

1-Dimethylamino-3-dimethyliminio-1-(*p*-methylphenyl)prop-1-ene perchlorate

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$

R factor = 0.075

wR factor = 0.272

Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{14}\text{H}_{21}\text{N}_2^+ \cdot \text{ClO}_4^-$, the planar vinamidinium chain forms a dihedral angle of $84.0(1)^\circ$ with the benzene ring. The molecular packing is stabilized by $\text{C}-\text{H} \cdots \text{O}$ interactions.

Comment

The title compound, (I), was studied, as a member of a series of vinamidinium systems, to explore the possibility of these compounds showing non-linear optical properties.

The molecular structure of (I) with the atom-numbering scheme is shown in Fig. 1. The title molecule is non-planar; the two planar segments, *viz.* the benzene ring and the vinamidinium chain ($\text{N1}/\text{C1}/\text{C2}/\text{C3}/\text{N2}$), have a dihedral angle of $84.0(1)^\circ$. The $\text{N}-\text{C}$ bonds (see Table 1) at both ends of the vinamidinium chain have lengths intermediate between a single and double bond, as a result of delocalization of the electrons. Similar features have been observed in a related structure (Girija *et al.*, 2004).

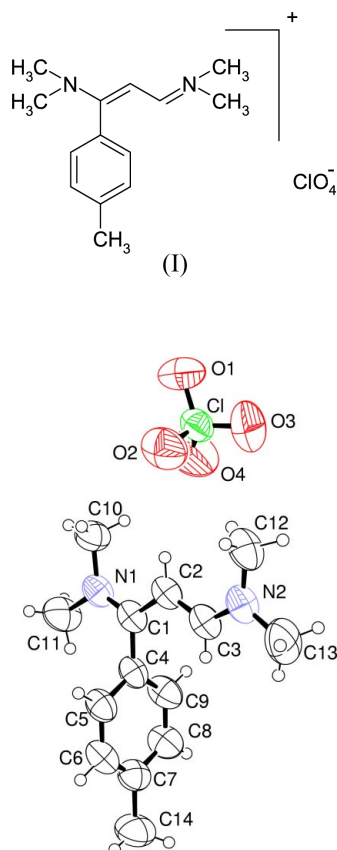


Figure 1
ORTEP-3 (Farrugia, 1997) diagram of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

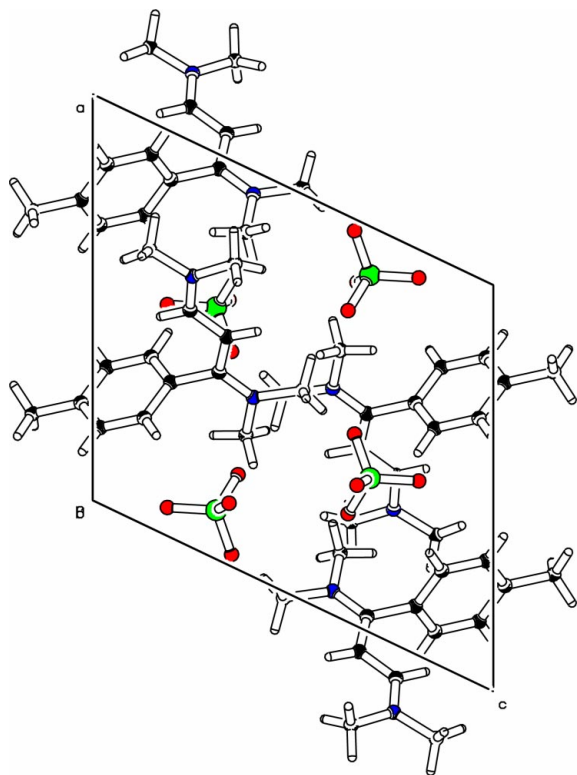


Figure 2
The molecular packing in the unit cell, viewed down the *b* axis.

The crystal structure of (I) is stabilized by a number of C—H···O interactions (Table 2), including two bifurcated C—H···O interactions.

The compound does not exhibit second harmonic generation (SHG), since it has crystallized in a centrosymmetric space group.

Experimental

A formylating agent was obtained by the action of phosphoryl chloride and dimethylformamide in chloroform. To this reagent, *p*-methylacetophenone, dissolved in the same solvent, was added. The mixture was heated under reflux, extracted with chloroform and treated with a saturated solution of sodium perchlorate in water. The compound was filtered off and dried over P₂O₅ (Holy *et al.*, 1965). Crystals were grown from ethanol at room temperature by slow evaporation.

Crystal data

C₁₄H₂₁N₂⁺·ClO₄[−]
M_r = 316.78
 Monoclinic, *P*2₁/*a*
a = 11.601 (3) Å
b = 12.409 (2) Å
c = 12.664 (4) Å
 β = 115.501 (18)°
V = 1645.4 (8) Å³
Z = 4
D_x = 1.279 Mg m^{−3}

D_m not measured
 MoK α radiation
 Cell parameters from 23 reflections
 θ = 3.5–4.4°
 μ = 0.25 mm^{−1}
T = 293 (2) K
 Prism, colourless
 0.3 × 0.2 × 0.1 mm

Data collection

Rigaku AFC-7S diffractometer
 $\omega/2\theta$ scans
 3045 measured reflections
 2890 independent reflections
 1649 reflections with *I* > 2 σ (*I*)
*R*_{int} = 0.100
 θ_{\max} = 25.0°

h = 0 → 13
k = 0 → 14
l = −15 → 13
 3 standard reflections
 every 150 reflections
 intensity decay: 10.4%

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.075
wR(*F*²) = 0.272
S = 1.08
 2890 reflections
 196 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1546P)^2 + 0.6164P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.42 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.34 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.024 (6)

Table 1

Selected geometric parameters (Å, °).

N1—C1	1.319 (6)	N2—C13	1.476 (8)
N1—C10	1.474 (6)	C1—C2	1.402 (6)
N1—C11	1.459 (7)	C1—C4	1.491 (6)
N2—C3	1.316 (6)	C2—C3	1.383 (7)
N2—C12	1.446 (7)		
C1—N1—C10	122.2 (4)	C12—N2—C13	116.5 (4)
C1—N1—C11	123.2 (4)	N1—C1—C2	122.2 (4)
C10—N1—C11	114.6 (4)	N1—C1—C4	116.8 (4)
C3—N2—C12	123.8 (4)	N2—C3—C2	126.4 (4)
C3—N2—C13	119.6 (5)		
N1—C1—C4—C9	95.5 (5)	N1—C1—C4—C5	−84.7 (5)
C2—C1—C4—C9	95.1 (6)	C2—C1—C4—C5	−84.7 (5)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C2—H2···O2	0.93	2.81	3.740 (6)	177
C10—H10C···O4	0.96	2.55	3.465 (9)	159
C12—H12B···O2	0.96	2.75	3.599 (9)	147
C12—H12B···O3	0.96	2.95	3.750 (8)	141
C5—H5···O1 ⁱ	0.93	2.71	3.589 (7)	159
C5—H5···O2 ⁱ	0.93	2.84	3.643 (8)	146
C6—H6···O1 ⁱⁱ	0.93	2.72	3.601 (6)	158
C14—H14A···O4 ⁱⁱ	0.96	2.85	3.796 (8)	168
C8—H8···O1 ⁱⁱⁱ	0.93	2.93	3.754 (8)	148
C9—H9···O1 ^{iv}	0.93	2.82	3.458 (6)	127
C10—H10A···O4 ^v	0.96	2.80	3.725 (10)	163
C13—H13A···O3 ^{vi}	0.96	2.94	3.740 (8)	142
C12—H12A···O3 ^{vi}	0.96	2.52	3.356 (9)	145
C12—H12C···O4 ^{vii}	0.96	2.51	3.416 (7)	156

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

H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and allowed to ride on their parent atoms, with *U*_{iso}(H) set at 1.5*U*_{eq}(C) for the methyl H atoms and at 1.2*U*_{eq}(C) for the other H atoms. A rotating group model was used for the methyl groups.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *TEXSAN*; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 1990).

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