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The crystal structure of 1,1′-(2,5-dimethylpyrazine-1,4-diyl)bis(ethan-1-one), $C_{10}H_{14}N_2O_2$

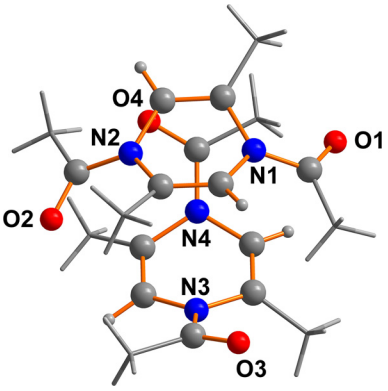

Table 1: Data collection and handling.

Crystal:	Colourless, block
Size:	0.23 × 0.20 × 0.18 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
θ_{max}, completeness:	25.0°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21244, 3707, 0.049
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2577
$N(param)_{refined}$:	261
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

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Abstract

$C_{10}H_{14}N_2O_2$, monoclinic, $C2/c$ (no. 15), a = 15.7853(7) Å, b = 8.5036(4) Å, c = 31.4964(17) Å, β = 90.371(2)°, V = 4,227.7(4) Å3, Z = 8, $R_{gt}(F)$ = 0.0451, $wR_{ref}(F_2)$ = 0.1611, T = 655 K.

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The two independent molecules constructing the asymmetric unit are shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

To a 100 ml round-bottom flask, add 2,5-dimethylpyrazine (500 mg; 4.627 mmol), which was fully dissolved with ethylene glycol dimethyl ether (20 mL), followed by the addition of acetic anhydride (945 mg; 9.254 mmol) and zinc powder (908 mg; 13.881 mmol). In an nitrogen atmosphere, the reflux reaction was heated at 90 °C for about 1 h. After the reaction was completed and cooled to room temperature, 150 mL of water was added to the reaction system, the aqueous layer was extracted with ethyl acetate (50 mL × 3), the anhydrous sodium sulfate dried the organic layer for about 2 h, and the organic layer was concentrated by filtration and decompression. Methanol was used to recrystallize the desired compound (white solid, 629 mg, 70 %). Single crystals were obtained by crystallization of the title compound from a mixture of dichloromethane (1 mL) and n-hexane (2 mL).

2 Experimental details

Using Olex2,[2] the structure was solved with the SHELXT[3] structure solution program and refined with the SHELXL[4] refinement package. All hydrogen atoms were placed in the calculated positions.

3 Comment

Some 1,4-dihydropyrazines are important building blocks that make up natural products[5] and have important biological activities that can be used in biological reagents.[6–8] Based on the original compound, 2,5-dimethylpyrazine, we synthesized the structure of the new compound described in this paper with moderate yields. The introduction of the N-position acetyl group has caused some significant changes in the properties of 2,5-dimethylpyrazine itself. The reaction route adopted in this

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.24384 (9)	0.87090 (18)	0.58105 (5)	0.0514 (4)
H1	0.236148	0.897896	0.552675	0.062*
C2	0.32097 (9)	0.86955 (18)	0.59735 (5)	0.0494 (4)
C3	0.25733 (9)	0.86641 (18)	0.66645 (5)	0.0509 (4)
H3	0.265251	0.889716	0.695045	0.061*
C4	0.18021 (9)	0.87047 (18)	0.65016 (5)	0.0491 (4)
C5	0.39661 (10)	0.9283 (2)	0.57345 (6)	0.0681 (5)
H5A	0.432171	0.841141	0.566015	0.102*
H5B	0.427961	1.000446	0.590962	0.102*
H5C	0.378050	0.981039	0.548089	0.102*
C6	0.10545 (10)	0.9325 (2)	0.67416 (6)	0.0673 (5)
H6A	0.067027	0.847866	0.680039	0.101*
H6B	0.077032	1.010932	0.657401	0.101*
H6C	0.124485	0.978382	0.700387	0.101*
C7	0.10357 (9)	0.75191 (19)	0.58967 (6)	0.0518 (4)
C8	0.10541 (11)	0.7178 (2)	0.54328 (6)	0.0653 (5)
H8A	0.055085	0.661496	0.535275	0.098*
H8B	0.154306	0.655206	0.536919	0.098*
H8C	0.108017	0.814865	0.527763	0.098*
C9	0.39752 (9)	0.74860 (19)	0.65749 (6)	0.0535 (4)
C10	0.39676 (11)	0.7128 (2)	0.70373 (6)	0.0675 (5)
H10A	0.447151	0.655771	0.711261	0.101*
H10B	0.347892	0.650244	0.710215	0.101*
H10C	0.394742	0.809208	0.719538	0.101*
C11	0.16379 (10)	0.33453 (19)	0.62817 (5)	0.0520 (4)
H11	0.106410	0.313974	0.631726	0.062*
C12	0.19591 (10)	0.33159 (18)	0.58965 (5)	0.0508 (4)
C13	0.33397 (10)	0.32153 (19)	0.62215 (5)	0.0514 (4)
H13	0.390196	0.292106	0.618499	0.062*
C14	0.30175 (10)	0.32220 (18)	0.66071 (5)	0.0488 (4)
C15	0.14668 (11)	0.2737 (2)	0.55192 (6)	0.0699 (5)
H15A	0.092728	0.234519	0.561036	0.105*
H15B	0.177581	0.190783	0.538300	0.105*
H15C	0.138196	0.358605	0.532273	0.105*
C16	0.34922 (12)	0.2595 (2)	0.69844 (6)	0.0674 (5)
H16A	0.400419	0.209591	0.689164	0.101*
H16B	0.314636	0.184168	0.713009	0.101*
H16C	0.362998	0.344448	0.717326	0.101*
C17	0.18317 (10)	0.44476 (19)	0.69924 (5)	0.0523 (4)
C18	0.09135 (10)	0.4841 (2)	0.69857 (6)	0.0663 (5)
H18A	0.076952	0.538949	0.724170	0.099*
H18B	0.058750	0.389053	0.696609	0.099*
H18C	0.079087	0.549726	0.674500	0.099*
C19	0.31871 (10)	0.44046 (19)	0.55163 (5)	0.0535 (4)
C20	0.41292 (10)	0.4592 (2)	0.55144 (6)	0.0719 (5)
H20A	0.429758	0.511359	0.525816	0.108*
H20B	0.439196	0.357455	0.552836	0.108*
H20C	0.430284	0.520645	0.575527	0.108*
N1	0.17212 (7)	0.83179 (15)	0.60611 (4)	0.0493 (4)
N2	0.32870 (7)	0.82713 (16)	0.64120 (4)	0.0489 (4)
N3	0.28460 (7)	0.36483 (16)	0.58603 (4)	0.0509 (4)
N4	0.21472 (7)	0.36835 (15)	0.66441 (4)	0.0497 (4)
O1	0.04496 (7)	0.71149 (15)	0.61227 (4)	0.0687 (4)
O2	0.45659 (7)	0.70951 (16)	0.63468 (4)	0.0734 (4)
O3	0.27425 (8)	0.49029 (16)	0.52281 (4)	0.0703 (4)
O4	0.22886 (8)	0.48006 (16)	0.72939 (4)	0.0698 (4)

paper has good substrate applicability and functional group compatibility, It provides a convenient and efficient reaction route for the introduction of different substituents at the N position of 2,5-dimethylpyrazine.

Both molecules in the title compound adopt a non-planar conformation, with two acetyl groups located in different planes, separated by a dihedral angle of 56.2°. In the crystal structure, the bond lengths of C–O and C–N are in the range of 1.2199(19) Å–1.227(2) Å and 1.371(2) Å–1.4341(19) Å, respectively. Geometric parameters are all in the expected regions.[9,10]

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Conflict of interest: The authors declare no conflicts of interest regarding this article.

References

1. SAINT. *Program for Data Extraction and Reduction*; Bruker AXS, Inc: Madison.
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX2: a Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Cryst.* **2009**, *42*, 339–341.
3. Sheldrick, G. M. *SHELXTL* – 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. Li, F.; Mao, S.; Sun, Y.; Lu, C.; An, Y. Dipotassium Phosphate Catalyzes the Synthesis of 1,4-dihydropyrano [2,3-c]pyrapyran by Multi-Component One-Pot Reaction Azole Derivatives. *Appl. Chem.* **2018**, *35*, 1201–1207.
6. Fourrey, J. L.; Beauhaire, J.; Yuan, C. W. Preparation of Stable 1,4-Dihydropyrazines. *J. Chem. Soc., Perkin Trans. 1* **1987**, *89*, 1841–1843.
7. Chaignaud, M.; Gillaizeau, I.; Ouhamou, N.; Coudert, G. New Highlights in the Synthesis and Reactivity of 1,4-Dihydropyrazine Derivatives. *Tetrahedron* **2008**, *35*, 8059–8806.
8. Tandon, V. K.; Yadav, D. B.; Maurya, H. K.; Chaturvedi, A. K.; Shukla, P. K. Design, Synthesis, and Biological Evaluation of 1,2,3-Trisubstituted-1,4-Dihydrobenzo[g]Quinoxaline-5,10-Diones and Related Compounds as Antifungal and Antibacterial Agents. *Bioorg. Med. Chem.* **2006**, *17*, 6.
9. Wang, L. H.; Song, G. Q.; Tai, X. S. The Crystal Structure of 6-Amino-Pyridini-2-Carboxylate, $C_6H_6N_2O_2$. *Z. Kristallogr. N. Cryst. Struct.* **2024**, *239*, 979–980.
10. Fan, Q.; Duan, X.; Yan, H. Solid State [2 + 2] Photocycloaddition for Constructing Dimers of *N*,*N*′-diacyl-1,4-dihydropyrazines Based on Thiourea-induced Assembly. *CrystEngComm* **2018**, *20*, 1151.