

Wen-Qiang Zou,<sup>a,b</sup> Yan Li,<sup>a,b</sup>  
Fa-Kun Zheng,<sup>a\*</sup> Guo-Cong Guo<sup>a</sup>  
and Jin-Shun Huang<sup>a</sup><sup>a</sup>State Key Laboratory of Structural Chemistry,  
Fujian Institute of Research on the Structure of  
Matter, Chinese Academy of Sciences, Fuzhou,  
Fujian 350002, People's Republic of China, and  
<sup>b</sup>Graduate School of Chinese Academy of  
Sciences, Beijing 100039, People's Republic of  
China

Correspondence e-mail: zfk@ms.fjirsm.ac.cn

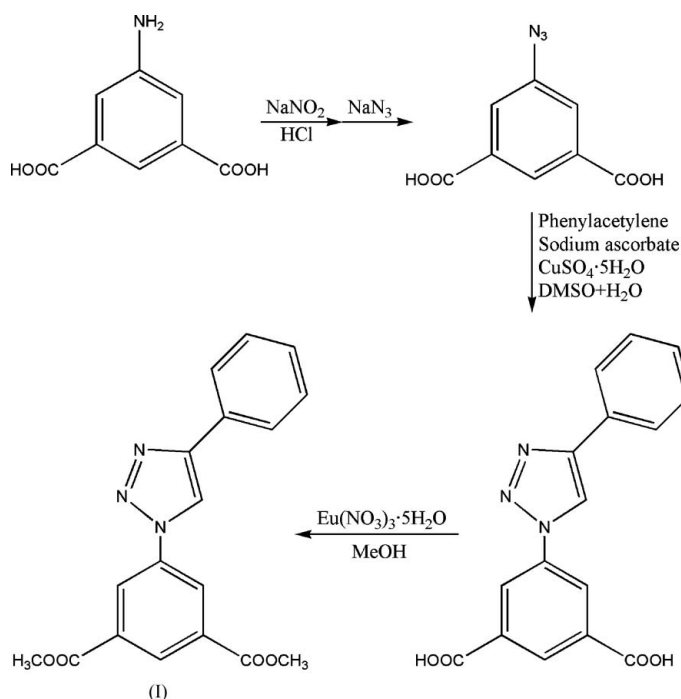
## Key indicators

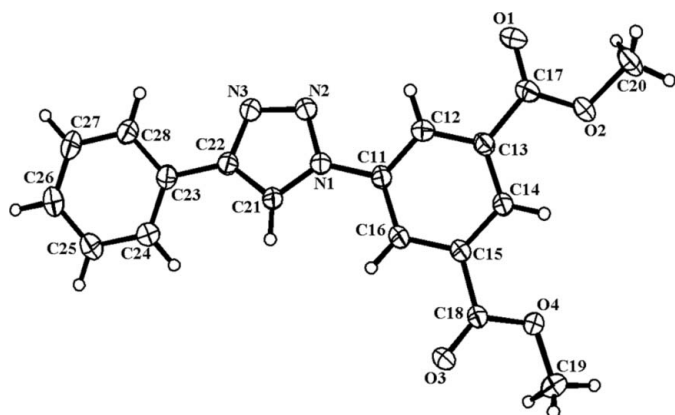
Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.056  
 $wR$  factor = 0.179  
Data-to-parameter ratio = 13.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Dimethyl 5-(4-phenyl-1*H*-1,2,3-triazol-1-yl)-  
benzene-1,3-dicarboxylateThe cycloaddition reaction of 3,5-dicarboxyphenyl azide and  
phenylacetylene, followed by esterification with methanol  
produced the title compound,  $\text{C}_{18}\text{H}_{15}\text{N}_3\text{O}_4$ . The molecular  
skeleton is essentially planar.

Received 22 June 2006

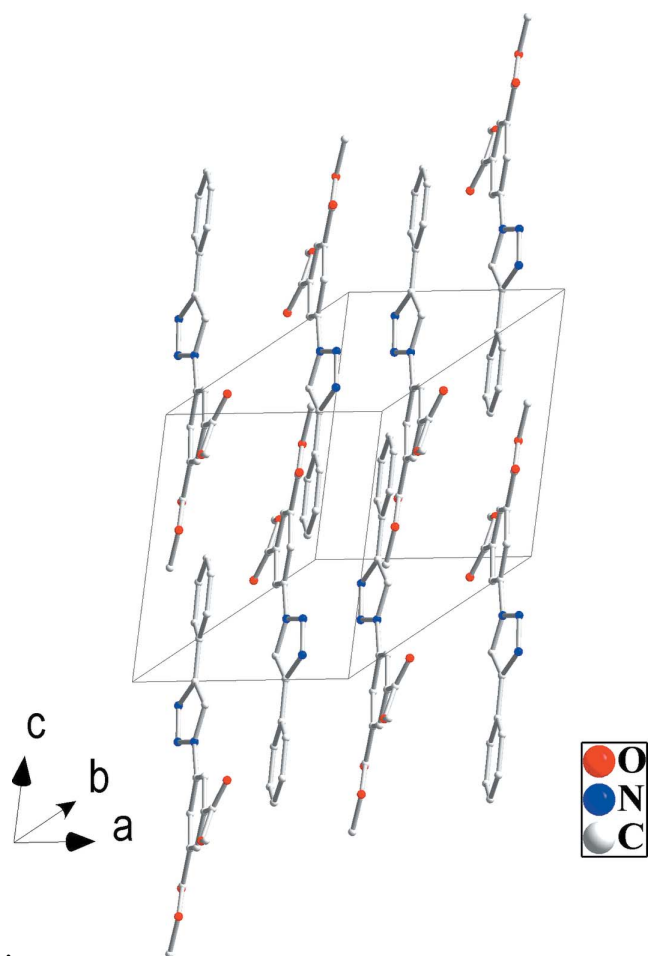
Accepted 25 July 2006

## Comment

Substituted 1,2,3-triazoles can be synthesized by the  
copper(I)-catalysed 1,3-dipolar cycloaddition of terminal  
alkynes to azides (Rostovtsev *et al.*, 2002; Wang *et al.*, 2003;  
Himo *et al.*, 2005; Zhang *et al.*, 2005). Recent interest in  
substituted 1,2,3-triazoles has arisen from their wide range of  
industrial applications, such as agrochemicals, corrosion inhi-  
bitors, dyes, optical brighteners and biologically active agents  
(Orgueira *et al.*, 2005). 1,2,3-Triazole, having strong  $\sigma$ -donor  
and weak  $\pi$ -acceptor properties in the ring, is also a potential  
ligand for building coordination complexes with intriguing  
structures and specific magnetic properties (Bronisz, 2005;  
Devi *et al.*, 2002). Here we describe the synthesis and structure  
of a new substituted 1*H*-1,2,3-triazole, namely dimethyl 5-(4-  
phenyl-1*H*-1,2,3-triazol-1-yl)benzene-1,3-dicarboxylate, (I).The molecular skeleton of (I) is essentially planar (Fig. 1).  
The 1*H*-1,2,3-triazole ring is planar, with a mean deviation of  
0.0011 Å from the plane. The dihedral angle between the  
triazole ring and the C11–C16 benzene ring is 14.34 (17)°, and  
the dihedral angle between the triazole ring and the C23–C28



**Figure 1**  
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.



**Figure 2**  
A packing diagram. H atoms have been omitted for clarity.

phenyl ring is  $14.93(18)^\circ$ . The two benzene rings are approximately coplanar, with a dihedral angle of  $0.87(17)^\circ$ . The bond lengths C21–N1, C22–N3 and N1–N2 (Table 1) are shorter than a C–N single-bond length of  $1.483 \text{ \AA}$  (Banerjee *et al.*, 2002) and an N–N single-bond length of  $1.467 \text{ \AA}$  (Janas & Sobota, 2005), which suggests that the C–N and N–N bonds have partial double-bond character. The bond lengths of N2–N3 and C21–C22 are shorter than those

of N1–N2 and C22–C23, showing some double-bond character.

The packing of the molecules in the crystal structure is shown in Fig. 2. No weak supramolecular interactions such as  $\pi$ – $\pi$  and hydrogen-bonding interactions are found.

## Experimental

The title compound was prepared by the procedure shown in the scheme. 5-Aminoisophthalic acid ( $1.811 \text{ g}$ ,  $10 \text{ mmol}$ ) was dissolved in hot hydrochloric acid [prepared from  $4 \text{ ml}$  concentrated HCl and  $10 \text{ ml}$  water], then  $100 \text{ ml}$  of water was added and the solution was cooled to  $273 \text{ K}$  in an ice-salt bath. A solution of sodium nitrite ( $0.828 \text{ g}$ ,  $12 \text{ mmol}$ ) in water ( $2 \text{ ml}$ ) was added dropwise while stirring for  $10 \text{ min}$ . With vigorous stirring, a solution of sodium azide ( $0.650 \text{ g}$ ,  $10 \text{ mmol}$ ) in water ( $2 \text{ ml}$ ) was added, and the resulting white precipitate was isolated by filtration, washed with water, and dried in air [yield:  $1.935 \text{ g}$ ,  $93.4\%$  (based on 5-aminoisophthalic acid)]. The next step was modified according to the literature (Rostovtsev *et al.*, 2002). 3,5-Dicarboxyphenyl azide ( $1.036 \text{ g}$ ,  $5 \text{ mmol}$ ) and phenylacetylene ( $0.510 \text{ g}$ ,  $5 \text{ mmol}$ ) were dissolved in  $25 \text{ ml}$  of a 4:1 DMSO/water mixture. Sodium ascorbate ( $0.099 \text{ g}$ ,  $0.5 \text{ mmol}$ , in  $0.5 \text{ ml}$  of water) was added, followed by copper(II) sulfate pentahydrate ( $0.015 \text{ g}$ ,  $0.06 \text{ mmol}$ , in  $0.3 \text{ ml}$  of water). The mixture was stirred at  $333 \text{ K}$  for  $24 \text{ h}$ , then poured into  $100 \text{ ml}$  of ice-cold water. The off-white precipitate was isolated by filtration and washed with cold water, then dried in air [yield  $1.381 \text{ g}$ ,  $89.3\%$  (based on 3,5-dicarboxyphenyl azide)]. A mixture of  $\text{Eu}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  ( $0.107 \text{ g}$ ,  $0.25 \text{ mmol}$ ), 1-(3,5-dicarboxyphenyl)-4-phenyl-1H-1,2,3-triazole ( $0.077 \text{ g}$ ,  $0.25 \text{ mmol}$ ) and methanol ( $8 \text{ ml}$ ) was heated in a sealed Teflon-lined stainless steel autoclave at  $453 \text{ K}$  for  $3 \text{ d}$ , then slowly cooled to room temperature. The product was isolated and washed with water, and colorless crystals suitable for X-ray diffraction studies were obtained [yield  $0.0565 \text{ g}$ ,  $67\%$  [based on 1-(3,5-dicarboxyphenyl)-4-phenyl-1H-1,2,3-triazole]].

### Crystal data

$\text{C}_{18}\text{H}_{15}\text{O}_4\text{N}_3$	$V = 794.8(4) \text{ \AA}^3$
$M_r = 337.33$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.410 \text{ Mg m}^{-3}$
$a = 8.758(3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.718(2) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 10.197(2) \text{ \AA}$	$T = 293(2) \text{ K}$
$\alpha = 80.696(19)^\circ$	Prism, colorless
$\beta = 69.50(2)^\circ$	$0.18 \times 0.16 \times 0.10 \text{ mm}$
$\gamma = 79.47(2)^\circ$	

### Data collection

Rigaku AFC-7R diffractometer	2132 reflections with $I > 2\sigma(I)$
$\omega$ – $2\theta$ scans	$R_{\text{int}} = 0.016$
Absorption correction: $\psi$ scan	$\theta_{\text{max}} = 25.5^\circ$
( <i>ABSCOR</i> ; Rigaku, 2002)	3 standard reflections
$T_{\text{min}} = 0.986$ , $T_{\text{max}} = 0.999$	every 200 reflections
3217 measured reflections	intensity decay: none
2962 independent reflections	

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.12P)^2 + 0.0605P]$
$R[F^2 > 2\sigma(F^2)] = 0.056$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.179$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
2962 reflections	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
226 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters (Å, °).

C11—N1	1.428 (3)	C20—O2	1.441 (3)
C13—C17	1.497 (3)	C21—N1	1.348 (3)
C15—C18	1.487 (3)	C21—C22	1.363 (3)
C17—O1	1.188 (3)	C22—N3	1.363 (3)
C17—O2	1.323 (3)	C22—C23	1.470 (3)
C18—O3	1.205 (3)	N1—N2	1.352 (3)
C18—O4	1.328 (3)	N2—N3	1.301 (3)
C19—O4	1.450 (3)		
O1—C17—O2	123.6 (2)	C21—C22—C23	130.6 (2)
O1—C17—C13	124.0 (2)	C21—N1—N2	110.19 (19)
O2—C17—C13	112.3 (2)	C21—N1—C11	130.1 (2)
O3—C18—O4	123.8 (2)	N2—N1—C11	119.72 (19)
O3—C18—C15	124.1 (2)	N3—N2—N1	107.04 (19)
O4—C18—C15	112.07 (19)	N2—N3—C22	109.75 (19)
N1—C21—C22	105.5 (2)	C17—O2—C20	116.9 (2)
N3—C22—C21	107.5 (2)	C18—O4—C19	116.7 (2)
N3—C22—C23	121.9 (2)		

H atoms were placed in calculated positions, with C—H distances of 0.95 Å, and included in the refinement in a riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *WinAFC* (Rigaku, 2002); cell refinement: *WinAFC*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge the financial support of the NSF for Distinguished Young Scientists of China (20425104) and the NSF of Fujian Province (Nos. A0420002 and 2005I017).

## References

- Banerjee, S., Mukherjee, A. K., Goswami, D., De, A. U. & Helliwell, M. (2002). *Cryst. Res. Technol.* **37**, 309–317.
- Brandenburg, K. (1999). *DIAMOND*. Version 2.1c. Crystal Impact GbR, Bonn, Germany.
- Bronisz, R. (2005). *Inorg. Chem.* **44**, 4463–4465.
- Devi, R. N., Rabu, P., Golub, V. O., O'Connor, C. J. & Zubieta, J. (2002). *Solid State Sci.* **4**, 1095–1102.
- Himo, F., Lovell, T., Hilgraf, R., Rostovtsev, V. V., Noodleman, L., Sharpless, K. B. & Fokin, V. V. (2005). *J. Am. Chem. Soc.* **127**, 210–216.
- Janas, Z. & Sobota, P. (2005). *Coord. Chem. Rev.* **249**, 2144–2155.
- Orgueira, H. A., Fokas, D., Isome, Y., Chan, P. C.-M. & Baldino, C. M. (2005). *Tetrahedron Lett.* **46**, 2911–2914.
- Rigaku (2002). *WinAFC Diffractometer Control Software*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MS (2004). *CrystalStructure*. Version 3.1.0. Rigaku/MS, The Woodlands, Texas, USA.
- Rostovtsev, V. V., Green, L. C., Fokin, V. V. & Sharpless, K. B. (2002). *Angew. Chem. Int. Ed.* **41**, 2596–2599.
- Siemens (1994). *SHELXTL*. Release 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Wang, Q., Chan, T. R., Hilgraf, R., Fokin, V. V., Sharpless, K. B. & Finn, M. G. (2003). *J. Am. Chem. Soc.* **125**, 3192–3193.
- Zhang, L., Chen, X. G., Xue, P., Sun, H. H. Y., Williams, I. D., Sharpless, K. B., Fokin, V. V. & Jia, G. C. (2005). *J. Am. Chem. Soc.* **127**, 15998–15999.