NEW CATALYSTS FOR THE INDIRECT LIQUEFACTION OF COAL. QUARTERLY TECHNICAL REPORT, NOVEMBER 1, 1982-JANUARY 31, 1983

VIRGINIA COMMONWEALTH UNIV., RICHMOND.
DEPT. OF CHEMISTRY

1983
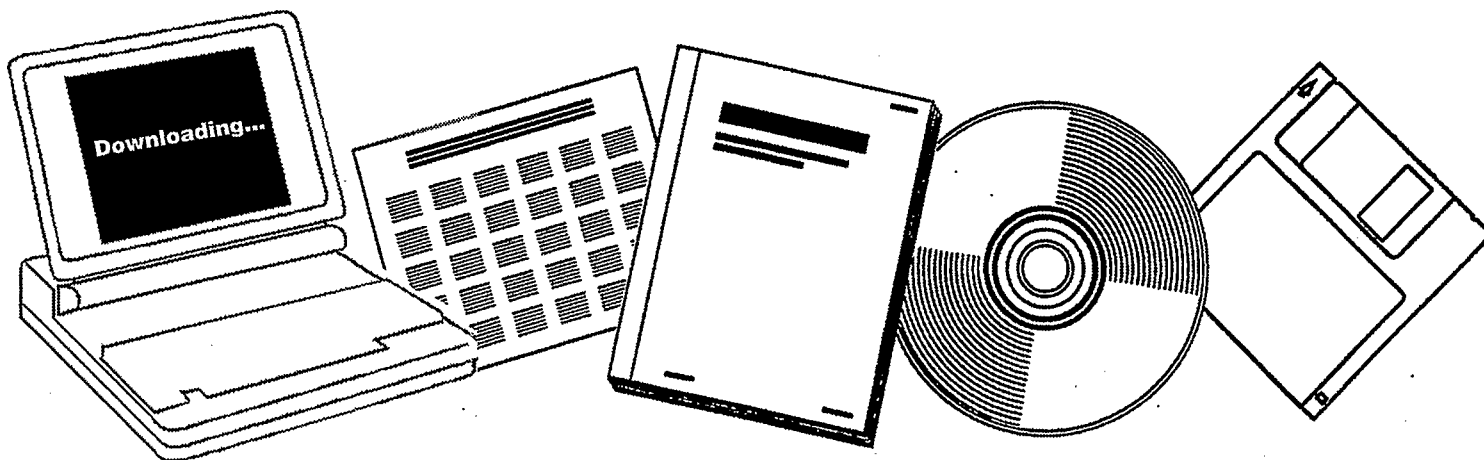
NEW CATALYSTS FOR THE INDIRECT LIQUEFACTION OF COAL

Quarterly Technical Report

Grant No: DE-FG22-80PC30228

Period Covered: November 1, 1982 - January 31, 1983

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MASTER

Abstract

Some ZSM-5 supported ruthenium (Ru/ZSM-5) catalysts have been prepared and characterized. The catalysts were prepared from $Ru_3(CO)_{12}$ and the acid form of ZSM-5 by the extraction procedure. Characterization of the catalysts has been achieved by using infrared spectroscopy, X-ray powder diffractometry, X-ray photoelectron spectroscopy, ion-scattering spectrometry and chemisorption. The chemical composition of the catalysts has been identified.

Ru/ZSM-5 catalysts with weight % ruthenium loading from ~1% to ~8% have been prepared from $Ru_3(CO)_{12}$ and the acid (H$^+$) form of ZSM-5 by the extraction technique (EX) or by dry grinding (DG). As-prepared (EX) catalysts are designated AP, those calcined in air at 500°C are designated H-500. The catalysts have been characterized by a variety of techniques.

1. Infrared spectroscopy

Infrared (IR) spectroscopy was used throughout this study to identify the presence of certain functional groups in the samples. Initially, IR spectra were obtained for the starting materials of the catalyst preparation. In Table 1, the major IR bands are listed for typical samples of $Ru_3(CO)_{12}$, NH_4^+-ZSM-5 and H$^+$-ZSM-5. The $Ru_3(CO)_{12}$ has bands in the region for terminal carbonyl groups, $\gamma(CO)$ of approximately 2100 to 2000 cm^{-1}. This $Ru_3(CO)_{12}$ spectrum is in agreement with that reported in the literature. Table 1, also, includes the major IR bands for ZSM-5; the ZSM-5 samples are in their NH_4^+ form and H$^+$ form with one sample heated to 538°C and the other sample heated to 350°C. The spectra of the two heated samples are similar, indicating that both procedures removed the NH_3 and H_2O present in ZSM-5. The spectra for the NH_4^+-ZSM-5 samples have additional bands due to the $\gamma(N-H)$ located around 3500 to 3100 cm^{-1}, and the $\delta(N-H)$ positioned around 1640 to 1500 cm^{-1}. Furthermore, there are $\nu(O-H)$ bands occurring at approximately 3500 to 3200 cm^{-1} and $\gamma(OH)$ at 1600 cm^{-1}. All the spectra for ZSM-5 contain bands for $\gamma(Si-O)$ and $\delta(Si-O)$ which are around 1070 to 800 cm^{-1} and 660 to 490 cm^{-1} respectively.

Table 1: Major IR Bands for Typical $Ru_3(CO)_{12}$ and ZSM-5 Samples

SAMPLE	MAJOR IR BANDS[a] (cm^{-1})
$Ru_3(CO)_{12}$	2061(vs); 2015(vs); 2000(s); 1992(s); 591(m); 577(m); 546(w); 464(w); 447(m)
NH_4^+-ZSM-5	3282(w); 1881(vw); 1637(vw); 1096(s); 718(vw); 541(w); 457(m)
H^+-ZSM-5[b]	1103(s); 719(vw); 540(w); 452(m)
NH_4^+-ZSM-5	3641(s); 3432(s); 1878(vw); 1635(m); 1092(vs); 601(vw); 542(w); 449(m)
H^+-ZSM-5[c]	1222(w); 1097(vs); 719(vw); 542(w); 450(m)

[a] vs = very strong; s = strong; m = moderate; w = weak; vw = very weak

[b] NH_4^+-ZSM-5 heated at 538°C

[c] NH_4^+-ZSM-5 heated at 350°C

IR spectra were obtained on the solid catalyst samples after preparation, and oftentimes, on the residue of the filtrate after evapopration of the solvent. The bands of a typical IR spectrum for a ~5% Ru/ZSM-5 sample are listed in Table 2. The IR spectrum obtained on the catalyst sample contains two bands (2054 cm^{-1} and 1991 cm^{-1}) in the terminal carbonyl stretching frequency region. The carbonyl bands present in the Ru supported catalysts are shifted to lower wave numbers in comparison to the $Ru_3(CO)_{12}$. These shifts may be due to the carbonyl interactions with the support or a change in the symmetry that occurs following adsorption. The spectrum of the catalyst, also, contains a very weak band due to the presence of water (1636 cm^{-1}) which probably was adsorbed from the atmosphere

during the preparation and/or handling of the catalyst. The remaining bands are due to the support. The residue obtained from evaporation of the filtrate has a spectrum indicating that it is $Ru_3(CO)_{12}$.

Table 2: Typical IR Spectra for a Catalyst Sample

SAMPLE	MAJOR IR BANDS[a] (cm^{-1})
~5% Ru/EX/ZSM-5	2054(w); 1991(w); 1635(vw); 1222(s); 1098(vs); 719(vw); 542(m); 450(s)
Residue from ~5% Ru/ZSM-5 filtrate	2059(m); 2025(m); 2000(w); 591(vw); 578(vw); 448(vw)

[a] vs = very strong; s = strong; m = moderate; w = weak; vw = very weak

The DG samples were, also, studied by IR spectroscopy; Table 3 lists the major bands obtained from the spectra. As the percentage of ruthenium was increased, the intensity of the bands in the carbonyl region (2100 to 2000 cm^{-1}) increased proportionally which indicates that a relationship can be drawn between the intensity of the carbonyl bands and the amount of ruthenium carbonyl in the sample. Gallezot and coworkers reported that the intensity of the IR bands in the carbonyl region increased with the amount of adsorbed $Mo(CO)_6$ on HY zeolite (1) which supports the findings in this study.

Air exposure and heating studies were performed on the EX and DG samples. IR spectra were obtained in order to detect any changes in the carbonyl region upon air exposure or heating of the samples; Tables 4 and 5 summarize these IR studies. The disappearance of

Table 3: The IR Data Obtained for the DG Samples

SAMPLE	MAJOR IR BANDS[a] (cm^{-1})
0.95% Ru/DG/ZSM-5	2060(vw); 2027(vw); 1643(vw); 1220(m); 1094(s); 719(vw); 544(w); 453(m)
2.90% Ru/DG/ZSM-5	2060(w); 2027(w); 2000(vw); 1637(vw); 1221(m); 1094(s); 719(vw); 547(w); 450(m)
5.94% Ru/DG/ZSM-5	2060(m); 2037(m); 2025(m); 2000(m); 1656(vw); 1222(m); 1095(s); 720(vw); 546(w); 449(m)

[a] vs = very strong; s = strong; m = moderate; w = weak;
vw = very weak

these carbonyl bands signified the decomposition of the $Ru_3(CO)_{12}$ in the samples. It can be seen from the data in Table 4 that exposure to air for as long as 24 h does not change the IR spectrum of ∿5% Ru/ZSM-5. However, heating at 150°C for an hour caused the disappearance of the carbonyl peaks. Table 5 shows that the DG and EX catalyst samples behave in the same manner when heated; each sample experienced $Ru_3(CO)_{12}$ decomposition when heated at 100°C for 30 min although no decomposition was observed when heated at 50°C for 30 min. The ∿10% Ru/ZSM-5 sample was studied for further heating effects so it was heated at 80°C for 5 min, no change occurred; however, heating at 92°C for 5 min caused the disappearance of the carbonyl bands. Therefore, heating for a short time at around 92°C can cause the decarbonylation of the ruthenium carbonyl in these materials. The Ru/ZSM-5 samples became black when decarbonylation

Table 4: The IR Results from the Air Exposure and Heating Studies on a ~5% Ru/EX/ZSM-5 Catalyst

AIR EXPOSURE (h)	HEATING TIME 150°C (h)	γ(CO) (cm^{-1})
0	0	2054; 1991
2	0	2053; 1992
8	0	2058; 1996
24	0	2060; 1995
26	1	no bands
50	24	no bands

Table 5: Summary of the IR Results from the Heating Studies

SAMPLE	HEATING TIME (min)	TEMPERATURE (°C)	γ(CO) (cm^{-1})
5.49% Ru/DG/ZSM-5	0	--	2060; 2037 2025; 2000
	30	50	2060; 2037 2025; 2000
	30	100	no bands
~5% Ru/EX/ZSM-5	0	--	2058; 1995
	30	50	2059; 1996
	30	100	no bands
~10% Ru/EX/ZSM-5	0	--	2054; 1994
	15	70	2055; 1995
	5	80	2059; 1998
	5	92	no bands
	30	50	2057; 1995
	30	100	no bands

had occurred indicating the presence of RuO_2 on the support; this conclusion was confirmed by XRPD studies. Therefore, the air exposure and heating studies indicate that the catalysts are stable in the presence of air, but decarbonylation occurs when the samples were heated.

2. Thermogravimetric Analysis

The technique of Thermogravimetric Analysis (TGA) measures the weight loss or gain of a sample in a controlled atmosphere. TGA was used in the early stages of this work to gain an insight into the thermal stability of the materials. NH_4^+-ZSM-5 was studied by the use of TGA from 25°C to 600°C in He. During the TGA experiment, the weight loss of the sample occurred before 350°C due to the removal of NH_3 and H_2O. Therefore, the ZSM-5 was heated to 350°C in order to obtain the H^+ form of the ZSM-5; the loss of NH_3 and H_2O by the sample was confirmed by IR spectroscopy.

The catalysts were also studied by TGA to indicate their stabilities. The samples used in this study were ∿5% Ru/ZSM-5, ∿10% Ru/ZSM-5 and ∿10% Ru/ZSM-5 heated at 500°C for 18 h. Three gases were used in the following order He, H_2 and CO. A temperature range of 25°C to 600°C was used, and the sample was heated at a rate of $10°C \cdot min^{-1}$. For the ∿5% Ru/ZSM-5 and ∿10% Ru/ZSM-5 samples, the curves showed a loss in weight in all three gases which decreased in the order of the introduction of the gases ($He > H_2 > CO$). Upon heating, $Ru_3(CO)_{12}$ is known to continuously envolve carbon monoxide. IR spectra obtained for the samples before the TGA study indicate the presence of the carbonyl groups; however, after the TGA study the carbonyl bands were absent. These results support

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the conclusion that carbon monoxide is lost by the sample during the TGA experiment. Another source reported a loss of H_2O adsorbed during the catalyst preparation by ruthenium zeolite catalysts when investigated by TGA using N_2 (2). In the preparation of the Ru/ZSM-5 catalysts in this study, H_2O was adsorbed by the support which lead to additional weight loss in the TGA experiment. It has been reported that $Ru_3(CO)_{12}$ does not decompose completely to the metal during the TGA investigation, but partial sublimation occurs (3). The RuO_2 present in the sample due to the carbonyl decomposition could have been reduced to Ru° causing a weight loss in H_2. However, if Ru° was present it did not adsorb H_2 or CO which would of been reflected as a weight gain by the TGA curve.

A weight loss was observed for the heated ~10% Ru/ZSM-5 sample in the three gases, but the order was different from that obtained for the unheated samples. The sample lost most of its weight in H_2, the second gas, followed by He and CO. Also, the overall weight loss was smaller for the heated sample. The IR spectrum obtained on the pretreated sample did not contain the carbonyl bands; therefore, the evolution of carbon monoxide had already occurred. The conditions during the TGA experiment produced a weight loss in the sample due to loss of H_2O which was adsorbed when the sample was cooled in air, the reduction of RuO_2 to Ru° and the sublimation of ruthenium.

3. X-ray Powder Diffractometry

A characteristic diffraction pattern is obtained from X-ray Powder Diffractometry (XRPD) for each substance whether it is in the pure state or present in a mixture. Due to this ability of XRPD, it was used in this study for identification purposes. To

confirm the structure of ZSM-5, diffraction patterns on each batch of ZSM-5 were obtained. A representative pattern is shown in Figure 1. Patterns were, also, obtained for the catalysts, and the results are summarized in Table 6. Each pattern indicated the presence of the support, ZSM-5. The patterns for the AP cata- lysts indicate the presence of only the ZSM-5 which is due to the fact that the source, Cu K_α, does not allow a species of less than 60 Å to be detected. Therefore, the metal species is thought to be highly dispersed on the support with a size less than 60 Å. Sim- ilar results were obtained by McVicker and Vannice on a 1.3% Ru/Al_2O_3 catalyst promoted with 0.45% K (4).

RuO$_2$ was detected by XRPD on the H-500 samples. The pre- treatment is thought to cause the agglomeration of the metal species on the support; this agglomeration leads to particle sizes greater than 60 Å which are detected by the diffractometer. There is a dif- ference within the patterns obtained from XRPD for the Used catalysts. In the spectra for the two lower loadings (0.98% and 2.88%) no metal species was detected. However, the 7.32% and 8.13% Used catalysts contained Ru°. It appears that after the catalytic evaluation the 7.32% and 8.13% samples still have some of the active Ru° of larger than 60 Å present while for the 0.98% and 2.88% samples, the Ru° or other metal species is of a small size.

From XRPD, it is concluded that the AP catalysts contain a high degree of dispersion of the metal species on the support, that the pretreatment of the H-500 samples causes an increase in the par- ticle size of the metal species and that the higher loadings of Ru contain Ru° after catalytic evaluation.
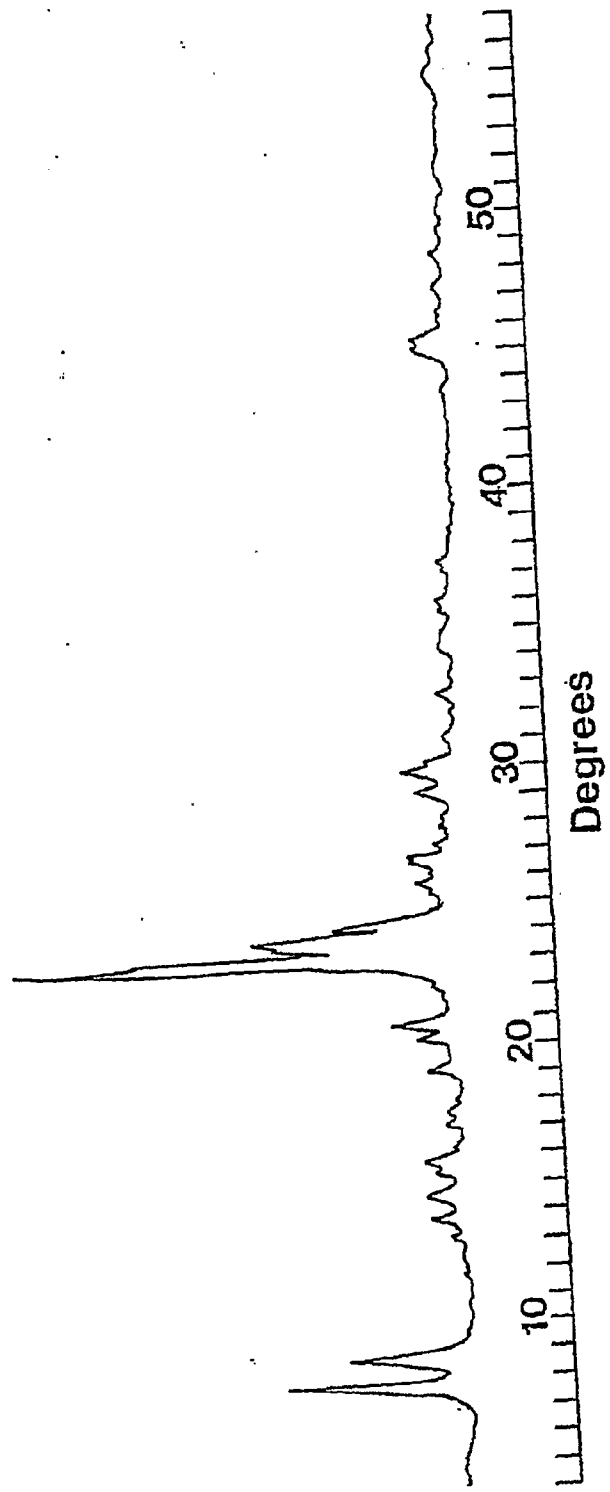
Figure 1. A representative XRPD spectrum of ZSM-5.

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Table 6.: Summary of the XRPD Results[a] on the Catalyst Samples

% Ru/ZSM-5	AP	H-500	USED
0.98	--	RuO_2	--
2.88	--	RuO_2	--
7.32	--	b	Ru°
8.13	--	RuO_2	Ru°

[a]All spectra contained the pattern for ZSM-5

[b]This spectrum was not obtained

4. Ion-Scattering Spectrometry

The technique of Ion-Scattering Spectrometry (ISS) was used for the detection of elements present in the sample. The depth profiling capability of ISS enables the surface and subsurface of the sample to be studied. The [4]He ion penetrates the sample at ~0.5 $\overset{\circ}{A} \cdot min^{-1}$ exposing the elements within the surface. Figure 2 a representative ISS scan; the ruthenium peak is located at 0.845 E/E_o, the overlapping aluminum and silicon peaks at 0.57 E/E_o and the oxygen peak at 0.385 E/E_o. From the scans obtained by ISS, the Ru to Si-Al ratios were calculated from the peak heights. In Figure 3, these ratios for the AP catalysts are plotted versus the sputtering times of 0-5 min, 5-10 min, 30-35 min and 60-65 min. The curves indicate that the Ru/Si-Al ratios increase with higher Ru loading. Also, for a particular loading the ratio decreases with longer sputtering time which implies that the amount of Ru decreases with
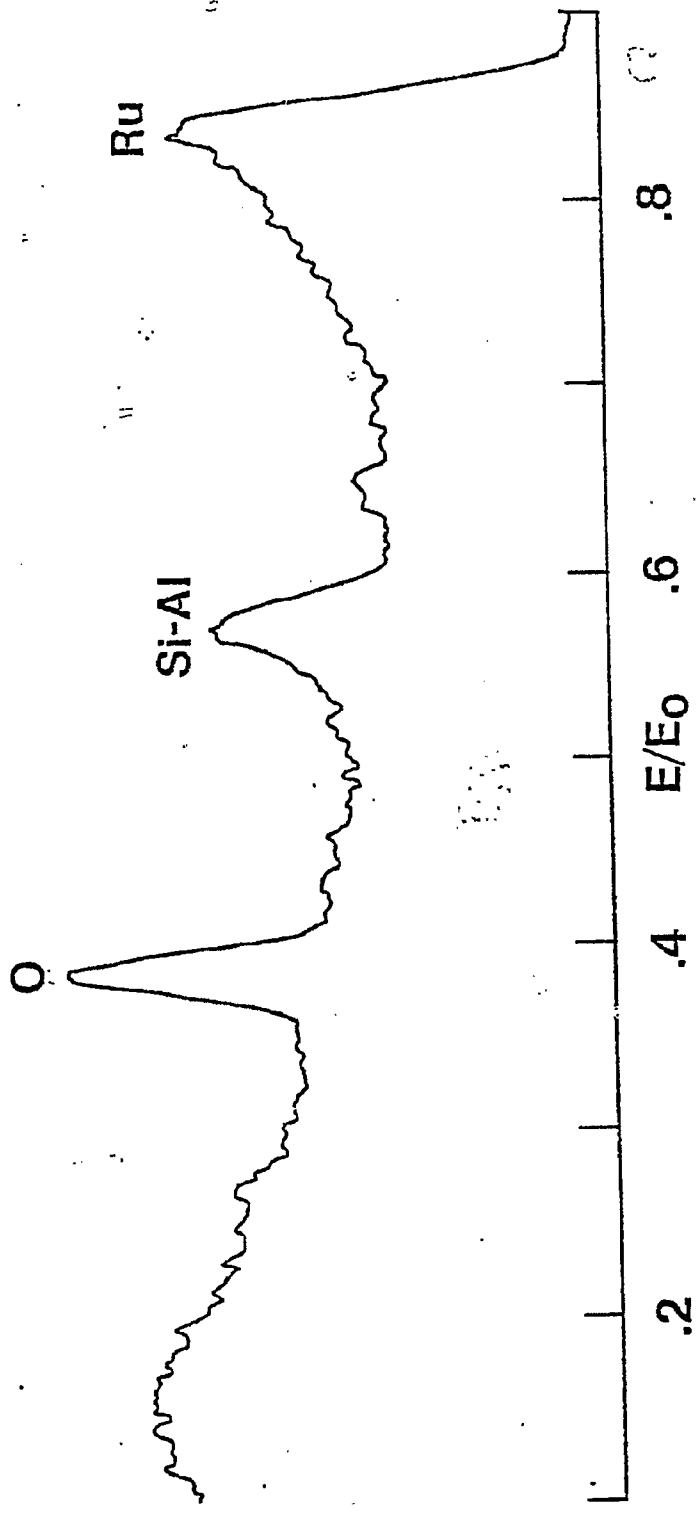
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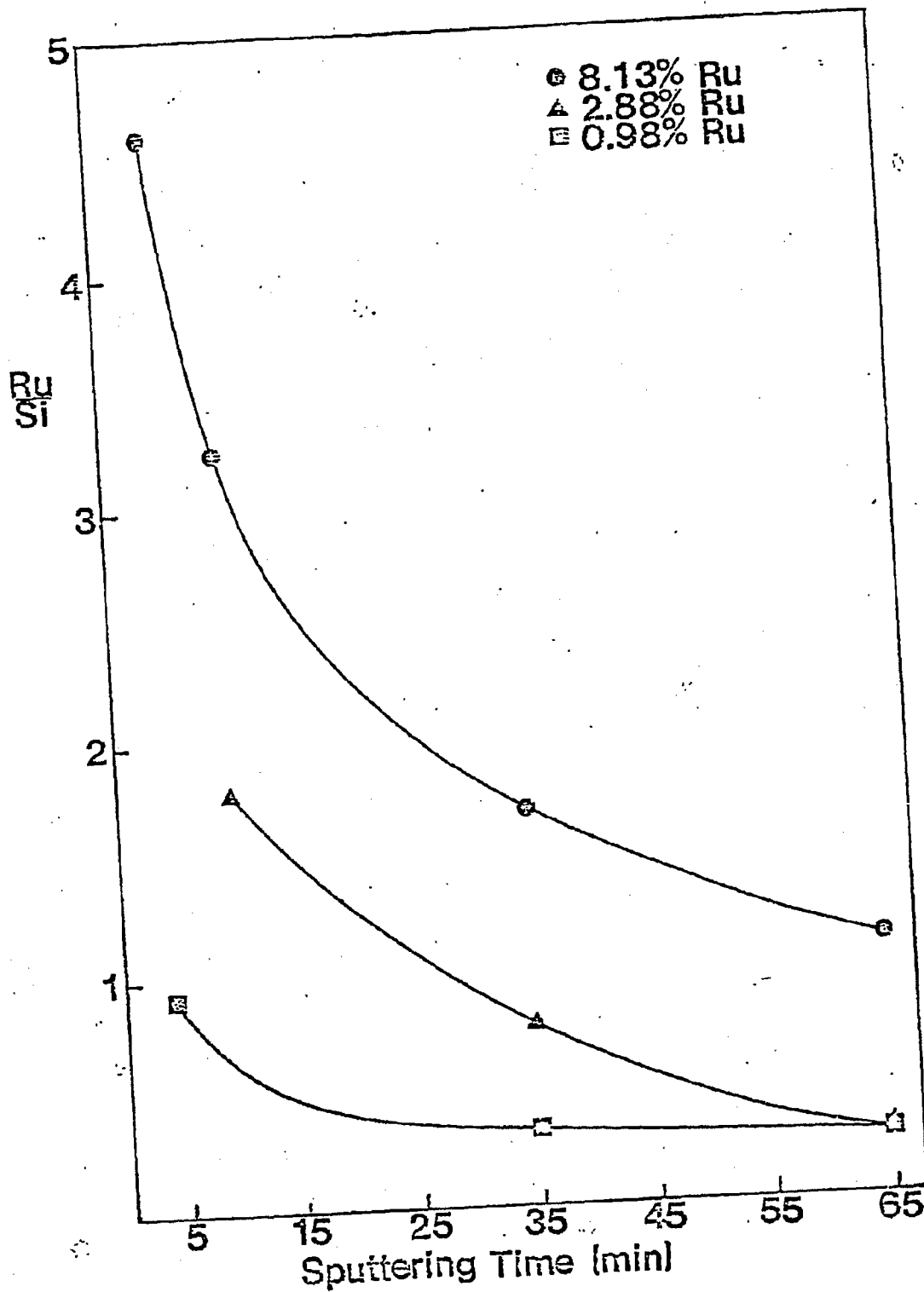Figure 2: A representative ISS scan of a Ru/ZSM-5 catalyst.

Figure 3: The ISS results of the AP catalysts.

depth. Even after 65 min of sputtering, an estimated depth of 32.5 Å, Ru was still observed; therefore, the Ru is not just on the surface of the sample, but Ru penetrates into the support. The Used catalysts were also investigated by ISS; the Ru/Si-Al ratios obtained from these scans were significantly lower than the AP catalysts. In Figure 4 the ratios are plotted for three Used catalysts. From these curves, an increase in the Ru/Si-Al ratio was observed as the sputtering time increased. This trend is opposite in direction to the trend for the AP catalysts, and may be due to surface contamination, probably carbon deposits, obtained during the evaluation. Once below this contamination, the Ru is detected by the instrumentation causing the increase in the Ru/Si-Al ratio.

5. X-ray Photoelectron Spectroscopy

The instrumental technique of X-ray Photoelectron Spectroscopy (XPS/ESCA) is a powerful tool used in the characterization of surface and subsurface atomic layers of solids; for example, heterogeneous catalysts. ESCA provides insight into the elemental composition and oxidation states of species present is a sample. Non-sputtered (NON-SP) and sputtered (SP) ESCA spectra were obtained for the catalysts; ^{40}Ar was used to sputter into the sample for 10 min which would be a depth of ~70 Å. After the binding energies (B.E.) were corrected for charging, they were compared to the literature values to determine the ruthenium species present in the sample. For this study, the binding energies used were:

	Ru $3d_{5/2}$	Ru $3p_{3/2}$
Ru°	280.0	461.1
RuO_2	280.5	462.3

USED

● 8.13% Ru
▲ 2.88% Ru
▣ 0.98% Ru
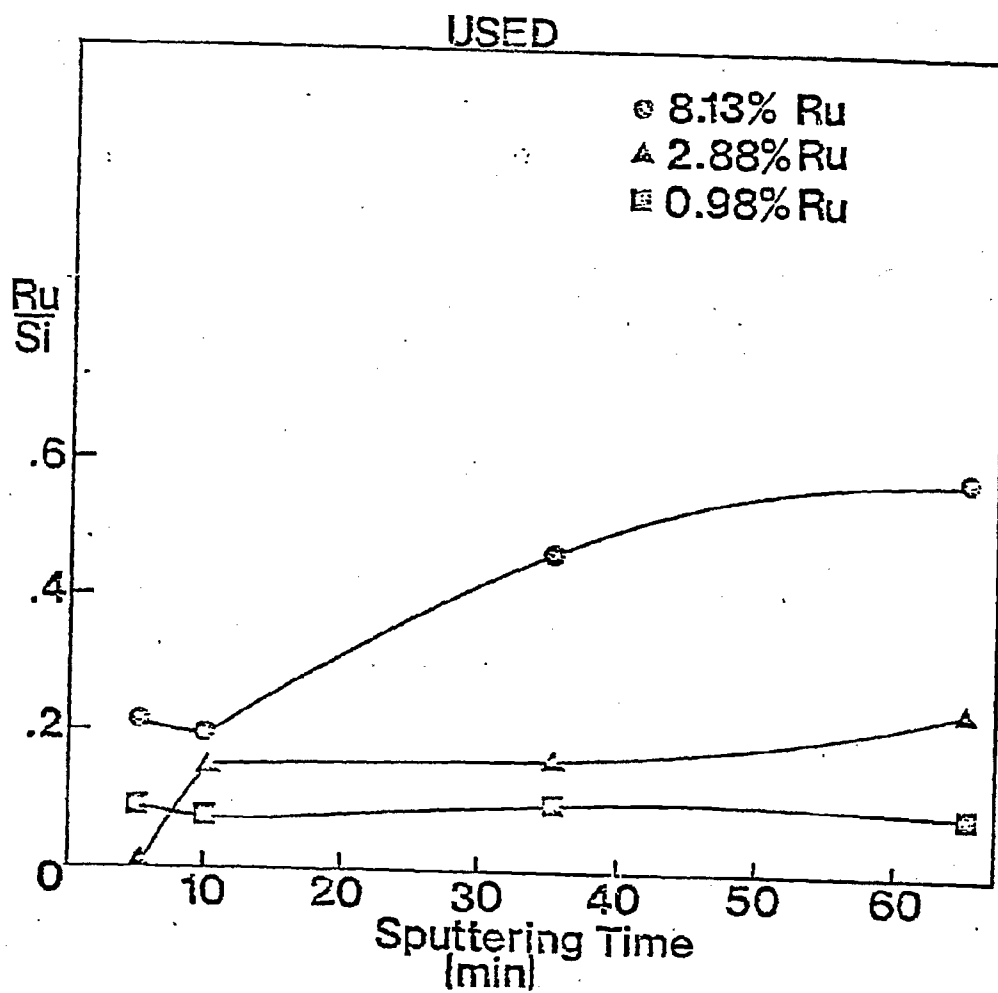
$\frac{Ru}{Si}$

Sputtering Time
(min)

Figure 4.   The ISS results of the Used catalysts.

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The carbon 1s peak overlaps the Ru $3d_{3/2}$ peak in the region of 286 to 283 eV; therefore, this Ru band cannot be clearly distinguished and was not used in the determination of the Ru species present in the sample.

Initially, ESCA scans were obtained for the AP catalysts. The Ru $3p_{3/2}$ peaks for the non-sputtered and sputtered scans for the AP catalysts are shown in Figure 5. Table 7 summarizes these results, and from the binding energies it was determined that the metal species present was RuO_2. The H-500 catalysts were investigated by ESCA; however, the sputtered scans were not obtained due to technical difficulties. Table 8 contains the binding energies of the H-500 samples, these values also indicate the presence of RuO_2; recall that the XRPD results indicated the presence of RuO_2 in the H-500 catalysts. Therefore, the AP and H-500 catalysts contain the same metal species (RuO_2), but the particle size of this species is larger for the H-500 samples than for the AP catalysts.

The Used catalysts differ from the AP and H-500 catalysts. Table 10 contains the binding energies for the Used samples. The binding energies obtained for the 0.98%, 2.88% and 7.32% Used catalysts do not clearly indicate the presence of Ru° or RuO_2. The peaks of the Used catalysts are slightly broader than the peaks obtained for the AP catalysts. Due to the breadth of the peaks and because the binding energies are between Ru° and RuO_2, it appears that these Used catalysts contain a mixture of Ru° and RuO_2. The results obtained for the 8.13% Used catalyst indicated that Ru° was on the surface and RuO_2 under the surface. Figure 6 shows the $3p_{3/2}$ peaks for the AP and Used catalysts showing the difference of the
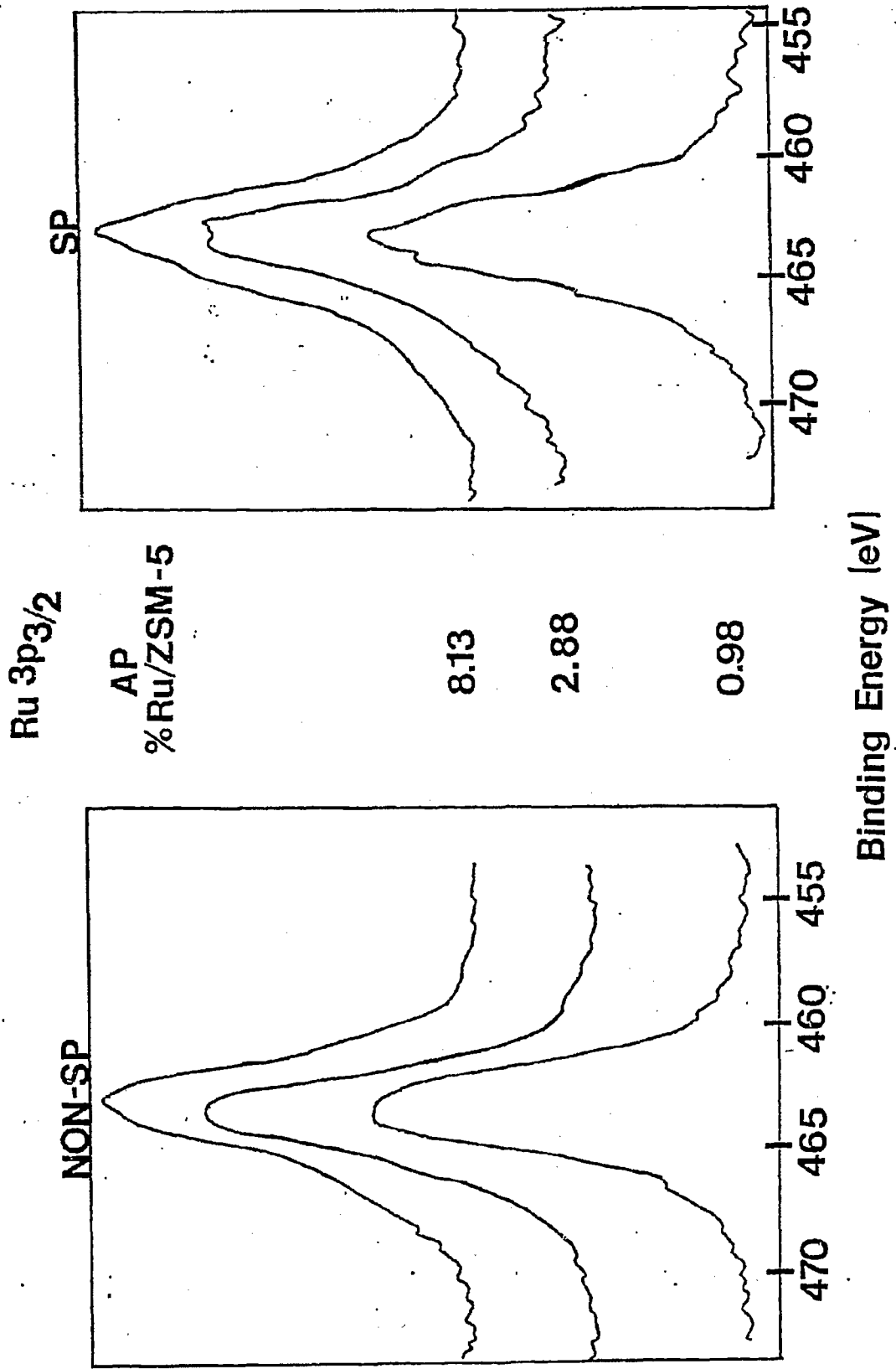
Ru 3p3/2

AP %Ru/ZSM-5
8.13
2.88
0.98

Binding Energy (eV)

Figure 5: The Ru $3p_{3/2}$ peaks for the AP catalysts.

Table 7 : The ESCA Results for the AP Catalyts

% Ru/ZSM-5	PEAK	BINDING ENERGY	
		NON-SP	SP
0.98	Si	103.00	102.85
	Ru $3p_{3/2}$	463.10	462.80
	Ru $3d_{5/2}$	281.60	280.55
2.88	Si	103.22	102.75
	Ru $3p_{3/2}$	463.22	462.65
	Ru $3d_{5/2}$	281.82	280.60
8.13	Si	102.95	103.85
	Ru $3p_{3/2}$	463.00	463.45
	Ru $3d_{5/2}$	281.20	281.30

Table 8: The ESCA Results for the H-500 Catalysts

% Ru/ZSM-5	PEAK	NON-SP BINDING ENERGY
0.98	Si	102.85
	Ru $3p_{3/2}$	462.65
	Ru $3d_{5/2}$	a
2.88	Si	102.95
	Ru $3p_{3/2}$	462.95
	Ru $3d_{5/2}$	280.55

[a] Peak indistinguishable

Table 9: The ESCA Results for the Used Catalysts

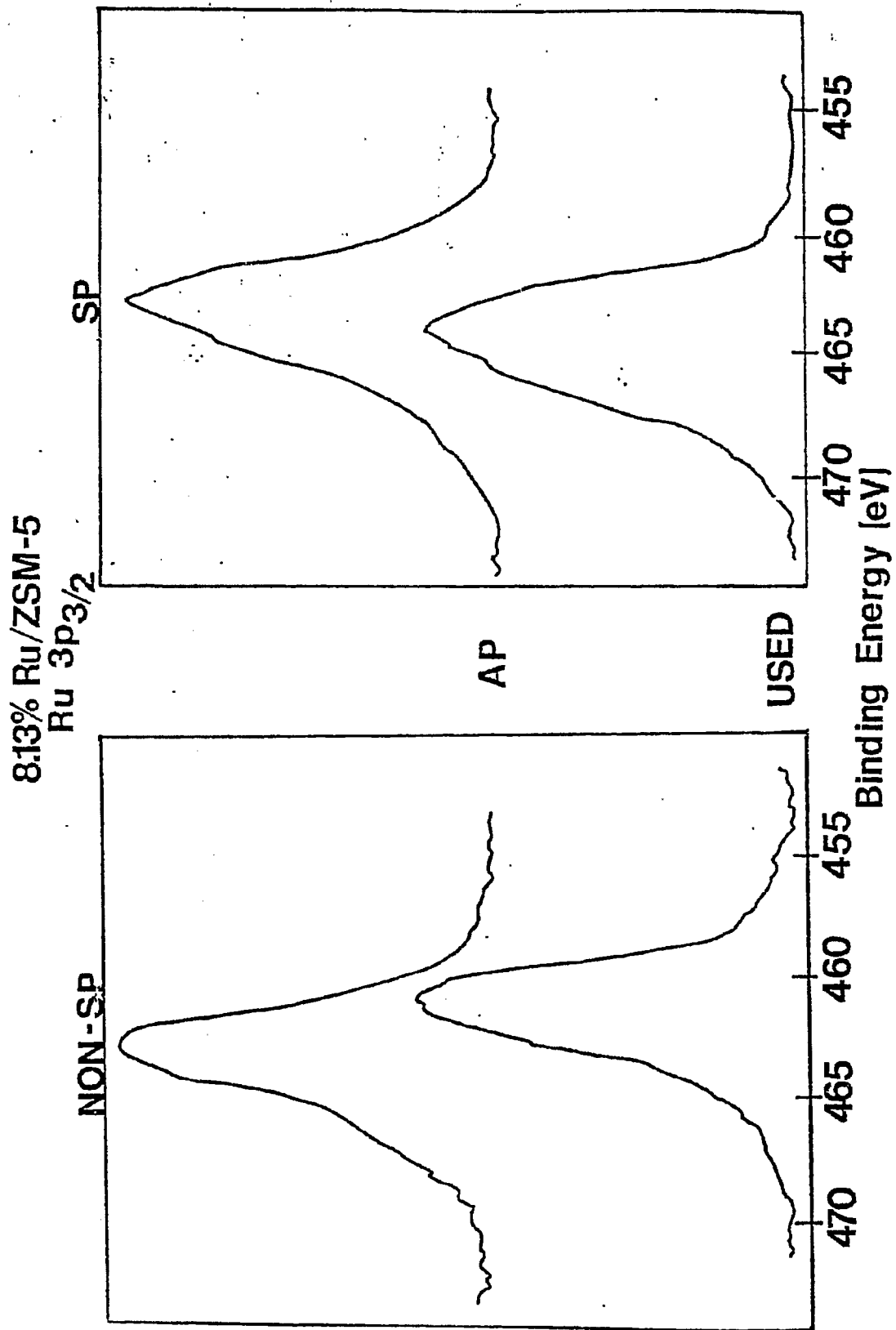| % Ru/ZSM-5	PEAK	BINDING ENERGY NON-SP	SP
0.98	Si	102.80	102.80
	Ru $3p_{3/2}$	461.50	462.10
	Ru $3d_{5/2}$	280.10	280.10
2.88	Si	102.85	102.90
	Ru $3p_{3/2}$	461.65	462.00
	Ru $3d_{5/2}$	280.10	280.10
7.32	Si	102.90	102.80
	Ru $3p_{3/2}$	461.45	461.60
	Ru $3d_{5/2}$	279.80	280.10
8.13	Si	103.05	102.90
	Ru $3p_{3/2}$	461.25	462.30
	Ru $3d_{5/2}$	279.65	280.30

Figure 6. The Ru 3p$_{3/2}$ peaks for the AP and Used 8.13% Ru/ZSM-5 catalysts.

non-sputtered peak from RuO_2 for the AP to Ru^o for the Used catalyst.
However, the sputtered peaks are both characteristic of RuO_2. The
ESCA data for this Used catalyst indicates that the metal species
below the surface was not totally reduced by the hydrogen employed
during reduction.

6. Hydrogen and Carbon Monoxide Chemisorption

The AP catalysts of 0.98%, 2.88% and 8.13% Ru/ZSM-5 were studied
by hydrogen and carbon monoxide chemisorption. The amounts of CO
adsorbed during the CO chemisorption experiments were 2 to 3 times
higher than the measurements obtained from the H_2 chemisorption data
due to multiple CO adsorption. These results are in agree-
ment with the data obtained on Ru/silica samples by Kobayashi and
Shriasaki (5,6). Since multiple CO chemisorption occurred with
the Ru samples, the CO adsorption measuremnts are not reliable for
the prediction of the average Ru particle size and dispersion. How-
ever, the H_2 chemisorption measurements were found by Irwin to pre-
dict Ru metal particle size and dispersion fairly accurately (7).
The surface stoichiometry of $H/Ru_{(s)}$ was assumed to be 1.0 which was
found to be in agreement with previously reported values (8,9).

Table 10 contains the Ru particle sizes and dispersions obtained
from the H_2 chemisorption measurements on the AP catalysts. In the
determination of the particle sizes and dispersions, the assumption
was made that the atomic ruthenium surface area was 8.17 $Å^2$/Ru atom
(7). From the reported data, there is a slight increase in the
particle size with an increase in metal loading. Figure 7 is a
plot of the particle size versus the evacuation time which indicates
that the determined particle size for the 8.13% sample remained

Table 10:   The H_2 Chemisorption Results on the AP Catalysts

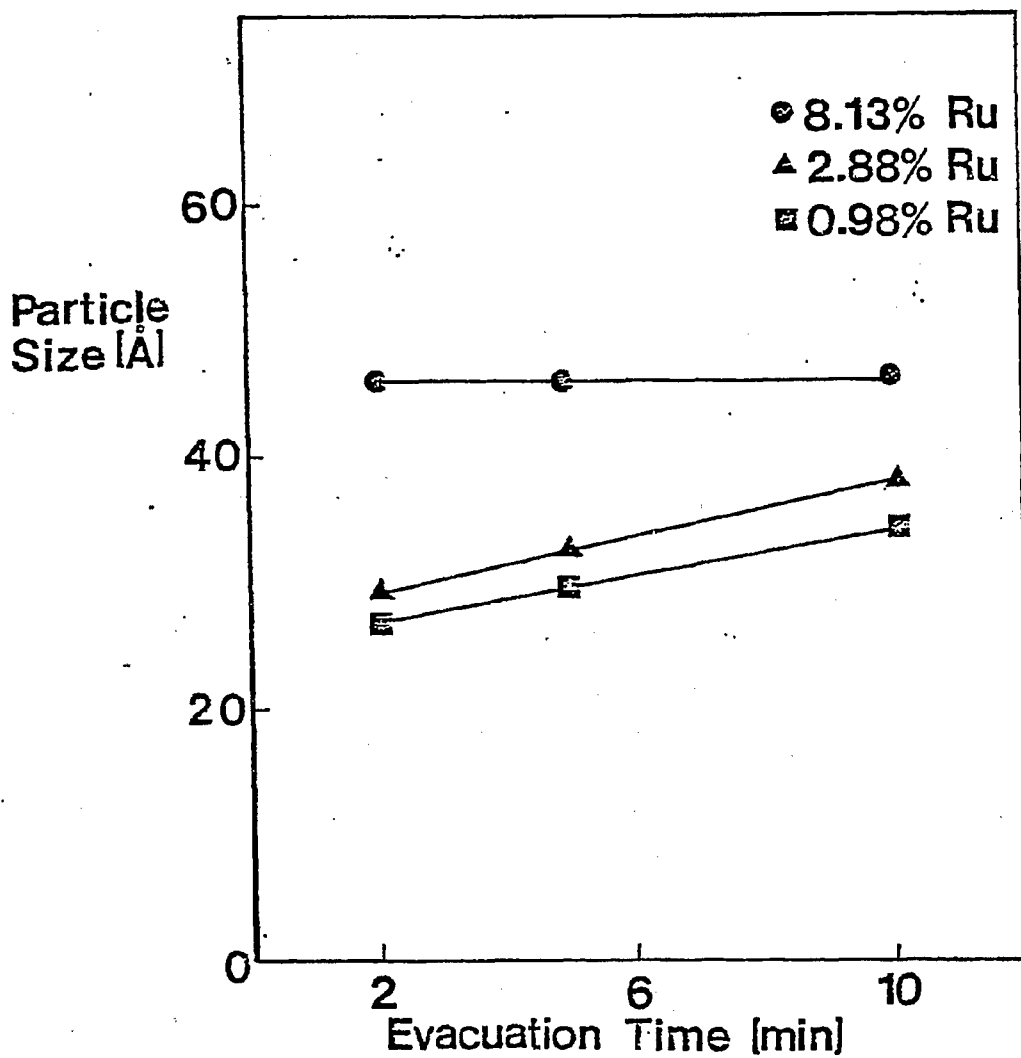| % Ru/ZSM-5	DISPERSION (%)	PARTICLE SIZE (Å)	EVACUATION TIME (min)
0.98	31.5	26.8	2
	28.4	29.8	5
	24.8	34.1	10
2.88	28.7	29.3	2
	25.6	32.9	5
	22.0	38.4	10
8.13	18.4	45.9	2
	18.4	45.9	5
	18.1	46.6	10

Figure 7. A plot of the particle size versus evacuation time obtained from the H_2 chemisorption data for the AP catalysts.

fairly constant while the calculated particle size for the lower loadings increased with increasing evacuation time. Therefore, the lower metal loadings, with smaller Ru particle sizes, appeared to strongly reversibly chemisorb H_2 requiring the longer evacuation times to desorb it from the surface (7). Measurements obtained from H_2 chemisorption indicated that the particle sizes for all three samples were below 60 Å, the detection limit of the copper X-ray source used in XRPD.

7. Conclusions

The following conclusions can be drawn from the characterization data obtained on the catalysts.

1. It was determined that complete decomposition of the $Ru_3(CO)_{12}$ did not occur during the preparation procedure; however, calcination of the sample did cause the decompositon of the $Ru_3(CO)_{12}$ structure and agglomeration of the metal species.

2. The metal species was determined to be RuO_2 for the AP and H-500 samples with particle sizes of less than 60 Å for the AP catalysts and greater than 60 Å for the H-500 catalysts. Therefore, the extraction technique produces a high degree of dispersion of the metal species on the AP catalyst.

3. The characterization by ESCA, also, indicated that the Used catalysts did not have complete reduction of the metal species during the catalytic evaluation; because $Ru°$ and RuO_2 species were detected.

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"Synthesis Gas Conversion with Ru/ZSM-5 Catalysts Prepared from $Ru_3(CO)_{12}$". G. A. Melson, R. Y. Eaton, J. M. Stencel and C. A. Spitler, 34th Southeastern Regional Meeting, American Chemical Society, Birmingham, AL, November, 1982.

"^{57}Fe Mössbauer Spectroscopic Characterization of Supported Iron Fischer-Tropsch Catalysts". P. P. Vaishnava, P. I. Ktorides, P. A. Montano, K. J. Mbadcam, and G. A. Melson. 1982 Annual Meeting, Materials Research Society, Boston, MA. November, 1982.

Invited Seminar

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