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(2*SR*,3*RS*)-Benzyl[4-chloro-1-(4-chlorophenyl)-1-methoxycarbonyl-2-butyl]ammonium chloride

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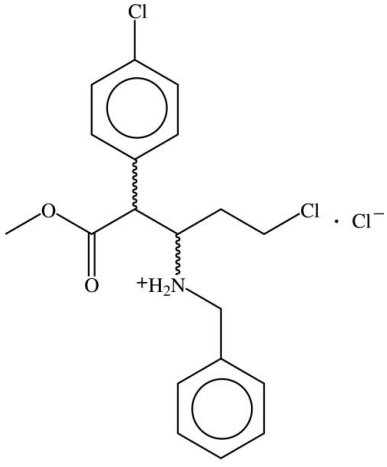
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.159; data-to-parameter ratio = 19.5.

In the racemic hydrochloride salt of the title ester, $C_{19}H_{22}Cl_2NO_2^+\cdot Cl^-$, the pentanoic acid chain shows a mixture of *trans* and *gauche* orientations to give an overall helical conformation. The dihedral angle between the two aromatic rings is 26.11 (10)°. The charged secondary amine function participates in two N—H···Cl hydrogen bonds.

Related literature

For a related structure, see: Froimowitz *et al.* (1998).



Experimental

Crystal data

$C_{19}H_{22}Cl_2NO_2^+\cdot Cl^-$
M_r = 402.73
Triclinic, $P\overline{1}$
a = 9.263 (2) Å
b = 10.432 (3) Å
c = 11.490 (3) Å
α = 115.954 (3)°
β = 93.925 (3)°
γ = 103.015 (3)°
V = 954.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 296 (2) K
0.60 × 0.38 × 0.18 mm

Data collection

Bruker APEX II CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.782, T_{max} = 0.915
8767 measured reflections
4422 independent reflections
3320 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.159
S = 1.03
4422 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2···Cl3	0.90	2.28	3.0890 (18)	150
N1—H1···Cl3^{i}	0.90	2.59	3.2447 (18)	131

Symmetry code: (i) $-x, -y+1, -z$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2801).

References

Bruker (2007). *APEX2* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Froimowitz, M., Wu, K.-M., George, C., VanDerveer, D., Shi, Q. & Deutsch, H. M. (1998). *Struct. Chem.* **9**, 295–303.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2008). E**64**, o2038 [**doi:10.1107/S1600536808030742**]

(2*SR*,3*RS*)-Benzyl[4-chloro-1-(4-chlorophenyl)-1-methoxycarbonyl-2-butyl]ammonium chloride

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Comment

The title molecule, (I), is a β-amino acid ester resulting from a rhodium-catalyzed C—H insertion reaction between methyl-(4-chlorophenyl)diazo acetate and Boc-protected *N*-benzyl-3-chloropropanamine. Full details will be published elsewhere.

Experimental

Colourless blocks of (I) were grown by diffusion of diethylether into 100 µl of a solution containing about 5 mg of the title compound in methanol.

Refinement

The H atoms were positioned with idealized geometry (N–H = 0.90 Å, C–H = 0.93-0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(methyl C).

Figures

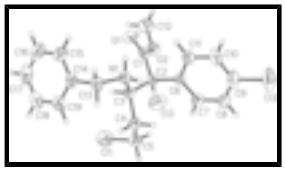

Fig. 1. : The molecular structure of (I). Displacement ellipsoids are shown at the 50% probability level and H-atoms are shown as spheres of arbitrary size.

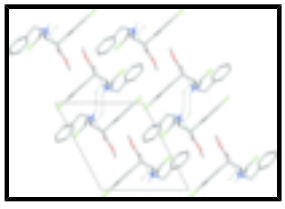

Fig. 2. : Molecular packing and unit cell of (I) viewed along the *a* axis. Hydrogen bonding is indicated by dashed lines, H-atoms bonded to C have been omitted for clarity.

(2SR,3RS)-Benzyl[4-chloro-1-(4-chlorophenyl)-1-methoxycarbonyl- 2-butyl]ammonium chloride

Crystal data

$C_{19}H_{22}Cl_2NO_2^+\cdot Cl^-$	$Z = 2$
$M_r = 402.73$	$F_{000} = 420$
Triclinic, $P\bar{1}$	$D_x = 1.401$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.263$ (2) Å	Cell parameters from 3509 reflections
$b = 10.432$ (3) Å	$\theta = 2.0$–$28.7°$

c = 11.490 (3) Å	μ = 0.49 mm^{-1}
α = 115.954 (3)º	T = 296 (2) K
β = 93.925 (3)º	Block, colourless
γ = 103.015 (3)º	0.60 × 0.38 × 0.18 mm
V = 954.8 (4) Å^3	

Data collection

Bruker APEXII CCD diffractometer	4422 independent reflections
Radiation source: fine-focus sealed tube	3320 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
Detector resolution: 8.3 pixels mm^{-1}	θ_{max} = 28.7º
T = 296(2) K	θ_{min} = 2.0º
sets of exposures each taken over 0.5° ω rotation scans	h = −12→10
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	k = −13→12
T_{min} = 0.782, T_{max} = 0.915	l = −15→15
8767 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H-atom parameters constrained
$wR(F^2)$ = 0.159	$w = 1/[\sigma^2(F_o^2) + (0.097P)^2 + 0.1562P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.002
4422 reflections	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
227 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Crystal grew over 48 h.

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

Refinement. Data were collected by measuring three sets of exposures with the detector set at 2θ = 29°, crystal-to-detector distance 6.00 cm. Refinement of F^2 against ALL reflections.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.64470 (8)	0.98821 (8)	0.38456 (7)	0.0691 (2)
Cl2	0.26825 (11)	−0.01534 (7)	−0.06864 (6)	0.0787 (3)
Cl3	0.17315 (6)	0.47020 (7)	−0.12520 (5)	0.05352 (19)
O1	0.11480 (18)	0.6582 (2)	0.40412 (15)	0.0512 (4)
O2	0.28993 (18)	0.62358 (19)	0.52192 (14)	0.0473 (4)
N1	0.18786 (18)	0.67376 (17)	0.17013 (15)	0.0330 (3)
H1	0.1142	0.6505	0.2107	0.040*
H2	0.1838	0.5903	0.0966	0.040*
C1	0.2347 (2)	0.6371 (2)	0.41927 (17)	0.0346 (4)
C2	0.3435 (2)	0.6218 (2)	0.32432 (17)	0.0321 (4)
H21	0.4452	0.6583	0.3773	0.039*
C3	0.3372 (2)	0.7230 (2)	0.25943 (17)	0.0314 (4)
H31	0.3497	0.8237	0.3299	0.038*
C4	0.4634 (2)	0.7305 (2)	0.18192 (19)	0.0377 (4)
H41	0.4571	0.6302	0.1166	0.045*
H42	0.4464	0.7852	0.1349	0.045*
C5	0.6210 (2)	0.8022 (3)	0.2633 (2)	0.0457 (5)
H51	0.6930	0.8014	0.2058	0.055*
H52	0.6418	0.7446	0.3064	0.055*
C6	0.3211 (2)	0.4594 (2)	0.22692 (18)	0.0338 (4)
C7	0.4464 (2)	0.4091 (3)	0.1945 (2)	0.0431 (5)
H71	0.5425	0.4744	0.2344	0.052*
C8	0.4308 (3)	0.2642 (3)	0.1046 (2)	0.0507 (6)
H81	0.5154	0.2315	0.0833	0.061*
C9	0.2888 (3)	0.1685 (2)	0.0466 (2)	0.0495 (6)
C10	0.1623 (3)	0.2137 (3)	0.0776 (2)	0.0494 (5)
H101	0.0668	0.1469	0.0380	0.059*
C11	0.1778 (2)	0.3591 (2)	0.1681 (2)	0.0423 (5)
H111	0.0925	0.3903	0.1899	0.051*
C12	0.1968 (3)	0.6398 (3)	0.6215 (2)	0.0590 (7)
H121	0.2542	0.6480	0.6984	0.088*
H122	0.1100	0.5545	0.5872	0.088*
H123	0.1646	0.7278	0.6445	0.088*
C13	0.1553 (3)	0.7857 (2)	0.1302 (2)	0.0416 (5)
H131	0.2240	0.7983	0.0735	0.050*
H132	0.0535	0.7461	0.0790	0.050*
C14	0.1697 (2)	0.9353 (2)	0.2434 (2)	0.0387 (4)
C15	0.0533 (3)	0.9580 (3)	0.3120 (3)	0.0589 (6)
H151	−0.0319	0.8791	0.2907	0.071*
C16	0.0627 (4)	1.0968 (3)	0.4118 (3)	0.0726 (8)
H161	−0.0156	1.1104	0.4583	0.087*
C17	0.1861 (4)	1.2151 (3)	0.4434 (3)	0.0646 (7)
H171	0.1906	1.3089	0.5097	0.077*
C18	0.3036 (3)	1.1941 (3)	0.3761 (2)	0.0562 (6)
H181	0.3883	1.2736	0.3974	0.067*

C19	0.2947 (3)	1.0547 (3)	0.2771 (2)	0.0456 (5)
H191	0.3742	1.0408	0.2322	0.055*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0507 (4)	0.0516 (4)	0.0684 (4)	−0.0046 (3)	0.0023 (3)	0.0063 (3)
Cl2	0.1402 (8)	0.0425 (4)	0.0514 (4)	0.0395 (4)	0.0239 (4)	0.0128 (3)
Cl3	0.0418 (3)	0.0546 (4)	0.0430 (3)	0.0064 (2)	0.0086 (2)	0.0077 (2)
O1	0.0410 (9)	0.0740 (12)	0.0479 (8)	0.0263 (8)	0.0165 (7)	0.0306 (8)
O2	0.0516 (9)	0.0586 (10)	0.0389 (7)	0.0191 (8)	0.0122 (6)	0.0269 (7)
N1	0.0316 (8)	0.0309 (8)	0.0339 (7)	0.0081 (6)	0.0027 (6)	0.0138 (6)
C1	0.0379 (10)	0.0281 (9)	0.0313 (9)	0.0053 (8)	0.0045 (7)	0.0103 (7)
C2	0.0278 (9)	0.0334 (10)	0.0332 (8)	0.0074 (7)	0.0039 (7)	0.0149 (7)
C3	0.0289 (9)	0.0284 (9)	0.0306 (8)	0.0048 (7)	0.0015 (7)	0.0106 (7)
C4	0.0349 (10)	0.0377 (11)	0.0375 (9)	0.0058 (8)	0.0077 (8)	0.0170 (8)
C5	0.0351 (11)	0.0457 (12)	0.0527 (12)	0.0067 (9)	0.0097 (9)	0.0220 (10)
C6	0.0390 (10)	0.0330 (10)	0.0340 (9)	0.0136 (8)	0.0097 (7)	0.0176 (8)
C7	0.0411 (11)	0.0451 (12)	0.0520 (11)	0.0188 (9)	0.0169 (9)	0.0261 (10)
C8	0.0627 (15)	0.0514 (14)	0.0564 (13)	0.0329 (12)	0.0298 (11)	0.0307 (11)
C9	0.0855 (18)	0.0354 (11)	0.0353 (10)	0.0277 (12)	0.0167 (11)	0.0175 (9)
C10	0.0565 (14)	0.0359 (11)	0.0491 (12)	0.0099 (10)	−0.0034 (10)	0.0175 (10)
C11	0.0390 (11)	0.0360 (11)	0.0491 (11)	0.0110 (9)	0.0030 (9)	0.0180 (9)
C12	0.0731 (17)	0.0678 (17)	0.0396 (11)	0.0207 (14)	0.0212 (11)	0.0263 (12)
C13	0.0464 (12)	0.0381 (11)	0.0405 (10)	0.0126 (9)	−0.0013 (8)	0.0194 (9)
C14	0.0373 (11)	0.0360 (11)	0.0447 (10)	0.0133 (8)	0.0010 (8)	0.0201 (9)
C15	0.0377 (12)	0.0492 (14)	0.0818 (17)	0.0139 (10)	0.0119 (11)	0.0227 (13)
C16	0.0671 (19)	0.0614 (18)	0.087 (2)	0.0355 (15)	0.0280 (16)	0.0221 (15)
C17	0.083 (2)	0.0421 (14)	0.0609 (15)	0.0286 (14)	0.0017 (14)	0.0136 (12)
C18	0.0662 (16)	0.0382 (12)	0.0588 (14)	0.0058 (11)	−0.0061 (12)	0.0246 (11)
C19	0.0476 (12)	0.0427 (12)	0.0502 (11)	0.0100 (9)	0.0065 (9)	0.0267 (10)

Geometric parameters (Å, °)

Cl1—C5	1.776 (2)	C8—C9	1.370 (4)
Cl2—C9	1.745 (2)	C8—H81	0.9300
O1—C1	1.196 (3)	C9—C10	1.372 (3)
O2—C1	1.330 (2)	C10—C11	1.379 (3)
O2—C12	1.454 (3)	C10—H101	0.9300
N1—C3	1.502 (2)	C11—H111	0.9300
N1—C13	1.508 (3)	C12—H121	0.9600
N1—H1	0.9000	C12—H122	0.9600
N1—H2	0.9000	C12—H123	0.9600
C1—C2	1.517 (3)	C13—C14	1.501 (3)
C2—C6	1.522 (3)	C13—H131	0.9700
C2—C3	1.544 (3)	C13—H132	0.9700
C2—H21	0.9800	C14—C15	1.381 (3)
C3—C4	1.526 (3)	C14—C19	1.381 (3)
C3—H31	0.9800	C15—C16	1.376 (4)

C4—C5	1.508 (3)	C15—H151	0.9300
C4—H41	0.9700	C16—C17	1.370 (4)
C4—H42	0.9700	C16—H161	0.9300
C5—H51	0.9700	C17—C18	1.380 (4)
C5—H52	0.9700	C17—H171	0.9300
C6—C7	1.387 (3)	C18—C19	1.379 (3)
C6—C11	1.395 (3)	C18—H181	0.9300
C7—C8	1.374 (3)	C19—H191	0.9300
C7—H71	0.9300		
C1—O2—C12	116.08 (18)	C9—C8—H81	120.4
C3—N1—C13	115.50 (15)	C7—C8—H81	120.4
C3—N1—H1	108.4	C8—C9—C10	121.5 (2)
C13—N1—H1	108.4	C8—C9—Cl2	119.32 (19)
C3—N1—H2	108.4	C10—C9—Cl2	119.2 (2)
C13—N1—H2	108.4	C9—C10—C11	119.5 (2)
H1—N1—H2	107.5	C9—C10—H101	120.3
O1—C1—O2	124.21 (18)	C11—C10—H101	120.3
O1—C1—C2	124.68 (17)	C10—C11—C6	120.2 (2)
O2—C1—C2	111.11 (17)	C10—C11—H111	119.9
C1—C2—C6	111.18 (15)	C6—C11—H111	119.9
C1—C2—C3	111.11 (15)	O2—C12—H121	109.5
C6—C2—C3	114.25 (14)	O2—C12—H122	109.5
C1—C2—H21	106.6	H121—C12—H122	109.5
C6—C2—H21	106.6	O2—C12—H123	109.5
C3—C2—H21	106.6	H121—C12—H123	109.5
N1—C3—C4	109.14 (15)	H122—C12—H123	109.5
N1—C3—C2	111.71 (14)	C14—C13—N1	114.43 (16)
C4—C3—C2	112.34 (16)	C14—C13—H131	108.7
N1—C3—H31	107.8	N1—C13—H131	108.7
C4—C3—H31	107.8	C14—C13—H132	108.7
C2—C3—H31	107.8	N1—C13—H132	108.7
C5—C4—C3	115.28 (16)	H131—C13—H132	107.6
C5—C4—H41	108.5	C15—C14—C19	118.5 (2)
C3—C4—H41	108.5	C15—C14—C13	120.1 (2)
C5—C4—H42	108.5	C19—C14—C13	121.3 (2)
C3—C4—H42	108.5	C16—C15—C14	120.4 (2)
H41—C4—H42	107.5	C16—C15—H151	119.8
C4—C5—Cl1	111.82 (16)	C14—C15—H151	119.8
C4—C5—H51	109.3	C17—C16—C15	120.8 (3)
Cl1—C5—H51	109.3	C17—C16—H161	119.6
C4—C5—H52	109.3	C15—C16—H161	119.6
Cl1—C5—H52	109.3	C16—C17—C18	119.6 (2)
H51—C5—H52	107.9	C16—C17—H171	120.2
C7—C6—C11	118.70 (19)	C18—C17—H171	120.2
C7—C6—C2	119.31 (18)	C19—C18—C17	119.6 (2)
C11—C6—C2	121.99 (17)	C19—C18—H181	120.2
C8—C7—C6	121.0 (2)	C17—C18—H181	120.2
C8—C7—H71	119.5	C18—C19—C14	121.2 (2)
C6—C7—H71	119.5	C18—C19—H191	119.4

C9—C8—C7	119.1 (2)	C14—C19—H191	119.4
C1—C2—C3—C4	170.19 (15)	C3—C2—C6—C11	−84.9 (2)
C2—C3—C4—C5	−65.1 (2)	C11—C6—C7—C8	1.2 (3)
C3—C4—C5—Cl1	−59.2 (2)	C2—C6—C7—C8	−178.37 (18)
C3—C2—C1—O2	−145.64 (16)	C6—C7—C8—C9	−0.2 (3)
C2—C1—O2—C12	178.60 (18)	C7—C8—C9—C10	−0.8 (3)
C1—C2—C6—C7	−138.62 (18)	C7—C8—C9—Cl2	179.55 (17)
C2—C3—N1—C13	164.81 (16)	C8—C9—C10—C11	0.7 (3)
C3—N1—C13—C14	−53.8 (2)	Cl2—C9—C10—C11	−179.63 (16)
N1—C13—C14—C15	−80.7 (3)	C9—C10—C11—C6	0.3 (3)
C12—O2—C1—O1	−1.5 (3)	C7—C6—C11—C10	−1.3 (3)
O1—C1—C2—C6	−93.9 (2)	C2—C6—C11—C10	178.28 (18)
O2—C1—C2—C6	85.91 (19)	N1—C13—C14—C19	102.7 (2)
O1—C1—C2—C3	34.5 (3)	C19—C14—C15—C16	−0.1 (4)
C13—N1—C3—C4	−70.3 (2)	C13—C14—C15—C16	−176.8 (2)
C1—C2—C3—N1	−66.78 (18)	C14—C15—C16—C17	1.1 (5)
C6—C2—C3—N1	60.0 (2)	C15—C16—C17—C18	−1.4 (5)
C6—C2—C3—C4	−63.0 (2)	C16—C17—C18—C19	0.7 (4)
N1—C3—C4—C5	170.40 (17)	C17—C18—C19—C14	0.3 (3)
C3—C2—C6—C7	94.6 (2)	C15—C14—C19—C18	−0.6 (3)
C1—C2—C6—C11	41.8 (2)	C13—C14—C19—C18	176.10 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2···Cl3	0.90	2.28	3.0890 (18)	150
N1—H1···Cl3^{i}	0.90	2.59	3.2447 (18)	131

Symmetry codes: (i) −*x*, −*y*+1, −*z*.

Fig. 1

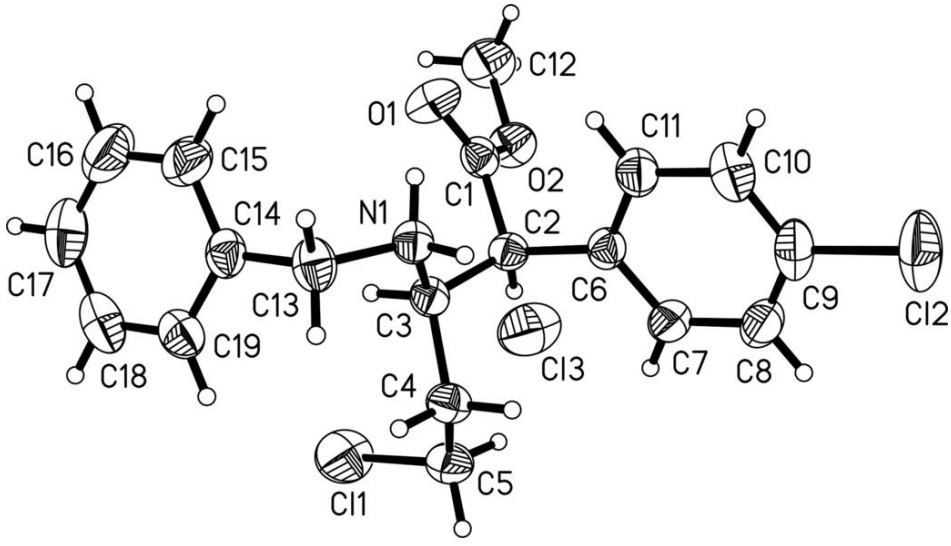
C12
O1
C11
C16
C15
C1
O2
C10
C14
N1
C2
C17
C13
C6
C9
Cl2
C3
Cl3
C19
C7
C8
C18
C4
C5
Cl1

Fig. 2