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

O(2)—C(2)	1.204 (9)	N(1)—C(7)	1.418 (9)
O(5)—C(5)	1.229 (9)	N(4)—C(5)	1.365 (9)
O(10)—C(1′)	1.478 (7)	N(4)—C(9)	1.403 (8)
N(1)—C(2)	1.375 (9)		
C(2)—N(1)—C(6)	119.4 (6)	N(4)—C(3)—C(2)	115.0 (7)
C(3)—N(4)—C(5)	117.2 (7)	N(4)—C(5)—C(6)	117.7 (8)
N(1)—C(2)—C(3)	116.4 (7)	N(1)—C(6)—C(5)	117.4 (6)

Table 2. *Torsion angles (°)*

C(2)—N(1)—C(6)—C(5)	φ_1	−32.4 (9)
C(5)—N(4)—C(3)—C(2)	φ_2	−41.0 (8)
N(1)—C(6)—C(5)—N(4)	ψ_1	20.1 (9)
N(4)—C(3)—C(2)—N(1)	ψ_2	28.9 (9)
C(3)—C(2)—N(1)—C(6)	ω_1	7 (1)
C(6)—C(5)—N(4)—C(3)	ω_2	16.2 (9)

The θ-scan width used was $(1.30 + 0.30\tan\theta)°$ at a speed of 16° min^{-1} (in ω). The weak reflections were rescanned a maximum of four times and the counts accumulated to ensure good counting statistics. Stationary background counts were made on each side of the reflection with a 2:1 ratio of peak to background counting time. The maximum limit of 120° in 2θ was used. Higher-angle data collection was not possible and would have resulted in hardware collision. Owing to the small size and weakly diffracting nature of the crystal, collection of data to any higher angle would not have significantly improved the data to parameter ratio in this particular case. The reader should however treat with discretion the somewhat detailed comparisons given in the discussion. The structure was solved by the direct methods program *SIR*92 (Altomare *et al.*, 1994) and expanded using Fourier techniques (*DIRDIF*92; Beurskens *et al.*, 1992). H atoms were located from a difference map and fixed at ideal positions with C—H = 0.96 Å and U_{iso} = $1.2U_{eq}$(C).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992*b*). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: TA1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Glimepiride

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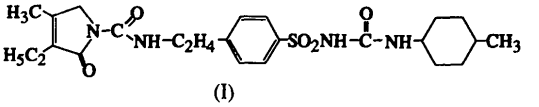
Abstract

Glimepiride, 1-{4-[2-(3-ethyl-4-methyl-2-oxo-3-pyrroline-1-carboxyamido)ethyl]phenylsulfonyl}-3-(*trans*-4-methylcyclohexyl)urea, $C_{24}H_{34}N_4O_5S$, is a drug used in the treatment of non-insulin dependent diabetes mellitus.

Comment

Glimepiride, (I), is a second-generation sulfonylurea compound used in the treatment of non-insulin dependent diabetes mellitus (Holmes, Heel, Brogden, Speight & Avery, 1984). Structural data on glimepiride is limited and so far only one stable polymorphic form is known; its solubility is 1.2 mg l^{-1} (293 K) at pH 7 (Iwata, 1997). The crystal structure of this stable form determined by X-ray crystallographic analysis is reported here.



(I)

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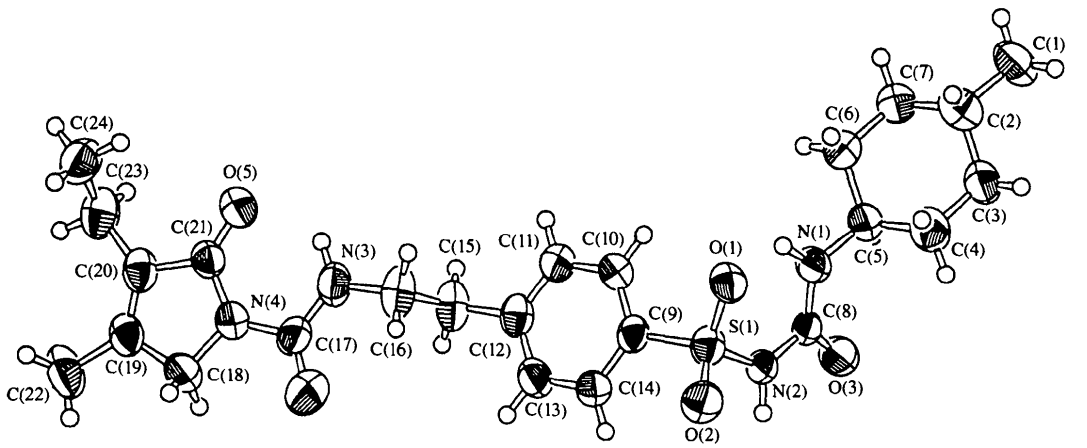

Fig. 1. The molecular structure and atomic numbering scheme of (I). The displacement ellipsoids are drawn at the 50% probability level.

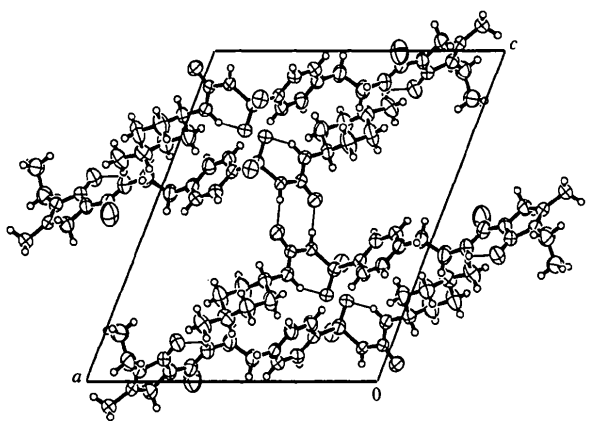

Fig. 2. The packing of (I) viewed down the *b* axis. Thin lines indicate the intra- and intermolecular hydrogen-bonding interactions.

Experimental

Crystals of (I) were grown by diffusing ethanol into a chloroform solution of (I) at room temperature.

Crystal data

$C_{24}H_{34}N_4O_5S$
M_r = 490.62
Monoclinic
$P2_1/n$
a = 15.279 (4) Å
b = 9.812 (3) Å
c = 18.178 (4) Å
β = 111.96 (2)°
V = 2527 (1) Å^3
Z = 4
D_x = 1.289 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 28.20–28.47°
μ = 0.1482 mm^{-1}
T = 297 K
Irregular
0.30 × 0.25 × 0.20 mm
Colorless

Data collection

Rigaku AFC-7*R* diffractometer
R_{int} = 0.023
θ_{max} = 60.06°
$\omega/2\theta$ scans
Absorption correction: none
4180 measured reflections
4015 independent reflections
3272 reflections with $I > 3\sigma(I)$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 11$
$l = -20 \rightarrow 18$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F
R = 0.046
wR = 0.077
S = 2.430
3272 reflections
307 parameters
H atoms not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max}$ = 0.016
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected bond lengths (Å)*

S(1)—O(1)	1.431 (2)	C(3)—C(4)	1.515 (5)
S(1)—O(2)	1.420 (2)	C(4)—C(5)	1.505 (4)
S(1)—N(2)	1.643 (2)	C(5)—C(6)	1.502 (4)
S(1)—C(9)	1.759 (3)	C(6)—C(7)	1.519 (4)
O(3)—C(8)	1.230 (3)	C(9)—C(10)	1.384 (4)
O(4)—C(17)	1.193 (4)	C(9)—C(14)	1.381 (3)
O(5)—C(21)	1.217 (3)	C(10)—C(11)	1.368 (4)
N(1)—C(5)	1.468 (3)	C(11)—C(12)	1.386 (4)
N(1)—C(8)	1.318 (3)	C(12)—C(13)	1.388 (4)
N(2)—C(8)	1.401 (3)	C(12)—C(15)	1.499 (4)
N(3)—C(16)	1.457 (4)	C(13)—C(14)	1.384 (4)
N(3)—C(17)	1.341 (4)	C(15)—C(16)	1.503 (4)
N(4)—C(17)	1.408 (4)	C(18)—C(19)	1.499 (5)
N(4)—C(18)	1.447 (4)	C(19)—C(20)	1.323 (4)
N(4)—C(21)	1.388 (4)	C(19)—C(22)	1.494 (4)
C(1)—C(2)	1.520 (4)	C(20)—C(21)	1.469 (4)
C(2)—C(3)	1.502 (5)	C(20)—C(23)	1.493 (4)
C(2)—C(7)	1.518 (4)	C(23)—C(24)	1.492 (5)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *MULTAN*88 (Debaerdemaeker, Germain, Main, Refaat & Woolfson, 1988). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: TA1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-(Carbamoylmethyl)pyridinium Perchlorate and *N*-(Carbamoylmethyl)-*N'*-methyl-4,4'-bipyridinium Diperchlorate Hydrate

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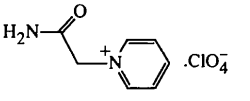
Abstract

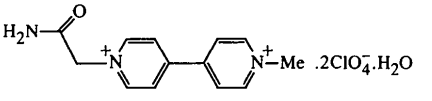
The title compounds, $C_7H_9N_2O^+.ClO_4^-$, (1), and $C_{13}H_{15}N_3O^{2+}.2ClO_4^-.H_2O$, (2), have been synthesized and their crystal structures determined. The C—N(carbamoyl) bond in each compound has some double-bond character [bond lengths 1.319 (5) for (1) and 1.325 (7) Å for (2)], indicating that the sp^2 hybridization is delocalized over the O—C—N(carbamoyl) unit. The carbamoylmethyl plane is canted with respect to the attached pyridinium plane at an angle of 63.5 (2) for (1) and 79.1 (2)° for (2). Two major conformers have been found in the molecular mechanics calculations performed on the *N*-carbamoylmethylpyridinium cation.

Comment

We have recently shown that the amidate-bridged *cis*-diammineplatinum(II) dimers, $[Pt_2^{II}(NH_3)_4(\mu\text{-amidato-}N,O)_2]^{2+}$ (amidato is acetamidato, *etc.*), are generally efficient as H_2-producing catalysts in a well known photosystem consisting of edta, tris(2,2'-bipyridine)-ruthenium(II) and methyl viologen (*N*,*N'*-dimethyl-4,4'-bipyridinium salt) (Sakai, Kizaki, Tsubomura & Matsumoto, 1993). In this system, the methyl viologen cation serves as an electron acceptor which oxidatively quenches the excited state of the ruthenium complex. The resulting radical cation reduces water to molecular hydrogen in the presence of a suitable catalyst such as colloidal platinum or the platinum(II) dimers mentioned above. From the standpoint of so-called 'supramolecular chemistry', it is worth developing systems in which more than two components of the above essential chemical species are combined into a single bifunctional molecule in an appropriate spatial arrangement. With the aim of obtaining new platinum dimers covalently attached to pyridinium moieties, with quenching properties similar to those of methyl viologen, the title amides, (1) and (2), have been prepared and characterized. Other important similar examples may be the *N*-carboxyalkyl-*N'*-methyl-4,4'-bipyridinium compounds reported by Willner *et al.* (1994).



(1)



(2)

The crystal structures of (1) and (2) are shown in Figs. 1 and 2, respectively. The carbamoylmethyl plane is canted with respect to the attached pyridinium plane at an angle of 63.5 (2) for (1) and 79.1 (2)° for (2). Both the carbamoylmethyl and pyridinium moieties are close to being planar with the four- and six-atom r.m.s. deviations less than 0.006 Å. It is notable that the orientation of the carbamoyl unit in (1) is very similar to that in (2), suggesting that this conformation is the thermodynamically favourable one. Perhaps this is due to the electrostatic attraction between the positively charged pyridinium N atom and the negatively charged carbamoyl O atom, however, it must be noted that this conformation also minimizes steric interactions between the O(1) atom and the *ortho*-H atoms [H1 and H5 in (1),

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