Crystal structure of (2,4,6-trimethyl-phenyl)-[6-(2,4,6-trimethyl-phenyl)-pyridin-2-yl]-amine, $C_{23}H_{26}N_2$

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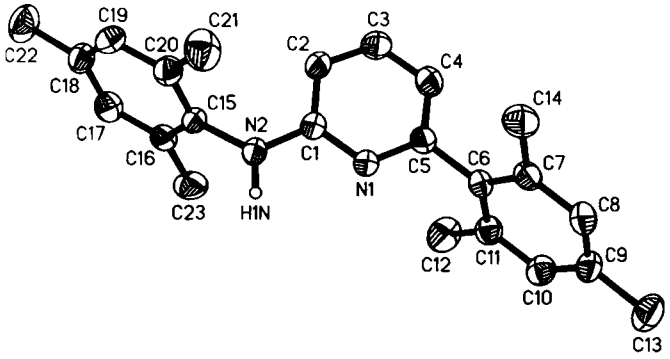

Abstract

$C_{23}H_{26}N_2$, monoclinic, $C12/c1$ (no. 15), $a = 29.968(2)$ Å, $b = 8.156(1)$ Å, $c = 21.365(2)$ Å, $\beta = 132.154(7)°$, $V = 3871.4$ Å^3, $Z = 8$, $R_{gt}(F) = 0.058$, $wR_{ref}(F^2) = 0.146$, $T = 193$ K.

Source of material

The title compound can be synthesised in accordance to a published procedure [1]. Re-crystallization from hexane gave colorless crystals suitable for X-ray crystal structure analysis.

Discussion

Recently, we described the preparation of sterically demanding aminopyridinato ligands by the introduction of 2,6-alkylphenyl substituents at the amido nitrogen atom and in the 6-position of the pyridine ring [1]. Deprotonation of this bulky ligands and salt metathesis reactions with lanthanide halogenides lead to di- and trivalent lanthanide complexes [2]. The title compound is a new member of this type of ligands.

The amino H atom lies almost in plane of the pyridine ring with a deviation of only 0.0068 Å. The amino H atoms form strong intermolecular hydrogen bonds with the adjacent pyridine N atoms (d(H1N···$N_{pyridine}$) = 2.172 Å). These intermolecular hydrogen bonds form a dimeric structure in the solid state. The twisted arrangement of the bulky phenyl substituents to the pyridine ring underlines these intermolecular hydrogen-bonding interactions and the dihedral angles are 98.9° and 99.8°. Both phenyl substituents are slightly twisted, forming a dihedral angle of 6.5°.

Experimental details

All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms except H13A – H13C and H22A – H22C were refined with isotropic displacement parameters. The atoms H13A – H22C are in calculated positions and were refined applying the riding model with fixed U_{iso} values. These hydrogen atoms belong to disordered methyl groups.

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Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.3 x 0.4 x 0.5 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.66 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω/φ
$2\theta_{max}$:	51.64°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21869, 3654
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2466
$N(param)_{refined}$:	306
Programs:	SIR97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1N)	8*f*	1.023(1)	−0.051(3)	0.219(2)	0.054(8)
H(2)	8*f*	0.9206(9)	−0.205(3)	0.015(1)	0.032(6)
H(3)	8*f*	0.816(1)	−0.163(3)	−0.078(2)	0.053(7)
H(4)	8*f*	0.778(1)	−0.026(3)	−0.023(2)	0.052(7)
H(8)	8*f*	0.779(1)	0.037(3)	0.213(1)	0.039(6)
H(10)	8*f*	0.8233(9)	0.474(3)	0.173(1)	0.035(6)
H(13A)	8*f*	0.7609	0.2803	0.2518	0.089
H(13B)	8*f*	0.7431	0.4341	0.1923	0.089
H(13C)	8*f*	0.8091	0.4223	0.2842	0.089
H(17)	8*f*	1.100(1)	−0.059(3)	0.081(1)	0.042(6)
H(19)	8*f*	1.0685(9)	−0.514(3)	0.115(1)	0.038(6)
H(22A)	8*f*	1.1303	−0.2768	0.0465	0.090
H(22B)	8*f*	1.0863	−0.4319	0.0094	0.090
H(22C)	8*f*	1.1508	−0.4371	0.1034	0.090
H(141)	8*f*	0.781(2)	−0.204(4)	0.100(2)	0.09(1)
H(142)	8*f*	0.848(2)	−0.220(5)	0.168(2)	0.11(1)
H(143)	8*f*	0.808(2)	−0.220(4)	0.194(2)	0.10(1)
H(211)	8*f*	1.028(2)	−0.569(5)	0.176(2)	0.12(1)
H(212)	8*f*	0.973(1)	−0.440(3)	0.138(2)	0.066(9)
H(213)	8*f*	1.035(2)	−0.419(4)	0.232(2)	0.10(1)
H(121)	8*f*	0.834(2)	0.356(4)	0.038(2)	0.09(1)
H(122)	8*f*	0.905(2)	0.342(4)	0.130(2)	0.10(1)
H(123)	8*f*	0.864(1)	0.480(4)	0.112(2)	0.071(9)
H(231)	8*f*	1.065(2)	0.169(5)	0.101(2)	0.10(1)
H(232)	8*f*	1.072(1)	0.162(4)	0.179(2)	0.070(9)
H(233)	8*f*	1.008(2)	0.152(4)	0.090(2)	0.08(1)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.94172(9)	–0.0861(3)	0.1161(1)	0.031(1)	0.034(1)	0.034(1)	0.0016(8)	0.023(1)	–0.0009(9)
C(2)	8*f*	0.9040(1)	–0.1487(3)	0.0335(1)	0.035(1)	0.048(1)	0.035(1)	0.001(1)	0.024(1)	–0.008(1)
C(3)	8*f*	0.8435(1)	–0.1251(3)	–0.0176(2)	0.039(1)	0.065(2)	0.033(1)	–0.002(1)	0.020(1)	–0.014(1)
C(4)	8*f*	0.8205(1)	–0.0435(3)	0.0118(2)	0.028(1)	0.065(2)	0.037(1)	0.000(1)	0.018(1)	–0.009(1)
C(5)	8*f*	0.86007(9)	0.0129(3)	0.0939(1)	0.029(1)	0.033(1)	0.034(1)	0.0001(8)	0.0207(9)	–0.0005(9)
C(6)	8*f*	0.83812(9)	0.1020(3)	0.1300(1)	0.024(1)	0.036(1)	0.032(1)	0.0015(8)	0.0176(9)	–0.0004(9)
C(7)	8*f*	0.81509(9)	0.0139(3)	0.1590(1)	0.028(1)	0.032(1)	0.038(1)	–0.0010(9)	0.020(1)	–0.0003(9)
C(8)	8*f*	0.7948(1)	0.0994(3)	0.1919(1)	0.036(1)	0.044(1)	0.046(1)	–0.002(1)	0.031(1)	0.001(1)
C(9)	8*f*	0.79747(9)	0.2686(3)	0.1978(1)	0.034(1)	0.045(1)	0.044(1)	0.006(1)	0.028(1)	–0.001(1)
C(10)	8*f*	0.8205(1)	0.3529(3)	0.1691(1)	0.042(1)	0.030(1)	0.047(1)	0.0054(9)	0.031(1)	0.002(1)
C(11)	8*f*	0.84013(9)	0.2735(3)	0.1339(1)	0.035(1)	0.035(1)	0.039(1)	0.0023(9)	0.025(1)	0.0029(9)
C(12)	8*f*	0.8623(2)	0.3697(4)	0.0992(2)	0.077(2)	0.043(2)	0.075(2)	–0.004(2)	0.062(2)	0.002(2)
C(13)	8*f*	0.7757(1)	0.3593(3)	0.2348(2)	0.066(2)	0.059(2)	0.076(2)	0.005(1)	0.057(2)	–0.008(1)
C(14)	8*f*	0.8128(1)	–0.1702(3)	0.1564(2)	0.053(2)	0.032(1)	0.061(2)	–0.006(1)	0.039(2)	–0.003(1)
C(15)	8*f*	1.03300(9)	–0.1683(3)	0.1460(1)	0.028(1)	0.040(1)	0.030(1)	0.0021(9)	0.0186(9)	–0.0059(9)
C(16)	8*f*	1.05643(9)	–0.0629(3)	0.1231(1)	0.030(1)	0.035(1)	0.031(1)	0.0008(8)	0.017(1)	–0.0013(9)
C(17)	8*f*	1.0846(1)	–0.1307(3)	0.0977(1)	0.038(1)	0.043(1)	0.040(1)	–0.001(1)	0.029(1)	0.003(1)
C(18)	8*f*	1.08915(9)	–0.2985(3)	0.0934(1)	0.036(1)	0.048(1)	0.039(1)	0.004(1)	0.027(1)	–0.003(1)
C(19)	8*f*	1.0660(1)	–0.3993(3)	0.1176(2)	0.043(1)	0.030(1)	0.048(1)	0.001(1)	0.031(1)	–0.005(1)
C(20)	8*f*	1.03841(9)	–0.3385(3)	0.1449(1)	0.037(1)	0.036(1)	0.042(1)	–0.0005(9)	0.028(1)	–0.0009(9)
C(21)	8*f*	1.0152(2)	–0.4518(4)	0.1729(2)	0.059(2)	0.051(2)	0.076(2)	–0.002(1)	0.051(2)	0.008(2)
C(22)	8*f*	1.1165(1)	–0.3671(4)	0.0602(2)	0.060(2)	0.079(2)	0.057(2)	0.013(1)	0.046(1)	–0.003(1)
C(23)	8*f*	1.0501(1)	0.1197(3)	0.1238(2)	0.051(2)	0.032(1)	0.052(2)	0.003(1)	0.027(1)	–0.003(1)
N(1)	8*f*	0.91993(7)	–0.0086(2)	0.1461(1)	0.0283(9)	0.033(1)	0.0317(9)	0.0006(7)	0.0204(8)	–0.0018(7)
N(2)	8*f*	1.00277(8)	–0.1022(3)	0.1706(1)	0.0276(9)	0.057(1)	0.035(1)	0.0022(8)	0.0196(9)	–0.0120(9)

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