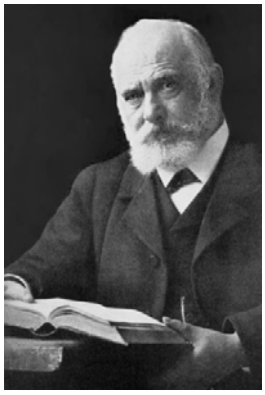
Fig. 37-1



William R. Fittig (1835 -1910)
http://en.wikipedia.org/wiki/Wilhelm_Rudolph_Fittig

Experiment 37

ACID CATALYSIS - REACTION OF PINACOL

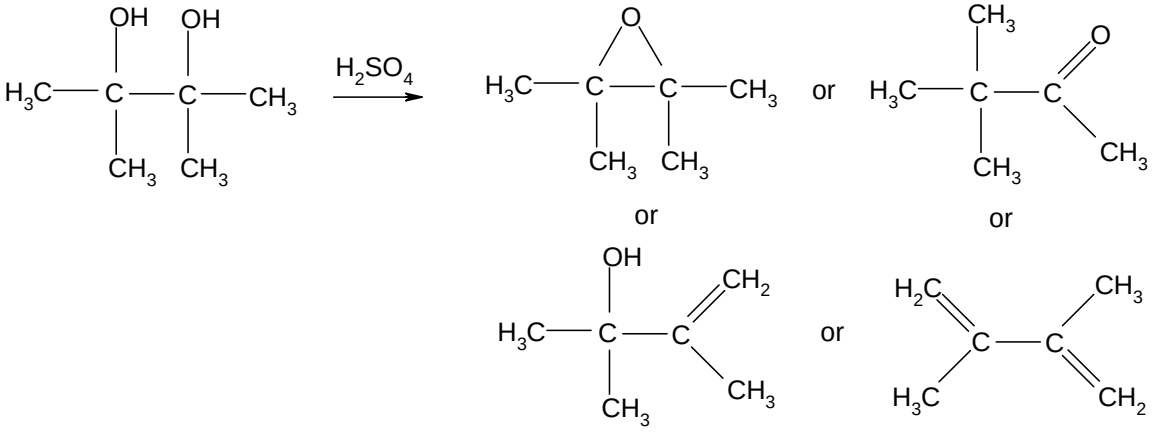
Text Topics

Acid catalysis, reaction mechanisms, carbocation rearrangements.

Discussion

In the biological world, catalysis is often achieved with enzymes that specifically control the rate and direction of a reaction. In the organic laboratory, chemists often must be satisfied with more general catalysts such as hydrogen ion. While increased acid concentration often does increase the rate of a reaction, rates of competing reactions are commonly increased as well. Chemists who can design and synthesize catalysts that are as efficient and specific as enzymes will be recognized as leaders in their field. For today's experiment, the catalyst will be sulfuric acid and it will be your goal to determine the structure of the product, if any, of the reaction.

The starting material for the reaction will be 2,3-dimethyl-2,3-butanediol (common name pinacol). In the presence of acid, in addition to the absence of a reaction, at least four different products seem plausible.



As part of the ***Prelaboratory Exercise***, you will be asked to provide reasonable mechanisms from pinacol to each of the products.

Procedure

Fig. 37-2

Add 0.75 g of 2,3-dimethyl-2,3-butanediol and a magnetic spin vane to a 5 mL conical vial. Assemble an apparatus with the conical vial equipped with a Hickman still and condenser in a sand bath on a magnetic stirrer unit. Add 2 mL of 3 M sulfuric acid to the vial and heat (about 100ºC) until about 1 mL has distilled and been collected. Use a disposable pipet to transfer the liquid collected to a clean conical flask. The liquid should consist of a bottom aqueous layer and an organic layer. Draw off the aqueous layer with another disposable pipet and properly dispose of it. Add 1 mL of water to the organic layer and thoroughly mix the contents of the vial. Draw off the aqueous layer and discard again. Repeat the above washing with 1 mL of water one more time. Use a pipet to transfer the organic layer to a clean vial containing a small amount of sodium sulfate. Remove the organic layer using a pipet and save it in a stoppered container. Determine the ir, H-nmr and mass spectra of the product. Determine if the resulting organic liquid is starting material or one of the suggested possible products.

References

Wojciechowsi, Brenda J.; Deal, S. T. *J. Chem Ed.*, **1996**, *73*, 85.
Arrington,C. A.; Hill, J. B.; Radfar, R.; Whisnant, D. M.; Bass, C. G. *J. Chem Ed.*, **2008**, *85*, 290.
Schoffstall, A. M.; Gaddis, B. A.; Druelinger, M. L. *Microscale and Miniscale Organic Chemistry Laboratory Experiments*, McGraw-Hill, **2000**, 378-83.

Prelaboratory Preparation - *Experiment 37*

First, be sure to list all the goals of the experiment. Write detailed mechanisms for pathways from the starting material to each of the possible products. State which product is most likely to be formed and give reasons for your selection. Prepare a table for insertion of useful and observed data such as molecular mass, mass, moles, melting points and percent yields and recoveries. Present a method for distinguishing among the 4 possible products.

Observations

Report all relevant observations including physical properties and spectra.

Conclusions

This section should include the following:

1. Were the goals of the experiment achieved? Explain your answer.
2. What was the identity of your product and did it agree with your prediction? Explain your answer.
3. How could the percent yield and recoveries have been improved?
4. Could conditions have been changed to favor a different product? If so, explain how.