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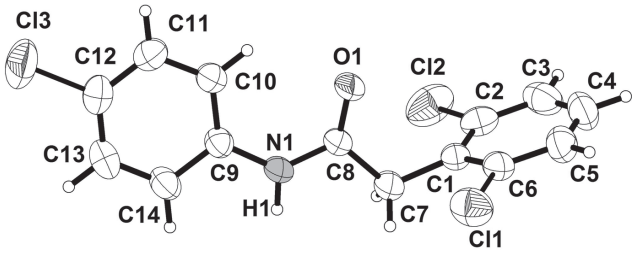
# Crystal structure of *N*-(4-chlorophenyl)-2-(2,6-dichlorophenyl)acetamide, $C_{14}H_{10}Cl_3NO$

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## Abstract

$C_{14}H_{10}Cl_3NO$, monoclinic, $P2_1/n$ (no. 14), $a = 13.7865(2)$ Å, $b = 13.0725(2)$ Å, $c = 15.9606(2)$ Å, $\beta = 100.7371(14)°$, $V = 2826.11(7)$ Å$^3$, $Z = 8$, $R_{gt}(F) = 0.0662$, $wR_{ref}(F^2) = 0.1980$, $T = 293(2)$ K.

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One of two crystallographically independent molecules of title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

## Source of material

Dichlorophenylacetic acid (48.78 mmol) and EDCI (1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride, 58.54 mmol) was stirred in dichloromethane (150 mL) in the presence of DMAP (4-dimethylaminopyridine, 58.54 mmol). Then *p*-chloroaniline (48.78 mmol) was added to the reaction. The mixture was stirred overnight at room temperature under nitrogen and monitored by TLC. The reaction mixture was diluted with dichloromethane, washed with 5% hydrochloric acid, water, and brine successively; then dried over anhydrous sodium sulfate and evaporated under reduced

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**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Needle, clear light colorless |
| Size: | 0.25 × 0.08 × 0.07 mm |
| Wavelength: | Cu $K\alpha$ radiation (1.54184 Å) |
| $\mu$: | 5.79 mm$^{-1}$ |
| Diffractometer, scan mode: | Xcalibur, Atlas, Gemini ultra, $\omega$-scans |
| $\theta_{max}$, completeness: | 66.8°, >97% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 21143, 5011, 0.034 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3840 |
| $N(param)_{refined}$: | 343 |
| Programs: | CrysAlis$^{PRO}$ [1], SHELX [2], OLEX2 [3] |

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.55802(8) | 0.01716(11) | 0.40796(8) | 0.0894(4) |
| Cl2 | 0.82090(12) | 0.19198(12) | 0.23948(9) | 0.1062(5) |
| Cl3 | 0.50552(11) | 0.75709(9) | 0.46331(10) | 0.0995(4) |
| O1 | 0.68997(18) | 0.27541(19) | 0.42003(15) | 0.0627(7) |
| N1 | 0.55920(19) | 0.3437(2) | 0.32923(16) | 0.0478(6) |
| H1 | 0.5170 | 0.3297 | 0.2839 | 0.057* |
| C1 | 0.6919(2) | 0.0972(2) | 0.3231(2) | 0.0492(8) |
| C2 | 0.7841(3) | 0.0936(3) | 0.2995(2) | 0.0608(9) |
| C3 | 0.8480(3) | 0.0114(4) | 0.3213(3) | 0.0724(12) |
| H3 | 0.9090 | 0.0104 | 0.3045 | 0.087* |
| C4 | 0.8208(3) | −0.0670(4) | 0.3672(3) | 0.0744(12) |
| H4 | 0.8632 | −0.1224 | 0.3809 | 0.089* |
| C5 | 0.7320(3) | −0.0664(3) | 0.3939(2) | 0.0659(10) |
| H5 | 0.7140 | −0.1201 | 0.4259 | 0.079* |
| C6 | 0.6698(2) | 0.0160(3) | 0.3720(2) | 0.0528(8) |
| C7 | 0.6195(3) | 0.1809(3) | 0.2943(2) | 0.0632(10) |
| H7A | 0.5533 | 0.1530 | 0.2870 | 0.076* |
| H7B | 0.6294 | 0.2053 | 0.2391 | 0.076* |
| C8 | 0.6272(2) | 0.2705(2) | 0.3548(2) | 0.0465(7) |
| C9 | 0.5474(2) | 0.4397(2) | 0.36607(19) | 0.0451(7) |
| C10 | 0.6122(2) | 0.4784(3) | 0.4359(2) | 0.0508(8) |
| H10 | 0.6649 | 0.4388 | 0.4629 | 0.061* |
| C11 | 0.5983(3) | 0.5757(3) | 0.4651(2) | 0.0574(9) |
| H11 | 0.6422 | 0.6018 | 0.5115 | 0.069* |
| C12 | 0.5206(3) | 0.6340(3) | 0.4264(3) | 0.0593(9) |
| C13 | 0.4544(3) | 0.5962(3) | 0.3580(3) | 0.0634(10) |
| H13 | 0.4012 | 0.6359 | 0.3321 | 0.076* |
| C14 | 0.4675(3) | 0.4991(3) | 0.3282(2) | 0.0557(8) |
| H14 | 0.4225 | 0.4731 | 0.2825 | 0.067* |
| Cl4 | 0.37025(12) | 0.81605(13) | 0.98439(9) | 0.1080(5) |
| Cl5 | 0.68995(11) | 0.95153(17) | 0.86637(11) | 0.1251(6) |

**Table 2** (continued)

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl6 | 0.74619(15) | 0.24376(11) | 0.81217(12) | 0.1220(6) |
| O2 | 0.53723(19) | 0.6919(2) | 0.85168(16) | 0.0659(7) |
| N2 | 0.6758(2) | 0.6529(2) | 0.94636(19) | 0.0615(8) |
| H2 | 0.7178 | 0.6747 | 0.9894 | 0.074* |
| C15 | 0.5284(3) | 0.8907(3) | 0.9219(2) | 0.0601(10) |
| C16 | 0.4276(3) | 0.9010(3) | 0.9274(3) | 0.0712(11) |
| C17 | 0.3710(4) | 0.9794(4) | 0.8868(3) | 0.0848(15) |
| H17 | 0.3048 | 0.9852 | 0.8908 | 0.102* |
| C18 | 0.4130(5) | 1.0480(4) | 0.8408(3) | 0.0917(17) |
| H18 | 0.3747 | 1.1014 | 0.8140 | 0.110* |
| C19 | 0.5094(5) | 1.0415(4) | 0.8323(3) | 0.0955(17) |
| H19 | 0.5366 | 1.0887 | 0.7998 | 0.115* |
| C20 | 0.5659(3) | 0.9618(3) | 0.8740(3) | 0.0721(11) |
| C21 | 0.5896(3) | 0.8069(3) | 0.9689(3) | 0.0740(12) |
| H21A | 0.5610 | 0.7871 | 1.0175 | 0.089* |
| H21B | 0.6554 | 0.8332 | 0.9905 | 0.089* |
| C22 | 0.5981(3) | 0.7134(3) | 0.9154(2) | 0.0563(9) |
| C23 | 0.6927(3) | 0.5557(3) | 0.9123(2) | 0.0551(8) |
| C24 | 0.7809(3) | 0.5335(3) | 0.8890(3) | 0.0718(11) |
| H24 | 0.8299 | 0.5833 | 0.8938 | 0.086* |
| C25 | 0.7978(3) | 0.4374(4) | 0.8584(3) | 0.0832(13) |
| H25 | 0.8581 | 0.4222 | 0.8432 | 0.100* |
| C26 | 0.7254(4) | 0.3648(3) | 0.8507(3) | 0.0734(12) |
| C27 | 0.6362(3) | 0.3865(3) | 0.8727(3) | 0.0704(11) |
| H27 | 0.5866 | 0.3374 | 0.8659 | 0.084* |
| C28 | 0.6208(3) | 0.4808(3) | 0.9045(2) | 0.0623(9) |
| H28 | 0.5611 | 0.4948 | 0.9213 | 0.075* |

pressure. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from ethyl acetate at room temperature.

## Experimental details

All H atoms were positioned geometrically and constrained to ride on the parent atoms, N-H = 0.86 Å, C-H = 0.93∼0.97 Å with $U_{iso}(H) = 1.2U_{eq}(C, N)$.

## Discussion

The title compound is an important impurity of diclofenac sodium. It has been recorded by European Pharmacopoeia 9.0. Diclofenac sodium to be the most widely used non-steroidal anti-inflammatory drugs [4, 5]. It exhibits anti-inflammatory, analgesic and antipyretic activities mainly through the inhibition of prostaglandin synthesis [6].

The crystal structure of the title compound contains two crystallographically independent molecules in the asymmetric unit. The main difference is the dihedral angles between the planes of the aryl moieties. The value is 80.7° and 53.5° for the two molecules, respectively. In the crystal structure, the components are linked by intermolecular N—H···O hydrogen bonds to form chains propagating along [101]. The crystal structure is further stabilized by π-π interactions, with centroid–centroid distances Cg1—$Cg1^{i}$ 3.718 Å and Cg2—$Cg2^{ii}$ 3.828 Å (Cg1: C9/C14, symmetry code (i): $1-x, 1-y, 1-z$; Cg2: C15/C20, symmetry code (ii): $1-x, -y, -z$).

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