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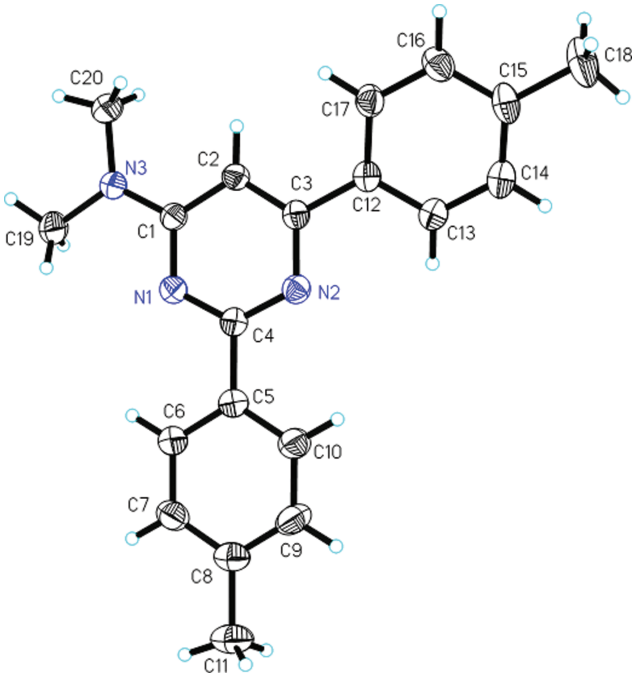
The crystal structure of *N,N*-dimethyl-2,6-di-*p*-tolylpyrimidin-4-amine, $C_{20}H_{21}N_3$



https://doi.org/10.1515/ncrs-2017-0161
Received June 13, 2017; accepted August 29, 2017; available online September 16, 2017

Abstract

$C_{20}H_{21}N_3$, Orthorhombic, *Pbca*, $a = 12.2422(16)$ Å, $b = 7.1996(10)$ Å, $c = 39.037(5)$ Å, $V = 3440.6(8)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0686$, $wR_{ref}(F^2) = 0.1723$, $T = 293(2)$ K.

CCDC no.: 1571314

The asymmetric unit of the title 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.

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Table 1: Data collection and handling.

Crystal:	Prismatic, colorless
Size:	0.20 × 0.17 × 0.11 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.07 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17561, 3040, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2585
$N(param)_{refined}$:	213
Programs:	Bruker programs [1], SHELX [2, 3]

Source of material

The title product was prepared by the modified method reported by Wu's group [5]. Under an argon atmosphere, KO^{t}Bu (224.4 mg, 2.0 mmol), DMAc (2.0 mL) and 4-methylbenzonitrile (393 mg, 3.0 mmol) were successively added into a Schlenk tube. Then the mixture was stirred vigorously at 110°C for 16 h. The reaction mixture was cooled to room temperature, diluted with ethyl acetate (30 mL), and washed with saturated brine. The organic phases were dried over anhydrous Na_2SO_4, filtered, and then the solvent was removed under reduced pressure. The residue was purified by flash column chromatography (SiO_2) to give the title product in 87% yield as a white solid. Crystals were grown in a mixture of ethyl acetate and petroleum ether.

Experimental details

All hydrogen atoms attached to C atoms were introduced using the HFIX command in the SHELXL program [2, 3]. The C–H distances in CH_3 were restrained to 0.96 Å with U_{iso} values to be $1.5U_{eq}$(C). Vinylic and aromatic C–H distances were restrained to 0.93 Å with U_{iso} values to be $1.2U_{eq}$(C) [4].

Discussion

Pyrimidines are frequently found in natural products. Some of them show biological and pharmaceutical activities or can be used as functional materials [6–11].

The bond lengths and angles derived from the title structure are within normal ranges. All ring moieties are almost all in one plane. This study is part of our attempt to synthesize new pyrimidine derivatives with potential applications in medicine and advanced materials.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.68796(14)	0.0993(3)	0.09334(4)	0.0463(5)
N2	0.57627(15)	0.0463(3)	0.14241(4)	0.0468(5)
N3	0.87524(15)	0.0998(3)	0.09666(5)	0.0539(5)
C1	0.77849(17)	0.0797(3)	0.11265(5)	0.0428(5)
C2	0.76975(17)	0.0410(3)	0.14778(5)	0.0436(5)
H2	0.8317	0.0254	0.1613	0.052*
C3	0.66729(17)	0.0267(3)	0.16157(5)	0.0426(5)
C4	0.59221(17)	0.0811(3)	0.10917(5)	0.0430(5)
C5	0.49294(18)	0.1011(3)	0.08759(6)	0.0461(5)
C6	0.5009(2)	0.1356(4)	0.05278(6)	0.0560(6)
H6	0.5694	0.1464	0.0427	0.067*
C7	0.4081(2)	0.1541(4)	0.03299(7)	0.0626(7)
H7	0.4156	0.1769	0.0097	0.075*
C8	0.3051(2)	0.1399(4)	0.04665(7)	0.0597(7)
C9	0.2981(2)	0.1057(4)	0.08149(8)	0.0695(8)
H9	0.2295	0.0952	0.0916	0.083*
C10	0.38968(19)	0.0867(4)	0.10160(6)	0.0618(7)
H10	0.3820	0.0639	0.1249	0.074*
C11	0.2041(2)	0.1577(5)	0.02488(8)	0.0807(9)
H11A	0.2246	0.1905	0.0019	0.121*
H11B	0.1576	0.2525	0.0342	0.121*
H11C	0.1657	0.0415	0.0246	0.121*
C12	0.65013(17)	−0.0134(3)	0.19860(5)	0.0437(5)
C13	0.55324(19)	−0.0930(3)	0.20983(6)	0.0520(6)
H13	0.4988	−0.1209	0.1940	0.062*
C14	0.5363(2)	−0.1316(4)	0.24406(6)	0.0600(7)
H14	0.4710	−0.1863	0.2509	0.072*
C15	0.6147(2)	−0.0905(4)	0.26836(6)	0.0605(7)
C16	0.7109(2)	−0.0103(4)	0.25720(6)	0.0602(7)
H16	0.7647	0.0193	0.2731	0.072*
C17	0.7287(2)	0.0268(3)	0.22295(6)	0.0537(6)
H17	0.7945	0.0797	0.2161	0.064*
C18	0.5960(3)	−0.1290(5)	0.30602(7)	0.0947(11)
H18A	0.5935	−0.0138	0.3183	0.142*
H18B	0.6546	−0.2041	0.3147	0.142*
H18C	0.5280	−0.1937	0.3089	0.142*
C19	0.8801(2)	0.1354(4)	0.06020(6)	0.0664(8)
H19A	0.8715	0.0207	0.0480	0.100*
H19B	0.9494	0.1898	0.0546	0.100*
H19C	0.8226	0.2193	0.0539	0.100*
C20	0.97742(19)	0.0842(4)	0.11494(7)	0.0617(7)
H20A	0.9755	0.1625	0.1348	0.093*
H20B	1.0363	0.1222	0.1003	0.093*
H20C	0.9884	−0.0424	0.1219	0.093*

Acknowledgements: I acknowledge the financial support from Natural Science Foundation of Zhejiang Province (No. LY13B020009) for the publication fee.

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