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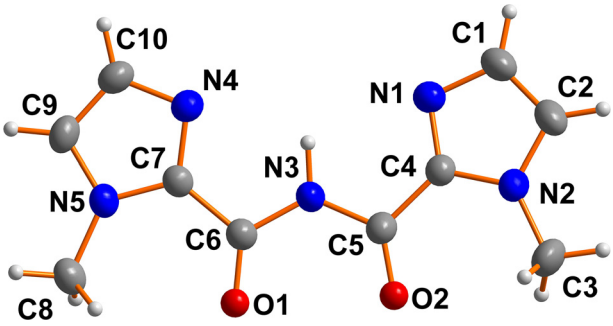
The crystal structure of 1-methyl-*N*-(1-methyl-1*H*-imidazole-2-carbonyl)-1*H*-imidazole-2-carboxamide, $C_{10}H_{11}N_5O_2$



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Abstract

$C_{10}H_{11}N_5O_2$, orthorhombic, $Pca2_1$ (no. 29), a = 22.6173(13) Å, b = 4.8730(3) Å, c = 9.9921(5) Å, V = 1101.27(11) Å^3, Z = 4, $R_{gt}(F)$ = 0.0440, $wR_{ref}(F^2)$ = 0.0999, T = 200(2) K.

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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:	Colorless prism
Size:	0.36 × 0.18 × 0.14 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Rigaku SCXmini, ω
θ_{max}, completeness:	27.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9422, 2514, 0.062
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1893
$N(param)_{refined}$:	160
Programs:	Rigaku [1], Olex2 [2], SHELX [3, 4], Diamond [5]

Source of materials

All chemicals were purchased from commercial sources and used as received without further purification. In a 25 mL reaction vessel, a solution of (1*H*-imidazol-2-yl) methanamine (10 mmol), copper acetate (2 mmol), 80 mL of toluene was added to a 150 mL round bottom flask. The resulted mixture was stirred for 24 h in an ice bath under the protection of nitrogen atmosphere. The solvent is removed by evaporation and then extracted by dichloromethane. Finally, the samples were purified by recrystallization. Yield: 3.80 g, 86%.

Experimental details

All H atoms bond to C atoms were introduced using the HFIX command in the SHELXL-2014 program [1], with the value of 0.93 Å or 0.96 Å for C–H bonds distances. All H atoms were allowed for as riding atoms with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C)$ for hydrogen atoms bonded to imidazole ring and methyl group, respectively.

Comment

Amide type compounds are important chemical intermediates in organic chemistry, which play an important role in medicinal chemistry, life science and other fields [6–8]. Owing to strong intermolecular hydrogen bonding, amide type compounds have irreplaceable functional properties [9]. Up to now, Amide structure has a high occupancy of constant high (more than 25%) in the known pharmacochemical analysis database. Imidazole is a class of important nitrogen-containing heterologous compound, which form hydrogen bonds with receptors and enzymes in organisms by the special thick ring structure of imidazole. The advantages of amide group structure combined with the properties of imidazole fully exploit its potential in

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.62241 (11)	0.2754 (5)	0.3186 (2)	0.0487 (7)
O2	0.71828 (11)	0.5702 (6)	0.4308 (3)	0.0598 (8)
N1	0.64554 (12)	1.0242 (6)	0.6563 (3)	0.0398 (7)
N3	0.61961 (13)	0.6080 (6)	0.4818 (3)	0.0374 (6)
N5	0.49537 (11)	0.1962 (5)	0.3633 (2)	0.0344 (6)
C1	0.67431 (17)	1.1990 (8)	0.7413 (3)	0.0473 (9)
H1	0.655297	1.327570	0.798346	0.057*
C4	0.68851 (14)	0.8859 (7)	0.5959 (3)	0.0348 (7)
C5	0.67876 (15)	0.6744 (7)	0.4949 (3)	0.0385 (8)
C6	0.59508 (14)	0.4048 (7)	0.4022 (3)	0.0352 (7)
C7	0.53172 (13)	0.3713 (6)	0.4284 (3)	0.0324 (7)
C8	0.50937 (15)	−0.0042 (8)	0.2583 (3)	0.0396 (8)
H8A	0.514130	0.091038	0.172602	0.059*
H8B	0.477156	−0.137847	0.251260	0.059*
H8C	0.546187	−0.099464	0.281020	0.059*
C3	0.80109 (15)	0.8593 (9)	0.6008 (4)	0.0579 (11)
H3A	0.831987	0.962287	0.647759	0.087*
H3B	0.806373	0.879858	0.503975	0.087*
H3C	0.803822	0.664707	0.624840	0.087*
N2	0.74294 (12)	0.9653 (6)	0.6394 (2)	0.0390 (7)
C2	0.73397 (17)	1.1635 (8)	0.7329 (3)	0.0458 (9)
H2	0.763403	1.258361	0.782576	0.055*
N4	0.50276 (12)	0.5213 (6)	0.5187 (3)	0.0383 (6)
C10	0.44543 (15)	0.4393 (8)	0.5080 (4)	0.0446 (9)
H10	0.413622	0.511276	0.559240	0.053*
C9	0.44012 (14)	0.2400 (7)	0.4138 (4)	0.0426 (8)
H9	0.404793	0.148751	0.388029	0.051*
H3	0.5933 (19)	0.694 (8)	0.544 (4)	0.066 (13)*

biological function. Up to date, a great number of imidazole amide compounds have been reported [10–13]. However, there are relatively few reports about diimidazole amides compounds. Herein, we report the crystal structure of a new imidazole-based amide compound, which was synthesized by simple method in high yield.

The single X-ray diffraction analysis shows that the crystal structure agrees well with the expected structure of the title compound 1 (see the structure drawing). The two imidazole cores as the main bodies are connected with the N-formylforamide group. The N-formylformamide bond distances are 1.386(4) Å for C6–N3 and 1.383(4) Å for C5–N3. The C6–N3–C5 bond angel is 127.5(3) Å. The O1–C6 and O2–C5 bond distances are 1.215(4) and 1.211(4) Å, respectively. The N3–C6–O1 and N3–C5–O2 bond angels are 124.2(3) and 124.5(3) Å.

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

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