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5-{[4-(4-Bromophenyl)thiazol-2-yl]amino-methylene}-2,2-dimethyl-1,3-dioxane-4,6-dione

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.088
Data-to-parameter ratio = 12.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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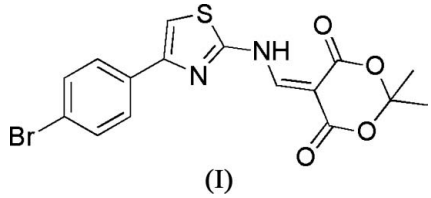
In the title compound, $C_{16}H_{13}BrN_2O_4S$, the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation. The amino H atom makes an intramolecular contact to a carbonyl O atom, forming a six-membered ring.

Comment

Thiazoles and their derivatives are found to be associated with various biological activities such antibacterial and anti-inflammatory (Holla *et al.*, 2003). In addition, phenylthiazole derivatives have been investigated as inhibitors of kynurenine 3-hydroxylase (Röver *et al.*, 1997). Prompted by these reports and in continuation of our search for bioactive molecules from Meldrum's acid derivatives (Joussef *et al.*, 2005*a*,*b*; da Silva *et al.*, 2005*a*,*b*; da Silva, Joussef, Foro & Schmidt, 2006; da Silva, Joussef, Andrighetti-Fröhner *et al.* 2006), the structure of the title compound, (I), has been determined and the results are presented here (Fig. 1).



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation with the C13 atom at the flap position. The torsion angle C11—C10—N2—C1 of −176.8 (2)° and the C10—N2 and C10—C11 bond distances (Table 1) indicate electron delocalization. The delocalization of the N atom lone pair into the Meldrum's acid ring may be favoured in the

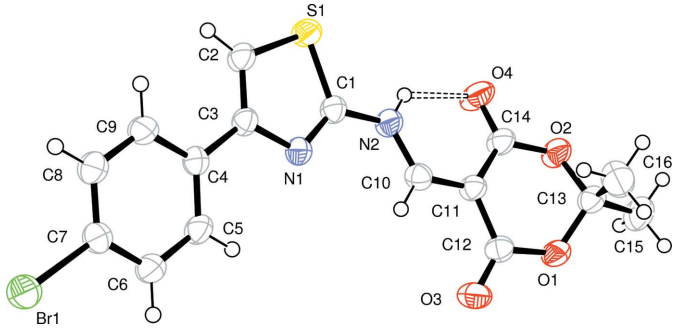


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids (arbitrary spheres for H atoms). Dashed lines indicate a hydrogen bond.

direction of one of the two available carbonyl groups C12═O3 and C14═O4 (Blake *et al.*, 2003). The thiazole ring and the benzene ring are slightly twisted with respect to each other, with a dihedral angle of 9.3 (1)° between the mean planes. The amino H atom is intramolecularly hydrogen bonded to O4 (Table 2), forming a six-membered ring.

Experimental

The title compound was prepared according to the literature procedure (Cassis *et al.*, 1985) and was recrystallized from methanol.

Crystal data

$C_{16}H_{13}BrN_2O_4S$
M_r = 409.25
Triclinic, $P\overline{1}$
a = 7.8262 (6) Å
b = 9.4869 (7) Å
c = 11.8847 (7) Å
α = 70.969 (5)°
β = 79.213 (6)°
γ = 77.059 (6)°
V = 806.72 (10) Å^3
Z = 2
D_x = 1.685 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 4.90 mm^{-1}
T = 299 (2) K
Thick needle, yellow
0.60 × 0.25 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.198, T_{max} = 0.479
3288 measured reflections
2774 independent reflections
2603 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 66.9°
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.088
S = 1.14
2774 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.4106P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0133 (7)

Table 1
Selected bond lengths (Å).

C10—N2	1.331 (3)	C10—C11	1.370 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···O4	0.86	2.06	2.704 (3)	131

All H atoms were positioned with idealized geometry [N—H = 0.86 Å, and C—H = 0.93 (aromatic) and 0.96 Å (methyl)], and were refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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References

Blake, A. J., McNab, H. & Withell, K. (2003). *Acta Cryst.* E**59**, o841–o842.
Cassis, R., Tapia, R. & Valderrama, J. A. (1985). *Synth. Commun.* **15**, 125–129.
Enraf–Nonius (1996). *CAD-4-PC*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.
Holla, B. S., Malini, K. V., Rao, B. S., Sarojini, B. K. & Kumari, N. S. (2003). *Eur. J. Med. Chem.* **38**, 313–318.
Joussef, A. C., da Silva, L. E., Bortoluzzi, A. J. & Foro, S. (2005*a*). *Acta Cryst.* E**61**, o2642–o2643.
Joussef, A. C., da Silva, L. E., Bortoluzzi, A. J. & Foro, S. (2005*b*). *Acta Cryst.* E**61**, o2873–o2874.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Röver, S., Cesura, A. M., Huguenin, P., Kettler, R. & Szente, A. (1997). *J. Med. Chem.* **40**, 4378–4385.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Silva, L. E. da, Joussef, A. C., Andrighetti-Fröhner, C. R., Nunes, R. J. & Bortoluzzi, A. J. (2006). *Acta Cryst.* E**62**, o1062–o1063.
Silva, L. E. da, Joussef, A. C., Foro, S. & Schmidt, B. (2006). *Acta Cryst.* E**62**, o742–o743.
Silva, L. E. da, Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*a*). *Acta Cryst.* E**61**, o4121–o4122.
Silva, L. E. da, Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*b*). *Acta Cryst.* E**61**, o4252–o4253.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1987). *REDU4*. Version 6.2c. Stoe & Cie GmbH, Darmstadt, Germany.