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Cage opening of 1,2-$(CH_2OH)_2$-*closo*-1,2-$C_2B_{10}H_{10}$ with 2-(aminomethyl)-pyridine: synthesis and structure of a cobalt salt of functionalized *nido*-$C_2B_9H_{10}$ mono anion†

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The reaction of 1,2-$(CH_2OH)_2$-*closo*-1,2-C_2B_{10} H_{10} carborane with 2-(aminomethyl)-pyridine in methylene chloride at room temperature led to the partial cage degradation of the *closo* carborane and its conversion to the *nido* derivative. The addition of cobalt chloride hexahydrate in methanol to the mixture produced orange crystals 1. The crystal structure of the orange compound has been determined. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: 2-(aminomethyl)-pyridine; cage degradation; cobalt(III); *nido*-carborane; synthesis; X-ray diffraction analysis

INTRODUCTION

The carborane synthon, [*nido*-7,8-$C_2B_9H_{12}$]$^-$, and its substituted derivatives were first prepared by cage degradation of *closo*-1,2-$C_2B_{10}H_{12}$ and its substituted derivatives by refluxing in methanolic potassium hydroxide as shown in Equation (1):

$$B_{10}C_2H_{12} + CH_3O^- + 2CH_3OH \rightarrow B_9C_2H_{12}^- + B(OCH_3)_3 + H_2 \quad (1)$$

The resulting anion [7,8-$C_2B_9H_{12}$]$^-$ and several of its carbon-substituted derivatives were isolated as heavy metal salts and have been characterized by NMR and X-ray single crystal diffraction studies.[1–8] Also, for many of the primary sources, consult the references and reviews cited in the pertinent volumes of the Boron Compound series of the *Gmelin Handbook of Inorganic Chemistry*[9–21] and Jelinek *et al.*[22] The bridging proton from the open face of [7,8-$C_2B_9H_{12}$]$^-$ can be removed by strong base to give [*nido*-7,8-$C_2B_9H_{11}$]$^{2-}$, which can bind to a metal in η^5 fashion. This discovery led to the development of the field of metallacarboranes.[1–22]

Previous attempts to effect similar cage degradation using weak bases such as amines were unsuccessful, however, Zakharin and Kalnin[23] described the facile degradation of 1,2-dicarba-*closo*-dodecarborane using piperidine. The degraded carborane was proposed to be a zwitterion, $B_9C_2H_{12}^{(-)}C_5H_{10}NH_2^+$. The reaction of the 11-vertex *closo*-2,3-$C_2B_9H_{11}$ carborane and *closo*-1,8-$C_2B_9H_{11}$ with nitrogen-containing bases, such as pyridine or ethylamine, produced the corresponding charge-compensated 3-Me_3N-*nido*-7,9-$C_2B_9H_{11}$ without cage degradation.[24,25] In our previous NMR studies with 1-(2-methylpyridyl)-2-dicarba-*closo*-carborane, we observed that this functionalized carborane undergoes a cage-opening reaction in dimethylsulfoxide at room temperature.[26] This encouraged us to investigate the use of a chelating amine, such as 2-(aminomethyl)-pyridine, to effect a low-temperature cage degradation reaction of functionalized 1,2-$C_2B_{10}H_{10}R_2$ (R = CH_2OH) carboranes. We report here a high-yield room-temperature cage degradation of 1,2-$(CH_2OH)_2$-*closo*1,2-$C_2B_{10}H_{10}$ to produce the [7,8-$(CH_2OH)_2$-*nido*-7,8-$C_2B_9H_{10}$]$^-$ derivatives.

RESULTS AND DISCUSSION

Synthesis and characterization

The cage degradation of 1,2-$(CH_2OH)_2$-*closo*-1,2-$C_2B_{10}H_{10}$ was accomplished at room temperature using 2-(aminomethyl)-

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†Dedicated to Professor Thomas P. Fehlner on the occasion of his 65th birthday, in recognition of his outstanding contributions to organometallic and inorganic chemistry.

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pyridine to produced a yellow solid. Although the exact nature of the yellow product has not been ascertained, preliminary analysis by NMR and FTIR is suggestive of a zwitterion, comprising of the degraded cage carborane anion and a boron atom chelated by the 2-(aminomethyl)-pyridine as the cation. Addition of cobalt(II) chloride led to the displacement of the boron in the chelated cation, and subsequent air oxidation led to the isolation of the orange crystals. The IR spectra of the yellow and the orange compounds exhibited several bands in the region 2500–2600 cm^{-1} assigned to the various B–H stretching vibrations. In addition, the orange crystals showed bands at 1614 and 1600 cm^{-1} and are due to the $\nu_{C=N}$ vibration of the coordinated pyridine. In the yellow solid, the $\nu_{C=N}$ stretches were observed at 1616 and 1604 cm^{-1}. The $\nu_{C=N}$ stretch in the free ligand was observed at 1607 and 1592 cm^{-1}, an indication that the 2-(aminomethyl)-pyridine is coordinated to the cobalt in the orange crystal or to the boron in the yellow solid. The electronic spectra of the orange compound contains a broad band in the region 600–650 nm, notable for symmetric cobalt(III) ion in an octahedral field, and confirming that the cobalt(II) has been oxidized, most likely by oxygen to the corresponding cobalt(III) ion. The orange crystals were further analyzed by single-crystal X-ray diffraction.

Crystal and molecular structure of $[Co(III)(C_6H_8N_2)_3][(C_2B_9H_{10}(CH_2OH)_2)^-]_2\,[Cl]^-\,[C_2B_{10}H_{10}(CH_2OH)_2]\cdot MeOH$ (1)

The crystal structure reveals that **1** is a cluster consisting of an octahedral $[Co(C_6H_8N_2)_3]^{3+}$ cation, a $[Cl^-]$, two $[7,8\text{-}(CH_2OH)_2\text{-}\textit{nido}\text{-}7,8\text{-}C_2B_9H_{10}]^-$, a $1,2\text{-}(CH_2OH)_2\text{-}\textit{closo}\text{-}1,2\text{-}C_2B_{10}H_{10}$ and a methanol solvate. The ORTEP plot of the interaction sphere about the two inversion-related Cl^- ions is shown in Fig. 1. The thermal ellipsoids are drawn at the 50% probability level. The hydrogen atoms and solvate molecule have been omitted for clarity.

The geometry around the cobalt is octahedral. The six nitrogen atoms from the three 2-(aminomethyl)-pyridine are the ligating atoms with Co–N distances ranging from 1.945(2) to 1.964(2) Å. These distances are comparable to Co(III)–N distances observed in related complexes,[27,28] and, as expected, they are significantly shorter than the distances of 1.989(14) to 2.081(11) Å observed in Co(II)–N complexes. Figures 2 and 3 show an ORTEP view of the two *nido* carborane anions $[7,8\text{-}(CH_2OH)_2\text{-}\textit{nido}\text{-}7,8\text{-}C_2B_9H_{10}]^-$.

Both carborane anions show some similarities, but they also exhibit significant differences in their structures. The expected *nido*-icosahedral fragment geometry with an *exo*-polyhedral hydrogen atom attached to each cage boron atom and a localized *endo*-hydrogen atom, which penetrates the open pentagonal face of the *nido*-carborane cage and is directed towards the empty 12th vertex, were exhibited by both ions. However, this *endo*-polyhedral hydrogen atom located near the capping site is found to be bridging two boron atoms in one anion, with a B–H–B bridge between B12 and B13 and a B12–B13 distance of 1.828(5) Å, which is slightly longer than the B11–B12 distance of 1.815(5) Å. In the other anion, the *endo*-polyhedral hydrogen atom is bridged to three boron atoms (B21, B22 and B23) in the open C_2B_3 pentagonal face.

The ORTEP view of the $[Co(C_6H_8N_2)_3]^{3+}$ cation is given in Fig. 4.

All six $-NH_2$ protons are involved in each of the two types of hydrogen bond, namely with oxygen atoms of the ROH groups and with the Cl^- ion. Also, all six of the carborane ROH

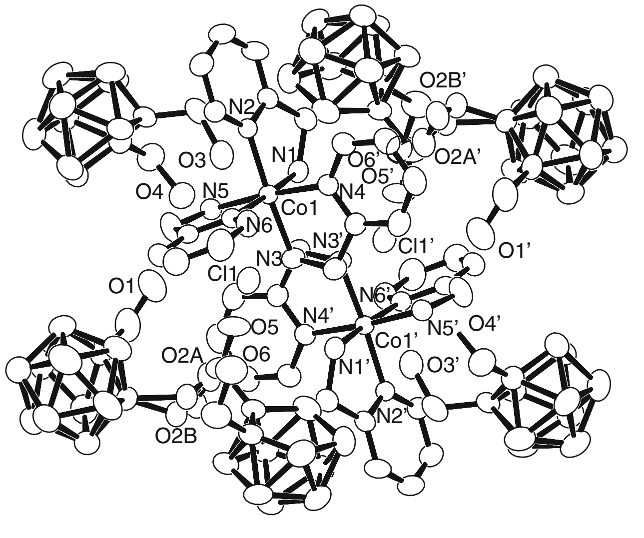

Figure 1. ORTEP view of $[Co(III)(C_6H_8N_2)_3][(C_2B_9H_{10}(CH_2OH)_2)^-]_2[Cl]^-[C_2B_{10}H_{10}(CH_2OH)_2]\cdot MeOH$ (**1**). H atoms omitted for clarity.

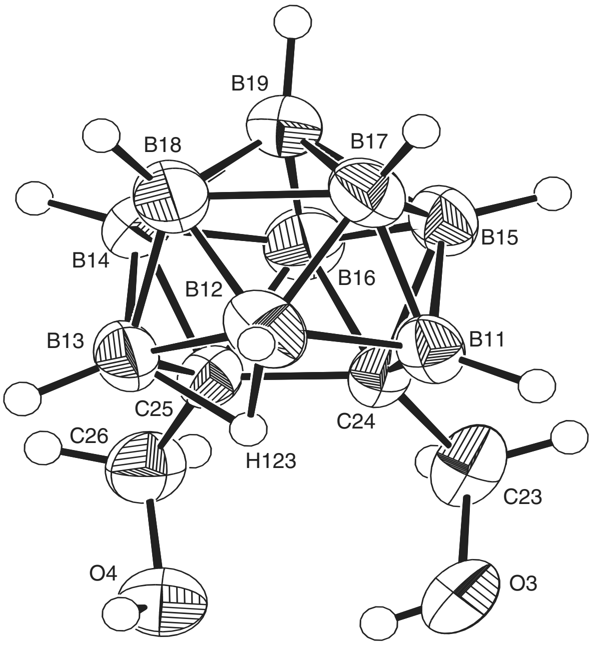

Figure 2. ORTEP diagram of the molecular structure of the anion $[7,8\text{-}(CH_2OH)_2\text{-}\textit{nido}\text{-}7,8\text{-}C_2B_9H_{10}]^-$.

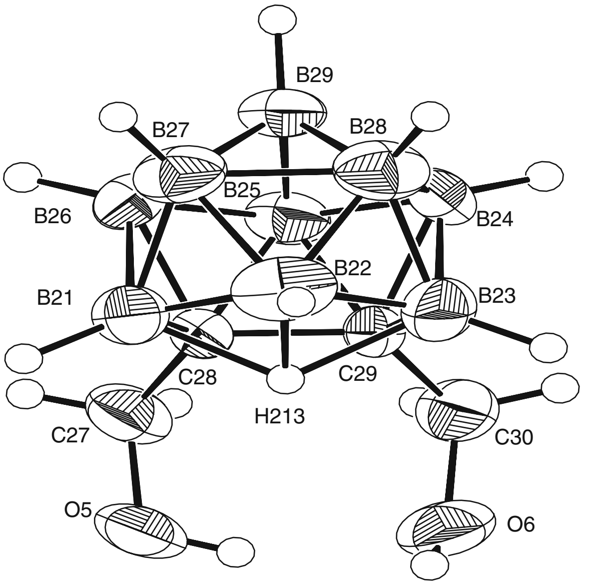

Figure 3. ORTEP diagram of the molecular structure of the other carborane anion $[7,8\text{-}(CH_2OH)_2\text{-}nido\text{-}7,8\text{-}C_2B_9H_{10}]^-$.

protons are involved in three types of hydrogen bond, namely with the Cl^- ion, with the hydrido hydrogen atom and with the oxygen atoms of the ROH groups (Table 1). It is of interest to note that the hydrido H213, which bridges three boron atoms, is engaged in only one hydrogen bond interaction, whereas the hydrido H123, which bridges two boron atoms, is engaged in two hydrogen bond interactions. The methanol solvate apparently does not engage in noteworthy hydrogen bonding.

EXPERIMENTAL

Synthesis of $Co(III)(C_6H_8N_2)_3Cl[C_2B_9H_{10}(CH_2OH)_2]_2[C_2B_{10}H_{10}(CH_2OH)_2]\cdot MeOH$ (1)

2 ml of 2-(aminomethyl)-pyridine ($d = 1.049\ g\ ml^{-1}$, 0.0194 mol) were added to 3.94 g (0.0194 mol) of $1,2\text{-}(CH_2OH)_2\text{-}closo\text{-}1,2\text{-}C_2B_{10}H_{10}$ carborane in 50 ml of methylene chloride and the mixture was stirred in air for 72 h. Subsequently, the solvent was removed by slow evaporation in air to give a yellow solid. To the yellow solid, a solution of 2.3 g (0.0097 mol) of cobalt(II) chloride hexahydrate dissolved in 50 ml of methanol was added and stirred for 2 h. Subsequent filtration and slow evaporation of the solvents deposited orange crystals, identified as $Co(III)(C_6H_8N_2)_3Cl[C_2B_9H_{10}(CH_2OH)_2]_2[C_2B_{10}H_{10}(CH_2OH)_2]\cdot MeOH$ (**1**), 4.1 g, (61.7% yield), M.P.: decomposed at 250 °C. Elemental analysis (Scwarzkopf, NY). Found: C, 36.6; H, 6.93; N, 7.70. Calc: C, 35.67; H, 7.2; N, 8.01.

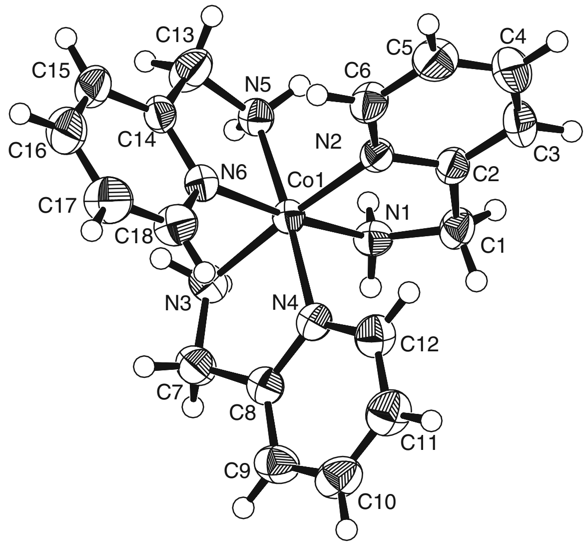

Figure 4. ORTEP diagram of the molecular structure of the $[Co(C_6H_8N_2)_3]^{3+}$ in **1**. The thermal ellipsoids are drawn at the 50% probability level. The hydrogen atoms are open circles of arbitrary diameter.

IR (KBr): $\nu_{C=N}$ 1600 (m), 1614 (m) ν_{BH} 2581(s), 2595 (vs), 2603 cm^{-1} (vs).

Electronic spectra: methanol, 630 nm, broad symmetric cobalt(III) ion in an octahedral field.

Structure determination of $Co(III)(C_6H_8N_2)_3Cl[C_2B_9H_{10}(CH_2OH)_2)]_2[C_2B_{10}H_{10}(CH_2OH)_2]\cdot MeOH$ (1)

An orange crystal of **1** was glued to the end of a glass fiber with epoxy and placed on a Rigaku X-ray diffractometer with graphite monochromatized Mo Kα (0.710 73 Å). The pertinent crystallographic data are summarized in Table 2.

The data were collected at 293(2) K. Of the 9173 reflections collected $3.05 < 2\theta < 62.95°$, 8772 reflections were considered

Table 1. Hydrogen bonding interactions in the crystal structure of **1**

H bond	Distance (Å)	H bond	Distance (Å)	H bond	Distance (Å)
H123 ··· H4O	2.96	H123 ··· H1O	3.06	H213 ··· H6O	3.06
Cl1 ··· H5N2	2.32	Cl1 ··· H1N2′	2.40	Cl1 ··· H3N2	2.57
Cl1 ··· H2OA	2.76	Cl1 ··· H1O	2.86	Cl1 ··· H3N2′	2.87
O6 ··· H5O	1.93	O5 ··· H3N1	1.95	O1 ··· H4O	2.03
O4 ··· H3O	2.08	O3 ··· H1N1	2.09	O3 ··· H5N1	2.16

Table 2. Crystal data and structure refinement for (**1**) $[Co(III)(C_6H_8N_2)_3][(C_2B_9H_{10}(CH_2OH)_2)^-]_2[Cl]^-$ $[C_2B_{10}H_{10}(CH_2OH)_2]\cdot MeOH$

Empirical formula	$C_{30.5}H_{74}B_{28}ClCoN_6O_{6.5}$
Formula weight	1026.02
Temperature (K), wavelength (Å)	293(2), 1.5418
Crystal color, habit	Orange, hexagonal block
Crystal system, space group	Triclinic, $P\overline{1}$
No., θ range (deg) for cell data	25, $30.1 < \theta < 37.5$
Unit cell dimensions	
a (Å)	13.664(1)
b (Å)	14.221(1)
c (Å)	15.533(1)
α (deg)	101.63(1)
β (deg)	104.10(1)
γ (deg)	104.06(1)
Volume $(Å)^3$	2724.3(4)
Z, calculated density (Mg m^{-3})	2, 1.251
Absorption coefficient (mm^{-1}), $F(000)$	3.276, 1070
Crystal size (mm^3)	$0.50 \times 0.40 \times 0.25$
θ range (deg) for data collection	3.05 to 62.95
Limiting indices	$0 \le h \le 15, -16 \le k \le 15, -17 \le l \le 17$
Reflections collected/unique	9173/8772 ($R_{int} = 0.0199$)
Completeness to $\theta = 62.95°$ (%)	99.7
Max. and min. transmission	0.4947 and 0.2912
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	8772/827/867
Goodness-of-fit on F^2	1.030
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.046, wR_2 = 0.120$
R indices (all data)	$R_1 = 0.063, wR_2 = 0.126$
Extinction coefficient	0.002 00(18)
Largest diff. peak and hole (e$^-$ Å^{-3})	0.433 and −0.333

unique ($R_{int} = 0.0199$). Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods and full matrix least squares refinements were performed using SHELXTL-PLUS.[29] The positional parameters for the hydrogen atoms bonded to the boron, nitrogen and oxygen atoms were refined and those for the hydrogen atoms bonded to carbon atoms were fixed at their calculated geometry values (sp^3 or sp^2). All non-hydrogen atoms were refined anisotropically. The final refinement converged at $R = 0.046$, $wR_2 = 0.120$ and GOF = 1.030.

For the non-hydrogen atoms, the coordinates and equivalent isotropic displacement parameters are given in Table 3.

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (10^3 Å^2) for $[Co(III)(C_6H_8N_2)_3][(C_2B_9H_{10}(CH_2OH)_2)^-]_2[Cl]^-[C_2B_{10}H_{10}(CH_2OH)_2]\cdot MeOH$ (**1**)

	x	y	z	U_{eq}[a]
Co(1)	1594(1)	−3103(1)	2195(1)	29(1)
N(1)	590(2)	−2995(2)	1118(1)	36(1)
N(2)	1422(2)	−1837(1)	2800(1)	32(1)
C(1)	591(2)	−1935(2)	1217(2)	42(1)
C(2)	877(2)	−1416(2)	2218(2)	36(1)
C(3)	651(2)	−547(2)	2549(2)	49(1)
C(4)	987(3)	−88(2)	3477(2)	58(1)
C(5)	1551(3)	−504(2)	4069(2)	57(1)
C(6)	1751(2)	−1380(2)	3712(2)	45(1)
N(3)	1713(2)	−4345(2)	1488(2)	38(1)
N(4)	2789(2)	−2452(1)	1805(1)	34(1)
C(7)	2701(2)	−4181(2)	1228(2)	46(1)
C(8)	3175(2)	−3093(2)	1323(2)	36(1)
C(9)	3993(2)	−2753(2)	976(2)	48(1)
C(10)	4446(2)	−1731(2)	1144(2)	54(1)
C(11)	4055(2)	−1079(2)	1631(2)	54(1)
C(12)	3237(2)	−1451(2)	1956(2)	45(1)
N(5)	419(2)	−3856(2)	2533(1)	36(1)
N(6)	2471(2)	−3274(1)	3321(1)	33(1)
C(13)	758(2)	−4112(3)	3403(2)	55(1)
C(14)	1934(2)	−3807(2)	3776(2)	35(1)
C(15)	2440(2)	−4062(2)	4533(2)	48(1)
C(16)	3519(2)	−3784(2)	4830(2)	58(1)
C(17)	4079(2)	−3231(3)	4385(2)	60(1)
C(18)	3539(2)	−2984(2)	3635(2)	47(1)
B(1)	−1359(3)	−7367(3)	2624(2)	56(1)
B(2)	−2291(4)	−7284(3)	3236(2)	70(1)
B(3)	−1096(4)	−7513(3)	3747(3)	78(1)
B(4)	−827(3)	−8329(3)	2872(3)	65(1)
B(5)	−1866(3)	−9472(3)	2473(3)	63(1)
B(6)	−3041(3)	−9230(3)	1977(3)	63(1)
B(7)	−3321(3)	−8441(3)	2836(3)	67(1)
B(8)	−2304(4)	−8182(3)	3874(3)	75(1)
B(9)	−1403(3)	−8822(3)	3648(3)	66(1)
B(10)	−2769(3)	−9384(3)	3090(3)	66(1)
C(19)	−3314(3)	−7604(3)	1356(3)	74(1)
C(20)	−2692(2)	−7946(2)	2126(2)	49(1)
C(21)	−1858(2)	−8564(2)	1910(2)	47(1)
O(1)	−2820(3)	−6618(3)	1362(2)	91(1)
C(22A)	−1749(5)	−8834(3)	940(3)	66(2)
O(2A)	−1662(3)	−8026(2)	536(2)	71(1)
C(22B)	−1499(14)	−8447(17)	1043(9)	66(2)
O(2B)	−2389(10)	−8825(8)	232(7)	71(1)
B(11)	−1420(3)	−2933(2)	3559(2)	47(1)
B(12)	−2024(3)	−3953(3)	3986(2)	59(1)
B(13)	−3293(3)	−4559(3)	3073(2)	53(1)
B(14)	−4055(3)	−3698(3)	3074(3)	63(1)
B(15)	−2159(3)	−2068(3)	3568(3)	58(1)
B(16)	−3391(3)	−2687(3)	2735(3)	61(1)

Table 3. (*Continued*)

	x	y	z	U_{eq}[a]
B(17)	−2016(3)	−2695(3)	4426(3)	62(1)
B(18)	−3224(4)	−3739(3)	4115(3)	72(1)
B(19)	−3275(3)	−2555(3)	3901(3)	70(1)
C(23)	−1897(2)	−2725(2)	1843(2)	55(1)
O(3)	−1417(2)	−3429(2)	1470(2)	56(1)
C(24)	−2283(2)	−3001(2)	2615(2)	41(1)
C(25)	−3342(2)	−3898(2)	2340(2)	44(1)
C(26)	−3923(2)	−4417(2)	1326(2)	56(1)
O(4)	−3345(2)	−4973(2)	891(1)	58(1)
B(21)	2198(3)	−7210(3)	816(2)	57(1)
B(22)	3440(3)	−7404(3)	698(3)	67(1)
B(23)	4084(3)	−7430(3)	1849(3)	59(1)
B(24)	3355(3)	−8539(3)	2057(3)	55(1)
B(25)	2147(3)	−8369(2)	2102(2)	54(1)
B(26)	1452(3)	−8321(3)	1012(2)	54(1)
B(27)	2228(3)	−8410(3)	289(2)	62(1)
B(28)	3458(3)	−8556(3)	958(3)	64(1)
B(29)	2222(3)	−9144(2)	1099(2)	54(1)
C(27)	1667(3)	−6613(2)	2360(2)	62(1)
O(5)	2216(2)	−5565(2)	2621(2)	70(1)
C(28)	2203(2)	−7244(2)	1850(2)	41(1)
C(29)	3266(2)	−7357(2)	2424(2)	41(1)
C(30)	3699(3)	−6822(2)	3446(2)	65(1)
O(6)	4119(2)	−5748(2)	3610(2)	75(1)
Cl(1)	−773(1)	−5729(1)	756(1)	52(1)
O(7)	4911(14)	52(14)	5560(12)	269(8)
C(31)	4690(20)	200(20)	4603(12)	269(8)

[a] U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Crystallographic data for the structure determination have been deposited with the Cambridge Crystallographic Data Centre as CCDC no. 198534. Copies may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (www.ccdc.cam.ac.uk).

FTIR and electronic measurements

FTIR spectra were recorded on a Nicolet Magna IR 760 spectrometer in KBr pellets. Visible spectra were recorded on Perkin Elmer Lambda II in methanol.

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