Acid promoted synthesis of cyclic 1,3-dione fused symmetrical 2,8-dioxabicyclo [3.3.1]nonanes

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Supplementary Data

Contents:
Crystallographic data for the compounds 4a & 3b
1H-NMR and 13C-NMR spectrums for all new compounds

Crystallographic data for 4a and 3b compounds

Data collection and Structure solution: X-ray data for two compounds (4a and 3b) were collected at room temperature using the Bruker Smart Apex CCD diffractometer with graphite monochromated MoKα radiation (λ=0.71073Å) with ω-scan method. Preliminary lattice parameters and orientation matrices were obtained from four sets of frames. Unit cell dimensions were determined using 3351 reflections for 4a and 8574 reflections for 3b crystal data sets. Integration and scaling of intensity data were accomplished using SAINT program. The structures were solved by Direct Methods using SHELXS97 and refinement was carried out by full-matrix least-squares technique using SHELXL97. Anisotropic displacement parameters were included for all non-hydrogen atoms. All H atoms were positioned geometrically and treated as riding on their parent C atoms, with C-H distances of 0.93–0.97 Å, and with Uiso(H) = 1.2Ueq (C) or 1.5Ueq for methyl atoms.

2. Sheldrick, G. M. SHELXS97 and SHELXL97, Programs for crystal structure solution and refinement; University of Göttingen: Germany, 1997.
Crystal data for 4a: C$_{25}$H$_{28}$O$_4$, $M = 392.47$, colorless block, 0.43 x 0.38 x 0.29 mm$^3$, triclinic, space group $P1$ (No. 1), $a = 5.7508(11)$, $b = 10.2102(19)$, $c = 10.3282(19)$ Å, $\alpha = 60.929(2)$, $\beta = 80.487$ (3), $\gamma = 76.750(3)^{\circ}$, $V = 514.89(17)$ Å$^3$, $Z = 1$, $D_c = 1.266$ g/cm$^3$, $F_{000} = 210$, CCD area detector, MoK$\alpha$ radiation, $\lambda = 0.71073$ Å, $T = 293(2)$K, $2\theta_{\text{max}} = 50.0^{\circ}$, 4878 reflections collected, 3541 unique ($R_{\text{int}} = 0.0182$), Final $GooF = 1.032$, $R1 = 0.0392$, $wR2 = 0.1066$, $R$ indices based on 3384 reflections with $I > 2\sigma(I)$ (refinement on $F^2$), 266 parameters, 3 restraints, $\mu = 0.084$ mm$^{-1}$. CCDC 1056215 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Figure caption: ORTEP diagram of 4a with the atom-numbering. Displacement thermal ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.
Crystal data for 3b: C_{25}H_{29}ClO_{5}, M = 444.93, colorless needle, 0.40 x 0.16 x 0.07 mm³, monoclinic, space group I2/a (No. 15), a = 19.0793(11), b = 11.3954(7), c = 22.0551(13) Å, β = 98.7630(10)°, V = 4739.2(5) Å³, Z = 8, D_e = 1.247 g/cm³, F_{000} = 1888, CCD area detector, MoKα radiation, λ = 0.71073 Å, T = 293(2)K, 2θ_{max} = 50.0°, 20502 reflections collected, 4183 unique (R_{int} = 0.0190), Final Goof = 1.024, RL = 0.0431, wR2 = 0.1191, R indices based on 3429 reflections with I > 2σ(I) (refinement on F²), 292 parameters, 1 restraint, µ = 0.193 mm⁻¹. **CCDC 1056216** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

Figure caption: ORTEP diagram of 3b with the atom-numbering. Displacement thermal ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.
$^1$H-NMR and $^{13}$C-NMR spectrums:

$^1$H NMR (500 MHz) spectrum of $4a$ in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of $4a$ in CDCl$_3$. 
$^1$H NMR (300 MHz) spectrum of 4b in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4b in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4c in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4c in CDCl$_3$. 
$^1$H NMR (300 MHz) spectrum of 4d in CDCl$_3$.

$^{13}$C NMR (75 MHz) spectrum of 4d in CDCl$_3$. 
\( ^1H \) NMR (300 MHz) spectrum of 4e in CDCl\(_3\).

\( ^{13}C \) NMR (125 MHz) spectrum of 4e in CDCl\(_3\).
$^1$H NMR (500 MHz) spectrum of 4f in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4f in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of $4g$ in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of $4g$ in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4h in CDCl$_3$.

$^{13}$C NMR (100 MHz) spectrum of 4h in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4i in CDCl₃.

$^{13}$C NMR (75 MHz) spectrum of 4i in CDCl₃.
$^1$H NMR (500 MHz) spectrum of 4j in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4j in CDCl$_3$. 
$^1$H NMR (300 MHz) spectrum of 4k in CDCl$_3$. 

$^{13}$C NMR (125 MHz) spectrum of 4k in CDCl$_3$. 

S 14
$^1$H NMR (300 MHz) spectrum of 4I in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4I in CDCl$_3$. 
$^1$H NMR (300 MHz) spectrum of 4m in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4m in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4n in CDCl$_3$. 

$^{13}$C NMR (125 MHz) spectrum of 4n in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4o in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4o in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4p in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4p in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4q in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4q in CDCl$_3$.
\[ ^1H \text{ NMR (500 MHz) spectrum of 4r in CDCl}_3. \]

\[ ^{13}C \text{ NMR (125 MHz) spectrum of 4r in CDCl}_3. \]
$^1$H NMR (500 MHz) spectrum of 4s in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4s in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 4t in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 4t in CDCl$_3$. 

S 23
$^1$H NMR (300 MHz) spectrum of 3a in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 3a in CDCl$_3$. 
$^1$H NMR (300 MHz) spectrum of 3b in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 3b in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 5a in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 5a in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 5b in CDCl$_3$. 

$^{13}$C NMR (125 MHz) spectrum of 5b in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of 5c in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 5c in CDCl$_3$. 
$^1$H NMR (300 MHz) spectrum of 5d in CDCl$_3$.

$^{13}$C NMR (75 MHz) spectrum of 5d in CDCl$_3$. 
$^1$H NMR (400 MHz) spectrum of 5e in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 5e in CDCl$_3$. 
$^1$H NMR (400 MHz) spectrum of 5f in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 5f in CDCl$_3$. 
$\textbf{1H NMR (300 MHz) spectrum of 5g in CDCl}_3.$

$\textbf{13C NMR (100 MHz) spectrum of 5g in CDCl}_3.$
$^1$H NMR (300 MHz) spectrum of $5h$ in CDCl$_3$.

$^{13}$C NMR (100 MHz) spectrum of $5h$ in CDCl$_3$. 
$^1$H NMR (500 MHz) spectrum of Si in CDCl$_3$.

$^{13}$C NMR (100 MHz) spectrum of Si in CDCl$_3$. 
$^1$H NMR (300 MHz) spectrum of 5j in CDCl$_3$.

$^{13}$C NMR (125 MHz) spectrum of 5j in CDCl$_3$. 