

## *n*-Dodecylammonium bromide monohydrate

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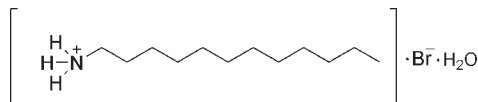
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.081; data-to-parameter ratio = 18.2.

In the title compound,  $\text{C}_{12}\text{H}_{28}\text{N}^+\cdot\text{Br}^-\cdot\text{H}_2\text{O}$ , the ionic pairs formed by *n*-dodecylammonium cations and bromide anions are arranged into thick layers; these layers are linked in a nearly perpendicular fashion [the angle between the layers is  $85.84(5)^\circ$ ] by hydrogen-bonding interactions involving the water molecules. The methylene part of the alkyl chain in the cation adopts an all-*trans* conformation. In the crystal structure, molecules are linked by intermolecular  $\text{N}-\text{H}\cdots\text{Br}$ ,  $\text{O}-\text{H}\cdots\text{Br}$  and  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

### Related literature

Long-chain *n*-alkylammonium halides are widely used as surfactants (Aratono *et al.*, 1998; Tornblom *et al.*, 2000) and as models for biological membranes (Ringsdorf *et al.*, 1988). They exhibit polymorphism at room temperature: for solid-solid phase transitions in *n*-alkylammonium chlorides, see: Terreros *et al.* (2000). For a related structure, see: Lundén (1974).



### Experimental

#### Crystal data

$\text{C}_{12}\text{H}_{28}\text{N}^+\cdot\text{Br}^-\cdot\text{H}_2\text{O}$

$M_r = 284.28$

Monoclinic,  $Cc$

$a = 4.7921(5)$  Å

$b = 42.810(4)$  Å

$c = 7.8573(8)$  Å

$\beta = 105.798(2)^\circ$

$V = 1551.0(3)$  Å<sup>3</sup>

$Z = 4$

Mo  $K\alpha$  radiation

$\mu = 2.63$  mm<sup>-1</sup>

$T = 293$  K

$0.42 \times 0.14 \times 0.06$  mm

#### Data collection

Siemens SMART CCD area-detector diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.404$ ,  $T_{\max} = 0.858$

4760 measured reflections  
2644 independent reflections  
1665 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.047$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.081$

$S = 0.92$

2644 reflections

145 parameters

5 restraints

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\max} = 0.40$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

Absolute structure: Flack (1983), 945 Friedel pairs

Flack parameter: 0.048 (19)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                                      | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1A}\cdots\text{Br1}^{\text{i}}$  | 0.89     | 2.53        | 3.380 (4)   | 159           |
| $\text{N1}-\text{H1B}\cdots\text{Br1}$             | 0.89     | 2.47        | 3.340 (4)   | 166           |
| $\text{N1}-\text{H1C}\cdots\text{O1}$              | 0.89     | 1.97        | 2.834 (7)   | 165           |
| $\text{O1}-\text{H1}\cdots\text{Br1}^{\text{ii}}$  | 0.83 (4) | 2.76 (6)    | 3.329 (5)   | 128 (6)       |
| $\text{O1}-\text{H2}\cdots\text{Br1}^{\text{iii}}$ | 0.97 (4) | 2.49 (5)    | 3.361 (5)   | 149 (5)       |

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

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); 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 DIAMOND (Brandenburg, 1998); 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: LX2141).

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**supplementary materials**

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## ***n*-Dodecylammonium bromide monohydrate**

**W. Dan, Y. Di, D. He, W. Yang and Y. Kong**

### **Comment**

Long-chain *n*-alkylammonium halides are widely used as surfactants (Aratono *et al.*, 1998; Tornblom *et al.*, 2000) and as models for biological membranes (Ringsdorf *et al.*, 1988). They exhibit polymorphism at room temperature; solid-solid phase transitions occurred in *n*-alkylammonium chlorides (Terreros *et al.*, 2000). As a part of the studies on novel potential phase transfer materials with the thermochemical properties such as *n*-alkylammonium chlorides, we report the crystal structure of the title compound (Fig. 1).

Atoms C3–C12 are coplanar in the title compound; however, atoms C2–C12 are coplanar in *n*-dodecylammonium bromide (Ludén, 1974). Although the methylene chain had the extended all-*trans* conformation, it is slightly curved in the vicinity of the ammonium group, to accommodate the hydrogen-bonding interactions. The hydrogen bonds of *n*-dodecylammonium bromide monohydrate are more stronger than that of *n*-dodecylammonium bromide because of N—H $\cdots$ O and O—H $\cdots$ Br hydrogen bonds. Only torsion angle C1–C2–C3–C4 deviates significantly from 180°, with a value of 170.6 (5)°. The crystal packing (Fig. 2) is stabilized by intermolecular N—H $\cdots$ Br, N—H $\cdots$ O and O—H $\cdots$ Br hydrogen bonds (Table 1).

### **Experimental**

*n*-Dodecylammonium bromide monohydrate was prepared by the addition of hydrobromic acid to an ethanol solution of *n*-dodecylamine. The resulting precipitate was filtered off and recrystallized several times from chloroform. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in chloroform at room temperature. Analysis, calculated for C<sub>12</sub>H<sub>30</sub>BrNO (Mr = 284.28): C 50.69, H 10.64, N 4.93, O 5.63, Br 28.11%; found: C 50.67, H 10.65, N 4.91, O 5.64, Br 28.13%.

### **Refinement**

The reported Flack parameter was obtained by TWIN/BASF procedure in SHELXL (Sheldrick, 2008). Water molecule bound H atoms were located in difference Fourier maps and their positional parameters refined with a distance restraint [O1—H1 = 0.85 (5) & O1—H2 = 0.80 (5) Å] and a angle restraint. The H atoms of C and N atoms were positioned geometrically, with methylene C—H distances of 0.97 Å, methyl C—H distances of 0.96 Å, N—H 0.89 Å and refined as riding on their parent atoms. The  $U_{\text{iso}}(\text{H})$  values were set at  $1.2U_{\text{eq}}$  for the methylene H atoms and at  $1.5U_{\text{eq}}$  for other H atoms.

### **Figures**



Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

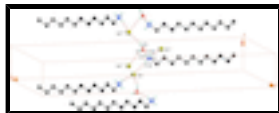


Fig. 2. N—H...Br, N—H...O and O—H...Br interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i)  $x + 1, -y + 1, z + 1/2$ ; (ii)  $x, -y + 1, z - 1/2$ ; (iii)  $x + 1, -y + 1, z - 1/2$ ]

## *n*-Dodecylammonium bromide monohydrate

### Crystal data

$C_{12}H_{28}N^+ \cdot Br^- \cdot H_2O$

$M_r = 284.28$

Monoclinic, *Cc*

Hall symbol: C -2yc

$a = 4.7921$  (5) Å

$b = 42.810$  (4) Å

$c = 7.8573$  (8) Å

$\beta = 105.798$  (2)°

$V = 1551.0$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 608$

$D_x = 1.217$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1266 reflections

$\theta = 2.9$ – $25.0$ °

$\mu = 2.63$  mm<sup>-1</sup>

$T = 293$  K

Acicular, colourless

$0.42 \times 0.14 \times 0.06$  mm

### Data collection

Siemens SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

Detector resolution: 10 pixels mm<sup>-1</sup>

$\phi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.404$ ,  $T_{\max} = 0.858$

4760 measured reflections

2644 independent reflections

1665 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 27.0$ °,  $\theta_{\min} = 1.9$ °

$h = -6 \rightarrow 5$

$k = -54 \rightarrow 50$

$l = -6 \rightarrow 10$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.081$

$S = 0.92$

2644 reflections

145 parameters

5 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0277P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.40$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

Absolute structure: Flack (1983), 945 Friedel pairs

Flack parameter: 0.048 (19)

*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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

|     | x           | y             | z            | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|---------------|--------------|----------------------------------|
| Br1 | 0.2086 (2)  | 0.524590 (10) | 0.38293 (16) | 0.05643 (18)                     |
| O1  | 0.8268 (12) | 0.45062 (13)  | 0.1580 (6)   | 0.0760 (15)                      |
| H1  | 0.734 (13)  | 0.4485 (18)   | 0.053 (6)    | 0.114*                           |
| H2  | 0.993 (11)  | 0.4589 (15)   | 0.124 (9)    | 0.114*                           |
| N1  | 0.7249 (9)  | 0.46990 (8)   | 0.4809 (6)   | 0.0502 (12)                      |
| H1A | 0.8831      | 0.4731        | 0.5697       | 0.075*                           |
| H1B | 0.6083      | 0.4864        | 0.4696       | 0.075*                           |
| H1C | 0.7755      | 0.4671        | 0.3810       | 0.075*                           |
| C1  | 0.5729 (12) | 0.44200 (13)  | 0.5176 (8)   | 0.0496 (17)                      |
| H1D | 0.3952      | 0.4392        | 0.4238       | 0.059*                           |
| H1E | 0.5212      | 0.4449        | 0.6276       | 0.059*                           |
| C2  | 0.7567 (11) | 0.41323 (10)  | 0.5313 (7)   | 0.0480 (14)                      |
| H2A | 0.9345      | 0.4161        | 0.6249       | 0.058*                           |
| H2B | 0.8084      | 0.4104        | 0.4213       | 0.058*                           |
| C3  | 0.6028 (11) | 0.38403 (11)  | 0.5696 (7)   | 0.0496 (14)                      |
| H3A | 0.5795      | 0.3855        | 0.6881       | 0.059*                           |
| H3B | 0.4108      | 0.3831        | 0.4875       | 0.059*                           |
| C4  | 0.7635 (11) | 0.35416 (10)  | 0.5545 (7)   | 0.0466 (14)                      |
| H4A | 0.7908      | 0.3531        | 0.4367       | 0.056*                           |
| H4B | 0.9540      | 0.3550        | 0.6382       | 0.056*                           |
| C5  | 0.6137 (11) | 0.32477 (11)  | 0.5877 (7)   | 0.0484 (14)                      |
| H5A | 0.5955      | 0.3254        | 0.7076       | 0.058*                           |
| H5B | 0.4194      | 0.3245        | 0.5082       | 0.058*                           |
| C6  | 0.7625 (11) | 0.29500 (11)  | 0.5641 (7)   | 0.0488 (14)                      |
| H6A | 0.9550      | 0.2951        | 0.6458       | 0.059*                           |
| H6B | 0.7859      | 0.2947        | 0.4453       | 0.059*                           |
| C7  | 0.6105 (11) | 0.26515 (11)  | 0.5920 (7)   | 0.0454 (13)                      |
| H7A | 0.5909      | 0.2651        | 0.7117       | 0.055*                           |
| H7B | 0.4168      | 0.2651        | 0.5117       | 0.055*                           |
| C8  | 0.7621 (11) | 0.23602 (11)  | 0.5640 (7)   | 0.0484 (14)                      |
| H8A | 0.9555      | 0.2361        | 0.6447       | 0.058*                           |
| H8B | 0.7827      | 0.2362        | 0.4446       | 0.058*                           |
| C9  | 0.6128 (11) | 0.20610 (11)  | 0.5905 (7)   | 0.0495 (14)                      |

## supplementary materials

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|      |             |              |            |             |
|------|-------------|--------------|------------|-------------|
| H9A  | 0.4180      | 0.2062       | 0.5116     | 0.059*      |
| H9B  | 0.5957      | 0.2058       | 0.7107     | 0.059*      |
| C10  | 0.7642 (12) | 0.17604 (11) | 0.5585 (7) | 0.0508 (14) |
| H10A | 0.7842      | 0.1766       | 0.4389     | 0.061*      |
| H10B | 0.9579      | 0.1757       | 0.6387     | 0.061*      |
| C11  | 0.6143 (13) | 0.14651 (12) | 0.5818 (8) | 0.0592 (16) |
| H11A | 0.5946      | 0.1459       | 0.7014     | 0.071*      |
| H11B | 0.4205      | 0.1468       | 0.5016     | 0.071*      |
| C12  | 0.7623 (15) | 0.11758 (13) | 0.5502 (9) | 0.080 (2)   |
| H12A | 0.7850      | 0.1179       | 0.4325     | 0.121*      |
| H12B | 0.6483      | 0.0998       | 0.5634     | 0.121*      |
| H12C | 0.9496      | 0.1163       | 0.6342     | 0.121*      |

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$   | $U^{13}$   | $U^{23}$    |
|-----|------------|------------|------------|------------|------------|-------------|
| Br1 | 0.0519 (3) | 0.0577 (3) | 0.0587 (3) | 0.0057 (6) | 0.0134 (2) | 0.0063 (5)  |
| O1  | 0.078 (3)  | 0.085 (4)  | 0.069 (3)  | 0.006 (3)  | 0.025 (3)  | 0.006 (3)   |
| N1  | 0.051 (3)  | 0.038 (3)  | 0.056 (3)  | 0.006 (2)  | 0.005 (2)  | 0.0026 (19) |
| C1  | 0.050 (4)  | 0.032 (3)  | 0.067 (5)  | 0.001 (3)  | 0.017 (3)  | 0.004 (3)   |
| C2  | 0.052 (3)  | 0.036 (3)  | 0.057 (4)  | -0.001 (3) | 0.016 (3)  | 0.000 (2)   |
| C3  | 0.053 (4)  | 0.043 (3)  | 0.059 (4)  | 0.001 (3)  | 0.027 (3)  | 0.007 (3)   |
| C4  | 0.047 (3)  | 0.039 (3)  | 0.056 (4)  | 0.000 (3)  | 0.017 (3)  | 0.001 (3)   |
| C5  | 0.057 (4)  | 0.041 (3)  | 0.052 (3)  | 0.001 (3)  | 0.024 (3)  | -0.005 (3)  |
| C6  | 0.053 (3)  | 0.044 (3)  | 0.049 (3)  | -0.003 (3) | 0.013 (3)  | 0.000 (3)   |
| C7  | 0.048 (3)  | 0.036 (3)  | 0.056 (3)  | -0.001 (3) | 0.020 (3)  | 0.001 (3)   |
| C8  | 0.050 (3)  | 0.042 (3)  | 0.055 (3)  | -0.003 (3) | 0.017 (3)  | -0.001 (3)  |
| C9  | 0.051 (4)  | 0.046 (3)  | 0.056 (4)  | -0.007 (3) | 0.021 (3)  | -0.001 (3)  |
| C10 | 0.058 (4)  | 0.048 (3)  | 0.047 (3)  | 0.000 (3)  | 0.015 (3)  | 0.001 (3)   |
| C11 | 0.073 (4)  | 0.041 (3)  | 0.067 (4)  | -0.011 (3) | 0.026 (3)  | -0.001 (3)  |
| C12 | 0.104 (5)  | 0.041 (4)  | 0.097 (5)  | 0.003 (4)  | 0.028 (4)  | -0.007 (3)  |

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

|        |           |          |           |
|--------|-----------|----------|-----------|
| O1—H1  | 0.83 (4)  | C6—C7    | 1.516 (6) |
| O1—H2  | 0.97 (4)  | C6—H6A   | 0.9700    |
| N1—C1  | 1.468 (7) | C6—H6B   | 0.9700    |
| N1—H1A | 0.8900    | C7—C8    | 1.490 (6) |
| N1—H1B | 0.8900    | C7—H7A   | 0.9700    |
| N1—H1C | 0.8900    | C7—H7B   | 0.9700    |
| C1—C2  | 1.501 (7) | C8—C9    | 1.509 (7) |
| C1—H1D | 0.9700    | C8—H8A   | 0.9700    |
| C1—H1E | 0.9700    | C8—H8B   | 0.9700    |
| C2—C3  | 1.522 (6) | C9—C10   | 1.532 (7) |
| C2—H2A | 0.9700    | C9—H9A   | 0.9700    |
| C2—H2B | 0.9700    | C9—H9B   | 0.9700    |
| C3—C4  | 1.514 (7) | C10—C11  | 1.490 (7) |
| C3—H3A | 0.9700    | C10—H10A | 0.9700    |
| C3—H3B | 0.9700    | C10—H10B | 0.9700    |

|             |           |               |            |
|-------------|-----------|---------------|------------|
| C4—C5       | 1.506 (6) | C11—C12       | 1.481 (8)  |
| C4—H4A      | 0.9700    | C11—H11A      | 0.9700     |
| C4—H4B      | 0.9700    | C11—H11B      | 0.9700     |
| C5—C6       | 1.496 (6) | C12—H12A      | 0.9600     |
| C5—H5A      | 0.9700    | C12—H12B      | 0.9600     |
| C5—H5B      | 0.9700    | C12—H12C      | 0.9600     |
| H1—O1—H2    | 91 (5)    | C5—C6—H6B     | 108.3      |
| C1—N1—H1A   | 109.5     | C7—C6—H6B     | 108.3      |
| C1—N1—H1B   | 109.5     | H6A—C6—H6B    | 107.4      |
| H1A—N1—H1B  | 109.5     | C8—C7—C6      | 114.3 (4)  |
| C1—N1—H1C   | 109.5     | C8—C7—H7A     | 108.7      |
| H1A—N1—H1C  | 109.5     | C6—C7—H7A     | 108.7      |
| H1B—N1—H1C  | 109.5     | C8—C7—H7B     | 108.7      |
| N1—C1—C2    | 111.6 (5) | C6—C7—H7B     | 108.7      |
| N1—C1—H1D   | 109.3     | H7A—C7—H7B    | 107.6      |
| C2—C1—H1D   | 109.3     | C7—C8—C9      | 114.9 (4)  |
| N1—C1—H1E   | 109.3     | C7—C8—H8A     | 108.5      |
| C2—C1—H1E   | 109.3     | C9—C8—H8A     | 108.5      |
| H1D—C1—H1E  | 108.0     | C7—C8—H8B     | 108.5      |
| C1—C2—C3    | 112.4 (4) | C9—C8—H8B     | 108.5      |
| C1—C2—H2A   | 109.1     | H8A—C8—H8B    | 107.5      |
| C3—C2—H2A   | 109.1     | C8—C9—C10     | 115.3 (4)  |
| C1—C2—H2B   | 109.1     | C8—C9—H9A     | 108.5      |
| C3—C2—H2B   | 109.1     | C10—C9—H9A    | 108.5      |
| H2A—C2—H2B  | 107.9     | C8—C9—H9B     | 108.5      |
| C4—C3—C2    | 113.2 (4) | C10—C9—H9B    | 108.5      |
| C4—C3—H3A   | 108.9     | H9A—C9—H9B    | 107.5      |
| C2—C3—H3A   | 108.9     | C11—C10—C9    | 115.3 (4)  |
| C4—C3—H3B   | 108.9     | C11—C10—H10A  | 108.5      |
| C2—C3—H3B   | 108.9     | C9—C10—H10A   | 108.5      |
| H3A—C3—H3B  | 107.8     | C11—C10—H10B  | 108.5      |
| C5—C4—C3    | 114.6 (4) | C9—C10—H10B   | 108.5      |
| C5—C4—H4A   | 108.6     | H10A—C10—H10B | 107.5      |
| C3—C4—H4A   | 108.6     | C12—C11—C10   | 114.8 (5)  |
| C5—C4—H4B   | 108.6     | C12—C11—H11A  | 108.6      |
| C3—C4—H4B   | 108.6     | C10—C11—H11A  | 108.6      |
| H4A—C4—H4B  | 107.6     | C12—C11—H11B  | 108.6      |
| C6—C5—C4    | 115.1 (4) | C10—C11—H11B  | 108.6      |
| C6—C5—H5A   | 108.5     | H11A—C11—H11B | 107.5      |
| C4—C5—H5A   | 108.5     | C11—C12—H12A  | 109.5      |
| C6—C5—H5B   | 108.5     | C11—C12—H12B  | 109.5      |
| C4—C5—H5B   | 108.5     | H12A—C12—H12B | 109.5      |
| H5A—C5—H5B  | 107.5     | C11—C12—H12C  | 109.5      |
| C5—C6—C7    | 115.9 (4) | H12A—C12—H12C | 109.5      |
| C5—C6—H6A   | 108.3     | H12B—C12—H12C | 109.5      |
| C7—C6—H6A   | 108.3     |               |            |
| N1—C1—C2—C3 | 179.9 (4) | C5—C6—C7—C8   | 178.9 (4)  |
| C1—C2—C3—C4 | 170.6 (5) | C6—C7—C8—C9   | -179.7 (5) |

## supplementary materials

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|             |            |                |            |
|-------------|------------|----------------|------------|
| C2—C3—C4—C5 | -178.7 (4) | C7—C8—C9—C10   | 178.9 (4)  |
| C3—C4—C5—C6 | 177.0 (5)  | C8—C9—C10—C11  | -179.1 (5) |
| C4—C5—C6—C7 | -178.4 (4) | C9—C10—C11—C12 | 179.9 (5)  |

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

| $D-H\cdots A$                     | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|----------|-------------|-------------|---------------|
| N1—H1A $\cdots$ Br1 <sup>i</sup>  | 0.89     | 2.53        | 3.380 (4)   | 159.          |
| N1—H1B $\cdots$ Br1               | 0.89     | 2.47        | 3.340 (4)   | 166.          |
| N1—H1C $\cdots$ O1                | 0.89     | 1.97        | 2.834 (7)   | 165.          |
| O1—H1 $\cdots$ Br1 <sup>ii</sup>  | 0.83 (4) | 2.76 (6)    | 3.329 (5)   | 128 (6)       |
| O1—H2 $\cdots$ Br1 <sup>iii</sup> | 0.97 (4) | 2.49 (5)    | 3.361 (5)   | 149 (5)       |

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



Fig. 1

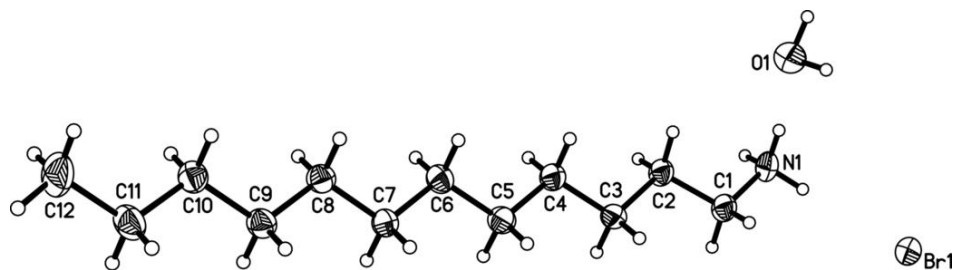


Fig. 2

