Refinement was carried out based on all independent reflections. The threshold $I > 2\sigma(I)$ was used only for calculation of the R factor. The hydroxyl H atoms were located from difference syntheses and only displacement parameters were refined, assuming ideal bond lengths (O—H 0.96 Å).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku Corporation, 1998). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1531). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1993). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation & Rigaku Corporation (1998). *TEXSAN. Single Crystal Structure Analysis Software*. Version 1.9. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA, and Rigaku Corporation, 3-9-12 Akishima, Tokyo, Japan.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Reddy, D. S., Ovchinnikov, Y. E., Shishkin, O. V., Struchkov, Y. T. & Desiraju, G. R. (1996). *J. Am. Chem. Soc.* **118**, 4085–4089.

Acta Cryst. (1999). **C55**, 594–596

N-Propylimidazolidinetrione and *N*-methyl-*N*′-phenylethylimidazolidinetrione

Ryouko Yoshihara,[a] Hiroyuki Hosomi,[a] Hiromu Aoyama[b] and Shigeru Ohba[a]

[a]*Department of Chemistry, Faculty of Science and Technology, Keio University, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan, and* [b]*Department of Material Chemistry, Faculty of Textile Science and Technology, Shinsyu University, Tokida 3-15-1, Ueda 386-0081, Japan. E-mail: ohba@chem.keio.ac.jp*

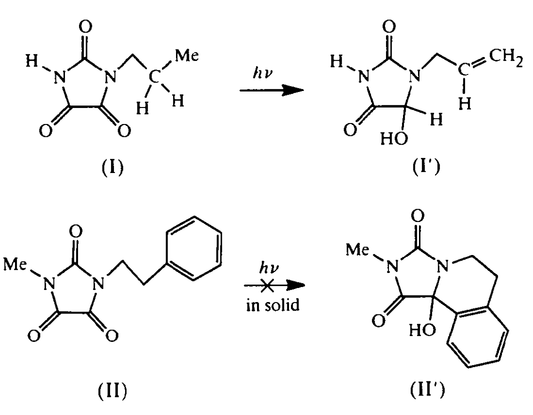
(*Received 1 September 1998; accepted 15 December 1998*)

Abstract

In crystals of *N*-propylimidazolidinetrione, $C_6H_8N_2O_3$, (I), and *N*-methyl-*N*′-phenylethylimidazolidinetrione, $C_{12}H_{12}N_2O_3$, (II), the *N*-alkyl side chains have *trans* conformations. Photoirradiation of (I) gives the *N*-allyl derivative both in solution and in the solid state. (II) is photoreactive, yielding a tricyclic compound in solution, but not in the solid state. The photostability of (II) in the crystalline state may be due to the reaction cavity which does not allow the *N*′-phenylethyl moiety to make a large conformational change for cyclization.

Comment

The photoreactivities of imidazolidinetriones in solution vary with the substituents on the N atoms (Aoyama *et al.*, 1984). An intramolecular disproportionation reaction occurs for (I) and a cyclization reactions occurs for (II). The intermediate of the reactions seems to be the 1,4-biradical formed by γ-hydrogen abstraction by the α-dicarbonyl group (Aoyama *et al.*, 1984). Recently, the photoreactivities in the solid state have also been investigated for these compounds by one of the authors (HA). The yield of (I′) was *ca* 30% after irradiation for 12 h with a 1000 W high-pressure Hg lamp, but there was no yield of (II′). The yields of the photoproducts were estimated *via* their ^{1}H NMR spectra. The crystal structure analyses have been carried out to investigate the geometrical factors which might determine the reactivity in the solid state. The distances between the carbonyl oxygen, O3, and one of the γ-H atoms bonded to C10 are 3.42 (4) Å in (I), and 3.17 Å (no standard uncertainty due to a riding model) in (II). The C8=O3···H angles are 74.6 (7) and 76.4°, respectively. Therefore, the conformations in (I) and (II) are both suitable for intramolecular hydrogen abstraction. It is supposed that the reaction of (I) takes place with little structural change, but that of (II) does not because a large conformational change of the ethylphenyl group is not allowed in the crystal. The C6=O1 bond length in (I) is 0.022 (4) Å longer than that in (II). This elongation of the carbonyl bond is attributed to the hydrogen bonding of O1 in (I), which does not occur in (II). Similar effects were reported for imidazolidinetrione (Weber & Craven, 1987).



Prismatic crystals of (I) have normal hardness, which may be due to the intermolecular cyclic N—H···O hydrogen bonds. Thin plate-like crystals of (II) are very soft and easily curved. The crystallinity of *N*-isobutyl-*N*′-methylimidazolidinetrione, (III), is also poor and the crystals sublime in air. Crystal data for (III): $C_7H_{12}N_2O_3$, M_r = 172.18, monoclinic, $P2_1/a$, a = 8.569 (6), b = 5.728 (5), c = 19.601 (5) Å, β = 101.76 (4)°, V = 941.9 (9) Å^3, Z = 4.

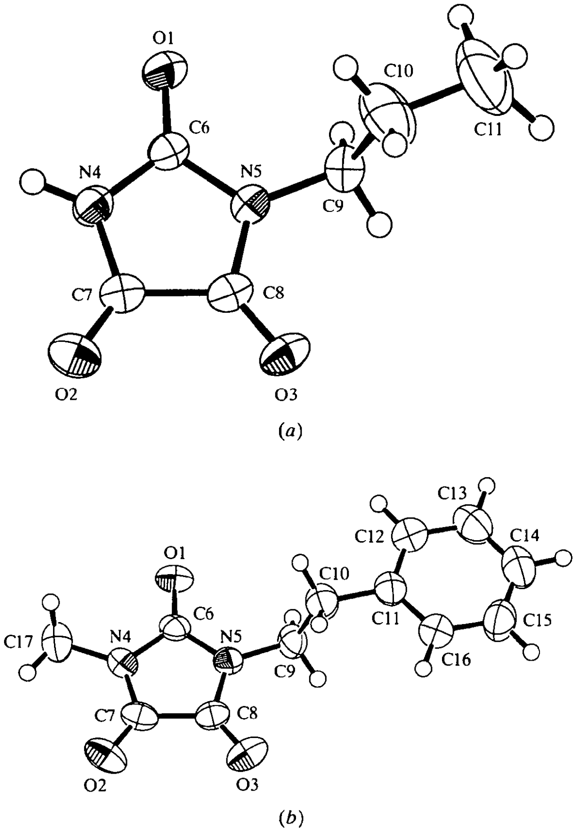


Fig. 1. The molecular structures of (*a*) (I) and (*b*) (II). Displacement ellipsoids are plotted at the 50% probability level. H atoms are shown as spheres of an arbitrary radius.

Experimental

The crystals of compounds (I) and (II) were prepared by a literature method (Larson *et al.*, 1973) and were grown by slow evaporation of methanol solutions.

Compound (I)

Crystal data

$C_6H_8N_2O_3$
M_r = 156.14
Monoclinic
$P2_1/n$
a = 11.503 (4) Å
b = 4.975 (2) Å
c = 13.304 (3) Å
β = 99.86 (2)°
V = 750.2 (4) Å^3
Z = 4
D_x = 1.382 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 14.6–14.9°
μ = 0.112 mm^{-1}
T = 296 K
Prism
0.6 × 0.2 × 0.2 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.946, $T_{\max}$ = 0.978
2829 measured reflections
2715 independent reflections

1549 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
$\theta_{\max}$ = 32.5°
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 7$
$l = -20 \rightarrow 20$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F
R = 0.047
wR = 0.087
S = 1.07
2715 reflections
132 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o) + 0.00226|F_o|^2]$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.21 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) for (I)*

O1—C6	1.208 (2)	N5—C8	1.372 (2)
O2—C7	1.200 (2)	N5—C9	1.459 (2)
O3—C8	1.205 (2)	C7—C8	1.536 (2)
N4—C6	1.376 (2)	C9—C10	1.509 (3)
N4—C7	1.374 (2)	C10—C11	1.505 (4)
N5—C6	1.390 (2)		
C6—N4—C7	111.7 (1)	N4—C7—C8	104.4 (1)
C6—N5—C8	111.0 (1)	N5—C8—C7	105.1 (1)
N4—C6—N5	107.8 (1)		
N5—C9—C10—C11	−178.9 (3)	C6—N5—C9—C10	88.3 (2)

Table 2. *Hydrogen-bonding geometry (Å, °) for (I)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O1$^{\text{i}}$	0.92 (2)	1.97 (2)	2.881 (2)	170 (2)

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

Compound (II)

Crystal data

$C_{12}H_{12}N_2O_3$
M_r = 232.24

Mo $K\alpha$ radiation
λ = 0.71073 Å

Monoclinic
$C2/c$
$a = 49.87$ (2) Å
$b = 5.406$ (3) Å
$c = 8.471$ (3) Å
$\beta = 88.66$ (3)°
$V = 2282.9$ (17) Å^3
$Z = 8$
$D_x = 1.351$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 11.8$–14.8°
$\mu = 0.099$ mm^{-1}
$T = 296$ K
Plate
$0.3 \times 0.3 \times 0.1$ mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.960$, $T_{\max} = 0.990$
2667 measured reflections
2638 independent reflections

1124 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.055$
$\theta_{\max} = 27.5$°
$h = 0 \rightarrow 64$
$k = 0 \rightarrow 7$
$l = -11 \rightarrow 11$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.052$
$wR = 0.116$
$S = 1.10$
2638 reflections
154 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o) + 0.00265|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) for (II)*

O1—C6	1.186 (4)	N5—C6	1.397 (4)
O2—C7	1.200 (4)	N5—C8	1.367 (4)
O3—C8	1.204 (4)	N5—C9	1.457 (4)
N4—C6	1.391 (4)	C7—C8	1.539 (5)
N4—C7	1.354 (4)	C9—C10	1.510 (4)
N4—C17	1.461 (4)	C10—C11	1.509 (4)
C6—N4—C7	112.2 (3)	N5—C8—C7	104.5 (3)
C6—N5—C8	111.7 (2)	N5—C9—C10	112.7 (3)
N4—C6—N5	106.5 (3)	C9—C10—C11	111.4 (3)
N4—C7—C8	105.1 (3)		
N5—C9—C10—C11	173.8 (3)	C6—N5—C9—C10	94.1 (4)

In (I), all H atoms were located from difference syntheses. In (II), all H-atom positions were calculated geometrically with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(parent atom). A riding model was used in their refinement (C—H 0.96 Å).

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software* for (I); *MSC/AFC Diffractometer Control Software* for (II). For both compounds, data reduction: *TEXSAN* (Molecular Structure Corporation, 1998); program(s) used to solve structures: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structures: *TEXSAN*; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1052). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435–436.

Aoyama, H., Ohnota, M., Sakamoto, M. & Omote, Y. (1984). *Tetrahedron Lett.* **25**, 3327–3330.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Larson, D. B., Arnet, J. F. & McGlynn, S. P. (1973). *J. Am. Chem. Soc.* **95**, 6928–6935.

Molecular Structure Corporation (1993). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1998). *TEXSAN. Single Crystal Structure Analysis Software*. Version 1.9. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Weber, H.-P. & Craven, B. M. (1987). *Acta Cryst.* B**43**, 202–209.

Acta Cryst. (1999). C**55**, 596–599

3,3,6,6-Tetramethyl-9-(3-nitrophenyl)-3,4,5,6,9,10-hexahydroacridine-1,8(2*H*,7*H*)-dione

HÉCTOR NOVOA DE ARMAS,[a]† NORBERT M. BLATON,[b] OSWALD M. PEETERS,[b] CAMIEL J. DE RANTER,[b] MARGARITA SUÁREZ NAVARRO,[c] YAMILA VERDECIA REYES,[c] ESTAEL OCHOA RODRÍGUEZ[c] AND ESPERANZA SALFRÁN[c]

[a]*Centro de Química Farmacéutica, Departamento de Análisis, Apartado 16042, La Habana, Cuba,* [b]*Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Faculteit Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium, and* [c]*Laboratorio de Síntesis Orgánica, Universidad de La Habana, Apartado 10400, La Habana, Cuba. E-mail: hector.novoa@farm.kuleuven.ac.be*

(*Received 5 October 1998; accepted 3 December 1998*)

Abstract

In the title compound, $C_{23}H_{26}N_2O_4$, the central ring adopts a distorted boat conformation, while the two outer rings are almost in ideal envelope conformations.

---

† Present address: Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Faculteit Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium.