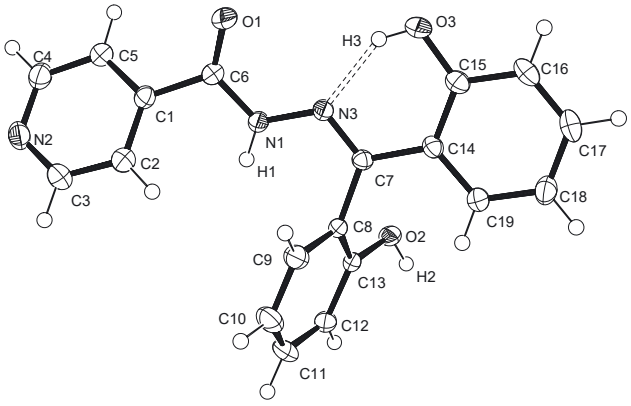
Itumeleng B. Setshedi, Andreas Lemmerer and Mark G. Smith*

The crystal structure of *N′*-[bis(2-hydroxyphenyl) methylidene]pyridine-4-carbohydrazide, $C_{19}H_{15}N_3O_3$



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Abstract

$C_{19}H_{15}N_3O_3$, monoclinic, *C*2/*c* (no. 15), a = 20.6917(6) Å, b = 10.1392(3) Å, c = 16.9544(5) Å, β = 115.739(1)°, V = 3204.07(17) Å^3, Z = 8, $R_{gt}(F)$ = 0.0364, $wR_{ref}(F^2)$ = 0.1009, T = 173 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Mark G. Smith**, Department of Chemistry, University of South Africa, Unisa Science Campus, 28 Pioneer Avenue, Florida, Roodepoort, Gauteng, South Africa,
E-mail: smithm2@unisa.ac.za. https://orcid.org/0000-0003-2553-2540
Itumeleng B. Setshedi, Department of Life Science, University of South Africa, Unisa Science Campus, 28 Pioneer Avenue, Florida, Roodepoort, Gauteng, South Africa. https://orcid.org/0000-0001-8865-4735
Andreas Lemmerer, Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, Johannesburg, Gauteng, South Africa. https://orcid.org/0000-0003-1569-2831

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.36 × 0.25 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture Photon, ω
θ_{max}, completeness:	28.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	23,999, 3872, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3340
$N(param)_{refined}$:	238
Programs:	Bruker [1], SHELX [2, 3], ORTEP-3 [4], WinGX [5], PLATON [6]

Source of material

All reagents used were used without further purification. An amount of 0.0857 g of isonicotinic acid hydrazide (isoniazid) (0.6249 mmol) and 0.1340 g of 2,2′-dihydroxybenzophenone (0.6255 mmol) were added into a vial. *p*-Hydroxybenzoic acid 0.086 g (0.6255 mmol) was added to catalyze the reaction. The solids were dissolved in 3 mL of AP-grade methanol and refluxed in a closed screw-top vial for 24 h at 67 °C, and were left to stand at room temperature slightly open to the atmosphere. Yellow plates were afforded after 3 days.

Experimental details

C-bound hydrogen atoms were located in the difference map then positioned geometrically and were allowed to ride on their respective parent atoms with thermal displacement parameters 1.2 times of the parent C atom. The coordinates and isotropic displacement parameters of the N-bound and O-bound H atoms involved in hydrogen bonding interactions were allowed to refine freely.

Comment

Isoniazid has been modified to enhance activity against multidrug resistant mycobacteria [7]. Several modified

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.18530 (6)	0.48217 (11)	0.00875 (7)	0.0229 (2)
C2	−0.18090 (7)	0.59801 (12)	−0.03244 (8)	0.0313 (3)
H2A	−0.135895	0.629967	−0.02665	0.038*
C3	−0.24367 (7)	0.66582 (13)	−0.08216 (8)	0.0357 (3)
H3A	−0.240643	0.744564	−0.110745	0.043*
C4	−0.31206 (6)	0.51342 (12)	−0.05324 (8)	0.0309 (3)
H4	−0.357842	0.483541	−0.060549	0.037*
C5	−0.25246 (6)	0.43823 (11)	−0.00302 (8)	0.0266 (2)
H5	−0.257386	0.358104	0.022907	0.032*
C6	−0.12008 (6)	0.39996 (11)	0.06116 (7)	0.0222 (2)
C7	0.06093 (5)	0.48892 (10)	0.18322 (7)	0.0188 (2)
C8	0.05681 (5)	0.63347 (10)	0.16316 (7)	0.0192 (2)
C9	0.03440 (6)	0.67069 (11)	0.07594 (8)	0.0273 (2)
H9	0.023726	0.604289	0.032532	0.033*
C10	0.02733 (7)	0.80184 (12)	0.05115 (8)	0.0352 (3)
H10	0.012886	0.825523	−0.008337	0.042*
C11	0.04170 (7)	0.89814 (12)	0.11462 (9)	0.0341 (3)
H11	0.036033	0.988493	0.09816	0.041*
C12	0.06415 (6)	0.86438 (10)	0.20145 (8)	0.0261 (2)
H12	0.0739	0.931475	0.244231	0.031*
C13	0.07259 (5)	0.73183 (10)	0.22665 (7)	0.0195 (2)
C14	0.13043 (6)	0.42320 (10)	0.23167 (7)	0.0203 (2)
C15	0.13587 (6)	0.28567 (11)	0.24744 (7)	0.0244 (2)
C16	0.20348 (7)	0.22849 (12)	0.29311 (8)	0.0302 (3)
H16	0.207129	0.136371	0.30448	0.036*
C17	0.26498 (7)	0.30374 (13)	0.32192 (8)	0.0321 (3)
H17	0.310523	0.263249	0.35287	0.038*
C18	0.26061 (6)	0.43870 (13)	0.30584 (8)	0.0301 (3)
H18	0.302982	0.490499	0.325066	0.036*
C19	0.19407 (6)	0.49667 (11)	0.26166 (7)	0.0246 (2)
H19	0.191331	0.589034	0.25127	0.03*
N1	−0.06094 (5)	0.47504 (9)	0.10727 (6)	0.0233 (2)
N2	−0.30829 (6)	0.62593 (10)	−0.09190 (7)	0.0324 (2)
N3	0.00444 (5)	0.41459 (9)	0.15336 (6)	0.02117 (19)
O1	−0.12054 (4)	0.28042 (8)	0.05804 (6)	0.0318 (2)
O2	0.09561 (4)	0.69679 (8)	0.31188 (5)	0.02388 (18)
O3	0.07849 (5)	0.20449 (8)	0.21997 (6)	0.0337 (2)
H1	−0.0664 (8)	0.5585 (16)	0.1199 (10)	0.037 (4)*
H2	0.1230 (10)	0.768 (2)	0.3467 (12)	0.062 (5)*
H3	0.0405 (11)	0.2535 (19)	0.1922 (12)	0.058 (5)*

isoniazid derivative have been synthesized [8–12]. In this paper we present the crystal structure of isoniazid modified with 2,2′-dihydroxybenzophenone. The compound crystallizes in the monoclinic *C*2/*c* space group and the asymmetric unit contains one molecule. There is one hydroxyl group on each of the benzophenone rings. As expected from Etter's rules of hydrogen bonding [13], one of the hydroxyl groups forms a six-membered intramolecular hydrogen bonded ring with the hydrazide nitrogen atom (N3) and is not involved in anyntermolecular interactions. The other hydroxyl group is hydrogen bonded to the pyridyl nitrogen atom of an adjacent molecule *via* the robust O–H···$N_{pyridine}$ heterosynthon. Its oxygen atom acts as an acceptor to the amide hydrogen atom donor (H1). The packing of the crystal forms one-dimensional ribbons with a two-one screw axis. These hydrogen bonding patterns form two rings, namely an R_4^4 (18) ring involving four molecules, and an R_4^4 (14) ring involving three molecules.

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