Synthesis and Structural Characterization of a Fused Bispyrone and Preparation of 
the First Metal Bispyrylium Complexes

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SUPPORTING INFORMATION

General techniques:

All reactions were performed with freshly distilled solvents and recently purchased materials. Infrared spectra were recorded using a thin film supported on KBr discs or dispersed in a KBr pellet. $^1$H and $^{13}$C NMR spectra were recorded in Fourier transform mode at the field strength specified on a 400 MHz Bruker Avance spectrometer. Spectra were obtained in DMSO, CDCl$_3$ or CD$_3$OD solutions in 5 mm diameter tubes, and chemical shifts in ppm are quoted relative to the residual signals of the solvent used DMSO ($\delta$H 2.51 ppm or $\delta$C 39.9) chloroform ($\delta$H 7.26 ppm, or $\delta$C 77.0 ppm) methanol ($\delta$H 3.31, 4.87 $\delta$C 49.1). Multiplicities in the $^1$H NMR spectra are described as: s = singlet, d = doublet, t = triplet, q= quartet, m = multiplet, br = broad; coupling constants are reported in Hz. High Resolution Mass spectra are reported with ion mass/charge (m/z) ratios as values in atomic mass units.

Table 1: Synthesis and properties of metal bispyrylium perchlorates formed from 4.

<table>
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<tr>
<th>Reagent Product</th>
<th>Product</th>
<th>$\lambda_{max}$ (nm) ($\epsilon$ 10$^3$)</th>
<th>Color</th>
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<tbody>
<tr>
<td>Mg(ClO$_4$)$_2$</td>
<td>7 (M=Mg)</td>
<td>303 (19.2)</td>
<td>Colorless</td>
</tr>
<tr>
<td>Ni(ClO$_4$)$_2$•6MeCN</td>
<td>7 (M=Ni)</td>
<td>304 (4.48)</td>
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<tr>
<td>Fe(ClO$_4$)$_2$•6MeCN</td>
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<td>301 (5.99)</td>
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<tr>
<td>Li(ClO$_4$)$_2$</td>
<td>8 (M=Li)</td>
<td>[a]</td>
<td>Colorless</td>
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<tr>
<td>Cu(ClO$_4$)$_2$•6H$_2$O</td>
<td>9 (M=Cu)</td>
<td>318 (5.54)</td>
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</tr>
<tr>
<td>Co(ClO$_4$)$_2$•6H$_2$O</td>
<td>9 (M=Co)</td>
<td>298 (30.0)</td>
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<tr>
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[a] not determined.
Fig. 3. Reaction of Fused bispyrone 4 with divalent metal perchlorates.

Fig. 8. Reaction of fused bispyrone 4 with hydrated divalent metal perchlorates.

X-ray Crystallography. X-ray diffraction intensities for 4, 6, 7 (M=Mg), 7 (M=Ni), 8, 9 (M=Cu), 9 (M=Co), 9 (M=Ni), 9 (M=Zn), 9 (M=Fe) and 10 were collected at 173(2) K on a Bruker Apex CCD diffractometer using MoKα radiation λ= 0.71073 Å.¹ Space groups were determined based on systematic absences or intensity statistics. Absorption corrections in all cases were applied by SADABS.² Structures were solved by direct methods and Fourier techniques and refined on \( F^2 \) using full matrix least-squares procedures. All non-H atoms were refined with anisotropic thermal parameters. H atoms in all structures mainly were found from the F-map and refined with isotropic thermal parameters except some cases indicated below. In the crystal structures of 4, and 7 (M=Mg) besides the main molecule there are solvent water molecules. The –OH group and the carbonyl O atom in 6 are disordered over two positions related by a mirror plane. These O atoms were refined as sharing the same position and the H atom in the disordered –OH group was refined with occupation factor \( \mu = 0.5 \). Other H atoms in 6 were found on the residual density and refined with isotropic thermal parameters. \( \text{ClO}_4 \) anions in 7 (M=Mg), 7 (M= Ni), 9 (M = Fe) and 10 are disordered over two positions and were refined with restrictions; the average Cl-O bond length was used as the target for corresponding distances. H atoms in 7 (M=Mg) were found from the F-map and refined with isotropic thermal parameters except those in the terminal Me-groups. H atoms in a solvent water molecule in 7 (M=Mg) involved in H-bonds were found from the F-map and refined with isotropic thermal parameters. The thermal parameters for these H atoms are relatively high and indicate that H atoms in the solvent molecule seem to be disordered, but we did not find a good solution for this disorder. H atoms in the CH₃OH molecules coordinated to the Li-atoms in 8 were refined in calculated positions. H atoms in an insolated solvent methanol molecule in 8 were not found and not taken into consideration in the refinement. All calculations were performed by the Bruker SHELXTL (v. 6.10) package.³
Crystallographic Data for 4•(H$_2$O): C$_{10}$H$_{10}$O$_5$, M = 210.18, 0.27 x 0.25 x 0.06 mm, T = 173(2) K, monoclinic, space group $P2_1/c$, $a = 7.0711(2)$ Å, $b = 25.5598(9)$ Å, $c = 11.1908(4)$ Å, $\beta = 104.521(1)^\circ$, $V = 1957.97(11)$ Å$^3$, $Z = 8$, $D_c = 1.426$ Mg/m$^3$, $\mu = 0.116$ mm$^{-1}$, $F(000) = 880$, $2\theta_{\text{max}} = 54.00^\circ$, 19210 reflections, 4263 independent reflections [R$_{\text{int}} = 0.0315$], R$_1 = 0.0450$, wR$_2 = 0.1021$ and GOF = 1.025 for 4263 reflections (351 parameters) with $I > 2\sigma(I)$, R$_1 = 0.0783$, wR$_2 = 0.1219$ and GOF = 1.025 for all reflections, max/min residual electron density +0.184/-0.193 eÅ$^3$.

Crystallographic Data for 6: C$_{10}$H$_{9}$ClO$_8$, M = 292.62, 0.38 x 0.11 x 0.10 mm, T = 173(2) K, orthorhombic, space group $Pnma$, $a = 10.190(4)$ Å, $b = 12.552(4)$ Å, $c = 8.820(3)$ Å, $V = 1128.1(7)$ Å$^3$, $Z = 4$, $D_c = 1.723$ Mg/m$^3$, $\mu = 0.375$ mm$^{-1}$, $F(000) = 600$, $2\theta_{\text{max}} = 54.00^\circ$, 12297 reflections, 1291 independent reflections [R$_{\text{int}} = 0.0324$], R$_1 = 0.0363$, wR$_2 = 0.0974$ and GOF = 1.086 for 1291 reflections (114 parameters) with $I > 2\sigma(I)$, R$_1 = 0.0411$, wR$_2 = 0.1022$ and GOF = 1.086 for all reflections, max/min residual electron density +0.473/-0.221 eÅ$^3$.

Crystallographic Data for 7 (M=Mg)•H$_2$O: C$_{22}$H$_{26}$Cl$_2$MgO$_{19}$, M = 689.64, 0.23 x 0.19 x 0.12 mm, T = 173(2) K, monoclinic, space group $P2_1/c$, $a = 15.050(3)$ Å, $b = 18.021(3)$ Å, $c = 11.008(2)$ Å, $\beta = 103.023(3)^\circ$, $V = 2908.7(10)$ Å$^3$, $Z = 4$, $Z' = 2$, $D_c = 1.575$ Mg/m$^3$, $\mu = 0.331$ mm$^{-1}$, $F(000) = 1424$, $2\theta_{\text{max}} = 50.00^\circ$, 27023 reflections, 5118 independent reflections [R$_{\text{int}} = 0.0436$], R$_1 = 0.0579$, wR$_2 = 0.1501$ and GOF = 1.065 for 5118 reflections (500 parameters) with $I > 2\sigma(I)$, R$_1 = 0.0794$, wR$_2 = 0.1665$ and GOF = 1.174 for all reflections, max/min residual electron density +0.697/-0.431 eÅ$^3$.

Crystallographic Data for 7 (M=Ni): C$_{22}$H$_{24}$Cl$_2$NiO$_{18}$, M = 706.02, 0.31 x 0.20 x 0.14 mm, T = 173(2) K, monoclinic, space group $P2_1/n$, $a = 7.412(2)$ Å, $b = 14.508(3)$ Å, $c = 13.476(4)$ Å, $\alpha = 95.849(5)^\circ$, $\beta = 87.173(2)^\circ$, $\gamma = 76.390(2)^\circ$, $V = 1441.6(7)$ Å$^3$, $Z = 2$, $Z' = 0.5$, $D_c = 1.627$ Mg/m$^3$, $\mu = 0.940$ mm$^{-1}$, $F(000) = 724$, $2\theta_{\text{max}} = 50.00^\circ$, 9624 reflections, 3129 independent reflections [R$_{\text{int}} = 0.0436$], R$_1 = 0.0579$, wR$_2 = 0.1501$ and GOF = 1.065 for 5118 reflections (500 parameters) with $I > 2\sigma(I)$, R$_1 = 0.0794$, wR$_2 = 0.1665$ and GOF = 1.174 for all reflections, max/min residual electron density +0.697/-0.431 eÅ$^3$.

Crystallographic Data for 8: C$_{32.50}$H$_{32}$Cl$_2$Li$_2$O$_{22.50}$, M = 867.36, 0.38 x 0.27 x 0.16 mm, T = 173(2) K, triclinic, space group $P-1$, $a = 13.6532(18)$ Å, $b = 15.954(2)$ Å, $c = 18.398(2)$ Å, $\alpha = 79.690(2)^\circ$, $\beta = 87.173(2)^\circ$, $\gamma = 76.390(2)^\circ$, $V = 3832.0(9)$ Å$^3$, $Z = 4$, $Z' = 2$, $D_c = 1.503$ Mg/m$^3$, $\mu = 0.259$ mm$^{-1}$, $F(000) = 1788$, $2\theta_{\text{max}} = 50.00^\circ$, 37382 reflections, 13455 independent reflections [R$_{\text{int}} = 0.0402$], R$_1 = 0.0556$, wR$_2 = 0.1523$ and GOF = 1.033 for 13455 reflections (1219 parameters) with $I > 2\sigma(I)$, R$_1 = 0.0898$, wR$_2 = 0.1691$ and GOF = 1.033 for all reflections, max/min residual electron density +0.545/-0.289 eÅ$^3$.

Crystallographic Data for 9 (M=Cu): C$_{20}$H$_{20}$Cl$_2$CuO$_{18}$, M = 682.80, 0.32 x 0.27 x 0.16 mm, T = 173(2) K, monoclinic, space group $P2_1/n$, $a = 9.9449(10)$ Å, $b = 12.6414(13)$ Å, $c = 10.4593(11)$ Å, $\beta = 95.359(2)^\circ$, $V = 1309.2(2)$ Å$^3$, $Z = 2$, $Z' = 0.5$, $D_c = 1.732$
Crystallographic Data for 9 (M=Co): $\text{C}_{20}\text{H}_{20}\text{Cl}_{2}\text{CoO}_{18}$, M = 678.19, 0.19 x 0.16 x 0.12 mm, T = 173(2) K, monoclinic, space group $P2_1/n$, $a = 10.033(6)$ Å, $b = 12.586(8)$ Å, $c = 10.332(6)$ Å, $\beta = 95.922(9)^\circ$, $V = 1297.7(14)$ Å$^3$, $Z = 2$, $Z^\prime = 0.5$, $D_c = 1.736$ Mg/m$^3$, $\mu = 0.955$ mm$^{-1}$, $F(000) = 690$, $2\theta_{\text{max}} = 50.00^\circ$, 11277 reflections, 2279 independent reflections [R int = 0.0425], $R_1 = 0.0509$, wR2 = 0.1218 and GOF = 1.055 for 2279 reflections (227 parameters) with I>2σ(I), $R_1 = 0.0666$, wR2 = 0.1320 and GOF = 1.055 for all reflections, max/min residual electron density +0.924/-0.438 eÅ$^3$.

Crystallographic Data for 9 (M=Ni): $\text{C}_{20}\text{H}_{20}\text{Cl}_{2}\text{NiO}_{18}$, M = 677.97, 0.19 x 0.12 x 0.09 mm, T = 173(2) K, monoclinic, space group $P2_1/n$, $a = 9.952(3)$ Å, $b = 12.636(3)$ Å, $c = 10.439(3)$ Å, $\beta = 95.348(4)^\circ$, $V = 1307.0(6)$ Å$^3$, $Z = 2$, $Z^\prime = 0.5$, $D_c = 1.723$ Mg/m$^3$, $\mu = 1.033$ mm$^{-1}$, $F(000) = 692$, $2\theta_{\text{max}} = 50.00^\circ$, 12556 reflections, 2852 independent reflections [R int = 0.0295], $R_1 = 0.0295$, wR2 = 0.0723 and GOF = 1.066 for 2852 reflections (227 parameters) with I>2σ(I), $R_1 = 0.0368$, wR2 = 0.0775 and GOF = 1.066 for all reflections, max/min residual electron density +0.327/-0.412 eÅ$^3$.

Crystallographic Data for 9 (M=Zn): $\text{C}_{20}\text{H}_{20}\text{Cl}_{2}\text{ZnO}_{18}$, M = 684.63, 0.32 x 0.11 x 0.08 mm, T = 173(2) K, monoclinic, space group $P2_1/n$, $a = 9.973(3)$ Å, $b = 12.636(3)$ Å, $c = 10.432(3)$ Å, $\beta = 95.500(4)^\circ$, $V = 1308.5(6)$ Å$^3$, $Z = 2$, $Z^\prime = 0.5$, $D_c = 1.738$ Mg/m$^3$, $\mu = 1.229$ mm$^{-1}$, $F(000) = 696$, $2\theta_{\text{max}} = 54.00^\circ$, 14755 reflections, 2855 independent reflections [R int = 0.0382], $R_1 = 0.0387$, wR2 = 0.0944 and GOF = 1.082 for 2855 reflections (227 parameters) with I>2σ(I), $R_1 = 0.0530$, wR2 = 0.1044 and GOF = 1.082 for all reflections, max/min residual electron density +0.680/-0.305 eÅ$^3$.

Crystallographic Data for 9 (M=Fe): $\text{C}_{20}\text{H}_{20}\text{Cl}_{2}\text{FeO}_{18}$, M = 675.11, 0.39 x 0.22 x 0.10 mm, T = 173(2) K, monoclinic, space group $P2_1/n$, $a = 10.113(2)$ Å, $b = 12.627(3)$ Å, $c = 10.339(2)$ Å, $\beta = 95.402(4)^\circ$, $V = 1314.5(5)$ Å$^3$, $Z = 2$, $Z^\prime = 0.5$, $D_c = 1.706$ Mg/m$^3$, $\mu = 0.863$ mm$^{-1}$, $F(000) = 688$, $2\theta_{\text{max}} = 54.00^\circ$, 8603 reflections, 2848 independent reflections [R int = 0.0191], $R_1 = 0.0348$, wR2 = 0.0880 and GOF = 1.039 for 2848 reflections (237 parameters) with I>2σ(I), $R_1 = 0.0411$, wR2 = 0.0932 and GOF = 1.039 for all reflections, max/min residual electron density +0.334/-0.361 eÅ$^3$.

Crystallographic Data for 10: $\text{C}_{20}\text{H}_{20}\text{Cl}_{2}\text{BaO}_{18}$, M = 756.60, 0.39 x 0.24 x 0.08 mm, T = 173(2) K, triclinic, space group P-1, $a = 10.1152(15)$ Å, $b = 10.2248(15)$ Å, $c = 12.9429(19)$ Å, $\alpha = 92.527(2)^\circ$, $\beta = 99.751(2)^\circ$, $\gamma = 97.818(2)^\circ$, $V = 1303.9(3)$ Å$^3$, $Z = 2$, $D_c = 1.927$ Mg/m$^3$, $\mu = 1.815$ mm$^{-1}$, $F(000) = 748$, $2\theta_{\text{max}} = 54.00^\circ$, 14739 reflections, 5654 independent reflections [R int = 0.0153], $R_1 = 0.0205$, wR2 = 0.0635 and GOF = 1.186 for 5654 reflections (479 parameters) with I>2σ(I), $R_1 = 0.0209$, wR2 = 0.0639
and GOF = 1.190 for all reflections, max/min residual electron density +0.543/-0.453 eÅ³.

References:

1  Bruker (2000). SMART and SAINT, Bruker AXS Inc., Madison, Wisconsin, USA

SHELXTL-6.10 "Program for Structure Solution, Refinement and Presentation"
BRUKER AXS Inc., 5465 East Cheryl Parkway, Madison, WI 53711-5373 USA

General Procedure:

Bis-γ-pyrene 4 was prepared by dissolving 2.00 g (11.8 mmol) of dehydroacetic acid (3) in 5 mL of acetic anhydride and slowly adding the solution to 1.5 mL 60% perchloric acid in 20 mL of acetic anhydride at 0°C. The solution was stirred for 12 hrs at 0°C during which an orange precipitate formed. The solid was collected via filtration, washed with hexanes and identified as the perchlorate salt 6 (1.78 g, 6.09 mmol, 51% yield). The filtrate upon standing for one week, deposited additional crystalline 6, increasing the yield to 65%. NMR – [ppm δH (DMSO) 2.29 s (6H), 6.22 s (1H), 6.58 s (1H) 8.90 br s (1H), δC 19.4, 20.4, 98.9, 104.9, 115.3, 157.4, 164.6, 167.7, 169.7, 174.0] IR cm⁻¹ KBr 1733.8, 1652.7, 1646.4, 1622.8, 1575.6, 1559.1, 1539.7, 1114.3, 1086.4, 623.2. mp > 250°C.

A portion of 6 (500 mg, 1.71 mmol) was carefully neutralized with NaHCO₃ (143 mg 1.71 mmol) in H₂O (7 mL) and the mixture was extracted with CHCl₃, dried over Na₂SO₄ and evaporated to afford solid bis-γ-pyrene 4 as a colorless solid. The solid was recrystallized from either benzene or CHCl₃-cyclohexane to afford 4 as colorless needles. NMR – [ppm δH (CDCl₃) 2.33 s (3H), 6.18 s (1H), δC 19.10, 115.42, 160.55, 175.76]. IR (KBr) cm⁻¹ 1749.8, 1698.1, 1684.4, 1653.0, 1646.8, 1635.5, 1558.4, 1437.2. HRMS – calcd 192.0423 found 192.0431. mp = 200 °C (decomp). λmax 300 nm.
Metal complexes 7 were prepared by dissolving 2 equivalents of 4 (i.e. 10 mg 0.05 mmol) in dry MeOH (300 µL), pouring the solution into a solution of 1 equivalent of the metal perchlorate salt (0.026 mmol) in dry MeOH (300 µL), and allowing the solution to slowly evaporate. This gave crystalline salts 7, 8, and 10 in quantitative yields.

Rearranged metal complexes 9 were prepared as described above, except that 1 equivalent of the hydrated metal perchlorates (0.026 mmol) was used.

**General and crystallographic data for complexes 4, 6, 7, 8, 9, and 10.**

**Bis-γ-pyrone perchlorate salt 6:**
NMR – [ppm δH (DMSO) 2.29 s (6H), 6.22 s (1H), 6.58 s (1H) 8.90 br s (1H), δC 19.4, 20.4, 98.9, 104.9, 115.3, 157.4, 164.6, 167.7, 169.7, 174.0] IR cm⁻¹ KBr 1733.8, 1652.7, 1646.4, 1622.8, 1575.6, 1559.1, 1539.7, 1114.3, 1086.4, 623.2. mp > 250°C.

Crystal Structure:
Table 1. Crystal data and structure refinement for 6.

Identification code 6
Empirical formula C10 H9 Cl O8
Formula weight 292.62
Temperature 173(2) K
Wavelength 0.71073 Å
Crystal system Orthorhombic
Space group Pnma
Unit cell dimensions
\[ a = 10.190(4) \text{ Å} \]
\[ b = 12.552(4) \text{ Å} \]
\[ c = 8.820(3) \text{ Å} \]
Volume 1128.1(7) Å³
Z 4
Density (calculated) 1.723 Mg/m³
Absorption coefficient 0.375 mm⁻¹
F(000) 600
Crystal size 0.38 x 0.11 x 0.10 mm³
Theta range for data collection 2.82 to 26.99°
Index ranges -13≤h≤13, -16≤k≤16, -11≤l≤11
Reflections collected 12297
Independent reflections 1291 [R(int) = 0.0324]
Completeness to theta = 26.99° 100.0 %
Absorption correction Semi-empirical from equivalents
Max. and min. transmission 0.9634 and 0.8705
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters 1291 / 0 / 114
Goodness-of-fit on F² 1.086
Final R indices [I>2σ(I)] R1 = 0.0363, wR2 = 0.0974
R indices (all data) R1 = 0.0411, wR2 = 0.1022
Largest diff. peak and hole 0.473 and -0.221 e.Å⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for 6. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

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Table 3. Bond lengths [Å] and angles [°] for 6.

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<td>O(3)-Cl(1)-O(5)</td>
<td>110.15(8)</td>
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</tbody>
</table>
O(4)-Cl(1)-O(5) 108.77(8)
O(5)#1-Cl(1)-O(5) 109.34(12)
C(1)-O(1)-C(5) 118.91(13)
C(3)-O(2)-H(1) 109(3)
O(1)-C(1)-O(1)#2 111.74(19)
O(1)-C(1)-C(2) 124.13(10)
O(1)#2-C(1)-C(2) 124.13(10)
C(1)-C(2)-C(3)#2 118.67(11)
C(1)-C(2)-C(3) 118.67(11)
C(3)#2-C(2)-C(3) 122.6(2)
O(2)-C(3)-C(4) 123.07(16)
O(2)-C(3)-C(2) 120.40(16)
C(4)-C(3)-C(2) 116.53(16)
C(5)-C(4)-C(3) 120.36(16)
C(5)-C(4)-H(2) 118.2(11)
C(3)-C(4)-H(2) 121.5(12)
H(3)-C(6)-H(4) 112.2(18)
C(5)-C(6)-H(3) 106.1(14)
C(5)-C(6)-H(4) 109.0(14)
H(3)-C(6)-H(5) 112.7(18)
H(4)-C(6)-H(5) 108.7(18)

Symmetry transformations used to generate equivalent atoms:
#1 x, -y+1/2, z  #2 x, -y+3/2, z

Table 4. Anisotropic displacement parameters (Å² x 10³) for 6. The anisotropic
displacement factor exponent takes the form: -2\pi^2 [ a^2 U_{11} + ... + 2 h k a^* b^* U_{12} ]

<table>
<thead>
<tr>
<th></th>
<th>U₁¹</th>
<th>U₂²</th>
<th>U₃³</th>
<th>U₂₃</th>
<th>U₁₃</th>
<th>U₁₂</th>
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<td>0</td>
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<tr>
<td>O(1)</td>
<td>23(1)</td>
<td>27(1)</td>
<td>32(1)</td>
<td>1(1)</td>
<td>0(1)</td>
<td>-1(1)</td>
</tr>
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</table>
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3)
for 4.

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
</tr>
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<tbody>
<tr>
<td>H(1)</td>
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<td>7070(30)</td>
<td>-600(40)</td>
<td>42(13)</td>
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<tr>
<td>H(2)</td>
<td>6730(19)</td>
<td>4861(16)</td>
<td>740(20)</td>
<td>34(5)</td>
</tr>
<tr>
<td>H(3)</td>
<td>5020(20)</td>
<td>4151(19)</td>
<td>2290(20)</td>
<td>43(6)</td>
</tr>
<tr>
<td>H(4)</td>
<td>4640(20)</td>
<td>4836(18)</td>
<td>3730(30)</td>
<td>52(6)</td>
</tr>
<tr>
<td>H(5)</td>
<td>3720(30)</td>
<td>4844(17)</td>
<td>2280(20)</td>
<td>51(6)</td>
</tr>
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</table>

Table 6. Torsion angles [°] for 6.

<p>| | |</p>
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<th></th>
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<td>-0.1(3)</td>
</tr>
<tr>
<td>O(1)-C(1)-C(2)-C(3)#2</td>
<td>179.15(18)</td>
</tr>
<tr>
<td>O(1)#2-C(1)-C(2)-C(3)#2</td>
<td>-0.3(3)</td>
</tr>
<tr>
<td>O(1)-C(1)-C(2)-C(3)</td>
<td>0.3(3)</td>
</tr>
<tr>
<td>O(1)#2-C(1)-C(2)-C(3)</td>
<td>-179.15(18)</td>
</tr>
<tr>
<td>C(1)-C(2)-C(3)-O(2)</td>
<td>179.93(18)</td>
</tr>
<tr>
<td>C(3)#2-C(2)-C(3)-O(2)</td>
<td>1.2(3)</td>
</tr>
<tr>
<td>C(1)-C(2)-C(3)-C(4)</td>
<td>-0.3(3)</td>
</tr>
<tr>
<td>C(3)#2-C(2)-C(3)-C(4)</td>
<td>-179.03(14)</td>
</tr>
</tbody>
</table>
O(2)-C(3)-C(4)-C(5) 179.77(16)
C(2)-C(3)-C(4)-C(5) 0.0(2)
C(3)-C(4)-C(5)-O(1) 0.3(2)
C(3)-C(4)-C(5)-C(6) 179.78(16)
C(1)-O(1)-C(5)-C(4) -0.2(2)
C(1)-O(1)-C(5)-C(6) -179.80(16)

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z   #2 x,-y+3/2,z

Table 7. Hydrogen bonds for 6 [Å and °].

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O(2)-H(1)...O(2)#2</td>
<td>0.81(4)</td>
<td>1.85(4)</td>
<td>2.583(3)</td>
<td>151(4)</td>
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</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z   #2 x,-y+3/2,z

Dimethyl-bis-γ-pyrone 4:

NMR – [ppm δH (CDCl3) 2.33 s (3H), 6.18 s (1H), δC 19.10, 115.42, 160.55, 175.76]. IR (KBr) cm⁻¹ 1749.8, 1698.1, 1684.4, 1653.0, 1646.8, 1635.5, 1558.4, 1437.2. HRMS – calcd 192.0423 found 192.0431. mp = 200 °C (decomp). λmax 300 nm.

Crystal Structure:
Table 1. Crystal data and structure refinement for 4.

Identification code 4
Empirical formula C10 H10 O5
Formula weight 210.18
Temperature  296 K
Wavelength  0.71073 Å
Crystal system  Monoclinic
Space group  P2(1)/c
Unit cell dimensions  
a = 7.0711(2) Å  \( \alpha = 90^\circ \)
b = 25.5598(9) Å  \( \beta = 104.5210(10)^\circ \)
c = 11.1908(4) Å  \( \gamma = 90^\circ \)
Volume  1957.97(11) Å³
Z  8
Density (calculated)  1.426 Mg/m³
Absorption coefficient  0.116 mm⁻¹
F(000)  880
Crystal size  0.27 x 0.25 x 0.06 mm³
Theta range for data collection  1.59 to 27.00°.
Index ranges  -9 <= h <= 9, -32 <= k <= 32, -14 <= l <= 14
Reflections collected  19210
Independent reflections  4263 [R(int) = 0.0315]
Completeness to theta = 27.00°  99.9%
Absorption correction  Semi-empirical from equivalents
Max. and min. transmission  0.9931 and 0.9694
Refinement method  Full-matrix least-squares on F²
Data / restraints / parameters  4263 / 0 / 351
Goodness-of-fit on F²  1.025
Final R indices [I>2sigma(I)]  R₁ = 0.0450, wR₂ = 0.1021
R indices (all data)  R₁ = 0.0783, wR₂ = 0.1219
Largest diff. peak and hole  0.184 and -0.193 e.Å⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for 4. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
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</thead>
<tbody>
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<td>2017(2)</td>
<td>3837(1)</td>
<td>9586(1)</td>
<td>56(1)</td>
</tr>
<tr>
<td>O(2)</td>
<td>1304(2)</td>
<td>3067(1)</td>
<td>8855(1)</td>
<td>50(1)</td>
</tr>
<tr>
<td>O(3)</td>
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<td>4589(1)</td>
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<td>O(4)</td>
<td>-1466(2)</td>
<td>3562(1)</td>
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<td>66(1)</td>
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Table 3. Bond lengths [Å] and angles [°] for 4.

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</tr>
<tr>
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<td>1.3321(19)</td>
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<td>O(2)-C(8)</td>
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<td>O(3)-C(4)</td>
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</tr>
<tr>
<td>Bond</td>
<td>Distance (Å)</td>
</tr>
<tr>
<td>----------------------</td>
<td>--------------</td>
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<td>C(3)-C(4)</td>
<td>1.448(3)</td>
</tr>
<tr>
<td>C(3)-H(3)</td>
<td>0.94(2)</td>
</tr>
<tr>
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<tr>
<td>C(5)-C(6)</td>
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<tr>
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<td>Distance (Å)</td>
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C(2)-C(10)-H(10B) 109.6(16)
H(10A)-C(10)-H(10B) 110(2)
C(2)-C(10)-H(10C) 110.6(14)
H(10A)-C(10)-H(10C) 111(2)
H(10B)-C(10)-H(10C) 106(2)
C(8)-C(11)-H(11A) 111.1(13)
C(8)-C(11)-H(11B) 111.1(13)
H(11A)-C(11)-H(11B) 107.6(19)
C(8)-C(11)-H(11C) 110.2(11)
H(11A)-C(11)-H(11C) 107.3(17)
H(11B)-C(11)-H(11C) 109.4(17)
C(1')-O(1')-C(2') 118.37(14)
C(1')-O(2')-C(8') 117.88(13)
O(1')-C(1')-O(2') 108.22(14)
O(1')-C(1')-C(5') 125.85(16)
O(2')-C(1')-C(5') 125.93(16)
C(3')-C(2')-O(1') 120.49(18)
C(3')-C(2')-C(10') 128.9(2)
O(1')-C(2')-C(10') 110.58(19)
C(2')-C(3')-C(4') 123.17(19)
C(2')-C(3')-H(3') 117.6(12)
C(4')-C(3')-H(3') 119.2(12)
O(3')-C(4')-C(3') 122.96(18)
O(3')-C(4')-C(5') 122.70(18)
C(3')-C(4')-C(5') 114.34(17)
C(1')-C(5')-C(6') 117.70(16)
C(1')-C(5')-C(4') 117.75(16)
C(6')-C(5')-C(4') 124.53(16)
O(4')-C(6')-C(7') 122.24(17)
O(4')-C(6')-C(5') 123.48(17)
C(7')-C(6')-C(5') 114.26(16)
C(8')-C(7')-C(6') 123.27(18)
C(8')-C(7')-H(7') 119.2(12)
C(6')-C(7')-H(7') 117.5(12)
C(7')-C(8')-O(2') 120.69(16)
C(7')-C(8')-C(11') 127.60(19)
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\[
\begin{align*}
O(2')-C(8')-C(11') & \quad 111.71(17) \\
C(2')-C(10')-H(10D) & \quad 109.8(19) \\
C(2')-C(10')-H(10E) & \quad 112.2(16) \\
H(10D)-C(10')-H(10E) & \quad 107(3) \\
C(2')-C(10')-H(10F) & \quad 108.8(16) \\
H(10E)-C(10')-H(10F) & \quad 113(2) \\
C(8')-C(11')-H(11D) & \quad 108.2(15) \\
C(8')-C(11')-H(11E) & \quad 110.2(15) \\
H(11D)-C(11')-H(11E) & \quad 111(2) \\
C(8')-C(11')-H(11F) & \quad 110.9(14) \\
H(11D)-C(11')-H(11F) & \quad 111(2) \\
H(11E)-C(11')-H(11F) & \quad 106(2) \\
H(15)-O(15)-H(25) & \quad 107(3) \\
H(15)-O(15)-H(25) & \quad 106(3)
\end{align*}
\]

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å² x 10³) for jw33. The anisotropic displacement factor exponent takes the form: -2π² [ h²a*²U₁₁ + ... + 2 h k a* b* U₁₂ ]

<table>
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<tr>
<th>Atom</th>
<th>U₁₁</th>
<th>U₂₂</th>
<th>U₃₃</th>
<th>U₁₃</th>
<th>U₂₃</th>
<th>U₁₂</th>
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<tbody>
<tr>
<td>O(1)</td>
<td>77(1)</td>
<td>36(1)</td>
<td>48(1)</td>
<td>0(1)</td>
<td>3(1)</td>
<td>-2(1)</td>
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<tr>
<td>O(2)</td>
<td>65(1)</td>
<td>34(1)</td>
<td>48(1)</td>
<td>2(1)</td>
<td>10(1)</td>
<td>0(1)</td>
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<tr>
<td>O(3)</td>
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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 4.
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Table 6. Torsion angles [°] for 4.

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C(6)-C(7)-C(8)-O(2) = -2.0(3)
C(6)-C(7)-C(8)-C(11) = 176.99(19)
C(1)-O(2)-C(8)-C(7) = 0.4(2)
C(1)-O(2)-C(8)-C(11) = -178.73(16)
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C(2')-O(1')-C(1')-C(5') = -0.5(3)
C(8')-O(2')-C(1')-C(5') = 178.46(13)
C(1')-O(1')-C(2')-C(3') = 1.3(3)
C(1')-O(1')-C(2')-C(10') = -178.50(19)
C(2')-C(3')-C(4')-O(3') = -0.6(3)
C(2')-C(3')-C(4')-C(5') = 179.1(2)
O(1')-C(1')-C(5')-C(6') = 177.46(15)
O(2')-C(1')-C(5')-C(6') = -2.4(3)
O(1')-C(1')-C(5')-C(4') = -0.8(3)
O(2')-C(1')-C(5')-C(4') = 179.37(16)
O(3')-C(4')-C(5')-C(1') = -178.28(18)
C(3')-C(4')-C(5')-C(1') = 1.4(2)
O(3')-C(4')-C(5')-C(6') = 3.6(3)
C(3')-C(4')-C(5')-C(6') = -176.78(16)
C(1')-C(5')-C(6')-O(4') = -173.08(17)
C(4')-C(5')-C(6')-O(4') = 5.1(3)
C(1')-C(5')-C(6')-C(7) = 5.5(2)
C(4')-C(5')-C(6')-C(7) = -176.31(16)
O(4')-C(6')-C(7')-C(8') = 173.27(19)
C(5')-C(6')-C(7')-C(8') = -5.4(3)
C(6')-C(7')-C(8')-O(2') = 1.6(3)
C(6')-C(7')-C(8')-C(11') = -178.6(2)
C(1')-O(2')-C(8')-C(7) = 2.1(2)
C(1')-O(2')-C(8')-C(11') = -177.66(18)

Symmetry transformations used to generate equivalent atoms:
Table 7. Hydrogen bonds for 4 [Å and °].

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<th>d(D...A)</th>
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Symmetry transformations used to generate equivalent atoms:
#1 x+1,y,z    #2 -x+1,-y+1,-z+1

Metal Bis-γ-pyrene Complex 7 (M=Mg): General Procedure followed with 10 mg (0.0520 mmol) bis-γ-pyrene 5 and 5.8 mg (0.026 mmol) of Mg(II)(ClO₄)₂ yielding 15.8 mg of white crystals (quant.). NMR [δH (DMSO) 2.28 s (3H), 6.21 s (1H), δC 18.86, 106.33, 114.87, 161.63, 167.88, 175.72]. IR (KBr) cm⁻¹ 1674.4, 1640.4, 1487.2, 1256.7, 1182.8, 1120.4, 1023.8. HRMS calcd 507.0181 found 507.0160. mp > 250.

Crystal Structure:
Table 1. Crystal data and structure refinement for 7 (M=Mg)•H₂O

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<tr>
<td>Formula weight</td>
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</table>
Temperature 173(2) K
Wavelength 0.71073 Å
Crystal system Monoclinic
Space group P2(1)/c
Unit cell dimensions 
\[\begin{align*}
a &= 15.050(3) \text{ Å} \\
b &= 18.021(3) \text{ Å} \\
c &= 11.008(2) \text{ Å}
\end{align*}\]
\[a= 90^\circ, \quad b= 103.023(3)^\circ, \quad g = 90^\circ.\]
Volume 2908.7(10) Å³
Z 4
Density (calculated) 1.575 Mg/m³
Absorption coefficient 0.331 mm⁻¹
F(000) 1424
Crystal size 0.23 x 0.19 x 0.12 mm³
Theta range for data collection 1.39 to 25.00°
Index ranges -17<=h<=17, -21<=k<=21, -13<=l<=13
Reflections collected 27023
Independent reflections 5118 [R(int) = 0.0436]
Completeness to theta = 25.00° 100.0 %
Absorption correction Semi-empirical from equivalents
Max. and min. transmission 0.9613 and 0.9277
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters 5118 / 18 / 500
Goodness-of-fit on F² 1.065
Final R indices [I>2sigma(I)] R1 = 0.0579, wR2 = 0.1501
R indices (all data) R1 = 0.0794, wR2 = 0.1665
Largest diff. peak and hole 0.697 and -0.431 e.Å⁻³
Table 2. Atomic coordinates (x $10^4$) and equivalent isotropic displacement parameters ($\AA^2 x 10^3$) for 7 (M= Mg). $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

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Table 3. Bond lengths [Å] and angles [°] for 7 (M=Mg).

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O(9A)-Cl(1)-O(8A) 104.6(8)
O(8)-Cl(1)-O(8A) 141.7(6)
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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 7 (M=Mg).

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<td>-177.4(3)</td>
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<td>2.2(5)</td>
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<td>177.4(3)</td>
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<td>0.6(5)</td>
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<td>-2.1(5)</td>
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C(4)-C(5)-C(6)-O(4) -179.0(3)
Mg(1)-O(2)-C(7)-C(8) 174.7(2)
Mg(1)-O(2)-C(7)-C(5) -5.6(5)
C(6)-C(5)-C(7)-O(2) -179.3(3)
C(4)-C(5)-C(7)-O(2) -2.7(6)
C(6)-C(5)-C(7)-C(8) 0.3(5)
C(4)-C(5)-C(7)-C(8) 176.9(3)
O(2)-C(7)-C(8)-C(9) -179.1(3)
C(5)-C(7)-C(8)-C(9) 1.2(5)
C(7)-C(8)-C(9)-O(2) -1.2(5)
C(7)-C(8)-C(9)-C(10) 178.5(4)
C(6)-O(4)-C(9)-C(8) -0.5(5)
C(6)-O(4)-C(9)-C(10) 179.8(3)
O(2')#2-Mg(1')-O(1')-C(4') 3.5(3)
O(2')#2-Mg(1')-O(1')-C(4') -176.5(3)
O(5')#2-Mg(1')-O(1')-C(4') -64(100)
O(5')#2-Mg(1')-O(1')-C(4') 92.4(3)
O(5')#2-Mg(1')-O(1')-C(4') -87.6(3)
O(2')#2-Mg(1')-O(2')-C(7') 75(80)
O(2')#2-Mg(1')-O(2')-C(7') 175.7(3)
O(1')#2-Mg(1')-O(2')-C(7') -4.3(3)
O(5')#2-Mg(1')-O(2')-C(7') -94.0(3)
O(5')#2-Mg(1')-O(2')-C(7') 86.0(3)
O(2')#2-Mg(1')-O(5')-C(11') 161.6(4)
O(2')#2-Mg(1')-O(5')-C(11') -18.4(4)
O(1')#2-Mg(1')-O(5')-C(11') -105.7(4)
O(1')-Mg(1')-O(5')-C(11') 74.3(4)
O(5')#2-Mg(1')-O(5')-C(11') -31(100)
C(6')-O(3')-C(2')-C(3') -3.0(5)
C(6')-O(3')-C(2')-C(1') 177.2(3)
O(3')-C(2')-C(3')-C(4') 1.3(5)
C(1')-C(2')-C(3')-C(4') -179.0(4)
Mg(1')-O(1')-C(4')-C(3') 178.0(2)
Mg(1')-O(1')-C(4')-C(5') -2.2(5)
C(2')-C(3')-C(4')-O(1') -178.4(3)
C(2')-C(3')-C(4')-C(5') 1.8(5)
O(1')-C(4')-C(5')-C(6') 177.1(3)
C(3')-C(4')-C(5')-C(6') -3.2(5)
O(1')-C(4')-C(5')-C(7') 0.1(5)
C(3')-C(4')-C(5')-C(7') 179.8(3)
C(9')-O(4')-C(6')-O(3') -179.2(3)
C(9')-O(4')-C(6')-C(5') 0.3(5)
C(2')-O(3')-C(6')-O(4') -179.0(3)
C(2')-O(3')-C(6')-C(5') 1.5(5)
C(4')-C(5')-C(6')-O(4') -177.8(3)
C(7')-C(5')-C(6')-O(4') -0.5(5)
C(4')-C(5')-C(6')-O(3') 1.7(5)
C(7')-C(5')-C(6')-O(3') 178.9(3)
Mg(1')-O(2')-C(7')-C(8') -175.0(2)
Mg(1')-O(2')-C(7')-C(5') 3.9(5)
C(6')-C(5')-C(7')-O(2') -177.9(3)
C(4')-C(5')-C(7')-O(2') -0.9(5)
C(6')-C(5')-C(7')-C(8') 1.0(5)
C(4')-C(5')-C(7')-C(8') 178.0(3)
O(2')-C(7')-C(8')-C(9') 177.5(3)
C(5')-C(7')-C(8')-C(9') -1.4(5)
C(7')-C(8')-C(9')-O(2') 1.2(5)
C(7')-C(8')-C(9')-C(10') -177.4(4)
C(6')-O(4')-C(9')-C(8') -0.6(5)
C(6')-O(4')-C(9')-C(10') 178.3(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1    #2 -x,-y+2,-z+2

Table 7. Hydrogen bonds for 7 (M= Mg) [Å and °].

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O(5')-H(5'O)...O(9A)#3</td>
<td>0.93(2)</td>
<td>1.83(3)</td>
<td>2.728(12)</td>
<td>161(4)</td>
</tr>
<tr>
<td>O(5')-H(5'O)...O(8)#3</td>
<td>0.93(2)</td>
<td>1.98(3)</td>
<td>2.832(8)</td>
<td>151(4)</td>
</tr>
<tr>
<td>O(5)-H(5O)...O(1S)</td>
<td>0.95(2)</td>
<td>1.73(3)</td>
<td>2.656(4)</td>
<td>165(6)</td>
</tr>
<tr>
<td>O(1S)-H(1S)...O(7A)#4</td>
<td>1.00(2)</td>
<td>2.02(7)</td>
<td>2.872(19)</td>
<td>142(8)</td>
</tr>
</tbody>
</table>
Metal Bis-γ-pyrones complex 7 (M=Ni)

General Procedure was followed using 10 mg (0.0520 mmole) of bis-γ-pyrones 5 and 13.06 mg (0.026 mmole) of Ni(ClO₄)₂•6CH₃CN to yield 23 mg of light blue crystals (quant.). IR (KBr) cm⁻¹ 1670.8, 1625.5, 1486.8, 1256.9, 1183.4, 1120.0, 1024.5. HRMS calc 540.9684 found 540.9677. mp > 250°C.

Crystal structure:

<table>
<thead>
<tr>
<th>Distance</th>
<th>Bond Angle</th>
<th>Bond Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>O(1S)-H(1S)...O(8)</td>
<td>1.00(2)</td>
<td>2.12(5)</td>
</tr>
<tr>
<td>O(1S)-H(1S)...O(6)</td>
<td>1.00(2)</td>
<td>2.67(5)</td>
</tr>
<tr>
<td>O(1S)-H(2S)...O(6)</td>
<td>0.98(2)</td>
<td>2.07(3)</td>
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<tr>
<td>O(1S)-H(2S)...O(8A)</td>
<td>0.98(2)</td>
<td>2.55(7)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1  #2 -x,-y+2,-z+2  #3 x,y,z+1  
#4 x,-y+3/2,z+1/2

Table 1. Crystal data and structure refinement for 7 (M=Ni)

<table>
<thead>
<tr>
<th>Identification code</th>
<th>7 (M= Ni)</th>
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<tbody>
<tr>
<td>Empirical formula</td>
<td>C22 H24 Cl2 Ni O18</td>
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<tr>
<td>Property</td>
<td>Value</td>
</tr>
<tr>
<td>--------------------------------</td>
<td>------------------------</td>
</tr>
<tr>
<td>Formula weight</td>
<td>706.02</td>
</tr>
<tr>
<td>Temperature</td>
<td>173(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P2(1)/n</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td></td>
</tr>
<tr>
<td>a</td>
<td>7.412(2) Å</td>
</tr>
<tr>
<td>a°</td>
<td>90°</td>
</tr>
<tr>
<td>b</td>
<td>14.508(4) Å</td>
</tr>
<tr>
<td>b°</td>
<td>95.849(5)°</td>
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<tr>
<td>c</td>
<td>13.476(4) Å</td>
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<tr>
<td>g°</td>
<td>90°</td>
</tr>
<tr>
<td>Volume</td>
<td>1441.6(7) Å³</td>
</tr>
<tr>
<td>Z</td>
<td>2</td>
</tr>
<tr>
<td>Density (calculated)</td>
<td>1.627 Mg/m³</td>
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<tr>
<td>Absorption coefficient</td>
<td>0.940 mm⁻¹</td>
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<tr>
<td>F(000)</td>
<td>724</td>
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<tr>
<td>Crystal size</td>
<td>0.31 x 0.20 x 0.14 mm³</td>
</tr>
<tr>
<td>Theta range for data collection</td>
<td>2.07 to 27.00°</td>
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<tr>
<td>Index ranges</td>
<td>-9 ≤ h ≤ 6, -18 ≤ k ≤ 18, -17 ≤ l ≤ 17</td>
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<tr>
<td>Reflections collected</td>
<td>9624</td>
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<tr>
<td>Independent reflections</td>
<td>3129 [R(int) = 0.0306]</td>
</tr>
<tr>
<td>Completeness to theta = 27.00°</td>
<td>99.1 %</td>
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<tr>
<td>Absorption correction</td>
<td>Semi-empirical from equivalents</td>
</tr>
<tr>
<td>Max. and min. transmission</td>
<td>0.8796 and 0.7592</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on F²</td>
</tr>
<tr>
<td>Data / restraints / parameters</td>
<td>3129 / 7 / 254</td>
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<tr>
<td>Goodness-of-fit on F²</td>
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<tr>
<td>Final R indices [I&gt;2sigma(I)]</td>
<td>R1 = 0.0428, wR2 = 0.1012</td>
</tr>
<tr>
<td>R indices (all data)</td>
<td>R1 = 0.0542, wR2 = 0.1099</td>
</tr>
<tr>
<td>Largest diff. peak and hole</td>
<td>0.733 and -0.530 e.Å⁻³</td>
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</table>
Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for 7 (M= Ni). U(eq) is defined as one third of the trace of the orthogonalized U^ij tensor.

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
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<tbody>
<tr>
<td>Ni(1)</td>
<td>0</td>
<td>5000</td>
<td>0</td>
<td>28(1)</td>
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<tr>
<td>Cl(1)</td>
<td>2048(1)</td>
<td>3463(1)</td>
<td>3263(1)</td>
<td>49(1)</td>
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<tr>
<td>O(1)</td>
<td>1503(2)</td>
<td>3870(1)</td>
<td>-193(1)</td>
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<td>O(3)</td>
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<td>3287(3)</td>
<td>81(1)</td>
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<td>3319(13)</td>
<td>2323(10)</td>
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<td>4107(17)</td>
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<td>5419(3)</td>
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<td>59(1)</td>
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Table 3. Bond lengths [Å] and angles [°] for 7 (M= Ni).

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<th>Bond</th>
<th>Length/Angle</th>
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<td>Ni(1)-O(1)</td>
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<tr>
<td>Ni(1)-O(2)#1</td>
<td>2.0146(17)</td>
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<td>Ni(1)-O(5)#1</td>
<td>2.078(2)</td>
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<tr>
<td>Ni(1)-O(5)</td>
<td>2.078(2)</td>
</tr>
<tr>
<td>Cl(1)-O(8A)</td>
<td>1.325(14)</td>
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C(9)-C(10)  1.474(4)
C(10)-H(10A)  0.87(4)
C(10)-H(10B)  0.92(4)
C(10)-H(10C)  0.97(4)
C(11)-H(11A)  0.93(5)
C(11)-H(11B)  0.94(5)
C(11)-H(11C)  0.93(6)
O(1)#1-Ni(1)-O(1)  180.00(10)
O(1)#1-Ni(1)-O(2)  88.55(7)
O(1)-Ni(1)-O(2)  91.45(7)
O(1)#1-Ni(1)-O(2)#1  91.45(7)
O(1)-Ni(1)-O(2)#1  88.55(7)
O(2)-Ni(1)-O(2)#1  180.00(8)
O(1)#1-Ni(1)-O(5)#1  89.70(8)
O(1)-Ni(1)-O(5)#1  90.30(8)
O(2)-Ni(1)-O(5)#1  90.40(9)
O(2)#1-Ni(1)-O(5)#1  89.60(9)
O(1)#1-Ni(1)-O(5)  90.30(8)
O(1)-Ni(1)-O(5)  89.70(8)
O(2)-Ni(1)-O(5)  89.60(9)
O(2)#1-Ni(1)-O(5)  90.40(9)
O(5)#1-Ni(1)-O(5)  180.0
O(8A)-Cl(1)-O(9)  58.4(15)
O(8A)-Cl(1)-O(8)  135.5(10)
O(9)-Cl(1)-O(8)  110.6(3)
O(8A)-Cl(1)-O(9A)  122.9(17)
O(9)-Cl(1)-O(9A)  142.2(8)
O(8)-Cl(1)-O(9A)  38.0(9)
O(8A)-Cl(1)-O(6)  111.9(10)
O(9)-Cl(1)-O(6)  111.56(19)
O(8)-Cl(1)-O(6)  112.0(2)
O(9A)-Cl(1)-O(6)  102.4(9)
O(8A)-Cl(1)-O(7)  51.4(15)
O(9)-Cl(1)-O(7)  107.7(2)
O(8)-Cl(1)-O(7)  107.1(3)
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Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z

Table 4. Anisotropic displacement parameters (Å²x 10³) for 7 (M= Ni). The anisotropic displacement factor exponent takes the form: -2\pi²[ h*a²U_{11} + ... + 2 h k a*b* U_{12} ]

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Table 5. Hydrogen coordinates ($x \times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for 7 (M= Ni).

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Table 6. Torsion angles [°] for 7 (M= Ni).

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Table 7. Hydrogen bonds for 7 (M=Ni) [Å and °].

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<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
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Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z

Metal-bispyrone Complex 7 (M=Fe): IR KBr cm⁻¹ 1733.6, 1717.7, 1628.6, 1545.1, 1457.6, 1436.1, 1388.2, 1081.8, 621.8. HRMS – calcd 538.9680 found 538.9672. mp > 250 °C.

Lithium tris(bis-γ-pyrone) metal complex 8:
General Procedure was followed in which 10 mg (0.0520 mmoles) of bis-γ-pyrone 4 was poured into a solution of 2.8 mg (0.026 mmoles) LiClO₄ in dry MeOH yielding 12.8 mg of colorless crystals (quant.). IR (KBr) cm⁻¹ 1673.2, 1637.7, 1480.3, 1257.8, 1182.4, 1112.1, 1085.6, 624.8. HRMS – calcd 903.4295 found 903.1784 mp > 250 °C.

Crystal Structure:

Table 1. Crystal data and structure refinement for 8 (M = Li)

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<tr>
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<td>Wavelength</td>
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<tr>
<td>Crystal system</td>
<td>Triclinic</td>
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<tr>
<td>Space group</td>
<td>P-1</td>
</tr>
<tr>
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</tr>
</tbody>
</table>
Volume 3832.0(9) Å³
Z 4
Density (calculated) 1.503 Mg/m³
Absorption coefficient 0.259 mm⁻¹
F(000) 1788
Crystal size 0.38 x 0.27 x 0.16 mm³
Theta range for data collection 1.12 to 25.00°
Index ranges -16<=h<=16, -18<=k<=18, -21<=l<=21
Reflections collected 37382
Independent reflections 13455 [R(int) = 0.0402]
Completeness to theta = 25.00° 99.8 %
Absorption correction Semi-empirical from equivalents
Max. and min. transmission 0.9597 and 0.9078
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters 13455 / 2 / 1219
Goodness-of-fit on F² 1.033
Final R indices [I>2sigma(I)] R1 = 0.0556, wR2 = 0.1523
R indices (all data) R1 = 0.0898, wR2 = 0.1691
Largest diff. peak and hole 0.545 and -0.289 e.Å⁻³
Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for 8. U(eq) is defined as one third of the trace of the orthogonalized U^ij tensor.

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Symmetry transformations used to generate equivalent atoms:
Table 4. Anisotropic displacement parameters (Å² x 10³) for 8. The anisotropic displacement factor exponent takes the form: -2π² [ h²a²U₁¹ + ... + 2 h k a* b* U₁² ]

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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å²x 10^3) for 8.

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Table 6. Torsion angles [°] for 8.

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\begin{align*}
O(13)-\text{Li}(1)-\text{Li}(2)-O(9) & 11.5(5) \\
O(2)-\text{Li}(1)-\text{Li}(2)-O(9) & 121.6(5) \\
O(6)-\text{Li}(1)-\text{Li}(2)-O(9) & -113.6(5) \\
O(5)-\text{Li}(1)-\text{Li}(2)-O(9) & -91.5(5) \\
O(1)-\text{Li}(1)-\text{Li}(2)-O(9) & 98.5(5) \\
O(13)-\text{Li}(1)-\text{Li}(2)-O(10) & -172.0(3) \\
O(2)-\text{Li}(1)-\text{Li}(2)-O(10) & -61.9(5) \\
O(6)-\text{Li}(1)-\text{Li}(2)-O(10) & 62.9(5) \\
O(5)-\text{Li}(1)-\text{Li}(2)-O(10) & 85.0(4) \\
O(1)-\text{Li}(1)-\text{Li}(2)-O(10) & -84.9(4) \\
O(13)-\text{Li}(1)-\text{Li}(2)-O(1) & -87.1(3) \\
O(2)-\text{Li}(1)-\text{Li}(2)-O(1) & 23.0(3) \\
O(6)-\text{Li}(1)-\text{Li}(2)-O(1) & 147.9(4) \\
O(5)-\text{Li}(1)-\text{Li}(2)-O(1) & 169.9(4) \\
O(13)-\text{Li}(1)-\text{Li}(2)-O(5) & 103.0(3) \\
O(2)-\text{Li}(1)-\text{Li}(2)-O(5) & -146.9(4) \\
O(6)-\text{Li}(1)-\text{Li}(2)-O(5) & -22.1(3) \\
O(1)-\text{Li}(1)-\text{Li}(2)-O(5) & -169.9(4) \\
O(9)-\text{Li}(2)-O(1)-C(4) & 48.3(6) \\
O(10)-\text{Li}(2)-O(1)-C(4) & -66.2(5) \\
O(5)-\text{Li}(2)-O(1)-C(4) & -179.6(4) \\
\text{Li}(1)-\text{Li}(2)-O(1)-C(4) & 173.2(5) \\
O(9)-\text{Li}(2)-O(1)-\text{Li}(1) & -124.9(4) \\
O(10)-\text{Li}(2)-O(1)-\text{Li}(1) & 120.6(3) \\
O(5)-\text{Li}(2)-O(1)-\text{Li}(1) & 7.2(3) \\
O(13)-\text{Li}(1)-O(1)-C(4) & -77.0(4) \\
O(2)-\text{Li}(1)-O(1)-C(4) & 24.4(4) \\
O(6)-\text{Li}(1)-O(1)-C(4) & 114.6(7) \\
O(5)-\text{Li}(1)-O(1)-C(4) & 178.8(3)
\end{align*}
\]
Li(2)-Li(1)-O(1)-C(4) -174.5(4)
O(13)-Li(1)-O(1)-Li(2) 97.5(3)
O(2)-Li(1)-O(1)-Li(2) -161.1(3)
O(6)-Li(1)-O(1)-Li(2) -70.9(7)
O(5)-Li(1)-O(1)-Li(2) -6.7(3)
O(13)-Li(1)-O(2)-C(7) 70.9(5)
O(6)-Li(1)-O(2)-C(7) -175.2(4)
O(5)-Li(1)-O(2)-C(7) -86.5(8)
O(1)-Li(1)-O(2)-C(7) -37.5(6)
O(9)-Li(2)-O(5)-C(14) -69.6(5)
O(10)-Li(2)-O(5)-C(14) 42.6(5)
O(1)-Li(2)-O(5)-C(14) 155.7(3)
Li(1)-Li(2)-O(5)-C(14) 162.9(4)
O(9)-Li(2)-O(5)-Li(1) 127.5(4)
O(10)-Li(2)-O(5)-Li(1) -120.4(3)
O(1)-Li(2)-O(5)-Li(1) -7.3(3)
O(13)-Li(1)-O(5)-C(14) 110.4(3)
O(2)-Li(1)-O(5)-C(14) -92.5(7)
O(6)-Li(1)-O(5)-C(14) -2.0(4)
O(1)-Li(1)-O(5)-C(14) -157.4(3)
Li(2)-Li(1)-O(5)-C(14) -164.1(4)
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O(2)-Li(1)-O(5)-Li(2) 71.5(7)
O(6)-Li(1)-O(5)-Li(2) 162.1(3)
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Li(2)-Li(1)-O(6)-C(17) 9.3(6)
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O(9)-Li(2)-O(10)-C(27) -5.9(4)
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O(5)-Li(2)-O(10)-C(27) -133.8(3)
Li(1)-Li(2)-O(10)-C(27) 176.4(3)
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O(5)-Li(1)-O(13)-C(31) -44.8(6)
O(1)-Li(1)-O(13)-C(31) -127.1(5)
Li(2)-Li(1)-O(13)-C(31) -86.3(5)
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C(6)-O(3)-C(2)-C(1) -178.0(3)
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O(1)-C(4)-C(5)-C(6) 178.4(3)
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O(1)-C(4)-C(5)-C(7) -0.2(6)
C(3)-C(4)-C(5)-C(7) 179.7(3)
C(9)-O(4)-C(6)-O(3) 179.1(2)
C(9)-O(4)-C(6)-C(5) -1.1(5)
C(4)-C(5)-C(6)-O(4) 179.1(3)
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C(7)-C(5)-C(6)-O(4) 177.7(3)
Li(1)-O(2)-C(7)-C(8) -165.2(4)
Li(1)-O(2)-C(7)-C(5) 14.3(7)
C(6)-C(5)-C(7)-O(2) -175.3(4)
C(4)-C(5)-C(7)-O(2) 3.3(6)
C(6)-C(5)-C(7)-C(8) 4.3(5)
C(4)-C(5)-C(7)-C(8) -177.1(3)
O(2)-C(7)-C(8)-C(9) \quad 175.9(4)
C(5)-C(7)-C(8)-C(9) \quad -3.6(6)
C(7)-C(8)-C(9)-O(4) \quad 0.6(6)
C(7)-C(8)-C(9)-C(10) \quad -177.8(4)
C(6)-O(4)-C(9)-C(8) \quad 1.9(5)
C(6)-O(4)-C(9)-C(10) \quad -179.5(3)
C(16)-O(7)-C(12)-C(13) \quad -8.3(4)
C(16)-O(7)-C(12)-C(11) \quad 170.9(3)
O(7)-C(12)-C(13)-C(14) \quad 3.6(5)
C(11)-C(12)-C(13)-C(14) \quad -175.4(3)
Li(2)-O(5)-C(14)-C(15) \quad -149.8(3)
Li(1)-O(5)-C(14)-C(15) \quad 7.7(5)
Li(2)-O(5)-C(14)-C(13) \quad 29.8(5)
Li(1)-O(5)-C(14)-C(13) \quad -172.7(3)
C(12)-C(13)-C(14)-O(5) \quad -177.1(3)
C(12)-C(13)-C(14)-C(15) \quad 2.6(5)
O(5)-C(14)-C(15)-O(7) \quad -177.1(3)
C(14)-C(15)-C(16)-O(8) \quad -179.4(3)
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C(17)-C(15)-C(16)-O(7) \quad -177.4(3)
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C(27)-C(25)-C(26)-O(12) 2.2(5)
Li(2)-O(10)-C(27)-C(28) -177.7(3)
Li(2)-O(10)-C(27)-C(25) 2.8(5)
C(26)-C(25)-C(27)-O(10) -179.5(3)
C(24)-C(25)-C(27)-O(10) 3.3(5)
C(26)-C(25)-C(27)-C(28) 0.9(4)
C(24)-C(25)-C(27)-C(28) -176.3(3)
O(10)-C(27)-C(28)-C(29) 178.4(3)
C(25)-C(27)-C(28)-C(29) -2.0(5)
C(27)-C(28)-C(29)-O(12) 0.2(5)
C(27)-C(28)-C(29)-C(30) 179.7(4)
C(26)-O(12)-C(29)-C(28) 2.8(4)
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O(1')-Li(2')-O(5')-C(14') -168.3(3)
Li(1')-Li(2')-O(5')-C(14') -152.4(5)
O(9')-Li(2')-O(5')-Li(1') 99.8(3)
O(10')-Li(2')-O(5')-Li(1') -147.1(4)
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O(13')-Li(1')-O(5')-C(14') 71.0(4)
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O(1')-Li(1')-O(5')-C(14') 172.4(3)
Li(2')-Li(1')-O(6')-C(17') 41.5(6)
O(10')-Li(2')-O(9')-C(24') 0.2(4)
O(5')-Li(2')-O(9')-C(24') -122.6(4)
O(1')-Li(2')-O(9')-C(24') 122.9(4)
Li(1')-Li(2')-O(9')-C(24') -178.8(5)
O(2')-Li(1')-O(13')-C(31') -47.6(5)
O(6')-Li(1')-O(13')-C(31') -149.2(4)
O(1')-Li(1')-O(13')-C(31') 43.1(5)
O(5')-Li(1')-O(13')-C(31') 124.3(4)
\begin{align*}
L(2')-L(1')-O(13')-C(31') & \quad 83.0(4) \\
C(6')-O(3')-C(2')-C(3') & \quad -2.2(4) \\
C(6')-O(3')-C(2')-C(1') & \quad 178.6(3) \\
O(3')-C(2')-C(3')-C(4') & \quad 2.6(5) \\
C(1')-C(2')-C(3')-C(4') & \quad -178.4(3) \\
L(2')-O(1')-C(4')-C(3') & \quad 6.8(6) \\
L(1')-O(1')-C(4')-C(3') & \quad 171.6(3) \\
L(2')-O(1')-C(4')-C(5') & \quad -172.5(4) \\
L(1')-O(1')-C(4')-C(5') & \quad -7.7(5) \\
C(2')-C(3')-C(4')-O(1') & \quad 178.8(3) \\
C(2')-C(3')-C(4')-C(5') & \quad -1.8(5) \\
O(1')-C(4')-C(5')-C(6') & \quad -180.0(3) \\
C(3')-C(4')-C(5')-C(6') & \quad 0.7(5) \\
O(1')-C(4')-C(5')-C(7') & \quad -1.3(5) \\
C(3')-C(4')-C(5')-C(7') & \quad 179.3(3) \\
C(2')-O(3')-C(6')-O(4') & \quad -179.1(2) \\
C(2')-O(3')-C(6')-C(5') & \quad 1.1(5) \\
C(9')-O(4')-C(6')-O(3') & \quad 178.5(2) \\
C(9')-O(4')-C(6')-C(5') & \quad -1.7(5) \\
C(4')-C(5')-C(6')-O(3') & \quad -0.4(5) \\
C(7')-C(5')-C(6')-O(3') & \quad -179.2(3) \\
C(4')-C(5')-C(6')-O(4') & \quad 179.8(3) \\
C(7')-C(5')-C(6')-O(4') & \quad 1.1(5) \\
L(1')-O(2')-C(7')-C(8') & \quad -170.3(4) \\
L(1')-O(2')-C(7')-C(5') & \quad 11.2(6) \\
C(6')-C(5')-C(7')-O(2') & \quad 178.6(3) \\
C(4')-C(5')-C(7')-O(2') & \quad 0.0(6) \\
C(6')-C(5')-C(7')-C(8') & \quad 0.0(5) \\
C(4')-C(5')-C(7')-C(8') & \quad -178.6(3) \\
O(2')-C(7')-C(8')-C(9') & \quad -179.1(4) \\
C(5')-C(7')-C(8')-C(9') & \quad -0.5(5) \\
C(7')-C(8')-C(9')-O(4') & \quad -0.1(5) \\
C(7')-C(8')-C(9')-C(10') & \quad 178.9(4) \\
C(6')-O(4')-C(9')-C(8') & \quad 1.1(5) \\
C(6')-O(4')-C(9')-C(10') & \quad -178.0(3) \\
C(16')-O(7')-C(12')-C(13') & \quad 6.6(4)
\end{align*}
C(16')-O(7')-C(12')-C(11')  -172.6(3)
O(7')-C(12')-C(13')-C(14')  -1.6(5)
C(11')-C(12')-C(13')-C(14')  177.3(4)
Li(2')-O(5')-C(14')-C(13')  -19.5(6)
Li(1')-O(5')-C(14')-C(13')  -165.0(3)
Li(2')-O(5')-C(14')-C(15')  161.3(4)
Li(1')-O(5')-C(14')-C(15')  15.9(5)
C(12')-C(13')-C(14')-O(5')  176.9(3)
C(12')-C(13')-C(14')-C(15')  -3.9(5)
O(5')-C(14')-C(15')-C(16')  -176.3(3)
C(13')-C(14')-C(15')-C(16')  4.4(4)
O(5')-C(14')-C(15')-C(17')  7.1(5)
C(13')-C(14')-C(15')-C(17')  -172.2(3)
C(19')-O(8')-C(16')-O(7')  -177.4(3)
C(19')-O(8')-C(16')-C(15')  2.9(5)
C(12')-O(7')-C(16')-O(8')  174.3(2)
C(12')-O(7')-C(16')-C(15')  -6.0(5)
C(14')-C(15')-C(16')-O(8')  -180.0(3)
C(14')-C(15')-C(16')-O(7')  -3.1(5)
C(14')-C(15')-C(16')-O(7')  0.4(5)
C(17')-C(15')-C(16')-O(7')  177.3(3)
Li(1')-O(6')-C(17')-C(18')  153.3(4)
Li(1')-O(6')-C(17')-C(15')  -26.2(6)
C(16')-C(15')-C(17')-O(6')  -179.6(3)
C(14')-C(15')-C(17')-O(6')  -3.0(6)
C(16')-C(15')-C(17')-C(18')  0.8(5)
C(14')-C(15')-C(17')-C(18')  177.4(3)
O(6')-C(17')-C(18')-C(19')  -178.1(4)
C(15')-C(17')-C(18')-C(19')  1.5(5)
C(17')-C(18')-C(19')-O(8')  -1.8(6)
C(17')-C(18')-C(19')-C(20')  178.7(5)
C(16')-O(8')-C(19')-C(18')  -0.3(5)
C(16')-O(8')-C(19')-C(20')  179.2(4)
C(26')-O(11')-C(22')-C(23')  -1.2(5)
C(26')-O(11')-C(22')-C(21')  178.8(3)
O(11')-C(22')-C(23')-C(24')  -1.0(5)
C(21')-C(22')-C(23')-C(24') 179.0(4)
Li(2')-O(9')-C(24')-C(23') -178.5(3)
Li(2')-O(9')-C(24')-C(25') 0.3(5)
C(22')-C(23')-C(24')-O(9') -178.7(3)
C(22')-C(23')-C(24')-C(25') 2.5(5)
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C(23')-C(24')-C(25')-C(26') -1.8(4)
O(9')-C(24')-C(25')-C(27') -0.7(5)
C(23')-C(24')-C(25')-C(27') 178.1(3)
C(29')-O(12')-C(26')-C(25') 0.8(5)
C(22')-O(11')-C(26')-C(25') -177.4(3)
C(22')-O(11')-C(26')-C(25') 1.9(5)
C(24')-C(25')-C(26')-O(12') 178.8(3)
C(27')-C(25')-C(26')-O(12') -1.1(5)
C(24')-C(25')-C(26')-O(11') -0.3(5)
C(27')-C(25')-C(26')-O(11') 179.8(3)
Li(2')-O(10')-C(27')-C(28') 179.1(3)
Li(2')-O(10')-C(27')-C(25') 0.2(5)
C(26')-C(25')-C(27')-O(10') -179.6(3)
C(24')-C(25')-C(27')-O(10') 0.4(5)
C(26')-C(25')-C(27')-C(28') 1.4(4)
C(24')-C(25')-C(27')-C(28') -178.5(3)
O(10')-C(27')-C(28')-C(29') 179.3(3)
C(25')-C(27')-C(28')-C(29') -1.7(5)
C(27')-C(28')-C(29')-O(12') 1.5(5)
C(27')-C(28')-C(29')-C(30') -179.1(4)
C(26')-O(12')-C(29')-C(28') -1.0(4)
C(26')-O(12')-C(29')-C(28') 179.6(3)

Symmetry transformations used to generate equivalent atoms:
Table 7. Hydrogen bonds for 8 [Å and °].

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Symmetry transformations used to generate equivalent atoms:
#1 x,y,z-1

**Metal Bispyrone Complex 9 (M=Cu):** IR (KBr) cm⁻¹ 1668.8, 1605.3, 1558.4, 1491.0, 1260.8, 1183.2, 1106.6, 1071.5, 1028.9, 864.6, 623.5. HRMS – calcd 545.9626 found 545.9595. mp > 250 °C.

Crystal Structure:
Table 1. Crystal data and structure refinement for 9 (M= Cu).

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<td>Empirical formula</td>
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<td>Formula weight</td>
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Temperature 173(2) K
Wavelength 0.71073 Å
Crystal system Monoclinic
Space group P2(1)/n
Unit cell dimensions a = 9.9449(10) Å  a= 90°.
b = 12.6414(13) Å  b= 95.359(2)°.
c = 10.4593(11) Å  g = 90°.
Volume 1309.2(2) Å³
Z 2
Density (calculated) 1.732 Mg/m³
Absorption coefficient 1.125 mm⁻¹
F(000) 694
Crystal size 0.32 x 0.27 x 0.16 mm³
Theta range for data collection 2.53 to 27.00°.
Index ranges -12<=h<=12, -16<=k<=16, -13<=l<=13
Reflections collected 14330
Independent reflections 2858 [R(int) = 0.0158]
Completeness to theta = 27.00° 100.0 %
Absorption correction Semi-empirical from equivalents
Max. and min. transmission 0.8405 and 0.7148
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters 2858 / 0 / 227
Goodness-of-fit on F² 1.048
Final R indices [I>2sigma(I)] R1 = 0.0238, wR2 = 0.0690
R indices (all data) R1 = 0.0251, wR2 = 0.0702
Largest diff. peak and hole 0.312 and -0.229 e.Å⁻³
Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for 9 (M=Cu). U(eq) is defined as one third of the trace of the orthogonalized U^ij tensor.

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Table 3. Bond lengths [Å] and angles [°] for 9 (M=Cu).

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C(7)-C(8)-H(8) 118.7(12)
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C(8)-C(9)-C(10) 126.89(15)
O(3)-C(9)-C(10) 112.30(14)
C(9)-C(10)-H(10A) 107.6(14)
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H(10A)-C(10)-H(10B) 113(2)
C(9)-C(10)-H(10C) 110.3(13)
H(10A)-C(10)-H(10C) 107.3(19)
H(10B)-C(10)-H(10C) 111(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1
Table 4. Anisotropic displacement parameters (Å² x 10³) for 9 (M= Cu). The anisotropic displacement factor exponent takes the form: -2\(p^2\)\[ h^2a^*2U^{11} + ... + 2hk a^* b^* U^{12} \]

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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 9 (M=Cu).

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Table 6. Torsion angles [°] for 9 (M=Cu).

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C(3)-C(4)-C(5)-C(7) 0.28(19)
O(1)-C(4)-C(5)-C(6) 0.8(2)
C(3)-C(4)-C(5)-C(6) -179.07(13)
Cu(1)-O(2)-C(6)-O(3) -178.34(9)
Cu(1)-O(2)-C(6)-C(5) 2.0(2)
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C(7)-C(5)-C(6)-O(2) 179.08(14)
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C(7)-C(5)-C(6)-O(3) -0.53(19)
C(4)-C(5)-C(6)-O(3) 178.84(12)
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C(2)-O(4)-C(7)-C(8) 179.22(12)
C(6)-C(5)-C(7)-O(4) 179.20(12)
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C(6)-C(5)-C(7)-C(8) -0.2(2)
C(4)-C(5)-C(7)-C(8) -179.55(13)
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O(4)-C(7)-C(8)-C(9) -178.21(13)
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C(7)-C(8)-C(9)-O(3) -1.5(2)
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C(6)-O(3)-C(9)-C(8) 0.9(2)
C(6)-O(3)-C(9)-C(10) -178.02(14)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1
Table 7. Hydrogen bonds for 9 (M = Cu) [Å and °].

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<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
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</table>

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1    #2 x-1,y,z    #3 x-1/2,-y+1/2,z+1/2

**Metal Bispyrone Complex 9** (M=Co): IR (KBr) cm⁻¹ 1674.4, 1634.9, 1477.7, 1255.7, 1182.3, 1120.3, 1087.6, 623.3. HRMS – calcd 541.9662 found 541.9648. mp > 250 °C.

Crystal Structure:
Empirical formula  C20 H20 Cl2 Co O18
Formula weight  678.19
Temperature  173(2) K
Wavelength  0.71073 Å
Crystal system  Monoclinic
Space group  P2(1)/n
Unit cell dimensions  
\[ \begin{align*}
\text{a} &= 10.033(6) \text{ Å} & a &= 90^\circ, \\
\text{b} &= 12.586(8) \text{ Å} & b &= 95.922(9)^\circ, \\
\text{c} &= 10.332(6) \text{ Å} & g &= 90^\circ.
\end{align*} \]
Volume  1297.7(14) Å³
Z  2
Density (calculated)  1.736 Mg/m³
Absorption coefficient  0.955 mm⁻¹
F(000)  690
Crystal size  0.19 x 0.16 x 0.12 mm³
Theta range for data collection  2.56 to 24.99°.
Index ranges  -11<=h<=11, -14<=k<=14, -12<=l<=12
Reflections collected  11277
Independent reflections  2279 [R(int) = 0.0425]
Completeness to theta = 24.99°  100.0 %
Absorption correction  Semi-empirical from equivalents
Max. and min. transmission  0.8941 and 0.8394
Refinement method  Full-matrix least-squares on F²
Data / restraints / parameters  2279 / 0 / 227
Goodness-of-fit on F²  1.055
Final R indices [I>2sigma(I)]  R1 = 0.0509, wR2 = 0.1218
R indices (all data)  R1 = 0.0666, wR2 = 0.1320
Largest diff. peak and hole  0.924 and -0.438 e.Å⁻³

Table 2. Atomic coordinates ( x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for 9 (M= Co). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

<table>
<thead>
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<th>z</th>
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<td>Bond Lengths [Å]</td>
<td>Angles [°]</td>
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Table 3. Bond lengths [Å] and angles [°] for 9 (M= Co).
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<td>O(5)-H(5OB)</td>
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<tr>
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O(2)#1-Co(1)-O(2) 180.00(16)
O(8)-Cl(1)-O(9) 112.3(2)
O(8)-Cl(1)-O(7) 109.8(3)
O(9)-Cl(1)-O(7) 111.7(2)
O(8)-Cl(1)-O(6) 107.8(2)
O(9)-Cl(1)-O(6) 110.2(2)
O(7)-Cl(1)-O(6) 104.6(3)
C(4)-O(1)-Co(1) 130.1(3)
C(6)-O(2)-Co(1) 127.7(3)
C(6)-O(3)-C(9) 123.0(3)
C(7)-O(4)-C(2) 119.7(3)
Co(1)-O(5)-H(5OA) 121(5)
Co(1)-O(5)-H(5OB) 123(6)
H(5OA)-O(5)-H(5OB) 108(7)
C(2)-C(1)-H(1A) 112(3)
C(2)-C(1)-H(1B) 108(3)
H(1A)-C(1)-H(1B) 115(5)
C(2)-C(1)-H(1C) 112(4)
H(1A)-C(1)-H(1C) 97(5)
H(1B)-C(1)-H(1C) 111(5)
C(3)-C(2)-O(4) 120.6(4)
C(3)-C(2)-C(1) 127.1(5)
O(4)-C(2)-C(1) 112.3(4)
C(2)-C(3)-C(4) 123.5(4)
C(2)-C(3)-H(3) 116(4)
C(4)-C(3)-H(3) 120(4)
O(1)-C(4)-C(3) 120.9(4)
O(1)-C(4)-C(5) 124.5(4)
C(3)-C(4)-C(5) 114.6(4)
C(7)-C(5)-C(6) 117.4(4)
C(7)-C(5)-C(4) 119.9(4)
C(6)-C(5)-C(4) 122.7(4)
O(2)-C(6)-O(3) 114.3(4)
O(2)-C(6)-C(5) 127.8(4)
O(3)-C(6)-C(5) 117.9(3)
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<tr>
<td>H(10B)-C(10)-H(10C)</td>
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</table>

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z

Table 4. Anisotropic displacement parameters (Å² x 10⁶) for 9 (M= Co). The anisotropic displacement factor exponent takes the form: -2π² [ h²a²*U¹¹ + ... + 2 h k a* b* U²³ ]

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<th>Atom</th>
<th>U¹¹</th>
<th>U²²</th>
<th>U³³</th>
<th>U²³</th>
<th>U¹³</th>
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<td>4(1)</td>
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<td>-4(1)</td>
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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 9 (M=Co).

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<th>U(eq)</th>
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Table 6. Torsion angles [°] for 9 (M=Co).

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<td>Bond</td>
<td>Angle (°)</td>
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<tr>
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<tr>
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<tr>
<td>O(5)#1-Co(1)-O(2)-C(6)</td>
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Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z

Table 7. Hydrogen bonds for 9 (M= Co) [Å and °].

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<th>d(H...A)</th>
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Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z   #2 -x+1/2,y-1/2,-z+1/2   #3 -x+1,-y+1,-z+1

**Metal Bispyrone Complex 9 (M=Ni):** IR (KBr) cm⁻¹ 1670.7, 1624.5, 1487.2, 1257.5, 1183.3, 1108.0, 625.6. HRMS – calcld 540.9684 found 540.9677. mp > 250 °C.

Crystal Structure:
Table 1. Crystal data and structure refinement for 9 (M= Ni).

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<tr>
<td>Formula weight</td>
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Temperature 173(2) K
Wavelength 0.71073 Å
Crystal system Monoclinic
Space group P2(1)/n
Unit cell dimensions
\[ \begin{align*}
a &= 9.952(3) \text{ Å} & a &= 90^\circ. \\
b &= 12.636(3) \text{ Å} & b &= 95.348(4)^\circ. \\
c &= 10.439(3) \text{ Å} & g &= 90^\circ. \\
\end{align*} \]
Volume 1307.0(6) Å³
Z 2
Density (calculated) 1.723 Mg/m³
Absorption coefficient 1.033 mm⁻¹
F(000) 692
Crystal size 0.19 x 0.12 x 0.09 mm³
Theta range for data collection 2.54 to 26.99°.
Index ranges -12<=h<=12, -15<=k<=16, -13<=l<=13
Reflections collected 12556
Independent reflections 2852 [R(int) = 0.0295]
Completeness to theta = 26.99° 99.9 %
Absorption correction Semi-empirical from equivalents
Max. and min. transmission 0.9127 and 0.8278
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters 2852 / 0 / 227
Goodness-of-fit on F² 1.066
Final R indices [I>2sigma(I)] R1 = 0.0295, wR2 = 0.0723
R indices (all data) R1 = 0.0368, wR2 = 0.0775
Largest diff. peak and hole 0.327 and -0.412 e.Å⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for 9 (M= Ni). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

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Table 3. Bond lengths [Å] and angles [°] for 9 (M= Ni).

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C(5)-C(7)-C(8) 122.10(18)
C(9)-C(8)-C(7) 118.93(18)
C(9)-C(8)-H(8) 122.3(14)
C(7)-C(8)-H(8) 118.8(14)
C(8)-C(9)-O(3) 120.67(19)
C(8)-C(9)-C(10) 127.2(2)
O(3)-C(9)-C(10) 112.11(18)
C(9)-C(10)-H(10A) 111.2(19)
C(9)-C(10)-H(10B) 109(2)
H(10A)-C(10)-H(10B) 112(3)
C(9)-C(10)-H(10C) 106.8(16)
H(10A)-C(10)-H(10C) 107(2)
H(10B)-C(10)-H(10C) 110(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1

Table 4. Anisotropic displacement parameters (Å² x 10³) for 9 (M= Ni). The anisotropic displacement factor exponent takes the form: -2π²[ h²a*²U¹¹ + ... + 2 h k a* b* U¹² ]

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<td>-4(1)</td>
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<tr>
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<tr>
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<td>2(1)</td>
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<tr>
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<td>-3(1)</td>
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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å² x 10^3) for 9 (M= Ni).

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
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<td>5930(30)</td>
<td>1910(20)</td>
<td>7310(30)</td>
<td>54(8)</td>
</tr>
<tr>
<td>H(1B)</td>
<td>6640(30)</td>
<td>2940(20)</td>
<td>6810(30)</td>
<td>59(8)</td>
</tr>
<tr>
<td>H(1C)</td>
<td>5820(30)</td>
<td>2140(20)</td>
<td>5750(30)</td>
<td>51(7)</td>
</tr>
<tr>
<td>H(3)</td>
<td>3740(20)</td>
<td>3113(17)</td>
<td>4980(20)</td>
<td>35(6)</td>
</tr>
<tr>
<td>H(5A)</td>
<td>1810(40)</td>
<td>6610(30)</td>
<td>5250(40)</td>
<td>102(12)</td>
</tr>
<tr>
<td>H(5B)</td>
<td>670(30)</td>
<td>6870(30)</td>
<td>4370(30)</td>
<td>74(11)</td>
</tr>
<tr>
<td>H(8)</td>
<td>4080(20)</td>
<td>4123(18)</td>
<td>10080(20)</td>
<td>37(6)</td>
</tr>
<tr>
<td>H(10A)</td>
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<td>5180(20)</td>
<td>11730(30)</td>
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</tr>
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<td>H(10B)</td>
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<td>5210(30)</td>
<td>11360(30)</td>
<td>94(12)</td>
</tr>
<tr>
<td>H(10C)</td>
<td>2310(30)</td>
<td>6170(20)</td>
<td>11020(20)</td>
<td>61(8)</td>
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</table>

Table 6. Torsion angles [°] for 9 (M= Ni).

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<td>O(5)#1-Ni(1)-O(1)-C(4)</td>
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<tr>
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<td>90.25(17)</td>
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<tr>
<td>O(1)-Ni(1)-O(2)-C(6)</td>
<td>1.35(16)</td>
</tr>
<tr>
<td>O(1)#1-Ni(1)-O(2)-C(6)</td>
<td>-178.65(16)</td>
</tr>
<tr>
<td>O(2)#1-Ni(1)-O(2)-C(6)</td>
<td>-74(89)</td>
</tr>
<tr>
<td>Bond/Angle</td>
<td>Value</td>
</tr>
<tr>
<td>-----------</td>
<td>-----------</td>
</tr>
<tr>
<td>O(5)-Ni(1)-C(6)</td>
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<tr>
<td>O(5)-Ni(1)-O(2)</td>
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<tr>
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<tr>
<td>O(4)-C(2)-C(3)</td>
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<td>C(1)-C(2)-C(3)</td>
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</tr>
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<td>Ni(1)-O(1)-C(4)</td>
<td>-179.13(14)</td>
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<tr>
<td>Ni(1)-O(1)-C(5)</td>
<td>1.1(3)</td>
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<td>C(2)-C(3)-C(4)</td>
<td>-179.97(19)</td>
</tr>
<tr>
<td>O(1)-C(4)-C(5)</td>
<td>-0.1(3)</td>
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<td>O(1)-C(5)-C(7)</td>
<td>-179.61(18)</td>
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<td>C(1)-C(5)-C(7)</td>
<td>179.28(18)</td>
</tr>
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<td>Ni(1)-O(2)-C(6)</td>
<td>178.32(11)</td>
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<td>Ni(1)-O(2)-O(3)</td>
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<td>C(9)-O(3)-C(6)</td>
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<td>C(9)-O(3)-O(2)</td>
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<td>C(4)-C(5)-C(6)</td>
<td>1.4(3)</td>
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<td>C(4)-C(5)-C(7)</td>
<td>0.8(3)</td>
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<td>C(2)-O(4)-C(7)</td>
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<tr>
<td>C(2)-O(4)-C(8)</td>
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<tr>
<td>C(6)-C(5)-C(7)</td>
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<tr>
<td>C(4)-C(5)-O(4)</td>
<td>-179.26(17)</td>
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<tr>
<td>C(4)-C(5)-O(4)</td>
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<td>-0.1(3)</td>
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Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1

Table 7. Hydrogen bonds for 9 (M= Ni) [Å and °].

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<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O(5)-H(5B)...O(6)#2</td>
<td>0.82(3)</td>
<td>2.04(3)</td>
<td>2.854(3)</td>
<td>173(3)</td>
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</table>

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1    #2 x-1/2,-y+3/2,z-1/2

Metal-Bispyrone Complex 9 (M=Zn): IR (KBr) cm⁻¹ 1700.3, 1653.1, 1646.5, 1635.0, 1558.2, 1472.9, 1102.9, 623.7. HRMS – calcd 546.9622 found 546.9634. mp > 250 °C.

Crystal Structure:
Table 1. Crystal data and structure refinement for 9 (M= Zn).

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<thead>
<tr>
<th>Identification code</th>
<th>(9) (M= Zn)</th>
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<tr>
<td>Empirical formula</td>
<td>(\text{C}20\ \text{H}20\ \text{Cl}2\ \text{O}18\ \text{Zn})</td>
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Table 2. Atomic coordinates ($x \times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for 9 (M= Zn). U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

<table>
<thead>
<tr>
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<th>y</th>
<th>z</th>
<th>U(eq)</th>
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<td>0</td>
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<td>31(1)</td>
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<td>54(2)</td>
<td>34(1)</td>
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<td>O(3)</td>
<td>O(4)</td>
<td>O(5)</td>
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<td>1972(2)</td>
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</table>
Table 3. Bond lengths [Å] and angles [°] for 9 (M= Zn).

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<tr>
<td>Zn(1)-O(5)#1</td>
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<tr>
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<tr>
<td>Cl(1)-O(8)</td>
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<td>Cl(1)-O(9)</td>
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<td>C(10)-H(10B)</td>
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C(10)-H(10C) 0.91(4)

O(1)#1-Zn(1)-O(1) 180.00(11)
O(1)#1-Zn(1)-O(2)#1 87.82(7)
O(1)-Zn(1)-O(2)#1 92.18(7)
O(1)-Zn(1)-O(2) 87.82(7)
O(2)#1-Zn(1)-O(2) 180.00(11)
O(1)#1-Zn(1)-O(5)#1 88.80(10)
O(1)-Zn(1)-O(5)#1 91.20(10)
O(2)#1-Zn(1)-O(5)#1 91.11(9)
O(2)-Zn(1)-O(5)#1 88.89(9)
O(1)#1-Zn(1)-O(5) 91.20(10)
O(1)-Zn(1)-O(5) 88.80(10)
O(2)#1-Zn(1)-O(5) 88.89(9)
O(2)-Zn(1)-O(5) 91.11(9)
O(5)#1-Zn(1)-O(5) 180.00(18)
O(8)-Cl(1)-O(9) 111.87(14)
O(8)-Cl(1)-O(7) 111.01(15)
O(9)-Cl(1)-O(7) 109.51(15)
O(8)-Cl(1)-O(6) 109.87(15)
O(9)-Cl(1)-O(6) 108.51(14)
O(7)-Cl(1)-O(6) 105.87(15)
C(4)-O(1)-Zn(1) 129.72(18)
C(6)-O(2)-Zn(1) 127.26(18)
C(6)-O(3)-C(9) 122.6(2)
Zn(1)-O(5)-H(5A) 118(3)
Zn(1)-O(5)-H(5B) 114(4)
H(5A)-O(5)-H(5B) 112(5)
C(2)-C(1)-H(1A) 109(2)
C(2)-C(1)-H(1B) 112(2)
H(1A)-C(1)-H(1B) 108(3)
C(2)-C(1)-H(1C) 111(2)
H(1A)-C(1)-H(1C) 107(3)
H(1B)-C(1)-H(1C) 110(3)
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<tr>
<td>H(10B)-C(10)-H(10C)</td>
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</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y,-z
Table 4. Anisotropic displacement parameters (Å\(^2\times 10^3\)) for 9 (M= Zn). The anisotropic displacement factor exponent takes the form: 

\[-2\pi^2 \left[ h^2a^{*2}U_{11} + \ldots + 2hk a^{*} b^{*} U_{12} \right] \]

<table>
<thead>
<tr>
<th></th>
<th>(U_{11})</th>
<th>(U_{22})</th>
<th>(U_{33})</th>
<th>(U_{23})</th>
<th>(U_{13})</th>
<th>(U_{12})</th>
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<td>-1(1)</td>
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<tr>
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<td>3(1)</td>
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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 9 (M=Zn).

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<th>z</th>
<th>U(eq)</th>
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<td>2340(40)</td>
<td>50(10)</td>
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<tr>
<td>H(1B)</td>
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<td>44(9)</td>
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<td>H(1C)</td>
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<td>1880(30)</td>
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<td>1940(20)</td>
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<td>H(5A)</td>
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<td>51(13)</td>
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<tr>
<td>H(5B)</td>
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<td>-630(40)</td>
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<tr>
<td>H(8)</td>
<td>9060(40)</td>
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<td>41(9)</td>
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<td>H(10A)</td>
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<tr>
<td>H(10B)</td>
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<tr>
<td>H(10C)</td>
<td>7870(40)</td>
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<td>39(9)</td>
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Table 6. Torsion angles [°] for 9 (M=Zn).

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<th>Angle [°]</th>
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<tr>
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<td>O(5)#1-Zn(1)-O(1)-C(4)</td>
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<td>O(1)#1-Zn(1)-O(2)-C(6)</td>
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<td>O(1)-Zn(1)-O(2)-C(6)</td>
<td>-0.9(2)</td>
</tr>
<tr>
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<tr>
<td>O(5)#1-Zn(1)-O(2)-C(6)</td>
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<td>C(7)-O(4)-C(2)-C(3)</td>
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<td>C(7)-O(4)-C(2)-C(1)</td>
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</tr>
<tr>
<td>O(4)-C(2)-C(3)-C(4)</td>
<td>-1.3(5)</td>
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<tr>
<td>C(1)-C(2)-C(3)-C(4)</td>
<td>179.4(3)</td>
</tr>
<tr>
<td>Zn(1)-O(1)-C(4)-C(3)</td>
<td>-179.19(19)</td>
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<tr>
<td>Zn(1)-O(1)-C(4)-C(5)</td>
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</table>
Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z
Table 7. Hydrogen bonds for 9 (M= Zn) [Å and °].

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
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</thead>
<tbody>
<tr>
<td>O(5)-H(5A)...O(7)</td>
<td>0.70(4)</td>
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<td>2.794(4)</td>
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<td>0.73(4)</td>
<td>2.12(5)</td>
<td>2.851(4)</td>
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</table>

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z  #2 x-1/2,-y-1/2,z-1/2

**Metal-Bispyrone Complex 9** (M=Fe): IR (KBr) cm⁻¹ 1699.1, 1676.0, 1632.5, 1558.1, 1541.5, 1473.3, 1102.6, 623.4. HRMS – calcd 637.9165 found 637.9177. mp > 250 °C.

Crystal Structure:
Table 1. Crystal data and structure refinement for 9 (M= Fe).

<table>
<thead>
<tr>
<th>Identification code</th>
<th>9 (M= Fe)</th>
</tr>
</thead>
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<tr>
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<td>C20 H20 Cl2 Fe O18</td>
</tr>
<tr>
<td>Formula weight</td>
<td>675.11</td>
</tr>
</tbody>
</table>
Temperature 173(2) K
Wavelength 0.71073 Å
Crystal system Monoclinic
Space group P2(1)/n
Unit cell dimensions
\[ a = 10.113(2) \text{ Å} \]
\[ b = 12.627(3) \text{ Å} \]
\[ c = 10.339(2) \text{ Å} \]
\[ \alpha = 90^\circ \]
\[ \beta = 95.402(4)^\circ \]
\[ \gamma = 90^\circ \]
Volume 1314.5(5) Å\(^3\)
Z 2
Density (calculated) 1.706 Mg/m\(^3\)
Absorption coefficient 0.863 mm\(^{-1}\)
\(F(000)\) 688
Crystal size 0.39 x 0.22 x 0.10 mm\(^3\)
Theta range for data collection 2.55 to 27.00°.
Index ranges -12<=h<=10, -16<=k<=15, -13<=l<=13
Reflections collected 8603
Independent reflections 2848 [R(int) = 0.0191]
Completeness to theta = 27.00° 99.5 %
Absorption correction Semi-empirical from equivalents
Max. and min. transmission 0.9186 and 0.7295
Refinement method Full-matrix least-squares on \(F^2\)
Data / restraints / parameters 2848 / 0 / 237
Goodness-of-fit on \(F^2\) 1.039
Final R indices [I>2sigma(I)] R1 = 0.0348, wR2 = 0.0880
R indices (all data) R1 = 0.0411, wR2 = 0.0932
Largest diff. peak and hole 0.334 and -0.361 e.Å\(^{-3}\)

Table 2. Atomic coordinates (x 10\(^4\)) and equivalent isotropic displacement parameters (Å\(^2\)x 10\(^3\)) for \(9\) (M= Fe). \(U(eq)\) is defined as one third of the trace of the orthogonalized \(U^0\) tensor.

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>5000</td>
<td>5000</td>
<td>33(1)</td>
</tr>
<tr>
<td>Cl(1)</td>
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<td>6841(1)</td>
<td>2761(1)</td>
<td>35(1)</td>
</tr>
<tr>
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<td>6996(1)</td>
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</table>

Supplementary Material (ESI) for Chemical Communications
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### Table 3. Bond lengths [Å] and angles [°] for 9 (M= Fe).

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<tr>
<td>Fe(1)-O(2)</td>
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<tr>
<td>Fe(1)-O(5)#1</td>
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<tr>
<td>Fe(1)-O(5)</td>
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<tr>
<td>Cl(1)-O(8)</td>
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<tr>
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O(2)-C(6)  1.229(2)
O(3)-C(6)  1.359(2)
O(3)-C(9)  1.375(2)
O(4)-C(7)  1.348(2)
O(4)-C(2)  1.370(3)
O(5)-H(5A)  0.69(3)
O(5)-H(5B)  0.79(4)
C(1)-C(2)  1.481(3)
C(1)-H(1A)  0.95(3)
C(1)-H(1B)  0.89(3)
C(1)-H(1C)  0.95(3)
C(2)-C(3)  1.335(3)
C(3)-C(4)  1.425(3)
C(3)-H(3)  0.84(3)
C(4)-C(5)  1.448(3)
C(5)-C(7)  1.378(3)
C(5)-C(6)  1.437(3)
C(7)-C(8)  1.413(3)
C(8)-C(9)  1.328(3)
C(8)-H(8)  0.74(3)
C(9)-C(10)  1.471(3)
C(10)-H(9C)  0.94(3)
C(10)-H(10A)  0.95(3)
C(10)-H(10B)  1.01(4)
O(1)-Fe(1)-O(1)#1  180.00(8)
O(1)-Fe(1)-O(2)#1  94.25(6)
O(1)#1-Fe(1)-O(2)#1  85.75(6)
O(1)-Fe(1)-O(2)  85.75(6)
O(1)#1-Fe(1)-O(2)  94.25(6)
O(2)#1-Fe(1)-O(2)  180.0
O(1)-Fe(1)-O(5)#1  91.92(7)
O(1)#1-Fe(1)-O(5)#1  88.08(7)
O(2)#1-Fe(1)-O(5)#1  91.59(7)
O(2)-Fe(1)-O(5)#1  88.41(7)  
O(1)-Fe(1)-O(5)   88.08(7)  
O(1)#1-Fe(1)-O(5)  91.92(7)  
O(2)#1-Fe(1)-O(5)  88.41(7)  
O(2)-Fe(1)-O(5)   91.59(7)  
O(5)#1-Fe(1)-O(5) 180.0    
O(8A)-Cl(1)-O(6A) 116.5(9)  
O(8A)-Cl(1)-O(9)   107.2(6)  
O(6A)-Cl(1)-O(9)   100.6(5)  
O(8A)-Cl(1)-O(8)   131.9(5)  
O(6A)-Cl(1)-O(8)   80.9(6)   
O(9)-Cl(1)-O(8)    113.14(11) 
O(8A)-Cl(1)-O(7A) 119.8(8)  
O(6A)-Cl(1)-O(7A) 111.0(8)  
O(9)-Cl(1)-O(7A)   97.9(6)   
O(8)-Cl(1)-O(7A)   31.4(5)   
O(8A)-Cl(1)-O(6)   31.1(6)   
O(6A)-Cl(1)-O(6)   140.5(6)  
O(9)-Cl(1)-O(6)    110.30(13) 
O(8)-Cl(1)-O(6)    107.47(13) 
O(7A)-Cl(1)-O(6)   88.9(5)   
O(8A)-Cl(1)-O(7)   78.2(7)   
O(6A)-Cl(1)-O(7)   38.4(6)   
O(9)-Cl(1)-O(7)    111.72(12) 
O(8)-Cl(1)-O(7)    108.62(14) 
O(7A)-Cl(1)-O(7)   139.4(6)  
O(6)-Cl(1)-O(7)    105.20(13) 
C(4)-O(1)-Fe(1)    131.45(14) 
C(6)-O(2)-Fe(1)    129.19(13) 
C(6)-O(3)-C(9)     122.64(16) 
C(7)-O(4)-Cl(2)    119.63(16) 
Fe(1)-O(5)-H(5A)   115(3)    
Fe(1)-O(5)-H(5B)   119(2)    
H(5A)-O(5)-H(5B)   111(4)    
C(2)-C(1)-H(1A)    108.9(17) 
C(2)-C(1)-H(1B)    110.4(18)
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</tr>
<tr>
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</tr>
<tr>
<td>C(7)-C(8)-H(8)</td>
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</tr>
<tr>
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</tr>
<tr>
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<tr>
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</tr>
<tr>
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<td>112.1(15)</td>
</tr>
<tr>
<td>C(9)-C(10)-H(10A)</td>
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</tr>
<tr>
<td>H(9C)-C(10)-H(10A)</td>
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</tr>
<tr>
<td>C(9)-C(10)-H(10B)</td>
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<tr>
<td>H(9C)-C(10)-H(10B)</td>
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</tr>
<tr>
<td>H(10A)-C(10)-H(10B)</td>
<td>109(3)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1

Table 4. Anisotropic displacement parameters (Å² x 10³) for $9$ (M= Fe). The anisotropic displacement factor exponent takes the form: $-2p²[ h²a⁺²U¹¹ + ... + 2 h k a⁺ b⁺ U¹² ]$

<table>
<thead>
<tr>
<th></th>
<th>$U¹¹$</th>
<th>$U²²$</th>
<th>$U³³$</th>
<th>$U²³$</th>
<th>$U¹³$</th>
<th>$U¹²$</th>
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<td>-1(1)</td>
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<tr>
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<td>33(1)</td>
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<td>-2(1)</td>
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<td>1(1)</td>
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<td>1(1)</td>
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<td>37(1)</td>
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<td>2(1)</td>
<td>3(1)</td>
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<td>35(1)</td>
<td>-3(1)</td>
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<td>2(1)</td>
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<tr>
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<td>2(1)</td>
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<td>-7(1)</td>
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<tr>
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<td>34(1)</td>
<td>-2(1)</td>
<td>-4(1)</td>
<td>5(1)</td>
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<tr>
<td>C(10)</td>
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<td>40(1)</td>
<td>9(1)</td>
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<td>-2(1)</td>
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Table 5. Hydrogen coordinates ($x \times 10^4$) and isotropic displacement parameters ($\AA^2 \times 10^3$) for 9 ($M = \text{Fe}$).

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<tr>
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<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
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</thead>
<tbody>
<tr>
<td>H(1A)</td>
<td>5960(30)</td>
<td>8050(20)</td>
<td>7430(30)</td>
<td>60(8)</td>
</tr>
<tr>
<td>H(1B)</td>
<td>5840(30)</td>
<td>7900(20)</td>
<td>5970(30)</td>
<td>57(8)</td>
</tr>
<tr>
<td>H(1C)</td>
<td>6630(30)</td>
<td>7120(20)</td>
<td>6850(30)</td>
<td>57(8)</td>
</tr>
<tr>
<td>H(3)</td>
<td>3810(20)</td>
<td>6916(19)</td>
<td>5090(30)</td>
<td>44(7)</td>
</tr>
<tr>
<td>H(5A)</td>
<td>750(30)</td>
<td>3200(30)</td>
<td>4310(30)</td>
<td>66(12)</td>
</tr>
<tr>
<td>H(5B)</td>
<td>1720(30)</td>
<td>3440(30)</td>
<td>5050(30)</td>
<td>69(10)</td>
</tr>
<tr>
<td>H(8)</td>
<td>3950(30)</td>
<td>5830(20)</td>
<td>10100(30)</td>
<td>51(8)</td>
</tr>
<tr>
<td>H(9C)</td>
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<td>4884(19)</td>
<td>11840(30)</td>
<td>41(7)</td>
</tr>
<tr>
<td>H(10A)</td>
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<td>3860(30)</td>
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<td>66(9)</td>
</tr>
<tr>
<td>H(10B)</td>
<td>1330(40)</td>
<td>4820(20)</td>
<td>11440(30)</td>
<td>82(10)</td>
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</table>

Table 6. Torsion angles [$^\circ$] for 9 ($M = \text{Fe}$).

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<th></th>
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</thead>
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</tr>
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<td>O(5)#1-Fe(1)-O(1)-C(4)</td>
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<tr>
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<td>-92.41(19)</td>
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<tr>
<td>O(1)-Fe(1)-O(2)-C(6)</td>
<td>-0.09(18)</td>
</tr>
<tr>
<td>O(1)#1-Fe(1)-O(2)-C(6)</td>
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</tr>
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<td>O(2)-Fe(1)-O(2)-C(6)</td>
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<td>O(5)-Fe(1)-O(2)-C(6)</td>
<td>87.87(18)</td>
</tr>
<tr>
<td>C(7)-O(4)-C(2)-C(3)</td>
<td>-0.1(3)</td>
</tr>
<tr>
<td>C(7)-O(4)-C(2)-C(1)</td>
<td>-179.57(18)</td>
</tr>
<tr>
<td>O(4)-C(2)-C(3)-C(4)</td>
<td>-0.4(3)</td>
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<td>C(1)-C(2)-C(3)-C(4)</td>
<td>179.1(2)</td>
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<tr>
<td>Fe(1)-O(1)-C(4)-C(3)</td>
<td>-179.51(14)</td>
</tr>
<tr>
<td>Fe(1)-O(1)-C(4)-C(5)</td>
<td>0.8(3)</td>
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</tbody>
</table>
C(2)-C(3)-C(4)-O(1)  -179.4(2)
C(2)-C(3)-C(4)-C(5)  0.3(3)
O(1)-C(4)-C(5)-C(7)  179.82(19)
C(3)-C(4)-C(5)-C(7)  0.1(3)
O(1)-C(4)-C(5)-C(6)  -0.1(3)
C(3)-C(4)-C(5)-C(6)  -179.81(18)
Fe(1)-O(2)-C(6)-O(3)  -179.44(11)
Fe(1)-O(2)-C(6)-C(5)  0.7(3)
C(9)-O(3)-C(6)-O(2)  -179.68(17)
C(9)-O(3)-C(6)-C(5)  0.2(3)
C(7)-C(5)-C(6)-O(2)  179.39(17)
C(4)-C(5)-C(6)-O(2)  -0.7(3)
C(7)-C(5)-C(6)-O(3)  -0.5(3)
C(4)-C(5)-C(6)-O(3)  179.45(17)
C(2)-O(4)-C(7)-C(5)  0.5(3)
C(2)-O(4)-C(7)-C(8)  -179.89(17)
C(6)-C(5)-C(7)-O(4)  179.38(17)
C(4)-C(5)-C(7)-O(4)  -0.6(3)
C(6)-C(5)-C(7)-C(8)  -0.2(3)
C(4)-C(5)-C(7)-C(8)  179.88(18)
O(4)-C(7)-C(8)-C(9)  -178.43(18)
C(5)-C(7)-C(8)-C(9)  1.2(3)
C(7)-C(8)-C(9)-O(3)  -1.4(3)
C(7)-C(8)-C(9)-C(10)  177.4(2)
C(6)-O(3)-C(9)-C(8)  0.8(3)
C(6)-O(3)-C(9)-C(10)  -178.24(18)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1

Table 7. Hydrogen bonds for 9 (M= Fe) [Å and °].

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>0.69(3)</td>
<td>2.19(4)</td>
<td>2.871(4)</td>
<td>170(4)</td>
</tr>
<tr>
<td>O(5)-H(5A)...O(7A)#2</td>
<td>0.69(3)</td>
<td>2.30(4)</td>
<td>2.796(13)</td>
<td>130(4)</td>
</tr>
<tr>
<td>Bond</td>
<td>d (Å)</td>
<td>r (Å)</td>
<td>D (Å)</td>
<td>ϕ (°)</td>
</tr>
<tr>
<td>-----------------------------</td>
<td>-------</td>
<td>--------</td>
<td>-----------</td>
<td>--------</td>
</tr>
<tr>
<td>O(5)-H(5B)...O(6A)#3</td>
<td>0.79(4)</td>
<td>1.99(4)</td>
<td>2.771(12)</td>
<td>171(3)</td>
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<tr>
<td>O(5)-H(5B)...O(7)#3</td>
<td>0.79(4)</td>
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<td>2.796(3)</td>
<td>160(3)</td>
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</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1
#2 -x+1/2,y-1/2,-z+1/2
#3 -x+1,-y+1,-z+1

**Ba$_2$-(tetrakis)bispyrone Complex 10**

NMR- $\delta ^1$H (DMSO) 2.42 s (3H), 6.32 s (1H), δ $^{13}$C 18.88, 106.33, 114.82, 161.86, 167.88, 175.87; IR (KBr) cm$^{-1}$ 1671.7, 1647.7, 1462.5, 1254.7, 1181.1, 1118.9, 627.9. HRMS – calcd 620.9383 found 620.9388.
Table 1. Crystal data and structure refinement for 10.

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<td>Property</td>
<td>Value</td>
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<td>--------------------------------</td>
<td>-------------</td>
</tr>
<tr>
<td>Temperature</td>
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<tr>
<td>Wavelength</td>
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<td>Crystal system</td>
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<td>Space group</td>
<td>P-1</td>
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<td>Unit cell dimensions</td>
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<tr>
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<tr>
<td>b</td>
<td>10.2248(15) Å</td>
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<tr>
<td>c</td>
<td>12.9429(19) Å</td>
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<td>Volume</td>
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<td>Absorption coefficient</td>
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<td>F(000)</td>
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<tr>
<td>Crystal size</td>
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<tr>
<td>Independent reflections</td>
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<td>Semi-empirical from equivalents</td>
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<td>0.8684 and 0.5379</td>
</tr>
<tr>
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<td>Full-matrix least-squares on F²</td>
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<tr>
<td>Data / restraints / parameters</td>
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<tr>
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<tr>
<td>R indices (all data)</td>
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</tr>
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Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for 10. U(eq) is defined as one third of the trace of the orthogonalized U^ij tensor.

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<th>z</th>
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C(9)-C(10)-H(10B) 112(2)
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H(10B)-C(10)-H(10C) 109(3)
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C(12)-C(13)-C(14) 122.1(2)
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Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+2,-z+1
Table 4. Anisotropic displacement parameters (Å²x 10³) for 10. The anisotropic displacement factor exponent takes the form: -2p² [ h²a*²U¹¹ + ... + 2 h k a* b* U¹² ]

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C(10)  43(1)  29(1)  41(1)  11(1)  13(1)  4(1)  
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C(19)  27(1)  24(1)  34(1)  5(1)  7(1)  4(1) 
C(20)  33(1)  37(1)  49(2)  21(1)  9(1)  5(1) 

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **10**.

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Table 6. Torsion angles [°] for 10.

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<td>11.6(4)</td>
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O(13)-Ba(1)-O(5)-C(14) 10.4(3)
O(14A)-Ba(1)-O(5)-C(14) 45.5(3)
O(17)-Ba(1)-O(6)-C(17) 34.2(2)
O(2)-Ba(1)-O(6)-C(17) 114.81(18)
O(5)-Ba(1)-O(6)-C(17) 55.49(17)
O(1)-Ba(1)-O(6)-C(17) 171.16(19)
O(18)-Ba(1)-O(6)-C(17) -117.43(18)
O(13A)-Ba(1)-O(6)-C(17) -40.2(4)
O(9)-Ba(1)-O(6)-C(17) -70.6(2)
O(10)#1-Ba(1)-O(6)-C(17) 172.37(16)
O(13)-Ba(1)-O(6)-C(17) -44.2(3)
O(14A)-Ba(1)-O(6)-C(17) -8.9(3)
O(11)-Cl(1)-O(9)-Ba(1) -117.6(2)
O(12)-Cl(1)-O(9)-Ba(1) 2.9(2)
O(17)-Ba(1)-O(9)-Cl(1) 32.94(19)
O(2)-Ba(1)-O(9)-Cl(1) -24.1(2)
O(5)-Ba(1)-O(9)-Cl(1) 67.7(2)
O(6)-Ba(1)-O(9)-Cl(1) 162.20(17)
O(1)-Ba(1)-O(9)-Cl(1) -100.3(2)
O(18)-Ba(1)-O(9)-Cl(1) -148.1(2)
O(13A)-Ba(1)-O(9)-Cl(1) 134.4(4)
O(10)#1-Ba(1)-O(9)-Cl(1) -53.19(19)
O(13)-Ba(1)-O(9)-Cl(1) 137.7(3)
O(14A)-Ba(1)-O(9)-Cl(1) 96.6(2)
O(9)-Cl(1)-O(10)-Ba(1)#1 -125.28(12)
O(11)-Cl(1)-O(10)-Ba(1)#1 114.78(14)
O(12)-Cl(1)-O(10)-Ba(1)#1 -5.73(16)
O(15A)-Cl(2)-O(13)-Ba(1) 115.0(13)
O(16)-Cl(2)-O(13)-Ba(1) -111.4(10)
O(13A)-Cl(2)-O(13)-Ba(1) -31(6)
O(14)-Cl(2)-O(13)-Ba(1) 21.2(10)
O(15)-Cl(2)-O(13)-Ba(1) 134.2(6)
O(14A)-Cl(2)-O(13)-Ba(1) -9.5(10)
O(16A)-Cl(2)-O(13)-Ba(1) -119.4(11)
O(17)-Ba(1)-O(13)-Cl(2) -9.5(8)
O(2)-Ba(1)-O(13)-Cl(2)  66.0(11)
O(5)-Ba(1)-O(13)-Cl(2)  61.8(7)
O(6)-Ba(1)-O(13)-Cl(2)  122.6(8)
O(1)-Ba(1)-O(13)-Cl(2)  164.3(6)
O(18)-Ba(1)-O(13)-Cl(2) -149.7(8)
O(13A)-Ba(1)-O(13)-Cl(2)  43(7)
O(9)-Ba(1)-O(13)-Cl(2) -75.8(7)
O(10)#1-Ba(1)-O(13)-Cl(2) -91.7(8)
O(14A)-Ba(1)-O(13)-Cl(2)  6.9(8)
O(15A)-Cl(2)-O(13A)-Ba(1)  104.7(17)
O(16)-Cl(2)-O(13A)-Ba(1) -114.7(11)
O(14)-Cl(2)-O(13A)-Ba(1)  9.8(12)
O(13)-Cl(2)-O(13A)-Ba(1)  141(7)
O(15)-Cl(2)-O(13A)-Ba(1)  126.2(9)
O(14A)-Cl(2)-O(13A)-Ba(1) -16.7(11)
O(16A)-Cl(2)-O(13A)-Ba(1) -124.4(13)
O(17)-Ba(1)-O(13A)-Cl(2) -0.2(10)
O(2)-Ba(1)-O(13A)-Cl(2)  72.2(12)
O(5)-Ba(1)-O(13A)-Cl(2)  70.8(10)
O(6)-Ba(1)-O(13A)-Cl(2)  133.1(11)
O(1)-Ba(1)-O(13A)-Cl(2)  170.9(8)
O(18)-Ba(1)-O(13A)-Cl(2) -140.6(10)
O(9)-Ba(1)-O(13A)-Cl(2) -67.7(9)
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O(14A)-Ba(1)-O(13A)-Cl(2)  12.9(9)
O(15A)-Cl(2)-O(14A)-Ba(1) -110.6(9)
O(16)-Cl(2)-O(14A)-Ba(1)  122.9(9)
O(13A)-Cl(2)-O(14A)-Ba(1)  11.6(8)
O(14)-Cl(2)-O(14A)-Ba(1) -130.1(13)
O(13)-Cl(2)-O(14A)-Ba(1)  7.9(9)
O(15)-Cl(2)-O(14A)-Ba(1) -97.9(10)
O(16A)-Cl(2)-O(14A)-Ba(1)  113.6(12)
O(17)-Ba(1)-O(14A)-Cl(2)  155.3(11)
O(2)-Ba(1)-O(14A)-Cl(2) -160.1(7)
O(5)-Ba(1)-O(14A)-Cl(2) -122.1(10)
O(6)-Ba(1)-O(14A)-Cl(2)  -60.9(9)
O(1)-Ba(1)-O(14A)-Cl(2)  -60.7(14)
O(18)-Ba(1)-O(14A)-Cl(2)  17.2(10)
O(13A)-Ba(1)-O(14A)-Cl(2)  -9.7(7)
O(9)-Ba(1)-O(14A)-Cl(2)  80.2(9)
O(10)#1-Ba(1)-O(14A)-Cl(2)  118.1(8)
O(13)-Ba(1)-O(14A)-Cl(2)  -6.0(7)
C(6)-O(3)-C(2)-C(3)  -1.0(3)
C(6)-O(3)-C(2)-C(1)  178.7(2)
O(3)-C(2)-C(3)-C(4)  1.0(4)
C(1)-C(2)-C(3)-C(4)  -178.7(3)
Ba(1)-O(1)-C(4)-C(3)  -141.82(18)
Ba(1)-O(1)-C(4)-C(5)  36.5(3)
C(2)-C(3)-C(4)-O(1)  -176.5(2)
C(2)-C(3)-C(4)-C(5)  -1.9(3)
O(1)-C(4)-C(5)-C(6)  -175.6(2)
C(3)-C(4)-C(5)-C(6)  -178.0(2)
O(1)-C(4)-C(5)-C(7)  -0.4(3)
C(3)-C(4)-C(5)-C(7)  178.0(2)
C(2)-O(3)-C(6)-O(4)  -176.71(19)
C(2)-O(3)-C(6)-C(5)  2.2(3)
C(9)-O(4)-C(6)-O(3)  179.63(17)
C(9)-O(4)-C(6)-C(5)  0.7(3)
C(4)-C(5)-C(6)-O(3)  -3.2(3)
C(7)-C(5)-C(6)-O(3)  -178.7(2)
C(4)-C(5)-C(6)-O(4)  175.57(19)
C(7)-C(5)-C(6)-O(4)  0.0(3)
Ba(1)-O(2)-C(7)-C(8)  135.42(18)
Ba(1)-O(2)-C(7)-C(5)  -45.1(3)
C(6)-C(5)-C(7)-O(2)  179.9(2)
C(4)-C(5)-C(7)-O(2)  4.7(3)
C(6)-C(5)-C(7)-C(8)  -0.5(3)
C(4)-C(5)-C(7)-C(8)  -175.7(2)
O(2)-C(7)-C(8)-C(9)  179.9(2)
C(5)-C(7)-C(8)-C(9)  0.4(3)
C(7)-C(8)-C(9)-O(4)  0.3(3)
C(7)-C(8)-C(9)-C(10)  -179.8(2)
C(6)-O(4)-C(9)-C(8)  -0.9(3)
C(6)-O(4)-C(9)-C(10)  179.3(2)
C(16)-O(7)-C(12)-C(13)  -0.1(3)
C(16)-O(7)-C(12)-C(11)  179.0(2)
O(7)-C(12)-C(13)-C(14)  2.4(3)
C(11)-C(12)-C(13)-C(14)  -176.5(2)
Ba(1)-O(5)-C(14)-C(13)  -136.66(18)
Ba(1)-O(5)-C(14)-C(15)  41.2(3)
C(12)-C(13)-C(14)-O(5)  173.8(2)
C(12)-C(13)-C(14)-C(15)  -4.2(3)
O(5)-C(14)-C(15)-C(16)  179.3(2)
C(13)-C(14)-C(15)-C(16)  3.8(3)
O(5)-C(14)-C(15)-C(17)  1.3(3)
C(13)-C(14)-C(15)-C(17)  179.3(2)
C(12)-O(7)-C(16)-O(8)  178.35(17)
C(12)-O(7)-C(16)-C(15)  -0.2(3)
C(19)-O(8)-C(16)-O(7)  176.82(17)
C(19)-O(8)-C(16)-C(15)  -1.4(3)
C(14)-C(15)-C(16)-O(7)  178.5(2)
C(17)-C(15)-C(16)-O(7)  -1.6(4)
C(17)-C(15)-C(16)-O(8)  0.2(3)
Ba(1)-O(6)-C(17)-C(18)  179.2(2)
C(16)-O(8)-C(17)-O(6)  179.2(2)
C(14)-C(15)-C(17)-O(6)  3.7(3)
C(16)-C(15)-C(17)-C(18)  0.3(3)
C(14)-C(15)-C(17)-C(18)  -175.2(2)
O(6)-C(17)-C(18)-C(19)  -178.5(2)
C(15)-C(17)-C(18)-C(19)  0.4(3)
C(17)-C(18)-C(19)-O(8)  2.1(3)
C(16)-O(8)-C(19)-C(20)  -176.4(2)
Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+2,-z+1

Table 7. Hydrogen bonds for 10 [Å and °].

<table>
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<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
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<td>2.996(3)</td>
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<td>O(18)-H(1O)...O(18)#2</td>
<td>0.80(5)</td>
<td>2.64(4)</td>
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<td>0.74(4)</td>
<td>2.41(4)</td>
<td>3.056(3)</td>
<td>146(4)</td>
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Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+2,-z+1  #2 -x,-y+1,-z+1