Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-[(2-Bromophenylamino)methylene]-2,2-dimethyl-1,3-dioxane-4,6-dione

Luiz Everson da Silva,[a] Antonio Carlos Joussef,[b]* Ricardo José Nunes,[b] Carla Regina Andrighetti-Fröhner[b] and Adailton José Bortoluzzi[b]

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

Correspondence e-mail: luiz_everson@yahoo.de

Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.004 Å
R factor = 0.030
wR factor = 0.073
Data-to-parameter ratio = 13.1

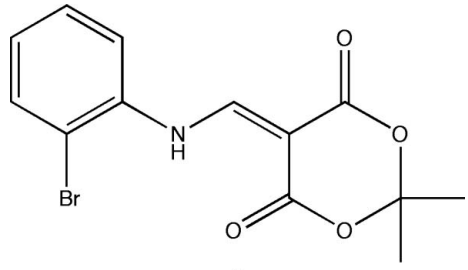
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 November 2005
Accepted 9 November 2005
Online 23 November 2005

In the title compound, $C_{13}H_{12}BrNO_4$, the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The molecules are linked by C—H···O hydrogen bonds. The amino H atom has one intramolecular contact to a carbonyl O atom, forming a six-membered ring.

Comment

Recently, we reported the structure of two arylaminomethylene Meldrum's acid derivatives (Joussef *et al.*, 2005*a*,*b*). As part of a continuing study of the conformational analysis in the solid state of Meldrum's acid derivatives, we report here the structure of the title compound, (I).



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The dihedral angle C6—N7—C8—C9 is 170.5 (3)° and the distances C6—N7 and C8—C9 indicate delocalization. The amino H atom has one intramolecular contact to atom O10, with an H···O distance of 2.12 (3) Å, forming a six-membered ring. The delocalization of the N-atom lone pair into the Meldrum's acid ring may be favoured in the direction of one of the two carbonyl groups (Blake *et al.*, 2003). One intramolecular contact between the amino H atom and Br, with a distance of 2.62 (3) Å, is also observed. The molecular packing of (I) is stabilized by C—H···O hydrogen

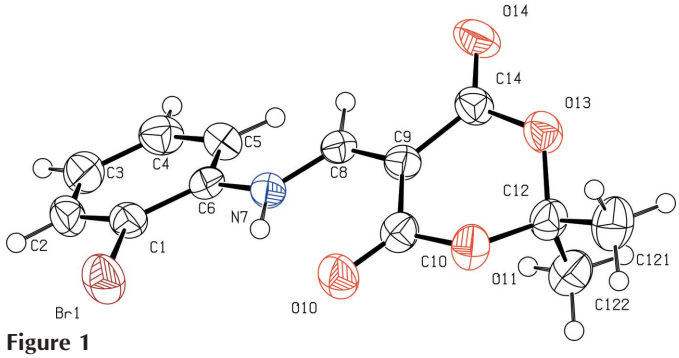


Figure 1
The molecular structure of (I), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

bonds. The bond lengths and angles are in good agreement with values retrieved from the Cambridge Structural Database (Version 5.25; Allen, 2002). Details of the hydrogen bonding are given in Table 1.

Experimental

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

Crystal data

$C_{13}H_{12}BrNO_4$
M_r = 326.15
Triclinic, $P\overline{1}$
a = 6.960 (5) Å
b = 9.298 (5) Å
c = 10.424 (5) Å
α = 95.570 (5)°
β = 101.043 (5)°
γ = 99.508 (5)°
V = 647.2 (7) Å^3
Z = 2
D_x = 1.674 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 4.1–16.9°
μ = 3.19 mm^{-1}
T = 193 (2) K
Irregula block, colorless
0.43 × 0.26 × 0.16 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.417, T_{max} = 0.601
2451 measured reflections
2312 independent reflections
1724 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.1°
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 0$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.073
S = 1.06
2312 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0298P)^2 + 0.241P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.024 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7···O10	0.83 (3)	2.12 (3)	2.734 (3)	130 (3)
C2—H2···O14^{i}	0.93	2.46	3.266 (4)	146

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

The amino H atom was located in a difference map and was refined freely. C-bound H atoms were positioned with idealized geometry and were refined as riding, with C—H = 0.93 Å (0.96 Å for methyl groups) and U^{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

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*.

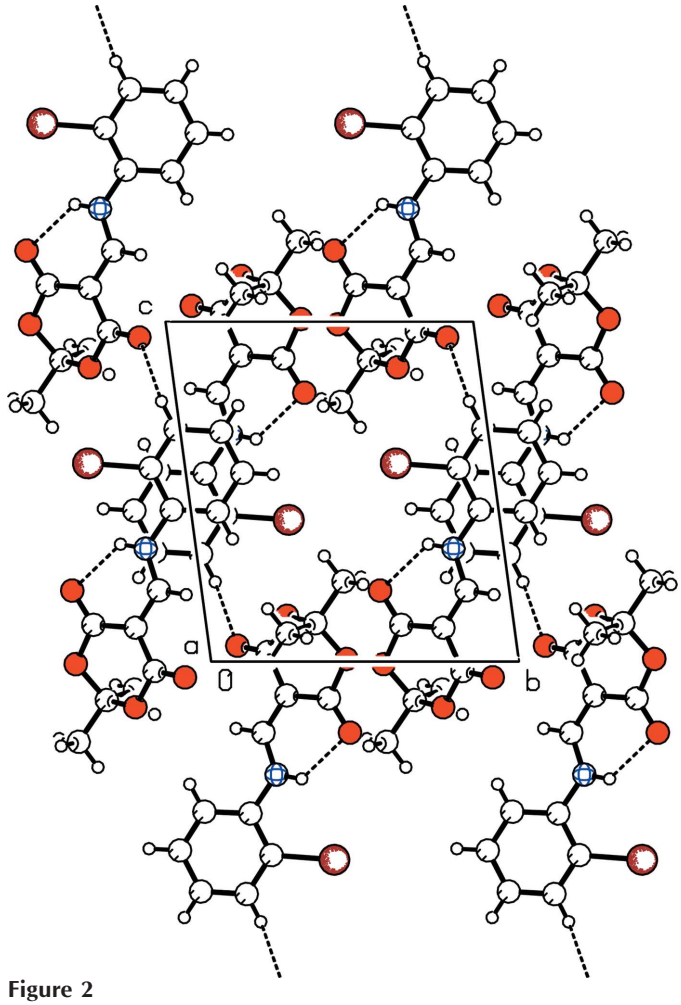


Figure 2
The molecular packing of (I), viewed along the *a* axis, with hydrogen bonding shown as dashed lines.

The authors thank Sabine Foro from TU–Darmstadt, Germany, and Claudia M. O. Simoes from Federal University of Santa Catarina State, Brazil, for their help and advice.

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