

1-Acetyl-3-(*p*-chlorobenzyl)-4-(*p*-chlorobenzylideneamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-oneNazan Ocak,<sup>a\*</sup> Ufuk Çoruh,<sup>b</sup>  
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## Key indicators

Single-crystal X-ray study

T = 293 K

Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$ 

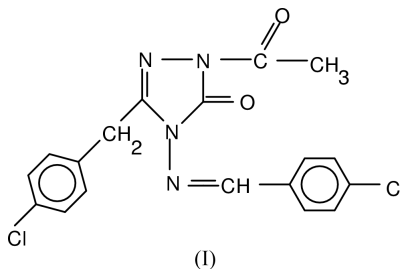
R factor = 0.060

wR factor = 0.133

Data-to-parameter ratio = 11.6

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound,  $\text{C}_{18}\text{H}_{14}\text{Cl}_2\text{N}_4\text{O}_2$ , the triazole ring and benzylideneamino moiety are each planar, and approximately coplanar with respect to each other. The crystal structure is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{N}$  intra- and intermolecular hydrogen bonds.

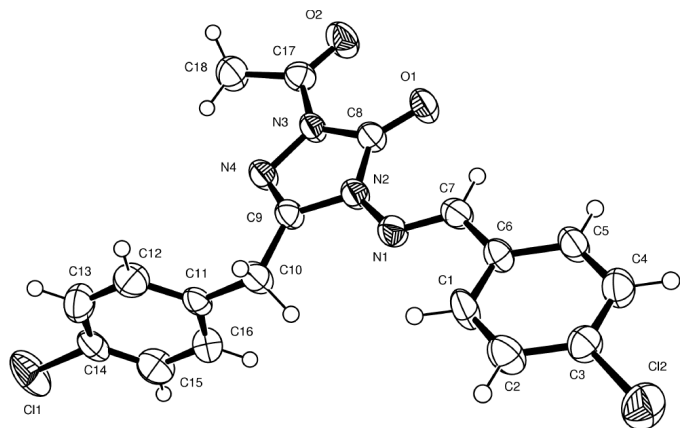
## Comment

The 1,2,4-Triazole ring systems are very interesting because of their similarity to the 1,3-imidazole bridging found in the copper–zinc protein superoxide dismutase (Feiters, 1990) and for their ability to act as a doubly bidentate chelating ligand (Wang *et al.*, 1998). Compounds containing these systems have been structurally characterized as part of a study of Schiff bases of amine- and thione-substituted triazoles (McCarrick *et al.*, 1999; Sen *et al.*, 1998) and their metal complexes (Sen *et al.*, 1996, 1997; Clark *et al.*, 1999). Most Schiff bases possess antibacterial, anticancer, *anti*-inflammatory and antitoxic activities (Williams, 1972), and sulfur-containing Schiff bases are particularly effective. The 4-amino-3-methyl-1,2,4-triazole-5-thione derivative of *p*-nitrophenylaldehyde has been structurally characterized and it has been shown to be highly effective as an inhibitor of *Staphylococcus aureus* (Liu *et al.*, 1999). Therefore, the crystal structure analysis of the title compound, (I), has been carried out.The title compound consists of one 1,2,4-triazole ring (ring A: N3/C8/N2/C9/N4) with an acetyl group substituted at N3, a benzylideneamino group, a *p*-chlorobenzyl group, and an O atom substituted at C8.The  $\text{N}=\text{C}$  bond lengths [ $\text{N1}=\text{C7} = 1.276(4) \text{ \AA}$  and  $\text{N4}=\text{C9} = 1.280(4) \text{ \AA}$ ] are close to the values reported in the literature (Puviasaran *et al.*, 1999; Liu *et al.*, 1999; Zhu *et al.*, 2000), while the presence of the acetyl group at N3 and the benzylideneamino group at C9 causes a small, but significant, stretching of the  $\text{N}-\text{N}$  bond [ $\text{N3}-\text{N4} = 1.404(4) \text{ \AA}$ ].The  $\text{Cl1}-\text{C14}$  and  $\text{Cl2}-\text{C3}$  bond lengths [ $1.744(4)$  and  $1.742(4) \text{ \AA}$ , respectively] conform to those found previously (Wang *et al.*, 1998; Puviasaran *et al.*, 1999).

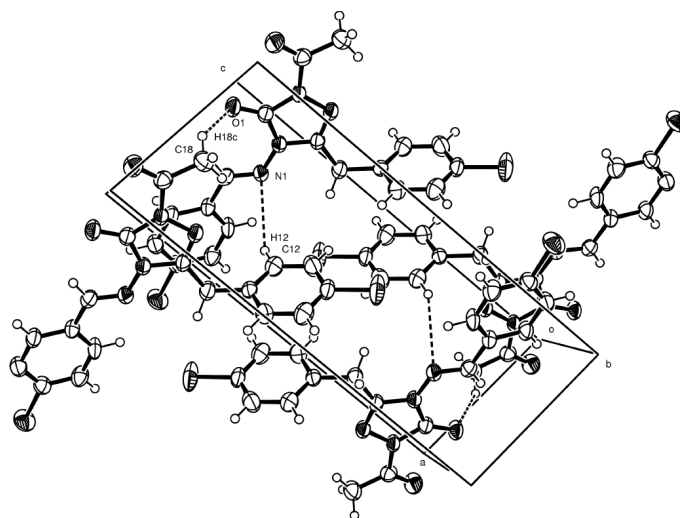
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**Figure 1**  
The molecule of the title compound, showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are shown at the 50% probability level.



**Figure 2**  
A packing diagram of the title compound, illustrating the hydrogen-bonding network.

The title compound consists of three planar rings. One is the triazole ring, ring *A*; the others are ring *B* (C1–C6) and ring *C* (C11–C16). In the triazole ring, the maximum deviation from planarity is 0.008 (3) Å for atom C9. Atom O1 is 0.024 (3) Å from the plane. The dihedral angles between rings *A/B*, *A/C* and *B/C* are 11.1 (1), 84.0 (1) and 89.1 (1)°, respectively.

The crystal structure of (1) is stabilized by four intramolecular and two intermolecular hydrogen bonds. Indeed all intramolecular hydrogen bonds seem to drive the orientation of the substituents with respect to the triazole rings as indicated by the near coplanarity of the groups involved. In the molecule, the *p*-chlorobenzyl and the acetyl group are able to form hydrogen bonds with the benzylideneamino and atom O1 of a symmetry-related molecule. For details of the C–H···N and C–H···O inter- and intramolecular hydrogen bonds found in the crystal see Table 2. These contacts generate infinite chains in the crystal and seem to force the molecule to adopt a twisted conformation.

## Experimental

3-(*p*-Chlorobenzyl)-4-(*p*-chlorobenzylideneamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one (0.01 mol) was treated with 10 ml of acetic anhydride and the mixture was refluxed for 1 h. The resulting product was filtered off and dried *in vacuo*. Several recrystallizations from ethyl acetate gave the pure title compound. Yield: 84%, m.p.: 461–462 K.

### Crystal data

C<sub>18</sub>H<sub>14</sub>Cl<sub>2</sub>N<sub>4</sub>O<sub>2</sub>  
*M<sub>r</sub>* = 389.23  
 Triclinic, *P* $\bar{1}$   
*a* = 6.7109 (10) Å  
*b* = 8.6329 (12) Å  
*c* = 15.833 (2) Å  
 $\alpha$  = 84.144 (3)°  
 $\beta$  = 79.466 (3)°  
 $\gamma$  = 76.252 (3)°  
*V* = 874.3 (2) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.478 Mg m<sup>−3</sup>  
 Mo *K*α radiation  
 Cell parameters from 870 reflections  
 $\theta$  = 2.4–22.2°  
 $\mu$  = 0.39 mm<sup>−1</sup>  
*T* = 293 (2) K  
 Prism, colourless  
 0.42 × 0.10 × 0.10 mm

### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 4127 measured reflections  
 2730 independent reflections

1873 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.027  
 $\theta_{\max}$  = 24.8°  
*h* = −7 → 7  
*k* = −8 → 10  
*l* = −18 → 17

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.060  
*wR* (*F*<sup>2</sup>) = 0.133  
*S* = 1.04  
 2730 reflections  
 235 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 0.3745P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.33 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.35 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å).

C11–C14	1.744 (4)	C7–N1	1.276 (4)
N3–N4	1.404 (4)	C3–Cl2	1.742 (4)
N4–C9	1.280 (4)		

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C10–H10 <i>B</i> ···N1	0.97	2.61	2.853 (5)	94
C1–H1···N1	0.93	2.50	2.792 (4)	99
C18–H18 <i>A</i> ···N4	0.96	2.22	2.754 (4)	114
C7–H7···O1	0.93	2.29	2.949 (4)	127
C12–H12···N1 <sup>i</sup>	0.93	2.90	3.678 (5)	142
C18–H18 <i>C</i> ···O1 <sup>i</sup>	0.96	2.77	3.513 (5)	135

Symmetry code: (i) *x* − 1, *y*, *z*.

The H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C–H distance at 0.93 Å, the methylene C–H distance 0.97 Å and methyl group C–H distance 0.96 Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1997) and *PARST* (Nardelli, 1995).

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