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N-(*tert*-Butyl)-*N*-[2-hydroxy-3-(2-isopropyl-5-methylphenoxy)propyl]ammonium hydrogen oxalate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.157
Data-to-parameter ratio = 15.7

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

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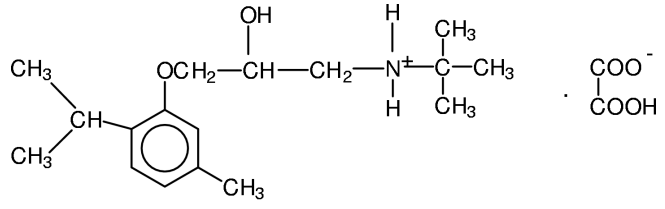
In the crystal of the title compound, $C_{17}H_{30}NO_2^+\cdot C_2HO_4^-$, the centrosymmetrically related cations form dimeric pairs through N—H···O hydrogen bonds. The anions are linked by O—H···O hydrogen bonds to form infinite one-dimensional chains along the *b* axis. The anions and the cations are linked by intermolecular N—H···O and O—H···O hydrogen bonds to form a molecular network.

Comment

The structure determination of the title compound, (I), was undertaken to study the stereochemistry of the molecule, the effect of substituents and the nature of hydrogen bonding. In (I), the oxalic acid H atom has been transferred to the N atom of the amine to form a cation. In the resulting anion, the C21—C24 distance of 1.529 (3) Å is longer than the normal Csp^2—Csp^2 distance and this is commonly observed in the oxalate moiety. The exocyclic angle O7—C1—C2 [114.97 (15)°] deviates significantly from the normal value of 120° because of the intermolecular non-bonded interaction between O7 and H18 (2.35 Å). The O7—C8—C9 angle [104.69 (13)°] deviates by *ca* 5° from the tetrahedral value. This may be due to the intramolecular non-bonded interaction between O7 and H10*A* (2.55 Å). The slight increase in the O7—C1—C6 angle [123.18 (15)°] may be due to the repulsion between H6 and H8*A*.



(I)

In the asymmetric unit, the cation and anion are linked by an O—H···O hydrogen bond involving the hydroxyl group (O16—H16) and the carboxylate atom O26. In the crystal, centrosymmetrically related cations form dimeric pairs through N—H···O hydrogen bonds; the anions are linked by O—H···O hydrogen bonds to form infinite one-dimensional chains along the *b* axis (Table 1). One of the H atoms, H11*B*, of the ammonium group is involved in intermolecular bifurcated N—H···O hydrogen bonds with O23(−1/2+*x*, 3/2−*y*, −1/2+*z*) and O25(−1/2+*x*, 3/2−*y*, −1/2+*z*). The crystal structure is stabilized by a network of N—H···O, O—H···O and C—H···O intermolecular hydrogen bonds.

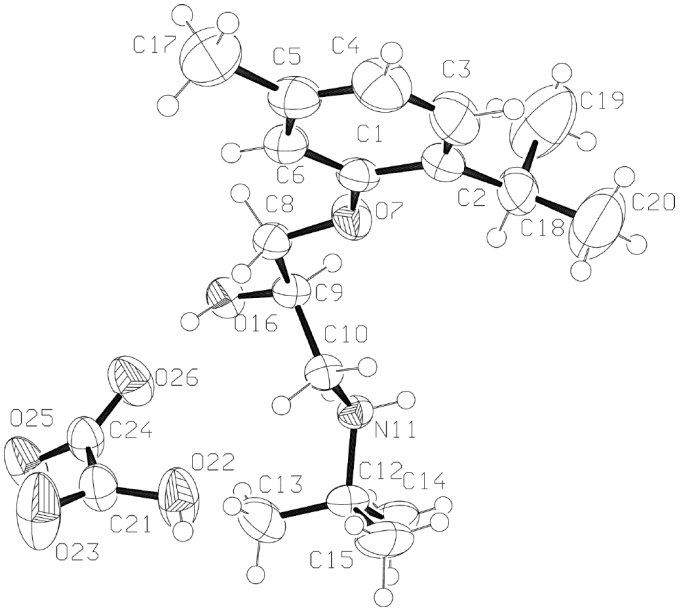


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound was prepared by refluxing a mixture of 1,2-epoxy-3-(2-isopropyl-5-methylphenoxy)propane (2 g, 9.7 m*M*) and *tert*-butylamine (10 ml, 137 m*M*) for 12 h. The completion of the reaction was confirmed by thin-layer chromatography. The excess *tert*-butylamine was removed under reduced pressure to give 1-*tert*-butylamino-3-(2-isopropyl-5-methylphenoxy)propan-2-ol (1.6 g, 59.06%), which could not be crystallized. The salt, (I), was prepared by refluxing 1-*tert*-butylamino-3-(2-isopropyl-5-methylphenoxy)propan-2-ol (2 g, 7.2 m*M*) and oxalic acid (1 g, 7.9 m*M*) in dry acetone (50 ml) for 30 min. The solvent was removed under reduced pressure and the solid residue was crystallized from dry acetone (0.91 g, 34.41%, m.p. 466–469 K).

Crystal data

$C_{17}H_{30}NO_2^+\cdot C_2HO_4^-$
M_r = 369.45
Monoclinic, $P2_1/n$
a = 12.908 (2) Å
b = 10.763 (1) Å
c = 15.784 (2) Å
β = 110.648 (11)°
V = 2052.0 (5) Å^3
Z = 4
D_x = 1.196 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 20–30°
μ = 0.73 mm^{-1}
T = 293 (2) K
Plate, white
0.30 × 0.15 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.812, T_{max} = 0.931
4071 measured reflections
3723 independent reflections
3066 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 67.9°
$h = -15 \rightarrow 14$
$k = 0 \rightarrow 12$
$l = -17 \rightarrow 18$
2 standard reflections
frequency: 120 min
intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.157
S = 1.06
3723 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0912P)^2 + 0.5356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11A···O16^{i}	0.90	2.05	2.913 (2)	159
N11—H11B···O25^{ii}	0.90	1.96	2.809 (2)	157
N11—H11B···O23^{ii}	0.90	2.53	3.047 (2)	117
O16—H16···O26	0.82	1.95	2.731 (2)	159
O22—H22···O25^{iii}	0.82	1.77	2.569 (2)	164
C9—H9···O23^{ii}	0.98	2.47	3.322 (3)	146
C10—H10B···O26	0.97	2.50	3.227 (2)	132

Symmetry codes: (i) $1-x, 2-y, 1-z$; (ii) $x-\frac{1}{2}, \frac{3}{2}-y, z-\frac{1}{2}$; (iii) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97.

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References

Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.