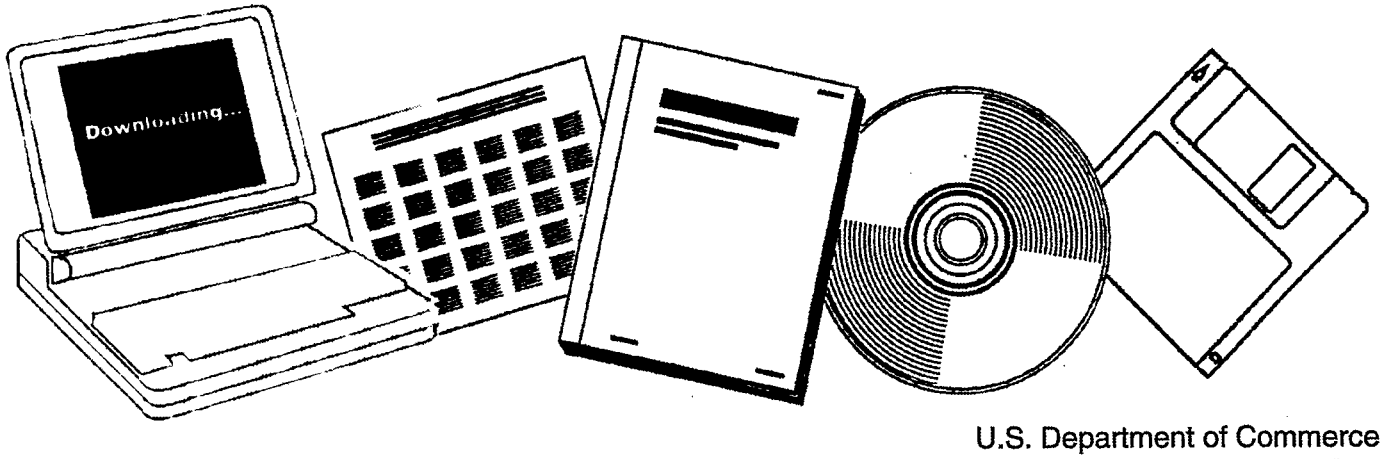
SELECTIVE CATALYTIC CRACKING OF FISCHER-TROPSCH LIQUIDS TO HIGH VALUE TRANSPORTATION FUELS. REPORT NUMBER 24: QUARTERLY TECHNICAL PROGRESS REPORT FOR FOURTH QUARTER FISCAL YEAR 1992 (JULY 1--SEPTEMBER 30, 1992)

AMOCO RESEARCH CENTER, NAPERVILLE, IL.
RESEARCH AND DEVELOPMENT DEPT

1992



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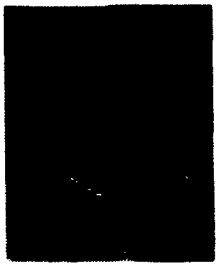

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THE SELECTIVE CATALYTIC CRACKING OF FISCHER-TROPSCH LIQUIDS TO HIGH VALUE TRANSPORTATION FUELS.

REPORT NO. 24

QUARTERLY TECHNICAL PROGRESS REPORT

FOR

FOURTH QUARTER FISCAL YEAR, 1992

(July 1, 1992 - September 30, 1992)

PROJECT MANAGER: D. M. WASHECHECK

PRINCIPAL INVESTIGATOR: W. J. REAGAN

WORK PERFORMED UNDER CONTRACT NO. DE-AC22-91PC90057

FOR

U.S. DEPARTMENT OF ENERGY
PITTSBURGH ENERGY TECHNOLOGY CENTER
PITTSBURGH, PENNSYLVANIA

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DISCLAIMER

EXECUTIVE SUMMARY

Amoco Oil Company, under a contract with the United States Department of Energy, is investigating a selective catalytic cracking process to convert the Fischer-Tropsch gasoline and wax fractions to high value transportation fuels. This report describes the work in the fourth quarter, fiscal year 1992, the fifth quarter of the two year project.

Task 1, Project Management Plan. The plan has been accepted by the Project Manager DOE/PETC. This report contains the most current and accurate information and projections of the scope of work, schedules, milestones, staffing/manpower plan and costs.

Task 2, Preparation of Feedstocks and Equipment Calibration. The work in this area is complete. The wax feedstock for this program, a commercial sample of Fischer-Tropsch product from Sasol, is a high melting point,(>220°F), high boiling range (50% boiling above 1000°F), largely paraffinic material.

Task 3, Catalytic Cracking Catalyst Screening Program. The wax feedstock readily converts over conventional fluid catalytic cracking (FCC) catalysts (85%+ conversion) to high yields of C_4- gas (high in propylene and C_4 olefins) and naphtha (C_5-430°F). Three different types of zeolite catalysts and one amorphous cracking catalyst show wide variations of product yields as a function of wax feedstock conversion. The Beta and HZSM-5 zeolite catalysts have higher target light olefin (isobutylene and isoamylenes) yields than the Y zeolite sample. The HSZM-5 sample also produces the highest yields of propylene. Further work continues on various commercial and experimental HZSM-5 samples. There are some clear wax conversion variations among the samples. However, product selectivity differences are small and difficult to measure accurately. A series of rare earth exchanged Y zeolite catalysts show the expected "rare earth effect": increased rare earth exchange gives higher naphtha and lower olefin yields. Several new FCC catalysts with low (10%) zeolite content have been prepared and analyzed. The zeolite Beta sample appears to have a lower hydrothermal stability than zeolite Y catalyst.

Task 4, Pilot Plant Tests. No pilot plant tests were conducted this Quarter.

The catalytic cracking test results from both the small scale test unit and the pilot plant indicate that the wax feedstock readily converts to C_4- gas and naphtha. These tests suggest that very mild process conditions and low activity catalysts are needed to lower the overall wax conversion. The target light olefin yields vary with catalyst type and the process conditions.

BACKGROUND

Fischer-Tropsch (F-T) synthesis technology produces liquid hydrocarbons from synthesis gas (hydrogen and carbon monoxide) derived from the gasification of coal. Domestic supplies of both high- and low-rank coals are extensive and represent a strategic resource to supplement dwindling petroleum reserves. The Fischer-Tropsch technology has been practiced commercially at Sasol in South Africa since the mid-1950's. The F-T liquid product consists of a broad range of normal paraffins (C_5-C_{50}) and a small quantity of oxygenates and olefins. The gasoline range C_5-C_{12} product fraction consists of linear paraffins and olefins of low octane number. The distillate fraction, C_{12}-C_{18}, is an excellent quality fuel. The largest product fraction, C_{18}+, is primarily wax and is useless as a transportation fuel. There are many studies on the upgrading of these F-T liquids. These products are further treated by conventional petroleum processes, such as hydrotreating, reforming and catalytic cracking to produce conventional gasoline and distillate fuels. There are no reported studies of the catalytic cracking processing of F-T liquids to produce C_3-C_8 olefins as feedstocks for the synthesis of gasoline range ethers and alcohols. This is the primary focus of this project.

Fuel oxygenates, particularly alcohols and ethers, represent a potential solution to environmental concerns due to conventional automotive fuels. Governmental regulations, most recently in the Clean Air Act Amendments of November, 1990, have resulted in the phase-out of lead additives, lowering of the Reid vapor pressure of gasoline and in some geographical areas, the mandated use of oxygenates. Recent studies of methyl tertiary butyl ether (MTBE) and tertiary amyl methyl ether (TAME) suggest that these compounds may reduce automotive carbon monoxide emissions, have high blending gasoline octane ratings, R+M/2, (MTBE-108, TAME-102) and have low Reid vapor pressure values. These ethers are produced commercially by the etherification of the appropriate olefin by methanol (MTBE, isobutylene; TAME, isoamylenes). These olefins are derived from conventional petroleum processes such as catalytic cracking or steam/thermal reforming.

There is a growing need for alternative sources of olefins for ethers and alcohols syntheses as demand for these materials escalates beyond the capacity of conventional petroleum processes. This project addresses this requirement for an alternative olefin feedstock for oxygenate synthesis.

PROGRAM OBJECTIVES

The objective of this program is to prepare high-value transportation fuels, including gasoline, distillate, and gasoline range ethers and alcohols from non-petroleum resources. A selective catalytic cracking process of Fischer-Tropsch liquids is proposed. The C_4-C_8 product olefins would then be etherified with methanol to prepare the target ethers. Alcohols will be produced by direct hydration of C_3-C_8 product olefins. The gasoline and distillate products are also expected to be superior to conventional fuels because of the unique combination catalysts to be used in this process.

PROJECT DESCRIPTION

A two year, multi-task program will be used to accomplish the objective to develop a selective catalytic cracking process to produce premium transportation fuels, including ethers and alcohols from Fischer-Tropsch gasoline and wax products.

Task 1. — Project Management Plan. A plan will be prepared which describes the work to be done, milestones, and manpower and cost requirements.

Task 2. -- Preparation of Feedstocks and Equipment Calibration. Suitable mixtures of Fischer-Tropsch waxes (C_{18}+) and light olefin components (C_5-C_{12}) will be prepared to simulate full range F-T liquids without the premium distillate products. The necessary analytical equipment will be calibrated for the detailed identification of C_4-C_8 olefins and ethers and other paraffin, aromatic and naphthene gasoline range components.

Task 3. -- Catalytic Cracking Catalyst Screening Program. Various zeolite catalysts and process variables will be studied with small scale test equipment.

Task 4. -- Pilot Plant Tests of the Optimized Catalyst and Process. The optimized process will be tested on a pilot plant scale. The target light olefin products, gasoline and distillate products will be produced in sufficient quantities for complete characterization.

Task 5. -- Preparation of C_5-C_8 Ethers and C_3-C_8 Alcohols. These products will be prepared from the pilot plant C_3-C_8 olefin products.

Task 6. -- Evaluation of Gasoline Blending Properties of Ethers and Alcohol Products. The gasoline blending properties of the product ethers and alcohols will be measured. The properties of the distillate products will also be evaluated.

Task 7. -- Scoping Economic Evaluation of the Proposed Processes. An economic analysis of the proposed process will be compared with conventional petroleum processes and ether and alcohol synthesis routes.

The DOE reporting requirements for this contract will be followed in all cases. This includes all project status, milestone schedule, and cost management reports. A final detailed project report will be submitted upon completion of the contract.

RESULTS AND DISCUSSION.

During this quarter, project activities center on Tasks 3 and 4 of the contract.

TASK 1. Project Management Plan.

The draft Project Management Plan has been accepted by the Program Manager at DOE/PETC. This completes Task 1 of the contract. This document contains the most current and accurate information and projections of the scope of work, schedules, milestones, staffing/manpower plan and costs. This plan contains the following sections:

Management Plan
Technical Plan
Milestone Schedule/Manpower Plan
Cost Plan
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The technical approach builds from small scale tests of the selective cracking concept to pilot plant scale verification of product yields. The screening test results will serve as a preliminary milestone of this process scheme. An assessment of project directions, scope of work and objectives after this milestone will be appropriate.

TASK 2. Feedstock Characterization.

The wax feedstock has been analyzed by various analytical methods. The boiling point and the carbon number distributions of the largely paraffinic material are consistent with literature reports of similar Fischer-Tropsch samples. No further work in this area is planned.

TASK 3. Screening Catalytic Cracking Tests.

Activities under Task 3 of the contract continue on the small scale test unit, the MYU (Micro Yields Unit).

The catalytic cracking results from both the small scale test unit (MYU) and the pilot plant indicate that the wax feedstock readily converts to high yields of product gas (C_4-) and gasoline. The HZSM-5 zeolite catalyst has the highest isobutylene and isoamylenes yields among the catalysts tested to date. However, the production of propylene is also high. A series of HZSM-5 samples with added rare earth oxide levels and selected commercial samples are under study. The main objective is to alter the gas selectivity from propylene to the desired C_4 and C_5 olefins. The initial results of these studies are detailed in the May and June monthly reports and the Quarterly Technical Status Report, Third Quarter, Fiscal Year, 1992. Further tests at an additional process condition (0.9 catalyst/oil weight ratio, 880°F) are now available. The wax conversion levels, (the sum of C_4- gas, gasoline and coke) of the various catalysts vary with the catalyst/oil ratio. (Figure 1) The rare earth oxide treated HZSM-5 samples and the Isocat material are lower in conversion than the other two commercial HZSM-5 samples, Intercat Corp.'s ZCatPlus and Davison's OH-S. This variation in activity does not correlate directly with the surface area of the samples. The results from nitrogen adsorption measurements (Table I) show similar BET surface areas for the catalysts. These measurements probably reflect the relative zeolite contents of the samples, which are about 25%. The catalytic activity (wax conversion) is a function of the available Bronsted acid site concentration, or framework aluminum content. This explains why the

Isocat sample has a lower catalytic activity. It has a higher silica to alumina ratio (low framework alumina) HZSM-5 zeolite. Since the rare earth samples also show lower activity, it is likely that the impregnation treatment has lowered the framework aluminum (acidity level) content. One method to determine the concentration of such active aluminum framework sites is to re-exchange the zeolite samples with ammonium ions and then to thermally treat the sample and titrate the evolved ammonia gas. The total number of sites and their relative strengths (the temperature of ammonia evolution is a measure of the strength of the acid site) can be determined in this manner. The results for temperature programmed desorption (TPD) measurements for fresh and steamed HZSM-5, the ZCatPlus sample, are shown in Figure 2. The steam treatment lowers the overall acid site concentration (~50%) and changes the distribution of the sites. Further studies of this type will relate such physical measurements of the catalysts to their wax conversion performance.

Another key aspect of these various HZSM-5 samples is to observe changes in product selectivity among the light gas and gasoline products. Figures 3-6 show plots of various product yields as a function of wax conversion. There are no major selectivity differences in product yields among these HZSM-5 samples. The Isocat sample has lower propylene and isobutylene yields than the other HZSM-5 samples but the differences are small. The total aromatic and olefin contents of the gasoline from the various catalysts vary with conversion. (Figures 7,8) At high conversion levels, (>90%), the olefins convert to aromatics. The variability in these measurements, especially at the higher conversion levels, would require additional test runs to verify these small changes.

This study of various HZSM-5 samples shows that activity (wax conversion levels) differences are readily apparent. However, product selectivity changes are small and difficult to measure accurately.

A series of rare earth exchanged zeolite Y catalysts are under study to determine the effects of rare earth ions on olefin selectivities. These catalysts have similar zeolite and matrix levels and vary only in rare earth content. The initial wax cracking test results and analytical data for these samples are found in the June, 1992 Monthly Progress Report and the Quarterly Technical Status Report, Third Quarter, Fiscal Year, 1992. For convenience, the analytical results are presented in Table II. Further wax cracking tests of these samples were carried out at two severity levels, catalyst to oil ratios of 0.75 and 0.1875 at 880°F. Figure 9 summarizes these tests. There are wide variations in the wax conversion levels (the sum of C_5-430°F naphtha, C_4- gas and coke) of the various samples. A multiple number (4-6) of tests runs on each of these samples did not resolve these variations. This lack of precision is especially noticeable at the test conditions of low catalyst to oil ratio (0.1875) and the low reaction temperature of 880°F. These lower wax conversion conditions may result in incomplete vaporization of the heavy wax feedstock, resulting in the wide swings in conversion. This situation will be studied further. Test runs at higher reaction temperature may provide an answer to this puzzling variability.

These multiple wax cracking test runs with the variable rare earth Y zeolite catalysts do represent an excellent source of product selectivity

information. Figures 10-16 present the conventional product yield versus conversion plots for all of these runs. It is difficult to distinguish among the various catalysts due to the scatter in the data. One approach to resolving this difficulty is to plot selected points from the power law equations that have been used to correlate the data for the various catalysts. Plots of this type (Figures 17-20) show that two of the high rare earth samples (1704,1705) have the expected "rare earth effect": higher gasoline and lower olefin yields. The other high rare earth sample (1706) has a product selectivity more like the low or no rare earth samples. Further analytical and wax cracking tests of these samples, especially the 1706 material, will continue.

These wax cracking test results confirm the findings of other workers with regular gas oil feedstocks that "zero" rare earth FCC catalysts provide the maximum yields of the desirable light olefins.

Several pilot plant runs of the catalytic cracking of Fischer-Tropsch wax show very high conversion levels. (>85-90%) The details of these runs have been reported in earlier reports. (see Quarterly Technical Status Reports for First and Second Quarter Fiscal Year, 1992, Report Nos. 10, 13).

These high conversion levels may not provide the optimum light olefin yields. The zeolite content is the major factor that affects catalyst activity. A series of FCC catalysts with low zeolite content (~10%) should address this issue with lower catalytic activity.

Table III presents the nominal compositions of several catalyst samples, with either zeolite Y or Beta and a low activity matrix. These preparations use conventional raw materials and experimental techniques. The BET surface areas of these catalysts, after a steam treatment at 1450°F, 5 hours, are listed in Table IV. The surface areas and pore volumes decrease with the lower zeolite levels, as expected. Figure 21 illustrates the differences in nitrogen pore size distribution among the catalysts. The 10% Beta zeolite catalyst (69 m2/g) has a significantly lower surface area than the 10% Y zeolite sample (90 m2/g). Similar differences are evident for the 40% Y and Beta samples. This may indicate that the Beta zeolite has a lower hydrothermal stability than the Y zeolite sample. The two different surface area results (Table IV) for the 40% Y sample (14040-48-1,-2) illustrates the variability in the steaming treatments. The influence of the matrix composition on zeolite stability is another issue that will be addressed with future catalyst variations.

The wax conversion properties of these new catalyst samples are currently under study.

TASK 4. Pilot Plant Tests.

No work in this area was performed this Quarter.

CONCLUSIONS

Task 1 of the contract, the Project Management Plan, and Task 2, Feedstock Characterization are complete.

Activities under Task 3, Screening Catalytic Cracking Tests, continue this Quarter.

The catalytic cracking results of both the small scale test unit (MYU) and the pilot plant indicate that the wax feedstock readily converts (>85%) to product gas (C_4-) and gasoline (C_5-430°F). The HZSM-5 zeolite catalyst produces the highest yields of the target isobutylene and isoamylenes. The beta zeolite and the amorphous catalyst have lower light olefin yields than the HZSM-5 sample but greater olefin yields than the Y zeolite. These high olefin yields come at the expense of gasoline product. The Y zeolite has the highest gasoline yields among the catalysts tested. Further catalytic cracking tests of both commercial and experimental HZSM-5 catalysts showed that product selectivity differences are small and difficult to measure. Two HZSM-5 samples, the rare earth impregnated samples and the commercial Isocat material are lower in wax conversion than two other commercial HZSM-5 samples. The catalytic cracking of the wax feedstock for a series of rare earth exchanged Y zeolite catalysts show a "rare earth effect": higher naphtha and lower olefin yields with higher rare earth levels. A series of FCC catalysts with low zeolite content (10%) with either Beta or Y zeolites have been prepared. The physical properties of these samples after hydrothermal treatment are consistent with a lower hydrothermal stability for Beta zeolite than for Y zeolite.

These variations in product yields will form the basis for economic evaluations of the various process and catalyst options that will follow in later stages of this project.

ACKNOWLEDGEMENT

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NOTICE

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Naperville, Illinois

TABLE I

B.E.T. SURFACE AREAS OF STEAMED HZSM-5 SAMPLES

Steam Treatment: 1450°F, 5 Hours, 100% Steam

Sample ID	Total B.E.T. Surface Area (m^2/g)	Zeolite Surface Area (m^2/g)	Matrix Surface Area (m^2/g)
ZCatPlus (#1)	70	39	31
ZCatPlus (#2)	70	30	40
1% ReO/ZCatPlus	63	41	22
2% ReO/ZCatPlus	62	38	24
3% ReO/ZCatPlus	60	42	18
OH-S (#1)	54	31	23
OH-S (#2)	56	25	31
Isocat	53	30	23

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3/8/93

TABLE II

DESCRIPTION OF RARE EARTH OXIDE/FAUJASITE CATALYSTS

Catalyst I.D.	Wt% Rare Earth	Steam Treatment
CCC-1701	<0.04	1450°F, 8.5 hours, 100% steam
CCC-1702	0.27	1450°F, 8.5 hours, 100% steam
CCC-1703	0.34	1450°F, 8.5 hours, 100% steam
CCC-1704	0.96	1450°F, 8.5 hours, 100% steam
CCC-1705	1.49	1450°F, 16 hours, 100% steam
CCC-1706	1.49	1450°F, 8 hours, 100% steam

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3/8/93

TABLE III

CHEMICAL COMPOSITIONS OF ZEOLITE FCC CATALYSTS

Catalyst ID	Nominal Composition	Comment
9669-146-148	40% Beta zeolite 20.8% Ludox AS-40 (silica sol) 4.2% Catapal alumina 35% Kaolin clay	Standard Beta
9669-152	40% LZY-84 zeolite 20.8% Ludox AS-40 (silica sol) 4.2% Catapal alumina 35% Kaolin clay	Standard Y
9669-153	34.7% Ludox AS-40 (silica sol) 7% Catapal alumina 58.3% Kaolin clay	Matrix only
9669-154	10% LZY-84 20.8% Ludox AS-40 (silica sol) 4.2% Catapal alumina 65% Kaolin clay	Low Y zeolite
9669-155	10% Beta 20.8% Ludox AS-40 (silica sol) 4.2% Catapal alumina 65% Kaolin clay	Low Beta

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3/8/93

TABLE IV

PHYSICAL PROPERTIES OF ZEOLITE FCC CATALYSTS

Steam Treatment: 1450°F, 5 hours, 100% steam

		BET Surface Area, m^2/g			
Catalyst ID	Description	Total	Zeolite	Matrix	Micropore Volume (cc/g)
14059-81-4	40% Beta	211	143	68	.066
14040-48-1	40% Y	253	183	70	.084
14040-48-2	40% Y	233	167	66	.077
14040-50-5	Matrix	55	3.0	52	.001
14040-50-6	10% Y	90	48	42	.022
14040-50-7	10% Beta	69	19	50	.009

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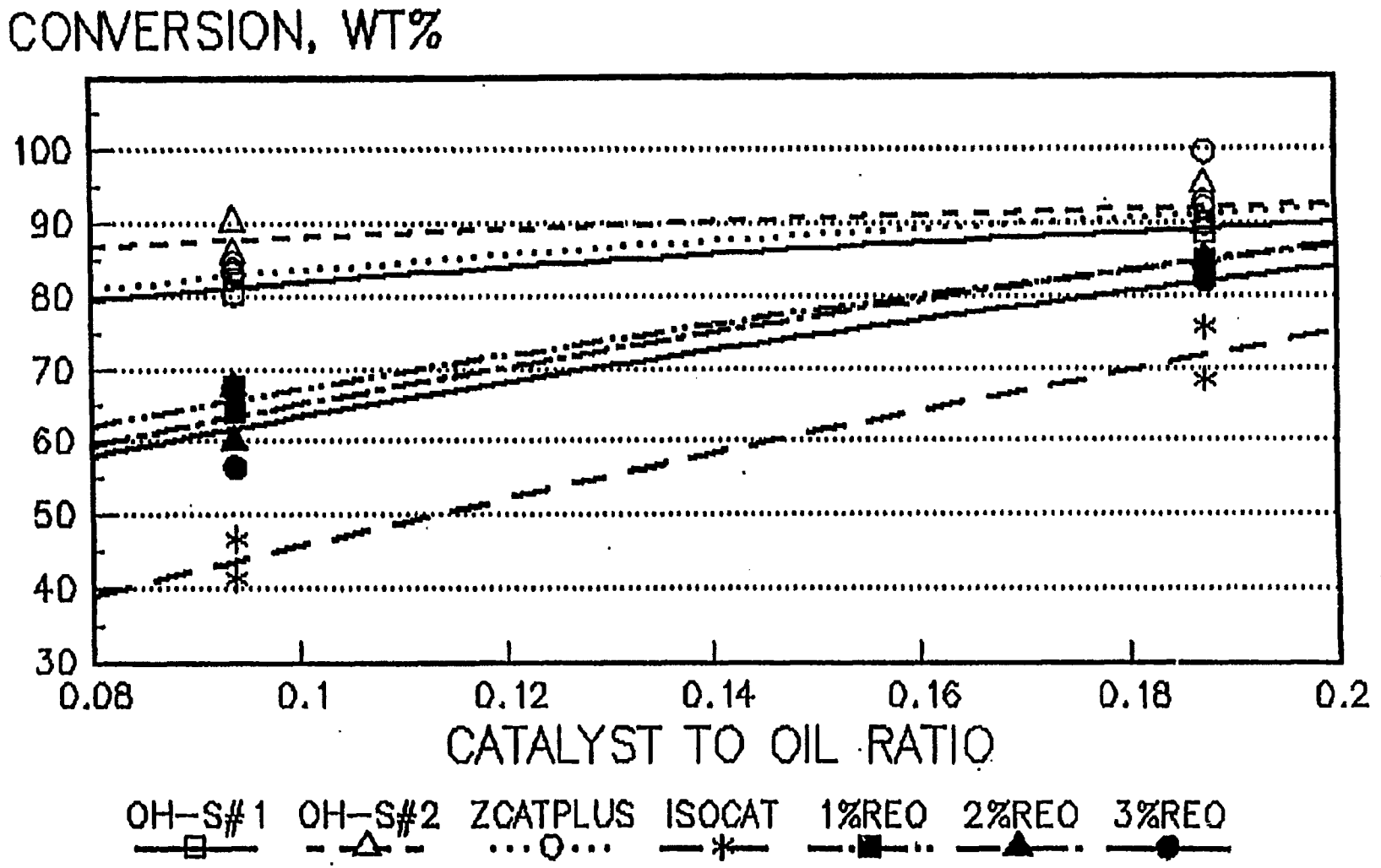
FIGURE 1

FISCHER-TROPSCH WAX CATALYTIC CRACKING ACTIVITY OF HZSM-5 SAMPLES



(17)

FIGURE 2

TPD AMMONIA OF HZSM-5 SAMPLES

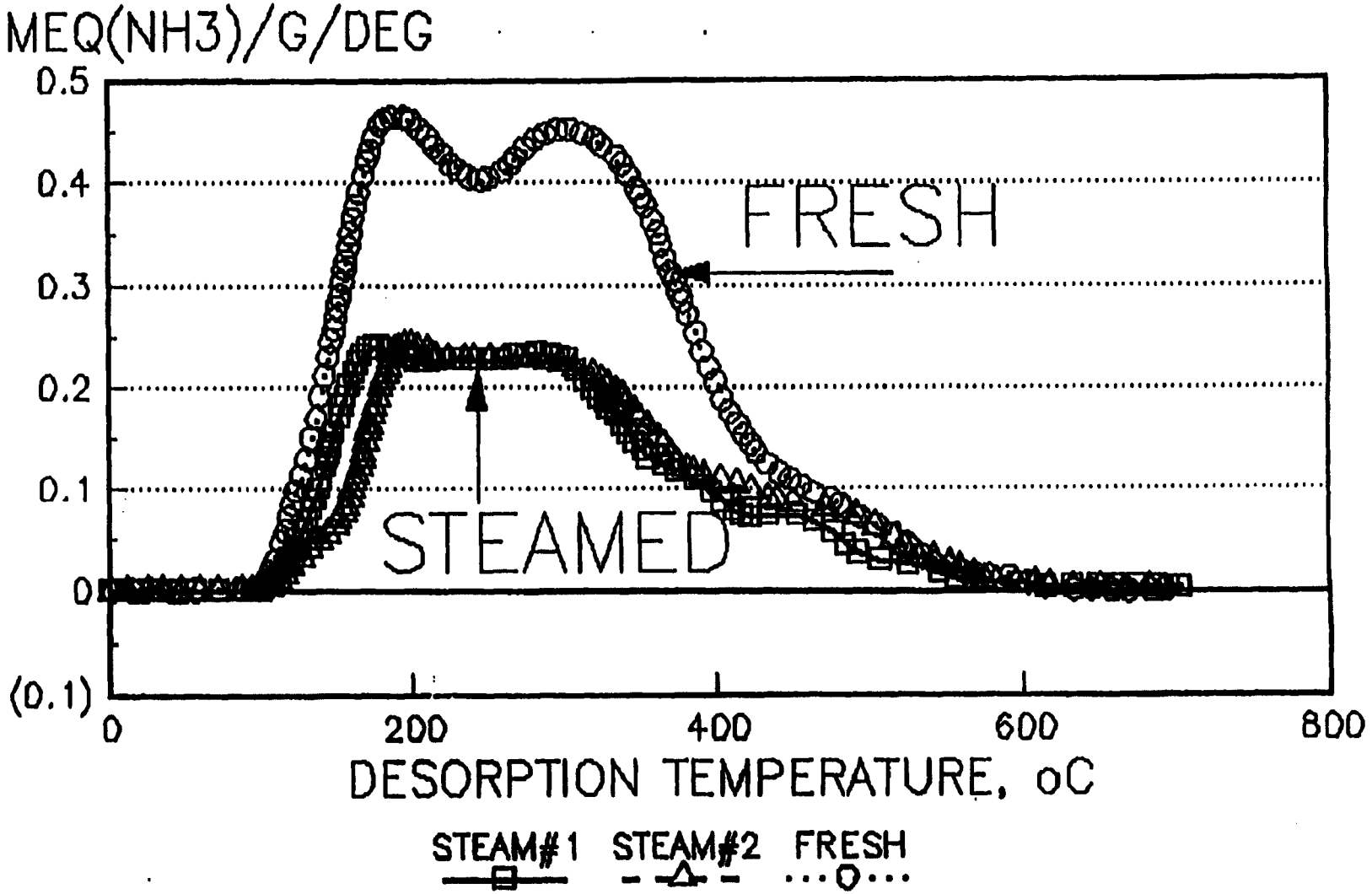


FIGURE 3

PRODUCT SELECTIVITIES OF HZSM-5 SAMPLES

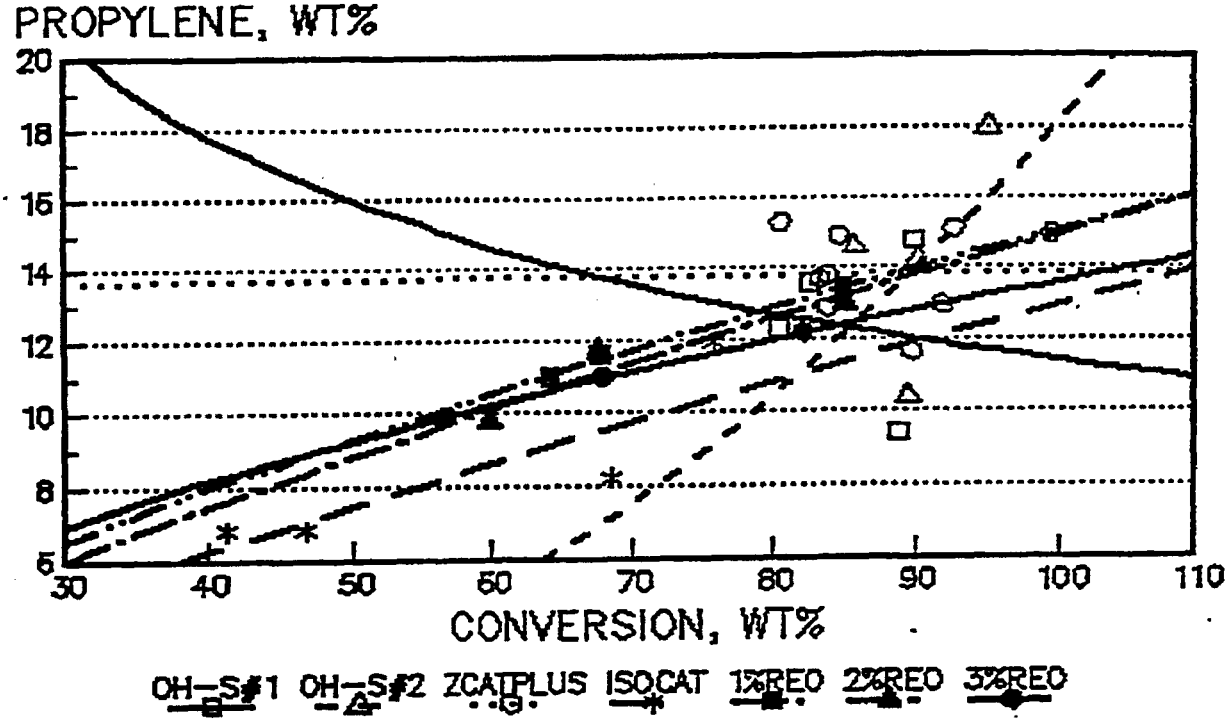


FIGURE 4

PRODUCT SELECTIVITIES OF HZSM-5 SAMPLES

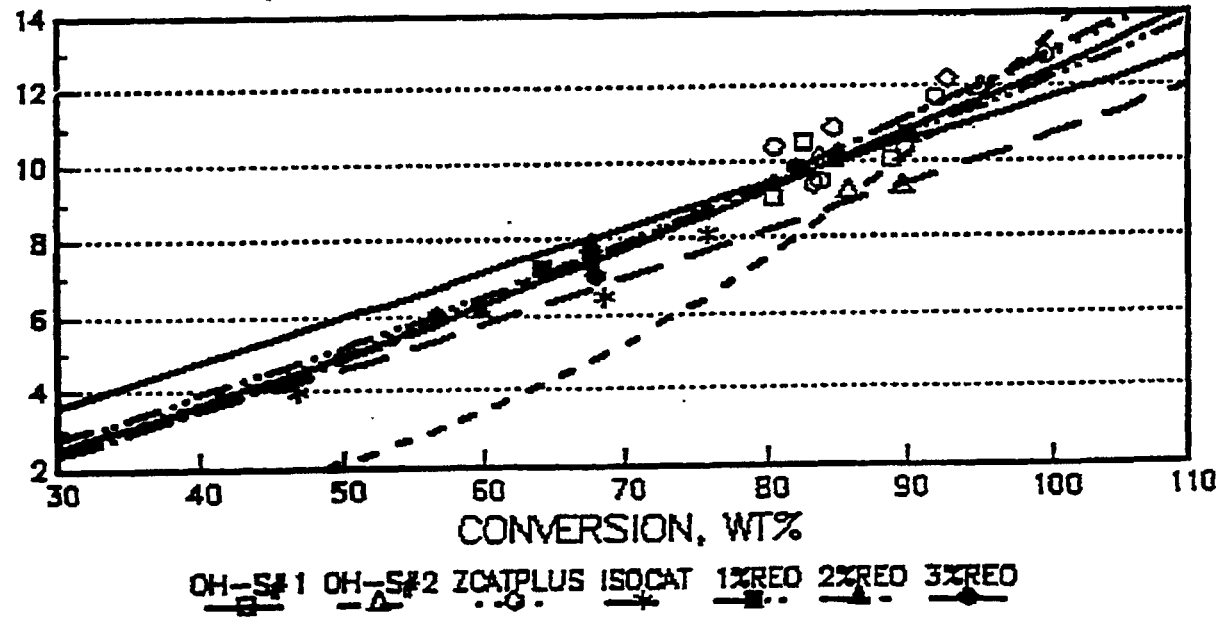


FIGURE 5

PRODUCT SELECTIVITIES OF HZSM-5 SAMPLES

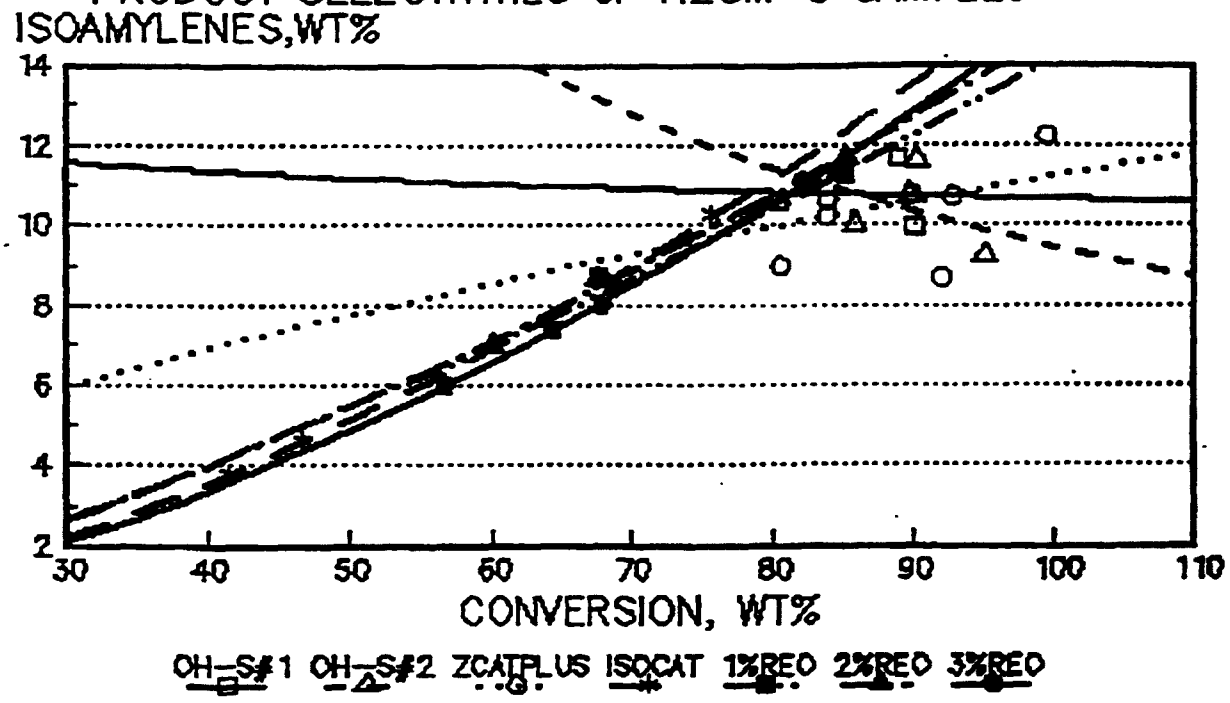


FIGURE 6

PRODUCT SELECTIVITIES OF HZSM-5 SAMPLES

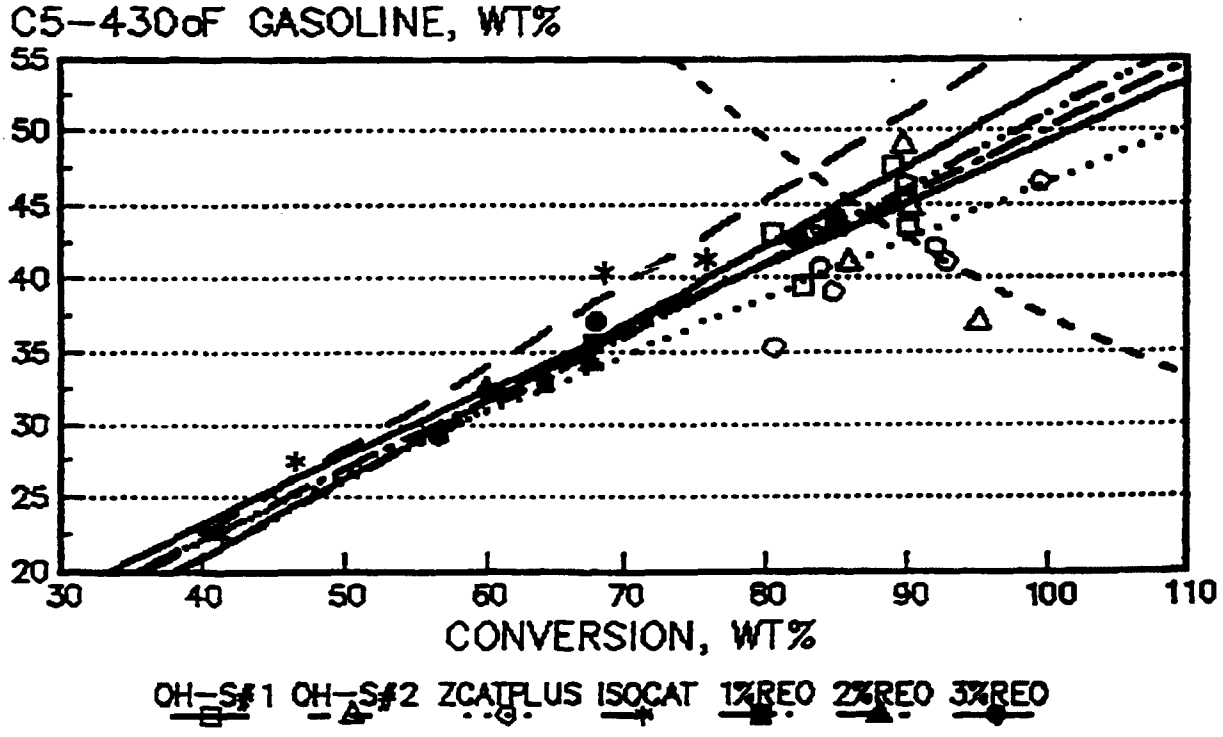


FIGURE 7
PRODUCT SELECTIVITIES OF HZSM-5 SAMPLES

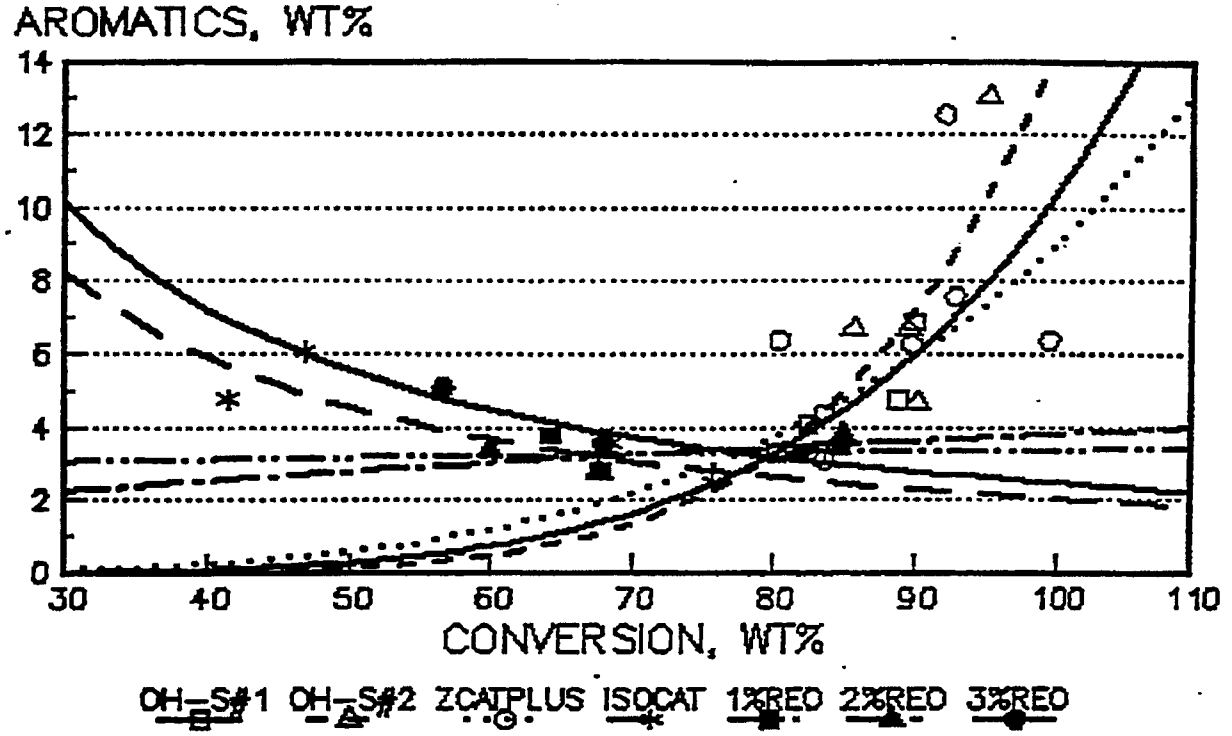


FIGURE 8
PRODUCT SELECTIVITIES OF HZSM-5 SAMPLES

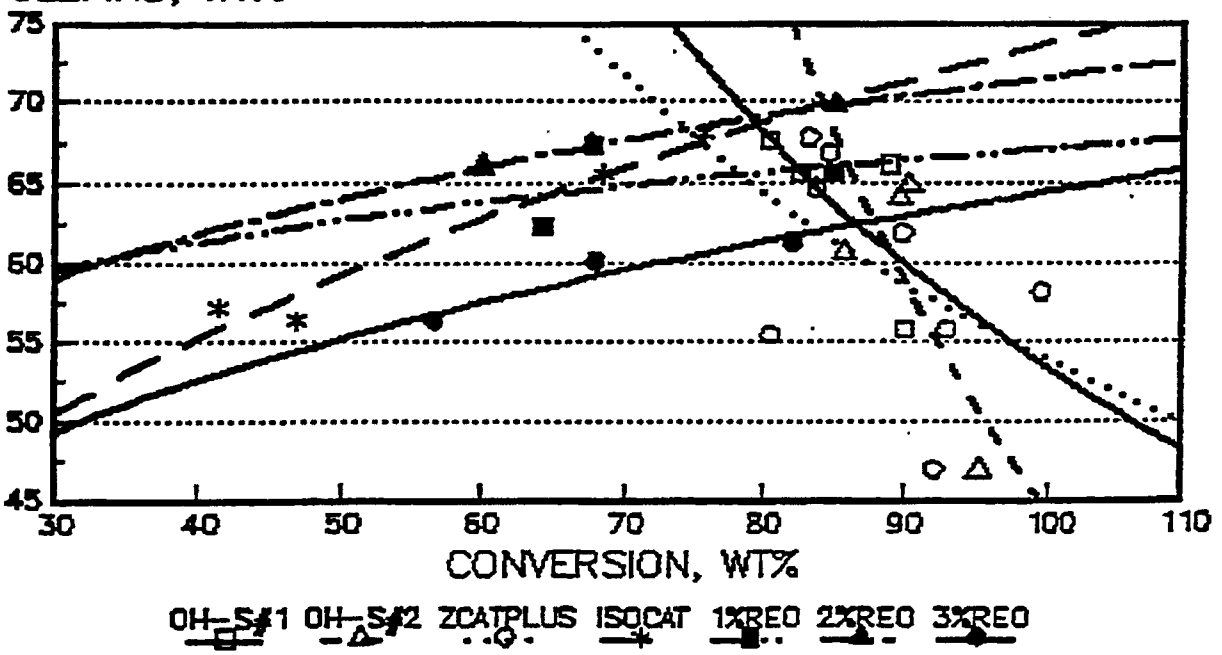


FIGURE 9

FISCHER-TROPSCH WAX CATALYTIC CRACKING
VARIABLE RARE EARTH Y ZEOLITE CATALYSTS

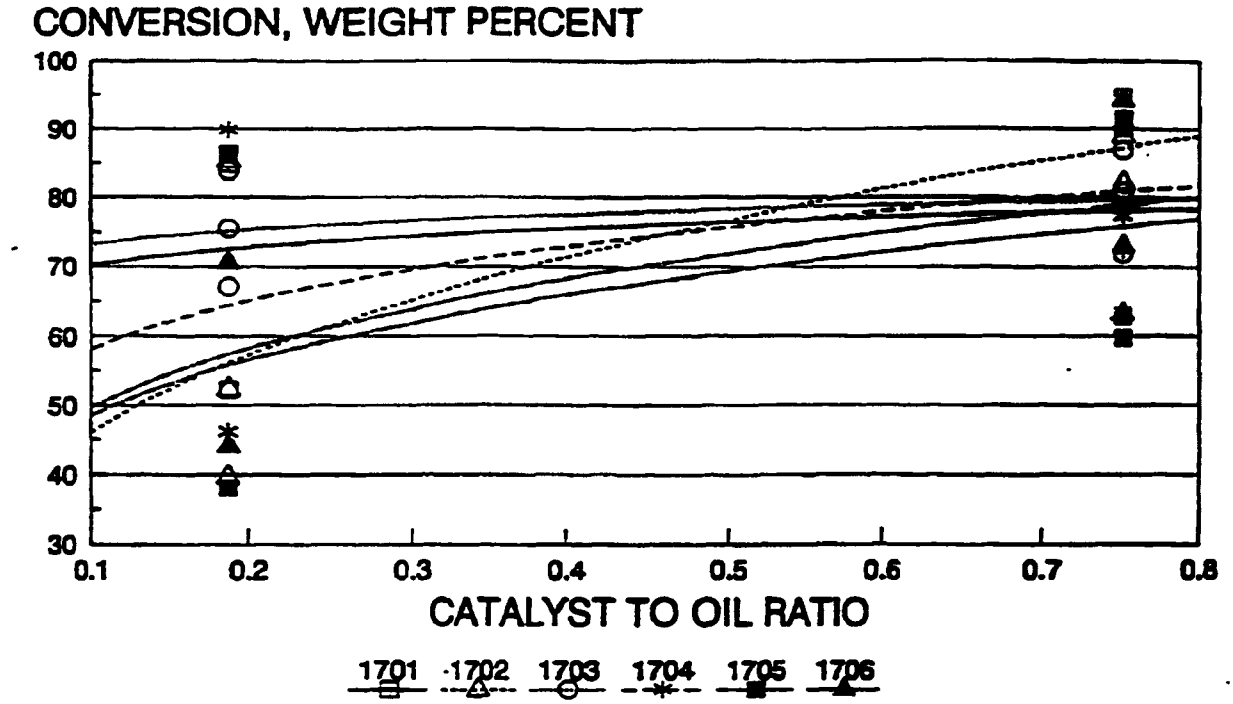


FIGURE 10

FISCHER-TROPSCH WAX CATALYTIC CRACKING
VARIABLE RARE EARTH Y ZEOLITE CATALYSTS

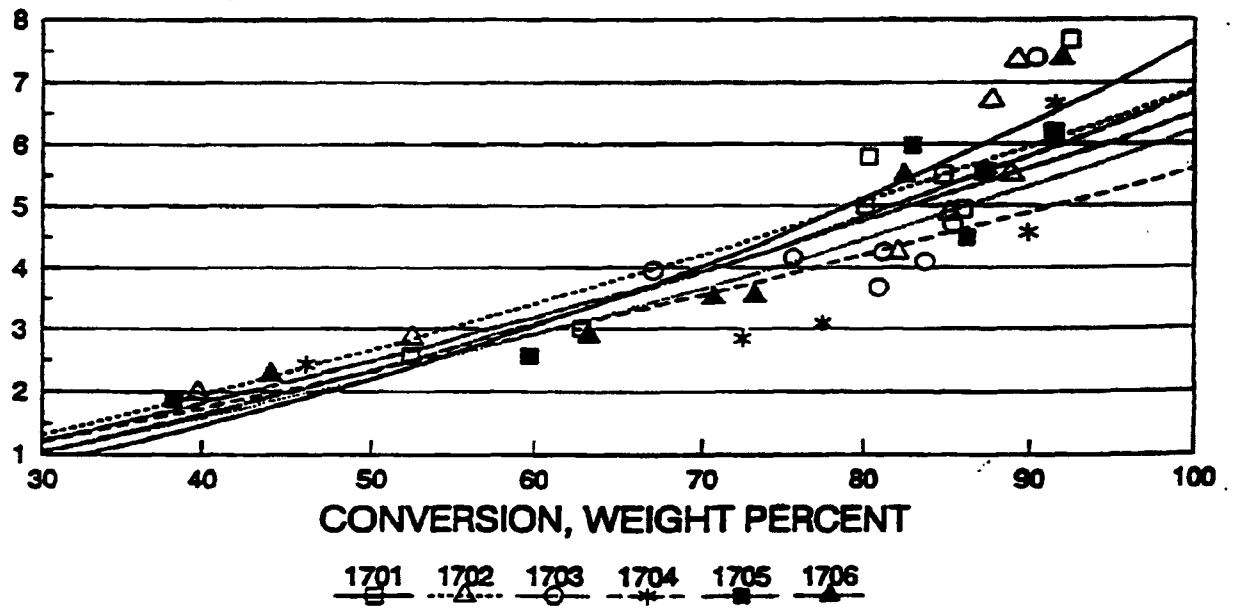


FIGURE 11

FISCHER-TROPSCH WAX CATALYTIC CRACKING

VARIABLE RARE EARTH Y ZEOLITE CATALYSTS

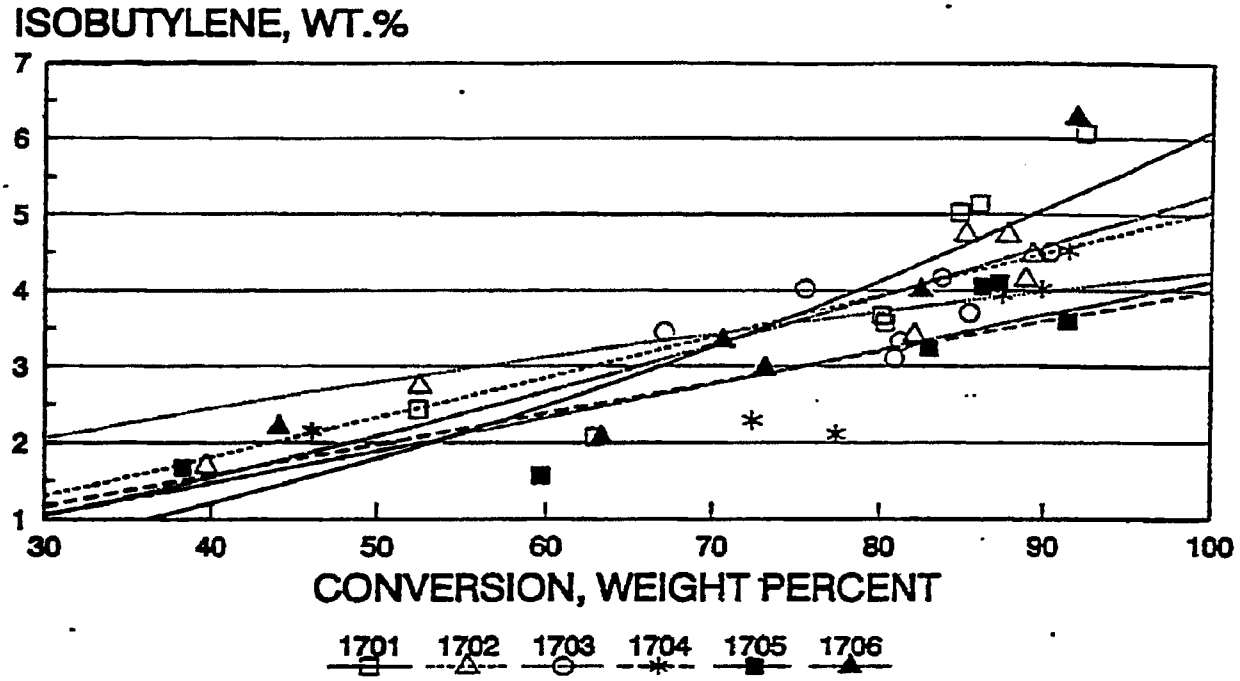


FIGURE 12

FISCHER-TROPSCH WAX CATALYTIC CRACKING

VARIABLE RARE EARTH Y ZEOLITE CATALYSTS

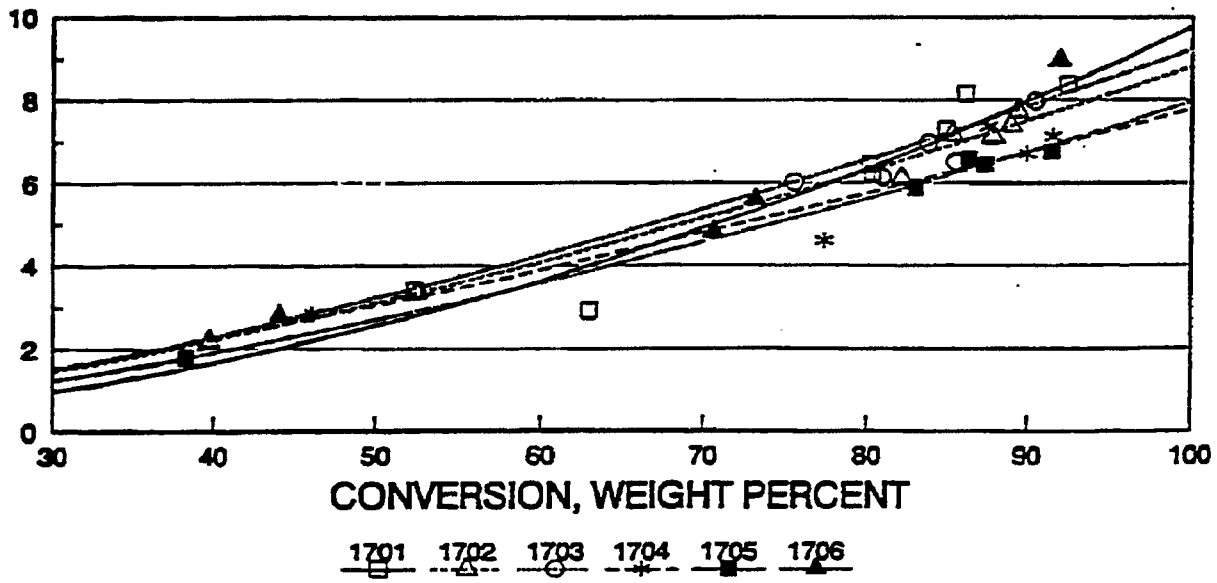


FIGURE 13
FISCHER-TROPSCH WAX CATALYTIC CRACKING
VARIABLE RARE EARTH Y CATALYSTS

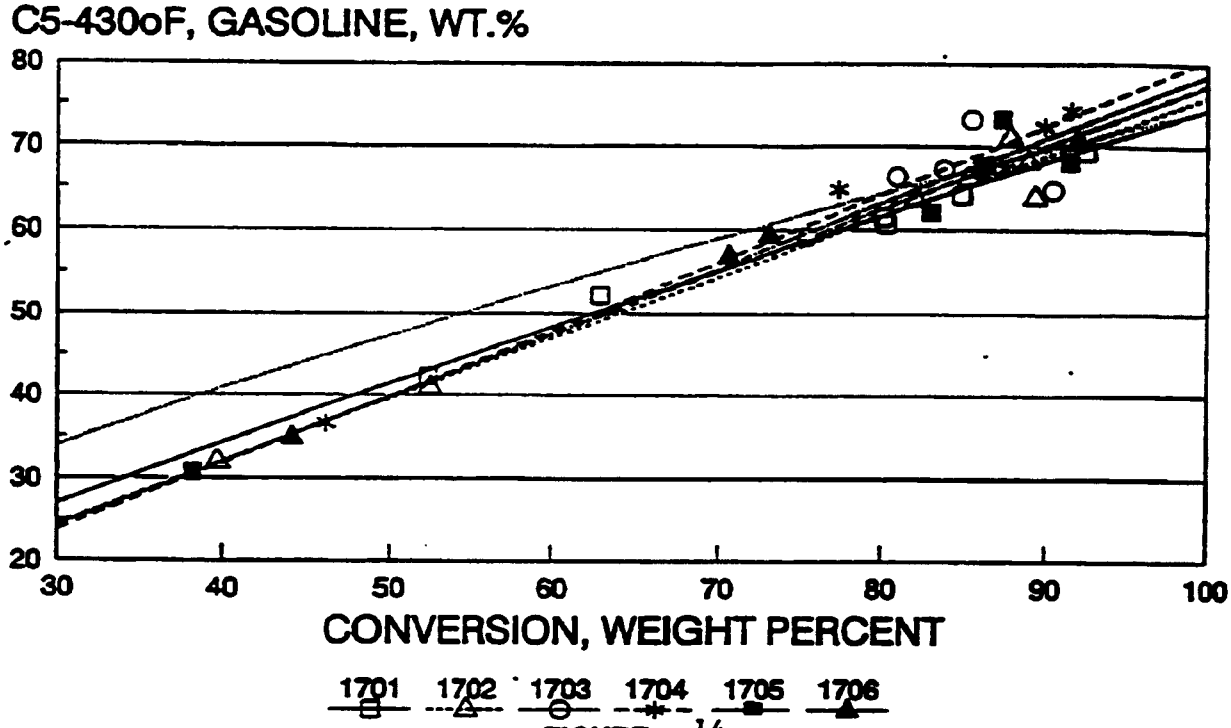


FIGURE 14
FISCHER-TROPSCH WAX CATALYTIC CRACKING
VARIABLE RARE EARTH Y CATALYSTS

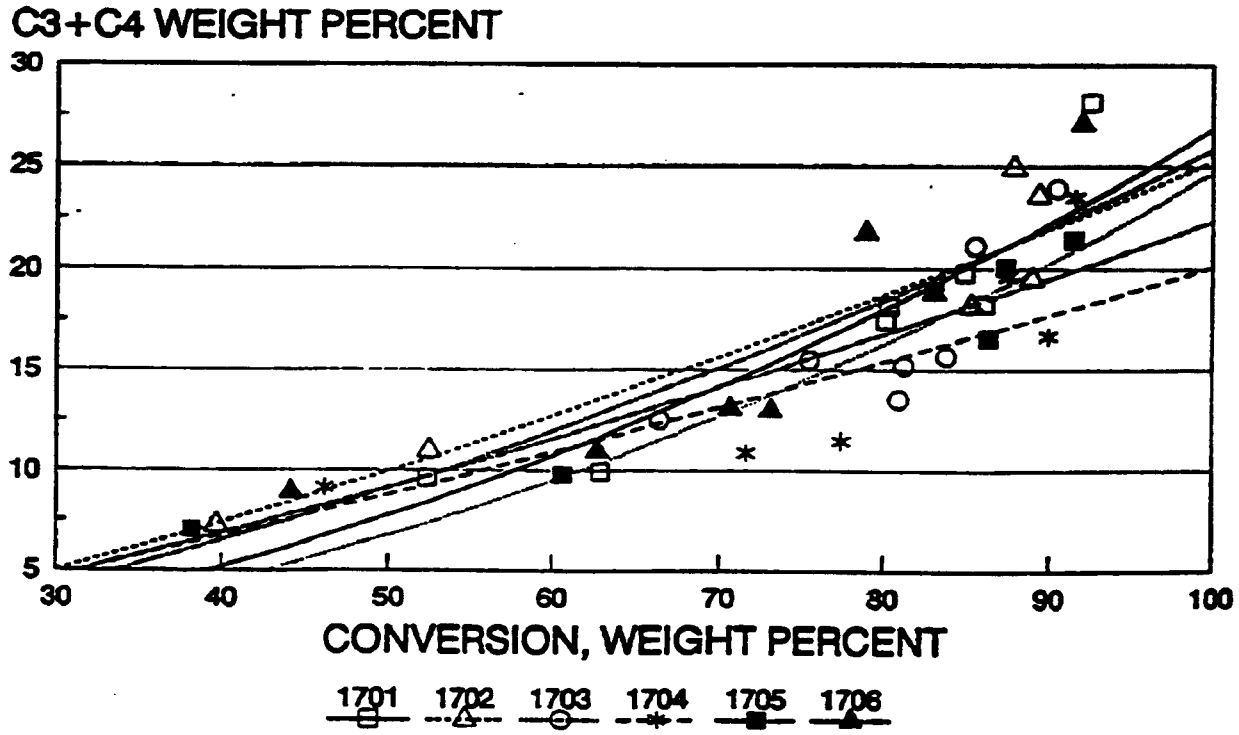


FIGURE 15
FISCHER-TROPSCH WAX CATALYTIC CRACKING
VARIABLE RARE EARTH Y CATALYSTS

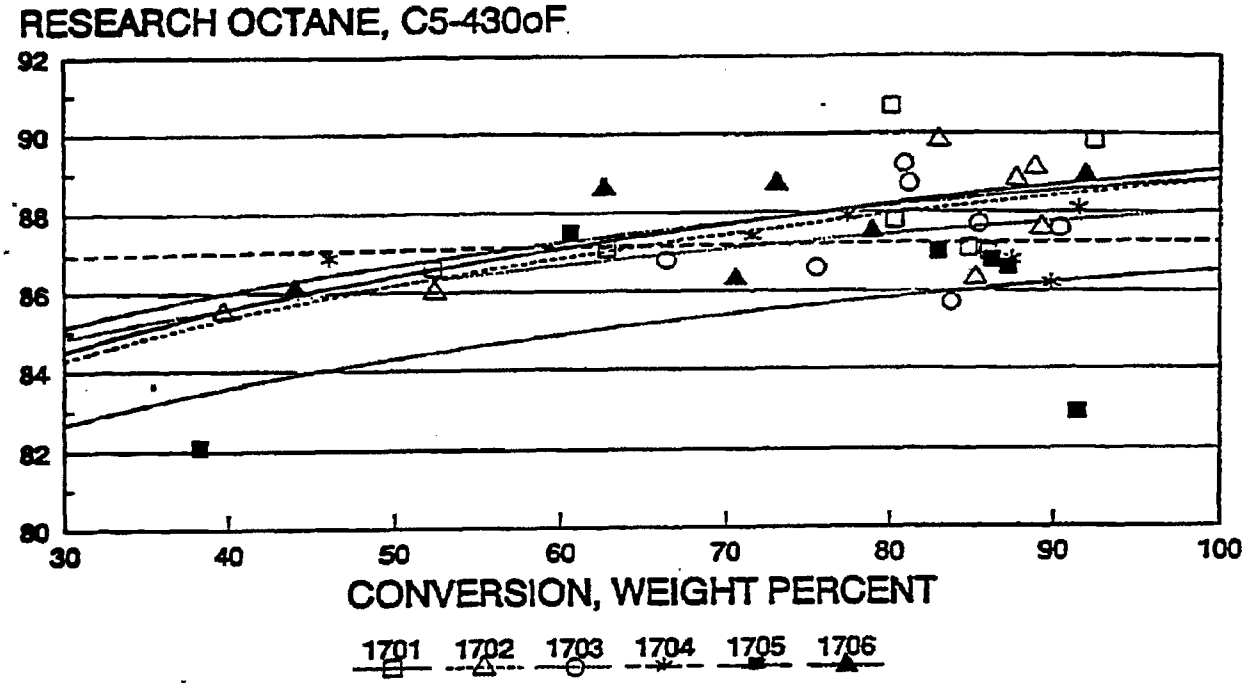


FIGURE 16
FISCHER-TROPSCH WAX CATALYTIC CRACKING
VARIABLE RARE EARTH Y CATALYSTS

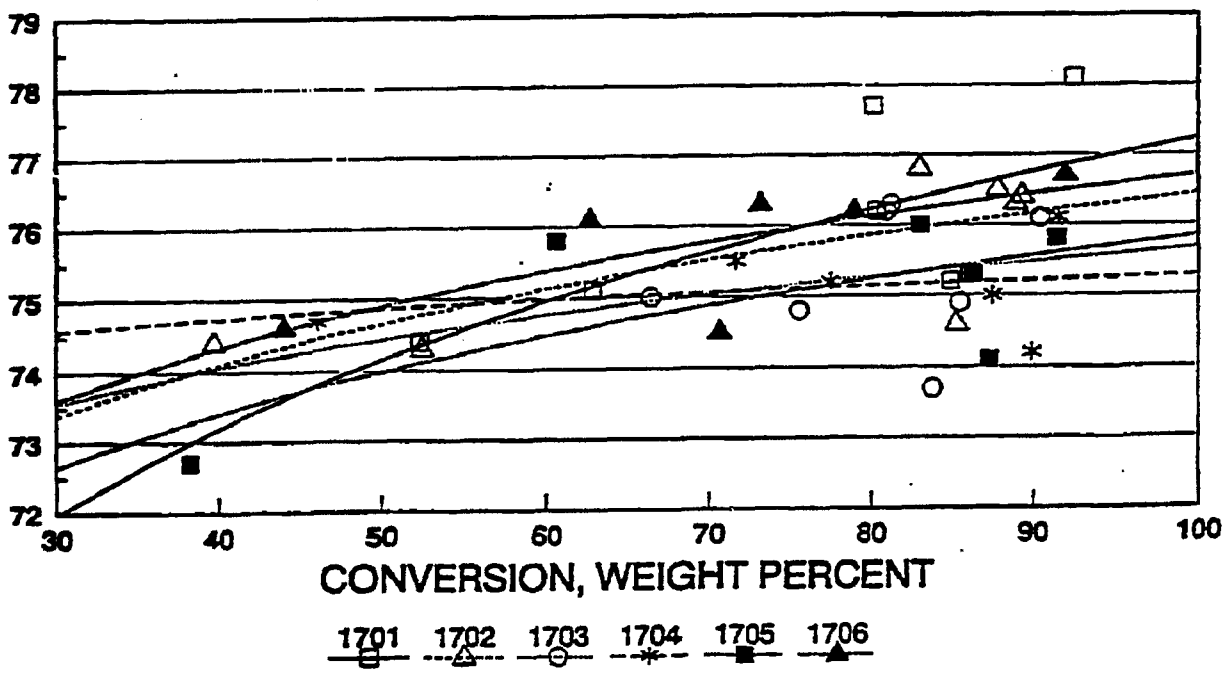


FIGURE 17
FISCHER-TROPSCH WAX CATALYTIC CRACKING
RARE EARTH Y CATALYSTS-INTERPOLATED DATA

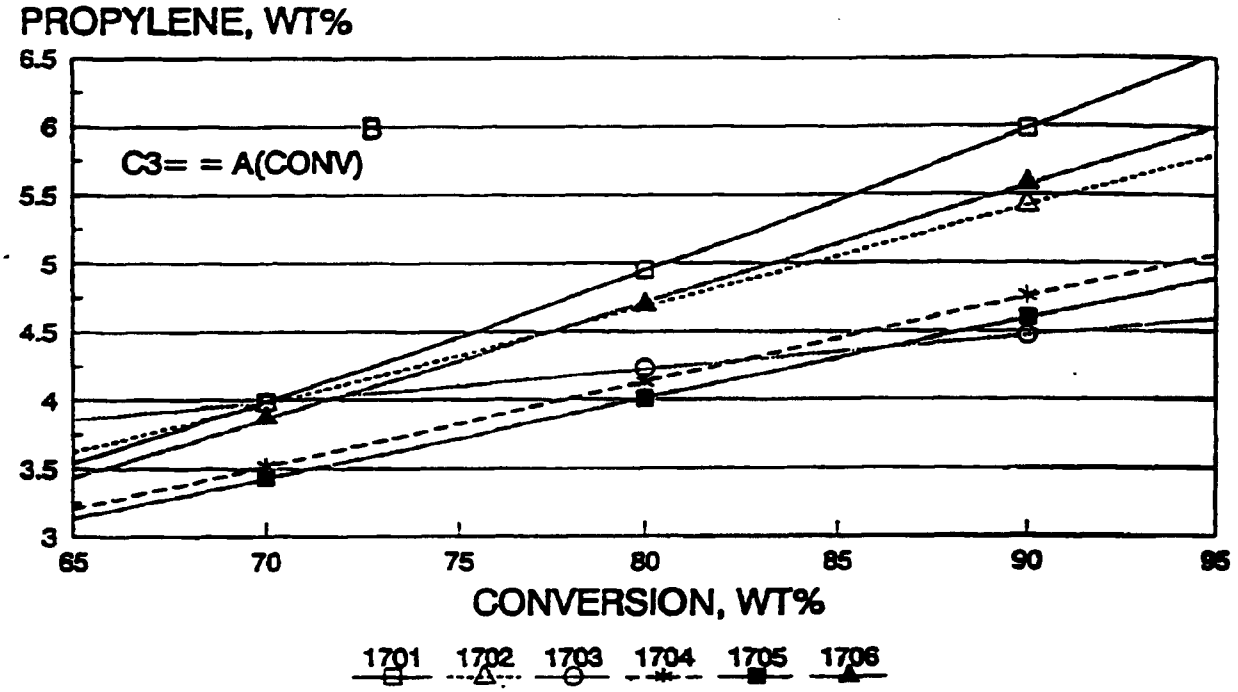


FIGURE 18
FISCHER-TROPSCH WAX CATALYTIC CRACKING
RARE EARTH Y CATALYSTS-INTERPOLATED DATA

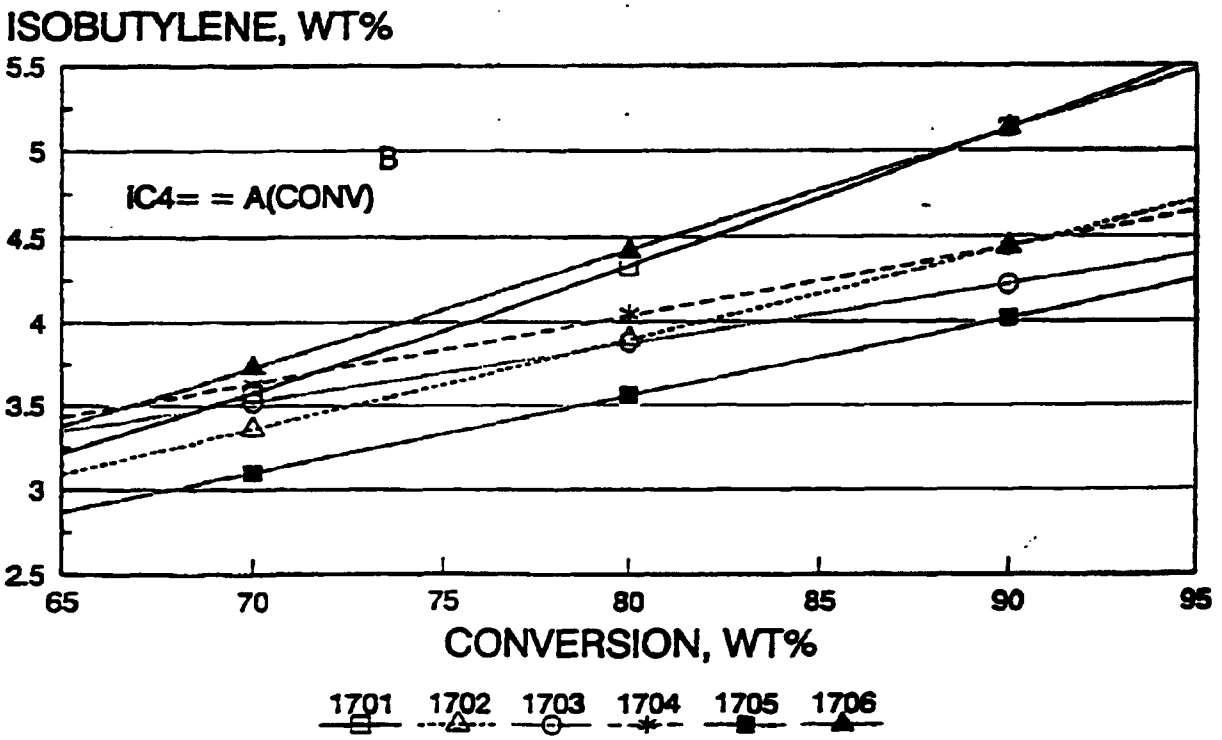


FIGURE 19
FISCHER-TROPSCH WAX CATALYTIC CRACKING
RARE EARTH Y CATALYSTS-INTERPOLATED DATA

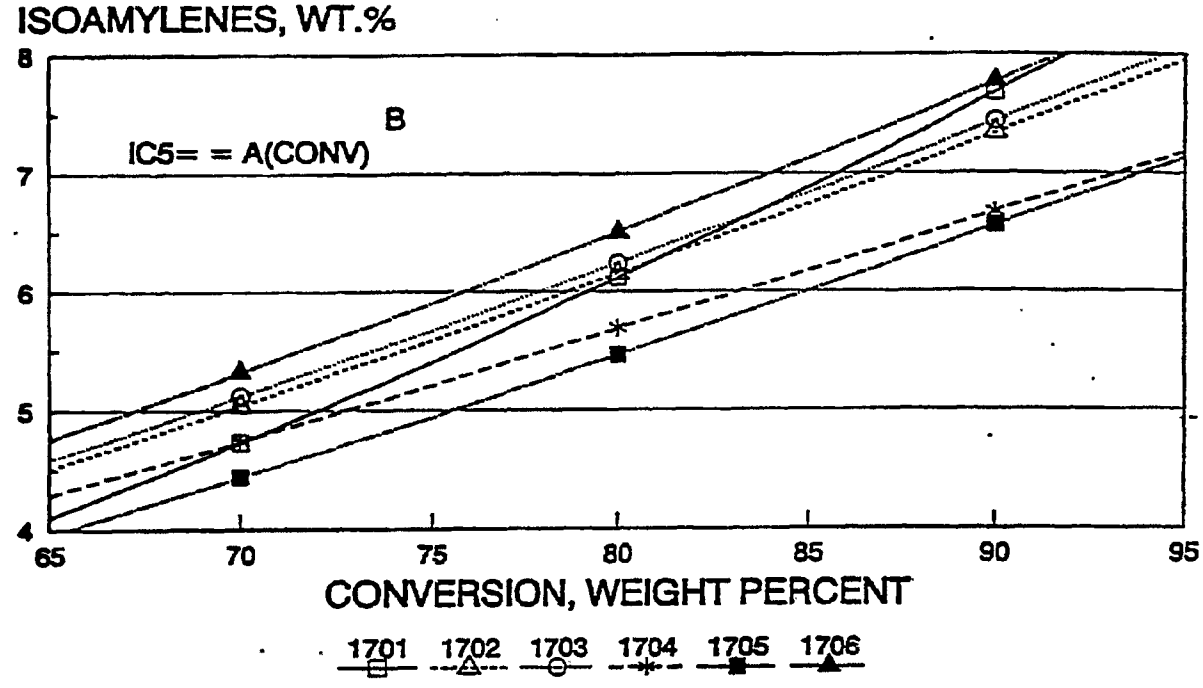


FIGURE 20
FISCHER-TROPSCH WAX CATALYTIC CRACKING
RARE EARTH Y CATALYSTS-INTERPOLATED DATA

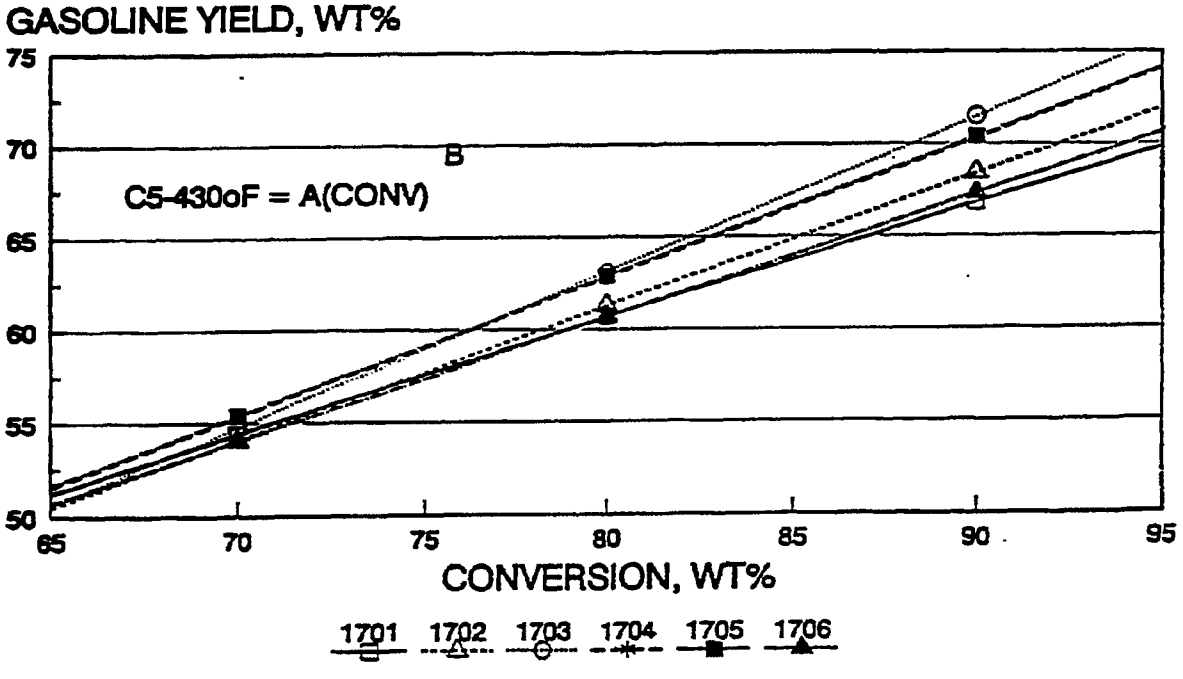
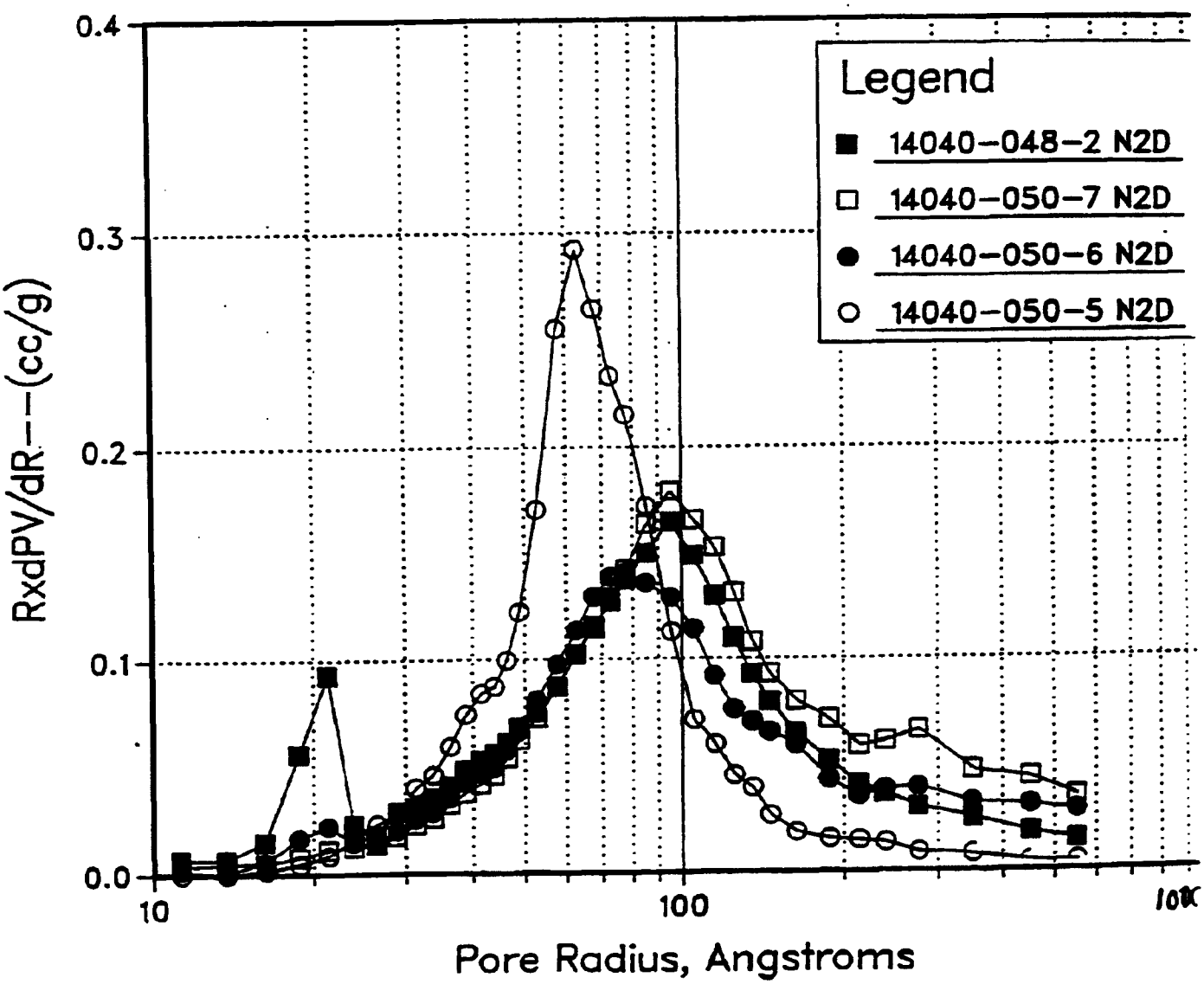


FIGURE 21

NITROGEN PORE SIZE DISTRIBUTION
STEAMED ZEOLITE CATALYSTS

Weighted Differential Pore Volume



QUARTERLY MANPOWER REPORT

For FOURTH QUARTER FISCAL YEAR, 1992

(July 1, 1992 - September 30, 1992)

TITLE: THE SELECTIVE CATALYTIC CRACKING OF FISCHER-TROPSCH LIQUIDS TO HIGH VALUE TRANSPORTATION FUELS

IDENTIFICATION NUMBER: DE-AC22-91PC90057

START DATE: July 1, 1992
COMPLETION DATE: September 31, 1992

PARTICIPANT NAME AND ADDRESS:

AMOCO OIL COMPANY
P. O. BOX 3011
NAPERVILLE, ILLINOIS 60566

	Manpower In Hours by Task							
Name	1	2	3	4	5	6	7	Total
W. J. Reagan	0	0	157	119	102	0	0	378
D. M. Washecheck	0	0	16	5	5	0	0	26
G. G. Glasrud	0	0	0	0	0	0	0	0
Other Professionals	0	0	0	0	0	0	0	0
Technical Support	0	0	262	56	56	0	0	374
Secretarial	0	0	3	3	2	0	0	8
Total Hours	0	0	438	183	165	0	0	786

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Technical Information Service
Springfield, VA 22161 (703) 605-6000

DE95001939

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