$C_{12}H_{26}NO_2^+.Br^-$

Table 2. *Selected geometric parameters (Å, °)*

O1—C31	1.194 (4)	C2—C3	1.503 (5)
O2—C31	1.314 (4)	C3—C4	1.526 (5)
N1—C1	1.521 (4)	C4—C5	1.495 (5)
N1—C32	1.492 (3)	C5—C6	1.519 (6)
N1—C33	1.508 (4)	C6—C7	1.496 (5)
N1—C34	1.510 (4)	C7—C8	1.518 (7)
C1—C2	1.519 (5)	C31—C32	1.507 (4)
C1—N1—C32	108.9 (2)	N1—C1—C2	115.7 (2)
C1—N1—C33	110.0 (2)	C1—C2—C3	110.2 (3)
C1—N1—C34	111.7 (2)	O1—C31—O2	125.5 (3)
C32—N1—C33	105.8 (2)	O1—C31—C32	126.8 (3)
C32—N1—C34	111.3 (2)	O2—C31—C32	107.7 (2)
C33—N1—C34	108.9 (2)	N1—C32—C31	117.8 (2)
C32—N1—C1—C2	169.3 (2)	O1—C31—C32—N1	9.1 (4)
C1—N1—C32—C31	60.3 (3)	O2—C31—C32—N1	−172.1 (2)
N1—C1—C2—C3	−177.4 (3)		

H2 was found by difference Fourier synthesis and all other H-atom positions were calculated assuming tetrahedral or staggered tetrahedral geometry and a C—H bond length of 0.95 Å. Isotropic displacement parameters for H atoms were calculated by multiplying the displacement parameter of the bonding partners by 1.3. The position of H2 was refined; all other H-atom parameters were fixed.

Data collection: CAD-4 diffractometer software (Enraf–Nonius, 1977). Cell refinement: CAD-4 diffractometer software. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN*. Molecular graphics: *PLATON*92 (Spek, 1990), *SCHAKAL*92 (Keller, 1992). Software used to prepare material for publication: *PLATON*92.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including torsion angles and contact distances, have been deposited with the IUCr (Reference: SE1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Carboxymethyl)dimethylundecyl-ammonium Bromide

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Abstract

The title compound, $C_{15}H_{32}NO_2^+.Br^-$, crystallizes to form layers with interdigitating hydrocarbon chains stabilized by $Br^- \cdots H$—O hydrogen bonds and $Br^- \cdots N^+$ contacts.

Comment

In a systematic study, analogies between the crystal structures and the self-assembling properties of the homologous *N-n*-alkylcarboxybetaines have been investigated. It is of special interest whether there are similarities between layers within the crystal structures of the amphiphilic homologues and the adsorption monolayers they form.

This is the second publication in a series describing the crystal structures of surface-active carboxybetaines. The aim is to find relationships between alkyl-chain length and molecular packing in the crystal. The questions to be addressed are whether there is an even–odd effect, *i.e.* whether the crystal structures of compounds with an even number of alkyl C atoms are different from the crystal structures of those with an odd number (Müller-Fahrnow, Hilgenfeld, Hesse, Saenger & Pfannemüller, 1988), and how long or how short the alkyl chain should be for the molecules to form a layered structure. In the first publication of this series the structure of the octyl betaine was presented (Rudert, Schulz, Reck, Vollhardt & Kriwanek, 1995).

The zigzag undecyl chain in the title compound, (I), is nearly planar. The atoms C2 to C11 have a maximum deviation from the least-squares plane of 0.08 (5) Å. C1, N1 and C31 deviate from the plane by 0.48 (5), 0.56 (5) and 0.90 (5) Å, respectively. The same kind of deviation of C1 from the plane was observed for the octyl betaine.

O1, O2, C31 and C32 are coplanar with a maximum deviation of 0.01 (3) Å. The conformation of the head region is the same as in the octyl betaine. The average C—C bond distance in the undecyl chain, excluding the short C10—C11 bond, is 1.47 Å. The C—C bond distances in the chain from C3 to C11 are alternately short and long.

(I)
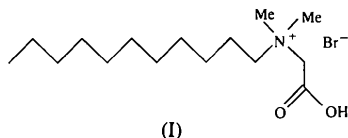
While the H atom of the carboxyl group in the octyl betaine was found by a difference Fourier synthesis, it could not be located in the title compound. However, as the two C—O bond lengths in the title compound are different and the head-group arrangement is the same as in the octyl betaine, the H atom must be bonded to O2.

The molecules form layers parallel to the (001) plane with interdigitating hydrocarbon chains. The zigzag planes of the chains are parallel $(T\|$ packing; see Abrahamsson, Dahlén, Löfgren & Pascher, 1978). In the polar region each Br⁻ ion is coordinated by three N atoms at distances of 4.231 (4), 4.264 (4) and 4.447 (4) Å (see Fig. 2). Two of the three Br···N contacts are intralayer contacts, the third connects different layers. One intralayer hydrogen bond, Br···H—O2, with a Br···O distance of 3.089 (4) Å, is formed.

All C···C distances between neighbouring alkyl chains are larger than 4 Å. Five C···C contacts are below 4.2 Å. The displacement parameters of the C atoms increase from C1 to C11 by a factor of two with a particularly large increase in thermal motion from C9 to C10. The terminal chain C atom, C11, is close to the polar group of a neighbouring molecule, C11···O1 being 3.442 (9) Å, which is distinctly shorter than in the octyl betaine.

The triclinic subcell has the cell constants a_s = 4.899, b_s = 5.253, c_s = 2.561 Å, α_s = 62.55, β_s = 112.96, γ_s = 99.27°. The direction of the chain axis is about $[20\bar{1}]$ and the angle between the polar plane and the chain axis is 50.3° (slightly less than for the octyl betaine). The area per alkyl chain is 25.40 Å² ($\frac{1}{2}absin\gamma$), which is only slightly greater than for the octyl betaine. This corresponds to a packing cross section per chain perpendicular to the chain axis of 19.54 Å².
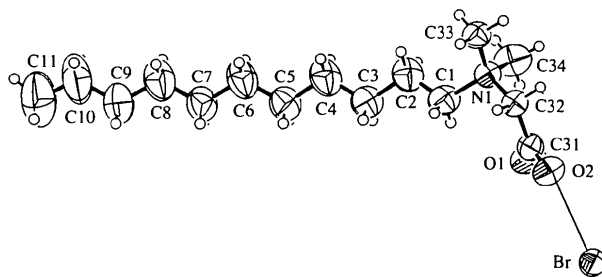

Fig. 1. ORTEPII (Johnson, 1976) drawing of the asymmetric unit. Displacement ellipsoids are shown at the 50% probability level for non-H atoms and H atoms are represented by spheres of arbitrary size. The hydrogen bond is represented by a thin line.
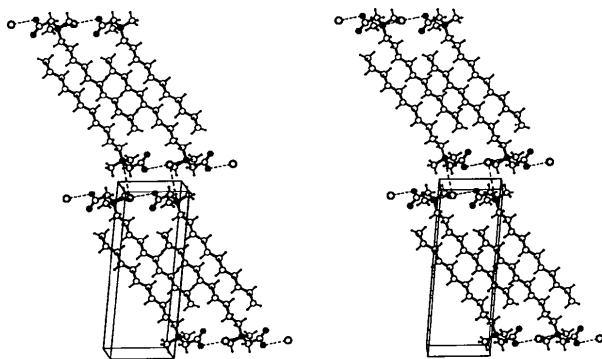
Experimental

The title compound was synthesized according to the detailed description given for N-n-alkyl-N,N-dimethylammonio acetic acid bromides (Wüstneck, Kriwanek, Herbst, Wasow & Haage, 1992). Equimolar quantities of N,N-dimethylglycine butyl ester and undecyl bromide were refluxed for 7 h in methanol. The product was hydrolyzed by addition of methanolic KOH and repeated refluxing for 1 h. Aqueous hydrobromic acid (48%) was added until the solution became acidic and the surplus inorganic salt was then removed. The title compound crystallized upon cooling. The product was filtered off, washed with acetone, dried using P_4O_{10} under vacuum, and recrystallized repeatedly from dry acetone containing small amounts of methanol.

Fig. 2. SCHAKAL92 (Keller, 1992) drawing of the molecular packing viewed along [010]. The Br⁻···N contacts and the hydrogen bonds are represented by dotted lines.

Crystal data

$C_{15}H_{32}NO_2^+.Br^-$	Mo $K\alpha$ radiation
M_r = 338.34	λ = 0.71073 Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
a = 6.791 (3) Å	θ = 10–16°
b = 7.554 (4) Å	μ = 2.200 mm⁻¹
c = 18.304 (7) Å	T = 294 K
α = 89.89 (5)°	Prismatic
β = 84.06 (4)°	0.65 × 0.38 × 0.20 mm
γ = 81.94 (8)°	Colourless
V = 924.7 (9) Å³	
Z = 2	
D_x = 1.22 Mg m⁻³	

Data collection

Enraf–Nonius CAD-4	R_{int} = 0.027
diffractometer	θ_{max} = 25.08°

$\theta/2\theta$ scans $h = -8 \rightarrow 8$
Absorption correction: $k = -8 \rightarrow 8$
  refined from ΔF (Walker $l = 0 \rightarrow 21$
  & Stuart, 1983)   2 standard reflections
3335 measured reflections   frequency: 30 min
3233 independent reflections   intensity decay: 6.69%
2926 observed reflections
  $[I > \sigma(I)]$

Refinement

Refinement on F $(\Delta/\sigma)_{max} = 0.011$
$R = 0.060$ $\Delta\rho_{max} = 0.71$ e Å^{-3}
$wR = 0.055$ $\Delta\rho_{min} = -1.48$ e Å^{-3}
$S = 0.928$ Extinction correction:
2926 reflections   Zachariasen (1977)
173 parameters   Extinction coefficient:
H atoms treated using a 5.1×10^{-7}
  riding model   Atomic scattering factors
$w = 1/\sigma^2(F_o)$ from *International Tables*
  where $\sigma(F_o^2) = [\sigma^2(I)$ *for X-ray Crystallography*
  $+ (pF_o^2)^2]$ and $p = 0.04$ (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br	0.75155 (8)	0.27720 (7)	0.11826 (3)	0.0701 (3)
O1	0.4360 (5)	0.8761 (5)	0.1544 (2)	0.088 (2)
O2	0.3848 (5)	1.1096 (5)	0.0802 (2)	0.081 (2)
N1	0.0755 (6)	0.7504 (5)	0.1107 (2)	0.062 (2)
C1	−0.0060 (9)	0.8059 (8)	0.1921 (3)	0.090 (4)
C2	−0.1455 (9)	0.7007 (9)	0.2292 (3)	0.093 (4)
C3	−0.2202 (9)	0.7698 (9)	0.3052 (3)	0.090 (4)
C4	−0.397 (1)	0.698 (1)	0.3404 (3)	0.103 (5)
C5	−0.474 (1)	0.7654 (9)	0.4150 (3)	0.097 (4)
C6	−0.665 (1)	0.708 (1)	0.4480 (4)	0.112 (5)
C7	−0.744 (1)	0.772 (1)	0.5213 (3)	0.102 (5)
C8	−0.934 (1)	0.717 (1)	0.5537 (4)	0.125 (6)
C9	−1.016 (1)	0.776 (1)	0.6269 (4)	0.120 (6)
C10	−1.204 (1)	0.719 (2)	0.6583 (5)	0.163 (8)
C11	−1.285 (2)	0.770 (1)	0.7249 (5)	0.19 (1)
C31	0.3429 (7)	0.9587 (7)	0.1096 (3)	0.067 (3)
C32	0.1660 (7)	0.9037 (6)	0.0775 (3)	0.060 (3)
C33	−0.0962 (7)	0.7282 (7)	0.0687 (3)	0.071 (3)
C34	0.2196 (9)	0.5832 (7)	0.1040 (4)	0.094 (4)

Table 2. *Selected geometric parameters* (Å, °)

O1—C31	1.207 (6)	C4—C5	1.476 (8)
O2—C31	1.314 (6)	C5—C6	1.50 (1)
N1—C1	1.572 (6)	C6—C7	1.451 (9)
N1—C32	1.486 (6)	C7—C8	1.47 (1)
N1—C33	1.489 (7)	C8—C9	1.44 (1)
N1—C34	1.482 (6)	C9—C10	1.47 (1)
C1—C2	1.438 (9)	C10—C11	1.32 (1)
C2—C3	1.499 (8)	C31—C32	1.499 (7)
C3—C4	1.48 (1)		
C1—N1—C32	106.9 (4)	N1—C1—C2	116.9 (5)
C1—N1—C33	109.0 (4)	C1—C2—C3	112.5 (6)
C1—N1—C34	113.8 (4)	O1—C31—O2	124.8 (5)
C32—N1—C33	106.4 (4)	O1—C31—C32	125.6 (5)
C32—N1—C34	111.5 (4)	O2—C31—C32	109.6 (4)
C33—N1—C34	108.9 (4)	N1—C32—C31	118.4 (4)
C32—N1—C1—C2	163.9 (5)	O1—C31—C32—N1	9.8 (8)
C1—N1—C32—C31	63.0 (5)	O2—C31—C32—N1	−171.4 (4)
N1—C1—C2—C3	−177.5 (5)		

Data collection: CAD-4 diffractometer software (Enraf–Nonius, 1977). Cell refinement: CAD-4 diffractometer soft-

ware. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: *MULTAN* (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP*II (Johnson, 1976), *SCHAKAL*92 (Keller, 1992). Software used to prepare material for publication: *MolEN BTABLE, PTABLE* and *CIF IN*.

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1-Trifluoroacetyl-3-trifluoromethyl-3a,8b-dihydro-1H,4H-indeno[1,2-c]pyrazole

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Abstract

The title structure, $C_{13}H_8F_6N_2O$, is the hydrolysis product of the rearranged 1,3-dipolar cycloadduct of 2,5-dichloro-1,1,1,6,6,6-hexafluoro-3,4-diazahexa-2,4-diene