SUPPORTING INFORMATION

Chloride Sensing via Suppression of Excited State Intramolecular Proton Transfer in Squaramides

*Mintu Porel, Vijayakumar Ramalingam, Maciej E. Domaradzki, Victor G. Young Jr., Vaidyanathan Ramamurthy and Rajeev S. Muthyala**

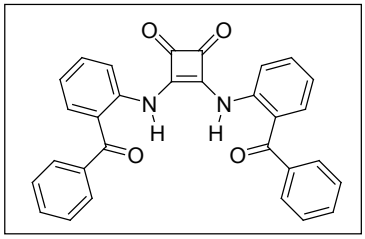
Center for Learning Innovation,
University of Minnesota Rochester,
111 S. Broadway
Rochester, MN 55904
muthy004@r.umn.edu

General Information:

Compounds **1**, **2** and **3** were prepared described earlier.[1] Melting points were recorded using Mel-Temp apparatus and are uncorrected. All tetra butyl ammonium salts were purchased from commercial sources and were used without further purification. HPLC grade acetonitrile and chloroform were used for all UV-Vis studies. ^1H and ^{13}C NMR were recorded at 400MHz and 100MHz, respectively. The chemical shifts are reported in ppm using the residual solvent peak as the internal standard. High resolution mass spectra were collected at the CUNY Mass Spectrometry Facility at Hunter College which is funded by NSF Grant 0521085 and NIH Grant 1S10RR022649-01.

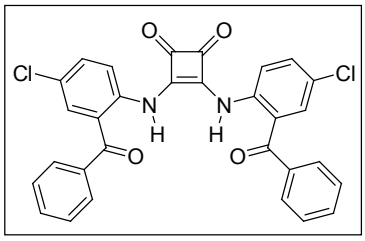
1. V. Ramalingam, M. E. Domaradzki, S. Jang and R. S. Muthyala, *Org. Lett.*, 2008, **10**, 3315.

Characterization of 3,4-bis (2-benzoyl phenyl amino) cyclobut-3-ene-1, 2-dione (**1**):



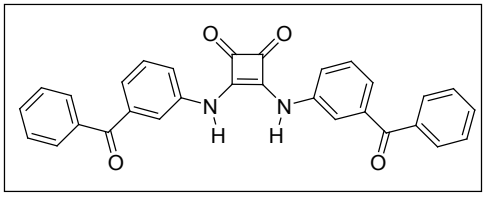
^{1}H NMR ($CDCl_3$): δ 11.4(2H, s), 8.14(2H, d, J=8.4Hz), 7.73(4H, d, J=6.8Hz), 7.72-7.59(6H, m), 7.50(4H, t, J=7.2), 7.16(2H, t, J=8Hz) ppm. ^{13}CNMR ($CDCl_3$): δ 199.9, 183.3, 166.32, 140.4, 138.3, 135.0, 134.3, 132.4, 129.8, 128.3, 122.6, 122.4, and 120.9 ppm. UV/vis (CH_3CN) $\lambda_{max,}$ nm (ε) 383 ($1.9x10^4$ $M^{-1}cm^{-1}$); 313, ($2.1x10^4$ $M^{-1}cm^{-1}$), HRMS calculated for $C_{30}H_{20}N_2O_4$ (M^++1) =473.1495 found 473.1492. mp. 223-224 °C

Characterization of 3,4-bis (2-benzoyl 4-chloro phenyl amino) cyclobut-3-ene-1, 2-dione (**2**):



^{1}H NMR ($CDCl_3$): δ11.30 (2H, s), 8.12(2H, d, J=8.4Hz), 7.72(4H, d, J=6.8Hz), 7.67-7.61(6H, m), 7.53(4H, t, J=8.0) ppm. ^{13}C NMR ($CDCl_3$): δ 198.8, 183.0, 166.0, 138.8, 137.5, 134.8, 133.4, 133.0, 129.8, 128.6, 128.1, 123.4, 122.57 ppm. UV/vis (CH_3CN) λ_{max}, nm (ε) 388 ($1.8x10^4$ $M^{-1}cm^{-1}$); 319, ($2.4x10^4$ $M^{-1}cm^{-1}$), HRMS calculated for $C_{30}H_{18}Cl_2N_2O_4$ (M^+) = 540.0643 found 540.0646. mp. 248-250 °

Characterization of 3,4-bis(3-benzoyl phenyl amino)cyclobut-3-ene-1,2-dione (**3**):



^{1}H NMR (CD_3CN): δ8.38 (2H, s), 7.82(4H, d, J=8.0Hz), 7.75(2H, s), 7.67(4H, m), 7.55(8H, m) ppm. ^{13}CNMR (CD_3CN): δ 195.2, 182.6, 165.4, 138.3, 138.2, 136.8, 132.3, 129.5, 129.1, 128.0, 124.9, 122.6 and 119.8 ppm. UV/vis (CH_3CN) λ_{max}, nm(ε) 320 ($3.2x10^4$ $M^{-1}cm^{-1}$). HRMS calculated for $C_{30}H_{20}N_2O_4$ (M^+) = 472.1423 found 472.1432. mp. 170-172 ^{o}C.

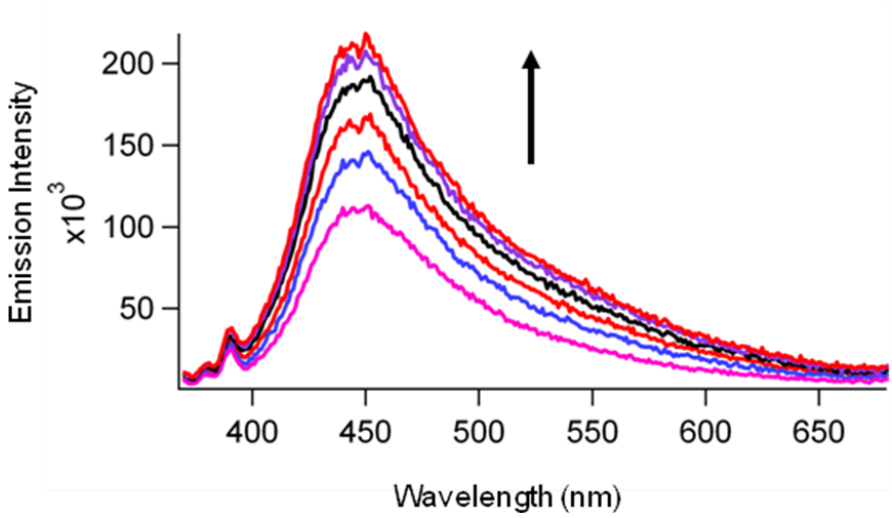


Figure S1 Emission titration spectra of squaramide **1** with tetrabutyl ammonium chloride in acetonitrile, λ_{ex} = 350 nm; [squaramide **1**] = 5×10^{-5} M and [tetrabutyl ammonium chloride] = 0 to 5×10^{-4} M.

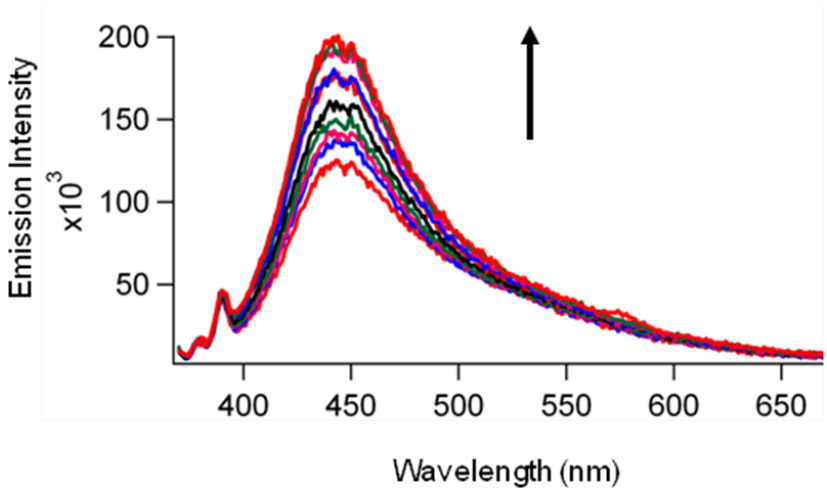


Figure S2 Emission titration spectra of squaramide **1** with tetrabutyl ammonium bromide in acetonitrile, λ_{ex} = 350 nm; [squaramide **1**] = 5×10^{-5} M and [tetrabutyl ammonium bromide] = 0 to 5×10^{-4} M.

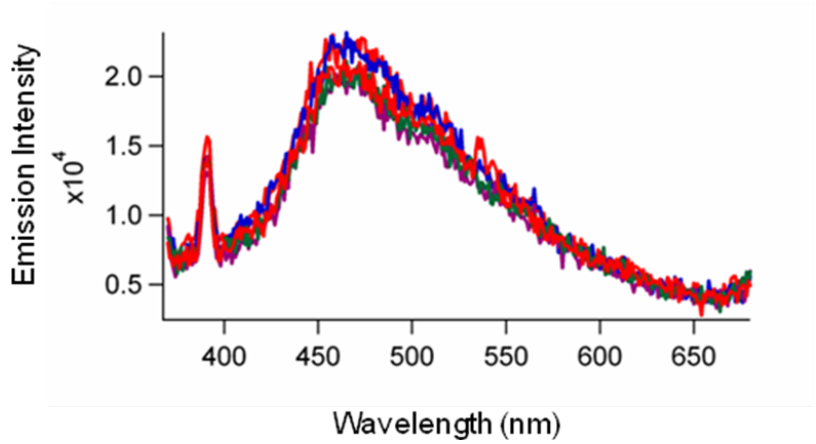


Figure S3 Emission titration spectra of squaramide **1** with tetrabutyl ammonium iodide in acetonitile, λ_{ex} = 350 nm; [squaramide **1**] = 5×10^{-5} M and [tetrabutyl ammonium iodide] = 0 to 5×10^{-4} M.

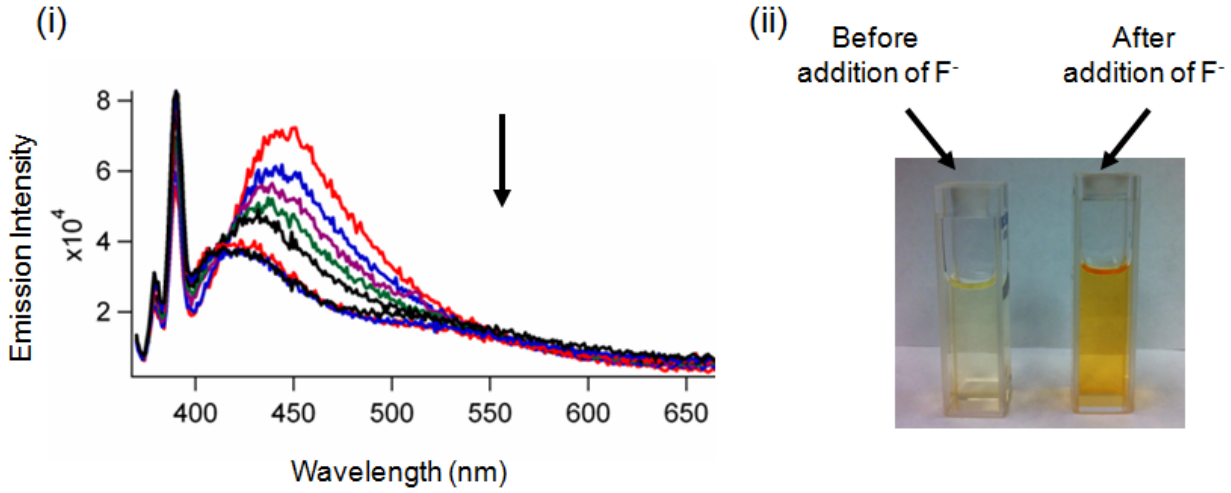


Figure S4 (i) Emission titration spectra of squaramide **1** with tetrabutyl ammonium fluoride in acetonitrile, λ_{ex} = 350 nm; [squaramide **1**] = 5×10^{-5} M and [tetrabutyl ammonium fluoride] = 0 to 3×10^{-4} M and (ii) color change of squaramide **1** in acetonitrile before and after addition of tetrabutyl ammonium fluoride.

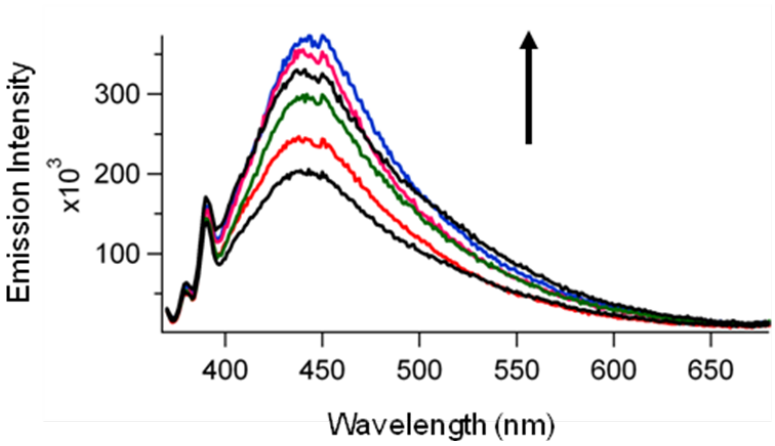


Figure S5 Emission titration spectra of squaramide **2** with tetrabutyl ammonium bromide in acetonitrile, λ_{ex} = 350 nm; [squaramide **2**] = 5×10^{-5} M and [tetrabutyl ammonium bromide] = 0 to 5×10^{-4} M.

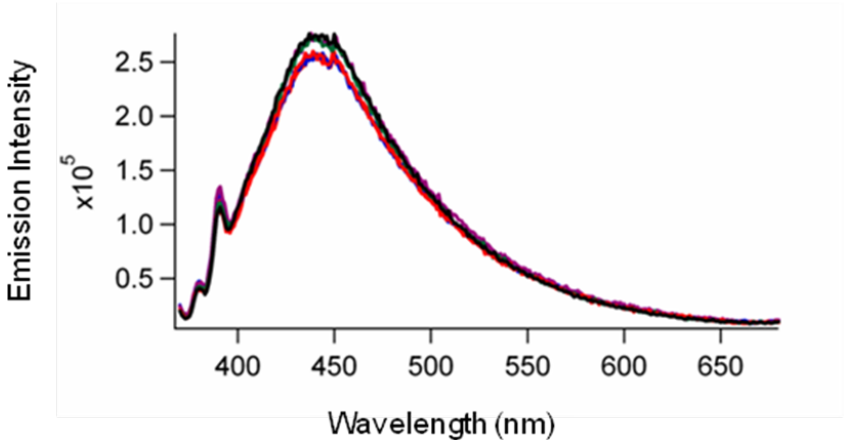


Figure S6 Emission titration spectra of squaramide **2** with tetrabutyl ammonium iodide in acetonitrile, λ_{ex} = 350 nm; [squaramide **2**] = 5×10^{-5} M and [tetrabutyl ammonium iodide] = 0 to 5×10^{-4} M.

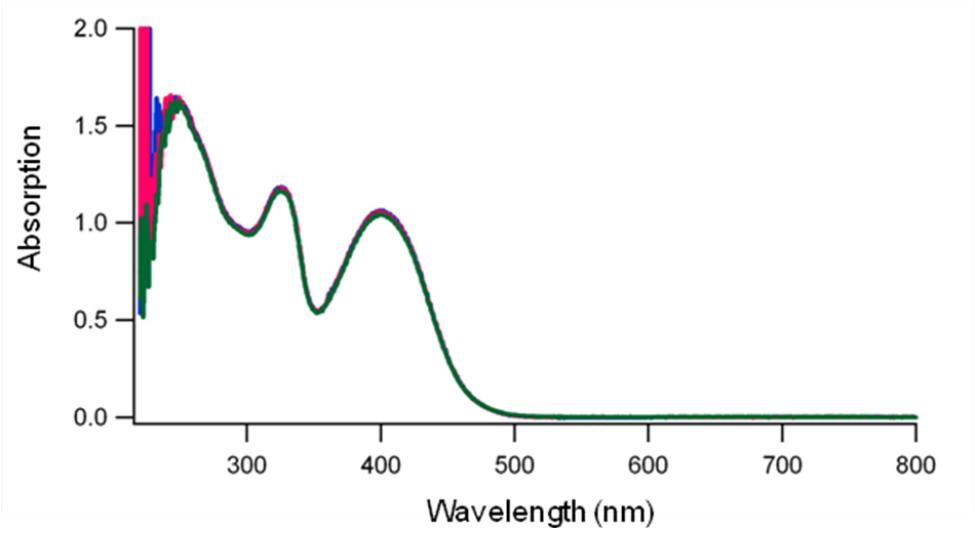


Figure S7 UV-visible titration spectra of squaramide **2** with tetrabutyl ammonium chloride in chloroform; [squaramide **2**] = 5×10^{-5} M and [tetrabutyl ammonium chloride] = 0 to 5×10^{-4} M.

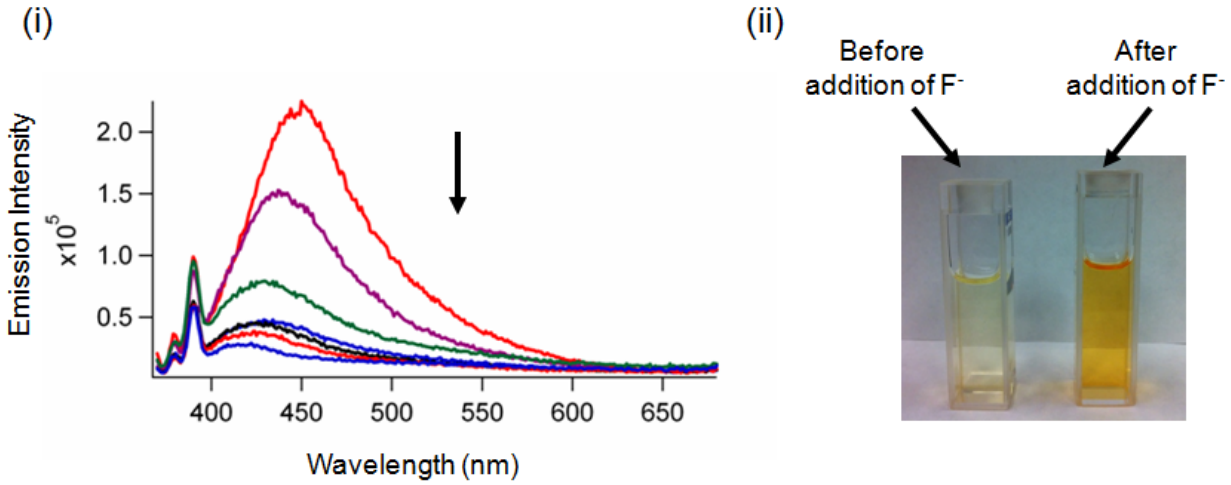


Figure S8 (i) Emission titration spectra of squaramide **2** with tetrabutyl ammonium fluoride in acetonitrile, λ_{ex} = 350 nm; [squaramide **2**] = 5×10^{-5} M and [tetrabutyl ammonium fluoride] = 0 to 3×10^{-4} M and (ii) color change of squaramide **2** in acetonitrile before and after addition of tetrabutyl ammonium fluoride.

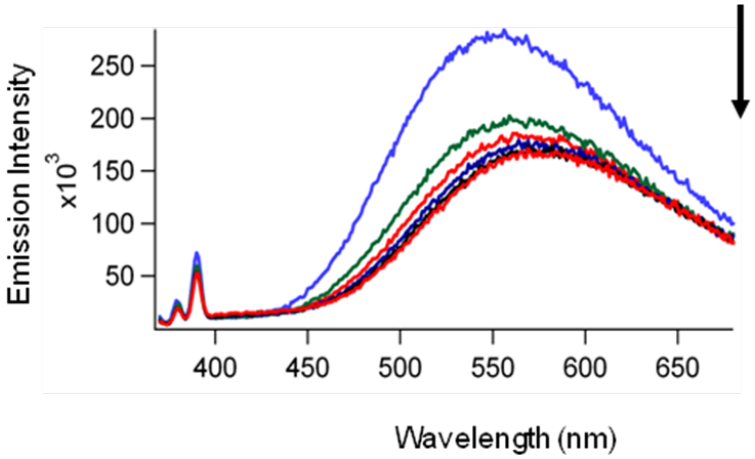


Figure S9 Emission titration spectra of squaramide **3** with tetrabutyl ammonium chloride in acetonitrile, λ_{ex} = 350 nm; [squaramide **3**] = 5×10^{-5} M and [tetrabutyl ammonium chloride] = 0 to 5×10^{-4} M.

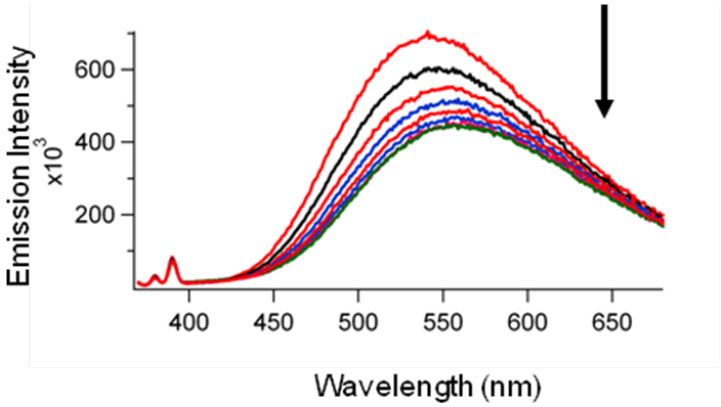


Figure S10 Emission titration spectra of squaramide **3** with tetrabutyl ammonium bromide in acetonitrile, λ_{ex} = 350 nm; [squaramide **3**] = 5×10^{-5} M and [tetrabutyl ammonium bromide] = 0 to 5×10^{-4} M.

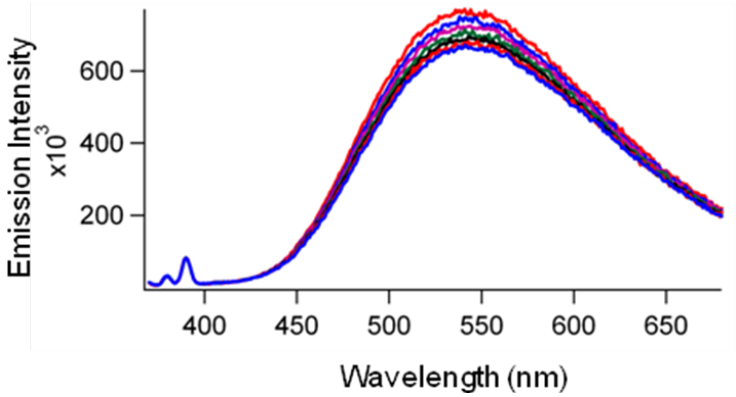


Figure S11 Emission titration spectra of squaramide **3** with tetrabutyl ammonium iodide in acetonitrile, λ_{ex} = 350 nm; [squaramide **3**] = 5×10^{-5} M and [tetrabutyl ammonium iodide] = 0 to 5×10^{-4} M.

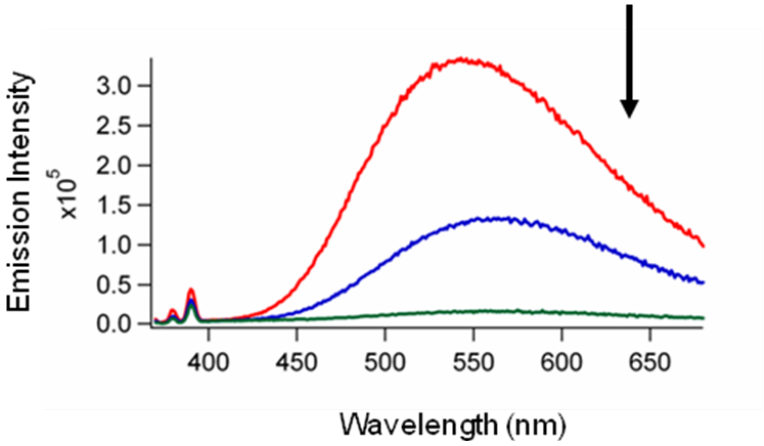
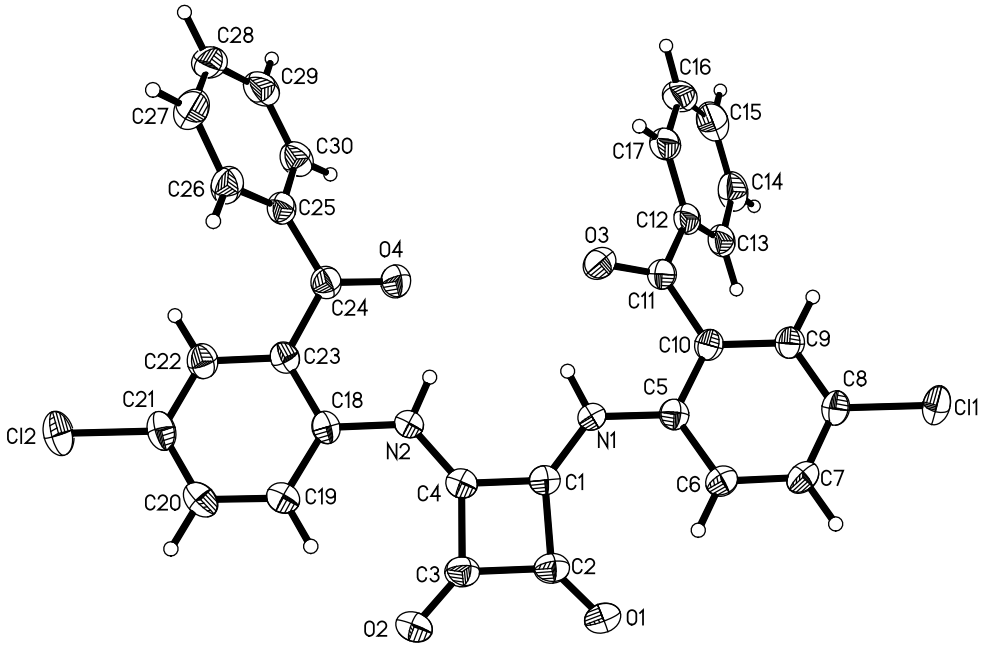
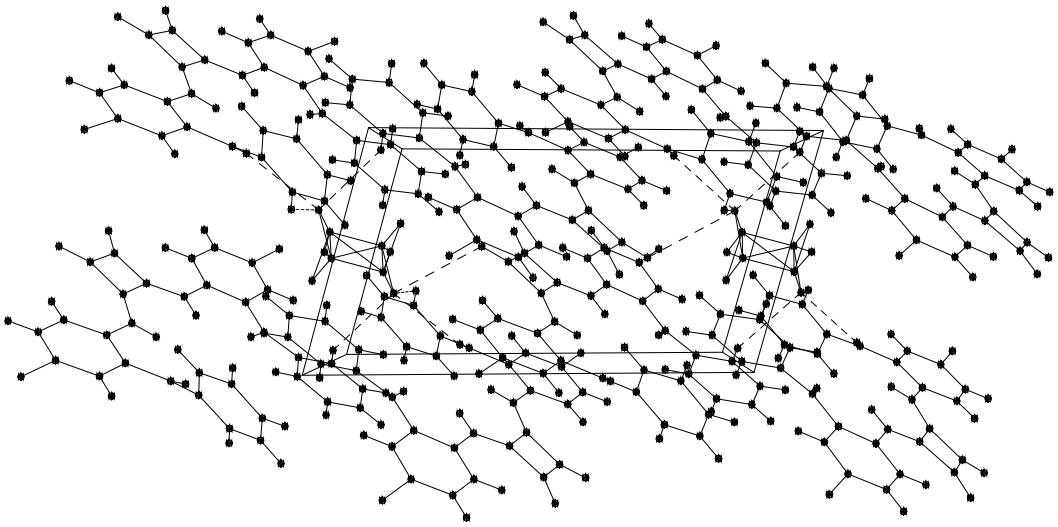


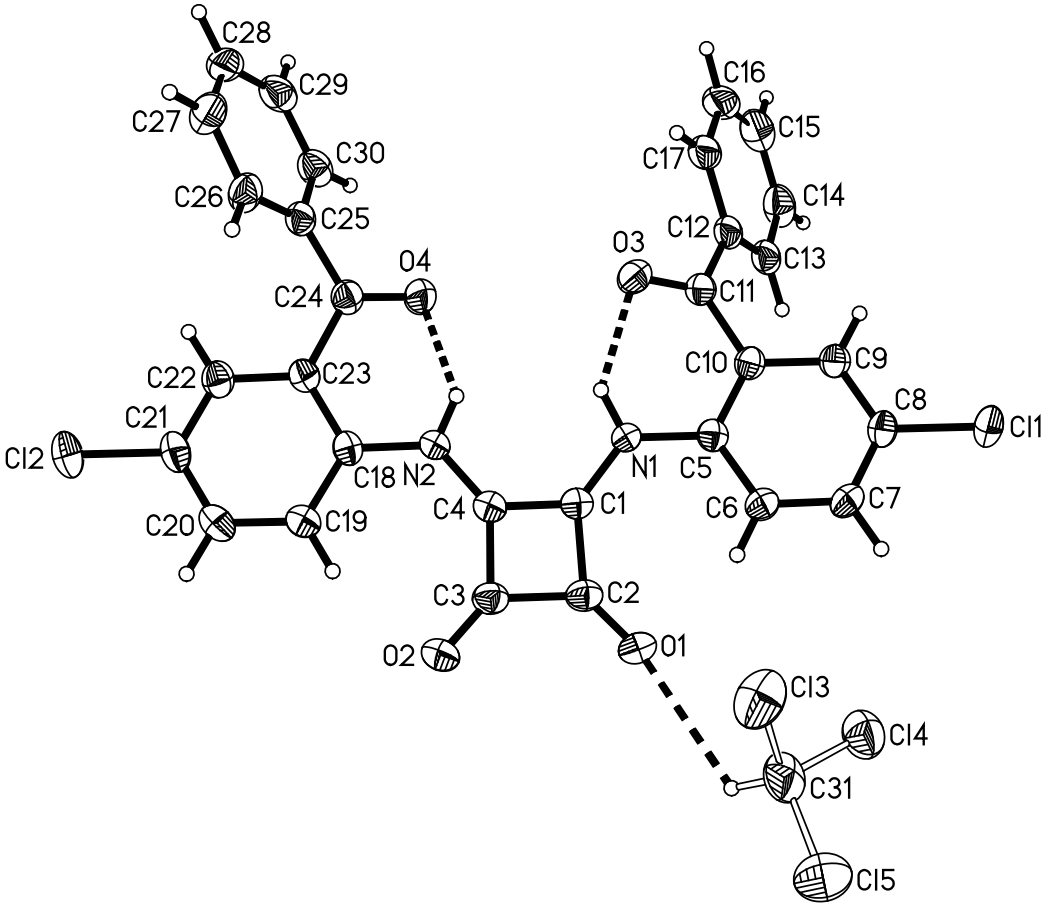
Figure S12 Emission titration spectra of squaramide **3** with tetrabutyl ammonium fluoride in acetonitrile, λ_{ex} = 350 nm; [squaramide **3**] = 5×10^{-5} M and [tetrabutyl ammonium fluoride] = 0 to 1×10^{-4} M.

CRYSTAL STRUCTURE REPORT

$C_{30.50}\,H_{18.50}\,Cl_{3.50}\,N_2\,O_4$







Data collection

A crystal (approximate dimensions 0.45x 0.40 x 0.40mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a CCD area detector diffractometer for a data collection at173(2) K.[1] A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 54 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 20 seconds and a detector distance of 4.9 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.80 Å. Four major sections of frames were collected with 0.30º steps in ω at four different ϕ settings and a detector position of -28º in2θ. The intensity data were corrected for absorption and decay (SADABS).[2] Final cell constants were calculated from 2991 strong reflections from the actual data collection after integration (SAINT).[3] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using Bruker SHELXTL[4] and refined using Bruker SHELXTL.[4] The space group P-1 was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0477$ and $wR2 = 0.1195$ (F^2, all data).

Structure description

The structure is the one suggested. The structure is a chloroform solvate. The chloroform is located on a crystallographic inversion center. There is one weak C-H···O type hydrogen bond between the solvent and the main molecule.

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, 192 Kolthoff Hall, Department of Chemistry, University of Minnesota. All calculations were performed using Pentium computers using the current SHELXTL suite of programs.

1 SMART V5.054, Bruker Analytical X-raySystems, Madison, WI (2001).

2 An empirical correction for absorption anisotropy,R. Blessing, *Acta Cryst.* **A51**, 33-38(1995).

3 SAINT+ V6.45, Bruker Analytical X-Ray Systems,Madison, WI (2003).

4 SHELXTL V6.14, Bruker Analytical X-Ray Systems,Madison, WI (2000).

5 A. Altomare, M. C. Burla, M. Camalli,G. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori,R. Spagna. Sir97: a new tool for crystal structure determination andrefinement. *J. Appl. Cryst.* **32**, 115-119(1998).

6 M. C. Burla, M. Camalli, B. Carrozzini,G. L. Cascarano, C. Giacovazzo, G. Polidori, R. Spagna. Sir2002: a newDirect Methods program for automatic solution and refinement of crystal structures. *J. Appl. Cryst.* (2003), in preparation.

7 A. L. Spek, *Acta. Cryst.***A46**, C34 (1990). PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, A. L.Spek (2000).

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R_1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = q \,/\, [\sigma^2(F_o^2) + (a*P)^2 + b*P + d + e*\sin(\theta)]$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (n-p)]^{1/2}$$

Table 1. Crystal data and structure refinement for 09041a.

Identification code	09041a	
Empirical formula	$C_{30.50}\,H_{18.50}\,Cl_{3.50}\,N_2\,O_4$	
Formula weight	601.05	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.4803(8) Å	α = 97.0370(10)°
	b = 13.4027(14) Å	β = 95.3740(10)°
	c = 13.7401(14) Å	γ = 104.6630(10)°
Volume	1311.4(2) $Å^3$	
Z	2	
Density (calculated)	1.522 Mg/m^3	
Absorption coefficient	0.443 mm^{-1}	
F(000)	614	
Crystal color, morphology	Yellow, Block	
Crystal size	0.45 x 0.40 x 0.40 mm^3	
Theta range for data collection	1.51 to 26.37°	
Index ranges	$-9 \le h \le 9, -16 \le k \le 16, 0 \le l \le 17$	
Reflections collected	15222	

Independent reflections	5334 [R(int) = 0.0342]
Observed reflections	3945
Completeness to theta = 26.37°	99.4%
Absorption correction	Multi-scan
Max. and min. transmission	0.8427 and 0.8256
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5334 / 0 / 379
Goodness-of-fit on F^2	1.034
Final R indices [I>2sigma(I)]	R1 = 0.0477, wR2 = 0.1078
R indices (all data)	R1 = 0.0723, wR2 = 0.1195
Largest diff. peak and hole	0.611 and -0.354 e.Å^{-3}

Table 2. Atomic coordinates(x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 09041a. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cl1	-69(1)	6166(1)	795(1)	36(1)
Cl2	5149(1)	2502(1)	10232(1)	54(1)
O1	5257(2)	7053(1)	5259(1)	44(1)
O2	5970(3)	6424(1)	7399(1)	47(1)
O3	-748(2)	3040(1)	3862(1)	40(1)
O4	1162(3)	2080(1)	5909(1)	43(1)
N1	1878(2)	4771(1)	4589(1)	26(1)
N2	2969(3)	4034(1)	6557(1)	28(1)
C1	3136(3)	5295(2)	5369(2)	26(1)
C2	4559(3)	6312(2)	5648(2)	31(1)
C3	4948(3)	5993(2)	6647(2)	32(1)
C4	3592(3)	4973(2)	6271(2)	26(1)
C5	1421(3)	5108(2)	3693(2)	26(1)
C6	2091(3)	6144(2)	3560(2)	31(1)
C7	1637(3)	6455(2)	2668(2)	32(1)
C8	497(3)	5748(2)	1907(2)	29(1)

C9	-189(3)	4716(2)	2018(2)	29(1)
C10	274(3)	4378(2)	2910(2)	27(1)
C11	-524(3)	3268(2)	3030(2)	30(1)
C12	-1039(3)	2424(2)	2164(2)	32(1)
C13	-10(3)	2429(2)	1373(2)	36(1)
C14	-409(4)	1556(2)	643(2)	45(1)
C15	-1846(4)	693(2)	706(2)	50(1)
C16	-2884(4)	689(2)	1478(2)	48(1)
C17	-2484(4)	1542(2)	2216(2)	40(1)
C18	3466(3)	3699(2)	7445(2)	27(1)
C19	4703(3)	4364(2)	8217(2)	30(1)
C20	5210(3)	3997(2)	9067(2)	35(1)
C21	4490(3)	2963(2)	9160(2)	35(1)
C22	3222(3)	2297(2)	8424(2)	32(1)
C23	2670(3)	2649(2)	7554(2)	28(1)
C24	1312(3)	1899(2)	6764(2)	31(1)
C25	121(3)	916(2)	7006(2)	31(1)
C26	-852(4)	907(2)	7823(2)	37(1)
C27	-2081(4)	-16(2)	7961(2)	46(1)
C28	-2297(4)	-928(2)	7319(2)	45(1)
C29	-1315(4)	-919(2)	6521(2)	44(1)
C30	-120(4)	-2(2)	6360(2)	38(1)

C31	5277(9)	9426(6)	4839(6)	54(2)
Cl3	3144(3)	8866(2)	5253(1)	66(1)
Cl4	5007(2)	9084(1)	3546(1)	56(1)
Cl5	5894(5)	10774(2)	5134(2)	96(1)

Table 3. Bond lengths [Å] and angles [°] for 09041a.

Cl(1)-C(8)	1.743(2)	C(7)-C(8)	1.378(3)
Cl(2)-C(21)	1.743(2)	C(7)-H(7A)	0.9500
O(1)-C(2)	1.210(3)	C(8)-C(9)	1.380(3)
O(2)-C(3)	1.212(3)	C(9)-C(10)	1.402(3)
O(3)-C(11)	1.234(3)	C(9)-H(9A)	0.9500
O(4)-C(24)	1.229(3)	C(10)-C(11)	1.491(3)
N(1)-C(1)	1.350(3)	C(11)-C(12)	1.487(3)
N(1)-C(5)	1.404(3)	C(12)-C(13)	1.388(3)
N(1)-H(1A)	0.8800	C(12)-C(17)	1.399(3)
N(2)-C(4)	1.347(3)	C(13)-C(14)	1.394(3)
N(2)-C(18)	1.401(3)	C(13)-H(13A)	0.9500
N(2)-H(2A)	0.8800	C(14)-C(15)	1.382(4)
C(1)-C(4)	1.401(3)	C(14)-H(14A)	0.9500
C(1)-C(2)	1.483(3)	C(15)-C(16)	1.373(4)
C(2)-C(3)	1.511(3)	C(15)-H(15A)	0.9500
C(3)-C(4)	1.481(3)	C(16)-C(17)	1.383(4)
C(5)-C(6)	1.392(3)	C(16)-H(16A)	0.9500
C(5)-C(10)	1.412(3)	C(17)-H(17A)	0.9500
C(6)-C(7)	1.383(3)	C(18)-C(19)	1.394(3)
C(6)-H(6A)	0.9500	C(18)-C(23)	1.414(3)

C(19)-C(20)	1.379(3)	C(26)-H(26A)	0.9500
C(19)-H(19A)	0.9500	C(27)-C(28)	1.381(4)
C(20)-C(21)	1.380(4)	C(27)-H(27A)	0.9500
C(20)-H(20A)	0.9500	C(28)-C(29)	1.375(4)
C(21)-C(22)	1.375(3)	C(28)-H(28A)	0.9500
C(22)-C(23)	1.402(3)	C(29)-C(30)	1.381(4)
C(22)-H(22A)	0.9500	C(29)-H(29A)	0.9500
C(23)-C(24)	1.493(3)	C(30)-H(30A)	0.9500
C(24)-C(25)	1.486(3)	C(31)-Cl(5)	1.734(8)
C(25)-C(30)	1.387(3)	C(31)-Cl(4)	1.759(8)
C(25)-C(26)	1.393(3)	C(31)-Cl(3)	1.761(7)
C(26)-C(27)	1.388(4)	C(31)-H(31A)	1.0000

C(1)-N(1)-C(5)	128.57(18)	O(1)-C(2)-C(1)	138.0(2)
C(1)-N(1)-H(1A)	115.7	O(1)-C(2)-C(3)	134.1(2)
C(5)-N(1)-H(1A)	115.7	C(1)-C(2)-C(3)	87.74(17)
C(4)-N(2)-C(18)	128.97(19)	O(2)-C(3)-C(4)	138.4(2)
C(4)-N(2)-H(2A)	115.5	O(2)-C(3)-C(2)	133.7(2)
C(18)-N(2)-H(2A)	115.5	C(4)-C(3)-C(2)	87.90(17)
N(1)-C(1)-C(4)	129.2(2)	N(2)-C(4)-C(1)	128.8(2)
N(1)-C(1)-C(2)	138.7(2)	N(2)-C(4)-C(3)	139.1(2)
C(4)-C(1)-C(2)	92.04(18)	C(1)-C(4)-C(3)	92.07(18)

C(6)-C(5)-N(1)	121.3(2)	C(13)-C(12)-C(11)	122.4(2)
C(6)-C(5)-C(10)	119.5(2)	C(17)-C(12)-C(11)	117.6(2)
N(1)-C(5)-C(10)	119.17(19)	C(12)-C(13)-C(14)	120.0(2)
C(7)-C(6)-C(5)	120.2(2)	C(12)-C(13)-H(13A)	120.0
C(7)-C(6)-H(6A)	119.9	C(14)-C(13)-H(13A)	120.0
C(5)-C(6)-H(6A)	119.9	C(15)-C(14)-C(13)	119.6(3)
C(8)-C(7)-C(6)	120.4(2)	C(15)-C(14)-H(14A)	120.2
C(8)-C(7)-H(7A)	119.8	C(13)-C(14)-H(14A)	120.2
C(6)-C(7)-H(7A)	119.8	C(16)-C(15)-C(14)	120.7(2)
C(7)-C(8)-C(9)	120.7(2)	C(16)-C(15)-H(15A)	119.7
C(7)-C(8)-Cl(1)	119.34(17)	C(14)-C(15)-H(15A)	119.7
C(9)-C(8)-Cl(1)	119.99(18)	C(15)-C(16)-C(17)	120.3(3)
C(8)-C(9)-C(10)	120.0(2)	C(15)-C(16)-H(16A)	119.8
C(8)-C(9)-H(9A)	120.0	C(17)-C(16)-H(16A)	119.8
C(10)-C(9)-H(9A)	120.0	C(16)-C(17)-C(12)	119.8(3)
C(9)-C(10)-C(5)	119.1(2)	C(16)-C(17)-H(17A)	120.1
C(9)-C(10)-C(11)	119.4(2)	C(12)-C(17)-H(17A)	120.1
C(5)-C(10)-C(11)	121.36(19)	C(19)-C(18)-N(2)	122.2(2)
O(3)-C(11)-C(12)	119.0(2)	C(19)-C(18)-C(23)	119.5(2)
O(3)-C(11)-C(10)	119.8(2)	N(2)-C(18)-C(23)	118.28(19)
C(12)-C(11)-C(10)	121.15(19)	C(20)-C(19)-C(18)	120.6(2)
C(13)-C(12)-C(17)	119.6(2)	C(20)-C(19)-H(19A)	119.7

C(18)-C(19)-H(19A)	119.7	C(27)-C(26)-H(26A)	120.3
C(19)-C(20)-C(21)	120.0(2)	C(25)-C(26)-H(26A)	120.3
C(19)-C(20)-H(20A)	120.0	C(28)-C(27)-C(26)	120.7(3)
C(21)-C(20)-H(20A)	120.0	C(28)-C(27)-H(27A)	119.6
C(22)-C(21)-C(20)	120.7(2)	C(26)-C(27)-H(27A)	119.6
C(22)-C(21)-Cl(2)	119.50(19)	C(29)-C(28)-C(27)	119.8(2)
C(20)-C(21)-Cl(2)	119.83(19)	C(29)-C(28)-H(28A)	120.1
C(21)-C(22)-C(23)	120.6(2)	C(27)-C(28)-H(28A)	120.1
C(21)-C(22)-H(22A)	119.7	C(28)-C(29)-C(30)	120.2(2)
C(23)-C(22)-H(22A)	119.7	C(28)-C(29)-H(29A)	119.9
C(22)-C(23)-C(18)	118.6(2)	C(30)-C(29)-H(29A)	119.9
C(22)-C(23)-C(24)	119.1(2)	C(29)-C(30)-C(25)	120.5(3)
C(18)-C(23)-C(24)	122.32(19)	C(29)-C(30)-H(30A)	119.8
O(4)-C(24)-C(25)	119.0(2)	C(25)-C(30)-H(30A)	119.8
O(4)-C(24)-C(23)	121.0(2)	Cl(5)-C(31)-Cl(4)	109.0(4)
C(25)-C(24)-C(23)	120.02(19)	Cl(5)-C(31)-Cl(3)	111.1(4)
C(30)-C(25)-C(26)	119.5(2)	Cl(4)-C(31)-Cl(3)	107.9(4)
C(30)-C(25)-C(24)	118.6(2)	Cl(5)-C(31)-H(31A)	109.6
C(26)-C(25)-C(24)	121.8(2)	Cl(4)-C(31)-H(31A)	109.6
C(27)-C(26)-C(25)	119.3(2)	Cl(3)-C(31)-H(31A)	109.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for 09041a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	51(1)	34(1)	30(1)	11(1)	7(1)	19(1)
Cl2	57(1)	65(1)	39(1)	19(1)	-12(1)	16(1)
O1	44(1)	34(1)	47(1)	12(1)	-2(1)	-5(1)
O2	53(1)	34(1)	41(1)	2(1)	-13(1)	-3(1)
O3	49(1)	31(1)	34(1)	8(1)	2(1)	0(1)
O4	55(1)	37(1)	27(1)	7(1)	-1(1)	-1(1)
N1	28(1)	22(1)	27(1)	6(1)	1(1)	3(1)
N2	28(1)	26(1)	26(1)	3(1)	-2(1)	3(1)
C1	24(1)	25(1)	29(1)	3(1)	5(1)	8(1)
C2	29(1)	26(1)	37(1)	2(1)	4(1)	7(1)
C3	32(1)	26(1)	35(1)	2(1)	2(1)	6(1)
C4	23(1)	28(1)	26(1)	1(1)	5(1)	8(1)
C5	25(1)	28(1)	28(1)	5(1)	4(1)	10(1)
C6	34(1)	24(1)	33(1)	5(1)	4(1)	7(1)
C7	36(1)	24(1)	38(1)	9(1)	7(1)	9(1)
C8	34(1)	30(1)	28(1)	8(1)	6(1)	15(1)

C9	30(1)	29(1)	29(1)	5(1)	4(1)	10(1)
C10	29(1)	27(1)	28(1)	5(1)	5(1)	10(1)
C11	29(1)	28(1)	33(1)	7(1)	0(1)	8(1)
C12	34(1)	27(1)	34(1)	6(1)	-5(1)	11(1)
C13	41(1)	29(1)	36(1)	6(1)	-7(1)	13(1)
C14	61(2)	42(2)	36(1)	3(1)	-3(1)	24(1)
C15	70(2)	32(1)	46(2)	-5(1)	-16(2)	21(1)
C16	49(2)	26(1)	60(2)	4(1)	-16(1)	5(1)
C17	39(1)	31(1)	46(2)	8(1)	-8(1)	8(1)
C18	28(1)	31(1)	23(1)	4(1)	3(1)	11(1)
C19	29(1)	30(1)	29(1)	1(1)	2(1)	7(1)
C20	29(1)	43(1)	30(1)	-1(1)	-2(1)	11(1)
C21	34(1)	47(2)	27(1)	8(1)	0(1)	18(1)
C22	35(1)	33(1)	32(1)	7(1)	2(1)	14(1)
C23	29(1)	29(1)	26(1)	3(1)	1(1)	10(1)
C24	36(1)	29(1)	29(1)	4(1)	2(1)	10(1)
C25	35(1)	27(1)	31(1)	7(1)	-2(1)	10(1)
C26	50(2)	29(1)	34(1)	8(1)	4(1)	12(1)
C27	50(2)	46(2)	45(2)	18(1)	10(1)	10(1)
C28	42(2)	30(1)	59(2)	19(1)	-11(1)	1(1)
C29	49(2)	30(1)	48(2)	2(1)	-14(1)	9(1)
C30	42(1)	34(1)	35(1)	0(1)	-3(1)	13(1)

C31	31(3)	73(5)	64(4)	23(4)	0(3)	21(4)
Cl3	58(1)	75(1)	79(1)	31(1)	33(1)	29(1)
Cl4	53(1)	70(1)	45(1)	14(1)	6(1)	16(1)
Cl5	132(3)	45(1)	93(2)	-1(1)	-22(2)	9(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for 09041a.

	x	y	z	U(eq)
H1A	1262	4133	4649	32
H2A	2134	3566	6127	33
H6A	2864	6638	4084	37
H7A	2115	7160	2578	38
H9A	-975	4235	1489	34
H13A	966	3027	1330	43
H14A	302	1553	106	54
H15A	-2117	98	209	60
H16A	-3883	96	1506	57
H17A	-3189	1531	2757	47
H19A	5201	5076	8157	36
H20A	6056	4456	9589	42
H22A	2715	1592	8505	39
H26A	-677	1526	8280	45
H27A	-2781	-21	8504	56

H28A	-3122	-1559	7428	54
H29A	-1459	-1546	6080	53
H30A	541	1	5802	45
H31A	6276	9145	5147	65

Table 6. Torsion angles [°] for 09041a.

C5-N1-C1-C4	179.2(2)	C1-N1-C5-C6	-9.3(3)
C5-N1-C1-C2	-3.8(4)	C1-N1-C5-C10	170.0(2)
N1-C1-C2-O1	-7.0(5)	N1-C5-C6-C7	179.3(2)
C4-C1-C2-O1	170.7(3)	C10-C5-C6-C7	0.0(3)
N1-C1-C2-C3	178.5(3)	C5-C6-C7-C8	0.9(3)
C4-C1-C2-C3	-3.79(17)	C6-C7-C8-C9	-0.9(4)
O1-C2-C3-O2	8.0(5)	C6-C7-C8-Cl1	179.40(18)
C1-C2-C3-O2	-177.1(3)	C7-C8-C9-C10	0.0(3)
O1-C2-C3-C4	-171.3(3)	Cl1-C8-C9-C10	179.63(17)
C1-C2-C3-C4	3.58(16)	C8-C9-C10-C5	1.0(3)
C18-N2-C4-C1	177.7(2)	C8-C9-C10-C11	178.1(2)
C18-N2-C4-C3	2.0(4)	C6-C5-C10-C9	-1.0(3)
N1-C1-C4-N2	4.8(4)	N1-C5-C10-C9	179.71(19)
C2-C1-C4-N2	-173.3(2)	C6-C5-C10-C11	-178.1(2)
N1-C1-C4-C3	-178.1(2)	N1-C5-C10-C11	2.6(3)
C2-C1-C4-C3	3.86(18)	C9-C10-C11-O3	-152.4(2)
O2-C3-C4-N2	-6.4(5)	C5-C10-C11-O3	24.7(3)
C2-C3-C4-N2	172.8(3)	C9-C10-C11-C12	28.3(3)
O2-C3-C4-C1	176.9(3)	C5-C10-C11-C12	-154.6(2)
C2-C3-C4-C1	-3.79(17)	O3-C11-C12-C13	-142.8(2)

C10-C11-C12-C13	36.5(3)	C19-C18-C23-C22	-2.9(3)
O3-C11-C12-C17	29.6(3)	N2-C18-C23-C22	177.4(2)
C10-C11-C12-C17	-151.1(2)	C19-C18-C23-C24	178.9(2)
C17-C12-C13-C14	-0.6(3)	N2-C18-C23-C24	-0.8(3)
C11-C12-C13-C14	171.7(2)	C22-C23-C24-O4	-160.6(2)
C12-C13-C14-C15	0.8(4)	C18-C23-C24-O4	17.6(3)
C13-C14-C15-C16	0.1(4)	C22-C23-C24-C25	19.2(3)
C14-C15-C16-C17	-1.2(4)	C18-C23-C24-C25	-162.6(2)
C15-C16-C17-C12	1.3(4)	O4-C24-C25-C30	42.9(3)
C13-C12-C17-C16	-0.5(3)	C23-C24-C25-C30	-136.9(2)
C11-C12-C17-C16	-173.1(2)	O4-C24-C25-C26	-132.6(2)
C4-N2-C18-C19	3.1(4)	C23-C24-C25-C26	47.6(3)
C4-N2-C18-C23	-177.1(2)	C30-C25-C26-C27	-1.5(4)
N2-C18-C19-C20	-177.7(2)	C24-C25-C26-C27	173.9(2)
C23-C18-C19-C20	2.5(3)	C25-C26-C27-C28	2.2(4)
C18-C19-C20-C21	-0.1(4)	C26-C27-C28-C29	-1.3(4)
C19-C20-C21-C22	-2.0(4)	C27-C28-C29-C30	-0.2(4)
C19-C20-C21-Cl2	179.52(18)	C28-C29-C30-C25	0.9(4)
C20-C21-C22-C23	1.6(4)	C26-C25-C30-C29	0.0(4)
Cl2-C21-C22-C23	-179.92(18)	C24-C25-C30-C29	-175.6(2)
C21-C22-C23-C18	0.9(3)		
C21-C22-C23-C24	179.1(2)		

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for 09041a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1A...O3	0.88	1.94	2.645(2)	135.8
N2-H2A...O4	0.88	1.91	2.625(2)	136.6
C31-H31A...O1	1.00	2.74	3.298(8)	115.5

Symmetry transformations used to generate equivalent atoms: