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Crystal structures and packing motifs of 2,3-diphenylquinoxaline and 2,3-diphenylbenzo[*g*]quinoxaline

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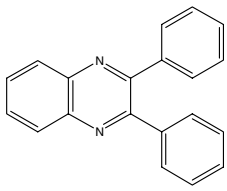
White plates of 2,3-diphenylquinoxaline crystallize in the monoclinic space group $P2_1/n$ with $Z = 4$ and $a = 6.0325(3)$ Å, $b = 10.9516(6)$ Å, $c = 22.5985(13)$ Å, and $\beta = 95.107(2)^\circ$. The phenyl rings in 2,3-diphenylquinoxaline form torsion angles of 36.88(5)° and 53.32(4)° with the plane defined by the quinoxaline moiety. Yellow plates of 2,3-diphenylbenzo[*g*]quinoxaline crystallize in the monoclinic space group $C2/c$ with $Z = 8$ and $a = 25.0621(16)$ Å, $b = 7.7190(5)$ Å, $c = 21.2225(14)$ Å, and $\beta = 123.674(2)^\circ$. The phenyl rings in 2,3-diphenylbenzo[*g*]quinoxaline form torsion angles of 46.89(3)° and 43.42(3)° with the plane defined by the benzo[*g*]quinoxaline moiety. Packing in 2,3-diphenylquinoxaline can best be described as following the herringbone motif; whereas the packing in 2,3-diphenylbenzo[*g*]quinoxaline crystals can also be described as herringbone—albeit a herringbone pattern made up of head-to-tail oriented neighbors.

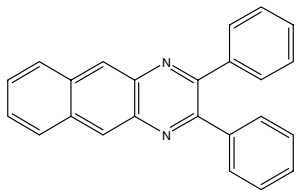
KEY WORDS: Diphenylquinoxaline; diphenylbenzo[*g*]quinoxaline; ring torsion; aromatic heterocycles.

Introduction

Quinoxalines and benzoquinoxalines have found wide use as electronic and polymeric materials,[1] biomaterials,[2] and ligands for transition metals. We have been interested in the crystal structures and metal complexes of 2,3-dithienylquinoxalines and benzo[*g*]quinoxalines in attempts to design bidentate [N,S] ligand systems utilizing quinoxaline nitrogen and thienyl sulfurs;[3] however, the majority of the published literature deals with metal complexes with 2-mono- and 2,3-di-2-pyridylquinoxalines, 2,3-di-2-pyridylbenzo[*g*]quinoxalines, and 2,3-diphenylquinoxaline. Crystal structure determinations of di-2-pyridyl compounds have shown bidentate (N,N) binding from quinoxaline and nearby pyridyl ring to Ag^+,[4] Co^{2+},[4b,5] Cu^+,[6]



I



II

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Cu^{2+},[7] Fe^{2+},[8] Os,[9] Re^{+},[10] Rh^{3+},[11] and Ru^{2+};[12] whereas 2,3-diphenylquinoxaline, **I**, has formed monodentate arrays with Na^{+},[13] K^{+},[13] and Cu^{+}.[14,15] As a ligand, **I** has shown the ability to bind Cu^{+} using its quinoxaline nitrogens with the general formula of $[Cu(\mathbf{I})_2]ClO_4$ (where the perchlorate oxygen acts as the third ligand)[14] or form polymeric networks with formulas, $[Cu(\mathbf{I})OH_2]ClO_4{\cdot}EtOH$[15] and $[Cu(\mathbf{I})OH_2]BF_4{\cdot}H_2O$[14] where a water molecule serves as the neutral third ligand. The ligand has also shown to form polymeric networks with 1,2-dimethoxyethane and Na^{+} and K^{+} with the general formula, $[M(\mathbf{I})(dme)]_n$ (where $M = Na^{+}$ and K^{+}).[13] To date, there have been no crystal structure determinations reported of metal complexes with 2,3-diphenylbenzo[g]quinoxaline, **II**. The dipyridyl- and diphenylquinoxaline reactions

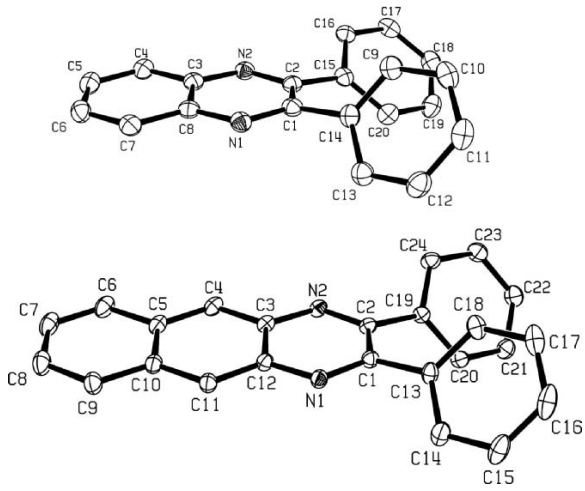


Fig. 1. ORTEPs of **I** [top] and **II** [bottom] at the 50% probability ellipsoid level.[22] [Hydrogens have been omitted for clarity.].

Table 1. Crystal Data and Structure Refinement Information for 2,3-diphenylquinoxaline (**I**) and 2,3-diphenylbenzo[*g*]quinoxaline (**II**)

	I	**II**
IUPAC name	2,3-Diphenylquinoxaline	2,3-diphenyl benzo[*g*]quinoxaline
CCDC no.	265060	265061
Color/shape	Colorless plate	Yellow plate
Chemical formula	$C_{20}H_{14}N_2$	$C_{24}H_{16}N_2$
Formula weight (g/mol)	282.33	332.39
Melting point (°C)	125	190
Temperature (K)	298(2)	100(2)
Wavelength	Cu K_α ($\lambda = 1.54178$ Å)	Mo K_α ($\lambda = 0.71073$ Å)
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$C2/c$
Unit cell dimensions		
a (Å)	6.0325(3)	25.0621(16)
b (Å)	10.9516(6)	7.7190(5)
c (Å)	22.5985(13)	21.2225(14)
β (°)	95.107(2)	123.674(2)
Volume (Å^3)	1487.05(14)	3416.7(4)
Z	4	8
Density (calculated, g/cm^3)	1.261	1.292
F_{000}	592	1392
Absorption coefficient (mm^{-1})	0.579	0.076
Diffractometer/scan	Siemens SMART/CCD	Siemens SMART/CCD
θ range for data collection	3.93 to 64.96	1.95 to 33.83
Reflections measured	2361	23836
Independent/observed reflections	2361 ($R_{int} = 0.0172$/2276 [$I>2\sigma(I)$])	6126 ($R_{int} = 0.0297$/4779 [$I>2\sigma(I)$])
Min./max. trans. factor	0.80/0.95	0.85/0.95
Data/restraints/parameters	2361/0/255	6126/0/283
Goodness of fit on F^2	1.111	1.016
Final R indices [$I>2\sigma(I)$]	$R_1 = 0.0372$, $wR_2 = 0.0950$	$R_1 = 0.0460$, $wR_2 = 0.1205$
R indices (all data)	$R_1 = 0.0382$, $wR_2 = 0.0943$	$R_1 = 0.0619$, $wR_2 = 0.1335$

served as templates for our reactions with 2,3-di-2-thienylquinoxalines; however, the main product after reaction of our dithienylquinoxaline ligand with the copper(II) perchlorate was a protonated 2,3-di-2-thienylquinoxaline—leading to the crystallization and structure determination of a 2,3-dithienylquinoxalin-1-ium perchlorate salt.[16] During our reinvestigations into the method to which our dithienylquinoxaline was protonated, we synthesized and characterized **I** and **II** as model ligands that did not react to form quinoxaline-1-ium perchlorates. In this paper we describe the crystal structures of these two compounds that were used as a reference in our studies: 2,3-diphenylquinoxaline, **I**, and 2,3-diphenylbenzo[*g*]quinoxaline, **II**.

Experimental

The synthesis of 2,3-diphenylquinoxaline (**I**) is straightforward and has been published elsewhere.[17] In general, it consists of either the direct combination of benzil with 1,2-diaminobenzene in warmed ethanol or a direct solvent-free reaction where equal molar amounts of each reactant are placed in a test tube which is then placed in boiling water. The latter reaction is interesting because benzil and impure 1,2-diaminobenzene melt under 100°C; whereas diphenylquinoxaline has a melting point of 125°C[17] and therefore precipitates upon formation. Once purified by recrystallization, crystals were formed by slowly evaporating solutions of **I** in warmed ethanol.

The synthesis of 2,3-diphenylbenzo[*g*]quinoxaline (**II**) was performed as follows. To a 250 mL round bottom flask equipped with a reflux condenser, 0.158 g (1 mmol) 2,3-diaminonapthalene was combined with 0.210 g (1 mmol) benzil in toluene. The reaction mixture was gently refluxed for 43 h. The resulting bright yellow solution was cooled to room temperature then chilled to 0°C until product precipitated. The suspension was vacuum filtered and purified by flash chromatography. The reaction yielded 0.254 g of **II** as a bright yellow solid (77%). R_f 0.72 (SiO_2, 90% Pet. Ether: 10% EtOAc); mp 190°C; IR (Nujol) 990, 890, 730, 710 cm^{-1};^{1}H NMR (400 MHz, $CDCl_3$) 8.761 (s, 2H), 8.128 (dd, 2H, $J = 6.4$ Hz, 3.2 Hz), 7.587 (d, 2H, $J = 6.4$ Hz), 7.583 (d, 4H, $J = 6.4$ Hz), 7.378 (m, 6H);^{13}C NMR (400 MHz, $CDCl_3$) 154.100, 139.230, 137.956, 134.039, 129.684, 128.531, 128.277, 127.576, 127.552, 126.179; MS calcd for $C_{24}H_{16}N_2$: M^+:332, measured: 332.

Single crystal data was collected at room temperature for **I** and 100 K for **II** using a Bruker Kappa Diffractometer equipped with a 4 k CCD detector utilizing the SMART software package.[18] The data was integrated using SAINT.[19] Absorption corrections were applied to data by running SADABS on merged data collection runs.[20] Structure refinement and solution was performed using SHELXTL.[21] For both structures, hydrogen atom positions were refined as were their isotropic thermal parameters. After inspection of output files during the initial phases of data refinement for both **I** and **II**, the extinction coefficients were not refined. ORTEPs (50% probability ellipsoids) of **I** and **II** are shown (without hydrogen atoms) in Fig. 1; whereas primary crystal data and structure refinement information for **I** and **II** are shown in Table 1. Supplementary materials for **I** and **II** include complete *cif* files containing fractional coordinates, anisotropic thermal parameters, bond lengths, angles, torsions, and additional geometric and refinement statistics.

Results

Packing in **I** can best be described as herringbone as is packing in **II**—albeit a herringbone pattern made up of head-to-tail oriented neighbors. (Packing diagrams for **I** (100) and **II** (010) are illustrated in Fig. 2.) In **I** and **II**, all N–C, C–C, and C–H bond lengths and angles are within the expected values for quinoxalines. However, not all N–C bonds in each heterocycle are equivalent. N–C bonds between N1–C8 and N2–C3 in **I** and N1–C12 & N2–C3 in **II** are longer than the corresponding N1–C1 and N2–C2

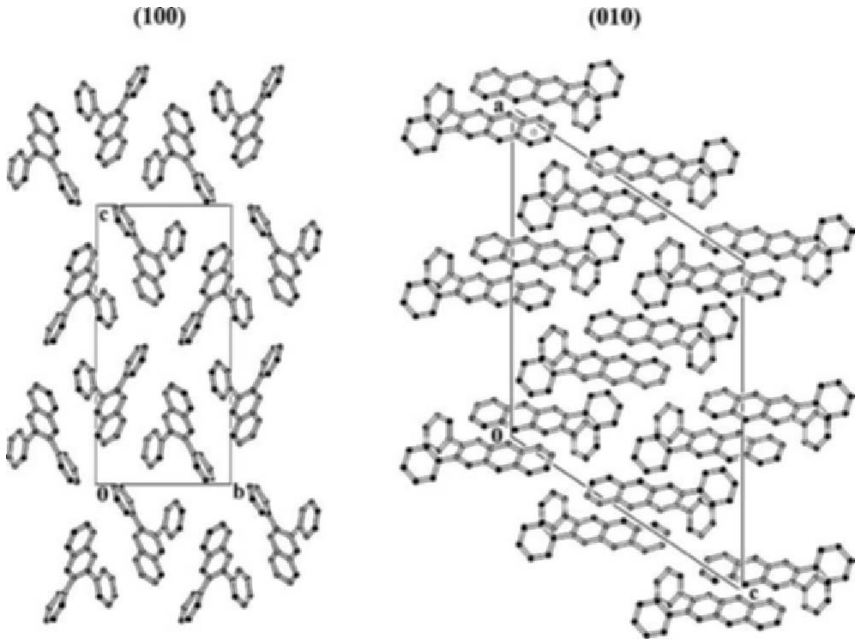


Fig. 2. Packing diagrams showing the (100) face of **I** [left] and the (010) face of **II** [right].[23] [Hydrogen atoms have been omitted for clarity.].

bonds in **I** and N1–C1 and N2–C2 bonds in **II** (Table 2). This non-equivalence is common for quinoxalines. From a statistical analysis of organic compounds containing the quinoxaline moiety in the CSD, the N–C bond lengths are shorter for those bonds close to the 2- and 3-positions on the quinoxaline heterocycle as shown in Table 2.[24]

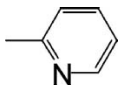
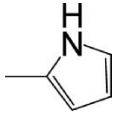
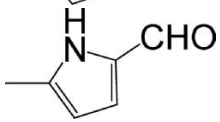
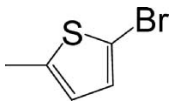
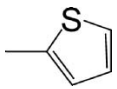
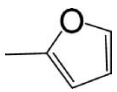
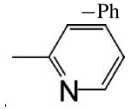
Table 2. Crystal Data and Structure Refinement Information for 2,3-diphenylquinoxaline **(I)** and 2,3-Diphenylbenzo[*g*]quinoxaline **(II)** and all Purely Organic Molecules in the CSD Containing the Quinoxaline Moiety.

Quinoxaline C–N Bonds[a]	Compounds	Label	Distance (e.s.d.)
$N–C_m$			
	I	N1–C8	1.3715(19) Å
	I	N2–C3	1.3672(19) Å
	II	N1–C12	1.3764(11) Å
	II	N2–C3	1.3774(11) Å
	CSD[24]		1.3738 Å (±0.0195)[b]
$N–C_a$			
	I	N1–C1	1.3209(19) Å
	I	N2–C2	1.3250(19) Å
	II	N1–C1	1.3106(11) Å
	II	N2–C2	1.3115(11) Å
	CSD[24]		1.3162 Å (±0.0251)[b]

[a] $N–C_m$ represents the N–C bond pointing toward the middle of the quinoxaline moiety; whereas $N–C_a$ represents the nitrogen–carbon bond toward the carbons at the 2- and 3-position on the heterocycle. [b] Standard deviation.

I and **II** display a slight bowing of the quinoxaline moiety. The root-mean-square deviation from ideal planarity for the quinoxaline atoms in **I** was 0.0350(12) Å; whereas for **II**, the value was 0.0406(8) Å for all fitted non-hydrogen atoms in the benzo[*g*]quinoxaline unit. Using an illustrative technique commonly employed for porphyrins,[25] deviations for each individual atom from the mean plane are shown in Fig. 3 and indicate a bowing of the ideally planar heterocycle. This distortion of the quinoxaline moiety could originate internally from the unfavorable steric interactions between the rings substituted at the 2- and 3-positions or could stem from packing effects. A simple case for the former can be made from examination of existing 2,3-diarylquinoxalines and benzo[*g*]quinoxalines in the literature. There are crystal structures of six other simple, non-ionic, heteroatomic, 2,3-disubstituted quinoxalines to which to compare to **I** as well as one crystal structure of

Table 3. Geometric Relationships and Packing Motifs in 2,3-diphenylquinoxaline (**I**), 2,3-diphenylbenzo[*g*]quinoxaline (**II**), and Related Compounds

	Compound name	CSD code	*R*-group	Ring torsion angles (RTA, °)	Ring torsion angles (RTA, °)	Plane deformation (rms, Å)	Reference
I	2,3-diphenylquinoxaline		-Ph	36.88(5)	53.32(4)	0.0350(12)	This work
a	2,3-bis(2-pyridyl)quinoxaline	JEWLIG		27.99	43.96	0.0744	(26)
b	2,3-bis(2-pyrrolyl)quinoxaline	AWIXOT		1.13	88.76	0.0263	(27)
c	2,3-bis(5-formyl-2-pyrrolyl)quinoxaline	AWIXUZ		32.76	32.76	0.0577	(27)
d	2,3-bis(5-bromo2-thienyl)quinoxaline	ETIXAG		26.60	27.02	0.0607	(3b)
e	2,3-di-2-thienylquinoxaline	GUWCEG		9.31	83.38	0.0081	(3a)
f	2,3-di-2-furylquinoxaline	LUQWID		7.27	64.85	0.0320	(28)
II	2,3-diphenylbenzo[*g*]quinoxaline		−Ph	43.42(3)	46.89(3)	0.0406(8)	This work
g	2,3-bis(2-pyridyl)benzo[*g*]quinoxaline	SOSREX		24.73	46.05	0.0874	(26)

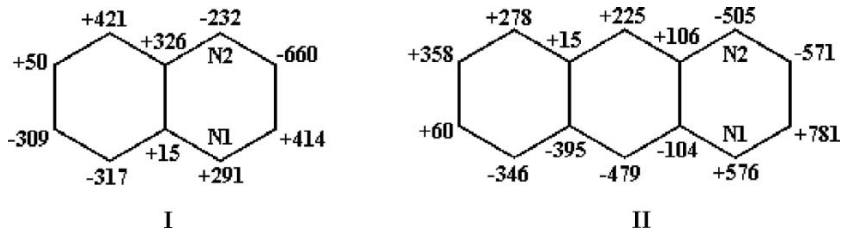


Fig. 3. Deviation of atoms from the mean plane in units of 0.001 Å in the quinoxaline and benzo[*g*]quinoxaline moieties in compounds **I** and **II**, respectively.

a 2,3-bis(2-pyridyl)benzo[*g*]quinoxaline[11] which is analogous to **II**. Immediately evident are the similarities in ring torsions for the phenyl and pyridyl quinoxalines and benzo[*g*]quinoxalines despite difference in packing—suggesting that steric interactions predominate.[11,26] From inspection of Table 3, it is evident that as one ring becomes coplanar with the quinoxaline, the buckling of the quinoxaline diminishes suggesting that steric strain is diminished (Fig. 4). This is seen with all 2,3-diarylquinoxalines with unsubstituted 5-membered heterocyclic rings (*b*[27], e[3a], & f[28]). However, the lower steric interactions of five-membered heterocyclic rings as opposed to six-membered rings at the 2- and 3-positions on quinoxalines are not the only factor

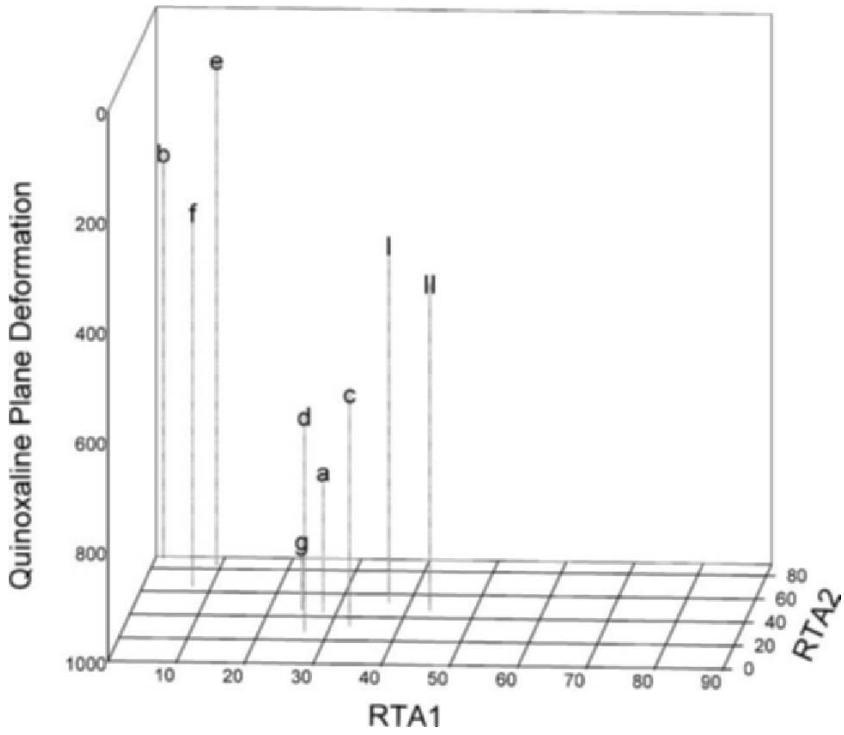


Fig. 4. Graph showing the ring torsion angle 1 (*x*-axis), ring torsion angle 2 (*y*-axis), and deviation of atoms from the mean plane of the quinoxaline and/or benzo[*g*]quinoxaline moiety in units of 0.001 Å (*z*-axis) for the quinoxaline and benzo[*g*]quinoxalines in Table 3. All unsubstituted five-membered heterocycles (b, e, & f) have one ring nearly coplanar with the quinoxaline and hence result in less quinoxaline buckling..

in ring torsions and quinoxaline deformation. Structural studies on a larger ring system, that of 2,3-bis(benzimidazol-2-yl)quinoxaline[29] which has ring torsion angles of 22.17° and 41.04°, only has a quinoxaline plane deformation of 0.0319 Å which is comparable to the deformation in 2,3-di-2-furylquinoxaline (f).[28] Therefore, it is interesting to bring into play the nature of packing. The majority of the compounds in the table adopt variants of herringbone packing motifs—utilizing a mixture of π–π interactions and intermolecular C–H interactions; but crystals of 2,3-bis(benzimidazol-2-yl)quinoxaline are more heavily directed by π–π layer stacking.[29] Molecules in the bulkier quinoxaline pack as tilted head-to-tail dimers that utilize stacking interactions between the quinoxaline moiety and a neighboring molecule's larger ring substituent. Therefore, the deformation of the quinoxaline moiety is not due to intramolecular steric effects alone.

Supplementary material CCDC 265060 and 265061 contain supplementary crystallographic data for this paper. These data can be obtained free of charge by contacting The Cambridge Crystallographic Data Centre via mail sent to 12 Union Road, Cambridge CB2 1EZ, UK, [Fax: +44(0)1223-336033.],via email [data_request@ccdc.cam.ac.uk], or via the internet [www.ccdc.cam.ac.uk/data_request/cif].

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