2-Hydroxyethylammonium [2-(2,6-dichloroanilino)-phenyl]acetate monohydrate

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In the solid-state structure of the title compound derived from diclofenac, C₂H₈NO⁺·C₁₄H₁₀Cl₂NO₂⁻·H₂O, the asymmetric unit contains one cation, one anion and a water molecule, all in general positions. A complex network of hydrogen bonds is present in the crystal structure.

Structure description

The pharmaceutical diclofenac (D) is widely used as a non-steroidal anti-inflammatory drug, to treat pain and inflammatory diseases (Skoutakis et al., 1988; Moser et al., 1990). The Cambridge Structural Database (CSD version 5.42, last update February 2021; Groom et al., 2016) includes crystallographic data for 50 entries with the term ‘diclofenac’. Among them, there are 21 entries where diclofenac is present in the form of a salt, and in three entries, diclofenac forms salts with aliphatic amines: with (R) and (S)-phenylethylammonium (Lemmerer et al., 2010), with diethyl ammonium (Castellari et al., 2001) and with tris(2-ammonioethyl)amine (Lynch et al., 2003). In this article, we present another complex in the form of a diclofenac salt with an amino-containing compound, namely monoethanolamine. Ethanolamine is always present in significant quantities in the human and animal body with a complete protein diet. Its formation occurs during the decarboxylation of serine, and in one of the metabolic variants, it turns into glycine (the simplest aliphatic amino acid; Wishart et al., 2007). In addition, monoethanolamine is used in some cosmetic products (Knaak et al., 1997). Therefore, the interaction of these compounds seems to be interesting for investigation.

The crystal structure of the title compound has one monoethanolamine (MEA) cation, one 2-(2,6-dichloroanilino)phenylacetate anion, and one water molecule in the asymmetric unit, and crystallizes in space group P2₁/c (Fig. 1). The diclofenac anion is stabilized by one intramolecular hydrogen bond between the amino group and atom O1 of the carboxylic group: N1—H1···O1 [2.884 (3) Å, 128.9°; see
Table 1

| Hydrogen-bond geometry (Å, °). | D—H · · · A | D—H | H · · · A | D · · · A | D—H · · · A |
|-------------------------------|-------------|------|-----------|-----------|-------------|
| N1—H1···O1                 | 0.86        | 2.27 | 2.884 (3) | 129       |
| N2—H2A···O2                 | 0.89        | 1.96 | 2.811 (4) | 160       |
| N2—H2B···O1W                | 0.89        | 2.15 | 2.947 (4) | 148       |
| N2—H2C···O1W                | 0.89        | 1.92 | 2.802 (3) | 169       |
| O3—H3A···O1W                | 0.82        | 1.96 | 2.770 (4) | 168       |
| O1W—H1WB···O2              | 0.85        | 1.87 | 2.690 (3) | 161       |
| O1W—H1WA···O1W              | 0.85        | 2.00 | 2.809 (3) | 158       |

Symmetry codes: (i) x, y + 1, z; (ii) x, y – 1/2, –z + 1/2; (iii) x, y – 1, z; (iv) x, y + 3, z + 1/2.

gate parallel to the (100) plane, where the chains are related by the glide plane c [O1W···O1w, symmetry code: (iv) x, y + 3, z + 1/2, 2.809 (3) Å] and the inversion centre [N2···O2, symmetry code: (i) x, y + 1, z + 1, 2.811 (4) Å, Fig. 2]. The layers are linked by Y···Cg π–ring interactions, for C3···Cl and C7···Cl1 bonds, for which the X···Cg separations and y angles range from 3.533 to 3.958 Å.

In order to visualize the intermolecular interactions in the crystal of the title compound, a Hirshfeld surface analysis was carried out using Crystal Explorer 17.5 (Turner et al., 2017). The Hirshfeld surface mapped over dnorm shows the expected bright-red spots near atoms O1 and O2, involved in the O···H and N···O hydrogen-bonding interactions (Fig. 3). Fingerprint plots (Fig. 4) reveal that H···H, H···C/Cl···H, H···Cl/Cl···H and O···O contacts make the greatest contributions to the surface contacts (Table 1), while H···N/N···H, C···C and O···O contacts are much less significant.

Synthesis and crystallization

To a solution of 0.1 g (0.52 mmol) of D in 4 ml of ethanol, 32 μL of monoethanolamine was added. The mixture was kept
in an ultrasonic bath (30 kHz) at 298 K for 5 min. The solution was then placed in a loosely closed bottle and kept at 298 K for 10 days. The precipitated prismatic crystals were selected for the single-crystal X-ray diffraction analysis.

**Refinement**

Crystal data, data collection and structure refinement details are summarized in Table 2.

**Funding information**

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**Table 2**

**Experimental details.**

| Crystal data | Chemical formula | C₂H₈NO⁺–C₆H₅ClNO₃⁻H₂O |
|--------------|------------------|--------------------------|
| M, a, b, c (Å) | 375.24           | Monoclinic, P2₁/c        |
| Temperature (K) | 293              | 19.1257 (10), 9.3864 (5), |
|                  |                  | 10.0502 (6)              |
| β (°) | 103.546 (6) |
| V (Å³) | 1754.05 (17) |
| Z | 4 |
| μ (mm⁻¹) | 3.53 |
| Crystal size (mm) | 0.31 × 0.28 × 0.1 |

**Data collection**

| Agilent Technologies Xcalibur, Ruby |

**Absorption correction**

Multi-scan (CrysAlis PRO).

| T_min, T_max | 0.556, 1.000 |
| No. of measured, independent and observed | 12416, 3621, 2431 |
| H-atoms treatment | H-atom parameters constrained |
| Δρ_max, Δρ_min (e Å⁻³) | 0.40, –0.35 |

**Diffractometer**

Agilent Technologies Xcalibur.

**Computer programs:**

CrysAlis PRO (Agilent, 2014), SHELXT2018/2 (Sheldrick, 2015α), SHELXL2018/3 (Sheldrick, 2015β), XP (Siemens, 1994), Mercury (Macrae et al., 2020) and pubCIF (Westrip, 2010).

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full crystallographic data

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Crystal data

\[
\begin{align*}
C_9H_{16}NO^+\cdot C_{14}H_9Cl_2NO_2^-\cdot H_2O & \quad F(000) = 784 \\
M_r = 375.24 & \quad D_a = 1.421 \text{ Mg m}^{-3} \\
\text{Monoclinic, } P2_1/c & \quad \text{Cu Kα radiation, } \lambda = 1.54184 \text{ Å} \\
a = 19.1257 (10) \text{ Å} & \quad \text{Cell parameters from 2166 reflections} \\
b = 9.3864 (5) \text{ Å} & \quad \theta = 4.7-73.9^\circ \\
c = 10.0502 (6) \text{ Å} & \quad \mu = 3.53 \text{ mm}^{-1} \\
\beta = 103.546 (6)^\circ & \quad T = 293 \text{ K} \\
V = 1754.05 (17) \text{ Å}^3 & \quad \text{Prism, colourless} \\
Z = 4 & \quad 0.31 \times 0.28 \times 0.1 \text{ mm} \\
\end{align*}
\]

Data collection

Agilent Technologies Xcalibur, Ruby diffractometer

Radiation source: fine-focus sealed tube

\(\omega\) scans

Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)

\(T_{\text{min}} = 0.356, T_{\text{max}} = 1.000\)

12416 measured reflections

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.051\)

\(wR(F^2) = 0.134\)

\(S = 1.01\)

3621 independent reflections

222 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H-atom parameters constrained

\(w = \frac{1}{[\sigma^2(F_c^2) + (0.0614P)^2]}\)

where \(P = (F_c^2 + 2F_s^2)/3\)

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

\(\Delta\rho_{\text{max}} = 0.40 \text{ e Å}^{-3}\)

\(\Delta\rho_{\text{min}} = -0.35 \text{ e Å}^{-3}\)

Special details

Refinement. All hydrogen atoms were placed in idealized positions and refined as riding to their carrier atoms.

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

|     | \(x\)       | \(y\)       | \(z\)       | \(U_{iso}/U_{eq}\) |
|-----|-------------|-------------|-------------|---------------------|
| Cl1 | 0.46202 (4) | 0.95109 (8) | 0.76764 (8) | 0.0441 (2)         |
| Cl2 | 0.24250 (4) | 1.20690 (9) | 0.40514 (9) | 0.0500 (2)         |
| Atom   | U11  | U22  | U33  | U12  | U13  | U23  |
|--------|------|------|------|------|------|------|
| O1     | 0.1500 (11) | 0.9926 (2) | 0.5897 (2) | 0.0472 (6) |      |      |
| O1W    | 0.07192 (13) | 0.6316 (3) | 0.8457 (2) | 0.0558 (6) |      |      |
| H1WA   | 0.103624 | 0.612215 | 0.917982 | 0.084* |      |      |
| H1WB   | 0.085392 | 0.710654 | 0.818732 | 0.084* |      |      |
| N1     | 0.30390 (13) | 0.9959 (2) | 0.6226 (3) | 0.0379 (6) |      |      |
| H1     | 0.269351 | 1.041181 | 0.645204 | 0.045* |      |      |
| O2     | 0.08825 (11) | 0.8748 (3) | 0.7146 (2) | 0.0592 (7) |      |      |
| N2     | 0.03573 (14) | 0.1808 (3) | 0.4921 (3) | 0.0446 (6) |      |      |
| H2A    | 0.003371 | 0.148791 | 0.419647 | 0.054* |      |      |
| H2B    | 0.015651 | 0.187499 | 0.563284 | 0.054* |      |      |
| H2C    | 0.072670 | 0.120586 | 0.512028 | 0.054* |      |      |
| O3     | 0.07091 (17) | 0.3857 (3) | 0.6928 (3) | 0.0762 (9) |      |      |
| H3A    | 0.073883 | 0.464400 | 0.729146 | 0.114* |      |      |
| C2     | 0.42880 (15) | 1.0634 (3) | 0.6305 (3) | 0.0327 (6) |      |      |
| C14    | 0.14604 (15) | 0.9162 (3) | 0.6906 (3) | 0.0371 (7) |      |      |
| C1     | 0.35476 (15) | 1.0754 (3) | 0.5751 (3) | 0.0322 (6) |      |      |
| C12    | 0.26283 (14) | 0.7823 (3) | 0.7158 (3) | 0.0342 (6) |      |      |
| C7     | 0.30532 (15) | 0.8463 (3) | 0.6360 (3) | 0.0342 (6) |      |      |
| C8     | 0.34720 (15) | 0.7613 (3) | 0.5707 (3) | 0.0382 (7) |      |      |
| H8     | 0.374195 | 0.803323 | 0.515422 | 0.046* |      |      |
| C6     | 0.33381 (16) | 1.1786 (3) | 0.4739 (3) | 0.0359 (6) |      |      |
| C13    | 0.21653 (15) | 0.8708 (3) | 0.7865 (3) | 0.0383 (7) |      |      |
| H13A   | 0.243062 | 0.955166 | 0.824815 | 0.046* |      |      |
| H13B   | 0.206067 | 0.816317 | 0.861507 | 0.046* |      |      |
| C9     | 0.34871 (16) | 0.6146 (3) | 0.5877 (3) | 0.0447 (8) |      |      |
| H9     | 0.377768 | 0.559121 | 0.546037 | 0.054* |      |      |
| C5     | 0.38298 (18) | 1.2604 (3) | 0.4257 (3) | 0.0430 (7) |      |      |
| H5     | 0.366991 | 1.328721 | 0.358405 | 0.052* |      |      |
| C4     | 0.45510 (18) | 1.2404 (3) | 0.4774 (3) | 0.0459 (8) |      |      |
| H4     | 0.488229 | 1.292653 | 0.442936 | 0.055* |      |      |
| C3     | 0.47873 (16) | 1.1423 (3) | 0.5811 (3) | 0.0408 (7) |      |      |
| H3     | 0.527698 | 1.129421 | 0.617432 | 0.049* |      |      |
| C11    | 0.26455 (17) | 0.6344 (3) | 0.7292 (3) | 0.0436 (8) |      |      |
| H11    | 0.236429 | 0.590723 | 0.781473 | 0.052* |      |      |
| C10    | 0.30743 (18) | 0.5508 (3) | 0.6661 (3) | 0.0488 (8) |      |      |
| H10    | 0.308209 | 0.452307 | 0.676839 | 0.059* |      |      |
| C16    | 0.06160 (19) | 0.3234 (3) | 0.4609 (4) | 0.0515 (8) |      |      |
| H16A   | 0.020800 | 0.385304 | 0.426179 | 0.062* |      |      |
| H16B   | 0.088673 | 0.314490 | 0.390895 | 0.062* |      |      |
| C15    | 0.1082 (2) | 0.3869 (4) | 0.5871 (4) | 0.0593 (10) |      |      |
| H15A   | 0.152356 | 0.332453 | 0.614785 | 0.071* |      |      |
| H15B   | 0.120699 | 0.483999 | 0.568925 | 0.071* |      |      |

Atomic displacement parameters (Å²)

| Atom   | U11  | U22  | U33  | U12  | U13  | U23  |
|--------|------|------|------|------|------|------|
| Cl1    | 0.0413 (4) | 0.0418 (4) | 0.0442 (4) | 0.0030 (3) | 0.0002 (3) | 0.0044 (3) |
| Cl2    | 0.0424 (4) | 0.0481 (5) | 0.0552 (5) | 0.0075 (3) | 0.0025 (4) | 0.0094 (4) |
| Atom | U1 (Å²) | U2 (Å²) | U3 (Å²) | U4 (Å²) | U5 (Å²) | U6 (Å²) |
|------|---------|---------|---------|---------|---------|---------|
| O1   | 0.0363  | 0.0534  | 0.0497  | 0.0053  | 0.0057  | 0.0195  |
| O1W  | 0.0600  | 0.0558  | 0.0496  | −0.0060 | 0.0087  | 0.0047  |
| N1   | 0.0349  | 0.0309  | 0.0525  | 0.0053  | 0.0196  | 0.0055  |
| O2   | 0.0325  | 0.0799  | 0.0625  | −0.0032 | 0.0054  | 0.0235  |
| N2   | 0.0409  | 0.0497  | 0.0404  | 0.0072  | 0.0037  | −0.0030 |
| O3   | 0.106   | 0.0641  | 0.0689  | −0.0190 | 0.0403  | −0.0214 |
| C2   | 0.0376  | 0.0276  | 0.0331  | 0.0030  | 0.0087  | −0.0018 |
| C14  | 0.0317  | 0.0414  | 0.0364  | 0.0012  | 0.0043  | −0.0003 |
| C1   | 0.0351  | 0.0267  | 0.0347  | 0.0014  | 0.0080  | −0.0029 |
| C12  | 0.0283  | 0.0355  | 0.0351  | 0.0000  | −0.0003 | 0.0050  |
| C7   | 0.0300  | 0.0310  | 0.0377  | 0.0010  | 0.0002  | 0.0027  |
| C8   | 0.0328  | 0.0415  | 0.0389  | 0.0001  | 0.0056  | −0.0017 |
| C6   | 0.0373  | 0.0322  | 0.0374  | 0.0013  | 0.0071  | −0.0013 |
| C13  | 0.0352  | 0.0428  | 0.0351  | −0.0017 | 0.0044  | 0.0068  |
| C9   | 0.0415  | 0.0377  | 0.0498  | 0.0045  | 0.0004  | −0.0099 |
| C5   | 0.057   | 0.0333  | 0.0410  | −0.0019 | 0.0157  | 0.0027  |
| C4   | 0.0494  | 0.0401  | 0.053   | −0.0111 | 0.0226  | −0.0036 |
| C3   | 0.0346  | 0.0380  | 0.0505  | −0.0056 | 0.0115  | −0.0067 |
| C11  | 0.0441  | 0.0387  | 0.0447  | −0.0035 | 0.0039  | 0.0110  |
| C10  | 0.052   | 0.0313  | 0.058   | −0.0001 | 0.0025  | 0.0041  |
| C16  | 0.059   | 0.0442  | 0.049   | 0.0028  | 0.0073  | 0.0046  |
| C15  | 0.055   | 0.064   | 0.058   | −0.0107 | 0.0104  | −0.0099 |

Geometric parameters (Å, °)

| Bond       | Distance (Å) |
|------------|--------------|
| Cl1—C2     | 1.734 (3)    |
| C12—C6     | 1.741 (3)    |
| O1—C14     | 1.259 (3)    |
| O1W—H1WA   | 0.8501       |
| O1W—H1WB   | 0.8504       |
| N1—C1      | 1.396 (3)    |
| N1—C7      | 1.410 (3)    |
| N1—H1      | 0.8600       |
| O2—C14     | 1.248 (3)    |
| N2—C16     | 1.486 (4)    |
| N2—H2A     | 0.8900       |
| N2—H2B     | 0.8900       |
| N2—H2C     | 0.8900       |
| O3—C15     | 1.412 (4)    |
| O3—H3A     | 0.8200       |
| C2—C3      | 1.389 (4)    |
| C2—C1      | 1.400 (4)    |
| C14—C13    | 1.523 (4)    |
| C1—C6      | 1.394 (4)    |
| C12—C11    | 1.395 (4)    |
| C12—C7     | 1.403 (4)    |
| C12—C13    | 1.508 (4)    

| Bond                  | Angle       | Torsion                        | Bond                  | Angle       | Torsion                        |
|----------------------|-------------|--------------------------------|----------------------|-------------|--------------------------------|
| H1WA—O1W—H1WB        | 104.5       | C14—C13—H13A                  | 109.0                |             |                                |
| C1—N1—C7             | 124.4 (2)   | C12—C13—H13B                  | 109.0                |             |                                |
| C1—N1—H1             | 117.8       | C14—C13—H13B                  | 109.0                |             |                                |
| C7—N1—H1             | 117.8       | H13A—C13—H13B                 | 107.8                |             |                                |
| C16—N2—H2A           | 109.5       | C10—C9—C8                     | 120.3 (3)            |             |                                |
| C16—N2—H2B           | 109.5       | C10—C9—H9                     | 119.8                |             |                                |
| H2A—N2—H2B           | 109.5       | C8—C9—H9                      | 119.8                |             |                                |
| C16—N2—H2C           | 109.5       | C4—C5—C6                      | 119.8 (3)            |             |                                |
| H2A—N2—H2C           | 109.5       | C4—C5—H5                      | 120.1                |             |                                |
| C15—O3—H3A           | 109.5       | C5—C4—C3                      | 120.0 (3)            |             |                                |
| C3—C2—C1             | 122.1 (3)   | C5—C4—H4                      | 120.0                |             |                                |
| C3—C2—Cl1            | 117.0 (2)   | C3—C4—H4                      | 120.0                |             |                                |
| C1—C2—Cl1            | 120.9 (2)   | C4—C3—C2                      | 119.5 (3)            |             |                                |
| O2—C14—O1            | 123.9 (3)   | C4—C3—H3                      | 120.2                |             |                                |
| O2—C14—C13           | 118.9 (3)   | C2—C3—H3                      | 120.2                |             |                                |
| O1—C14—C13           | 117.2 (3)   | C10—C11—C12                   | 121.4 (3)            |             |                                |
| C6—C1—N1             | 121.1 (3)   | C10—C11—H11                   | 119.3                |             |                                |
| C6—C1—C2             | 115.9 (3)   | C12—C11—H11                   | 119.3                |             |                                |
| N1—C1—C2             | 122.8 (3)   | C9—C10—C11                    | 119.6 (3)            |             |                                |
| C11—C12—C7           | 118.5 (3)   | C9—C10—H10                    | 120.2                |             |                                |
| C11—C12—C13          | 120.4 (3)   | C11—C10—H10                   | 120.2                |             |                                |
| C7—C12—C13           | 121.1 (3)   | N2—C16—C15                    | 110.0 (3)            |             |                                |
| C8—C7—C12            | 119.7 (3)   | N2—C16—H16A                   | 109.7                |             |                                |
| C8—C7—N1             | 121.6 (3)   | C15—C16—H16A                  | 109.7                |             |                                |
| C12—C7—N1            | 118.7 (3)   | N2—C16—H16B                   | 109.7                |             |                                |
| C9—C8—C7             | 120.4 (3)   | C15—C16—H16B                  | 109.7                |             |                                |
| C9—C8—H8             | 119.8       | H16A—C16—H16B                 | 108.2                |             |                                |
| C7—C8—H8             | 119.8       | O3—C15—C16                    | 109.2 (3)            |             |                                |
| C5—C6—C1             | 122.5 (3)   | O3—C15—H15A                   | 109.8                |             |                                |
| C5—C6—Cl2            | 118.4 (2)   | O3—C15—H15B                   | 109.8                |             |                                |
| C1—C6—Cl2            | 119.1 (2)   | C16—C15—H15A                  | 109.8                |             |                                |
| C12—C13—C14          | 112.7 (2)   | C16—C15—H15B                  | 109.8                |             |                                |
| C12—C13—H13A         | 109.0       | H15A—C15—H15B                 | 108.3                |             |                                |
| C7—N1—C1—C6          | 131.3 (3)   | C2—C1—C6—C12                  | −176.9 (2)           |             |                                |
| C7—N1—C1—C2          | −52.7 (4)   | C11—C12—C13—C14               | −100.2 (3)           |             |                                |
| C3—C2—C1—C6          | −4.4 (4)    | C7—C12—C13—C14                | 80.1 (3)             |             |                                |
| C11—C2—C1—C6         | 174.1 (2)   | O2—C14—C13—C12                | 117.9 (3)            |             |                                |
| C3—C2—C1—N1          | 179.4 (3)   | O1—C14—C13—C12                | −61.2 (4)            |             |                                |
| C11—C2—C1—N1         | −2.1 (4)    | C7—C8—C9—C10                  | 1.8 (4)              |             |                                |
| C11—C12—C7—C8        | 0.9 (4)     | C1—C6—C5—C4                   | 0.3 (5)              |             |                                |
| C13—C12—C7—C8        | −179.4 (3)  | C12—C6—C5—C4                  | −179.8 (2)           |             |                                |
| C11—C12—C7—N1        | −179.8 (3)  | C6—C5—C4—C3                   | −2.4 (5)             |             |                                |
| C13—C12—C7—N1        | −0.1 (4)    | C5—C4—C3—C2                   | 0.9 (5)              |             |                                |
| C1—N1—C7—C8          | −16.6 (4)   | C1—C2—C3—C4                   | 2.6 (4)              |             |                                |
| C1—N1—C7—C12         | 164.1 (3)   | C11—C2—C3—C4                  | −176.0 (2)           |             |                                |
| C12—C7—C8—C9         | −2.0 (4)    | C7—C12—C11—C10                | 0.3 (4)              |             |                                |
N1—C7—C8—C9 178.7 (3) C13—C12—C11—C10 −179.3 (3)
N1—C1—C6—C5 179.3 (3) C8—C9—C10—C11 −0.5 (5)
C2—C1—C6—C5 3.0 (4) C12—C11—C10—C9 −0.5 (5)
N1—C1—C6—Cl2 −0.6 (4) N2—C16—C15—O3 −53.2 (4)

Hydrogen-bond geometry (Å, º)

| D—H···A  | D—H | H···A | D···A | D—H···A |
|----------|------|-------|-------|----------|
| N1—H1···O1 | 0.86 | 2.27 | 2.884 (3) | 129 |
| N2—H2A···O2i | 0.89 | 1.96 | 2.811 (4) | 160 |
| N2—H2B···O1Wii | 0.89 | 2.15 | 2.947 (4) | 148 |
| N2—H2C···O1iii | 0.89 | 1.92 | 2.802 (3) | 169 |
| O3—H3A···O1W | 0.82 | 1.96 | 2.770 (3) | 168 |
| O1W···H1W···O2 | 0.85 | 1.87 | 2.690 (3) | 161 |
| O1W···H1WA···O1iv | 0.85 | 2.00 | 2.809 (3) | 158 |

Symmetry codes: (i) −x, −y+1, −z+1; (ii) −x, y−1/2, −z+3/2; (iii) x, y−1, z; (iv) x, −y+3/2, z+1/2.