

REGULAR STRUCTURAL PAPERS

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2-Methyl-4,6-diphenyl-1,2,3-triazinium Iodide

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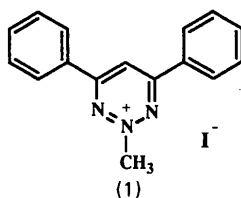
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Abstract

The planarity and endocyclic bond distances of the 1,2,3-triazine ring indicate extensive delocalization of electron density. The triazine ring and the iodine anion lie on the crystallographic twofold rotation axis. The bond angle involving the three N atoms is very large [129.0(6)°] because of the ionic contribution of the central N⁺ atom, which acts as a counter cation to I⁻.

Comment

We are interested in the chemistry of heterocyclic compounds containing many N atoms. An X-ray analysis of the title compound (1) was undertaken to investigate the structure of 1,2,3-triazinium salts and provide information for theoretical studies of this class of compounds. The preparation of compound (1) was reported by Ohsawa, Arai, Ohnishi, Kaihoh, Itoh, Yamaguchi, Igeta & Iitaka (1985).



The triazine ring is almost planar, with a maximum displacement from the least-squares plane of 0.013 Å for N5. This planarity as well as the endocyclic bond distances indicate an extensive delocalization of electron density. The bond lengths and angles of the triazine ring are almost consistent with those found in unsubstituted 1,2,3-triazine (Yamaguchi, Ohsawa, Arai, Ohnishi, Igeta & Iitaka, 1983). However, the N5–N3–N5' angle in compound (1) is 7.2° larger [129.0(6)°] than that in the unsubstituted compound. This may possibly be ascribed to the ionic contribution of the positively charged N3 atom, which acts as a counter cation to I⁻. The distances

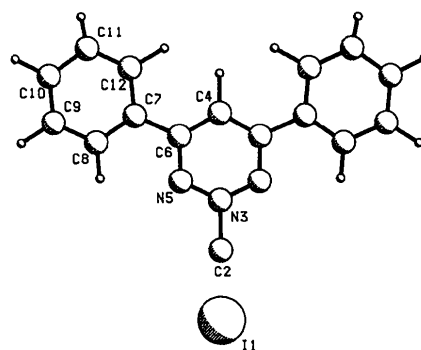


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule.

between I1 and C2, and I1 and N3 are 3.693(9) and 5.163(6) Å, respectively. The dihedral angle between the triazine and phenyl rings is 5.4°.

Experimental

Crystal data

C₁₆H₁₄N₃⁺·I⁻

M_r = 375.21

Monoclinic

*C*2/*c*

a = 18.234 (2) Å

b = 11.984 (1) Å

c = 7.468 (1) Å

β = 113.95 (2)°

Cell parameters from 20
reflections

θ = 27.0–30.5°

V = 1491.3 (4) Å³

Z = 4

D_x = 1.671 Mg m⁻³

Cu Kα₁

λ = 1.5405 Å

μ = 16.98 mm⁻¹

T = 297 K

Prism

0.30 × 0.15 × 0.11 mm

Reddish

Data collection

Rigaku AFC-5 diffractometer

ω/2θ scans

Absorption correction:

none

1307 measured reflections

1117 independent reflections

1085 observed reflections

[*F* > 3σ(*F*)]

*R*_{int} = 0.023

θ_{max} = 60°

h = -20 → 20

k = 0 → 13

l = 0 → 8

3 standard reflections

monitored every 150

reflections

intensity variation: <3%

Refinement

Refinement on *F*²

Final *R* = 0.048

ω*R* = 0.047

S = 1.398

1085 reflections

174 parameters

All H-atom parameters re-
fined

w = 1/[σ²(*F*) + 0.035*F*²]

(Δ/σ)_{max} = 0.176

Δρ_{max} = 0.276 e Å⁻³

Δρ_{min} = -0.312 e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Data collection: *AFD* (Rigaku Corporation, 1985a). Cell refinement: *AFD*. Data reduction: *RCRYSTAN* (Rigaku Corpora-

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
H1	1.0000	0.28813 (4)	0.2500	0.084 (1)
C2	1.0000	0.5963 (7)	0.2500	0.081 (3)
N3	1.0000	0.7189 (4)	0.2500	0.066 (2)
C4	1.0000	0.9359 (6)	0.2500	0.067 (2)
N5	1.0686 (2)	0.7655 (4)	0.3536 (6)	0.068 (1)
C6	1.0694 (2)	0.8788 (4)	0.3593 (6)	0.063 (1)
C7	1.1461 (2)	0.9296 (4)	0.4837 (6)	0.062 (1)
C8	1.2106 (3)	0.8628 (5)	0.6002 (8)	0.077 (1)
C9	1.2830 (3)	0.9120 (5)	0.7205 (9)	0.084 (2)
C10	1.2919 (3)	1.0254 (6)	0.7251 (9)	0.086 (2)
C11	1.2281 (3)	1.0926 (6)	0.6084 (9)	0.087 (2)
C12	1.1551 (3)	1.0450 (4)	0.4888 (8)	0.074 (1)

Table 2. Geometric parameters (\AA , $^\circ$)

C2—N3	1.469 (10)	C7—C6	1.461 (6)
N3—N5	1.300 (4)	C8—C9	1.392 (7)
C4—C6	1.378 (6)	C9—C10	1.368 (10)
N5—C6	1.358 (6)	C10—C11	1.393 (8)
C7—C12	1.392 (7)	C11—C12	1.391 (8)
C7—C8	1.397 (7)		
N5—N3—C2	115.5 (3)	C12—C7—C6	120.2 (4)
N5—N3—N5'	129.0 (6)	C8—C7—C6	120.3 (4)
C6—C4—C6'	120.4 (7)	C9—C8—C7	119.9 (6)
N3—N5—C6	116.4 (4)	C10—C9—C8	120.6 (5)
N5—C6—C4	118.8 (4)	C9—C10—C11	119.9 (5)
N5—C6—C7	115.6 (4)	C12—C11—C10	120.3 (6)
C4—C6—C7	125.6 (4)	C11—C12—C7	119.8 (5)
C12—C7—C8	119.5 (4)		

tion, 1985b). Program(s) used to solve structure: *SAPI85* (Yao, Zheng, Qian, Han, Gu & Fan, 1985). Program(s) used to refine structure: *RCRYSTAN*. Molecular graphics: *ACV* (Stardent Computer Inc., 1990). Software used to prepare material for publication: *XPACK* (Yamaguchi, 1987).

The ω -scan width was $(1.3 + 0.14 \tan \theta)^\circ$ and the scan speed was $16^\circ \text{ min}^{-1}$. Refinement was by the full-matrix least-squares method. The methyl H atoms were refined with occupancy factor 0.5.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54928 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1000]

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Unusual Conformation of (*R,R*)-*N,N,N',N'*-Tetramethyltartramide (I) and its *O,O'*-Dibenzoyl Derivative (II)†

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Abstract

Despite the conformational stability of optically active tartaric acid and its salts, all of which adopt the energetically preferred –synclinal conformation in the solid state, the tetramethyl amide derivatives (I) and (II) adopt an unprecedented +synclinal conformation. In this conformation both hydroxy (or benzoxyloxy) and amide groups are in *gauche* arrangements, and the H atoms are *trans*. Compound (I) has a twofold rotation axis coinciding with the crystallographic diad axis lying at $(x, 0, \frac{1}{6})$. A three-dimensional network of O—H...O hydrogen bonds connects the molecules of (I) in the crystal while the molecules of (II) are mainly held together by van der Waals forces.

Comment

It is generally accepted that for optically active tartaric acid and its derivatives as well as for its salts there is a strong preference for a planar carbon-chain conformation. When applied to the *O,O'*-dibenzoyl derivative of (*R,R*)-*N,N,N',N'*-tetramethyltartramide, the exciton chirality method revealed the presence of a bent carbon-chain conformation (Gawroński, Gawrońska & Rychlewska, 1989). In order to compare the circular dichroism conformational results in solution with the conformation of tartramides in the solid state, the X-ray analysis of (*R,R*)-*N,N,N',N'*-tetramethyltartramide (I) and its *O,O'*-dibenzoyl derivative (II) was undertaken. The

† Alternative nomenclature: (I) (*R,R*)-2,3-dihydroxy-*N,N,N',N'*-tetramethylbutanediamide; (II) (*R,R*)-2,3-dibenzoyloxy-*N,N,N',N'*-tetramethylbutanediamide.