and then crystallized from a 3:2 mixture of hexane–diethyl ether. Crystals suitable for X-ray experiments were obtained by slow evaporation of the solvent. The melting point was found to be 397 K. $[\alpha]_D = -11.9$ (c 9.5 g dm^{-3}, chloroform).

Crystal data

$C_{16}H_{26}N_2O_3$
$M_r = 294.39$
Monoclinic
$P2_1$
$a = 6.358\,(5)$ Å
$b = 10.463\,(5)$ Å
$c = 12.842\,(5)$ Å
$\beta = 94.57\,(5)°$
$V = 851.6\,(9)$ Å^3
$Z = 2$
$D_x = 1.148$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 38 reflections
$\theta = 10.10$–$25.03°$
$\mu = 0.079$ mm^{-1}
$T = 173\,(2)$ K
Transparent block
$0.28 \times 0.12 \times 0.10$ mm
Colourless

Data collection

Siemens $P4$ diffractometer
$2\theta/\omega$ scans
Absorption correction: none
2349 measured reflections
1841 independent reflections
1458 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$

$\theta_{\max} = 25.49°$
$h = -1 \rightarrow 7$
$k = -1 \rightarrow 12$
$l = -15 \rightarrow 15$
3 standard reflections every 97 reflections intensity decay: 7.24%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.148$
$S = 1.025$
1749 reflections
195 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0889P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.198$ e Å^{-3}
$\Delta\rho_{\min} = -0.235$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O2—C12	1.239 (4)	N2—C12	1.323 (5)
O1—N1	1.457 (4)	N2—C2	1.465 (6)
O3—C12	1.338 (5)	C4—C3	1.523 (6)
O3—C13	1.472 (6)	C3—C2	1.543 (5)
N1—C5	1.461 (5)	C2—C1	1.526 (6)
N1—C3	1.472 (5)		
O1—N1—C5	105.2 (3)	C1—C2—C3	111.8 (3)
O1—N1—C3	107.4 (3)	O2—C12—N2	125.6 (4)
C5—N1—C3	115.0 (3)	O2—C12—O3	123.8 (4)
N2—C2—C1	109.0 (3)	N2—C12—O3	110.5 (3)
N2—C2—C3	112.6 (3)		
C1—C2—C3—C4	−59.3 (4)	C1—C2—C3—N1	173.8 (3)
N1—C3—C2—N2	50.8 (4)	C4—C3—C2—N2	177.7 (3)

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXTL-Plus*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1318). Services for accessing these data are described at the back of the journal.

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Trimethyl Isocyanurate and Triethyl Isocyanurate†

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Abstract

The crystal structures of trimethyl isocyanurate, $C_6H_9N_3O_3$, (1), and triethyl isocyanurate, $C_9H_{15}N_3O_3$, (2), contain topologically similar C—H⋯O hydrogen-bonded networks. In (1), there are two symmetry-independent molecules and each forms its own layer structure. In (2), two of the ethyl groups point one way with respect to the heterocyclic ring, while the third points in the opposite direction.

Comment

The crystal structure of trimethyl isocyanurate, (1), has been reported previously by Belaj & Nachbaur (1987) to

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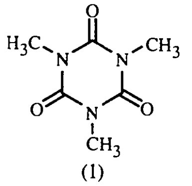
† Alternative names: 1,3,5-trimethyl-1,3,5-triazine-2,4,6(1*H*,3*H*,5*H*)-trione and 1,3,5-triethyl-1,3,5-triazine-2,4,6(1*H*,3*H*,5*H*)-trione.

low accuracy and without H-atom positions. Compound (1) in its molecular complex with 1,3,5-trinitrobenzene has been used in the construction of supramolecular networks stabilized by C—H···O hydrogen bonds (Thalladi *et al.*, 1995) and in this light, we have redetermined the structure of pure (1).


(1)

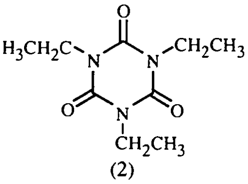

(2)

Fig. 1 is an *ORTEP*II (Johnson, 1976) diagram of the two symmetry independent molecules of (1), referred to hereafter as *A* and *B*. The dihedral angle between their mean planes is 81.69 (2)°. The crystal structure of (1) contains many C—H···O hydrogen bonds (Table 1) and the complex three-dimensional packing of the structure can be understood by an analysis of these bonds. The *A* molecules are assembled into a corrugated layer structure parallel to (001) as shown in Fig. 2. Each molecule of *A* is connected to four screw-axis-related neighbours along [010] with a total of six C—H···O hydrogen bonds; molecules are connected in pairs as C—H···O dimers [C···O 3.229 (4) and 3.679 (4) Å] and singly by isolated C—H···O bonds [C···O 3.362 (4) Å]. Adjacent inversion-related layers along [001] are linked by additional C—H···O hydrogen bonds [C···O 3.662 (4) and 3.437 (4) Å] to give a bilayer structure. These *A* bilayers are separated by layers which comprise stacked diads of *B* molecules (Fig. 3). The diads are linked with 'catemeric' C—H···O hydrogen bonds [C···O 3.725 (5) Å]. Bilayers of *A* and layers of *B* alternate along [001] and are related by C—H···O hydrogen bonds [C···O 3.513 (5), 3.185 (5) and 3.251 (4) Å] to complete the structure.

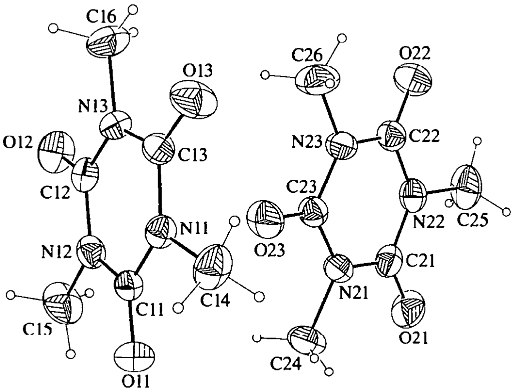


Fig. 1. An *ORTEP*II (Johnson, 1976) diagram of the two symmetry independent molecules of (1) in the asymmetric unit. Displacement ellipsoids are plotted at the 30% probability level. For clarity, H atoms are drawn as small circles.

The crystal structure of triethyl isocyanurate, (2), was subsequently studied to ascertain its relationship to (1). Fig. 4 shows an *ORTEP*II (Johnson, 1976) diagram of (2). The molecular geometry of (2) shows that the central heterocyclic ring and the three methylene groups

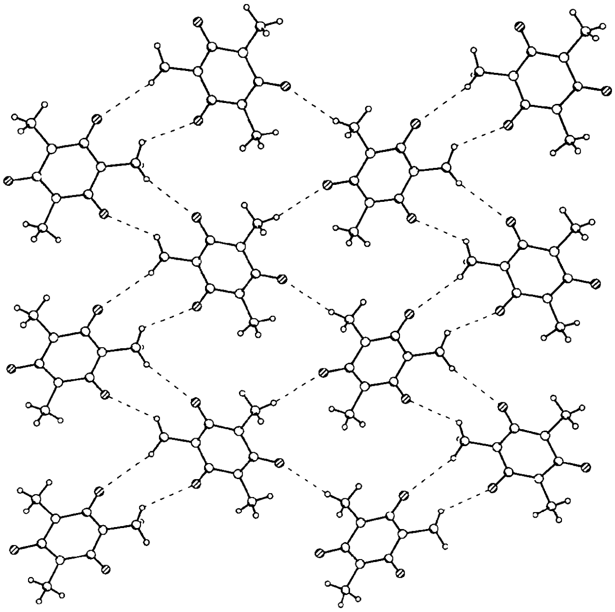


Fig. 2. Corrugated layer structure of *A* molecules in (1) perpendicular to [001]; [010] is horizontal. The C—H···O bonds are indicated.

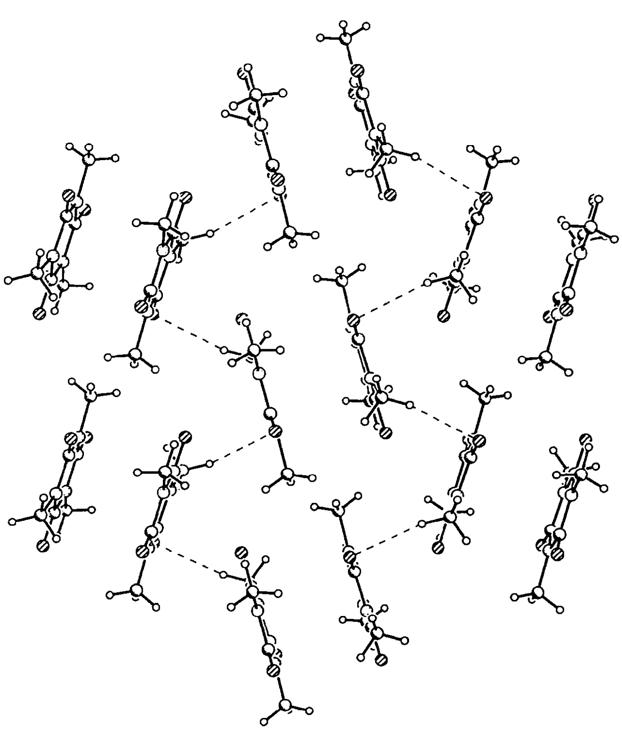


Fig. 3. Close packing of the *B* diads in (1). Note the 'catemer' synthon arrangement.

are coplanar (the deviation from the mean plane being only 0.6°). While two ethyl groups project in one direction with respect to the heterocyclic ring, the third projects in the opposite direction. Molecules related by translation along [010] and [100] are connected by C—H···O hydrogen bonds [C···O 3.350 (2) and 3.493 (3) Å] (Table 2) involving methylene and methyl groups, respectively, and this produces a square network (Fig. 5). The structure is completed with 2_1-related networks along [001], the inter-network packing being stabilized by the close packing of methyl groups.

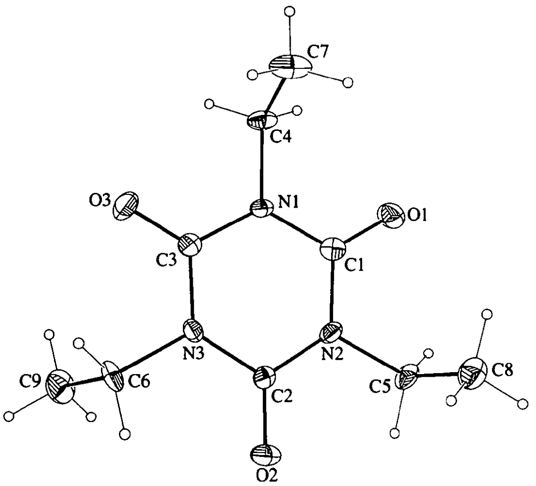


Fig. 4. An *ORTEPII* (Johnson, 1976) diagram of (2). Displacement ellipsoids are plotted at the 50% probability level. For clarity, H atoms are drawn as small circles.

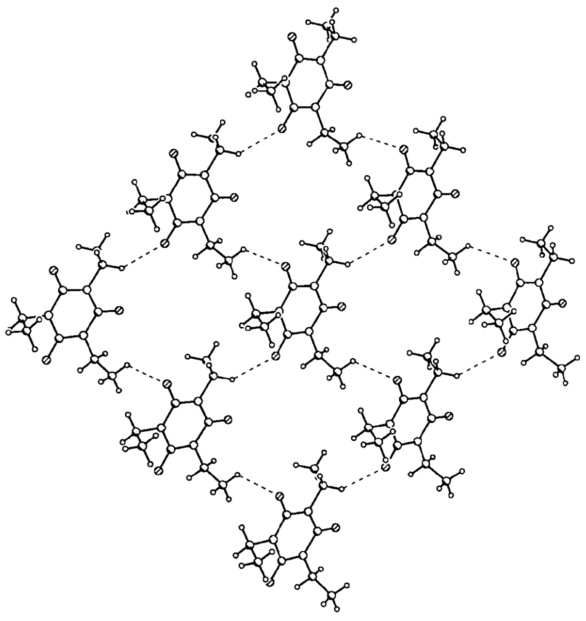


Fig. 5. C—H···O-mediated square networks in (2). Note the topological similarity to the network of *A* molecules in (1) shown in Fig. 2.

It may be seen from Figs. 3 and 5 that each *A* molecule in (1) and each molecule in (2) is hydrogen bonded to four near neighbours leading to topologically similar supramolecular networks in the two crystal structures.

Experimental

Compounds (1) and (2) were prepared according to the literature procedure of Fukui, Tanimoto & Kitano (1965).

Compound (1)

Crystal data

$C_6H_9N_3O_3$
$M_r = 171.155$
Monoclinic
$P2_1/a$
$a = 8.142\,(1)$ Å
$b = 13.393\,(1)$ Å
$c = 14.822\,(1)$ Å
$\beta = 100.88\,(7)°$
$V = 1587.2\,(3)$ Å^3
$Z = 8$
$D_x = 1.433$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 2$–$25°$
$\mu = 0.116$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.34 × 0.32 × 0.26 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2754 measured reflections
2508 independent reflections
1814 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.022$
$\theta_{\rm max} = 24.98°$
$h = -4 \rightarrow 9$
$k = -4 \rightarrow 15$
$l = -17 \rightarrow 17$
2 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.163$
$S = 1.088$
2508 reflections
218 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0967P)^2 + 0.3347P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.007$
$\Delta\rho_{\rm max} = 0.241$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.188$ e Å^{-3}
Extinction correction: *SHELXL93*
Extinction coefficient: 0.058 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °) for (1)*

D—H···A	D—H	H···A	D···A	D—H···A
C14—H141···O11$^{\rm i}$	0.96	2.602 (4)	3.229 (4)	123.2 (3)
C14—H142···O13$^{\rm ii}$	0.96	2.773 (4)	3.679 (4)	157.7 (3)
C14—H143···O12$^{\rm iii}$	0.96	2.841 (4)	3.662 (4)	144.0 (3)
C15—H151···O11$^{\rm iv}$	0.96	2.790 (4)	3.437 (4)	125.5 (4)
C16—H162···O12$^{\rm v}$	0.96	2.558 (4)	3.362 (4)	141.4 (4)
C16—H163···O22$^{\rm vi}$	0.96	2.955 (5)	3.513 (5)	118.3 (3)
C24—H242···O13$^{\rm vii}$	0.96	2.744 (5)	3.185 (5)	108.8 (4)
C25—H252···O12$^{\rm viii}$	0.96	2.808 (4)	3.251 (4)	109.0 (3)
C25—H253···O22$^{\rm ii}$	0.96	2.836 (5)	3.725 (5)	154.6 (3)

Symmetry codes: (i) $x-\frac{1}{2}, \frac{3}{2}-y, z$; (ii) $\frac{1}{2}+x, \frac{3}{2}-y, z$; (iii) $-x, 2-y, -z$; (iv) $1-x, 2-y, -z$; (v) $x-\frac{1}{2}, \frac{5}{2}-y, z$; (vi) $-x, 2-y, 1-z$; (vii) $1+x, y, z$; (viii) $1-x, 2-y, 1-z$.

Compound (2)

Crystal data

$C_9H_{15}N_3O_3$
$M_r = 213.24$
Orthorhombic
$P2_12_12_1$
$a = 7.835$ (2) Å
$b = 8.144$ (1) Å
$c = 16.820$ (5) Å
$V = 1073.3$ (4) Å^3
$Z = 4$
$D_x = 1.320$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 750 reflections
$\theta = 2.78$–$28.81°$
$\mu = 0.100$ mm^{-1}
$T = 293$ (2) K
Prism
0.20 × 0.15 × 0.10 mm
White

Data collection

Enraf–Nonius FAST area-detector diffractometer
Rotation scans
Absorption correction: none
1568 measured reflections
1568 independent reflections

1380 reflections with $I > 2\sigma(I)$
$\theta_{max} = 28.81°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.107$
$S = 1.074$
1568 reflections
136 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.1546P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.296$ e Å^{-3}
$\Delta\rho_{min} = -0.227$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H61···O1^{i}	0.97	2.572 (2)	3.350 (2)	137.4 (2)
C7—H71···O3^{ii}	0.96	2.667 (3)	3.305 (3)	124.3 (2)
C7—H73···O2^{iii}	0.96	2.876 (3)	3.493 (3)	123.0 (2)
C8—H81···O1^{iv}	0.96	2.604 (3)	3.299 (3)	129.5 (2)
C8—H82···O3^{v}	0.96	2.728 (3)	3.340 (3)	122.2 (2)

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iii) $x-1, y, z$; (iv) $\frac{1}{2}+x, \frac{5}{2}-y, 2-z$; (v) $\frac{1}{2}+x, \frac{3}{2}-y, 2-z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989) for (1); *MADNES* (Pflugrath & Messerschmidt, 1989) for (2). Cell refinement: *CAD*-4 *Software* for (1); *MADNES* for (2). Data reduction: *CAD*-4 *Software* for (1); *MADNES* for (2). For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978); software used to prepare material for publication: *SHELXL*93.

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3α,4β-Dihydroxy-5α-androstan-17-one

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Abstract

The ring conformation of the title compound, 3α,4β-dihydroxy-5α-androstan-17-one, $C_{19}H_{30}O_3$, is similar to that of the 5β-epimer except for the *trans A/B* ring junction and the geometry of ring *D*, which adopts a slightly deformed 13,14-half-chair conformation instead of the more common 14α-envelope conformation. The molecules are linked together by a two-dimensional network of hydrogen bonds involving the carbonyl and hydroxyl groups.

Comment

The title steroid, (I), is one of the intermediate compounds obtained during the synthesis of formestane (Tavares da Silva, Sá e Melo & Campos Neves, 1996), an irreversible aromatase inhibitor which is known to be a very effective drug in the treatment of breast cancer. This work is part of a project to study the conformation and intermolecular interactions of steroids related to formestane. The crystal structure of the 5β-epimer, also obtained as an intermediate in the formestane syn-