Supporting Information

Probing covalency in halogen bonds through Donor K-edge X-ray Absorption Spectroscopy: polyhalides as coordination complexes

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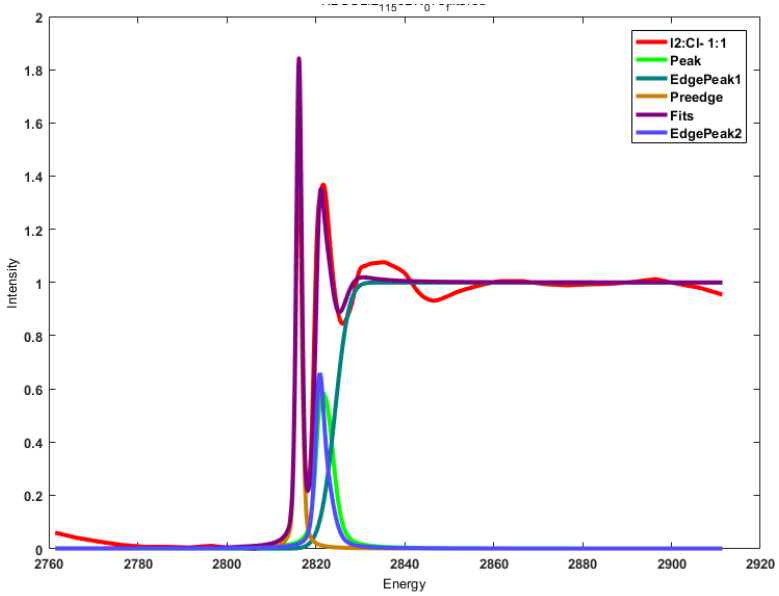
1. X-ray Absorption Spectroscopy – Fitting

Results of BlueprintXAS fitting routines for the experimental data.

$[NBu_4][I_2Cl]$

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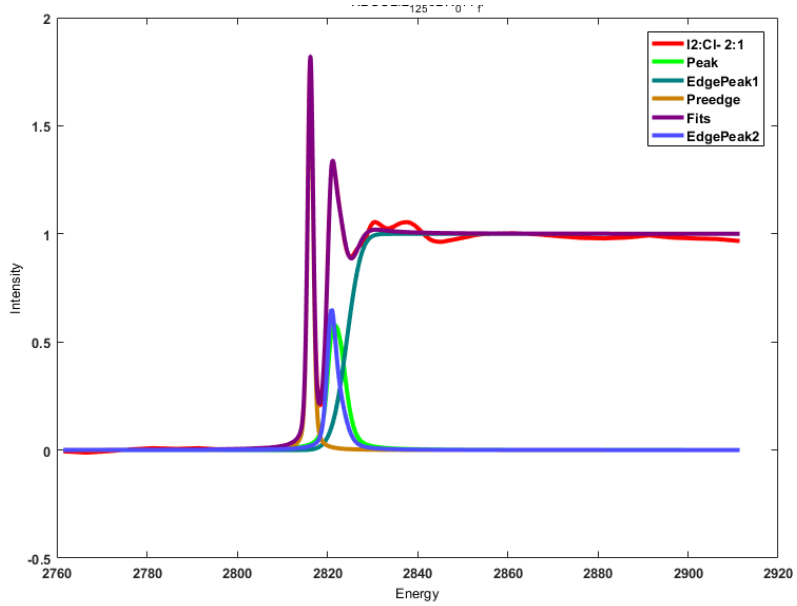
Num of Fits       87
SSE       0.0785433
rsquare   0.996328
fy-Projection, <f|y>       0.999836
```

Table of Coefficients

Coefficient Name	Identifier(ID)	Average	Standard Dev
Degree-Pre-edge	M1	2.49631	0.859466
Degree-Spline1	N1	2.48686	0.937439
E0 (edge-Spline)	E0	2825.12	0.591702
hwhm	W1	2.95225	0.354827
Pre-edge, Max	B1	2810.03	3.00571
Spline, Min	C1	2823.65	1.23927
Intensity	I1	2.01592	0.918122
Position	O1	2822.08	0.798055
hwhm	W2	2.05909	0.837748
Gaussian, fracti	G2	0.470512	0.0893283
Intensity	**I2**	**1.2309**	**0.0586109**
Position	O2	2816.21	0.00344701
hwhm	W3	0.715495	0.0155986
Gaussian, fracti	G3	0.593103	0.0367047
Intensity	I3	1.36364	0.852886
Position	O3	2821.13	0.790714
hwhm	W4	1.41184	0.45678
Gaussian, fracti	G4	0.556676	0.0824875
Intens		**0.461078**	**0.0596354**

[NBu_4][I_4Cl]

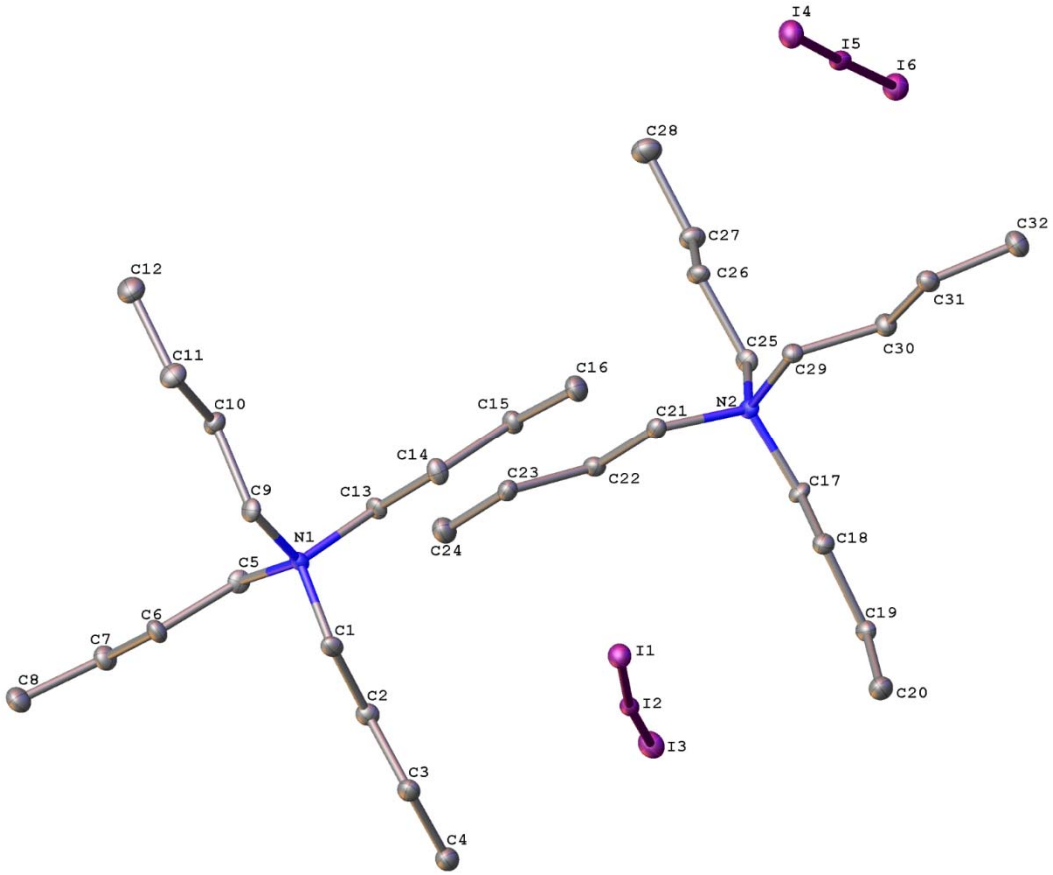


Fit Results for NBUCLI2_1_2_50BN_077_fl
Num of Fits 73
SSE 0.0279142
rsquare 0.99879
fy-Projection, <f|y> 0.999972

Coefficient Name	Identifier(ID)	Average	Standard Dev
Degree-Pre-edge	M1	2.50044	0.806518
Degree-Spline1	N1	2.62854	0.905923
E0 (edge-Spline)	E0	2824.2	0.706002
hwhm	W1	2.93857	0.347731
Pre-edge, Max	B1	2810.11	3.10245
Spline, Min	C1	2823.43	1.15782
Intensity	I1	1.5132	0.654563
Position	O1	2821.97	0.781686
hwhm	W2	1.72476	0.556447
Gaussian, fraction	G2	0.478286	0.0953849
Intensity	**I2**	**1.48991**	**0.0416267**
Position	O2	2816.26	0.00161486
hwhm	W3	0.740242	0.0110056
Gaussian, fraction	G3	0.592608	0.0179774
Intensity	I3	1.17341	0.515586
Position	O3	2821.35	0.733435
hwhm	W4	1.34854	0.439796
Gaussian, fraction	G4	0.545577	0.0892616
Intens		**0.466285**	**0.0393856**

2. X-ray Crystallographic Data

CCDC 1534358 (PK025) $[NBu_4][I_3]$ with ~2% $[NBu_4][I_2Cl]$ (disordered)



Data Collection

A yellow blade crystal of $C_{16}H_{36}I_3N$ having approximate dimensions of 0.03 x 0.12 x 0.27 mm was mounted on a cryo-loop. All measurements were made on a Bruker APEX DUO diffractometer with a TRIUMPH curved-crystal monochromator with Mo-Kα radiation.

The data were collected at a temperature of -183.0 ± 0.1°C to a maximum 2θ value of 61.1°. Data were collected in a series of φ and ω scans in 0.5° oscillations using 5.0-second exposures. The crystal-to-detector distance was 40.20 mm.

Data Reduction

Of the 51116 reflections that were collected, 13372 were unique (R_{int} = 0.046); equivalent reflections were merged. Data were collected and integrated using the Bruker SAINT[1] software package. The linear absorption coefficient, μ, for Mo-Kα radiation is 42.76 cm^{-1}. Data were corrected for absorption effects using the multi-scan technique (SADABS[2]), with minimum and maximum transmission coefficients of 0.703 and 0.880, respectively. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[3]. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were placed in calculated positions. The final cycle of full-matrix least-squares refinement[4] on F^2 was based on 13372 reflections and 369 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1\ (I>2.00\sigma(I)) = \Sigma\ ||Fo| - |Fc||\ /\ \Sigma\ |Fo| = 0.030$$

$$wR2\ (all\ data) = [\ \Sigma\ (\ w\ (Fo^2 - Fc^2)^2\ )/\ \Sigma\ w(Fo^2)^2]^{1/2} = 0.064$$

The standard deviation of an observation of unit weight[5] was 1.09. The weighting scheme was based on counting statistics. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.09 and –0.96 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf" were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All refinements were performed using the XL[10] via the OLEX2[11] interface.

References

(1) SAINT. Version 8.34A Bruker AXS Inc., Madison, Wisconsin, USA. (1997-2013).

(2) SADABS 2014/5 - Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Crystallogr.* **48**.

(3) XT: Sheldrick, G. M.; Acta Cryst., A71, 3-8 (2015).

(4) Least Squares function minimized:

$$\Sigma w(F_o^2-F_c^2)^2$$

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

$$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where: N_o = number of observations

N_v = number of variables

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

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

(8) 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).

(9) 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).

(10) XL Sheldrick, G. M.; Acta Cryst., C71, 3-8 (2015).

(11) OLEX2 – V1.2.8 Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H., OLEX2: A complete structure solution, refinement and analysis program (2009). J. Appl. Cryst., 42, 339-341.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{16}H_{36}I_3N$
Formula Weight	623.16
Crystal Colour, Habit	yellow, blade
Crystal Dimensions	0.03 x 0.12 x 0.27 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 9.463(3) Å
	b = 15.553(5) Å
	c = 15.805(5) Å
	α = 83.658(7)^o
	β = 74.285(6)^o
	γ = 78.500(6)^o
	V = 2190.4(11) Å^3
Space Group	*P* -1 (#2)
Z value	4
D_{calc}	1.890 g/cm^3
F_{000}	1192.00
μ(Mo-Kα)	42.76 cm^{-1}

B. Intensity Measurements

Diffractometer	Bruker APEX DUO
Radiation	Mo-Kα (λ = 0.71073 Å)
Data Images	1937 exposures @ 5.0 seconds
Detector Position	40.20 mm
$2\theta_{max}$	61.1^{o}
No. of Reflections Measured	Total: 51116
	Unique: 13372 (R_{int} = 0.046)
Corrections	Absorption (T_{min} = 0.703, T_{max}= 0.880)
	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (XT)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w=1/(\sigma^2(Fo^2)+(0.0000P)^2 + 4.2509P)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>0.00σ(I))	13372
No. Variables	369
Reflection/Parameter Ratio	36.24
Residuals (refined on F^2, all data): R1; wR2	0.041; 0.064
Goodness of Fit Indicator	1.09
No. Observations (I>2.00σ(I))	11164
Residuals (refined on F^2): R1; wR2	0.030; 0.060
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	1.09 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.96 $e^-/Å^3$

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for pk025. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
N1	806(3)	9688.4(16)	7350.5(16)	9.1(5)
C1	817(3)	9810(2)	6373(2)	11.9(6)
C2	2330(3)	9843(2)	5736(2)	13.3(6)
C3	2226(4)	9844(2)	4786(2)	15.2(6)
C4	3753(4)	9856(2)	4127(2)	18.5(7)
C5	1643(3)	10336(2)	7572(2)	11.3(6)
C6	987(4)	11295(2)	7433(2)	13.5(6)
C7	2005(4)	11897(2)	7571(2)	15.1(6)
C8	1278(4)	12860(2)	7520(2)	18.5(7)
C9	-818(3)	9847(2)	7881(2)	11.5(6)
C10	-1061(3)	9742(2)	8875(2)	13.2(6)
C11	-2727(4)	9858(2)	9332(2)	16.2(6)
C12	-3013(4)	9812(2)	10329(2)	19.9(7)
C13	1579(3)	8759.1(19)	7563(2)	11.5(6)
C14	769(3)	8025(2)	7502(2)	13.7(6)
C15	1652(3)	7129(2)	7711(2)	13.0(6)
C16	872(4)	6388(2)	7610(2)	17.9(7)
N2	6827(3)	5039.6(16)	7293.1(17)	9.3(5)
C17	7804(3)	5014(2)	6350.5(19)	11.2(6)
C18	6994(3)	5038(2)	5634(2)	13.0(6)
C19	8080(3)	5096(2)	4725(2)	12.7(6)
C20	7261(4)	5229(2)	3995(2)	17.8(7)
C21	5833(3)	5943.6(19)	7450(2)	11.0(6)

C22	6627(3)	6695(2)	7493(2)	12.3(6)
C23	5522(3)	7565(2)	7566(2)	12.5(6)
C24	6304(4)	8331(2)	7608(2)	19.2(7)
C25	7872(3)	4841(2)	7897(2)	12.4(6)
C26	7121(3)	4842(2)	8876(2)	12.4(6)
C27	8289(4)	4739(2)	9401(2)	15.5(6)
C28	7595(4)	4712(2)	10383(2)	20.8(7)
C29	5789(3)	4367.1(19)	7470(2)	10.8(6)
C30	6589(3)	3416(2)	7368(2)	13.7(6)
C31	5462(4)	2812(2)	7428(2)	13.4(6)
C32	6221(4)	1852(2)	7429(2)	19.4(7)
I1	3772.7(2)	7344.0(2)	5192.5(2)	14.45(5)
I2	6831.4(2)	7544.3(2)	5048.7(2)	11.11(4)
I3	9965.8(2)	7581.3(2)	4969.9(2)	16.55(5)
I4	1825.7(2)	2779.4(2)	10030.0(2)	18.17(5)
I5	4984.5(2)	2517.2(2)	9928.8(2)	13.08(4)
I6	8220.7(2)	2200.1(2)	9805.4(2)	17.41(5)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for pk025. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	10.0(11)	7.8(11)	10.5(12)	0.8(9)	-4.8(9)	-1.2(9)
C1	15.8(14)	11.7(14)	10.2(14)	0.6(11)	-6.8(12)	-3.1(11)
C2	14.4(14)	13.7(15)	12.9(15)	0.1(11)	-5.3(12)	-3.1(11)
C3	19.5(16)	12.5(15)	12.9(15)	0.4(11)	-5.0(12)	-0.6(12)
C4	26.3(18)	13.8(15)	14.5(16)	-0.8(12)	-3.6(14)	-3.6(13)

C5	9.9(13)	13.8(14)	12.9(14)	-2.1(11)	-5.9(11)	-3.2(11)
C6	17.5(15)	8.2(13)	15.2(15)	0.4(11)	-6.1(12)	-1.4(11)
C7	17.3(15)	12.9(15)	16.9(16)	-1.6(12)	-5.1(13)	-5.1(12)
C8	24.1(17)	12.7(15)	19.0(17)	0.1(12)	-4.7(14)	-5.9(13)
C9	7.8(13)	11.6(14)	13.8(15)	-2.5(11)	-2.3(11)	1.3(10)
C10	13.2(14)	14.3(15)	11.2(14)	-2.2(11)	-2.2(12)	-1.1(11)
C11	12.5(14)	20.0(16)	15.5(16)	-1.8(12)	-1.8(12)	-3.5(12)
C12	19.6(16)	20.7(17)	17.5(17)	-1.6(13)	-1.0(13)	-3.7(13)
C13	11.5(13)	9.6(13)	13.0(14)	1.2(11)	-5.7(11)	1.6(11)
C14	13.8(14)	11.2(14)	18.2(16)	-1.9(11)	-7.7(12)	-1.6(11)
C15	14.4(14)	10.7(14)	14.0(15)	-0.6(11)	-5.4(12)	0.3(11)
C16	23.8(17)	12.1(15)	18.4(17)	-1.8(12)	-4.8(14)	-4.7(13)
N2	6.2(11)	10.8(12)	9.9(12)	0.0(9)	-2.4(9)	0.7(9)
C17	9.4(13)	14.5(14)	8.4(14)	-2.2(11)	0.3(11)	-1.6(11)
C18	14.7(14)	12.3(14)	11.9(14)	-0.4(11)	-2.3(12)	-3.5(11)
C19	13.2(14)	11.2(14)	11.9(14)	-0.4(11)	-1.9(12)	-0.1(11)
C20	24.2(17)	15.4(16)	13.1(15)	-1.1(12)	-3.9(13)	-2.8(13)
C21	11.5(13)	11.1(14)	10.0(14)	-0.7(10)	-3.9(11)	0.9(11)
C22	13.9(14)	12.1(14)	10.5(14)	1.2(11)	-3.8(11)	-1.5(11)
C23	14.9(14)	11.5(14)	10.4(14)	-1.8(11)	-2.9(12)	-0.9(11)
C24	27.4(18)	13.8(15)	17.2(17)	-1.2(12)	-6.4(14)	-4.5(13)
C25	10.0(13)	12.3(14)	14.4(15)	-0.2(11)	-4.8(11)	1.2(11)
C26	13.0(14)	13.4(14)	9.8(14)	1.5(11)	-3.7(11)	0.2(11)
C27	18.3(15)	17.7(16)	11.8(15)	0.2(12)	-6.0(12)	-4.0(12)
C28	24.7(18)	23.5(18)	14.7(16)	1.0(13)	-6.9(14)	-4.4(14)
C29	10.4(13)	11.2(14)	10.5(14)	0(1)	-1.8(11)	-2.8(11)

C30	13.3(14)	12.4(14)	14.5(15)	-0.8(11)	-2.5(12)	-2.0(11)
C31	16.3(14)	12.3(14)	12.7(15)	0.6(11)	-5.2(12)	-3.4(11)
C32	24.7(17)	11.7(15)	20.8(17)	-0.2(12)	-4.6(14)	-3.2(13)
I1	12.64(9)	14.06(10)	15.95(10)	-1.21(7)	-2.68(8)	-1.93(7)
I2	13.89(9)	9.01(9)	10.08(9)	0.04(6)	-2.94(7)	-1.68(7)
I3	14.39(9)	15.52(10)	20.88(11)	0.25(8)	-6.88(8)	-2.74(8)
I4	16.75(10)	16.79(10)	20.42(11)	-2.59(8)	-4.08(8)	-1.79(8)
I5	17.11(9)	12.58(9)	9.58(9)	-0.08(7)	-2.94(7)	-3.67(7)
I6	16.18(10)	17.33(10)	19.09(11)	-1.66(8)	-4.25(8)	-3.84(8)

Table 4 Bond Lengths for pk025.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C1	1.532(4)	N2	C25	1.520(4)
N1	C5	1.521(4)	N2	C29	1.527(4)
N1	C9	1.523(4)	C17	C18	1.524(4)
N1	C13	1.530(4)	C18	C19	1.529(4)
C1	C2	1.518(4)	C19	C20	1.530(4)
C2	C3	1.532(4)	C21	C22	1.527(4)
C3	C4	1.537(5)	C22	C23	1.530(4)
C5	C6	1.514(4)	C23	C24	1.538(4)
C6	C7	1.539(4)	C25	C26	1.517(4)
C7	C8	1.521(5)	C26	C27	1.528(4)
C9	C10	1.521(4)	C27	C28	1.511(5)
C10	C11	1.529(4)	C29	C30	1.525(4)
C11	C12	1.521(5)	C30	C31	1.535(4)

C13	C14	1.521(4)	C31	C32	1.523(4)
C14	C15	1.528(4)	I1	I2	2.9180(9)
C15	C16	1.529(4)	I2	I3	2.9468(9)
N2	C17	1.526(4)	I4	I5	2.8978(9)
N2	C21	1.532(4)	I5	I6	2.9590(9)

Table 5 Bond Angles for pk025.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C5	N1	C1	110.2(2)	C17	N2	C29	110.9(2)
C5	N1	C9	110.4(2)	C25	N2	C17	106.9(2)
C5	N1	C13	107.9(2)	C25	N2	C21	110.9(2)
C9	N1	C1	107.5(2)	C25	N2	C29	110.9(2)
C9	N1	C13	110.6(2)	C29	N2	C21	106.9(2)
C13	N1	C1	110.3(2)	C18	C17	N2	115.8(2)
C2	C1	N1	115.6(2)	C17	C18	C19	110.1(3)
C1	C2	C3	110.3(3)	C18	C19	C20	111.1(3)
C2	C3	C4	111.4(3)	C22	C21	N2	115.9(2)
C6	C5	N1	114.9(2)	C21	C22	C23	109.8(2)
C5	C6	C7	111.1(3)	C22	C23	C24	110.6(3)
C8	C7	C6	111.2(3)	C26	C25	N2	115.5(2)
C10	C9	N1	115.3(2)	C25	C26	C27	110.1(3)
C9	C10	C11	110.4(3)	C28	C27	C26	112.0(3)
C12	C11	C10	111.8(3)	C30	C29	N2	114.4(2)
C14	C13	N1	115.3(2)	C29	C30	C31	110.2(3)
C13	C14	C15	110.8(2)	C32	C31	C30	110.6(3)

C14	C15	C16	110.8(3)	I1	I2	I3	174.814(10)
C17	N2	C21	110.5(2)	I4	I5	I6	178.372(10)

Table 6 Torsion Angles for pk025.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
N1	C1	C2	C3	172.5(2)	N2	C17	C18	C19	174.7(2)
N1	C5	C6	C7	-173.4(3)	N2	C21	C22	C23	174.8(2)
N1	C9	C10	C11	-176.3(2)	N2	C25	C26	C27	-173.6(3)
N1	C13	C14	C15	179.0(3)	N2	C29	C30	C31	-171.9(2)
C1	N1	C5	C6	61.8(3)	C17	N2	C21	C22	-71.2(3)
C1	N1	C9	C10	179.2(2)	C17	N2	C25	C26	179.5(3)
C1	N1	C13	C14	-67.7(3)	C17	N2	C29	C30	61.0(3)
C1	C2	C3	C4	-178.7(3)	C17	C18	C19	C20	-173.4(3)
C5	N1	C1	C2	49.0(3)	C21	N2	C17	C18	-69.5(3)
C5	N1	C9	C10	-60.6(3)	C21	N2	C25	C26	59.0(3)
C5	N1	C13	C14	171.9(3)	C21	N2	C29	C30	-178.5(2)
C5	C6	C7	C8	-174.4(3)	C21	C22	C23	C24	-179.8(3)
C9	N1	C1	C2	169.4(2)	C25	N2	C17	C18	169.8(3)
C9	N1	C5	C6	-56.9(3)	C25	N2	C21	C22	47.1(3)
C9	N1	C13	C14	51.0(3)	C25	N2	C29	C30	-57.6(3)
C9	C10	C11	C12	-176.5(3)	C25	C26	C27	C28	-178.5(3)
C13	N1	C1	C2	-70.0(3)	C29	N2	C17	C18	48.8(3)
C13	N1	C5	C6	-177.8(3)	C29	N2	C21	C22	168.1(2)
C13	N1	C9	C10	58.8(3)	C29	N2	C25	C26	-59.6(3)
C13	C14	C15	C16	-177.5(3)	C29	C30	C31	C32	-173.8(3)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for pk025.

Atom	*x*	*y*	*z*	U(eq)
H1A	414.68	9321.75	6226.12	14
H1B	133.46	10362.97	6281.58	14
H2A	3060.9	9327.81	5863.3	16
H2B	2682.78	10380.9	5812.61	16
H3A	1509.2	10366.02	4657.33	18
H3B	1848.08	9313.31	4715.74	18
H4A	3646.04	9868.97	3525.34	28
H4B	4453.81	9327.32	4237.58	28
H4C	4131.41	10379	4196.63	28
H5A	1672.76	10218.78	8196.54	14
H5B	2684.92	10226.85	7207.48	14
H6A	-5.93	11432.02	7850.87	16
H6B	854.41	11408.02	6827.87	16
H7A	2219.8	11743.33	8153.38	18
H7B	2963.38	11802.03	7114.47	18
H8A	1094.64	13017.68	6936.68	28
H8B	1942.37	13226.37	7618.87	28
H8C	330.16	12954.83	7971.79	28
H9A	-1286.06	10449.76	7723.48	14
H9B	-1341.35	9433.1	7703.35	14
H10A	-616.63	10185.06	9070.6	16
H10B	-558.52	9151.24	9042.65	16

H11A	-3236.02	10432.8	9131.7	19
H11B	-3155.15	9392.1	9160.98	19
H12A	-2699.17	10314.1	10504.97	30
H12B	-2444.57	9264.27	10524.64	30
H12C	-4079.6	9828.8	10599.52	30
H13A	2586.27	8651.34	7155.03	14
H13B	1707.43	8730.26	8167.51	14
H14A	629.48	8045.65	6901.21	16
H14B	-229.18	8112.11	7922.21	16
H15A	1752.36	7099.19	8321.19	16
H15B	2665.01	7053.15	7307.77	16
H16A	799.85	6406.6	7001.36	27
H16B	-131.27	6463.37	8009.18	27
H16C	1447.9	5820.85	7755.77	27
H17A	8333.66	5520.49	6232.11	13
H17B	8568.12	4472.61	6306.26	13
H18A	6174.4	5552.37	5693.89	16
H18B	6553.21	4500.25	5699.83	16
H19A	8832.84	4549.73	4641.45	15
H19B	8609.27	5593.54	4687.76	15
H20A	6509.94	5767.42	4080.59	27
H20B	6770.57	4725.34	4016.86	27
H20C	7977.4	5278.76	3421.75	27
H21A	5316.68	6097.82	6971.79	13
H21B	5058.73	5897.61	8009.43	13
H22A	7453.56	6726.59	6956.83	15

H22B	7053.76	6586.9	8009.88	15
H23A	4696.91	7531.89	8103.16	15
H23B	5092.04	7669.77	7050.71	15
H24A	6777.95	8210.97	8098.17	29
H24B	5567.55	8875.68	7696.32	29
H24C	7064.33	8395.13	7054.43	29
H25A	8503.28	4257.49	7770.11	15
H25B	8541.14	5280.93	7752.33	15
H26A	6553.43	4352.76	9049.06	15
H26B	6411.3	5400.34	9007.51	15
H27A	9014.94	4190.02	9250.82	19
H27B	8838.86	5236.65	9233.14	19
H28A	8383.31	4614.52	10695.18	31
H28B	7022.59	4232.42	10549.12	31
H28C	6930.76	5271.86	10540.45	31
H29A	5141.44	4420.86	8076.24	13
H29B	5135.93	4511.18	7060.21	13
H30A	7118.86	3232.28	7836.92	16
H30B	7336.12	3367.37	6791.95	16
H31A	4651.88	2911.04	7974.1	16
H31B	5011.96	2954.88	6920.83	16
H32A	6672.05	1711.97	7928.2	29
H32B	6998.22	1749.44	6878.17	29
H32C	5481.56	1475.53	7480.06	29

CCDC 1534357 (PK023) $[NBu_4][I_5]$ with ~5% $[NBu_4][I_4Cl]$

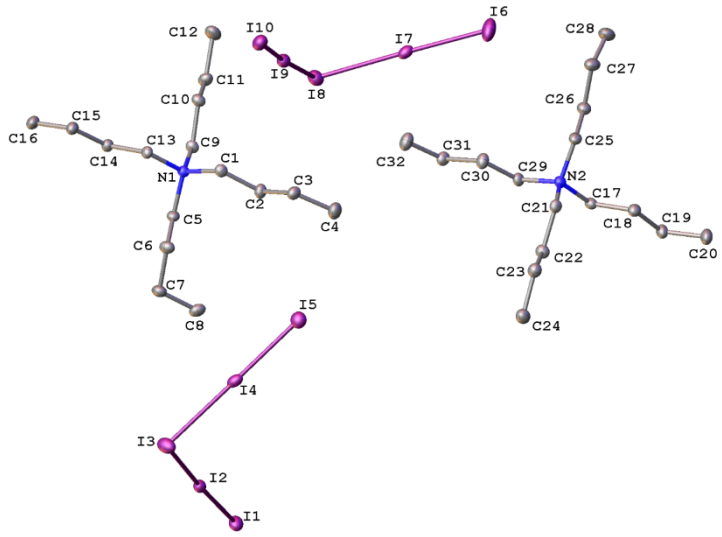


Fig. 1. ORTEP-style picture of the major disordered fragment.

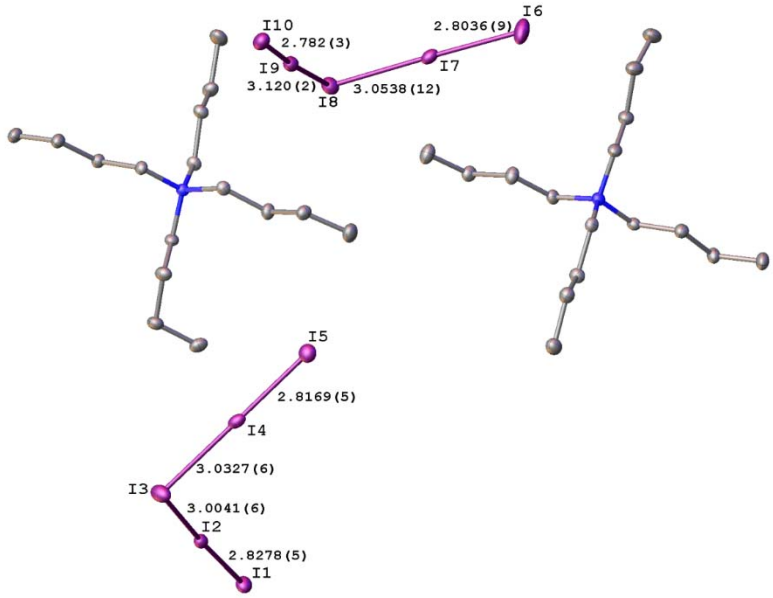


Fig. 2. I – I bond distances for the major disordered fragment.

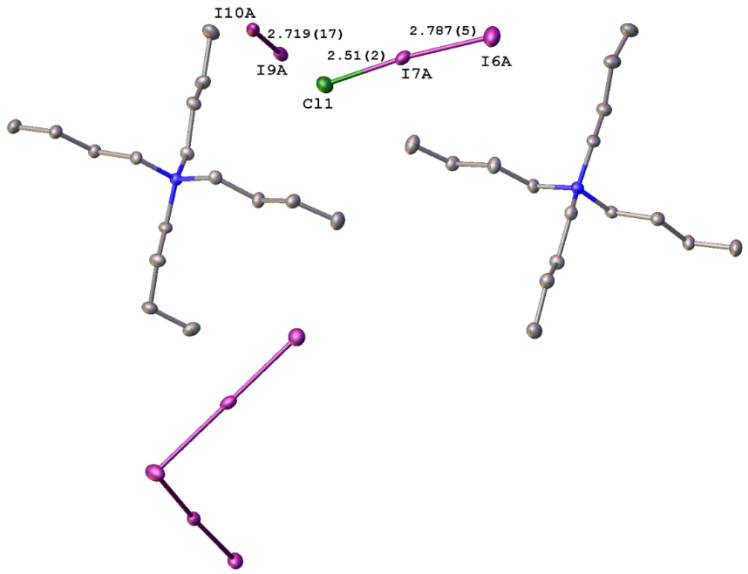


Fig. 3. Bond distances for the minor disordered fragment. The I9A – Cl1 distance is 3.402 A

Data Collection

A brown blade crystal of $C_{16}H_{36}NI_{4.93}Cl_{0.07}$ having approximate dimensions of 0.07 x 0.13 x 0.29 mm was mounted on a glass fiber. All measurements were made on a Bruker X8 APEX II diffractometer with cross-coupled multilayer optics Mo-Kα radiation.

The data were collected at a temperature of -173.0 $\pm$ 2^{o}C to a maximum 2θ value of 60.2^{o}. Data were collected in a series of ϕ and ω scans in 0.50^{o} oscillations with 5.0-second exposures. The crystal-to-detector distance was 39.79 mm.

Data Reduction

Of the 66866 reflections that were collected, 15334 were unique (R_{int} = 0.037); equivalent reflections were merged. Data were collected and integrated using the Bruker SAINT[1] software package. The linear absorption coefficient, μ, for Mo-Kα radiation is 58.70 cm^{-1}. Data were corrected for absorption effects using the multi-scan technique (SADABS[2]), with minimum and maximum transmission coefficients of 0.470 and 0.663, respectively. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[3]. The material crystallizes with two complete salt moieties in the asymmetric unit. Each contains an ordered NBu4+ cation, however one I_5^- appears to be a

mixture of I_5^- and I_2Cl^- and I_2. The I_2Cl^- fragment is present with an occupancy of 0.147(2), The final cycle of full-matrix least-squares refinement[4] on F^2 was based on 15334 reflections and 445 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1\ (I>2.00\sigma(I)) = \Sigma\ ||Fo| - |Fc||\ /\ \Sigma\ |Fo| = 0.031$$

$$wR2\ (all\ data) = [\ \Sigma\ (\ w\ (Fo^2 - Fc^2)^2\ )/\ \Sigma\ w(Fo^2)^2]^{1/2} = 0.055$$

The standard deviation of an observation of unit weight[5] was 1.10. The weighting scheme was based on counting statistics. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.22 and –1.09 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All refinements were performed using the SHELXL-2014[10] via the Olex2[11] interface.

References

(1) SAINT. Version 8.34A. Bruker AXS Inc., Madison, Wisconsin, USA. (1997-2013).

(2) SADABS 2014/5 - Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Crystallogr.* **48**.

(3) SHELXT: Sheldrick, G. M.; Acta Cryst., A71, 3-8 (2015).

(4) Least Squares function minimized:

$$\Sigma w(F_o^2 - F_c^2)^2$$

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

$$[\Sigma w(F_o^2 - F_c^2)^2/(N_o - N_v)]^{1/2}$$

where: N_o = number of observations

N_v = number of variables

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

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

(8) 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).

(9) 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).

(10) SHELXL-2014 - Sheldrick, G. M.; Acta Cryst., C71, 3-8 (2015).

(11) OLEX2 – V1.2.8 – Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H., OLEX2: A complete structure solution, refinement and analysis program (2009). J. Appl. Cryst., 42, 339-341.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{16}H_{36}NI_{4.93}Cl_{0.07}$
Formula Weight	870.21
Crystal Colour, Habit	brown, blade
Crystal Dimensions	0.07 x 0.13 x 0.29 mm
Crystal System	monoclinic
Lattice Type	primitive
Lattice Parameters	a = 19.948(3) Å
	b = 28.361(5) Å
	c = 9.3012(16) Å
	$\alpha = 90^o$
	$\beta = 96.590(4)^o$
	$\gamma = 90^o$
	V = 5227.3(15) $Å^3$
Space Group	$P\ 2_1/c$ (#14)
Z value	8
D_{calc}	2.212 g/cm^3
F_{000}	3211.00

μ(MoKα)	58.70 cm^{-1}

B. Intensity Measurements

Diffractometer	Bruker X8 APEX II
Radiation	MoKα (λ = 0.71073 Å)
	graphite monochromated
Data Images	1166 exposures @ 5.0 seconds
Detector Position	39.79 mm
$2\theta_{max}$	60.2^{o}
No. of Reflections Measured	Total: 66866
	Unique: 15334 (R_{int} = 0.037)
Corrections	Absorption (T_{min} = 0.470, T_{max}= 0.663)
	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (XT)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w=1/(\sigma^2(Fo^2)+(0.0060P)^2 + 12.0155P)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>0.00σ(I))	15334
No. Variables	445
Reflection/Parameter Ratio	34.46
Residuals (refined on F^2, all data): R1; wR2	0.040; 0.055
Goodness of Fit Indicator	1.10
No. Observations (I>2.00σ(I))	13136
Residuals (refined on F^2): R1; wR2	0.031; 0.053
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	1.22 $e^-/Å^3$
Minimum peak in Final Diff. Map	-1.09 $e^-/Å^3$

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for pk023. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
N1	6209.4(13)	3851.4(10)	6756(3)	13.8(5)
C1	5686.4(16)	3521.8(12)	5980(3)	16.3(6)
C2	5348.7(16)	3184.5(12)	6940(3)	19.2(7)
C3	4904.7(17)	2834.3(12)	6026(4)	20.1(7)
C4	4522.3(19)	2509.7(14)	6944(4)	28.4(8)
C5	6752.7(15)	3576.1(12)	7689(3)	16.4(6)
C6	7102.9(17)	3191.7(13)	6919(4)	21.2(7)
C7	7608.5(17)	2923.8(13)	7980(4)	22.7(7)
C8	7279.3(18)	2624.8(12)	9056(4)	23.0(7)
C9	5885.4(16)	4182.8(12)	7758(3)	16.0(6)
C10	5380.3(16)	4530.5(12)	7035(3)	18.7(7)
C11	5117.8(17)	4848.6(12)	8172(4)	21.5(7)
C12	4596.0(18)	5196.9(14)	7507(5)	29.6(8)
C13	6516.0(15)	4129.0(12)	5587(3)	15.2(6)
C14	6993.2(16)	4522.7(12)	6143(3)	17.0(6)
C15	7299.1(16)	4762.9(12)	4907(4)	19.8(7)
C16	7787.3(17)	5145.4(13)	5476(4)	23.9(7)
N2	1218.9(13)	1198.7(10)	5530(3)	15.6(5)
C17	1012.7(15)	954.9(12)	6866(3)	15.6(6)
C18	483.6(16)	573.4(12)	6596(4)	19.6(7)
C19	316.8(16)	364.8(13)	8022(4)	20.7(7)
C20	-236.9(18)	-4.2(14)	7784(4)	27.6(8)

C21	1480.0(16)	842.1(12)	4501(3)	16.5(6)
C22	2067.9(16)	538.2(13)	5138(3)	19.6(7)
C23	2322.1(17)	238.7(13)	3951(4)	22.1(7)
C24	2894.5(18)	-86.5(13)	4538(4)	26.2(8)
C25	619.7(16)	1440.9(12)	4676(4)	19.3(7)
C26	247.5(17)	1804.8(13)	5492(4)	21.9(7)
C27	-264.2(18)	2070.1(14)	4434(4)	26.9(8)
C28	-718.1(19)	2391.0(15)	5206(5)	34.2(9)
C29	1765.1(15)	1556.3(12)	6055(3)	16.9(6)
C30	2067.8(17)	1822.9(13)	4882(4)	21.4(7)
C31	2587.2(17)	2182.1(12)	5534(3)	19.3(7)
C32	2914.2(19)	2437.6(14)	4364(4)	27.8(8)
I1	8707.2(2)	823.8(2)	14071.4(2)	25.04(5)
I2	8828.7(2)	1058.9(2)	11158.2(2)	18.97(5)
I3	8878.3(2)	1282.7(2)	8015.2(3)	31.13(6)
I4	7373.5(2)	1415.7(2)	7210.6(2)	20.76(5)
I5	5973.4(2)	1565.2(2)	6607.9(3)	26.93(6)
I6	830.9(4)	3199.8(4)	5331.1(10)	41.14(17)
I7	2152.5(3)	3551.1(4)	5754.7(10)	21.54(12)
I8	3597.7(5)	3921.1(2)	6276.3(10)	27.17(14)
I9	3836.0(14)	3881.4(11)	3021.6(19)	23.4(2)
I10	3956.9(13)	3834.6(12)	75(3)	27.9(3)
I6A	1064.6(19)	3181(2)	5448(6)	36.3(8)
I7A	2362.1(19)	3559(2)	5840(6)	21.3(7)
Cl1	3569(12)	3827(6)	6290(30)	27.17(14)
I9A	3801(8)	3871(6)	2724(11)	21.6(13)

I10A	3895(7)	3852(6)	-169(15)	26.1(17)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for pk023. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	15.0(12)	14.9(13)	11.4(11)	-1.2(10)	1.3(9)	0.3(10)
C1	17.6(14)	17.9(17)	13.3(14)	-3.6(12)	1.7(11)	-0.2(12)
C2	19.7(15)	20.1(17)	17.9(15)	-1.8(13)	1.8(12)	-4.8(13)
C3	22.3(16)	17.6(17)	19.5(16)	-0.3(13)	-0.8(12)	-2.3(13)
C4	27.0(18)	24(2)	35(2)	-1.0(16)	5.1(15)	-7.0(15)
C5	16.3(14)	17.0(16)	15.2(14)	2.1(12)	-1.1(11)	1.3(12)
C6	22.1(16)	23.2(19)	18.8(16)	1.3(14)	4.8(13)	5.7(14)
C7	19.4(16)	23.2(19)	25.3(17)	3.0(14)	1.6(13)	6.3(14)
C8	31.4(18)	16.1(17)	21.3(16)	-0.3(14)	2.3(14)	2.9(14)
C9	17.1(14)	18.0(17)	13.1(14)	-4.1(12)	3.0(11)	1.7(12)
C10	17.3(15)	21.6(18)	16.8(15)	-1.2(13)	0.4(12)	2.0(13)
C11	25.7(17)	17.1(17)	22.8(17)	-0.4(13)	7.7(13)	2.8(14)
C12	21.3(17)	26(2)	42(2)	-4.0(17)	5.5(16)	6.3(15)
C13	16.2(14)	17.7(16)	11.8(13)	1.3(12)	2.9(11)	-1.0(12)
C14	17.5(15)	16.1(16)	17.6(15)	-0.2(12)	2.3(12)	-1.3(12)
C15	19.2(15)	18.4(17)	21.3(16)	3.2(13)	0.1(12)	-2.3(13)
C16	22.8(17)	17.7(18)	30.8(19)	5.0(15)	1.4(14)	-2.8(14)
N2	14.9(12)	17.2(14)	14.4(12)	-0.5(10)	0.1(9)	0(1)
C17	15.1(14)	18.9(17)	12.9(14)	1.0(12)	1.6(11)	2.0(12)
C18	18.7(15)	20.7(18)	18.7(15)	2.3(13)	-0.7(12)	-0.8(13)

C19	16.8(15)	26.0(19)	19.2(15)	4.7(14)	2.1(12)	-2.8(13)
C20	20.6(17)	30(2)	33(2)	7.8(16)	3.0(14)	-6.4(15)
C21	18.2(15)	17.3(16)	13.6(14)	-2.8(12)	0.6(11)	-3.2(12)
C22	20.5(16)	22.3(18)	16.1(15)	-1.7(13)	2.4(12)	0.9(13)
C23	23.2(17)	21.6(18)	21.8(16)	-0.4(14)	3.5(13)	-1.1(14)
C24	23.0(17)	23.2(19)	32(2)	-1.9(15)	2.6(14)	-0.5(15)
C25	16.8(15)	20.3(18)	19.7(15)	4.3(13)	-2.7(12)	-0.3(13)
C26	19.7(16)	22.4(18)	22.8(16)	2.9(14)	-0.6(13)	1.6(14)
C27	24.8(18)	22.0(19)	31.7(19)	2.6(15)	-5.9(15)	1.6(15)
C28	23.6(19)	26(2)	52(3)	5.7(19)	1.0(17)	7.2(16)
C29	15.4(14)	18.1(17)	16.7(14)	-2.2(12)	-0.2(11)	-2.9(12)
C30	23.2(16)	24.6(19)	16.4(15)	0.0(14)	2.6(12)	-6.3(14)
C31	21.7(16)	19.0(17)	16.6(15)	-2.2(13)	-1.1(12)	-3.8(13)
C32	32.5(19)	27(2)	24.7(18)	0.3(15)	6.0(15)	-9.7(16)
I1	21.75(11)	31.84(14)	20.63(10)	4.46(9)	-1.41(8)	-2.02(9)
I2	17.32(9)	19.27(11)	19.94(10)	-0.23(8)	0.47(7)	-1.05(8)
I3	29.33(12)	43.44(16)	21.86(11)	6.26(11)	8.23(9)	9.57(11)
I4	32.47(12)	15.42(11)	15.32(9)	1.19(8)	6.70(8)	2.18(9)
I5	29.43(12)	31.73(14)	20.21(11)	0.05(9)	5.36(9)	-1.09(10)
I6	42.0(4)	49.7(3)	31.4(2)	6.6(2)	2.6(3)	-23.2(4)
I7	31.8(3)	16.45(18)	16.49(18)	1.69(13)	3.2(3)	-1.1(3)
I8	23.50(17)	31.9(3)	25.51(14)	-1.6(3)	0.08(11)	1.1(3)
I9	20.2(4)	23.2(4)	26.2(6)	1.9(5)	-0.1(4)	-0.6(3)
I10	30.0(6)	24.2(5)	30.7(5)	3.1(3)	8.2(3)	-0.5(4)
I6A	34.2(19)	42.2(14)	32.1(14)	7.8(10)	2.7(16)	-8.2(19)
I7A	29.5(18)	16(1)	18.5(11)	1.4(7)	2.6(15)	-1.8(17)

Cl1	23.50(17)	31.9(3)	25.51(14)	-1.6(3)	0.08(11)	1.1(3)
I9A	15.9(13)	20.0(14)	27(4)	-4(3)	-4(3)	-0.7(9)
I10A	17.4(17)	17.9(16)	45(5)	5(3)	11(3)	3.4(13)

Table 4 Bond Lengths for pk023.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C1	1.520(4)	C19	C20	1.519(5)
N1	C5	1.524(4)	C21	C22	1.520(4)
N1	C9	1.519(4)	C22	C23	1.526(5)
N1	C13	1.526(4)	C23	C24	1.520(5)
C1	C2	1.518(4)	C25	C26	1.524(5)
C2	C3	1.523(4)	C26	C27	1.532(5)
C3	C4	1.519(5)	C27	C28	1.521(5)
C5	C6	1.518(5)	C29	C30	1.510(5)
C6	C7	1.530(5)	C30	C31	1.528(5)
C7	C8	1.518(5)	C31	C32	1.516(5)
C9	C10	1.512(4)	I1	I2	2.8278(6)
C10	C11	1.527(5)	I2	I3	3.0041(6)
C11	C12	1.515(5)	I3	I4	3.0327(6)
C13	C14	1.519(4)	I4	I5	2.8169(6)
C14	C15	1.523(4)	I6	I7	2.8036(9)
C15	C16	1.512(5)	I7	I8	3.0538(12)
N2	C17	1.520(4)	I8	I9	3.120(2)
N2	C21	1.524(4)	I9	I10	2.782(3)

N2	C25	1.521(4)	I6A	I7A	2.787(5)
N2	C29	1.527(4)	I7A	Cl1	2.51(2)
C17	C18	1.512(4)	I9A	I10A	2.719(17)
C18	C19	1.524(5)			

Table 5 Bond Angles for pk023.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	N1	C5	111.1(2)	C25	N2	C21	106.1(2)
C1	N1	C13	106.7(2)	C25	N2	C29	110.9(3)
C5	N1	C13	110.7(2)	C18	C17	N2	116.0(3)
C9	N1	C1	110.8(2)	C17	C18	C19	110.5(3)
C9	N1	C5	107.0(2)	C20	C19	C18	111.6(3)
C9	N1	C13	110.6(2)	C22	C21	N2	115.8(2)
C2	C1	N1	115.8(2)	C21	C22	C23	109.8(3)
C1	C2	C3	110.6(3)	C24	C23	C22	112.1(3)
C4	C3	C2	112.3(3)	N2	C25	C26	116.4(3)
C6	C5	N1	115.8(3)	C25	C26	C27	109.9(3)
C5	C6	C7	110.9(3)	C28	C27	C26	112.3(3)
C8	C7	C6	113.6(3)	C30	C29	N2	115.5(3)
C10	C9	N1	116.1(3)	C29	C30	C31	110.9(3)
C9	C10	C11	110.0(3)	C32	C31	C30	111.2(3)
C12	C11	C10	112.1(3)	I1	I2	I3	176.643(11)
C14	C13	N1	115.0(2)	I2	I3	I4	97.135(10)
C13	C14	C15	111.2(3)	I5	I4	I3	176.867(11)

C16	C15	C14	110.7(3)	I6	I7	I8	178.75(5)
C17	N2	C21	110.8(2)	I7	I8	I9	94.56(6)
C17	N2	C25	111.2(2)	I10	I9	I8	176.18(12)
C17	N2	C29	106.9(2)	Cl1	I7A	I6A	174.6(5)
C21	N2	C29	111.0(2)				

Table 6 Torsion Angles for pk023.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
N1	C1	C2	C3	-172.9(3)	N2	C17	C18	C19	-179.1(3)
N1	C5	C6	C7	-176.9(3)	N2	C21	C22	C23	-173.9(3)
N1	C9	C10	C11	-178.1(3)	N2	C25	C26	C27	171.0(3)
N1	C13	C14	C15	177.2(3)	N2	C29	C30	C31	177.9(3)
C1	N1	C5	C6	53.9(4)	C17	N2	C21	C22	-56.6(3)
C1	N1	C9	C10	-65.2(3)	C17	N2	C25	C26	57.1(4)
C1	N1	C13	C14	172.9(3)	C17	N2	C29	C30	177.1(3)
C1	C2	C3	C4	-176.4(3)	C17	C18	C19	C20	177.4(3)
C5	N1	C1	C2	55.2(3)	C21	N2	C17	C18	-57.4(3)
C5	N1	C9	C10	173.5(3)	C21	N2	C25	C26	177.7(3)
C5	N1	C13	C14	-66.1(3)	C21	N2	C29	C30	56.1(4)
C5	C6	C7	C8	68.8(4)	C21	C22	C23	C24	-177.8(3)
C9	N1	C1	C2	-63.6(3)	C25	N2	C17	C18	60.4(4)
C9	N1	C5	C6	175.0(3)	C25	N2	C21	C22	-177.5(3)
C9	N1	C13	C14	52.3(3)	C25	N2	C29	C30	-61.5(4)
C9	C10	C11	C12	-178.6(3)	C25	C26	C27	C28	172.2(3)

C13	N1	C1	C2	175.9(3)	C29	N2	C17	C18	-178.5(3)
C13	N1	C5	C6	-64.5(3)	C29	N2	C21	C22	62.0(3)
C13	N1	C9	C10	52.9(3)	C29	N2	C25	C26	-61.7(4)
C13	C14	C15	C16	-178.5(3)	C29	C30	C31	C32	177.7(3)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for pk023.

Atom	*x*	*y*	*z*	U(eq)
H1A	5339	3712	5441	20
H1B	5902	3338	5284	20
H2A	5076	3362	7550	23
H2B	5691	3015	7563	23
H3A	4584	3006	5358	24
H3B	5184	2646	5461	24
H4A	4250	2296	6327	43
H4B	4238	2694	7491	43
H4C	4838	2334	7594	43
H5A	7092	3797	8106	20
H5B	6549	3433	8482	20
H6A	7337	3331	6165	25
H6B	6769	2973	6465	25
H7A	7880	2722	7436	27
H7B	7909	3149	8508	27
H8A	6952	2419	8544	34
H8B	7059	2826	9687	34

H8C	7618	2440	9618	34
H9A	5661	3994	8429	19
H9B	6241	4359	8321	19
H10A	5007	4362	6508	22
H10B	5593	4721	6349	22
H11A	4919	4655	8871	26
H11B	5493	5020	8682	26
H12A	4790	5389	6812	44
H12B	4452	5394	8252	44
H12C	4215	5029	7035	44
H13A	6759	3911	5031	18
H13B	6152	4264	4934	18
H14A	7351	4394	6825	20
H14B	6749	4754	6651	20
H15A	7534	4531	4383	24
H15B	6942	4899	4239	24
H16A	7999	5277	4690	36
H16B	8126	5014	6178	36
H16C	7547	5389	5920	36
H17A	1413	817	7395	19
H17B	845	1192	7485	19
H18A	649	327	6006	23
H18B	78	705	6071	23
H19A	719	222	8525	25
H19B	173	615	8628	25
H20A	-93	-255	7196	41

H20B	-639	138	7304	41
H20C	-329	-129	8701	41
H21A	1110	636	4143	20
H21B	1617	1012	3677	20
H22A	1924	335	5884	24
H22B	2430	738	5578	24
H23A	1952	51	3486	27
H23B	2477	444	3224	27
H24A	3046	-264	3757	39
H24B	2738	-299	5230	39
H24C	3262	98	4999	39
H25A	299	1200	4310	23
H25B	777	1595	3846	23
H26A	568	2026	5978	26
H26B	16	1648	6220	26
H27A	-24	2258	3787	32
H27B	-541	1844	3852	32
H28A	-1000	2572	4505	51
H28B	-445	2600	5841	51
H28C	-996	2203	5760	51
H29A	1574	1783	6676	20
H29B	2125	1392	6641	20
H30A	1713	1984	4273	26
H30B	2283	1603	4282	26
H31A	2368	2410	6103	23
H31B	2932	2022	6174	23

H32A	2575	2603	3743	42
H32B	3135	2213	3805	42
H32C	3240	2659	4803	42

Table 8 Atomic Occupancy for pk023.

Atom	***Occupancy***	**Atom**	***Occupancy***	**Atom**	***Occupancy***
I6	0.8524(16)	I7	0.8524(16)	I8	0.8524(16)
I9	0.8524(16)	I10	0.8524(16)	I6A	0.1476(16)
I7A	0.1476(16)	Cl1	0.1476(16)	I9A	0.1476(16)
I10A	0.1476(16)				