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Comparative studies of the photochemical and thermal reactivities of the molybdenum–molybdenum single bond in $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_6]$ and triple bond in $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_4]$ towards nitrite and nitrate

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Abstract

Irradiation at $\lambda = 507$ and 391 nm of $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_6]$ in a degassed tetrahydrofuran (THF) or THF-MeOH solution containing nitrite gives $[Mo(\eta^5\text{-}C_5H_5)(CO)_2NO]$ and several oxo complexes including $[\{Mo(\eta^5\text{-}C_5H_5)(O)_2\}_2O]$ in good yields. The quantum yields for the disappearance of $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_6]$ in the reaction with NO_2^- depend on the nitrite concentration, thus suggesting participation of the metal-radical intermediate in the reduction of nitrite. Reactions of $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_4]$ with nitrite or nitrate in the dark give the same nitrosyl and oxo complexes as above. An oxygen atom in nitrite or nitrate is mainly transferred onto the molybdenum atom both in the photochemical and the dark reactions.

Introduction

In recent years there has been considerable interest in the chemistry of complexes containing metal–metal bonds. One of the most widely investigated classes of compounds is the (η^5-cyclopentadienyl)molybdenum carbonyl dimer, for example, $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_6]$ (**1a**) and $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_4]$ (**2a**) [1,2]. The Mo–Mo single bond in **1a** is cleaved by irradiation at $\simeq 400$ or $\simeq 500$ nm to give a very reactive 17-electron metal-radical intermediate which can be trapped by NO and CCl_4 with production of $[Mo(\eta^5\text{-}C_5H_5)(CO)_2NO]$ (**3a**) [3] and $[Mo(\eta^5\text{-}C_5H_5)(CO)_3Cl]$ (**4a**) [4], respectively. The Mo≡Mo triple bond in **2a** has been shown to play an important role in the formation of μ-nitrene molybdenum complexes on the reactions with organic nitro compounds [5].

We have become interested in comparative studies on the behavior of both single and triple bonds in the molybdenum dimers towards particular substrates, for

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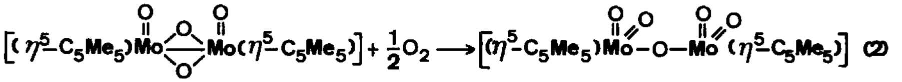
example, NO_2^- and NO_3^-. Some of the reactions of **1a** and **2a** with NO_2^- and NO_3^- have previously been published [6]. This paper reports in more detail on this and related reactions.

Results and discussion

Photochemical reactions. A deep red solution of **1a** and excess $NaNO_2$ in a degassed tetrahydrofuran (THF) or THF-MeOH solution did not change color for a long time when kept in the dark, and almost all of the starting material was recovered. Under irradiation at $\lambda \approx 400$ nm or in sunlight, however, the solution changed rapidly from deep red to orange yellow. The reaction mixture was dried and then extracted into benzene. An orange crystalline solid, isolated from the extract, was identified as the nitrosyl complex **3a** [7] (with 90% yield based on the stoichiometry of eq. 1) by

$$\underset{\mathbf{1a}}{[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_6]} + NO_2^- \xrightarrow{h\nu} \underset{\mathbf{3a}}{[Mo(\eta^5\text{-}C_5H_5)(CO)_2NO]} + 4CO + \underset{\mathbf{A}}{[(\eta^5\text{-}C_5H_5)Mo{=}O]^-} \quad (1)$$

IR (2020, 1945 (ν CO), 1691 cm^{-1} (ν NO)), ^1H NMR (C_5H_5 5.76 ppm in acetone-d_6) spectroscopy, and elemental analysis. The pale-yellow residue which remained after the extraction was not pure and was found to be composed of several oxo complexes in addition to $[\{Mo(\eta^5\text{-}C_5H_5)(O)_2\}_2O]$ (5a) [8] which was identified by comparing the IR spectrum of the mixture with that of an authentic sample of **5a** [ν CH η^5-C_5H_5 3100 cm^{-1}, ν_{as} Mo=O 930, 920 cm^{-1}, ν_s Mo=O 898, 850 cm^{-1}, ν_{as} Mo–O–Mo 770 cm^{-1}). When $[Mo_2(\eta^5\text{-}C_5Me_5)_2(CO)_6]$ (**1b**) was used for the reaction instead of **1a**, $[\{Mo(\eta^5\text{-}C_5Me_5)(O)_2\}_2O]$ (**5b**) and $[Mo(\eta^5\text{-}C_5Me_5)(O)(\mu\text{-}O)]_2$ (**6b**) were obtained in 42 and 37% yields (vide infra), respectively, as well as the nitrosyl complex $[Mo(\eta^5\text{-}C_5Me_5)(CO)_2NO]$ (**3b**) as a crude product in 48% yield. The oxo complexes **5a**, **5b**, and **6b** are probably produced by the reaction of **A** which was not isolated with excess $NaNO_2$ in the system and/or with oxygen in air during the isolation work-up of products. Furthermore, we found that **5b** and **6b** were also obtained from the reaction of **3b** with $NaNO_2$ in THF-methanol (1 : 1). Thus, the yields of the oxo complexes (**5b** and **6b**) are based on the molar ratio of these oxo complexes to the starting dimer **1b** used for the reaction. These new oxo complexes were characterized by IR: 914, 907 (sh) (ν_{as} Mo=O), 882 (νs Mo=O), 765 cm^{-1} (νas Mo–O–Mo) for **5b**; 917 (ν_{as} Mo=O), 714 cm^{-1} (ν_{as} Mo–O–Mo) for **6b**, ^1H NMR: $C_5(CH_3)_5$, 2.17 (s) ppm for **5b**, 1.95 (s) ppm for **6b** in $CDCl_3$, molecular weight measurement: found 530 for **5b**, 511 for **6b** in CH_2Cl_2, and elemental analysis. Complex **6b** in chloroform reacted slowly with oxygen to give **5b** (eq. 2).



$$[(\eta^5\text{-}C_5Me_5)Mo(O)(\mu\text{-}O)_2Mo(O)(\eta^5\text{-}C_5Me_5)] + \tfrac{1}{2}O_2 \longrightarrow [(\eta^5\text{-}C_5Me_5)Mo(O)_2\text{–}O\text{–}Mo(O)_2(\eta^5\text{-}C_5Me_5)] \quad (2)$$

6b **5b**

Recently **6b** was also prepared by the reaction of $[Mo_2(\eta^5\text{-}C_5Me_5)_2(CO)_4]$ (**2b**) with an equimolar amount of bis(t-butylisocyanamide)peroxonickel [10].

Gas phases in the reaction systems were analyzed by IR absorption spectroscopy. CO (ν: 2160 cm^{-1}) and very small amounts of CO_2 (ν_2: 2349 cm^{-1}, ν_3: 667 cm^{-1})

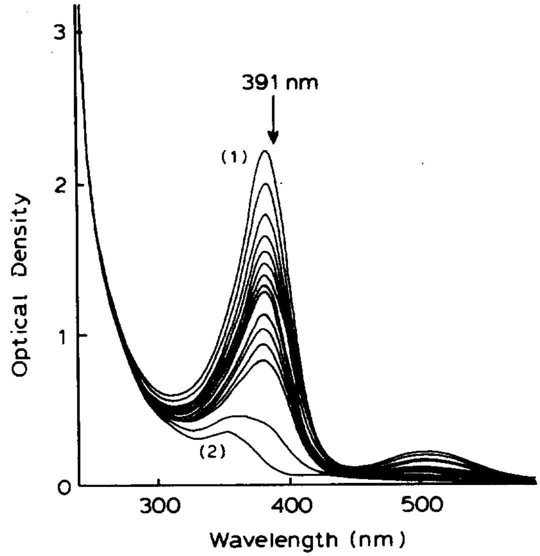


Fig. 1. Changes in spectral absorption of **1a** in MeOH-THF containig $NaNO_2$ with time and irradiation at 391 nm at 25 °C. Curves (1) and (2) represent the spectra after irradiation for 0 and 45 min, respectively. The initial concentration of the starting complex and $NaNO_2$ were 0.13 and 7.3 mmol dm^{-3}, respectively.

were detected. The ability of CO to reduce coordinated NO_2^- to coordinated NO (eq. 3) is well documented [11]. In our case, however, one of the oxygen atoms in NO_2^- was transferred to the molybdenum atom in **1a** to produce oxide species (**A**) (eq. 1). The transfer of the oxygen atom to coordinated CO to give CO_2 might also take place, though, only as a minor process (eq. 4).

$$M(NO_2)Ln + CO \longrightarrow M(NO)Ln + CO_2 \qquad (3)$$

$$\mathbf{1a} + NO_2^- \xrightarrow{h\nu} \mathbf{3a} + CO_2 + [Mo(\eta^5\text{-}C_5H_5)(CO)_3]^- \qquad (4)$$

Insight into the mechanism of the photochemical reduction of NO_2^- by **1a** is provided by the following studies. The complex **1a** shows characteristic UV-visible absorption bands in the region 350–550 nm: λ_{max} 385 (ϵ 17700, σ–σ^* transition of the Mo–Mo bond) and 505 nm (ϵ 1650, $d\pi$–σ^* transition of the Mo–Mo bond) [4], whereas **3a** has no absorption maxima in this region (ϵ 430 and 385 nm, ϵ 58.2 at 505 nm). The irradiation of **1a** at the σ–σ^* or $d\pi$–σ^* transition in the presence of $NaNO_2$ gave rise to a significant decrease in the optical densities over the region of 350 to 420 nm; the disappearance of **1a** was conveniently monitored by spectroscopy (Fig. 1). Wavelengths at 391 and 507 nm isolated by interference filters were used for the irradiations. Quantum yield measurements at these wavelengths were based on the disappearance of the band at 385 nm due to **1a**. The quantum yields (ϕ_d) for the disappearance of **1a** in the reaction with NO_2^- (56 equiv.) were 0.064 (irradiation at 507 nm) and 0.065 (at 391 nm), and did not show any marked dependence on wavelength. However, those with NO_2^- concentrations of 1.0, 7.8, 15.6, and 30.2 mmol dm^{-3} with a constant initial concentration of **1a** (0.13 mmol dm^{-3}) were 0.025, 0.065, 0.081, and 0.092 respectively, with error limits of ±0.007

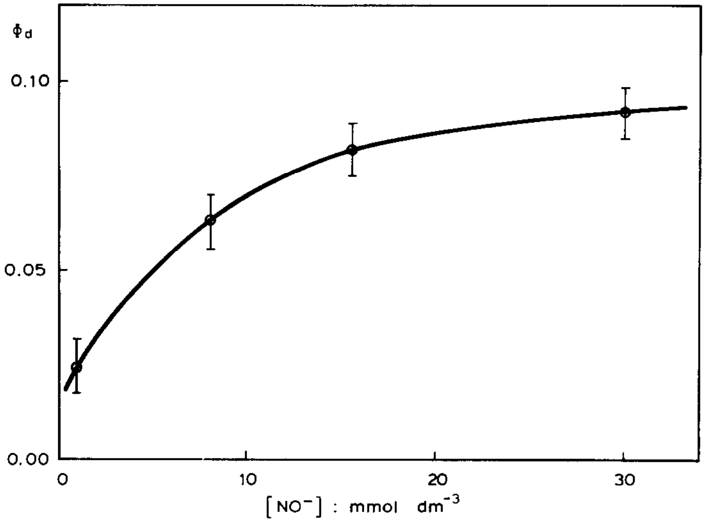
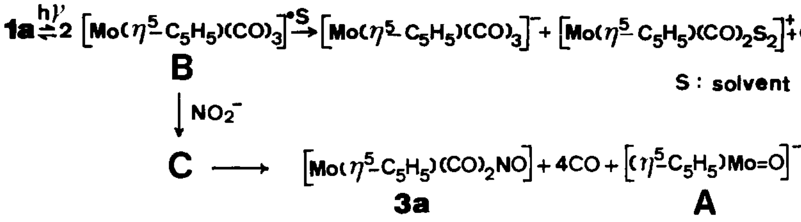


Fig. 2. The dependence of the ϕ_d values on the $NaNO_2$-concentration in the photolysis of **1a** (0.13 mmol dm^{-3}) in THF-MeOH at 391 nm. Error limits of the ϕ_d values were ±0.007.

for each, on irradiation at 391 nm (Fig. 2). The irradiation of **1a** brings about homolytic cleavage of the metal–metal bond and concomitant formation of a 17-electron metal-radical intermediate **B** and disproportionation occurs in polar or co-ordinating solvents (eq. 5-1) [12].

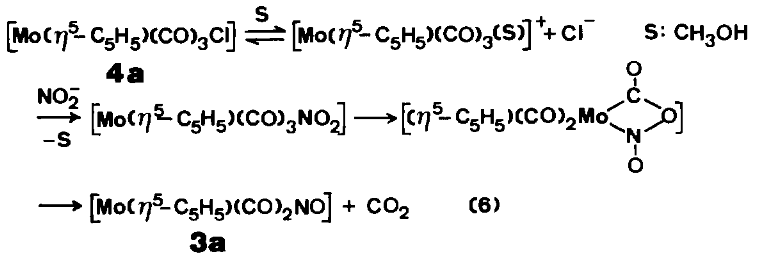


$$\mathbf{1a} \overset{h\nu}{\rightleftharpoons} 2\underset{\mathbf{B}}{[Mo(\eta^5\text{-}C_5H_5)(CO)_3]^{\bullet}} \xrightarrow{S} [Mo(\eta^5\text{-}C_5H_5)(CO)_3]^- + [Mo(\eta^5\text{-}C_5H_5)(CO)_2S_2]^+ + CO \quad (5\text{-}1)$$

S : solvent

$$\mathbf{B} \xrightarrow{NO_2^-} \mathbf{C} \longrightarrow \underset{\mathbf{3a}}{[Mo(\eta^5\text{-}C_5H_5)(CO)_2NO]} + 4CO + \underset{\mathbf{A}}{[(\eta^5\text{-}C_5H_5)Mo{=}O]^-} \quad (5\text{-}2)$$

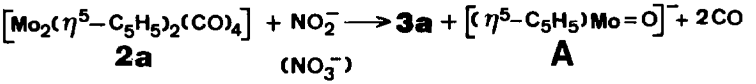
Dependence of the quantum yield on the NO_2^- concentration indicates that the metal-radical intermediate **B** rather than the ionic species participates in the reaction to form the nitrosyl complex. The reaction seems to proceed through a reactive intermediate (or an activated complex) **C** in which the bond rupture of N–O in NO_2^- ($\mathbf{B} + NO_2^- \rightarrow \mathbf{3a} + O^- + CO$) and O^--transfer to molybdenum atom ($\mathbf{B} + O^- \rightarrow \mathbf{A} + 3CO$) are facilitated (eq. 5–2). The reaction of **4a** with $NaNO_2$ in MeOH in the dark gave the nitrosyl complex **3a** with the evolution of a considerable amount of CO_2. This observation suggests that the reaction of **4a** with $NaNO_2$ proceeds through the following oxygen atom transfer to CO (eq. 6).



Photolysis of **1a** generates intermediates such as $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_5]$, $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_5(S)]$, and $[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_4(S)]$ (S: solvent) in addition to the 17-electron organometallic radical [1,3]. Thus, we tried to find out whether other paths to **3a** via these intermediates were present or not. After photolysis of **1a** for 30 min in the absence of NO_2^- the resulting deep red solution was allowed to react with the anion in the dark for 24 h. The reaction, however, gave **3a** in very low yield. This result indicates either that the intermediates play no role in the formation of the nitrosyl complex or that the intermediates were produced in insuffcient quantity to lead to the formation of the nitrosyl complex.

The photochemical reactions of **1a** and **1b** with NO_3^- proceeded at room temperature, to give traces of **3a** and **3b**, respectively.

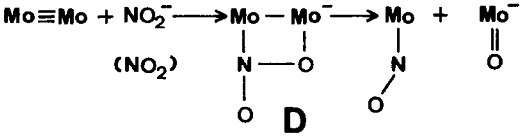
Dark reactions. The reaction of **2a** which has a Mo≡Mo triple bond, with excess NO_2^- and NO_3^- inMeOH-THF at 25 °C in the dark gave **3a** (23 and 14% yields based on the stoichiometry of eq. 7, respectively), the molybdenum oxide species, and **1a** which might be formed by the subsequent reaction of the liberated CO with **2a** (eq. 7 and 8). In the reaction of **2b** with the anions, **3b** and the molybdenum oxide species **5b** and **6b** were also obtained (NO_2^-: 62, 27 and 36% yields, NO_3^-: 18, 34 and 10% yields, respectively; the yield of **3b** is based on the analogous stoichiometry of eq. 7 whereas those of **5b** and **6b** are based on the molar ratio of the oxo complex to starting dimer **2b** used for the reaction), but **1b**, the corresponding dimer of **1a**, was not obtained. No carbon dioxide gas was detected in these reaction systems.



$$\underset{\mathbf{2a}}{\left[Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_4\right]} + \underset{(NO_3^-)}{NO_2^-} \longrightarrow \mathbf{3a} + \underset{\mathbf{A}}{\left[(\eta^5\text{-}C_5H_5)Mo{=}O\right]^-} + 2CO \qquad (7)$$

$$\mathbf{2a} + 2CO \longrightarrow \mathbf{1a} \qquad (8)$$

It is well known that molybdenum complexes reduce NO_3^- to NO_2 under anhydrous conditions [13]. In the present case, the reactions of **2a** and **2b** with NO_3^- may also give NO_2 in the initial step, which reacts directly with the Mo≡Mo bond to form the NO complexes without disproportionation, which would give NO_3^- and NO_2^-. Although the evolution of NO_2 was not examined in the reaction systems, **2a** and **2b** react readily with NO_2 gas to give **3a** and **3b**, respectively, under the same conditions. A possible mechanism for the formation of the nitrosyl complex from the dark reactions of **2a** or **2b** with NO_2^- and NO_2 is shown in eq. 9.



(9)

The anion NO_2^- and NO_2 have to interact with the Mo atoms before the oxygen atom is transferred onto one of them. Nitrite groups are known to bridge two metal atoms through N and O atoms as in intermediate (**D**) [14]. The reactions of **2a** with ethyl nitrite and t-butyl nitrite also gave the nitrosyl complex **3a** in low yields, but the yield was better in the former reaction. Steric hindrance by the alkyl groups in the nitrites appears to affect the stability of the intermediate **D** and the yield of **3a**.

The results presented here suggest that the Mo–Mo single bond is selective towards one-electron reduction ($NO_2^- \rightarrow NO$) in the photochemical reaction, whereas the Mo≡Mo triple bond is effective in both the one-electron reduction ($NO_2^- \rightarrow NO$) and the three-electron reduction ($NO_3^- \rightarrow NO$) in the dark reaction. The three-electron reduction is thought to involve a two-electron reduction of the NO_2 produced by the preceding one-electron reduction of NO_3^-.

Experimental

Air-sensitive materials were handled under nitrogen or argon, unless otherwise stated. Solvents were redistilled under argon and other reagents were purged with nitrogen before use. The starting materials, **1a** [15], **1b** [16], **2a** [17], **2b** [16], and **4a** [7] were prepared by literature methods. $Mo(CO)_6$ was obtained from Aldrich Chemical Company Inc. and used as received. NO_2 gas was purchased from Seitetsu Kagaku and used without further purification. Sodium nitrite and sodium nitrate were recrystallized from ethanol and water, respectively. Electronic spectra were recorded on a Hitachi 330 spectrophotometer. Infrared spectra were obtained by using a JASCO DS-701G (4000–200 cm^{-1}) spectrophotometer. ^1H NMR spectra were taken with a JEOL JNM MH-100 spectrometer. Molecular weights were measured in dichloromethane at 25 °C by vapor pressure osmometry with an instrument manufactured by Knauer (Berlin, West Germany). All photochemical reactions were carried out under argon in Schlenk flasks or tubes. A high-pressure mercury lamp (Ushio UM-103B, 100 W) and a xenon-lamp (Ushio DSB-501A, 500W) equipped with interference filters of 391 or 507 nm (Nippon Shinku Kogaku) for wavelength selection were used as the light source for the photochemical synthesis and for studies of quantum yields, respectively.

Quantum yield measurements were made with the Hitachi 330 spectrophotometer using matched quartz 1.00 cm cells fitted on with rubber septum caps. The disappearance of **1a** was monitored at 385 nm. The xenon-lamp intensity was measured by ferrioxalate actinometry; lamp intensities at 391 and 507 nm were 1.98×10^{-7} and 4.62×10^{-7} einstein/min, respectively [18].

Photochemical reaction of 1a with $NaNO_2$

A solution of **1a** (0.2065 g, 4.213×10^{-4} mol) and $NaNO_2$ (0.0956 g, 1.39×10^{-3} mol) in THF-MeOH (1 : 1, 20 cm^3) was photolyzed with sunlight (or with a pressure mercury lamp in a water-cooled pyrex jacket at 15 °C) for 40 h (or 15 min when the lamp was used) with stirring. The color of the solution changed from red-violet to yellow-orange. The solvent was evaporated to dryness to leave a yellow-orange solid which was extracted with benzene (20 $cm^3 \times 2$). A pale-yellow residue after the extraction with benzene was applied to the IR assay and found to be a mixture of **5a** and several unidentified oxo complexes. The yellow-orange extract was evaporated to give a yellow crystalline solid, **3a**. The solid was purified by sublimation at 50–60 °C/0.005 Torr onto a water-cooled probe for 3 d (yield, 0.0937 g, 90.0%; Anal. Found: C, 33.41; H, 2.12; N, 5.59. $C_7H_5MoNO_3$ calc: C, 34.03; H, 2.04; N, 5.67%) or by rapid column chromatography on deactivated neutral alumina (2 g) with n-pentane as eluent (50 cm^3).

Both the photochemical reaction of **1b** (0.3156 g, 5.006×10^{-4} mol) with $NaNO_2$ (0.1107 g, 1.604×10^{-3} mol), and the isolation of products were carried out by methods similar to those above. The reaction mixture was separated on a Florisil column with ether as eluent into **3b**, **5b** and **6b**. The results of the elemental analyses of these products were as follows: **3b** (yield as crude product, 0.0762 g, 48.0%; Anal. Found: C, 45.23; H, 4.54; N, 4.33%. $C_{12}H_{15}MoNO_3$ calc: C, 45.44; H, 4.77; N, 4.42%), **5b** (yield, 0.114 g, 42.0%; Anal. Found: C, 44.33; H, 5.55. $C_{20}H_{30}Mo_2O_5$ calc: C, 44.29; H, 5.58%), **6b** (yield, 0.0975 g, 37.0%; Anal. Found: C, 45.68; H, 5.70%. $C_{20}H_{30}Mo_2O_4$ calc: C, 45.64; H, 5.75%).

An evacuated gas cell (Ø 4 cm × 10 cm) was filled with the gas phase from the reaction system of **1a** or **1b** and $NaNO_2$ for IR assay.

*Reaction of **2a** with $NaNO_2$ in the dark*

Compound **2a** (0.1740 g, 4.008×10^{-4} mol) and $NaNO_2$ (0.0600 g, 8.696×10^{-4} mol) were placed in a two-necked flask (50 cm^3) wrapped with aluminum foil and dissolved in MeOH-THF (1 : 2 by volume, 30 cm^3). After stirring the solution for 4 d at 25 °C, the reddish-purple solid which separated (**1a**) was filtered off (yield, 0.0834 g, 42.5%). The filtrate was dried and the residue was extracted with C_6H_6 (10 cm^3). After extraction the pale-yellow residue was found by IR spectroscopy to be a mixture of **5a** and several unidentified oxo complexes. The extract was again dried under vacuum to give **3a** (yield, 0.0228 g, 23.0%). The gases evolved during the reaction were also examined by IR measurement (as above).

Similarly, reactions of **2a** with $NaNO_3$ (**3a**; yield, 14%), ethyl nitrite (**3a**; yield, 12%), or t-butyl nitrite (**3a**: yield, 3%) in the dark were carried out as described above. Reaction of **2b** (0.3305 g, 5.754×10^{-4} mol) with $NaNO_2$ (0.1191 g, 1.726×10^{-3} mol) or $NaNO_3$ (0.1467 g, 1.726×10^{-3} mol) in the dark was also carried out in MeOH-THF (1 : 2 by volume, 30 cm^3) at 25 °C. After stirring the solution for 4 d, the solvent was evaporated to dryness to leave a yellow-orange solid. The solid was dissolved in the minimum amount of CH_2Cl_2 and chromatographed on a column of Florisil. Compound **3b** (NO_2^-: 0.1132 g, 62% yield, NO_3^-; 0.0329 g, 18% yield) was obtained from first band, **6b** (NO_2^-: 0.1090 g, 36% yield, NO_3^-: 0.0303 g, 10% yield) from second, and **5b** (NO_2^-: 0.0843 g, 27% yield, NO_3^-: 0.1061 g, 34% yield) from third by using ether as eluent.

*Reaction of **4a** with $NaNO_2$ in the dark*

Compound **4a** (0.2153 g, 7.68×10^{-4} mol) and $NaNO_2$ (0.0530 g, 7.681×10^{-4} mol) were placed in a two-necked flask (50 cm^3) and dissolved in CH_3OH (30 cm^3). After stirring of the solution for 24 h at 25 °C, the solvent was evaporated to leave a brown solid which was extracted three times with n-pentane (10 $cm^3 \times 3$). The extract was then dried to give a yellow crystalline solid of **3a** (yield, 0.0455 g, 24.0%). The gas phase was checked by IR spectroscopy.

Acknowledgements

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