Copper(II)-salt-promoted oxidative ring-opening reactions of bicyclic cyclopropanol derivatives via radical pathways

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Abstract
Copper(II)-salt-promoted oxidative ring-opening reactions of bicyclic cyclopropanol derivatives were investigated. The regioselectivities of these processes were found to be influenced by the structure of cyclopropanols as well as the counter anion of the copper(II) salts. A mechanism involving rearrangement reactions of radical intermediates and their competitive trapping by copper ions is proposed.

Introduction
Radical ions are key intermediates in electron-transfer (ET) reactions of organic molecules [1-5] and they often undergo fragmentations to yield free radicals and ions [6-10]. The ensuing reaction pathways followed by the resulting radicals are governed not only by their intrinsic nature but also by the nature of co-existing redox reagents. In principle, radical intermediates in ET-promoted reactions have a tendency to participate in further ET processes to generate ionic species when stoichiometric amounts of redox reagents are used (Scheme 1) [1-10]. In contrast, radical intermediates formed by a photoinduced ET (PET) are less likely to undergo these secondary reactions, because steady-state concentrations of PET-generated redox reagents are used (Scheme 1) [1-10].
reagents are low [11-19]. When radical intermediates and ions derived from their precursor radical ions undergo different rearrangement reactions, it is often possible to distinguish respective reaction pathways of radicals and ions by examining the product distributions of the reactions of substrates that contain appropriate probe moieties (Scheme 2).

In past studies, we developed unique families of substances (exemplified by probes I and II in Figure 1) that act as radical ion probes [20] and found that radical intermediates in their reaction pathways undergo efficient 5-exo hexenyl radical cyclization reactions [21], (Figure 1) [22-30]. For example, PET reactions of probe I with amines were observed to produce a spirocyclic ketone product while its reduction reaction induced by samarium diiodide (SmI$_2$) gives rise to a cyclopropanol (left in Scheme 3) [22,24,27]. On the other hand, the same spirocyclic ketone is obtained in the 9,10-dicyanoanthracene (DCA) and biphenyl (BP) sensitized PET reaction of probe II, while reactions of this substrate with certain oxidants afford ring-expanded ketone and enone products (right in Scheme 3) [23,25,26,28-30].

Careful examination of the reaction of probe II with FeCl$_3$ revealed that a small quantity of the spirocyclic ketone was also formed [23,28]. This observation prompted us to explore the possibility that the free radical rearrangement route becomes more predominant when oxidizing reagents weaker than Fe(III) are used to promote the reaction. Based on a consideration of the redox potentials of Fe and Cu ions (Eº in H$_2$O, V versus NHE), +0.77 for Fe(III)/Fe(II), +0.17 for copper(II)/copper(I) [31], we chose to explore the use of copper(II) reagents in this effort. Although various ET reagents have been employed to promote reactions of cyclopropanol derivatives [32-47], the employment of copper(III) reagents to induce reactions has not been extensively studied [36,39]. In the investigation described below, we have explored copper(II)-salt-promoted oxidative ring-opening reactions of selected bicyclic cyclopropanol derivatives.

![Scheme 2](image2.png)

**Scheme 2:** Using rearrangements of radicals and ions to distinguish mechanistic pathways for ET-reactions.

![Scheme 3](image3.png)

**Scheme 3:** Reductive ET reactions of the probe I (left) and oxidative ET reactions of probe II (right).
Results and Discussion

In the initial phase of this effort, we examined the reaction of cyclopropyl silyl ether 1a (0.40 mmol) with copper(II) acetate, Cu(OAc)$_2$ (1.1 equiv) for 1 h at room temperature (Scheme 4). Under these conditions no reaction takes place, which we attribute to the steric bulk of the silyl substituent causing interference in the reaction of the substrate with Cu(OAc)$_2$. In accordance with this reasoning, we found that inclusion of $n$-Bu$_4$NF (1.2 equiv) in the reaction mixture led to a reaction that completely consumes 1a and produced the expected spirocyclic ketone 2, albeit in low yield, and spirocyclic ketone 3 possessing an exo-methylene moiety as the major product. Interestingly, ketone 3 was previously observed as a product of the DCA–BP-sensitized PET reaction of 1a in the presence of Cu(OAc)$_2$ [25]. Only a trace amount of ring-expanded enone 4 along with small amounts of desilylated alcohol 1b (ca. 8%) and ketone 5 were detected in the product mixture by using $^1$H NMR analysis. Treatment of 1a (0.19 mmol) with $n$-Bu$_4$NF (2.0 equiv) in THF for 1 h followed by hydrolysis gave a mixture of 1b and 5 (12:88). Therefore, 5 may not result from the copper(II)-oxidation reaction.

Based on the above observations, we anticipated that sterically less hindered cyclopropanols would more efficiently undergo copper(II)-induced oxidation reactions than the corresponding silyl ethers. To probe this prediction, cyclopropanols 1, prepared by SmI$_2$-promoted intramolecular Barbier reaction of the corresponding $\alpha$-bromomethyl cycloalkanones 6 [28], were subjected to reactions promoted by various copper(II) salts, CuX$_2$ (Scheme 5).

The results of the reaction of 1b with Cu(OAc)$_2$ (Scheme 6) are summarized in Table 1. As expected, this process produces ketone 3 as the major product along with both 2 and ring-expanded enone 4 as minor products. Moreover, the order of addition of 1b and Cu(OAc)$_2$ does not significantly affect the product distribution (compare Table 1, entry 1 to entry 2). An exploration of solvent effects revealed that MeCN is more suitable than DMF while the solubility of Cu(OAc)$_2$ is higher in the latter solvent (compare Table 1, entry 1 to entry 5). In entry 5 (Table 1), ring-opened ketone 5 was obtained. In other experiments (see below), the formations of 5 (see Table 2), and other ring-opened ketones 22 (see Table 3) and 25 (see Scheme 11) are also observed. These products might be formed by deprotonation of the corresponding cyclopropanols 1. It should be noted that THF is not an appropriate solvent for this reaction (Table 1, entry 8), a finding that is in contrast to the previous
Table 1: Reaction of cyclopropanol 1b with Cu(OAc)₂.¹

| entry | Cu(OAc)₂ (equiv) | solvent | conv of 1b² (%) | 2 | yields³ (%) |
|-------|-----------------|---------|----------------|---|-------------|
| 1     | 1.1             | MeCN    | 91             | 0 | 70          | ~5ᵈ |
| 2ᵃ    | 1.1             | MeCN    | 100            | 0 | 70          | ~8ᵈ |
| 3     | 0.5             | MeCN    | 82             | 5 | 47          | ~2ᵈ |
| 4ᵇ    | 2.2             | MeCN    | 100            | 0 | 62          | 4   |
| 5ᶜ    | 1.1             | DMF     | 60             | 0 | 35          | trace |
| 6     | 2.2             | DMF     | 69             | 1 | 40          | ~1ᵈ |
| 7ᵇ    | 1.1             | CH₂Cl₂  | 85             | 10| 38          | trace |
| 8     | 1.1             | THF     | 28             | trace | 6 | trace |

¹1b derived from 6b (0.40 mmol) was added to Cu(OAc)₂ in a solvent (4 mL). ²Determined by ¹H NMR based on the yield of the isolated products (see Experimental). ³Isolated or determined by ¹H NMR. ⁴Crude yields. ⁵Cu(OAc)₂ was added to 1b in a solvent. ⁶Ketone 5 (~5%) was obtained.

The observations described above suggest that the mechanism for this reaction shown in Scheme 7 is plausible. Because copper(II) is a relatively weak outer-sphere SET oxidant [1], addition of the hydroxy group of 1b to Cu(OAc)₂ takes place initially to produce Lewis base–acid complex 7, followed by inner-sphere ET involving elimination of CuOAc and AcOH, which gives cyclopropoxy radical 8. Either external or internal bond cleavage of 8 generates the respective primary alkyl radical 9 or tertiary alkyl radical 10. An equilibrium interconverting 9 and 10 through 8 [22-30] might occur (see below). A mechanism on the fragmentation of initially formed
metal–organic complexes, giving β-ketoalkyl radicals [40], cyclopropoxy radicals [25,28,48-50], or β-metalated carbonyls [39], is still controversial [35-47]. Thus, we believe the reaction follows the pathways shown in Scheme 7 although the possibility of direct formations of 9 and 10, a concerted ET and cyclopropane ring opening, cannot be ruled out. Rapid 5-exo cyclization of hexenyl radical moiety in 9 produces spirocyclic primary alkyl radical 11. Hydrogen-atom abstraction by 11 then leads to formation of spirocyclic ketone product 2, while trapping of 11 by CuOAc followed by β-H elimination (either hydride elimination or deprotonation) [39] of the resulting organocopper intermediate 12 generates the exocyclic methylene analogue 3 as the major product [25]. Protonation of 12 might be an alternative route for the formation of 2 (not shown in Scheme 7). Reactions of alkyl radicals with copper(II) are well documented [51,52], and it has been also suggested that copper(I) efficiently reacts with alkyl radicals [39]. As described, 1.1 equiv of Cu(OAc)₂ leads to nearly complete reaction of 1b (see entry 1 and entry 2 in Table 1). Thus, CuOAc which is generated after initial ET between Cu(OAc)₂ and 1b may capture the primary alkyl radical 11. In addition, although not predominant, oxidation of 10 by Cu(OAc)₂ gives rise to tertiary carbocation 13 [51,52], which is then deprotonated to form enone 4.

Studies of the effect of the counter ion on copper(II)-promoted reactions of 1b (Scheme 8) gave the results summarized in Table 2. While no reaction occurred when copper(II) acetylacetone, Cu(acac)₂, is used, (Table 2, entry 1), copper(II) 2-ethylhexanoate, Cu(ehex)₂, serves as an effective oxidant in transforming 1b to 3 in a yield that is comparable to the process promoted by Cu(OAc)₂ (compare Table 2, entry 2 to entry 3). Noticeable amounts of 2 are also generated in this reaction. When CuCl₂ is employed to oxidize 1b, only ring-expanded ketones 4 and 14 are produced along with a lesser amount of chloro ketone 15, and competitive formation of 2 and 3 does not occur (Table 2, entry 4). An increase in the amount of CuCl₂ causes a slight increase in the conversion of 1b and the total yield of ring-expanded products 4 and 14 (compare Table 2, entry 5 to entry 4). Interestingly, CuCl₂ (1.1 equiv) could also promote the reaction of silyl ether 1a to produce 4 (23%), 14 (4%) and 15 (3%) at 89% conversion of 1a. Although the origin

![Scheme 8: Reaction of cyclopropanol 1b with various copper(II) salts (CuX₂).](image-url)

| entry | X                  | Conv of 1b (%) | yields (%) | 2 | 3 | 4 |
|-------|--------------------|----------------|------------|---|---|---|
| 1     | acetyl acetonate (acac) | 0             | No reaction |   |   |   |
| 2     | 2-ethyl hexanoate (ehex) | 94            | 5          | 63 |   |   |
| 3     | OAc                | 91            | 0          | 70 |   |   |
| 4     | Cl                 | 63            | 0          | 0  | ~25% |   |
| 5     | Cl                 | 71            | 0          | 0  | 34% (9)%   |   |
| 6     | OTf                | 77            | trace      | 0  | ~11% (34)% |   |

*a1b derived from 6b (0.40 mmol) was added to CuX₂ (1.1 equiv for entries 1–4, 6; 2.2 equiv for entry 5) in MeCN (4 mL). *bDetermined by ¹H NMR based on the yield of the isolated products (see Experimental). *cIsolated or determined by ¹H NMR. *dNumber in the parenthesis is the yield of chloro adduct 14. *eKetone 5 (9%) and chloro ketone 15 (4%) were obtained. *fNumber in parentheses is the yield of acetamide 16.
of 15 is uncertain, one possibility is that it is formed by halogen substitution of unconverted bromide 6b to 1b by SmI₂. The formation of chloro ketone 23 (see Table 3) may be similarly explained. Finally, reaction of 1b with Cu(OTf)₂ leads to formation of ring-expanded products 4 and 16 and a negligible amount of 2 (Table 2, entry 6). Acetamide 16 is probably produced in this process through a Ritter reaction between cation 13 and the solvent acetonitrile (Scheme 9).

Hypothetically, both the Lewis acidity and oxidizing ability of CuX₂ should depend on the basicity of the counter ion (X⁻: conjugate base of HX). Based on the acidity order HX, TfOH > HCl > AcOH ~ 2-ethyl hexanoic acid > acetylacetone [53,54], it is possible to assign Cu(acac)₂, which is ineffective in promoting the reaction, as the weakest oxidant. On the other hand, CuCl₂ and Cu(OTf)₂ induce reactions that follow a different pathway from those promoted by copper(II) carboxylates. These observations suggest that a rapid equilibrium does indeed exist between isomeric radical intermediates 9 and 10 (Scheme 7) and that the thermodynamically less stable isomer 9 undergoes fast hexenyl-radical cyclization leading to the formation of 11 in reactions promoted by copper(II) carboxylates. On the other hand, a fast oxidation of the more stable isomer 10 by stronger oxidants such as CuCl₂ or Cu(OTf)₂ occurs to give the stable tertiary carbocation 13, which is then captured by Cl⁻ or MeCN.

In order to explore the generality of the proposed counteranion-dependent reactivity switch in the nature of copper(II)-promoted reactions of 1, the pentenyl-substituted cyclo-

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Scheme 9: Formation of acetoamide 16 from the cation 13.

Scheme 10: Reaction of cyclopropanol 1c with various copper(II) salts (CuX₂).

Table 3: Reaction of cyclopropanol 1c with various copper(II) salts (CuX₂).^a

| entry | X      | additive | conv of 1c (%) | yields (%) | 19 | 20 |
|-------|--------|----------|----------------|------------|----|----|
| 1     | OAc    | –        | 95             | 55         | 0  | 0  |
| 2     | OAc    | pyridine (1.2 equiv) | –65f | 33         | 0  | 0  |
| 3     | ehex   | –        | 160           | 33         | 0  | 0  |
| 4e    | Cl     | –        | 63             | 0          | 28(8)f | 0  |
| 5     | OTf    | –        | –93²          | 0          | 13(33)f | 0  |

^aCuX₂ (1.1 equiv) was added to 1c derived from 6c (0.4 mmol) in MeCN (4 mL). ^bDetermined by ¹H NMR based on the yield of the isolated products (see Experimental). ^cIsolated or determined by ¹H NMR. ^dBased on the crude yield of 1c. ^eKetone 22 (14%) and chloro ketone 23 (5%) were obtained. ^fNumber in parentheses is the yield of chloro adduct 21. ^gNumber in parentheses is the yield of acetoamide 24.
propanol 1c was employed as the substrate (Scheme 10 and Table 3). A major product of the reaction of 1c promoted by Cu(OAc)$_2$ was observed to be the exo-methylene containing spirocyclic ketone 19 (Table 3, entry 1), which is produced in the DCA–BP sensitized PET reaction of silyl ether of 1e in the presence of Cu(OAc)$_2$ [25]. Contrary to the expectation that a base could assist the deprotonation of the complex between copper and 1c (similar to 7 in Scheme 7), the addition of pyridine was found to decelerate the reaction (Table 3, entry 2). This observation suggests that coordination of pyridine to copper reduces the oxidizing ability of Cu(OAc)$_2$. Cu(ehex)$_2$ was also effective to give 19 although the yield was relatively low (Table 3, entry 3). Reaction of 1c with CuCl$_2$ was observed to form ring-expanded ketones 20 and 21, along with small amounts of 22 and 23. However, competitive generation of 19 does not take place (Table 3, entry 4). Finally, reaction of 1c with Cu(OTf)$_2$ leads to the formation of ring-expanded enone 20 and acetoamide 24 (Table 3, entry 5).

As described above, observation of the occurrence of hexenyl-radical cyclization processes serves as good evidence for the involvement of radical intermediates in mechanistic pathways for reactions of 1b and 1c. In order to gain more information about these processes, we explored an oxidation reaction of substrate 1d, which does not contain an alkene tether and whose reaction pathway, thus, cannot involve radical intermediates that undergo hexenyl-radical cyclization. We observed that reaction of the methyl-substituted cyclopropanol 1d with Cu(OAc)$_2$ leads to formation of the ring-expanded enone 25 as a major product along with a trace amount of ketone 26 (Scheme 11).

The Cu(OAc)$_2$-promoted reactions of 1c and 1d are compared in Scheme 12. The ring-expanded tertiary alkyl radical 27, formed as an intermediate in the reaction of 8 (R = (CH$_2$)$_3$CH=CH$_2$), undergoes rapid 5-exo hexenyl cyclization along the route for the production of spirocyclic ketone 19. Thus, oxidation of 27 followed by deprotonation to give enone 20 is a minor contributor. If an external bond cleavage of 8 occurs, cyclization of heptenyl-radical moiety in the resulting primary alkyl radical (not shown in Scheme 12) is expected. However, the exo-cyclization of heptenyl radical is two orders of magnitude slower than that of the hexenyl radical [55]. In contrast, because no competitive radical-rearrangement process exists, the corresponding radical intermediate 28 formed from 8 (R = Me) undergoes sequential oxidation and deprotonation to give enone 25 as a major product.

**Conclusion**

Various copper(II) salts promote ring-opening reactions of bicyclic cyclopropanol derivatives. Using substrates that possess hexenyl moieties, we observed that the nature of the counter anion of copper(II) salts has a significant impact on the product distributions. The results suggest that reaction pathways followed by radical intermediates derived from these substrates are strongly influenced by post ring-opening steps.
Thus, cyclopropane bond cleavage, which is reversible, does not serve as a product-determining step if a rapid follow-up reaction like hexenyl-radical cyclization does not exist. The results show that by using a proper choice of copper(II) salts it is possible to control the reaction pathways followed by radical and ionic intermediates derived from the initially formed Lewis base–acid complexes if the radicals and ions are capable of undergoing different rearrangement reactions.

Experimental

General: NMR spectra were recorded in CDCl₃ with Me₄Si as an internal standard at 400 MHz for ¹H NMR and 100 MHz for ¹³C NMR. Column chromatography was performed with silica gel (Wakogel C-200). Preparative TLC was performed on 20 cm × 20 cm plates coated with silica gel (Wakogel B-5F).

Preparation of cyclopropanols 1: Cyclopropanol derivatives 1 were prepared from the corresponding bromo ketones 6 following previously reported procedures [25, 28]. Products 1a–1c, and 1d were determined by using ¹H NMR analysis of the crude reaction mixtures were performed in a similar manner. Because cyclopropanes 1 have a tendency to partially decompose during silica-gel chromatography, their conversion in reactions was determined by using ¹H NMR analysis of the crude reaction mixtures. When product isolations were not performed, yields were also determined by ¹H NMR, and crude yields are reported in some cases.

Reaction of cyclopropanols 1 with copper(II) salts: A typical experiment using 1b is described (Table 1, entry 1). To Cu(OAc)₂ (79.9 mg, 0.44 mmol) in MeCN (4 mL) was added 1b (85.7 mg, 0.40 mmol). In some experiments, the order of addition was reversed (see entry 2 in Table 1 and Table 3). The resulting mixture was stirred under N₂ at room temperature for 1 h, diluted with water and extracted with Et₂O. The extract was washed with water, saturated aqueous Na₂SO₄, saturated aqueous NaHCO₃, and brine, dried over anhydrous MgSO₄, and concentrated in vacuo giving a residue that was subjected to TLC (AcOEt:n-hexane 20:1), and 3 (59.3 mg, 0.28 mmol, 70%) and 4 (<5 mg, ~0.02 mmol, ~5%) were obtained. Other reactions were performed in a similar manner. Because cyclopropanes 1 have a tendency to partially decompose during silica-gel chromatography, their conversion in reactions was determined by using ¹H NMR analysis of the crude reaction mixtures. When product isolations were not performed, yields were also determined by ¹H NMR, and crude yields are reported in some cases.

1-Hydroxy-3-(4-pentenyl)-6,7-benzobicyclo[4.1.0]heptane (1c): Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 8.05–8.02 (m, 1H), 7.51–7.46 (m, 1H), 7.34–7.23 (m, 2H), 5.77–5.70 (m, 1H), 5.00–4.91 (m, 2H), 3.77 (d, J = 10.4 Hz, 1H), 3.64 (d, J = 10.4 Hz, 1H), 3.13–2.90 (m, 2H), 2.34–2.16 (m, 2H), 2.04–1.98 (m, 2H), 1.78–1.24 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 198.7, 143.0, 138.0, 133.5, 131.3, 128.8, 128.1, 126.8, 115.0, 48.6, 39.3, 33.9, 32.7, 30.9, 24.8, 22.7; IR (neat) νmax (cm⁻¹): 2938, 1680, 1600, 1454, 1304, 1224, 991, 910, 743; HRMS–ESI (m/z): [M + H⁺]⁺ calcd for C₁₄H₁₉O₇⁹Br, 307.0692; found, 307.0687; [M + H⁺]⁺ calcd for C₁₆H₁₉O₈¹Br, 309.0672; found, 306.0665.

3-(3-Butenyl)-3-chloro-1-benzosuberone (14): Brown liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.80 (d, J = 7.6 Hz, 1H), 7.41 (t, J = 6.6 Hz, 1H), 7.32–7.29 (m, 2H), 5.88–5.78 (m, 1H), 5.00–4.91 (m, 2H), 3.13–2.90 (m, 2H), 2.34–2.16 (m, 2H), 2.04–1.98 (m, 2H), 1.78–1.24 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 198.7, 143.0, 138.0, 133.5, 131.3, 128.8, 128.1, 126.8, 115.0, 48.6, 39.3, 33.9, 32.7, 30.9, 24.8, 22.7; IR (neat) νmax (cm⁻¹): 2938, 1680, 1600, 1454, 1304, 1224, 991, 910, 743; HRMS–ESI (m/z): [M + H⁺]⁺ calcd for C₁₄H₁₉O₇⁹Br, 307.0692; found, 307.0687; [M + H⁺]⁺ calcd for C₁₆H₁₉O₈¹Br, 309.0672; found, 306.0665.
3-(Acetylamino)-3-(3-butenyl)-1-benzosuberone (16): Yellow solid; mp 105.0–107.0 °C; 1H NMR (400 MHz, CDCl3) δ 7.77 (d, J = 6.4 Hz, 1H), 7.41 (t, J = 6.8 Hz, 1H), 7.30 (t, J = 6.4 Hz, 1H), 7.25 (d, J = 8.0 Hz, 1H), 5.87–5.77 (m, 1H), 5.56 (bs, 1H), 5.06–4.95 (m, 2H), 3.12–2.97 (m, 4H), 2.46–2.40 (m, 1H), 2.27–2.20 (m, 1H), 2.10–2.03 (m, 3H), 1.99–1.95 (m, 4H); 13C NMR (100 MHz, CDCl3) δ 201.3, 170.7, 144.2, 138.2, 138.1, 132.1, 130.3, 128.6, 126.6, 115.0, 57.5, 50.9, 39.3, 36.1, 31.2, 28.3, 24.2; IR (neat) νmax (cm⁻¹) 3308, 3209, 2246, 1665, 1599, 1548, 1450, 1298, 1232, 912, 732; HRMS–ESI (m/z): [M + Na]+ calcld for C16H17O3Cl, 271.1567; found, 294.1463.

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