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Dibenzylammonium hydrogen maleate and a redetermination at 120 K of bis(dibenzylamino)methane

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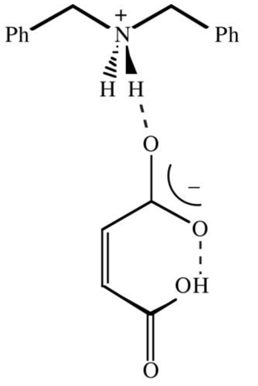
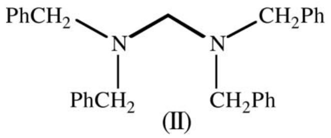
In dibenzylammonium hydrogen maleate [or dibenzylammonium (2*Z*)-3-carboxyprop-2-enoate], $C_{14}H_{16}N^+\cdot C_4H_3O_4^-$, (I), the anion contains a fairly short and nearly linear O—H⋯O hydrogen bond, with an O⋯O distance of 2.4603 (16) Å, but with the H atom clearly offset from the mid-point of the O⋯O vector. The counter-ions in (I) are linked by two N—H⋯O hydrogen bonds to form $C_2^2(6)$ chains and these chains are weakly linked into sheets by a C—H⋯O hydrogen bond. Bis(dibenzylamino)methane, $C_{29}H_{30}N_2$, (II), crystallizes with two independent molecules lying across twofold rotation axes in the space group *C*2/*c*, and the molecules are conformationally chiral; there are no direction-specific intermolecular interactions in the crystal structure of (II).

Comment

We report here the molecular and supramolecular structures of dibenzylammonium hydrogen maleate, (I) (Fig. 1), and a redetermination of bis(dibenzylamino)methane, (II) (Fig. 2). Aminoethers are valuable building blocks in organic synthesis as they are important precursors in the synthesis of a wide range of pharmaceutical products (Pinder & Wieringa, 1993; Franchini *et al.*, 2003; Cavalluzzi *et al.*, 2007; Huang *et al.*, 2009). In recent years, a series of selective serotonin (5-HT)-reuptake inhibitor antidepressants (*e.g.* Fluexetine and Paroxetine) and selective norepinephrine-reuptake inhibitor antidepressants (*e.g.* Tomoxetine and Viloxazine) have been developed (Pinder & Wieringa, 1993), and several of these compounds contain a γ-aminoether functionality, which could be related to their biological activity. Continuing with our current studies on the synthetic utility of benzylamines (Castillo *et al.*, 2009; Abonía *et al.*, 2010), the aminal (II) was isolated as a possible intermediate in a synthetic route to novel γ-aminoether derivatives starting from secondary benzylamines and using a four-component strategy mediated by a Mannich-type reaction (Abonía *et al.*, 2013). In order to confirm the role of (II) in this route, it has been prepared from the reaction of dibenzylamine with paraformaldehyde in acetonitrile and then used to obtain the γ-aminoethers, thus confirming its intermediacy. As a model for the crystallization and characterization of new secondary benzylamine derivatives needed in this synthetic route we have prepared and crystallized the dibenzylammonium hydrogen maleate salt, (I).



(I)



(II)

The structure of compound (II) has been reported previously using diffraction data collected at 298 K [Cambridge Structural Database (CSD; Allen, 2002) refcode MIKQON (Lu *et al.*, 2007)], but the refinement converged to a rather high R_1 value of 0.0794, while the value of wR_2 was also quoted as 0.0794, suggesting that unweighted data were used

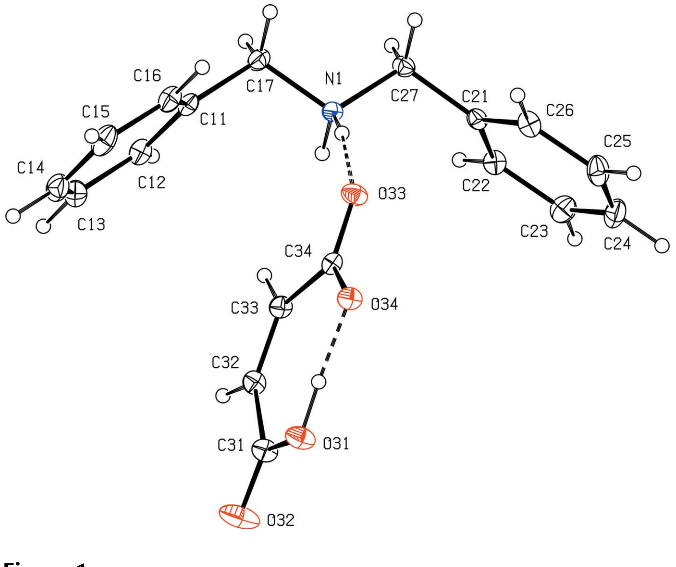


Figure 1
The ionic components of salt (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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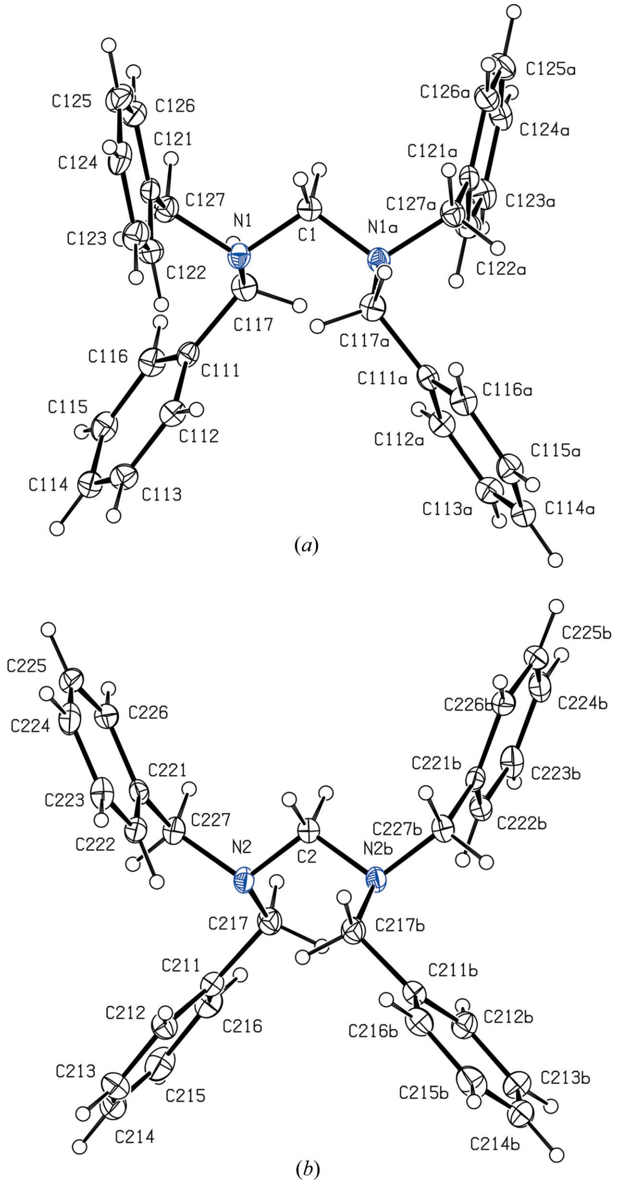


Figure 2
The independent molecular components of compound (II), showing the atom-labelling scheme for (*a*) a type 1 molecule and (*b*) a type 2 molecule. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (a) $-x + 1, y, -z + \frac{1}{2}$; (b) $-x + 1, y, -z + \frac{3}{2}$.]

in the final refinements. Alongside the high *R* index is the rather low precision, with s.u. values for the bonded C—C distances ranging from 0.006 to 0.014 Å, with a mean s.u. of 0.009 Å. Accordingly, we have now taken the opportunity to reinvestigate compound (II) using diffraction data collected at 120 K, resulting in a lower *R* value and significantly better precision, with s.u. values for the bonded C—C distances typically equal to 0.002 Å.

In the cation of salt (I), the central backbone between atoms C11 and C21 (Fig. 1) is almost planar, as indicated by the relevant torsion angles (Table 1), while the two independent phenyl rings are almost orthogonal to this central plane. The anion of (I) contains a rather short and almost linear O—H···O hydrogen bond, forming an *S*(7) motif (Bernstein *et al.*, 1995) (Fig. 1 and Table 2); however, despite the short O···O distance, the hydroxy H atom is clearly offset from the mid-point of the O···O vector. The C—O distances in the anion (Table 1) are fully consistent with the location of the hydroxy H atom as deduced from a difference map. Similarly short and nearly linear O—H···O hydrogen bonds are found in the hydrogen maleate salts formed with both 1,2-bis(pyridin-4-yl)ethane and 4,4′-bipyridyl, where the O···O distances are 2.4528 (18) and 2.463 (2) Å, respectively, with O—H···O angles of 178 and 176°, respectively (Bowes *et al.*, 2003); in both of these salts, the H atom in the O—H···O hydrogen bond is clearly offset from the mid-point of the O···O vector, just as in salt (I).

The C—C—C—O torsion angles in the anion of (I) (Table 1) show that both of the carboxyl fragments are rotated slightly away from the plane defined by atoms C31–C34, such that atoms O31 and O34 (those involved in the intra-anion hydrogen bond) are both displaced to one side of the C31–C34 plane, by 0.143 (2) and 0.134 (2) Å, respectively, while atoms O32 and O33 are displaced to the other side of this plane, by 0.117 (2) and 0.115 (2) Å, respectively, corresponding to a disrotatory motion of the carboxyl groups around the C31—C32 and C33—C34 bonds. By contrast, the anions in the salts formed with 1,2-bis(pyridin-4-yl)ethane and 4,4′-bipyridyl are both effectively planar, with maximum deviations from the mean plane of the eight non-H atoms of the anions of 0.041 (2) Å in the former salt and only 0.013 (2) Å in the latter (Bowes *et al.*, 2003).

In the asymmetric unit selected for salt (I) (Fig. 1), the two ions are linked by an N—H···O hydrogen bond (Table 2), while a second N—H···O hydrogen bond links such ion pairs

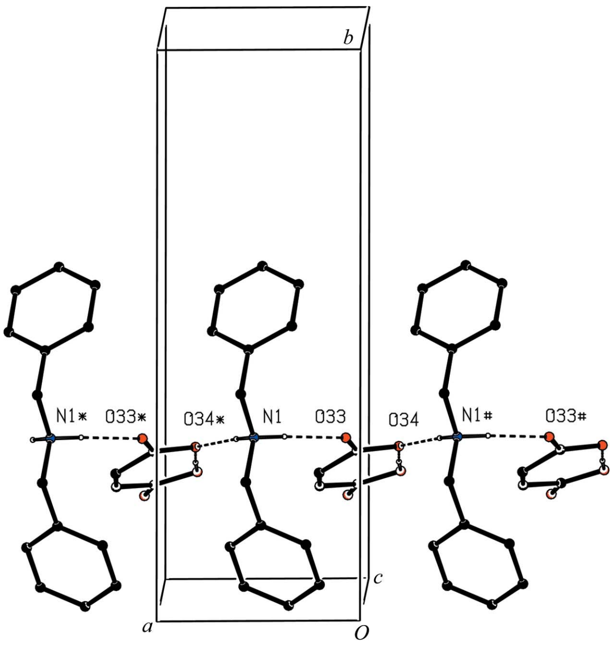


Figure 3
Part of the crystal structure of salt (I), showing the formation of a hydrogen-bonded chain along [100]. For the sake of clarity, H atoms bonded to C atoms have all been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($x + 1, y, z$) and ($x - 1, y, z$), respectively.

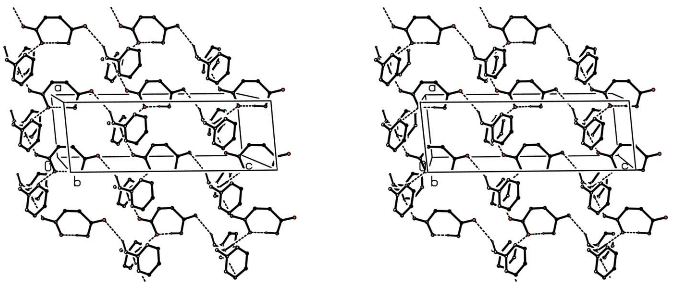
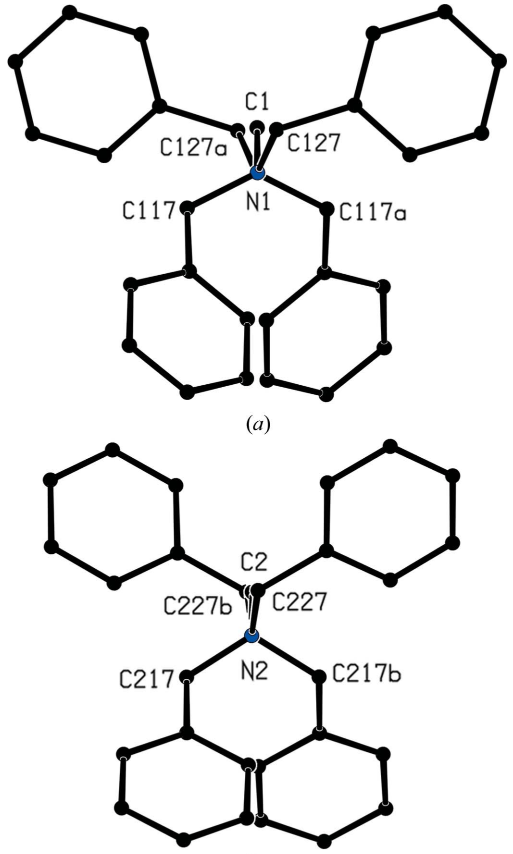


Figure 4
A stereoview of part of the crystal structure of salt (I), showing the formation of a hydrogen-bonded sheet parallel to (010). For the sake of clarity, H atoms not involved in the motifs shown have been omitted

which are related by translation to form a $C_2^2(6)$ (Bernstein *et al.*, 1995) chain running parallel to the [100] direction (Fig. 3). There are also two short intermolecular C—H···O contacts present in the structure of (I) (Table 2). One of these interactions, having a C—H···O angle of 153°, weakly links the chains along [100] into a sheet parallel to (010) and built from $S(7)$ and $R_7^6(25)$ rings (Fig. 4). In the other such interaction, which lies within the (010) sheet, the C—H···O angle is only 131°, so that this contact is probably not structurally significant (Wood *et al.*, 2009). It is notable that the strong and nearly linear O—H···O and N—H···O hydrogen bonds in the structure of (I) all involve the negatively charged O atoms of the carboxylate group, whereas the C—H···O contacts involve the formally neutral O32 atom of the carboxylic acid unit.

Compound (II) crystallizes in the space group $C2/c$ with two independent molecules both lying across twofold rotation axes. Comparison of the unit-cell dimensions at 298 K (Lu *et al.*, 2007) and at 120 K indicates that no phase change has occurred between these two temperatures. The molecules of (II) exhibit no internal symmetry other than the twofold rotation axis and hence they are conformationally chiral, although the space group accommodates equal numbers of the two conformational enantiomers. There is thus considerable flexibility available in the choice of the asymmetric unit, but that selected here contains two independent molecules of the same enantiomeric form, as indicated by the general similarity of corresponding torsion angles (Table 3). On this basis, the type 1 molecule containing atom C1 lies across the rotation axis along ($\frac{1}{2}$, y, $\frac{1}{4}$), while the type 2 molecule containing atom C2 lies across the axis along ($\frac{1}{2}$, y, $\frac{3}{4}$).

In each molecule of (II), the arrangement of the benzyl groups is probably dominated by a combination of steric factors and the mutual repulsion of the lone pairs on the two N atoms, which adopt an anticlinal conformation relative to the N···N direction (Fig. 5). The projections along the N···N vectors, together with the detailed comparison of the relevant torsion angles (Table 3) confirm that there are sufficient conformational differences between the two independent molecules to preclude the possibility of any additional crystallographic symmetry.



(*a*)

(*b*)

Figure 5
Projections of the molecular structures in compound (II) along the N···N vectors, showing the molecular conformations for (*a*) a type 1 molecule and (*b*) a type 2 molecule. For the sake of clarity, H atoms have all been omitted. [Symmetry codes: (a) $-x + 1, y, -z + \frac{1}{2}$; (b) $-x + 1, y, -z + \frac{3}{2}$.]

Despite the bulk of the dibenzylamino group, the interbond angles (Table 3) at the central C1 and C2 atoms are unexceptional, and the C—N bond lengths involving atoms C1 and C2 are typical of their type [mean value (Allen *et al.*, 1987) 1.469 Å]. Both of the N atoms in compound (II) are markedly pyramidal, with the sum of the interbond angles being 332.4 (2)° at N1 and 330.4 (2)° at N2. Despite this, there are no C—H···N hydrogen bonds within the crystal structure of (II); indeed, there are no direction-specific interactions of any type, as both C—H···π(arene) hydrogen bonds and aromatic π–π stacking interactions are also absent.

Experimental

For the synthesis of salt (I), a mixture of dibenzylamine (80 mg) and an excess of maleic acid (161 mg, 2.5 equivalents) in ethyl acetate (2 ml) was stirred at ambient temperature for 1 h. The resulting solid product was collected by filtration, washed with cold ethyl acetate (2 × 1 ml) and dried at ambient temperature to provide product (I) as colourless crystals (yield 86%, m.p. 482 K). FT–IR (KBr): 3032, 2828, 2751, 2636, 1705 (C═O), 1629 (C═C), 1384, 1361, 1214, 1086 (C—O) cm^{-1}. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in air, from a solution in ethyl acetate. For the synthesis of compound (II), a mixture of dibenzylamine (201 mg, 2.0 mmol) and paraformaldehyde (15 mg, 1.0 mmol) in acetonitrile (2 ml) was stirred at ambient temperature for 3 h. The resulting solid product was collected by filtration, washed with acetonitrile (1 ml) and dried at ambient temperature to provide aminal (II) (yield 93%, m.p. 372 K). FT–IR (KBr): 3058, 2925, 2877, 1599, 739, 697 cm^{-1}. Colourless crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in air, from a solution in ethanol.

Compound (I)

Crystal data

$C_{14}H_{16}N^{+}\cdot C_4H_3O_4^{-}$
M_r = 313.34
Monoclinic, $P2_1/c$
a = 5.7551 (5) Å
b = 16.1579 (19) Å
c = 17.1230 (19) Å
β = 95.464 (8)°
V = 1585.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 120 K
0.32 × 0.30 × 0.26 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min}$ = 0.971, $T_{\max}$ = 0.976
22681 measured reflections
3639 independent reflections
2621 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.110
S = 1.08
3639 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.18 e Å^{-3}
$\Delta\rho_{\min}$ = −0.25 e Å^{-3}

Compound (II)

Crystal data

$C_{29}H_{30}N_2$
M_r = 406.55
Monoclinic, $C2/c$
a = 25.548 (6) Å
b = 12.9426 (16) Å
c = 18.555 (3) Å
β = 130.428 (11)°
V = 4670.4 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 120 K
0.42 × 0.24 × 0.23 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min}$ = 0.972, $T_{\max}$ = 0.985
18316 measured reflections
5366 independent reflections
3223 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.115
S = 1.01
5366 reflections
281 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.25 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e Å^{-3}

All H atoms were located in difference maps. H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (aromatic and alkene) or 0.99 Å (CH_2) and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C). H atoms bonded to N or O atoms were permitted to ride at the positions deduced from difference maps, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(N) or $1.5U_{\rm eq}$(O), giving the N—H and O—H distances shown in Table 2.

For both compounds, data collection: *COLLECT* (Hooft, 1998); cell refinement: *DIRAX*/*LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

Table 1
Selected geometric parameters (Å, °) for (I).

C31—O31	1.3120 (19)	C34—O33	1.2402 (18)
C31—O32	1.220 (2)	C34—O34	1.2842 (18)
C27—N1—C17—C11	−177.63 (12)	C33—C32—C31—O31	6.5 (3)
N1—C17—C11—C12	−85.51 (18)	C33—C32—C31—O32	−174.08 (19)
C17—N1—C27—C21	−179.37 (12)	C32—C33—C34—O33	173.64 (17)
N1—C27—C21—C22	90.51 (17)	C32—C33—C34—O34	−7.5 (3)
C31—C32—C33—C34	0.9 (3)		

Table 2
Hydrogen-bond geometry (Å, °) for (I).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1*A*⋯O33	0.91	1.94	2.8412 (16)	171
N1—H1*B*⋯O34$^{\rm i}$	0.90	1.96	2.8582 (16)	175
O31—H31⋯O34	0.98	1.48	2.4603 (16)	177
C27—H27*A*⋯O32$^{\rm ii}$	0.99	2.55	3.290 (2)	131
C27—H27*B*⋯O32$^{\rm iii}$	0.99	2.54	3.452 (2)	153

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

Table 3
Selected geometric parameters (Å, °) for (II).

C1—N1	1.4612 (17)	C2—N2	1.4678 (17)
N1—C1—N1$^{\rm i}$	110.27 (17)	N2—C2—N2$^{\rm ii}$	110.83 (17)
N1$^{\rm i}$—C1—N1—C117	−70.90 (10)	N2$^{\rm ii}$—C2—N2—C217	−64.21 (10)
N1$^{\rm i}$—C1—N1—C127	165.41 (12)	N2$^{\rm ii}$—C2—N2—C227	174.01 (12)
C1—N1—C117—C111	163.10 (13)	C2—N2—C217—C211	169.98 (13)
N1—C117—C111—C112	−39.3 (2)	N2—C217—C211—C212	−29.9 (2)
C1—N1—C127—C121	−68.89 (15)	C2—N2—C227—C221	−63.51 (15)
N1—C127—C121—C122	−50.98 (19)	N2—C227—C221—C222	−37.5 (2)

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

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: YF3037). Services for accessing these data are described at the back of the journal.

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supplementary materials

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Dibenzylammonium hydrogen maleate and a redetermination at 120 K of bis(dibenzylamino)methane

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Computing details

For both compounds, data collection: *COLLECT* (Hooft, 1998); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

(I) Dibenzylammonium (2*Z*)-3-carboxyprop-2-enoate

Crystal data

$C_{14}H_{16}N^{+}\cdot C_4H_3O_4^{-}$
$M_r = 313.34$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.7551$ (5) Å
$b = 16.1579$ (19) Å
$c = 17.1230$ (19) Å
$\beta = 95.464$ (8)°
$V = 1585.0$ (3) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.313$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3639 reflections
$\theta = 2.8$–27.5°
$\mu = 0.09$ mm^{-1}
$T = 120$ K
Block, colourless
0.32 × 0.30 × 0.26 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker–Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ & ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.971$, $T_{\max} = 0.976$
22681 measured reflections
3639 independent reflections
2621 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$
$\theta_{\max} = 27.5°$, $\theta_{\min} = 2.8°$
$h = -7\rightarrow7$
$k = -20\rightarrow20$
$l = -22\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.110$
$S = 1.08$
3639 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.5921P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.25$ e Å^{-3}

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

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
N1	0.5366 (2)	0.29720 (8)	0.32397 (7)	0.0191 (3)
H1A	0.3868	0.2977	0.3365	0.023*
H1B	0.6253	0.2914	0.3695	0.023*
C17	0.5742 (3)	0.22299 (10)	0.27401 (9)	0.0257 (4)
H17A	0.7414	0.2192	0.2652	0.031*
H17B	0.4832	0.2296	0.2223	0.031*
C11	0.5009 (3)	0.14446 (10)	0.31198 (9)	0.0228 (3)
C12	0.6548 (3)	0.10306 (10)	0.36598 (10)	0.0271 (4)
H12	0.8073	0.1246	0.3788	0.033*
C13	0.5884 (3)	0.03080 (11)	0.40131 (11)	0.0336 (4)
H13	0.6947	0.0033	0.4385	0.040*
C14	0.3674 (3)	−0.00141 (11)	0.38249 (11)	0.0344 (4)
H14	0.3218	−0.0510	0.4066	0.041*
C15	0.2136 (3)	0.03868 (11)	0.32862 (11)	0.0343 (4)
H15	0.0623	0.0164	0.3153	0.041*
C16	0.2788 (3)	0.11154 (11)	0.29361 (10)	0.0283 (4)
H16	0.1712	0.1391	0.2569	0.034*
C27	0.5999 (3)	0.37606 (10)	0.28506 (9)	0.0225 (3)
H27A	0.5025	0.3821	0.2345	0.027*
H27B	0.7653	0.3736	0.2738	0.027*
C21	0.5641 (3)	0.45008 (10)	0.33596 (9)	0.0209 (3)
C22	0.7442 (3)	0.47900 (10)	0.38888 (9)	0.0251 (4)
H22	0.8900	0.4510	0.3940	0.030*
C23	0.7124 (3)	0.54831 (11)	0.43418 (10)	0.0318 (4)
H23	0.8362	0.5677	0.4702	0.038*
C24	0.5000 (3)	0.58949 (11)	0.42711 (11)	0.0336 (4)
H24	0.4783	0.6370	0.4583	0.040*
C25	0.3199 (3)	0.56126 (11)	0.37469 (11)	0.0323 (4)
H25	0.1746	0.5896	0.3696	0.039*
C26	0.3511 (3)	0.49167 (10)	0.32962 (10)	0.0267 (4)
H26	0.2262	0.4721	0.2941	0.032*
C31	0.0391 (3)	0.19287 (11)	0.62431 (10)	0.0265 (4)
C32	0.2231 (3)	0.19375 (10)	0.56848 (9)	0.0235 (3)
H32	0.3669	0.1696	0.5888	0.028*
C33	0.2229 (3)	0.22217 (10)	0.49537 (9)	0.0224 (3)
H33	0.3650	0.2140	0.4724	0.027*
C34	0.0356 (3)	0.26483 (10)	0.44335 (9)	0.0204 (3)
O31	−0.17349 (18)	0.21716 (8)	0.60052 (7)	0.0306 (3)
H31	−0.1673	0.2373	0.5466	0.046*
O32	0.0899 (2)	0.16877 (10)	0.69127 (7)	0.0472 (4)
O33	0.08704 (18)	0.29195 (7)	0.37947 (6)	0.0273 (3)
O34	−0.17021 (17)	0.27045 (7)	0.46603 (6)	0.0243 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0174 (6)	0.0214 (7)	0.0185 (6)	0.0016 (5)	0.0018 (5)	0.0008 (5)
C17	0.0295 (9)	0.0243 (9)	0.0242 (8)	0.0014 (7)	0.0076 (7)	−0.0042 (7)
C11	0.0237 (8)	0.0210 (8)	0.0243 (8)	0.0021 (6)	0.0055 (6)	−0.0056 (6)
C12	0.0249 (8)	0.0245 (9)	0.0319 (9)	0.0000 (7)	0.0021 (7)	−0.0053 (7)
C13	0.0409 (10)	0.0260 (10)	0.0334 (10)	0.0036 (8)	0.0011 (8)	−0.0004 (8)
C14	0.0419 (10)	0.0231 (9)	0.0404 (10)	−0.0051 (8)	0.0161 (8)	−0.0040 (8)
C15	0.0255 (9)	0.0310 (10)	0.0475 (11)	−0.0050 (7)	0.0084 (8)	−0.0142 (9)
C16	0.0255 (8)	0.0273 (9)	0.0318 (9)	0.0038 (7)	0.0012 (7)	−0.0075 (7)
C27	0.0233 (8)	0.0231 (8)	0.0213 (8)	−0.0026 (6)	0.0030 (6)	0.0021 (6)
C21	0.0213 (8)	0.0196 (8)	0.0220 (8)	−0.0013 (6)	0.0036 (6)	0.0034 (6)
C22	0.0216 (8)	0.0251 (9)	0.0283 (9)	0.0000 (7)	0.0008 (7)	0.0002 (7)
C23	0.0290 (9)	0.0314 (10)	0.0346 (9)	−0.0048 (7)	0.0011 (7)	−0.0053 (8)
C24	0.0376 (10)	0.0213 (9)	0.0440 (10)	−0.0028 (7)	0.0145 (8)	−0.0061 (8)
C25	0.0245 (9)	0.0249 (9)	0.0488 (11)	0.0047 (7)	0.0105 (8)	0.0041 (8)
C26	0.0215 (8)	0.0253 (9)	0.0330 (9)	−0.0007 (7)	0.0012 (7)	0.0043 (7)
C31	0.0213 (8)	0.0328 (10)	0.0252 (8)	−0.0006 (7)	0.0012 (6)	0.0032 (7)
C32	0.0166 (7)	0.0270 (9)	0.0267 (8)	0.0030 (6)	0.0003 (6)	0.0017 (7)
C33	0.0163 (7)	0.0272 (9)	0.0242 (8)	0.0031 (6)	0.0038 (6)	0.0000 (7)
C34	0.0177 (7)	0.0206 (8)	0.0227 (8)	0.0004 (6)	0.0011 (6)	−0.0037 (6)
O31	0.0203 (6)	0.0458 (8)	0.0264 (6)	0.0057 (5)	0.0057 (5)	0.0092 (5)
O32	0.0293 (7)	0.0836 (11)	0.0290 (7)	0.0082 (7)	0.0044 (5)	0.0217 (7)
O33	0.0230 (6)	0.0354 (7)	0.0239 (6)	0.0043 (5)	0.0050 (5)	0.0064 (5)
O34	0.0170 (5)	0.0314 (7)	0.0245 (6)	0.0032 (5)	0.0025 (4)	0.0019 (5)

Geometric parameters (Å, º)

N1—C27	1.4987 (19)	C21—C26	1.393 (2)
N1—C17	1.501 (2)	C22—C23	1.384 (2)
N1—H1A	0.9085	C22—H22	0.9500
N1—H1B	0.8954	C23—C24	1.387 (2)
C17—C11	1.504 (2)	C23—H23	0.9500
C17—H17A	0.9900	C24—C25	1.382 (3)
C17—H17B	0.9900	C24—H24	0.9500
C11—C12	1.390 (2)	C25—C26	1.385 (2)
C11—C16	1.393 (2)	C25—H25	0.9500
C12—C13	1.385 (2)	C26—H26	0.9500
C12—H12	0.9500	C31—O31	1.3120 (19)
C13—C14	1.384 (3)	C31—O32	1.220 (2)
C13—H13	0.9500	C31—C32	1.493 (2)
C14—C15	1.378 (3)	C32—C33	1.333 (2)
C14—H14	0.9500	C32—H32	0.9500
C15—C16	1.389 (3)	C33—C34	1.499 (2)
C15—H15	0.9500	C33—H33	0.9500
C16—H16	0.9500	C34—O33	1.2402 (18)
C27—C21	1.506 (2)	C34—O34	1.2842 (18)
C27—H27A	0.9900	O31—H31	0.9822
C27—H27B	0.9900	O34—H31	1.4792

C21—C22	1.391 (2)		

C27—N1—C17	111.82 (11)	C21—C27—H27B	109.3
C27—N1—H1A	112.0	H27A—C27—H27B	108.0
C17—N1—H1A	109.5	C22—C21—C26	118.97 (15)
C27—N1—H1B	109.5	C22—C21—C27	120.64 (14)
C17—N1—H1B	108.1	C26—C21—C27	120.37 (14)
H1A—N1—H1B	105.6	C23—C22—C21	120.44 (15)
N1—C17—C11	111.48 (12)	C23—C22—H22	119.8
N1—C17—H17A	109.3	C21—C22—H22	119.8
C11—C17—H17A	109.3	C22—C23—C24	120.12 (16)
N1—C17—H17B	109.3	C22—C23—H23	119.9
C11—C17—H17B	109.3	C24—C23—H23	119.9
H17A—C17—H17B	108.0	C25—C24—C23	119.90 (17)
C12—C11—C16	118.67 (16)	C25—C24—H24	120.0
C12—C11—C17	120.42 (14)	C23—C24—H24	120.0
C16—C11—C17	120.91 (15)	C24—C25—C26	120.04 (16)
C13—C12—C11	120.74 (16)	C24—C25—H25	120.0
C13—C12—H12	119.6	C26—C25—H25	120.0
C11—C12—H12	119.6	C25—C26—C21	120.52 (16)
C14—C13—C12	120.08 (17)	C25—C26—H26	119.7
C14—C13—H13	120.0	C21—C26—H26	119.7
C12—C13—H13	120.0	O32—C31—O31	121.36 (15)
C15—C14—C13	119.77 (17)	O32—C31—C32	118.92 (15)
C15—C14—H14	120.1	O31—C31—C32	119.71 (14)
C13—C14—H14	120.1	C33—C32—C31	131.85 (15)
C14—C15—C16	120.33 (16)	C33—C32—H32	114.1
C14—C15—H15	119.8	C31—C32—H32	114.1
C16—C15—H15	119.8	C32—C33—C34	130.74 (14)
C15—C16—C11	120.40 (16)	C32—C33—H33	114.6
C15—C16—H16	119.8	C34—C33—H33	114.6
C11—C16—H16	119.8	O33—C34—O34	123.11 (14)
N1—C27—C21	111.45 (12)	O33—C34—C33	117.90 (13)
N1—C27—H27A	109.3	O34—C34—C33	118.98 (14)
C21—C27—H27A	109.3	C31—O31—H31	105.9
N1—C27—H27B	109.3	C34—O34—H31	109.1

C27—N1—C17—C11	−177.63 (12)	C26—C21—C22—C23	−0.4 (2)
N1—C17—C11—C12	−85.51 (18)	C27—C21—C22—C23	178.40 (15)
N1—C17—C11—C16	94.80 (17)	C21—C22—C23—C24	0.0 (3)
C16—C11—C12—C13	−0.4 (2)	C22—C23—C24—C25	0.0 (3)
C17—C11—C12—C13	179.86 (15)	C23—C24—C25—C26	0.4 (3)
C11—C12—C13—C14	0.6 (3)	C24—C25—C26—C21	−0.7 (3)
C12—C13—C14—C15	−0.1 (3)	C22—C21—C26—C25	0.7 (2)
C13—C14—C15—C16	−0.5 (3)	C27—C21—C26—C25	−178.07 (15)
C14—C15—C16—C11	0.6 (3)	C31—C32—C33—C34	0.9 (3)
C12—C11—C16—C15	−0.2 (2)	C33—C32—C31—O31	6.5 (3)
C17—C11—C16—C15	179.54 (15)	C33—C32—C31—O32	−174.08 (19)
C17—N1—C27—C21	−179.37 (12)	C32—C33—C34—O33	173.64 (17)

N1—C27—C21—C22	90.51 (17)	C32—C33—C34—O34	−7.5 (3)
N1—C27—C21—C26	−90.76 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O33	0.91	1.94	2.8412 (16)	171
N1—H1*B*···O34^{i}	0.90	1.96	2.8582 (16)	175
O31—H31···O34	0.98	1.48	2.4603 (16)	177
C27—H27*A*···O32^{ii}	0.99	2.55	3.290 (2)	131
C27—H27*B*···O32^{iii}	0.99	2.54	3.452 (2)	153

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

(II) Bis(dibenzylamino)methane

Crystal data

$C_{29}H_{30}N_2$	$F(000) = 1744$
$M_r = 406.55$	$D_x = 1.156$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 5366 reflections
$a = 25.548 (6)$ Å	$\theta = 2.8$–$27.5°$
$b = 12.9426 (16)$ Å	$\mu = 0.07$ mm^{-1}
$c = 18.555 (3)$ Å	$T = 120$ K
$\beta = 130.428 (11)°$	Block, colourless
$V = 4670.4 (16)$ Å^3	0.42 × 0.24 × 0.23 mm
$Z = 8$	

Data collection

Bruker–Nonius KappaCCD diffractometer	$T_{min} = 0.972$, $T_{max} = 0.985$
Radiation source: Bruker–Nonius FR591 rotating anode	18316 measured reflections
Graphite monochromator	5366 independent reflections
Detector resolution: 9.091 pixels mm^{-1}	3223 reflections with $I > 2\sigma(I)$
φ & ω scans	$R_{int} = 0.055$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 27.5°$, $\theta_{min} = 2.8°$
	$h = -33 \rightarrow 33$
	$k = -16 \rightarrow 16$
	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.115$	$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 1.028P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
5366 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
281 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5000	0.50145 (17)	0.2500	0.0307 (5)	

H1A	0.5412	0.4566	0.2885	0.037*	0.50
H1B	0.4588	0.4566	0.2115	0.037*	0.50
N1	0.49758 (6)	0.56599 (9)	0.31241 (9)	0.0276 (3)	
C117	0.56415 (8)	0.61702 (12)	0.38317 (11)	0.0320 (4)	
H11A	0.5818	0.6415	0.3519	0.038*	
H11B	0.5973	0.5658	0.4319	0.038*	
C111	0.56044 (8)	0.70735 (12)	0.43112 (10)	0.0266 (4)	
C112	0.50511 (8)	0.77576 (12)	0.38141 (11)	0.0298 (4)	
H112	0.4674	0.7637	0.3163	0.036*	
C113	0.50456 (8)	0.86085 (13)	0.42574 (11)	0.0335 (4)	
H113	0.4661	0.9061	0.3911	0.040*	
C114	0.55938 (9)	0.88093 (13)	0.51999 (12)	0.0349 (4)	
H114	0.5590	0.9402	0.5499	0.042*	
C115	0.61455 (9)	0.81410 (13)	0.57019 (11)	0.0362 (4)	
H115	0.6526	0.8275	0.6349	0.043*	
C116	0.61463 (8)	0.72733 (13)	0.52643 (11)	0.0337 (4)	
H116	0.6523	0.6807	0.5621	0.040*	
C127	0.47741 (8)	0.50487 (13)	0.35771 (11)	0.0315 (4)	
H12A	0.4843	0.5469	0.4078	0.038*	
H12B	0.5076	0.4435	0.3882	0.038*	
C121	0.40329 (8)	0.46988 (12)	0.28810 (10)	0.0276 (4)	
C122	0.35101 (8)	0.54050 (13)	0.22746 (11)	0.0315 (4)	
H122	0.3622	0.6111	0.2298	0.038*	
C123	0.28296 (9)	0.50947 (14)	0.16380 (11)	0.0367 (4)	
H123	0.2479	0.5587	0.1229	0.044*	
C124	0.26594 (9)	0.40668 (14)	0.15969 (12)	0.0373 (4)	
H124	0.2193	0.3851	0.1160	0.045*	
C125	0.31716 (9)	0.33627 (14)	0.21932 (12)	0.0398 (4)	
H125	0.3058	0.2657	0.2167	0.048*	
C126	0.38526 (9)	0.36730 (12)	0.28316 (12)	0.0343 (4)	
H126	0.4201	0.3178	0.3241	0.041*	
C2	0.5000	0.97739 (17)	0.7500	0.0296 (5)	
H2A	0.4848	1.0223	0.7765	0.035*	0.50
H2B	0.5152	1.0223	0.7235	0.035*	0.50
N2	0.44225 (6)	0.91302 (10)	0.67439 (9)	0.0279 (3)	
C217	0.46209 (8)	0.85271 (12)	0.62851 (12)	0.0325 (4)	
H21A	0.5069	0.8190	0.6780	0.039*	
H21B	0.4684	0.9004	0.5929	0.039*	
C211	0.41026 (8)	0.77066 (12)	0.56140 (11)	0.0287 (4)	
C212	0.36882 (8)	0.72257 (12)	0.57501 (11)	0.0342 (4)	
H212	0.3710	0.7436	0.6259	0.041*	
C213	0.32439 (9)	0.64434 (13)	0.51542 (13)	0.0443 (5)	
H213	0.2963	0.6121	0.5257	0.053*	
C214	0.32050 (10)	0.61278 (15)	0.44124 (13)	0.0492 (5)	
H214	0.2901	0.5586	0.4007	0.059*	
C215	0.36114 (10)	0.66048 (15)	0.42603 (12)	0.0469 (5)	
H215	0.3587	0.6392	0.3749	0.056*	
C216	0.40538 (9)	0.73944 (13)	0.48572 (11)	0.0366 (4)	
H216	0.4327	0.7726	0.4746	0.044*	

C227	0.38256 (8)	0.97856 (12)	0.60460 (11)	0.0304 (4)
H22A	0.3456	0.9347	0.5514	0.036*
H22B	0.3959	1.0292	0.5790	0.036*
C221	0.35458 (7)	1.03623 (12)	0.64375 (10)	0.0273 (4)
C222	0.35201 (8)	0.99267 (13)	0.71004 (11)	0.0311 (4)
H222	0.3692	0.9247	0.7326	0.037*
C223	0.32499 (8)	1.04652 (14)	0.74361 (11)	0.0370 (4)
H223	0.3245	1.0160	0.7897	0.044*
C224	0.29869 (8)	1.14460 (14)	0.71023 (12)	0.0400 (4)
H224	0.2800	1.1817	0.7331	0.048*
C225	0.29975 (8)	1.18829 (13)	0.64341 (12)	0.0397 (4)
H225	0.2809	1.2552	0.6194	0.048*
C226	0.32808 (8)	1.13546 (12)	0.61092 (11)	0.0335 (4)
H226	0.3295	1.1671	0.5660	0.040*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0330 (13)	0.0289 (12)	0.0330 (12)	0.000	0.0227 (11)	0.000
N1	0.0252 (7)	0.0311 (7)	0.0289 (7)	−0.0022 (6)	0.0187 (6)	−0.0009 (6)
C117	0.0220 (8)	0.0381 (9)	0.0330 (9)	0.0009 (7)	0.0166 (8)	0.0001 (7)
C111	0.0227 (8)	0.0317 (9)	0.0269 (8)	−0.0009 (7)	0.0167 (7)	0.0035 (7)
C112	0.0230 (8)	0.0338 (9)	0.0250 (8)	−0.0001 (7)	0.0122 (7)	0.0026 (7)
C113	0.0281 (9)	0.0334 (9)	0.0348 (9)	0.0033 (7)	0.0185 (8)	0.0047 (7)
C114	0.0406 (10)	0.0341 (9)	0.0374 (10)	−0.0026 (8)	0.0285 (9)	−0.0011 (8)
C115	0.0333 (10)	0.0449 (10)	0.0226 (8)	−0.0055 (8)	0.0147 (8)	−0.0006 (8)
C116	0.0256 (9)	0.0411 (10)	0.0271 (8)	0.0046 (8)	0.0137 (8)	0.0061 (8)
C127	0.0295 (9)	0.0349 (9)	0.0304 (9)	0.0034 (8)	0.0196 (8)	0.0055 (7)
C121	0.0309 (9)	0.0320 (9)	0.0283 (8)	−0.0004 (7)	0.0230 (8)	0.0006 (7)
C122	0.0319 (9)	0.0295 (9)	0.0335 (9)	0.0004 (8)	0.0214 (8)	0.0009 (7)
C123	0.0315 (9)	0.0455 (11)	0.0323 (9)	0.0034 (8)	0.0203 (8)	0.0062 (8)
C124	0.0327 (9)	0.0517 (11)	0.0314 (9)	−0.0101 (9)	0.0226 (8)	−0.0017 (8)
C125	0.0459 (11)	0.0368 (10)	0.0442 (10)	−0.0102 (9)	0.0325 (10)	−0.0015 (8)
C126	0.0387 (10)	0.0325 (9)	0.0395 (10)	0.0026 (8)	0.0289 (9)	0.0055 (8)
C2	0.0235 (12)	0.0298 (12)	0.0308 (12)	0.000	0.0156 (11)	0.000
N2	0.0199 (7)	0.0326 (7)	0.0279 (7)	0.0012 (6)	0.0139 (6)	−0.0042 (6)
C217	0.0291 (9)	0.0361 (9)	0.0375 (9)	−0.0005 (8)	0.0239 (8)	−0.0032 (8)
C211	0.0266 (8)	0.0290 (9)	0.0289 (8)	0.0047 (7)	0.0172 (7)	0.0029 (7)
C212	0.0328 (9)	0.0359 (9)	0.0357 (9)	−0.0023 (8)	0.0230 (8)	−0.0011 (8)
C213	0.0384 (11)	0.0351 (10)	0.0556 (12)	−0.0058 (9)	0.0287 (10)	−0.0014 (9)
C214	0.0339 (10)	0.0385 (11)	0.0467 (11)	−0.0010 (9)	0.0135 (9)	−0.0123 (9)
C215	0.0448 (11)	0.0533 (12)	0.0318 (10)	0.0126 (10)	0.0200 (9)	−0.0065 (9)
C216	0.0383 (10)	0.0402 (10)	0.0351 (9)	0.0067 (8)	0.0255 (8)	0.0044 (8)
C227	0.0248 (9)	0.0371 (9)	0.0262 (8)	0.0027 (7)	0.0151 (7)	0.0019 (7)
C221	0.0185 (8)	0.0305 (9)	0.0259 (8)	−0.0040 (7)	0.0112 (7)	−0.0049 (7)
C222	0.0244 (8)	0.0333 (9)	0.0320 (9)	0.0026 (7)	0.0167 (8)	0.0032 (7)
C223	0.0261 (9)	0.0512 (11)	0.0315 (9)	−0.0004 (8)	0.0178 (8)	−0.0035 (8)
C224	0.0234 (9)	0.0495 (11)	0.0363 (10)	0.0019 (8)	0.0146 (8)	−0.0115 (9)
C225	0.0287 (9)	0.0291 (9)	0.0440 (10)	0.0036 (8)	0.0158 (9)	−0.0046 (8)
C226	0.0282 (9)	0.0298 (9)	0.0317 (9)	−0.0011 (7)	0.0146 (8)	0.0003 (7)

Geometric parameters (Å, º)

C1—N1^{i}	1.4612 (17)	C2—N2	1.4678 (17)
C1—N1	1.4612 (17)	C2—N2^{ii}	1.4678 (17)
C1—H1A	0.9900	C2—H2A	0.9900
C1—H1B	0.9900	C2—H2B	0.9900
N1—C117	1.4685 (19)	N2—C217	1.4663 (19)
N1—C127	1.4694 (19)	N2—C227	1.4700 (19)
C117—C111	1.509 (2)	C217—C211	1.514 (2)
C117—H11A	0.9900	C217—H21A	0.9900
C117—H11B	0.9900	C217—H21B	0.9900
C111—C116	1.391 (2)	C211—C212	1.385 (2)
C111—C112	1.393 (2)	C211—C216	1.387 (2)
C112—C113	1.380 (2)	C212—C213	1.381 (2)
C112—H112	0.9500	C212—H212	0.9500
C113—C114	1.382 (2)	C213—C214	1.376 (3)
C113—H113	0.9500	C213—H213	0.9500
C114—C115	1.378 (2)	C214—C215	1.385 (3)
C114—H114	0.9500	C214—H214	0.9500
C115—C116	1.387 (2)	C215—C216	1.387 (2)
C115—H115	0.9500	C215—H215	0.9500
C116—H116	0.9500	C216—H216	0.9500
C127—C121	1.512 (2)	C227—C221	1.506 (2)
C127—H12A	0.9900	C227—H22A	0.9900
C127—H12B	0.9900	C227—H22B	0.9900
C121—C126	1.389 (2)	C221—C222	1.393 (2)
C121—C122	1.391 (2)	C221—C226	1.394 (2)
C122—C123	1.384 (2)	C222—C223	1.381 (2)
C122—H122	0.9500	C222—H222	0.9500
C123—C124	1.386 (2)	C223—C224	1.381 (2)
C123—H123	0.9500	C223—H223	0.9500
C124—C125	1.374 (2)	C224—C225	1.379 (2)
C124—H124	0.9500	C224—H224	0.9500
C125—C126	1.385 (2)	C225—C226	1.386 (2)
C125—H125	0.9500	C225—H225	0.9500
C126—H126	0.9500	C226—H226	0.9500
N1—C1—N1^{i}	110.27 (17)	N2—C2—N2^{ii}	110.83 (17)
N1^{i}—C1—H1A	109.6	N2—C2—H2A	109.5
N1—C1—H1A	109.6	N2^{ii}—C2—H2A	109.5
N1^{i}—C1—H1B	109.6	N2—C2—H2B	109.5
N1—C1—H1B	109.6	N2^{ii}—C2—H2B	109.5
H1A—C1—H1B	108.1	H2A—C2—H2B	108.1
C1—N1—C117	110.24 (11)	C217—N2—C2	110.21 (11)
C1—N1—C127	110.87 (12)	C217—N2—C227	110.33 (12)
C117—N1—C127	111.29 (12)	C2—N2—C227	109.87 (12)
N1—C117—C111	113.35 (13)	N2—C217—C211	113.81 (13)
N1—C117—H11A	108.9	N2—C217—H21A	108.8
C111—C117—H11A	108.9	C211—C217—H21A	108.8
N1—C117—H11B	108.9	N2—C217—H21B	108.8

C111—C117—H11B	108.9	C211—C217—H21B	108.8
H11A—C117—H11B	107.7	H21A—C217—H21B	107.7
C116—C111—C112	118.00 (15)	C212—C211—C216	118.43 (15)
C116—C111—C117	120.15 (14)	C212—C211—C217	121.96 (15)
C112—C111—C117	121.78 (14)	C216—C211—C217	119.57 (15)
C113—C112—C111	120.70 (14)	C213—C212—C211	120.79 (17)
C113—C112—H112	119.7	C213—C212—H212	119.6
C111—C112—H112	119.7	C211—C212—H212	119.6
C112—C113—C114	120.70 (16)	C214—C213—C212	120.46 (18)
C112—C113—H113	119.6	C214—C213—H213	119.8
C114—C113—H113	119.6	C212—C213—H213	119.8
C115—C114—C113	119.34 (16)	C213—C214—C215	119.60 (17)
C115—C114—H114	120.3	C213—C214—H214	120.2
C113—C114—H114	120.3	C215—C214—H214	120.2
C114—C115—C116	120.12 (15)	C214—C215—C216	119.72 (17)
C114—C115—H115	119.9	C214—C215—H215	120.1
C116—C115—H115	119.9	C216—C215—H215	120.1
C115—C116—C111	121.11 (15)	C211—C216—C215	120.98 (17)
C115—C116—H116	119.4	C211—C216—H216	119.5
C111—C116—H116	119.4	C215—C216—H216	119.5
N1—C127—C121	112.46 (12)	N2—C227—C221	114.00 (12)
N1—C127—H12A	109.1	N2—C227—H22A	108.8
C121—C127—H12A	109.1	C221—C227—H22A	108.8
N1—C127—H12B	109.1	N2—C227—H22B	108.8
C121—C127—H12B	109.1	C221—C227—H22B	108.8
H12A—C127—H12B	107.8	H22A—C227—H22B	107.6
C126—C121—C122	118.12 (15)	C222—C221—C226	118.11 (15)
C126—C121—C127	121.38 (14)	C222—C221—C227	122.29 (14)
C122—C121—C127	120.50 (14)	C226—C221—C227	119.57 (14)
C123—C122—C121	121.02 (16)	C223—C222—C221	121.17 (15)
C123—C122—H122	119.5	C223—C222—H222	119.4
C121—C122—H122	119.5	C221—C222—H222	119.4
C122—C123—C124	120.06 (16)	C224—C223—C222	120.12 (17)
C122—C123—H123	120.0	C224—C223—H223	119.9
C124—C123—H123	120.0	C222—C223—H223	119.9
C125—C124—C123	119.42 (16)	C225—C224—C223	119.51 (17)
C125—C124—H124	120.3	C225—C224—H224	120.2
C123—C124—H124	120.3	C223—C224—H224	120.2
C124—C125—C126	120.55 (16)	C224—C225—C226	120.61 (16)
C124—C125—H125	119.7	C224—C225—H225	119.7
C126—C125—H125	119.7	C226—C225—H225	119.7
C125—C126—C121	120.83 (16)	C225—C226—C221	120.45 (16)
C125—C126—H126	119.6	C225—C226—H226	119.8
C121—C126—H126	119.6	C221—C226—H226	119.8

N1$^{\rm i}$—C1—N1—C117	−70.90 (10)	N2$^{\rm ii}$—C2—N2—C217	−64.21 (10)
N1$^{\rm i}$—C1—N1—C127	165.41 (12)	N2$^{\rm ii}$—C2—N2—C227	174.01 (12)
C1—N1—C117—C111	163.10 (13)	C2—N2—C217—C211	169.98 (13)
C127—N1—C117—C111	−73.45 (16)	C227—N2—C217—C211	−68.51 (17)

N1—C117—C111—C116	143.96 (15)	N2—C217—C211—C212	−29.9 (2)
N1—C117—C111—C112	−39.3 (2)	N2—C217—C211—C216	152.47 (14)
C116—C111—C112—C113	0.2 (2)	C216—C211—C212—C213	0.9 (2)
C117—C111—C112—C113	−176.60 (15)	C217—C211—C212—C213	−176.75 (15)
C111—C112—C113—C114	1.1 (2)	C211—C212—C213—C214	0.0 (3)
C112—C113—C114—C115	−1.0 (2)	C212—C213—C214—C215	−0.5 (3)
C113—C114—C115—C116	−0.4 (2)	C213—C214—C215—C216	0.1 (3)
C114—C115—C116—C111	1.7 (3)	C212—C211—C216—C215	−1.3 (2)
C112—C111—C116—C115	−1.6 (2)	C217—C211—C216—C215	176.41 (15)
C117—C111—C116—C115	175.26 (15)	C214—C215—C216—C211	0.8 (3)
C1—N1—C127—C121	−68.89 (15)	C217—N2—C227—C221	174.78 (13)
C117—N1—C127—C121	168.02 (13)	C2—N2—C227—C221	−63.51 (15)
N1—C127—C121—C126	129.53 (15)	N2—C227—C221—C222	−37.5 (2)
N1—C127—C121—C122	−50.98 (19)	N2—C227—C221—C226	144.65 (14)
C126—C121—C122—C123	−0.2 (2)	C226—C221—C222—C223	−1.0 (2)
C127—C121—C122—C123	−179.67 (14)	C227—C221—C222—C223	−178.85 (14)
C121—C122—C123—C124	0.0 (2)	C221—C222—C223—C224	1.2 (2)
C122—C123—C124—C125	0.1 (3)	C222—C223—C224—C225	−0.1 (2)
C123—C124—C125—C126	0.1 (3)	C223—C224—C225—C226	−1.2 (2)
C124—C125—C126—C121	−0.2 (3)	C224—C225—C226—C221	1.5 (2)
C122—C121—C126—C125	0.3 (2)	C222—C221—C226—C225	−0.4 (2)
C127—C121—C126—C125	179.78 (15)	C227—C221—C226—C225	177.57 (14)

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