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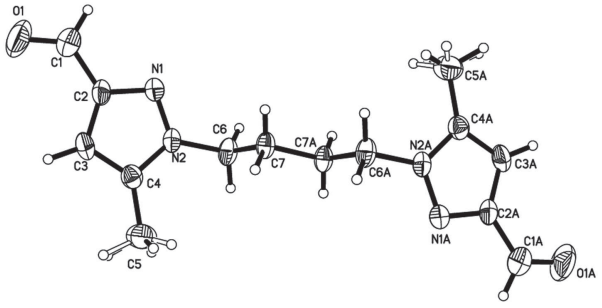
Crystal structure of 1,1′-(butane-1,4-diyl)bis(5-methyl-1*H*-pyrazole-3-carbaldehyde), $C_{14}H_{18}N_4O_2$



DOI 10.1515/ncrs-2015-0196
Received February 9, 2016; accepted February 29, 2016; available online March 25, 2016

Abstract

$C_{14}H_{18}N_4O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 4.4260(9)$ Å, $b = 14.502(4)$ Å, $c = 11.455(3)$ Å, $\beta = 96.319(5)°$, $V = 730.8(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0712$, $wR_{ref}(F^2) = 0.1946$, $T = 296(2)$ K.

CCDC no.: 1418841

The crystal structure of the title compound is shown in the Figure. Details of the measurement method and a list of the atoms including displacement parameters and atomic coordinates are given in Tables 1–3.

Table 1: Data collection and handling.

Crystal:	Brown, needle, size 0.16×0.18×0.74 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.86 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II D8, φ and ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1283, 128
$N(param)_{refined}$:	115
Programs:	Bruker programs [11], SHELX [12]

Table 2: Fractional atomic cordinates and equivalent isotopic displacement coefficients (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*e*	0.9867	0.1117	0.3993	0.079
H(3A)	4*e*	0.7858	0.2474	0.1158	0.055
H(5A)	4*e*	0.2259	0.4332	0.1686	0.099
H(5B)	4*e*	0.3743	0.3916	0.0587	0.099
H(5C)	4*e*	0.5577	0.4655	0.1427	0.099
H(5CX)	4*e*	0.19(2)	0.416(7)	0.13(1)	0.07(3)
H(5BX)	4*e*	0.49(2)	0.473(5)	0.175(6)	0.02(2)
H(5AX)	4*e*	0.53(2)	0.409(5)	0.058(6)	0.03(3)
H(6A)	4*e*	0.2737	0.3600	0.4640	0.052
H(6B)	4*e*	0.2063	0.4273	0.3535	0.052
H(7B)	4*e*	0.682(7)	0.501(2)	0.408(3)	0.044(8)
H(7A)	4*e*	0.742(9)	0.440(2)	0.513(3)	0.06(1)

Source of material

The synthesis of 1,1′-(butane-1,4-diyl)bis(5-methyl-1*H*-Pyrazole-3 carbaldehyde) starting from (butane-1,4-diylbis (5-methyl-1*H*-pyrazole-1,3-diyl))dimethanol was prepared according reported procedure [1]. (Butane-1,4-diylbis(5-methyl-1*H*-pyrazole-1,3-diyl))dimethanol (9.00 mmol) and activated manganese dioxide (54 mmol) were mixed in 1,4-dioxane (100 mL) and the suspension was stirred under reflux for 48 h. The reaction was monitored by TLC (MeOH / DCM = 1/9). The obtained suspension was filtered and washed with boiling 1,4-dioxane and MeOH.

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Table 3: Fractional coordinates and atomic displacement coefficients (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*e*	1.0766(8)	0.0871(2)	0.2498(3)	0.100(2)	0.068(2)	0.108(2)	0.029(2)	0.019(2)	−0.031(2)
N(1)	4*e*	0.6708(6)	0.2584(2)	0.3829(2)	0.054(2)	0.040(2)	0.036(1)	0.005(1)	0.004(1)	−0.007(1)
N(2)	4*e*	0.5250(5)	0.3313(2)	0.3321(2)	0.038(1)	0.038(2)	0.038(1)	0.000(1)	0.007(1)	−0.011(1)
C(1)	4*e*	0.963(1)	0.1326(3)	0.3202(4)	0.080(3)	0.053(2)	0.065(2)	0.013(2)	0.008(2)	−0.010(2)
C(2)	4*e*	0.7924(7)	0.2169(2)	0.2954(3)	0.047(2)	0.039(2)	0.040(2)	0.001(2)	0.006(1)	−0.013(1)
C(3)	4*e*	0.7248(8)	0.2637(2)	0.1901(3)	0.057(2)	0.049(2)	0.034(2)	−0.005(2)	0.013(1)	−0.012(1)
C(4)	4*e*	0.5530(7)	0.3376(2)	0.2149(2)	0.044(2)	0.039(2)	0.038(2)	−0.006(2)	0.005(1)	−0.004(1)
C(5)	4*e*	0.416(1)	0.4134(3)	0.1399(4)	0.078(3)	0.061(3)	0.057(2)	0.002(2)	−0.004(2)	0.010(2)
C(6)	4*e*	0.3697(7)	0.3953(2)	0.4040(3)	0.035(2)	0.045(2)	0.053(2)	−0.002(1)	0.013(1)	−0.016(2)
C(7)	4*e*	0.5820(7)	0.4659(2)	0.4642(3)	0.036(2)	0.039(2)	0.043(2)	−0.003(1)	0.015(1)	−0.011(1)

After removing the solvent, the resultant white solid was recrystallized from MeOH to give the target product. Yield: 47%.

Experimental details

Data reduction and cell refinement were carried out by Bruker SAINT and APEX2 [11]. The hydrogen atoms of the methyl group are disordered over two positions. The hydrogen atoms were placed on calculated positions with the SHELX program (AFIX 23 and 43 option) [12].

Discussion

5- or 6-membered heterocyclic rings are known for their diverse pharmaceutical potentials. For example, pyrazoles have been extensively explored for development of pharmaceutically important molecules. Heterocyclic chelating ligands are of particular interest, such as *bis*(pyrazol-1-yl)methanes and 1,2-*bis*(pyrazol-1-yl)ethanes, which have received considerable attention in relation to their coordination chemistry [2–10]. The title molecule has a center of symmetry at the midpoint of the central C—C bond. The two aldehyde groups lie on opposite sides of that plane and their pseudo-torsion angle is 180°, as required by symmetry. The pyrazole rings are planar. The molecules are linked *via* a non-classical hydrogen bond C7—H7B$\cdots$O1.

Acknowledgements: The authors extend their sincere appreciation to the Deanship of Scientific Research at king Saud University for its funding this Prolific Research group (PRG-1437-29).

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