AN ABSTRACT OF THE THESIS OF

MARILYN XIAOHONG ZHOU for the Master of Science Degree in Chemistry presented on April, 27 1992.

Title: The Use of Selenium(IV) Oxide to Oxidize Aromatic Methyl Groups

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Abstract:

In this series of experiments, the effect of selenium(IV) dioxide (SeO₂) on aromatic methyl group was investigated. The products obtained when 2-picoline, 4-picoline and 8-methyl quinoline were oxidized by selenium dioxide were 2-pyridine carboxylic acid, 4-pyridine carboxylic acid and 8-quinoline aldehyde, respectively. The yield corresponding to each product above is 50%, 77% and 49%, respectively. Other compounds such as 2,6-lutidine, 2-methyl quinoline and 4methyl quinoline were also studied. The products were 2,6pyridine dicarboxylic acid, 2-quinoline aldehyde and 4quinoline aldehyde. Additionally, the compounds which did not react with SeO₂ are toluene, 2-nitrotoluene, 4-nitrotoluene, 2,4-dinitrotoluene, 2-chlorotoluene, and 4-chlorotoluene. The mechanism for reactions involving selenium(IV) oxide is discussed in detail. The products were analyzed qualitively by using FTIR, NMR, and other physical and chemical methods.

Approved for the Major Division

Alfred Erein Committee Member

Jorge Ballesto Committee Member

Sie <u>L. Jump</u> Committee Chaifman <u>Jayk N. Vourill</u> Approved for the Graduate Council

The Use of Selenium(IV) Oxide to Oxidize Aromatic Methyl Groups

A Thesis

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Note: Figure 3-4 and 7-21 are FTIR spectra. Figure 5 and 6 are NMR spectra.

Introduction

Selenium dioxide is often used as an oxidant. Methyl or methylene groups α to a carbonyl can be oxidized with selenium dioxide to give, respectively, α -keto aldehydes and α diketones.

$$R - C - CH_2 - R' \xrightarrow{SeO_2} R - C - C - R'$$
(1)

Internal alkynes have been oxidized to α -diketones by several oxidizing agents, including SeO₂ with a small amount of H₂SO₄.

Treatment of double-bond compounds with selenium dioxide introduces an OH group into the allylic position.



Six-membered alicyclic rings can be aromatized in a number of ways. Aromatization is accomplished most easily if there are already one or two double bonds in the ring or if the ring is fused to an aromatic ring.

$$2 \qquad + \qquad Se0_2 \qquad --- \qquad 2 \qquad + \qquad Se + 2 H_2 0 \qquad (4)$$

Since the aromatic pyridine ring is very stable, generally, it is very difficult to oxidize. But in most cases, the alkyl group on the aromatic ring such as pyridine and quinoline can be oxidized fairly easily by other oxidant reagent. Some alkyl-substituted pyridine can be oxidized to corresponding carboxylic acid by HNO_3 , $KMnO_4$, or O_3 (1). In this series of experiments, our goals were to optimize the conditions for the reactions; simplify the isolation of products; improve the yields; and know the generality of the reactions.

The SeO₂ is a non-metallic compound, in the form of a white crystalline solid. The Lewis structure of SeO₂ is



in which there is comparatively high electron density around the higher electronegativity element, oxygen. The bond length is between that of a typical single bond and a double bond. Thus, the electron density around the selenium is lower than that in an oxygen. SeO_2 could be an electron accepter, or a Lewis acid, and the selenium is the center of this Lewis acid. The reactions involving SeO_2 acting as oxidant would undergo Lewis acid-base reactions, which result in the Se-O bond being broken followed by the element selenium produced.

Reagents:

2-picoline, 4-picoline, 8-methyl quinoline, 2,6-lutidine, 2-methyl quinoline, 4-methyl quinoline, ethyl ether, methanol, silver nitrate, sodium hydroxide, ammonia, acetone (NMR grade), sodium bicarbonate (5 g/100 ml water), hydrochloric acid, copper(II) sulfate, pHydrion MIKRO 0 to 11 (pH paper).

Apparatus:

Heating mantle, hot plate, temperature controller, 25 ml round bottom flask (size 19/22), condenser (size 19/22), filter flask, vacuum pump, etc.

Instrument:

FTIR, Bomen Michelson Series NMR, Hitachi Perkin Elmer (High resolution 24-B)

Procedure:

A. 8-methyl quinoline

0.5 g SeO₂ was mixed with 2.0 ml 8-methyl quinoline in a 25 ml round bottom flask, which was set in a heating mantle with temperature controller (See Figure 1 in the appendix). The mixture was heated in a refluxed manner for 5 hours at a temperature between 100 °C to 150 °C. During the reaction, the color of the liquid changed little by little, from colorless to yellowish-brown and then to dark brown. Finally, the original white "needle" solid, SeO_2 , changed to a black fine solid, selenium.

Next, the mixture was extracted with ethyl ether and the liquid was run through an alumina column. The ethyl ether eluant was set in the hood to evaporate the ether. A white crystalline solid was obtained. The solid left in the flask after ethyl ether extraction was a mixture of black solid and white crystals. The weight of the product was obtained by adding the weight value of the extracted portion together with the value of the solid mixture portion subtracted from the calculated weight of selenium.

B. 2-picoline

0.5 g SeO₂ added to 2.0 ml 2-picoline in a 25 ml round bottom flask (See Figure 1 in the appendix) was set in a heating mantle with a temperature controller. The mixture was heated for about 5 to 6 hours at a temperature below 120 °C. Finally, the color of the liquid changed from colorless to dark brown and a solid was formed.

Then the dark brown liquid was poured out of the flask. The solid mixture, which looked almost black, was taken out of the flask, weighed, and sublimated by using the equipment setup as in Figure 2 of the appendix. An off-white or slightly yellow platelet solid was collected from the test tube. The weight of the product was obtained by subtracting the calculated weight of selenium from the weight of the solid

mixture.

C. 4-picoline

0.5 g SeO₂ added to 2.0 ml 4-picoline in a 25 ml round bottom flask (the same as in Figure 1) was set in a heating mantle with a temperature controller. The mixture was heated for about 5 to 6 hours at a temperature below 120 °C. The color of the liquid changed from colorless yellow to dark red. The solid changed from white crystals, SeO₂, to a black fine solid, Se, mixed with an orange-red color solid.

The liquid was then poured out and the solid was transferred to a filter flask to sublimate (Figure 2 in the appendix). An orange-colored solid was collected from the test tube. The weight of the product was obtained in the same manner as the one of 2-picoline.

D. 2,6-lutidine

0.5 g SeO₂ was added with 1.5 ml colorless 2,6-lutidine in a 25 ml round bottom flask (the same as in Figure 1) which was set in a heating mantle with temperature controller. The mixture was heated for about 10 hours at a temperature between 60-80 °C. When the reaction was finished, a yellow liquid and a black fine solid were obtained. The liquid was transferred to an evaporating dish. Then a white "needle" solid was crystallized from the liquid.

Results

A. 8-methyl quinoline

The weight of the product from the oxidation of 8-methyl quinoline was 0.341 g. The percent yield was 49%. Some pure solid was white crystal. Although contaminated by some impurities, most of the solid was slightly yellow. The melting point of the solid was 77-82 °C. Tollens test (the procedure following reference 3) was positive. When sodium bicarbonate (NaHCO₃, 5 g per 100 ml water) was added drop-wise to the solid product, nothing happened. The solid didn't dissolve in NaHCO₃ aqueous solution. Therefore, the product couldn't be carboxylic acid, but might be an aldehyde.

When analyzed with FTIR in nujol oil by using nujol oil as the reference, the sample showed the characteristic transmittance peaks at 2964.7-2864.4 cm⁻¹, which was the C-H stretch resonance peak in the aldehyde group (CHO), and 1701.7 cm⁻¹, which was the C=O characteristic peak. There was no broad peak in the 2500-2800 cm⁻¹ region where an O-H stretch resonance peak in the carboxylic acid group (COOH) should be. See Figure 4.

When analyzed with NMR by using acetone (NMR grade) as solvent, the sample showed the multiple peaks at 7.0-9.0 ppm, which are aromatic protons on the pyridine, and a characteristic peak of -CHO at 11.55 ppm, which was a little further from which is supposed to be. See Figures 5 and 6 in

the appendix. The reason accounting for this is that the zero position (5% TMS as the reference) shifted to the left.

The white "needle" product from 8-methyl quinoline oxidized by SeO, should be 8-quinoline aldehyde.

$$A_{g}/D_{g} + M_{u}OH \longrightarrow M_{0}NO_{g} + A_{f}OH$$
 (5)

$$Ag011 + 2 NH_10H \longrightarrow Ag(NH_3)_20H + 2 H_20$$
(6)

$$(H) + 2 A_{g}(NH_{3})_{2}OH \longrightarrow (H) + 2 A_{g}(H_{3})_{2}OH \longrightarrow (7)$$

B. 2-picoline

The raw product formed from the oxidation of 2-picoline finished was about 0.6 g. Because the apparatus for sublimation was not very efficient, only about 30 mg was collected. The solid was white. Under the microscope, the product was a tiny granulated solid. The melting point of the solid was 125-130 °C. The product dissolved in water easily. When the product was added with NaECO₃, a Lot of bubbles appeared. When it was added to an aqueous CuSO₄ solution, a blue precipitate appeared.

When analyzed with FTIR in a KBr disk (KBr disk as the reference), the product showed an intensive C=O characteristic

peak at 1719.6 cm⁻¹, a broad O-H (in COOH group) stretch peak at 2500-2800 cm⁻¹, and a weaker O-H (in water) peak at 3391-3450 cm⁻¹. See Figure 8. Therefore, the product was 2-pyridine carboxylic acid.



C. 4-picoline

The raw product formed from the oxidation of 4-picoline finished was reddish orange in color. After sublimation, the product was orange, and looked like a fine powder. The weight of the impure product was 0.312 g. The yield was 77%. Only about 25 mg of sublimated product was collected. When the melting point was checked, most of the product sublimated beginning at 260 °C, and melted at 285-295 °C. The pH of its aqueous solution was about 4. When the product was mixed with NaHCO₃ aqueous solution, bubbles appeared. When the product was added with aqueous CuSO₄ solution, a blue precipitate was obtained.

The product was analyzed with FTIR using a KBr disk

(another KBr disk was used as the reference). The product showed an intensive C=O characteristic peak at 1722.2 cm⁻¹, a medium broad O-H (in the COOH group) stretch peak at 2423.6-2374.6 cm⁻¹, and an O-H (in water) peak at 3434.8 cm⁻¹. See Figure 10. Thus, the reaction produced 4-pyridine carboxylic acid.



A comparison of the physical properties of the experimental results with the literature values is listed in Table I in the appendix.

D. 2,6-lutidine

The raw product was a white needle solid, 0.15 g. The product dissolved in water easily, but dissolved in ethyl ether with difficulty. When the aqueous solution of the product was mixed with NaHCO₃ solution, bubbles appeared. Its aqueous solution had a pH of about 4.

The FTIR spectrum (Figure 13) showed several

characteristic peaks, including a sharp peak at 1717.3 cm^{-1} (C=O), a broad peak at 2498 cm^{-1} (O-H in the COOH), and a peak at 3468.3 cm^{-1} (O-H in water). All the results show that the product should be 2,6-pyridine dicarboxylic acid.

$$HOOC - N - COOH + 2 N_{a}HCO_{3} - N - COCN_{a} + 2 CO_{2} + 2 H_{2}O \qquad (12)$$

E. Others

Following the same procedure, 3-picoline, lepidine, quinaldine, and quinoline were also studied.

2.0 ml of 3-picoline was heated with 0.5 g SeO_2 for 6 hours at a temperature just below 120 °C. The FTIR spectrum (Figure 15) showed a sharp peak at 1722.4 cm⁻¹ (C=O), a broad peak at 2385.2 cm⁻¹ (O-H in the COOH), and a peak at 3389.1 cm⁻¹ (O-H in water), which indicated that the product should be 3-pyridine carboxylic acid. The color of the liquid changed from colorless to yellow. The solid was black and there was no other kind of solid mixed in it. No product was obtained by using ethyl ether extraction and running through an alumina column.

The reaction of quinaldine (2-methyl quinoline) with SeO_2 occurred between 100-120 °C. The solid product was black.

Extracted by ethyl ether, a red liquid mixture with high viscosity was obtained, but the procedure failed to yield a pure product. No reaction occurred when a small portion of the liquid mixture was added with NaHCO₃ aqueous solution. A pH test showed about 7. The product did not mix well with water. The spectrum (Figure 17) from the liquid mixture of 2-methyl quinoline heated for 11 hours showed peaks at 3384 cm⁻¹ (O-H in water), 2923-3055 cm⁻¹ (C-H), 1706.2 cm⁻¹ (C=O). No peak occurred at the 2500-2800 cm⁻¹ region (O-H in COOH), which indicated that there was some 2-quinoline aldehyde produced.

2.0 ml lepidine (4-methyl quinoline) was heated with 0.5 g SeO₂ for 11 hours. Both the solid and liquid mixture were black and sticky. After the liquid mixture was extracted by ethyl ether and run through an aluminum oxide column, a brown liquid mixture with high viscosity was obtained. This mixture did not mix well with water, but mixed well with 6 M HC1. There was no reaction with NaHCO₃. The spectrum of the liquid mixture from 4-methyl quinoline oxidized for 11 hours (Figure 19) showed peaks at 1713.3 cm⁻¹ (C=O), 2870.3-3066.9 cm⁻¹ (C-H), and 3384.4 cm⁻¹ (O-H), which indicates that there was some 4-quinoline aldehyde produced.

After the mixture of 2.0 ml quinoline with 0.5 g SeO_2 was heated for 17 hours at about 120 °C, the liquid was dark and reddish black, not too viscous. The solid was black. The spectra of the quinoline before and after heating with SeO_2 were almost the same. See Figure 20 and 21. Thus, the aromatic

rings on quinoline won't open when heated with SeO2.

F. Unsuccessful reactions whose the yield of the products were too small for the products to be collected

1. 0.500 g SeO₂ and 1.5 ml 2-nitrotoluene were heated for about 14 hours at about 100 °C. Both black and white solid were obtained. A white "needle" solid was obtained which was studied by FTIR and the spectra was the same as the one of SeO₂.

2. 0.500 g SeO₂ and 1.5 ml 2-nitrotoluene with 7 ml water as solvent were heated for about 54 hours at about 100 °C. No desired product was produced.

3. 0.500 g SeO₂ and 2.000 g 4-nitrotoluene (solid) with 5.5 ml benzene as solvent were heated for about 24 hours. No desired product was produced.

4. 0.500 g SeO_2 and 2.000 g 4-nitrotoluene with 5 ml pyridine as solvent were heated for about 24 hours. No desired product was obtained.

5. 0.500 g SeO₂ and 2.500 g 2,4-dinitrotoluene with 6 ml benzene as solvent were heated for 48 hours. The spectrum of the solid product was extremely difficult to interpret, but did indicate that some kind of reaction had occurred.

6. 0.500 g SeO₂ and 5 ml pyridine were heated for about 15 hours. The spectra before and after pyridine was heated are the same.

The yield of product from 8-methyl quinoline, 2-picoline, and 4-picoline was fair, because some product was lost during either extraction or separation. Another reason for the low yield from 2-picoline and 4-picoline is that some products were not isolated in the liquid portion of the reaction mixture. The three reactions went to completion; no SeO₂ was left.

When the Tollens test was done on the products from 2picoline and 4-picoline, which are carboxylic acids, positive results were obtained. The reason for this phenomenon is that the products are not pure enough.

The spectra of the product mixtures from 2-methyl quinoline and 4-methyl quinoline show that some reactions occurred just like the suggested reaction equations in the introduction section. But the products were not obtained. The suggested reason is that the activation energies for the above three reactions are so high that the rates of the reactions are very slow. Moreover, the two compounds are both light and heat sensitive species. Even if there were no SeO_2 added to them, the two compounds become darker and sticky. Thus, even though the spectra does show the desired information, only traces of the products were obtained.

The most successful reactions and their assumed stoichiometry are shown below.

$$2 \left| \int_{-\infty}^{\infty} CH_3 + 3 S_e O_2 - 2 \left| \int_{-\infty}^{\infty} COOH + 3 S_e \right| + 2H_2 0 \quad (13)$$



$$H_{2} + S_{c} \ell_{2} \longrightarrow H_{2} + S_{e} + H_{2}$$
(15)

Other reactions are also possible.

$$H_{3}C - \begin{pmatrix} N \\ - \end{pmatrix} - \begin{pmatrix} CH_{3} \\ + \end{pmatrix} + 3 \\ Se \\ 0_{2} \end{pmatrix} \longrightarrow H^{0}OC - \begin{pmatrix} N \\ - \end{pmatrix} - \begin{pmatrix} CCOH \\ + \end{pmatrix} + 3 \\ Se \end{pmatrix} + 2 \\ H_{2}O \end{pmatrix} (16)$$

$$2 \begin{pmatrix} N \\ - \\ CH_{3} \end{pmatrix} + 3 \\ Se \\ 0_{2} \end{pmatrix} \longrightarrow 2 \begin{pmatrix} N \\ - \\ - \end{pmatrix} + 3 \\ Se \end{pmatrix} + 2 \\ H_{2}O \end{pmatrix} (17)$$

$$H_{3}C - \begin{pmatrix} N \\ - \\ - \end{pmatrix} + \\ Se \\ 0_{2} \end{pmatrix} \longrightarrow 0 \\ H^{0}C - \begin{pmatrix} N \\ - \\ - \end{pmatrix} + \\ Se \\ - \end{pmatrix} + \\ H_{2}O \end{pmatrix} (18)$$

$$(18)$$

$$(18)$$

$$(18)$$

$$(18)$$

$$(19)$$

ĊНО

The postulated mechanism of one of the typical methyl quinoline oxidation reactions, the 8-methyl quinoline oxidation reaction, is shown below.

Scheme 1:







The postulated mechanism of one of the typical methyl pyridine oxidation reactions, the 2-picoline oxidation reaction, is shown below.

Scheme 2:

Step 1:













Step 2:





The postulated mechanism of the 4-picoline oxidation reaction is shown below.

Scheme 3:

Step 1:





Step 3:



Step 4:







When water was added to the mixture of SeO_2 and 4picoline (0.500 g SeO_2 , 0.5 ml H₂O, and 1.0 ml 4-picoline), the reaction occurred at a lower temperature than without water, which is around 75 °C. The solid mixture was separated by decanting the liquid and then sublimating. The melting point of the sublimated solid was 319 °C, which is almost the same as the one obtained following the same procedure without water. The FTIR spectrum of the sublimated solid obtained by water dissolving SeO_2 first was very similar to the one without water (Figure 11). Since the temperature for the one with water is lower than the one without water, 4-pyridine carboxylic acid was obtained more easily when water was added than it was obtained when no water was added. Thus, water assists the reactions.

Furthermore, there is no reaction when toluene was heated with SeO_2 . The only difference between the methyl pyridine and toluene is that the pyridine ring contains a nitrogen, while the benzene ring does not. Thus, the lone pair of electrons of nitrogen on the ring may play a very important role in these reactions. One important function of the lone pair of electrons on the nitrogen atom is a Lewis base center which can take part in the reaction like those suggested in the mechanism. Especially when a methyl group is on the para and ortho positions, the resonance forms showed that nitrogenselenium bond formation or carbocation formation would be more important in 2-picoline and 4-picoline than in 3-picoline.

The resonance forms of carbanions:

1. 2-picoline



2. 4-picoline



3. 3-picoline



Conclusion

The products from 2-picoline, 4-picoline and 8-methyl quinoline oxidized by selenium dioxide were 2-pyridine carboxylic acid, 4-pyridine carboxylic acid, and 8-quinoline aldehyde, respectively, and the percentage yields were 50%, 76.8%, and 49.1%, respectively. 2,6-pyridine carboxylic acid was also the product when 2,6-lutidine was oxidized by SeO₂, and 3-pyridine carboxylic acid was the product from 3picoline. Aldehydes were the products from lepidine and quinaldine oxidation reactions.

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Appendix

	color, state		m.p. (°C)		b.p. (°C)	
	lit	exp	lit	exp	lit	exp
8-quinoline		white		77-82		
aldehyde		crys				
2-pyridine ca-	nd (w	white	136.7	126-	sub	sub
rboxylic acid	al)	granu		130		
4-pyridine ca-	plat	orang	319	285-	sub	sub
rboxylic acid	elet	plat		295	260	260
					15va	va

Table I: The comparison of the physical properties of the experimental results with the literature values

Note for the shorthands:

m.p.= melting point	b.p.= boiling point
lit = literature	exp = experimental
crys = crystals	nd = needle
w = white	al = alcohol
granu = granulate	<pre>sub = sublimation</pre>
plat = platelet	va = vacuum

Figure 1: The equipment setup for experiments



Figure 2: The equipment setup for sublimation







Transmittance



Figure 4: Solid From 8-methyl quinoline product mixture after ether extraction Reference: nujol Scan: 16



Figure 5: Solid from 8-methyl quinoline oxidized mixture after ether extraction (I) Reference: TMS (5%) H1 level: normal Sovlent: acetone (NMR grade) Gain: x1 Amplitude: 10 Filter Hz: normal Sweep time: 150/sec X axis: normal Date: Jan 30, 1992



Figure 6: Solid from 8-methyl quinoline oxidized mixture after ehter extraction (II) Reference: TMS (5%) Solvent: acetone (NMR grade) Amplitude: 10 H1 level: normal Gain: x1 Filter Hz: normal Sweep time: 150/sec X axis: x10 Date: Jan 30, 1992













altarisolid of 4-pic ox, by Se02 diss, in H20 in KBr

16

Scan:

Reference: KBr disk





solid from evap. liquid of 2,6-lutidine ox in kBr

16

Scan:

disk

KBr

Reference:

2,6-lutidine after evaperation



Reference: air Scan: 16

















Reference: air Scan: 16



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marilyn Kaohong Thou

Signature of Author

May 8, 1992 Date

The use of selenium (11) oxide to oridize aromatic medyl groups Title of Thesis/Research Project

ana Seelect impature of Graduate Office Staff Member

May 1, 1992 Date Réceived