Cobalt-Mediated [3+2]-Annulation Reaction of Alkenes with α,β-Unsaturated Ketones and Imines

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Supplementary Information

X-ray Structure Report
for Compound 3

Discussion

The compound crystallizes with one molecule in the asymmetric unit.The molecule has approximate, non-crystallographic, miror symmetry. Intra-molecular distances and angles are in normal ranges. There is an inter-molecular hydrogen bond from the hydroxyl (O3) to one of the nitrosyl oxygens on a neighboring molecule related by an inversion center at 1/2,0,1/2. Figures and the usual tables are provided.

Experimental

Data Collection

A fragment of a black rhomboidal crystal of $CoO_4N_2C_{19}H_{27}$ having approximate dimensions of 0.17 x 0.22 x 0.24 mm was mounted on a Kapton loop using Paratone N hydrocarbon oil. All measurements were made on a SMART[1] CCD area detectorwith graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix, obtained from a least-squares refinement using the measured positions of 3574 centered reflections with $I > 10\sigma$ in the range $7.00 < 2\theta < 51.00^o$ corresponded to a primitive monoclinic cell with dimensions:

a = 9.428(1) Å
b = 17.121(2) Å $\beta = 112.149(2)^o$
c = 12.367(2) Å
V = 1849.0(4) Å^3

For Z = 4 and F.W. = 406.37, the calculated density is 1.46 g/cm^3. The systematic absences of:

h0l: $l \neq 2n$
0k0: $k \neq 2n$

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -125 $\pm$ 1^{o}C. Frames corresponding to an arbitrary hemisphereof data were collected using ω scans of 0.3^{o}counted for a total of 10.0 seconds per frame.

Data Reduction

Data were integrated by the program SAINT[2] to a maximum 2θ value of 50.8°.The data were corrected for Lorentz and polarization effects. Data were analyzed for agreement and possible absorptionusing XPREP[3]. An empirical absorption correction based on comparison of redundant and equivalent reflections was applied using SADABS[4]. (Tmax = 1.00, Tmin = 0.91).

Structure Solution and Refinement

The structure was solved by direct methods[5] and expanded using Fourier techniques[6]. Non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen atoms were included in calculated positions but not refined. The hydroxyl hydrogen was included as found in a difference Fourier map.The final cycle of full-matrix least-squares refinement[7] was based on 2638 observed reflections ($I > 3.00\sigma(I)$) and 235 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.033$$

$$R_w = [(\Sigma \, w \, (|Fo| - |Fc|)^2 \, / \, \Sigma \, w \, Fo^2)]^{1/2} = 0.041$$

The standard deviation of an observation of unit weight[8] was 1.92. The weighting scheme was based on counting statistics and included a factor (p = 0.030) to downweight the intense reflections. Plots of $\Sigma \, w \, (|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.45 and -0.20 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[9]. Anomalous dispersion effects were included in Fcalc[10]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[11]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[12]. All calculations were performed using the teXsan[13] crystallographic software package of Molecular Structure Corporation.

References

(1) SMART: Area-Detector Software Package,
Bruker Analytical X-ray Systems, Inc.: Madison, WI, (2001-03)

(2) SAINT: SAX Area-Dectector Integration Program, V7.06;
Bruker Analytical X-ray Systems Inc.: Madison, WI, (2005)

(3) XPREP:(v 6.12) Part of the SHELXTL Crystal Structure Determination Package,
Bruker Analytical X-ray Systems, Inc.: Madison, WI, (2001)

(4) SADABS: Bruker-Nonius Area Detector Scaling and Absorption v. 2.10
Bruker Analytical X-ray Systems, Inc.: Madison, WI (2005).

(5) SIR97: Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G., Spagna, R. SIR97: A new tool for crystal structure determination and refinement. J. App. Cryst. (1998)

(6) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(7) Least-Squares:

Function minimized: $\Sigma w(|F_o|-|F_c|)^2$

(8) Standard deviation of an observation of unit weight:

$[\Sigma w(|F_o|-|F_c|)^2/(N_o-N_v)]^{1/2}$
where: N_o = number of observations
N_v = number of variables

(9) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(10) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(11) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(12) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(13) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$CoO_4N_2C_{19}H_{27}$
Formula Weight	406.37
Crystal Color, Habit	black, rhomboidal
Crystal Dimensions	0.17 X 0.22 X 0.24 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 9.428(1)Å b = 17.121(2) Å c = 12.367(2) Å β = 112.149(2)^o V = 1849.0(4) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.460 g/cm^3
F_{000}	856.00
μ(MoKα)	9.55 cm^{-1}

B. Intensity Measurements

Diffractometer	Bruker SMART CCD
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Detector Position	60.00 mm
Exposure Time	10.0 seconds per frame.
Scan Type	ω (0.3 degrees per frame)
$2\theta_{max}$	50.8^o
No. of Reflections Measured	Total: 8556 Unique: 3575 (R_{int} = 0.019)
Corrections	Lorentz-polarization Absorption (Tmax = 1.00 Tmin = 0.91)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares
Function Minimized	Σ w (|Fo| - |Fc|)^2
Least Squares Weights	$1/\sigma^2(Fo) = 4Fo^2/\sigma^2(Fo^2)$
p-factor	0.0300
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	2638
No. Variables	235
Reflection/Parameter Ratio	11.23
Residuals: R; Rw; Rall	0.033 ; 0.041; 0.046
Goodness of Fit Indicator	1.92
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.45 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.20 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
Co1	0.12508(4)	0.08662(2)	0.18621(3)	1.466(8)	
O1	0.3377(2)	0.0157(1)	0.3798(2)	3.66(5)	
O2	0.1768(2)	0.1807(1)	0.0230(2)	3.41(5)	
O3	0.6835(2)	0.0747(1)	0.4364(2)	3.83(5)	
O4	0.4304(2)	-0.0035(1)	0.1010(2)	3.37(5)	
N1	0.3051(2)	0.0528(1)	0.2855(2)	2.02(5)	
N2	0.2254(2)	0.1351(1)	0.1093(2)	1.85(5)	
C1	0.4442(3)	0.0733(1)	0.2623(2)	1.84(6)	
C2	0.3956(3)	0.1247(2)	0.1532(2)	1.79(5)	
C3	0.4899(3)	0.2004(2)	0.1883(2)	2.42(6)	
C4	0.4199(4)	0.2632(2)	0.2406(3)	3.48(7)	
C5	0.3853(4)	0.2345(2)	0.3442(3)	3.69(8)	
C6	0.5041(4)	0.1776(2)	0.4239(3)	3.54(8)	
C7	0.5682(3)	0.1217(2)	0.3584(2)	2.44(6)	
C8	0.6348(3)	0.1685(2)	0.2849(2)	2.49(6)	
C9	0.5045(3)	-0.0008(2)	0.2239(3)	3.10(7)	
C10	0.4346(3)	0.0749(2)	0.0656(3)	2.98(7)	
C11	-0.0331(3)	0.0249(2)	0.2361(2)	1.98(6)	
C12	-0.0131(3)	0.1022(2)	0.2829(2)	1.93(6)	
C13	-0.0579(3)	0.1555(1)	0.1871(2)	1.87(6)	
C14	-0.1031(3)	0.1109(1)	0.0818(2)	1.83(5)	
C15	-0.0878(3)	0.0311(2)	0.1128(2)	1.96(6)	
C16	-0.0059(4)	-0.0493(2)	0.3040(3)	3.32(7)	
C17	0.0451(3)	0.1218(2)	0.4100(3)	3.05(7)	
C18	-0.0554(3)	0.2426(2)	0.1937(3)	2.98(7)	
C19	-0.1630(3)	0.1419(2)	-0.0394(3)	2.82(7)	
C101	-0.0590	0.0849	0.1801	0.3000	0.000
H1	0.5125	0.2206	0.1250	2.8996	
H2	0.4898	0.3056	0.2654	4.1730	
H3	0.3272	0.2807	0.1819	4.1730	
H4	0.3803	0.2786	0.3893	4.4288	
H5	0.2887	0.2090	0.3153	4.4288	
H6	0.5864	0.2070	0.4770	4.2460	
H7	0.4572	0.1478	0.4661	4.2460	
H8	0.6919	0.1362	0.2536	2.9868	
H9	0.6982	0.2096	0.3287	2.9868	
H10	0.4799	-0.0458	0.2583	3.7238	
H11	0.6124	0.0022	0.2456	3.7238	
H12	0.5337	0.0876	0.0677	3.5816	

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy (continued)

atom	x	y	z	B_{eq}	occ
H13	0.3613	0.0829	-0.0112	3.5816	
H14	-0.0119	-0.0921	0.2536	3.9810	
H15	-0.0814	-0.0550	0.3371	3.9810	
H16	0.0928	-0.0480	0.3645	3.9810	
H17	0.1344	0.0920	0.4505	3.6570	
H18	0.0693	0.1759	0.4203	3.6570	
H19	-0.0315	0.1101	0.4402	3.6570	
H20	0.0024	0.2586	0.2715	3.5802	
H21	-0.0098	0.2631	0.1430	3.5802	
H22	-0.1572	0.2617	0.1707	3.5802	
H23	-0.2632	0.1622	-0.0575	3.3876	
H24	-0.0976	0.1823	-0.0460	3.3876	
H25	-0.1666	0.1010	-0.0924	3.3876	
H26	-0.1104	-0.0114	0.0597	2.3533	
H27	0.6641	0.0394	0.4934	2.6593	

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co1	0.0175(2)	0.0193(2)	0.0186(2)	0.0003(1)	0.0064(1)	0.0013(2)
O1	0.026(1)	0.070(2)	0.039(1)	0.003(1)	0.0078(10)	0.035(1)
O2	0.032(1)	0.056(1)	0.037(1)	-0.005(1)	0.0077(10)	0.027(1)
O3	0.028(1)	0.069(2)	0.039(1)	-0.002(1)	0.0025(10)	0.030(1)
O4	0.044(1)	0.037(1)	0.054(1)	-0.004(1)	0.028(1)	-0.020(1)
N1	0.021(1)	0.031(1)	0.025(1)	-0.0006(10)	0.0087(10)	0.007(1)
N2	0.024(1)	0.023(1)	0.021(1)	-0.0031(9)	0.0063(10)	0.0006(10)
C1	0.018(1)	0.026(2)	0.028(1)	0.002(1)	0.010(1)	0.000(1)
C2	0.021(1)	0.024(1)	0.024(1)	-0.004(1)	0.010(1)	-0.002(1)
C3	0.025(2)	0.029(2)	0.035(2)	-0.006(1)	0.009(1)	0.002(1)
C4	0.034(2)	0.024(2)	0.064(2)	-0.006(1)	0.008(2)	-0.010(2)
C5	0.033(2)	0.045(2)	0.058(2)	-0.004(1)	0.013(2)	-0.034(2)
C6	0.034(2)	0.069(2)	0.031(2)	-0.017(2)	0.011(1)	-0.020(2)
C7	0.019(1)	0.045(2)	0.025(1)	-0.001(1)	0.003(1)	0.001(1)
C8	0.024(2)	0.033(2)	0.036(2)	-0.007(1)	0.010(1)	-0.006(1)
C9	0.028(2)	0.029(2)	0.065(2)	0.005(1)	0.022(2)	0.003(2)
C10	0.036(2)	0.051(2)	0.034(2)	-0.008(1)	0.023(1)	-0.010(1)
C11	0.018(1)	0.029(2)	0.029(1)	-0.001(1)	0.010(1)	0.008(1)
C12	0.018(1)	0.034(2)	0.024(1)	0.001(1)	0.011(1)	0.001(1)
C13	0.016(1)	0.025(1)	0.032(2)	0.002(1)	0.011(1)	0.003(1)
C14	0.015(1)	0.026(1)	0.027(1)	0.001(1)	0.007(1)	0.004(1)
C15	0.016(1)	0.027(1)	0.027(1)	-0.003(1)	0.003(1)	-0.004(1)
C16	0.035(2)	0.040(2)	0.048(2)	-0.003(1)	0.013(2)	0.019(2)
C17	0.029(2)	0.061(2)	0.030(2)	-0.001(1)	0.015(1)	-0.003(1)
C18	0.032(2)	0.027(2)	0.056(2)	0.004(1)	0.018(2)	-0.003(1)
C19	0.028(2)	0.041(2)	0.031(2)	0.005(1)	0.003(1)	0.011(1)

The general temperature factor expression:
$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
CO1	N1	1.776(2)	CO1	N2	1.779(2)
CO1	C11	2.101(3)	CO1	C12	2.091(3)
CO1	C13	2.093(3)	CO1	C14	2.091(3)
CO1	C15	2.093(3)	CO1	C101	1.7087(4)
O1	N1	1.260(3)	O2	N2	1.261(3)
O3	C7	1.403(3)	O4	C9	1.413(4)
O4	C10	1.418(4)	N1	C1	1.486(3)
N2	C2	1.498(3)	C1	C2	1.530(4)
C1	C7	1.555(4)	C1	C9	1.536(4)
C2	C3	1.539(4)	C2	C10	1.528(4)
C3	C4	1.528(4)	C3	C8	1.537(4)
C4	C5	1.518(5)	C5	C6	1.532(5)
C6	C7	1.519(4)	C7	C8	1.514(4)
C11	C12	1.428(4)	C11	C15	1.417(4)
C11	C16	1.490(4)	C12	C13	1.428(4)
C12	C17	1.495(4)	C13	C14	1.429(4)
C13	C18	1.494(4)	C14	C15	1.411(4)
C14	C19	1.487(4)			

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O3	H27	1.00	C3	H1	0.95
C4	H2	0.95	C4	H3	0.95
C5	H4	0.95	C5	H5	0.95
C6	H6	0.95	C6	H7	0.95
C8	H8	0.95	C8	H9	0.95
C9	H10	0.95	C9	H11	0.95
C10	H12	0.95	C10	H13	0.95
C15	H26	0.95	C16	H14	0.95
C16	H15	0.95	C16	H16	0.95
C17	H17	0.95	C17	H18	0.95
C17	H19	0.95	C18	H20	0.95
C18	H21	0.95	C18	H22	0.95
C19	H23	0.95	C19	H24	0.95
C19	H25	0.95			

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N1	CO1	N2	87.68(10)	N1	CO1	C101	135.66(7)
N2	CO1	C101	136.06(7)	C9	O4	C10	104.0(2)
CO1	N1	O1	129.8(2)	CO1	N1	C1	118.2(2)
O1	N1	C1	111.9(2)	CO1	N2	O2	130.1(2)
CO1	N2	C2	117.6(2)	O2	N2	C2	112.2(2)
N1	C1	C2	108.3(2)	N1	C1	C7	116.3(2)
N1	C1	C9	108.8(2)	C2	C1	C7	104.8(2)
C2	C1	C9	103.0(2)	C7	C1	C9	114.6(2)
N2	C2	C1	108.2(2)	N2	C2	C3	115.4(2)
N2	C2	C10	108.7(2)	C1	C2	C3	106.7(2)
C1	C2	C10	103.5(2)	C3	C2	C10	113.6(2)
C2	C3	C4	114.6(2)	C2	C3	C8	99.8(2)
C4	C3	C8	108.7(2)	C3	C4	C5	113.2(3)
C4	C5	C6	114.3(3)	C5	C6	C7	113.5(2)
O3	C7	C1	112.6(2)	O3	C7	C6	110.8(2)
O3	C7	C8	109.1(2)	C1	C7	C6	114.0(2)
C1	C7	C8	100.7(2)	C6	C7	C8	109.0(2)
C3	C8	C7	101.9(2)	O4	C9	C1	105.6(2)
O4	C10	C2	105.4(2)	C12	C11	C15	107.8(2)
C12	C11	C16	126.4(2)	C15	C11	C16	125.9(3)
C11	C12	C13	107.7(2)	C11	C12	C17	125.1(2)
C13	C12	C17	127.3(3)	C12	C13	C14	107.9(2)
C12	C13	C18	126.8(2)	C14	C13	C18	125.3(2)
C13	C14	C15	107.8(2)	C13	C14	C19	126.7(2)
C15	C14	C19	125.5(2)	C11	C15	C14	108.9(2)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C7	O3	H27	122.2	C2	C3	H1	111.1
C4	C3	H1	111.1	C8	C3	H1	111.1
C3	C4	H2	108.5	C3	C4	H3	108.5
C5	C4	H2	108.5	C5	C4	H3	108.5
H2	C4	H3	109.5	C4	C5	H4	108.2
C4	C5	H5	108.3	C6	C5	H4	108.3
C6	C5	H5	108.3	H4	C5	H5	109.5
C5	C6	H6	108.5	C5	C6	H7	108.5
C7	C6	H6	108.5	C7	C6	H7	108.5
H6	C6	H7	109.5	C3	C8	H8	111.3
C3	C8	H9	111.3	C7	C8	H8	111.3
C7	C8	H9	111.3	H8	C8	H9	109.5
O4	C9	H10	110.4	O4	C9	H11	110.4
C1	C9	H10	110.4	C1	C9	H11	110.4
H10	C9	H11	109.5	O4	C10	H12	110.5
O4	C10	H13	110.5	C2	C10	H12	110.5
C2	C10	H13	110.5	H12	C10	H13	109.5
CO1	C15	H26	125.2	C11	C15	H26	125.6
C14	C15	H26	125.6	C101	C15	H26	179.9
C11	C16	H14	109.5	C11	C16	H15	109.5
C11	C16	H16	109.5	H14	C16	H15	109.5
H14	C16	H16	109.5	H15	C16	H16	109.5
C12	C17	H17	109.5	C12	C17	H18	109.5
C12	C17	H19	109.5	H17	C17	H18	109.5
H17	C17	H19	109.5	H18	C17	H19	109.5
C13	C18	H20	109.5	C13	C18	H21	109.5
C13	C18	H22	109.5	H20	C18	H21	109.5
H20	C18	H22	109.5	H21	C18	H22	109.5
C14	C19	H23	109.5	C14	C19	H24	109.5
C14	C19	H25	109.5	H23	C19	H24	109.5
H23	C19	H25	109.5	H24	C19	H25	109.5

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
CO1	N1	C1	C2	2.2(3)	CO1	N1	C1	C7	119.8(2)
CO1	N1	C1	C9	-109.0(2)	CO1	N2	C2	C1	-1.5(3)
CO1	N2	C2	C3	-120.9(2)	CO1	N2	C2	C10	110.2(2)
O1	N1	CO1	N2	173.5(3)	O1	N1	CO1	C101	1.7(3)
O1	N1	C1	C2	-174.6(2)	O1	N1	C1	C7	-56.9(3)
O1	N1	C1	C9	74.2(3)	O2	N2	CO1	N1	-174.9(2)
O2	N2	CO1	C101	-3.2(3)	O2	N2	C2	C1	176.1(2)
O2	N2	C2	C3	56.8(3)	O2	N2	C2	C10	-72.2(3)
O3	C7	C1	N1	95.1(3)	O3	C7	C1	C2	-145.4(2)
O3	C7	C1	C9	-33.3(3)	O3	C7	C6	C5	176.0(2)
O3	C7	C8	C3	167.3(2)	O4	C9	C1	N1	89.9(2)
O4	C9	C1	C2	-24.8(3)	O4	C9	C1	C7	-138.1(2)
O4	C10	C2	N2	-89.3(3)	O4	C10	C2	C1	25.6(3)
O4	C10	C2	C3	140.8(2)	N1	CO1	N2	C2	2.3(2)
N1	CO1	C101	C11	-29.1(2)	N1	CO1	C101	C12	43.1(2)
N1	CO1	C101	C13	115.1(2)	N1	CO1	C101	C14	-172.6(2)
N1	CO1	C101	C15	-101.0(2)	N1	C1	C2	N2	-0.4(3)
N1	C1	C2	C3	124.3(2)	N1	C1	C2	C10	-115.6(2)
N1	C1	C7	C6	-32.3(3)	N1	C1	C7	C8	-148.9(2)
N2	CO1	N1	C1	-2.6(2)	N2	CO1	C101	C11	162.8(2)
N2	CO1	C101	C12	-125.0(2)	N2	CO1	C101	C13	-53.0(2)
N2	CO1	C101	C14	19.3(2)	N2	CO1	C101	C15	90.9(2)
N2	C2	C1	C7	-125.1(2)	N2	C2	C1	C9	114.7(2)
N2	C2	C3	C4	33.7(3)	N2	C2	C3	C8	149.6(2)
C1	N1	CO1	C101	-174.4(1)	C1	C2	C3	C4	-86.4(3)
C1	C2	C3	C8	29.5(3)	C1	C7	C6	C5	-55.7(3)
C1	C7	C8	C3	48.7(2)	C1	C9	O4	C10	42.1(3)
C2	N2	CO1	C101	174.0(1)	C2	C1	C7	C6	87.2(3)
C2	C1	C7	C8	-29.4(3)	C2	C3	C4	C5	54.6(3)
C2	C3	C8	C7	-48.6(3)	C2	C10	O4	C9	-42.4(3)
C3	C2	C1	C7	-0.4(3)	C3	C2	C1	C9	-120.6(2)
C3	C4	C5	C6	37.4(3)	C3	C8	C7	C6	-71.6(3)
C4	C3	C2	C10	160.2(2)	C4	C3	C8	C7	71.7(3)
C4	C5	C6	C7	-37.1(4)	C5	C4	C3	C8	-56.1(3)
C5	C6	C7	C8	56.0(3)	C6	C7	C1	C9	-160.6(3)
C7	C1	C2	C10	119.7(2)	C8	C3	C2	C10	-83.9(3)
C8	C7	C1	C9	82.8(3)	C9	C1	C2	C10	-0.5(3)
C11	C12	C13	C14	-0.8(3)	C11	C12	C13	C18	-179.5(3)
C11	C15	C14	C13	-0.2(3)	C11	C15	C14	C19	-177.5(2)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C12	C11	C15	C14	-0.4(3)	C12	C13	C14	C15	0.6(3)
C12	C13	C14	C19	177.9(2)	C13	C12	C11	C15	0.7(3)
C13	C12	C11	C16	-177.6(3)	C14	C13	CO1	C15	-37.6(1)
C14	C13	C12	C17	179.0(2)	C14	C15	C11	C16	178.0(3)
C15	C11	C12	C17	-179.1(2)	C15	C14	C13	C18	179.4(2)
C16	C11	C12	C17	2.5(4)	C17	C12	C13	C18	0.3(4)
C18	C13	C14	C19	-3.4(4)					

Table 8. Non-bonded Contacts out to 3.75 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
O1	O3	2.819(3)	65603	O1	O1	3.411(4)	65603
O2	C17	3.690(4)	55404	O3	C16	3.519(4)	65603
O3	C17	3.635(4)	65501	O4	C10	3.056(3)	65503
O4	O4	3.234(4)	65503	O4	C19	3.332(4)	3
O4	C15	3.348(3)	3	O4	C14	3.572(3)	3
C8	C12	3.517(4)	65501	C8	C13	3.543(4)	65501
C8	C17	3.672(4)	65501	C8	C18	3.728(4)	65501
C10	C10	3.499(6)	65503	C10	C15	3.663(4)	3
C16	C18	3.607(4)	54502				

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ±4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1) X, Y, Z (2) -X, 1/2+Y, 1/2-Z (3) -X, -Y, -Z
(4) X, 1/2-Y, 1/2+Z

Table 9. Least Squares Planes

Plane number 1

Atoms defining plane	Distance
C11	-0.003(3)
C12	0.005(3)
C13	-0.004(3)
C14	0.002(3)
C15	0.001(3)

Additional Atoms	Distance
CO1	1.709
C16	-0.047
C17	0.022
C18	0.009
C19	-0.047

Plane number 2

Atoms defining plane	Distance
CO1	-0.1
N1	0.0
N2	0.0
C101	0.0

Additional Atoms	Distance
C1	0.034
C2	0.039

Summary

plane	mean deviation	χ^2
1	0.0030	8.9
2	0.0272	0.0

Dihedral angles between planes (^{o})

plane	1
2	92.90