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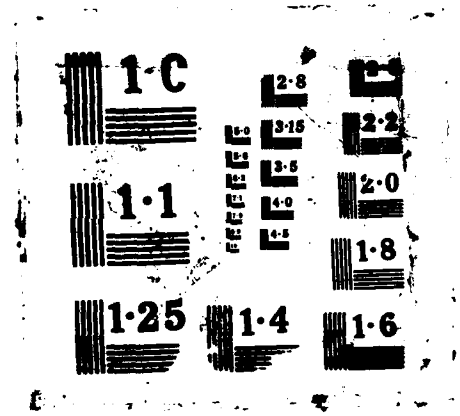
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DECOMPOSITION OF METHANOL, ACETALDEHYDE, AND ACETONE ON SUPPORTED RHODIUM CATALYSTS

BY

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Decomposition of Methanol, Acetaldehyde, and Acetone on Supported Rhodium Catalysts

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Abstract

The decomposition processes for methanol, acetaldehyde, and acetone over supported rhodium catalytic films have been investigated using infrared spectroscopy. An interesting support effect was observed for methanol decomposition with methane being the primary product over Rh/TiO_2 and dimethyl ether being formed over Rh/Al_2O_3. In the latter case the Al_2O_3 support was the active catalyst. For acetaldehyde decomposition the principle products were methane and acetone for both supported rhodium catalysts. Acetone decomposition produced only inactive carbon with no other infrared-detectable surface species. It is possible that a metallic rhodium gem dicarbonyl adsorbate was produced during the decomposition of acetaldehyde over Rh/Al_2O_3. Previous observations of the rhodium gem dicarbonyl species have referred to dispersed Rh^+ ions.

Introduction

A considerable amount of work has been performed in these laboratories and elsewhere focused upon the elucidation of the mechanisms for the hydrogenation reactions of CO and CO_2 over supported rhodium catalysts.[1] The product distributions for these two reactions depend makedly upon the reaction conditions, with variables including (at least) temperature, pressure, nature of the support, and the presence or absence of alkali metal promotors. For example, a recent study of CO hydrogenation over Rh/TiO_2 showed that the presence of potassium ions poisoned the methanation reaction, but enhanced the yield of the oxygenated products acetaldehyde and acetone.[2] Our interest is currently directed toward the detection of intermediates in the hydrogenation reactions mentioned above using infrared spectroscopy as a primary analytical probe. We have been able to detect a rhodium carbonyl hydride species during the production of methane and higher hydrocarbons[1,3,4] in accord with the previous work of Solymosi and coworkers.[5-8] We now wish to extend our work to the detection of intermediates leading to the oxygenated products. The current paper will describe our observations concerning the reverse processes (decomposition of methanol, acetaldehyde, and acetone over supported rhodium catalyst films). The decomposition reactions of methanol over Rh(111)[9] and on the supports employed here[10-14] have been investigated previously, but not by using the infrared cell-reactor techniques employed in these laboratories.

Experimental Section

The catalytic sample preparation procedure developed here for Rh/TiO_2 and Rh/Al_2O_3 films has been described elsewhere[1-4] but will be reiterated briefly here. Slurries of $RhCl_3 \cdot 3H_2O$, support material (Degussa Titanium Dioxide P25, 50 m^2g^{-1} or Degussa Aluminum Oxide C, 100 m^2g^{-1}), in some cases KCl, and water/acetone were sprayed from an atomizer onto a 25 mm CaF_2 infrared window held at ca. 360 K. Evaporation of the solvents left a thin film (ca. 4.3 mg cm^{-2}) of $RhCl_3 \cdot 3H_2O/TiO_2$ (or Al_2O_3), which in some runs contained potassium ions, adhered to the CaF_2 window. The window containing the catalytic film was mounted inside a Pyrex infrared cell reactor[1] which was then evacuated to 10^{-6} Torr using an ion pump. The sample was reduced by 85 Torr doses of hydrogen at 480 K for periods of 5, 5, 10, and 20 min, each reduction cycle being followed by evacuation. Prior studies in these laboratories and elsewhere have shown that this procedure provides a mixture of isolated Rh^+ ions and Rh^0 clusters; the Cl^- is lost as HCl. Following heating for 1 h at 490 K under vacuum, the samples were evacuated overnight to 10^{-6} Torr at ambient temperature. Then 20.0 Torr of either methanol, acetaldehyde, or acetone was introduced into the cell reactor. These materials had been purified by repetitive freeze-thaw evacuation cycles. After recording the infrared spectrum (Perkin-Elmer 983 with data system) at 300 K, the catalyst sample was brought to 440 K, and the infrared spectra were monitored as a function of time. Gaseous products were analyzed at the conclusion of the experiments using chromatography (Carle 400). Pressure measurements were made with an MKS Baratron capacitance manometer (± 0.01 Torr). Runs involving support material only were made using the same general procedure.

Results and Discussion

Figures 1 and 2 show the infrared spectra for the methanol decomposition process over 2.2% Rh/TiO_2 and 2.2% Rh/Al_2O_3, respectively. A comparison of the analogous spectra in the two figures reveals that there was a significant support effect. Spectrum 1a shows that even at ambient temperature the Rh/TiO_2 film began to catalyze the decomposition of methanol, as some adsorbed

CO features were detected near 2000 cm^{-1} which overlap the C-O stretching overtone band[15] for CH_3OH (2035, P; 2056, Q; 2078, R). Heating for 1.5 h at 440 K (Fig. 1b) caused significant decomposition as evidenced by the declination of band intensity in the C-H stretch region (2850 and 2950 cm^{-1}) and the CO stretch region (1012, P; 1034, Q; 1060, R)[15], with a concomitant enhancement of the adsorbed CO bands in the range 1850-2120cm^{-1}. Based upon extensive prior work in these laboratories,[1,16,17] these bands can be assigned to CO stretching modes as follows: 1850, bridged dirhodium carbonyl; 1975, carbonyl bridged between Rh and the support TiO_2; 2037, the rhodium carbonyl hydride species; 2050, a "linear CO" rhodium species; 2120, CO adsorbed on Rh^{+2} (or at least in an oxidation state higher than +1)[16,18]. It is interesting that there was no evidence of sharp twin bands at 2030 and 2100 cm^{-1} indicating that the isolated Rh^{+1} "gem dicarbonyl" species was not formed during the decomposition of methanol. It is well known that CO gas causes disruption of Rh clusters to produce highly dispersed Rh^{+1} sites,[19,20] but evidently methanol is not capable of causing similar behavior. Figure 1c shows the result of further decomposition at 440 K (after 5.5 h); the bands attributed to methanol and CO surface species have greatly declined in intensity, and those present at 1304/3015 cm^{-1} and 2349 cm^{-1} indicate the formation of gas-phase methane and carbon dioxide. Analysis by gas chromatography indicated that at this point the gas-phase methanol was completely decomposed. Comparison with spectrum 1e indicates that under the same reaction conditions TiO_2 alone causes some decomposition of CH_3OH, but no CH_4, CO_2, or CO surface species were detected. It is clear that Rh plays an active role in the decomposition of CH_3OH when TiO_2 is the support. Spectrum 1d shows the result of adding potassium ions (K:Rh=1) to the 2.2% Rh/TiO_2 catalyst. This caused a substantial decrease in the rate of decomposition of

CH_3OH (86% decomposition indicated by GC), an elimination of CH_4 as a product, and a decline in the production of CO_2 and most adsorbed CO species. However, the bridged dirhodium carbonyl species was significantly enhanced. The increase of this CO species of Rh and the poisoning of the methanation reaction in the presence of potassium ions has been noted previously for the hydrogenation of CO and CO_2 over supported rhodium films.[2]

In contrast to the results noted for 2.2% Rh/TiO_2 above, for the 2.2% Rh/Al_2O_3 catalyst only the rhodium carbonyl hydride and bridged dirhodium carbonyl adsorbed CO species were produced in significant amounts (Fig. 2b, c), no decomposition occurred at 300 K, CH_4 and CO_2 were not produced in significant amounts, and the primary decomposition product was dimethyl ether (bands at 1180 and 1110 cm^{-1}, verified by gas chromatography). However, a comparison of spectra 2c and 2e indicates that the dimethyl ether production is catalyzed primarily by the Al_2O_3 support rather than by rhodium. Spectrum 2d shows that the presence of potassium ions poisons the production of dimethyl ether indicating that the K^+ is spread over the support as well as on Rh. The K^+ ions must also interact with Rh because the carbonyl hydride species was diminished, and the bridged dirhodium carbonyl species was enhanced when potassium was present. We would postulate that the potassium poisons the active Rh methanation sites by aiding the formation of inactive carbon on these sites and enhancing the formation of the bridged dirhodium carbonyl species, which we have noted before does not yield methane under the conditions (440 K and low pressure) of our experiments.[2] As noted before, the potassium ions also poison the production of dimethyl ether on the Al_2O_3 support. This is probably a result of blockage of Lewis acid sites, as pointed out by Busca <u>et</u> <u>al</u>. in their studies of the interaction of methanol with KOH doped TiO_2.[14]

Thus it might be postulated that under the conditions of our experiments, Rh/TiO_2 catalyzes the rupture of the C-O bond in methanol to produce active carbon (CH_3) which subsequently leads to CH_4 and CO_2, as products, while Rh/Al_2O_3 assists the rupture of the O-H bond in methanol as well to form CH_3O which subsequently leads to the production of dimethyl ether. In the latter case the Rh performs little useful purpose with the Al_2O_3 providing the catalysis.

Figures 3 and 4 present the infrared spectra for the decomposition of acetaldehyde over 2.2% Rh/TiO_2 and 2.2% Rh/Al_2O_3, respectively. In this case the results were similar for the two different supported catalysts. Spectrum 3a indicates that little acetaldehyde decomposed at 300 K, but upon heating at 440 K (spectra 3b,c), marked decomposition occurred (85% as measured by GC) to form CH_4 (1304 cm^{-1}), CO_2 (2349 cm^{-1}), CO (2143 cm^{-1}), adsorbed bridged rhodium carbonyl species (1800-2000 cm^{-1}), surface carbonate and acetate species (1410, 1445, 1526, 1576 cm^{-1}), and acetone (1240, 1340 cm^{-1}). Decomposition occurred over the pure TiO_2 support material also (Fig. 3e), but only surface acetate bands were observed. Thus it can be concluded that the acetate species was confined primarily to the TiO_2 support. It should be noted that Orita <u>et al</u>. have detected adsorbed acetate ions during the formation of acetaldehyde from the hydrogenation of CO over Rh/SiO_2.[21] Prior work in these laboratories suggested that acetaldehyde and acetone were produced from the bridged dirhodium carbonyl species during the hydrogenation of CO over 2.2% Rh/TiO_2, while methane was produced from the other adsorbed CO species.[2] Spectrum 3c shows that the only adsorbed CO/Rh species produced during the decomposition of acetaldehyde was the bridged carbonyl species. Spectrum 3d indicates that the presence of potassium ions poisoned, to some extent, the decomposition of acetaldehyde (reduced to 54%) and eliminated the formation of CH_4 and acetone. Most likely the potassium ions cause the formation of inactive carbon at the expense of the active carbon surface species which normally yield hydrocarbon and oxygenated products.

Figure 4 shows that the decomposition of acetaldehyde over 2.2% Rh/Al_2O_3 again produced CH_4, although very little CO_2 or CO, surface acetate, and acetone. Spectrum 4e indicates that the acetate species was primarily confined to the Al_2O_3 support. Again potassium ions (spectrum 4d) poisoned the production of CH_4 and acetone. The most interesting feature appears in spectrum 4b. Upon initial heating at 440 K, twin adsorbed CO bands occurred at 2012 and 2082 cm^{-1}. It is well known[16] that the presence of sharp twin bands which grow and decline in intensity together in this region of the spectrum are indicative of the presence of a rhodium gem dicarbonyl species. However, it is also a fact that these twin bands generally appear at ca. 2030 and 2100 cm^{-1} and refer to a highly dispersed Rh^+ gem dicarbonyl.[16] The gem dicarbonyl bands in spectrum 4b which do grow proportionately appear 18 cm^{-1} lower which could very well mean that acetaldehyde decomposes over Rh/Al_2O_3 to yield a rhodium gem dicarbonyl species with Rh in a zero oxidation state. If so, to our knowledge, this is the first infrared data which has been interpreted as evidence for the existence of a metallic Rh gem dicarbonyl species. Yates and Cavanagh also observed abnormally low frequency infrared bands (2021 and 2088 cm^{-1}) for a Rh gem dicarbonyl species during the decomposition of formaldehyde over 2.2% Rh/Al_2O_3, but they attributed the low frequency of the bands as being due to support perturbation of the CO modes by the presence of oxide-bound species derived from H_2CO.[22] It is possible that the Rh gem dicarbonyl species produced from H_2CO also involved metallic rhodium rather than the usual Rh^+.

Finally, a study of acetone decomposition over 2.2% Rh/TiO_2 and 2.2% Rh/Al_2O_3 was also performed here. However, the results were not inspiring. The infrared bands for gas-phase acetone disappeared, and GC indicated complete decomposition, upon heating at 440 K, but no adsorbed CO species or any other products could be detected by infrared. It would thus appear that acetone decomposes to inactive carbon over supported rhodium.

Conclusions

From this study it may be concluded that: (1) methanol decomposes over supported Rh at 440 K to produce methane if TiO_2 is the support or dimethyl ether if Al_2O_3 is the support; (2) acetaldehyde decomposes over supported Rh at 440 K to produce methane and acetone; (3) acetone decomposes over supported Rh at 440 K to produce inactive carbon only; (4) potassium ions poison the formation of all products by presumably causing the production of large amounts of inactive carbon on the surface; and (5) the new Rh gem dicarbonyl state produced during the decomposition of acetaldehyde over 2.2% Rh/Al_2O_3 probably refers to metallic rhodium rather than the usual Rh^+ species.

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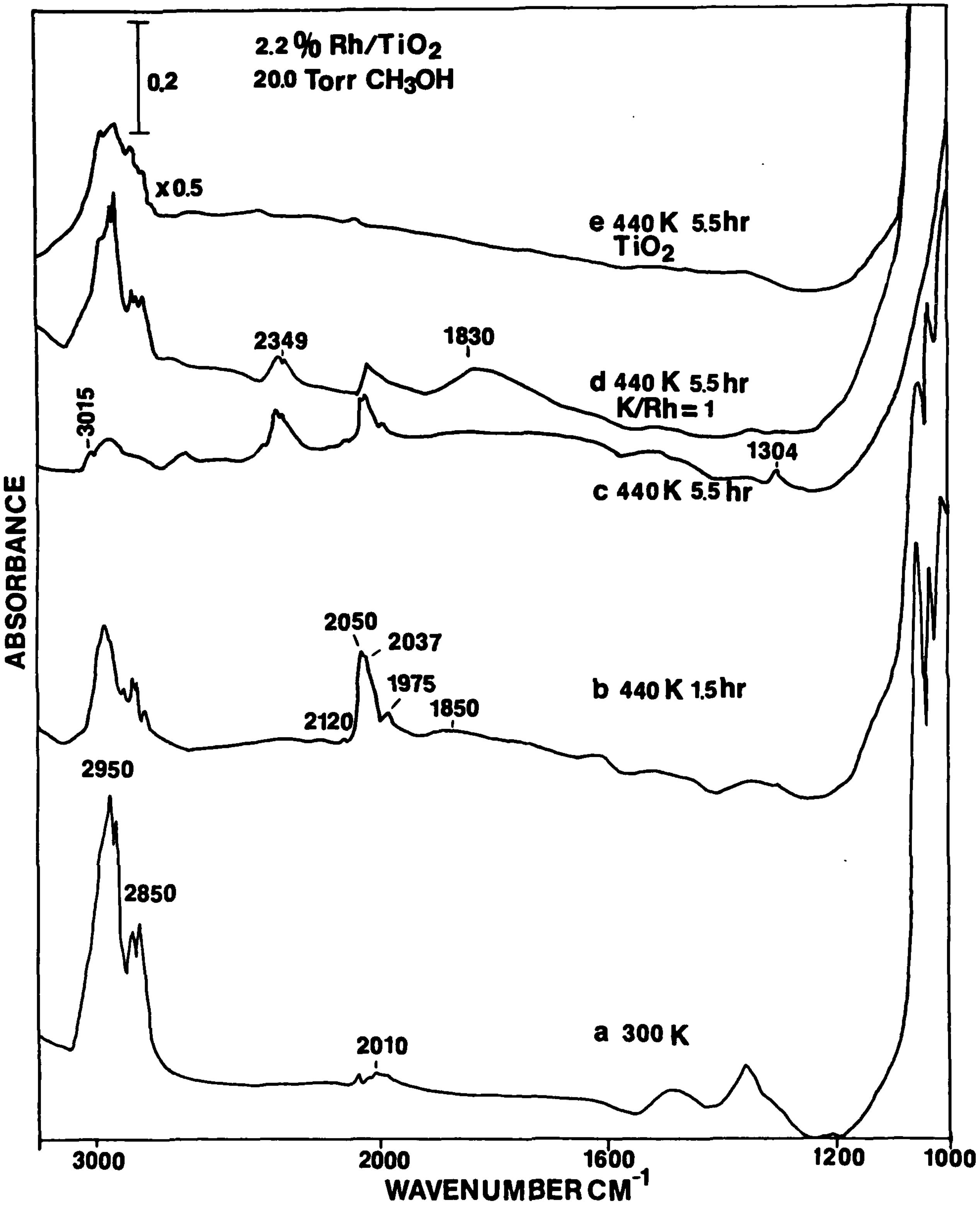
Figure Captions

Figure 1. Infrared spectra for the decomposition of 20.0 Torr methanol over: (a, b, c) a 2.2% Rh/TiO_2 film (4.3 mg cm^{-2}); (d) a 2.2% Rh/TiO_2 film containing K^+; (e) a TiO_2 film.

Figure 2. Infrared spectra for the decomposition of 20.0 Torr methanol over: (a, b, c) a 2.2% Rh/Al_2O_3 film (4.3 mg cm^{-2}); (d) a 2.2% Rh/Al_2O_3 film containing K^+; (e) an Al_2O_3 film.

Figure 3. Infrared spectra for the decomposition of 20.0 Torr acetaldehyde over: (a, b, c) a 2.2% Rh/TiO_2 film (4.3 mg cm^{-2}); (d) a 2.2% Rh/TiO_2 film containing K^+; (e) a TiO_2 film.

Figure 4. Infrared spectra for the decomposition of 20.0 Torr acetaldehyde over: (a, b, c) a 2.2% Rh/Al_2O_3 film (4.3 mg cm^{-2}); (d) a 2.2% Rh/Al_2O_3 film containing K^+; (e) an Al_2O_3 film.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TiO₂
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1830
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K/Rh = 1
3015
1304
c 440 K 5.5 hr
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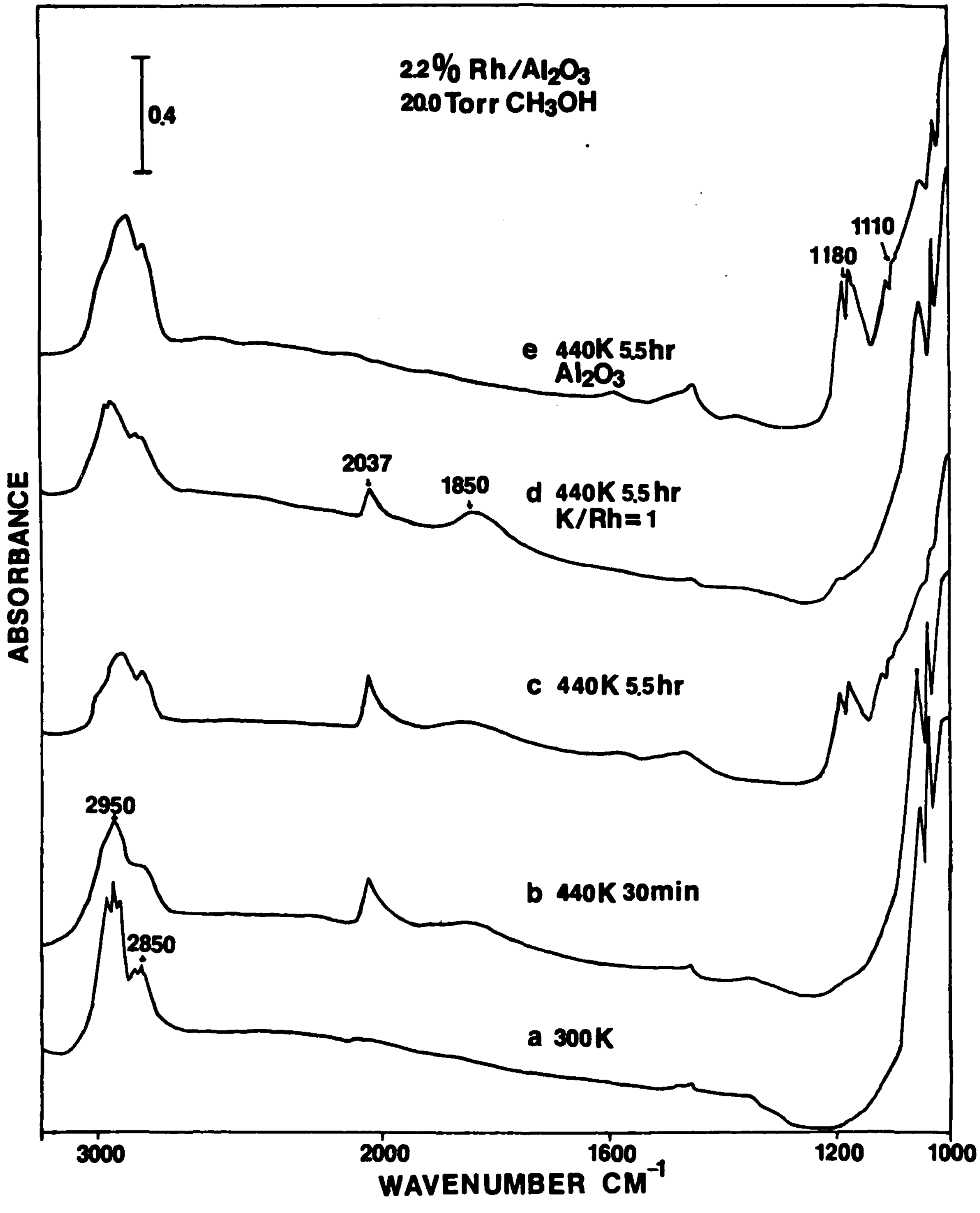

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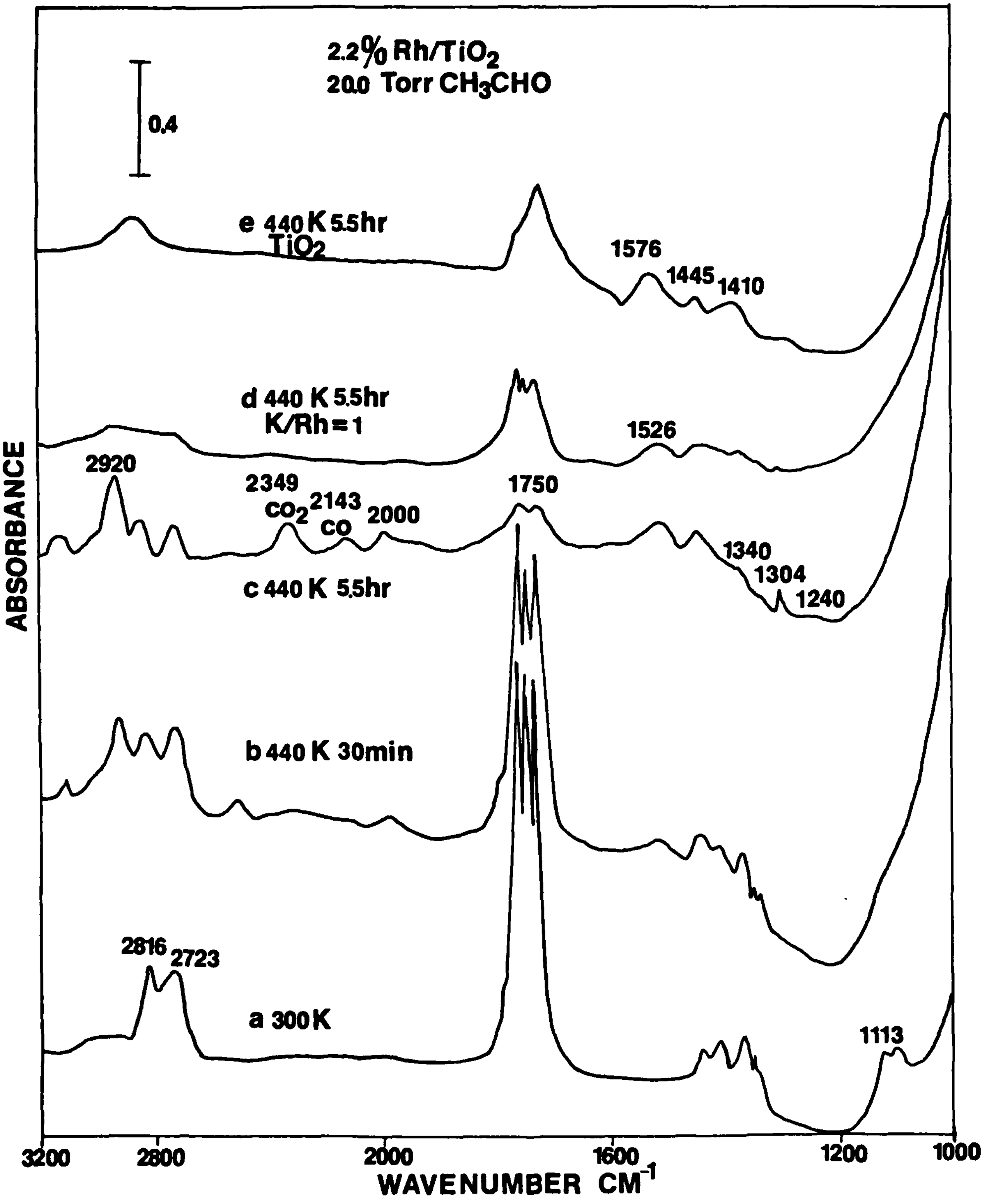

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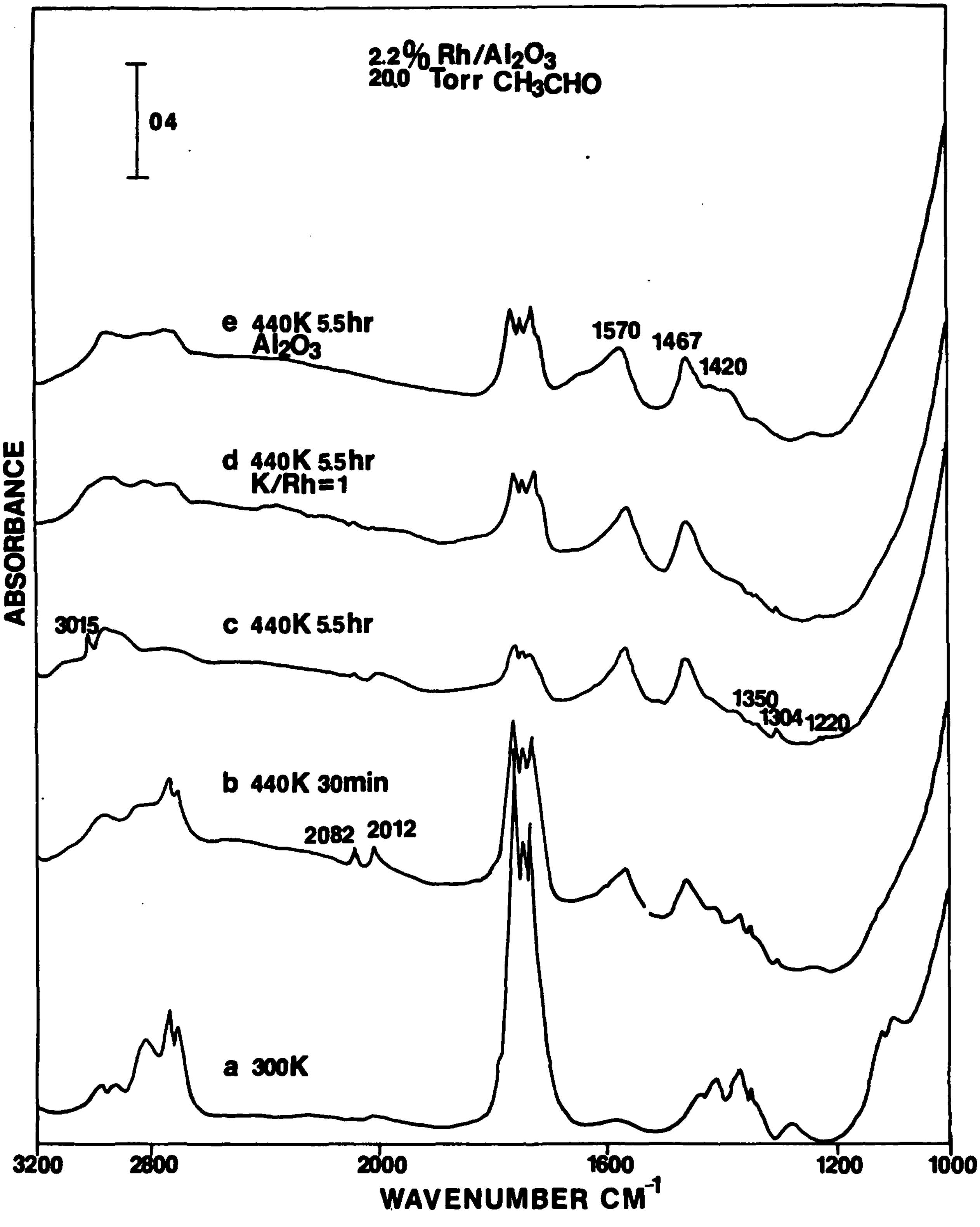

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