Supplementary information for:

Pyrazine analogues of dipyrrolylquinoxalines

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SYNTHESIS

General: All reactions were conducted under dry argon unless otherwise stated. All solvents were of reagent grade quality and purchased commercially. All starting materials were purchased from Aldrich Chemical Co. and used without further purification. NMR spectra used in the characterization of products were recorded on Varian INOVA 500, Varian Mercury 400 or Varian UNITY+ 300 instruments. The NMR spectra were referenced to solvent and the spectroscopic solvents were purchased from Cambridge Isotope Laboratories. All high-resolution (HR) chemical ionization (CI) mass spectra were recorded on a VG ZAB-2E instrument. Elemental analyses were performed by Atlantic Microlabs, Inc., Atlanta, GA, and are reported as percentages. Melting points were measured using a Meltemp II apparatus. TLC analyses were carried out using Baker-flex Silica gel IB-F sheets. Column chromatography was performed on Whatman silica gel 60Å (230 – 400 mesh). 1,2-Bis-(1*H*-pyrrol-2-yl)-ethane-dione **1** was prepared according to literature procedures.[1]

5,6-Bis-(1H-pyrrol-2-yl)-pyrazine-2,3-dicarbonitrile, 2. To a CH_2Cl_2 solution (50 ml) of 1,2-bis-(1*H*-pyrrol-2-yl)-ethane-dione **1** (100 mg, 0.53 mmol) excess (2 equiv.) BF_3•Et_2O was added. After 5 min. of stirring, diaminomaleonitrile (144 mg, 1.33 mmol) was added, as a powder, in small portions to the upper solution. The resulting mixture was heated to, and maitainat, reflux with stirring for 12 h. After cooling and follow-up evaporative removal of the volatile components, the crude product was purified by column chromatography using a 9:1 CH_2Cl_2:EtOAc mixture as the eluent. This gave product **2** as a yellow powder with a green tint (75 mg, 55%). M.p. = 186°C decomp. ^{1}H NMR (400 MHz, acetone $-d_6$) δ (ppm) = 6.25-6.27 (m, 2H, pyrrole H), 7.09-7.11 (m, 2H, pyrrole H), 7.20-7.22 (m, 2H, pyrrole H), 11.22 (broad s, 2H, NH). ^{13}C NMR (100 MHz, acetone $-d_6$) δ (ppm) = 111.06, 114.97, 115.59, 125.84, 126.58, 127.33, 144.54. HRMS (CI^+): calcd for $C_{14}H_9N_6$ $[M + H]^+$ 261.0888; found *m/z*: 261.0893. Anal. calcd for $C_{14}H_8N_6$: C, 64.61; H, 3.10; N, 32.29; found C, 64.40; H, 3.24; N, 32.15.

5-(5-Formyl-1H-pyrrol-2-yl)-6-(1H-pyrrol-2-yl)-pyrazine-2,3-dicarbonitrile, 3.
Phosphorus oxychloride (0.28 ml; 3 mmol) was added to 0.7 ml (9 mmol) of DMF and cooled to 0°C. The resulting mixture was stirred at this temperature for 5 min, then allowed to warm to room temperature whereupon it was stirred for an additional 10 min.

1,2-Dichloroethane (7 ml) was added to the mixture followed by a dropwise addition of a solution of **2** (650 mg, 2.5 mmol) in 40 ml 1,2-dichloroethane. When the addition was complete (ca 40 min.) the mixture was heated to reflux and maintained there for 30 min. After cooling the reaction mixture to 0°C, 5 ml of saturated NaOAc were added in one batch. The mixture was then heated at reflux for an additional hour. Upon cooling once again, the organic phase was separated and washed with water, an aqueous solution of $NaHCO_3$, and brine. After removing the volatile components from the organic phase using a rotary evaporator, the resulting crude product was subject to column chromatography (silica gel, eluent: 1 % MeOH in CH_2Cl_2). The desired products were obtained as the 3rd fraction of the column. Evaporation to dryness yields **3** (446 mg, 62%) in the form of orange powder. m.p. = 212-214°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 6.32-6.34 (m, 1H, pyrrole H), 6.96-6.99 (m, 1H, pyrrole H), 7.15-7.18 (m, 2H, pyrrole H), 7.29-7.31 (m, 1H, pyrrole H), 9.64 (broad s, 1H, NH), 9.67 (s, 1H, CHO), 10.13 (borad s, 1H, NH). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 111.06, 114.97, 115.59, 125.84, 126.58, 127.33, 144.54. HRMS (CI^+): calcd for $C_{15}H_9N_6O$ $[M + H]^+$ 289.0838; found *m/z*: 289.0844.

5,6-Bis-(5-formyl-1H-pyrrol-2-yl)-pyrazine-2,3-dicarbonitrile, 4. Phosphorus oxychloride (0.7 ml; 7.5 mmol) was added to 1.3 ml (16.8 mmol) of DMF and cooled to 0°C. The resulting mixture was stirred at this temperature for 5 min, then allowed to warm to room temperature whereupon it was stirred for an additional 10 min. 1,2-Dichloroethane (7 ml) was added to the mixture followed by a dropwise addition of a solution of **2** (650 mg, 2.5 mmol) in 40 ml 1,2-dichloroethane. When the addition was complete (ca 40 min.) the mixture was heated to reflux and maintained there for 30 min. After cooling the reaction mixture to 0°C, 7 ml of saturated NaOAc were added in one batch. The mixture was then heated at reflux for an additional hour. Upon cooling once again, the organic phase was separated and washed with water, an aqueous solution of $NaHCO_3$, and brine. After removing the volatile components from the organic phase using a rotary evaporator, the resulting crude product was subject to column chromatography (silica gel, eluent: 1 % MeOH in CH_2Cl_2). The desired products were obtained as the 5th fraction of the column. Evaporation to dryness yields **4** (560 mg, 71%) as yellow powder. m.p. = 232°C decomp. ^{1}H NMR (400 MHz, acetone $-d_6$) δ(ppm) = 6.80 (2, 2H, pyrrole H, J = 4.0 Hz), 7.08 (d, 2H, pyrrole H, J = 4.0 Hz), 9.75 (s, 2H, CHO), 12.00 (broad s, 2H, NH). ^{13}C NMR (100 MHz, acetone $-d_6$) δ(ppm) = 114.0, 114.9, 115.7, 119.3, 129.2, 132.4, 136.6, 145.2, 180.2. HRMS (CI^+): calcd for $C_{15}H_9N_6O$ $[M + H]^+$ 289.0838; found *m/z*: 289.0844.

5-{5-[Bis-(1H-pyrrol-2-yl)-methyl]-1H-pyrrol-2-yl}-6-(1H-pyrrol-2-yl)-pyrazine-2,3-dicarbonitrile, 5. Compound **3** (50 mg, 0.173 mmol) was suspended in 3 ml (43.20 mmol) of freshly distilled pyrrole. To this mixture, 9 µl (1.52 mmol) of TFA were added. The reaction mixture was then stirred at room temperature for 2 h after which time the excess pyrrole was removed *in vacuo*. The crude product was then purified by column chromatography over silica gel using 0-5% MeOH in CH_2Cl_2as the eluent. The product was isolated as the 5th fraction and yield a deep-red – black powder after evaporative removal of the solvent (35 mg, 50%). M.p. = 118-120°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 5.60 (s, 1H, CH), 6.06-6.09 (m, 4H, pyrrole H), 6.17-6.19 (m, 2H, pyrrole H), 6.26-6.28 (m, 1H, pyrrole H), 6.73-6.74 (m, 2H, pyrrole H), 7.06-7.07 (m, 1H, pyrrole H),

7.26-7.28 (m, 1H, pyrrole H), 7.29-7.31 (m, 1H, pyrrole H), 8.09 (broad s, 2H, NH), 9.44 (broad s, 1H, NH), 9.57 (broad s, 1H, NH). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 37.7, 107.2, 108.7, 110.3, 111.3, 113.6, 113.8, 115.4, 116.4, 117.9, 124.9, 125.7, 126.3, 129.4, 139.9, 142.7. HRMS (CI^+): calcd for $C_{23}H_{17}N_8$ $[M + H]^+$ 405.1576; found *m/z*: 405.1567. Anal. calcd for $(C_{23}H_{16}N_8)_3 \bullet CH_2Cl_2 \bullet CH_3OH$: C, 64.10; H, 4.09; N, 25.27; found C, 63.70; H, 3.93; N, 25.01.

5,6-Bis-[5-(1H-pyrrol-2-ylmethyl)-1H-pyrrol-2-yl]-pyrazine-2,3-dicarbonitrile, 6. Compound **4** (50 mg; 0.158 mmol) was dissolved in 5 ml dry THF. A 0.83 M solution of lithium pyrrolidinoborane (0.28 ml; ca. 0.237 mmol)[2] was added dropwise at room temperature. Once the addition was complete, the reaction mixture was stirred at room temperature for an additional 6 h. The reaction was then quenched with 1 ml H_2O. After removing the solvent using a rotary evaporator, the resulting diol (whose production was confirmed by MS) was suspended without any further purification in 2 ml of freshly distilled pyrrole (28.80 mmol). To this mixture, 9 µl of TFA (1.52 mmol) were added. The mixture was then stirred at room temperature for an additional 2 h. After removal of the solvent using a rotary evaporator, the crude product was purified by column chromatography over silica gel using 0-5% MeOH in CH_2Cl_2 as the eluent. Compound **5** was isolated as the dominant fraction and obtained in the form of a reddish-orange powder (18.6 mg, 28 %) after removal of the solvent. ^{1}H NMR (500 MHz, acetone $-d_6$) δ (ppm) = 4.68 (s, 4H, CH_2), 6.17-6.18 (m, 2H, pyrrole H), 6.26-6.28 (m, 2H, pyrrole H), 7.05-7.06 (m, 2H, pyrrole H), 7.16-7.17 (m, 2H, pyrrole H), 7.21-7.22 (m, 2H, pyrrole H), 10.99 (broad s, 2H, NH), 11.20 (broad s, 2H, NH). ^{13}C NMR (125 MHz, acetone $-d_6$) δ (ppm) = 57.6, 109.1, 111.2, 115.2, 115.7, 116.5, 125.9, 126.4, 126.9, 127.7, 141.9, 144.7. MS (CI^+): calcd. for $C_{24}H_{19}N_8$ $[M + H]^+$ 419, found *m/z*: 419

5,6-Bis-{5-[bis-(1H-pyrrol-2-yl)-methyl]-1H-pyrrol-2-yl}-pyrazine-2,3-dicarbonitrile, 7. Compound **4** (27 mg, 0.085 mmol) was suspended in 3 ml (43.20 mmol) of freshly distilled pyrrole. To the resulting mixture, 9 µl of TFA (1.52 mmol) were added. The reaction mixture was then stirred at room temperature for 2 h, after which time the excess pyrrole was removed *in vacuo* and the crude product was purified by column chromatography over silica gel using 0-5% MeOH in CH_2Cl_2 as the eluent. The product was isolated as the 5th fraction and obtained in the form of a deep-red – black powder after removal of the solvents (39 mg, 84%). M.p. = 116-118°C. ^{1}H NMR (500 MHz, CD_2Cl_2) δ(ppm) = 5.59 (s, 2H, CH), 6.04-6.05 (m, 4H, pyrrole H), 6.07-6.09 (m, 2H, pyrrole H), 6.16-6.17 (m, 4H, pyrrole H), 6.73-6.75 (m, 4H, pyrrole H), 7.25-7.26 (m, 2H, pyrrole H), 8.17 (broad s, 2H, NH), 9.44 (broad s, 2H, NH). ^{13}C NMR (125 MHz, CD_2Cl_2) δ(ppm) = 38.0, 107.5, 109.0, 110.5, 114.4, 116.6, 118.3, 125.7, 126.4, 130.2, 140.4, 143.2. . HRMS (CI^+): calcd for $C_{32}H_{25}N_{10}$ $[M + H]^+$ 549.2263; found *m/z*: 549.2272. Anal. calcd for $C_{32}H_{24}N_{10} \bullet 0.5CH_2Cl_2$: C, 62.56; H, 4.14; N, 22.11; found C, 62.88; H, 4.43; N, 21.81.

BINDING STUDIES

UV-Vis Anion Recognition Study:

Stock solutions of the host molecules being studied were made up in dichloromethane with the final concentrations being between 2.9×10^{-5} M and 4.2×10^{-5} M. The hosts themselves were synthesized using the procedures described in the Experimental Section (**7**) or in accord with the previously reported procedures (**8**).[3] For instance, a 2 mL aliquot of a 1.05×10^{-4} M solution of receptor **7** was diluted to 5 mL yielding a stock 4.2×10^{-5} M solution of 7.

Stock solutions of the guest were prepared by dissolving 10 - 100 equivalents of tetrabutylammonium salts of the anions used in this study in 1.5 - 2.5 ml of stock solution of the hosts, prepared as described above. Making up the anion source solutions in this way allowed the binding studies to be carried out without having to make mathematical corrections to account for the changes in host concentration.

The general procedure for the UV-Vis binding studies involved making sequential additions of titrant (anionic guest) using Hamilton® pipettes to a 2 mL aliquot of the host stock solution in the spectrometric cell. The data was then collated and combined to produce plots that showed the changes in host (i.e., receptor **7** or **8**) spectral features as a function of changes in the concentration of the guest.

Calculations of Equilibrium Constants, K_a:

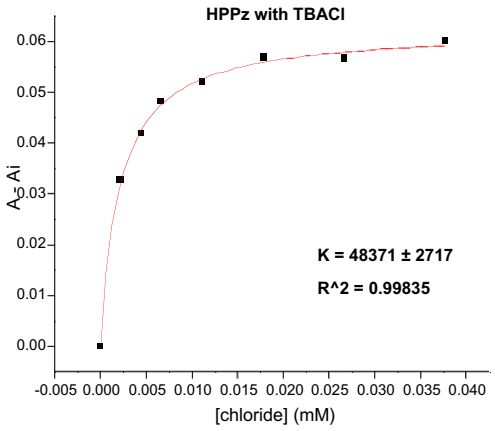
Equilibrium constants were calculated using equation 4.5 of Connors[4] where [L] = [anion]. The resulting equation, of the form, $y = B \times K \times x / (1 + K \times x)$, was computer fit using Origin version 7.0, where x = [anion], $y = \Delta A$, $B = \Delta\varepsilon \times b$, K = the equilibrium constant. The change in absorbance, ΔA, was calculated at λ where the spectral change was maximum.

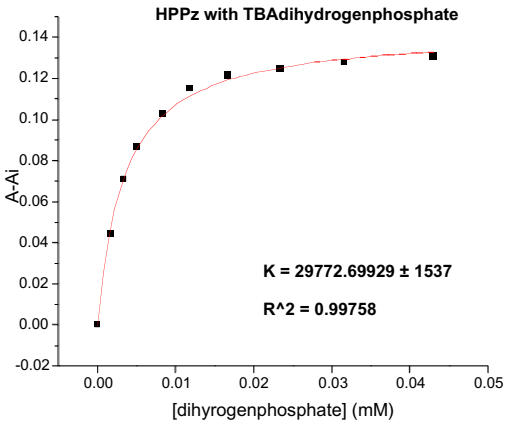
Job Plot Construction:

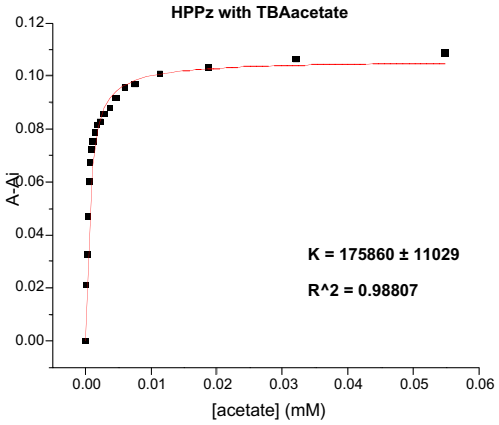
A stock solution of the host (e.g., receptor **7**) was prepared as described for the K_a determination experiments. The guest stock solution was prepared by dissolving 1 - 2 equivalents of the tetrabutylammonium salts of the studied anions is the same solvent (dichloromethane) as the one used for the host stock solution.

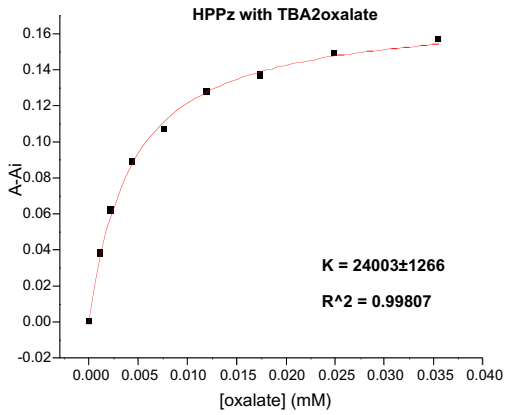
The general procedure for the UV-Vis Job titrations involved making sequential additions of titrant (anionic guest) using Hamilton® pipettes to a 1.5 ml aliquot of the host stock solution in a spectrometric cell. The data was then collated and combined to produce data files from which so-called Job plots could be constructed. These latter were produced as described by Connors[5], namely by plotting the molar fraction of guest (X_G) as a function of [host] $\times \Delta A$. The plots themselves were generated using Kaleidagraph software version 3.5.2. The change in absorbance, ΔA, was calculated at λ where the spectral change was maximum. In accord with accepted practice, the maxima of the resulting graph was considered indicative of the stoichiometry of the host:gust complex, namely a maxima

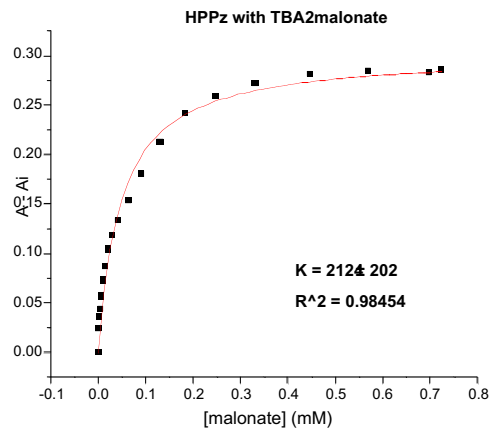
where $X_G = 0.5$ was considered indicative of a 1:1 host:guest complex, while a maxima with $X_G = 0.33$ was taken as being diagnostic for a 2:1 host:guest complex.

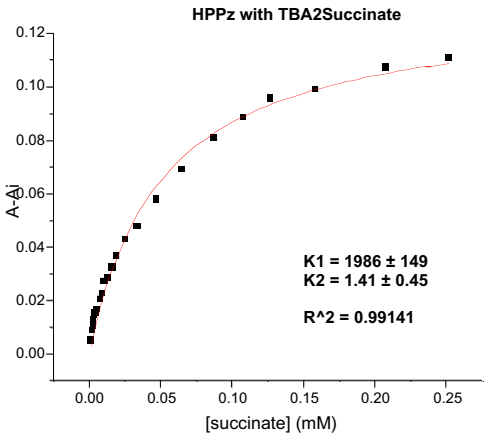


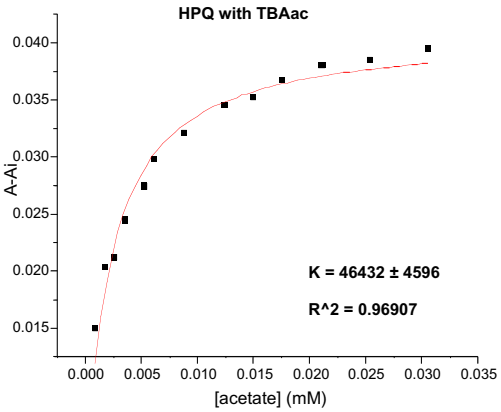


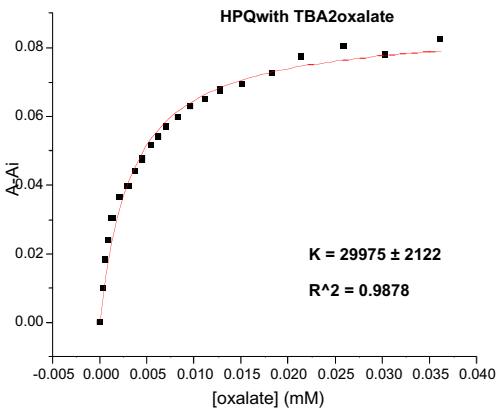


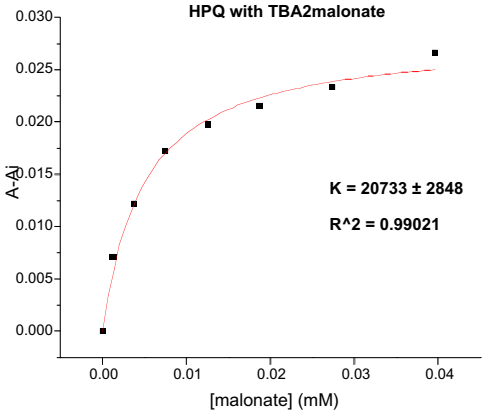


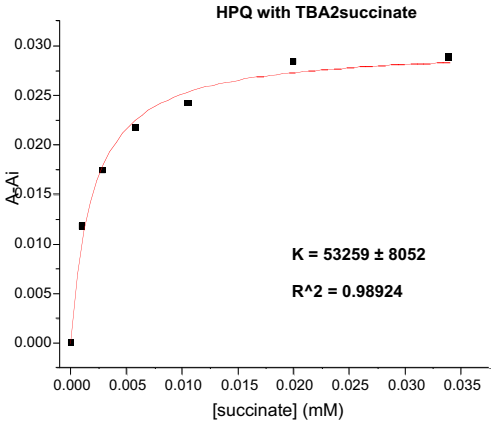





HPQ with TBAac
A-Ai
[acetate] (mM)
K = 46432 ± 4596
R^2 = 0.96907


HPQwith TBA2oxalate
A-Ai
[oxalate] (mM)
K = 29975 ± 2122
R^2 = 0.9878


HPQ with TBA2malonate
A-Ai
[malonate] (mM)
K = 20733 ± 2848
R^2 = 0.99021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A-Ai
[succinate] (mM)
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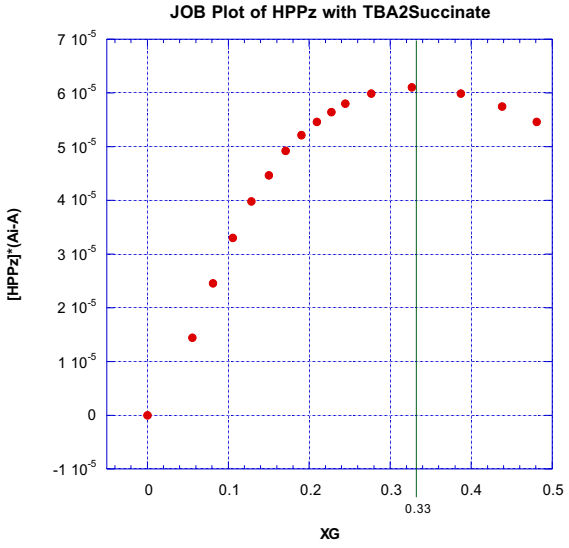
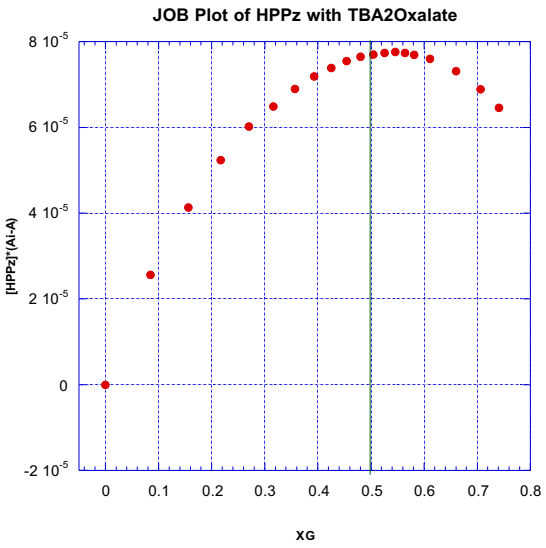
R^2 = 0.98924


JOB Plot of HPPz with TBA2Succinate
[HPPz]*(Ai-A)
XG
0.33


JOB Plot of HPPz with TBA2Oxalate
[HPPz]*(Ai-A)
XG

X-RAY EXPERIMENTAL

Crystallographic Material for **2**.

X-ray Experimental.

Table 1. Crystallographic Data for **2**.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of **2**.

Table 3. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of **1**.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **1**.

Table 5. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of **2**.

Table 6. Torsion Angles (^{o}) for the non-hydrogen atoms of **2**.

Table 7. Observed and calculated structure factor amplitudes for **2**. Values for Fo, Fc and s(Fo) have been multiplied by 10.

Figure 1. View of molecule 1 of **2** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary size.

Figure 2. View of molecule 2 of **2** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary size.

Figure 3. View of the fit by least-squares of atoms of molecule 1 onto the equivalent atoms of molecule 2. All non-H atoms were used in the fit.

Figure 4. Unit cell packing diagram for **2**. The view is approximately down the **a** axis. Molecules 1 are shown in wireframe form while molecules 2 are displayed as ball-and-stick. The molecules stack in columns parallel to **a**. Molecules 1 and 2 alternate within a column. Dashed lines are indicative of a H-bonding interaction. The geometry of these interactions are: N1-H1···N18 (related by 1-x, 1-y, 1-z); N···N 2.958(2)Å, H···N 2.12(2)Å, N-H···N 161(2)°; N1'-H1'···N18' (related by 2-x, 1-y, 1-z); N···N 3.136(3)Å, H···N 2.30(2)Å, N-H···N 165(2)°; N13'-H13'···N20' (related by 1-x, 2-y, 1-z); N···N 3.063(2)Å, H···N 2.31(2)Å, N-H···N 152(2)°.

X-ray Experimental for receptor **2**, $2(C_{14}H_8N_6) - CH_2Cl_2$:

Crystals grew as pale yellow lathes by slow evaporation from a methylene chloride solution. The data crystal was a long lathe that had approximate dimensions; 0.40x0.15x0.05 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKa radiation (l = 0.71073Å). A total of 753 frames of data were collected using w-scans with a scan range of 1° and a counting time of 92 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.[6] The structure was solved by direct methods using SIR92[7] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[8] The hydrogen atoms were observed in a ΔF map and refined with isotropic displacement parameters. There are two crystallographically independent molecules of the dicyano molecule per asymmetric unit. These molecules alternate along the **a** axis and are H-bound in an extended two dimensional array. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(s(F_o))^2 + (0.0502*P)^2 + (0.3704*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.119, with R(F) equal to 0.0464 and a goodness of fit, S, = 1.02. Definitions used for calculating R(F),$R_w(F^2)$ and the goodness of fit, S, are given below.[9] The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (8.7(14)x10^{-6})* F_c^2 l^3/(\sin 2q)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[10] All figures were generated using SHELXTL/PC.[11] Tables of positional and thermal parameters, bond lengths and angles, torsion angles, figures and lists of observed and calculated structure factors are located in tables 1 through 7.

Table 1. Crystal data and structure refinement for **2**.

Empirical formula	C29 H18 Cl2 N12	
Formula weight	605.45	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.3374(1) Å	α= 79.514(1)°.
	b = 12.8235(2) Å	β= 80.208(1)°.
	c = 15.6794(3) Å	γ = 84.107(1)°.
Volume	1425.60(4) $Å^3$	
Z	2	
Density (calculated)	1.410 Mg/m^3	
Absorption coefficient	0.272 mm^{-1}	
F(000)	620	
Crystal size	0.40 x 0.15 x 0.05 mm	
Theta range for data collection	2.93 to 27.50°.	
Index ranges	-9<=h<=9, -16<=k<=16, -19<=l<=20	
Reflections collected	11939	
Independent reflections	6522 [R(int) = 0.0299]	
Completeness to theta = 27.50°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6522 / 0 / 461	
Goodness-of-fit on F^2	1.023	
Final R indices [I>2sigma(I)]	R1 = 0.0464, wR2 = 0.1016	
R indices (all data)	R1 = 0.0862, wR2 = 0.1186	
Extinction coefficient	$8.7(14)x10^{-6}$	
Largest diff. peak and hole	0.274 and -0.380 $e.Å^{-3}$	

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

	x	y	z	U(eq)
Cl1A	-1775(1)	10819(1)	9030(1)	74(1)
Cl2A	-2156(1)	11413(1)	7186(1)	71(1)
C1A	-1114(4)	10506(2)	7972(2)	59(1)
N1'	7498(2)	4633(1)	7531(1)	32(1)
C2'	6621(2)	5621(1)	7631(1)	28(1)
C3'	5562(3)	5493(2)	8462(1)	31(1)
C4'	5841(3)	4431(2)	8856(1)	37(1)
C5'	7044(3)	3921(2)	8268(1)	37(1)
C6'	6882(2)	6483(1)	6891(1)	27(1)
N7'	7849(2)	6177(1)	6161(1)	29(1)
C8'	7946(3)	6867(1)	5405(1)	29(1)
C9'	6966(3)	7854(1)	5363(1)	30(1)
N10'	6133(2)	8215(1)	6090(1)	29(1)
C11'	6181(2)	7583(1)	6866(1)	27(1)
C12'	5551(2)	8109(1)	7618(1)	28(1)
N13'	4597(2)	9096(1)	7501(1)	31(1)
C14'	4340(3)	9505(2)	8254(1)	37(1)
C15'	5152(3)	8793(2)	8866(1)	36(1)
C16'	5916(3)	7919(2)	8472(1)	31(1)
C17'	9053(3)	6519(2)	4638(1)	34(1)
N18'	9902(2)	6256(1)	4020(1)	42(1)
C19'	6812(3)	8545(2)	4533(1)	35(1)
N20'	6679(3)	9089(1)	3879(1)	47(1)
N1	2343(2)	7650(1)	5619(1)	29(1)
C2	1603(2)	7381(1)	6493(1)	25(1)
C3	482(3)	8262(1)	6718(1)	29(1)
C4	586(3)	9056(2)	5977(1)	35(1)
C5	1745(3)	8657(2)	5311(1)	35(1)
C6	2023(2)	6316(1)	6934(1)	25(1)
N7	3108(2)	5690(1)	6424(1)	27(1)
C8	3329(2)	4654(1)	6739(1)	27(1)

C9	2386(3)	4210(1)	7543(1)	29(1)
N10	1432(2)	4826(1)	8093(1)	28(1)
C11	1345(2)	5883(1)	7830(1)	25(1)
C12	570(2)	6485(1)	8524(1)	26(1)
N13	-537(2)	5999(1)	9255(1)	32(1)
C14	-906(3)	6646(2)	9867(1)	38(1)
C15	-9(3)	7545(2)	9549(1)	38(1)
C16	930(3)	7454(2)	8706(1)	32(1)
C17	4543(3)	4002(1)	6187(1)	32(1)
N18	5470(2)	3463(1)	5758(1)	41(1)
C19	2390(3)	3071(2)	7832(1)	37(1)
N20	2399(3)	2173(1)	8055(1)	56(1)

Table 3. Bond lengths [Å] and angles [°] for **2**.

Cl1A-C1A	1.753(3)	C17'-N18'	1.146(2)
Cl2A-C1A	1.754(3)	C19'-N20'	1.144(2)
C1A-H1AA	1.03(3)	N1-C5	1.349(2)
C1A-H1AB	0.95(3)	N1-C2	1.381(2)
N1'-C5'	1.351(3)	N1-H1	0.87(2)
N1'-C2'	1.384(2)	C2-C3	1.390(2)
N1'-H1'	0.85(2)	C2-C6	1.442(2)
C2'-C3'	1.390(3)	C3-C4	1.395(3)
C2'-C6'	1.452(3)	C3-H3	0.96(2)
C3'-C4'	1.399(3)	C4-C5	1.371(3)
C3'-H3'	0.974(18)	C4-H4	0.97(2)
C4'-C5'	1.372(3)	C5-H5	0.96(2)
C4'-H4'	0.99(2)	C6-N7	1.344(2)
C5'-H5'	0.97(2)	C6-C11	1.437(2)
C6'-N7'	1.341(2)	N7-C8	1.334(2)
C6'-C11'	1.447(2)	C8-C9	1.384(3)
N7'-C8'	1.340(2)	C8-C17	1.445(3)
C8'-C9'	1.387(2)	C9-N10	1.337(2)
C8'-C17'	1.446(3)	C9-C19	1.447(3)
C9'-N10'	1.336(2)	N10-C11	1.341(2)
C9'-C19'	1.449(3)	C11-C12	1.451(2)
N10'-C11'	1.338(2)	C12-N13	1.375(2)
C11'-C12'	1.448(2)	C12-C16	1.384(3)
C12'-N13'	1.380(2)	N13-C14	1.356(2)
C12'-C16'	1.385(3)	N13-H13	0.87(2)
N13'-C14'	1.354(3)	C14-C15	1.359(3)
N13'-H13'	0.83(2)	C14-H14	0.95(2)
C14'-C15'	1.368(3)	C15-C16	1.402(3)
C14'-H14'	0.95(2)	C15-H15	0.93(2)
C15'-C16'	1.399(3)	C16-H16	0.89(2)
C15'-H15'	0.91(2)	C17-N18	1.144(2)
C16'-H16'	0.987(18)	C19-N20	1.141(2)
Cl1A-C1A-Cl2A	111.95(14)	Cl1A-C1A-H1AA	107.6(14)

Cl2A-C1A-H1AA	106.3(14)	C14'-N13'-C12'	109.99(17)
Cl1A-C1A-H1AB	109.9(18)	C14'-N13'-H13'	125.9(13)
Cl2A-C1A-H1AB	106.8(18)	C12'-N13'-H13'	123.8(13)
H1AA-C1A-H1AB	114(2)	N13'-C14'-C15'	108.07(17)
C5'-N1'-C2'	109.94(17)	N13'-C14'-H14'	120.2(12)
C5'-N1'-H1'	126.8(15)	C15'-C14'-H14'	131.7(12)
C2'-N1'-H1'	123.3(15)	C14'-C15'-C16'	107.69(18)
N1'-C2'-C3'	106.27(16)	C14'-C15'-H15'	127.8(12)
N1'-C2'-C6'	117.98(16)	C16'-C15'-H15'	124.5(13)
C3'-C2'-C6'	135.61(16)	C12'-C16'-C15'	107.85(17)
C2'-C3'-C4'	107.85(17)	C12'-C16'-H16'	124.5(10)
C2'-C3'-H3'	126.6(11)	C15'-C16'-H16'	127.6(10)
C4'-C3'-H3'	125.5(11)	N18'-C17'-C8'	178.5(2)
C5'-C4'-C3'	107.59(19)	N20'-C19'-C9'	179.5(2)
C5'-C4'-H4'	124.6(12)	C5-N1-C2	109.83(16)
C3'-C4'-H4'	127.8(12)	C5-N1-H1	126.0(15)
N1'-C5'-C4'	108.32(18)	C2-N1-H1	124.2(15)
N1'-C5'-H5'	121.4(12)	N1-C2-C3	106.34(15)
C4'-C5'-H5'	130.2(12)	N1-C2-C6	118.21(15)
N7'-C6'-C11'	119.20(16)	C3-C2-C6	135.33(17)
N7'-C6'-C2'	113.60(15)	C2-C3-C4	107.90(17)
C11'-C6'-C2'	127.18(16)	C2-C3-H3	126.3(12)
C8'-N7'-C6'	118.78(15)	C4-C3-H3	125.8(12)
N7'-C8'-C9'	120.81(16)	C5-C4-C3	107.49(17)
N7'-C8'-C17'	117.10(15)	C5-C4-H4	126.0(12)
C9'-C8'-C17'	122.05(17)	C3-C4-H4	126.5(12)
N10'-C9'-C8'	121.26(16)	N1-C5-C4	108.43(18)
N10'-C9'-C19'	116.92(16)	N1-C5-H5	119.1(13)
C8'-C9'-C19'	121.82(17)	C4-C5-H5	132.5(13)
C9'-N10'-C11'	118.69(15)	N7-C6-C11	119.31(15)
N10'-C11'-C6'	119.36(16)	N7-C6-C2	114.41(15)
N10'-C11'-C12'	114.36(15)	C11-C6-C2	126.25(15)
C6'-C11'-C12'	126.25(16)	C8-N7-C6	118.13(15)
N13'-C12'-C16'	106.40(16)	N7-C8-C9	121.78(16)
N13'-C12'-C11'	119.29(16)	N7-C8-C17	117.07(16)
C16'-C12'-C11'	133.55(16)	C9-C8-C17	121.10(16)

N10-C9-C8	120.76(16)	C12-N13-H13	122.3(14)
N10-C9-C19	117.03(16)	N13-C14-C15	108.32(19)
C8-C9-C19	122.22(16)	N13-C14-H14	118.6(13)
C9-N10-C11	118.38(15)	C15-C14-H14	133.0(13)
N10-C11-C6	119.79(15)	C14-C15-C16	107.66(18)
N10-C11-C12	113.93(15)	C14-C15-H15	124.2(13)
C6-C11-C12	126.27(15)	C16-C15-H15	128.1(13)
N13-C12-C16	106.53(16)	C12-C16-C15	107.65(19)
N13-C12-C11	119.29(16)	C12-C16-H16	124.9(12)
C16-C12-C11	133.21(18)	C15-C16-H16	127.4(12)
C14-N13-C12	109.81(17)	N18-C17-C8	178.1(2)
C14-N13-H13	127.9(14)	N20-C19-C9	179.7(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2**. 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}
Cl1A	108(1)	67(1)	55(1)	-16(1)	-15(1)	-31(1)
Cl2A	94(1)	65(1)	53(1)	-15(1)	-7(1)	-3(1)
C1A	59(2)	52(2)	68(2)	-22(1)	0(1)	-3(1)
N1'	34(1)	30(1)	33(1)	-6(1)	-5(1)	2(1)
C2'	27(1)	26(1)	32(1)	-6(1)	-9(1)	-2(1)
C3'	30(1)	32(1)	32(1)	-8(1)	-5(1)	-2(1)
C4'	39(1)	35(1)	34(1)	-1(1)	-5(1)	-6(1)
C5'	40(1)	29(1)	42(1)	-1(1)	-9(1)	-1(1)
C6'	22(1)	30(1)	31(1)	-7(1)	-8(1)	-1(1)
N7'	28(1)	29(1)	31(1)	-7(1)	-7(1)	-1(1)
C8'	28(1)	31(1)	30(1)	-6(1)	-5(1)	-3(1)
C9'	30(1)	30(1)	29(1)	-3(1)	-6(1)	-5(1)
N10'	27(1)	29(1)	31(1)	-4(1)	-6(1)	-3(1)
C11'	21(1)	27(1)	32(1)	-3(1)	-6(1)	-2(1)
C12'	25(1)	25(1)	33(1)	-5(1)	-5(1)	0(1)
N13'	36(1)	25(1)	32(1)	-3(1)	-6(1)	1(1)
C14'	40(1)	27(1)	43(1)	-11(1)	-2(1)	1(1)
C15'	42(1)	34(1)	36(1)	-12(1)	-6(1)	-4(1)
C16'	31(1)	28(1)	35(1)	-6(1)	-7(1)	-1(1)
C17'	34(1)	32(1)	34(1)	-4(1)	-5(1)	-1(1)
N18'	45(1)	42(1)	37(1)	-7(1)	-4(1)	2(1)
C19'	41(1)	29(1)	35(1)	-6(1)	-6(1)	-2(1)
N20'	63(1)	39(1)	38(1)	-1(1)	-11(1)	-3(1)
N1	29(1)	28(1)	29(1)	-5(1)	-3(1)	1(1)
C2	24(1)	27(1)	24(1)	-5(1)	-3(1)	-4(1)
C3	28(1)	28(1)	31(1)	-7(1)	-4(1)	0(1)
C4	37(1)	28(1)	39(1)	-5(1)	-7(1)	6(1)
C5	37(1)	33(1)	33(1)	-1(1)	-5(1)	1(1)
C6	23(1)	26(1)	28(1)	-7(1)	-4(1)	-1(1)
N7	28(1)	27(1)	27(1)	-6(1)	-3(1)	0(1)
C8	29(1)	25(1)	29(1)	-9(1)	-5(1)	2(1)

C9	33(1)	25(1)	29(1)	-6(1)	-6(1)	0(1)
N10	32(1)	26(1)	28(1)	-7(1)	-4(1)	0(1)
C11	22(1)	27(1)	28(1)	-6(1)	-5(1)	0(1)
C12	26(1)	28(1)	24(1)	-3(1)	-5(1)	1(1)
N13	36(1)	29(1)	27(1)	-2(1)	-3(1)	3(1)
C14	46(1)	42(1)	23(1)	-6(1)	-4(1)	10(1)
C15	46(1)	38(1)	32(1)	-16(1)	-13(1)	6(1)
C16	32(1)	32(1)	33(1)	-8(1)	-6(1)	-2(1)
C17	36(1)	27(1)	30(1)	-4(1)	-4(1)	0(1)
N18	46(1)	36(1)	39(1)	-11(1)	1(1)	6(1)
C19	46(1)	30(1)	31(1)	-7(1)	3(1)	1(1)
N20	75(2)	30(1)	54(1)	-5(1)	7(1)	0(1)

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

	x	y	z	U(eq)
H1AA	-1590(40)	9780(20)	7975(16)	67(7)
H1AB	180(40)	10530(20)	7805(19)	87(10)
H1'	8200(30)	4510(17)	7060(15)	44(6)
H3'	4720(30)	6032(15)	8719(12)	28(5)
H4'	5300(30)	4089(16)	9447(14)	42(6)
H5'	7590(30)	3197(17)	8330(13)	40(6)
H13'	4320(30)	9407(15)	7024(13)	26(5)
H14'	3680(30)	10178(16)	8284(12)	36(5)
H15'	5200(30)	8853(16)	9434(14)	38(6)
H16'	6660(30)	7289(15)	8730(12)	28(5)
H1	3060(30)	7222(18)	5319(15)	49(7)
H3	-250(30)	8314(15)	7277(14)	39(5)
H4	-20(30)	9764(16)	5941(13)	40(6)
H5	2170(30)	8959(17)	4709(15)	50(6)
H13	-910(30)	5370(18)	9291(14)	42(6)
H14	-1690(30)	6416(16)	10400(15)	45(6)
H15	-20(30)	8102(17)	9860(13)	41(6)
H16	1670(30)	7913(16)	8350(13)	32(5)

Table 6. Torsion angles [°] for **2**.

C5'-N1'-C2'-C3'	-1.1(2)	N13'-C14'-C15'-C16'	-0.4(2)
C5'-N1'-C2'-C6'	-177.53(16)	N13'-C12'-C16'-C15'	0.7(2)
N1'-C2'-C3'-C4'	0.9(2)	C11'-C12'-C16'-C15'	170.3(2)
C6'-C2'-C3'-C4'	176.4(2)	C14'-C15'-C16'-C12'	-0.2(2)
C2'-C3'-C4'-C5'	-0.4(2)	N7'-C8'-C17'-N18'	144(8)
C2'-N1'-C5'-C4'	0.8(2)	C9'-C8'-C17'-N18'	-34(8)
C3'-C4'-C5'-N1'	-0.3(2)	N10'-C9'-C19'-N20'	-81(28)
N1'-C2'-C6'-N7'	5.3(2)	C8'-C9'-C19'-N20'	100(28)
C3'-C2'-C6'-N7'	-169.9(2)	C5-N1-C2-C3	1.0(2)
N1'-C2'-C6'-C11'	-176.48(16)	C5-N1-C2-C6	177.62(16)
C3'-C2'-C6'-C11'	8.4(3)	N1-C2-C3-C4	-0.9(2)
C11'-C6'-N7'-C8'	-8.0(2)	C6-C2-C3-C4	-176.7(2)
C2'-C6'-N7'-C8'	170.43(16)	C2-C3-C4-C5	0.5(2)
C6'-N7'-C8'-C9'	-4.3(3)	C2-N1-C5-C4	-0.7(2)
C6'-N7'-C8'-C17'	178.00(17)	C3-C4-C5-N1	0.1(2)
N7'-C8'-C9'-N10'	10.6(3)	N1-C2-C6-N7	-0.5(2)
C17'-C8'-C9'-N10'	-171.88(18)	C3-C2-C6-N7	174.89(19)
N7'-C8'-C9'-C19'	-170.14(17)	N1-C2-C6-C11	-178.23(16)
C17'-C8'-C9'-C19'	7.4(3)	C3-C2-C6-C11	-2.8(3)
C8'-C9'-N10'-C11'	-3.3(3)	C11-C6-N7-C8	8.1(2)
C19'-C9'-N10'-C11'	177.39(16)	C2-C6-N7-C8	-169.84(15)
C9'-N10'-C11'-C6'	-9.1(2)	C6-N7-C8-C9	4.1(3)
C9'-N10'-C11'-C12'	169.07(16)	C6-N7-C8-C17	-178.34(16)
N7'-C6'-C11'-N10'	15.1(3)	N7-C8-C9-N10	-10.1(3)
C2'-C6'-C11'-N10'	-163.11(17)	C17-C8-C9-N10	172.45(17)
N7'-C6'-C11'-C12'	-162.83(17)	N7-C8-C9-C19	170.31(18)
C2'-C6'-C11'-C12'	19.0(3)	C17-C8-C9-C19	-7.2(3)
N10'-C11'-C12'-N13'	14.3(2)	C8-C9-N10-C11	2.8(3)
C6'-C11'-C12'-N13'	-167.71(17)	C19-C9-N10-C11	-177.55(16)
N10'-C11'-C12'-C16'	-154.2(2)	C9-N10-C11-C6	9.3(2)
C6'-C11'-C12'-C16'	23.8(3)	C9-N10-C11-C12	-169.58(15)
C16'-C12'-N13'-C14'	-1.0(2)	N7-C6-C11-N10	-15.1(2)
C11'-C12'-N13'-C14'	-172.37(17)	C2-C6-C11-N10	162.48(17)
C12'-N13'-C14'-C15'	0.9(2)	N7-C6-C11-C12	163.58(16)

C2-C6-C11-C12	-18.8(3)
N10-C11-C12-N13	-21.7(2)
C6-C11-C12-N13	159.52(17)
N10-C11-C12-C16	145.37(19)
C6-C11-C12-C16	-33.4(3)
C16-C12-N13-C14	1.6(2)
C11-C12-N13-C14	171.81(16)
C12-N13-C14-C15	-1.4(2)
N13-C14-C15-C16	0.7(2)
N13-C12-C16-C15	-1.2(2)
C11-C12-C16-C15	-169.40(19)
C14-C15-C16-C12	0.3(2)
N7-C8-C17-N18	-142(6)
C9-C8-C17-N18	36(6)
N10-C9-C19-N20	175(100)
C8-C9-C19-N20	-5(54)

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
2	0	0	417	436	3	-3	5	0	183	171	1	2	10	0	61	64	7	-5-13	1	0	20	1	-4	-7	1	54	54	4
3	0	0	218	229	1	-2	5	0	13	8	10	3	10	0	25	29	10	-4-13	1	133	128	4	-3	-7	1	211	208	4
4	0	0	157	160	1	-1	5	0	217	217	1	4	10	0	60	64	4	-3-13	1	86	82	5	-2	-7	1	280	268	3
5	0	0	115	120	2	0	5	0	177	163	2	5	10	0	45	46	16	-2-13	1	107	112	4	-1	-7	1	192	193	2
6	0	0	37	28	6	1	5	0	26	29	2	6	10	0	48	18	21	-1-13	1	181	172	4	0	-7	1	179	177	1
7	0	0	33	29	11	2	5	0	276	276	2	7	10	0	19	7	18	0-13	1	32	6	13	1	-7	1	297	287	2
8	0	0	119	120	6	3	5	0	424	428	5	-6	11	0	29	6	23	1-13	1	16	2	16	2	-7	1	150	159	1
9	0	0	85	76	7	4	5	0	93	90	3	-5	11	0	64	55	5	2-13	1	25	31	13	3	-7	1	219	219	2
-9	1	0	86	87	7	5	5	0	24	31	7	-4	11	0	120	129	3	3-13	1	40	37	15	4	-7	1	124	127	1
-8	1	0	23	28	23	6	5	0	222	226	5	-3	11	0	126	130	2	4-13	1	73	66	10	5	-7	1	75	79	2
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5	8	16	53	58	12	5	1	17	28	10	28	1	-4	18	46	62	16	0	8	18	37	1	18						

Figure 1. View of molecule 1 of **2** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary size.

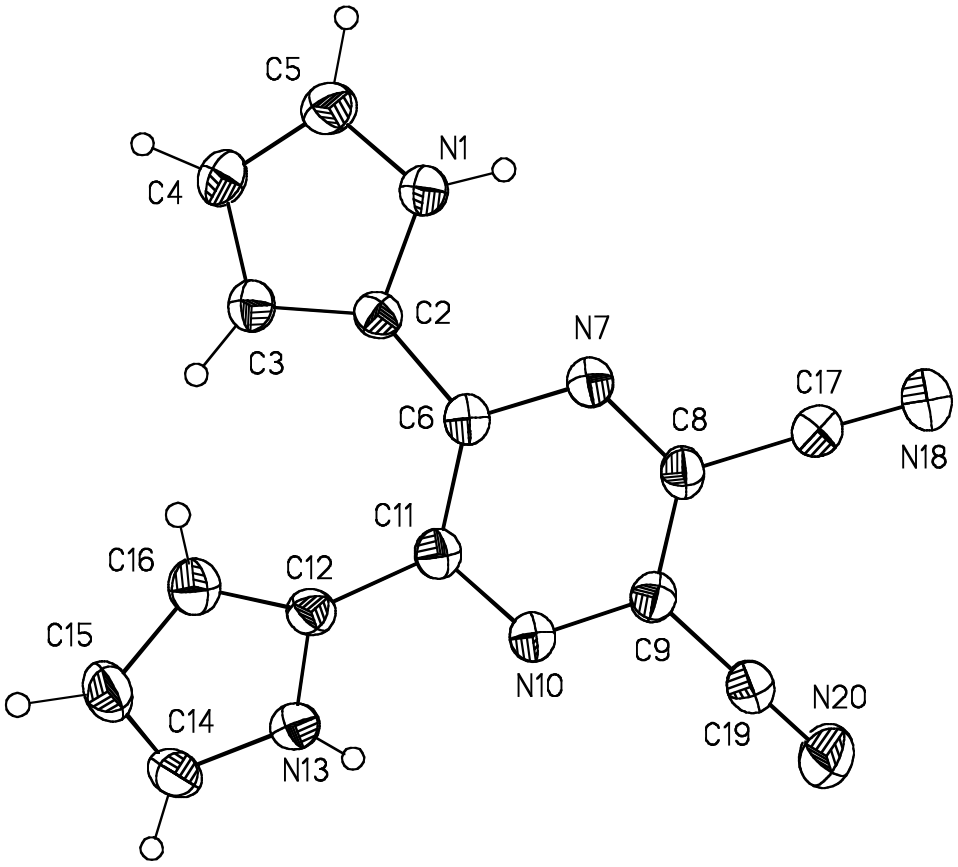


Figure 2. View of molecule 2 of **2** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary size.

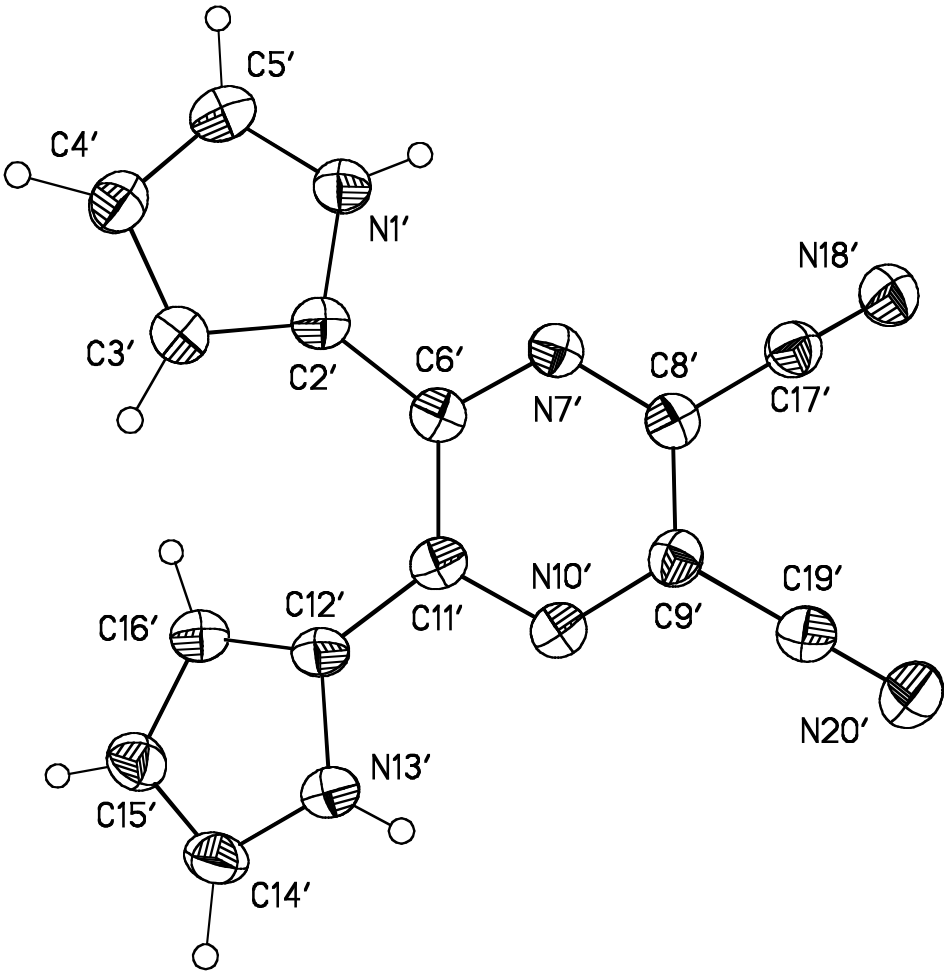


Figure 3. View of the fit by least-squares of atoms of molecule 1 onto the equivalent atoms of molecule 2. All non-H atoms were used in the fit.

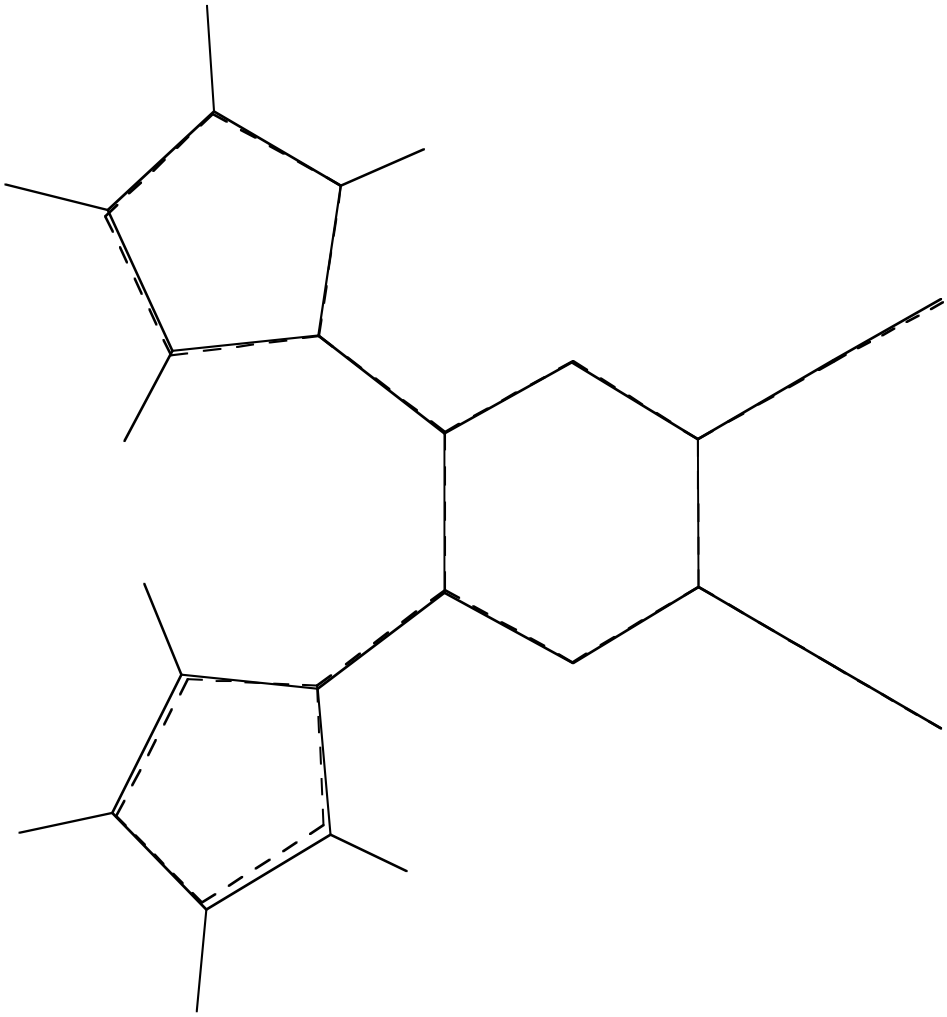
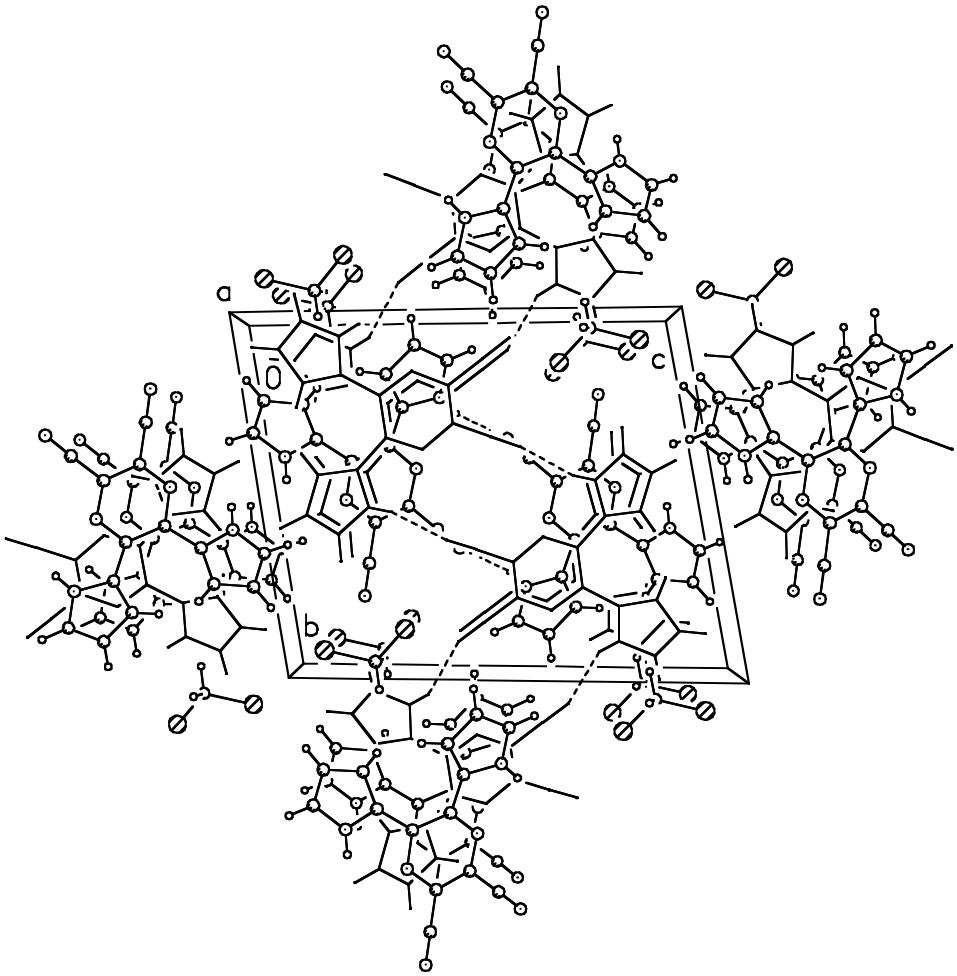


Figure 4. Unit cell packing diagram for **2**. The view is approximately down the **a** axis. Molecules 1 are shown in wireframe form while molecules 2 are displayed as ball-and-stick. The molecules stack in columns parallel to **a**. Molecules 1 and 2 alternate within a column. Dashed lines are indicative of a H-bonding interaction. The geometry of these interactions are: N1-H1···N18 (related by 1-x, 1-y, 1-z); N···N 2.958(2)Å, H···N 2.12(2)Å, N-H···N 161(2)°; N1'-H1'···N18' (related by 2-x, 1-y, 1-z); N···N 3.136(3)Å, H···N 2.30(2)Å, N-H···N 165(2)°; N13'-H13'···N20' (related by 1-x, 2-y, 1-z); N···N 3.063(2)Å, H···N 2.31(2)Å, N-H···N 152(2)°.



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[9] $R_W(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection.

$R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$.

$S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

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