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4-Methyl-*N*-8-quinolybenzenesulphonamide, C₁₆H₁₄N₂O₂S. The Active Component of LIX 34

BY G. GERMAIN AND J. P. DECLERCQ

Laboratoire de Chimie-Physique et de Cristallographie, Université de Louvain, 1 Place Louis Pasteur, B 1348, Louvain-la-Neuve, Belgique

AND J. M. CASTRESANA, M. P. ELIZALDE AND J. M. ARRIETA*

Departamento de Química, Facultad de Ciencias, Universidad del País Vasco, Apartado 644, Bilbao, Spain

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Abstract. $M_r = 298.35$, triclinic, space group $P\bar{1}$, $a = 6.675$ (2), $b = 15.340$ (3), $c = 16.065$ (4) Å, $\alpha = 116.07$ (2), $\beta = 96.81$ (2), $\gamma = 97.92$ (2)°, $V = 1433.5$ (6) Å³, $Z = 4$, $D_x = 1.38$ Mg m⁻³, $F(000) = 624$, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 0.23$ mm⁻¹. Final $R = 0.061$ for 1277 observed reflections [$I \geq 2.5\sigma(I)$]. The two molecules of the asymmetric unit are almost identical. The S atom has a distorted tetrahedral coordination.

Introduction. One of the most recent commercial chelating agents is LIX 34, a quinolylsulphonamide which has been used in industrial solvent-extraction processes to recover copper(II) (Kordosky, 1976).

4-Methyl-*N*-8-quinolybenzenesulphonamide, with a similar structure to the active component in LIX 34, has been synthesized (Cox & Castresana, 1982). In this paper its crystalline structure is described.

Experimental. Colourless prismatic crystals grown from acetone; data collected from a crystal of approximately 0.4 × 0.1 × 0.1 mm; cell parameters determined by least squares from the setting angles of 15 reflections; 3043 independent reflections measured with graphite-monochromated Mo $K\alpha$ radiation using ω scans up to $2\theta = 42^\circ$; one standard reflection measured every fifty reflections showed only random deviation from its mean intensity; Lorentz and polarization but not absorption corrections were applied; 1277 reflections [$I \geq 2.5\sigma(I)$] considered as observed and included in the refinement; direct methods with *MULTAN* 80 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); an *E* map showed clearly all the non-H atoms of the molecule; refinement with *SHELX* 76 (Sheldrick, 1976) with anisotropic thermal parameters for the non-H atoms; H positions determined by a difference Fourier synthesis introduced into the refinement with an overall isotropic temperature factor. The final R and R_w were 0.061 and 0.048 respectively [$w = 1/\sigma^2(F)$].

* Author to whom correspondence should be addressed.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (\AA^2)

	Molecule A				Molecule B			
	x	y	z	B_{eq}	x	y	z	B_{eq}
N(1)	6323 (14)	3136 (6)	966 (8)	4.80	2772 (16)	9845 (8)	4070 (8)	4.79
C(2)	6454 (18)	3324 (9)	262 (9)	5.26	3272 (18)	10746 (11)	4768 (10)	5.92
C(3)	5061 (24)	3768 (9)	-74 (8)	6.29	1836 (28)	11393 (10)	5021 (9)	7.19
C(4)	3450 (21)	4052 (8)	387 (10)	5.96	-67 (23)	11046 (11)	4501 (10)	5.76
C(5)	3336 (17)	3876 (7)	1148 (9)	3.75	-628 (20)	10091 (10)	3740 (10)	4.56
C(6)	1762 (19)	4114 (7)	1653 (10)	5.15	-2608 (21)	9684 (11)	3148 (10)	5.70
C(7)	1654 (18)	3912 (9)	2424 (10)	5.84	-3006 (18)	8725 (11)	2412 (10)	5.77
C(8)	3217 (21)	3492 (8)	2701 (9)	6.21	-1560 (19)	8175 (8)	2196 (8)	5.00
C(9)	4691 (19)	3238 (7)	2208 (8)	3.76	383 (18)	8545 (8)	2772 (8)	3.75
C(10)	4737 (18)	3422 (7)	1433 (8)	3.79	842 (20)	9522 (9)	3556 (8)	3.62
N(11)	6313 (14)	2866 (7)	2482 (7)	4.64	2039 (13)	8029 (6)	2624 (6)	4.60
S(12)	6161 (5)	2088 (2)	2920 (2)	4.97	1781 (5)	6837 (2)	2199 (2)	4.49
O(13)	8096 (11)	1816 (5)	2927 (5)	6.84	513 (11)	6376 (5)	1271 (5)	5.73
O(14)	5475 (11)	2555 (5)	3798 (5)	6.69	3828 (11)	6695 (5)	2328 (5)	6.24
C(15)	4222 (17)	1026 (7)	2114 (8)	3.81	388 (20)	6445 (7)	2895 (7)	3.33
C(16)	2284 (18)	957 (8)	2298 (8)	3.90	-1686 (17)	6085 (7)	2251 (7)	3.60
C(17)	797 (17)	150 (9)	1652 (9)	5.15	-2612 (18)	5808 (7)	3215 (9)	4.67
C(18)	1257 (24)	-557 (9)	821 (9)	5.47	-1519 (23)	5914 (8)	4067 (10)	4.82
C(19)	3245 (24)	-478 (10)	673 (9)	6.24	567 (20)	6312 (8)	4319 (8)	4.94
C(20)	4752 (18)	317 (10)	1311 (9)	5.06	1556 (17)	6589 (7)	3762 (9)	4.69
C(21)	-374 (17)	-1440 (7)	102 (8)	7.19	-2629 (17)	5609 (8)	4706 (8)	6.86

Table 1* gives the final atomic parameters with their B_{eq} values (Willis & Pryor, 1975) following the numbering shown in Fig. 1, instead of the IUPAC numbering given in the title. Fig. 1 shows a perspective view of the molecule drawn with *PLUTO* (Motherwell & Clegg, 1978).

Discussion. As can be seen from Tables 2 and 3, the bond distances and angles of the two molecules in the asymmetric unit are very similar. The isoquinoline groups are planar within experimental error in the two molecules, the largest atomic deviation from the mean plane for each molecule being 0.03 (4) [C(8A)] and

0.04 (4) \AA [C(8B)]. The phenyl groups are also planar, the largest deviations from their respective mean planes being 0.02 (3) [C(18A)] and 0.02 (3) \AA [C(15B)].

The S atoms show a distorted tetrahedral coordination with a mean angle of 109.4° (range: 105.1 – 119.4°) for molecule A, and 109.4° (range: 105.8 – 120.5°) for molecule B. The distances S(12)–N(11) and S(12)–C(15) are 1.63 (1) and 1.79 (1) \AA for molecule A and 1.62 (1) and 1.78 (2) \AA for molecule B. These values are longer than those reported by Houttemane, Boivin, Nowogrocki, Thomas & Bonte (1981) in *N*-(1-ethyl-2-pyrrolidinyl)methyl-2-methoxy-5-sulphamoylbenzamide. This can be explained by the bridging function of our sulphonamide group between the phenyl and isoquinoline groups.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38057 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

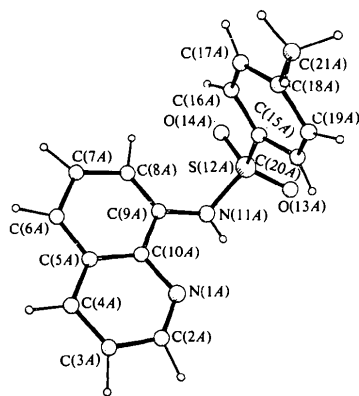


Fig. 1. Perspective view of molecule A with the atom numbering.

Table 2. Bond distances (\AA)

	Molecule A	Molecule B
C(2)–N(1)	1.29 (2)	1.30 (2)
C(10)–N(1)	1.39 (2)	1.36 (2)
C(3)–C(2)	1.42 (2)	1.45 (2)
C(4)–C(3)	1.40 (2)	1.34 (2)
C(5)–C(4)	1.37 (2)	1.40 (2)
C(6)–C(5)	1.41 (2)	1.43 (2)
C(10)–C(5)	1.40 (2)	1.39 (2)
C(7)–C(6)	1.41 (2)	1.39 (2)
C(8)–C(7)	1.44 (2)	1.36 (2)
C(9)–C(8)	1.34 (2)	1.40 (2)
C(10)–C(9)	1.40 (2)	1.43 (1)
N(11)–C(9)	1.42 (2)	1.44 (2)
S(12)–N(11)	1.63 (1)	1.62 (1)
O(13)–S(12)	1.43 (1)	1.44 (1)
O(14)–S(12)	1.44 (1)	1.44 (1)
C(15)–S(12)	1.79 (1)	1.78 (1)
C(16)–C(15)	1.38 (2)	1.38 (2)
C(20)–C(15)	1.40 (2)	1.43 (2)
C(17)–C(16)	1.38 (1)	1.34 (2)
C(18)–C(17)	1.41 (2)	1.41 (2)
C(19)–C(18)	1.39 (2)	1.39 (2)
C(21)–C(18)	1.52 (1)	1.53 (2)
C(20)–C(19)	1.37 (2)	1.35 (2)

Table 3. Bond angles ($^{\circ}$)

	Molecule A	Molecule B
C(10)—N(1)—C(2)	116.7 (1.2)	118.0 (1.1)
C(3)—C(2)—N(1)	124.4 (1.2)	122.4 (1.1)
C(4)—C(3)—C(2)	119.3 (1.4)	118.0 (1.1)
C(5)—C(4)—C(3)	116.4 (1.4)	120.6 (1.5)
C(6)—C(5)—C(4)	121.3 (1.3)	123.5 (1.4)
C(10)—C(5)—C(4)	121.4 (1.2)	117.4 (1.2)
C(10)—C(5)—C(6)	117.3 (1.4)	119.1 (1.1)
C(7)—C(6)—C(5)	120.4 (1.3)	118.8 (1.4)
C(8)—C(7)—C(6)	118.7 (1.2)	122.8 (1.1)
C(9)—C(8)—C(7)	121.1 (1.4)	119.7 (0.9)
C(10)—C(9)—C(8)	119.0 (1.3)	119.2 (1.2)
N(11)—C(9)—C(8)	122.9 (1.3)	125.0 (0.9)
N(11)—C(9)—C(10)	117.9 (1.2)	115.8 (1.0)
C(5)—C(10)—N(1)	121.8 (1.3)	123.6 (0.9)
C(9)—C(10)—N(1)	114.9 (1.2)	116.1 (1.2)
C(9)—C(10)—C(5)	123.3 (1.2)	120.3 (1.1)
S(12)—N(11)—C(9)	126.6 (0.9)	124.8 (0.7)
O(13)—S(12)—N(11)	105.1 (0.6)	107.9 (0.5)
O(14)—S(12)—N(11)	107.2 (0.5)	104.3 (0.4)
O(14)—S(12)—O(13)	119.4 (0.5)	120.5 (0.5)
C(15)—S(12)—N(11)	106.9 (0.6)	108.5 (0.5)
C(15)—S(12)—O(13)	109.6 (0.5)	105.8 (0.5)
C(15)—S(12)—O(14)	107.9 (0.5)	109.5 (0.6)
C(16)—C(15)—S(12)	118.9 (0.7)	123.0 (1.0)
C(20)—C(15)—S(12)	117.8 (0.9)	115.2 (0.9)
C(20)—C(15)—C(16)	123.3 (0.9)	121.6 (1.2)
C(17)—C(16)—C(15)	118.0 (1.0)	119.2 (1.1)
C(18)—C(17)—C(16)	120.3 (1.2)	121.3 (1.1)
C(19)—C(18)—C(17)	120.1 (1.0)	118.7 (1.5)
C(21)—C(18)—C(17)	120.6 (1.3)	120.1 (1.2)
C(21)—C(18)—C(19)	119.3 (1.1)	121.2 (1.3)
C(20)—C(19)—C(18)	120.5 (1.1)	121.5 (1.3)
C(19)—C(20)—C(15)	117.8 (1.2)	117.5 (1.1)

The chelating effect of the title molecule could be explained by the presence of the two N atoms. We shall try to confirm this point by determining the structure of a metal-LIX 34 chelate.

The molecular packing is shown in Fig. 2.

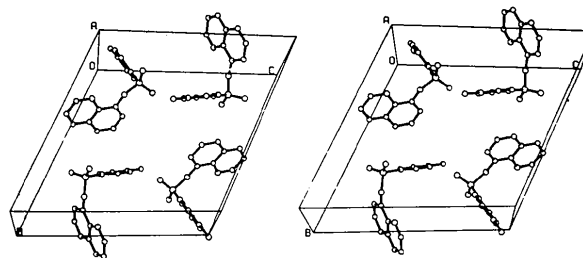


Fig. 2. Stereoscopic view of the unit cell.

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Nicotinic Acid, C₆H₅NO₂: Refinement

BY A. KUTOGLU AND C. SCHERINGER

Institut für Mineralogie der Universität Marburg, Lahnberge, D-3550 Marburg, Federal Republic of Germany

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Abstract. $M_r = 123.1$, monoclinic, $P2_1/c$, $a = 7.186$ (2), $b = 11.688$ (3), $c = 7.231$ (2) Å, $\beta = 113.55$ (6) $^{\circ}$, $Z = 4$. Wright & King's [*Acta Cryst.* (1953), 6, 305–317] determination of the crystal structure ($R = 0.22$) was confirmed. 6893 reflections were measured and $R(F) = 0.051$ was obtained for the 802 'observed' independent X-ray data.

Introduction. The crystal structure of nicotinic acid has already been determined by Wright & King (1953) with photographic methods ($R = 0.22$). Our reinvestigation was performed to derive more accurate parameters as a preliminary to the study of the electron density distribution in the molecule. This work is part of our project on electron density determination in derivatives