Crystal structure of 3-phenylquinoxalin-2(1*H*)-one, $C_{14}H_{10}N_2O$

Xandri Schoultz, Thomas I. A. Gerber, Eric C. Hosten and Richard Betz*

Nelson Mandela Metropolitan University, Summerstrand Campus, Department of Chemistry, University Way, Summerstrand, PO Box 77000, Port Elizabeth 6031, South Africa

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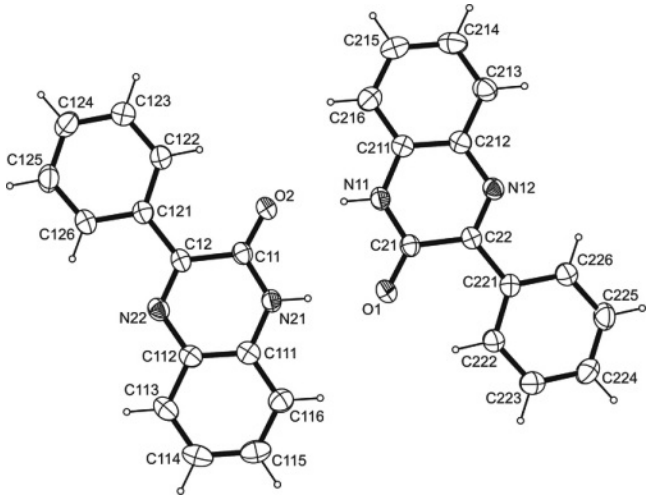

Abstract

$C_{14}H_{10}N_2O$, orthorhombic, *Pna*2$_1$ (no. 33), a = 17.6745(7) Å, b = 5.3858(2) Å, c = 22.4721(9) Å, V = 2139.1 Å^3, Z = 8, $R_{gt}(F)$ = 0.0386, $wR_{ref}(F^2)$ = 0.1092, T = 200 K.

Table 1. Data collection and handling.

Crystal:	colourless platelets, size 0.127×0.184×0.434 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.89 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18687, 2719
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2273
$N(param)_{refined}$:	307
Programs:	SHELX, WinGX, MERCURY, PLATON [12–15]

Source of material

The compound was obtained upon reacting (1*H*-benzo[*d*]-imidazol-2-yl)(phenyl)methanone with $ReO_2I(PPh_3)_2$ in methanol. Crystals suitable for the diffraction study were obtained upon slow evaporation of the solvent at ambient conditions.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). Nitrogen-bound H atoms were placed in calculated positions (N–H 0.88 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(N). Due to the absence of a strong anomalous scatterer, the Flack parameter is meaningless. Thus, Friedel opposites (2335 pairs) have been merged and the item was removed from the CIF.

* Correspondence author (e-mail: Richard.Betz@nmmu.ac.za)

Discussion

Next to cardiovascular diseases, cancer has become one of the main fatal diseases in industrialized countries. Apart from classical surgery, chemo- and radiotherapeutic treatments have entered the arsenal of possible cures for certain types of cancer. All methods, however, suffer from their own set of problematic side-effects and, as a consequence, the development of radiopharmaceuticals – combining the advantages of chemotherapy as well as radiation methods while at the same time avoiding their unique respective undesired side-effects – has been a topic of research [1, 2]. Tailoring and fine-tuning of the envisioned radiopharmaceuticals' properties such as lipophilicity and, in particular, inertness is of paramount importance with respect to possible future *in vivo* applications in contemporary medicine and requires sound knowledge about structural parameters of the ligands applied if a more heuristic approach in the synthesis is to triumph over pure trial-and-error as it is encountered in this specific field of coordination chemistry up to the present day. In continuation of our interest in rhenium-based coordination compounds that might serve as radiopharmaceuticals, a rhenium(V) starting material was reacted with a derivative of benzimidazole. A structural analysis of the crystalline reaction product showed the formation of an unexpected compound, namely a derivative of quinoxalinone. The crystal structures of other quinoxalinone derivatives featuring a methyl group [3, 4], a carboxyl group [5] or a pyrrol moiety [6] as substituents on the heterocyclic scaffold have been reported earlier. The title compound is a derivative of quinoxalinone bearing a phenyl group on the scaffold of the heterocyclic ring. The asymmetric unit is comprised of two complete molecules. Both molecules are essentially flat (r.m.s. of all fitted non-hydrogen atoms = 0.0388 Å and 0.0576 Å, respectively) and nearly co-planar as the least-squares planes defined by their respective non-hydrogen atoms enclose an angle of only 0.057(6)°. The O=C–N(H) bond lengths along the intracyclic amide moieties hardly differ in between the two crystallographically independant molecules present in the asymmetric unit with 1.348(3) Å and 1.350(3) Å for the two C–N bond lengths and 1.241(3) Å for both C=O bond lengths. In comparison to other quinoxalinone derivatives whose metrical parameters have been deposited with the Cambridge Structural Database [7] the two C–N bond lengths found in the title compound slightly deviate to shorter values than reported as the most common values while the opposite is true for the C=O bonds that show marked elongation with respect to the values reported for similar compounds. The latter finding might be in agreement with an increased degree of amide-type resonance stabilization of this respective functional group in the title compound. A conformational analysis of the heterocyclic six-membered rings according to Cremer & Pople [8] is precluded by the small puckering amplitudes (τ = 1.6° and 1.9°, respectively). In the crystal, classical hydrogen bonds of the N–H···O type are observed next to C–H···O and C–H···N contacts, the range of the

latter ones invariably falling below the sum of van-der-Waals radii of the atoms participating in them [9]. The intermolecular N–H···O contacts connect the two molecules present in the asymmetric unit to centrosymmetric dimers. The C–H···O and C–H···N contacts are all intramolecular and are supported by hydrogen atoms in *ortho* position on the phenyl moieties bonded to the heterocyclic scaffold. In terms of graph-set analysis [10, 11], the descriptor for the classical hydrogen bonds is *DD* on the unary and $R^2_2(8)$ on the binary level while the contacts involving C–H groups require a *S*(5)*S*(6) descriptor on the unary level each for the two different acceptor atoms. C···O interactions between the two different molecules present in the asymmetric unit and their symmetry-related equivalents extend these dimers to infinite chains along the crystallographic *b* axis. In addition, two C–H···π contacts can be observed that stem from the hydrogen atoms in *para* position on the phenyl groups attached to the heterocyclic scaffold and always have the all-carba aromatic system of the quinoxalinone moiety as acceptor. The shortest intercentroid distance between two centers of gravity was measured at 4.0895(14) Å and is apparent between the two different six-membered ring systems of the quinoxalinone moiety of the same molecule present in the asymmetric unit and its symmetry-related equivalent.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11)	4*a*	0.3354	0.4938	0.2064	0.034
H(21)	4*a*	0.4272	0.5180	0.1218	0.034
H(113)	4*a*	0.4855	1.0029	–0.0712	0.043
H(114)	4*a*	0.5826	0.7204	–0.0885	0.049
H(115)	4*a*	0.6048	0.3966	–0.0219	0.048
H(116)	4*a*	0.5306	0.3523	0.0629	0.042
H(122)	4*a*	0.2385	1.0557	0.1478	0.042
H(123)	4*a*	0.1399	1.3416	0.1514	0.049
H(124)	4*a*	0.1267	1.6392	0.0768	0.046
H(125)	4*a*	0.2117	1.6459	–0.0023	0.048
H(126)	4*a*	0.3084	1.3561	–0.0081	0.043
H(213)	4*a*	0.2697	–0.0035	0.3960	0.043
H(214)	4*a*	0.1749	0.2845	0.4143	0.050
H(215)	4*a*	0.1552	0.6156	0.3486	0.047
H(216)	4*a*	0.2309	0.6612	0.2644	0.042
H(222)	4*a*	0.5206	–0.0623	0.1807	0.038
H(223)	4*a*	0.6180	–0.3546	0.1790	0.047
H(224)	4*a*	0.6333	–0.6334	0.2575	0.047
H(225)	4*a*	0.5496	–0.6211	0.3379	0.047
H(226)	4*a*	0.4531	–0.3294	0.3408	0.040

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*a*	0.43871(9)	0.2385(3)	0.17357(9)	0.0355(8)	0.035(1)	0.030(1)	0.0014(8)	0.0050(7)	0.0089(7)
O(2)	4*a*	0.32409(9)	0.7723(3)	0.15563(9)	0.0364(9)	0.035(1)	0.029(1)	0.0039(8)	0.0059(7)	0.0090(7)
N(11)	4*a*	0.3434(1)	0.3715(4)	0.23162(9)	0.030(1)	0.027(1)	0.029(1)	–0.0008(8)	–0.0007(8)	0.0058(8)
N(12)	4*a*	0.3682(1)	–0.0066(4)	0.31076(8)	0.029(1)	0.031(1)	0.0231(9)	–0.0001(8)	–0.0010(8)	0.0017(8)
N(21)	4*a*	0.4181(1)	0.6388(4)	0.09638(9)	0.0292(9)	0.028(1)	0.028(1)	–0.0001(8)	–0.0004(8)	0.0061(8)
N(22)	4*a*	0.3900(1)	1.0116(4)	0.01641(9)	0.028(1)	0.035(1)	0.026(1)	–0.0026(9)	–0.0018(8)	0.0033(8)
C(11)	4*a*	0.3609(1)	0.7964(5)	0.1090(1)	0.027(1)	0.028(1)	0.024(1)	–0.003(1)	–0.0028(8)	0.002(1)
C(12)	4*a*	0.3472(1)	0.9930(4)	0.0633(1)	0.026(1)	0.027(1)	0.024(1)	–0.0056(9)	–0.0036(9)	0.0030(9)
C(21)	4*a*	0.4005(1)	0.2137(4)	0.2195(1)	0.026(1)	0.025(1)	0.026(1)	–0.0023(9)	–0.0023(9)	0.002(1)
C(22)	4*a*	0.4126(1)	0.0146(4)	0.2649(1)	0.027(1)	0.024(1)	0.023(1)	–0.0038(9)	–0.0025(9)	0.0000(9)
C(111)	4*a*	0.4631(1)	0.6540(5)	0.0466(1)	0.025(1)	0.031(1)	0.029(1)	–0.007(1)	–0.0033(9)	–0.0027(9)
C(112)	4*a*	0.4488(1)	0.8479(5)	0.0069(1)	0.026(1)	0.034(2)	0.028(1)	–0.004(1)	–0.0008(9)	–0.001(1)
C(113)	4*a*	0.4945(2)	0.8716(6)	–0.0439(1)	0.035(1)	0.046(2)	0.026(1)	–0.006(1)	0.002(1)	0.003(1)
C(114)	4*a*	0.5518(2)	0.7064(6)	–0.0541(1)	0.033(1)	0.055(2)	0.033(1)	–0.009(1)	0.008(1)	–0.008(1)
C(115)	4*a*	0.5652(2)	0.5113(6)	–0.0142(1)	0.029(1)	0.043(2)	0.048(2)	–0.002(1)	0.004(1)	–0.012(1)
C(116)	4*a*	0.5214(1)	0.4845(5)	0.0358(1)	0.031(1)	0.035(1)	0.039(1)	–0.001(1)	–0.000(1)	–0.002(1)
C(121)	4*a*	0.2841(1)	1.1727(5)	0.0692(1)	0.027(1)	0.029(1)	0.027(1)	–0.0034(9)	–0.0039(9)	0.0013(9)
C(122)	4*a*	0.2330(1)	1.1740(5)	0.1167(1)	0.035(1)	0.034(1)	0.035(1)	0.002(1)	0.002(1)	0.008(1)
C(123)	4*a*	0.1746(2)	1.3453(6)	0.1192(1)	0.039(1)	0.044(2)	0.041(1)	0.006(1)	0.007(1)	0.007(1)
C(124)	4*a*	0.1665(2)	1.5212(5)	0.0749(1)	0.037(1)	0.038(2)	0.040(1)	0.010(1)	–0.006(1)	–0.002(1)
C(125)	4*a*	0.2169(2)	1.5242(5)	0.0280(1)	0.045(2)	0.039(2)	0.035(1)	0.007(1)	–0.007(1)	0.009(1)
C(126)	4*a*	0.2746(2)	1.3525(5)	0.0247(1)	0.042(1)	0.039(2)	0.027(1)	0.004(1)	0.000(1)	0.007(1)
C(211)	4*a*	0.2966(1)	0.3554(4)	0.2806(1)	0.026(1)	0.028(1)	0.027(1)	–0.003(1)	–0.0017(9)	–0.0024(9)
C(212)	4*a*	0.3093(1)	0.1583(5)	0.3194(1)	0.027(1)	0.032(1)	0.024(1)	–0.002(1)	–0.0030(8)	–0.001(1)
C(213)	4*a*	0.2624(1)	0.1318(6)	0.3695(1)	0.035(1)	0.046(2)	0.027(1)	0.001(1)	0.001(1)	0.003(1)
C(214)	4*a*	0.2061(2)	0.3016(6)	0.3801(1)	0.035(1)	0.056(2)	0.033(1)	0.002(1)	0.006(1)	–0.007(1)
C(215)	4*a*	0.1944(2)	0.4992(6)	0.3409(1)	0.031(1)	0.042(2)	0.044(2)	0.005(1)	0.003(1)	–0.008(1)
C(216)	4*a*	0.2391(1)	0.5271(5)	0.2911(1)	0.032(1)	0.032(1)	0.042(1)	0.001(1)	–0.001(1)	–0.001(1)
C(221)	4*a*	0.4761(1)	–0.1641(4)	0.2607(1)	0.025(1)	0.025(1)	0.028(1)	–0.0032(9)	–0.0040(9)	0.0012(9)
C(222)	4*a*	0.5262(1)	–0.1750(5)	0.2129(1)	0.031(1)	0.035(1)	0.030(1)	0.003(1)	0.001(1)	0.005(1)
C(223)	4*a*	0.5843(2)	–0.3494(5)	0.2119(1)	0.036(1)	0.043(2)	0.037(1)	0.005(1)	0.005(1)	0.001(1)
C(224)	4*a*	0.5935(2)	–0.5151(5)	0.2583(1)	0.034(1)	0.036(2)	0.046(2)	0.009(1)	–0.003(1)	0.002(1)
C(225)	4*a*	0.5439(2)	–0.5068(5)	0.3060(1)	0.040(1)	0.040(2)	0.038(1)	0.004(1)	–0.006(1)	0.011(1)
C(226)	4*a*	0.4863(1)	–0.3338(5)	0.3076(1)	0.033(1)	0.036(1)	0.032(1)	0.002(1)	0.002(1)	0.005(1)

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