Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2-Dimethyl-5-[(1,2,4-triazin-3-ylamino)-methylene]-1,3-dioxane-4,6-dione

Antonio C. Joussef,[a] Luiz Everson da Silva,[a] Adailton J. Bortoluzzi[a] and Sabine Foro[b]*

[a]Departmento de Química–UFSC, 88040-900 Florianópolis, SC, Brazil, and [b]Clemens–Schöpf-Institut für Organische Chemie und Biochemie, Technische Universität Darmstadt, Petersenstrasse 22, D-64287 Darmstadt, Germany

Correspondence e-mail: foro@tu-darmstadt.de

Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ() = 0.000 Å
Disorder in main residue
R factor = 0.055
wR factor = 0.161
Data-to-parameter ratio = 8.4

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

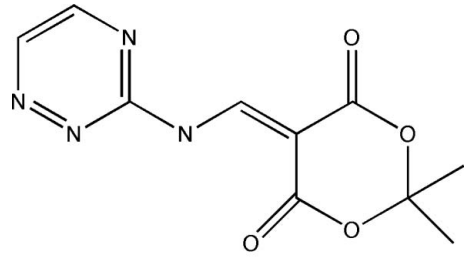
© 2005 International Union of Crystallography
Printed in Great Britain – all rights reserved

Received 14 June 2005
Accepted 3 August 2005
Online 12 August 2005

In the title compound, $C_{10}H_{10}N_4O_4$, the triazine ring is nearly planar. The 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The molecule is disordered and the structure has been refined using a split model.

Comment

Various derivatives of Meldrum's acid have been investigated by X-ray methods (Gould *et al.*, 1998; Blake *et al.*, 1997, Blake, Gould *et al.*, 1994; Blake & McNab, 1995; Blake, McNab & Morrow, 1994). As part of a continuing study of the 5-methoxymethylene Meldrum's acid derivatives as potential antiviral, antitrypanosomal and leishmanicidal, we report the structure of the title compound, (I).



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation and the triazine ring is nearly planar. The molecule is disordered and the structure has therefore been refined using a split model. Because of the disorder some of the bond lengths and angles are unusual. The disorder components with displacement ellipsoids are shown separately in Figs. 1 and 2. The total disordered molecular structure is shown in Fig. 3. Details of the intra- and intermolecular hydrogen bonds are given in Table 1.

Experimental

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

Crystal data

$C_{10}H_{10}N_4O_4$
M_r = 250.22
Monoclinic, $P2_1/n$
a = 6.379 (5) Å
b = 18.239 (5) Å
c = 10.101 (5) Å
β = 107.331 (5)°
V = 1121.9 (11) Å^3
Z = 4
D_x = 1.481 Mg m^{-3}
D_m = ? Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 4.9–15.4°
μ = 0.12 mm^{-1}
T = 193 (2) K
Prism, colorless
0.50 × 0.20 × 0.16 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2028 measured reflections
1924 independent reflections
1191 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 25.1^\circ$
$h = -7 \rightarrow 7$
$k = -21 \rightarrow 0$
$l = -12 \rightarrow 0$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.161$
$S = 1.05$
1924 reflections
230 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.8549P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N7*A*—H7*A*···O14*A*	0.86	2.08	2.724 (5)	131
N7*B*—H7*B*···O10*B*	0.86	2.13	2.777 (19)	132
C4*B*—H4*B*···O10*A*^{i}	0.93	2.04	2.842 (14)	143
C4*B*—H4*B*···O10*B*^{i}	0.93	1.90	2.802 (19)	162

Symmetry code: (i) $-x+2, -y+1, -z+2$.

The structure is disordered and was refined with a split model. Refinement was performed with restraints for both components. The corresponding site-occupation factors were refined but later fixed to 0.80 and 0.20. All non-H atoms of the minor occupied component were refined isotropically and a common variable was used for the isotropic displacement factor of the ring N1*B*–C6*B*. The triazine ring is additionally disordered, such that atoms N2*A* and C4*A*, as well as N2*B* and C4*B*, occupy the same position. Therefore, these atoms were refined with site-occupation factors 0.5:0.5. All H atoms were positioned with idealized geometry and were refined with isotropic displacement parameters (set to 1.2 times U_{eq} of the parent atom) using a riding model with C—H = 0.93 Å (0.96 Å for methyl groups) and N—H = 0.86 Å.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4/PC Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

References

Blake, A. J., Gould, R. O., Irving, I., McNab, H. & Morrow, M. (1994). *Acta Cryst.* C**50**, 1935–1938.
Blake, A. J. & McNab, H. (1995). *Acta Cryst.* C**51**, 527–529.
Blake, A. J., McNab, H. & Morrow, M. (1994). *Acta Cryst.* C**50**, 1716–1717.

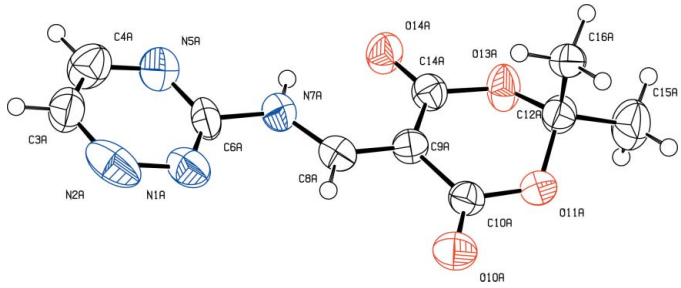


Figure 1
The molecular structure of the major component (site-occupation factor = 0.80), with the atom labelling, and with displacement ellipsoids drawn at the 50% probability level.

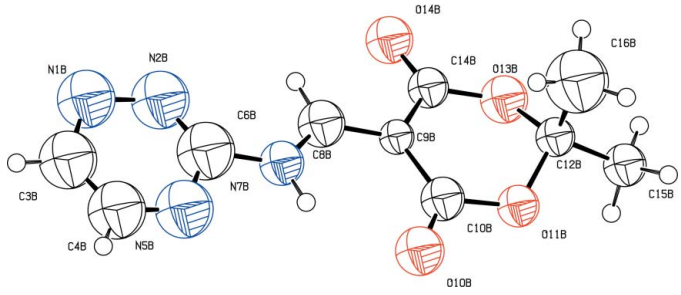


Figure 2
The molecular structure of the minor component (site-occupation factor = 0.20), with displacement spheres drawn at the 50% probability level. (Please note: this fragment was refined isotropically.)

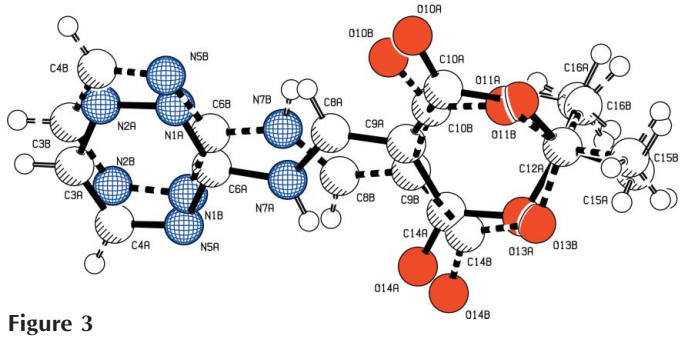


Figure 3
The disordered molecular structure of the title compound, with the atom labelling (the bonds of the minor component are drawn with dashed lines).

Cassis, R., Tapia, R. & Valderrama, J. A. (1985). *Synth. Commun.* **15**, 125–128.
Enraf–Nonius (1993). *CAD-4/PC Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.
Gould, R. O., Harris, S. G., McNab, H., Parsons, S. & Withell, K. (1998). *Acta Cryst.* C**54**, 234–236.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.