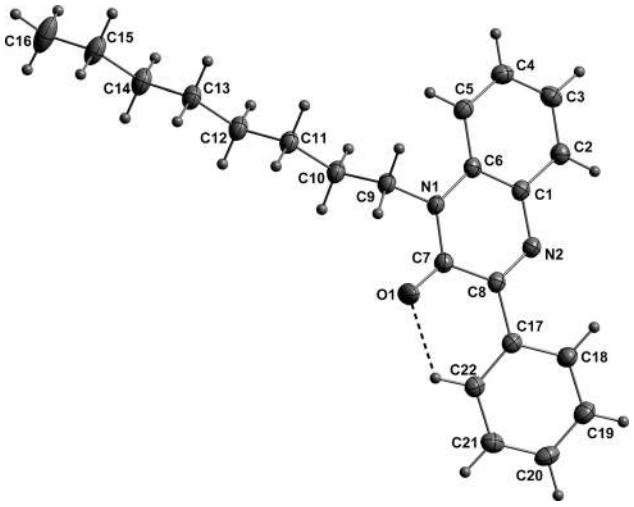
Nadeem Abad, Souad Ferfra, El Mokhtar Essassi, Joel T. Mague and Youssef Ramli*

Synthesis and crystal structure of 1-octyl-3-phenylquinoxalin-2(1*H*)-one, $C_{22}H_{26}N_2O$



https://doi.org/10.1515/ncrs-2020-0404
Received July 26, 2020; accepted August 28, 2020; published online October 6, 2020

Abstract

$C_{22}H_{26}N_2O$, monoclinic, *C*2/*c* (no. 15), a = 47.4887(9) Å, b = 5.0220(1) Å, c = 16.4551(3) Å, β = 108.993(1)°, V = 3710.70(12) Å^3, Z = 8, $R_{gt}(F)$ = 0.0345, $wR_{ref}(F^2)$ = 0.0935, T = 150(2) K.

CCDC Nr.: 2025941

The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.18 × 0.14 × 0.12 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	0.57 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE PHOTON 100, ω
θ_{max}, completeness:	72.5°, >99%
$N(hkl)_{meas.}$, $N(hkl)_{unique}$, R_{int}:	14888, 3644, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3231
$N(param)_{refined}$:	331
Programs:	Bruker [1], SHELX [2–4], Diamond [5]

Source of material

3-Phenylquinoxalin-2-one 1 (4.5 mmol), potassium carbonate (5.85 mmol) and tetrakis(*n*-butyl)ammonium bromide (0.5 mmol) in DMF (20 mL) were added to 1-bromooctane (9 mmol). Stirring was maintained at room temperature for 24 h. The crude residue was filtered and the solvent removed. The residue was extracted with water. The organic compounds were purified by column chromatography using hexane-ethyl acetate (*v*/*v*, 9/1). A portion of the product was dissolved in ethanol, the solution was filtered and the filtrate was left undisturbed for 7 days to form colorless block crystals.

Experimental details

Crystal data, data collection and structure refinement details are summarized in Table 1. Hydrogen atoms were added using riding models [4].

Comment

Quinoxaline and its derivatives have received considerable attention due to their pharmacological activity [6, 7] and industrial properties [8–10]. Our research group has

***Corresponding author: Youssef Ramli**, Laboratory of Medicinal Chemistry, Drug Sciences Research Center, Faculty of Medicine and Pharmacy, Mohammed V University, Rabat, Morocco, E-mail: y.ramli@um5s.net.ma. https://orcid.org/0000-0002-6885-5692
Nadeem Abad, Laboratory of Medicinal Chemistry, Drug Sciences Research Center, Faculty of Medicine and Pharmacy, Mohammed V University, Rabat, Morocco; and Laboratory of Heterocyclic Organic Chemistry, Faculty of Sciences, Mohammed V University, Rabat, Morocco, E-mail: abadnadeem3@gmail.com
Souad Ferfra and El Mokhtar Essassi, Laboratory of Heterocyclic Organic Chemistry, Faculty of Sciences, Mohammed V University, Rabat, Morocco, E-mail: emessassi@yahoo.fr (E.M. Essassi)
Joel T. Mague, Department of Chemistry, Tulane University, New Orleans, LA 70118, USA, E-mail: joelt@tulane.edu

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.37437 (2)	0.16657 (17)	0.45497 (6)	0.0368 (2)
N1	0.38156 (2)	0.49212 (17)	0.36879 (5)	0.0222 (2)
N2	0.44122 (2)	0.34815 (17)	0.41442 (5)	0.0219 (2)
C1	0.42983 (2)	0.5480 (2)	0.35551 (6)	0.0216 (2)
C2	0.44919 (2)	0.6824 (2)	0.32061 (6)	0.0251 (2)
H2	0.4704 (3)	0.630 (3)	0.3419 (8)	0.028 (3)*
C3	0.43885 (2)	0.8820 (2)	0.26107 (7)	0.0274 (2)
H3	0.4528 (3)	0.975 (3)	0.2376 (9)	0.036 (3)*
C4	0.40877 (2)	0.9525 (2)	0.23542 (7)	0.0280 (2)
H4	0.4012 (3)	1.095 (3)	0.1932 (8)	0.031 (3)*
C5	0.38934 (2)	0.8274 (2)	0.26973 (7)	0.0264 (2)
H5	0.3684 (3)	0.882 (3)	0.2512 (8)	0.036 (3)*
C6	0.39973 (2)	0.6243 (2)	0.33069 (6)	0.0216 (2)
C7	0.39167 (2)	0.2841 (2)	0.42594 (6)	0.0238 (2)
C8	0.42397 (2)	0.2217 (2)	0.44838 (6)	0.0212 (2)
C9	0.35041 (2)	0.5759 (2)	0.35087 (7)	0.0250 (2)
H9A	0.3451 (3)	0.519 (3)	0.4022 (8)	0.031 (3)*
H9B	0.3501 (3)	0.775 (3)	0.3471 (8)	0.030 (3)*
C10	0.32942 (2)	0.4528 (2)	0.26888 (7)	0.0281 (2)
H10A	0.3285 (3)	0.252 (3)	0.2781 (9)	0.040 (4)*
H10B	0.3378 (3)	0.480 (3)	0.2221 (8)	0.031 (3)*
C11	0.29832 (2)	0.5728 (2)	0.24530 (7)	0.0287 (2)
H11A	0.2896 (3)	0.545 (3)	0.2937 (9)	0.042 (4)*
H11B	0.2999 (3)	0.774 (3)	0.2375 (9)	0.040 (4)*
C12	0.27681 (2)	0.4609 (2)	0.16224 (8)	0.0311 (3)
H12A	0.2751 (3)	0.263 (3)	0.1679 (10)	0.050 (4)*
H12B	0.2852 (3)	0.488 (3)	0.1154 (9)	0.042 (4)*
C13	0.24592 (2)	0.5850 (2)	0.13698 (7)	0.0305 (3)
H13A	0.2368 (3)	0.550 (3)	0.1833 (9)	0.040 (4)*
H13B	0.2477 (3)	0.784 (3)	0.1309 (9)	0.042 (4)*
C14	0.22466 (2)	0.4791 (3)	0.05239 (8)	0.0335 (3)
H14A	0.2230 (3)	0.273 (3)	0.0567 (10)	0.049 (4)*
H14B	0.2338 (3)	0.509 (3)	0.0055 (9)	0.044 (4)*
C15	0.19380 (3)	0.6022 (3)	0.02715 (8)	0.0374 (3)
H15A	0.1846 (3)	0.570 (3)	0.0730 (10)	0.050 (4)*
H15B	0.1960 (4)	0.812 (4)	0.0221 (11)	0.062 (5)*
C16	0.17279 (3)	0.4975 (4)	−0.05752 (9)	0.0515 (4)
H16A	0.1526 (4)	0.587 (3)	−0.0718 (10)	0.058 (5)*
H16B	0.1699 (5)	0.298 (5)	−0.0545 (13)	0.082 (6)*
H16C	0.1812 (4)	0.530 (4)	−0.1049 (12)	0.063 (5)*
C17	0.43803 (2)	0.0145 (2)	0.51375 (6)	0.0219 (2)
C18	0.46925 (2)	0.0058 (2)	0.54544 (7)	0.0263 (2)
H18	0.4807 (3)	0.139 (3)	0.5266 (8)	0.029 (3)*
C19	0.48400 (2)	−0.1845 (2)	0.60508 (7)	0.0298 (2)
H19	0.5058 (3)	−0.187 (3)	0.6259 (9)	0.035 (3)*
C20	0.46794 (3)	−0.3703 (2)	0.63474 (7)	0.0287 (2)
H20	0.4778 (3)	−0.506 (3)	0.6765 (9)	0.035 (3)*
C21	0.43712 (2)	−0.3632 (2)	0.60437 (7)	0.0269 (2)
H21	0.4252 (3)	−0.490 (3)	0.6244 (8)	0.033 (3)*
C22	0.42213 (2)	−0.1728 (2)	0.54443 (6)	0.0237 (2)
H22	0.4000 (3)	−0.169 (3)	0.5242 (8)	0.030 (3)*

recently reported the synthesis of novel quinoxaline-based compounds [11–14]. In continuation of our efforts toward the discovery of novel quinoxaline derivatives [15–21].

The dihydroquinoxaline moiety excepting N1 is planar within 0.0139(9) Å (r.m.s. deviation of the fitted atoms = 0.0072) with N1 0.0458(11) Å out of the above plane. The C17…C22 benzene ring is inclined by only 12.90(4)° from the aforementioned plane, due in part by a weak, intramolecular C22–H22…O1 hydrogen bond (see the Figure). In the crystal, the molecules form oblique stacks extending in the *b*-axis direction and parallel to the *ab* plane. The stacks are associated through "intercalation" of the "fully extended" *n*-octyl groups to form a typical micellar array.

In the crystal, the molecules are linked through C–H…π(ring) interactions and intercalation of the *n*-octyl groups.

Acknowledgments: The corresponding author thanks the National Research Foundation for financial support.
Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: The support of NSF-MRI Grant #1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. *APEX3 – Diffractometer Control and Data Reduction Software Package*; Bruker AXS Inc.: Madison, WI, USA, 2016.
2. Krause L., Herbst-Irmer R., Sheldrick G. M., Stalke D. Comparison of silver and molybdenum microfocus X-ray sources for single-crystal structure determination. *J. Appl. Crystallogr.* 2015, *48*, 3–10.
3. Sheldrick G. M. SHELXT – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
4. Sheldrick G. M. Crystal structure refinement with *SHELXL*. *Acta Crystallogr.* 2015, *C71*, 3–8.
5. Brandenburg K. *DIAMOND. Visual Crystal Structure Information System, Ver. 4.0*; Crystal Impact: Bonn, Germany, 2015.
6. Ramli Y., Essassi E. M. Advances in synthetic approaches, functionalization and biological properties of quinoxaline derivatives. In *Advances in Chemistry Research*; Taylor J. C., Ed; Nova Science Publishers: New York, *Vol. 27*, 2015, pp. 109–160.

7. Ramli Y., Moussaif A., Karrouchi K., Essassi E. M. Pharmacological profile of quinoxalinone. *J. Chem.* 2014, *2014*, Article 563406; https://doi.org/10.1155/2014/563406.
8. Zarrouk A., Zarrok H., Ramli Y., Bouachrine M., Hammouti B., Sahibed-dinee A., Bentisse F. Inhibitive properties, adsorption and theoretical study of 3,7-dimethyl-1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one as efficient corrosion inhibitor for carbon steel in hydrochloric acid solution. *J. Mol. Liq.* 2016, *222*, 239–252.
9. Laabaissi T., Benhiba F., Missioui M., Rouifi Z., Rbaa M., Ouddaa H., Ramli Y., Guenbour A., Warad I., Zarrouk A. Coupling of chemical, electrochemical and theoretical approach to study the corrosion inhibition of mild steel by new quinoxaline compounds in 1 M HCl. *Heliyon* 2020, *6*, e03939.
10. El Ouali I., Hammouti B., Aouniti A., Ramli Y., Azougagh M., Essassi E. M., Bouachrine M. Thermodynamic characterisation of steel corrosion in HCl in the presence of 2-phenylthieno (3,2-*b*) quinoxaline. *J. Mater. Environ. Sci.* 2010, *1*, 1–8.
11. Abad N., Hajji M., Ramli Y., Belkhira M., Elmgirhi S. M. H., Habib M. A., Guerfel T., Mague J. T., Essassi E. M. A newly synthesized nitrogen-rich derivative of bicyclic quinoxaline—structural and conceptual DFT reactivity study. *J. Phys. Org. Chem.* 2020, *33*, e4055.
12. Abad N., Lgaz H., Atioglu Z., Akkurt M., Mague J. T., Ali I. H., Ill-Chung M., Salghih R., Essassi E. M., Ramli Y. Synthesis, crystal structure, Hirshfeld surface analysis, DFT computations and molecular dynamics study of 2-(benzyloxy)-3-phenylquinoxaline. *J. Mol. Struct.* 2020, *1221*, 128727.
13. Missioui M., Essassi E. M., Mague J. T., Ramli Y. Synthesis and crystal structure of (*E*)-1-benzyl-3-(4-methoxystyryl)quinoxalin-2(1*H*)-one, $C_{24}H_{20}N_2O_2$. *Z. Kristallogr. NCS* 2020, *235*; https://doi.org/10.1515/ncrs-2020–0300.
14. Ramli Y., Essassi E. M. Condensation study of hydrazonoyl chloride with 3-methylquinoxalin-2-one and 3-styrylquinoxalin-2-one. *J. Mar. Chim. Heterocycl.* 2019, *18*, 39–47.
15. Missioui M., El Fal M., Taoufik J., Essassi E. M., Mague J. T., Ramli Y. 2-(3–Methyl-2-oxo-1,2-dihydroquinoxalin-1-yl)acetic acid dihydrate. *IUCrData* 2018, *3*, x180882.
16. Ramli Y., El Bakri Y., El Ghayati L., Essassi E. M., Mague J. T. 1–Benzyl-3-methylquinoxalin-2(1*H*)-one. *IUCrData* 2018, *3*, x180390.
17. Missioui M., Mague J. T., El Fal M., Taoufik J., Essassi E. M., Ramli Y. Ethyl 2-[(3-methylquinoxalin-2-yl)sulfanyl]acetate. *IUCrData* 2017, *2*, x171763.
18. Ramli Y., Karrouchi K., Essassi E. M., El Ammari L. *N*′-Phenyl-*N*′-[3-(2,4,5-triphenyl-2,5-dihydro-1*H*-pyrazol-3-yl)quinoxalin-2-yl] benzohydrazide. *Acta Crystallogr.* 2013, *E69*, o1320–o1321.
19. Ramli Y., Slimani R., Zouihri H., Lazar S., Essassi E. M. 3-Methyl-1-(prop-2-en-1-yl)quinoxalin-2(1*H*)-one. *Acta Crystallogr.* 2010, *E66*, o1767.
20. Benzeid H., Essassi E. M., Saffon N., Garrigues B., Ng S. W. 1-Methyl-3-phenylquinoxalin-2(1*H*)-one. *Acta Crystallogr.* 2009, *E65*, o2323.
21. Benzeid H., Saffon N., Garrigues B., Essassi E. M., Ng S. W. 1-Benzyl-3-phenylquinoxalin-2(1*H*)-one. *Acta Crystallogr.* 2009, *E65*, o2685.