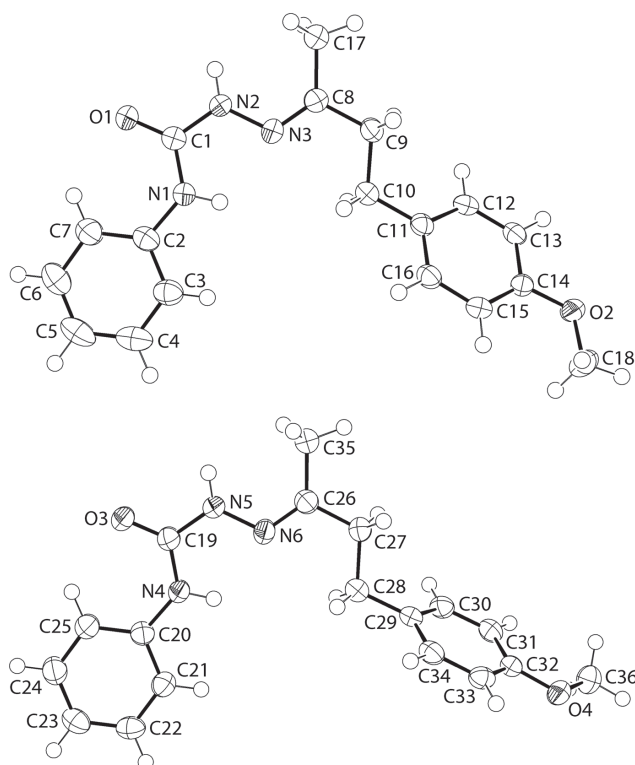


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Crystal structure of 1-[(Z)-[4-(4-methoxyphenyl)butan-2-ylidene]amino]-3-phenylurea, C₁₈H₂₁N₃O₂



$\beta = 89.689(4)^\circ$, $\gamma = 80.666(4)^\circ$, $V = 1683.74(14) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.055$, $wR_{\text{ref}}(F^2) = 0.133$, $T = 100 \text{ K}$.

CCDC no.: 1830404

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	$0.44 \times 0.29 \times 0.10 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	0.8 cm^{-1}
Diffractometer, scan mode:	SuperNova Dual, ω scans
$2\theta_{\text{max}}$, completeness:	55° , $>99\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15637, 7710, 0.040
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4964
$N(\text{param})_{\text{refined}}$:	431
Programs:	Agilent [1], SHELX [2, 3], ORTEP [4]

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

To a solution of 4-phenylsemicarbazide (0.151 g, 1 mmol) in heated absolute ethanol (20 mL) was added slowly a heated ethanol solution (20 mL) of 4-methoxy-2-butanone (0.102 g, 1 mmol) while stirring for 20 min. The white precipitate was filtered, washed with cold ethanol and dried in vacuo. Single crystals were grown at room temperature from slow evaporation of a mixture of ethanol and acetonitrile (1:1 v/v). IR (cm^{-1}) 3338 (N–H), 1665 (C=O), 1597 (C=N), 1239 (C–N), 1025 (C=S). MS: m/z 311.25 $[M]^+$.

Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95–0.99 $\text{\AA}$) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C})$. The N-bound H-atoms were located in a difference Fourier map but were refined with a distance restraint of N–H = $0.88 \pm 0.01 \text{ \AA}$, and with $U_{\text{iso}}(\text{H})$ set to $1.2 U_{\text{eq}}(\text{N})$.

Comment

It is well known that the condensation of semicarbazides with aldehydes/ketones gives rise to a class of potential Schiff base ligands. These molecules attract interest in

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Abstract

C₁₈H₂₁N₃O₂, triclinic, $P\bar{1}$ (no. 2), $a = 8.5155(4) \text{ \AA}$, $b = 10.6415(4) \text{ \AA}$, $c = 19.0732(10) \text{ \AA}$, $\alpha = 80.918(4)^\circ$,

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.62938(16)	0.17977(12)	0.45757(7)	0.0325(4)
O2	0.20571(15)	0.95078(12)	−0.03809(6)	0.0274(3)
N1	0.65711(19)	0.26364(15)	0.34056(8)	0.0265(4)
H1N	0.621(2)	0.3328(14)	0.3101(9)	0.032*
N2	0.4832(2)	0.37184(15)	0.41117(8)	0.0278(4)
H2N	0.437(2)	0.3733(18)	0.4526(7)	0.033*
N3	0.44148(18)	0.46161(14)	0.35062(8)	0.0240(4)
C1	0.5933(2)	0.26540(17)	0.40598(10)	0.0252(4)
C2	0.7657(2)	0.16579(17)	0.31674(10)	0.0250(4)
C3	0.8087(2)	0.18784(19)	0.24596(10)	0.0288(5)
H3	0.7658	0.2666	0.2167	0.035*
C4	0.9134(2)	0.0962(2)	0.21768(12)	0.0353(5)
H4	0.9415	0.1122	0.1692	0.042*
C5	0.9768(3)	−0.0181(2)	0.25965(12)	0.0380(5)
H5	1.0492	−0.0810	0.2405	0.046*
C6	0.9343(3)	−0.04031(19)	0.32977(12)	0.0378(5)
H6	0.9779	−0.1194	0.3586	0.045*
C7	0.8291(2)	0.05033(18)	0.35935(11)	0.0315(5)
H7	0.8010	0.0336	0.4078	0.038*
C8	0.3416(2)	0.56302(17)	0.35577(10)	0.0239(4)
C9	0.2972(2)	0.65680(17)	0.28843(9)	0.0239(4)
H9A	0.3307	0.7399	0.2933	0.029*
H9B	0.1799	0.6727	0.2823	0.029*
C10	0.3715(2)	0.61082(17)	0.22189(9)	0.0238(4)
H10A	0.4886	0.5933	0.2288	0.029*
H10B	0.3366	0.5282	0.2170	0.029*
C11	0.3316(2)	0.70338(16)	0.15329(9)	0.0211(4)
C12	0.1768(2)	0.76840(16)	0.13697(9)	0.0213(4)
H12	0.0956	0.7563	0.1705	0.026*
C13	0.1385(2)	0.84978(16)	0.07345(9)	0.0215(4)
H13	0.0326	0.8937	0.0641	0.026*
C14	0.2555(2)	0.86751(16)	0.02301(9)	0.0211(4)
C15	0.4095(2)	0.80246(16)	0.03690(10)	0.0231(4)
H15	0.4896	0.8123	0.0026	0.028*
C16	0.4454(2)	0.72220(17)	0.10202(10)	0.0235(4)
H16	0.5516	0.6789	0.1116	0.028*
C17	0.2642(2)	0.59499(18)	0.42328(10)	0.0302(5)
H17A	0.1897	0.5354	0.4386	0.045*
H17B	0.2064	0.6836	0.4149	0.045*
H17C	0.3461	0.5868	0.4604	0.045*
C18	0.3235(2)	0.9762(2)	−0.08930(10)	0.0324(5)
H18A	0.4076	1.0111	−0.0676	0.049*
H18B	0.2746	1.0390	−0.1295	0.049*
H18C	0.3694	0.8959	−0.1060	0.049*
O3	0.33971(16)	0.34631(12)	0.54839(7)	0.0327(4)
O4	0.82627(15)	−0.44318(12)	1.03135(7)	0.0284(3)
N4	0.32024(18)	0.26414(14)	0.66636(8)	0.0252(4)
H4N	0.361(2)	0.1956(14)	0.6972(9)	0.030*
N5	0.48823(19)	0.15496(15)	0.59335(8)	0.0268(4)
H5N	0.537(2)	0.1558(18)	0.5519(7)	0.032*
N6	0.53493(18)	0.06471(14)	0.65343(8)	0.0245(4)
C19	0.3792(2)	0.26156(17)	0.59999(10)	0.0251(4)
C20	0.2120(2)	0.36143(17)	0.69096(10)	0.0231(4)
C21	0.1890(2)	0.34831(18)	0.76388(10)	0.0259(4)
H21	0.2450	0.2756	0.7942	0.031*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C22	0.0855(2)	0.44006(18)	0.79290(10)	0.0283(4)
H22	0.0713	0.4302	0.8428	0.034*
C23	0.0029(2)	0.54582(18)	0.74927(11)	0.0295(5)
H23	−0.0687	0.6087	0.7688	0.035*
C24	0.0255(2)	0.55916(19)	0.67699(11)	0.0323(5)
H24	−0.0312	0.6319	0.6470	0.039*
C25	0.1297(2)	0.46828(18)	0.64711(10)	0.0289(5)
H25	0.1443	0.4792	0.5972	0.035*
C26	0.6373(2)	−0.03487(17)	0.64658(10)	0.0243(4)
C27	0.6851(2)	−0.13092(17)	0.71260(9)	0.0246(4)
H27A	0.8021	−0.1428	0.7188	0.030*
H27B	0.6570	−0.2149	0.7057	0.030*
C28	0.6084(2)	−0.09378(17)	0.78052(10)	0.0266(4)
H28A	0.6317	−0.0079	0.7865	0.032*
H28B	0.4915	−0.0868	0.7757	0.032*
C29	0.6656(2)	−0.18903(17)	0.84620(9)	0.0224(4)
C30	0.6270(2)	−0.31287(17)	0.85763(10)	0.0252(4)
H30	0.5632	−0.3376	0.8232	0.030*
C31	0.6792(2)	−0.40153(17)	0.91802(10)	0.0249(4)
H31	0.6523	−0.4858	0.9242	0.030*
C32	0.7710(2)	−0.36547(17)	0.96925(10)	0.0219(4)
C33	0.8103(2)	−0.24187(17)	0.95914(10)	0.0241(4)
H33	0.8729	−0.2165	0.9938	0.029*
C34	0.7577(2)	−0.15610(17)	0.89839(10)	0.0250(4)
H34	0.7853	−0.0721	0.8921	0.030*
C35	0.7140(3)	−0.06337(19)	0.57839(10)	0.0321(5)
H35A	0.6314	−0.0586	0.5421	0.048*
H35B	0.7772	−0.1501	0.5863	0.048*
H35C	0.7835	0.0000	0.5624	0.048*
C36	0.7948(3)	−0.57233(18)	1.04164(11)	0.0332(5)
H36A	0.6798	−0.5715	1.0446	0.050*
H36B	0.8472	−0.6199	1.0858	0.050*
H36C	0.8360	−0.6144	1.0016	0.050*

terms of potential biological activity, most notably in the context of their anti-convulsant properties with the 4-(4-fluorophenoxy)benzaldehyde semicarbazone being the subject of considerable investigations in this regard [5]. In connection with on-going studies of the biological activity of transition metal thiosemicarbazone complexes [6], recently the synthesis and characterization of a new Schiff base ligand, derived from the reaction of aryl semicarbazide and vanillylacetone, was described [7]. In continuation of these studies, the title Schiff base molecule was characterized.

Two independent molecules comprise the asymmetric unit of the title compound. As seen from the Figure (70% displacement ellipsoids), the molecules present many similarities but, with the obvious difference related to the relative disposition of the methoxyphenyl residues. The molecule comprises a di-substituted urea residue. At one end, there is a phenyl ring while at the other, an imine (*Z*-configuration) group connects the urea residue to the 4-methoxyphenyl ring

via an ethane link. The four atoms of the urea core are strictly planar with a r.m.s. deviation of 0.0004 Å for the fitted atoms [0.0014 Å for the O3-molecule]. The amine-N—H and imine-N atoms are syn, a disposition that enables the formation of intramolecular amine-N—H $\cdots$ N(imine) hydrogen bonds [N1—H1n $\cdots$ N3: 2.111(16) Å and 113.8(14)°; N4—H4n $\cdots$ N6: 2.125(16) Å and 113.7(13)°]. The dihedral angle between the CN₂O plane and the adjacent phenyl ring is 3.85(13)° [7.54(12)°], consistent with a co-planar relationship. The dihedral angles between the outer phenyl rings are 37.56(8) and 66.56(5)° for the O1- and O3-molecules, respectively.

The most prominent feature of the molecular packing is the formation of an eight-membered { $\cdots$ HNCO}₂ amide synthon formed between the two independent molecules comprising the asymmetric unit [N2—H2n $\cdots$ O3: 2.001(14) Å and 171.0(18)°; N5—H5n $\cdots$ O1: 1.956(14) Å and 172.8(17)°].

There is a sole literature precedent for molecules of this type as discussed recently [7]. The structure of this molecule, derived from the reaction of semicarbazide and vanillylacetone, presents very similar features to that described above.

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