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# A nimesulide intermediate

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## Abstract

The X-ray structure of the title compound, $C_{13}H_{13}NO_3S$, has been determined. The two phenyl rings make a dihedral angle of 73.5 (1)°, indicating the non-planarity of the molecule. The orientation of the methanesulfonanilide moiety to the phenyl ring is given by torsion angle C13—S—N—C12 − 69.2 (2)°. The molecules are linked into pairs by N—H···O hydrogen bonding over an inversion centre.


## Comment

The structure of the title compound was examined as part of an extensive study of Non-steroidal anti-inflammatory drugs (Ravikumar, 1993, 1994). We report here the crystal structure of 2-phenoxymethanesulfonanilide, (I), the precursor used in the synthesis of Nimesulide, (II), an anti-inflammatory drug (Moore & Harrington, 1974; Swingle & Moore, 1984; Ward & Brodgen, 1988). A view of the molecule with atom numbering in shown in Fig. 1. The S═O distances [1.424 (2)–1.438 (2) Å] are within the range observed for other sulfanalide drugs (Chaterjee *et al.*, 1981). The structure of Nimesulide has been reported (Dupont *et al.*, 1995). Bond distances and angles are similar to those observed in Nimesulide except C8—C9—C10 angle, 120.5 (2)°. The widening of the corresponding angle 123.1 (3)° observed in Nimesulide may be attributed to the effect of the nitro group substituted at C9. The dihedral angle between the two phenyl rings is 73.5 (1)° [74.7 (1)° in Nimesulide (II)] indicating that the molecule is twisted. In the crytal, the molecules exists as N—H···O hydrogen bond dimers around inversion centres. The amino N atom and atom O2 of the sulfoxide participates in the hydrogen bond formation [N···O2(−*x*, −*y* + 1, −*z*) 2.949 Å, H0A···O2 2.153Å and N—H0A···O2 147.1°].

## Experimental

The title compound was provided by NATCO Laboratory, Hyderabad, India, and recrystallized from benzene by slow evaporation.

## Refinement

H atoms were located from difference Fourier map; they were positioned geometrically and included as riding atoms with fixed isotropic displacement parameter in the structure-factor calculations.

## Computing details

Data collection: P3 Diffractometer Program (Siemens, 1991); cell refinement: *SHELXTL-Plus* (Sheldrick, 1991); data reduction: *SHELXTL-Plus*; program(s) used to solve structure: *SHELXTL-Plus*; program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXL93*.

# CIF access

## 2-phenoxymethanesulfonanilide

*Crystal data*

| | |
|---|---|
| $C_{13}H_{13}NO_3S$ | $\gamma = 102.78\ (1)^{\circ}$ |
| $M_r = 263.30$ | $V = 627.0\ (1)\ Å^3$ |
| Triclinic, $P\overline{1}$ | $Z = 2$ |
| $a = 8.146\ (1)$ Å | Mo $K\alpha$ |
| $b = 8.927\ (1)$ Å | $\mu = 0.26\ mm^{-1}$ |
| $c = 9.290\ (1)$ Å | $T = 293\ (2)$ K |
| $\alpha = 101.14\ (1)^{\circ}$ | $0.16 \times 0.16 \times 0.16$ mm |
| $\beta = 100.95\ (1)^{\circ}$ | |

*Data collection*

| | |
|---|---|
| Siemens R3m/V diffractometer | $R_{int} = 0.018$ |
| Absorption correction: none | 2 standard reflections |
| 2669 measured reflections | every 98 reflections |
| 2481 independent reflections | intensity decay: ≤1% |
| 2077 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | 164 parameters |
| $wR(F^2) = 0.116$ | H-atom parameters constrained |
| $S = 1.04$ | $\Delta\rho_{max} = 0.20\ e\ Å^{-3}$ |
| 2481 reflections | $\Delta\rho_{min} = -0.16\ e\ Å^{-3}$ |

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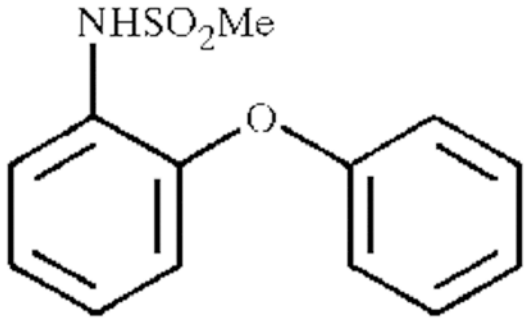
**Scheme 1**



# supplementary materials

## 2-phenoxymethanesulfonanilide

*Crystal data*

| | |
|---|---|
| $C_{13}H_{13}NO_3S$ | $Z = 2$ |
| $M_r = 263.30$ | $F_{000} = 276$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.395$ Mg m$^{-3}$ |
| $a = 8.146$ (1) Å | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $b = 8.927$ (1) Å | Cell parameters from 25 reflections |
| $c = 9.290$ (1) Å | $\theta = 6$–$15^o$ |
| $\alpha = 101.14$ (1)º | $\mu = 0.26$ mm$^{-1}$ |
| $\beta = 100.95$ (1)º | $T = 293$ (2) K |
| $\gamma = 102.78$ (1)º | Transparent cube, colorless |
| $V = 627.0$ (1) Å$^3$ | 0.16 × 0.16 × 0.16 mm |

*Data collection*

| | |
|---|---|
| Siemens R3m/V diffractometer | $R_{int} = 0.018$ |
| Radiation source: fine-focus sealed tube | $\theta_{max} = 26.1^o$ |
| Monochromator: graphite | $\theta_{min} = 2.3^o$ |
| $T = 293$(2) K | $h = 0 \rightarrow 10$ |
| $\omega/2\theta$ scans | $k = -11 \rightarrow 10$ |
| Absorption correction: none | $l = -11 \rightarrow 11$ |
| 2669 measured reflections | 2 standard reflections |
| 2481 independent reflections | every 98 reflections |
| 2077 reflections with $I > 2\sigma(I)$ | intensity decay: ≤1% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | H-atom parameters constrained |
| $wR(F^2) = 0.116$ | Calculated $w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.2251P]$ where $P = (F_o^2 + 2F_c^2)/3$ ? |
| $S = 1.04$ | $\Delta\rho_{max} = 0.20$ e Å$^{-3}$ |
| 2481 reflections | $\Delta\rho_{min} = -0.16$ e Å$^{-3}$ |
| 164 parameters | Extinction correction: SHELXL93, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.160 (10) |

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| S | 0.05903 (7) | 0.58633 (6) | 0.26066 (6) | 0.0377 (2) |
| N | 0.1093 (2) | 0.4278 (2) | 0.1847 (2) | 0.0371 (4) |
| H0A | 0.1102 | 0.4075 | 0.0862 | 0.080* |
| O1 | −0.1320 (2) | 0.1599 (2) | 0.1693 (2) | 0.0486 (4) |
| O2 | 0.0399 (2) | 0.6717 (2) | 0.1448 (2) | 0.0503 (4) |
| O3 | 0.1836 (2) | 0.6620 (2) | 0.4003 (2) | 0.0531 (4) |
| C1 | −0.3990 (3) | 0.0271 (3) | 0.2121 (3) | 0.0479 (5) |
| H1A | −0.4031 | 0.1257 | 0.2728 | 0.080* |
| C2 | −0.5316 (3) | −0.1088 (3) | 0.1868 (3) | 0.0560 (6) |
| H2B | −0.6275 | −0.1039 | 0.2321 | 0.080* |
| C3 | −0.5256 (3) | −0.2503 (3) | 0.1005 (3) | 0.0534 (6) |
| H3B | −0.6190 | −0.3438 | 0.0832 | 0.080* |
| C4 | −0.3878 (3) | −0.2559 (3) | 0.0376 (3) | 0.0580 (6) |
| H4A | −0.3828 | −0.3539 | −0.0234 | 0.080* |
| C5 | −0.2539 (3) | −0.1200 (3) | 0.0609 (3) | 0.0542 (6) |
| H5A | −0.1568 | −0.1224 | 0.0168 | 0.080* |
| C6 | −0.2620 (3) | 0.0187 (2) | 0.1485 (2) | 0.0386 (5) |
| C7 | 0.0271 (3) | 0.1787 (2) | 0.2627 (2) | 0.0358 (4) |
| C8 | 0.0686 (3) | 0.0706 (3) | 0.3433 (3) | 0.0455 (5) |
| H8A | −0.0158 | −0.0268 | 0.3344 | 0.080* |
| C9 | 0.2340 (3) | 0.1031 (3) | 0.4339 (3) | 0.0490 (6) |
| H9A | 0.2625 | 0.0294 | 0.4908 | 0.080* |
| C10 | 0.3574 (3) | 0.2410 (3) | 0.4439 (3) | 0.0505 (6) |
| H10A | 0.4715 | 0.2634 | 0.5082 | 0.080* |
| C11 | 0.3165 (3) | 0.3482 (3) | 0.3621 (2) | 0.0439 (5) |
| H11A | 0.4036 | 0.4429 | 0.3681 | 0.080* |
| C12 | 0.1513 (3) | 0.3192 (2) | 0.2725 (2) | 0.0345 (4) |
| C13 | −0.1435 (3) | 0.5254 (3) | 0.3009 (3) | 0.0560 (6) |
| H13A | −0.1768 | 0.6163 | 0.3467 | 0.080* |
| H13B | −0.2285 | 0.4683 | 0.2089 | 0.080* |
| H13C | −0.1359 | 0.4577 | 0.3693 | 0.080* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S | 0.0433 (3) | 0.0326 (3) | 0.0355 (3) | 0.0083 (2) | 0.0071 (2) | 0.0088 (2) |
| N | 0.0437 (10) | 0.0346 (9) | 0.0334 (8) | 0.0083 (7) | 0.0110 (7) | 0.0103 (7) |
| O1 | 0.0388 (8) | 0.0389 (8) | 0.0626 (10) | 0.0023 (6) | −0.0017 (7) | 0.0228 (7) |
| O2 | 0.0670 (10) | 0.0396 (8) | 0.0474 (9) | 0.0152 (7) | 0.0118 (8) | 0.0187 (7) |
| O3 | 0.0588 (10) | 0.0418 (8) | 0.0453 (9) | 0.0078 (7) | −0.0021 (8) | 0.0009 (7) |
| C1 | 0.0479 (13) | 0.0438 (12) | 0.0505 (13) | 0.0135 (10) | 0.0123 (10) | 0.0063 (10) |
| C2 | 0.0447 (13) | 0.0577 (14) | 0.066 (2) | 0.0088 (11) | 0.0212 (12) | 0.0147 (12) |
| C3 | 0.0472 (13) | 0.0451 (12) | 0.0599 (14) | −0.0005 (10) | 0.0084 (11) | 0.0138 (11) |
| C4 | 0.065 (2) | 0.0378 (12) | 0.067 (2) | 0.0090 (11) | 0.0196 (13) | 0.0039 (11) |
| C5 | 0.0512 (13) | 0.0463 (12) | 0.069 (2) | 0.0123 (10) | 0.0268 (12) | 0.0112 (11) |
| C6 | 0.0361 (10) | 0.0346 (10) | 0.0431 (11) | 0.0056 (8) | 0.0035 (9) | 0.0151 (8) |
| C7 | 0.0366 (10) | 0.0368 (10) | 0.0347 (10) | 0.0097 (8) | 0.0084 (8) | 0.0105 (8) |
| C8 | 0.0490 (12) | 0.0411 (11) | 0.0490 (12) | 0.0118 (10) | 0.0111 (10) | 0.0184 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.0556 (14) | 0.0497 (13) | 0.0460 (12) | 0.0239 (11) | 0.0059 (10) | 0.0169 (10) |
| C10 | 0.0443 (12) | 0.0568 (14) | 0.0469 (12) | 0.0202 (10) | 0.0006 (10) | 0.0077 (11) |
| C11 | 0.0386 (11) | 0.0432 (11) | 0.0447 (12) | 0.0086 (9) | 0.0074 (9) | 0.0038 (9) |
| C12 | 0.0390 (10) | 0.0341 (10) | 0.0313 (9) | 0.0113 (8) | 0.0100 (8) | 0.0069 (8) |
| C13 | 0.0521 (14) | 0.065 (2) | 0.0568 (14) | 0.0189 (12) | 0.0214 (12) | 0.0168 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S—O3 | 1.424 (2) | C3—C4 | 1.366 (4) |
| S—O2 | 1.438 (2) | C4—C5 | 1.389 (3) |
| S—N | 1.627 (2) | C5—C6 | 1.366 (3) |
| S—C13 | 1.753 (2) | C7—C8 | 1.390 (3) |
| N—C12 | 1.438 (2) | C7—C12 | 1.402 (3) |
| O1—C7 | 1.371 (2) | C8—C9 | 1.382 (3) |
| O1—C6 | 1.409 (2) | C9—C10 | 1.381 (4) |
| C1—C6 | 1.368 (3) | C10—C11 | 1.390 (3) |
| C1—C2 | 1.381 (3) | C11—C12 | 1.381 (3) |
| C2—C3 | 1.376 (3) | | |
| O3—S—O2 | 118.96 (10) | C5—C6—C1 | 121.7 (2) |
| O3—S—N | 108.05 (10) | C5—C6—O1 | 120.3 (2) |
| O2—S—N | 105.55 (9) | C1—C6—O1 | 117.9 (2) |
| O3—S—C13 | 107.87 (12) | O1—C7—C8 | 124.8 (2) |
| O2—S—C13 | 108.39 (11) | O1—C7—C12 | 114.6 (2) |
| N—S—C13 | 107.51 (11) | C8—C7—C12 | 120.6 (2) |
| C12—N—S | 120.98 (13) | C9—C8—C7 | 119.3 (2) |
| C7—O1—C6 | 118.1 (2) | C10—C9—C8 | 120.5 (2) |
| C6—C1—C2 | 118.7 (2) | C9—C10—C11 | 120.2 (2) |
| C3—C2—C1 | 120.8 (2) | C12—C11—C10 | 120.3 (2) |
| C4—C3—C2 | 119.5 (2) | C11—C12—C7 | 119.1 (2) |
| C3—C4—C5 | 120.4 (2) | C11—C12—N | 120.6 (2) |
| C6—C5—C4 | 118.9 (2) | C7—C12—N | 120.3 (2) |
| O3—S—N—C12 | 47.0 (2) | C6—O1—C7—C12 | 177.4 (2) |
| O2—S—N—C12 | 175.27 (15) | O1—C7—C8—C9 | 180.0 (2) |
| C13—S—N—C12 | −69.2 (2) | C12—C7—C8—C9 | 0.3 (3) |
| C6—C1—C2—C3 | 0.6 (4) | C7—C8—C9—C10 | −0.4 (4) |
| C1—C2—C3—C4 | −0.9 (4) | C8—C9—C10—C11 | −0.4 (4) |
| C2—C3—C4—C5 | 0.3 (4) | C9—C10—C11—C12 | 1.3 (3) |
| C3—C4—C5—C6 | 0.5 (4) | C10—C11—C12—C7 | −1.5 (3) |
| C4—C5—C6—C1 | −0.7 (4) | C10—C11—C12—N | −178.5 (2) |
| C4—C5—C6—O1 | −177.4 (2) | O1—C7—C12—C11 | −179.1 (2) |
| C2—C1—C6—C5 | 0.2 (3) | C8—C7—C12—C11 | 0.7 (3) |
| C2—C1—C6—O1 | 176.9 (2) | O1—C7—C12—N | −2.0 (3) |
| C7—O1—C6—C5 | −74.1 (3) | C8—C7—C12—N | 177.8 (2) |
| C7—O1—C6—C1 | 109.1 (2) | S—N—C12—C11 | −82.6 (2) |
| C6—O1—C7—C8 | −2.3 (3) | S—N—C12—C7 | 100.4 (2) |