

INTRAMOLECULAR ALKENE-OXIRANE CYCLOADDITIONS. SYNTHESIS AND STRUCTURE

OF 5-OXAPENTACYCLO[7.3.0.0^{3,7}.0^{4,12}.0^{6,10}]DODECANE-2,8-DIONE

Alan P. Marchand* and G. Madhusudhan Reddy

Department of Chemistry, University of North Texas
Denton, Texas 76203-5068

William H. Watson* and Ram Kashyap

Department of Chemistry, Texas Christian University
Fort Worth, Texas 76129

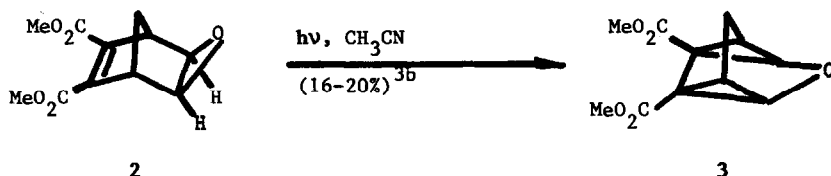
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Abstract: MCPBA promoted epoxidation of the C(9)-C(10) double bond in *endo*-tricyclo[6.2.1.0^{2,7}]undeca-4,9-diene-3,6-dione (**4**) followed by intramolecular [2 + 2] photocyclization of the resulting *exo* epoxide (**5**) afforded the title compound, **1**, in 16% overall yield. The structure of symmetrically hydrated **1** (i.e., **1a**) was determined by single crystal X-ray crystallographic methods. Proton and carbon-13 NMR spectral assignments are given for epoxide **5**.

Synthesis of 1. The synthesis of the title compound, **1**, was investigated as part of a continuing program that is concerned with the synthesis and chemistry of novel polycyclic cage compounds.¹ In the present study, **1** was synthesized by employing an intramolecular alkene-oxirane [2 + 2] photocyclization process² analogous to that reported previously by Prinzbach and coworkers.³ This interesting reaction provides a potentially general method for synthesizing novel heteropolycyclic oxa-cage systems.

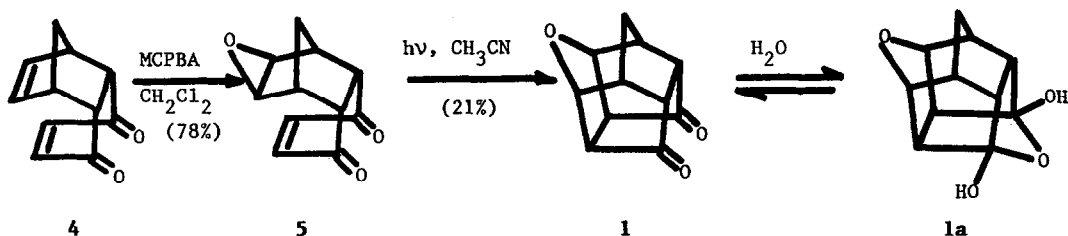
Although several formal intramolecular [$\sigma_2 + \pi_2$] cycloadditions of alkenes to cyclopropanes have been reported,^{3a} less is known about the corresponding reaction between alkenes and oxiranes. It has been suggested that reactions of this type are strongly geometry-dependent.³ One example of intramolecular alkene-oxirane [2 + 2] photocyclization is provided by the results of an earlier study by Prinzbach and coworkers.^{3b} They reported that irradiation of an acetonitrile solution of tricyclic epoxide **2** with a Hanovia 450 W high pressure mercury lamp (Vycor filter) at -20 °C afforded the corresponding tetracyclic ether, **3**, in 16-20% yield (Scheme 1).

Scheme 1



Our route for synthesizing **1** is outlined in Scheme 2. The starting material, **4**, can be prepared in large quantities via Diels-Alder cycloaddition of cyclopentadiene to *p*-benzoquinone.⁴ Selective epoxidation of the isolated C=C double bond in **4** was carried out by reacting **4** with *m*-chloroperbenzoic acid (MCPBA), thereby affording **5** in 78% yield. The fact that MCPBA epoxidation occurred exclusively at the exo face of the isolated C=C double bond in **4** was established via analysis of proton and carbon-13 NMR spectra of the epoxidation product, **5** (*vide infra*).

Scheme 2



Photolysis of a solution of **5** in HPLC grade acetonitrile⁵ was performed at 30 °C under argon by using a Hanovia 450 W medium pressure mercury lamp (Vycor filter). Irradiation was stopped after 2 h, and the reaction mixture was concentrated in vacuo. The corresponding cage diketone, **1**, was isolated from the residue in 21% yield. Its structure was established via ¹H and ¹³C NMR spectral analysis, which indicated (i) the disappearance of lowfield absorptions due to vinylic protons or carbons that are present in **5** but absent in **1** and (ii) the presence of only six carbon resonances in the proton noise-decoupled ¹³C NMR spectrum of **1**, a result that is consistent with the existence of a twofold symmetry element in **1**. Unequivocal evidence for the structure of **1** was obtained via X-ray crystallographic analysis of the corresponding symmetrical hydrate, **1a**.

X-ray Structure of 1a. A structure drawing of **1a** is shown in Figure 1, below.

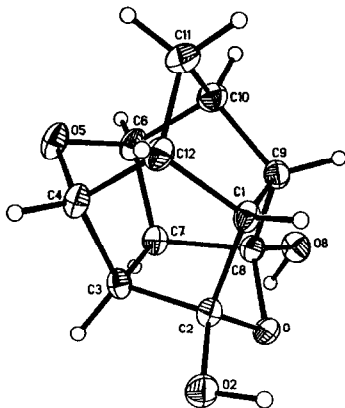
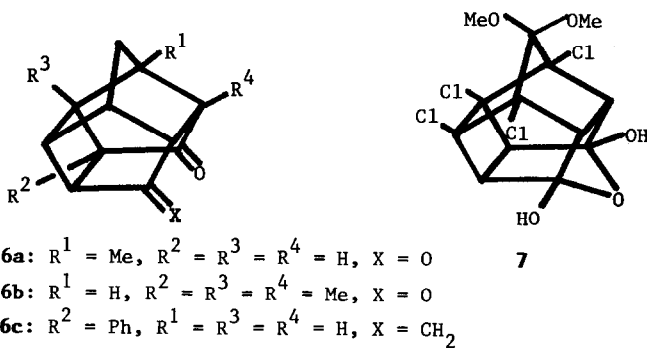


Figure 1. Structure drawing of **1a**.

It is instructive to compare selected bond lengths in **1a** with the corresponding bond lengths in substituted pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-diones (PCUD-8,11-diones) **6a**,⁶ **6b**,⁷ and **6c**⁸ and in a hydrated substituted PCUD-8,11-dione, **7** (Scheme 3).⁹

Scheme 3



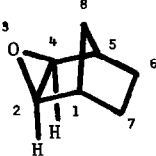
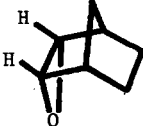
In **6a-6c**, the C(1)-C(7) and C(9)-C(10) bond lengths average 1.590 (5) Å and 1.589 (1) Å, respectively. The fact that these two C-C bonds in **6a-6c** are unusually long can be attributed to a through-bond interaction between the two flanking pi-systems.¹⁰ Hydration of the carbonyl groups in PCUD-8,11-diones leads to loss of the pi-system with concomitant shortening of these carbon-carbon σ -bonds [e.g., 1.534 (3) Å and 1.545 (3) Å for the C(1)-C(7) and C(9)-C(10) bonds in **7**].⁹ For comparison, The corresponding bond distances in hydrated diketone **1a** are 1.549 (2) Å and 1.556 (2) Å for the C(3)-C(7) and C(1)-C(9) bonds, respectively (see Figure 1 for atom numbering system).

The two hydroxyl groups in **1a** form intermolecular hydrogen bonds. H(O2) interacts with the O(5') ether oxygen in an adjacent molecule such that O(2) \cdots O(5') = 2.739 (2) Å, H(O2) \cdots O(5') = 1.78 (2) Å, and the O-H \cdots O bond angle O(2)-H(O2) \cdots O(5') = 174.5 (8)^o. In addition, H(O8) interacts with the bridging hydrate oxygen, O', of an adjacent molecule such that O(8) \cdots O = 2.781 (2) Å and the O-H \cdots O bond angle O(8)-H(O8) \cdots O' = 164.7 (8)^o.

Analysis of the ¹H and ¹³C NMR spectra of 5. Carbon-13 spectra of two model systems, exo-2,3- and endo-2,3-epoxynorbornanes (i.e., isomeric 3-oxatricyclo[3.2.1.0^{2,4}]oct-6-enes **8a** and **8b**) have been reported previously (Table I).¹¹ Of particular significance is the large reported difference in chemical shift between the bridging methylene carbon atom, i.e., C(8), in these two isomers (note that C(8) in **8a** resonates ca. 24 ppm upfield of the corresponding carbon atom in **8b**). Since the corresponding carbon atom in **5**, i.e., C(12), resonates at δ 25.04, we conclude that formation of **5** must have occurred via epoxidation of **4** from the *exo* face of its C(9)-C(10) double bond.

The two-dimensional ¹H-¹H COSY NMR spectrum of **5** reveals that only the signal centered at δ 3.12 is correlated with as many as four other proton signals (i.e., those at δ 0.82, 1.44, 2.97, and 3.07). We therefore assign the signal at δ 3.12 to the bridgehead protons, H(1) and H(8).

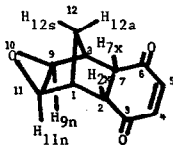
Table 1. Carbon-13 NMR spectral assignments in **8a** and **8b**.¹¹

		
	8a	8b
C(1), C(5)	δ 36.8	δ 37.6
C(2), C(4)	51.2	62.0
C(6), C(7)	25.2	25.6
C(8)	26.3	50.6

The signal centered at δ 2.97 is correlated strongly with the signal at δ 0.82 [H(12a)].¹² We anticipate that this results from W-coupling ($^4J_{\text{HH}}$) between H(9n)/H(11n) and H(12a).¹³ This suggestion receives support from the results of a nuclear magnetic resonance double resonance experiment. Double irradiation of the signal centered at δ 2.97 results in collapse of the signal at δ 0.82 to a tripled AB pattern. On this basis, the signal at δ 2.97 can be assigned to H(9n) and H(11n).

The remaining signal, a doubled doublet at δ 3.07 ($J = 2.7$ Hz, $J' = 1.5$ Hz), must arise from H(2x) and H(7x). Examination of the COSY spectrum suggests that these protons are correlated strongly with the bridgehead protons, H(1) and H(8), respectively and weakly with H(12s). Long-range $^4J_{\text{HH}}$ couplings in norbornanes and/or norbornenes that are analogous to the $^4J_{\text{H}(2x)\text{H}(12s)}$ coupling that we observe in **5** are, to our knowledge, unprecedented.^{13b}

Corresponding ^{13}C assignments were made via analysis of the two-dimensional ^1H - ^{13}C heteronuclear correlated (HETCOR) NMR spectrum of **5**. Proton and ^{13}C NMR spectral assignments in **5** thereby obtained are summarized in Table II.

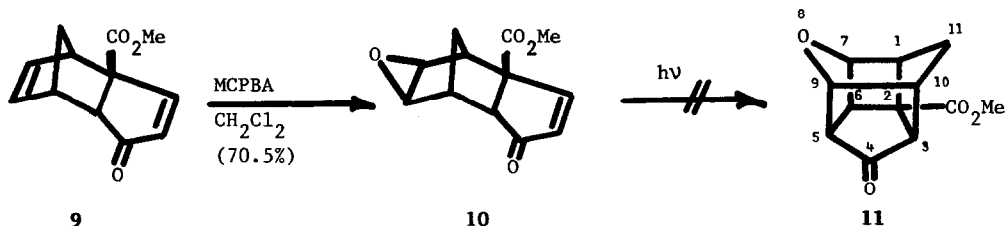
Table II. NMR spectral assignments in **5**.

proton chemical shift assignments in 5			carbon-13 chemical shift assignments in 5		
proton	δ , ppm	remarks	carbon atom	δ , ppm	remarks
1(8)	3.12	COSY: crosspeaks with 2x, 11n, 12a, 12s	1(8)	41.99 (d)	HETCOR: crosspeak with H(1)
2x(7x)	3.07	dd, $J = 2.7$ Hz, $J' = 1.5$ Hz	2(7)	48.68 (d)	HETCOR: crosspeak with H(2)
4(5)	6.69	sharp singlet	3(6)	198.33 (s)	carbonyl carbon atom
9n(11n)	2.97	COSY: crosspeak with H(12a)	4(5)	141.97 (d)	vinyl carbon atom
12s	1.44	tripled AB, $J_{AB} = 10.8$ Hz, $J_{1,12s} = 2.0$ Hz	9(11)	48.26 (d)	HETCOR: crosspeak with H(9)
12a	0.82	AB, $J_{AB} = 10.8$ Hz	12	25.04 (t)	HETCOR: crosspeaks with H(12a) and H(12s)

Synthesis of **10 and Attempted Photocyclization of **10** to **11**.** An attempt was made to utilize the readily available tricyclic ketoester, **9**, as starting material to synthesize the corresponding 2-substituted-8-oxapentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane, **11** (Scheme 4). Thus, MCPBA oxidation of **9**¹⁴ afforded the corresponding epoxide, **10**, in 70.5% yield. The bridging methylene carbon atom in **10** resonates at δ 28.64. Hence, by analogy to the ¹³C chemical shifts of the corresponding methylene carbon atom in **5** and in **8a**, we conclude that formation of **10** must have occurred via epoxidation of **9** from the exo face of the isolated alkene double bond.

Several attempts were made to promote intramolecular photocyclization of **10** to **11** (see Experimental Section). However, for reasons that are not apparent to us, none of these attempts proved to be successful.

Scheme 4



Experimental Section

Melting points are uncorrected. High-resolution mass spectra were obtained by personnel at the Midwest Center for Mass Spectrometry, Department of Chemistry, University of Nebraska-Lincoln, Lincoln, NE 68588. Compound **4** was synthesized via Diels-Alder cycloaddition of cyclopentadiene (freshly cracked from the corresponding dimer) to *p*-benzoquinone; the reaction was performed in methanol at $-78\text{ }^{\circ}\text{C}$.^{4c}

Epoxidation of 4. A solution of **4** (870 mg, 5.0 mmol) in methylene chloride (30 mL) was cooled externally to $0\text{ }^{\circ}\text{C}$. To this solution was added with stirring *m*-chloroperbenzoic acid (1.12 g, 6.5 mmol). The cold bath was removed, and the reaction mixture was allowed to warm slowly to ambient temperature and then stirred for 3 days. The reaction mixture was washed sequentially with 5% aqueous sodium bicarbonate solution (3 x 30 mL) and water (2 x 20 mL). The organic layer was dried (anhydrous sodium sulfate) and filtered, and the filtrate was concentrated in vacuo. The residue was triturated with diethyl ether (15 mL), and the solid residue was isolated by suction filtration. The residue was washed with ether (20 mL) and then air-dried, thereby affording the corresponding monoepoxide, **5** (740 mg, 78%) as a pale yellow microcrystalline solid: mp $140\text{--}142\text{ }^{\circ}\text{C}$; IR (KBr) 2982 (m), 1652 (s), 1380 (m), 1290 (s), 1279 (s), 1181 (m), 868 cm^{-1} (s); ^1H NMR (CDCl_3) δ 0.82 (AB, $\underline{J}_{\text{AB}} = 10.8\text{ Hz}$, 1 H), 1.44 (tripled AB, $\underline{J}_{\text{AB}} = 10.8\text{ Hz}$, $\underline{J}' = 2.0\text{ Hz}$, 1 H), 2.97 (m, 2 H), 3.07 (dd, $\underline{J} = 2.7\text{ Hz}$, $\underline{J}' = 1.5\text{ Hz}$, 2 H), 3.12 (m, 2 H), 6.69 (s, 2 H); ^{13}C NMR (CDCl_3) δ 25.04 (t), 41.99 (d), 48.26 (d), 48.68 (d), 141.97 (d), 198.33 (s). Anal. Calcd for $\text{C}_{11}\text{H}_{10}\text{O}_3$: \underline{M}_r 190.0630. Found (high-resolution mass spectroscopy): \underline{M}_r 190.0626.

Photocyclization of 5. A solution of **5** (200 mg, 1.05 mmol) in HPLC grade acetonitrile (250 mL) under argon was irradiated with a 450 watt medium pressure Hg lamp (Vycor filter) at $30\text{ }^{\circ}\text{C}$ for 2 h. The reaction mixture was concentrated in vacuo, and the residue was purified via column chromatography on silica gel by using 1:1 ethyl acetate-hexane mixed solvent as eluent. Compound **1** (41.6 mg, 21%) was thereby obtained as a colorless microcrystalline solid: mp $270\text{--}280\text{ }^{\circ}\text{C}$ (dec.); IR (KBr) 3009 (s), 1740 (s), 1183 (s), 1124 (s), 885 cm^{-1} (s); ^1H NMR (CDCl_3) δ 1.50 (tripled AB, $\underline{J}_{\text{AB}} = 11.9\text{ Hz}$, $\underline{J}' = 4.0\text{ Hz}$, 1 H), 2.37 (AB, $\underline{J}_{\text{AB}} = 11.9\text{ Hz}$, 1 H), 2.63 (d, $\underline{J} = 2.6\text{ Hz}$, 2 H), 2.73 (d, $\underline{J} = 2.8\text{ Hz}$, 2 H), 2.93-3.06 (m, 2 H), 4.81-4.97 (m, 2 H); ^{13}C NMR (CDCl_3) δ 27.35 (t), 43.37 (d), 54.51 (d), 54.77 (d), 77.60 (d), 207.76 (s).

This sample of **1** was further recrystallized from acetone in an effort to obtain a pure sample for elemental microanalysis. During this recrystallization procedure, **1** gradually was converted into the corresponding ketone hydrate, **1a**, the structure of which was established by X-ray crystallographic methods (*vide infra*). Analytically pure **1a**, mp 301–302 °C (dec.), was thereby obtained; IR (KBr) 3275 (s), 2968 (s), 1378 (s), 1346 (s), 1324 (s), 1306 (s), 1257 (m), 1206 (s), 1185 (s), 1162 (s), 1077 (s), 1026 (s), 933 (s), 889 (s) 747 cm⁻¹ (s); ¹H NMR (acetone-d₆) δ 1.28 (AB, J_{AB} = 10.2 Hz, 1 H), 1.86 (AB, J_{AB} = 10.2 Hz, 1 H), 2.34–3.18 (m, 6 H), 4.29–4.72 (m, 2 H), 5.81 (s, 2 H); ¹³C NMR (acetone-d₆) δ 30.91 (t), 44.02 (d), 58.55 (d), 60.36 (d), 81.05 (d), 108.90 (s). Anal. Calcd for C₁₁H₁₂O₄: C, 63.45; H, 5.81. Found: C, 63.37; H, 5.75.

X-ray Crystal Structure of 1a.¹⁵ A crystal of dimensions 0.35 x 0.33 x 0.30 mm was mounted on a Nicolet R3m/μ update of a P2/1 diffractometer. Data were collected by using the ω-scan mode (3 < 2θ < 55°), a variable scan rate, and graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The space group is triclinic, $\bar{P}1$, with a = 6.541 (1), b = 6.639 (1), c = 10.808 (1) Å, α = 85.43 (1), β = 87.99 (1), γ = 73.56 (1)°, v = 448.1 (1) Å³, Z = 2, and D(calcd) = 1.541 g·cm⁻³. The structure was solved via direct methods. All atoms were located in difference maps and were refined by using least-squares techniques with H-atom thermal parameters isotropic. Of the 2249 independent reflections collected, 1982 had intensities greater than 3σ(I), and refinement with 185 parameters led to a final R of 0.0411 (R_{all} = 0.0462), wR = 0.0568 with S = 1.690, (Δ/σ)_{max} = 0.017 with largest peaks in the final difference map of 0.36 and -0.27 eÅ⁻³. The function minimized was Σw(|F_o - |F_c||² with w = [σ²(F_o) + 0.00067 F_o²]⁻¹. A ψ-scan absorption correction was applied (transmission factors 0.889 to 0.873), and a secondary isotropic extinction correction was applied. All computer programs were supplied by Nicolet for a Desktop 30 Microclipse and Nova 4/C configuration. Atomic scattering factors and anomalous dispersion corrections were taken from the International Tables for X-ray Crystallography.¹⁶

Epoxidation of 9. A solution of **9** (204 mg, 1.0 mmol) in methylene chloride (15 mL) was cooled externally to 0 °C. To this solution was added with stirring m-chloroperbenzoic acid (224 mg, 1.3 mmol). The cold bath was removed, and the reaction mixture was allowed to warm slowly to ambient temperature and then stirred for 2 days. The reaction mixture

was washed sequentially with 5% aqueous sodium bicarbonate solution (3 x 15 mL) and water (2 x 20 mL). The organic layer was dried (anhydrous sodium sulfate) and filtered, and the filtrate was concentrated in vacuo. The residue was purified via column chromatography on silica gel by using 15% ethyl acetate-hexane mixed solvent as eluent. The corresponding exo monoepoxide, **10** (155 mg, 70.5%) was thereby obtained as a colorless microcrystalline solid: mp 101-102 °C; IR (film) 2960 (m), 1684 (s), 1436 (m), 1253 (s), 1231 (s), 1035 (m), 844 cm⁻¹ (m); ¹H NMR (CDCl₃) δ 1.33 (AB, J_{AB} = 10.5 Hz, 1 H), 1.61 (AB, J_{AB} = 10.5 Hz, 1 H), 2.84-3.27 (m, 5 H), 3.75 (s, 3 H), 6.11 (AB, J_{AB} = 5.7 Hz, 1 H), 7.48 (AB, J_{AB} = 5.7 Hz, 1 H); ¹³C NMR (CDCl₃) δ 28.64 (t), 39.07 (d), 42.17 (d), 48.60 (d), 49.06 (d), 52.90 (q), 54.78 (d), 65.07 (s), 136.53 (d), 159.96 (d), 172.28 (s), 206.99 (s); Anal. Calcd for C₁₂H₁₂O₄: C, 65.45; H, 5.49. Found: C, 65.60; H, 5.46.

Attempted Photolysis of 10. A solution of **10** (200 mg, 0.90 mmol) in HPLC grade acetonitrile (250 mL) under argon was irradiated with a 450 watt medium pressure Hg lamp (Vycor filter). After 1.5 h, thin layer chromatographic analysis of the reaction mixture indicated the absence of starting material. The reaction mixture then was concentrated in vacuo. An attempt was made to purify the residue via column chromatography on silica gel by using a 10-100% ethyl acetate-hexane mixed solvent gradient elution scheme. However, no identifiable product could be isolated from the eluate thereby obtained.

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References and Footnotes

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2. The term "[2 + 2] alkene-oxirane photocyclization" is used here in a formal sense. It is not known whether such photochemical processes are, in fact, concerted [$\sigma_s^2 + \pi_s^2$] photocycloadditions, or if instead they occur, e.g., via photochemical disrotatory electrocyclic ring opening of the oxirane to form an intermediate carbonyl ylide which subsequently undergoes (thermal) intramolecular [$\pi_s^4 + \pi_s^2$] cycloaddition to an alkene C=C moiety.
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5. The yield of the desired photocyclization product is affected dramatically by the purity of the acetonitrile solvent. None of the desired cycloadduct could be obtained when a solution of **5** in HPLC grade acetonitrile was irradiated with Pyrex filtered light. Photolysis of **5**, when performed in benzene, acetone, or ethyl acetate solution, afforded only polymeric material.
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12. Signals in the proton NMR spectrum of model system **8a** at δ 0.67 and 1.33 (AB pattern) have been assigned previously to H(8a) and H(8s), respectively. δ 1.1^a Accordingly, we assign the signals at δ 0.82 and 1.44 in **5** to H(12a) and H(12s), respectively.
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15. Tables of atomic coordinates and isotropic thermal parameters, bond lengths, bond angles, anisotropic thermal parameters, H-atom coordinates, and isotopic thermal parameters (5 pages) for **1a** are available upon request from the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CV2 1EW, U. K. Requests should be accompanied by the full literature citation for this article.
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