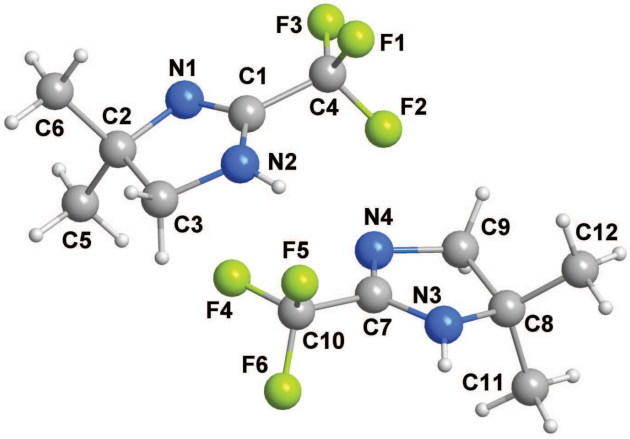
A. Musa Said*, Ismail Warad*, O. Seraj Alzahrani, Abdelkader Zarrouk and Dieter Lentz*

Crystal structure of 4,4-dimethyl-2-(trifluoromethyl)-4,5-dihydro-1*H*-imidazole, $C_6H_9F_3N_2$



https://doi.org/10.1515/ncrs-2018-0582
Received December 14, 2018; accepted January 15, 2019; available online February 4, 2019

Abstract

$C_6H_9F_3N_2$, monoclinic, $P2_1/n$ (no. 14), $a = 10.6224(9)$ Å, $b = 11.8639(9)$ Å, $c = 13.3139(11)$ Å, $\beta = 105.903(3)°$, $V = 1613.6(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0618$, $wR_{ref}(F^2) = 0.1629$, $T = 102(2)$ K [1–3].

CCDC no.: 1884790

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

***Corresponding authors: A. Musa Said,** Department of Chemistry, Taibah University, 30002 Al-Madina, Al-Mounawara, Saudi Arabia, e-mail: musasaid04@gmail.com; **Ismail Warad,** Department of Chemistry, AN-Najah National University, P.O. Box 7, Nablus, Palestine, e-mail: warad@najah.edu; and **Dieter Lentz,** Institut für Chemie und Biochemie, Anorganische Chemie, Freie Universität Berlin, Fabeckstr. 34-36, 14195 Berlin, Germany, e-mail: dlentz@zedat.fu-berlin.de
O. Seraj Alzahrani: Department of Chemistry, Taibah University, PO Box 30002, Code 14177, Al-Madina Al-Mounawara, Kingdom of Saudi Arabia
Abdelkader Zarrouk: Laboratory of Materials, Nanotechnology and Environment, Faculty of Sciences, Mohammed V University, Av. IbnBattouta, Box 1014, Agdal-Rabat, Morroco

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.40 × 0.20 × 0.18 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	Bruker Venture Photon, φ and ω
θ_{max}, completeness:	27.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25043, 3722, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3126
$N(param)_{refined}$:	211
Programs:	Bruker [1], SHELX [2, 3]

Source of material

Stoichiometric amount of 2-methylpropane-1,2-diamine (1.68 g, 19.06 mmol) was dissolved in 50 mL of dioxone in one neck round bottom flask equipped with a reflux condenser and magnetic stirring bar. Ethyl 2,2,2-trifluoroacetate (2.71 mL, 3.97 g, 38.12 mmol) was added dropwise and the mixture was stirred at moderate heating (50 °C) overnight. The solvent was completely removed and viscous colorless product was obtained. The product was further purified by distillation under reduced pressure (0.1 mm Hg, RT), (Yield 79%). M.P. 55−57 °C.

Experimental details

Hydrogen atoms were placed at the calculated positions and included using a riding model with $U_{iso}(H) = 1.2U_{eq}$ or $1.5U_{eq}$ of the adjacent non-hydrogen atom.

Comment

The development of imidazole-based molecules has speedily increased recently, since it can be used extensively in medicinal chemistry as man-made drugs, agrochemicals, artificial acceptors, biomimetic catalysts, supramolecular material [4–6]. Several imidazole medicinal drugs have been used for treatment of broad types of diseases, imidazole derivatives demonstrated anti-inflammatory, analgesic, antiviral, antitubercular, antimicrobial, antimalarial, anti-diabetic and anticancer activity [7–10].

The molecule $C_6H_9F_3N_2$ crystallizes in the space group $P2_1/n$. The asymmetric unit of the title structure contains

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6367(2)	0.27964(17)	0.58791(15)	0.0254(4)
C2	0.73966(18)	0.44033(16)	0.63748(15)	0.0220(4)
C3	0.6742(2)	0.40100(18)	0.72269(16)	0.0281(4)
H3A	0.739069	0.395447	0.791899	0.034*
H3B	0.603163	0.452885	0.727600	0.034*
C4	0.5809(3)	0.1758(2)	0.52623(18)	0.0413(6)
C5	0.6921(2)	0.55617(19)	0.59481(18)	0.0333(5)
H5A	0.729385	0.574908	0.537231	0.050*
H5B	0.719890	0.612439	0.650379	0.050*
H5C	0.596356	0.555871	0.569313	0.050*
C6	0.8882(2)	0.4379(2)	0.67844(19)	0.0346(5)
H6A	0.916767	0.361909	0.703466	0.052*
H6B	0.916464	0.491723	0.736064	0.052*
H6C	0.927006	0.458485	0.622156	0.052*
C7	0.40653(17)	0.18527(15)	0.85401(14)	0.0181(4)
C8	0.22696(18)	0.08125(16)	0.77486(14)	0.0214(4)
C9	0.31326(19)	0.11170(18)	0.70118(15)	0.0241(4)
H9A	0.349257	0.042471	0.678059	0.029*
H9B	0.260652	0.152188	0.638705	0.029*
C10	0.49772(19)	0.25035(17)	0.94148(14)	0.0232(4)
C11	0.0969(2)	0.1437(2)	0.74606(18)	0.0326(5)
H11A	0.052868	0.132647	0.801053	0.049*
H11B	0.041604	0.114349	0.679815	0.049*
C12	0.2065(2)	−0.04492(18)	0.78123(18)	0.0350(5)
H12A	0.291734	−0.082610	0.804080	0.053*
H12B	0.158469	−0.073403	0.712299	0.053*
H12C	0.156267	−0.060202	0.831429	0.053*
F1	0.6175(4)	0.08556(15)	0.57764(17)	0.1328(15)
F2	0.4514(2)	0.1738(2)	0.5063(2)	0.1104(11)
F3	0.6078(2)	0.16886(14)	0.43734(13)	0.0660(6)
F4	0.58725(15)	0.30726(13)	0.90982(10)	0.0436(4)
F5	0.56104(13)	0.18335(11)	1.01875(10)	0.0353(3)
F6	0.43133(14)	0.32464(12)	0.98252(11)	0.0413(4)
N1	0.69927(17)	0.35430(14)	0.55252(13)	0.0246(4)
H1N	0.567(3)	0.259(3)	0.706(2)	0.051(9)*
N2	0.6232(2)	0.2912(17)	0.68486(15)	0.0371(5)
H2N	0.283(3)	0.137(2)	0.928(2)	0.036(7)*
N3	0.31069(16)	0.12340(15)	0.87538(13)	0.0252(4)
N4	0.42054(16)	0.18443(13)	0.76162(12)	0.0207(3)

two independent molecules (figure), with a high similarity in their structural parameters like bond lengths and angles. The similarity of these two structures extends to their hydrogen-bonding motifs. The molecules in the title structure are connected *via* intermolecular hydrogen bonds, of N—H···N type (N2···N4: 2.91 Å; N1···N3′: 2.93 Å; $' = 0.5 + x, 0.5 - y, -0.5 + z$).

Acknowledgements: M. A. Said is thankful to Alexander von Humboldt Stiftung, Germany for the opportunities provided.

References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. Acta Crystallogr. **A71** (2015) 3–8.
3. Sheldrick, G. M.: SHELXL-2017/1. Acta Crystallogr. **C71** (2015) 3–8.
4. Forte, B.; Malgesini, B.; Piutti, C.; Quartieri, F.; Scolaro, A.; Papeo, G.: A submarine journey: the pyrrole-imidazole alkaloids. Drugs **7** (2009) 705–753.
5. Jin, Z.: Muscarine, imidazole, oxazole, and thiazole alkaloids. Nat. Prod. Rep. **28** (2011) 1143–1191.
6. Jiang, H.; Zhou, C.; Luo, K.; Chen, H.; Lan, J.; Xie, R.: Chiral imidazole metalloenzyme models: synthesis and enantioselective hydrolysis for α-amino acid esters. J. Mol. Catal. A: Chem. **260** (2006) 288–294.
7. Congiu, C.; Cocco, M.; Onnis, V.: Design, synthesis, and *in vitro* antitumor activity of new 1,4-diarylimidazole-2-ones and their 2-thione analogues. Bioorg. Med. Chem. Lett **18** (2008) 989–993.
8. Venkatesan, A. M.; Agarwal, A.; Abe, T.; Ushirogochi, H.; Ado, M.; Tsuyoshi, T.; Santos, O. D.; Li, Z.; Francisco, G.; Lin, Y. I.; Peterson, P. J.; Yang, Y.; Weiss, W. J.; Shales, D. M.; Manzour, T. S.: 5,5,6-Fused tricycles bearingimidazole and pyrazole 6-methylidene penems as broad-spectrum inhibitors of β-lactamases. Bioorg. Med. Chem. **16** (2008) 1890–1902.
9. Bizhayev, M.: Biological activities of the natural imidazole-containing peptidomimetics *n*-acetylcarnosine, carcinine and L-carnosine in ophthalmic and skin care products. Life Sci. **78** (2006) 2343–2357.
10. Roman, G.; Riley, J.; Vlahakis, J.; Kinobe, R.; Brien, J.; Nakatsu, K.; Szrek, W.: Heme oxygenase inhibition by 2-oxy-substituted 1-(1*H*-imidazol-1-yl)-4-phenylbutanes: effect of halogen substitution in the phenyl ring. Bioorg. Med. Chem. **15** (2007) 3225–3234.