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rac-tert-Butyl{2-hydroxy-2-[4-hydroxy-3-(hydroxymethyl)phenyl]ethyl}azanium acrylate

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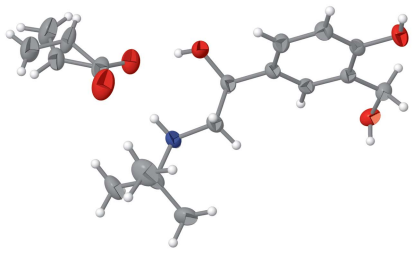
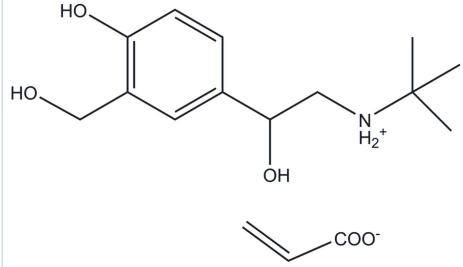
Structural data: full structural data are available from iucrdata.iucr.org

The title salt, $C_{13}H_{22}NO_3^+\cdot C_3H_3O_2^-$, comprises one salbutamol cation and an acrylate anion. The acrylate anion is linked to the salbutamol cation *via* an O—H···O and an N—H···O hydrogen bond. The C=C group of the acrylate anion is disordered over two positions, with refined site occupancies of 0.812 (7) and 0.188 (7). The crystal structure is stabilized by N—H···O and O—H···O hydrogen-bonding interactions.

3D view



Chemical scheme



Structure description

Salbutamol is known as a short-action selective β2-adrenergic receptor agonist for the treatment of pulmonary diseases (Saleh *et al.*, 2000). However, due to its low dissolution in water, salbutamol is usually transformed into its salt form with some acids to improve its solubility. The title compound is a novel salt of salbutamol.

The asymmetric unit of the title compound is shown in Fig. 1. The compound comprises a protonated salbutamol cation and a deprotonated acrylic acid anion which are linked *via* an O—H···O and an N—H···O hydrogen bond, forming an $R_2^2(9)$ motif (Table 1 and Fig. 1). In the crystal, cations and anions are linked by O—H···(O,N) hydrogen bonds, forming a three-dimensional network (Fig. 2).

Synthesis and crystallization

Salbutamol (0.479 g, 2 mmol) was reacted with acrylic acid (0.144 g, 2 mmol) in 10 ml methanol. The mixture was stirred and the methanol was evaporated at room temperature, yielding salbutamol acrylate. After recrystallization of the raw product from ethanol, the crystals were dissolved in ethanol and the solution was filtered. Finally, the solvent was evaporated slowly and single crystals suitable for the diffraction experiment were obtained.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.82	1.98	2.7817 (14)	166
O2—H2···O1^{ii}	0.82	1.96	2.7816 (14)	174
O3—H3···O4	0.82	1.90	2.7194 (15)	179
N1—H1A···O4^{iii}	0.89	1.92	2.8022 (16)	171
N1—H1B···O5	0.89	1.96	2.8292 (18)	166

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

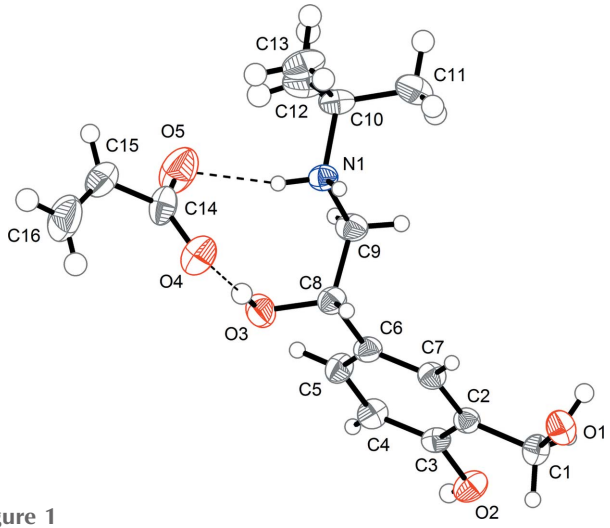


Figure 1
Perspective view of the cation and anion of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The minor-occupied sites of the disordered atoms are not shown.

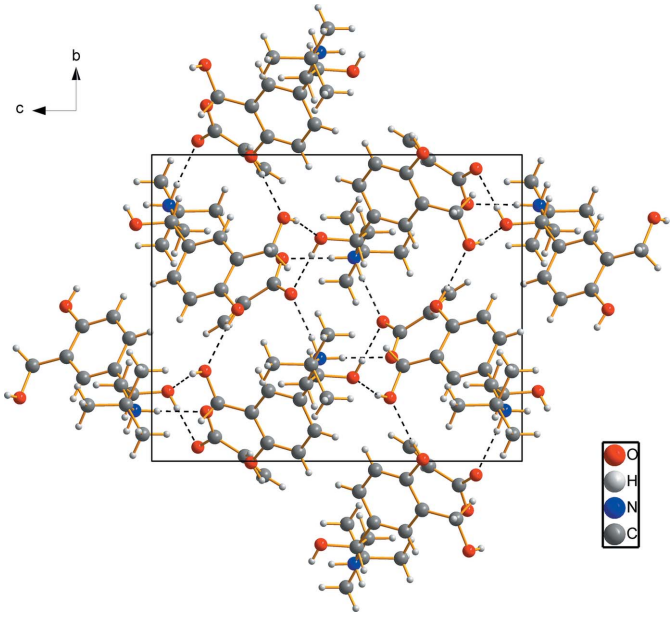


Figure 2
The crystal packing of the title compound, viewed perpendicular to the *bc* plane. N—H···O and O—H···O hydrogen bonds are shown as dashed lines. The minor-occupied sites of the disordered atoms are not shown.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{22}NO_3^+\cdot C_3H_3O_2^-$
M_r	311.37
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	295
a, b, c (Å)	9.5304 (4), 11.9927 (5), 14.5120 (7)
β (°)	91.815 (2)
V (Å^3)	1657.82 (13)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.4 × 0.3 × 0.2
Data collection	
Diffractometer	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.719, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	39441, 3825, 3468
R_{int}	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.651
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.125, 1.04
No. of reflections	3825
No. of parameters	224
No. of restraints	3
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.27

Computer programs: *APEX2* (Bruker, 2015), *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C═C group of the acrylate anion is disordered over two positions, with refined site occupancies of 0.812 (7) and 0.188 (7). Bond lengths and angles involving the disordered atoms were restrained to be equal. H atoms were refined using a riding model, with aromatic C—H = 0.93 Å, methyl C—H = 0.96 Å, methylene C—H = 0.97 Å and tertiary C—H = 0.98 Å. U_{iso}(H) values were set at 1.5U_{eq}(C,O) for methyl and hydroxy groups and at 1.2U_{eq}(C,N) for the remaining atoms. The torsion angles of the hydroxy and methyl groups were allowed to refine.

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full crystallographic data

IUCrData (2017). **2**, x171189 [https://doi.org/10.1107/S2414314617011890]

rac-tert-Butyl{2-hydroxy-2-[4-hydroxy-3-(hydroxymethyl)phenyl]ethyl}azanium acrylate

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Crystal data

$C_{13}H_{22}NO_3^+·C_3H_3O_2^-$
M_r = 311.37
Monoclinic, $P2_1/c$
a = 9.5304 (4) Å
b = 11.9927 (5) Å
c = 14.5120 (7) Å
β = 91.815 (2)°
V = 1657.82 (13) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.248 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9984 reflections
θ = 2.7–27.5°
μ = 0.09 mm^{-1}
T = 295 K
Block, colourless
0.4 × 0.3 × 0.2 mm

Data collection

CCD area detector diffractometer
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
T_{min} = 0.719, T_{max} = 0.746
39441 measured reflections
3825 independent reflections
3468 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.6°, θ_{min} = 3.3°
h = −12→12
k = −15→15
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.125
S = 1.04
3825 reflections
224 parameters
3 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.7417P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.08955 (11)	0.29361 (8)	0.85654 (7)	0.0385 (2)	
H1	0.1603	0.2851	0.8894	0.058*	
O2	−0.03078 (10)	−0.00530 (8)	0.73205 (7)	0.0388 (2)	
H2	−0.0449	−0.0630	0.7028	0.058*	
O3	0.32888 (12)	0.27262 (10)	0.45673 (7)	0.0466 (3)	
H3	0.3638	0.3271	0.4321	0.070*	
O4	0.44223 (13)	0.45453 (9)	0.37554 (8)	0.0500 (3)	
N1	0.60151 (11)	0.33496 (9)	0.54666 (7)	0.0299 (2)	
H1A	0.5788	0.4020	0.5678	0.036*	
H1B	0.5899	0.3369	0.4856	0.036*	
O5	0.61642 (17)	0.33915 (13)	0.35229 (10)	0.0711 (4)	
C3	0.06403 (12)	0.05912 (10)	0.68766 (8)	0.0278 (3)	
C7	0.19586 (13)	0.22911 (11)	0.68475 (9)	0.0295 (3)	
H7	0.2221	0.2964	0.7121	0.035*	
C2	0.10425 (12)	0.15933 (10)	0.73002 (8)	0.0268 (2)	
C6	0.24953 (13)	0.20124 (11)	0.59972 (9)	0.0299 (3)	
C8	0.34961 (13)	0.28039 (11)	0.55336 (9)	0.0322 (3)	
H8	0.3288	0.3568	0.5727	0.039*	
C4	0.11790 (14)	0.02944 (11)	0.60353 (9)	0.0332 (3)	
H4	0.0915	−0.0377	0.5760	0.040*	
C10	0.75646 (13)	0.31554 (12)	0.56937 (11)	0.0372 (3)	
C1	0.04384 (15)	0.18869 (11)	0.82191 (9)	0.0336 (3)	
H1C	−0.0578	0.1891	0.8155	0.040*	
H1D	0.0702	0.1313	0.8663	0.040*	
C5	0.21116 (14)	0.09956 (11)	0.56033 (9)	0.0342 (3)	
H5	0.2484	0.0784	0.5045	0.041*	
C14	0.54440 (18)	0.42130 (13)	0.32981 (10)	0.0425 (3)	
C9	0.49975 (14)	0.25237 (12)	0.58317 (10)	0.0354 (3)	
H9A	0.5077	0.2514	0.6500	0.042*	
H9B	0.5230	0.1785	0.5611	0.042*	
C13	0.83340 (17)	0.41218 (16)	0.52486 (14)	0.0549 (4)	
H13A	0.8128	0.4124	0.4597	0.082*	
H13B	0.9327	0.4036	0.5358	0.082*	
H13C	0.8031	0.4813	0.5511	0.082*	
C12	0.8007 (2)	0.20572 (16)	0.52759 (16)	0.0609 (5)	
H12A	0.7579	0.1453	0.5598	0.091*	
H12B	0.9009	0.1988	0.5327	0.091*	
H12C	0.7711	0.2035	0.4638	0.091*	
C11	0.78183 (17)	0.31788 (17)	0.67316 (12)	0.0541 (4)	
H11A	0.7478	0.3870	0.6973	0.081*	
H11B	0.8806	0.3113	0.6872	0.081*	
H11C	0.7331	0.2568	0.7006	0.081*	
C15	0.5977 (3)	0.4841 (2)	0.24830 (16)	0.0471 (7)	0.812 (7)
H15	0.6919	0.4772	0.2345	0.057*	0.812 (7)
C16	0.5186 (4)	0.5469 (3)	0.19678 (19)	0.0701 (9)	0.812 (7)

H16A	0.4241	0.5550	0.2093	0.084*	0.812 (7)
H16B	0.5561	0.5842	0.1470	0.084*	0.812 (7)
C15A	0.5151 (15)	0.4941 (9)	0.2405 (6)	0.053 (3)	0.188 (7)
H15A	0.4254	0.5194	0.2243	0.064*	0.188 (7)
C16A	0.6210 (14)	0.5165 (11)	0.1918 (8)	0.069 (4)	0.188 (7)
H16C	0.7095	0.4902	0.2096	0.083*	0.188 (7)
H16D	0.6094	0.5591	0.1385	0.083*	0.188 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0476 (6)	0.0325 (5)	0.0349 (5)	0.0089 (4)	−0.0042 (4)	−0.0048 (4)
O2	0.0392 (5)	0.0306 (5)	0.0473 (6)	−0.0095 (4)	0.0115 (4)	0.0010 (4)
O3	0.0444 (6)	0.0602 (7)	0.0349 (5)	−0.0197 (5)	−0.0041 (4)	0.0119 (5)
O4	0.0610 (7)	0.0387 (6)	0.0514 (6)	0.0049 (5)	0.0162 (5)	0.0048 (5)
N1	0.0262 (5)	0.0284 (5)	0.0351 (5)	−0.0015 (4)	0.0023 (4)	0.0035 (4)
O5	0.0847 (10)	0.0674 (9)	0.0627 (8)	0.0246 (8)	0.0264 (7)	0.0068 (7)
C3	0.0244 (5)	0.0245 (5)	0.0345 (6)	0.0000 (4)	0.0020 (4)	0.0036 (5)
C7	0.0291 (6)	0.0276 (6)	0.0317 (6)	−0.0040 (5)	−0.0005 (5)	−0.0014 (5)
C2	0.0255 (5)	0.0270 (6)	0.0280 (6)	0.0019 (4)	−0.0003 (4)	0.0017 (4)
C6	0.0255 (5)	0.0315 (6)	0.0328 (6)	−0.0028 (5)	0.0015 (4)	0.0029 (5)
C8	0.0300 (6)	0.0328 (6)	0.0338 (6)	−0.0043 (5)	0.0027 (5)	0.0038 (5)
C4	0.0361 (6)	0.0258 (6)	0.0378 (7)	−0.0017 (5)	0.0033 (5)	−0.0043 (5)
C10	0.0246 (6)	0.0370 (7)	0.0503 (8)	0.0012 (5)	0.0041 (5)	−0.0020 (6)
C1	0.0387 (7)	0.0324 (6)	0.0299 (6)	0.0015 (5)	0.0039 (5)	0.0014 (5)
C5	0.0359 (6)	0.0347 (7)	0.0322 (6)	−0.0006 (5)	0.0069 (5)	−0.0035 (5)
C14	0.0602 (9)	0.0360 (7)	0.0318 (7)	−0.0045 (6)	0.0101 (6)	−0.0046 (5)
C9	0.0301 (6)	0.0338 (7)	0.0421 (7)	−0.0056 (5)	−0.0003 (5)	0.0092 (5)
C13	0.0365 (8)	0.0535 (10)	0.0755 (12)	−0.0114 (7)	0.0156 (8)	0.0007 (8)
C12	0.0475 (9)	0.0467 (9)	0.0888 (14)	0.0142 (7)	0.0091 (9)	−0.0114 (9)
C11	0.0361 (8)	0.0702 (11)	0.0554 (10)	0.0000 (8)	−0.0105 (7)	0.0037 (8)
C15	0.0484 (14)	0.0491 (12)	0.0446 (13)	−0.0059 (11)	0.0156 (10)	−0.0001 (9)
C16	0.086 (2)	0.0770 (18)	0.0488 (14)	0.0114 (15)	0.0205 (13)	0.0207 (14)
C15A	0.067 (8)	0.059 (6)	0.034 (5)	0.020 (5)	0.023 (5)	0.015 (4)
C16A	0.078 (9)	0.075 (8)	0.055 (7)	−0.019 (7)	0.009 (6)	0.019 (6)

Geometric parameters (Å, º)

O1—H1	0.8200	C10—C11	1.518 (2)
O1—C1	1.4183 (16)	C1—H1C	0.9700
O2—H2	0.8200	C1—H1D	0.9700
O2—C3	1.3656 (14)	C5—H5	0.9300
O3—H3	0.8200	C14—C15	1.504 (3)
O3—C8	1.4130 (16)	C14—C15A	1.580 (10)
O4—C14	1.2602 (19)	C9—H9A	0.9700
N1—H1A	0.8900	C9—H9B	0.9700
N1—H1B	0.8900	C13—H13A	0.9600
N1—C10	1.5208 (16)	C13—H13B	0.9600

N1—C9	1.4950 (16)	C13—H13C	0.9600
O5—C14	1.238 (2)	C12—H12A	0.9600
C3—C2	1.3979 (17)	C12—H12B	0.9600
C3—C4	1.3858 (18)	C12—H12C	0.9600
C7—H7	0.9300	C11—H11A	0.9600
C7—C2	1.3895 (17)	C11—H11B	0.9600
C7—C6	1.3914 (18)	C11—H11C	0.9600
C2—C1	1.5108 (17)	C15—H15	0.9300
C6—C8	1.5179 (17)	C15—C16	1.288 (4)
C6—C5	1.3908 (18)	C16—H16A	0.9300
C8—H8	0.9800	C16—H16B	0.9300
C8—C9	1.5193 (18)	C15A—H15A	0.9300
C4—H4	0.9300	C15A—C16A	1.279 (14)
C4—C5	1.3876 (19)	C16A—H16C	0.9300
C10—C13	1.526 (2)	C16A—H16D	0.9300
C10—C12	1.515 (2)		

C1—O1—H1	109.5	C4—C5—C6	120.62 (12)
C3—O2—H2	109.5	C4—C5—H5	119.7
C8—O3—H3	109.5	O4—C14—C15	122.80 (17)
H1A—N1—H1B	107.2	O4—C14—C15A	97.9 (4)
C10—N1—H1A	108.0	O5—C14—O4	122.95 (14)
C10—N1—H1B	108.0	O5—C14—C15	114.02 (17)
C9—N1—H1A	108.0	O5—C14—C15A	137.5 (4)
C9—N1—H1B	108.0	N1—C9—C8	111.61 (11)
C9—N1—C10	117.27 (10)	N1—C9—H9A	109.3
O2—C3—C2	117.08 (11)	N1—C9—H9B	109.3
O2—C3—C4	122.65 (11)	C8—C9—H9A	109.3
C4—C3—C2	120.27 (11)	C8—C9—H9B	109.3
C2—C7—H7	119.0	H9A—C9—H9B	108.0
C2—C7—C6	121.92 (11)	C10—C13—H13A	109.5
C6—C7—H7	119.0	C10—C13—H13B	109.5
C3—C2—C1	118.80 (11)	C10—C13—H13C	109.5
C7—C2—C3	118.53 (11)	H13A—C13—H13B	109.5
C7—C2—C1	122.66 (11)	H13A—C13—H13C	109.5
C7—C6—C8	119.78 (11)	H13B—C13—H13C	109.5
C5—C6—C7	118.42 (11)	C10—C12—H12A	109.5
C5—C6—C8	121.79 (11)	C10—C12—H12B	109.5
O3—C8—C6	109.18 (10)	C10—C12—H12C	109.5
O3—C8—H8	108.8	H12A—C12—H12B	109.5
O3—C8—C9	111.64 (11)	H12A—C12—H12C	109.5
C6—C8—H8	108.8	H12B—C12—H12C	109.5
C6—C8—C9	109.56 (11)	C10—C11—H11A	109.5
C9—C8—H8	108.8	C10—C11—H11B	109.5
C3—C4—H4	119.9	C10—C11—H11C	109.5
C3—C4—C5	120.19 (12)	H11A—C11—H11B	109.5
C5—C4—H4	119.9	H11A—C11—H11C	109.5
N1—C10—C13	105.57 (12)	H11B—C11—H11C	109.5

C12—C10—N1	109.07 (12)	C14—C15—H15	118.5
C12—C10—C13	110.33 (14)	C16—C15—C14	122.9 (3)
C12—C10—C11	112.05 (15)	C16—C15—H15	118.5
C11—C10—N1	109.59 (11)	C15—C16—H16A	120.0
C11—C10—C13	110.03 (14)	C15—C16—H16B	120.0
O1—C1—C2	113.46 (11)	H16A—C16—H16B	120.0
O1—C1—H1C	108.9	C14—C15A—H15A	121.7
O1—C1—H1D	108.9	C16A—C15A—C14	116.6 (12)
C2—C1—H1C	108.9	C16A—C15A—H15A	121.7
C2—C1—H1D	108.9	C15A—C16A—H16C	120.0
H1C—C1—H1D	107.7	C15A—C16A—H16D	120.0
C6—C5—H5	119.7	H16C—C16A—H16D	120.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{i}	0.82	1.98	2.7817 (14)	166
O2—H2···O1^{ii}	0.82	1.96	2.7816 (14)	174
O3—H3···O4	0.82	1.90	2.7194 (15)	179
N1—H1*A*···O4^{iii}	0.89	1.92	2.8022 (16)	171
N1—H1*B*···O5	0.89	1.96	2.8292 (18)	166

Symmetry codes: (i) x, $-y+1/2$, $z+1/2$; (ii) $-x$, $y-1/2$, $-z+3/2$; (iii) $-x+1$, $-y+1$, $-z+1$.