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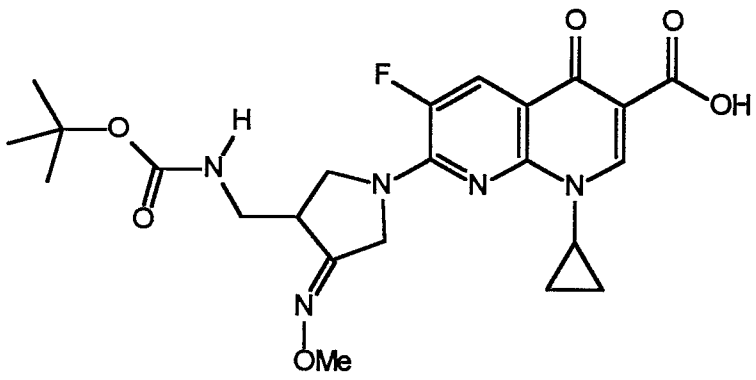
X-ray data of *t*-BOC analog of LB20304



t-BOC analog of LB20304

Data collection and processing

White plate crystals were grown in a solution of *t*-BOC analog of LB20304 in *N,N*-dimethylformamide at -5 °C. A plate was chosen (size of 0.5x0.5x0.1 mm) and mounted on the end of a glass fiber. Data were collected at ambient temperature on a Enlaf-Nonius CAD4 diffractometer using graphite-monochromatized Mo Kα radiation.

Crystal orientation and cell parameters were determined from 25 machine centered reflections with maximum 2θ of 50 °. Three check reflections, monitered every 100 reflections showed no significant decay over the course of data collection.

Data sets were processed using the MOLEN program package (Enlaf-Nonius, Delft, the Netherlands). Empirical absorption correction was applied.

Structure Solution and Refinement

All non-hydrogen atoms of the molecule were located by use of the direct method program MULTAN. The carboxylic acid hydrogen atom was located in the difference electron density map. All the other hydrogen atoms were generated at the ideal positions and were included in the structure factor calculation but not refined. Atom scattering factors were taken from the tabulation of Cromer and Waber.[1] All the heavy atoms were then allowed to refine isotropically.

Final R factors and other data are included in Table 1. Table of experimental details (Table 1), table of positional parameters (Table 2), table of general displacement parameter expressions (Table 3), table of bond distances (Table 4), table of bond angles (Table 5) are also listed. Standard deviations are given in parentheses.

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1. Cromer, D. T.; Waber, J. T. In *International tables for X-ray Crystallography*; Ibers, J. A.; Hamilton, H. C., Eds.; Kynoch Press: Birmingham, England, 1974; Vol 4.

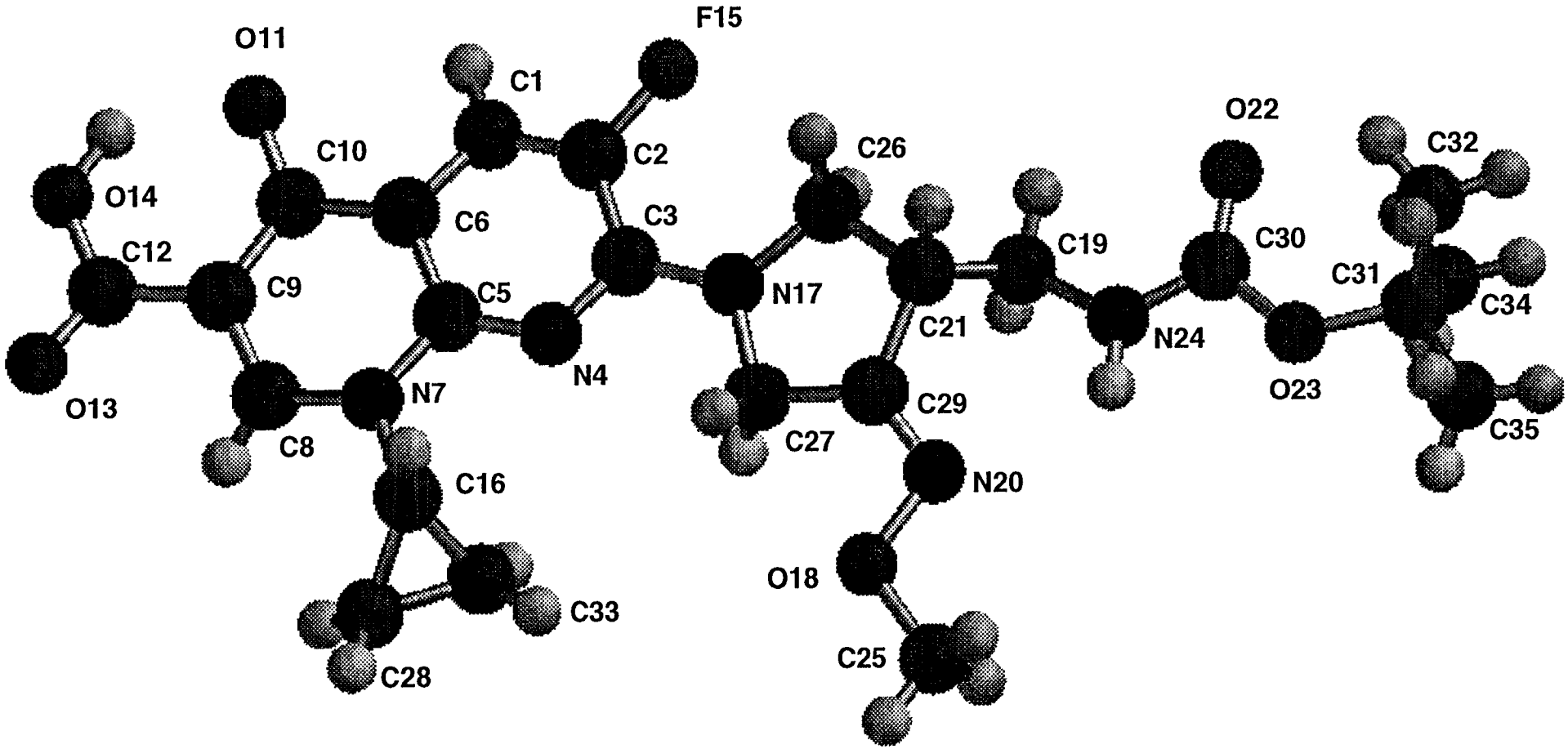
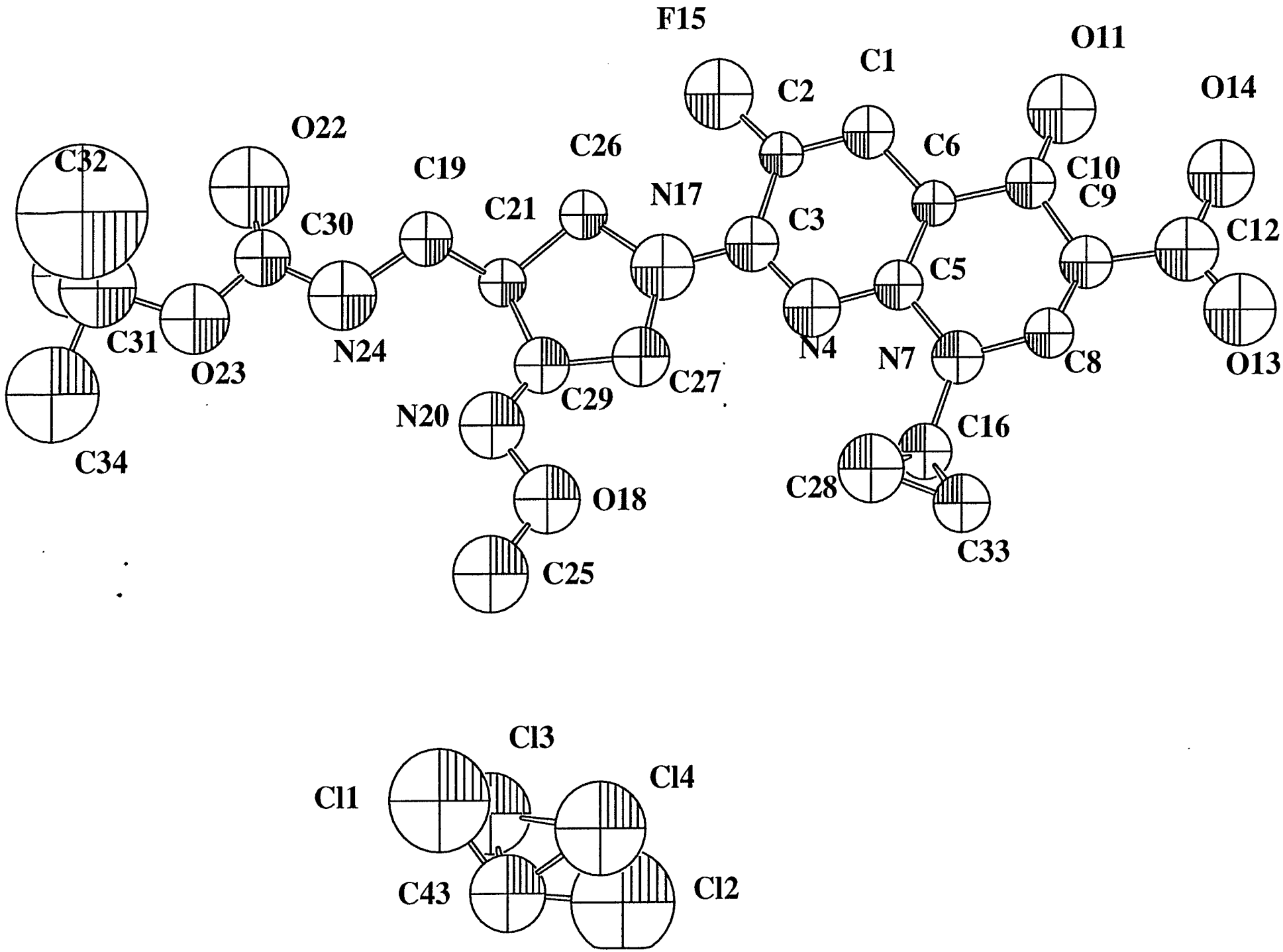
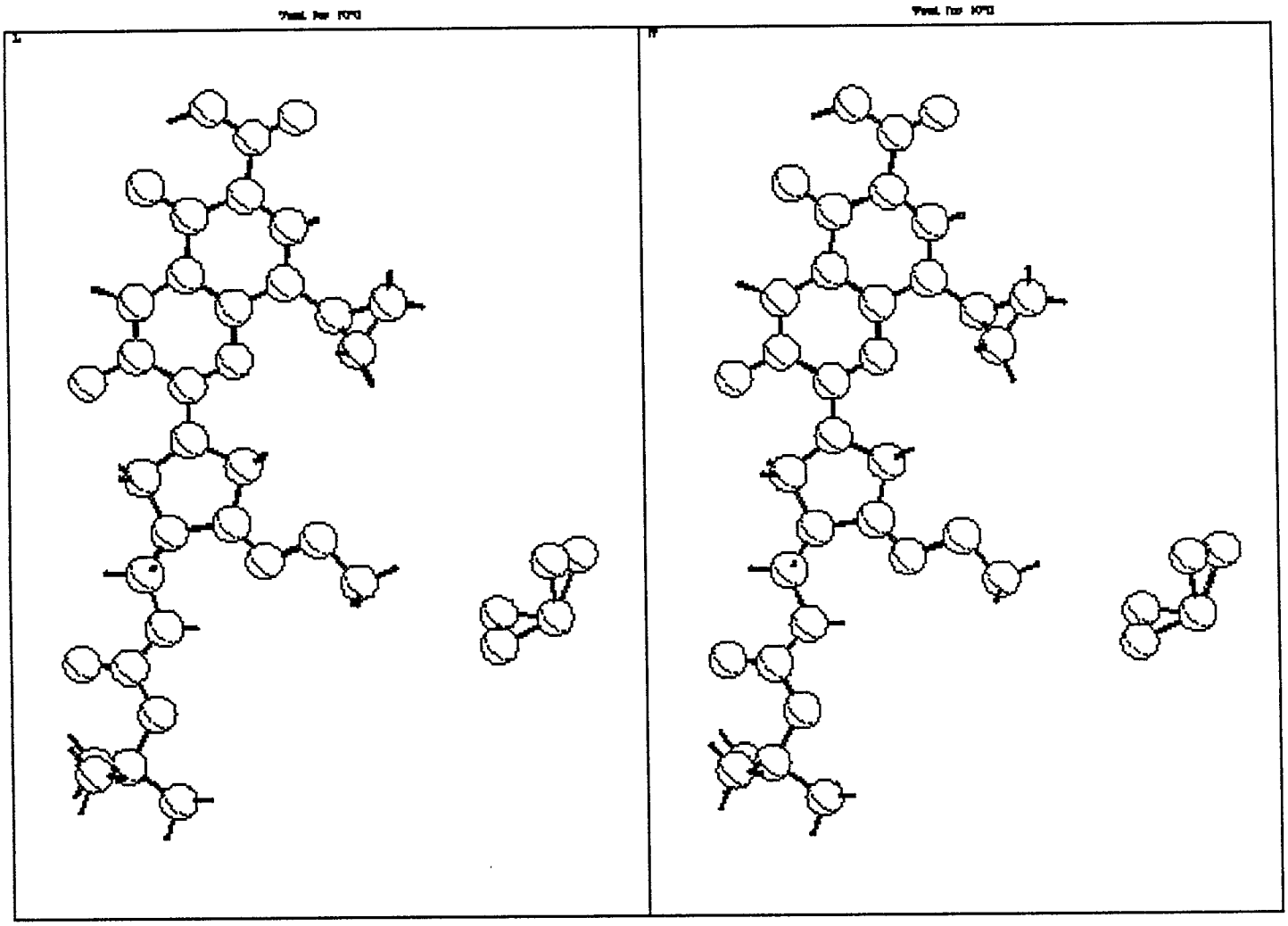


Figure 1. X–ray structure of t–Boc analog of LB20304


F15
C1
O11
C2
O14
O22
C6
C32
C19
C26
C10
C9
C30
C21
N17
C3
C12
C5
C31
N24
N4
N7
C8
O13
O23
C29
C27
C34
N20
C16
C28
O18
C33
C25
Cl3
Cl4
Cl1
C43
Cl2



Stereoview

ABSTRACT

F.W.= 573.43; Space Group: C2 (# 5); a = 21.496(9)Å, b = 7.141(2)Å, c = 18.502(8)Å, β = 104.56(2) °; V = 2749(3)Å3; Z = 4; Dcalc = 1.386g/cm³; Radiation = MoKα (λ = 0.71073Å); μ = 2.9cm-1; F(000) = 1196; Temperature = 23±1 °; Final R = 0.116; Number of Unique Reflections= 1290.

Table of Experimental Details

Crystal Description:	white ,plate 0.50 x 0.50 x 0.10 mm
Instrument:	Enraf-Nonius CAD4 diffractometer
Corrections:	Lorentz-polarization
	Empirical absorption (from91.15 to99.82 on I)
Maximum 2θ :	50.0 °
hkl ranges:	h= 0- 22 k= 0- 8 l=-20- 21
No. of refl. measured:	2048 total, 1989 unique
Reflections included:	1290 with Fo >1.0σ(Fo)
Solution:	Direct methods
Hydrogen atoms:	Fixed and included in the structure factor
Parameters refined:	276
Unweighted agreement factor:	0.116
Weighted agreement factor:	0.115
Esd of obs. of unit weight:	11.0
Convergence, largest shift:	0.24
Minimization function:	w(|Fo|-|Fc|)2
Least-squares weights:	4Fo/σ(Fo)
Instrument instability factor:	0.060
High peak in final diff. map:	0.48 (9) e/Å3
Low peak in final diff. map:	0.00 (9) e/Å3

Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(Å2)

CL1	0.3296(9)	0.215(4)	0.621(1)	18.1(5)*
CL2	0.469(1)	0.154(5)	0.610(1)	19.6(6)*
CL3	0.351(2)	0.117(7)	0.594(2)	11.3(9)*
CL4	0.431(3)	0.27(1)	0.582(3)	15(2)*
F15	0.0832(2)	0.177(3)	-0.0316(3)	8.3(3)
O11	0.2619(3)	0.180(4)	-0.1645(4)	8.0(3)
O13	0.4573(3)	0.178(4)	-0.0893(5)	9.0(4)
O14	0.3750(3)	0.185(3)	-0.1824(4)	7.6(3)
O18	0.2165(3)	0.183(3)	0.3448(4)	7.8(3)
O22	-0.0977(4)	0.195(4)	0.2388(5)	11.0(4)
O23	-0.0533(3)	0.198(4)	0.3605(4)	8.4(3)
N4	0.2438(3)	0.165(3)	0.0869(4)	5.9(2)
N7	0.3428(3)	0.158(2)	0.0613(4)	4.8(2)
N17	0.1491(3)	0.183(4)	0.1227(4)	7.3(3)
N20	0.1497(4)	0.185(4)	0.3135(5)	7.4(3)
N24	0.0084(4)	0.211(4)	0.2834(5)	8.1(4)
C1	0.1809(5)	0.200(3)	-0.0632(6)	4.8(2)*
C2	0.1487(4)	0.192(2)	-0.0107(4)	3.5(1)*
C3	0.1814(4)	0.184(2)	0.0676(5)	5.1(2)
C5	0.2764(4)	0.179(3)	0.0353(5)	4.2(2)*
C6	0.2485(3)	0.169(2)	-0.0424(4)	3.5(2)
C8	0.3799(4)	0.205(3)	0.0122(5)	4.2(2)*
C9	0.3561(5)	0.176(3)	-0.0611(6)	4.7(2)*
C10	0.2880(4)	0.192(3)	-0.0944(5)	4.3(2)*
C12	0.3998(4)	0.191(5)	-0.1105(5)	6.9(4)
C16	0.3733(6)	0.123(2)	0.1400(7)	5.2(3)*
C19	0.0233(6)	0.250(2)	0.2117(7)	5.0(2)*
C21	0.0716(5)	0.129(2)	0.1940(5)	3.9(2)*
C25	0.2298(7)	0.192(7)	0.4259(7)	10.1(6)
C26	0.0792(4)	0.166(3)	0.1146(5)	4.1(2)*
C27	0.1875(4)	0.189(3)	0.1999(5)	6.0(3)
C28	0.3708(9)	0.261(4)	0.196(1)	7.8(4)*

C29	0.1399(4)	0.164(3)	0.2445(5)	5.4(3)
C30	-0.0515(4)	0.210(3)	0.2883(5)	5.4(3)
C31	-0.1142(4)	0.232(4)	0.3829(4)	10.5(6)
C32	-0.146(2)	0.382(8)	0.355(3)	31(1)
C33	0.4316(6)	0.234(3)	0.1796(7)	5.7(3)*
C34	-0.0880(7)	0.151(9)	0.4688(8)	15(1)
C35	-0.1534(6)	0.043(7)	0.353(1)	13(1)
C43	0.406(1)	0.143(6)	0.651(1)	10.2(6)*
H1	0.328	0.208	-0.206	6.0
H2	0.421	0.258	0.030	5.5
H3	0.159	0.221	-0.114	6.2
H4	0.052	-0.032	0.184	6.0
H5	0.042	0.190	0.326	10.4
H6	0.363	-0.007	0.132	6.7
H7	0.365	0.220	0.243	10.2
H8	0.348	0.373	0.182	10.2
H9	0.449	0.327	0.154	7.4
H10	0.466	0.174	0.215	7.4
H11	0.062	0.063	0.082	5.3
H12	0.058	0.276	0.095	5.3
H13	0.209	0.303	0.211	7.8
H14	0.218	0.088	0.209	7.8
H15	0.275	0.188	0.447	12.6
H16	0.213	0.304	0.440	12.6
H17	0.211	0.087	0.444	12.6
H18	-0.184	0.388	0.372	37.7
H19	-0.121	0.487	0.370	37.7
H20	-0.158	0.372	0.302	37.7
H21	-0.122	0.153	0.494	20.0
H22	-0.076	0.018	0.466	20.0
H23	-0.052	0.217	0.495	20.0
H24	-0.194	0.047	0.365	16.6
H25	-0.160	0.033	0.301	16.6

H26	-0.130	-0.064	0.377	16.6
H27	0.038	0.374	0.212	6.5
H28	-0.015	0.235	0.173	6.5

Starred atoms were refined isotropically.
Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as:
(4/3) * [a2*β11 + b2*β22 + c2*β33 + ac(cosβ)*β13]

Table of General Displacement Parameter Expressions - U's

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
F15	0.040(2)	0.22(1)	0.054(2)	-0.026(7)	0.013(2)	-0.032(7)
O11	0.064(3)	0.19(1)	0.051(3)	0.01(1)	0.021(2)	-0.01(1)
O13	0.064(3)	0.20(2)	0.089(4)	0.00(1)	0.012(2)	-0.00(1)
O14	0.077(3)	0.16(1)	0.066(3)	0.022(8)	0.041(2)	-0.008(8)
O18	0.053(3)	0.19(1)	0.052(3)	-0.023(8)	0.020(2)	0.020(8)
O22	0.062(3)	0.28(2)	0.077(4)	0.052(8)	0.024(3)	-0.02(1)
O23	0.065(3)	0.20(1)	0.061(3)	-0.046(8)	0.022(2)	-0.022(9)
N4	0.048(3)	0.14(1)	0.036(3)	-0.047(5)	0.019(2)	-0.011(7)
N7	0.038(3)	0.103(9)	0.043(3)	0.017(5)	0.015(2)	-0.013(6)
N17	0.044(3)	0.18(1)	0.055(3)	-0.019(9)	0.023(2)	-0.00(1)
N20	0.054(3)	0.17(1)	0.066(4)	0.049(7)	0.027(3)	0.005(9)
N24	0.057(3)	0.20(2)	0.062(4)	-0.012(9)	0.031(2)	0.032(8)
C3	0.062(3)	0.080(8)	0.058(4)	-0.066(5)	0.025(3)	-0.028(7)
C6	0.053(3)	0.033(6)	0.055(3)	0.004(6)	0.030(2)	-0.006(6)
C12	0.064(4)	0.16(2)	0.051(4)	0.02(1)	0.033(3)	0.01(1)
C25	0.095(6)	0.23(3)	0.061(5)	-0.05(2)	0.032(4)	0.00(2)
C27	0.057(3)	0.13(1)	0.045(3)	0.057(7)	0.024(3)	0.007(8)
C29	0.053(4)	0.10(1)	0.052(4)	-0.011(8)	0.020(3)	0.011(9)
C30	0.052(3)	0.10(1)	0.065(4)	0.013(7)	0.030(3)	0.026(7)
C31	0.051(3)	0.31(2)	0.049(3)	-0.077(8)	0.038(2)	-0.076(9)
C32	0.31(1)	0.56(4)	0.36(4)	0.36(1)	0.22(2)	0.25(3)
C34	0.124(7)	0.39(6)	0.095(6)	-0.03(3)	0.076(4)	0.02(2)

C35	0.054(4)	0.33(5)	0.123(8)	0.01(1)	0.053(4)	0.02(2)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2\{h^2a^2U(1,1) + k^2b^2U(2,2) + l^2c^2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2klbcU(2,3)\}]$ where a,b, and c are reciprocal lattice constants.

Table of Bond Distances in Angstroms

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
CL1	CL3	1.03(5)	N17	C26	1.48(1)
CL1	C43	1.67(3)	N17	C27	1.46(1)
CL2	CL4	1.19(7)	N20	C29	1.25(1)
CL2	C43	1.71(4)	N24	C19	1.47(2)
CL3	CL4	2.10(8)	N24	C30	1.31(1)
CL3	C43	1.37(4)	C1	C2	1.33(1)
CL4	C43	1.76(8)	C1	C6	1.42(1)
F15	C2	1.367(9)	C2	C3	1.44(1)
O11	C10	1.28(1)	C5	C6	1.41(1)
O13	C12	1.20(1)	C6	C10	1.44(1)
O14	C12	1.30(1)	C8	C9	1.34(1)
O18	N20	1.406(9)	C9	C10	1.44(1)
O18	C25	1.46(1)	C9	C12	1.47(2)
O22	C30	1.18(1)	C16	C28	1.44(3)
O23	C30	1.35(1)	C16	C33	1.50(2)
O23	C31	1.49(1)	C19	C21	1.45(2)
N4	C3	1.30(1)	C21	C26	1.54(1)
N4	C5	1.32(1)	C21	C29	1.55(1)
N7	C5	1.39(1)	C27	C29	1.48(1)
N7	C8	1.39(1)	C28	C33	1.43(3)
N7	C16	1.46(1)	C31	C32	1.31(6)
N17	C3	1.37(1)	C31	C34	1.65(3)
C31	C35	1.61(5)			

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
CL3	CL1	C43	55.(2)	C1	C2	C3	121.5(7)
CL4	CL2	C43	72.(4)	N4	C3	N17	118.3(7)
CL1	CL3	CL4	100.(4)	N4	C3	C2	119.1(8)
CL1	CL3	C43	87.(3)	N17	C3	C2	122.4(7)
CL4	CL3	C43	56.(3)	N4	C5	N7	115.1(8)
CL2	CL4	CL3	94.(5)	N4	C5	C6	124.3(8)
CL2	CL4	C43	68.(4)	N7	C5	C6	118.7(9)
CL3	CL4	C43	41.(2)	C1	C6	C5	114.2(8)
N20	O18	C25	109.9(8)	C1	C6	C10	122.2(8)
C30	O23	C31	120.7(8)	C5	C6	C10	120.1(7)
C3	N4	C5	119.2(8)	N7	C8	C9	119(1)
C5	N7	C8	117.0(8)	C8	C9	C10	121.(1)
C5	N7	C16	121.8(8)	C8	C9	C12	118.5(9)
C8	N7	C16	120.2(7)	C10	C9	C12	117.9(9)
C3	N17	C26	128.2(7)	O11	C10	C6	119.1(8)
C3	N17	C27	117.4(7)	O11	C10	C9	125(1)
C26	N17	C27	114.4(8)	C6	C10	C9	114.2(8)
O18	N20	C29	108.2(8)	O13	C12	O14	117.(1)
C19	N24	C30	120.4(9)	O13	C12	C9	124(1)
C2	C1	C6	118.7(9)	O14	C12	C9	118.1(8)
F15	C2	C1	119.1(7)	N7	C16	C28	121.(2)
F15	C2	C3	119.3(8)	N7	C16	C33	120.(1)
C28	C16	C33	58.(1)	O23	C31	C32	116.(3)
N24	C19	C21	115.(1)	O23	C31	C34	96.(1)
C19	C21	C26	112.(1)	O23	C31	C35	101.(2)
C19	C21	C29	113.(1)	C32	C31	C34	133.(3)
C26	C21	C29	103.7(8)	C32	C31	C35	112.(2)
N17	C26	C21	105.4(7)	C34	C31	C35	93.(3)
N17	C27	C29	104.0(7)	C16	C33	C28	59.(1)
C16	C28	C33	63.(1)	CL1	C43	CL2	131.(2)

N20	C29	C21	121.8(9)	CL1	C43	CL3	38.(2)
N20	C29	C27	126(1)	CL	C43	CL4	93.(3)
C21	C29	C27	111.6(8)	CL2	C43	CL3	107.(3)
O22	C30	O23	122.(1)	CL2	C43	CL4	40.(2)
O22	C30	N24	127.(1)	CL3	C43	CL4	83.(3)
O23	C30	N24	110.0(7)				

Numbers in parentheses are estimated standard deviations in the least significant digits.