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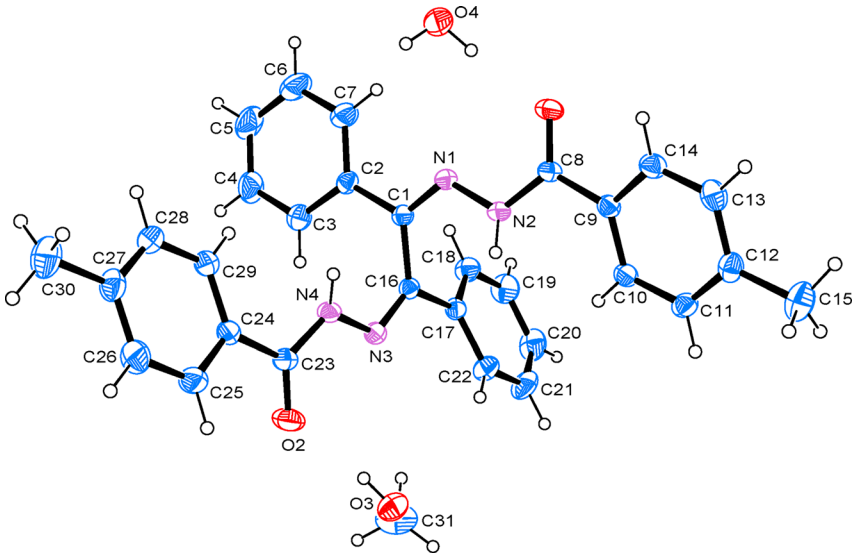
Crystal structure of *N′*,*N‴*-((1*E*,2*E*)-1,2-diphenylethane-1,2-diylidene) bis(4-methylbenzohydrazide) – water – methanol (1/1/1), $C_{31}H_{32}N_4O_4$



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Abstract

$C_{31}H_{32}N_4O_4$, monoclinic, *P*2/*c* (no. 13), a = 23.938(2) Å, b = 9.1931(8) Å, c = 12.6327(10) Å, β = 91.828(7)°, V = 2778.6(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0544, $wR_{ref}(F^2)$ = 0.1388, T = 296(2) 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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.13 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	SuperNova, φ and ω
θ_{max}, completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12635, 4956, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3371
$N(param)_{refined}$:	373
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

All chemicals were purchased from commercial sources and used as received without further purification. The 4-methylbenzohydrazide (5 mmol, 0.751 g) and benzaldehyde (2.5 mmol, 0.526 g) were dissolved in MeOH (20 mL). The mixture was refluxed for 8 h, and then the precipitate was collected by filtration. The solid was filtered out and the title product was obtained by recrystallization from methanol and water (9:1).

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.74432 (7)	0.7393 (2)	0.42355 (14)	0.0319 (5)
C2	0.78656 (8)	0.8224 (2)	0.36580 (15)	0.0360 (5)
C3	0.83091 (9)	0.8909 (3)	0.41865 (18)	0.0498 (6)
H3	0.834733	0.882492	0.491887	0.060*
C4	0.86948 (10)	0.9713 (3)	0.3646 (2)	0.0660 (7)
H4A	0.898989	1.016375	0.401246	0.079*
C5	0.86417 (11)	0.9844 (3)	0.2566 (2)	0.0759 (8)
H5	0.890024	1.038425	0.219705	0.091*
C6	0.82037 (12)	0.9173 (3)	0.2033 (2)	0.0745 (8)
H6	0.816647	0.926780	0.130118	0.089*
C7	0.78226 (10)	0.8370 (3)	0.25613 (17)	0.0561 (7)
H7	0.753129	0.791584	0.218543	0.067*
C8	0.62583 (8)	0.5277 (2)	0.36624 (15)	0.0365 (5)
C9	0.58608 (8)	0.4353 (2)	0.42391 (14)	0.0342 (5)
C10	0.57737 (8)	0.4465 (2)	0.53167 (15)	0.0368 (5)
H10	0.598034	0.512902	0.572295	0.044*
C11	0.53834 (8)	0.3599 (2)	0.57893 (16)	0.0420 (5)
H11	0.533149	0.368921	0.651284	0.050*
C12	0.50661 (8)	0.2597 (2)	0.52106 (17)	0.0441 (5)
C13	0.51507 (9)	0.2501 (2)	0.41298 (18)	0.0491 (6)
H13	0.494090	0.184349	0.372329	0.059*
C14	0.55399 (8)	0.3363 (2)	0.36489 (16)	0.0431 (5)
H14	0.558862	0.328142	0.292383	0.052*
C15	0.46370 (10)	0.1669 (3)	0.5731 (2)	0.0682 (7)
H15A	0.481843	0.085862	0.607607	0.102*
H15B	0.437201	0.131823	0.520457	0.102*
H15C	0.444626	0.223724	0.624489	0.102*
C16	0.74538 (7)	0.7481 (2)	0.54284 (14)	0.0313 (5)
C17	0.70449 (8)	0.8471 (2)	0.58996 (15)	0.0352 (5)
C18	0.68577 (9)	0.9689 (2)	0.53431 (17)	0.0473 (6)
H18	0.697555	0.984259	0.465769	0.057*
C19	0.64998 (10)	1.0674 (3)	0.5789 (2)	0.0613 (7)
H19	0.638591	1.149530	0.541035	0.074*
C20	0.63121 (10)	1.0449 (3)	0.6787 (2)	0.0650 (7)
H20	0.607223	1.111545	0.708971	0.078*
C21	0.64816 (10)	0.9222 (3)	0.73434 (19)	0.0610 (7)
H21	0.634931	0.905760	0.801722	0.073*
C22	0.68445 (9)	0.8240 (3)	0.69099 (16)	0.0486 (6)
H22	0.695616	0.742040	0.729284	0.058*
C23	0.85852 (8)	0.5276 (2)	0.62120 (15)	0.0414 (5)
C24	0.90105 (8)	0.4410 (2)	0.56546 (15)	0.0392 (5)
C25	0.93227 (9)	0.3398 (3)	0.62359 (17)	0.0514 (6)
H25	0.925920	0.326614	0.695191	0.062*
C26	0.97268 (10)	0.2590 (3)	0.57514 (19)	0.0582 (7)
H26	0.993094	0.191112	0.614889	0.070*
C27	0.98368 (9)	0.2760 (3)	0.46932 (18)	0.0509 (6)
C28	0.95258 (9)	0.3780 (3)	0.41252 (17)	0.0502 (6)
H28	0.959201	0.391369	0.341035	0.060*
C29	0.91213 (8)	0.4601 (3)	0.45911 (16)	0.0453 (6)
H29	0.892108	0.528686	0.419274	0.054*
C30	1.02798 (10)	0.1879 (3)	0.4167 (2)	0.0748 (8)
H30A	1.055343	0.156412	0.468985	0.112*
H30B	1.045741	0.246632	0.364681	0.112*
H30C	1.011105	0.104594	0.382897	0.112*
C31	0.77931 (14)	0.7276 (3)	0.9140 (2)	0.0845 (9)
H31A	0.750633	0.730511	0.965370	0.127*
H31B	0.769878	0.793245	0.857047	0.127*
H31C	0.814320	0.756206	0.946846	0.127*
H4	0.8113 (8)	0.552 (2)	0.4972 (17)	0.049 (7)*
H4B	0.7312 (16)	0.540 (5)	0.152 (3)	0.152 (18)*
H4C	0.6772 (12)	0.579 (3)	0.170 (2)	0.089 (11)*
N1	0.70647 (6)	0.67069 (18)	0.36962 (11)	0.0346 (4)
N2	0.66836 (7)	0.59019 (19)	0.42412 (13)	0.0351 (4)
H2	0.6762 (8)	0.557 (2)	0.4857 (17)	0.046 (6)*
N3	0.78002 (6)	0.68008 (19)	0.60525 (12)	0.0358 (4)
N4	0.81760 (7)	0.5887 (2)	0.55857 (13)	0.0370 (4)
O1	0.61953 (6)	0.5495 (2)	0.27115 (10)	0.0604 (5)
O2	0.86159 (7)	0.5467 (2)	0.71686 (11)	0.0682 (6)
O3	0.78378 (7)	0.58687 (18)	0.87436 (12)	0.0566 (5)
H3A	0.805138	0.586638	0.824882	0.085*
O4	0.70195 (9)	0.5666 (2)	0.12220 (14)	0.0627 (5)

Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq} (parent C-atom). The H-atom of the hydroxyl group in the methanol molecule is refined with U_{iso}(H) = 1.5 U_{eq} (O). The H-atoms at the N-atoms and in the water molecule are refined using isotropic approximation.

Comment

Generally, Schiff bases are synthesized through the condensation reaction of aldehydes or ketones with primary amines [5–7]. Schiff base compounds and their metal complexes have been studied for many years and successfully applied in many fields [8, 9]. Bisaroyl hydrazones are known to be a class of versatile Schiff bases ligands, capable of generating different molecular architectures and coordination polyhedra [10]. So, the synthesis and crystal structure of bisaroyl hydrazone derivatives are of great significance to studying the application.

Single-crystal structure analysis revealed that the title compound crystallized in the monoclinic space group *P*2/*c*. The ORTEP diagram is presented in Figure 1. Bond lengths and angles are all in the expected ranges. The title molecule exhibits an *E* configuration. The bond length of C1=N1, C16=N3, C8–N2 and C23–N4 are 1.282(2), 1.288(2), 1.361(2), and 1.361(2) Å, respectively, which are similar to those reported in the literature [11, 12]. The bond length of C8=O1

and C23=O2 are 1.222(2) and 1.221(2) Å, respectively, they are similar to those reported in the literature [13, 14].

The methanol and water in the structure participates in the construction of hydrogen bonds. The oxygen atom O3 of methanol provides one hydrogen bond to O2 of the acylhydrazone molecule (O3···O2 = 2.793(2) Å), the oxygen atom O4 of water provides three hydrogen bonds to O1 of the acylhydrazone molecule (O4···O1 = 2.774(2) Å), N3″ and N4″ of another acylhydrazone molecule (O4···N3″ = 2.951(3) Å; O4···N4″ = 3.239(3) Å; ″ = x, $-y$, $z - 1/2$), the nitrogen atom N2 of acylhydrazone provides one hydrogen bond to O4 of the water molecule (N2···O4′ = 2.976(2) Å; ′ = x, $-y$, $z + 1/2$), the nitrogen atom N4 of acylhydrazone provides one hydrogen bond to O3 of the methanol molecule (N2···O3′ = 2.925(2) Å; ′ = x, $-y$, $z + 1/2$).

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