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Decomposition of Decanol and Decanal over Ni- and H-ZSM-5

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The decomposition of 1-decanol and decanal in the presence of hydrogen by using Ni- and H-ZSM-5 embedded within a silica binder was investigated. Temperatures and pressures for the decanol decomposition ranged from 572 to 666 K and 0.57 to 1.45 MPa, whereas for decanal the ranges were 622 to 722 K and 0.58 to 1.22 MPa. Decanol was converted to decene through the intermediate didecyl ether. Decanal was convered to nonane by the elimination of carbon monoxide; to a β-hydroxy carbonyl compound, an aldo, which subsequently dehydrated to yield C_{20} aldehyde. The aldehyde, subsequently, was converted to hydrocarbons. All of the above are acid-catalyzed reactions. A side reaction catalyzed by the reactor walls which occurred with both compounds was the hydrogenation of decanal or dehydrogenation of decanol.

INTRODUCTION

The reactions of various oxygenated hydrocarbons on ZSM-5 have been examined since Chang and Silvestri (*1*) published their landmark paper in 1977. By far most of the studies have concentrated on low-molecular-weight alcohols, methanol in particular, due to interest in the Mobil process, methanol to gasoline (MTG), in which ZSM-5 plays a crucial role. According to Meisel and Weisz (*2*), ZSM-5 can also be used to upgrade Fischer–Tropsch (F-T) liquids to a hydrocarbon mixture which contains approximately 60% gasoline range compounds. Upgrading F-T liquids with ZSM-5 would eliminate the complex refining schemes necessary to produce gasoline from F-T liquids, convert the oxygenates to hydrocarbons, and increase the gasoline yield.

Fischer–Tropsch liquids (produced with an iron oxide catalyst) usually contain about 10% oxygenated hydrocarbons, i.e., alcohols, aldehydes, ketones, ethers, esters, and carboxylic acids. The reactions of alcohols and ethers over ZSM-5 or other zeolite catalysts have been studied extensively. However, the reactions of aldehydes, ketones, esters, and carboxylic acids for the most part have received little attention. Our investigation of the reactions of two F-T model compounds, 1-decanol and decanal, on ZSM-5 leads us to propose series mechanisms involving condensation reactions for the decomposition of both model compounds.

EXPERIMENTAL

Catalyst preparation and analysis. The two catalyst were prepared as follows: 1 g of the NH_4-ZSM-5 was slurried with 200 ml of a solution containing 12.7% $Ni(NO_3)_2 \cdot 6H_2O$ and refluxed at 361 K for 4 h. The exchanged catalyst was then filtered and dried at 381 K for 13 h. The catalyst was then mixed with a solution of Ludox AS40, extruded, dried, and calcined at 811 K for 7 h. The Ni-ZSM-5 extrudates were then crushed to particles in the size of 100 to 400 μm with a density of approximately 0.75 g/ml. Therefore, the Ni-ZSM-5 catalyst contained 69% silica binder and 31% zeolite. An analysis of this material by ICP indicated the following: Ni = 0.078% and A1 = 1.64%. An analysis by XPS indicated that the nickel was on the external surface of the particles. Hence, during the calcination the

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TABLE 1

Reaction Conditions for 1-Decanol Decomposition

Catalyst	Temp (K)	Pressure (atm)	CT[a] (s-g/cm^3)	WHSV[b] (h^{-1})
Ni-ZSM-5	598–619	5.73–14.50	1.18–2.80	14.17–31.13
H-ZSM-5	572–666	8.55–10.74	0.99–2.03	19.63–32.12

[a] Contact time (grams catalyst/volumetric flow rate at reactor T, P).

[b] Weight hourly space velocity (grams fed per hour per gram of catalyst).

nickel migrated from inside of the pores of the catalysts to the external surfaces.

The hydrogen form of the ZSM-5 was prepared in a similar fashion as the Ni-ZSM-5 except an 11% solution of NH_4Cl was used. The final catalyst contained 20% zeolite and 80% binder. Particles of 100 to 300 μm with a bulk density of 0.79 g/ml were used in the reactor. An XPS analysis of the catalyst did not detect any Al on the surface of the particles. Hence, only the binder could be seen. Both catalysts were purged in situ at reaction temperature by flowing pure hydrogen through the catalyst bed to remove any water or oxygen which may have been present in the microreactor system and to reduce the Ni cations.

The reactions were studied using a 316 SS microreactor system designed to operate up to 21 atm (1 atm-1.01 × 10^5 N/m^2) and 775 K. The microreactor was immersed in an isothermal eutectic salt bath. The products were collected at atmospheric pressure using cold traps and were analyzed using separate chromatographs for the vapor and liquid products.

TABLE 2

Primary Product Distribution for 1-Decanol/H-ZSM-5

Temp (K)	Conv[a] (%)	C_{10}[b] (%)	C_{20} ether[b] (%)	C_9 A[b] (%)
572	1.27	0.00	99.61	0.00
596	8.07	31.28	68.49	0.00
598	13.23	58.59	37.98	0.00
599	26.94	74.87	23.83	0.00
600	13.42	50.38	46.50	0.00
618	45.29	83.30	13.66	1.59
645	60.91	84.83	5.63	5.11
666	66.75	83.75	6.28	7.90

[a] Conversions are on a decanal, *n*-nonane, CO, and CO_2 free basis.

[b] Product distributions are on a decanal, *n*-nonane, CO, CO_2, and H_2O free basis.

TABLE 3

Primary Product Distribution for 1-Decanol/Ni-ZSM-5

Temp (K)	Conv[a] (%)	C_{10}[b] (%)	C_{20} ether[b] (%)
598	23.71	97.84	0.00
598	32.80	90.83	6.58
598	33.14	95.32	2.90
598	66.85	96.54	1.62
619	40.85	92.55	3.90
619	65.66	95.56	1.42
619	68.37	95.06	1.54

[a] Conversions are on a decanal, *n*-nonane, CO, and CO_2 free basis.

[b] Product distributions are on a decanal, *n*-nonane, CO, CO_2, and H_2O free basis.

The experiments lasted between 15 min and 1 h depending on how long it took to collect a sufficient liquid sample for weighing and subsequent GC analysis. Outlet vapor samples and flow rates were taken three times per run to certify that a steady state had been achieved. Generally five runs could be made before any loss of activity occurred with the Ni-ZSM-5 whereas H-ZSM-5 started to show loss of activity after three runs. The catalysts were regenerated with air until neither CO or CO_2 were present in the outlet stream and then reused. The experimental procedure has been described in greater detail by Riley (*3*).

RESULTS

Results for 1-Decanol

The range of reaction conditions are given in Table 1 and the run conditions and product distributions of each run are given in Tables 4 and 5. The primary product distributions, after correcting for decanal for-

TABLE 4

1-Decanol Decomposition over H-ZSM-5

Catalyst/run	H/1	H/2	H/3	H/4	H/5	H/6	H/7	H/8
Temperature (K)	618	572	645	666	596	598	599	600
Pressure (atm)	8.76	8.98	8.57	9.98	9.98	12.05	10.74	8.55
Conversion[a] (%)	67.43	10.73	81.98	99.99	18.90	26.95	43.71	25.70
Mass closure[b] (%)	98.8	102.1	102.2	100.7	105.5	99.4	97.4	99.7
CT[c] (s-g/cm^3)	1.00	1.11	1.05	1.06	1.18	1.01	2.03	0.99
H_2/feed (mol/mol)	2.29	2.30	3.50	2.30	2.30	2.14	2.20	2.18
WHSV[d] (h^{-1})	30.7	30.5	20.4	30.6	30.5	45.3	19.6	32.1
	Product distribution (wt%)							
C_1–C_2	0.24	0.00	0.37	0.31	0.01	0.78	0.17	1.14
C_3	0.21	0.09	0.99	0.67	0.10	0.27	0.22	0.25
C_4	0.31	0.00	1.15	0.67	0.00	0.34	0.24	0.19
C_5	0.16	0.00	0.67	0.32	0.01	0.21	0.10	0.00
n-Nonane	0.58	0.00	0.00	1.28	0.00	0.00	0.00	0.00
C_9 Aromatics	1.01	0.00	3.69	5.09	0.00	0.00	0.00	0.00
C_{10}	53.14	0.00	61.26	53.30	17.07	27.34	41.86	25.59
Ether	8.72	24.22	4.06	4.04	37.39	17.72	13.32	23.62
Decanal	35.21	75.26	27.30	33.68	45.14	52.74	43.00	49.01
CO	0.13	0.00	0.13	0.37	0.07	0.19	0.24	0.08
CO_2	0.27	0.42	0.36	0.26	0.21	0.41	0.86	0.11

[a] Total conversion.
[b] Mass closure (mass fed/mass collected) * 100%.
[c] Contact time (grams ZSM-5/volumetric flow rate at reactor *T*, *P*).
[d] Weight hourly space velocity (grams fed per hour per gram of ZSM-5).

TABLE 5

1-Decanol Decomposition over Ni-ZSM-5

Catalyst/run	Ni/1	Ni/2	Ni/3	Ni/4	Ni/5	Ni/6	Ni/7
Temperature (K)	598	598	598	598	619	619	619
Pressure (atm)	14.0	8.96	14.50	6.99	9.69	5.73	7.80
Conversion[a] (%)	69.07	37.09	26.20	36.10	79.10	91.23	59.97
Mass closure[b] (%)	99.2	102.9	99.9	105.7	96.6	99.5	101.8
CT[c] (s-g/cm^3)	2.80	1.64	1.84	2.19	1.18	1.71	1.47
H_2/feed (mol/mol)	3.28	2.39	2.07	2.22	2.04	2.08	3.03
WHSV[d] (h^{-1})	14.17	19.22	30.55	11.79	31.12	12.57	15.26
	Product distribution (wt%)						
C_1–C_2	0.33	0.40	0.49	0.42	0.25	0.37	0.56
C_3	0.52	0.78	0.47	0.54	0.88	0.93	0.75
C_4	0.58	0.81	0.56	0.51	0.91	0.86	0.79
C_5	0.34	0.31	0.43	0.18	0.37	0.29	0.25
n-Nonane	0.00	4.04	0.00	1.93	0.00	6.98	3.21
C_9 Aromatics	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C_{10}	92.97	80.22	87.74	88.00	76.47	68.37	61.41
Ether	1.56	5.81	0.00	2.67	1.14	1.11	2.59
Decanal	3.63	7.64	10.32	5.75	19.56	20.76	30.11
CO	0.0	0.00	0.00	0.00	0.22	0.34	0.32
CO_2	0.07	0.00	0.00	0.00	0.20	0.00	0.00

[a] Total conversion.
[b] Mass closure (mass fed/mass collected) * 100%.
[c] Contact time (grams ZSM-5/volumetric flow rate at reactor *T*, *P*).
[d] Weight hourly space velocity (grams fed per hour per gram of ZSM-5).

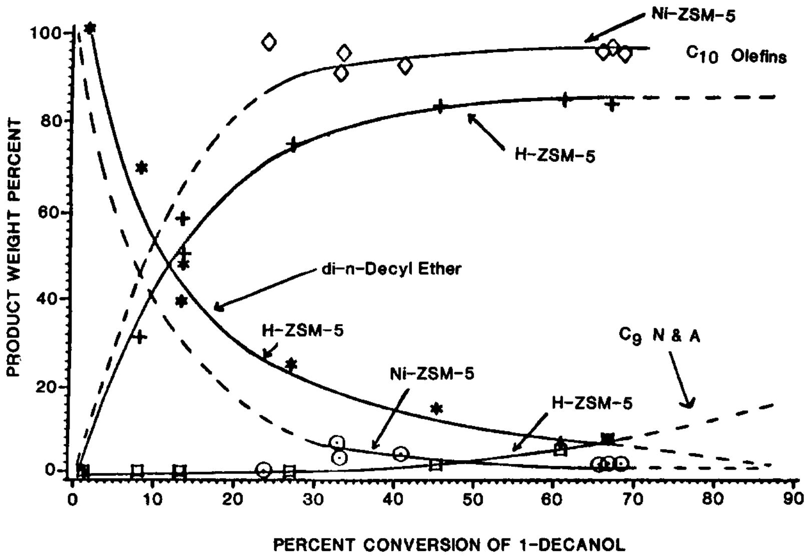


FIG. 1. Primary product distribution for decomposition of 1-decanol on Ni and H-ZSM-5 at 572 to 666 K.

mation by the reactor wall, are given in Tables 2 and 3. Table 2 gives the primary product distributions for 1-decanol dehydrating on H-ZSM-5. The primary products were di-*n*-decyl ether and decenes with some C_9 naphthenes and aromatics at higher temperatures. Table 3 shows the primary product distribution for 1-decanol on Ni-ZSM-5. Again most of the products were decenes and di-*n*-decyl ether. Slight traces of *n*-decane were found when using the Ni-ZSM-5 catalyst but C_9 naphthenes and aromatics were seen only on H-ZSM-5. Light hydrocarbon gas formation was less than 5 wt% in all cases.

Figure 1 shows the primary product distributions given in Tables 2 and 3 as a function of 1-decanol conversion (corrected for decanal formation). For H-ZSM-5, Fig. 1 shows that the concentration of di-*n*-decylether decreases as 1-decanol conversion increases. Simultaneously, the decenes increase with conversion. This behavior strongly suggests a series mechanism with dehydration proceeding via ether formation. The data collected on Ni-ZSM-5 were not low enough conversions to show the same features as observed with H-ZSM-5, but di-*n*-decyl ether was present over the entire temperature range examined.

Activation energies for dehydration were calculated for both catalyst. In both cases, the activation energy was found to be 21 kcal/gmole. This is roughly two thirds the energy of activation for cracking of *n*-heptane on Ni-ZSM-5 (*6*).

Results for Decanal

The range of reaction conditions are given in Table 6 and the product distributions and run conditions for each run are given in Tables 7 and 8. The product distributions of decanal were much more complex than those of 1-decanol due to three competing reactions. Qualitatively both

TABLE 6

Reaction Conditions for Decanal Decomposition

Catalyst	Temp (K)	Pressure (atm)	CT[a] (s-g/cm^3)	WHSV[b] (h^{-1})
Ni-ZSM-5	674–719	5.81–12.22	0.967–1.675	12.75–40.08
H-ZSM-5	622–722	5.93–10.57	0.653–1.156	24.36–54.01

[a] Contact time (grams catalyst/volumetric flow rate at reactor *T*, *P*).

[b] Weight hourly space velocity (grams fed per hour per gram of catalyst).

TABLE 7

Decanal Decomposition over H-ZSM-5

Catalyst/run	H/1	H/2	H/3	H/4	H/5	H/6	H/7
Temperature (K)	676	676	676	722	673	622	622
Pressure (atm)	7.67	5.93	9.40	8.86	7.39	9.55	10.57
Total conversion[a] (%)	26.6	25.3	19.0	59.6	17.4	26.6	30.7
Conv. to nonane[b] (%)	5.6	5.7	4.0	13.3	3.3	0.9	1.42
Mass closure[c] (%)	100.7	100.4	102.4	99.0	101.3	98.1	96.3
Contact time[d] (s-g/cm^3)	0.78	0.92	0.65	0.84	0.75	1.05	1.16
H_2/feed (mol/mol)	2.01	1.74	1.77	2.08	2.07	2.02	2.01
WHSV[e] (h^{-1})	33.01	24.36	54.01	33.31	33.40	34.23	34.32
			Product distribution (wt%)				
C_1–C_2	0.70	0.66	0.61	1.34	0.92	0.42	0.03
C_3	0.95	0.99	0.69	3.94	1.38	0.39	0.29
C_4	0.71	0.76	0.48	3.98	0.83	0.29	0.15
C_5	0.37	0.40	0.25	3.97	0.70	0.39	0.0
C_6–C_8	0.0	0.0	0.0	13.90	0.0	0.0	0.0
C_9 Aromatics	0.0	0.0	0.0	12.54	0.0	0.0	0.0
n-Nonane	16.87	18.37	15.43	19.25	14.58	3.07	4.47
C_{10}	13.98	4.49	5.33	10.57	0.0	29.95	25.90
Oxygenates[f]	59.01	66.72	69.85	21.84	73.29	41.95	45.29
Decanol	0.0	0.0	0.0	0.32	0.0	21.79	22.05
CO	3.85	4.38	4.60	5.08	4.76	1.07	1.45
CO_2	3.56	3.23	2.76	3.27	3.52	0.68	0.36

[a] Total conversion is grams converted to products/grams decanal fed.

[b] Conversion to nonane is moles nonane formed per mole decanal fed.

[c] Mass closure (mass fed/mass collected) * 100%.

[d] Contact time (grams catalyst/volumetric flow rate at reactor T, P).

[e] Weight hourly space velocity (grams fed per hour per gram of catalyst).

[f] Oxygenates (C_{18}–C_{20} aldol condensation products) other than decanol.

catalyst cause the formation of 20 to 70 wt% of oxygenated condensation products. These C_{18}–C_{20} oxygenated products were primarily C_{20} aldols and unsaturated aldehydes. The other compounds in the C_{18}–C_{20} range were most likely cross-condensation between decanol and decanal but were not identified. The C_{10}'s, olefins, traces of *n*-decane, and 1-decanol were between 0 and 50 wt%. Normal nonane composed between 3 and 28 wt%, and C_9 naphthenes and aromatics were produced at high temperatures on H-ZSM-5. C_1–C_8 compounds formed the remaining products and were less than 12% at temperatures below 700 K, but approached 35% at higher temperatures.

The cracking of decanal to *n*-nonane and CO was favored by high temperatures. The formation of condensation products was favored by lower temperatures. The conditions which favored hydrogenation of decanal to 1-decanol were not clear from these experiments, in part, due to the greater reactivity of decanol.

DISCUSSION

1-Decanol

The dehydration of 1-decanol over H-ZSM-5 and Ni-ZSM-5 appears to occur through a series of mechanism via di-*n*-decyl ether formation. The following mechanism is proposed:

$$2\ [\text{1-Decanol}] \rightleftarrows \text{Di-}n\text{-decyl ether} + H_2O \quad (1)$$

$$\text{Di-}n\text{-decyl ether} \rightarrow 2\ \text{Decene} + H_2O \quad (2)$$

TABLE 8

Decanal Decomposition over Ni-ZSM-5

Catalyst/run	Ni/1	Ni/2	Ni/3	Ni/4	Ni/5	Ni/6	Ni/7	Ni/8	Ni/9
Temperature (K)	675	674	673	697	697	697	719	719	719
Pressure (atm)	8.07	5.81	9.88	8.28	11.58	6.03	6.60	12.22	9.52
Total conversion[a] (%)	25.1	22.7	11.2	22.4	17.1	18.4	53.4	20.53	25.52
Conv. to nonane[b] (%)	3.8	4.6	3.3	5.9	5.8	4.8	8.5	6.6	6.6
Mass closure[c] (%)	98.3	100.0	102.5	99.9	100.1	101.0	94.4	99.7	98.7
Contact time[d] (s-g/cm^3)	1.28	1.57	1.09	1.29	0.97	1.56	1.68	0.99	1.12
H_2/feed (mol/mol)	2.21	2.00	1.92	2.30	2.01	1.97	2.02	2.01	3.07
WHSV[e] (h^{-1})	20.46	12.91	32.53	19.73	40.20	13.11	12.75	40.08	20.64
				Product distribution (wt%)					
C_1–C_2	0.99	0.95	1.09	1.68	1.56	2.18	2.35	2.66	2.83
C_3	2.63	1.90	1.30	4.26	2.94	4.77	7.46	5.94	5.90
C_4	2.42	1.47	0.99	3.16	2.02	3.21	5.68	3.67	3.49
C_5	1.42	0.86	1.07	1.84	1.40	1.50	6.91	2.47	2.37
C_6–C_8	0.0	2.37	0.0	0.0	0.0	0.0	11.85	0.0	0.0
C_9 Aromatics	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
n-Nonane	13.57	16.57	20.09	22.04	28.12	20.56	15.38	27.39	22.58
C_{10}	43.62	25.81	8.53	17.68	7.23	0.0	19.94	0.0	7.45
Oxygenates[f]	27.84	40.52	57.39	37.69	43.66	53.72	20.40	40.43	39.96
Decanol	0.0	2.20	0.0	0.0	0.0	0.0	0.0	0.0	0.0
CO	4.41	4.21	5.75	6.80	8.48	8.46	6.69	12.85	10.50
CO_2	3.10	3.14	3.79	4.84	4.59	5.60	3.34	4.59	4.93

[a] Total conversion is grams converted to products/grams decanal fed.

[b] Conversion to nonane is moles nonane formed per mole decanal fed.

[c] Mass closure (mass fed/mass collected) * 100%.

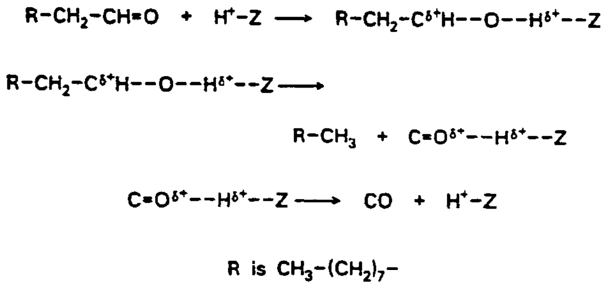
[d] Contact time (grams catalyst/volumetric flow rate at reactor *T*, *P*).

[e] Weight hourly space velocity (grams fed per hour per gram of catalyst).

[f] Oxygenates (C_{18}–C_{20} aldol condensation products) other than decanol.

This mechanism is essentially the same as described by Derouane *et al.* (*4*) for ethanol dehydration. The major difference is that Derouane *et al.* proposed a low temperature mechanism, diethyl ether formation, and then dehydration to ethylene; and a high temperature mechanism, direct dehydration to ethylene. The results illustrated in Fig. 1 and presented in Tables 2 and 3 indicate that only a single sequence occurs when 1-decanol is dehydrated over ZSM-5 and that failure to detect either intermediates may be due to ether formation being the rate-limiting step with subsequent ether dehydration to olefins being relatively fast.

The dehydration of 1-decanol by a consecutive ether–olefin mechanism was an unexpected result. The selectivity between olefin and ether formation is dependent on the chain length of the alcohol (Yue and Olaofe (*5*)) and ether formation is favored by shorter chain lengths. This raises the question of whether or not the reaction takes place on the surface of the catalyst. At high space velocities the decanol molecules may not have time to diffuse into the pores, forcing the reaction to occur on the surface in which case the long chain length would not matter. In addition, this work was done at pressures between 5.7 and 14.5 atm, whereas most studies of alcohol decomposition over zeolite catalyst have been done at pressures near or below atmospheric pressure. Condensation reactions, like ether formation, are generally favored by higher pressures.



$$R{-}CH_2{-}CH{=}O + H^+{-}Z \longrightarrow R{-}CH_2{-}C^{\delta+}H{-}{-}O{-}{-}H^{\delta+}{-}{-}Z$$

$$R{-}CH_2{-}C^{\delta+}H{-}{-}O{-}{-}H^{\delta+}{-}{-}Z \longrightarrow R{-}CH_3 + C{=}O^{\delta+}{-}{-}H^{\delta+}{-}{-}Z$$

$$C{=}O^{\delta+}{-}{-}H^{\delta+}{-}{-}Z \longrightarrow CO + H^+{-}Z$$

R is $CH_3{-}(CH_2)_7{-}$

FIG. 2. Mechanism for decanal decomposition to *n*-nonane and CO.

Decanal

The decomposition of decanal over Ni-ZSM-5 and H-ZSM-5 was observed to occur through three separate pathways. One mechanism was the reduction of decanal to 1-decanol:

$$\text{Decanal} + H_2 \rightleftarrows \text{1-Decanol}$$

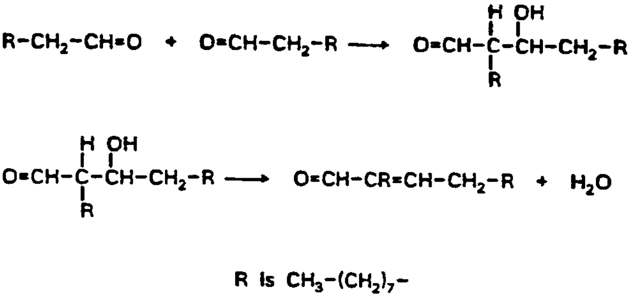
This reaction was catalyzed by the 316 SS reactor wall, which becomes activated over a long period of time. Ni-ZSM-5 probably has the ability to promote this reaction but the metal loading of the catalyst was insignificant when compared to the amount of Ni in the stainless steel.

Another pathway for decomposition was cleavage of CO to form *n*-nonane. We propose that CO cleavage is acid catalyzed as shown in Fig. 2. The third and least expected pathway was aldol condensation in which two decanal molecules condense to form a β-hydroxy carbonyl compound, an aldol. The aldol is not particularly stable under the reaction conditions and usually dehydrates to give an unsaturated aldehyde as shown in Fig. 3. The unsaturated C_{20} aldehyde was positively identified with gas chromatography–mass spectrometry.

The decomposition of decanal over ZSM-5 appeared to occur by three distinct pathways, one of which was aldol condensation. Chang and Sylvestri (*1*) found acid-catalyzed aldol condensation taking place with acetone but did not report any with propanal. Since the oxygen in propanal must be eliminated as CO, CO_2, or H_2O, the only way under the given reaction conditions to achieve 99.9% conversion with no production of CO or CO_2 would require aldol condensation between two propanal molecules with subsequent dehydration. Thus aldol condensation is a typical pathway for aldehydes and ketones reacting on H-ZSM-5 catalysts.

CONCLUSIONS

This study has shown that classical acid-catalyzed reactions of alcohols and alde-



$$R{-}CH_2{-}CH{=}O + O{=}CH{-}CH_2{-}R \longrightarrow O{=}CH{-}\underset{R}{\overset{H}{C}}{-}\overset{OH}{CH}{-}CH_2{-}R$$

$$O{=}CH{-}\underset{R}{\overset{H}{C}}{-}\overset{OH}{CH}{-}CH_2{-}R \longrightarrow O{=}CH{-}CR{=}CH{-}CH_2{-}R + H_2O$$

R is $CH_3{-}(CH_2)_7{-}$

FIG. 3. Mechanism for decanal aldol condensation.

hydes, which have been observed for smaller oxygenates also occur with the relatively large 1-decanol and decanal. The question of where the condensation reactions take place, the surface or the pore, has not been fully answered in this study since surface acid site poisoning was not performed. However, the products showed the same type of molecular sieving common to ZSM-5 which would indicate that the condensation reactions do in fact take place in the pores of H-ZSM-5.

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