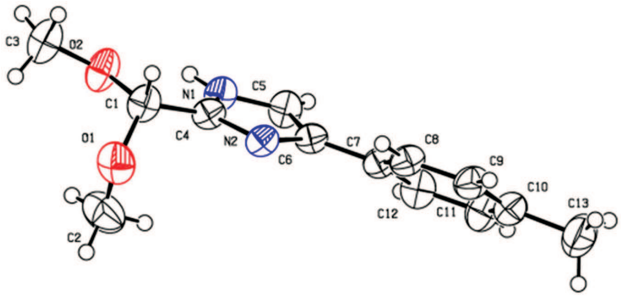
Wan-li Liu, Hong Sui, Gai-mei She*, Hai-Yuan Quan and Dong-Mei She

The crystal structure of 2-(dimethoxymethyl)-4-(4-methylphenyl)-1*H*-imidazole—petroleum ether-chloroform (3/1), $C_{27}H_{33}Cl_3N_4O_4$



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Abstract

$C_{27}H_{33}Cl_3N_4O_4$, monoclinic, $P2_1/n$ (no. 14), $a = 7.1343(4)$ Å, $b = 22.4930(10)$ Å, $c = 9.7835(4)$ Å, $\beta = 106.876(5)$, $V = 1502.37(13)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0605$, $wR_{ref}(F^2) = 0.1517$, $T = 179.99(10)$ K

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The title molecule of 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.

Table 1: Crystal collection and handling.

Crystal:	Block, colorless
Size:	0.4 × 0.2 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (λ = 0.71073 Å)
μ:	0.343 mm^{-1}
Diffractometer, scan mode:	XtaLAB AFC12, Φ-scans
θ_{max}, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9882, 3449, 0.0237
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2471
$N(param)_{refined}$:	199
Programs:	CrysAlis^{PRO} [1], OLEX2 [2], SHELX [3]

Source of material

The title molecule was synthesized according to a method reported previously [4]. By cyclocondensation of acylmethylamine $ZCOCH_2NH_2$ mineral acid salt (Z = alkyl) with acetimidate ester $AOC(:NH)CH(OR)_2$ (A = alkyl). Thus, 0.77 g 28% NaOMe in MeOH was added to 12 mL MeOH, followed by adding dropwise 2.58 g $(EtO)_2CHCN$, and the mixt. was allowed to react at room temp. for 1 h to give a solution of $(EtO)_2CHC(:NH)OMe$ in MeOH. To the latter solution was added portionwise 1.85 g 4-methylphenacylamine hydrochloride and then adding dropwise 1.93 g 28% NaOMe in MeOH. The mixture was allowed to react at room temp. for 1 h and then under refluxing for 2 h. Crystals of the title compound was obtained in a mixed solvent of petroleum ether and chloroform (v/v = 3:1). After stirring for 10 min at room temperature, the mixture was filtered and the filtrate was allowed to stand at room temperature for a week [5]. The solvent partially evaporated and colourless block crystals were obtained.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. Data reduction and empirical absorption correction were performed using the CrysAlis^{PRO} program [1]. Using Olex2 [2], the crystal structure was solved by Direct Methods and refined with the SHELX program system [3].

Comment

The title compound, 2-(dimethoxymethyl)-4-(4-methyl phenyl)-1*H*-imidazole and other imidazole derivatives are important agricultural intermediates [6]. It is important to

***Corresponding author: Gai-mei She,** School of Pharmacy, Ningxia Medical University, Yin chuan 750004, P.R. China; and School of Chinese Pharmacy, Beijing University of Chinese Medicine, Beijing 102488, P.R. China, e-mail: shegaimei@126.com
Wan-li Liu: School of Pharmacy, Ningxia Medical University, Yin chuan 750004, P.R. China; and State Key Laboratory for Biology of Plant Diseases and Insect Pests Institute of Plant Protection, Chinese Academy of Agricultural Sciences, Beijing 100193, P.R. China
Hong Sui: School of Pharmacy, Ningxia Medical University, Yin chuan 750004, P.R. China
Hai-yuan Quan and Dong-mei She: State Key Laboratory for Biology of Plant Diseases and Insect Pests Institute of Plant Protection, Chinese Academy of Agricultural Sciences, Beijing 100193, P.R. China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1[a]	0.7380(6)	0.9834(2)	1.0377(7)	0.2075(17)
Cl2[a]	0.4369(12)	1.03526(19)	1.1120(7)	0.228(2)
Cl3[a]	0.3640(10)	0.9696(2)	0.8713(4)	0.226(2)
C14[a]	0.5216(13)	1.0160(3)	0.9642(9)	0.124(3)
H14[a]	0.535(3)	1.0531(7)	0.921(2)	0.149
O1	1.0818(2)	0.82421(7)	0.84418(15)	0.0585(4)
O2	1.1315(2)	0.79755(7)	0.62672(14)	0.0538(4)
N1	0.8019(2)	0.73629(8)	0.56148(15)	0.0407(4)
H1A	0.836(3)	0.7523(9)	0.486(2)	0.049
N2	0.8188(2)	0.71581(7)	0.78543(15)	0.0377(4)
C1	1.0890(3)	0.77795(10)	0.74974(19)	0.0450(5)
H1	1.1925	0.7503	0.7996	0.054
C2	0.9330(4)	0.86695(12)	0.7872(3)	0.0761(8)
H2A	0.9569	0.8859	0.7060	0.114
H2B	0.9338	0.8963	0.8587	0.114
H2C	0.8078	0.8476	0.7585	0.114
C3	1.3273(4)	0.81779(13)	0.6519(3)	0.0639(7)
H3A	1.3531	0.8248	0.5623	0.096
H3B	1.4161	0.7882	0.7048	0.096
H3C	1.3449	0.8541	0.7058	0.096
C4	0.9023(3)	0.74391(9)	0.70041(17)	0.0374(4)
C5	0.6455(3)	0.70124(9)	0.55780(19)	0.0446(5)
H5	0.5500	0.6884	0.4766	0.054
C6	0.6554(3)	0.68843(8)	0.69643(18)	0.0370(4)
C7	0.5243(3)	0.65141(9)	0.75170(19)	0.0392(4)
C8	0.5963(3)	0.61962(9)	0.8787(2)	0.0428(5)
H8	0.7282	0.6224	0.9296	0.051
C9	0.4738(3)	0.58414(10)	0.9297(2)	0.0492(5)
H9	0.5251	0.5630	1.0140	0.059
C10	0.2757(3)	0.57921(10)	0.8581(2)	0.0514(5)
C11	0.2040(3)	0.61126(11)	0.7325(2)	0.0559(6)
H11	0.0715	0.6090	0.6831	0.067
C12	0.3258(3)	0.64663(10)	0.6794(2)	0.0506(5)
H12	0.2743	0.6674	0.5945	0.061
C13	0.1406(4)	0.54248(13)	0.9168(3)	0.0739(8)
H13A	0.0546	0.5198	0.8410	0.111
H13B	0.0644	0.5682	0.9582	0.111
H13C	0.2164	0.5160	0.9887	0.111

[a] = 0.5

determine the structure of synthetic compounds by X-ray single crystal diffraction before the study of the biological activity. The molecule is mainly composed of a substituted (1*H*)-imidazole ring and a methylphenyl moiety. The asymmetric unit of the title compound contains one title molecule and one half occupied chloroform molecule. In the figure the solvent molecules have been omitted for clarity [7].

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