

Data collection

Siemens SMART CCD
PLATFORM diffractometer
 ω scans
Absorption correction:
multi-scan (SADABS;
Sheldrick, 1996)
 $T_{\min} = 0.749$, $T_{\max} = 0.924$
9854 measured reflections
2084 independent reflections
(plus 1470 Friedel-related reflections)

3384 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$
 $\theta_{\text{max}} = 27.86^\circ$
 $h = -6 \rightarrow 8$
 $k = -14 \rightarrow 14$
 $l = -25 \rightarrow 26$
Intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.115$
 $S = 1.243$
3554 reflections
157 parameters
H atoms treated by a
mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.2776P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.019$

$\Delta\rho_{\text{max}} = 0.21 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$
Extinction correction:
SHELXL (Sheldrick, 1997)
Extinction coefficient:
0.0009 (18)
Scattering factors from
International Tables for
Crystallography (Vol. C)
Absolute structure:
Flack (1983)
Flack parameter =
-0.07 (14)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Si—C	1.8551 (18)	B2—C	1.618 (3)
Si—C101	1.878 (2)	B2—N2	1.659 (3)
Si—C102	1.877 (2)	C1—N1	1.483 (3)
Si—C103	1.877 (2)	C1—C2	1.510 (3)
B1—C	1.613 (3)	C2—N2	1.505 (3)
B1—N1	1.652 (3)		
C—Si—C101	112.20 (10)	B2—C—Si	109.94 (13)
C—Si—C102	112.29 (10)	N1—C1—C2	118.62 (19)
C101—Si—C102	104.68 (11)	N2—C2—C1	118.92 (19)
C—Si—C103	111.96 (10)	C11—N1—C1	111.4 (2)
C101—Si—C103	107.88 (12)	C11—N1—C12	108.0 (2)
C102—Si—C103	107.42 (12)	C1—N1—C12	105.92 (18)
C—B1—N1	114.01 (14)	C11—N1—B1	110.82 (17)
C—B2—N2	115.93 (15)	C1—N1—B1	113.02 (16)
B1—C—B2	125.86 (15)	C12—N1—B1	107.39 (16)
B1—C—Si	106.54 (12)		

H atoms on C, B1 and B2 were located by difference map and were refined individually. Other H atoms were refined as riding, with U_{iso} values 1.3 times those of the parent atoms, and C—H distances of 0.97 \AA for CH_2 and 0.96 \AA for CH_3 groups.

Data collection: SMART (Siemens, 1995). Cell refinement: SMART and SAINT (Siemens, 1995). Data reduction: SAINT. Program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999). Program(s) used to refine structure: SHELXL (Sheldrick, 1997). Molecular graphics: SHELXL. Software used to prepare material for publication: SHELXL.

This work was supported by Northern Illinois University and by grants from the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the National Science Foundation.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1209). Services for accessing these data are described at the back of the journal.

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
Hosmane, N. S. (1999). Unpublished results.
Hosmane, N. S., Jia, L., Zhang, H., Bausch, J. W., Prakash, G. K. S., Williams, R. E. & Onak, T. P. (1991). *Inorg. Chem.* **30**, 3793–3795.
Hosmane, N. S., Zhang, H., Wang, Y., Lu, K.-J., Thomas, C. J., Ezhova, M. B., Helfert, S. C., Collins, J. D., Maguire, J. A., Gray, T. G., Baumann, F. & Kaim, W. (1996). *Organometallics*, **15**, 2425–2427.
Lipscomb, W. N. (1963). *Boron Hydrides*, p. 89. New York: Benjamin.
Lipscomb, W. N. (1966). *Science*, **153**, 373–378.
Lipscomb, W. N. & Britton, D. (1960). *J. Chem. Phys.* **33**, 275–280.
Muetterties, E. L. & Knoth, W. H. (1968). *Polyhedral Boranes*, p. 40. New York: Marcel Dekker.
Parry, R. W. (1998). *The Borane, Carborane, and Carbocation Continuum*, edited by J. Casanova, pp. 191–214. New York: Wiley.
Sheldrick, G. M. (1996). *SADABS. Program for the Empirical Absorption Correction of Area Detector Data*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1995). *SMART and SAINT. Data Collection and Reduction Software for the SMART System*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Williams, R. E. (1998). *The Borane, Carborane, and Carbocation Continuum*, edited by J. Casanova, pp. 3–56. New York: Wiley.
Zhang, H., Wang, Y., Saxena, A. K., Oki, A. R., Maguire, J. A. & Hosmane, N. S. (1993). *Organometallics*, **12**, 3933–3944.

Acta Cryst. (1999). **C55**, 2109–2111

4,6-Diethoxy-1,3,5-triazin-2(1H)-one

MARK GREENBERG, VITALY SHTEIMAN AND MENAHEM KAFTORY

Department of Chemistry, Technion-Israel Institute of Technology, Haifa 32000, Israel. E-mail: kaftory@tx.technion.ac.il

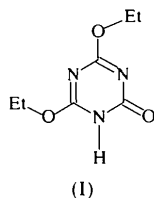
(Received 7 June 1999; accepted 7 September 1999)

Abstract

The molecules of the title compound, $\text{C}_7\text{H}_{11}\text{N}_3\text{O}_3$, are almost planar owing to an extensive conjugation of $\text{C}=\text{N}$ and $\text{C}=\text{O}$ double bonds resulting from five purely sp^2 hybridized atoms and one amide-N atom in the ring. The strong hydrogen $\text{N—H} \cdots \text{O}=\text{C}$ bonds produce a dimer across a centre of symmetry. The dimers are interconnected by weaker hydrogen bonds to form a layer structure.

Comment

The structure of the title compound, (I) (Fig. 1), has been studied in relation to the tendency of some cyanuric acid derivatives to undergo rearrangements in the solid or liquid states (Paoloni *et al.*, 1968; Tosato, 1979; Tosato & Soccorsi, 1982; Tosato, 1984; Kaftory & Handelsman-Benory, 1994). In contrast to its methoxy analogue, which undergoes O to N methyl transfer in the solid state (Greenberg & Kaftory, 1999), thermally induced rearrangements of ethyl groups in (I) take place only in the melt.



The bond angles and distances in (I) are in close agreement with those of the methoxy analogue (Główka & Bertolasi, 1987). The ring is approximately planar (deviations of the atoms from the ring mean plane do not exceed 0.01 Å) owing to an extensive conjugation of C=O and C=N double bonds resulting from five purely *sp*² hybridized atoms and one amide-N atom in the ring. The N—C bond lengths [1.297–1.358 (2) Å], except N1—C2 [1.382 (2) Å], are indicative of bond

orders significantly greater than 1. The ethoxy groups deviate only slightly from the ring mean plane (the displacements of the terminal C atoms are less than 0.20 Å) and the appropriate C—C—O—C torsion angles are 0.7 (2) and 5.6 (2)°.

The molecules are connected in pairs through N—H···O=C hydrogen bonds (see Table 1). The N···O distance [2.775 (2) Å] is comparable with those found for other *s*-triazines that also form strong hydrogen bonds: 2.784 (2) and 2.805 (1) Å in isocyanuric acid (Coppens & Vos, 1971); 2.791 (3) and 2.822 (3) Å in 4,6-dimethoxy-1,3,5-triazine-2(1*H*)-one (Główka & Bertolasi, 1987); 2.817 (3) and 2.853 (4) Å in 6-methoxy-1,3,5-triazine-2,4(1*H*,3*H*,5*H*,6*H*)-dione (Gilardi & George, 1987); 2.803 (1) and 2.845 (1) Å in 5-azauracil (Lowe & Schwalbe, 1998).

Adjacent dimers in (I) are linked by weaker C—H···O=C [3.380 (3) Å] and C—H···O—C [3.518 (2) Å] hydrogen bonds to form a layer structure. The interplanar stacking distance of 3.28 (1) Å is similar to those found in 2,4,6-trimethoxy-1,3,5-triazine and 3-methyl-4,6-dimethoxy-1,3,5-triazine-2(3*H*)-one [3.26 (1) and 3.24 (1) Å, respectively; Handelsman-Benory *et al.*, 1995], and is characteristic of a normal van der Waals electrostatic interaction.

Experimental

The title compound was synthesized by ethylation of cyanuric chloride according to the method of Dudley *et al.* (1951) and separation by silica-gel column chromatography. Colourless crystals of (I) were obtained from an ethyl acetate solution by slow evaporation of the solvent at room temperature.

Crystal data

C₇H₁₁N₃O₃
M_r = 185.19
 Triclinic
P $\bar{1}$
a = 7.393 (1) Å
b = 7.736 (1) Å
c = 8.679 (1) Å
 α = 91.01 (1)°
 β = 105.43 (1)°
 γ = 109.98 (1)°
V = 446.4 (1) Å³
Z = 2
D_x = 1.378 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 1571 reflections
 θ = 2.45–25.00°
 μ = 0.109 mm⁻¹
T = 293 (2) K
 Prism
 0.18 × 0.14 × 0.12 mm
 Colourless

Data collection

Nonius KappaCCD diffractometer
 φ scans
 Absorption correction: none
 1571 measured reflections
 1571 independent reflections
 1048 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.028
 θ _{max} = 25°
h = 0 → 8
k = -9 → 8
l = -10 → 9
 Intensity decay: none

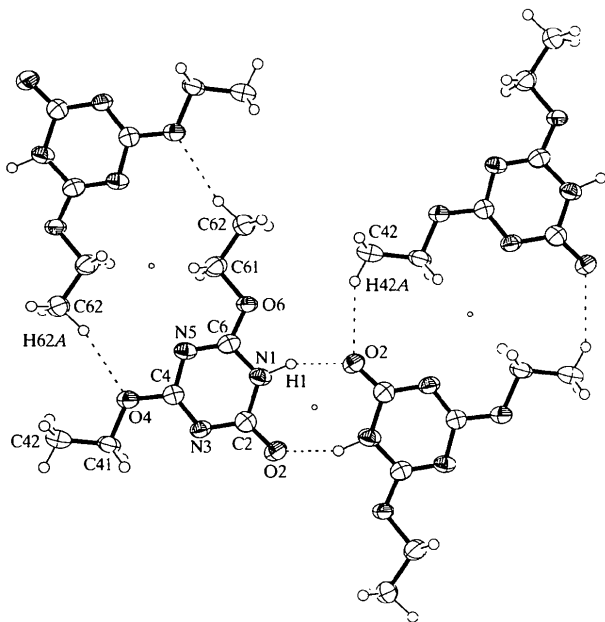


Fig. 1. An ORTEP (Johnson & Burnett, 1997) diagram of the layer structure of (I). The hydrogen bonds (see Table 1) are indicated by dashed lines. Centres of symmetry are drawn as small circles. Ellipsoids of atomic displacement for all atoms are drawn at the 50% probability level.

RefinementRefinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.092$ $S = 0.901$

1571 reflections

162 parameters

All H-atom parameters
refined $w = 1/[\sigma^2(F_o^2) + (0.0615P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Scattering factors from

*International Tables for
Crystallography* (Vol. C)Table 1. *Hydrogen-bonding geometry* (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...O2'	0.91 (2)	1.86 (2)	2.775 (2)	175 (2)
C62—H62A...O4 ⁱⁱ	1.01 (2)	2.51 (2)	3.518 (2)	178 (2)
C42—H42A...O2 ⁱⁱⁱ	1.01 (2)	2.45 (2)	3.380 (3)	152 (1)

Symmetry codes: (i) $-x, 1 - y, 2 - z$; (ii) $1 - x, 1 - y, 1 - z$;
(iii) $-1 - x, -y, 1 - z$.The refined N—H distance is 0.91 (2) \AA and the refined C—H distances are in the range 0.95 (2)–1.03 (2) \AA .Data collection: *COLLECT* (Nonius, 1998). Cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997). Data reduction: *DENZO-SMN*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *ORTEPIII* (Johnson & Burnett, 1997). Software used to prepare material for publication: *SHELXL97*.

The authors wish to thank Drs M. Kapon and M. Botoshansky for their valuable assistance. This research was mainly supported by the Israel Science Foundation, and partially supported by the Technion VPR Fund – E. and M. Mendelson Research Fund, and by the Fund for the Promotion of Research at the Technion.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1365). Services for accessing these data are described at the back of the journal.

References

- Coppens, P. & Vos, A. (1971). *Acta Cryst.* **B27**, 146–158.
 Dudley, J. R., Thurston, J. T., Schaefer, F. C., Holm-Hansen, D., Hull, C. J. & Adams, P. (1951). *J. Am. Chem. Soc.* **73**, 2986–2990.
 Gilardi, R. & George, C. (1987). *Acta Cryst.* **C43**, 363–364.
 Głowska, M. L. & Bertolasi, V. (1987). *Acta Cryst.* **C43**, 149–151.
 Greenberg, M. & Kaftory, M. (1999). In preparation.
 Handelsman-Benory, E., Botoshansky, M. & Kaftory, M. (1995). *Acta Cryst.* **C51**, 2421–2424.
 Johnson, C. K. & Burnett, M. N. (1997). *ORTEPIII*. Version 1.0.2. University of Glasgow, Scotland.
 Kaftory, M. & Handelsman-Benory, E. (1994). *Mol. Cryst. Liq. Cryst.* **240**, 241–249.
 Lowe, P. R. & Schwalbe, C. H. (1998). *Acta Cryst.* **C54**, 1936–1938.
 Nonius (1998). *COLLECT. KappaCCD Data Collection Software*. Nonius BV, Delft, The Netherlands.
 Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
 Paoloni, L., Tosato, M. L. & Cignitti, M. (1968). *J. Heterocycl. Chem.* **5**, 533–544.
 Sheldrick, G. M. (1997a). *SHELXS97. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

- Tosato, M. L. (1979). *J. Chem. Soc. Perkin Trans. 2*, pp. 1371–1375.
 Tosato, M. L. (1984). *J. Chem. Soc. Perkin Trans. 2*, pp. 1593–1599.
 Tosato, M. L. & Soccorsi, L. (1982). *J. Chem. Soc. Perkin Trans. 2*, pp. 1321–1326.

Acta Cryst. (1999). **C55**, 2111–2114**Two Schiff base derivatives of 4-amino-3-propyl-1,2,4-triazole-5-thione**ROBERT M. McCARRICK,^a PHILIP J. SQUATTRITO,^{a*}
RAJKUMAR NOREN SINGH,^b RAM N. HANDA^b AND
SURENDRA N. DUBEY^b^a*Department of Chemistry, Central Michigan University, Mount Pleasant, Michigan 48859, USA, and* ^b*Department of Chemistry, Kurukshetra University, Kurukshetra 136 119, Haryana, India. E-mail: p.squattrito@cmich.edu*

(Received 18 June 1999; accepted 6 September 1999)

Abstract

The Schiff base compounds 4-isopropylideneamino-3-propyl-4,5-dihydro-1*H*-1,2,4-triazole-5-thione, C₈H₁₄N₄S, (I), and 4-[1-(2-hydroxyphenyl)ethylideneamino]-3-propyl-4,5-dihydro-1*H*-1,2,4-triazole-5-thione, C₁₃H₁₆N₄OS, (II), contain essentially planar triazole rings in which the corresponding bond distances and angles are equivalent within experimental error. Both compounds show the same *anti* conformation of the propyl groups, however, they differ in the positioning of the functional groups on N atom. The isopropylidene group in (I) and the 1-(2-hydroxyphenyl)ethylideneamino group in (II) are rotated approximately 77° in opposite directions about the N—N bond relative to the triazole ring. In the case of (II), the adopted conformation permits an intramolecular hydrogen bond between the hydroxyl-H atom and the amine-N atom. Both compounds form hydrogen-bonded dimers through N—H...S interactions involving the thione-S atom and the adjacent protonated N atom on the ring. The structural features of these compounds are compared with those of similar amine- and thione-substituted triazoles and their Schiff base derivatives.

Comment

As part of a continuing study of Schiff bases of amine- and thione-substituted triazoles (Sen *et al.*, 1998) and their metal complexes (Sen *et al.*, 1996, 1997; Clark *et al.*, 1999), we have structurally characterized the compounds 4-isopropylideneamino-3-propyl-1,2,4-triazole-5-thione, (I), and 4-[1-(2-hydroxyphenyl)ethylideneamino]-3-propyl-1,2,4-triazole-5-thione, (II). As