

to yield 6.87 g of pale-yellow, liquid pyrolysate. A very small amount of carbon was deposited in the pyrolysis tube. Titration at 0 °C of a small portion of the pyrolysate dissolved in 95% ethanol indicated that the pyrolysate contained 86% titratable acid (calculated as hendecenoic acid).

The acid was separated from the lactone by extraction, as described previously, by the use of a 0.5 N sodium bicarbonate solution and then 0.5 N hydrochloric acid. The solvent was removed from the final ether extracts by distillation under reduced pressure and the residue was dried in a vacuum desiccator to yield 3.98 g (58% based on the weight of the pyrolysate) of 10-hendecenoic acid, mp 17–19.6 °C,  $n_D^{25}$  1.4465 [reported<sup>36</sup> mp 25.5 °C,  $n_D^{24}$  1.44642]. A mixture melting point determination with an authentic sample of 10-hendecenoic acid,  $n_D^{25}$  1.4463, exhibited a melting point of 18.5–21.3 °C.

While a solution of 2.28 g (0.011 mol) of 10-hendecenoic acid in 10 mL of purified petroleum ether was stirred in an ice bath, 2.2 g (0.027 mol) of bromine was added dropwise. A crystalline precipitate was collected by filtration and recrystallized from petroleum ether to yield 2.24 g (56%) of 10,11-dibromohendecanoic acid, mp 38.3–39.6 °C (reported<sup>36</sup> mp 38.5 °C). A mixture melting point determination with an authentic sample of the dibromo compound showed no depression.

**Registry No.**—1a, 96-48-0; 1b, 108-29-2; 2, 502-44-3; 4, 2549-59-9; 3, 6008-27-1; 5, 1725-03-7; 5-hexenoic acid, 1577-22-6; *p*-bromophenacyl bromide, 99-73-0; *p*-bromophenacyl 5-hexenoate, 63657-88-5; 6-heptenoic acid, 1119-60-4.

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## Pyrolysis of Unsaturated Compounds. 1. Pyrolysis of Vinyl Ethers<sup>1,2</sup>

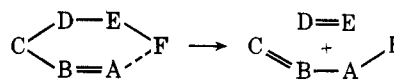
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Pyrolyses of vinyl ethers proceed at a temperature 40–50 °C lower than the corresponding esters. Thus, the pyrolysis of *n*-butyl vinyl ether at 480 °C gave 1-butene (71%), acetaldehyde (42%), and recovered vinyl ether (8%), while at 450 °C a 45% recovery of starting material was noted. Similarly, 2-ethylhexyl vinyl ether at 450 °C gave a 47% recovery of starting vinyl ether plus a 20% yield of 2-ethyl-1-hexene and a 29% yield of acetaldehyde. Pyrolysis of 2-ethoxy-1-butene at 460 °C gave nearly a quantitative conversion to methyl ethyl ketone and ethylene, while at 425 °C gave a 73% conversion to ketone and a 17% recovery of starting vinyl ether. Pyrolysis of  $\beta$ -butoxyethyl acetate at 520 °C gave a 65% yield of 1-butene and a 46% yield of acetaldehyde, but only a 12% yield of *n*-butyl vinyl ether and a 14% recovery of starting ester. Apparently, the ester pyrolyzes to give the intermediate vinyl ether, but at the high temperature (74 °C above the temperature required for 50% pyrolysis of the vinyl ether) the *n*-butyl vinyl ether undergoes extensive secondary pyrolysis. On the other hand, the corresponding  $\alpha$ -butoxyethyl acetate pyrolyzes at 380 °C to give a 43% yield of *n*-butyl vinyl ether with <4% secondary pyrolysis to 1-butene and acetaldehyde. Under temperatures at which other vinyl ethers pyrolyze methyl vinyl ether is relatively stable, but at 500 °C a 15% yield of propionaldehyde was noted. These results support the concept that unsaturated materials decompose by a concerted molecular mechanism in which the cyclic six-membered transition state resembles the products more than the starting materials.

Previous work in these laboratories has shown that pyrolysis of esters was a very excellent synthetic tool for the preparation of highly strained dienes,<sup>3,4</sup> isomers of aromatic compounds,<sup>5–7</sup> and highly reactive monomers.<sup>8–10</sup> Since we were able to confirm that the mechanism involved a quasi six-membered ring intermediate,<sup>11–13</sup> we became interested in finding out what other atoms would allow the six-membered cyclic mechanism to operate. In ester pyrolysis, A and C are



oxygen atoms, but pyrolysis of compounds with other atoms in these two positions are well known. For instance, the Chugaev reaction which involves the pyrolysis of a xanthate ester involves a sulfur atom at position A. Nace and O'Connor<sup>14</sup> proposed that the fact the xanthates pyrolyzed at a lower

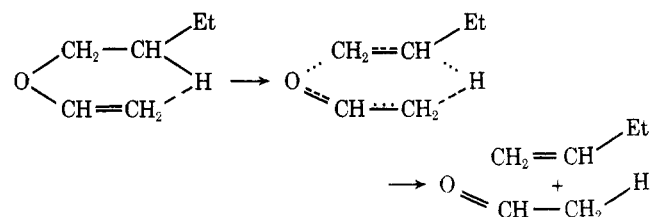
temperature than do esters could be attributed to the conversion of the thione system to a thiol system in the transition state.

Similarly, amides, which have a nitrogen atom in position C, require higher temperatures for pyrolysis than do esters.<sup>15</sup> This fact can be rationalized on the basis that the process of going from the lower energy carbon-oxygen double bond to the higher energy carbon-nitrogen double bond in the transition state produces a slower rate of reaction. For these reasons, it was of interest to study other elements in positions A and C in the ring. The pyrolysis of vinyl ethers in which A is carbon and C is oxygen was, therefore, investigated.

The pyrolyses of very few vinyl ethers have been reported in the literature. The pyrolyses of allyl vinyl ether and allyl  $\alpha$ -methylvinyl ether result in the Claisen or the oxy-Cope rearrangement to give the corresponding allyl acetaldehyde or allyl acetone, respectively, at about 250 °C.<sup>16,17</sup> The thermal decomposition of vinyl ethyl ether gave as the major products ethylene and acetaldehyde plus a considerable amount of ethane, methane, formaldehyde, and carbon monoxide as secondary products.<sup>18,19</sup> The thermal decomposition of isopropyl vinyl ether in the presence of toluene in the temperature range 447–521 °C yielded propylene and acetaldehyde as the main products.<sup>20</sup> The production of butadiene from the pyrolysis of ethyl vinyl ether<sup>21</sup> has also been reported. The pyrolysis of methyl vinyl ether above 500 °C gives as the main product propionaldehyde,<sup>22</sup> apparently formed by an ionic rearrangement. In contrast, phenyl vinyl ether has been reported to be quite thermally stable.<sup>23</sup>

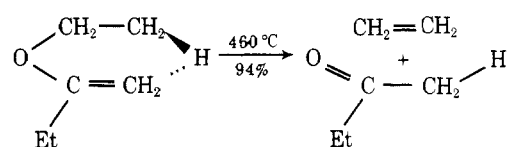
Since much of the previous work was carried out to determine the kinetics of pyrolysis, no yields of products were reported and no direct comparison was made between the ease of pyrolysis of vinyl ethers and esters. Therefore, we thought that it would be of interest to study the pyrolysis under the identical conditions used for ester pyrolysis so that a direct comparison between the two reactions could be made. In addition, an effort could be made to maximize the yield of the various products with the idea that the pyrolysis might be adapted for synthetic procedures in selected cases. Thus, when *n*-butyl vinyl ether was pyrolyzed at 480 °C over Pyrex helices, the products consisted of 1-butene in a 71% yield, acetaldehyde (42%), and recovered *n*-butyl vinyl ether (8%). In an effort to determine the temperature at which 50% of the vinyl ether was decomposed, the temperature of pyrolysis was lowered to 450 °C. Under these conditions, 45% of the *n*-butyl vinyl ether was recovered unchanged. Extrapolation from these two points led to the conclusion that at 446 °C 50% of the vinyl ether would have pyrolyzed. Under comparable conditions in the same apparatus, it was determined that a temperature of 490 °C was required to pyrolyze 50% of *n*-butyl acetate. Since the plots of the extent of thermal decomposition vs. temperature give "S"-shaped curves, the temperature required for 50% decomposition is a very convenient point to compare the relative ease of pyrolysis of the two compounds. It is apparent from these data that the vinyl ether pyrolyzes at a temperature approximately 40–50 °C lower than the corresponding ester. This fact would tend to rule out one type of mechanism that has been proposed for ester pyrolysis, which involves a nucleophilic attack of the hydrogen atom by the oxygen or sulfur atom with its unshared pair of electrons in the quasi-six-membered ring mechanism. Since the terminal CH<sub>2</sub> group of the vinyl ether neither has an unshared pair of electrons nor is very nucleophilic, the formation of the new bond between the carbon and hydrogen atoms must involve a nonionic concerted mechanism. One can rationalize the fact that the vinyl ether decomposed at a lower temperature by the fact that the starting material has a higher energy or less stable carbon-to-carbon double bond, while the carbonyl derivative formed as the product contains a much lower

energy or more stable carbon-oxygen double bond. This has been interpreted by DePuy and Bishop<sup>24</sup> as supporting the concept that the transition state in ester pyrolysis resembles the products more than it does the starting material. In other words, the transition state has more double-bond character between the carbon-oxygen atoms than between the two carbon atoms that originally contained the vinyl group.



In a very similar manner 2-ethylhexyl vinyl ether was pyrolyzed at 450 °C to give a 47% recovery of the starting material and a 20% yield of 2-ethyl-1-hexene plus a 29% yield of acetaldehyde. In addition, small amounts of 1-butene, propylene, and 2-methyl-1-butene were detected. The formation of the propylene and 2-methyl-1-butene undoubtedly occurs by a secondary pyrolysis of the 2-ethyl-1-hexene by a cyclic six-membered ring similar to the mechanism that was suggested for the decomposition of the vinyl ether. A separate study of the pyrolysis of olefins will be reported in a subsequent paper. When the temperature of the pyrolysis was lowered to 420 °C, the two major products, 2-ethyl-1-hexene and acetaldehyde, were still obtained, but 68% of the vinyl ether was recovered unchanged.

In order to determine what the effect of a substituent in the vinyl group would have, 2-ethoxy-1-butene was synthesized by the following set of reactions:  $\alpha$ -chloroethyl ethyl ether, which was prepared from acetaldehyde and ethanol in the presence of anhydrous hydrogen chloride, was brominated to give a 46% yield of  $\alpha,\beta$ -dibromoethyl ethyl ether. After the dibromo ether was added to ethylmagnesium bromide, the reaction mixture was decomposed by the addition of ice followed by acidification with hydrochloric acid to give a 69% yield of  $\beta$ -ethoxybutyl bromide. Heating the bromo ether with an excess of potassium hydroxide produced the 2-ethoxy-1-butene in a 66% yield. Pyrolysis of the 2-ethoxy-1-butene at 460 °C yielded nearly a quantitative conversion to methyl



ethyl ketone (94% isolated yield) and ethylene. When the temperature of pyrolysis was lowered to 420 °C, a 17% recovery of unchanged starting material was obtained plus a 73% yield of methyl ethyl ketone. In a similar experiment pyrolysis of isopropenyl ethyl ether at 425 °C gave a 31% conversion to acetone. It is quite obvious from these experiments that the introduction of a substituent in the 2 position lowers the temperature necessary for pyrolysis and produces a ketone which is stable to further decomposition under these conditions. This would suggest that enol ethers could be used as blocking groups for ketones and the ketone group could be regenerated by pyrolysis in quite satisfactory yields.

Since a vinyl ether pyrolyzes at a temperature lower than the corresponding ester, one would predict that the pyrolysis of  $\beta$ -alkoxyethyl acetate would not be an efficient synthetic procedure for the preparation of vinyl ethers. Instead, one would predict that the pyrolysis of the  $\alpha$ -alkoxyethyl acetates would proceed at a low enough temperature to be useful for the synthesis of vinyl ethers. In order to verify this prediction, *n*-butyl Cellusolve was converted to the corresponding 2-



ported<sup>35</sup> bp 118.6–119.6 °C,  $n_D^{20}$  1.4155), and 9.5 g (47% recovery) of unchanged 2-ethylhexyl vinyl ether, bp 176–180 °C.

In order to determine the identities and yields of the volatile pyrolysis products, 10.0 g (0.065 mol) of 2-ethylhexyl vinyl ether was pyrolyzed at 450 °C in a parallel run by the same method described previously. The pyrolysate (8.7 g), which was water-white, was cooled in a dry ice bath and used directly for a vapor-phase chromatographic analysis. By calibrating the chromatograph for both retention times and area responses with authentic compounds, it was determined that the yields of the various volatile products were acetaldehyde (29%), 1-butene (6%), propylene (7%), and 2-methyl-1-butene (3%). The 2-ethyl-1-hexene also was identified by an infrared spectrum in a carbon tetrachloride solution. The characteristic bands at 900 and 1650  $\text{cm}^{-1}$  indicated the presence of the terminal methylene group, and strong bands were also evident at 1210 and 1475  $\text{cm}^{-1}$ .

When 20.0 g (0.13 mol) of 2-ethylhexyl vinyl ether was pyrolyzed at 420 °C, 18.5 g of water-white pyrolysate was obtained. Fractionation of this material through an 8-in., helix-packed column yielded 2.0 g (14%) of 2-ethyl-1-hexene and 13.6 g (68% recovery) of unchanged vinyl ether.

**2-Butoxyethyl Acetate.** To a mixture of 153 g (1.5 mol) of acetic anhydride and 10 g of acetic acid was added dropwise over a period of 3 h 118 g (1.0 mol) of 2-butoxyethanol. After the acetic acid and the excess acetic anhydride had been removed by vacuum distillation, the residue was distilled under reduced pressure through an 18-in. Vigreux column to yield 125 g (78%) of 2-butoxyethyl acetate, bp 119–121 °C (95 mm),  $n_D^{21}$  1.4128 [reported<sup>32</sup> bp 190 °C (760 mm),  $n_D^{18}$  1.4144].

**1-Butoxyethyl Acetate.** The general method that has since been reported by Erickson and Woskow<sup>25</sup> was used for the preparation of the 1-butoxyethyl acetate. A mixture of 74 g (1 mol) of *n*-butyl alcohol and 44 g (1 mol) of acetaldehyde was saturated with dry hydrogen chloride, and the mixture was allowed to stand overnight. The excess hydrogen chloride and other volatiles were removed under reduced pressure, and the crude residue was treated directly with 82 g (1 mol) of anhydrous sodium acetate for 2 h at room temperature and then heated for 2 h on a steam bath with stirring. After the mixture had been cooled to room temperature, 100 mL of ethyl ether was added. The solid precipitate was removed by filtration and washed with ether.

After most of the ether had been removed from the filtrate by flash distillation, the residue was dried over anhydrous sodium carbonate and fractionated through an 8-in. Vigreux column to yield 112 g (70%) of 1-butoxyethyl acetate, bp 55–56 °C (20 mm),  $n_D^{20}$  1.4068 [reported<sup>33</sup> bp 54–54.5 °C (19 mm),  $n_D^{20}$  1.4062].

**2-Ethoxy-1-butene.** By modification of the method of Lloyd, Swallen, and Boord,<sup>30</sup> a mixture of 118.4 g (2.66 mol) of absolute ethanol and 117.0 g (2.66 mol) of acetaldehyde, cooled to 0 °C, was treated with 127.7 g (3.5 mol) of dry hydrogen chloride. The reaction mixture separated into two layers and the upper layer, which consisted of 203 g (74%) of crude  $\alpha$ -chloroethyl ethyl ether, was cooled in an ice bath at 0–5 °C, and 350 g of bromine was added immediately. After the mixture was allowed to stand overnight, it was distilled under reduced pressure to yield 200 g (46%) of  $\alpha,\beta$ -dibromo ethyl ether, bp 87–91 °C (20 mm) [reported<sup>30</sup> bp 90–91 °C (20 mm)]. The dibromo ether was added to an ether solution of ethylmagnesium bromide [prepared from 164 g (1.5 mol) of ethyl bromide and 36.6 g (1.5 mol) of magnesium turnings in 1000 mL of anhydrous ether]. When the reaction mixture was worked up in the usual manner, distillation

through an 18-in. Vigreux column gave 108 g (69%) of 1-bromo-2-ethoxybutane, bp 155–156 °C,  $n_D^{25}$  1.4419 (reported<sup>30</sup> bp 154–156 °C,  $n_D^{25}$  1.4427).

Treatment of this bromo ether by heating with a large excess of finely powdered, technical grade potassium hydroxide over a period of 8 h gave 39.4 g (66%) of crude 2-ethoxy-1-butene. Fractionation through a 6-in., helix-packed column gave pure 2-ethoxy-1-butene, bp 85–86 °C,  $n_D^{25}$  1.4002 (reported<sup>30</sup> bp 85–87 °C,  $n_D^{24.5}$  1.4001). Vapor-phase chromatography over two different columns gave in each case a single sharp peak for this sample of 2-ethoxy-1-butene, and an infrared spectrum in a carbon tetrachloride solution gave strong peaks at 880, 950, 978, 1050, 1098, 1120, 1242, 1286, and 1658  $\text{cm}^{-1}$ , indicative of the presence of a terminal methylene group and the presence of an ether group.

**Registry No.**—Acetic anhydride, 108-24-7; 2-butoxyethanol, 111-76-2.

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