SUPPORTING INFORMATION

Ozonolysis of Bicyclic 1,2-Dioxines: Initial Scope and Mechanistic Insights

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Crystallographic Details

Crystallographic data for 22

Crystallographic data for 33a

NMR Spectra

1H and 13C NMR for compounds 8e, 13, 7, 16, 17, 11a, 18a, 18b,
22, 25, 26, 27, 33a and 34.

1H and 13C VT NMR (ambient and +50 °C) for compounds 18d and 21.
CRYSTALLOGRAPHIC DETAILS

Details of crystal structure determination of C$_{17}$H$_{16}$O$_3$. (22): Crystal data for C$_{17}$H$_{16}$O$_3$: $M = 268.30$, $T = 98(2)$ K, triclinic, $P$-1, $a = 5.6262(15)$, $b = 10.040(3)$, $c = 12.165(3)$ Å, $\alpha = 95.510(4)$, $\beta = 90.569(6)$, $\gamma = 100.990(7)^\circ$, $V = 671.2(3)$ Å$^3$, $Z = 2$, $D_x = 1.328$, $F(000) = 284$, $\mu = 0.090$ mm$^{-1}$, no. of unique data (Rigaku AFC12k/SATURN724 CCD using Mo K$\alpha$ radiation so that $\theta_{\text{max}} = 26.5^\circ$) = 2740, no. of parameters = 184, $R$ (2417 data with $I \geq 2\sigma(I)) = 0.053$, $wR$ (all data) = 0.145. The structure was solved by direct-methods (SHELXS-97) and refined (anisotropic displacement parameters, H atoms in the riding model approximation, and a weighting scheme $w = 1/[\sigma^2(F_o)^2 + (0.071P)^2+0.33P]$ where $P = (F_o^2 + 2F_c^2)/3$) with SHELXL-97 on $F^2$. CCDC deposition number: 743807.

Figure S1: Molecular structure and crystallographic numbering scheme for 22. Diagram drawn with 50% displacement ellipsoids.
Details of crystal structure determination of \(\text{C}_{11}\text{H}_{18}\text{O}_5\). (33a): Crystal data for \(\text{C}_{11}\text{H}_{18}\text{O}_5\): \(M = 230.25\), \(T = 98(2)\) K, monoclinic, \(P2_1/c\), \(a = 10.686(3)\), \(b = 7.0118(19)\), \(c = 16.046(5)\) Å, \(\beta = 107.784(3)\)º, \(V = 1144.9(6)\) Å\(^3\), \(Z = 4\), \(D_x = 1.336\), \(F(000) = 496\), \(\mu = 0.105\) mm\(^{-1}\), no. of unique data (Rigaku AFC12κ/SATURN724 CCD using Mo Kα radiation so that \(\theta_{\text{max}} = 26.5º\)) = 2352, no. of parameters = 146, \(R\) (2203 data with \(I \geq 2\sigma(I)\)) = 0.044, \(wR\) (all data) = 0.115. The structure was solved by direct-methods (SHELXS-97) and refined (anisotropic displacement parameters, H atoms in the riding model approximation, and a weighting scheme \(w = 1/[\sigma^2(F_o^2) + (0.053P)^2+0.748P]\) where \(P = (F_o^2 + 2F_c^2)/3\) with SHELXL-97 on \(F^2\). CCDC deposition number: 743808.

Figure S2: Molecular structure and crystallographic numbering scheme for 33a. Diagram drawn with 50% displacement ellipsoids.
\(^1\)H AND \(^{13}\)C NMR SPECTRA

\(8e (\text{CDCl}_3, 300\text{MHz})\)

\[
\text{MeO}_2\text{C} - \overset{\text{O}}{\text{O}} - \text{CO}_2\text{Me}
\]
$8e$ (CDCl$_3$, 300MHz)

MeO$_2$C$\xrightarrow{\text{O}}$ CO$_2$Me
$^{13}$ (CDCl$_3$, 300MHz)
7 (CDCl₃, 300MHz)
16 (CDCl₃, 300MHz)
$^{17}$(CDCl$_3$, 300MHz)
17 (CDCl₃, 300MHz)
11a (CDCl₃, 300MHz)
11a (CDCl₃, 300MHz)
$18a$ (CDCl$_3$, 600MHz)
$^{18a}$ (CDCl$_3$, 600 MHz)
$^{18b} (CDCl_3, 300MHz)$
18b (CD$_3$Cl, 300MHz)
22 (CDCl₃, 300MHz)
$^{22}$ (CDCl$_3$, 300MHz)
26 (CDCl₃, 600MHz)
27 (CDCl₃, 200MHz)
33a (CDCl₃, 300MHz)
33a (CDCl₃, 300MHz)
34 (CDCl₃, 300MHz)
VT $^1$H AND $^{13}$C NMR SPECTRA

18d (Ambient Temperature, CDCl$_3$, 300MHz)
18d (Ambient Temperature, CDCl$_3$, 300MHz)
18d (+50 °C, CDCl₃, 300 MHz)
$^{18d}$ ($+50 \, ^\circ\text{C}, \text{CDCl}_3, 300\text{MHz}$)
21 (Ambient Temperature, CDCl₃, 300MHz)
$21$ (Ambient Temperature, CDCl$_3$, 300MHz)
$^5^1\ (\pm 50^\circ \text{C}, \text{CDCl}_3, 300\text{MHz})$