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1,3,7-Trimethylxanthenium 2,4,6-trinitrophenolate

A. Chandramohan,[a] D. Gayathri,[b] D. Velmurugan,[b]* K. Ravikumar[c] and M. A. Kandhaswamy[a]

[a]Department of Chemistry, Sri Ramakrishna Mission Vidhyala College of Arts and Science, Coimbatore 641 020, India, [b]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and [c]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India

Correspondence e-mail: d_velu@yahoo.com

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Key indicators

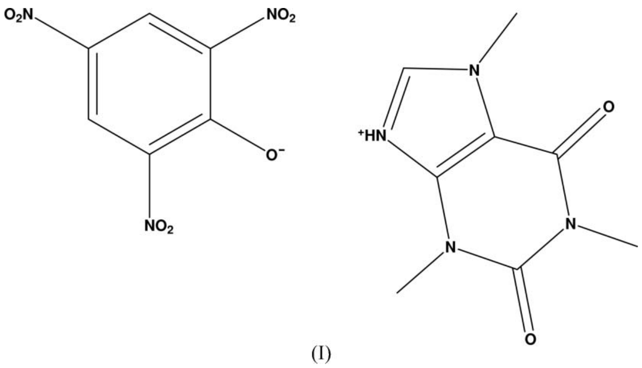
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.046
wR factor = 0.140
Data-to-parameter ratio = 12.9

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

The caffeinium and picrate ions in the title compound, $C_8H_{11}N_4O_2^+ \cdot C_6H_2N_3O_7^-$, are connected by intermolecular N—H···O and C—H···O interactions.

Comment

Molecular complexes of picric acid have shown very good nonlinear optical properties (Krishnakumar & Nagalakshmi, 2007). We present here the crystal structure of the title compound, (I). The title compound showed NLO activity. The second harmonic generation (SHG) efficiency was estimated to be 20 mV.



(I)

Bond lengths and angles of compound (I) are comparable with the literature values (Allen *et al.*, 1987).

The crystal packing of (I) is stabilized by N—H···O and C—H···O intermolecular interactions (Table 1).

Experimental

Analytical grade caffeine (1.94 g, 0.01 mol) and picric acid (2.29 g, 0.01 mol) were dissolved in chloroform (20 ml) separately in a 1:1 molar ratio and the two solutions were mixed together. The mixture was stirred well for about 15 min, when a yellow precipitate of the charge-transfer complex caffeinium picrate was obtained. This was filtered off and repeatedly recrystallized by slow evaporation of a 1:1 (*v*/*v*) solvent mixture of chloroform and methanol to obtain good quality crystals.

Crystal data

$C_8H_{11}N_4O_2^+ \cdot C_6H_2N_3O_7^-$
M_r = 423.31
Monoclinic, $P2_1/n$
a = 8.2996 (10) Å
b = 9.0014 (11) Å
c = 23.210 (3) Å
β = 91.474 (2)°
V = 1733.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 (2) K
0.29 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
18720 measured reflections
4035 independent reflections
3243 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.140$
$S = 1.04$
4035 reflections
312 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.31$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O3	0.86	1.83	2.671 (2)	167
C5—H5···O9*A*	0.93	2.36	2.915 (4)	118
C6—H6*A*···O2$^{\rm i}$	0.96	2.43	3.350 (2)	161
C7—H7*A*···O6$^{\rm ii}$	0.96	2.54	3.308 (2)	137
C8—H8*A*···O3	0.96	2.31	3.256 (2)	169

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+2, -y+1, -z+1$.

All H atoms were refined using a riding model, with C—H = 0.93 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for aromatic H, C—H = 0.96 Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C) for methyl H, and N—H = 0.86 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(N) for the NH group. The methyl groups were allowed to rotate but not to tip. Two nitro groups are disordered over two sites with occupation factors of 0.70 (1) for O4*A* and O5*A*, 0.30 (1) for O4*B* and O5*B*, 0.80 (2) for O8*A* and O9*A*, and 0.20 (2) for O8*B* and O9*B*. The occupancy factors were fixed in the final refinement.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

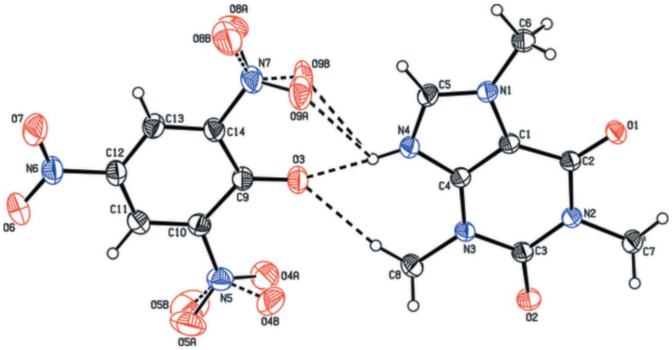


Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids. The minor sites of the disordered atoms are not shown. Dashed lines indicate hydrogen bonds.

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