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6,6′-Dimethoxy-2,2′-(ethane-1,2-diyldiammoniodimethylene)diphenol dinitrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.012 Å
R factor = 0.099
wR factor = 0.289
Data-to-parameter ratio = 11.4

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

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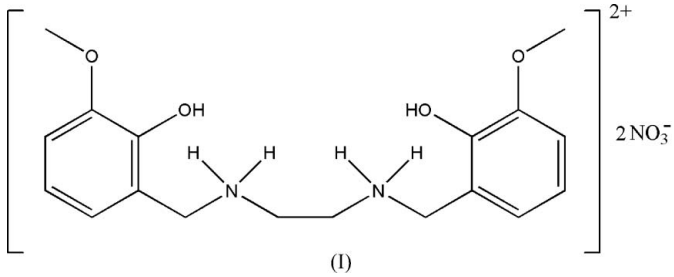
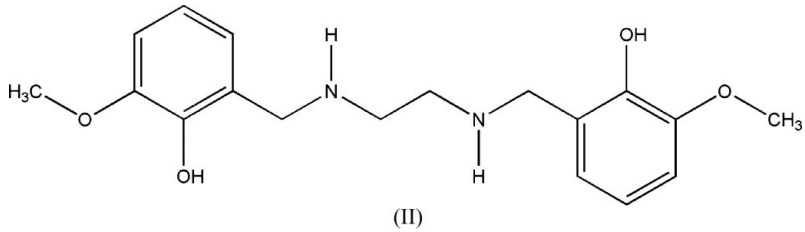
In the title compound, $C_{18}H_{26}N_2O_4^{2+}\cdot 2NO_3^-$, the ions are linked into two chains by C—H···O hydrogen bonds; these chains are linked into sheets parallel to (001). The asymmetric unit consists of one cation, two half-cations and four anions. Two of the cations have a centre of symmetry at the mid-point of the central C—C bond.

Comment

o-Vanillin diamine derivatives are potentially multidentate ligands. During the course of studies on the coordination chemistry of *o*-vanillin ethylenediamine derivatives, we obtained crystals of the title compound, (I); we report here its crystal structure. The asymmetric unit consists of one cation, two half-cations and four anions. Two of the cations have a centre of symmetry at the mid-point of the central C—C bond (Fig. 1). The bond lengths and angles are normal (Allen *et al.*, 1987) and are in agreement with those in three similar compounds (Xia *et al.*, 2006, 2007*a*,*b*). The most closely related compound is 6,6′-dimethoxy-2,2′-(ethane-1,2-diyldiimino-dimethylene)diphenol, (II) (Xia *et al.*, 2006).



(I)



(II)

The principal difference between (I) and (II) concerns the intermolecular aggregation. In (I), the ions are linked into two chains of rings by C—H···O hydrogen bonds. Atoms C7 and C15 in the molecule at (*x*, *y*, *z*) act as hydrogen-bond donors to methoxy atoms O4 in the molecule at $(1-x, 2-y, 1-z)$ and O2 in the molecule at $(1-x, 1-y, 1-z)$, respectively, generating a chain of $R_2^2(26)$ rings (Bernstein *et al.*, 1995) along the *b*-axis direction (Fig. 2 and Table 1). In addition, atoms C27 and C36 in the molecule at (*x*, *y*, *z*) act as donors to

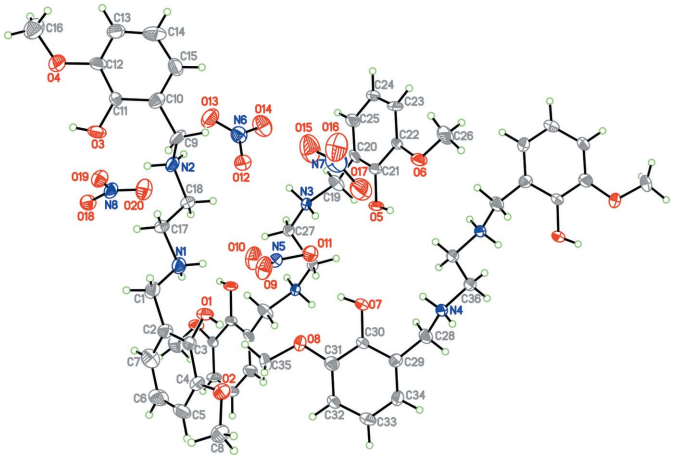


Figure 1
The structure of the independent ions of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms in the N4 cation are related to labelled atoms by ($-x$, $-y$, $1-z$). Unlabelled atoms in the N3 cation are related to labelled atoms by ($-x$, $1-y$, $1-z$).

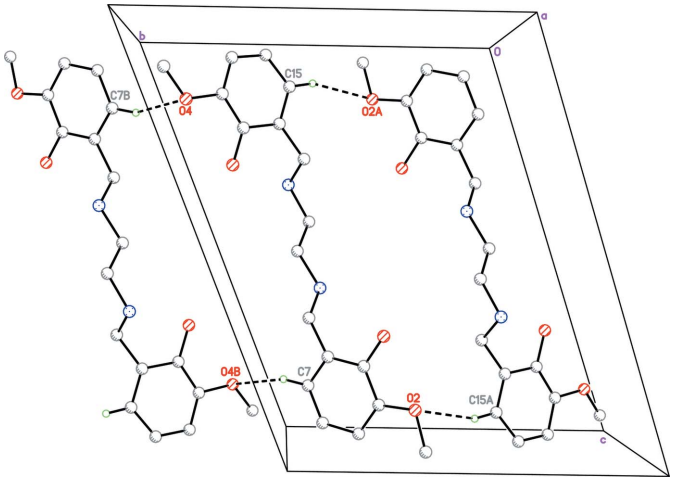


Figure 2
A portion of the crystal structure of (I), showing the formation of a hydrogen-bonded sheet built from C—H···O interactions. For clarity, H atoms not involved in hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (A) $1-x$, $1-y$, $1-z$; (B) $1-x$, $2-y$, $1-z$.]

hydroxy atoms O7 in the molecule at ($-x$, $1-y$, $1-z$) and O5 in the molecule at ($-x$, $-y$, $1-z$), respectively, generating a chain of $R_2^2(16)$ rings along the *b*-axis direction (Fig. 3 and Table 1). The nitrate anions occupy positions between the two chains, linking the ions into a sheet parallel to the (001) plane. There are no other types of intermolecular hydrogen bonds in the structure of (I) and there are no direction-specific interactions between adjacent sheets. By contrast, in (II), the molecules are linked into sheets by means of C—H···O and O—H···N hydrogen bonds; the latter are absent from the structure of (I).

Experimental

To a solution of 6,6′-dimethoxy-2,2′-(ethane-1,2-diyldiimino-dimethylene)diphenol (1 mmol) in methanol (20 ml) was added a solution of zinc(II) nitrate (1 mmol) in methanol (10 ml). The mixed solution was stirred for 2 h and then filtered. The solution was allowed to stand, slowly producing crystals of (I).

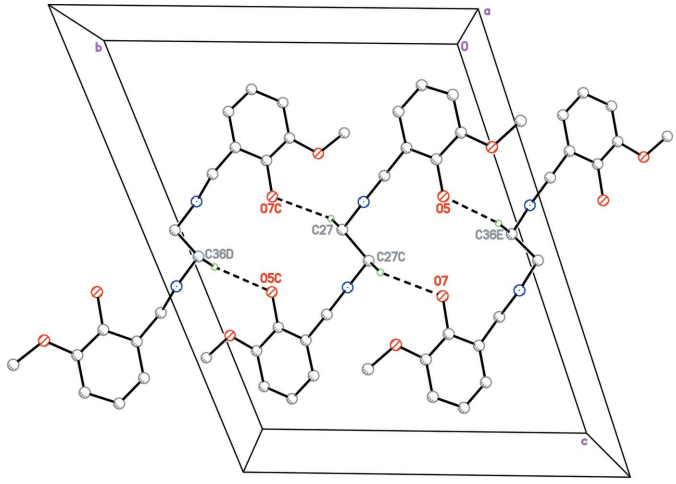


Figure 3
A portion of the crystal structure of (I), showing the formation of a hydrogen-bonded sheet built from C—H···O interactions. For clarity, H atoms not involved in hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (C) $-x$, $1-y$, $1-z$; (D) x, $1+y$, z; (E) $-x$, $-y$, $1-z$.]

Crystal data

$C_{18}H_{26}N_2O_4^{2+}\cdot 2NO_3^-$
M_r = 458.43
Triclinic, $P\overline{1}$
a = 11.799 (11) Å
b = 12.417 (11) Å
c = 16.354 (15) Å
α = 109.127 (12)°
β = 110.774 (12)°
γ = 90.582 (13)°
V = 2095 (3) Å^3
Z = 4
D_x = 1.454 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
Block, colourless
0.38 × 0.19 × 0.14 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.983
9489 measured reflections
6553 independent reflections
2443 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.099
$wR(F^2)$ = 0.289
S = 0.91
6553 reflections
577 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.139P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C36—H36*B*···O5^{i}	0.97	2.56	3.477 (9)	158
C27—H27*A*···O7^{ii}	0.97	2.62	3.473 (9)	147
C15—H15···O2^{iii}	0.93	2.62	3.511 (10)	161
C7—H7*A*···O4^{iv}	0.93	2.62	3.480 (11)	155

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

All H atoms were located in difference Fourier maps and then repositioned geometrically. They were refined as riding, with C—H distances of 0.93 (aryl), 0.96 (methyl) or 0.97 Å (methylene), N—H = 0.90 Å and O—H = 0.82 Å. U_{iso}(H) values were set at xU_{eq}(carrier atom), where x = 1.5 for methyl and hydroxy, x = 1.2 for the other H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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