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2-Methylpropan-2-aminium 2-(methoxycarbonyl)benzoate

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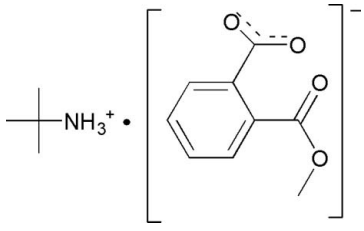
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; some non-H atoms missing; disorder in main residue; R factor = 0.047; wR factor = 0.137; data-to-parameter ratio = 11.8.

In the title compound, $C_4H_{12}N^+\cdot C_9H_7O_4^-$, two C atoms and the N atom of the cation lie on a mirror plane, while all the atoms of the anion are disordered about a mirror plane. In the crystal, N—H···O hydrogen bonds link the components into chains along [010]. In the anion, the mean planes of the methoxycarbonyl and carboxylate groups form dihedral angles of 83.0 (2) and 83.2 (2)°, respectively, with the aromatic ring.

Related literature

For the applications of phthalimides and *N*-substituted phthalimides, see: Lima *et al.* (2002). For related structures, see: Li (2011); Liang (2011).



Experimental

Crystal data

$C_4H_{12}N^+\cdot C_9H_7O_4^-$
M_r = 253.29
Monoclinic, $P2_1/m$
a = 9.2939 (8) Å
b = 7.0159 (6) Å
c = 10.5536 (11) Å
β = 103.322 (1)°
V = 669.63 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.49 × 0.43 × 0.32 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.956, T_{max} = 0.971
4353 measured reflections
1797 independent reflections
1137 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
S = 1.03
1797 reflections
152 parameters
14 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2*N*···O4	0.925 (18)	1.749 (19)	2.674 (3)	178.3 (17)
N1—H2*N*···O3^{i}	0.925 (18)	2.042 (18)	2.926 (3)	159.4 (16)
N1—H1*N*···O3^{ii}	0.92 (3)	1.96 (2)	2.825 (3)	156 (1)
N1—H1*N*···O3^{iii}	0.92 (3)	1.96 (2)	2.825 (3)	156 (1)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5313).

References

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supplementary materials

Acta Cryst. (2011). E**67**, o2705 [**doi:10.1107/S1600536811037688**]

2-Methylpropan-2-aminium 2-(methoxycarbonyl)benzoate

J. Li

Comment

Phthalimides and N-substituted phthalimides are animportant class of compounds because of their interesting biological activities (Lima *et al.*, 2002). Tert-butylaminium 2-(methoxycarbonyl)benzoate is an intermediate in the preparation of N-substituted phthalimides. The crystal structures of propan-1-aminium 3,4,5,6-tetrabromo-2-(methoxycarbonyl)benzoate *N*,*N*-dimethylformamide monosolvate (Li, 2011) and butane-1,4-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate] (Liang, 2011) have already been reported. In this paper, the structure of the title compound is reported. The asymmetric unit of the title compound, 2-methylpropan-2-aminium 2-(methoxycarbonyl)benzoate, (I), is shown in Fig. 1. Atoms C11 and C11a of the cation lie symmetrically on a mirror plane (C10C12N1) while all the atoms of the anion are disordered over a mirror plane. In the crystal, N—H···O hydrogen bonds link the components into one-dimensional chains along [010].

Experimental

A mixture of phthalic anhydride (1.52 g, 0.01 mol) and methanol (15 ml) was refluxed for 30 min. Then *tert*-butylamine (0.73 g, 0.01 mol) was added to the above solution and mixed for 30 min at room temperature. The solution was kept at room temperature for 5 d. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis.

Refinement

H atoms bonded to C atoms were placed in calculated positions and refined in a riding-model approximation with C—H = 0.93-0.96 Å and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}). H atoms bonded to N were refined independently with isotropic displacement parameters.

Figures

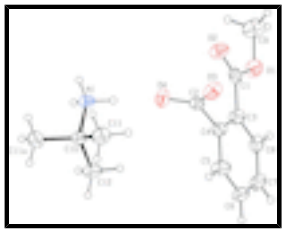

Fig. 1. The asymmetric unit of (I), drawn with 30% probability ellipsoids. The disorder is not shown (symmetry code (a): x, -y+3/2, z).

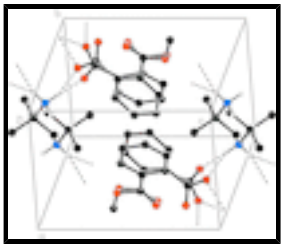

Fig. 2. Part of the crystal structure of the title compound with hydrogen bonds shown as dashed lines.

2-Methylpropan-2-aminium 2-(methoxycarbonyl)benzoate

Crystal data

$C_4H_{12}N^+·C_9H_7O_4^-$	$F(000) = 272$
$M_r = 253.29$	$D_x = 1.256$ Mg m^{-3}
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yb	Cell parameters from 1362 reflections
$a = 9.2939$ (8) Å	$\theta = 2.6$–$25.8°$
$b = 7.0159$ (6) Å	$\mu = 0.09$ mm^{-1}
$c = 10.5536$ (11) Å	$T = 298$ K
$\beta = 103.322$ (1)°	Block, colorless
$V = 669.63$ (11) Å^3	0.49 × 0.43 × 0.32 mm
$Z = 2$	

Data collection

Bruker SMART CCD diffractometer	1797 independent reflections
Radiation source: fine-focus sealed tube	1137 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.021$
φ and ω scans	$\theta_{max} = 28.4°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$h = -12\rightarrow11$
$T_{min} = 0.956$, $T_{max} = 0.971$	$k = -9\rightarrow9$
4353 measured reflections	$l = -6\rightarrow14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.047$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.137$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.1129P]$ where $P = (F_o^2 + 2F_c^2)/3$
1797 reflections	$(\Delta/\sigma)_{max} < 0.001$
152 parameters	$\Delta\rho_{max} = 0.16$ e Å^{-3}

14 restraints $\Delta\rho_{min} = -0.17$ 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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.8688 (2)	0.2267 (9)	0.39796 (18)	0.0599 (11)	0.50
O2	0.9747 (2)	0.2711 (10)	0.60624 (19)	0.0596 (10)	0.50
O3	0.8655 (3)	0.1366 (4)	0.8604 (3)	0.0541 (7)	0.50
O4	0.8413 (3)	0.4448 (4)	0.8097 (3)	0.0544 (7)	0.50
C1	0.8655 (3)	0.2433 (7)	0.5222 (2)	0.0440 (7)	0.50
C2	0.8117 (3)	0.2730 (9)	0.7889 (2)	0.0366 (9)	0.50
C3	0.7136 (3)	0.2236 (5)	0.5430 (2)	0.0470 (9)	0.50
C4	0.6905 (3)	0.2377 (6)	0.6689 (2)	0.0407 (6)	0.50
C5	0.5479 (3)	0.2184 (7)	0.6863 (3)	0.0574 (13)	0.50
H5A	0.5315	0.2273	0.7698	0.069*	0.50
C6	0.4291 (3)	0.1862 (4)	0.5818 (3)	0.0547 (9)	0.50
H6A	0.3342	0.1727	0.5955	0.066*	0.50
C7	0.4520 (4)	0.1743 (4)	0.4584 (3)	0.0566 (9)	0.50
H7A	0.3725	0.1551	0.3879	0.068*	0.50
C8	0.5922 (3)	0.1906 (3)	0.4389 (3)	0.0483 (8)	0.50
H8A	0.6070	0.1797	0.3551	0.058*	0.50
C9	1.0136 (3)	0.2437 (12)	0.3673 (3)	0.0716 (11)	0.5
H9A	1.0025	0.2370	0.2747	0.107*	0.50
H9B	1.0762	0.1417	0.4083	0.107*	0.50
H9C	1.0573	0.3637	0.3989	0.107*	0.50
N1	0.87003 (19)	0.7500	0.96516 (19)	0.0428 (5)	
C10	0.7457 (2)	0.7500	1.0343 (2)	0.0417 (5)	
C11	0.76193 (19)	0.5723 (3)	1.11787 (18)	0.0606 (5)	
H11A	0.7587	0.4617	1.0637	0.091*	
H11B	0.6826	0.5666	1.1621	0.091*	
H11C	0.8547	0.5761	1.1808	0.091*	
C12	0.6025 (2)	0.7500	0.9300 (2)	0.0601 (7)	
H12A	0.5986	0.8615	0.8766	0.090*	0.50
H12B	0.5200	0.7504	0.9706	0.090*	
H12C	0.5983	0.6381	0.8769	0.090*	0.50
H2N	0.8597 (18)	0.643 (2)	0.9127 (18)	0.060 (5)*	

H1N	0.960 (3)	0.7500	1.024 (3)	0.060 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0603 (11)	0.080 (3)	0.0415 (9)	−0.011 (2)	0.0155 (8)	−0.014 (2)
O2	0.0444 (9)	0.088 (3)	0.0444 (9)	−0.012 (2)	0.0060 (7)	−0.006 (2)
O3	0.0524 (14)	0.0535 (17)	0.0458 (16)	−0.0050 (13)	−0.0106 (12)	0.0165 (13)
O4	0.0625 (17)	0.0448 (16)	0.0515 (17)	−0.0075 (12)	0.0042 (13)	−0.0091 (12)
C1	0.0504 (12)	0.0467 (15)	0.0338 (12)	0.006 (5)	0.0073 (10)	−0.014 (4)
C2	0.0340 (9)	0.042 (2)	0.0338 (10)	0.0011 (13)	0.0088 (8)	0.0018 (14)
C3	0.0417 (12)	0.059 (3)	0.0369 (12)	0.0012 (18)	0.0015 (9)	−0.0022 (19)
C4	0.0364 (10)	0.0458 (15)	0.0364 (11)	0.005 (3)	0.0014 (8)	0.001 (3)
C5	0.0396 (12)	0.086 (4)	0.0453 (13)	−0.001 (2)	0.0064 (10)	0.001 (2)
C6	0.0357 (14)	0.056 (2)	0.068 (2)	−0.0041 (11)	0.0018 (14)	−0.0015 (14)
C7	0.0465 (16)	0.059 (2)	0.0528 (18)	−0.0036 (13)	−0.0126 (14)	−0.0040 (14)
C8	0.0556 (17)	0.046 (2)	0.0366 (14)	−0.0011 (12)	−0.0028 (12)	−0.0038 (11)
C9	0.0723 (18)	0.086 (3)	0.0611 (19)	−0.015 (5)	0.0253 (15)	0.032 (5)
N1	0.0340 (9)	0.0543 (12)	0.0378 (10)	0.000	0.0037 (7)	0.000
C10	0.0327 (10)	0.0499 (12)	0.0418 (11)	0.000	0.0069 (8)	0.000
C11	0.0581 (10)	0.0617 (11)	0.0619 (10)	−0.0050 (8)	0.0137 (8)	0.0120 (9)
C12	0.0361 (11)	0.0756 (17)	0.0636 (15)	0.000	0.0010 (11)	0.000

Geometric parameters (Å, °)

O1—C1	1.323 (3)	C8—H8A	0.9300
O1—C9	1.460 (3)	C9—H9A	0.9600
O2—C1	1.200 (3)	C9—H9B	0.9600
O3—C2	1.249 (6)	C9—H9C	0.9600
O4—C2	1.244 (7)	N1—C10	1.501 (3)
C1—C3	1.484 (4)	N1—H2N	0.925 (18)
C2—C4	1.509 (3)	N1—H1N	0.92 (3)
C3—C4	1.397 (4)	C10—C11^{i}	1.515 (2)
C3—C8	1.400 (4)	C10—C11	1.515 (2)
C4—C5	1.386 (4)	C10—C12	1.519 (3)
C5—C6	1.388 (4)	C11—H11A	0.9600
C5—H5A	0.9300	C11—H11B	0.9600
C6—C7	1.370 (5)	C11—H11C	0.9600
C6—H6A	0.9300	C12—H12A	0.9600
C7—C8	1.369 (5)	C12—H12B	0.9600
C7—H7A	0.9300	C12—H12C	0.9600
C1—O1—C9	116.4 (2)	O1—C9—H9B	109.5
O2—C1—O1	122.5 (2)	H9A—C9—H9B	109.5
O2—C1—C3	125.3 (2)	O1—C9—H9C	109.5
O1—C1—C3	112.2 (2)	H9A—C9—H9C	109.5
O4—C2—O3	126.5 (4)	H9B—C9—H9C	109.5
O4—C2—C4	113.5 (3)	C10—N1—H2N	107.8 (11)
O3—C2—C4	119.9 (3)	C10—N1—H1N	110.8 (15)

C4—C3—C8	119.0 (3)	H2N—N1—H1N	110.9 (13)
C4—C3—C1	119.6 (2)	N1—C10—C11^{i}	107.38 (11)
C8—C3—C1	121.4 (2)	N1—C10—C11	107.38 (11)
C5—C4—C3	118.7 (2)	C11^{i}—C10—C11	110.77 (19)
C5—C4—C2	117.2 (2)	N1—C10—C12	106.99 (18)
C3—C4—C2	124.1 (2)	C11^{i}—C10—C12	112.01 (11)
C4—C5—C6	121.4 (3)	C11—C10—C12	112.01 (11)
C4—C5—H5A	119.3	C10—C11—H11A	109.5
C6—C5—H5A	119.3	C10—C11—H11B	109.5
C7—C6—C5	119.7 (3)	H11A—C11—H11B	109.5
C7—C6—H6A	120.1	C10—C11—H11C	109.5
C5—C6—H6A	120.1	H11A—C11—H11C	109.5
C8—C7—C6	120.0 (3)	H11B—C11—H11C	109.5
C8—C7—H7A	120.0	C10—C12—H12A	109.5
C6—C7—H7A	120.0	C10—C12—H12B	109.5
C7—C8—C3	121.2 (3)	H12A—C12—H12B	109.5
C7—C8—H8A	119.4	C10—C12—H12C	109.5
C3—C8—H8A	119.4	H12A—C12—H12C	109.5
O1—C9—H9A	109.5	H12B—C12—H12C	109.5

Symmetry codes: (i) *x*, −*y*+3/2, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2N···O4	0.925 (18)	1.749 (19)	2.674 (3)	178.3 (17)
N1—H2N···O3^{ii}	0.925 (18)	2.042 (18)	2.926 (3)	159.4 (16)
N1—H1N···O3^{iii}	0.92 (3)	1.96 (2)	2.825 (3)	156.(1)
N1—H1N···O3^{iv}	0.92 (3)	1.96 (2)	2.825 (3)	156.(1)

Symmetry codes: (ii) *x*, −*y*+1/2, *z*; (iii) −*x*+2, −*y*+1, −*z*+2; (iv) −*x*+2, *y*+1/2, −*z*+2.

Fig. 1

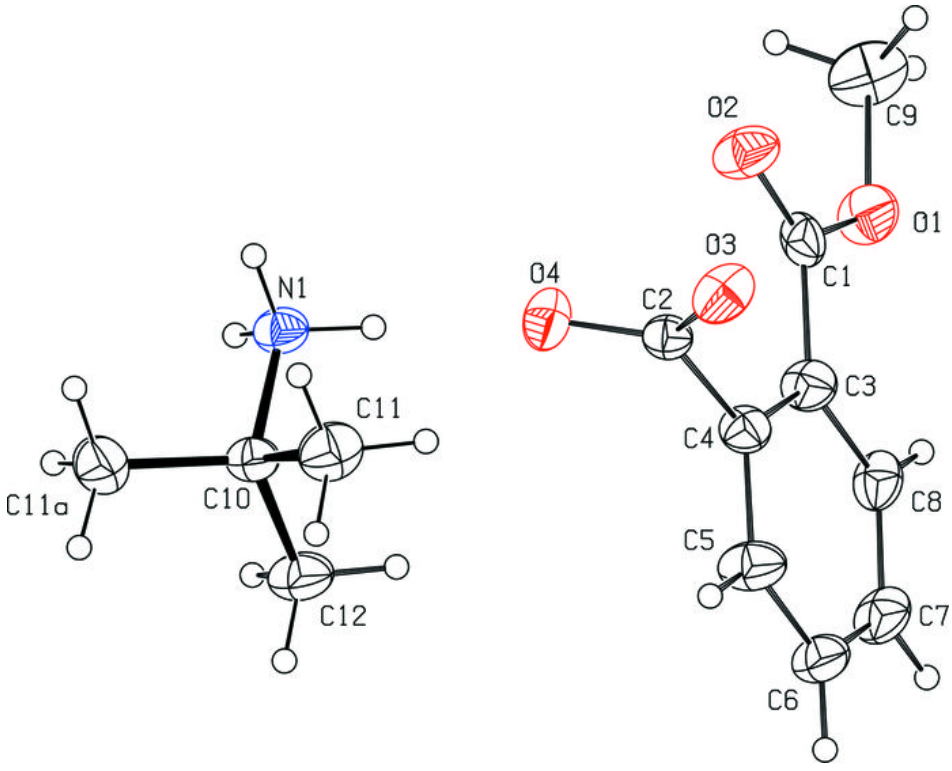

Fig. 2