

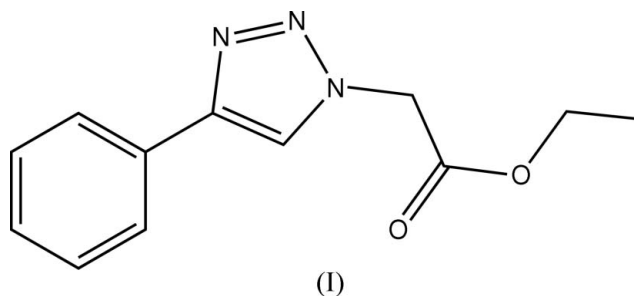
Ethyl 2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)acetateReceived 4 July 2006
Accepted 6 July 2006Xiao-Ru Zhang, Min-Hua Xu,
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Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(C-C) = 0.002$ Å
 R factor = 0.045
 wR factor = 0.116
Data-to-parameter ratio = 14.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.In the molecule of the title compound, $C_{12}H_{13}N_3O_2$, the benzene and triazole rings are each planar and form a dihedral angle of $18.36(9)^\circ$.

Comment

The copper(I)-catalysed union of terminal alkynes and organic azides to give 1,4-disubstituted 1,2,3-triazoles (Rostovtsev *et al.*, 2002) exhibits remarkably broad scope and good selectivity. The best click reaction (Kolb & Sharpless, 2003) has quickly found applications in chemistry, biology and materials science. The title compound, (I), was synthesized in high yield *via* a [3 + 2] cycloaddition reaction between azide and acetylene compounds. The chemical structure of (I), has now been confirmed by single-crystal X-ray diffraction analysis.In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Rings *A* (C1–C6) and *B* (N1–N3/C7/C8) are each planar, and form a dihedral angle of $18.36(9)^\circ$.

Experimental

NaN₃ (7.94 g, 122 mmol), phenylacetylene (21 ml, 190 mmol), CuSO₄·5H₂O (2.35 g, 9.4 mmol) and L-ascorbic acid sodium salt (3.72 g, 187 mmol) were added successively to a solution of 2-ethyl 2-chloroacetate (10 ml, 94 mmol) in 50 ml of DMF/H₂O (1:1). The mixture was stirred at 323 K for 48 h. NH₃·H₂O (25 ml) was then added, the solvent was extracted with ethyl acetate, washed with water, and the organic phase was dried over anhydrous Na₂SO₄. After evaporation, the resulting solid was recrystallized from ethyl acetate/petroleum ether (1:3), yielding the title compound (I) (yield 15.6 g, 72.2%). A solution of (I) in ethyl acetate was allowed to stand at room temperature for 2 d and colorless needle-shaped crystals suitable for X-ray crystallographic analysis were grown by slow evaporation.

Crystal data

$C_{12}H_{13}N_3O_2$
 $M_r = 231.25$
 Triclinic, $P\bar{1}$
 $a = 5.6646$ (15) Å
 $b = 8.644$ (2) Å
 $c = 12.393$ (3) Å
 $\alpha = 85.549$ (4)°
 $\beta = 81.179$ (4)°
 $\gamma = 74.569$ (4)°

$V = 577.6$ (3) Å³
 $Z = 2$
 $D_x = 1.330$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 294$ (2) K
 Needle, colorless
 $0.38 \times 0.19 \times 0.14$ mm

Data collection

Siemens SMART 1000 CCD area-
 detector diffractometer
 ω scans
 Absorption correction: multi-scan
SADABS (Sheldrick, 1996)
 $T_{\min} = 0.965$, $T_{\max} = 0.987$

3338 measured reflections
 2209 independent reflections
 1811 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\text{max}} = 26.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.117$
 $S = 1.05$
 2209 reflections
 154 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.0941P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

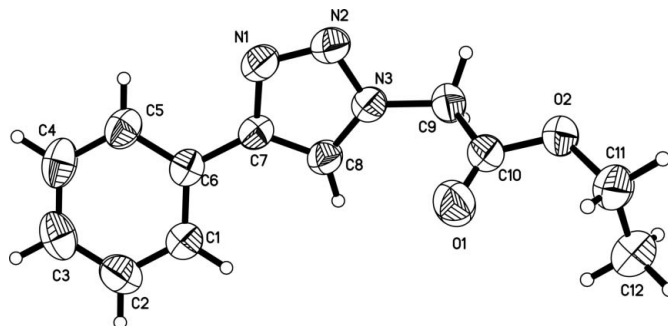


Figure 1

The molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Kolb, H. C. & Sharpless, K. B. (2003). *Drug Discovery Today*, **8**, 1128–1137.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
 Rostovtsev, V. V., Green, L. G., Fokin, V. V. & Sharpless, K. B. (2002). *Angew. Chem. Int. Ed.* **41**, 2596–2599.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.
 Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-Ray Systems Inc., Madison, Wisconsin, USA.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.