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Denise Mondieig, Philippe Négrier, Jean-Michel Léger, Stéphane Massip, B. Benali, et al.. Crystal Structure of 2-S-(1'-Deoxy-2',3':4',5'-di-O-isopropylidene-D,L-xylit-1'-ylthio)-3-methylquinoxaline. X-Ray Structure Analysis Online, 2010, 26 (1), pp.1-2. 10.2116/xraystruct.26.1 . hal-00495000

HAL Id: hal-00495000

<https://hal.science/hal-00495000>

Submitted on 9 Jan 2018

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Crystal Structure of 2-*S*-(1'-Deoxy-2',3':4',5'-di-*O*-isopropylidene-D,L-xylitol-1'-ylthio)-3-methylquinoxaline

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The single-crystal structure of 2-*S*-(1'-deoxy-2',3':4',5'-di-*O*-isopropylidene-D,L-xylitol-1'-ylthio)-3-methylquinoxaline (C₂₀H₂₆N₂O₄S) was determined at 293 K and refined to *R*1 = 0.0510 and *wR*2 = 0.1514 for 5083 observed reflections. This crystal is orthorhombic, *Pna*2₁ with *Z* = 8. Two molecules constitute the asymmetric unit. The unit-cell dimensions are: *a* = 14.590(3)Å, *b* = 9.672(2)Å, *c* = 28.074(5)Å and *V* = 3964(2)Å³.

(Received September 19, 2008; Accepted December 8, 2008; Published on web January 10, 2010)

Quinoxaline derivatives are biologically interesting compounds, and have been used in various pharmaceutical applications. For example, in colon cancer therapies¹ they interact with α -aminobutyric acid A (GABAA) and benzodiazepine receptor;² they inhibit aldose reductases,³ and they are potent angiotensin II receptor antagonists.⁴ The quinoxalines biodisponibility can be improved by their grafting on the monosaccharide moiety.⁵

2-*S*-(1'-Deoxy-2',3':4',5'-di-*O*-isopropylidene-D,L-xylitol-1'-ylthio)-3-methylquinoxaline was synthesized by the condensation of 2,3:4,5-di-*O*-isopropylidene-6-*O*-tosyl-D,L-xylitol (1.35 g, 3.50 mmol) with 3-methyl-1*H*-quinoxaline-2-thione (0.60 g, 3.50 mmol), K₂CO₃ (0.48 g, 4.20 mmol) and Bu₄NBr (0.2 g, 0.6 mmol) in DMF (50 mL).

X-ray data were collected with an Axis Rapid diffractometer (RIGAKU, Tokyo) equipped with Mm007 microfocus rotating anode generator. A data collection unit-cell refinement was achieved by using a Crystalclear software package. Diffraction data were collected at 293 K using monochromatic Cu-*K* α

radiation (λ = 1.54178 Å). The crystal and experimental data are given in Table 1. The asymmetric unit contains two molecules (see ORTEP plot in Fig. 2).

The structure was solved by direct methods and refined by full-matrix least-squares to a final reliability value of 0.0510.

Table 1 Crystal data and structure refinement

Empirical formula: C ₂₀ H ₂₆ N ₂ O ₄ S
Formula weight = 390.49
Crystal system: orthorhombic
Space group: <i>Pna</i> 2 ₁ , <i>Z</i> = 8
Cell dimensions: <i>a</i> = 14.590(3)Å, <i>b</i> = 9.672(2)Å, <i>c</i> = 28.074(5)Å
<i>V</i> = 3963.8(13)Å ³
<i>T</i> = 293(2)K
<i>D</i> _{calc} = 1.309 g/cm ³
No. reflections used = 5904
μ (Cu <i>K</i> α) = 1.685 mm ⁻¹ , λ = 1.54178 Å
2 θ _{max} = 71.88°
Crystal size = 0.20 × 0.15 × 0.15 mm
<i>F</i> (0 0 0) = 1664
<i>R</i> _{all} = 0.0565; <i>R</i> ₁ = 0.0510
Max. and min. transmission 0.7862 and 0.7293
Refinement method = Full-matrix least-squares on <i>F</i> ²
No. of parameters = 497
Goodness-of-fit on <i>F</i> ² = 1.023
(Δ / σ) _{max} = 0.001
($\Delta\rho$) _{max} = 0.4914 e.Å ⁻³ ($\Delta\rho$) _{min} = -0.264 e.Å ⁻³
Measurement: Rigaku RAXIS RAPID (Mm007 microfocus rotating anode)
Program system: Crystalclear crystal software package.
Structure determination: SHELXL-97
CCDC deposition number: 693144

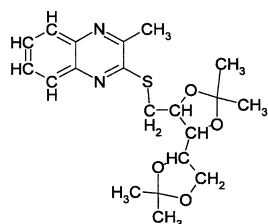


Fig. 1 Chemical diagram of 2-*S*-(1'-deoxy-2',3':4',5'-di-*O*-isopropylidene-D,L-xylitol-1'-ylthio)-3-methylquinoxaline.

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Table 2 Selected Bond lengths [Å], bond angles [°] and torsion angles [°]

Bond Length			
C(2)-C(3)	1.390(7)	C(52)-C(53)	1.417(7)
N(7)-C(8)	1.291(5)	N(57)-C(58)	1.309(6)
C(21)-O(25)	1.432(5)	C(71)-O(75)	1.403(5)
C(22)-O(23)	1.402(6)	C(72)-O(73)	1.426(6)
C(24)-O(25)	1.416(6)	C(74)-O(75)	1.433(6)
Bond angles			
C(2)-C(1)-C(6)	119.5(4)	C(52)-C(51)-C(56)	121.3(4)
C(1)-C(2)-C(3)	121.2(5)	C(51)-C(52)-C(53)	119.7(5)
C(3)-C(4)-C(5)	118.9(4)	C(53)-C(54)-C(55)	120.5(4)
C(9)-C(8)-S(12)	116.9(3)	C(59)-C(58)-S(62)	118.1(4)
N(10)-C(9)-C(8)	119.4(4)	N(60)-C(59)-C(58)	120.9(4)
O(15)-C(16)-C(20)	111.8(4)	O(65)-C(66)-C(69)	107.4(4)
O(15)-C(16)-C(19)	107.7(4)	O(65)-C(66)-C(70)	110.8(4)
O(17)-C(16)-C(19)	109.2(4)	O(67)-C(66)-C(70)	110.4(4)
C(21)-C(18)-C(14)	114.3(3)	C(71)-C(68)-C(64)	115.5(3)
O(25)-C(21)-C(18)	110.8(3)	O(75)-C(71)-C(68)	112.1(4)
O(23)-C(22)-C(21)	105.9(4)	O(73)-C(72)-C(71)	104.6(4)
C(22)-O(23)-C(24)	109.5(4)	C(74)-O(73)-C(72)	107.0(4)
Torsion angles			
O(25)-C(21)-C(22)-O(23)	-9.1(5)	O(75)-C(71)-C(72)-O(73)	-4.3(5)
C(18)-C(21)-C(22)-O(23)	-129.6(4)	C(68)-C(71)-C(72)-O(73)	117.9(4)
C(21)-C(22)-O(23)-C(24)	-8.3(5)	C(71)-C(72)-O(73)-C(74)	23.5(5)
C(22)-O(23)-C(24)-O(25)	22.9(5)	C(72)-O(73)-C(74)-O(75)	-33.7(5)
C(22)-O(23)-C(24)-C(27)	138.2(5)	C(72)-O(73)-C(74)-C(77)	-149.7(4)
C(22)-O(23)-C(24)-C(26)	-97.7(5)	C(72)-O(73)-C(74)-C(76)	84.9(5)

Table 3 Hydrogen bonding geometry

D-H...A	D-H[Å]	H...A[Å]	D...A[Å]	D-H...A[°]
C13 H13 N7	0.970	2.462	2.813(6)	100.99
C14 H14 O25	0.981	2.473	2.911(5)	106.74
C22 H22A O17	0.971	2.508	2.919(7)	105.36
C63 H63 O57	0.970	2.456	2.799(6)	100.44
C64 H64 O75	0.979	2.560	2.963(6)	104.68

least-square plane is lower than 0.2 Å). The dihedral angle between the quinoxaline rings of the two molecules forming an asymmetric unit is 0.3°. Selected bond lengths, angles and torsion angles corresponding to the highest differences between the two molecules of the asymmetric unit are listed in Table 2. The highest dissimilarity is situated on one of the dioxolane rings.

Pseudo-chelate type bondings appear to involve N7...H13-C13, O25...H14A-C14, N57...H63-C63 and O75...H64A-C64 (see Table 3). The relative configurations of C14, C18, C21, C64, C68 and C71 are *S*, *R*, *S*, *R*, *S* and *R*, respectively. The absolute structure was not determined by an X-ray measurement.

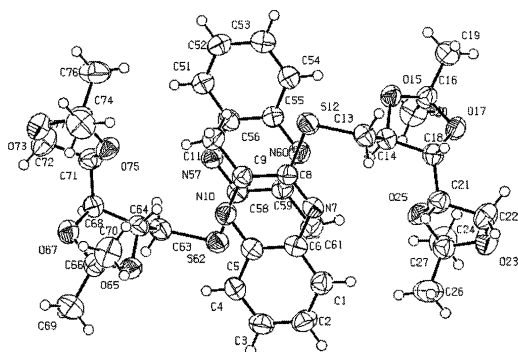


Fig. 2 ORTEP structure 2-*S*-(1'-deoxy-2',3':4',5'-di-*O*-isopropylidene-D,L-xylitol-1'-ylthio)-3-methylquinoxaline, showing 50% probability ellipsoids.

The non-hydrogen atoms were refined anisotropically. The H atoms were allowed to ride on the parent atoms in the model. The quinoxaline rings are quasi-planar (the deviation from the

Acknowledgments

This work has been supported by the French-Morocco cooperation CNRS/CNRST N°18606

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