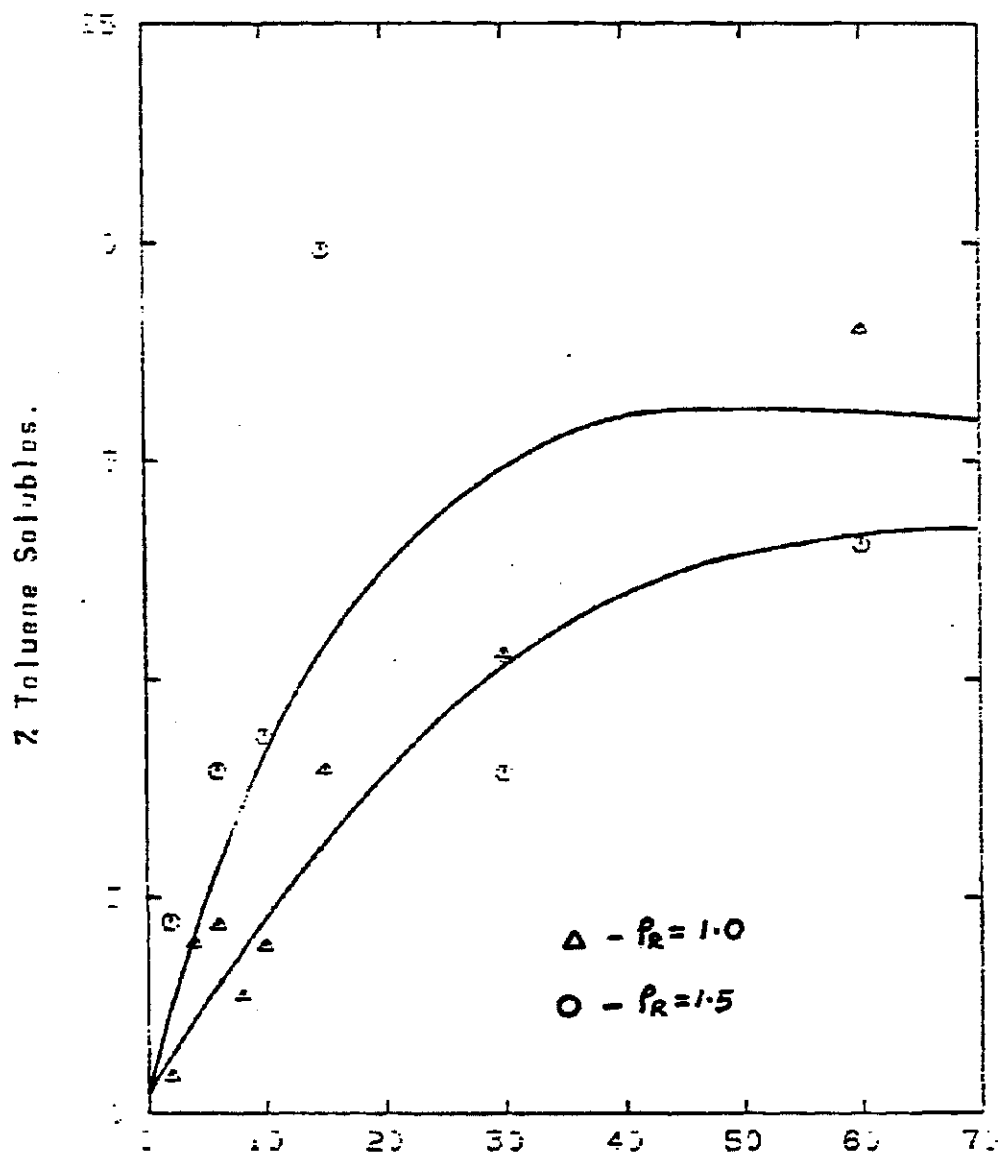


Figure 6 Reaction Time in Minutes.

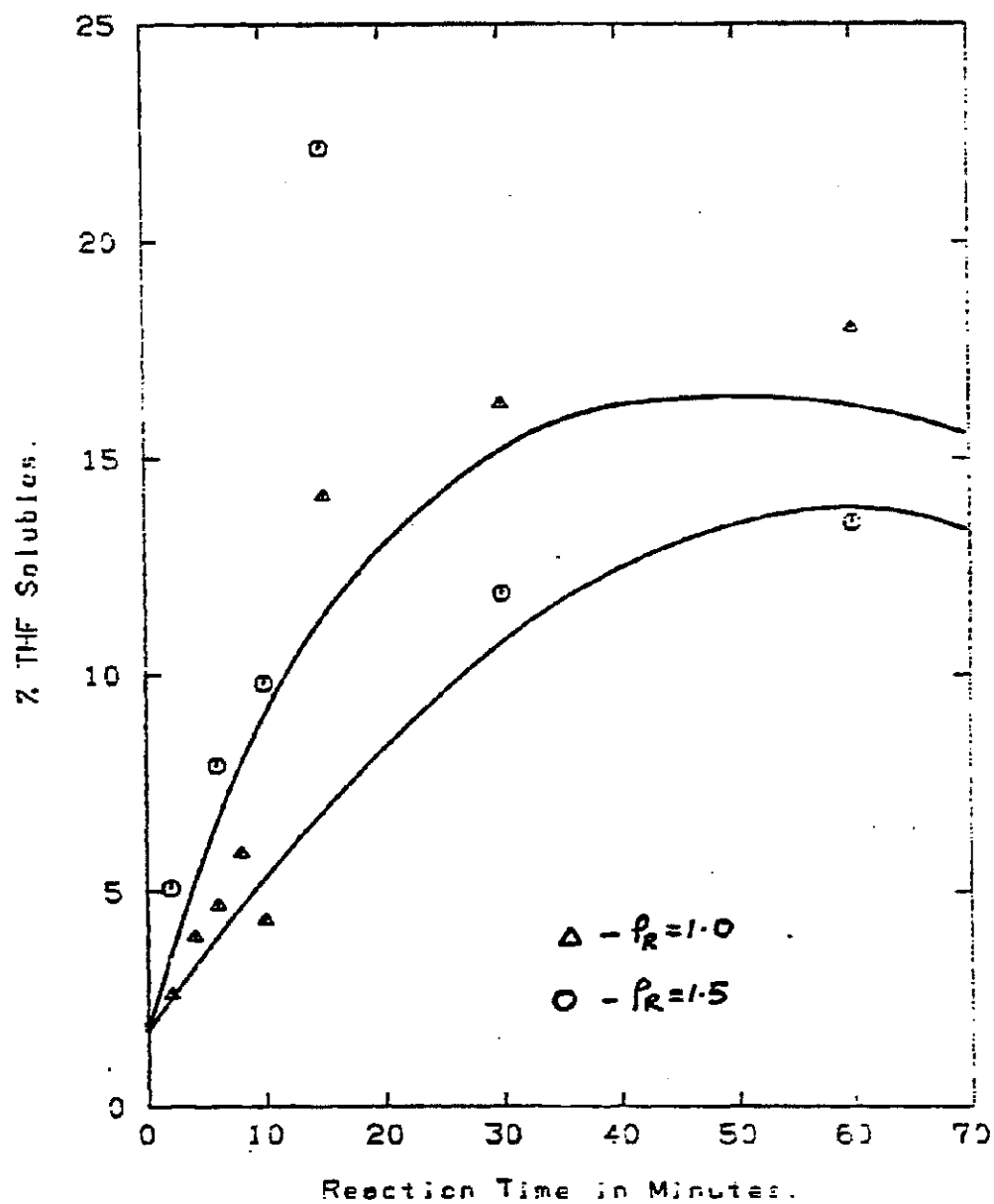


Figure 7 Effect of Reaction Time and Degree of SO Triole on % THF Solubles

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