

Dehydration of 2- methylcyclohexanol formal report essay sample essay



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A desiccation reaction of an intoxicant consequences in an olefine.

This type of reaction requires an intoxicant. an acerb accelerator and heat.

Generally strong concentrated acids. like sulphuric acid and phosphorous acid. are used as the acerb accelerator.

The acerb accelerator protonates the intoxicant. to do a much better go forthing group. Weakest bases make the best go forthing groups. so one time the intoxicant is protonated the go forthing group foliages and produces in a carbocation and H₂O. In order to organize the dual bond. one of the beta Hs.

Hs on a C adjacent to the carbocation. must be removed or eliminated.

Therefore. another name for a desiccation reaction is beta riddance. Water acts as a nucleophile and attacks the carbocation.

taking a beta H ; the negatrons from the C-H bond move to do a C-C pi-bond.

The general mechanism is displayed by equation (1. 0) Equation (1. 0) shows a reaction with a primary intoxicant. Normally primary intoxicants form primary carbocation. However.

primary carbocations are excessively unstable to organize as intermediates. so they can undergo a rearrangement or E2 mechanism. A rearrangement occurs when an alkyl group or a H on the adjacent C of the carbocation displacement to delocalize the positive charge to ensue in a more stable carbocation. A rearrangement about ever occurs whenever a carbocation can organize a more stable molecule.

An E2 mechanism means that the reaction is a concerted process and the rate-determining step is bimolecular. Two species are involved in one step of the mechanism. If the substrate in equation (1.0) were to undergo an E2 mechanism so the beta H would be eliminated (the nucleophile would attack the electrophilic carbon). C=C pi bond would be formed.

and the leaving group would go forth all in the rate-determining step. Additionally, equation (1.0) merely shows one product, which is not true for all substrates. Certain substrates can bring forth more than one product when they undergo desiccation.

such as 2-methylcyclohexanol, construction shown on right. The number of products ensuing can be determined by the number of groups of beta Cs on the molecule. For illustration, after the substrate is protonated and leaves, the carbocation will hold two groups of beta Hs.

so 2-methylcyclohexanol will hold at least two products as a consequence of beta elimination. The products of 2-methylcyclohexanol show regioselectivity. Significant products are not produced in equal amounts. Zaitsev's rule can predict which product will be the major, most favored product. Zaitsev's rule states that the more substituted alkene will be the favored product; the double bond incorporating the most non-hydrogen substituents will be the major product.

In this lab, 2-methylcyclohexanol will undergo a desiccation with the acid-catalyst, phosphorous acid. The merchandises will be contained in the same flask because they will be produced at the same clip.

Gas chromatography will be used to divide the mixture of merchandises into its constituents. Because each compound has a different affinity to the GC column two separate extremums will look in the chromatograph. The compound that produces the extremum with the greatest comparative country will be the major merchandise. The gas chromatograph is connected to the mass spectrometer.

so the individualities of the two compounds can besides be determined. A mass spectrometer measures the mass of ions of the sample and lucifers it with its built in database to find the compound (s) nowadays. There is no demand for running GC's of known compounds. Additionally IR spectra will be run on the get downing stuff and concluding merchandise to find the functional groups present.

Finally potassium permanganate will be used to find unsaturation. Potassium permanganate (KMnO_4) . deep purple in colour. acts an oxidising agent in a reaction with olefines or acetylenes to bring forth a glycol and a decreased signifier of manganese (MnO_2) .

which is brown. Equation (2. 0) shows a generic illustration of this reaction. Methane seriess and aromatic compounds do non respond with KMnO_4 ; hence.

no colour alteration occurs. Lack of colour alteration indicates that no olefine was present in the reaction. The intent of this lab is to find the merchandises of the desiccation of 2-methylcyclohexanol via distillment and set up if Zaitsev's regulation holds true in this reaction by analysing consequences from gas chromatography and the mass spectrometer. performed prior to the experiment. IR spectrometry will be used to analyse the merchandises obtained from the experiment.

The merchandises will besides be tested with KMnO_4 to corroborate that 2-methylcyclohexanol had undergone a desiccation to organize unsaturated compounds. Given the construction of 2-methylcyclohexane it will likely organize two merchandises. methylcyclohexene and 3-methylcyclohexene. as shown by equation (3. 0) .

If Zaitsev's regulation holdstrue. methylcyclohexene will be the major merchandise as it is more substituted than 3-methylcyclohexene. The trial for unsaturation will be positive. colour alteration visible.

ExperimentalThe powermite was set to 6 to heat the sand bath. placed on an drawn-out doodly-squat. To a 25 milliliter unit of ammunition bottom flask 5. 0 milliliter (4. 21 g or 0. 0369 moles) of 2-methylcyclohexanol (from Acros Organics with 99 % pureness) was added to the flask.

To the unit of ammunition underside flask. 2. 0 milliliter of 85 % phosphorous acid (from Fisher Scientific) and two boiling rocks were added. The flask was swirled to blend. The setup for distillment. shown in Figure 1 was set up.

A 10 milliliter having flask was used. and the 25 milliliter unit of ammunition bottom flask with 2-methylcyclohexanol. phosphorous acid and boiling rocks was used for the boiling flask. Both having and boiling flasks were attached. Water tubing was connected and the boiling flask was placed in a sand bath. The thermometer read 27 °C at the beginning of the distillment.

Because the temperature rose really rapidly three proceedingss into the distillment. the powermite was turned down to 3. The first bead occurred at 90 °C. Six proceedingss subsequently.

the temperature began to drop so the powermite was increased to 6. However. the temperature did non lift significantly. At about 79. 5 °C. 14 proceedingss after the distillment began.

the bulk of the contents in the boiling flask had distilled so the distillment was stopped. Contentss of the having flask were cloudy. The boiling flask. whose contents were bright xanthous.

was cooled earlier discarded. The consequence of the distillment weighed 3. 84 g. Contentss from the having flask were transferred to a extractor tubing. to which 2 milliliter of 10 % Na carbonate solution (lab sample) was added. Upon transportation to the extractor tubing.

separate beds were seeable. The tubing was swirled easy to blend. The extractor tubing was capped and inverted one time. The cap was so removed to throw out any gas force per unit area. Again the cap was replaced and the tubing inverted several more times. The tubing was placed in a beaker after the cap was removed to let beds to divide.

The top bed was cloudy but the bottom bed was clear. The organic bed was pipetted to a smaller beaker, to which a little sum of anhydrous Na sulphate (from Acros Organics) was added to absorb extra H₂O. The beaker was so swirled and allowed to sit for 10 min. A clean dry distillment setup was set up with a 10 milliliter boiling flask and a 5 milliliter having flask.

The power touch was turned to 8 to heat the sand bath. When the organic solution in the beaker was clear, the solution was decanted into the boiling flask and two boiling rocks were added. The flask was so attached to the distillment set up. Water tubing was connected and heat was applied to the boiling flask. The thermometer read 30 °C at the beginning of the distillment.

The contents of the boiling flask distilled in less than a minute, the thermometer read 79 °C. Because the lone distillment merchandise to be collected was between 102 -111°C, the distillment was repeated. The sand bath was allowed to chill.

and a little sum of the hot sand was replaced with cool sand. The thermometer read 31 °C at the beginning of the distillment. The contents of boiling flask were the contents of the having flask from the old distillment, massed at 1.1 g.

The powermite was set to 4.5. The first bead occurred at 73 °C. The distillment took about five proceedings, completing at 90 °C. The having concluding merchandise was massed at 0.

88 g or 0.00917 moles. An IR was run on this sample every bit good as on the dry sample (done antecedently by another group) . A little sample of

the concluding merchandise was combined with about three beads of 0.8 M KMnO_4 in a disposable trial tubing to prove for unsaturation. The tubing was agitated.

and little brown and ruddy musca volitans appeared. The trial tubing was allowed to sit as a negative control was run. In another trial tubing, cyclohexane was combined with three beads of KMnO_4 .

The tubing was agitated but no alteration in colour observed. This tubing was allowed to sit every bit good. After five proceedings, the tubing incorporating the sample from the distillment had turned brown and separated into two beds with the sample on top. The negative control was still violet.

besides separated with the sample as the top bed. Finally the GC-MS information was analyzed to find the cogency of Zaitsev's regulation in this reaction. Table 1.0 Summary of GC Retention Time Relative Area (100 %) 2.7624.

02.803.502.8972.5 Figure 2.0 shows the GC.

done by the teacher. Merely the important extremums were identified in Table 1.0. The country was calculated presuming, entire country equaled the amount of the countries of the three extremums (keeping clip = 2.76.

2.80. and 2.89) . Table 2.0 IR spectral information of 2-methylcyclohexanol
Peak Frequency (cm^{-1}) Peak Assignment
3356-OH stretch
2927-CH stretch
1449-CH₂
1372-CH₃
Table 3.

0 IR spectral information of merchandises of desiccation of 2-methylcyclohexanol
Peak Frequency (cm^{-1}) Peak

Assignment3356impurity3002C= C2925-CH32857-CH32836-CH21440CH

bending
Discussion
After the first distillment. two beds formed in the receiving flask. The top bed was the organic bed. and contained the merchandises of the desiccation of 2-methylcyclohexanol.

The bottom bed was the acidic bed and contained the by-product of the reaction. H_3O^+ . In order to neutralize the bed acid Na carbonate was added. Since the expected organic merchandises were olefines.

it was of import to neutralize the acid to forestall a rearward reaction. The 2nd distillment served as a purification measure in instance any H_2O was present after the separation of the beds. Presence of H_2O can do drosss and excess extremums in IRs. The most important extremum on the IR of 2-methylcyclohexanol. Figure 4.

0. is the wide intoxicant stretch from 3200-3500 cm^{-1} . Extremums in the 3000-2800 cm^{-1} country normally indicate CH stretch nowadays in methane series ; there is a important wide extremum at 2927 cm^{-1} . A extremum at 1372 indicates the presence of a methyl group. The information from this IR is consistent with the construction of 2-methylcyclohexanol.

The IR of the concluding merchandises of the desiccation of 2-methylcyclohexanol indicates the presence of a dual bond ; there is a extremum at 3002 cm^{-1} . Peaks Ate 2925 and 2857 cm^{-1} indicate the presence of a methyl group. Extremums in the 1500-1200 cm^{-1} normally indicate CH flexing due to $-\text{CH}_2-$; there are several little extremums in that
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scope. This information is consistent with the construction of the expected merchandises from the desiccation of 2-methylcyclohexanol. In add-on to these extremums.

Figure 5. 0 shows a minor stretch at 3356 cm^{-1} . Generally stretches in this country indicate the presence of -OH groups. The intent of the desiccation was to take the intoxicant group and replace it with a dual bond. It is likely that the extremum at 3356 cm^{-1} is due to the presence of H_2O .

However, H_2O was non expected to be in the concluding merchandise because the 2nd distillment. This mistake is likely because the merchandises acquired from the 2nd distillment were from a temperature scope below 90 $^{\circ}\text{C}$. The first clip the purification distillment was run, the temperature didn't even make 80 $^{\circ}\text{C}$.

Because the concluding temperature changed by 10 $^{\circ}\text{C}$ after running a 2nd distillment, it is possible that running the distillment two more times would hold given the coveted consequences (no H_2O). The consequences of the trial for unsaturation confirmed the desiccation of an methane series to an methane series. In the reaction with KMnO_4 .

the olefine (the merchandise) was oxidized to bring forth an intoxicant and MnO_2 . KMnO_4 is a deep purple colour and MnO_2 is a brown colour, doing the presence of the olefine more noticeable. The intent of the negative control was to corroborate there was nil incorrect with the get downing stuff.

The negative control determined that there were no olefines already present in the 2-methylcyclohexanol that could do the reaction with KMnO_4 to

continue. The consequences of the negative control indicated that there were no alkenes nowadays in the get downing stuff as there was no colour alteration. The concluding per centum output shows that merely 24 % of the concluding merchandises were really recovered. The major beginning of mistake could hold occurred during the separation of the organic bed and aqueous bed. It was hard to state when all of the organic bed had been removed from the extractor tubing to beaker so.

less of the organic bed may hold been removed so perceived. Additionally, because there was still material left in the boiling flask, some of the merchandise may hold non been distilled. Table 1.

0 shows a sum-up of the three chief extremums on the GC. all others are irrelevant. The extremum with keeping clip of 2. 76 had a comparative country of 24. 0 % .

The extremum with a keeping clip of 2. 80 had a comparative country of 3. 50 % . The concluding extremum, with a keeping clip of 2.

89 had a comparative country of 72. 5 % . The two extremums with the greatest country represent the two major merchandises, while a more undistinguished extremum likely represents the minor merchandise.

The mass spectrometer was consistent with this information as the machine identified two compounds with keeping times of 2. 76 and 2. 89. The compound with a keeping clip 2. 76 had the construction of 3-methylcyclohexene. The compound with a keeping clip of 2.

89 had the construction of methylcyclohexene. Sum uping informations from the GC and the mass spectrometer. two merchandises resulted from the desiccation of 2-methylcyclohexanol: methylcyclohexene and 3-methylcyclohexene. Methylcyclohexene was present in greatest sum. therefore it must be the major merchandise. Methylcyclohexene is more substituted than 3-methylcyclohexene.

therefore Zaitsev's regulation holds true for this reaction. Dehydration of 2-methylcyclohexanol followed Mechanism 1. 0 to give methylcyclohexene and 3-methylcyclohexene. In the first measure.

the -OH was protonated to do a better go forthing group. In measure two the go forthing group left. ensuing in a carbocation. Beta H riddance took topographic point in measure three.

There were two sets of beta H. If the nucleophile. H₂O. attacked the H on the C attached to the methyl group. methylcyclohexene was produced (A) . If H₂O attacked the other beta H.

shown by B. 3-methylcyclohexene was produced. The GC besides showed another minor merchandise. This is the merchandise produced if a 1. 2 hydride displacement occurs.

Mechanism 2. 0 shows that the hydride displacement would take topographic point at measure when the formation of the carbocation takes topographic point. The hydride displacement would take topographic point to organize a third carbocation. a more stable carbocation. However.

it is of import to maintain in head that any carbocation is unstable. In measure 3 of Mechanism 2. 0. the nucleophile could assail either of the two sets of beta H. Notice. the beta Hs are different from the 1s seen in Mechanism 1.

0. Elimination of a beta H. seen in A1. consequences in methylcyclohexene. However riddance of a H attached to the methyl group consequences in methylene cyclohexane. Because methylenecyclohexene is the least substituted compound.

it is the minor merchandise. Additionally. dual bonds inside the ring are more stable than dual bonds outside of the ring. Therefore though a third carbocation is more stable than a secondary carbocation.

the merchandise is the least substituted and least stable. Zaitsev's regulation favours Mechanism 1. 0 over Mechanism 2. 0. However some of the molecules undergo a hydride displacement. so a little sum of methylene cyclohexane is produced.

In the GC. the extremum with a keeping clip of 2. 80 is 2-methyl-methylene cyclohexane and makes up 3. 50 % of the merchandises. The anticipation holds true for the desiccation of 2-methylcyclohexanol as methylcyclohexene was the major merchandise. ConclusionThe two major merchandises of this desiccation were methylcyclohexene and 3-methylcyclohexene.

Methylcyclohexene constituted most of the concluding merchandise. about 73 % . and 3-methylcyclohexne made up 24 % of the concluding merchandise (obtained via analysis of a antecedently done GC and mass

spectrometer). The two major merchandises were formed largely by following Mechanism 1. 0, although one of the merchandises of Mechanism 2.

0 was besides methylcyclohexane. The other merchandise of Mechanism 2, 0 formed a minor merchandise, methylene cyclohexane, doing up approximately 4 % of the concluding merchandise. As predicted,

Zaitsev's rule held true for this reaction since the most substituted merchandise, methylcyclohexene, was the major merchandise. The trial for unsaturation confirmed the dependability of the get downing stuff.

The IR's of the get downing stuff and concluding merchandises were consistent with constructions of the compounds. However, there was taint by H₂O in the IR of the concluding merchandise. The per centum output was besides really low.

which can be accounted for loss in distillment every bit good as during separation of organic bed from the aqueous bed. A really of import lesson was learned from this experiment: desiccation of 2-methylcyclohexanol is a particular instance in carbocation rearrangement. Although the formation of a third carbocation from a secondary carbocation, via hydride displacement, was possible.

most of the molecules did non undergo a displacement. The displacement resulted in two merchandises where one (methylcyclohexene) was the most substituted of all three merchandises ensuing from a desiccation of 2-methylcyclohexanol, and another (methylene cyclohexane) which was the

least stable of the three merchandises. This reaction followed Zaitsev's regulation and so the bulk of the molecule did non undergo a hydride displacement.

but formed two more stable merchandises than methylene cyclohexane. Mentions • " Introduction to IR Spectrum Interpretation. " Elizabethtown College. Department of Chemistry and Biochemistry. Chem 113.

2009. • " Dehydration of 2-methylcyclohexanol. " Elizabethtown College. Department of Chemistry and Biochemistry. Chem 113. 2009.