other in the ground state. Since it is generally assumed that if an EDA molecule exists in a twisted conformation in the ground state, the emission is likely to take place from a twisted geometry, it is quite likely that the single Stokes-shifted fluorescence band that is observed for the present system originates from the TICT state.

Experimental

The title compound was prepared according to a general procedure (Bridger *et al.*, 1968). A mixture of carbazole (1 g, 3 mmol) and sodium hydride (72 mg, 3 mmol) was stirred in dry dimethylformamide (15 ml) under a nitrogen atmosphere for 2 h. The sodium salt of carbazole so formed was then heated at 393 K with 4-fluorobenzonitrile (0.363 g, 3 mmol) and sodium iodide (0.456 g, 6 mmol) for about 20 h. The product, along with unreacted reactants, was precipitated by adding water to the reaction mixture. The precipitate was dried and the title compound was separated by column chromatography on a silica-gel column. A mixture of ethyl acetate and hexane (50:50) was used as the eluent. Colourless crystals were obtained from absolute ethanol upon slow evaporation of the solvent.

Crystal data

$C_{19}H_{12}N_2$
M_r = 268.31
Monoclinic
$P2_1/c$
a = 8.2739 (12) Å
b = 20.681 (4) Å
c = 8.5913 (11) Å
β = 104.937 (12)°
V = 1420.4 (4) Å^3
Z = 4
D_x = 1.255 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 1.5–27.5°
μ = 0.075 mm^{-1}
T = 293 (2) K
Block
1.04 × 0.72 × 0.72 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
3457 measured reflections
3251 independent reflections
2043 reflections with $I > 2\sigma(I)$

R_{int} = 0.016
θ_{max} = 27.48°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 26$
$l = -11 \rightarrow 10$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.115
S = 1.046
2043 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0610P)^2 + 0.9105P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.034 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *Xtal*3.4 (Hall *et al.*, 1995). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1320). Services for accessing these data are described at the back of the journal.

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Nitro derivatives of glutethimide

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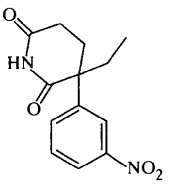
Abstract

Crystallographic experiments performed on single crystals of *m*-nitroglutethimide [3-ethyl-3-(3′-nitrophenyl)piperidine-2,6-dione, $C_{13}H_{14}N_2O_4$], (I), and *o*,*p*-

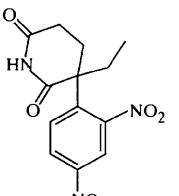
dinitroglutethimide [3-ethyl-3-(2′,4′-dinitrophenyl)-piperidine-2,6-dione, $C_{13}H_{13}N_3O_6$], (II), have unambiguously confirmed the molecular structures. From HPLC data it has been found that both of the title compounds are by-products in the nitration of glutethimide. The six-membered glutarimide rings of (I) and (II) have an envelope conformation with the C4 atom out of the plane, *i.e.* with C4-*exo* puckering in (I), but with C4-*endo* puckering in (II). Steric hindrances are, most likely, responsible for the observed conformational dissimilarity. The molecular structure of compound (I), unlike compound (II), is very similar to that of aminoglutethimide. Enantiomeric molecules in both compounds are linked together through pairs of symmetric $R_2^2(8)$ intermolecular hydrogen bonds forming centrosymmetric racemic dimers.

Comment

Aminoglutethimide is the generic name for 3-(4′-aminophenyl)-3-ethylpiperidine-2,6-dione. This non-steroidal drug inhibits several steps in the pathways of adrenal steroidogenesis, of which the principal ones appear to be the inhibition of peripheral (non-glandular) aromatase (conversion of androgens to estrogens), and of desmolase (conversion of cholesterol to pregnenolone), as well. Aromatase inhibition has been considered to be of greater relevance in determining clinical response. Therefore, aminoglutethimide has been used in endocrine therapy against metastatic hormone-dependent mammary tumours, especially in post-menopausal women (Santen *et al.*, 1978; Lönning & Kvinnsland, 1988). The *R*(+)-enantiomer of aminoglutethimide has been shown to be more active than the *S*(−) enantiomer (Finch *et al.*, 1975).



(I)



(II)

The intention of this paper is to confirm unambiguously the existence of *m*-nitroglutethimide, *i.e.* 3-ethyl-3-(3′-nitrophenyl)piperidine-2,6-dione, (I), as the major by-product in the nitration of glutethimide and of another by-product, *o,p*-dinitroglutethimide, *i.e.* 3-ethyl-3-(2′,4′-dinitrophenyl)piperidine-2,6-dione, (II), as well. In addition, detailed analysis and comparison of conformational properties of both compounds and their possible similarity with aminoglutethimide have been elucidated, as an experimental support for the structure–reactivity relationship studies performed by Danilovski *et al.* (1999). Figs. 1 and 2 depict only molecular structures of the *R*(+)-enantiomer from the racemic mixture found in the unit cell of both compounds, and the numbering scheme used.

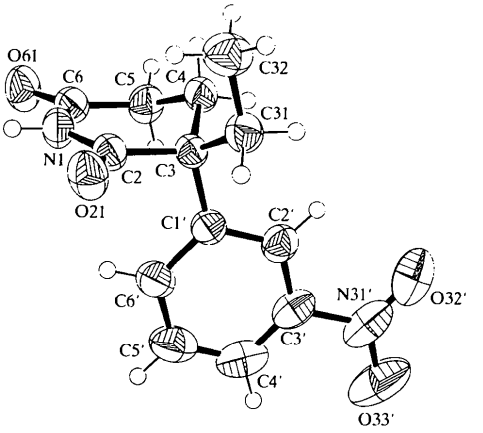


Fig. 1. *ORTEP*III (Johnson & Burnett, 1997) view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level, while H atoms are represented as spheres of an arbitrary radius.

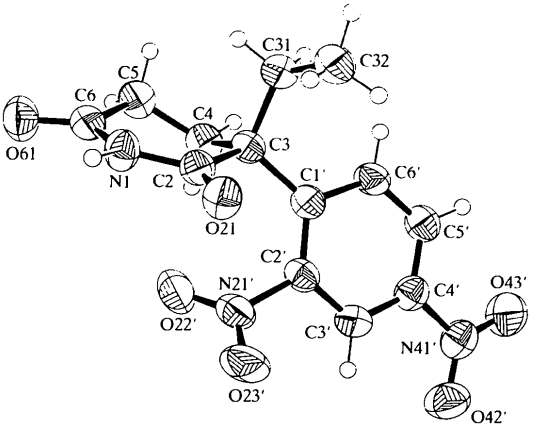


Fig. 2. *ORTEP*III (Johnson & Burnett, 1997) view of (II) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level, while H atoms are represented as spheres of an arbitrary radius.

Main conformational features of both compounds could be summarized by the presence of the six-membered glutarimide ring (piperidine-2,6-dione moiety) in its characteristic conformation, *i.e.* a non-planar, envelope conformation with C4 atom on the flap (glutarimide least-squares plane is defined throughout by atoms N1, C2, C3, C5 and C6). In compound (I) C4-*exo* puckering, regarding the C3-phenyl substituent, has been noticed with the C4 atom 0.623 (2) Å above the least-squares plane, while in compound (II) the C4 atom is 0.585 (2) Å below this plane adopting C4-*endo* puck-

ering [torsion angle C1′—C3—C4—C5 is 67.8 (2) in (I) and −170.7 (2)° in (II), while C31—C3—C4—C5 is −170.4 (2) in (I) and 66.9 (2)° in (II), respectively]. In addition, while the C3-phenyl substituent adopts an axial position in (I), it assumes the opposite, equatorial position in (II) [angles between the C3—C1′ bond and the normal to the glutarimide plane are as follows, 11.31 (8) in (I) and 58.22 (9)° in (II)]. Steric factors are, most likely, responsible for the observed dissimilarity. The phenyl-ring *ortho*-nitro substitution at the C2′ position in (II) causes significant steric hindrance and forces the phenyl ring from an axial to equatorial position at the C3 atom. Moreover, this phenyl-ring alteration from an axial in (I) to an equatorial position in (II) is accompanied by C4-*exo* to *endo* puckering alteration, as well. Conformational analysis of glutethimide has indicated thermodynamic preference for C4-*exo* puckering and its corresponding C3-phenyl axial orientation (Danilovski *et al.*, 1999). Accordingly, bond-angle analysis has elucidated the observed stability of C4-*exo* puckering in (I) and revealed the more significant deformation of the tetrahedral coordination at the C3 atom in (II) compared to (I) [bond angles C1′—C3—C4, C1′—C3—C31 and C1′—C3—C2 are 106.9 (1) 112.9 (1) and 112.1 (1) in (II) and 109.6 (1), 110.3 (1) and 107.9 (1)° in (I), respectively]. Nitro groups are conjugated to the phenyl ring making an angle with its least-squares plane of 5.34 (9) for the *meta*-nitro group in (I) and 5.85 (7)° for the *para*-nitro group in (II), indicating preserved coplanarity. But, due to steric hindrance the *ortho*-nitro group in (II) is twisted from coplanarity by 48.67 (7)°.

The *meta*-nitro derivative of glutethimide (I) is structurally very similar to aminoglutethimide. Molecules of both compounds have appeared to be L shaped with the phenyl moiety and the glutarimide ring forming the vertical arm and the base, respectively (Van Roey *et al.*, 1991). Dihedral angles formed by the least-squares planes of phenyl and glutarimide rings are 90.86 (6) in (I) and 88.8° in aminoglutethimide (Van Roey *et al.*, 1991). On the other hand, the dinitro derivative of glutethimide, (II), has a significantly smaller value of 60.65 (6)° for this dihedral angle.

Packing diagrams for both compounds are shown in Figs. 3 and 4 depicting the formation of centrosymmetric dimers in the unit cell. This dimerization is realised by one symmetric pair of $R_2^2(8)$ intermolecular hydrogen bonds (notation according to graph-set analysis; Bernstein *et al.*, 1995) between N1 and O61 in (I) [d(N1···O61) is 3.02 Å] or N1 and O21 in (II) [d(N1···O21) is 2.95 Å] with the corresponding atoms

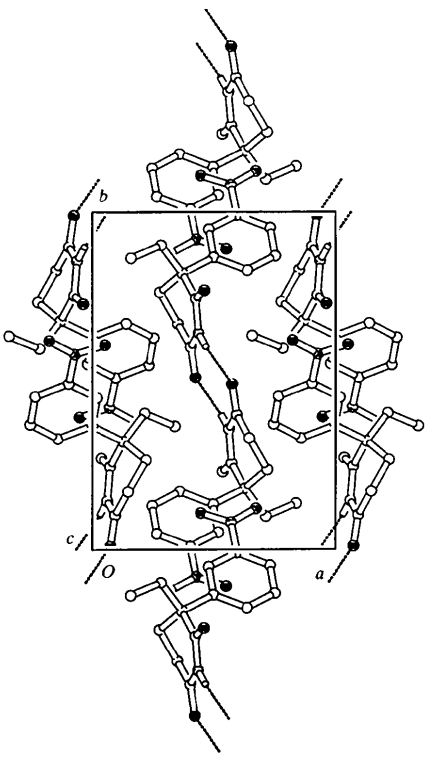


Fig. 3. The packing diagram (*PLUTON*97; Spek, 1997) of (I) viewed down the *c* axis. Non-hydrogen-bonding H atoms are omitted for clarity. Hydrogen bonds are shown as a dashed line.

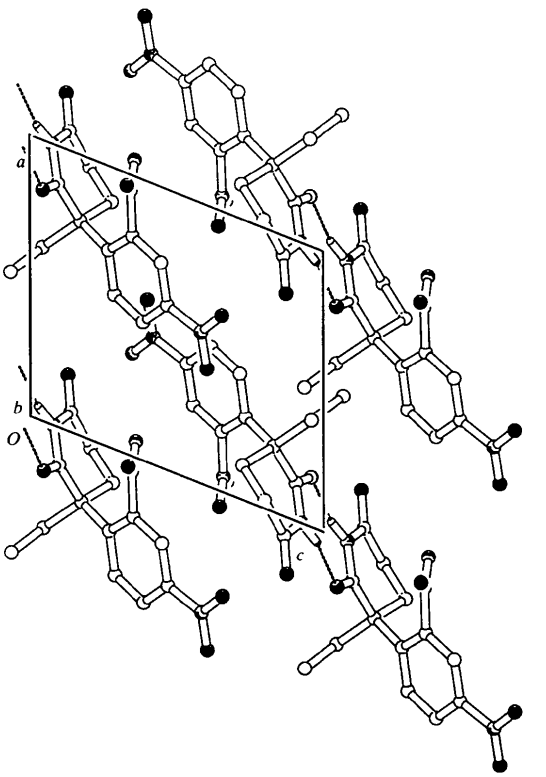


Fig. 4. The packing diagram (*PLUTON*97; Spek, 1997) of (II) viewed down the *b* axis. Non-hydrogen-bonding H atoms are omitted for clarity. Hydrogen bonds are shown as a dashed line.

of the inversion-centre-related enantiomeric molecule (Tables 3 and 4). Hence, the O61—C6 bond in (I) is significantly (about 6σ) longer than the O21—C2 bond, while in compound (II) this is reversed, *i.e.* the O21—C2 bond is (about 3σ) longer than the O61—C6 one.

Experimental

We have repeated the synthesis of aminoglutethimide as published (Adamo *et al.*, 1989). The key reaction step is nitration of glutethimide, *i.e.* 3-phenyl-3-ethylpiperidine-2,6-dione, using a mixture of concentrated H_2SO_4 and HNO_3 acids. Products of such electrophilic substitution had been expected to be *para*- and *ortho*-nitro derivatives. However, experimental yields have revealed a mixture of *para*- and *meta*-nitro derivatives in an approximate ratio of 3.6 to 1, containing less than 1% of *ortho*-nitro derivative (constitution in area % of the reaction mixture after a half hour of nitration time, determined by HPLC, was 54% *para*- and 15% *meta*-mononitro analogues, as well as 7% *ortho,para*-dinitro analogues). Simultaneously, with prolonged nitration time, the percentage of dinitro products had exceeded the percentage of mononitro products. The appearance of *meta*-nitro derivative, as the major by-product, has already been reported in the literature (Foster *et al.*, 1983; Hartmann & Batzl, 1986; Vinkler *et al.*, 1976; Stájer *et al.*. 1979).

A colourless and irregularly shaped crystal of compound (I) was chosen for X-ray data collection. It was obtained by slow evaporation of a methanol solution at room temperature. A colourless prism of compound (II) was obtained by evaporation of a mixture of acetone and petroleum ether in a 1:2 ratio. Crystals of both compounds exhibited fairly low diffraction power, so the first observed reflections [$I > 2\sigma(I)$] appeared at 0.86 Å resolution for (I), and 0.88 Å for (II). Moreover, in the 0.89–1.00 Å resolution shell, only 23.7% of reflections were observed in compound (I), and only 26.1% in compound (II).

Compound (I)

Crystal data

$C_{13}H_{14}N_2O_4$
M_r = 262.26
Monoclinic
$P2_1/n$
a = 9.4376 (7) Å
b = 13.168 (2) Å
c = 10.035 (2) Å
β = 96.71 (2)°
V = 1238.6 (3) Å^3
Z = 4
D_x = 1.406 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 20 reflections
θ = 15–30°
μ = 0.885 mm^{-1}
T = 293 K
Irregular shape
0.28 × 0.21 × 0.17 mm
Colourless

Data collection

Philips PW1100 diffractometer upgraded by Stoe & Cie
ω scans
Absorption correction: none
2130 measured reflections
2025 independent reflections
1556 reflections with $I > 2\sigma(I)$

R_{int} = 0.081
θ_{max} = 63.5°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 11$
3 standard reflections
frequency: 90 min
intensity decay: 3.5%

Refinement

Refinement on F^2
$R(F)$ = 0.039
$wR(F^2)$ = 0.111
S = 1.084
2017 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.246P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.153 e Å^{-3}
$\Delta\rho_{min}$ = −0.172 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.0037 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O21—C2	1.214 (2)	N1—C2	1.387 (2)
O61—C6	1.226 (2)	N1—C6	1.377 (2)
C2—C3—C4	110.4 (1)	C1′—C3—C4	109.6 (1)
C2—C3—C31	108.1 (1)	C1′—C3—C31	110.3 (1)
C4—C3—C31	110.5 (1)	C1′—C3—C2	107.9 (1)
O32′—N31′—C3′—C2′	−5.1 (3)	C3—C4—C5—C6	52.4 (2)
O33′—N31′—C3′—C2′	175.7 (2)	C31—C3—C1′—C6′	130.6 (2)
O32′—N31′—C3′—C4′	174.3 (2)	C31—C3—C1′—C2′	−51.8 (2)
C2—C3—C4—C5	−50.9 (2)	C31—C3—C4—C5	−170.4 (2)
C2—C3—C31—C32	−57.1 (2)	C4—C3—C1′—C6′	−107.5 (2)
C2—C3—C1′—C2′	−169.7 (2)	C1′—C3—C4—C5	67.8 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O61^i	0.86	2.17	3.022 (2)	168

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

Compound (II)

Crystal data

$C_{13}H_{13}N_3O_6$
M_r = 307.26
Triclinic
$P\bar{1}$
a = 8.5629 (18) Å
b = 8.8741 (15) Å
c = 10.594 (2) Å
α = 114.483 (8)°
β = 108.831 (8)°
γ = 92.235 (8)°
V = 679.3 (3) Å^3
Z = 2
D_x = 1.502 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 22 reflections
θ = 15–30°
μ = 1.036 mm^{-1}
T = 293 K
Prism
0.54 × 0.33 × 0.26 mm
Colourless

Data collection

Philips PW1100 diffractometer upgraded by Stoe & Cie
ω scans
Absorption correction: none
2391 measured reflections
2015 independent reflections
1597 reflections with $I > 2\sigma(I)$

R_{int} = 0.067
θ_{max} = 63.5°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -1 \rightarrow 11$
3 standard reflections
frequency: 90 min
intensity decay: 3.6%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.033$
$R(F) = 0.039$	$\Delta\rho_{max} = 0.254$ e Å^{-3}
$wR(F^2) = 0.114$	$\Delta\rho_{min} = -0.197$ e Å^{-3}
$S = 1.026$	Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
2007 reflections	Extinction coefficient: 0.039 (3)
200 parameters	Scattering factors from *International Tables for Crystallography* (Vol. C)
H-atom parameters constrained	
$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2 + 0.275P]$ where $P = (F_o^2 + 2F_c^2)/3$	

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

O21—C2	1.220 (2)	N1—C2	1.373 (2)
O61—C6	1.214 (2)	N1—C6	1.376 (2)
C4—C3—C31	109.2 (1)	C2—C3—C1′	112.1 (1)
C2—C3—C4	110.6 (1)	C1′—C3—C4	106.9 (1)
C2—C3—C31	105.2 (1)	C1′—C3—C31	112.9 (1)
O22′—N21′—C2′—C3′	−130.4 (2)	C2—C3—C31—C32	66.1 (2)
O22′—N21′—C2′—C1′	47.3 (2)	C2—C3—C1′—C2′	34.8 (2)
O23′—N21′—C2′—C3′	47.5 (2)	C3—C4—C5—C6	−53.2 (2)
O23′—N21′—C2′—C1′	−134.8 (2)	C31—C3—C1′—C6′	−31.9 (2)
O42′—N41′—C4′—C3′	−1.3 (3)	C31—C3—C1′—C2′	153.4 (2)
O42′—N41′—C4′—C5′	−179.4 (2)	C31—C3—C4—C5	−66.9 (2)
O43′—N41′—C4′—C5′	0.2 (3)	C4—C3—C1′—C6′	88.2 (2)
O43′—N41′—C4′—C3′	178.3 (2)	C1′—C3—C4—C5	170.7 (2)
C2—C3—C4—C5	48.4 (2)		

Table 4. *Hydrogen-bonding geometry (Å, °) for (II)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O21^{i}	0.86	2.13	2.951 (3)	160

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

For both compounds, data collection: *STADI*4 (Stoe & Cie, 1996*a*); cell refinement: *STADI*4; data reduction: *XRED* (Stoe & Cie, 1996*b*); program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *ORTEP*III (Johnson & Burnett, 1997) and *PLUTON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1321). Services for accessing these data are described at the back of the journal.

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4,6-Bis(benzyloxy)-1,3,5-triazin-2-ylamine

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Abstract

The title compound, $C_{17}H_{16}N_4O_2$, was synthesized by the reaction of 4,6-dichloro-1,3,5-triazin-2-ylamine, obtained from cyanuric chloride, with powdered sodium hydroxide in benzyl alcohol. It crystallizes as a centrosymmetric hydrogen-bonded dimer, with only one of the two H atoms of the amino group involved in a hydrogen bond. Whereas one of the two phenyl rings is nearly coplanar with the triazine ring, the other one is nearly perpendicular to it.

Comment

DNA analogues are of considerable interest in medicinal chemistry and molecular biology, mainly because of their possible use as therapeutic agents and their potential applications in diagnostics and as biomolecular tools (Dueholm *et al.*, 1994). PNA (peptide nucleic acid) is a DNA mimic (Egholm *et al.*, 1992; Nielsen, 1996) in which the phosphate–sugar backbone has been replaced