Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(2,6-Dioxo-3,4,5,6-tetrahydro-2*H*-pyran-3-yl)-2,3-dihydro-1*H*-isoindole-1,3-dione

Shao-Song Qian,[a]* Hong-You Cui[b] and Bo-Sheng Zhao[a]

[a]School of Life Sciences, ShanDong University of Technology, ZiBo 255049, People's Republic of China, and [b]School of Chemical Engineering, ShanDong University of Technology, ZiBo 255049, People's Republic of China

Correspondence e-mail: njuqss@tom.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.164
Data-to-parameter ratio = 13.2

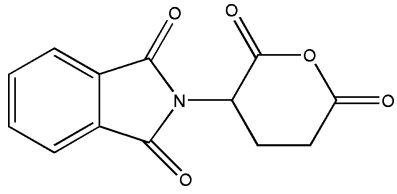
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 March 2006
Accepted 3 May 2006

The title compound, $C_{13}H_9NO_5$, contains two molecules in the asymmetric unit, in which the 3-substituted dihydropyran-2,6-dione is not planar.

Comment

The title compound, (I), has attracted attention as an intermediate for the synthesis of glutamine and of γ-dipeptides of glutamic acid (King & Kidd, 1949; Kokai, 1981). More recently, L-theanine was reported as an antitumour agent (Sadzuka *et al.*, 2000, 2002). The title compound has been used as an intermediate for the manufacture of L-theanine (Qian *et al.*, 2005). Here, we report its crystal structure.



(I)

In the crystal structure of (I) which has two molecules in the asymmetric unit (Fig. 1), the 3-substituted-dihydro-pyran-2,6-dione is not planar, which is as expected. The C—N bond lengths are in the range 1.397 (3)–1.452 (2) Å, which is between the normal value of a C—N single bond and a C═N double bond, due to conjugation effects. All other bond lengths are within normal ranges (Allen *et al.*, 1987).

Experimental

N-Phthaloyl-L-glutamic acid was prepared according to the literature method of Nefkens *et al.* (1960). L-Glutamic acid (14.7 g, 0.1 mol) was reacted with *N*-carboethoxyphthalimide (21.9 g, 0.1 mol) in water (200 ml) with sodium carbonate (23.3 g, 0.21 mol) to give *N*-phthaloyl-L-glutamic acid (yield 19.9 g, 72%). The title compound, (I) (11.9 g) was obtained by reacting *N*-phthaloyl-L-glutamic acid (13.9 g, 0.05 mol) in acetic anhydride (30 ml) under reflux for 20 min. A small quantity (0.1 g) of (I) was dissolved in acetic acid (20 ml) and single crystals suitable for X-ray diffraction were obtained by spontaneous evaporation of the solvent.

Crystal data

$C_{13}H_9NO_5$
M_r = 259.21
Monoclinic, $P2_1/n$
a = 11.868 (2) Å
b = 10.254 (2) Å
c = 19.833 (4) Å
β = 106.51 (3)°
V = 2314.1 (8) Å^3
Z = 8
D_x = 1.488 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
Prism, colourless
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.955, T_{max} = 0.966
4544 measured reflections

4544 independent reflections
3113 reflections with $I > 2\sigma(I)$
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.164
S = 1.07
4544 reflections
343 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

All H atoms attached to C atoms were placed in geometric positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å. They were treated as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

This project was sponsored by the State Innovation Foundation (People's Republic of China).

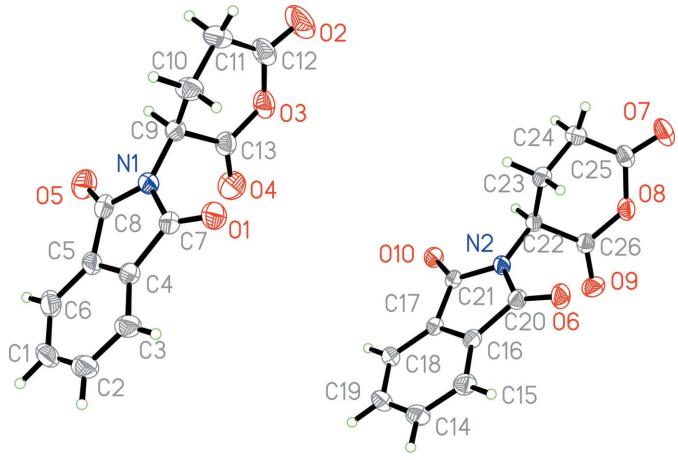


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

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.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.

King, F. E. & Kidd, D. A. A. (1949). *J. Chem. Soc.* pp. 3315–3319.

Kokai, T. K. (1981). Jpn Patent JP 56097260.

Nefkens, G. H. L., Tesser, G. I. & Nivard, R. J. F. (1960). *Helv. Chim. Acta*, **79**, 688–698.

Qian, S. S., Chen, R. & Liu, Y. (2005). *JingXi HuaGong* 22, 845–847. (In Chinese.)

Sadzuka, Y., Sugiyama, T. & Sonobe, T. (2000). *Cancer Lett.* **158**, 119–124.

Sadzuka, Y., Yamashita, Y., Kishmoto, S., Fukushima, S., Takeuchi, Y. & Sonobe, T. (2002). *Pharm. Soc. Jpn*, **122**, 995–999.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.