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

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SUPPORTING INFORMATION

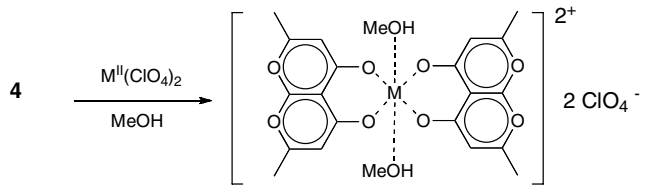
General techniques:

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_3OD solutions in 5 mm diameter tubes, and chemical shifts in ppm are quoted relative to the residual signals of the solvent used DMSO (δH 2.51 ppm or δC 39.9) chloroform (δH 7.26 ppm, or δC 77.0 ppm) methanol (δH 3.31, 4.87 δC 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**.

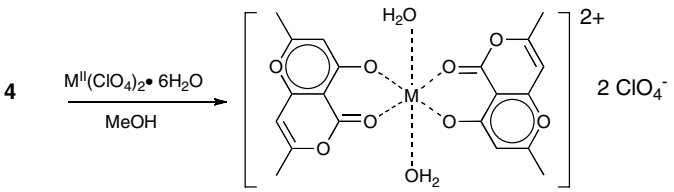
	Reagent	Product	λ_{max}(nm) (ε 10^3)	Color
1	$Mg(ClO_4)_2$	**7** (M=Mg)	303 (19.2)	Colorless
2	$Ni(ClO_4)_2$•6MeCN	**7** (M=Ni)	304 (4.48)	Light Blue
3	$Fe(ClO_4)_2$•6MeCN	**7** (M=Fe)	301 (5.99)	Orange
4	$Li(ClO_4)_2$	**8** (M=Li)	[a]	Colorless
5	$Cu(ClO_4)_2$•$6H_2O$	**9** (M=Cu)	318 (5.54)	Light Blue
6	$Co(ClO_4)_2$•$6H_2O$	**9** (M=Co)	298 (30.0)	Red
7	$Ni(ClO_4)_2$•$6H_2O$	**9** (M=Ni)	310 (1.00)	Light Blue
8	$Zn(ClO_4)_2$•$6H_2O$	**9** (M=Zn)	299 (17.1)	Colorless
9	$Fe(ClO_4)_2$•$6H_2O$	**9** (M=Fe)	300 (3.44)	Red
10	$Ba(ClO_4)_2$	**10** (M=Ba)	300 (0.95)	Colorless

a] not determined.



7 (M = Mg, Ni, Fe)

Fig. 3. Reaction of Fused bispyrone **4** with divalent metal perchlorates.



9 (M = Cu, Co, Ni Fe, Zn)

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 μ=0.5. Other H atoms in **6** were found on the residual density and refined with isotropic thermal parameters. 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 CH_3OH 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_2O)**: $C_{10}H_{10}O_5$, 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, β = 104.521(1)°, V = 1957.97(11) Å^3, Z = 8, D_c = 1.426 Mg/m^3, μ = 0.116 mm^{-1}, F(000) = 880, $2\theta_{max}$ = 54.00°, 19210 reflections, 4263 independent reflections [R_{int} = 0.0315], R1 = 0.0450, wR2 = 0.1021 and GOF = 1.025 for 4263 reflections (351 parameters) with I>2σ(I), R1 = 0.0783, wR2 = 0.1219 and GOF = 1.025 for all reflections, max/min residual electron density +0.184/-0.193 eÅ^3.

Crystallographic Data for **6**: $C_{10}H_9ClO_8$, 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, μ = 0.375 mm^{-1}, F(000) = 600, $2\theta_{max}$ = 54.00°, 12297 reflections, 1291 independent reflections [R_{int} = 0.0324], R1 = 0.0363, wR2 = 0.0974 and GOF = 1.086 for 1291 reflections (114 parameters) with I>2σ(I), R1 = 0.0411, wR2 = 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_2O**: $C_{22}H_{26}Cl_2MgO_{19}$, 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, β = 103.023(3)°, V = 2908.7(10) Å^3, Z = 4, Z'=2, D_c = 1.575 Mg/m^3, μ = 0.331 mm^{-1}, F(000) = 1424, $2\theta_{max}$ = 50.00°, 27023 reflections, 5118 independent reflections [R_{int} = 0.0436], R1 = 0.0579, wR2 = 0.1501 and GOF = 1.065 for 5118 reflections (500 parameters) with I>2σ(I), R1 = 0.0794, wR2 = 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)**:** $C_{22}H_{24}Cl_2NiO_{18}$, 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(4) (3) Å, c = 13.476(4) Å, β = 95.849(5)°, V = 1441.6(7) Å^3, Z = 2, Z'=0.5 D_c = 1.627 Mg/m^3, μ = 0.940 mm^{-1}, F(000) = 724, $2\theta_{max}$ = 54.00°, 9624 reflections, 3129 independent reflections [R_{int} = 0.0306], R1 = 0.0428, wR2 = 0.1012 and GOF = 1.040 for 3129 reflections (254 parameters) with I>2σ(I), R1 = 0.0542, wR2 = 0.1099 and GOF = 1.044 for all reflections, max/min residual electron density +0.733/-0.530 eÅ^3.

Crystallographic Data for **8**: $C_{32.50}H_{32}Cl_2Li_2O_{22.50}$, 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, α = 79.690(2)°, β = 87.173(2)°, γ = 76.390(2)°, V = 3832.0(9) Å^3, Z = 4, Z'=2, D_c = 1.503 Mg/m^3, μ = 0.259 mm^{-1}, F(000) = 1788, $2\theta_{max}$ = 50.00°, 37382 reflections, 13455 independent reflections [R_{int} = 0.0402], R1 = 0.0556, wR2 = 0.1523 and GOF = 1.033 for 13455 reflections (1219 parameters) with I>2σ(I), R1 = 0.0898, wR2 = 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): $C_{20}H_{20}Cl_2CuO_{18}$, 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, β = 95.359(2)°, V = 1309.2(2) Å^3, Z = 2, Z'=0.5, D_c = 1.732

Mg/m^3, μ = 1.125 mm^{-1}, F(000) = 694, $2\theta_{max}$ = 54.00°, 14330 reflections, 2858 independent reflections [R_{int} = 0.0158], R1 = 0.0238, wR2 = 0.0690 and GOF = 1.048 for 2858 reflections (227 parameters) with I>2σ(I), R1 = 0.0251, wR2 = 0.0702 and GOF = 1.048 for all reflections, max/min residual electron density +0.312/-0.229 eÅ^3.

Crystallographic Data for **9** (M=Co): $C_{20}H_{20}Cl_2CoO_{18}$, 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, β = 95.922(9)°, V = 1297.7(14) Å^3, Z = 2, Z'=0.5, D_c = 1.736 Mg/m^3, μ = 0.955 mm^{-1}, F(000) = 690, $2\theta_{max}$ = 50.00°, 11277 reflections, 2279 independent reflections [R_{int} = 0.0425], R1 = 0.0509, wR2 = 0.1218 and GOF = 1.055 for 2279 reflections (227 parameters) with I>2σ(I), R1 = 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): $C_{20}H_{20}Cl_2NiO_{18}$, 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, β = 95.348(4)°, V = 1307.0(6) Å^3, Z = 2, Z'=0.5, D_c = 1.723 Mg/m^3, μ = 1.033 mm^{-1}, F(000) = 692, $2\theta_{max}$ = 54.00°, 12556 reflections, 2852 independent reflections [R_{int} = 0.0295], R1 = 0.0295, wR2 = 0.0723 and GOF = 1.066 for 2852 reflections (227 parameters) with I>2σ(I), R1 = 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): $C_{20}H_{20}Cl_2ZnO_{18}$, 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, β = 95.500(4)°, V = 1308.5(6) Å^3, Z = 2, Z'=0.5, D_c = 1.738 Mg/m^3, μ = 1.229 mm^{-1}, F(000) = 696, $2\theta_{max}$ = 54.00°, 14755 reflections, 2855 independent reflections [R_{int} = 0.0382], R1 = 0.0387, wR2 = 0.0944 and GOF = 1.082 for 2855 reflections (227 parameters) with I>2σ(I), R1 = 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): $C_{20}H_{20}Cl_2FeO_{18}$, 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, β = 95.402(4)°, V = 1314.5(5) Å^3, Z = 2, Z'=0.5, D_c = 1.706 Mg/m^3, μ = 0.863 mm^{-1}, F(000) = 688, $2\theta_{max}$ = 54.00°, 8603 reflections, 2848 independent reflections [R_{int} = 0.0191], R1 = 0.0348, wR2 = 0.0880 and GOF = 1.039 for 2848 reflections (237 parameters) with I>2σ(I), R1 = 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**: $C_{20}H_{20}Cl_2BaO_{18}$, 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, α = 92.527(2)°, β = 99.751(2)°, γ = 97.818(2)°, V = 1303.9(3) Å^3, Z = 2, D_c = 1.927 Mg/m^3, μ = 1.815 mm^{-1}, F(000) = 748, $2\theta_{max}$ = 54.00°, 14739 reflections, 5654 independent reflections [R_{int} = 0.0153], R1 = 0.0205, wR2 = 0.0635 and GOF = 1.186 for 5654 reflections (479 parameters) with I>2σ(I), R1 = 0.0209, wR2 = 0.0639

and GOF = 1.190 for all reflections, max/min residual electron density +0.543/-0.453 eÅ^3.

References:

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

2 G. M. Sheldrick, (1995). *SADABS*, University of Göttingen, Germany.

3 G. M. Sheldrick, (2008). *Acta Cryst.* A64, 112-122.

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-pyrone **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 ∂_H (DMSO) 2.29 s (6H), 6.22 s (1H), 6.58 s (1H) 8.90 br s (1H), ∂_C 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 $NaHCO_3$ (143 mg 1.71 mmol) in H_2O (7 mL) and the mixture was extracted with $CHCl_3$, dried over Na_2SO_4 and evaporated to afford solid bis-γ-pyrone **4** as a colorless solid. The solid was recrystallized from either benzene or $CHCl_3$-cyclohexane to afford **4** as colorless needles. NMR – [ppm ∂_H ($CDCl_3$) 2.33 s (3H), 6.18 s (1H), ∂_C 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). λ_{max} 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 ∂_H (DMSO) 2.29 s (6H), 6.22 s (1H), 6.58 s (1H) 8.90 br s (1H), ∂_C 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:

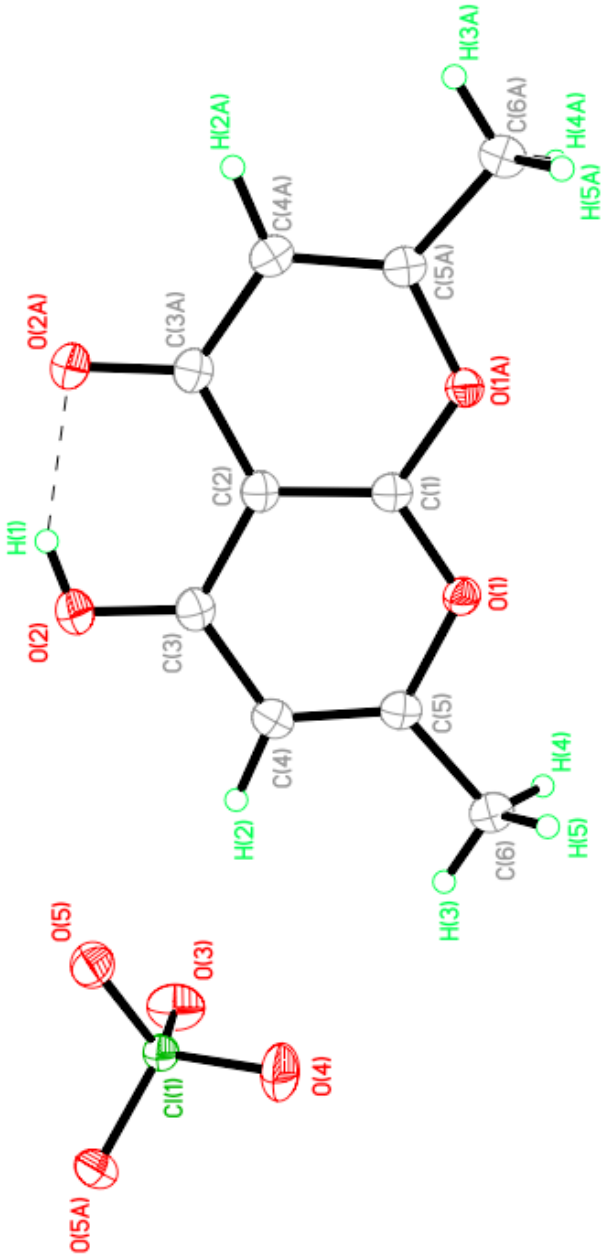


Table 1. Crystal data and structure refinement for **6**.

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) Å	a= 90°.
	b = 12.552(4) Å	b= 90°.
	c = 8.820(3) Å	g = 90°.
Volume	1128.1(7) $Å^3$	
Z	4	
Density (calculated)	1.723 Mg/m^3	
Absorption coefficient	0.375 mm^{-1}	
F(000)	600	
Crystal size	0.38 x 0.11 x 0.10 mm^3	
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 F^2	
Data / restraints / parameters	1291 / 0 / 114	
Goodness-of-fit on F^2	1.086	
Final R indices [I>2sigma(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 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **6**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	7547(1)	2500	364(1)	28(1)
O(1)	4911(1)	6632(1)	2401(1)	27(1)
O(2)	8069(1)	6471(1)	-398(2)	38(1)
O(3)	8367(2)	2500	1667(2)	53(1)

O(4)	6200(2)	2500	822(3)	55(1)
O(5)	7791(2)	3435(1)	-535(2)	45(1)
C(1)	5489(2)	7500	1898(2)	27(1)
C(2)	6554(2)	7500	959(3)	28(1)
C(3)	7081(2)	6501(1)	476(2)	30(1)
C(4)	6443(2)	5568(1)	1030(2)	31(1)
C(5)	5395(2)	5652(1)	1964(2)	28(1)
C(6)	4628(2)	4773(2)	2626(2)	34(1)

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

Cl(1)-O(3)	1.421(2)
Cl(1)-O(4)	1.431(2)
Cl(1)-O(5)#1	1.4378(14)
Cl(1)-O(5)	1.4378(14)
O(1)-C(1)	1.3157(17)
O(1)-C(5)	1.3810(19)
O(2)-C(3)	1.268(2)
O(2)-H(1)	0.81(4)
C(1)-O(1)#2	1.3157(17)
C(1)-C(2)	1.365(3)
C(2)-C(3)#2	1.429(2)
C(2)-C(3)	1.429(2)
C(3)-C(4)	1.426(2)
C(4)-C(5)	1.353(2)
C(4)-H(2)	0.97(2)
C(5)-C(6)	1.472(2)
C(6)-H(3)	0.92(2)
C(6)-H(4)	0.97(2)
C(6)-H(5)	0.98(3)
O(3)-Cl(1)-O(4)	109.63(14)
O(3)-Cl(1)-O(5)#1	110.15(8)
O(4)-Cl(1)-O(5)#1	108.77(8)
O(3)-Cl(1)-O(5)	110.15(8)

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)
C(4)-C(5)-O(1)	121.40(14)
C(4)-C(5)-C(6)	127.03(16)
O(1)-C(5)-C(6)	111.57(14)
C(5)-C(6)-H(3)	106.1(14)
C(5)-C(6)-H(4)	109.0(14)
H(3)-C(6)-H(4)	112.2(18)
C(5)-C(6)-H(5)	108.0(13)
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 ($Å^2 x\ 10^3$) for **6**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	27(1)	28(1)	29(1)	0	2(1)	0
O(1)	23(1)	27(1)	32(1)	1(1)	0(1)	-1(1)

O(2)	34(1)	39(1)	41(1)	-2(1)	10(1)	3(1)
O(3)	61(1)	51(1)	47(1)	0	-21(1)	0
O(4)	35(1)	69(1)	61(1)	0	17(1)	0
O(5)	59(1)	34(1)	43(1)	9(1)	13(1)	4(1)
C(1)	25(1)	28(1)	28(1)	0	-6(1)	0
C(2)	23(1)	32(1)	28(1)	0	-3(1)	0
C(3)	25(1)	36(1)	30(1)	-1(1)	-3(1)	0(1)
C(4)	30(1)	31(1)	31(1)	-3(1)	-4(1)	2(1)
C(5)	27(1)	29(1)	29(1)	-1(1)	-7(1)	0(1)
C(6)	37(1)	31(1)	35(1)	2(1)	-2(1)	-3(1)

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

	x	y	z	U(eq)
H(1)	8280(40)	7070(30)	-600(40)	42(13)
H(2)	6730(19)	4861(16)	740(20)	34(5)
H(3)	5020(20)	4151(19)	2290(20)	43(6)
H(4)	4640(20)	4836(18)	3730(30)	52(6)
H(5)	3720(30)	4844(17)	2280(20)	51(6)

Table 6. Torsion angles [°] for **6**.

C(5)-O(1)-C(1)-O(1)#2	179.45(11)
C(5)-O(1)-C(1)-C(2)	-0.1(3)
O(1)-C(1)-C(2)-C(3)#2	179.15(18)
O(1)#2-C(1)-C(2)-C(3)#2	-0.3(3)
O(1)-C(1)-C(2)-C(3)	0.3(3)
O(1)#2-C(1)-C(2)-C(3)	-179.15(18)
C(1)-C(2)-C(3)-O(2)	179.93(18)
C(3)#2-C(2)-C(3)-O(2)	1.2(3)
C(1)-C(2)-C(3)-C(4)	-0.3(3)
C(3)#2-C(2)-C(3)-C(4)	-179.03(14)

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 °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2)-H(1)...O(2)#2	0.81(4)	1.85(4)	2.583(3)	151(4)

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 ∂_H ($CDCl_3$) 2.33 s (3H), 6.18 s (1H), ∂_C 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). λ_{max} 300 nm.

Crystal Structure:

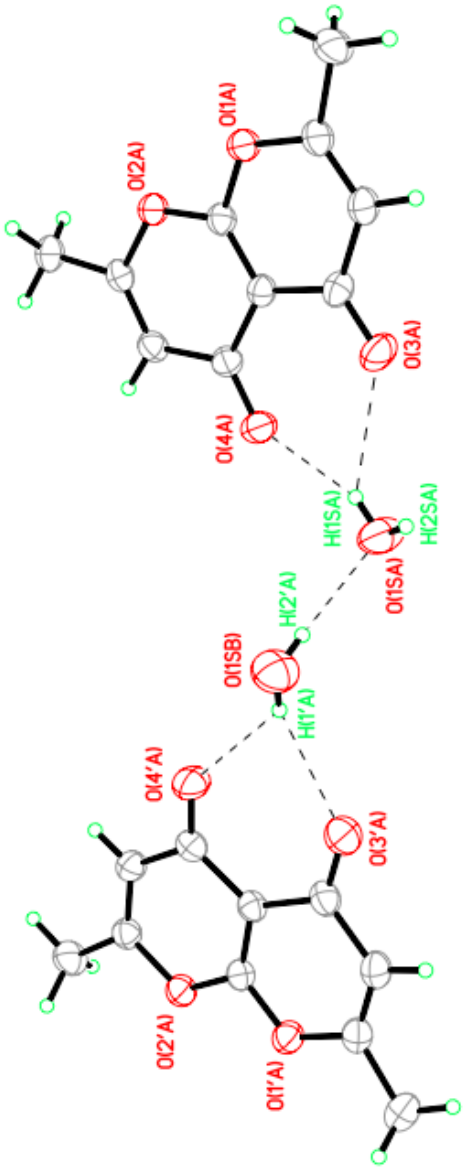


Table 1. Crystal data and structure refinement for **4**.

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	α= 90°.
	b = 25.5598(9) Å	β= 104.5210(10)°.
	c = 11.1908(4) Å	γ = 90°.
Volume	1957.97(11) $Å^3$	
Z	8	
Density (calculated)	1.426 Mg/m^3	
Absorption coefficient	0.116 mm^{-1}	
F(000)	880	
Crystal size	0.27 x 0.25 x 0.06 mm^3	
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 F^2	
Data / restraints / parameters	4263 / 0 / 351	
Goodness-of-fit on F^2	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 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **4**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2017(2)	3837(1)	9586(1)	56(1)
O(2)	1304(2)	3067(1)	8855(1)	50(1)
O(3)	-230(2)	4589(1)	6362(1)	72(1)
O(4)	-1466(2)	3562(1)	5420(1)	66(1)

C(1)	1160(2)	3574(1)	8573(2)	44(1)
C(2)	2047(3)	4379(1)	9535(2)	56(1)
C(3)	1266(3)	4625(1)	8486(2)	57(1)
C(4)	379(3)	4356(1)	7344(2)	52(1)
C(5)	318(2)	3784(1)	7454(2)	43(1)
C(6)	-567(3)	3422(1)	6459(2)	47(1)
C(7)	-337(3)	2872(1)	6796(2)	49(1)
C(8)	521(2)	2711(1)	7931(2)	44(1)
C(10)	3066(5)	4595(1)	10752(2)	75(1)
C(11)	768(4)	2168(1)	8406(2)	55(1)
O(1')	4080(2)	1544(1)	800(1)	55(1)
O(2')	4522(2)	2337(1)	1479(1)	53(1)
O(3')	6550(3)	880(1)	4107(1)	83(1)
O(4')	7424(2)	1916(1)	4940(1)	71(1)
C(1')	4803(2)	1834(1)	1798(2)	45(1)
C(2')	4229(3)	1004(1)	910(2)	54(1)
C(3')	5041(3)	785(1)	1992(2)	58(1)
C(4')	5828(3)	1084(1)	3107(2)	54(1)
C(5')	5676(2)	1651(1)	2939(2)	45(1)
C(6')	6451(3)	2034(1)	3907(2)	49(1)
C(7')	6013(3)	2572(1)	3538(2)	51(1)
C(8')	5130(3)	2712(1)	2397(2)	50(1)
C(10')	3396(5)	749(1)	-297(3)	78(1)
C(11')	4652(5)	3247(1)	1918(3)	68(1)
O(1S)	7905(3)	4433(1)	3636(2)	86(1)
O(1S')	9418(3)	1038(1)	6693(2)	92(1)

Table 3. Bond lengths [Å] and angles [°] for **4**.

O(1)-C(1)	1.326(2)
O(1)-C(2)	1.385(2)
O(2)-C(1)	1.3321(19)
O(2)-C(8)	1.384(2)
O(3)-C(4)	1.228(2)
O(4)-C(6)	1.230(2)

C(1)-C(5)	1.356(2)
C(2)-C(3)	1.324(3)
C(2)-C(10)	1.480(3)
C(3)-C(4)	1.448(3)
C(3)-H(3)	0.94(2)
C(4)-C(5)	1.469(2)
C(5)-C(6)	1.462(2)
C(6)-C(7)	1.454(2)
C(7)-C(8)	1.328(2)
C(7)-H(7)	0.958(19)
C(8)-C(11)	1.480(2)
C(10)-H(10A)	0.98(3)
C(10)-H(10B)	0.96(3)
C(10)-H(10C)	0.97(3)
C(11)-H(11A)	0.93(2)
C(11)-H(11B)	0.98(2)
C(11)-H(11C)	1.01(2)
O(1')-C(1')	1.332(2)
O(1')-C(2')	1.386(2)
O(2')-C(1')	1.336(2)
O(2')-C(8')	1.391(2)
O(3')-C(4')	1.224(2)
O(4')-C(6')	1.225(2)
C(1')-C(5')	1.355(2)
C(2')-C(3')	1.327(3)
C(2')-C(10')	1.483(3)
C(3')-C(4')	1.450(3)
C(3')-H(3')	0.93(2)
C(4')-C(5')	1.462(2)
C(5')-C(6')	1.462(2)
C(6')-C(7')	1.447(3)
C(7')-C(8')	1.323(3)
C(7')-H(7')	0.910(19)
C(8')-C(11')	1.478(3)
C(10')-H(10D)	0.98(3)
C(10')-H(10E)	0.94(3)

C(10')-H(10F)	0.92(3)
C(11')-H(11D)	0.97(3)
C(11')-H(11E)	0.90(2)
C(11')-H(11F)	0.97(2)
O(1S)-H(1S)	0.77(3)
O(1S)-H(2S)	0.87(3)
O(1S')-H(1'S)	0.80(4)
O(1S')-H(2'S)	0.81(3)

C(1)-O(1)-C(2)	118.69(14)
C(1)-O(2)-C(8)	118.04(13)
O(1)-C(1)-O(2)	107.48(14)
O(1)-C(1)-C(5)	126.07(15)
O(2)-C(1)-C(5)	126.44(16)
C(3)-C(2)-O(1)	120.31(17)
C(3)-C(2)-C(10)	129.52(19)
O(1)-C(2)-C(10)	110.16(17)
C(2)-C(3)-C(4)	123.25(18)
C(2)-C(3)-H(3)	117.1(11)
C(4)-C(3)-H(3)	119.7(11)
O(3)-C(4)-C(3)	122.51(17)
O(3)-C(4)-C(5)	123.03(17)
C(3)-C(4)-C(5)	114.45(16)
C(1)-C(5)-C(6)	117.26(15)
C(1)-C(5)-C(4)	117.14(16)
C(6)-C(5)-C(4)	125.60(15)
O(4)-C(6)-C(7)	121.55(17)
O(4)-C(6)-C(5)	123.92(16)
C(7)-C(6)-C(5)	114.52(15)
C(8)-C(7)-C(6)	122.90(17)
C(8)-C(7)-H(7)	117.5(11)
C(6)-C(7)-H(7)	119.5(11)
C(7)-C(8)-O(2)	120.76(15)
C(7)-C(8)-C(11)	128.23(17)
O(2)-C(8)-C(11)	111.00(16)
C(2)-C(10)-H(10A)	109.1(16)

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)

O(2')-C(8')-C(11')	111.71(17)
C(2')-C(10')-H(10D)	109.8(19)
C(2')-C(10')-H(10E)	112.2(16)
H(10D)-C(10')-H(10E)	107(3)
C(2')-C(10')-H(10F)	108.8(16)
H(10D)-C(10')-H(10F)	113(2)
H(10E)-C(10')-H(10F)	106(2)
C(8')-C(11')-H(11D)	108.2(15)
C(8')-C(11')-H(11E)	110.2(15)
H(11D)-C(11')-H(11E)	111(2)
C(8')-C(11')-H(11F)	110.9(14)
H(11D)-C(11')-H(11F)	111(2)
H(11E)-C(11')-H(11F)	106(2)
H(1S)-O(1S)-H(2S)	107(3)
H(1'S)-O(1S')-H(2'S)	106(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jw33. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	77(1)	36(1)	48(1)	0(1)	3(1)	-2(1)
O(2)	65(1)	34(1)	48(1)	2(1)	10(1)	0(1)
O(3)	96(1)	48(1)	62(1)	16(1)	1(1)	0(1)
O(4)	86(1)	56(1)	48(1)	3(1)	3(1)	1(1)
C(1)	51(1)	35(1)	47(1)	0(1)	13(1)	-2(1)
C(2)	72(1)	35(1)	59(1)	-2(1)	13(1)	-3(1)
C(3)	71(1)	33(1)	64(1)	2(1)	13(1)	-2(1)
C(4)	55(1)	42(1)	57(1)	8(1)	12(1)	3(1)
C(5)	45(1)	39(1)	48(1)	4(1)	14(1)	2(1)
C(6)	50(1)	46(1)	46(1)	1(1)	14(1)	0(1)
C(7)	54(1)	43(1)	49(1)	-6(1)	14(1)	-2(1)
C(8)	46(1)	37(1)	53(1)	-4(1)	18(1)	-2(1)
C(10)	110(2)	47(1)	61(1)	-8(1)	6(1)	-9(1)

C(11)	60(1)	37(1)	68(1)	5(1)	18(1)	2(1)
O(1')	59(1)	51(1)	52(1)	-1(1)	6(1)	-3(1)
O(2')	61(1)	43(1)	50(1)	7(1)	7(1)	1(1)
O(3')	119(1)	51(1)	66(1)	15(1)	1(1)	1(1)
O(4')	90(1)	58(1)	53(1)	5(1)	-5(1)	3(1)
C(1')	42(1)	44(1)	49(1)	1(1)	12(1)	-3(1)
C(2')	54(1)	48(1)	63(1)	-2(1)	17(1)	-5(1)
C(3')	64(1)	42(1)	69(1)	0(1)	19(1)	-2(1)
C(4')	59(1)	45(1)	59(1)	8(1)	14(1)	-1(1)
C(5')	41(1)	45(1)	49(1)	4(1)	11(1)	-1(1)
C(6')	47(1)	50(1)	50(1)	6(1)	14(1)	2(1)
C(7')	54(1)	47(1)	50(1)	-4(1)	13(1)	0(1)
C(8')	54(1)	44(1)	53(1)	1(1)	17(1)	-1(1)
C(10')	96(2)	65(2)	72(2)	-15(1)	15(2)	-14(2)
C(11')	90(2)	49(1)	67(2)	11(1)	22(1)	8(1)
O(1S)	105(1)	84(1)	59(1)	12(1)	5(1)	-13(1)
O(1S')	102(2)	85(1)	83(1)	12(1)	11(1)	17(1)

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

	x	y	z	U(eq)
H(1S)	8180(50)	4270(12)	4240(30)	127(13)
H(1'S)	8580(50)	1113(13)	6090(30)	128(14)
H(2S)	8650(50)	4711(13)	3750(30)	135(13)
H(2'S)	8850(50)	947(14)	7210(30)	145(17)
H(3)	1320(30)	4993(8)	8500(16)	60(5)
H(3')	5100(30)	422(8)	2026(17)	65(6)
H(7)	-870(30)	2611(7)	6193(18)	58(5)
H(7')	6400(30)	2824(7)	4123(18)	58(6)
H(10A)	4420(40)	4472(10)	10970(20)	111(10)
H(10B)	2410(40)	4485(10)	11360(20)	103(9)
H(10C)	3010(30)	4975(11)	10740(20)	102(8)

H(10D)	2130(50)	908(12)	-690(30)	143(13)
H(10E)	4200(40)	790(10)	-850(30)	113(10)
H(10F)	3320(40)	397(10)	-180(20)	98(9)
H(11A)	2070(30)	2095(8)	8760(20)	77(7)
H(11B)	40(30)	2112(8)	9030(20)	87(7)
H(11C)	310(30)	1911(8)	7706(18)	69(6)
H(11D)	5180(40)	3494(10)	2580(20)	99(8)
H(11E)	3350(40)	3286(9)	1650(20)	84(8)
H(11F)	5170(30)	3312(9)	1210(20)	89(8)

Table 6. Torsion angles [°] for **4**.

C(2)-O(1)-C(1)-O(2)	-179.49(14)
C(2)-O(1)-C(1)-C(5)	1.4(3)
C(8)-O(2)-C(1)-O(1)	-179.43(13)
C(8)-O(2)-C(1)-C(5)	-0.4(2)
C(1)-O(1)-C(2)-C(3)	-1.3(3)
C(1)-O(1)-C(2)-C(10)	-180.0(2)
O(1)-C(2)-C(3)-C(4)	-1.1(3)
C(10)-C(2)-C(3)-C(4)	177.3(3)
C(2)-C(3)-C(4)-O(3)	-175.3(2)
C(2)-C(3)-C(4)-C(5)	3.1(3)
O(1)-C(1)-C(5)-C(6)	-179.36(16)
O(2)-C(1)-C(5)-C(6)	1.7(3)
O(1)-C(1)-C(5)-C(4)	0.8(3)
O(2)-C(1)-C(5)-C(4)	-178.15(16)
O(3)-C(4)-C(5)-C(1)	175.59(18)
C(3)-C(4)-C(5)-C(1)	-2.9(2)
O(3)-C(4)-C(5)-C(6)	-4.3(3)
C(3)-C(4)-C(5)-C(6)	177.27(16)
C(1)-C(5)-C(6)-O(4)	176.02(17)
C(4)-C(5)-C(6)-O(4)	-4.1(3)
C(1)-C(5)-C(6)-C(7)	-2.9(2)
C(4)-C(5)-C(6)-C(7)	176.92(16)
O(4)-C(6)-C(7)-C(8)	-175.75(18)

C(5)-C(6)-C(7)-C(8)	3.2(2)
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)
C(2')-O(1')-C(1')-O(2')	179.31(14)
C(2')-O(1')-C(1')-C(5')	-0.5(3)
C(8')-O(2')-C(1')-O(1')	178.46(13)
C(8')-O(2')-C(1')-C(5')	-1.7(2)
C(1')-O(1')-C(2')-C(3')	1.3(3)
C(1')-O(1')-C(2')-C(10')	-178.50(19)
O(1')-C(2')-C(3')-C(4')	-0.6(3)
C(10')-C(2')-C(3')-C(4')	179.1(2)
C(2')-C(3')-C(4')-O(3')	178.9(2)
C(2')-C(3')-C(4')-C(5')	-0.7(3)
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 °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1S)-H(1S)...O(4)#1	0.77(3)	2.22(3)	2.948(2)	157(3)
O(1S)-H(1S)...O(3)#1	0.77(3)	2.50(3)	3.031(2)	128(3)
O(1S')-H(1'S)...O(3')	0.80(4)	2.40(4)	3.116(3)	150(3)
O(1S')-H(1'S)...O(4')	0.80(4)	2.45(3)	3.081(3)	136(3)
O(1S)-H(2S)...O(3)#2	0.87(3)	2.13(3)	2.993(3)	168(3)

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

Metal Bis-γ-pyrone Complex 7 (M=Mg): General Procedure followed with 10 mg (0.0520 mmol) bis-γ-pyrone **5** and 5.8 mg (0.026 mmol) of $Mg(II)(ClO_4)_2$ yielding 15.8 mg of white crystals (quant.). NMR [∂_H (DMSO) 2.28 s (3H), 6.21 s (1H), ∂_C 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:

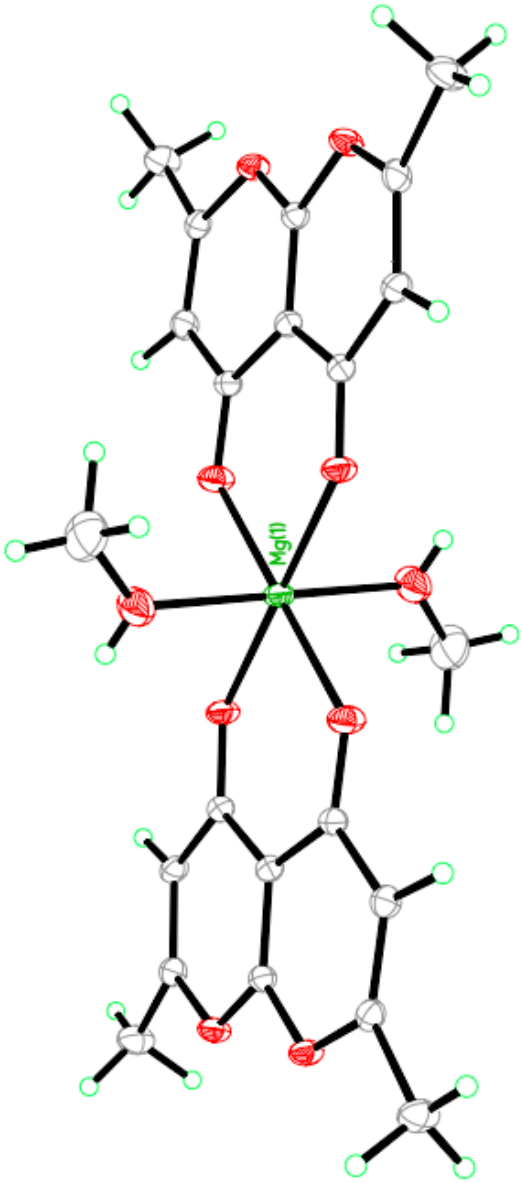


Table 1. Crystal data and structure refinement for **7** (M=Mg)•H_2O

Identification code	**7** (M=Mg)
Empirical formula	C22 H26 Cl2 Mg O19
Formula weight	689.64

Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 15.050(3) Å	a= 90°.
	b = 18.021(3) Å	b= 103.023(3)°.
	c = 11.008(2) Å	g = 90°.
Volume	2908.7(10) $Å^3$	
Z	4	
Density (calculated)	1.575 Mg/m^3	
Absorption coefficient	0.331 mm^{-1}	
F(000)	1424	
Crystal size	0.23 x 0.19 x 0.12 mm^3	
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 F^2	
Data / restraints / parameters	5118 / 18 / 500	
Goodness-of-fit on F^2	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 ($Å^2$x 10^3) for **7** (M= Mg). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Mg(1)	5000	5000	5000	24(1)
O(1)	5590(2)	6014(1)	5558(2)	26(1)
O(2)	4595(2)	4999(1)	6673(2)	30(1)
O(3)	5438(2)	7362(1)	8464(2)	26(1)
O(4)	4658(2)	6589(1)	9302(2)	24(1)
O(5)	3834(2)	5563(2)	4205(3)	40(1)
C(1)	6373(4)	8300(2)	7952(4)	36(1)
C(2)	5927(3)	7575(2)	7599(3)	26(1)
C(3)	5967(3)	7137(2)	6624(3)	25(1)
C(4)	5536(2)	6424(2)	6452(3)	22(1)
C(5)	5055(2)	6206(2)	7407(3)	21(1)
C(6)	5050(2)	6698(2)	8345(3)	21(1)
C(7)	4623(2)	5491(2)	7470(3)	23(1)
C(8)	4226(2)	5383(2)	8534(3)	25(1)
C(9)	4243(2)	5917(2)	9391(3)	23(1)
C(10)	3851(3)	5894(3)	10506(4)	33(1)
C(11)	3025(3)	5319(3)	3386(5)	56(1)
Mg(1')	0	10000	10000	23(1)
O(1')	503(2)	9992(1)	8410(2)	29(1)
O(2')	-480(2)	8956(1)	9526(2)	27(1)
O(3')	244(2)	8484(1)	5609(2)	26(1)
O(4')	-523(2)	7704(1)	6446(2)	24(1)
O(5')	1208(2)	9539(2)	10982(3)	43(1)
C(1')	1095(3)	9161(3)	4442(4)	33(1)
C(2')	730(2)	9131(2)	5581(3)	24(1)
C(3')	817(2)	9629(2)	6503(3)	25(1)
C(4')	431(2)	9513(2)	7572(3)	22(1)
C(5')	-33(2)	8813(2)	7600(3)	20(1)
C(6')	-101(2)	8351(2)	6599(3)	21(1)
C(7')	-473(2)	8571(2)	8583(3)	22(1)
C(8')	-917(3)	7863(2)	8389(3)	26(1)

C(9')	-943(2)	7458(2)	7362(3)	24(1)
C(10')	-1401(3)	6737(2)	7007(4)	34(1)
C(11')	2069(3)	9867(3)	11392(6)	70(2)
Cl(1)	2111(1)	7668(1)	2106(1)	44(1)
O(6)	2867(3)	7999(3)	2822(5)	108(2)
O(7)	1389(7)	7713(8)	2684(12)	164(5)
O(8)	1875(6)	8070(4)	967(6)	94(2)
O(9)	2227(5)	6935(3)	1810(8)	79(2)
O(7A)	2298(12)	7327(15)	1090(20)	164(5)
O(8A)	1831(11)	7048(8)	2736(13)	94(2)
O(9A)	1324(8)	8090(6)	1720(16)	79(2)
Cl(2)	2573(1)	1208(1)	5021(1)	31(1)
O(10)	2518(2)	1991(2)	5146(3)	58(1)
O(11)	3491(2)	1062(2)	4874(4)	45(1)
O(12)	1966(3)	964(2)	3975(5)	74(2)
O(13)	2499(4)	829(2)	6119(4)	71(2)
O(11A)	1681(9)	1052(9)	5217(15)	45(1)
O(12A)	3162(14)	898(10)	5780(20)	74(2)
O(13A)	2492(18)	841(11)	3934(16)	71(1)
O(1S)	3596(2)	6937(2)	4949(3)	57(1)

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

Mg(1)-O(5)#1	2.046(3)
Mg(1)-O(5)	2.046(3)
Mg(1)-O(1)#1	2.064(2)
Mg(1)-O(1)	2.064(2)
Mg(1)-O(2)#1	2.066(2)
Mg(1)-O(2)	2.066(2)
Mg(1)-H(5O)	2.60(5)
O(1)-C(4)	1.248(4)
O(2)-C(7)	1.241(4)
O(3)-C(6)	1.324(4)
O(3)-C(2)	1.383(4)
O(4)-C(6)	1.332(4)
O(4)-C(9)	1.376(4)
O(5)-C(11)	1.411(5)
O(5)-H(5O)	0.95(2)
C(1)-C(2)	1.479(5)
C(1)-H(1A)	0.92(5)
C(1)-H(1B)	0.85(5)
C(1)-H(1C)	1.01(5)
C(2)-C(3)	1.345(5)
C(3)-C(4)	1.432(5)
C(3)-H(3)	0.92(4)
C(4)-C(5)	1.459(5)
C(5)-C(6)	1.363(5)
C(5)-C(7)	1.450(5)
C(7)-C(8)	1.443(5)
C(8)-C(9)	1.344(5)
C(8)-H(8)	0.99(4)
C(9)-C(10)	1.477(5)
C(10)-H(10A)	1.01(5)
C(10)-H(10B)	0.96(5)
C(10)-H(10C)	0.93(5)
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800

C(11)-H(11C)	0.9800
Mg(1')-O(2')	2.041(2)
Mg(1')-O(2')#2	2.041(2)
Mg(1')-O(1')#2	2.057(2)
Mg(1')-O(1')	2.057(2)
Mg(1')-O(5')	2.068(3)
Mg(1')-O(5')#2	2.068(3)
O(1')-C(4')	1.250(4)
O(2')-C(7')	1.250(4)
O(3')-C(6')	1.330(4)
O(3')-C(2')	1.381(4)
O(4')-C(6')	1.320(4)
O(4')-C(9')	1.378(4)
O(5')-C(11')	1.403(6)
O(5')-H(5'O)	0.93(2)
C(1')-C(2')	1.478(5)
C(1')-H(1'A)	1.06(5)
C(1')-H(1'B)	0.97(5)
C(1')-H(1'C)	0.90(5)
C(2')-C(3')	1.340(5)
C(3')-C(4')	1.440(5)
C(3')-H(3')	0.96(4)
C(4')-C(5')	1.445(5)
C(5')-C(6')	1.367(5)
C(5')-C(7')	1.458(5)
C(7')-C(8')	1.433(5)
C(8')-C(9')	1.339(5)
C(8')-H(8')	0.94(4)
C(9')-C(10')	1.481(5)
C(10')-H(10D)	0.86(5)
C(10')-H(10E)	0.97(5)
C(10')-H(10F)	1.00(5)
C(11')-H(11D)	0.9800
C(11')-H(11E)	0.9800
C(11')-H(11F)	0.9800
Cl(1)-O(7A)	1.362(11)

Cl(1)-O(6)	1.367(4)
Cl(1)-O(7)	1.379(7)
Cl(1)-O(9)	1.382(6)
Cl(1)-O(9A)	1.391(10)
Cl(1)-O(8)	1.422(6)
Cl(1)-O(8A)	1.428(11)
Cl(2)-O(12A)	1.211(14)
Cl(2)-O(12)	1.371(4)
Cl(2)-O(13)	1.414(4)
Cl(2)-O(10)	1.422(3)
Cl(2)-O(11)	1.451(3)
Cl(2)-O(13A)	1.349(16)
Cl(2)-O(11A)	1.435(12)
O(1S)-H(1S)	1.00(2)
O(1S)-H(2S)	0.98(2)

O(5)#1-Mg(1)-O(5)	180.0
O(5)#1-Mg(1)-O(1)#1	87.60(11)
O(5)-Mg(1)-O(1)#1	92.40(11)
O(5)#1-Mg(1)-O(1)	92.40(11)
O(5)-Mg(1)-O(1)	87.60(11)
O(1)#1-Mg(1)-O(1)	180.000(1)
O(5)#1-Mg(1)-O(2)#1	89.12(11)
O(5)-Mg(1)-O(2)#1	90.88(11)
O(1)#1-Mg(1)-O(2)#1	85.92(9)
O(1)-Mg(1)-O(2)#1	94.08(9)
O(5)#1-Mg(1)-O(2)	90.89(11)
O(5)-Mg(1)-O(2)	89.11(11)
O(1)#1-Mg(1)-O(2)	94.08(9)
O(1)-Mg(1)-O(2)	85.92(9)
O(2)#1-Mg(1)-O(2)	180.000(1)
O(5)#1-Mg(1)-H(5O)	160.8(11)
O(5)-Mg(1)-H(5O)	19.2(10)
O(1)#1-Mg(1)-H(5O)	111.3(11)
O(1)-Mg(1)-H(5O)	68.7(10)
O(2)#1-Mg(1)-H(5O)	95.5(13)

O(2)-Mg(1)-H(5O)	84.5(13)
C(4)-O(1)-Mg(1)	131.2(2)
C(7)-O(2)-Mg(1)	131.3(2)
C(6)-O(3)-C(2)	118.3(3)
C(6)-O(4)-C(9)	118.4(3)
C(11)-O(5)-Mg(1)	130.6(3)
C(11)-O(5)-H(5O)	114(4)
Mg(1)-O(5)-H(5O)	115(4)
C(2)-C(1)-H(1A)	109(3)
C(2)-C(1)-H(1B)	116(3)
H(1A)-C(1)-H(1B)	110(4)
C(2)-C(1)-H(1C)	109(3)
H(1A)-C(1)-H(1C)	109(4)
H(1B)-C(1)-H(1C)	102(4)
C(3)-C(2)-O(3)	120.8(3)
C(3)-C(2)-C(1)	129.0(3)
O(3)-C(2)-C(1)	110.2(3)
C(2)-C(3)-C(4)	122.2(3)
C(2)-C(3)-H(3)	118(2)
C(4)-C(3)-H(3)	120(2)
O(1)-C(4)-C(3)	121.8(3)
O(1)-C(4)-C(5)	122.7(3)
C(3)-C(4)-C(5)	115.5(3)
C(6)-C(5)-C(7)	117.6(3)
C(6)-C(5)-C(4)	117.3(3)
C(7)-C(5)-C(4)	125.0(3)
O(3)-C(6)-O(4)	108.7(3)
O(3)-C(6)-C(5)	125.8(3)
O(4)-C(6)-C(5)	125.5(3)
O(2)-C(7)-C(8)	121.5(3)
O(2)-C(7)-C(5)	123.0(3)
C(8)-C(7)-C(5)	115.5(3)
C(9)-C(8)-C(7)	121.9(3)
C(9)-C(8)-H(8)	119(2)
C(7)-C(8)-H(8)	119(2)
C(8)-C(9)-O(4)	121.1(3)

C(8)-C(9)-C(10)	128.1(3)
O(4)-C(9)-C(10)	110.8(3)
C(9)-C(10)-H(10A)	110(3)
C(9)-C(10)-H(10B)	111(3)
H(10A)-C(10)-H(10B)	107(4)
C(9)-C(10)-H(10C)	106(3)
H(10A)-C(10)-H(10C)	110(4)
H(10B)-C(10)-H(10C)	113(4)
O(5)-C(11)-H(11A)	109.5
O(5)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
O(5)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
O(2')-Mg(1')-O(2')#2	180.000(1)
O(2')-Mg(1')-O(1')#2	92.72(9)
O(2')#2-Mg(1')-O(1')#2	87.28(9)
O(2')-Mg(1')-O(1')	87.28(9)
O(2')#2-Mg(1')-O(1')	92.72(9)
O(1')#2-Mg(1')-O(1')	180.000(1)
O(2')-Mg(1')-O(5')	88.95(11)
O(2')#2-Mg(1')-O(5')	91.05(11)
O(1')#2-Mg(1')-O(5')	90.38(11)
O(1')-Mg(1')-O(5')	89.62(12)
O(2')-Mg(1')-O(5')#2	91.05(11)
O(2')#2-Mg(1')-O(5')#2	88.95(11)
O(1')#2-Mg(1')-O(5')#2	89.62(12)
O(1')-Mg(1')-O(5')#2	90.38(12)
O(5')-Mg(1')-O(5')#2	180.000(1)
C(4')-O(1')-Mg(1')	130.2(2)
C(7')-O(2')-Mg(1')	131.0(2)
C(6')-O(3')-C(2')	118.5(3)
C(6')-O(4')-C(9')	118.5(3)
C(11')-O(5')-Mg(1')	129.6(3)
C(11')-O(5')-H(5'O)	112(3)
Mg(1')-O(5')-H(5'O)	118(3)

C(2')-C(1')-H(1'A)	109(3)
C(2')-C(1')-H(1'B)	110(3)
H(1'A)-C(1')-H(1'B)	108(4)
C(2')-C(1')-H(1'C)	107(3)
H(1'A)-C(1')-H(1'C)	115(4)
H(1'B)-C(1')-H(1'C)	108(4)
C(3')-C(2')-O(3')	120.7(3)
C(3')-C(2')-C(1')	128.8(3)
O(3')-C(2')-C(1')	110.5(3)
C(2')-C(3')-C(4')	122.2(3)
C(2')-C(3')-H(3')	117(2)
C(4')-C(3')-H(3')	120(2)
O(1')-C(4')-C(3')	121.3(3)
O(1')-C(4')-C(5')	123.3(3)
C(3')-C(4')-C(5')	115.4(3)
C(6')-C(5')-C(4')	117.7(3)
C(6')-C(5')-C(7')	116.8(3)
C(4')-C(5')-C(7')	125.4(3)
O(4')-C(6')-O(3')	108.6(3)
O(4')-C(6')-C(5')	126.1(3)
O(3')-C(6')-C(5')	125.3(3)
O(2')-C(7')-C(8')	121.8(3)
O(2')-C(7')-C(5')	122.7(3)
C(8')-C(7')-C(5')	115.5(3)
C(9')-C(8')-C(7')	122.3(3)
C(9')-C(8')-H(8')	116(2)
C(7')-C(8')-H(8')	121(2)
C(8')-C(9')-O(4')	120.8(3)
C(8')-C(9')-C(10')	128.6(3)
O(4')-C(9')-C(10')	110.6(3)
C(9')-C(10')-H(10D)	110(3)
C(9')-C(10')-H(10E)	113(3)
H(10D)-C(10')-H(10E)	107(4)
C(9')-C(10')-H(10F)	107(2)
H(10D)-C(10')-H(10F)	112(4)
H(10E)-C(10')-H(10F)	108(4)

O(5')-C(11')-H(11D)	109.5
O(5')-C(11')-H(11E)	109.5
H(11D)-C(11')-H(11E)	109.5
O(5')-C(11')-H(11F)	109.5
H(11D)-C(11')-H(11F)	109.5
H(11E)-C(11')-H(11F)	109.5
O(7A)-Cl(1)-O(6)	111.8(7)
O(7A)-Cl(1)-O(7)	137.4(7)
O(6)-Cl(1)-O(7)	110.4(4)
O(7A)-Cl(1)-O(9)	46.7(11)
O(6)-Cl(1)-O(9)	114.8(4)
O(7)-Cl(1)-O(9)	108.6(6)
O(7A)-Cl(1)-O(9A)	108.4(13)
O(6)-Cl(1)-O(9A)	118.6(5)
O(7)-Cl(1)-O(9A)	53.4(7)
O(9)-Cl(1)-O(9A)	126.6(5)
O(7A)-Cl(1)-O(8)	64.0(13)
O(6)-Cl(1)-O(8)	107.3(4)
O(7)-Cl(1)-O(8)	108.1(7)
O(9)-Cl(1)-O(8)	107.4(4)
O(9A)-Cl(1)-O(8)	55.0(7)
O(7A)-Cl(1)-O(8A)	100.7(12)
O(6)-Cl(1)-O(8A)	111.1(6)
O(7)-Cl(1)-O(8A)	58.1(7)
O(9)-Cl(1)-O(8A)	55.4(7)
O(9A)-Cl(1)-O(8A)	104.6(8)
O(8)-Cl(1)-O(8A)	141.7(6)
O(12A)-Cl(2)-O(12)	132.8(9)
O(12A)-Cl(2)-O(13)	51.1(13)
O(12)-Cl(2)-O(13)	113.4(3)
O(12A)-Cl(2)-O(10)	116.1(9)
O(12)-Cl(2)-O(10)	110.9(2)
O(13)-Cl(2)-O(10)	112.3(2)
O(12A)-Cl(2)-O(11)	55.0(13)
O(12)-Cl(2)-O(11)	108.6(3)
O(13)-Cl(2)-O(11)	105.5(3)

O(10)-Cl(2)-O(11)	105.6(2)
O(12A)-Cl(2)-O(13A)	107.1(15)
O(12)-Cl(2)-O(13A)	35.6(10)
O(13)-Cl(2)-O(13A)	120.9(9)
O(10)-Cl(2)-O(13A)	125.2(9)
O(11)-Cl(2)-O(13A)	73.1(11)
O(12A)-Cl(2)-O(11A)	111.7(15)
O(12)-Cl(2)-O(11A)	66.1(7)
O(13)-Cl(2)-O(11A)	61.4(7)
O(10)-Cl(2)-O(11A)	95.9(6)
O(11)-Cl(2)-O(11A)	158.2(7)
O(13A)-Cl(2)-O(11A)	97.9(12)
H(1S)-O(1S)-H(2S)	115(7)

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for **7** (M= Mg). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Mg(1)	31(1)	23(1)	19(1)	-5(1)	8(1)	-1(1)
O(1)	37(1)	24(1)	20(1)	-5(1)	12(1)	-4(1)
O(2)	44(2)	24(1)	26(1)	-6(1)	14(1)	-9(1)
O(3)	36(1)	20(1)	24(1)	-4(1)	15(1)	-2(1)
O(4)	30(1)	25(1)	21(1)	-4(1)	12(1)	-3(1)
O(5)	35(2)	36(2)	45(2)	-5(1)	-2(1)	5(1)
C(1)	54(3)	23(2)	36(3)	-4(2)	21(2)	-10(2)
C(2)	37(2)	22(2)	23(2)	5(2)	13(2)	0(2)
C(3)	33(2)	24(2)	19(2)	2(2)	9(2)	-1(2)
C(4)	25(2)	22(2)	20(2)	1(1)	5(1)	1(1)
C(5)	24(2)	22(2)	18(2)	0(1)	6(1)	1(1)
C(6)	22(2)	22(2)	20(2)	0(1)	5(1)	0(1)
C(7)	28(2)	20(2)	20(2)	-1(1)	6(1)	0(1)
C(8)	28(2)	23(2)	25(2)	3(2)	8(2)	-1(2)

C(9)	23(2)	26(2)	21(2)	3(2)	7(1)	2(2)
C(10)	39(2)	33(2)	31(2)	-2(2)	20(2)	-4(2)
C(11)	48(3)	58(3)	53(3)	0(2)	-6(2)	0(2)
Mg(1')	31(1)	22(1)	18(1)	-3(1)	8(1)	1(1)
O(1')	40(2)	24(1)	25(1)	-5(1)	13(1)	-8(1)
O(2')	41(2)	26(1)	18(1)	-5(1)	13(1)	-3(1)
O(3')	32(1)	28(1)	20(1)	-5(1)	13(1)	-5(1)
O(4')	32(1)	22(1)	22(1)	-3(1)	11(1)	-3(1)
O(5')	34(2)	41(2)	49(2)	7(1)	-1(1)	3(1)
C(1')	40(2)	34(2)	31(2)	-3(2)	19(2)	-4(2)
C(2')	27(2)	26(2)	23(2)	2(2)	10(2)	-2(2)
C(3')	28(2)	24(2)	25(2)	3(2)	9(2)	-1(2)
C(4')	23(2)	23(2)	20(2)	2(1)	4(1)	4(1)
C(5')	23(2)	21(2)	17(2)	0(1)	5(1)	2(1)
C(6')	21(2)	23(2)	21(2)	2(1)	8(1)	1(1)
C(7')	25(2)	22(2)	19(2)	2(1)	4(1)	3(1)
C(8')	34(2)	23(2)	23(2)	3(2)	12(2)	-1(2)
C(9')	29(2)	21(2)	24(2)	5(1)	10(2)	1(2)
C(10')	48(3)	25(2)	34(2)	-3(2)	18(2)	-5(2)
C(11')	39(3)	72(4)	90(4)	-11(3)	-4(3)	6(3)
Cl(1)	47(1)	37(1)	47(1)	-2(1)	8(1)	1(1)
O(6)	76(3)	84(3)	139(4)	3(3)	-26(3)	-38(3)
O(7)	119(7)	231(12)	176(9)	-150(9)	108(7)	-84(8)
O(8)	126(6)	82(5)	67(4)	24(3)	8(4)	16(4)
O(9)	73(4)	38(3)	117(6)	-8(3)	5(4)	11(3)
O(7A)	119(7)	231(12)	176(9)	-150(9)	108(7)	-84(8)
O(8A)	126(6)	82(5)	67(4)	24(3)	8(4)	16(4)
O(9A)	73(4)	38(3)	117(6)	-8(3)	5(4)	11(3)
Cl(2)	37(1)	25(1)	35(1)	1(1)	14(1)	2(1)
O(10)	52(2)	24(2)	96(3)	0(2)	11(2)	3(1)
O(11)	33(2)	56(2)	50(2)	-8(2)	17(2)	8(2)
O(12)	55(3)	49(3)	91(4)	-10(2)	-35(3)	2(2)
O(13)	116(4)	53(3)	66(3)	18(2)	64(3)	9(3)
O(11A)	33(2)	56(2)	50(2)	-8(2)	17(2)	8(2)
O(12A)	55(3)	49(3)	91(4)	-10(2)	-35(3)	2(2)
O(13A)	116(1)	53(1)	65(1)	16(2)	63(2)	9(2)

O(1S)	63(2)	43(2)	63(2)	-2(2)	9(2)	13(2)

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

	x	y	z	U(eq)
H(11A)	2599	5735	3182	84
H(11B)	2747	4925	3787	84
H(11C)	3170	5128	2620	84
H(11D)	2499	9498	11839	105
H(11E)	2023	10281	11952	105
H(11F)	2285	10051	10672	105
H(1A)	5930(30)	8640(30)	8020(40)	56(15)
H(1B)	6710(30)	8470(30)	7480(50)	54(15)
H(1C)	6820(30)	8250(20)	8780(40)	45(13)
H(1'A)	550(30)	9070(30)	3650(50)	61(14)
H(1'B)	1540(30)	8770(30)	4470(40)	49(13)
H(1'C)	1380(30)	9600(30)	4450(40)	45(13)
H(3)	6260(20)	7315(19)	6040(30)	20(9)
H(3')	1160(30)	10070(20)	6430(30)	26(10)
H(5'O)	1240(30)	9024(12)	11050(40)	58(15)
H(5O)	3830(40)	6072(15)	4410(60)	100(20)
H(8)	3950(30)	4900(20)	8650(40)	34(11)
H(8')	-1210(20)	7660(20)	8980(30)	25(9)
H(10A)	4350(30)	5980(20)	11280(40)	48(13)
H(10B)	3410(30)	6280(30)	10470(40)	50(13)
H(10C)	3610(30)	5420(30)	10530(40)	48(13)
H(10D)	-1010(30)	6410(30)	6890(40)	42(13)
H(10E)	-1700(30)	6540(30)	7630(40)	48(13)
H(10F)	-1880(30)	6820(20)	6230(40)	41(12)
H(1S)	3170(50)	6970(50)	5520(70)	200(40)
H(2S)	3400(50)	7210(40)	4160(40)	130(30)

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

O(5)#1-Mg(1)-O(1)-C(4)	-100.0(3)
O(5)-Mg(1)-O(1)-C(4)	80.0(3)
O(1)#1-Mg(1)-O(1)-C(4)	94(100)
O(2)#1-Mg(1)-O(1)-C(4)	170.7(3)
O(2)-Mg(1)-O(1)-C(4)	-9.3(3)
O(5)#1-Mg(1)-O(2)-C(7)	101.7(3)
O(5)-Mg(1)-O(2)-C(7)	-78.3(3)
O(1)#1-Mg(1)-O(2)-C(7)	-170.6(3)
O(1)-Mg(1)-O(2)-C(7)	9.4(3)
O(2)#1-Mg(1)-O(2)-C(7)	-56(100)
O(5)#1-Mg(1)-O(5)-C(11)	112(100)
O(1)#1-Mg(1)-O(5)-C(11)	-10.8(4)
O(1)-Mg(1)-O(5)-C(11)	169.2(4)
O(2)#1-Mg(1)-O(5)-C(11)	75.2(4)
O(2)-Mg(1)-O(5)-C(11)	-104.8(4)
C(6)-O(3)-C(2)-C(3)	3.6(5)
C(6)-O(3)-C(2)-C(1)	-175.0(3)
O(3)-C(2)-C(3)-C(4)	-2.1(6)
C(1)-C(2)-C(3)-C(4)	176.2(4)
Mg(1)-O(1)-C(4)-C(3)	-176.1(2)
Mg(1)-O(1)-C(4)-C(5)	5.5(5)
C(2)-C(3)-C(4)-O(1)	-178.6(3)
C(2)-C(3)-C(4)-C(5)	-0.2(5)
O(1)-C(4)-C(5)-C(6)	179.4(3)
C(3)-C(4)-C(5)-C(6)	0.9(5)
O(1)-C(4)-C(5)-C(7)	2.8(5)
C(3)-C(4)-C(5)-C(7)	-175.6(3)
C(2)-O(3)-C(6)-O(4)	176.7(3)
C(2)-O(3)-C(6)-C(5)	-2.9(5)
C(9)-O(4)-C(6)-O(3)	-177.4(3)
C(9)-O(4)-C(6)-C(5)	2.2(5)
C(7)-C(5)-C(6)-O(3)	177.4(3)
C(4)-C(5)-C(6)-O(3)	0.6(5)
C(7)-C(5)-C(6)-O(4)	-2.1(5)

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(4)	-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')-Mg(1')-O(1')-C(4')	3.5(3)
O(2')#2-Mg(1')-O(1')-C(4')	-176.5(3)
O(1')#2-Mg(1')-O(1')-C(4')	-64(100)
O(5')-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(1')#2-Mg(1')-O(2')-C(7')	175.7(3)
O(1')-Mg(1')-O(2')-C(7')	-4.3(3)
O(5')-Mg(1')-O(2')-C(7')	-94.0(3)
O(5')#2-Mg(1')-O(2')-C(7')	86.0(3)
O(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(4')	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 °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5')-H(5'O)...O(9A)#3	0.93(2)	1.83(3)	2.728(12)	161(4)
O(5')-H(5'O)...O(8)#3	0.93(2)	1.98(3)	2.832(8)	151(4)
O(5)-H(5O)...O(1S)	0.95(2)	1.73(3)	2.656(4)	165(6)
O(1S)-H(1S)...O(7A)#4	1.00(2)	2.02(7)	2.872(19)	142(8)

O(1S)-H(1S)...O(8)#4	1.00(2)	2.12(5)	3.045(10)	154(8)
O(1S)-H(1S)...O(6)#4	1.00(2)	2.67(5)	3.575(7)	151(8)
O(1S)-H(2S)...O(6)	0.98(2)	2.07(3)	3.032(6)	164(6)
O(1S)-H(2S)...O(8A)	0.98(2)	2.55(7)	3.181(14)	122(5)

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

Metal Bis-γ-pyrone complex 7 (M=Ni)
General Procedure was followed using 10 mg (0.0520 mmoles) of bis-γ-pyrone **5** and 13.06 mg (0.026 mmoles) of $Ni(ClO_4)_2 \bullet 6CH_3CN$ 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 calcd 540.9684 found 540.9677. mp > 250˚C.

Crystal structure:

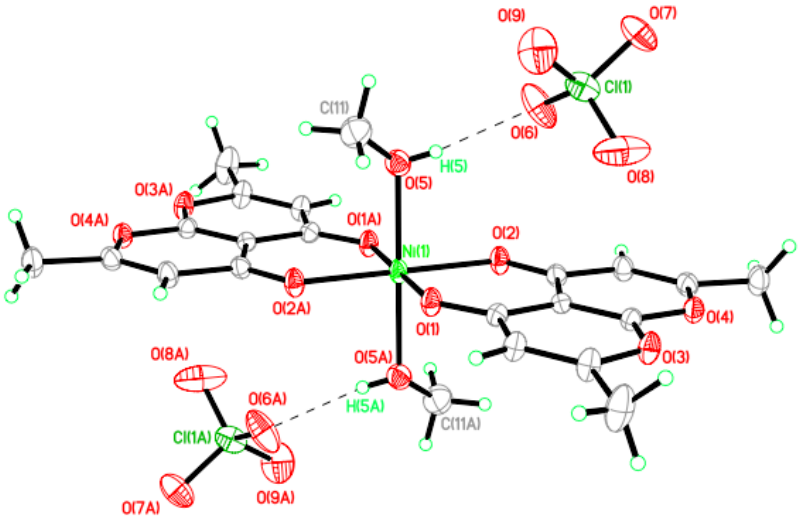


Table 1. Crystal data and structure refinement for **7** (M=Ni)

Identification code	**7** (M= Ni)
Empirical formula	C22 H24 Cl2 Ni O18

Formula weight	706.02	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 7.412(2) Å	a= 90°.
	b = 14.508(4) Å	b= 95.849(5)°.
	c = 13.476(4) Å	g = 90°.
Volume	1441.6(7) $Å^3$	
Z	2	
Density (calculated)	1.627 Mg/m^3	
Absorption coefficient	0.940 mm^{-1}	
F(000)	724	
Crystal size	0.31 x 0.20 x 0.14 mm^3	
Theta range for data collection	2.07 to 27.00°.	
Index ranges	-9<=h<=6, -18<=k<=18, -17<=l<=17	
Reflections collected	9624	
Independent reflections	3129 [R(int) = 0.0306]	
Completeness to theta = 27.00°	99.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8796 and 0.7592	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3129 / 7 / 254	
Goodness-of-fit on F^2	1.040	
Final R indices [I>2sigma(I)]	R1 = 0.0428, wR2 = 0.1012	
R indices (all data)	R1 = 0.0542, wR2 = 0.1099	
Largest diff. peak and hole	0.733 and -0.530 $e.Å^{-3}$	

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.

	x	y	z	U(eq)
Ni(1)	0	5000	0	28(1)
Cl(1)	2048(1)	3463(1)	3263(1)	49(1)
O(1)	1503(2)	3870(1)	-193(1)	33(1)
O(2)	-1631(2)	4288(1)	831(1)	35(1)
O(3)	600(3)	1232(1)	595(2)	37(1)
O(4)	-1684(2)	1547(1)	1406(1)	32(1)
O(5)	1681(3)	5316(2)	1284(2)	40(1)
O(6)	910(4)	4214(2)	2883(2)	75(1)
O(7)	1665(4)	3289(2)	4275(2)	63(1)
O(8)	1663(6)	2640(3)	2718(3)	103(2)
O(9)	3906(4)	3688(2)	3287(3)	81(1)
O(7A)	2870(20)	3319(13)	2323(10)	63(1)
O(8A)	3030(40)	3684(17)	4107(17)	103(2)
O(9A)	830(20)	2702(13)	3181(19)	81(1)
C(1)	2855(6)	603(2)	-301(4)	62(1)
C(2)	1968(4)	1449(2)	15(2)	36(1)
C(3)	2292(4)	2323(2)	-218(2)	32(1)
C(4)	1188(3)	3068(2)	62(2)	28(1)
C(5)	-301(3)	2812(2)	635(2)	25(1)
C(6)	-454(3)	1905(2)	865(2)	27(1)
C(7)	-1606(3)	3439(2)	997(2)	29(1)
C(8)	-2935(4)	3026(2)	1561(2)	34(1)
C(9)	-2960(4)	2121(2)	1750(2)	33(1)
C(10)	-4222(5)	1590(2)	2306(3)	45(1)
C(11)	3618(5)	5419(3)	1316(3)	59(1)

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

Ni(1)-O(1)#1	2.0143(17)
Ni(1)-O(1)	2.0143(17)
Ni(1)-O(2)	2.0146(17)
Ni(1)-O(2)#1	2.0146(17)
Ni(1)-O(5)#1	2.078(2)
Ni(1)-O(5)	2.078(2)
Cl(1)-O(8A)	1.325(14)
Cl(1)-O(9)	1.412(3)
Cl(1)-O(8)	1.416(4)
Cl(1)-O(9A)	1.423(16)
Cl(1)-O(6)	1.440(2)
Cl(1)-O(7)	1.442(3)
Cl(1)-O(7A)	1.476(13)
O(1)-C(4)	1.241(3)
O(2)-C(7)	1.251(3)
O(3)-C(6)	1.324(3)
O(3)-C(2)	1.378(3)
O(4)-C(6)	1.329(3)
O(4)-C(9)	1.375(3)
O(5)-C(11)	1.440(4)
O(5)-H(5)	0.73(3)
C(1)-C(2)	1.475(4)
C(1)-H(1A)	0.88(4)
C(1)-H(1B)	0.95(5)
C(1)-H(1C)	0.97(5)
C(2)-C(3)	1.334(4)
C(3)-C(4)	1.430(3)
C(3)-H(3)	0.88(3)
C(4)-C(5)	1.458(3)
C(5)-C(6)	1.359(3)
C(5)-C(7)	1.448(3)
C(7)-C(8)	1.435(4)
C(8)-C(9)	1.338(4)
C(8)-H(8)	0.91(3)

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)

O(9A)-Cl(1)-O(7)	75.8(10)
O(6)-Cl(1)-O(7)	107.72(15)
O(8A)-Cl(1)-O(7A)	122.1(16)
O(9)-Cl(1)-O(7A)	64.2(7)
O(8)-Cl(1)-O(7A)	61.0(8)
O(9A)-Cl(1)-O(7A)	97.9(12)
O(6)-Cl(1)-O(7A)	94.5(6)
O(7)-Cl(1)-O(7A)	157.6(6)
C(4)-O(1)-Ni(1)	127.19(16)
C(7)-O(2)-Ni(1)	127.25(16)
C(6)-O(3)-C(2)	118.49(19)
C(6)-O(4)-C(9)	118.83(19)
C(11)-O(5)-Ni(1)	124.0(2)
C(11)-O(5)-H(5)	109(3)
Ni(1)-O(5)-H(5)	107(3)
C(2)-C(1)-H(1A)	109(3)
C(2)-C(1)-H(1B)	109(3)
H(1A)-C(1)-H(1B)	112(4)
C(2)-C(1)-H(1C)	112(3)
H(1A)-C(1)-H(1C)	107(4)
H(1B)-C(1)-H(1C)	109(4)
C(3)-C(2)-O(3)	120.7(2)
C(3)-C(2)-C(1)	128.8(3)
O(3)-C(2)-C(1)	110.5(2)
C(2)-C(3)-C(4)	122.4(2)
C(2)-C(3)-H(3)	118(2)
C(4)-C(3)-H(3)	120(2)
O(1)-C(4)-C(3)	120.4(2)
O(1)-C(4)-C(5)	124.1(2)
C(3)-C(4)-C(5)	115.5(2)
C(6)-C(5)-C(7)	117.2(2)
C(6)-C(5)-C(4)	116.9(2)
C(7)-C(5)-C(4)	125.8(2)
O(3)-C(6)-O(4)	108.7(2)
O(3)-C(6)-C(5)	125.8(2)
O(4)-C(6)-C(5)	125.5(2)

O(2)-C(7)-C(8)	120.5(2)
O(2)-C(7)-C(5)	123.8(2)
C(8)-C(7)-C(5)	115.7(2)
C(9)-C(8)-C(7)	122.2(2)
C(9)-C(8)-H(8)	119.6(19)
C(7)-C(8)-H(8)	118.1(19)
C(8)-C(9)-O(4)	120.5(2)
C(8)-C(9)-C(10)	129.1(3)
O(4)-C(9)-C(10)	110.4(2)
C(9)-C(10)-H(10A)	112(2)
C(9)-C(10)-H(10B)	106(2)
H(10A)-C(10)-H(10B)	109(3)
C(9)-C(10)-H(10C)	109(2)
H(10A)-C(10)-H(10C)	108(3)
H(10B)-C(10)-H(10C)	113(3)
O(5)-C(11)-H(11A)	111(3)
O(5)-C(11)-H(11B)	112(3)
H(11A)-C(11)-H(11B)	102(4)
O(5)-C(11)-H(11C)	109(4)
H(11A)-C(11)-H(11C)	112(4)
H(11B)-C(11)-H(11C)	111(5)

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **7** (M= Ni). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	39(1)	18(1)	31(1)	2(1)	14(1)	2(1)
Cl(1)	47(1)	58(1)	42(1)	-1(1)	2(1)	18(1)
O(1)	41(1)	21(1)	40(1)	3(1)	16(1)	2(1)
O(2)	43(1)	22(1)	41(1)	5(1)	18(1)	3(1)
O(3)	37(1)	23(1)	52(1)	5(1)	13(1)	2(1)
O(4)	39(1)	24(1)	35(1)	3(1)	10(1)	-2(1)

O(5)	47(1)	37(1)	36(1)	0(1)	9(1)	1(1)
O(6)	72(2)	94(2)	62(2)	39(1)	28(1)	43(2)
O(7)	60(2)	86(2)	43(1)	21(1)	3(1)	25(2)
O(8)	109(3)	103(3)	87(3)	-47(2)	-37(2)	30(2)
O(9)	49(2)	91(2)	105(3)	14(2)	21(2)	11(2)
O(7A)	60(2)	86(2)	43(1)	21(1)	3(1)	25(2)
O(8A)	109(3)	103(3)	87(3)	-47(2)	-37(2)	30(2)
O(9A)	49(2)	91(2)	105(3)	14(2)	21(2)	11(2)
C(1)	58(2)	29(2)	104(3)	4(2)	36(2)	10(2)
C(2)	29(1)	28(1)	54(2)	5(1)	13(1)	4(1)
C(3)	29(1)	25(1)	44(2)	1(1)	12(1)	2(1)
C(4)	30(1)	24(1)	29(1)	0(1)	3(1)	-1(1)
C(5)	29(1)	24(1)	23(1)	1(1)	3(1)	-2(1)
C(6)	28(1)	24(1)	28(1)	0(1)	3(1)	0(1)
C(7)	34(1)	25(1)	27(1)	2(1)	7(1)	1(1)
C(8)	41(2)	28(1)	35(1)	1(1)	16(1)	1(1)
C(9)	38(2)	32(1)	30(1)	0(1)	10(1)	-1(1)
C(10)	57(2)	34(2)	48(2)	1(1)	25(2)	-6(2)
C(11)	51(2)	65(2)	61(2)	5(2)	-1(2)	-8(2)

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

	x	y	z	U(eq)
H(1A)	3810(60)	760(30)	-600(30)	71(12)
H(1B)	3180(70)	230(30)	270(40)	99(16)
H(1C)	2060(70)	250(30)	-780(40)	97(16)
H(3)	3180(50)	2430(20)	-590(20)	52(9)
H(5)	1480(50)	4990(20)	1660(30)	37(10)
H(8)	-3750(40)	3400(20)	1820(20)	45(8)
H(10A)	-5070(50)	1940(30)	2520(30)	61(11)
H(10B)	-4760(50)	1160(30)	1860(30)	68(11)
H(10C)	-3550(50)	1310(20)	2880(30)	58(10)

H(11A)	3910(60)	5990(30)	1050(30)	91(15)
H(11B)	4170(60)	5460(30)	1970(40)	97(15)
H(11C)	4080(80)	4930(40)	980(50)	130(20)

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

O(1)#1-Ni(1)-O(1)-C(4)	176(68)
O(2)-Ni(1)-O(1)-C(4)	7.0(2)
O(2)#1-Ni(1)-O(1)-C(4)	-173.0(2)
O(5)#1-Ni(1)-O(1)-C(4)	-83.4(2)
O(5)-Ni(1)-O(1)-C(4)	96.6(2)
O(1)#1-Ni(1)-O(2)-C(7)	174.2(2)
O(1)-Ni(1)-O(2)-C(7)	-5.8(2)
O(2)#1-Ni(1)-O(2)-C(7)	18(100)
O(5)#1-Ni(1)-O(2)-C(7)	84.5(2)
O(5)-Ni(1)-O(2)-C(7)	-95.5(2)
O(1)#1-Ni(1)-O(5)-C(11)	-124.7(3)
O(1)-Ni(1)-O(5)-C(11)	55.3(3)
O(2)-Ni(1)-O(5)-C(11)	146.8(3)
O(2)#1-Ni(1)-O(5)-C(11)	-33.2(3)
O(5)#1-Ni(1)-O(5)-C(11)	36(13)
C(6)-O(3)-C(2)-C(3)	4.0(4)
C(6)-O(3)-C(2)-C(1)	-173.7(3)
O(3)-C(2)-C(3)-C(4)	-4.0(4)
C(1)-C(2)-C(3)-C(4)	173.3(4)
Ni(1)-O(1)-C(4)-C(3)	173.68(18)
Ni(1)-O(1)-C(4)-C(5)	-5.1(3)
C(2)-C(3)-C(4)-O(1)	-177.9(3)
C(2)-C(3)-C(4)-C(5)	1.0(4)
O(1)-C(4)-C(5)-C(6)	-179.4(2)
C(3)-C(4)-C(5)-C(6)	1.8(3)
O(1)-C(4)-C(5)-C(7)	-0.7(4)
C(3)-C(4)-C(5)-C(7)	-179.6(2)
C(2)-O(3)-C(6)-O(4)	179.3(2)
C(2)-O(3)-C(6)-C(5)	-1.1(4)

C(9)-O(4)-C(6)-O(3)	-178.5(2)
C(9)-O(4)-C(6)-C(5)	1.9(4)
C(7)-C(5)-C(6)-O(3)	179.4(2)
C(4)-C(5)-C(6)-O(3)	-1.8(4)
C(7)-C(5)-C(6)-O(4)	-1.0(4)
C(4)-C(5)-C(6)-O(4)	177.8(2)
Ni(1)-O(2)-C(7)-C(8)	-176.37(18)
Ni(1)-O(2)-C(7)-C(5)	2.6(4)
C(6)-C(5)-C(7)-O(2)	-179.3(2)
C(4)-C(5)-C(7)-O(2)	2.1(4)
C(6)-C(5)-C(7)-C(8)	-0.2(3)
C(4)-C(5)-C(7)-C(8)	-178.9(2)
O(2)-C(7)-C(8)-C(9)	179.6(3)
C(5)-C(7)-C(8)-C(9)	0.6(4)
C(7)-C(8)-C(9)-O(4)	0.3(4)
C(7)-C(8)-C(9)-C(10)	-179.6(3)
C(6)-O(4)-C(9)-C(8)	-1.5(4)
C(6)-O(4)-C(9)-C(10)	178.4(2)

Symmetry transformations used to generate equivalent atoms:

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

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5)...O(6)	0.73(3)	2.07(3)	2.788(3)	172(3)

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) $LiClO_4$ 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:

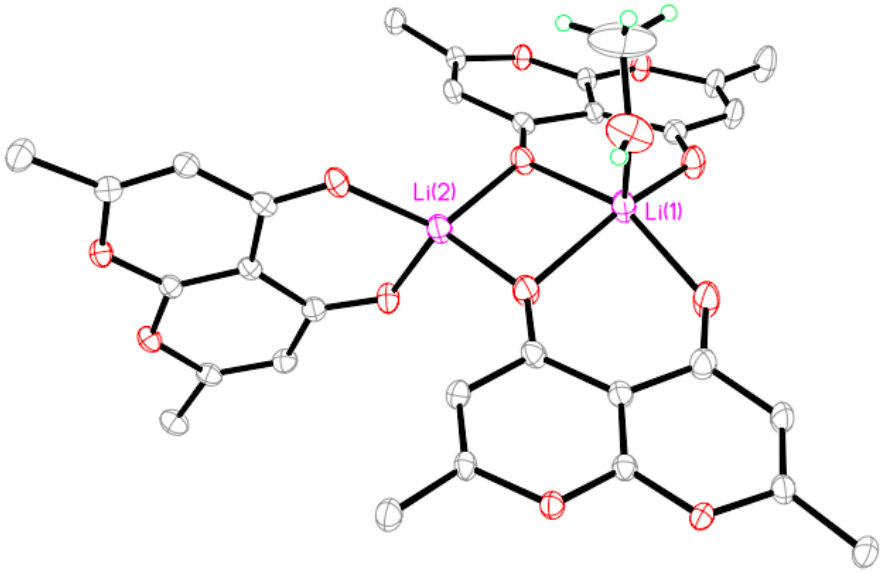


Table 1. Crystal data and structure refinement for **8** (M= Li)

Identification code	**8** (M = Li)	
Empirical formula	C32.50 H32 Cl2 Li2 O22.50	
Formula weight	867.36	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.6532(18) Å	a= 79.690(2)°.
	b = 15.954(2) Å	b= 87.173(2)°.
	c = 18.398(2) Å	g = 76.390(2)°.

Volume	3832.0(9) $Å^3$
Z	4
Density (calculated)	1.503 Mg/m^3
Absorption coefficient	0.259 mm^{-1}
F(000)	1788
Crystal size	0.38 x 0.27 x 0.16 mm^3
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 F^2
Data / restraints / parameters	13455 / 2 / 1219
Goodness-of-fit on F^2	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.

	x	y	z	U(eq)
Li(1)	2479(4)	3394(4)	2050(3)	34(1)
Li(2)	4698(4)	2975(4)	2007(3)	33(1)
O(1)	3674(2)	3351(2)	1261(1)	36(1)
O(2)	1625(2)	3354(2)	1227(1)	56(1)
O(3)	3421(2)	4178(1)	-957(1)	25(1)
O(4)	1861(2)	4115(1)	-988(1)	27(1)
O(5)	3696(2)	2834(2)	2776(1)	31(1)
O(6)	1641(2)	2827(2)	2817(1)	37(1)
O(7)	3828(2)	1266(1)	4782(1)	27(1)
O(8)	2277(2)	1181(2)	4774(1)	31(1)
O(9)	5744(2)	3536(1)	2110(1)	32(1)
O(10)	5561(2)	1871(2)	1891(1)	34(1)
O(11)	8757(2)	2579(1)	1877(1)	28(1)
O(12)	8609(2)	1326(1)	1684(1)	29(1)
O(13)	2254(2)	4655(2)	2029(2)	60(1)
C(1)	5090(3)	4347(3)	-1139(2)	34(1)
C(2)	4324(2)	4075(2)	-611(2)	25(1)
C(3)	4422(3)	3775(2)	116(2)	30(1)
C(4)	3595(2)	3589(2)	574(2)	29(1)
C(5)	2658(2)	3704(2)	193(2)	28(1)
C(6)	2646(2)	3986(2)	-549(2)	24(1)
C(7)	1723(3)	3557(3)	551(2)	39(1)
C(8)	901(3)	3674(3)	46(2)	36(1)
C(9)	972(2)	3939(2)	-673(2)	29(1)
C(10)	175(3)	4110(3)	-1236(2)	34(1)
C(11)	5456(3)	1364(3)	5073(2)	32(1)
C(12)	4638(2)	1626(2)	4521(2)	27(1)
C(13)	4603(2)	2135(2)	3863(2)	27(1)
C(14)	3715(2)	2385(2)	3404(2)	25(1)
C(15)	2857(2)	2069(2)	3731(2)	23(1)
C(16)	2976(2)	1537(2)	4396(2)	26(1)

C(17)	1850(3)	2325(2)	3407(2)	30(1)
C(18)	1102(3)	1944(3)	3844(2)	35(1)
C(19)	1310(3)	1404(2)	4487(2)	33(1)
C(20)	620(4)	968(4)	4988(3)	49(1)
C(21)	9218(3)	3834(3)	2098(3)	41(1)
C(22)	8383(3)	3412(2)	2040(2)	27(1)
C(23)	7400(3)	3721(2)	2121(2)	28(1)
C(24)	6671(2)	3229(2)	2047(2)	26(1)
C(25)	7084(2)	2354(2)	1892(2)	23(1)
C(26)	8100(2)	2101(2)	1821(2)	24(1)
C(27)	6497(2)	1712(2)	1839(2)	28(1)
C(28)	7086(3)	871(2)	1726(2)	29(1)
C(29)	8080(3)	692(2)	1652(2)	28(1)
C(30)	8782(3)	-134(3)	1538(3)	36(1)
C(31)	2217(5)	5119(3)	2602(2)	99(2)
Li(1')	2518(4)	7343(4)	3034(3)	42(2)
Li(2')	407(4)	7246(4)	3027(3)	35(1)
O(1')	1488(2)	6816(2)	3737(1)	34(1)
O(2')	3535(2)	6813(2)	3810(1)	49(1)
O(3')	1741(2)	5751(1)	5918(1)	25(1)
O(4')	3311(2)	5769(1)	5976(1)	26(1)
O(5')	1374(2)	7439(2)	2247(1)	33(1)
O(6')	3455(2)	7379(2)	2174(1)	42(1)
O(7')	1293(2)	8763(1)	140(1)	26(1)
O(8')	2870(2)	8778(2)	106(1)	33(1)
O(9')	-500(2)	8295(2)	3210(1)	35(1)
O(10')	-610(2)	6638(1)	2911(1)	32(1)
O(11')	-3557(2)	8788(1)	3306(1)	32(1)
O(12')	-3643(2)	7537(1)	3068(1)	31(1)
O(13')	2091(2)	8592(2)	3104(2)	62(1)
C(1')	53(3)	5598(3)	6061(2)	33(1)
C(2')	840(2)	5889(2)	5557(2)	27(1)
C(3')	742(3)	6254(2)	4843(2)	30(1)
C(4')	1569(2)	6487(2)	4408(2)	28(1)
C(5')	2510(2)	6326(2)	4799(2)	27(1)
C(6')	2518(2)	5966(2)	5528(2)	24(1)

C(7')	3452(3)	6503(2)	4468(2)	33(1)
C(8')	4297(3)	6267(2)	4968(2)	33(1)
C(9')	4217(2)	5921(2)	5679(2)	28(1)
C(10')	5021(3)	5639(3)	6243(2)	33(1)
C(11')	-376(3)	8725(3)	-89(2)	38(1)
C(12')	456(2)	8479(2)	451(2)	26(1)
C(13')	477(2)	8039(2)	1142(2)	27(1)
C(14')	1364(2)	7816(2)	1593(2)	26(1)
C(15')	2254(2)	8053(2)	1226(2)	25(1)
C(16')	2150(2)	8507(2)	526(2)	24(1)
C(17')	3269(3)	7796(2)	1545(2)	33(1)
C(18')	4035(3)	8080(3)	1061(2)	36(1)
C(19')	3840(3)	8552(2)	387(2)	36(1)
C(20')	4550(3)	8897(4)	-157(3)	47(1)
C(21')	-3775(3)	10226(3)	3532(3)	39(1)
C(22')	-3050(3)	9417(2)	3396(2)	31(1)
C(23')	-2060(3)	9266(2)	3358(2)	31(1)
C(24')	-1440(3)	8434(2)	3231(2)	28(1)
C(25')	-1995(2)	7797(2)	3120(2)	25(1)
C(26')	-3016(3)	8026(2)	3165(2)	28(1)
C(27')	-1542(3)	6922(2)	2962(2)	28(1)
C(28')	-2250(3)	6420(2)	2855(2)	30(1)
C(29')	-3236(3)	6709(2)	2914(2)	28(1)
C(30')	-4054(3)	6268(3)	2831(3)	42(1)
C(31')	1828(5)	8885(4)	3780(3)	108(2)
Cl(1)	2279(1)	6214(1)	9983(1)	32(1)
Cl(2)	7069(1)	8300(1)	1083(1)	35(1)
Cl(3)	7392(1)	6304(1)	4915(1)	30(1)
Cl(4)	8013(1)	1786(1)	3869(1)	30(1)
O(14)	2330(2)	6024(2)	10778(1)	45(1)
O(15)	1920(3)	7120(2)	9751(2)	65(1)
O(16)	3240(2)	5907(2)	9672(2)	51(1)
O(17)	1586(2)	5766(2)	9737(2)	60(1)
O(18)	7075(3)	8808(3)	383(2)	126(2)
O(19)	6587(2)	8859(2)	1585(2)	82(1)
O(20)	6515(2)	7658(2)	1091(2)	74(1)

O(21)	8083(2)	7900(2)	1319(1)	41(1)
O(22)	6968(2)	7211(2)	4690(1)	54(1)
O(23)	8378(2)	6066(2)	4601(1)	47(1)
O(24)	6753(2)	5823(2)	4653(2)	52(1)
O(25)	7434(2)	6096(2)	5705(1)	45(1)
O(26)	7990(2)	1202(2)	4540(2)	73(1)
O(27)	8587(2)	2395(2)	3937(2)	76(1)
O(28)	8474(2)	1282(2)	3324(2)	57(1)
O(29)	7006(2)	2240(2)	3653(1)	43(1)
O(1S)	1313(3)	9984(2)	1992(2)	86(1)
C(1S)	960(5)	10764(4)	2337(4)	115(2)

Table 3. Bond lengths [Å] and angles [°] for **8**.

Li(1)-O(13)	1.957(7)
Li(1)-O(2)	1.975(6)
Li(1)-O(6)	1.997(6)
Li(1)-O(5)	2.105(6)
Li(1)-O(1)	2.127(6)
Li(1)-Li(2)	2.944(8)
Li(2)-O(9)	1.889(6)
Li(2)-O(10)	1.916(6)
Li(2)-O(1)	1.923(6)
Li(2)-O(5)	1.940(6)
O(1)-C(4)	1.255(4)
O(2)-C(7)	1.237(4)
O(3)-C(6)	1.330(4)
O(3)-C(2)	1.377(4)
O(4)-C(6)	1.328(4)
O(4)-C(9)	1.386(4)
O(5)-C(14)	1.243(4)
O(6)-C(17)	1.230(4)
O(7)-C(16)	1.335(4)
O(7)-C(12)	1.390(4)
O(8)-C(16)	1.329(4)
O(8)-C(19)	1.389(4)
O(9)-C(24)	1.252(4)
O(10)-C(27)	1.246(4)
O(11)-C(26)	1.326(4)
O(11)-C(22)	1.388(4)
O(12)-C(26)	1.328(4)
O(12)-C(29)	1.385(4)
O(13)-C(31)	1.386(5)
O(13)-H(13A)	0.9500
C(1)-C(2)	1.478(5)
C(1)-H(1A)	0.94(4)
C(1)-H(1B)	0.97(3)
C(1)-H(1C)	0.95(4)

C(2)-C(3)	1.341(5)
C(3)-C(4)	1.429(5)
C(3)-H(3)	0.82(3)
C(4)-C(5)	1.448(4)
C(5)-C(6)	1.358(4)
C(5)-C(7)	1.456(5)
C(7)-C(8)	1.449(5)
C(8)-C(9)	1.320(5)
C(8)-H(8)	0.82(4)
C(9)-C(10)	1.484(5)
C(10)-H(10A)	0.85(4)
C(10)-H(10B)	1.02(3)
C(10)-H(10C)	1.02(4)
C(11)-C(12)	1.482(5)
C(11)-H(11A)	0.99(3)
C(11)-H(11B)	0.96(3)
C(11)-H(11C)	0.93(4)
C(12)-C(13)	1.327(4)
C(13)-C(14)	1.447(4)
C(13)-H(13)	0.93(3)
C(14)-C(15)	1.446(4)
C(15)-C(16)	1.353(4)
C(15)-C(17)	1.463(4)
C(17)-C(18)	1.447(5)
C(18)-C(19)	1.332(5)
C(18)-H(18)	0.92(4)
C(19)-C(20)	1.487(5)
C(20)-H(20A)	0.90(4)
C(20)-H(20B)	0.94(5)
C(20)-H(20C)	0.94(4)
C(21)-C(22)	1.471(5)
C(21)-H(21A)	0.81(4)
C(21)-H(21B)	0.94(4)
C(21)-H(21C)	0.99(3)
C(22)-C(23)	1.326(4)
C(23)-C(24)	1.430(5)

C(23)-H(23)	0.86(3)
C(24)-C(25)	1.451(4)
C(25)-C(26)	1.357(4)
C(25)-C(27)	1.460(4)
C(27)-C(28)	1.437(5)
C(28)-C(29)	1.324(5)
C(28)-H(28)	0.93(3)
C(29)-C(30)	1.479(5)
C(30)-H(30A)	0.96(4)
C(30)-H(30B)	0.90(4)
C(30)-H(30C)	0.90(4)
C(31)-H(31A)	0.9800
C(31)-H(31B)	0.9800
C(31)-H(31C)	0.9800
Li(1')-O(13')	1.966(7)
Li(1')-O(2')	1.974(6)
Li(1')-O(6')	1.987(6)
Li(1')-O(1')	2.085(6)
Li(1')-O(5')	2.142(7)
Li(1')-Li(2')	2.923(8)
Li(2')-O(9')	1.906(6)
Li(2')-O(10')	1.911(6)
Li(2')-O(5')	1.935(6)
Li(2')-O(1')	1.935(6)
O(1')-C(4')	1.252(4)
O(2')-C(7')	1.233(4)
O(3')-C(6')	1.329(4)
O(3')-C(2')	1.380(4)
O(4')-C(6')	1.337(4)
O(4')-C(9')	1.383(4)
O(5')-C(14')	1.246(4)
O(6')-C(17')	1.232(4)
O(7')-C(16')	1.340(4)
O(7')-C(12')	1.387(4)
O(8')-C(16')	1.326(4)
O(8')-C(19')	1.389(4)

O(9')-C(24')	1.250(4)
O(10')-C(27')	1.250(4)
O(11')-C(26')	1.327(4)
O(11')-C(22')	1.383(4)
O(12')-C(26')	1.325(4)
O(12')-C(29')	1.380(4)
O(13')-C(31')	1.406(5)
O(13')-H(13B)	0.9500
C(1')-C(2')	1.486(5)
C(1')-H(1D)	0.92(4)
C(1')-H(1E)	1.02(4)
C(1')-H(1F)	1.01(4)
C(2')-C(3')	1.338(5)
C(3')-C(4')	1.434(5)
C(3')-H(3')	0.99(3)
C(4')-C(5')	1.452(4)
C(5')-C(6')	1.360(4)
C(5')-C(7')	1.462(5)
C(7')-C(8')	1.446(5)
C(8')-C(9')	1.336(5)
C(8')-H(8')	0.92(3)
C(9')-C(10')	1.483(5)
C(10')-H(10D)	1.00(4)
C(10')-H(10E)	0.86(3)
C(10')-H(10F)	1.03(3)
C(11')-C(12')	1.482(5)
C(11')-H(11D)	0.87(4)
C(11')-H(11E)	0.94(5)
C(11')-H(11F)	0.93(4)
C(12')-C(13')	1.336(4)
C(13')-C(14')	1.439(4)
C(13')-H(13')	0.95(3)
C(14')-C(15')	1.456(4)
C(15')-C(16')	1.356(4)
C(15')-C(17')	1.469(4)
C(17')-C(18')	1.442(5)

C(18')-C(19')	1.333(5)
C(18')-H(18')	0.96(3)
C(19')-C(20')	1.483(5)
C(20')-H(20D)	0.89(4)
C(20')-H(20E)	0.99(4)
C(20')-H(20F)	0.95(5)
C(21')-C(22')	1.483(5)
C(21')-H(21D)	0.88(3)
C(21')-H(21E)	1.00(4)
C(21')-H(21F)	0.97(4)
C(22')-C(23')	1.317(5)
C(23')-C(24')	1.447(5)
C(23')-H(23')	1.00(4)
C(24')-C(25')	1.450(4)
C(25')-C(26')	1.357(4)
C(25')-C(27')	1.462(4)
C(27')-C(28')	1.433(5)
C(28')-C(29')	1.323(5)
C(28')-H(28')	0.84(3)
C(29')-C(30')	1.479(5)
C(30')-H(30D)	1.04(5)
C(30')-H(30E)	0.95(6)
C(30')-H(30F)	0.92(4)
C(31')-H(31D)	0.9800
C(31')-H(31E)	0.9800
C(31')-H(31F)	0.9800
Cl(1)-O(15)	1.405(3)
Cl(1)-O(16)	1.419(3)
Cl(1)-O(14)	1.442(2)
Cl(1)-O(17)	1.445(3)
Cl(2)-O(18)	1.395(3)
Cl(2)-O(20)	1.408(3)
Cl(2)-O(19)	1.429(3)
Cl(2)-O(21)	1.434(2)
Cl(3)-O(22)	1.419(3)
Cl(3)-O(23)	1.433(2)

Cl(3)-O(25)	1.433(2)
Cl(3)-O(24)	1.441(3)
Cl(4)-O(27)	1.408(3)
Cl(4)-O(26)	1.412(3)
Cl(4)-O(28)	1.428(3)
Cl(4)-O(29)	1.434(2)
O(1S)-C(1S)	1.467(6)

O(13)-Li(1)-O(2)	101.6(3)
O(13)-Li(1)-O(6)	112.6(3)
O(2)-Li(1)-O(6)	93.6(3)
O(13)-Li(1)-O(5)	104.7(3)
O(2)-Li(1)-O(5)	151.6(4)
O(6)-Li(1)-O(5)	86.2(2)
O(13)-Li(1)-O(1)	94.3(3)
O(2)-Li(1)-O(1)	86.1(2)
O(6)-Li(1)-O(1)	152.6(3)
O(5)-Li(1)-O(1)	81.4(2)
O(13)-Li(1)-Li(2)	98.1(3)
O(2)-Li(1)-Li(2)	124.3(3)
O(6)-Li(1)-Li(2)	125.1(3)
O(5)-Li(1)-Li(2)	41.13(16)
O(1)-Li(1)-Li(2)	40.70(16)
O(9)-Li(2)-O(10)	96.0(3)
O(9)-Li(2)-O(1)	125.8(3)
O(10)-Li(2)-O(1)	111.0(3)
O(9)-Li(2)-O(5)	122.1(3)
O(10)-Li(2)-O(5)	111.3(3)
O(1)-Li(2)-O(5)	91.2(3)
O(9)-Li(2)-Li(1)	137.7(3)
O(10)-Li(2)-Li(1)	126.2(3)
O(1)-Li(2)-Li(1)	46.16(18)
O(5)-Li(2)-Li(1)	45.54(17)
C(4)-O(1)-Li(2)	139.6(3)
C(4)-O(1)-Li(1)	126.9(3)
Li(2)-O(1)-Li(1)	93.1(2)

C(7)-O(2)-Li(1)	131.0(3)
C(6)-O(3)-C(2)	118.4(2)
C(6)-O(4)-C(9)	118.0(3)
C(14)-O(5)-Li(2)	134.3(3)
C(14)-O(5)-Li(1)	130.0(3)
Li(2)-O(5)-Li(1)	93.3(2)
C(17)-O(6)-Li(1)	132.7(3)
C(16)-O(7)-C(12)	117.7(2)
C(16)-O(8)-C(19)	117.9(3)
C(24)-O(9)-Li(2)	126.8(3)
C(27)-O(10)-Li(2)	125.7(3)
C(26)-O(11)-C(22)	117.6(2)
C(26)-O(12)-C(29)	118.3(2)
C(31)-O(13)-Li(1)	130.1(3)
C(31)-O(13)-H(13A)	114.9
Li(1)-O(13)-H(13A)	114.9
C(2)-C(1)-H(1A)	106(2)
C(2)-C(1)-H(1B)	111.0(18)
H(1A)-C(1)-H(1B)	111(3)
C(2)-C(1)-H(1C)	118(2)
H(1A)-C(1)-H(1C)	101(3)
H(1B)-C(1)-H(1C)	110(3)
C(3)-C(2)-O(3)	120.7(3)
C(3)-C(2)-C(1)	128.1(3)
O(3)-C(2)-C(1)	111.2(3)
C(2)-C(3)-C(4)	122.3(3)
C(2)-C(3)-H(3)	115(2)
C(4)-C(3)-H(3)	122(2)
O(1)-C(4)-C(3)	122.3(3)
O(1)-C(4)-C(5)	122.2(3)
C(3)-C(4)-C(5)	115.5(3)
C(6)-C(5)-C(4)	117.6(3)
C(6)-C(5)-C(7)	117.8(3)
C(4)-C(5)-C(7)	124.6(3)
O(4)-C(6)-O(3)	108.7(3)
O(4)-C(6)-C(5)	125.8(3)

O(3)-C(6)-C(5)	125.5(3)
O(2)-C(7)-C(8)	122.2(3)
O(2)-C(7)-C(5)	123.6(3)
C(8)-C(7)-C(5)	114.2(3)
C(9)-C(8)-C(7)	123.1(3)
C(9)-C(8)-H(8)	115(3)
C(7)-C(8)-H(8)	122(3)
C(8)-C(9)-O(4)	120.9(3)
C(8)-C(9)-C(10)	127.8(3)
O(4)-C(9)-C(10)	111.3(3)
C(9)-C(10)-H(10A)	109(3)
C(9)-C(10)-H(10B)	108.5(18)
H(10A)-C(10)-H(10B)	110(3)
C(9)-C(10)-H(10C)	110(2)
H(10A)-C(10)-H(10C)	109(3)
H(10B)-C(10)-H(10C)	109(3)
C(12)-C(11)-H(11A)	110.3(17)
C(12)-C(11)-H(11B)	114(2)
H(11A)-C(11)-H(11B)	104(3)
C(12)-C(11)-H(11C)	112(2)
H(11A)-C(11)-H(11C)	109(3)
H(11B)-C(11)-H(11C)	107(3)
C(13)-C(12)-O(7)	120.8(3)
C(13)-C(12)-C(11)	128.9(3)
O(7)-C(12)-C(11)	110.3(3)
C(12)-C(13)-C(14)	122.5(3)
C(12)-C(13)-H(13)	116(2)
C(14)-C(13)-H(13)	122(2)
O(5)-C(14)-C(15)	122.7(3)
O(5)-C(14)-C(13)	122.5(3)
C(15)-C(14)-C(13)	114.8(3)
C(16)-C(15)-C(14)	118.1(3)
C(16)-C(15)-C(17)	117.6(3)
C(14)-C(15)-C(17)	124.2(3)
O(8)-C(16)-O(7)	108.1(3)
O(8)-C(16)-C(15)	126.4(3)

O(7)-C(16)-C(15)	125.6(3)
O(6)-C(17)-C(18)	122.0(3)
O(6)-C(17)-C(15)	123.6(3)
C(18)-C(17)-C(15)	114.4(3)
C(19)-C(18)-C(17)	123.0(3)
C(19)-C(18)-H(18)	118(2)
C(17)-C(18)-H(18)	119(2)
C(18)-C(19)-O(8)	120.6(3)
C(18)-C(19)-C(20)	128.6(3)
O(8)-C(19)-C(20)	110.8(3)
C(19)-C(20)-H(20A)	109(3)
C(19)-C(20)-H(20B)	108(3)
H(20A)-C(20)-H(20B)	109(4)
C(19)-C(20)-H(20C)	112(2)
H(20A)-C(20)-H(20C)	105(4)
H(20B)-C(20)-H(20C)	114(4)
C(22)-C(21)-H(21A)	113(3)
C(22)-C(21)-H(21B)	114(2)
H(21A)-C(21)-H(21B)	104(4)
C(22)-C(21)-H(21C)	108.8(19)
H(21A)-C(21)-H(21C)	112(4)
H(21B)-C(21)-H(21C)	105(3)
C(23)-C(22)-O(11)	120.8(3)
C(23)-C(22)-C(21)	129.3(3)
O(11)-C(22)-C(21)	109.9(3)
C(22)-C(23)-C(24)	122.9(3)
C(22)-C(23)-H(23)	112(2)
C(24)-C(23)-H(23)	125(2)
O(9)-C(24)-C(23)	122.1(3)
O(9)-C(24)-C(25)	122.8(3)
C(23)-C(24)-C(25)	115.1(3)
C(26)-C(25)-C(24)	117.3(3)
C(26)-C(25)-C(27)	117.5(3)
C(24)-C(25)-C(27)	125.1(3)
O(11)-C(26)-O(12)	108.1(3)
O(11)-C(26)-C(25)	126.2(3)

O(12)-C(26)-C(25)	125.7(3)
O(10)-C(27)-C(28)	122.2(3)
O(10)-C(27)-C(25)	123.2(3)
C(28)-C(27)-C(25)	114.6(3)
C(29)-C(28)-C(27)	123.3(3)
C(29)-C(28)-H(28)	116.6(19)
C(27)-C(28)-H(28)	120.0(19)
C(28)-C(29)-O(12)	120.4(3)
C(28)-C(29)-C(30)	129.4(3)
O(12)-C(29)-C(30)	110.2(3)
C(29)-C(30)-H(30A)	109(2)
C(29)-C(30)-H(30B)	109(3)
H(30A)-C(30)-H(30B)	113(4)
C(29)-C(30)-H(30C)	109(2)
H(30A)-C(30)-H(30C)	107(3)
H(30B)-C(30)-H(30C)	109(4)
O(13)-C(31)-H(31A)	109.5
O(13)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
O(13)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
O(13')-Li(1')-O(2')	106.2(3)
O(13')-Li(1')-O(6')	101.1(3)
O(2')-Li(1')-O(6')	97.8(3)
O(13')-Li(1')-O(1')	101.8(3)
O(2')-Li(1')-O(1')	87.4(2)
O(6')-Li(1')-O(1')	154.1(4)
O(13')-Li(1')-O(5')	92.8(3)
O(2')-Li(1')-O(5')	159.3(4)
O(6')-Li(1')-O(5')	86.0(2)
O(1')-Li(1')-O(5')	80.9(2)
O(13')-Li(1')-Li(2')	89.9(3)
O(2')-Li(1')-Li(2')	128.7(3)
O(6')-Li(1')-Li(2')	127.0(3)
O(1')-Li(1')-Li(2')	41.38(17)

O(5')-Li(1')-Li(2')	41.43(17)
O(9')-Li(2')-O(10')	95.9(3)
O(9')-Li(2')-O(5')	113.6(3)
O(10')-Li(2')-O(5')	121.0(3)
O(9')-Li(2')-O(1')	113.3(3)
O(10')-Li(2')-O(1')	124.2(3)
O(5')-Li(2')-O(1')	90.2(3)
O(9')-Li(2')-Li(1')	112.7(3)
O(10')-Li(2')-Li(1')	151.4(3)
O(5')-Li(2')-Li(1')	47.09(19)
O(1')-Li(2')-Li(1')	45.42(18)
C(4')-O(1')-Li(2')	136.6(3)
C(4')-O(1')-Li(1')	129.2(3)
Li(2')-O(1')-Li(1')	93.2(3)
C(7')-O(2')-Li(1')	131.6(3)
C(6')-O(3')-C(2')	117.9(2)
C(6')-O(4')-C(9')	118.1(3)
C(14')-O(5')-Li(2')	137.7(3)
C(14')-O(5')-Li(1')	125.3(3)
Li(2')-O(5')-Li(1')	91.5(3)
C(17')-O(6')-Li(1')	127.4(3)
C(16')-O(7')-C(12')	117.8(2)
C(16')-O(8')-C(19')	117.9(3)
C(24')-O(9')-Li(2')	126.1(3)
C(27')-O(10')-Li(2')	126.5(3)
C(26')-O(11')-C(22')	118.0(3)
C(26')-O(12')-C(29')	118.0(3)
C(31')-O(13')-Li(1')	122.5(4)
C(31')-O(13')-H(13B)	118.7
Li(1')-O(13')-H(13B)	118.7
C(2')-C(1')-H(1D)	115(3)
C(2')-C(1')-H(1E)	109(2)
H(1D)-C(1')-H(1E)	105(3)
C(2')-C(1')-H(1F)	108(2)
H(1D)-C(1')-H(1F)	106(3)
H(1E)-C(1')-H(1F)	114(3)

C(3')-C(2')-O(3')	121.0(3)
C(3')-C(2')-C(1')	127.4(3)
O(3')-C(2')-C(1')	111.6(3)
C(2')-C(3')-C(4')	122.2(3)
C(2')-C(3')-H(3')	118.0(18)
C(4')-C(3')-H(3')	119.7(18)
O(1')-C(4')-C(3')	122.6(3)
O(1')-C(4')-C(5')	121.9(3)
C(3')-C(4')-C(5')	115.6(3)
C(6')-C(5')-C(4')	117.2(3)
C(6')-C(5')-C(7')	117.7(3)
C(4')-C(5')-C(7')	125.1(3)
O(3')-C(6')-O(4')	108.2(3)
O(3')-C(6')-C(5')	126.1(3)
O(4')-C(6')-C(5')	125.7(3)
O(2')-C(7')-C(8')	122.0(3)
O(2')-C(7')-C(5')	123.1(3)
C(8')-C(7')-C(5')	114.9(3)
C(9')-C(8')-C(7')	122.4(3)
C(9')-C(8')-H(8')	118(2)
C(7')-C(8')-H(8')	120(2)
C(8')-C(9')-O(4')	121.2(3)
C(8')-C(9')-C(10')	127.8(3)
O(4')-C(9')-C(10')	111.0(3)
C(9')-C(10')-H(10D)	113(2)
C(9')-C(10')-H(10E)	112(2)
H(10D)-C(10')-H(10E)	109(3)
C(9')-C(10')-H(10F)	111.5(18)
H(10D)-C(10')-H(10F)	103(3)
H(10E)-C(10')-H(10F)	107(3)
C(12')-C(11')-H(11D)	113(3)
C(12')-C(11')-H(11E)	112(3)
H(11D)-C(11')-H(11E)	110(4)
C(12')-C(11')-H(11F)	109(3)
H(11D)-C(11')-H(11F)	107(4)
H(11E)-C(11')-H(11F)	105(4)

C(13')-C(12')-O(7')	121.0(3)
C(13')-C(12')-C(11')	128.7(3)
O(7')-C(12')-C(11')	110.3(3)
C(12')-C(13')-C(14')	122.3(3)
C(12')-C(13')-H(13')	113(2)
C(14')-C(13')-H(13')	125(2)
O(5')-C(14')-C(13')	122.5(3)
O(5')-C(14')-C(15')	122.2(3)
C(13')-C(14')-C(15')	115.3(3)
C(16')-C(15')-C(14')	117.7(3)
C(16')-C(15')-C(17')	117.5(3)
C(14')-C(15')-C(17')	124.7(3)
O(8')-C(16')-O(7')	108.0(3)
O(8')-C(16')-C(15')	126.4(3)
O(7')-C(16')-C(15')	125.6(3)
O(6')-C(17')-C(18')	122.5(3)
O(6')-C(17')-C(15')	123.3(3)
C(18')-C(17')-C(15')	114.2(3)
C(19')-C(18')-C(17')	123.2(3)
C(19')-C(18')-H(18')	119(2)
C(17')-C(18')-H(18')	118(2)
C(18')-C(19')-O(8')	120.7(3)
C(18')-C(19')-C(20')	128.5(3)
O(8')-C(19')-C(20')	110.8(3)
C(19')-C(20')-H(20D)	109(3)
C(19')-C(20')-H(20E)	114(2)
H(20D)-C(20')-H(20E)	109(4)
C(19')-C(20')-H(20F)	110(3)
H(20D)-C(20')-H(20F)	114(4)
H(20E)-C(20')-H(20F)	101(3)
C(22')-C(21')-H(21D)	113(2)
C(22')-C(21')-H(21E)	112(2)
H(21D)-C(21')-H(21E)	111(3)
C(22')-C(21')-H(21F)	113(2)
H(21D)-C(21')-H(21F)	99(3)
H(21E)-C(21')-H(21F)	107(3)

C(23')-C(22')-O(11')	121.5(3)
C(23')-C(22')-C(21')	128.2(4)
O(11')-C(22')-C(21')	110.3(3)
C(22')-C(23')-C(24')	122.3(3)
C(22')-C(23')-H(23')	123(2)
C(24')-C(23')-H(23')	115(2)
O(9')-C(24')-C(23')	121.5(3)
O(9')-C(24')-C(25')	123.7(3)
C(23')-C(24')-C(25')	114.8(3)
C(26')-C(25')-C(24')	117.9(3)
C(26')-C(25')-C(27')	117.1(3)
C(24')-C(25')-C(27')	125.0(3)
O(12')-C(26')-O(11')	108.3(3)
O(12')-C(26')-C(25')	126.2(3)
O(11')-C(26')-C(25')	125.5(3)
O(10')-C(27')-C(28')	122.5(3)
O(10')-C(27')-C(25')	122.8(3)
C(28')-C(27')-C(25')	114.7(3)
C(29')-C(28')-C(27')	123.2(3)
C(29')-C(28')-H(28')	119(2)
C(27')-C(28')-H(28')	118(2)
C(28')-C(29')-O(12')	120.8(3)
C(28')-C(29')-C(30')	129.4(3)
O(12')-C(29')-C(30')	109.8(3)
C(29')-C(30')-H(30D)	113(3)
C(29')-C(30')-H(30E)	115(3)
H(30D)-C(30')-H(30E)	95(4)
C(29')-C(30')-H(30F)	110(2)
H(30D)-C(30')-H(30F)	113(3)
H(30E)-C(30')-H(30F)	110(4)
O(13')-C(31')-H(31D)	109.5
O(13')-C(31')-H(31E)	109.5
H(31D)-C(31')-H(31E)	109.5
O(13')-C(31')-H(31F)	109.5
H(31D)-C(31')-H(31F)	109.5
H(31E)-C(31')-H(31F)	109.5

O(15)-Cl(1)-O(16)	111.38(19)
O(15)-Cl(1)-O(14)	109.48(16)
O(16)-Cl(1)-O(14)	110.17(16)
O(15)-Cl(1)-O(17)	108.58(19)
O(16)-Cl(1)-O(17)	107.95(17)
O(14)-Cl(1)-O(17)	109.22(17)
O(18)-Cl(2)-O(20)	111.4(3)
O(18)-Cl(2)-O(19)	108.3(3)
O(20)-Cl(2)-O(19)	107.5(2)
O(18)-Cl(2)-O(21)	109.94(18)
O(20)-Cl(2)-O(21)	110.43(16)
O(19)-Cl(2)-O(21)	109.26(18)
O(22)-Cl(3)-O(23)	110.48(17)
O(22)-Cl(3)-O(25)	109.90(16)
O(23)-Cl(3)-O(25)	110.47(15)
O(22)-Cl(3)-O(24)	108.08(18)
O(23)-Cl(3)-O(24)	108.75(16)
O(25)-Cl(3)-O(24)	109.12(16)
O(27)-Cl(4)-O(26)	111.1(2)
O(27)-Cl(4)-O(28)	108.68(19)
O(26)-Cl(4)-O(28)	107.65(19)
O(27)-Cl(4)-O(29)	109.74(17)
O(26)-Cl(4)-O(29)	109.71(16)
O(28)-Cl(4)-O(29)	109.96(16)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **8**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Li(1)	24(3)	47(4)	28(3)	3(3)	0(3)	-6(3)
Li(2)	25(3)	43(4)	27(3)	-2(3)	0(2)	-4(3)
O(1)	27(1)	51(2)	24(1)	1(1)	-3(1)	-3(1)
O(2)	30(2)	114(3)	24(2)	1(2)	4(1)	-24(2)
O(3)	21(1)	29(1)	24(1)	-3(1)	-1(1)	-4(1)
O(4)	21(1)	35(1)	24(1)	-5(1)	-4(1)	-7(1)
O(5)	24(1)	40(1)	24(1)	2(1)	2(1)	-4(1)
O(6)	24(1)	50(2)	28(1)	8(1)	-4(1)	-6(1)
O(7)	21(1)	29(1)	28(1)	1(1)	-4(1)	-5(1)
O(8)	24(1)	37(1)	30(1)	5(1)	-3(1)	-12(1)
O(9)	22(1)	32(1)	36(1)	-5(1)	2(1)	1(1)
O(10)	23(1)	36(1)	43(2)	-5(1)	-1(1)	-6(1)
O(11)	23(1)	26(1)	32(1)	-2(1)	0(1)	-4(1)
O(12)	26(1)	25(1)	33(1)	-5(1)	4(1)	-1(1)
O(13)	82(2)	45(2)	39(2)	8(1)	2(2)	1(2)
C(1)	25(2)	43(3)	32(2)	-2(2)	-3(2)	-9(2)
C(2)	17(2)	22(2)	33(2)	-5(2)	-2(1)	0(1)
C(3)	21(2)	34(2)	31(2)	-6(2)	-6(2)	-2(2)
C(4)	23(2)	30(2)	28(2)	-3(2)	1(2)	2(2)
C(5)	23(2)	34(2)	25(2)	-5(2)	0(2)	-3(2)
C(6)	22(2)	23(2)	27(2)	-4(1)	-1(2)	-3(1)
C(7)	29(2)	57(3)	28(2)	-2(2)	-1(2)	-10(2)
C(8)	22(2)	57(3)	32(2)	-7(2)	2(2)	-14(2)
C(9)	21(2)	33(2)	35(2)	-8(2)	1(2)	-7(2)
C(10)	25(2)	48(3)	33(2)	-8(2)	-3(2)	-13(2)
C(11)	24(2)	42(3)	28(2)	-1(2)	-2(2)	-8(2)
C(12)	21(2)	28(2)	30(2)	-5(2)	1(2)	-4(2)
C(13)	19(2)	32(2)	30(2)	-5(2)	3(2)	-3(2)
C(14)	24(2)	24(2)	27(2)	-6(2)	3(2)	-2(1)
C(15)	18(2)	26(2)	23(2)	-4(1)	1(1)	-2(1)
C(16)	22(2)	26(2)	28(2)	-4(2)	1(2)	-7(2)

C(17)	27(2)	37(2)	26(2)	-5(2)	-2(2)	-5(2)
C(18)	22(2)	53(2)	30(2)	2(2)	-5(2)	-13(2)
C(19)	25(2)	42(2)	33(2)	0(2)	-6(2)	-13(2)
C(20)	35(3)	72(4)	42(3)	12(3)	-5(2)	-27(3)
C(21)	33(2)	38(3)	53(3)	-12(2)	-7(2)	-8(2)
C(22)	32(2)	23(2)	25(2)	-1(1)	-3(2)	-4(2)
C(23)	27(2)	22(2)	33(2)	-8(2)	0(2)	-1(2)
C(24)	28(2)	25(2)	21(2)	0(1)	0(2)	0(2)
C(25)	25(2)	25(2)	17(2)	1(1)	0(1)	-3(2)
C(26)	25(2)	26(2)	21(2)	-2(1)	1(1)	-5(2)
C(27)	26(2)	33(2)	21(2)	0(2)	-2(2)	-6(2)
C(28)	33(2)	24(2)	30(2)	-5(2)	0(2)	-9(2)
C(29)	33(2)	26(2)	24(2)	-3(2)	4(2)	-5(2)
C(30)	40(2)	28(2)	37(3)	-7(2)	6(2)	-3(2)
C(31)	179(7)	72(4)	37(3)	-10(3)	-4(3)	-11(4)
Li(1')	31(3)	56(4)	30(3)	9(3)	-2(3)	-4(3)
Li(2')	27(3)	38(3)	31(3)	3(3)	-1(3)	0(3)
O(1')	27(1)	48(2)	23(1)	5(1)	-2(1)	-7(1)
O(2')	30(1)	81(2)	28(2)	11(1)	2(1)	-12(1)
O(3')	21(1)	31(1)	22(1)	-2(1)	-1(1)	-6(1)
O(4')	22(1)	30(1)	25(1)	-1(1)	-1(1)	-6(1)
O(5')	30(1)	42(2)	24(1)	2(1)	1(1)	-7(1)
O(6')	26(1)	64(2)	26(1)	6(1)	-2(1)	-1(1)
O(7')	22(1)	27(1)	26(1)	4(1)	-3(1)	-5(1)
O(8')	24(1)	42(2)	30(1)	5(1)	-1(1)	-12(1)
O(9')	26(1)	35(1)	44(2)	-7(1)	-1(1)	-7(1)
O(10')	23(1)	32(1)	37(1)	-6(1)	1(1)	1(1)
O(11')	28(1)	26(1)	41(2)	-9(1)	6(1)	-2(1)
O(12')	26(1)	28(1)	38(1)	-6(1)	0(1)	-6(1)
O(13')	80(2)	66(2)	36(2)	-11(2)	5(2)	-11(2)
C(1')	28(2)	39(2)	30(2)	1(2)	3(2)	-7(2)
C(2')	26(2)	25(2)	31(2)	-6(2)	-2(2)	-6(2)
C(3')	24(2)	33(2)	31(2)	-3(2)	-2(2)	-6(2)
C(4')	29(2)	29(2)	24(2)	-3(2)	1(2)	-4(2)
C(5')	24(2)	29(2)	25(2)	0(2)	0(1)	-4(2)
C(6')	22(2)	24(2)	26(2)	-5(2)	0(2)	-2(1)

C(7')	26(2)	40(2)	26(2)	0(2)	1(2)	-3(2)
C(8')	23(2)	41(2)	33(2)	-2(2)	4(2)	-9(2)
C(9')	26(2)	25(2)	31(2)	-5(2)	1(2)	-4(2)
C(10')	26(2)	42(3)	30(2)	1(2)	-4(2)	-14(2)
C(11')	26(2)	51(3)	35(2)	-2(2)	-3(2)	-12(2)
C(12')	18(2)	28(2)	32(2)	-6(2)	2(2)	-5(1)
C(13')	18(2)	31(2)	30(2)	-6(2)	1(2)	-2(2)
C(14')	27(2)	26(2)	24(2)	-5(2)	5(2)	-2(2)
C(15')	20(2)	28(2)	24(2)	-3(2)	2(1)	-1(1)
C(16')	18(2)	28(2)	26(2)	-3(2)	-3(1)	-4(1)
C(17')	24(2)	38(2)	30(2)	-4(2)	-2(2)	4(2)
C(18')	20(2)	57(3)	28(2)	0(2)	-3(2)	-8(2)
C(19')	24(2)	50(2)	32(2)	-3(2)	0(2)	-11(2)
C(20')	29(2)	78(4)	34(3)	8(3)	-4(2)	-25(2)
C(21')	36(2)	29(2)	49(3)	-7(2)	9(2)	-1(2)
C(22')	35(2)	29(2)	29(2)	-6(2)	5(2)	-7(2)
C(23')	38(2)	26(2)	28(2)	-5(2)	-1(2)	-9(2)
C(24')	33(2)	29(2)	22(2)	1(2)	-5(2)	-8(2)
C(25')	29(2)	23(2)	20(2)	-1(1)	-1(1)	-4(2)
C(26')	31(2)	27(2)	24(2)	-2(2)	0(2)	-4(2)
C(27')	31(2)	30(2)	19(2)	-2(1)	0(2)	-3(2)
C(28')	35(2)	23(2)	30(2)	-6(2)	1(2)	-4(2)
C(29')	32(2)	26(2)	24(2)	-2(2)	-4(2)	-7(2)
C(30')	38(2)	42(3)	52(3)	-14(2)	-3(2)	-16(2)
C(31')	158(6)	108(5)	63(4)	-37(4)	15(4)	-29(4)
Cl(1)	27(1)	35(1)	29(1)	-4(1)	0(1)	0(1)
Cl(2)	25(1)	37(1)	40(1)	0(1)	-1(1)	-8(1)
Cl(3)	26(1)	33(1)	27(1)	-4(1)	0(1)	-1(1)
Cl(4)	26(1)	35(1)	29(1)	-4(1)	0(1)	-8(1)
O(14)	43(2)	59(2)	29(1)	4(1)	-1(1)	-14(1)
O(15)	107(3)	36(2)	37(2)	-3(1)	5(2)	11(2)
O(16)	27(1)	73(2)	53(2)	-24(2)	5(1)	0(1)
O(17)	41(2)	75(2)	71(2)	-26(2)	-8(2)	-19(2)
O(18)	52(2)	205(5)	76(3)	84(3)	-13(2)	-21(3)
O(19)	56(2)	71(2)	121(3)	-53(2)	0(2)	8(2)
O(20)	32(2)	47(2)	151(3)	-35(2)	-16(2)	-12(1)

O(21)	23(1)	50(2)	48(2)	0(1)	-5(1)	-7(1)
O(22)	79(2)	33(2)	37(2)	-1(1)	7(1)	7(1)
O(23)	24(1)	72(2)	44(2)	-17(1)	3(1)	-3(1)
O(24)	40(2)	64(2)	65(2)	-25(2)	1(1)	-23(1)
O(25)	46(2)	55(2)	28(1)	-1(1)	1(1)	-7(1)
O(26)	51(2)	108(3)	39(2)	28(2)	0(1)	-5(2)
O(27)	39(2)	53(2)	150(3)	-49(2)	-6(2)	-14(2)
O(28)	62(2)	59(2)	44(2)	-24(2)	1(1)	8(2)
O(29)	24(1)	52(2)	46(2)	4(1)	-5(1)	-3(1)
O(1S)	88(3)	78(2)	86(3)	-14(2)	-17(2)	-5(2)
C(1S)	92(5)	113(5)	133(6)	-81(4)	-41(4)	41(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **8**.

	x	y	z	U(eq)
H(13A)	2155	5004	1551	72
H(31A)	2085	5747	2402	148
H(31B)	2864	4938	2861	148
H(31C)	1677	5002	2948	148
H(13B)	2060	9013	2665	74
H(31D)	1650	9527	3694	162
H(31E)	2402	8671	4117	162
H(31F)	1251	8662	3999	162
H(1A)	4790(30)	4910(30)	-1380(20)	55(13)
H(1B)	5690(20)	4349(19)	-884(17)	25(9)
H(1C)	5270(30)	4050(20)	-1540(20)	47(12)
H(1D)	-140(30)	5910(30)	6440(20)	61(14)
H(1E)	340(30)	4970(30)	6320(20)	53(12)
H(1F)	-570(30)	5680(20)	5760(20)	48(11)
H(3)	4980(20)	3750(20)	286(17)	23(9)
H(3')	70(20)	6352(19)	4611(17)	27(9)
H(8)	340(30)	3600(20)	190(20)	45(12)

H(8')	4920(30)	6350(20)	4797(17)	29(9)
H(10A)	-310(30)	3890(30)	-1050(20)	54(13)
H(10B)	-70(20)	4770(20)	-1390(17)	25(9)
H(10C)	460(30)	3840(20)	-1690(20)	59(12)
H(10D)	4810(30)	5850(20)	6720(20)	42(11)
H(10E)	5570(30)	5800(20)	6082(17)	27(9)
H(10F)	5200(20)	4970(20)	6400(17)	29(9)
H(11A)	5630(20)	720(20)	5230(16)	20(8)
H(11B)	5270(20)	1580(20)	5530(20)	34(10)
H(11C)	6030(30)	1550(20)	4889(18)	37(10)
H(11D)	-920(30)	8560(30)	90(20)	53(13)
H(11E)	-530(30)	9330(30)	-270(20)	72(15)
H(11F)	-170(30)	8460(30)	-500(20)	59(13)
H(13)	5180(30)	2330(20)	3713(19)	41(10)
H(13')	-140(30)	7900(20)	1297(19)	39(10)
H(18)	450(30)	2090(20)	3680(20)	48(11)
H(18')	4700(30)	7970(20)	1254(19)	38(10)
H(20A)	510(30)	1200(30)	5400(20)	49(14)
H(20B)	940(40)	370(30)	5110(30)	87(18)
H(20C)	-30(30)	1070(20)	4780(20)	54(12)
H(20D)	4600(30)	8630(30)	-540(30)	68(16)
H(20E)	5230(30)	8830(20)	45(19)	45(11)
H(20F)	4340(30)	9520(30)	-280(30)	78(17)
H(21A)	9540(30)	3900(30)	1710(20)	54(15)
H(21B)	9710(30)	3510(20)	2448(19)	35(10)
H(21C)	8950(20)	4400(20)	2271(18)	33(10)
H(21D)	-4190(30)	10460(20)	3166(19)	30(11)
H(21E)	-3430(30)	10670(30)	3660(20)	54(12)
H(21F)	-4250(30)	10120(20)	3930(20)	37(10)
H(23)	7260(20)	4240(20)	2227(16)	16(8)
H(23')	-1680(30)	9710(20)	3436(19)	49(11)
H(28)	6770(20)	410(20)	1731(16)	24(9)
H(28')	-2020(20)	5910(20)	2773(18)	28(10)
H(30A)	9250(30)	-330(20)	1940(20)	54(13)
H(30B)	9080(30)	-40(30)	1090(30)	66(14)
H(30C)	8430(30)	-550(30)	1550(20)	49(12)

H(30D)	-4540(40)	6250(30)	3280(30)	86(16)
H(30E)	-4560(40)	6590(30)	2480(30)	110(20)
H(30F)	-3780(30)	5720(20)	2709(19)	39(11)

Table 6. Torsion angles [°] for **8**.

O(13)-Li(1)-Li(2)-O(9)	11.5(5)
O(2)-Li(1)-Li(2)-O(9)	121.6(5)
O(6)-Li(1)-Li(2)-O(9)	-113.6(5)
O(5)-Li(1)-Li(2)-O(9)	-91.5(5)
O(1)-Li(1)-Li(2)-O(9)	98.5(5)
O(13)-Li(1)-Li(2)-O(10)	-172.0(3)
O(2)-Li(1)-Li(2)-O(10)	-61.9(5)
O(6)-Li(1)-Li(2)-O(10)	62.9(5)
O(5)-Li(1)-Li(2)-O(10)	85.0(4)
O(1)-Li(1)-Li(2)-O(10)	-84.9(4)
O(13)-Li(1)-Li(2)-O(1)	-87.1(3)
O(2)-Li(1)-Li(2)-O(1)	23.0(3)
O(6)-Li(1)-Li(2)-O(1)	147.9(4)
O(5)-Li(1)-Li(2)-O(1)	169.9(4)
O(13)-Li(1)-Li(2)-O(5)	103.0(3)
O(2)-Li(1)-Li(2)-O(5)	-146.9(4)
O(6)-Li(1)-Li(2)-O(5)	-22.1(3)
O(1)-Li(1)-Li(2)-O(5)	-169.9(4)
O(9)-Li(2)-O(1)-C(4)	48.3(6)
O(10)-Li(2)-O(1)-C(4)	-66.2(5)
O(5)-Li(2)-O(1)-C(4)	-179.6(4)
Li(1)-Li(2)-O(1)-C(4)	173.2(5)
O(9)-Li(2)-O(1)-Li(1)	-124.9(4)
O(10)-Li(2)-O(1)-Li(1)	120.6(3)
O(5)-Li(2)-O(1)-Li(1)	7.2(3)
O(13)-Li(1)-O(1)-C(4)	-77.0(4)
O(2)-Li(1)-O(1)-C(4)	24.4(4)
O(6)-Li(1)-O(1)-C(4)	114.6(7)
O(5)-Li(1)-O(1)-C(4)	178.8(3)

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)	-22.7(5)
Li(2)-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)
O(13)-Li(1)-O(5)-Li(2)	-85.6(3)
O(2)-Li(1)-O(5)-Li(2)	71.5(7)
O(6)-Li(1)-O(5)-Li(2)	162.1(3)
O(1)-Li(1)-O(5)-Li(2)	6.6(3)
O(13)-Li(1)-O(6)-C(17)	-109.4(4)
O(2)-Li(1)-O(6)-C(17)	146.4(3)
O(5)-Li(1)-O(6)-C(17)	-5.1(5)
O(1)-Li(1)-O(6)-C(17)	58.1(9)
Li(2)-Li(1)-O(6)-C(17)	9.3(6)
O(10)-Li(2)-O(9)-C(24)	5.1(4)
O(1)-Li(2)-O(9)-C(24)	-116.1(4)
O(5)-Li(2)-O(9)-C(24)	124.9(4)
Li(1)-Li(2)-O(9)-C(24)	-177.7(3)
O(9)-Li(2)-O(10)-C(27)	-5.9(4)

O(1)-Li(2)-O(10)-C(27)	126.2(3)
O(5)-Li(2)-O(10)-C(27)	-133.8(3)
Li(1)-Li(2)-O(10)-C(27)	176.4(3)
O(2)-Li(1)-O(13)-C(31)	146.1(5)
O(6)-Li(1)-O(13)-C(31)	47.2(6)
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)
C(6)-O(3)-C(2)-C(3)	1.0(4)
C(6)-O(3)-C(2)-C(1)	-178.0(3)
O(3)-C(2)-C(3)-C(4)	-3.2(5)
C(1)-C(2)-C(3)-C(4)	175.7(4)
Li(2)-O(1)-C(4)-C(3)	-10.1(6)
Li(1)-O(1)-C(4)-C(3)	161.4(3)
Li(2)-O(1)-C(4)-C(5)	170.5(4)
Li(1)-O(1)-C(4)-C(5)	-18.0(5)
C(2)-C(3)-C(4)-O(1)	-176.3(3)
C(2)-C(3)-C(4)-C(5)	3.1(5)
O(1)-C(4)-C(5)-C(6)	178.4(3)
C(3)-C(4)-C(5)-C(6)	-1.0(5)
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)	-1.0(5)
C(2)-O(3)-C(6)-O(4)	-179.0(2)
C(2)-O(3)-C(6)-C(5)	1.1(5)
C(4)-C(5)-C(6)-O(4)	179.1(3)
C(7)-C(5)-C(6)-O(4)	-2.2(5)
C(4)-C(5)-C(6)-O(3)	-1.0(5)
C(7)-C(5)-C(6)-O(3)	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)	175.9(4)
C(5)-C(7)-C(8)-C(9)	-3.6(6)
C(7)-C(8)-C(9)-O(4)	0.6(6)
C(7)-C(8)-C(9)-C(10)	-177.8(4)
C(6)-O(4)-C(9)-C(8)	1.9(5)
C(6)-O(4)-C(9)-C(10)	-179.5(3)
C(16)-O(7)-C(12)-C(13)	-8.3(4)
C(16)-O(7)-C(12)-C(11)	170.9(3)
O(7)-C(12)-C(13)-C(14)	3.6(5)
C(11)-C(12)-C(13)-C(14)	-175.4(3)
Li(2)-O(5)-C(14)-C(15)	-149.8(3)
Li(1)-O(5)-C(14)-C(15)	7.7(5)
Li(2)-O(5)-C(14)-C(13)	29.8(5)
Li(1)-O(5)-C(14)-C(13)	-172.7(3)
C(12)-C(13)-C(14)-O(5)	-177.1(3)
C(12)-C(13)-C(14)-C(15)	2.6(5)
O(5)-C(14)-C(15)-C(16)	175.6(3)
C(13)-C(14)-C(15)-C(16)	-4.1(4)
O(5)-C(14)-C(15)-C(17)	-7.9(5)
C(13)-C(14)-C(15)-C(17)	172.4(3)
C(19)-O(8)-C(16)-O(7)	177.4(3)
C(19)-O(8)-C(16)-C(15)	-3.6(5)
C(12)-O(7)-C(16)-O(8)	-174.1(2)
C(12)-O(7)-C(16)-C(15)	6.9(5)
C(14)-C(15)-C(16)-O(8)	-179.4(3)
C(17)-C(15)-C(16)-O(8)	3.8(5)
C(14)-C(15)-C(16)-O(7)	-0.7(5)
C(17)-C(15)-C(16)-O(7)	-177.4(3)
Li(1)-O(6)-C(17)-C(18)	-174.8(4)
Li(1)-O(6)-C(17)-C(15)	5.9(6)
C(16)-C(15)-C(17)-O(6)	177.6(3)
C(14)-C(15)-C(17)-O(6)	1.1(5)
C(16)-C(15)-C(17)-C(18)	-1.7(5)
C(14)-C(15)-C(17)-C(18)	-178.3(3)
O(6)-C(17)-C(18)-C(19)	-179.5(4)
C(15)-C(17)-C(18)-C(19)	-0.2(5)

C(17)-C(18)-C(19)-O(8)	0.4(6)
C(17)-C(18)-C(19)-C(20)	-179.9(5)
C(16)-O(8)-C(19)-C(18)	1.4(5)
C(16)-O(8)-C(19)-C(20)	-178.4(4)
C(26)-O(11)-C(22)-C(23)	-1.4(4)
C(26)-O(11)-C(22)-C(21)	178.6(3)
O(11)-C(22)-C(23)-C(24)	0.0(5)
C(21)-C(22)-C(23)-C(24)	-180.0(4)
Li(2)-O(9)-C(24)-C(23)	178.5(3)
Li(2)-O(9)-C(24)-C(25)	-1.2(5)
C(22)-C(23)-C(24)-O(9)	-178.5(3)
C(22)-C(23)-C(24)-C(25)	1.2(5)
O(9)-C(24)-C(25)-C(26)	178.6(3)
C(23)-C(24)-C(25)-C(26)	-1.1(4)
O(9)-C(24)-C(25)-C(27)	-4.2(5)
C(23)-C(24)-C(25)-C(27)	176.1(3)
C(22)-O(11)-C(26)-O(12)	-178.3(2)
C(22)-O(11)-C(26)-C(25)	1.5(4)
C(29)-O(12)-C(26)-O(11)	175.7(2)
C(29)-O(12)-C(26)-C(25)	-4.1(5)
C(24)-C(25)-C(26)-O(11)	-0.2(5)
C(27)-C(25)-C(26)-O(11)	-177.6(3)
C(24)-C(25)-C(26)-O(12)	179.6(3)
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)
C(26)-O(12)-C(29)-C(30)	-176.8(3)

O(13')-Li(1')-Li(2')-O(9')	-7.2(3)
O(2')-Li(1')-Li(2')-O(9')	103.6(4)
O(6')-Li(1')-Li(2')-O(9')	-111.1(4)
O(1')-Li(1')-Li(2')-O(9')	100.9(3)
O(5')-Li(1')-Li(2')-O(9')	-101.7(3)
O(13')-Li(1')-Li(2')-O(10')	170.9(6)
O(2')-Li(1')-Li(2')-O(10')	-78.2(8)
O(6')-Li(1')-Li(2')-O(10')	67.0(8)
O(1')-Li(1')-Li(2')-O(10')	-81.0(6)
O(5')-Li(1')-Li(2')-O(10')	76.5(6)
O(13')-Li(1')-Li(2')-O(5')	94.4(3)
O(2')-Li(1')-Li(2')-O(5')	-154.7(5)
O(6')-Li(1')-Li(2')-O(5')	-9.5(3)
O(1')-Li(1')-Li(2')-O(5')	-157.4(4)
O(13')-Li(1')-Li(2')-O(1')	-108.1(3)
O(2')-Li(1')-Li(2')-O(1')	2.7(4)
O(6')-Li(1')-Li(2')-O(1')	148.0(5)
O(5')-Li(1')-Li(2')-O(1')	157.4(4)
O(9')-Li(2')-O(1')-C(4')	68.7(5)
O(10')-Li(2')-O(1')-C(4')	-46.6(6)
O(5')-Li(2')-O(1')-C(4')	-175.4(3)
Li(1')-Li(2')-O(1')-C(4')	168.3(5)
O(9')-Li(2')-O(1')-Li(1')	-99.6(3)
O(10')-Li(2')-O(1')-Li(1')	145.1(4)
O(5')-Li(2')-O(1')-Li(1')	16.3(3)
O(13')-Li(1')-O(1')-C(4')	-93.5(4)
O(2')-Li(1')-O(1')-C(4')	12.5(5)
O(6')-Li(1')-O(1')-C(4')	114.9(7)
O(5')-Li(1')-O(1')-C(4')	175.5(3)
Li(2')-Li(1')-O(1')-C(4')	-169.6(4)
O(13')-Li(1')-O(1')-Li(2')	76.1(3)
O(2')-Li(1')-O(1')-Li(2')	-177.9(3)
O(6')-Li(1')-O(1')-Li(2')	-75.4(8)
O(5')-Li(1')-O(1')-Li(2')	-14.9(3)
O(13')-Li(1')-O(2')-C(7')	87.1(5)
O(6')-Li(1')-O(2')-C(7')	-168.8(4)

O(1')-Li(1')-O(2')-C(7')	-14.4(5)
O(5')-Li(1')-O(2')-C(7')	-69.4(11)
Li(2')-Li(1')-O(2')-C(7')	-16.2(7)
O(9')-Li(2')-O(5')-C(14')	-52.6(6)
O(10')-Li(2')-O(5')-C(14')	60.5(6)
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)
O(1')-Li(2')-O(5')-Li(1')	-15.9(3)
O(13')-Li(1')-O(5')-C(14')	71.0(4)
O(2')-Li(1')-O(5')-C(14')	-131.5(9)
O(6')-Li(1')-O(5')-C(14')	-30.0(4)
O(1')-Li(1')-O(5')-C(14')	172.4(3)
Li(2')-Li(1')-O(5')-C(14')	157.5(4)
O(13')-Li(1')-O(5')-Li(2')	-86.6(3)
O(2')-Li(1')-O(5')-Li(2')	70.9(10)
O(6')-Li(1')-O(5')-Li(2')	172.4(3)
O(1')-Li(1')-O(5')-Li(2')	14.9(3)
O(13')-Li(1')-O(6')-C(17')	-56.9(5)
O(2')-Li(1')-O(6')-C(17')	-165.2(3)
O(1')-Li(1')-O(6')-C(17')	94.7(8)
O(5')-Li(1')-O(6')-C(17')	35.2(4)
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')	127.8(3)
O(1')-Li(2')-O(9')-C(24')	-131.1(3)
Li(1')-Li(2')-O(9')-C(24')	179.3(3)
O(9')-Li(2')-O(10')-C(27')	-0.5(4)
O(5')-Li(2')-O(10')-C(27')	-122.6(4)
O(1')-Li(2')-O(10')-C(27')	122.9(4)
Li(1')-Li(2')-O(10')-C(27')	-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)

Li(2')-Li(1')-O(13')-C(31')	83.0(4)
C(6')-O(3')-C(2')-C(3')	-2.2(4)
C(6')-O(3')-C(2')-C(1')	178.6(3)
O(3')-C(2')-C(3')-C(4')	2.6(5)
C(1')-C(2')-C(3')-C(4')	-178.4(3)
Li(2')-O(1')-C(4')-C(3')	6.8(6)
Li(1')-O(1')-C(4')-C(3')	171.6(3)
Li(2')-O(1')-C(4')-C(5')	-172.5(4)
Li(1')-O(1')-C(4')-C(5')	-7.7(5)
C(2')-C(3')-C(4')-O(1')	178.8(3)
C(2')-C(3')-C(4')-C(5')	-1.8(5)
O(1')-C(4')-C(5')-C(6')	-180.0(3)
C(3')-C(4')-C(5')-C(6')	0.7(5)
O(1')-C(4')-C(5')-C(7')	-1.3(5)
C(3')-C(4')-C(5')-C(7')	179.3(3)
C(2')-O(3')-C(6')-O(4')	-179.1(2)
C(2')-O(3')-C(6')-C(5')	1.1(5)
C(9')-O(4')-C(6')-O(3')	178.5(2)
C(9')-O(4')-C(6')-C(5')	-1.7(5)
C(4')-C(5')-C(6')-O(3')	-0.4(5)
C(7')-C(5')-C(6')-O(3')	-179.2(3)
C(4')-C(5')-C(6')-O(4')	179.8(3)
C(7')-C(5')-C(6')-O(4')	1.1(5)
Li(1')-O(2')-C(7')-C(8')	-170.3(4)
Li(1')-O(2')-C(7')-C(5')	11.2(6)
C(6')-C(5')-C(7')-O(2')	178.6(3)
C(4')-C(5')-C(7')-O(2')	0.0(6)
C(6')-C(5')-C(7')-C(8')	0.0(5)
C(4')-C(5')-C(7')-C(8')	-178.6(3)
O(2')-C(7')-C(8')-C(9')	-179.1(4)
C(5')-C(7')-C(8')-C(9')	-0.5(5)
C(7')-C(8')-C(9')-O(4')	-0.1(5)
C(7')-C(8')-C(9')-C(10')	178.9(4)
C(6')-O(4')-C(9')-C(8')	1.1(5)
C(6')-O(4')-C(9')-C(10')	-178.0(3)
C(16')-O(7')-C(12')-C(13')	6.6(4)

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(17')-C(15')-C(16')-O(8')	-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)
O(9')-C(24')-C(25')-C(26')	179.3(3)
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')-O(11')	-179.9(3)
C(29')-O(12')-C(26')-C(25')	0.8(5)
C(22')-O(11')-C(26')-O(12')	-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(30')	179.6(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **8** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(13)-H(13A)...O(14)#1	0.95	2.01	2.891(4)	153.1
O(13')-H(13B)...O(1S)	0.95	1.92	2.780(4)	150.2

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:

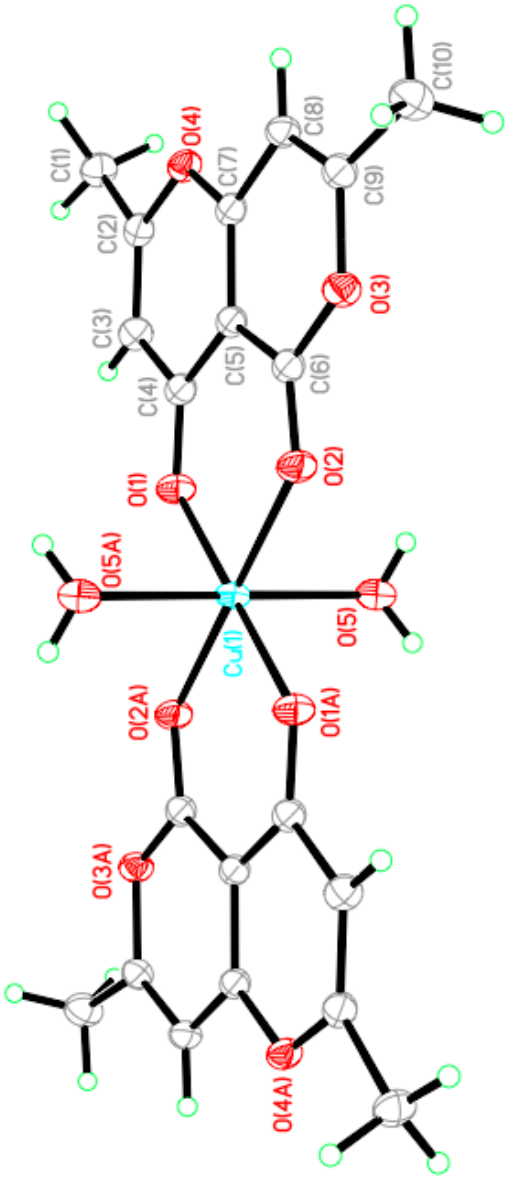


Table 1. Crystal data and structure refinement for **9** (M= Cu).

Identification code	**9** (M= Cu)
Empirical formula	C20 H20 Cl2 Cu O18
Formula weight	682.80

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) $Å^3$	
Z	2	
Density (calculated)	1.732 Mg/m^3	
Absorption coefficient	1.125 mm^{-1}	
F(000)	694	
Crystal size	0.32 x 0.27 x 0.16 mm^3	
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 F^2	
Data / restraints / parameters	2858 / 0 / 227	
Goodness-of-fit on F^2	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.

	x	y	z	U(eq)
Cu(1)	0	5000	5000	25(1)
Cl(1)	9259(1)	1858(1)	2307(1)	28(1)
O(1)	1645(1)	5827(1)	5043(1)	32(1)
O(2)	363(1)	4731(1)	6982(1)	31(1)
O(3)	1387(1)	4754(1)	8923(1)	30(1)
O(4)	4500(1)	6568(1)	7947(1)	29(1)
O(5)	1168(2)	3623(1)	4582(1)	42(1)
O(6)	9963(1)	910(1)	2039(1)	49(1)
O(7)	9845(1)	2759(1)	1736(1)	43(1)
O(8)	9310(1)	2016(1)	3683(1)	43(1)
O(9)	7848(1)	1775(1)	1837(1)	43(1)
C(1)	5863(2)	7527(1)	6622(2)	35(1)
C(2)	4607(2)	6902(1)	6717(2)	29(1)
C(3)	3665(2)	6660(1)	5755(2)	30(1)
C(4)	2500(1)	6035(1)	5966(1)	27(1)
C(5)	2430(1)	5694(1)	7276(1)	25(1)
C(6)	1344(2)	5049(1)	7664(2)	27(1)
C(7)	3437(1)	5979(1)	8209(1)	26(1)
C(8)	3424(2)	5662(1)	9503(2)	28(1)
C(9)	2408(2)	5047(1)	9824(2)	29(1)
C(10)	2241(2)	4596(2)	11102(2)	40(1)

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

Cu(1)-O(1)#1	1.9377(10)
Cu(1)-O(1)	1.9377(10)
Cu(1)-O(2)#1	2.0983(11)
Cu(1)-O(2)	2.0983(11)
Cu(1)-O(5)#1	2.1596(14)
Cu(1)-O(5)	2.1596(14)
Cl(1)-O(6)	1.4296(12)
Cl(1)-O(7)	1.4345(12)
Cl(1)-O(9)	1.4458(12)
Cl(1)-O(8)	1.4491(13)
O(1)-C(4)	1.2541(18)
O(2)-C(6)	1.2218(19)
O(3)-C(6)	1.365(2)
O(3)-C(9)	1.3702(19)
O(4)-C(7)	1.3424(17)
O(4)-C(2)	1.3674(19)
O(5)-H(5A)	0.78(3)
O(5)-H(5B)	0.76(3)
C(1)-C(2)	1.489(2)
C(1)-H(1A)	0.98(2)
C(1)-H(1B)	0.95(2)
C(1)-H(1C)	0.93(2)
C(2)-C(3)	1.344(2)
C(3)-C(4)	1.436(2)
C(3)-H(3)	0.89(2)
C(4)-C(5)	1.445(2)
C(5)-C(7)	1.3793(19)
C(5)-C(6)	1.441(2)
C(7)-C(8)	1.413(2)
C(8)-C(9)	1.342(2)
C(8)-H(8)	0.89(2)
C(9)-C(10)	1.478(2)
C(10)-H(10A)	0.95(3)
C(10)-H(10B)	0.96(3)

C(10)-H(10C)	0.94(2)
O(1)#1-Cu(1)-O(1)	180.00(6)
O(1)#1-Cu(1)-O(2)#1	89.85(4)
O(1)-Cu(1)-O(2)#1	90.15(4)
O(1)#1-Cu(1)-O(2)	90.15(4)
O(1)-Cu(1)-O(2)	89.85(4)
O(2)#1-Cu(1)-O(2)	180.0
O(1)#1-Cu(1)-O(5)#1	88.30(5)
O(1)-Cu(1)-O(5)#1	91.70(5)
O(2)#1-Cu(1)-O(5)#1	91.33(5)
O(2)-Cu(1)-O(5)#1	88.67(5)
O(1)#1-Cu(1)-O(5)	91.70(5)
O(1)-Cu(1)-O(5)	88.30(5)
O(2)#1-Cu(1)-O(5)	88.67(5)
O(2)-Cu(1)-O(5)	91.33(5)
O(5)#1-Cu(1)-O(5)	180.0
O(6)-Cl(1)-O(7)	110.99(8)
O(6)-Cl(1)-O(9)	110.49(8)
O(7)-Cl(1)-O(9)	109.60(8)
O(6)-Cl(1)-O(8)	109.73(8)
O(7)-Cl(1)-O(8)	109.06(8)
O(9)-Cl(1)-O(8)	106.88(8)
C(4)-O(1)-Cu(1)	129.95(10)
C(6)-O(2)-Cu(1)	125.31(10)
C(6)-O(3)-C(9)	122.93(12)
C(7)-O(4)-C(2)	119.70(12)
Cu(1)-O(5)-H(5A)	109.9(18)
Cu(1)-O(5)-H(5B)	112.2(18)
H(5A)-O(5)-H(5B)	112(3)
C(2)-C(1)-H(1A)	110.0(12)
C(2)-C(1)-H(1B)	108.3(13)
H(1A)-C(1)-H(1B)	107.4(18)
C(2)-C(1)-H(1C)	110.4(13)
H(1A)-C(1)-H(1C)	108.8(17)
H(1B)-C(1)-H(1C)	111.9(18)

C(3)-C(2)-O(4)	121.60(13)
C(3)-C(2)-C(1)	126.92(15)
O(4)-C(2)-C(1)	111.48(13)
C(2)-C(3)-C(4)	121.71(15)
C(2)-C(3)-H(3)	121.2(13)
C(4)-C(3)-H(3)	117.1(13)
O(1)-C(4)-C(3)	119.77(14)
O(1)-C(4)-C(5)	125.29(13)
C(3)-C(4)-C(5)	114.94(13)
C(7)-C(5)-C(6)	117.54(14)
C(7)-C(5)-C(4)	119.77(13)
C(6)-C(5)-C(4)	122.69(13)
O(2)-C(6)-O(3)	115.12(13)
O(2)-C(6)-C(5)	126.89(15)
O(3)-C(6)-C(5)	117.99(13)
O(4)-C(7)-C(5)	122.28(13)
O(4)-C(7)-C(8)	115.50(12)
C(5)-C(7)-C(8)	122.21(13)
C(9)-C(8)-C(7)	118.51(14)
C(9)-C(8)-H(8)	122.7(12)
C(7)-C(8)-H(8)	118.7(12)
C(8)-C(9)-O(3)	120.80(14)
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)
C(9)-C(10)-H(10B)	107.5(17)
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 ($Å^2 x\ 10^3$) for **9** (M= Cu). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cu(1)	20(1)	31(1)	22(1)	-1(1)	-2(1)	-2(1)
Cl(1)	26(1)	26(1)	33(1)	0(1)	1(1)	-1(1)
O(1)	25(1)	40(1)	29(1)	-2(1)	1(1)	-6(1)
O(2)	22(1)	37(1)	35(1)	-1(1)	-1(1)	-6(1)
O(3)	22(1)	33(1)	34(1)	1(1)	1(1)	-4(1)
O(4)	21(1)	29(1)	35(1)	-2(1)	1(1)	-5(1)
O(5)	41(1)	44(1)	39(1)	-3(1)	-12(1)	-2(1)
O(6)	44(1)	32(1)	74(1)	-4(1)	18(1)	6(1)
O(7)	48(1)	33(1)	49(1)	1(1)	17(1)	-8(1)
O(8)	44(1)	53(1)	31(1)	4(1)	-2(1)	3(1)
O(9)	33(1)	42(1)	52(1)	-3(1)	-11(1)	-4(1)
C(1)	26(1)	38(1)	42(1)	0(1)	4(1)	-8(1)
C(2)	23(1)	26(1)	37(1)	-1(1)	5(1)	0(1)
C(3)	25(1)	33(1)	34(1)	-1(1)	5(1)	-2(1)
C(4)	22(1)	27(1)	33(1)	-3(1)	3(1)	1(1)
C(5)	20(1)	24(1)	31(1)	-2(1)	2(1)	1(1)
C(6)	22(1)	26(1)	32(1)	-2(1)	2(1)	2(1)
C(7)	19(1)	23(1)	34(1)	-3(1)	2(1)	1(1)
C(8)	22(1)	29(1)	33(1)	-2(1)	-2(1)	0(1)
C(9)	24(1)	29(1)	34(1)	-1(1)	0(1)	3(1)
C(10)	34(1)	47(1)	37(1)	7(1)	1(1)	-5(1)

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

	x	y	z	U(eq)
H(1A)	5950(20)	8064(16)	7300(20)	45(6)
H(1B)	6620(20)	7061(17)	6760(20)	50(6)
H(1C)	5840(20)	7860(17)	5830(20)	46(6)
H(3)	3744(19)	6873(15)	4958(19)	36(5)
H(5A)	690(30)	3160(20)	4370(20)	56(7)
H(5B)	1670(30)	3473(19)	5140(20)	54(7)
H(8)	4100(20)	5862(15)	10066(19)	39(5)
H(10A)	2320(20)	3850(20)	11040(20)	58(6)
H(10B)	1370(30)	4810(20)	11340(30)	73(8)
H(10C)	2940(20)	4829(16)	11710(20)	43(6)

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

O(1)#1-Cu(1)-O(1)-C(4)	17(47)
O(2)#1-Cu(1)-O(1)-C(4)	-179.07(13)
O(2)-Cu(1)-O(1)-C(4)	0.93(13)
O(5)#1-Cu(1)-O(1)-C(4)	89.60(13)
O(5)-Cu(1)-O(1)-C(4)	-90.40(13)
O(1)#1-Cu(1)-O(2)-C(6)	178.50(12)
O(1)-Cu(1)-O(2)-C(6)	-1.50(12)
O(2)#1-Cu(1)-O(2)-C(6)	-99(100)
O(5)#1-Cu(1)-O(2)-C(6)	-93.21(13)
O(5)-Cu(1)-O(2)-C(6)	86.79(13)
C(7)-O(4)-C(2)-C(3)	0.4(2)
C(7)-O(4)-C(2)-C(1)	-179.10(13)
O(4)-C(2)-C(3)-C(4)	-0.3(2)
C(1)-C(2)-C(3)-C(4)	179.14(15)
Cu(1)-O(1)-C(4)-C(3)	179.05(10)
Cu(1)-O(1)-C(4)-C(5)	-0.8(2)

C(2)-C(3)-C(4)-O(1)	-179.93(14)
C(2)-C(3)-C(4)-C(5)	0.0(2)
O(1)-C(4)-C(5)-C(7)	-179.83(13)
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)
C(9)-O(3)-C(6)-O(2)	-179.44(13)
C(9)-O(3)-C(6)-C(5)	0.2(2)
C(7)-C(5)-C(6)-O(2)	179.08(14)
C(4)-C(5)-C(6)-O(2)	-1.6(2)
C(7)-C(5)-C(6)-O(3)	-0.53(19)
C(4)-C(5)-C(6)-O(3)	178.84(12)
C(2)-O(4)-C(7)-C(5)	-0.2(2)
C(2)-O(4)-C(7)-C(8)	179.22(12)
C(6)-C(5)-C(7)-O(4)	179.20(12)
C(4)-C(5)-C(7)-O(4)	-0.2(2)
C(6)-C(5)-C(7)-C(8)	-0.2(2)
C(4)-C(5)-C(7)-C(8)	-179.55(13)
O(4)-C(7)-C(8)-C(9)	-178.21(13)
C(5)-C(7)-C(8)-C(9)	1.2(2)
C(7)-C(8)-C(9)-O(3)	-1.5(2)
C(7)-C(8)-C(9)-C(10)	177.18(15)
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 °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5A)...O(8)#2	0.78(3)	2.07(3)	2.847(2)	175(3)
O(5)-H(5B)...O(9)#3	0.76(3)	2.06(3)	2.8061(19)	170(3)

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:

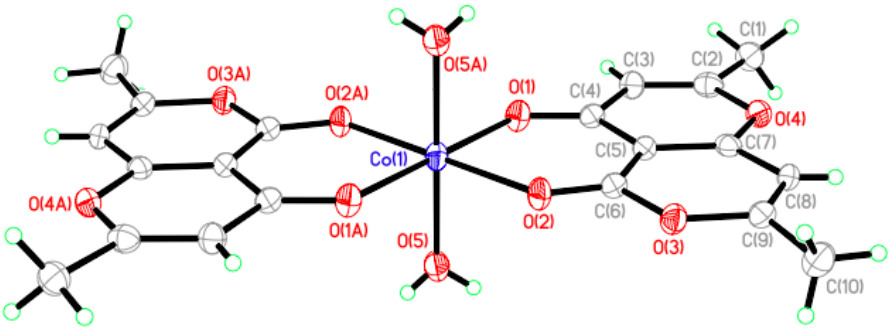


Table 1. Crystal data and structure refinement for **9** (M= Co).

Identification code	**9** (M= Co)

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	a = 10.033(6) Å	a= 90°.
	b = 12.586(8) Å	b= 95.922(9)°.
	c = 10.332(6) Å	g = 90°.
Volume	1297.7(14) $Å^3$	
Z	2	
Density (calculated)	1.736 Mg/m^3	
Absorption coefficient	0.955 mm^{-1}	
F(000)	690	
Crystal size	0.19 x 0.16 x 0.12 mm^3	
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 F^2	
Data / restraints / parameters	2279 / 0 / 227	
Goodness-of-fit on F^2	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 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **9** (M= Co). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Co(1)	0	5000	0	32(1)
Cl(1)	5748(1)	6853(1)	7757(1)	35(1)

O(1)	1746(3)	5847(2)	72(3)	38(1)
O(2)	431(3)	4759(2)	1980(3)	35(1)
O(3)	1419(3)	4779(2)	3950(3)	35(1)
O(4)	4537(3)	6578(2)	3047(3)	35(1)
O(5)	1056(4)	3652(3)	-392(4)	46(1)
O(6)	5772(4)	7005(3)	6378(3)	67(1)
O(7)	7127(4)	6788(3)	8259(4)	73(1)
O(8)	5159(4)	7766(3)	8259(4)	70(1)
O(9)	5042(4)	5910(3)	7999(4)	60(1)
C(1)	5871(5)	7550(4)	1724(6)	43(1)
C(2)	4655(4)	6919(3)	1808(4)	37(1)
C(3)	3723(4)	6684(4)	848(5)	34(1)
C(4)	2580(4)	6060(3)	1014(4)	33(1)
C(5)	2486(4)	5710(3)	2326(4)	30(1)
C(6)	1401(4)	5078(3)	2683(4)	30(1)
C(7)	3462(4)	5986(3)	3292(4)	31(1)
C(8)	3419(4)	5672(3)	4595(4)	32(1)
C(9)	2414(4)	5075(3)	4892(4)	34(1)
C(10)	2228(6)	4630(5)	6164(5)	46(1)

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

Co(1)-O(1)#1	2.045(3)
Co(1)-O(1)	2.045(3)
Co(1)-O(5)	2.062(4)
Co(1)-O(5)#1	2.062(4)
Co(1)-O(2)#1	2.069(3)
Co(1)-O(2)	2.069(3)
Cl(1)-O(8)	1.414(4)
Cl(1)-O(9)	1.418(3)
Cl(1)-O(7)	1.429(4)
Cl(1)-O(6)	1.440(4)
O(1)-C(4)	1.245(5)
O(2)-C(6)	1.221(5)
O(3)-C(6)	1.360(5)

O(3)-C(9)	1.372(5)
O(4)-C(7)	1.356(5)
O(4)-C(2)	1.366(5)
O(5)-H(5OA)	0.66(5)
O(5)-H(5OB)	0.73(7)
C(1)-C(2)	1.465(6)
C(1)-H(1A)	0.90(5)
C(1)-H(1B)	0.96(6)
C(1)-H(1C)	0.91(6)
C(2)-C(3)	1.324(6)
C(3)-C(4)	1.415(6)
C(3)-H(3)	0.82(5)
C(4)-C(5)	1.438(6)
C(5)-C(7)	1.369(6)
C(5)-C(6)	1.427(6)
C(7)-C(8)	1.408(6)
C(8)-C(9)	1.319(6)
C(8)-H(8)	0.77(5)
C(9)-C(10)	1.458(7)
C(10)-H(10A)	0.85(6)
C(10)-H(10B)	0.91(5)
C(10)-H(10C)	1.04(7)

O(1)#1-Co(1)-O(1)	180.0
O(1)#1-Co(1)-O(5)	91.10(16)
O(1)-Co(1)-O(5)	88.90(16)
O(1)#1-Co(1)-O(5)#1	88.90(16)
O(1)-Co(1)-O(5)#1	91.10(16)
O(5)-Co(1)-O(5)#1	180.0
O(1)#1-Co(1)-O(2)#1	87.12(11)
O(1)-Co(1)-O(2)#1	92.88(11)
O(5)-Co(1)-O(2)#1	89.18(14)
O(5)#1-Co(1)-O(2)#1	90.82(14)
O(1)#1-Co(1)-O(2)	92.88(11)
O(1)-Co(1)-O(2)	87.12(11)
O(5)-Co(1)-O(2)	90.82(14)

O(5)#1-Co(1)-O(2)	89.18(14)
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)

O(4)-C(7)-C(5)	121.7(4)
O(4)-C(7)-C(8)	115.9(4)
C(5)-C(7)-C(8)	122.4(4)
C(9)-C(8)-C(7)	118.9(4)
C(9)-C(8)-H(8)	123(4)
C(7)-C(8)-H(8)	118(4)
C(8)-C(9)-O(3)	120.4(4)
C(8)-C(9)-C(10)	126.9(4)
O(3)-C(9)-C(10)	112.6(4)
C(9)-C(10)-H(10A)	108(4)
C(9)-C(10)-H(10B)	109(3)
H(10A)-C(10)-H(10B)	109(5)
C(9)-C(10)-H(10C)	109(3)
H(10A)-C(10)-H(10C)	110(5)
H(10B)-C(10)-H(10C)	112(4)

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9** (M= Co). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Co(1)	26(1)	38(1)	31(1)	-2(1)	-3(1)	-2(1)
Cl(1)	35(1)	31(1)	38(1)	-1(1)	-2(1)	4(1)
O(1)	32(2)	47(2)	33(2)	2(1)	-1(1)	-4(1)
O(2)	26(2)	44(2)	32(2)	0(1)	-4(1)	-7(1)
O(3)	31(2)	38(2)	35(2)	3(1)	-4(1)	-4(1)
O(4)	30(2)	34(2)	41(2)	1(1)	-2(1)	-5(1)
O(5)	43(2)	43(2)	47(2)	-13(2)	-11(2)	5(2)
O(6)	80(3)	81(3)	40(2)	-14(2)	9(2)	-24(2)
O(7)	60(2)	56(2)	94(3)	10(2)	-39(2)	-2(2)
O(8)	87(3)	39(2)	92(3)	12(2)	51(2)	20(2)
O(9)	60(2)	38(2)	86(3)	-1(2)	22(2)	-9(2)
C(1)	38(3)	42(3)	50(3)	4(3)	4(2)	-7(2)

C(2)	35(2)	32(2)	46(3)	2(2)	9(2)	0(2)
C(3)	28(2)	38(2)	35(2)	0(2)	4(2)	-2(2)
C(4)	29(2)	30(2)	38(2)	-2(2)	-1(2)	5(2)
C(5)	28(2)	27(2)	33(2)	-1(2)	-2(2)	2(2)
C(6)	30(2)	28(2)	32(2)	0(2)	0(2)	3(2)
C(7)	26(2)	24(2)	43(2)	-3(2)	1(2)	2(2)
C(8)	28(2)	30(2)	34(2)	-1(2)	-9(2)	0(2)
C(9)	31(2)	31(2)	38(2)	0(2)	-3(2)	4(2)
C(10)	44(3)	50(3)	43(3)	8(2)	0(2)	-1(2)

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

	x	y	z	U(eq)
H(1A)	6620(50)	7170(40)	1930(50)	46(14)
H(1B)	5810(60)	7890(40)	880(60)	67(17)
H(1C)	6000(60)	8040(50)	2380(60)	65(18)
H(3)	3840(50)	6920(40)	130(50)	57(17)
H(5OA)	740(50)	3230(40)	-630(50)	31(17)
H(5OB)	1660(70)	3480(60)	10(70)	90(30)
H(8)	4020(50)	5810(40)	5080(50)	48(15)
H(10A)	2280(60)	3960(50)	6120(60)	70(20)
H(10B)	2900(50)	4860(30)	6760(50)	37(12)
H(10C)	1290(70)	4850(50)	6400(60)	81(19)

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

O(1)#1-Co(1)-O(1)-C(4)	-6(100)
O(5)-Co(1)-O(1)-C(4)	-91.9(4)
O(5)#1-Co(1)-O(1)-C(4)	88.1(4)
O(2)#1-Co(1)-O(1)-C(4)	178.9(4)
O(2)-Co(1)-O(1)-C(4)	-1.1(4)
O(1)#1-Co(1)-O(2)-C(6)	-179.9(3)

O(1)-Co(1)-O(2)-C(6)	0.1(3)
O(5)-Co(1)-O(2)-C(6)	88.9(4)
O(5)#1-Co(1)-O(2)-C(6)	-91.1(4)
O(2)#1-Co(1)-O(2)-C(6)	-142(3)
C(7)-O(4)-C(2)-C(3)	0.6(6)
C(7)-O(4)-C(2)-C(1)	179.8(4)
O(4)-C(2)-C(3)-C(4)	-1.4(7)
C(1)-C(2)-C(3)-C(4)	179.5(5)
Co(1)-O(1)-C(4)-C(3)	-178.8(3)
Co(1)-O(1)-C(4)-C(5)	1.4(6)
C(2)-C(3)-C(4)-O(1)	-178.7(4)
C(2)-C(3)-C(4)-C(5)	1.1(6)
O(1)-C(4)-C(5)-C(7)	179.8(4)
C(3)-C(4)-C(5)-C(7)	-0.1(6)
O(1)-C(4)-C(5)-C(6)	-0.5(6)
C(3)-C(4)-C(5)-C(6)	179.7(4)
Co(1)-O(2)-C(6)-O(3)	-179.2(2)
Co(1)-O(2)-C(6)-C(5)	0.6(6)
C(9)-O(3)-C(6)-O(2)	-178.9(3)
C(9)-O(3)-C(6)-C(5)	1.3(5)
C(7)-C(5)-C(6)-O(2)	179.2(4)
C(4)-C(5)-C(6)-O(2)	-0.6(7)
C(7)-C(5)-C(6)-O(3)	-1.0(5)
C(4)-C(5)-C(6)-O(3)	179.2(3)
C(2)-O(4)-C(7)-C(5)	0.5(6)
C(2)-O(4)-C(7)-C(8)	-179.8(4)
C(6)-C(5)-C(7)-O(4)	179.5(3)
C(4)-C(5)-C(7)-O(4)	-0.7(6)
C(6)-C(5)-C(7)-C(8)	-0.1(6)
C(4)-C(5)-C(7)-C(8)	179.6(4)
O(4)-C(7)-C(8)-C(9)	-178.5(4)
C(5)-C(7)-C(8)-C(9)	1.1(6)
C(7)-C(8)-C(9)-O(3)	-0.9(6)
C(7)-C(8)-C(9)-C(10)	176.9(4)
C(6)-O(3)-C(9)-C(8)	-0.3(6)
C(6)-O(3)-C(9)-C(10)	-178.4(4)

Symmetry transformations used to generate equivalent atoms:

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

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5OA)...O(6)#2	0.66(5)	2.24(5)	2.883(7)	166(6)
O(5)-H(5OB)...O(7)#3	0.73(7)	2.08(7)	2.768(6)	156(7)

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 – calcd 540.9684 found 540.9677. mp > 250 ˚C.

Crystal Structure:

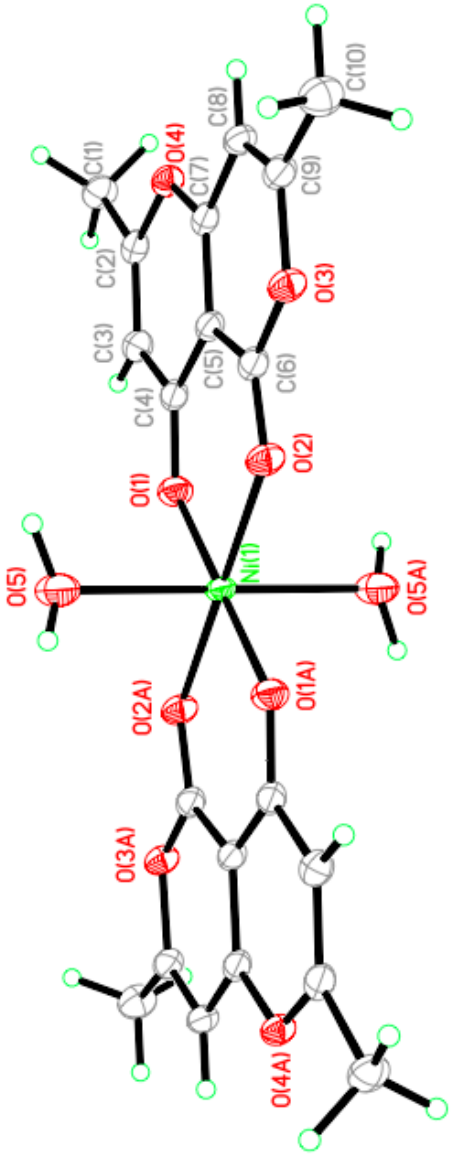


Table 1. Crystal data and structure refinement for **9** (M= Ni).

Identification code	**9** (M= Ni)
Empirical formula	C20 H20 Cl2 Ni O18
Formula weight	677.97

Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 9.952(3) Å	a= 90°.
	b = 12.636(3) Å	b= 95.348(4)°.
	c = 10.439(3) Å	g = 90°.
Volume	1307.0(6) $Å^3$	
Z	2	
Density (calculated)	1.723 Mg/m^3	
Absorption coefficient	1.033 mm^{-1}	
F(000)	692	
Crystal size	0.19 x 0.12 x 0.09 mm^3	
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 F^2	
Data / restraints / parameters	2852 / 0 / 227	
Goodness-of-fit on F^2	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 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **9** (M= Ni). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni(1)	0	5000	5000	23(1)
Cl(1)	4258(1)	6857(1)	7305(1)	30(1)
O(1)	1643(1)	4174(1)	5043(1)	33(1)
O(2)	362(1)	5270(1)	6983(1)	33(1)

O(3)	1385(1)	5247(1)	8926(1)	32(1)
O(4)	4498(1)	3433(1)	7949(1)	30(1)
O(5)	1172(2)	6377(1)	4581(2)	45(1)
O(6)	4308(2)	7011(1)	8680(2)	45(1)
O(7)	2852(2)	6776(1)	6832(2)	45(1)
O(8)	4959(2)	5911(1)	7038(2)	51(1)
O(9)	4843(2)	7756(1)	6738(2)	44(1)
C(1)	5857(2)	2473(2)	6624(2)	37(1)
C(2)	4602(2)	3099(2)	6712(2)	29(1)
C(3)	3659(2)	3343(2)	5759(2)	31(1)
C(4)	2506(2)	3964(2)	5969(2)	28(1)
C(5)	2430(2)	4306(2)	7277(2)	26(1)
C(6)	1345(2)	4949(2)	7665(2)	28(1)
C(7)	3433(2)	4021(2)	8212(2)	27(1)
C(8)	3418(2)	4340(2)	9504(2)	29(1)
C(9)	2411(2)	4954(2)	9828(2)	31(1)
C(10)	2243(3)	5403(2)	11105(2)	42(1)

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

Ni(1)-O(1)	1.9366(14)
Ni(1)-O(1)#1	1.9366(14)
Ni(1)-O(2)#1	2.0955(15)
Ni(1)-O(2)	2.0955(15)
Ni(1)-O(5)#1	2.1621(18)
Ni(1)-O(5)	2.1621(18)
Cl(1)-O(8)	1.4247(16)
Cl(1)-O(9)	1.4298(16)
Cl(1)-O(7)	1.4432(16)
Cl(1)-O(6)	1.4446(16)
O(1)-C(4)	1.259(2)
O(2)-C(6)	1.224(2)
O(3)-C(6)	1.365(3)
O(3)-C(9)	1.374(2)
O(4)-C(7)	1.343(2)

O(4)-C(2)	1.371(2)
O(5)-H(5A)	0.95(4)
O(5)-H(5B)	0.82(3)
C(1)-C(2)	1.489(3)
C(1)-H(1A)	1.01(3)
C(1)-H(1B)	0.98(3)
C(1)-H(1C)	1.00(3)
C(2)-C(3)	1.339(3)
C(3)-C(4)	1.425(3)
C(3)-H(3)	0.88(2)
C(4)-C(5)	1.441(3)
C(5)-C(7)	1.377(3)
C(5)-C(6)	1.439(3)
C(7)-C(8)	1.409(3)
C(8)-C(9)	1.335(3)
C(8)-H(8)	0.89(2)
C(9)-C(10)	1.474(3)
C(10)-H(10A)	0.96(4)
C(10)-H(10B)	0.98(4)
C(10)-H(10C)	0.97(3)

O(1)-Ni(1)-O(1)#1	180.00(8)
O(1)-Ni(1)-O(2)#1	90.15(5)
O(1)#1-Ni(1)-O(2)#1	89.85(6)
O(1)-Ni(1)-O(2)	89.85(6)
O(1)#1-Ni(1)-O(2)	90.15(5)
O(2)#1-Ni(1)-O(2)	180.0
O(1)-Ni(1)-O(5)#1	91.82(7)
O(1)#1-Ni(1)-O(5)#1	88.18(7)
O(2)#1-Ni(1)-O(5)#1	91.28(6)
O(2)-Ni(1)-O(5)#1	88.72(6)
O(1)-Ni(1)-O(5)	88.18(7)
O(1)#1-Ni(1)-O(5)	91.82(7)
O(2)#1-Ni(1)-O(5)	88.72(6)
O(2)-Ni(1)-O(5)	91.28(6)
O(5)#1-Ni(1)-O(5)	180.0

O(8)-Cl(1)-O(9)	111.08(10)
O(8)-Cl(1)-O(7)	110.51(10)
O(9)-Cl(1)-O(7)	109.51(10)
O(8)-Cl(1)-O(6)	109.60(10)
O(9)-Cl(1)-O(6)	109.05(10)
O(7)-Cl(1)-O(6)	106.99(10)
C(4)-O(1)-Ni(1)	130.17(13)
C(6)-O(2)-Ni(1)	125.15(14)
C(6)-O(3)-C(9)	122.67(16)
C(7)-O(4)-C(2)	119.51(15)
Ni(1)-O(5)-H(5A)	116(2)
Ni(1)-O(5)-H(5B)	110(2)
H(5A)-O(5)-H(5B)	109(3)
C(2)-C(1)-H(1A)	109.8(15)
C(2)-C(1)-H(1B)	108.6(16)
H(1A)-C(1)-H(1B)	106(2)
C(2)-C(1)-H(1C)	108.5(15)
H(1A)-C(1)-H(1C)	110(2)
H(1B)-C(1)-H(1C)	113(2)
C(3)-C(2)-O(4)	121.36(18)
C(3)-C(2)-C(1)	127.6(2)
O(4)-C(2)-C(1)	111.06(17)
C(2)-C(3)-C(4)	122.1(2)
C(2)-C(3)-H(3)	119.9(15)
C(4)-C(3)-H(3)	118.1(15)
O(1)-C(4)-C(3)	119.95(19)
O(1)-C(4)-C(5)	124.86(18)
C(3)-C(4)-C(5)	115.19(17)
C(7)-C(5)-C(6)	117.49(18)
C(7)-C(5)-C(4)	119.61(17)
C(6)-C(5)-C(4)	122.90(17)
O(2)-C(6)-O(3)	114.85(17)
O(2)-C(6)-C(5)	127.04(19)
O(3)-C(6)-C(5)	118.11(17)
O(4)-C(7)-C(5)	122.26(18)
O(4)-C(7)-C(8)	115.63(17)

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 ($Å^2 x\ 10^3$) for **9** (M= Ni). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	18(1)	29(1)	21(1)	1(1)	-2(1)	2(1)
Cl(1)	28(1)	27(1)	34(1)	0(1)	1(1)	-1(1)
O(1)	26(1)	41(1)	31(1)	3(1)	1(1)	5(1)
O(2)	24(1)	37(1)	37(1)	1(1)	-1(1)	7(1)
O(3)	24(1)	36(1)	35(1)	-1(1)	0(1)	4(1)
O(4)	22(1)	31(1)	36(1)	3(1)	2(1)	5(1)
O(5)	43(1)	46(1)	43(1)	3(1)	-11(1)	3(1)
O(6)	46(1)	55(1)	34(1)	5(1)	-2(1)	4(1)
O(7)	34(1)	44(1)	55(1)	-3(1)	-11(1)	-4(1)
O(8)	46(1)	33(1)	76(1)	-4(1)	18(1)	6(1)
O(9)	50(1)	33(1)	52(1)	0(1)	18(1)	-10(1)
C(1)	28(1)	39(1)	44(1)	0(1)	4(1)	8(1)
C(2)	24(1)	26(1)	39(1)	1(1)	6(1)	-2(1)
C(3)	26(1)	33(1)	34(1)	-1(1)	5(1)	1(1)

C(4)	22(1)	28(1)	35(1)	5(1)	3(1)	-2(1)
C(5)	20(1)	26(1)	32(1)	2(1)	2(1)	-1(1)
C(6)	23(1)	26(1)	35(1)	2(1)	2(1)	-2(1)
C(7)	20(1)	23(1)	37(1)	3(1)	4(1)	-1(1)
C(8)	23(1)	30(1)	35(1)	4(1)	-2(1)	0(1)
C(9)	25(1)	30(1)	37(1)	2(1)	-1(1)	-3(1)
C(10)	37(1)	48(2)	40(1)	-6(1)	2(1)	4(1)

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

	x	y	z	U(eq)
H(1A)	5930(30)	1910(20)	7310(30)	54(8)
H(1B)	6640(30)	2940(20)	6810(30)	59(8)
H(1C)	5820(30)	2140(20)	5750(30)	51(7)
H(3)	3740(20)	3113(17)	4980(20)	35(6)
H(5A)	1810(40)	6610(30)	5250(40)	102(12)
H(5B)	670(30)	6870(30)	4370(30)	74(11)
H(8)	4080(20)	4123(18)	10080(20)	37(6)
H(10A)	2950(40)	5180(20)	11730(30)	73(10)
H(10B)	1350(40)	5210(30)	11360(30)	94(12)
H(10C)	2310(30)	6170(20)	11020(20)	61(8)

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

O(1)#1-Ni(1)-O(1)-C(4)	28(100)
O(2)#1-Ni(1)-O(1)-C(4)	178.96(17)
O(2)-Ni(1)-O(1)-C(4)	-1.04(17)
O(5)#1-Ni(1)-O(1)-C(4)	-89.75(17)
O(5)-Ni(1)-O(1)-C(4)	90.25(17)
O(1)-Ni(1)-O(2)-C(6)	1.35(16)
O(1)#1-Ni(1)-O(2)-C(6)	-178.65(16)
O(2)#1-Ni(1)-O(2)-C(6)	-74(89)

O(5)#1-Ni(1)-O(2)-C(6)	93.18(17)
O(5)-Ni(1)-O(2)-C(6)	-86.82(17)
C(7)-O(4)-C(2)-C(3)	-0.4(3)
C(7)-O(4)-C(2)-C(1)	179.24(17)
O(4)-C(2)-C(3)-C(4)	0.5(3)
C(1)-C(2)-C(3)-C(4)	-179.1(2)
Ni(1)-O(1)-C(4)-C(3)	-179.13(14)
Ni(1)-O(1)-C(4)-C(5)	1.1(3)
C(2)-C(3)-C(4)-O(1)	-179.97(19)
C(2)-C(3)-C(4)-C(5)	-0.1(3)
O(1)-C(4)-C(5)-C(7)	179.61(18)
C(3)-C(4)-C(5)-C(7)	-0.2(3)
O(1)-C(4)-C(5)-C(6)	-0.9(3)
C(3)-C(4)-C(5)-C(6)	179.28(18)
Ni(1)-O(2)-C(6)-O(3)	178.32(11)
Ni(1)-O(2)-C(6)-C(5)	-1.7(3)
C(9)-O(3)-C(6)-O(2)	179.68(17)
C(9)-O(3)-C(6)-C(5)	-0.3(3)
C(7)-C(5)-C(6)-O(2)	-179.14(19)
C(4)-C(5)-C(6)-O(2)	1.4(3)
C(7)-C(5)-C(6)-O(3)	0.8(3)
C(4)-C(5)-C(6)-O(3)	-178.70(17)
C(2)-O(4)-C(7)-C(5)	0.1(3)
C(2)-O(4)-C(7)-C(8)	-179.18(16)
C(6)-C(5)-C(7)-O(4)	-179.26(17)
C(4)-C(5)-C(7)-O(4)	0.3(3)
C(6)-C(5)-C(7)-C(8)	-0.1(3)
C(4)-C(5)-C(7)-C(8)	179.44(17)
O(4)-C(7)-C(8)-C(9)	178.00(17)
C(5)-C(7)-C(8)-C(9)	-1.2(3)
C(7)-C(8)-C(9)-O(3)	1.8(3)
C(7)-C(8)-C(9)-C(10)	-177.3(2)
C(6)-O(3)-C(9)-C(8)	-1.1(3)
C(6)-O(3)-C(9)-C(10)	178.18(19)

Symmetry transformations used to generate equivalent atoms:

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

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5B)...O(6)#2	0.82(3)	2.04(3)	2.854(3)	173(3)

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:

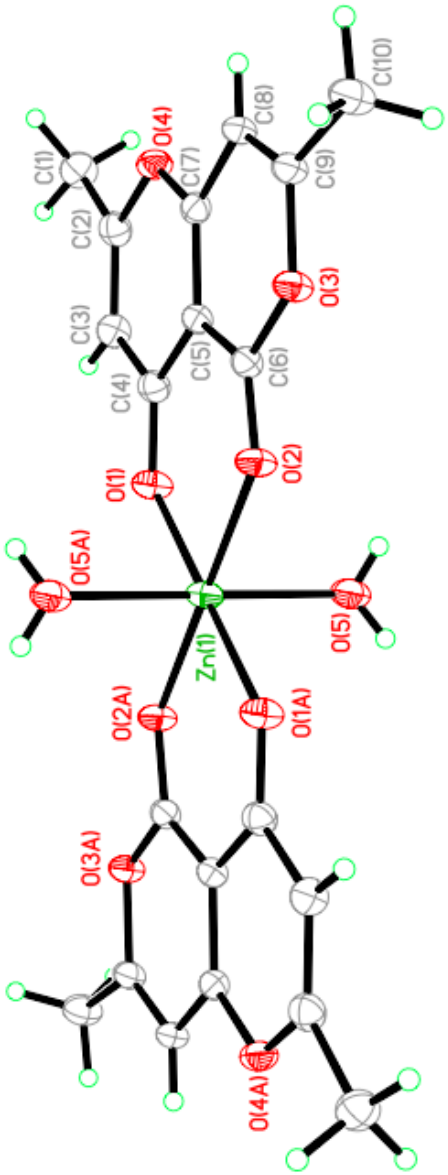


Table 1. Crystal data and structure refinement for **9** (M= Zn).

Identification code	**9** (M= Zn)
Empirical formula	C20 H20 Cl2 O18 Zn

Formula weight	684.63	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 9.973(3) Å	a= 90°.
	b = 12.636(3) Å	b= 95.500(4)°.
	c = 10.432(3) Å	g = 90°.
Volume	1308.5(6) $Å^3$	
Z	2	
Density (calculated)	1.738 Mg/m^3	
Absorption coefficient	1.229 mm^{-1}	
F(000)	696	
Crystal size	0.32 x 0.11 x 0.08 mm^3	
Theta range for data collection	2.54 to 27.00°.	
Index ranges	-12<=h<=12, -16<=k<=16, -13<=l<=13	
Reflections collected	14755	
Independent reflections	2855 [R(int) = 0.0382]	
Completeness to theta = 27.00°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9080 and 0.6944	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2855 / 0 / 227	
Goodness-of-fit on F^2	1.082	
Final R indices [I>2sigma(I)]	R1 = 0.0387, wR2 = 0.0944	
R indices (all data)	R1 = 0.0530, wR2 = 0.1044	
Largest diff. peak and hole	0.680 and -0.305 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **9** (M= Zn). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Zn(1)	5000	0	0	30(1)
Cl(1)	9255(1)	-1861(1)	2272(1)	31(1)
O(1)	6744(2)	851(2)	54(2)	34(1)

O(2)	5412(2)	-238(2)	1972(2)	31(1)
O(3)	6410(2)	-222(2)	3924(2)	31(1)
O(4)	9546(2)	1582(2)	3013(2)	31(1)
O(5)	6090(3)	-1354(2)	-395(2)	42(1)
O(6)	9269(2)	-2029(2)	3645(2)	50(1)
O(7)	7856(2)	-1767(2)	1792(2)	52(1)
O(8)	9977(2)	-921(2)	2031(2)	53(1)
O(9)	9823(2)	-2771(2)	1716(2)	52(1)
C(1)	10911(3)	2555(3)	1709(4)	40(1)
C(2)	9669(3)	1922(2)	1778(3)	33(1)
C(3)	8743(3)	1691(2)	816(3)	33(1)
C(4)	7582(3)	1062(2)	987(3)	29(1)
C(5)	7481(3)	715(2)	2303(3)	26(1)
C(6)	6397(3)	80(2)	2666(3)	27(1)
C(7)	8470(3)	989(2)	3251(3)	27(1)
C(8)	8428(3)	676(2)	4547(3)	29(1)
C(9)	7418(3)	66(2)	4848(3)	30(1)
C(10)	7226(4)	-388(3)	6114(3)	40(1)

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

Zn(1)-O(1)#1	2.0408(19)
Zn(1)-O(1)	2.0408(19)
Zn(1)-O(2)#1	2.0809(19)
Zn(1)-O(2)	2.0809(19)
Zn(1)-O(5)#1	2.089(3)
Zn(1)-O(5)	2.089(3)
Cl(1)-O(8)	1.424(2)
Cl(1)-O(9)	1.429(2)
Cl(1)-O(7)	1.442(2)
Cl(1)-O(6)	1.446(2)
O(1)-C(4)	1.250(3)
O(2)-C(6)	1.230(3)
O(3)-C(6)	1.366(3)
O(3)-C(9)	1.374(3)
O(4)-C(7)	1.351(3)
O(4)-C(2)	1.375(3)
O(5)-H(5A)	0.70(4)
O(5)-H(5B)	0.73(4)
C(1)-C(2)	1.482(4)
C(1)-H(1A)	0.93(4)
C(1)-H(1B)	0.97(4)
C(1)-H(1C)	0.92(4)
C(2)-C(3)	1.330(4)
C(3)-C(4)	1.429(4)
C(3)-H(3)	0.90(3)
C(4)-C(5)	1.455(4)
C(5)-C(7)	1.372(4)
C(5)-C(6)	1.426(4)
C(7)-C(8)	1.414(4)
C(8)-C(9)	1.330(4)
C(8)-H(8)	0.82(3)
C(9)-C(10)	1.469(4)
C(10)-H(10A)	0.93(4)
C(10)-H(10B)	0.95(5)

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)#1-Zn(1)-O(2)	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)
C(7)-O(4)-C(2)	119.3(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)

C(3)-C(2)-O(4)	121.3(3)
C(3)-C(2)-C(1)	127.3(3)
O(4)-C(2)-C(1)	111.4(3)
C(2)-C(3)-C(4)	122.7(3)
C(2)-C(3)-H(3)	117.1(19)
C(4)-C(3)-H(3)	120.1(19)
O(1)-C(4)-C(3)	120.8(3)
O(1)-C(4)-C(5)	124.7(3)
C(3)-C(4)-C(5)	114.6(2)
C(7)-C(5)-C(6)	117.5(2)
C(7)-C(5)-C(4)	119.5(2)
C(6)-C(5)-C(4)	122.9(2)
O(2)-C(6)-O(3)	114.0(2)
O(2)-C(6)-C(5)	127.6(3)
O(3)-C(6)-C(5)	118.4(2)
O(4)-C(7)-C(5)	122.5(2)
O(4)-C(7)-C(8)	115.3(2)
C(5)-C(7)-C(8)	122.2(3)
C(9)-C(8)-C(7)	118.7(3)
C(9)-C(8)-H(8)	123(2)
C(7)-C(8)-H(8)	118(2)
C(8)-C(9)-O(3)	120.6(3)
C(8)-C(9)-C(10)	127.2(3)
O(3)-C(9)-C(10)	112.2(3)
C(9)-C(10)-H(10A)	110(2)
C(9)-C(10)-H(10B)	108(2)
H(10A)-C(10)-H(10B)	107(3)
C(9)-C(10)-H(10C)	110(2)
H(10A)-C(10)-H(10C)	111(3)
H(10B)-C(10)-H(10C)	110(3)

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9** (M= Zn). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zn(1)	24(1)	38(1)	26(1)	-2(1)	-5(1)	-1(1)
Cl(1)	29(1)	30(1)	34(1)	-1(1)	-3(1)	3(1)
O(1)	29(1)	47(1)	26(1)	2(1)	-3(1)	-5(1)
O(2)	24(1)	42(1)	26(1)	1(1)	-4(1)	-6(1)
O(3)	26(1)	38(1)	27(1)	1(1)	-4(1)	-3(1)
O(4)	25(1)	34(1)	33(1)	-1(1)	-3(1)	-5(1)
O(5)	37(1)	46(2)	39(1)	-8(1)	-13(1)	6(1)
O(6)	52(1)	61(2)	35(1)	-6(1)	-2(1)	-9(1)
O(7)	41(1)	49(1)	63(2)	2(1)	-21(1)	2(1)
O(8)	47(1)	40(1)	72(2)	2(1)	15(1)	-6(1)
O(9)	60(2)	37(1)	62(2)	4(1)	24(1)	10(1)
C(1)	33(2)	43(2)	44(2)	3(2)	5(1)	-5(1)
C(2)	28(1)	33(2)	38(2)	3(1)	4(1)	1(1)
C(3)	32(2)	34(2)	32(2)	1(1)	5(1)	-1(1)
C(4)	25(1)	32(2)	31(1)	-1(1)	2(1)	3(1)
C(5)	22(1)	26(1)	29(1)	0(1)	-1(1)	2(1)
C(6)	27(1)	27(1)	27(1)	-1(1)	-2(1)	4(1)
C(7)	23(1)	24(1)	34(2)	-2(1)	-1(1)	2(1)
C(8)	26(1)	31(1)	29(1)	-2(1)	-8(1)	1(1)
C(9)	28(1)	32(2)	28(1)	-2(1)	-5(1)	3(1)
C(10)	38(2)	50(2)	31(2)	5(2)	-4(1)	-4(2)

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

	x	y	z	U(eq)
H(1A)	10950(30)	3080(30)	2340(40)	50(10)
H(1B)	10930(30)	2900(30)	880(30)	44(9)
H(1C)	11660(40)	2140(30)	1880(30)	47(10)
H(3)	8880(30)	1940(20)	30(30)	31(8)
H(5A)	6610(40)	-1510(30)	60(40)	51(13)
H(5B)	5670(50)	-1800(30)	-630(40)	67(16)
H(8)	9060(40)	850(30)	5070(30)	41(9)
H(10A)	7230(40)	-1120(30)	6070(40)	63(12)
H(10B)	6360(50)	-190(30)	6340(40)	68(13)
H(10C)	7870(40)	-150(20)	6720(30)	39(9)

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

O(1)#1-Zn(1)-O(1)-C(4)	10(100)
O(2)#1-Zn(1)-O(1)-C(4)	179.7(2)
O(2)-Zn(1)-O(1)-C(4)	-0.3(2)
O(5)#1-Zn(1)-O(1)-C(4)	88.5(2)
O(5)-Zn(1)-O(1)-C(4)	-91.5(2)
O(1)#1-Zn(1)-O(2)-C(6)	179.1(2)
O(1)-Zn(1)-O(2)-C(6)	-0.9(2)
O(2)#1-Zn(1)-O(2)-C(6)	0(100)
O(5)#1-Zn(1)-O(2)-C(6)	-92.1(2)
O(5)-Zn(1)-O(2)-C(6)	87.9(2)
C(7)-O(4)-C(2)-C(3)	0.8(4)
C(7)-O(4)-C(2)-C(1)	-179.9(3)
O(4)-C(2)-C(3)-C(4)	-1.3(5)
C(1)-C(2)-C(3)-C(4)	179.4(3)
Zn(1)-O(1)-C(4)-C(3)	-179.19(19)
Zn(1)-O(1)-C(4)-C(5)	0.4(4)

C(2)-C(3)-C(4)-O(1)	-179.1(3)
C(2)-C(3)-C(4)-C(5)	1.2(4)
O(1)-C(4)-C(5)-C(7)	179.6(3)
C(3)-C(4)-C(5)-C(7)	-0.8(4)
O(1)-C(4)-C(5)-C(6)	0.5(4)
C(3)-C(4)-C(5)-C(6)	-179.9(3)
Zn(1)-O(2)-C(6)-O(3)	-178.49(15)
Zn(1)-O(2)-C(6)-C(5)	1.9(4)
C(9)-O(3)-C(6)-O(2)	-179.2(2)
C(9)-O(3)-C(6)-C(5)	0.4(4)
C(7)-C(5)-C(6)-O(2)	179.1(3)
C(4)-C(5)-C(6)-O(2)	-1.8(4)
C(7)-C(5)-C(6)-O(3)	-0.4(4)
C(4)-C(5)-C(6)-O(3)	178.7(2)
C(2)-O(4)-C(7)-C(5)	-0.3(4)
C(2)-O(4)-C(7)-C(8)	-180.0(2)
C(6)-C(5)-C(7)-O(4)	179.5(2)
C(4)-C(5)-C(7)-O(4)	0.4(4)
C(6)-C(5)-C(7)-C(8)	-0.8(4)
C(4)-C(5)-C(7)-C(8)	-180.0(2)
O(4)-C(7)-C(8)-C(9)	-178.1(2)
C(5)-C(7)-C(8)-C(9)	2.2(4)
C(7)-C(8)-C(9)-O(3)	-2.2(4)
C(7)-C(8)-C(9)-C(10)	176.6(3)
C(6)-O(3)-C(9)-C(8)	1.0(4)
C(6)-O(3)-C(9)-C(10)	-178.0(3)

Symmetry transformations used to generate equivalent atoms:

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

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5A)...O(7)	0.70(4)	2.12(4)	2.794(4)	164(4)
O(5)-H(5B)...O(6)#2	0.73(4)	2.12(5)	2.851(4)	174(5)

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:

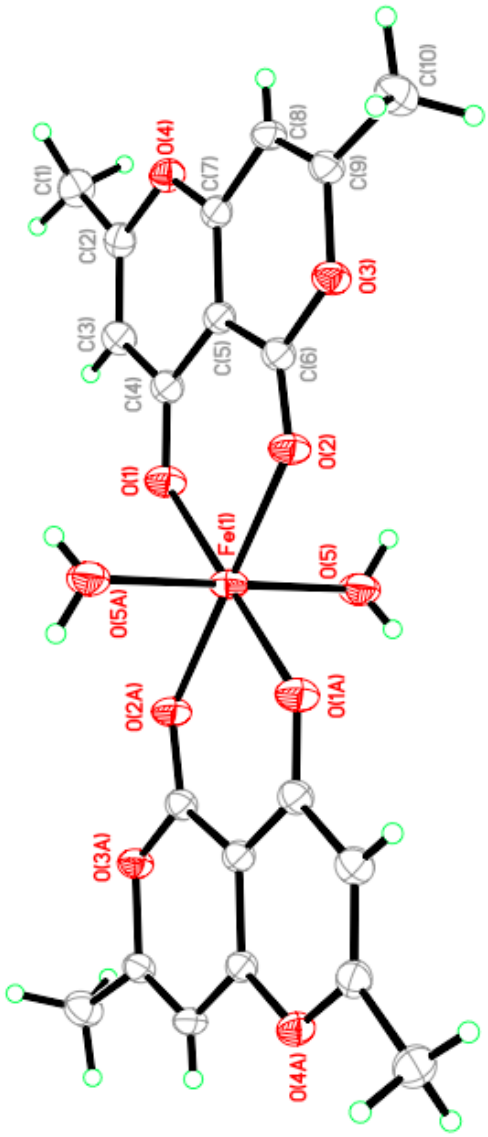


Table 1. Crystal data and structure refinement for **9** (M= Fe).

Identification code	**9** (M= Fe)
Empirical formula	C20 H20 Cl2 Fe O18
Formula weight	675.11

Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.113(2) Å	a= 90°.
	b = 12.627(3) Å	b= 95.402(4)°.
	c = 10.339(2) Å	g = 90°.
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^{ij} tensor.

	x	y	z	U(eq)
Fe(1)	0	5000	5000	33(1)
Cl(1)	5753(1)	6841(1)	2761(1)	35(1)
O(1)	1739(1)	5855(1)	5088(1)	38(1)
O(2)	452(1)	4761(1)	6996(1)	35(1)

O(3)	1426(1)	4777(1)	8973(1)	34(1)
O(4)	4528(1)	6584(1)	8048(1)	35(1)
O(5)	1123(2)	3625(2)	4546(2)	46(1)
O(6)	5733(2)	7011(2)	1370(2)	51(1)
O(7)	7152(2)	6769(2)	3231(2)	49(1)
O(8)	5206(2)	7769(2)	3308(2)	49(1)
O(6A)	6604(13)	6920(10)	3888(12)	51(1)
O(7A)	4655(13)	7575(11)	2782(13)	49(1)
O(8A)	6306(15)	6689(9)	1682(14)	49(1)
O(9)	5053(2)	5898(1)	3018(2)	60(1)
C(1)	5869(2)	7558(2)	6727(3)	43(1)
C(2)	4641(2)	6932(2)	6809(2)	35(1)
C(3)	3720(2)	6694(2)	5842(2)	35(1)
C(4)	2577(2)	6070(2)	6019(2)	32(1)
C(5)	2490(2)	5716(2)	7341(2)	29(1)
C(6)	1412(2)	5076(2)	7710(2)	30(1)
C(7)	3470(2)	5994(2)	8298(2)	30(1)
C(8)	3425(2)	5669(2)	9600(2)	32(1)
C(9)	2424(2)	5065(2)	9903(2)	33(1)
C(10)	2235(3)	4616(2)	11186(2)	45(1)

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

Fe(1)-O(1)	2.0581(15)
Fe(1)-O(1)#1	2.0581(15)
Fe(1)-O(2)#1	2.0932(15)
Fe(1)-O(2)	2.0932(15)
Fe(1)-O(5)#1	2.1506(18)
Fe(1)-O(5)	2.1506(18)
Cl(1)-O(8A)	1.308(12)
Cl(1)-O(6A)	1.385(11)
Cl(1)-O(9)	1.4241(18)
Cl(1)-O(8)	1.434(2)
Cl(1)-O(7A)	1.448(12)
Cl(1)-O(6)	1.452(2)

Cl(1)-O(7)	1.4547(19)
O(1)-C(4)	1.251(2)
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)-C(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)

H(1A)-C(1)-H(1B)	110(2)
C(2)-C(1)-H(1C)	110.9(17)
H(1A)-C(1)-H(1C)	105(2)
H(1B)-C(1)-H(1C)	111(2)
C(3)-C(2)-O(4)	120.99(19)
C(3)-C(2)-C(1)	127.4(2)
O(4)-C(2)-C(1)	111.63(19)
C(2)-C(3)-C(4)	123.0(2)
C(2)-C(3)-H(3)	119.6(18)
C(4)-C(3)-H(3)	117.4(17)
O(1)-C(4)-C(3)	121.31(19)
O(1)-C(4)-C(5)	124.21(18)
C(3)-C(4)-C(5)	114.49(18)
C(7)-C(5)-C(6)	117.73(18)
C(7)-C(5)-C(4)	119.66(18)
C(6)-C(5)-C(4)	122.60(18)
O(2)-C(6)-O(3)	115.07(17)
O(2)-C(6)-C(5)	126.79(19)
O(3)-C(6)-C(5)	118.14(17)
O(4)-C(7)-C(5)	122.22(18)
O(4)-C(7)-C(8)	116.17(17)
C(5)-C(7)-C(8)	121.61(18)
C(9)-C(8)-C(7)	119.1(2)
C(9)-C(8)-H(8)	121(2)
C(7)-C(8)-H(8)	120(2)
C(8)-C(9)-O(3)	120.81(19)
C(8)-C(9)-C(10)	126.9(2)
O(3)-C(9)-C(10)	112.29(19)
C(9)-C(10)-H(9C)	112.1(15)
C(9)-C(10)-H(10A)	108.6(18)
H(9C)-C(10)-H(10A)	111(2)
C(9)-C(10)-H(10B)	109.8(19)
H(9C)-C(10)-H(10B)	106(2)
H(10A)-C(10)-H(10B)	109(3)

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9** (M= Fe). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Fe(1)	25(1)	42(1)	31(1)	-4(1)	-3(1)	-1(1)
Cl(1)	33(1)	33(1)	38(1)	-1(1)	-2(1)	4(1)
O(1)	30(1)	51(1)	34(1)	0(1)	0(1)	-5(1)
O(2)	26(1)	46(1)	32(1)	-1(1)	-2(1)	-6(1)
O(3)	28(1)	40(1)	34(1)	1(1)	-2(1)	-3(1)
O(4)	26(1)	36(1)	41(1)	-1(1)	1(1)	-5(1)
O(5)	39(1)	49(1)	48(1)	-12(1)	-12(1)	8(1)
O(6)	49(1)	67(2)	36(1)	-5(1)	-3(1)	-5(1)
O(7)	38(1)	50(1)	57(1)	3(1)	-14(1)	4(1)
O(8)	52(1)	35(1)	61(1)	2(1)	21(1)	9(1)
O(6A)	49(1)	67(2)	36(1)	-5(1)	-3(1)	-5(1)
O(7A)	38(1)	50(1)	57(1)	3(1)	-14(1)	4(1)
O(8A)	52(1)	35(1)	61(1)	2(1)	21(1)	9(1)
O(9)	55(1)	44(1)	83(1)	-1(1)	22(1)	-5(1)
C(1)	34(1)	45(1)	51(2)	5(1)	7(1)	-7(1)
C(2)	29(1)	32(1)	44(1)	1(1)	4(1)	1(1)
C(3)	30(1)	39(1)	37(1)	1(1)	5(1)	1(1)
C(4)	27(1)	33(1)	37(1)	-1(1)	2(1)	3(1)
C(5)	24(1)	29(1)	35(1)	-3(1)	1(1)	2(1)
C(6)	26(1)	31(1)	33(1)	-1(1)	1(1)	2(1)
C(7)	24(1)	28(1)	38(1)	-2(1)	0(1)	2(1)
C(8)	26(1)	34(1)	34(1)	-3(1)	-7(1)	-1(1)
C(9)	30(1)	33(1)	34(1)	-2(1)	-4(1)	5(1)
C(10)	42(1)	52(2)	40(1)	9(1)	1(1)	-2(1)

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

	x	y	z	U(eq)
H(1A)	5960(30)	8050(20)	7430(30)	60(8)
H(1B)	5840(30)	7900(20)	5970(30)	57(8)
H(1C)	6630(30)	7120(20)	6850(30)	57(8)
H(3)	3810(20)	6916(19)	5090(30)	44(7)
H(5A)	750(30)	3200(30)	4310(30)	66(12)
H(5B)	1720(30)	3440(30)	5050(30)	69(10)
H(8)	3950(30)	5830(20)	10100(30)	51(8)
H(9C)	2850(30)	4884(19)	11840(30)	41(7)
H(10A)	2290(30)	3860(30)	11130(30)	66(9)
H(10B)	1330(40)	4820(20)	11440(30)	82(10)

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

O(1)#1-Fe(1)-O(1)-C(4)	57(100)
O(2)#1-Fe(1)-O(1)-C(4)	179.34(18)
O(2)-Fe(1)-O(1)-C(4)	-0.66(18)
O(5)#1-Fe(1)-O(1)-C(4)	87.59(19)
O(5)-Fe(1)-O(1)-C(4)	-92.41(19)
O(1)-Fe(1)-O(2)-C(6)	-0.09(18)
O(1)#1-Fe(1)-O(2)-C(6)	179.91(18)
O(2)#1-Fe(1)-O(2)-C(6)	18(99)
O(5)#1-Fe(1)-O(2)-C(6)	-92.13(18)
O(5)-Fe(1)-O(2)-C(6)	87.87(18)
C(7)-O(4)-C(2)-C(3)	-0.1(3)
C(7)-O(4)-C(2)-C(1)	-179.57(18)
O(4)-C(2)-C(3)-C(4)	-0.4(3)
C(1)-C(2)-C(3)-C(4)	179.1(2)
Fe(1)-O(1)-C(4)-C(3)	-179.51(14)
Fe(1)-O(1)-C(4)-C(5)	0.8(3)

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(19)
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 °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5A)...O(6)#2	0.69(3)	2.19(4)	2.871(4)	170(4)
O(5)-H(5A)...O(7A)#2	0.69(3)	2.30(4)	2.796(13)	130(4)

O(5)-H(5B)...O(6A)#3	0.79(4)	1.99(4)	2.771(12)	171(3)
O(5)-H(5B)...O(7)#3	0.79(4)	2.04(4)	2.796(3)	160(3)

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- δ ^{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.

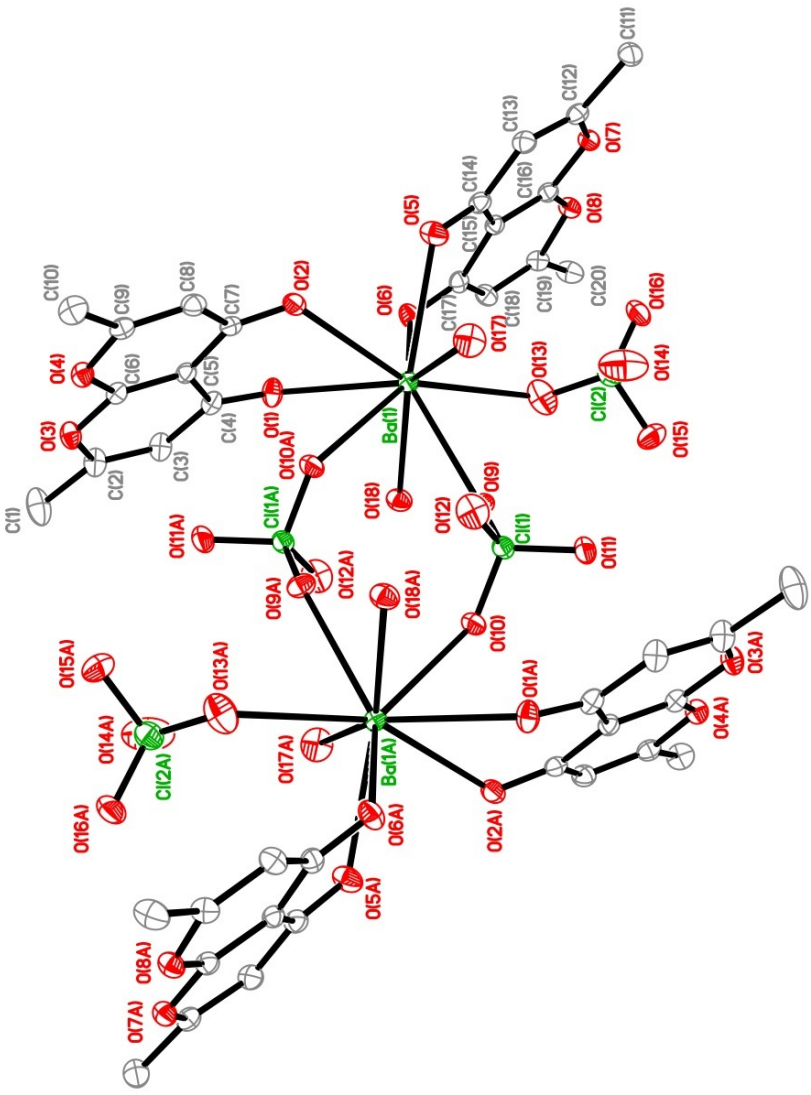


Table 1. Crystal data and structure refinement for **10**.

Identification code	**10**
Empirical formula	C20 H20 Ba Cl2 O18
Formula weight	756.60

Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.1152(15) Å	a= 92.527(2)°.
	b = 10.2248(15) Å	b= 99.751(2)°.
	c = 12.9429(19) Å	g = 97.818(2)°.
Volume	1303.9(3) $Å^3$	
Z	2	
Density (calculated)	1.927 Mg/m^3	
Absorption coefficient	1.815 mm^{-1}	
F(000)	748	
Crystal size	0.39 x 0.24 x 0.08 mm^3	
Theta range for data collection	1.60 to 27.00°.	
Index ranges	-12<=h<=12, -13<=k<=13, -16<=l<=16	
Reflections collected	14739	
Independent reflections	5654 [R(int) = 0.0153]	
Completeness to theta = 27.00°	99.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8684 and 0.5379	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5654 / 8 / 479	
Goodness-of-fit on F^2	1.186	
Final R indices [I>2sigma(I)]	R1 = 0.0205, wR2 = 0.0635	
R indices (all data)	R1 = 0.0209, wR2 = 0.0639	
Largest diff. peak and hole	0.543 and -0.453 $e.Å^{-3}$	

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.

	x	y	z	U(eq)
Ba(1)	2395(1)	7731(1)	5250(1)	23(1)
Cl(1)	669(1)	10748(1)	6452(1)	27(1)
Cl(2)	3611(1)	7685(1)	8257(1)	37(1)
O(1)	1167(2)	6090(2)	3477(1)	34(1)
O(2)	3257(2)	8259(2)	3387(1)	35(1)
O(3)	-419(2)	7004(2)	556(1)	31(1)
O(4)	1083(2)	8702(2)	522(1)	28(1)
O(5)	5095(2)	7470(2)	5372(1)	37(1)
O(6)	3131(2)	5235(2)	5515(1)	33(1)
O(7)	7445(2)	5977(2)	7756(1)	27(1)
O(8)	5929(2)	4337(2)	7920(1)	27(1)
O(9)	978(2)	9438(2)	6278(1)	33(1)
O(10)	-777(2)	10728(2)	6188(1)	36(1)
O(11)	1125(2)	11195(2)	7536(1)	47(1)
O(12)	1335(2)	11624(2)	5794(2)	47(1)
O(13)	2687(13)	6904(13)	7428(9)	69(3)
O(14)	4060(5)	9049(5)	8151(10)	76(3)
O(15)	2715(6)	7681(6)	9032(6)	49(1)
O(16)	4760(14)	7070(16)	8681(15)	47(3)
O(13A)	2854(16)	6990(17)	7326(9)	52(4)
O(14A)	4209(10)	8528(15)	7501(10)	81(5)
O(15A)	3000(20)	8210(20)	8970(11)	136(8)
O(16A)	4550(20)	6810(20)	8653(19)	37(4)
O(17)	3883(2)	10210(2)	5660(2)	48(1)
O(18)	-64(2)	6344(2)	5562(2)	35(1)
C(1)	-2293(4)	5319(3)	307(3)	53(1)
C(2)	-1039(3)	5950(2)	1016(2)	34(1)
C(3)	-504(3)	5630(2)	1968(2)	33(1)
C(4)	724(2)	6363(2)	2579(2)	27(1)
C(5)	1357(2)	7459(2)	2053(2)	24(1)
C(6)	712(2)	7705(2)	1091(2)	24(1)

C(7)	2581(2)	8371(2)	2520(2)	27(1)
C(8)	2951(2)	9431(2)	1870(2)	29(1)
C(9)	2240(2)	9574(2)	931(2)	27(1)
C(10)	2510(3)	10606(3)	192(2)	37(1)
C(11)	9316(3)	7662(3)	7864(2)	35(1)
C(12)	7940(2)	7103(2)	7313(2)	27(1)
C(13)	7192(2)	7575(2)	6497(2)	29(1)
C(14)	5820(2)	6967(2)	6066(2)	28(1)
C(15)	5352(2)	5755(2)	6535(2)	23(1)
C(16)	6202(2)	5366(2)	7356(2)	24(1)
C(17)	4001(2)	4977(2)	6237(2)	26(1)
C(18)	3743(2)	3875(2)	6875(2)	30(1)
C(19)	4653(2)	3596(2)	7671(2)	28(1)
C(20)	4481(3)	2539(3)	8413(2)	39(1)

Table 3. Bond lengths [Å] and angles [°] for **10**.

Ba(1)-O(17)	2.7472(18)
Ba(1)-O(2)	2.7507(16)
Ba(1)-O(5)	2.7586(17)
Ba(1)-O(6)	2.7731(16)
Ba(1)-O(1)	2.7842(16)
Ba(1)-O(18)	2.7917(18)
Ba(1)-O(13A)	2.804(15)
Ba(1)-O(9)	2.8269(16)
Ba(1)-O(10)#1	2.9247(17)
Ba(1)-O(13)	2.958(13)
Ba(1)-O(14A)	3.177(10)
Cl(1)-O(9)	1.4335(16)
Cl(1)-O(11)	1.4341(18)
Cl(1)-O(12)	1.442(2)
Cl(1)-O(10)	1.4423(16)
Cl(2)-O(15A)	1.322(11)
Cl(2)-O(16)	1.435(11)
Cl(2)-O(13A)	1.417(11)

Cl(2)-O(14)	1.427(4)
Cl(2)-O(13)	1.430(10)
Cl(2)-O(15)	1.461(5)
Cl(2)-O(14A)	1.483(7)
Cl(2)-O(16A)	1.438(16)
O(1)-C(4)	1.232(3)
O(2)-C(7)	1.230(3)
O(3)-C(6)	1.329(3)
O(3)-C(2)	1.382(3)
O(4)-C(6)	1.332(3)
O(4)-C(9)	1.382(3)
O(5)-C(14)	1.237(3)
O(6)-C(17)	1.235(3)
O(7)-C(16)	1.332(3)
O(7)-C(12)	1.381(3)
O(8)-C(16)	1.332(2)
O(8)-C(19)	1.384(3)
O(10)-Ba(1)#1	2.9247(17)
O(18)-H(1O)	0.80(5)
O(18)-H(2O)	0.74(4)
C(1)-C(2)	1.483(4)
C(1)-H(1A)	0.87(4)
C(1)-H(1B)	0.95(4)
C(1)-H(1C)	1.04(4)
C(2)-C(3)	1.333(3)
C(3)-C(4)	1.446(3)
C(3)-H(3)	0.96(3)
C(4)-C(5)	1.465(3)
C(5)-C(6)	1.356(3)
C(5)-C(7)	1.466(3)
C(7)-C(8)	1.448(3)
C(8)-C(9)	1.329(3)
C(8)-H(8)	0.86(3)
C(9)-C(10)	1.484(3)
C(10)-H(10A)	0.82(4)
C(10)-H(10B)	0.93(4)

C(10)-H(10C)	0.94(3)
C(11)-C(12)	1.478(3)
C(11)-H(11A)	0.81(5)
C(11)-H(11B)	0.87(4)
C(11)-H(11C)	0.99(5)
C(12)-C(13)	1.339(3)
C(13)-C(14)	1.447(3)
C(13)-H(13)	1.02(3)
C(14)-C(15)	1.461(3)
C(15)-C(16)	1.359(3)
C(15)-C(17)	1.466(3)
C(17)-C(18)	1.447(3)
C(18)-C(19)	1.328(3)
C(18)-H(18)	0.92(3)
C(19)-C(20)	1.489(3)
C(20)-H(20A)	0.80(4)
C(20)-H(20B)	0.96(4)
C(20)-H(20C)	1.08(4)

O(17)-Ba(1)-O(2)	76.35(6)
O(17)-Ba(1)-O(5)	72.07(6)
O(2)-Ba(1)-O(5)	68.78(5)
O(17)-Ba(1)-O(6)	131.36(6)
O(2)-Ba(1)-O(6)	100.06(5)
O(5)-Ba(1)-O(6)	62.04(5)
O(17)-Ba(1)-O(1)	136.81(5)
O(2)-Ba(1)-O(1)	62.45(5)
O(5)-Ba(1)-O(1)	102.41(6)
O(6)-Ba(1)-O(1)	71.59(5)
O(17)-Ba(1)-O(18)	139.93(6)
O(2)-Ba(1)-O(18)	128.62(5)
O(5)-Ba(1)-O(18)	141.41(6)
O(6)-Ba(1)-O(18)	79.99(6)
O(1)-Ba(1)-O(18)	69.34(5)
O(17)-Ba(1)-O(13A)	96.1(4)
O(2)-Ba(1)-O(13A)	151.5(3)

O(5)-Ba(1)-O(13A)	82.8(3)
O(6)-Ba(1)-O(13A)	64.2(3)
O(1)-Ba(1)-O(13A)	126.3(3)
O(18)-Ba(1)-O(13A)	74.3(4)
O(17)-Ba(1)-O(9)	69.58(6)
O(2)-Ba(1)-O(9)	123.10(5)
O(5)-Ba(1)-O(9)	134.27(5)
O(6)-Ba(1)-O(9)	136.64(5)
O(1)-Ba(1)-O(9)	122.54(5)
O(18)-Ba(1)-O(9)	70.36(5)
O(13A)-Ba(1)-O(9)	77.5(3)
O(17)-Ba(1)-O(10)#1	78.66(6)
O(2)-Ba(1)-O(10)#1	62.91(5)
O(5)-Ba(1)-O(10)#1	127.92(5)
O(6)-Ba(1)-O(10)#1	143.36(5)
O(1)-Ba(1)-O(10)#1	71.78(5)
O(18)-Ba(1)-O(10)#1	86.59(6)
O(13A)-Ba(1)-O(10)#1	143.4(3)
O(9)-Ba(1)-O(10)#1	66.60(5)
O(17)-Ba(1)-O(13)	98.3(2)
O(2)-Ba(1)-O(13)	155.0(3)
O(5)-Ba(1)-O(13)	86.3(3)
O(6)-Ba(1)-O(13)	64.8(2)
O(1)-Ba(1)-O(13)	124.5(2)
O(18)-Ba(1)-O(13)	70.7(2)
O(13A)-Ba(1)-O(13)	3.7(6)
O(9)-Ba(1)-O(13)	75.7(2)
O(10)#1-Ba(1)-O(13)	140.8(3)
O(17)-Ba(1)-O(14A)	60.33(17)
O(2)-Ba(1)-O(14A)	123.98(18)
O(5)-Ba(1)-O(14A)	65.4(3)
O(6)-Ba(1)-O(14A)	85.6(3)
O(1)-Ba(1)-O(14A)	157.2(3)
O(18)-Ba(1)-O(14A)	107.34(17)
O(13A)-Ba(1)-O(14A)	36.8(4)
O(9)-Ba(1)-O(14A)	74.4(4)

O(10)#1-Ba(1)-O(14A)	131.1(3)
O(13)-Ba(1)-O(14A)	39.7(3)
O(9)-Cl(1)-O(11)	109.61(11)
O(9)-Cl(1)-O(12)	109.29(11)
O(11)-Cl(1)-O(12)	109.91(13)
O(9)-Cl(1)-O(10)	109.11(10)
O(11)-Cl(1)-O(10)	109.45(11)
O(12)-Cl(1)-O(10)	109.45(12)
O(15A)-Cl(2)-O(16)	114.6(10)
O(15A)-Cl(2)-O(13A)	121.3(12)
O(16)-Cl(2)-O(13A)	112.1(9)
O(15A)-Cl(2)-O(14)	81.4(10)
O(16)-Cl(2)-O(14)	108.8(8)
O(13A)-Cl(2)-O(14)	114.5(10)
O(15A)-Cl(2)-O(13)	113.3(13)
O(16)-Cl(2)-O(13)	114.2(9)
O(13A)-Cl(2)-O(13)	9.7(13)
O(14)-Cl(2)-O(13)	120.7(8)
O(15A)-Cl(2)-O(15)	23.8(12)
O(16)-Cl(2)-O(15)	108.4(8)
O(13A)-Cl(2)-O(15)	107.5(8)
O(14)-Cl(2)-O(15)	105.1(4)
O(13)-Cl(2)-O(15)	98.1(6)
O(15A)-Cl(2)-O(14A)	120.5(9)
O(16)-Cl(2)-O(14A)	100.1(8)
O(13A)-Cl(2)-O(14A)	82.8(10)
O(14)-Cl(2)-O(14A)	40.9(5)
O(13)-Cl(2)-O(14A)	91.8(8)
O(15)-Cl(2)-O(14A)	142.4(8)
O(15A)-Cl(2)-O(16A)	114.9(13)
O(16)-Cl(2)-O(16A)	12.5(16)
O(13A)-Cl(2)-O(16A)	103.7(12)
O(14)-Cl(2)-O(16A)	121.2(11)
O(13)-Cl(2)-O(16A)	104.2(12)
O(15)-Cl(2)-O(16A)	103.7(10)
O(14A)-Cl(2)-O(16A)	108.8(11)

C(4)-O(1)-Ba(1)	130.44(14)
C(7)-O(2)-Ba(1)	129.07(14)
C(6)-O(3)-C(2)	118.51(17)
C(6)-O(4)-C(9)	118.13(17)
C(14)-O(5)-Ba(1)	127.55(14)
C(17)-O(6)-Ba(1)	124.16(13)
C(16)-O(7)-C(12)	118.56(17)
C(16)-O(8)-C(19)	117.78(17)
Cl(1)-O(9)-Ba(1)	148.38(10)
Cl(1)-O(10)-Ba(1)#1	131.12(11)
Cl(2)-O(13)-Ba(1)	120.4(8)
Cl(2)-O(13A)-Ba(1)	130.9(11)
Cl(2)-O(14A)-Ba(1)	107.2(4)
Ba(1)-O(18)-H(1O)	135(3)
Ba(1)-O(18)-H(2O)	109(3)
H(1O)-O(18)-H(2O)	110(4)
C(2)-C(1)-H(1A)	120(3)
C(2)-C(1)-H(1B)	108(2)
H(1A)-C(1)-H(1B)	103(3)
C(2)-C(1)-H(1C)	110(2)
H(1A)-C(1)-H(1C)	113(3)
H(1B)-C(1)-H(1C)	101(3)
C(3)-C(2)-O(3)	120.8(2)
C(3)-C(2)-C(1)	129.0(2)
O(3)-C(2)-C(1)	110.2(2)
C(2)-C(3)-C(4)	122.7(2)
C(2)-C(3)-H(3)	117.2(19)
C(4)-C(3)-H(3)	120.1(19)
O(1)-C(4)-C(3)	121.7(2)
O(1)-C(4)-C(5)	123.7(2)
C(3)-C(4)-C(5)	114.65(19)
C(6)-C(5)-C(4)	117.5(2)
C(6)-C(5)-C(7)	117.31(19)
C(4)-C(5)-C(7)	125.00(19)
O(3)-C(6)-O(4)	107.93(18)
O(3)-C(6)-C(5)	125.80(19)

O(4)-C(6)-C(5)	126.3(2)
O(2)-C(7)-C(8)	121.7(2)
O(2)-C(7)-C(5)	123.8(2)
C(8)-C(7)-C(5)	114.48(19)
C(9)-C(8)-C(7)	123.0(2)
C(9)-C(8)-H(8)	117.6(18)
C(7)-C(8)-H(8)	119.3(18)
C(8)-C(9)-O(4)	120.8(2)
C(8)-C(9)-C(10)	128.2(2)
O(4)-C(9)-C(10)	111.0(2)
C(9)-C(10)-H(10A)	110(3)
C(9)-C(10)-H(10B)	112(2)
H(10A)-C(10)-H(10B)	106(4)
C(9)-C(10)-H(10C)	110(2)
H(10A)-C(10)-H(10C)	109(4)
H(10B)-C(10)-H(10C)	109(3)
C(12)-C(11)-H(11A)	115(3)
C(12)-C(11)-H(11B)	116(3)
H(11A)-C(11)-H(11B)	116(4)
C(12)-C(11)-H(11C)	110(3)
H(11A)-C(11)-H(11C)	100(4)
H(11B)-C(11)-H(11C)	97(4)
C(13)-C(12)-O(7)	120.9(2)
C(13)-C(12)-C(11)	127.8(2)
O(7)-C(12)-C(11)	111.32(19)
C(12)-C(13)-C(14)	122.1(2)
C(12)-C(13)-H(13)	117.4(16)
C(14)-C(13)-H(13)	120.5(16)
O(5)-C(14)-C(13)	121.7(2)
O(5)-C(14)-C(15)	123.1(2)
C(13)-C(14)-C(15)	115.19(19)
C(16)-C(15)-C(14)	117.44(19)
C(16)-C(15)-C(17)	117.44(19)
C(14)-C(15)-C(17)	124.98(19)
O(7)-C(16)-O(8)	108.05(17)
O(7)-C(16)-C(15)	125.71(19)

O(8)-C(16)-C(15)	126.22(19)
O(6)-C(17)-C(18)	121.8(2)
O(6)-C(17)-C(15)	123.7(2)
C(18)-C(17)-C(15)	114.52(18)
C(19)-C(18)-C(17)	122.8(2)
C(19)-C(18)-H(18)	117.9(17)
C(17)-C(18)-H(18)	119.3(17)
C(18)-C(19)-O(8)	121.22(19)
C(18)-C(19)-C(20)	127.7(2)
O(8)-C(19)-C(20)	111.10(19)
C(19)-C(20)-H(20A)	114(3)
C(19)-C(20)-H(20B)	110(2)
H(20A)-C(20)-H(20B)	119(4)
C(19)-C(20)-H(20C)	109(2)
H(20A)-C(20)-H(20C)	95(3)
H(20B)-C(20)-H(20C)	108(3)

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **10**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ba(1)	24(1)	21(1)	24(1)	4(1)	6(1)	2(1)
Cl(1)	27(1)	25(1)	28(1)	-2(1)	0(1)	7(1)
Cl(2)	33(1)	35(1)	41(1)	6(1)	4(1)	3(1)
O(1)	50(1)	28(1)	23(1)	6(1)	4(1)	4(1)
O(2)	24(1)	54(1)	27(1)	9(1)	4(1)	6(1)
O(3)	36(1)	28(1)	25(1)	6(1)	0(1)	-2(1)
O(4)	31(1)	27(1)	25(1)	7(1)	6(1)	2(1)
O(5)	32(1)	43(1)	40(1)	23(1)	6(1)	9(1)
O(6)	35(1)	28(1)	33(1)	2(1)	-6(1)	7(1)
O(7)	24(1)	30(1)	26(1)	7(1)	4(1)	2(1)
O(8)	25(1)	27(1)	30(1)	10(1)	3(1)	4(1)
O(9)	38(1)	26(1)	39(1)	3(1)	14(1)	11(1)
O(10)	27(1)	45(1)	36(1)	-5(1)	-2(1)	14(1)
O(11)	40(1)	63(1)	33(1)	-16(1)	-8(1)	20(1)
O(12)	52(1)	30(1)	55(1)	10(1)	7(1)	-7(1)
O(13)	59(4)	76(5)	62(6)	-3(4)	-10(4)	0(4)
O(14)	55(2)	36(2)	142(8)	17(3)	38(3)	-6(2)
O(15)	51(3)	54(3)	47(2)	-4(2)	19(2)	19(2)
O(16)	32(4)	59(6)	45(4)	-11(4)	-9(3)	11(5)
O(13A)	60(6)	90(9)	11(3)	-12(4)	-5(3)	51(7)
O(14A)	62(5)	87(8)	77(7)	47(6)	-10(4)	-35(5)
O(15A)	232(17)	146(16)	50(6)	-39(8)	4(8)	139(13)
O(16A)	31(6)	50(7)	30(6)	11(6)	4(4)	6(5)
O(17)	54(1)	30(1)	55(1)	6(1)	12(1)	-9(1)
O(18)	28(1)	37(1)	40(1)	8(1)	6(1)	2(1)
C(1)	62(2)	44(2)	39(2)	9(1)	-9(1)	-22(2)
C(2)	43(1)	25(1)	30(1)	3(1)	5(1)	-6(1)
C(3)	45(1)	23(1)	30(1)	4(1)	6(1)	-2(1)
C(4)	37(1)	22(1)	25(1)	4(1)	9(1)	8(1)
C(5)	26(1)	25(1)	22(1)	4(1)	7(1)	6(1)
C(6)	28(1)	21(1)	24(1)	2(1)	8(1)	4(1)

C(7)	23(1)	34(1)	26(1)	3(1)	8(1)	8(1)
C(8)	24(1)	31(1)	33(1)	1(1)	8(1)	1(1)
C(9)	27(1)	25(1)	31(1)	3(1)	11(1)	3(1)
C(10)	43(1)	29(1)	41(1)	11(1)	13(1)	4(1)
C(11)	30(1)	40(1)	33(1)	5(1)	6(1)	-4(1)
C(12)	28(1)	27(1)	27(1)	3(1)	11(1)	2(1)
C(13)	30(1)	30(1)	30(1)	8(1)	9(1)	4(1)
C(14)	30(1)	31(1)	26(1)	7(1)	9(1)	9(1)
C(15)	26(1)	24(1)	21(1)	3(1)	6(1)	7(1)
C(16)	25(1)	24(1)	24(1)	4(1)	7(1)	5(1)
C(17)	30(1)	22(1)	26(1)	-2(1)	3(1)	6(1)
C(18)	27(1)	25(1)	35(1)	3(1)	2(1)	2(1)
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**.

	x	y	z	U(eq)
H(1A)	-2810(40)	4670(40)	520(30)	72(12)
H(1B)	-2040(40)	4920(40)	-300(30)	66(11)
H(1C)	-2830(40)	6040(40)	-30(30)	77(12)
H(3)	-990(30)	4900(30)	2250(30)	52(9)
H(8)	3680(30)	9980(30)	2090(20)	30(7)
H(10A)	2640(40)	10260(40)	-360(30)	74(13)
H(10B)	1780(40)	11060(40)	10(30)	70(11)
H(10C)	3280(30)	11220(30)	490(30)	50(9)
H(11A)	9900(50)	7200(50)	7800(40)	86(15)
H(11B)	9560(40)	8510(40)	7830(30)	78(13)
H(11C)	9350(50)	7670(50)	8630(40)	92(15)
H(13)	7620(30)	8400(30)	6190(20)	37(7)
H(18)	2900(30)	3360(30)	6750(20)	35(7)
H(20A)	5060(40)	2070(40)	8470(30)	72(12)

H(20B)	3550(40)	2130(30)	8310(30)	55(9)
H(20C)	4760(40)	2970(40)	9210(30)	71(11)
H(1O)	-310(40)	5610(50)	5710(30)	76(13)
H(2O)	-630(40)	6590(40)	5220(30)	56(11)

Table 6. Torsion angles [°] for **10**.

O(17)-Ba(1)-O(1)-C(4)	-26.7(2)
O(2)-Ba(1)-O(1)-C(4)	-45.83(19)
O(5)-Ba(1)-O(1)-C(4)	-103.6(2)
O(6)-Ba(1)-O(1)-C(4)	-158.2(2)
O(18)-Ba(1)-O(1)-C(4)	115.8(2)
O(13A)-Ba(1)-O(1)-C(4)	166.3(4)
O(9)-Ba(1)-O(1)-C(4)	67.6(2)
O(10)#1-Ba(1)-O(1)-C(4)	22.52(19)
O(13)-Ba(1)-O(1)-C(4)	162.3(4)
O(14A)-Ba(1)-O(1)-C(4)	-158.5(6)
O(17)-Ba(1)-O(2)-C(7)	-116.9(2)
O(5)-Ba(1)-O(2)-C(7)	167.3(2)
O(6)-Ba(1)-O(2)-C(7)	112.7(2)
O(1)-Ba(1)-O(2)-C(7)	49.7(2)
O(18)-Ba(1)-O(2)-C(7)	27.5(2)
O(13A)-Ba(1)-O(2)-C(7)	165.8(7)
O(9)-Ba(1)-O(2)-C(7)	-62.9(2)
O(10)#1-Ba(1)-O(2)-C(7)	-32.86(19)
O(13)-Ba(1)-O(2)-C(7)	162.8(6)
O(14A)-Ba(1)-O(2)-C(7)	-155.9(4)
O(17)-Ba(1)-O(5)-C(14)	110.4(2)
O(2)-Ba(1)-O(5)-C(14)	-167.7(2)
O(6)-Ba(1)-O(5)-C(14)	-53.00(19)
O(1)-Ba(1)-O(5)-C(14)	-114.1(2)
O(18)-Ba(1)-O(5)-C(14)	-41.8(2)
O(13A)-Ba(1)-O(5)-C(14)	11.6(4)
O(9)-Ba(1)-O(5)-C(14)	76.2(2)
O(10)#1-Ba(1)-O(5)-C(14)	169.43(18)

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(10)-Cl(1)-O(9)-Ba(1)	122.60(19)
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)
O(10)#1-Ba(1)-O(13A)-Cl(2)	-79.4(12)
O(13)-Ba(1)-O(13A)-Cl(2)	-128(8)
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)	2.8(3)
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)	-174.2(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)	-1.8(3)
C(17)-C(15)-C(16)-O(7)	-177.67(19)
C(14)-C(15)-C(16)-O(8)	176.06(19)
C(17)-C(15)-C(16)-O(8)	0.2(3)
Ba(1)-O(6)-C(17)-C(18)	130.18(18)
Ba(1)-O(6)-C(17)-C(15)	-48.6(3)
C(16)-C(15)-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)	-1.6(4)
C(17)-C(18)-C(19)-C(20)	176.6(2)
C(16)-O(8)-C(19)-C(18)	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 °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(18)-H(1O)...O(1)#2	0.80(5)	2.22(5)	2.996(3)	163(4)
O(18)-H(1O)...O(18)#2	0.80(5)	2.64(4)	3.080(4)	116(4)
O(18)-H(2O)...O(12)#1	0.74(4)	2.41(4)	3.056(3)	146(4)

Symmetry transformations used to generate equivalent atoms:

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