Orthorhombic
Pccn
$a = 19.856\,(3)$ Å
$b = 21.450\,(4)$ Å
$c = 7.228\,(5)$ Å
$V = 3078\,(2)$ Å^3
$Z = 8$
$D_x = 1.149$ Mg m^{-3}
D_m not measured

Cell parameters from 25
reflections
$\theta = 13.5-18.4°$
$\mu = 0.069$ mm^{-1}
$T = 294\,(2)$ K
Plate
$0.40 \times 0.35 \times 0.20$ mm
Colourless

Data collection

Rigaku AFC-5S diffractom-
eter
$\omega-2\theta$ scans
Absorption correction: none
3126 measured reflections
3126 independent reflections
1635 reflections with
$I > 2\sigma(I)$

$\theta_{max} = 25.00°$
$h = 0 \rightarrow 24$
$k = 0 \rightarrow 26$
$l = 0 \rightarrow 9$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.148$
$S = 1.022$
2713 reflections
199 parameters
H-atom treatment, see below
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.625P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.152$ e Å^{-3}
$\Delta\rho_{min} = -0.218$ e Å^{-3}
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O—C3	1.435 (3)	C4—C5	1.511 (4)
O—H	0.771 (16)	C4—C14	1.523 (3)
C1—C2	1.530 (3)	C4—H4	0.97 (3)
C2—C7	1.524 (4)	C5—C6	1.292 (4)
C2—C3	1.553 (3)	C5—H5	0.93 (3)
C2—H2	1.00 (3)	C6—H6A	1.03 (4)
C3—C8	1.544 (3)	C6—H6B	0.97 (3)
C3—C4	1.560 (3)		
C3—O—H	119 (2)	O—C3—C4	107.39 (18)
C7—C2—C1	109.6 (2)	C8—C3—C4	110.59 (18)
C7—C2—C3	113.9 (2)	C2—C3—C4	112.46 (19)
C1—C2—C3	112.6 (2)	C5—C4—C14	112.7 (2)
O—C3—C8	109.74 (18)	C5—C4—C3	110.2 (2)
O—C3—C2	105.18 (18)	C14—C4—C3	114.92 (19)
C8—C3—C2	111.25 (19)	C6—C5—C4	128.9 (3)
O—C3—C4—C5	−58.5 (2)	C8—C3—C4—C14	−170.14 (19)
C8—C3—C4—C5	61.2 (3)	C2—C3—C4—C14	−45.1 (3)
C2—C3—C4—C5	−173.7 (2)	C14—C4—C5—C6	12.1 (4)
O—C3—C4—C14	70.1 (2)	C3—C4—C5—C6	141.9 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O—H$\cdots$O^i	0.771 (16)	2.252 (17)	3.006 (4)	166 (3)

Symmetry code: (i) $1 - x, -y, -z$.

Non-H atoms were refined with anisotropic displacement factors. Methyl- and phenyl-H atoms were included at fixed ideal positions with isotropic displacement parameters (1.5 × that of the host atom). The rest of the H atoms were refined with fixed isotropic displacement parameters (1.5 × that of the host atom).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1995). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997). Software used to prepare material for publication: *SHELXL*97.

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4-Hydroxy-2-methyl-*N*-(5-methyl-1,3-thia-zol-2-yl)-2*H*-1,2-benzothiazine-3-carbox-amide 1,1-Dioxide

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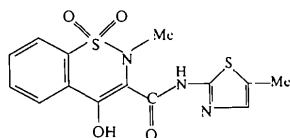
Abstract

The thiazine ring in the title compound, $C_{14}H_{13}N_3O_4S_2$, adopts a half-chair conformation. The methyl group at N3 is axial to the thiazine ring due to the tetrahedral

geometry of the N3 atom. The molecules are stabilized by intramolecular O—H···O and intermolecular N—H···O hydrogen bonds.

Comment

The title compound (meloxicam) is a new non-steroidal anti-inflammatory drug (NSAID) belonging to the oxicam group of compounds, of which peroxicam is a prototype. It has been approved in several countries for the treatment of rheumatoid arthritis and osteoarthritis (Noble & Balfour, 1996).

Meloxicam

The thiazine ring in the molecule is in a half-chair conformation. The angles S4—N3—C2 of 112.8(1)°, S4—N3—C19 of 115.1(2)° and C19—N3—C2 of 114.5(2)° indicate tetrahedral geometry of the N3 atom. The methyl group at N3 is axial to the thiazine ring. The O atoms O1 at C1 and O3 at S4 are equatorial, while O2 at S4 is axial. The methyl group at C15 is equatorial.

The thiazole ring and the group formed by atoms N3, C11, O4 and N12 are coplanar, and the dihedral angle formed by the mean planes of the thiazole and benzene rings is 11.81(8)°. The molecule is stabilized by an intramolecular O1—H1···O4 hydrogen bond [O1···O4 2.570(2), H1···O4 1.78(3) Å and O1—H1···O4 149(3)°] and the packing is stabilized by one intermolecular N12—H12···O3(1−x, 1−y, 1−z) hydrogen bond [N12···O3 3.029(2), H12···O3 2.25(3) Å and N12—H12···O3 165(3)°].
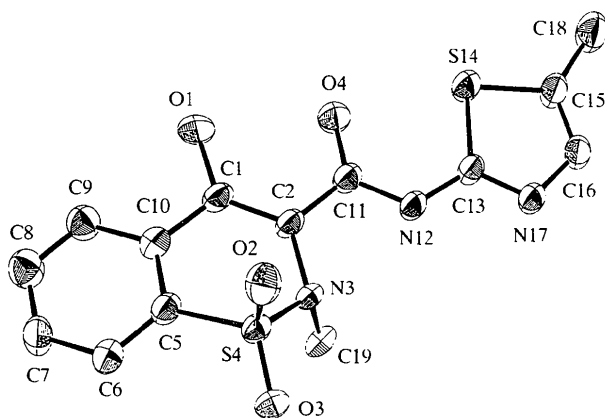

Fig. 1. *ORTEP*II (Johnson, 1976) diagram (50% probability ellipsoids) of the title molecule with the numbering scheme.

Experimental

Single crystals of meloxicam were obtained from a methanol solution by slow evaporation.

Crystal data

$C_{14}H_{13}N_3O_4S_2$	Cu $K\alpha$ radiation
$M_r = 351.39$	$\lambda = 1.5418$ Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
$a = 6.996(1)$ Å	$\theta = 3-23°$
$b = 8.106(1)$ Å	$\mu = 3.486$ mm^{-1}
$c = 13.602(1)$ Å	$T = 293(2)$ K
$\alpha = 85.68(1)°$	Prismatic
$\beta = 88.36(1)°$	$0.2 \times 0.2 \times 0.1$ mm
$\gamma = 74.88(1)°$	Yellow
$V = 742.5(2)$ Å^3	
$Z = 2$	
$D_x = 1.572$ Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	2427 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.018$
Absorption correction:	$\theta_{max} = 71.93°$
empirical via ψ scans	$h = 0 \rightarrow 8$
(North et al., 1968)	$k = -9 \rightarrow 9$
$T_{min} = 0.662, T_{max} = 0.706$	$l = -16 \rightarrow 16$
3054 measured reflections	3 standard reflections
2801 independent reflections	every 100 reflections
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.002$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{max} = 0.247$ e Å^{-3}
$wR(F^2) = 0.100$	$\Delta\rho_{min} = -0.517$ e Å^{-3}
$S = 1.061$	Extinction correction:
2801 reflections	SHELXL93
261 parameters	Extinction coefficient:
H atoms refined isotropically	0.0137(9)
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.514P]$	Scattering factors from International Tables for Crystallography (Vol. C)
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Selected geometric parameters (Å, °)*

S4—O3	1.426(2)	O1—C1	1.336(2)
S4—O2	1.429(2)	O4—C11	1.247(3)
S4—N3	1.636(2)	N3—C2	1.440(2)
S4—C5	1.751(2)	C2—C1	1.363(3)
S14—C15	1.723(3)	C10—C5	1.401(3)
S14—C13	1.727(2)	C10—C1	1.461(3)
O3—S4—N3—C2	169.63(15)	C18—C15—C16—N17	179.5(3)
C19—N3—C2—C1	93.0(3)	C5—C6—C7—C8	0.0(4)
C9—C10—C5—C6	2.9(3)	C9—C8—C7—C6	1.3(4)
O2—S4—C5—C10	75.6(2)	C7—C8—C9—C10	-0.5(4)
C10—C5—C6—C7	-2.1(4)	C5—C10—C9—C8	-1.6(4)
N3—C2—C1—O1	-178.2(2)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Data reduction: *XCAD*4 (Harms, 1996). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II

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Tetracyano-*p*-xylene†

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Abstract

The title compound, $C_{12}H_6N_4$, may be considered as the hydrogenated form of tetracyanoquinodimethane. The main property shown in the solid state is luminescence (blue–green emission) and it is due to the stacking interaction established between molecules along the z axis related by a c glide plane.

† IUPAC name: 2,2′-(*p*-phenylene)bis(propanedinitrile).

Comment

Over the last decade, organic semiconductors have attracted renewed interest owing to their potential applications as active layers in electronic and optoelectronic devices such as light-emitting diodes (Cimrova & Neher, 1996; Kalinowski *et al.*, 1996; Burrows *et al.*, 1996). Since its discovery in 1960, tetracyanoquinodimethane (TCNQ) has been among the most extensively studied semiconductors, owing to its ability to form small-band-gap charge-transfer complexes and organic metals suitable for technological uses (Epstein & Reiff, 1988; Bozio & Pecile, 1991; Miller, 1981–1983; Ferraro & Williams, 1987; Marks, 1990). Here we report the synthesis and the crystal structure of the hydrogenated form of TCNQ, namely the tetracyano-*p*-xylene (TCNX) obtained for the first time as a single crystal. As indicated by optical studies, TCNX seems to be a promising candidate for the realization of optoelectronic devices. The results of absorption measurements indicate that TCNX is a semiconductor with a direct optical absorption threshold below 450 nm, comparable to that of TCNQ. The most striking difference between the optical behaviour of TCNQ and TCNX is that TCNX shows an intense photoluminescence spectrum when excited with light of wavelength lower than 450 nm. Preliminary measurements of luminescence *versus* temperature show the presence of at least two emission bands, centred at approximately 480 nm and 530 nm, characterized by different mean lifetimes.

TCNX
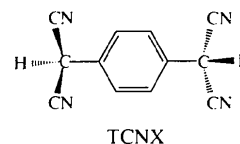
It is well known (Melby *et al.*, 1962) that AgTCNQ microcrystals can be easily obtained by dipping a metallic plate into a saturated solution of TCNQ. According to the literature (Duan *et al.*, 1993), the microcrystals form as a result of a corrosion process that the metal undergoes in the presence of the highly oxidizing TCNQ. We have noticed that when a silver plate is immersed in a TCNQ solution in ethanol in the presence of 1 mM 1,8-octanedithiol, AgTCNQ does not form and needle-shaped TCNX single crystals (mean dimension $0.1 \times 0.3 \times 5$ mm) grow in a few days. This result can be explained as a consequence of the presence of self-assembled dithiol layers that, according to the literature (Ulman, 1996), grow on silver when it is dipped in alcoholic solutions containing alkane dithiols. The dithiol layers act as an anti-corrosion film, preventing the metal oxidation and thus preventing the formation of the AgTCNQ complex. At the same time, the dithiol layers behave as a substrate for the spontaneous growth of the TCNX crystals.