

Sheldrick, G. M. (1990). *SHELXS86. Program for Crystal Structure Solution*. Univ. of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). **C51**, 926–927

N,N-Diethyl-4-(methylsulfonyl)-2-nitroaniline

DAVID D. MACNICOL AND PAUL R. MALLINSON

Chemistry Department, University of Glasgow, Glasgow G12 8QQ, Scotland

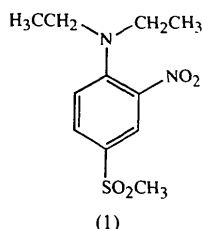
(Received 22 September 1994; accepted 9 November 1994)

Abstract

The title compound, C₁₁H₁₆N₂O₄S, shows significant rotation of the NEt₂ and NO₂ groups from the benzene plane, which reduces the steric compression of these *ortho* substituents. The methyl groups of the NEt₂ substituent adopt a *syn* arrangement, unlike the *anti* arrangement in *N,N*-diethyl-2,4-dinitroaniline.

Comment

The methyl groups in the NEt₂ unit of the title compound (1) adopt a *syn* arrangement. The NEt₂ and NO₂ N atoms are displaced by $-0.139(3)$ and $0.283(3)$ Å from the mean plane of the benzene ring whilst the S atom lies $0.008(1)$ Å from it. The orientations of the NEt₂ and NO₂ groups are described, respectively, by the torsion angles C5—C4—N2—C10 $22.6(4)$, C3—C4—N2—C8 $31.0(4)$, C4—C3—N1—O3 $38.1(4)$, C2—C3—N1—O4 $40.6(4)^\circ$. The NEt₂ N atom is slightly pyramidal, N2 being displaced from the plane of the directly attached C atoms by $0.070(3)$ Å. The nitro group is almost planar with the N atom $0.030(3)$ Å from the O3—O4—C3 plane. The differences between the two N—C(ethyl) and between the two C—C(ethyl) bond lengths are probably due to librational effects. One large anisotropic displacement parameter for C11 suggests either a large component of vibration amplitude or slight disorder.



The *syn* arrangement of the methyl groups contrasts with an *anti* arrangement in *N,N*-diethyl-2,4-dinitroaniline (MacNicol & Mallinson, unpublished results). The N2—C4 and N1—C3 distances in the title compound are $1.366(5)$ and $1.462(5)$ Å compared with $1.353(5)$ and $1.458(5)$ Å in the dinitro compound. The significantly closed angle C3—C4—C5 here, $114.0(4)^\circ$, may be compared with $115.2(4)^\circ$ in the dinitro compound.

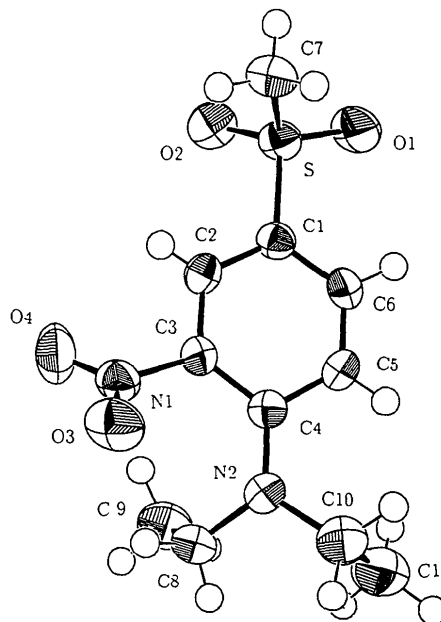


Fig. 1. A view of the molecular structure showing the atom labels. Displacement ellipsoids are shown at the 50% probability level; H atoms are shown as circles of arbitrary radii and are labelled according to the atoms to which they are attached.

Experimental

Crystals, m.p. 359 K, were prepared at Glasgow University by Dr C. W. N. Horning.

Crystal data

C₁₁H₁₆N₂O₄S
M_r = 272.32
 Monoclinic
*P*2₁/*n*
a = 5.035 (1) Å
b = 11.684 (1) Å
c = 21.792 (3) Å
 β = 90.54 (2) $^\circ$
V = 1282 (1) Å³
Z = 4
D_x = 1.41 Mg m⁻³

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 11–15 $^\circ$
 μ = 0.249 mm⁻¹
T = 293 K
 Needle
 0.8 × 0.1 × 0.1 mm
 Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer

*R*_{int} = 0.023
 θ _{max} = 27 $^\circ$

ω -2 θ scans	$h = 0 \rightarrow 6$
Absorption correction: none	$k = 0 \rightarrow 14$
2865 measured reflections	$l = -27 \rightarrow 27$
2797 independent reflections	2 standard reflections
1386 observed reflections [$I > 2\sigma(I)$]	frequency: 120 min intensity decay: none

Refinement

Refinement on F	$(\Delta/\sigma)_{\max} = 0.23$
$R = 0.044$	$\Delta\rho_{\max} = 0.6 \text{ e } \text{\AA}^{-3}$
$wR = 0.053$	$\Delta\rho_{\min} = -0.4 \text{ e } \text{\AA}^{-3}$
$S = 2.3$	Extinction correction: none
1386 reflections	Atomic scattering factors
223 parameters	from <i>International Tables</i>
All H-atom parameters	for <i>X-ray Crystallography</i>
refined except H11B	(1974, Vol. IV, Tables
which was not refined	2.2B and 2.3.1)
$w = 1/\sigma^2(F)$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j$$

	x	y	z	U_{eq}
C1	0.6510 (6)	0.2527 (3)	0.5213 (2)	0.041
C2	0.7487 (7)	0.3245 (3)	0.5662 (2)	0.043
C3	0.9105 (6)	0.2825 (3)	0.6118 (2)	0.040
C4	0.9737 (7)	0.1654 (3)	0.6179 (2)	0.047
C5	0.8763 (9)	0.0965 (4)	0.5695 (2)	0.057
C6	0.7225 (8)	0.1383 (3)	0.5228 (2)	0.051
C7	0.6773 (10)	0.3801 (5)	0.4144 (2)	0.059
C8	1.1001 (8)	0.1639 (4)	0.7289 (2)	0.058
C9	0.8259 (9)	0.1867 (6)	0.7510 (2)	0.075
C10	1.2456 (11)	0.0061 (5)	0.6587 (3)	0.079
C11	1.0771 (17)	-0.0869 (5)	0.6793 (3)	0.110
N1	1.0388 (6)	0.3703 (3)	0.6496 (1)	0.052
N2	1.1107 (7)	0.1187 (3)	0.6661 (2)	0.061
O1	0.3417 (5)	0.2130 (3)	0.4285 (1)	0.073
O2	0.2740 (5)	0.3922 (2)	0.4863 (1)	0.063
O3	1.2762 (5)	0.3600 (3)	0.6618 (1)	0.073
O4	0.9096 (6)	0.4537 (3)	0.6643 (1)	0.077
S	0.45238 (17)	0.30780 (9)	0.46148 (4)	0.049

Table 2. Selected geometric parameters (\AA , $^\circ$)

C1—C2	1.377 (5)	C7—S	1.753 (6)
C1—C6	1.384 (6)	C8—C9	1.491 (7)
C1—S	1.757 (4)	C8—N2	1.467 (6)
C2—C3	1.371 (5)	C10—C11	1.452 (10)
C3—C4	1.410 (5)	C10—N2	1.490 (7)
C3—N1	1.462 (5)	N1—O3	1.228 (5)
C4—C5	1.412 (6)	N1—O4	1.216 (5)
C4—N2	1.366 (5)	O1—S	1.430 (4)
C5—C6	1.364 (6)	O2—S	1.442 (3)
C4—C3—N1	122.0 (3)	O3—N1—O4	122.8 (4)
C3—C4—C5	114.0 (4)	C8—N2—C4	123.4 (4)
C3—C4—N2	124.8 (4)	C10—N2—C4	119.8 (4)
O3—N1—C3	118.6 (4)	C8—N2—C10	116.1 (4)
O4—N1—C3	118.5 (3)		
C6—C1—S—O1	13.2 (3)	C2—C1—S—O2	-40.1 (3)
C2—C1—S—C7	74.2 (4)		

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *GX*. Molecular graphics: *ORTEPII* (Johnson, 1971; Mallinson & Muir, 1985). Software used to prepare material for publication: *GX*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1134). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
 Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
 Johnson, C. K. (1971). *ORTEPII*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.
 Mallinson, P. R. & Muir, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

Acta Cryst. (1995). **C51**, 927–929

KR-25003, a Potent Analgesic Capsaicinoid

NO-SANG PARK, IL YEONG PARK AND JONG CHEOL LEE

*Korea Research Institute of Chemical Technology,
Taejon 305-606, Korea*

YANG BAE KIM

*College of Pharmacy, Seoul National University,
Seoul 151-742, Korea*

(Received 12 September 1994; accepted 28 November 1994)

Abstract

The crystal structure of *N*-[3-(3,4-dimethylphenyl)propyl](4-hydroxy-3-methoxyphenyl)acetamide, $\text{C}_{20}\text{H}_{25}\text{NO}_3$, was determined to a final R value of 0.047. The vanilloid, amide and dimethylphenyl groups of the compound are nearly perpendicular with respect to one another. The overall conformation in the crystalline state is somewhat different from that of other capsaicinoids. The molecules are stabilized by intermolecular hydrogen bonds.

Comment

Capsaicin, the pungent principle component of red pepper, is well known to have analgesic activity in animal species (Suzuki & Iwai, 1984). It has a different mechanism of action from the usual analgesics such as narcotic or non-steroidal anti-inflammatory drugs (NSAIDs). Although the undesirable side effects of natural capsaicin prevent its direct utilization in medicinal preparations for humans, the strong analgesic effect of the capsaicinoids, natural as well as synthetic, has been studied in detail (Dray, 1992).