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HIGH OCTANE ETHERS FROM SYNTHESIS GAS-DERIVED ALCOHOLS

Quarterly Technical Progress Report for the Period
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HIGH OCTANE ETHERS FROM SYNTHESIS GAS-DERIVED ALCOHOLS

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HIGH OCTANE ETHERS FROM SYNTHESIS GAS-DERIVED ALCOHOLS

OBJECTIVES OF THE RESEARCH

The objective of the proposed research is to synthesize high octane ethers, primarily methyl isobutyl ether (MIBE) and methyl tertiary butyl ether (MTBE), directly from $H_2/CO/CO_2$ coal-derived synthesis gas *via* alcohol mixtures that are rich in methanol and 2-methyl-1-propanol (isobutanol). The overall scheme involves gasification of coal, purification and shifting of the synthesis gas, higher alcohol synthesis, and direct synthesis of ethers.

The last stage of the synthesis involves direct coupling of synthesis gas-derived methanol and isobutanol that has been demonstrated by us to occur over superacid catalysts to yield methyl isobutyl ether (MIBE) at moderate pressures and a mixture of methanol and isobutene at low pressures. MIBE is an isomer of MTBE and a process is proposed whereby MTBE from the two alcohols is maximized and MIBE is minimized. This will be achieved by the proper choice of reaction conditions, i.e. intermediate pressures, and of inorganic acid catalysts that are stable at temperatures higher than 200°C, at which the carbonium ion reaction coupling of the two alcohols to MTBE is more effective than the oxonium ion or ester reaction coupling to MIBE.

Both organic and inorganic catalysts will be investigated, and the better catalysts of these classes will be subjected to long term performance studies. The long term performance studies of the combined process will extend to 1000 hr and detailed analytical data for all products will be provided. The project is divided into the following three tasks:

Task 1. Synthesis of High Octane Ethers from Alcohol Mixtures Containing Predominantly Methanol and 2-Methyl-1-Propanol over Superacid Resins

Task 2. Inorganic Catalysts for the Synthesis of High Octane Ethers from Alcohols

Task 3. Long Term Performance and Reaction Engineering for Scale-Up of the Alcohols-to-Ethers Process

The expected result of the proposed research is a novel process for producing ethers, in particular MTBE, in which all five carbons of the unsymmetric C_1 -O- C_4 ethers originate from coal-derived synthesis gas.

HIGH OCTANE ETHERS FROM SYNTHESIS GAS-DERIVED ALCOHOLS

SUMMARY OF TECHNICAL PROGRESS

The testing of strongly acidic catalysts for the conversion of a mixture of methanol and isobutanol to ethers and hydrocarbons was continued. Under standardized test conditions the catalysts tested this quarter were: phosphotungstic acid supported on zirconia ($\text{PW}_{12}/\text{ZrO}_2$), "niobic acid" ($\text{Nb}_2\text{O}_5 \cdot x\text{H}_2\text{O}$), and an iron and manganese doped sulfate-modified zirconia ($\text{Fe}/\text{Mn}/\text{ZrO}_2/\text{SO}_4^{2-}$). The overall activity of these catalysts followed the order of $\text{Fe}/\text{Mn}/\text{ZrO}_2/\text{SO}_4^{2-} > \text{PW}_{12}/\text{ZrO}_2 > \text{Nb}_2\text{O}_5 \cdot x\text{H}_2\text{O}$ with the $\text{Fe}/\text{Mn}/\text{ZrO}_2/\text{SO}_4^{2-}$ catalyst approaching $\text{ZrO}_2/\text{SO}_4^{2-}$ in both activity and selectivity for isobutene production.

The effect of the presence of water on the reaction of methanol and isobutanol over $\text{ZrO}_2/\text{SO}_4^{2-}$ was determined to be insignificant under the reaction conditions investigated. At 157°C and a feed of 2/1 molar ratio methanol/isobutanol under differential reaction conditions, the production of isobutene was unaffected by the addition of 0.05 and 0.10 parts of water to the 2/1 alcohol feed. A water effect might be observable if higher water addition rates were utilized.

Surface areas have been determined for $\text{Fe}/\text{Mn}/\text{ZrO}_2/\text{SO}_4^{2-}$, niobic acid, and phosphotungstic acid on silica. After calcination, the surface area for $\text{Fe}/\text{Mn}/\text{ZrO}_2/\text{SO}_4^{2-}$ was found to be 83 m²/g. Niobic acid and phosphotungstic acid on silica ($\text{PW}_{12}/\text{SiO}_2$) were found to have surface areas of 118 and 218 m²/g, respectively. ¹H NMR was used to determine if and how much 1-butene was present as a product when isobutanol was dehydrated over sulfate-modified zirconia. It was found to be present in small amounts, ≤3% of the product stream.

TECHNICAL PROGRESS

A. Results of Standard Tests Using Supported Heteropolyacid $\text{PW}_{12}/\text{ZrO}_2$, Nb_2O_5 , and $\text{Fe}/\text{Mn}/\text{ZrO}_2/\text{SO}_4^{2-}$ Catalysts

The screening of very strong solid acids as catalysts for the conversion of MeOH/i-BuOH mixtures was continued. New catalysts tested were (i) 20 wt% phosphotungstic acid supported on ZrO_2 ($\text{PW}_{12}/\text{ZrO}_2$), (ii) niobic acid ($\text{Nb}_2\text{O}_5 \cdot x\text{H}_2\text{O}$), and (iii) an iron and manganese doped sulfate-modified zirconia ($\text{Fe}/\text{Mn}/\text{ZrO}_2/\text{SO}_4^{2-}$). The preparation and pretreatment of these new catalysts follows.

Preparation

Supported heteropolyacids are used as heterogeneous catalysts in several industrial applications. They are reported to possess strong acidic character, as well as unique selectivity characteristics in hydration/dehydration reactions. The use of zirconia-supported phosphotungstic acid, designated herein as $\text{PW}_{12}/\text{ZrO}_2$, for the standard reaction of a 1/1 molar MeOH/i-BuOH mixture was investigated.

Phosphotungstic acid with a formula of $\text{H}_3\text{PW}_{12}\text{O}_{40}$, thus designated as PW_{12} , was obtained from Fisher Scientific, Co. This compound is also sometimes referred to by others as tungstophosphoric acid or 12-polytungstophosphoric acid. An aqueous solution consisting of 1.68 g of phosphotungstic acid in 5.5 ml of distilled water was prepared. The $\text{Zr}(\text{OH})_4$, 6.72 g, prepared from hydrolyzed $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, was impregnated with this solution, with constant mixing, to make a 20% loading of phosphotungstic acid by weight. The impregnated zirconium hydroxide, $\text{PW}_{12}/\text{Zr}(\text{OH})_4$, was placed in a 100°C oven overnight. The dried sample was then calcined to 300°C for 3 hr. This calcination temperature was chosen because it is below the decomposition range reported for heteropolyacids.

"Niobic acid", actually hydrated niobium oxide ($\text{Nb}_2\text{O}_5 \cdot x\text{H}_2\text{O}$), was calcined at 200°C for 2 hr. A literature report indicates that high surface area and strong acidity are maintained only with low calcination temperatures such as this (1).

Recently, an iron and manganese doped sulfate-modified zirconia catalyst ($\text{Fe/Mn/ZrO}_2/\text{SO}_4^{2-}$) has been reported to possess stronger and more populous acid sites than $\text{ZrO}_2/\text{SO}_4^{2-}$ (2). This catalyst was also reported to be many times more active for the low temperature isomerization of butane.

The $\text{Fe/Mn/ZrO}_2/\text{SO}_4^{2-}$ catalyst was prepared according to the method described in Ref. 2. This involved first dissolving 73 g of $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ in *ca.* 100 ml of distilled water. About 700 ml of aqueous ammonia was added, and the solution was stirred for 2 hr. The Zr(OH)_4 formed was filtered and washed with distilled water until free of chloride ion. This solid was put in an oven at 100°C overnight to dry. The solution used to impregnate the Zr(OH)_4 consisted of 1.133 g $(\text{NH}_4)_2\text{SO}_4$, 2.234 g of $\text{Fe(NO}_3)_3 \cdot 9\text{H}_2\text{O}$, and 0.658 g of a 51 wt% solution of $\text{Mn(NO}_3)_2$. These salts were dissolved in distilled water to make 26.5 ml that was used to impregnate 25 g of Zr(OH)_4 . The impregnated catalyst was dried in a 110°C oven overnight followed by calcination at 620°C for 3 hr. The catalyst preparation was designed to give 1.5% Fe, 0.5% Mn, and 4.0% SO_4^{2-} by weight supported on ZrO_2 .

Catalytic Testing Results

The samples were diluted with Pyrex beads, charged to the reactor, and tested using the following reaction conditions:

Temperature	90, 125, 150, 175°C , (also 200 and 225°C)
Pressure	1 atm
Methanol feed	1.72 mol/kg catalyst/hr
Isobutanol feed	1.72 mol/kg catalyst/hr
He + N_2 flow	16.0 mol/kg catalyst/hr
Catalyst weight	5.0 g

The catalytic results for these three samples are presented in two forms. One presentation of the data is in the form of rate of formation of each of the products, with the units of mol of product/kg catal/hr. This is also referred to as the space time yield or the productivity. In this case, the % conversion and % selectivity of each reactant can be directly calculated if the space time yields of all significant products are plotted, as they are in this report.

The second representation of the experimental data is %yield of each of the products. This latter quantity is defined as (% conversion of one of the reactant x % product selectivity)/100. Thus, for 50% conversion of isobutanol to isobutene with 75% selectivity, the %yield would be 37.5%. It can also be directly calculated from the rate of formation, i.e. %yield = (rate of formation of product/rate of reactant flow) x 100. For example, 0.645 mol isobutene/kg catal/hr under the present reaction conditions = 37.5 %yield. This last definition of %yield, as written, presumes that one mole of reactant forms one mole of product, e.g. isobutanol to isobutene (or MIBE). However, two moles of methanol can form one mole of dimethylether. In this case, it must be clear whether the %yield refers to comparisons of products containing the methyl group from methanol or molar conversions of methanol to products. As an example, the formation of 0.129 mol DME/kg catal/hr (20% conversion of methanol with 75% selectivity to DME) would correspond to a %yield of 15% because 0.258 mol of methanol/kg catal/hr would be converted to DME. If the basis were moles of products formed that contain one or more methyl groups derived from methanol, then the %yield of DME would be 7.5% ((0.129/1.72) x 100).

The zirconia-supported heteropolyacid catalyst, $\text{PW}_{12}/\text{ZrO}_2$, exhibited low activity and poor selectivity, as indicated in Figures 1-3, relative to other catalysts tested previously (3-4). This is especially true with respect to the $\text{SO}_4^{2-}/\text{ZrO}_2$ -based catalyst to be discussed in additional detail later in this report. As shown in Figure 1, isobutene is the principal

FIGURE 1

20 wt% $\text{PW}_{12}/\text{ZrO}_2$ Catalyst
MeOH/*i*-BuOH = 1/1 Reactant at 1 atm

FIGURE 2

20 wt% $\text{PW}_{12}/\text{ZrO}_2$ Catalyst
MeOH/i-BuOH = 1/1 Reactant at 1 atm

FIGURE 3

20 wt% $\text{PW}_{12}/\text{ZrO}_2$ Catalyst

MeOH/i-BuOH = 1/1 Reactant at 1 atm

product formed with a space time yield of $0.254 \text{ mol} \cdot \text{kg}^{-1} \cdot \text{hr}^{-1}$, while the rate of production of DME was $0.147 \text{ mol} \cdot \text{kg}^{-1} \cdot \text{hr}^{-1}$. This means that more methanol was converted to DME than the quantity of isobutanol that was converted to isobutene. However, based on the quantities of products formed, the molar selectivities were 29.2% DME, 50.5% isobutene, 8.5% *trans*-2-butene, 8.0% *cis*-2-butene, and 3.8% MIBE.

The overall conversions at 175°C corresponded to 20.7 mol% conversion of isobutanol to products and 18.2 mol% of methanol to products. The %yields are shown in Figure 2, and these are based on the conversion levels of the reactants. Just for comparison purposes, the %yields are shown in Figure 3 on the basis of product molar composition, where the only difference between Figures 2 and 3 are in the values of DME since DME contains two methyl groups, as already discussed. Earlier, it had been found that pure ZrO_2 was totally inactive for these reactions. Therefore, the activity of the $\text{PW}_{12}/\text{ZrO}_2$ catalyst can be attributed to the presence of the phosphotungstic acid ($\text{H}_3\text{PW}_{12}\text{O}_{40}$).

The niobic acid catalyst was essentially inactive at and below 175°C , as shown in Figures 4 and 5. At 200°C , the methanol and isobutanol conversions levels were 9.3 and 10.5%, respectively. When higher temperatures were used, most notably at 225°C , the activity and selectivity approached that seen for the best catalyst tested so far, the $\text{ZrO}_2/\text{SO}_4^{2-}$ catalyst. At 225°C , as shown in Figure 4, this $\text{Nb}_2\text{O}_5 \cdot \text{H}_2\text{O}$ niobic acid catalyst produced $1.0 \text{ mol} \cdot \text{kg}^{-1} \cdot \text{hr}^{-1}$ of isobutene and only $0.13 \text{ mol} \cdot \text{kg}^{-1} \cdot \text{hr}^{-1}$ of DME. In comparison, the $\text{ZrO}_2/\text{SO}_4^{2-}$ catalyst produced $1.1 \text{ mol} \cdot \text{kg}^{-1} \cdot \text{hr}^{-1}$ of isobutene and only $0.10 \text{ mol} \cdot \text{kg}^{-1} \cdot \text{hr}^{-1}$ of DME, which was achieved at the lower temperature of 175°C (3). With the Nb_2O_5 catalyst at 225°C , the reactant conversions were 70.2% isobutanol and 19.6% methanol to products, and Figure 5 shows that the %yields of isobutanol to isobutene and of methanol to DME were 58.7 and 15.5 %, respectively.

FIGURE 4

$\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ Catalyst

$\text{MeOH}/i\text{-BuOH} = 1/1$ Reactant at 1 atm

FIGURE 5

$\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ Catalyst
 $\text{MeOH}/i\text{-BuOH} = 1/1$ Reactant at 1 atm

The catalytic results for the Fe/Mn/ZrO₂/SO₄²⁻ catalyst were similar at the higher reaction temperatures employed to those of the ZrO₂/SO₄²⁻ catalyst (3). At 175°C, the Fe- and Mn-doped catalyst produced 0.91 mol*kg⁻¹*hr⁻¹ of isobutene and 0.03 mol*kg⁻¹*hr⁻¹ of DME, as shown in Figure 6. However, at lower temperature, the Fe/Mn/ZrO₂/SO₄²⁻ catalyst as prepared here was considerably less active than the ZrO₂/SO₄²⁻ catalyst. For example, at 150°C the yield of isobutene was only 0.24 mol*kg⁻¹*hr⁻¹ for Fe/Mn/ZrO₂/SO₄²⁻ compared to 0.61 mol*kg⁻¹*hr⁻¹ for ZrO₂/SO₄²⁻ (3).

This Fe/Mn/ZrO₂/SO₄²⁻ catalyst exhibited a greater selectivity in activating isobutanol relative to methanol than the PW₁₂/ZrO₂ catalyst and the Nb₂O₅·H₂O catalyst. The overall conversion levels for the former sulfated catalyst at 175°C were 61.3% isobutanol to products and 6.2% methanol to products. The %yields for this catalyst are shown in Figure 7. Figures 6 and 7 demonstrate that this catalyst was quite selective for the dehydration of isobutanol to isobutene, specifically 86.6 mol% of the C₄-containing products was isobutene and 84.4 mol% of all significant products, not including water, was isobutene.

B. The Effect of Water on Reaction of MeOH/i-BuOH Over ZrO₂/SO₄²⁻

A major product of any conversion of the methanol/isobutanol mixture over solid acid catalysts is water. The presence of water can influence the type of acid sites present, e.g. it can often convert Lewis acid sites into Brønsted acid sites. In addition, water may also lead to a retarding effect on the reaction, as was reported for alumina earlier (4). This latter effect may arise by competitive adsorption of water and partial blockage of active sites. In this experiment, reaction conditions were chosen where a 2/1 mixture of MeOH/i-BuOH was reacted over a 1% SO₄²⁻/ZrO₂ catalyst at 157°C and 1 atm within the differential regime as described in the previous quarterly report (5). Specifically, the

FIGURE 6

Fe/Mn/ZrO₂/SO₄²⁻ calcined at 620°C
MeOH/i-BuOH = 1/1

FIGURE 7

Fe/Mn/ZrO₂/SO₄²⁻ calcined at 620°C
MeOH/i-BuOH = 1/1

conditions were:

Temperature	157°C
Pressure	1 atm
Water feed	0, 1.08, 2.16 mol/kg catalyst/hr
Methanol feed	43.3 mol/kg catalyst/hr
Isobutanol feed	21.6 mol/kg catalyst/hr
He + N ₂ flow	762 mol/kg catalyst/hr
Catalyst weight	0.4 g

The catalyst was first exposed to the dry 2/1 MeOH/i-BuOH mixture, then to a MeOH/i-BuOH/H₂O = 2/1/0.05 mixture, followed by a 2/1/0.1 mixture. The feed was again switched to the dry 2/1 mixture and then back to the 2/1/0.1 MeOH/i-BuOH/H₂O mixture. The results of this experiment are shown in Figure 8. The high gas hourly space velocity (GHSV) of the carrier gas ensured low conversion levels of the reactants, e.g. at 100 hr on stream the conversion of isobutanol to products was about 4 mol% ($\approx 3.7\%$ yield of isobutene). It can be seen from Figure 8 that the catalyst gradually and steadily deactivated with time on stream. However, the presence or absence of water in the feed did not affect the yield or selectivity of the products, which consisted mainly of isobutene.

Another experiment with a fresh catalyst was carried out wherein it was first exposed to the water-containing reactant mixture and then to the dry alcohol reactant mixture. No difference in effect from the previous experiment was observed. These results differ from those observed earlier for alumina, where a strong inhibition, *ca.* 30%, in the production of DME, MIBE, and isobutene was noted (4). Those results were explained with the knowledge that alumina is known to possess strong acidity only in the form of Lewis acid sites that depend in number and strength on the degree of hydration of the surface. In the case of ZrO₂/SO₄²⁻, however, Arata has shown (6) that Lewis acid sites can be converted to strong Brønsted acid sites upon exposure to low partial pressures of water, i.e. 5 torr. In

FIGURE 8

$\text{ZrO}_2/\text{SO}_4^{2-}$ with and without H_2O in the reactants at 157°C and 1 atm

$\text{MeOH}/i\text{-BuOH}/\text{H}_2\text{O} = 2/1/x$ molar ratio

the present investigation, in the "dry" runs where the only water present was that formed as a product of the reaction, the partial pressure of water was less than 1 torr. In the "wet" runs, the partial pressure of water was about 3 torr. Under high conversion conditions, the water produced during reaction can reach rather high levels. For example, over the Fe/Mn/ZrO₂/SO₄²⁻ catalyst at 175°C (Figure 6), the water partial pressure generated by isobutanol dehydration corresponded to ≈42 torr, under non-differential reaction conditions. This approached the initial partial pressures of isobutanol and methanol reactants, which was ≈67 torr. Thus, perhaps additional experiments need to be carried out under differential reaction conditions, but where appreciably higher water injection rates are utilized to maintain higher levels of water partial pressures over the catalyst. We are currently investigating whether the state of this catalyst changes drastically in the presence of water during the course of different reaction conditions through direct spectroscopic means.

C. Surface Area Measurements

Surface areas have been determined for samples of Fe/Mn/ZrO₂/SO₄²⁻ and niobic acid (Nb₂O₅·xH₂O), prepared as described in Section A of this report, as well as the 12-phosphotungstic acid on silica catalyst (PW₁₂/SiO₂) that was prepared and tested as described in the previous quarterly report (5).

Experimental

Samples were analyzed with a Gemini 2360 Analyzer instrument from Micromeritics. Surface area was determined at -196°C by using the BET multipoint technique. Points were chosen so that p/p₀ varied from 0.05 to 0.3. This yielded a linear BET-plot and accurate determination of the surface area. Samples were generally degassed at 300°C for 2 hr prior

to exposure of N₂ at -196°C.

Results

Surface areas were determined for Fe/Mn/ZrO₂/SO₄²⁻ samples dried at 100°C and after calcination at 620°C. The uncalcined sample of Fe/Mn/ZrO₂/SO₄²⁻ was degassed at 120°C, whereas the calcined Fe/Mn/ZrO₂/SO₄²⁻, niobic acid, and phosphotungstic acid on silica (PW₁₂/SiO₂) catalysts were degassed at 300°C. The measured surface areas are given in Table 1. The surface area determined for calcined Fe/Mn/ZrO₂/SO₄²⁻ is in good agreement with the reported value of 97 m²/g (2).

Table 1. Surface Areas

Catalyst	Surface Area (m ² /g)
Fe/Mn/ZrO ₂ /SO ₄ ²⁻ dried to 100°C	219
Fe/Mn/ZrO ₂ /SO ₄ ²⁻ calcined to 620°C	83
Niobic Acid	118
(PW ₁₂ /SiO ₂)	218

D. ¹H NMR to Resolve Whether 1-Butene is a Product

Our main technique to identify reactants and products is gas chromatography (GC). Isobutene is an abundant product when isobutanol is dehydrated over acid catalysts. As shown, other possible dehydration products are cis-2-butene, trans-2-butene and 1-butene. By using GC, with our present method of separation using a Cp-Sil-5 capillary WCOT column, isobutene can be separated from cis-2-butene and trans-2-butene but not from 1-butene. To resolve if and how much 1-butene is present as a product over the sulfated zirconia, ¹H NMR was utilized to further distinguish the C₄ products.

Experimental

Products from dehydration of isobutanol over sulfated zirconia were collected in a N₂ (*l*) cooled trap. The product stream was dried with molecular sieves prior to the trap to remove as much water as possible. As standards, individual samples of pure 1-butene, isobutene, *cis*-2-butene, and *trans*-2-butene were collected from their respective gas cylinders.

A 500 MHz Bruker AM500 NMR instrument was utilized for these analyses. The spectra were acquired from 0 to 8 ppm at approximately -20°C. All samples were diluted approximately 1:4 with CDCl₃. The pure butene samples were used to calibrate peak positions.

Results

Isobutene has distinctive ¹H NMR peaks at 4.65 and 1.7 ppm (Figure 9), whereas 1-butene has groups of peaks at approximately 5.9, 4.9, 2.1, and 1.0 ppm (Figure 10). The spectra of the pure butenes were compared to the spectrum of the dehydration products of isobutanol (Figure 11).

From the NMR spectra, it is concluded that 1-butene was indeed present in the product stream in a small amount. By integrating the NMR peaks at 5.9 and 4.65 ppm, the quantity of 1-butene could be determined. Neither *cis*-2-butene nor *trans*-2-butene have any peaks in this region. It was found that the product stream consist of ≤3% 1-butene.

FIGURE 9

Spectrum of pure isobutene in CDCl_3

MAR1STD1
DATE 21-12-92
TIME 9:26

SOLVENT CCl_3
SF 500.135
SF0 500.130
SF02 500.130
SF2 500.135
SY 166.0
Q1 7941.000
SI 32768
ID 32768
SW 6024.096
SH2 6024.096
HZ/PT .368
VD 0.0

PW 1.0
RD 0.0
AQ 2.720
RG 1
NS 16
TE 300

DE 106.3
DR 12
DW 83
FW 7600
Q2 50000.000
QF 63L P0

LB 0.0
GB 0.0
NC 0
CX 20.00
CY 15.00
F1 7.500P
F2 .001P
MI .01
DC 1.000
HZ/CM 187.536
PPM/CM .375
IS 1
SR 5432.62

FIGURE 10

Spectrum of pure 1-butene in CDCl_3

HQ.DMSO
DATE 21-12-92
TIME 8:59

SOLVENT CCl_3
SF 500.135
SF0 500.130
SF02 500.130
SF2 500.135
SY 166.0
O1 7941.000
SI 32768
TD 32768
SM 6024.096
SW2 6024.096
HZ/PT .368
VD 0.0

PW .0
RD 0.0
AQ 2.720
RG 1
NS 16
TE 300

DE 105.3
DR 12
DW 83
FW 7500
O2 50000.000
OP 63L P0

LB 0.0
GB 0.0
NC -1
CX 20.00
CY 15.00
F1 7.500P
F2 .001P
MI .01
DC 1.000
HZ/CM 187.536
PPM/CM .375
IS 1
SR 5432.62

FIGURE 11

Spectrum of isobutanol
dehydration products

 FID3
 DATE 14-12-92
 TIME 9:59
 SOLVENT CDCl3
 SF 500.135
 SF0 500.130
 SF02 500.130
 SF2 500.135
 SY 166.0
 O1 7941.000
 SI 32768
 TD 32768
 SW 6024.096
 SW2 6024.096
 HZ/PT 368
 VD 0.0
 PW 2.0
 RD 0.0
 AQ 2.720
 RG 4
 NS 228
 TE 300
 DE 106.3
 DR 12
 DW 83
 FW 7600
 O2 50000.000
 DP 63L P0
 LB 0.0
 GB 0.0
 NC 5
 CX 20.00
 CY 0.0
 F1 8.000P
 F2 8.000P
 MI 0.01
 DC 1.000
 HZ/CM 200.056
 PPM/CM 400
 IS 1
 SR 5433.73

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