Synthesis of α-Amino-α'-diazomethyl Ketones Via Ring Opening of Substituted Cyclopropanones with Alkyl Azides. A Facile Route to N-Substituted 3-Azetidinones.

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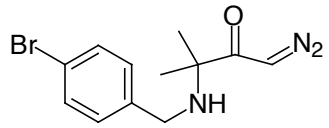
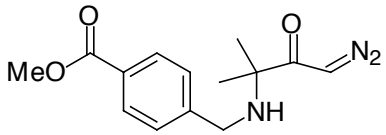
Supplementary Information

Characterization data for all new compounds and X-ray crystallographic analysis of compound **2c**

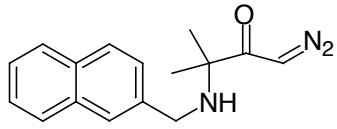
Characterization Data for New Compounds



3-(4-Bromophenyl)amino-1-diazo-3-methyl-butan-2-one (2b). Yellow oil (41%): R_f = 0.05 (20% ethyl acetate/hexanes); IR (neat) 3323, 3123, 2099, 1633, cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 8.3 Hz, 2H), 5.88 (br s, 1H), 3.60 (s, 2H), 1.64 (br s, 1H), 1.33 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 200.9, 139.8, 131.9, 130.1, 121.2, 62.6, 52.4, 48.0, 25.8; CIMS m/z (relative intensity) 298 (M+2, 55), 296 (58), 270 (97), 268 (100); HRMS calcd for $C_{12}H_{14}BrN_3O$: 296.0398, found: 296.0370.

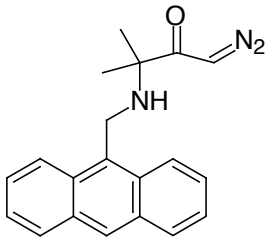


4-(3-Diazo-1,1-dimethyl-2-oxo-propylamino)benzoic acid methyl ester (2c). Yellow solid (46%): R_f = 0.05 (20% ethyl acetate/hexanes); mp 88-89 °C; IR (neat) 2099, 1707, 1633 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 5.90 (br s, 1H), 3.92 (s, 3H), 3.72 (s, 2H), 1.66 (br s, 1H), 1.35 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 200.8, 167.4, 146.1, 130.2, 129.3, 128.2, 62.7, 52.5, 52.4, 48.3, 25.8; CIMS m/z (relative intensity) 276 (MH^+, 27), 248 (100); HRMS calcd for $C_{14}H_{18}N_3O_3$: 276.1348, found: 276.1344.

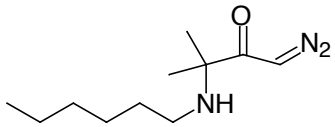


1-Diazo-3-methyl-3-(naphthalen-2-yl)amino-butan-2-one (2d). Yellow oil (38%): R_f = 0.07 (20% ethyl acetate/hexanes); IR (neat) 3323, 3119, 2099, 1633 cm^{-1}; ^1H NMR

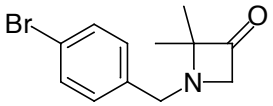
(400 MHz, $CDCl_3$) δ 7.80-7.83 (m, 4H), 7.45-7.48 (m, 3H), 5.99 (br s, 1H), 3.82 (s, 2H), 1.66 (br s, 1H), 1.38 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 201.2, 138.2, 133.9, 133.1, 128.5, 128.1, 126.9, 126.6, 126.5, 126.1, 62.7, 52.3, 48.8, 25.9; CIMS m/z (relative intensity) 268 (MH^+, 74), 198 (100); HRMS calcd for $C_{16}H_{17}N_3O$: 268.1450, found: 268.1445.



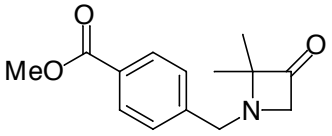
3-(Anthracen-9-yl)methyl-amino-1-diazo-3-methyl-butan-2-one (2e). Brown oil (54%): R_f = 0.07 (20% ethyl acetate/hexanes); IR (neat) 3323, 3057, 2099, 1628 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.41 (s, 1H), 8.33 (d, *J* = 8.9 Hz, 2H), 8.00 (d, *J* = 8.3 Hz, 2H), 7.54-7.58 (m, 2H), 7.45-7.49 (m, 2H), 5.91 (br s, 1H), 4.59 (s, 2H), 1.70 (br s, 1H), 1.50 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 201.0, 132.0, 131.5, 130.6, 129.6, 127.8, 126.6, 125.4, 124.4, 62.8, 52.6, 40.3, 25.7; CIMS m/z (relative intensity) 191 (100); HRMS calcd for $C_{20}H_{20}N_3O$: 318.1606, found: 318.1577.



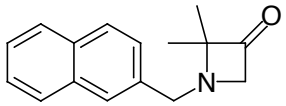
1-Diazo-3-hexylamino-3-methyl-butan-2-one (2f). Yellow oil (44%): R_f = 0.05 (20% ethyl acetate/hexanes); IR (neat) 3326, 3122, 2099, 1633 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.86 (br s, 1H), 2.44 (t, *J* = 7.0 Hz, 2H), 1.29-1.43 (m, 9H), 1.25 (s, 6H), 0.89 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 201.8, 62.3, 52.0, 44.3, 32.2, 31.3, 27.4, 25.7, 23.0, 14.4; CIMS m/z (relative intensity) 212 (MH^+, 100), 184 (75), 142 (72); HRMS calcd for $C_{11}H_{22}N_3O$: 212.1763, found: 212.1742.



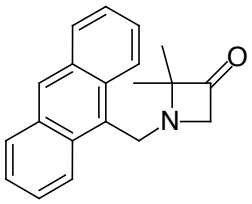
1-(4-Bromobenzyl)-2,2-dimethyl-azetidin-3-one (3b). Yellow oil (87%): R_f = 0.15 (20% ethyl acetate/hexanes); IR (neat) 2971, 1803 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.3 Hz, 2H), 4.02 (s, 2H), 3.79 (s, 2H), 1.28 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 208.6, 138.3, 131.9, 130.4, 121.3, 84.0, 70.9, 54.7, 20.4; CIMS m/z (relative intensity) 270 (M+2, 27), 268 (29), 70 (100); HRMS calcd for $C_{12}H_{14}BrNO$: 267.0259, found: 267.0238.



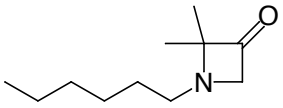
4-(2,2-Dimethyl-3-oxo-azetidin-1-yl)methylbenzoic acid methyl ester (3c). Yellow oil (77%): R_f = 0.16 (20% ethyl acetate/hexanes); IR (neat) 2973, 1804, 1721 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.47 (d, *J* = 8.1 Hz, 2H), 4.05 (s, 2H), 3.91 (s, 3H), 3.90 (s, 2H), 1.30 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 208.5, 167.3, 144.7, 130.1, 129.5, 128.6, 84.2, 71.0, 55.1, 52.5, 20.4; CIMS m/z (relative intensity) 248 (MH^+, 100), 70 (66); HRMS calcd for $C_{14}H_{18}NO_3$: 248.1287, found: 248.1276.



2,2-Dimethyl-1-(naphthalen-2-yl)methyl-azetidin-3-one (3d). Off-white solid (95%): R_f = 0.16 (20% ethyl acetate/hexanes); mp 81-82 °C; IR (neat) 2970, 1804 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.80-7.84 (m, 4H), 7.53-7.56 (dd, *J* = 1.5, 8.4 Hz, 1H), 7.45-7.48 (m, 2H), 4.08 (s, 2H), 4.00 (s, 2H), 1.33 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 209.1, 136.8, 133.8, 133.2, 128.5, 128.1, 127.2, 127.1, 126.5, 126.1; CIMS m/z (relative intensity) 240 (MH^+, 100), 141 (25), 70 (72); HRMS calcd for $C_{16}H_{18}NO$: 240.1388, found: 240.1407.

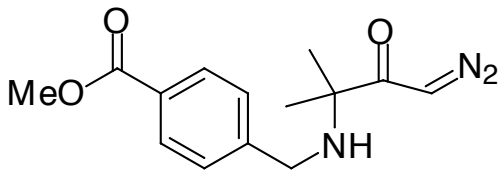


1-(Anthracen-9-yl)methyl-2,2-dimethyl-azetidin-3-one (3e). Yellow oil (100%): R_f = 0.18 (20% ethyl acetate/hexanes); IR (neat) 2966, 1800 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 8.8 Hz, 2H), 8.42 (s, 1H), 8.00 (d, *J* = 8.3 Hz, 2H), 7.51-7.55 (m, 2H), 7.44-7.48 (m, 2H), 4.80 (s, 2H), 3.96 (s, 2H), 1.48 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 208.8, 134.5, 132.0, 131.1, 129.7, 129.6, 129.4, 128.2, 127.6, 126.3, 126.1, 125.3, 124.7, 84.1, 70.2, 45.4, 20.4; CIMS m/z (relative intensity) 290(MH^+, 7), 209 (40), 191 (100); HRMS calcd for $C_{12}H_{16}NO$:, found:.



1-Hexyl-2,2-dimethyl-azetidin-3-one (3f). Yellow oil (72%): R_f = 0.14 (20% ethyl acetate/hexanes); IR (neat) 2927, 1806 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.01 (s, 2H), 2.62 (t, *J* = 7.1 Hz, 2H), 1.31-1.38 (m, 8H), 1.26 (s, 6H), 0.90 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 209.3, 83.3, 70.9, 51.4, 32.1, 29.8, 27.5, 23.0, 20.1, 14.4; CIMS m/z (relative intensity) 184 (MH^+, 100), 142(57), 91 (21); HRMS calcd for $C_{11}H_{22}NO$: 184.1701, found: 184.1692.

X-ray Crystallographic Analysis of Compound 2c



EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{14}H_{17}N_3O_3$
Formula Weight	275.31
Crystal Color, Habit	Clear, Prism
Crystal Dimensions	0.30 X 0.10 X 0.30 mm
Crystal System	triclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	16 (32.9 - 43.3°)
Omega Scan Peak Width at Half-height	0.30°
Lattice Parameters	a = 5.4514(5) Å b = 11.706(2) Å c = 12.161(1) Å α = 108.6(2) ° β = 93.785(9) ° γ = 101(1) ° V = 716(2) Å^3
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.278 g/cm^3
F_{000}	292.00
μ(CuKα)	7.56 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R
Radiation	CuKα (λ = 1.54178 Å) graphite monochromated
Attenuator	Ni foil (factors = 1.00, 3.83, 13.12, 50.79)
Take-off Angle	6.0°
Detector Aperture	6.0 mm horizontal 6.0 mm vertical

Crystal to Detector Distance	400 mm
Voltage, Current	50kV, 150mA
Temperature	23.0°C
Scan Type	ω-2θ
Scan Rate	8.0°/min (in ω) (up to 4 scans)
Scan Width	$(1.63 + 0.30 \tan \theta)^{o}$
$2\theta_{max}$	120.1°
No. of Reflections Measured	Total: 2247
Corrections	Unique: 2128 (R_{int} = 0.014) Lorentz-polarization Absorption (trans. factors: 0.9374 - 0.9994) Decay (2.70% decline)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	Σ w $(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$1/\sigma^2(Fo) = 4Fo^2/\sigma^2(Fo^2)$
p-factor	0.0092
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>0.01σ(I))	1789
No. Variables	249
Reflection/Parameter Ratio	7.18
Residuals: R; Rw	0.049 ; 0.038
Goodness of Fit Indicator	1.92
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.16 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.18 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
O(1)	1.0221(2)	0.9192(1)	0.3154(1)	5.38(3)
O(2)	-0.4644(2)	0.7707(1)	-0.2935(1)	6.41(4)
O(3)	-0.4974(2)	0.5717(1)	-0.3174(1)	5.09(3)
N(1)	0.4067(2)	0.7632(1)	0.1787(1)	3.44(3)
N(5)	0.8270(3)	1.1182(1)	0.3851(1)	3.94(3)
N(6)	0.9645(3)	1.2096(1)	0.4218(1)	5.29(4)
C(2)	0.6133(3)	0.7764(1)	0.2690(1)	3.27(4)
C(3)	0.7931(3)	0.9052(1)	0.3101(1)	3.35(4)
C(4)	0.6747(3)	1.0077(1)	0.3422(1)	4.04(4)
C(7)	0.4819(4)	0.7950(2)	0.0770(2)	5.02(5)
C(8)	0.2528(3)	0.7669(2)	-0.0126(1)	3.97(4)
C(9)	0.1122(3)	0.6471(2)	-0.0646(1)	4.29(5)
C(10)	-0.0959(3)	0.6202(2)	-0.1472(1)	3.95(4)
C(11)	-0.1686(3)	0.7137(1)	-0.1794(1)	3.57(4)
C(12)	-0.0285(3)	0.8335(2)	-0.1278(2)	4.54(5)
C(13)	0.1784(3)	0.8592(2)	-0.0449(2)	4.72(5)
C(14)	-0.3889(3)	0.6914(2)	-0.2677(1)	4.07(4)
C(15)	-0.7216(4)	0.5415(3)	-0.4013(2)	5.98(6)
C(16)	0.7615(4)	0.6770(2)	0.2232(2)	4.99(5)
C(17)	0.4898(4)	0.7598(2)	0.3741(2)	4.31(5)

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

atom	x	y	z	B_{eq}
H(1)	0.294(3)	0.808(1)	0.212(1)	3.2(3)
H(4)	0.496(3)	1.010(1)	0.338(1)	5.6(4)
H(7a)	0.564(3)	0.884(2)	0.096(1)	5.9(5)
H(7b)	0.597(3)	0.741(2)	0.040(2)	6.9(5)
H(9)	0.161(3)	0.584(1)	-0.043(1)	4.6(4)
H(10)	-0.191(3)	0.535(1)	-0.181(1)	5.2(4)
H(12)	-0.084(3)	0.896(2)	-0.149(1)	5.9(5)
H(13)	0.269(3)	0.943(1)	-0.003(1)	5.6(4)
H(15b)	-0.687(4)	0.581(2)	-0.460(2)	8.9(7)
H(15c)	-0.779(5)	0.450(2)	-0.429(2)	12.9(9)
H(15a)	-0.846(4)	0.581(2)	-0.367(2)	7.2(6)
H(16b)	0.852(3)	0.688(1)	0.156(1)	5.8(5)
H(16c)	0.648(3)	0.596(1)	0.198(1)	5.4(4)
H(16a)	0.897(3)	0.683(2)	0.283(2)	6.9(5)
H(17b)	0.382(3)	0.821(1)	0.400(1)	5.5(4)
H(17c)	0.385(3)	0.673(2)	0.351(1)	5.7(4)
H(17a)	0.614(3)	0.773(1)	0.441(1)	5.6(4)

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0294(7)	0.0554(8)	0.110(1)	0.0099(5)	0.0067(6)	0.0152(7)
O(2)	0.0690(9)	0.0729(9)	0.113(1)	0.0223(7)	-0.0090(8)	0.0476(9)
O(3)	0.0689(8)	0.0596(8)	0.0572(8)	0.0142(7)	-0.0172(6)	0.0147(6)
N(1)	0.0318(7)	0.0572(9)	0.0392(8)	0.0091(7)	0.0072(6)	0.0127(7)
N(5)	0.0454(8)	0.0520(9)	0.0506(9)	0.0189(8)	0.0005(7)	0.0114(7)
N(6)	0.059(1)	0.055(1)	0.076(1)	0.0090(8)	-0.0074(8)	0.0135(9)
C(2)	0.0290(8)	0.0465(9)	0.048(1)	0.0106(7)	0.0038(7)	0.0133(8)
C(3)	0.0340(9)	0.050(1)	0.0425(9)	0.0112(8)	0.0023(7)	0.0132(8)
C(4)	0.035(1)	0.045(1)	0.064(1)	0.0104(8)	-0.0025(8)	0.0047(9)
C(7)	0.053(1)	0.085(2)	0.044(1)	-0.004(1)	0.0037(9)	0.022(1)
C(8)	0.050(1)	0.062(1)	0.0337(9)	0.0032(9)	0.0073(8)	0.0138(8)
C(9)	0.063(1)	0.054(1)	0.046(1)	0.014(1)	-0.0018(9)	0.0182(9)
C(10)	0.059(1)	0.045(1)	0.040(1)	0.0088(9)	-0.0019(8)	0.0104(8)
C(11)	0.048(1)	0.048(1)	0.040(1)	0.0120(8)	0.0077(7)	0.0129(8)
C(12)	0.064(1)	0.052(1)	0.060(1)	0.013(1)	0.010(1)	0.022(1)
C(13)	0.062(1)	0.049(1)	0.057(1)	-0.002(1)	0.009(1)	0.010(1)
C(14)	0.051(1)	0.059(1)	0.050(1)	0.0166(9)	0.0101(8)	0.0217(9)
C(15)	0.064(1)	0.090(2)	0.069(2)	0.020(1)	-0.017(1)	0.025(1)
C(16)	0.044(1)	0.048(1)	0.094(2)	0.011(1)	0.012(1)	0.017(1)
C(17)	0.046(1)	0.070(1)	0.052(1)	0.011(1)	-0.0002(9)	0.029(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 Excluding Hydrogen(Å)

atom	atom	distance	atom	atom	distance
O1	C3	1.223(4)	O2	C14	1.201(7)
O3	C14	1.33(1)	O3	C15	1.450(7)
N1	C2	1.470(3)	N1	C7	1.463(4)
N5	N6	1.12(1)	N5	C4	1.32(2)
C2	C3	1.54(2)	C2	C16	1.52(1)
C2	C17	1.531(4)	C3	C4	1.43(1)
C7	C8	1.514(6)	C8	C9	1.39(2)
C8	C13	1.379(7)	C9	C10	1.381(6)
C10	C11	1.387(7)	C11	C12	1.38(2)
C11	C14	1.482(5)	C12	C13	1.381(6)

Table 4. Bond Lengths to Hydrogen(Å)

atom	atom	distance	atom	atom	distance
N1	H1	0.91(2)	C4	H4	0.98(2)
C7	H7a	1.00(2)	C7	H7b	1.00(3)
C9	H9	0.94(2)	C10	H10	0.97(2)
C12	H12	0.95(2)	C13	H13	0.97(2)
C15	H15b	0.98(3)	C15	H15c	1.00(3)
C15	H15a	0.94(3)	C16	H16b	1.01(2)
C16	H16c	0.97(2)	C16	H16a	0.98(2)
C17	H17b	1.00(2)	C17	H17c	1.01(2)
C17	H17a	0.98(2)			

Table 5. Bond Angles Excluding Hydrogen(°)

atom	atom	atom	angle	atom	atom	atom	angle
C14	O3	C15	116.4(9)	C2	N1	C7	115.9(4)
N6	N5	C4	177.0(2)	N1	C2	C3	112.7(7)
N1	C2	C16	109.8(6)	N1	C2	C17	106.4(3)
C3	C2	C16	109.7(2)	C3	C2	C17	108.0(4)
C16	C2	C17	110.1(3)	O1	C3	C2	122.5(9)
O1	C3	C4	121.9(9)	C2	C3	C4	115.6(2)
N5	C4	C3	116.2(2)	N1	C7	C8	109.9(5)
C7	C8	C9	120.6(8)	C7	C8	C13	121.2(8)
C9	C8	C13	118.2(2)	C8	C9	C10	121.1(8)
C9	C10	C11	120.2(8)	C10	C11	C12	118.8(2)
C10	C11	C14	123.1(8)	C12	C11	C14	118.1(8)
C11	C12	C13	120.4(8)	C8	C13	C12	121.3(8)
O2	C14	O3	122.6(3)	O2	C14	C11	124.9(7)
O3	C14	C11	112.5(9)				

Table 6. Bond Angles to Hydrogen(°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	N1	H1	109(1)	C7	N1	H1	110(1)
N5	C4	H4	114(2)	C3	C4	H4	130(2)
N1	C7	H7a	114(1)	N1	C7	H7b	108(2)
C8	C7	H7a	107(2)	C8	C7	H7b	108(2)
H7a	C7	H7b	111(2)	C8	C9	H9	119(1)
C10	C9	H9	120(2)	C9	C10	H10	119(2)
C11	C10	H10	121(1)	C11	C12	H12	117(1)
C13	C12	H12	122(2)	C8	C13	H13	117(1)
C12	C13	H13	122(2)	O3	C15	H15b	110(2)
O3	C15	H15c	106(2)	O3	C15	H15a	111(2)
H15b	C15	H15c	118(3)	H15b	C15	H15a	101(2)
H15c	C15	H15a	112(3)	C2	C16	H16b	112(1)
C2	C16	H16c	109(1)	C2	C16	H16a	111(2)
H16b	C16	H16c	110(2)	H16b	C16	H16a	104(2)
H16c	C16	H16a	110(2)	C2	C17	H17b	110(1)
C2	C17	H17c	109(1)	C2	C17	H17a	112(1)
H17b	C17	H17c	110(2)	H17b	C17	H17a	107(2)
H17c	C17	H17a	108(2)				

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O1	C3	C2	N1	-130.2(3)	O1	C3	C2	C16	-7.5(3)
O1	C3	C2	C17	112.6(3)	O1	C3	C4	N5	-4.2(3)
O2	C14	O3	C15	-2.9(4)	O2	C14	C11	C10	176.2(2)
O2	C14	C11	C12	-4.4(4)	O3	C14	C11	C10	-4.0(3)
O3	C14	C11	C12	175.4(2)	N1	C2	C3	C4	50.1(3)
N1	C7	C8	C9	-62.2(7)	N1	C7	C8	C13	118.0(7)
N5	C4	C3	C2	175.5(2)	N6	N5	C4	C3	-10(5)
C2	N1	C7	C8	175.3(2)	C3	C2	N1	C7	52.5(7)
C4	C3	C2	C16	172.8(2)	C4	C3	C2	C17	-67.2(3)
C7	N1	C2	C16	-70.2(6)	C7	N1	C2	C17	170.7(2)
C7	C8	C9	C10	-179.4(2)	C7	C8	C13	C12	179.1(2)
C8	C9	C10	C11	-0.3(4)	C8	C13	C12	C11	0.9(4)
C9	C8	C13	C12	-0.8(4)	C9	C10	C11	C12	0.4(3)
C9	C10	C11	C14	179.8(2)	C10	C9	C8	C13	0.5(4)
C10	C11	C12	C13	-0.7(4)	C11	C14	O3	C15	177.4(2)
C13	C12	C11	C14	179.9(2)					

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
O1	N1	3.24(2)	65501	O1	C4	3.48(2)	65501
O1	C17	3.59(3)	65501	O2	N5	2.91(2)	57502
O2	N6	3.134(5)	57502	O2	C4	3.23(2)	57502
O2	N6	3.235(5)	67502	O3	C16	3.55(2)	56502
N1	C16	3.60(2)	45501	N5	C17	3.54(1)	67602
N5	C12	3.559(5)	67502	N6	C14	3.34(2)	67502
N6	C15	3.49(2)	57502	N6	C12	3.498(4)	67502
N6	C11	3.532(7)	67502	C15	C15	3.52(2)	36402
C15	C15	3.56(2)	46402				

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 $\pm$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 is 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, -Y, -Z