final cycle 0·2. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1,\ldots,4$) (*International Tables for X-ray Crystallography*, 1974). Calculations performed on FACOM M-730 computer at Shionogi Research Laboratories. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A perspective view of the molecule with the atom-numbering system and a stereoview of the crystal packing drawn using the program *PLUTO* (Motherwell & Clegg, 1978) are presented in Figs. 1 and 2, respectively.

Related literature. The structure of the title compound reported here has been discussed in Kamisako, Suwa, Honda, Isoi, Nakai, Shiro & Machida (1987).

The author thanks Professor Wasuke Kamisako, Mukogawa Women's University, for the supply of crystals.

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* Lists of structure factors, anisotropic temperature factors of the non-H atoms and atomic coordinates of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51858 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

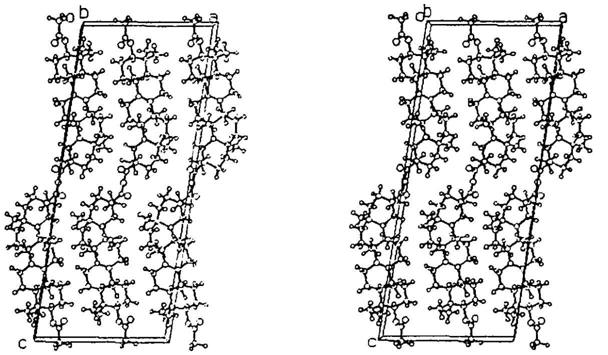

Fig. 2. A stereoview of the unit-cell packing.

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Structure of 2,3-Dihydro-6-(3-nitrobenzylidene)-imidazo[2,1-*b*]thiazol-5(6*H*)-one

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Abstract. $C_{12}H_9N_2O_3S$, $M_r = 261\cdot29$, monoclinic, $P2_1/n$, $a = 5\cdot3014\,(4)$, $b = 20\cdot8420\,(2)$, $c = 10\cdot8287\,(3)$ Å, $\beta = 98\cdot08\,(8)°$, $V = 1184\cdot6\,(5)$ Å^3, $Z = 4$, $D_x = 1\cdot47$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot27$ mm^{-1}, $F(000) = 540$, final $R = 0\cdot068$ for 1129 observed reflections [$I > 2\sigma(I)$]. The two fused five-membered rings forming the main part of the molecule are coplanar. The benzene ring is at an angle of 8·1 (7)° to this plane.

Experimental. The title compound (I) crystallized from glacial acetic acid in the form of a very thin yellow needle, monoclinic form. Crystal dimensions 0·2 × 0·15 × 0·4 mm. Preliminary data obtained from oscillation and Weissenberg photographs (Cu $K\alpha$ radiation); CAD-4 diffractometer. Final lattice parameters from least-squares refinement of 25 reflections ($4 < \theta < 12°$); no absorption correction applied; $\theta < 25°$ (h −6/6, k 0/24, l 0/9); ω–2θ scan technique; Mo $K\alpha$ radiation at room temperature; intensity of three ($\bar{2}7\bar{3}$, $7\bar{1}2$, $6\bar{1}5$) standard reflections

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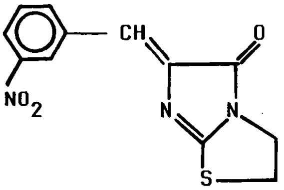
Table 1. *Positional parameters* ($\times 10^4$) *and* U_{eq} ($\times 10^3$) *for the non*-H *atoms*

	x	y	z	U_{eq}
S	1994 (3)	2140 (1)	1920 (1)	60 (0)
C4	327 (9)	2424 (2)	4122 (4)	57 (1)
C5	−265 (10)	1986 (2)	2989 (4)	74 (2)
C1	3221 (8)	2807 (2)	2732 (3)	43 (1)
N2	4906 (7)	3224 (2)	2503 (3)	42 (1)
N1	2131 (7)	2903 (2)	3811 (3)	45 (1)
C2	3200 (9)	3454 (2)	4375 (4)	46 (1)
O1	2710 (7)	3689 (1)	5342 (2)	63 (1)
C3	5015 (9)	3658 (2)	3504 (3)	41 (1)
C6	6443 (9)	4186 (2)	3685 (3)	45 (1)
C11	8300 (9)	4454 (2)	2948 (3)	41 (1)
C12	9090 (9)	4117 (2)	1940 (3)	41 (1)
C13	10885 (9)	4388 (2)	1313 (3)	41 (1)
N3	11761 (9)	4022 (2)	282 (3)	52 (1)
O3	13618 (7)	4225 (2)	−153 (3)	74 (1)
O2	10596 (7)	3534 (1)	−78 (3)	65 (1)
C14	11997 (9)	4975 (2)	1618 (4)	55 (1)
C15	11196 (10)	5311 (2)	2595 (4)	63 (2)
C16	9368 (10)	5055 (2)	3248 (4)	54 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C5—S	1·808 (4)	C1—S	1·722 (4)
C5—C4	1·526 (5)	N1—C4	1·455 (5)
N2—C1	1·296 (5)	N1—C1	1·389 (5)
C3—N2	1·407 (5)	C2—N1	1·383 (5)
O1—C2	1·217 (4)	C3—C2	1·500 (6)
C6—C3	1·334 (5)	C11—C6	1·462 (5)
C12—C11	1·411 (5)	C16—C11	1·393 (5)
C13—C12	1·367 (5)	N3—C13	1·479 (5)
C14—C13	1·377 (5)	O3—N3	1·225 (4)
O2—N3	1·226 (4)	C15—C14	1·385 (6)
C16—C15	1·384 (6)		
C1—S—C5	93·0 (2)	N1—C4—C5	107·1 (3)
C4—C5—S	109·1 (3)	N2—C1—S	131·7 (3)
N1—C1—S	112·0 (3)	N1—C1—N2	116·3 (4)
C3—N2—C1	103·9 (3)	C1—N1—C4	117·3 (3)
C2—N1—C4	135·2 (3)	C2—N1—C1	107·4 (4)
O1—C2—N1	126·2 (4)	C3—C2—N1	102·6 (4)
C3—C2—O1	131·3 (4)	C2—C3—N2	109·9 (4)
C6—C3—N2	127·2 (4)	C6—C3—C2	122·9 (4)
C11—C6—C3	130·2 (4)	C12—C11—C6	121·9 (4)
C16—C11—C6	119·9 (4)	C16—C11—C12	118·2 (4)
C13—C12—C11	118·8 (4)	N3—C13—C12	118·4 (4)
C14—C13—C12	123·5 (4)	C14—C13—N3	118·0 (4)
O3—N3—C13	117·9 (4)	O2—N3—C13	118·1 (4)
O2—N3—O3	124·0 (4)	C15—C14—C13	117·7 (5)
C16—C15—C14	120·5 (4)	C15—C16—C11	121·2 (4)

monitored every 70 reflections showed no significant fluctuations; 2862 reflections measured; after data reduction 1579 independent reflections; 1129 observed reflections with $I > 2\sigma(I)$.



(I)

Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986) with $R(E) = 0{\cdot}34$. E map provides positions for all non-H atoms. ΔF atoms showed the positions for all H atoms; isotropic thermal parameters of H atoms taken initially as 1·5 times the temperature factors for their parent C atoms. The parameters for H atoms were refined in the riding model. Refinement by full-matrix least squares with anisotropic temperature factors for all non-H atoms converged to $R = 0{\cdot}068$ {with $w = 1{\cdot}4373/[\sigma(F)^2 + 0{\cdot}0001(F)^2]$ to $wR = 0{\cdot}0413$} with the empirical extinction-correction coefficient $g = 0{\cdot}0008$; changes in all parameters $\Delta/\sigma < 0{\cdot}001$. The minimum and maximum peaks in the final $\Delta\rho$ map were $-0{\cdot}36$ and $0{\cdot}34$ e Å^{-3}; atomic scattering factors from *SHELX*76 (Sheldrick, 1976). All calculations performed using *SHELX*76 and *CSU* programs (Vicković, 1988) on an Amstrad 1512 PC. The atomic parameters for non-H atoms are given in Table 1.* Table 2 contains bond distances and angles, while the atom-labelling scheme is shown in Fig. 1 and a view of the molecular packing is shown in Fig. 2.

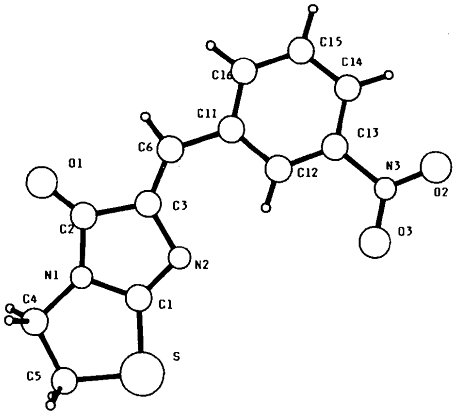


Fig. 1. View of the molecule with atom numbering.

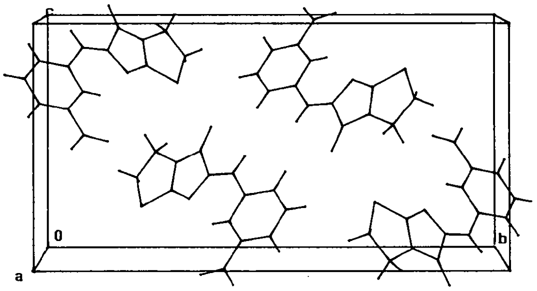


Fig. 2. Molecular packing.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51842 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The preparation of the title compound is an extension of the syntheses and X-ray structure analyses of annelated 2-thiohydantoin derivatives (Kieć-Kononowicz, Zejc, Mikołajczyk, Zatorski, Karolak-Wojciechowska & Wieczorek, 1980, 1981; Kieć-Kononowicz, Karolak-Wojciechowska & Kwiatkowski, 1988; Kieć-Kononowicz, Zatorski & Karolak-Wojciechowska, 1989; Karolak-Wojciechowska, Mikołajczyk, Zatorski, Kieć-Kononowicz & Zejc, 1985; Karolak-Wojciechowska & Kieć-Kononowicz, 1987). All synthesized compounds have been the subject of preliminary pharmacological screening. The aim of the work is further examination of structure–activity relationships.

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Crystal Structure and Absolute Configuration of (+)-Methadone Hydrobromide

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Abstract. $C_{21}H_{28}NO^+.Br^-$, M_r = 390·37, triclinic, $P1$, a = 9·577 (1), b = 9·591 (2), c = 13·482 (2) Å, α = 70·52 (1), β = 93·09 (1), γ = 119·32 (1)°, V = 1008·9 (3) Å^3, Z = 2, D_x = 1·285 g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 28·2 cm^{-1}, F(000) = 408, T = 298 K, R = 0·0346 for 2905 reflections with $I \geq$ 1·5$\sigma(I)$. There are two independent molecules in the asymmetric unit as predicted by solid-state NMR spectroscopy. The molecular conformations of the two molecules are similar to each other and also to the conformation previously observed in the monoclinic form.

Experimental. (+)-Methadone hydrobromide, $CH_3CH_2COC(Ph)_2CH_2CH(CH_3)N(CH_3)_2.HBr$, was crystallized as thick colorless plates from a concentrated solution of the salt in a 1:15 (v/v) mixture of methanol and ether at room temperature. In view of the polymorphous nature of methadone salts (Hanson & Ahmed, 1958) a powdered sample was checked by solid-state NMR which revealed the presence of a single crystalline form (Sumner, Moreland, Carroll, Brine & Boldt, 1989). An almost cubic fragment of approximate dimensions (0·50 × 0·50 × 0·40) mm was cut from a larger piece and used for all X-ray data collection using Cu $K\alpha$ radiation on a Nicolet $R3m/\mu$ four-circle automatic diffractometer equipped with a highly oriented graphite monochromator. Cell dimensions* were obtained by a least-squares refinement of the setting angles of 25 reflections with $50 \leq 2\theta \leq 65°$. Intensity data were collected by the $\omega/2\theta$-scan method up to 115° in 2θ at variable scan speeds between 3·9 and 29·3° min^{-1} depending on intensity; range in hkl: $0 < h < 10$, $-10 < k < 9$, $-14 < l < 14$. Stationary backgrounds were measured for 1/2 of scan time on each side of a peak. Two reflections (021, 211) were measured as standards after every 48 reflections to monitor the instrument and crystal stability. A maximum variation of only ± 1·0% in the intensity of either standard was observed. 2956 reflections were measured which yielded 2905 unique reflections with intensities $I \geq$ 1·5$\sigma(I)$; $R_{\rm int}$ = 0·0041. The intensities were corrected for background, Lorentz and polarization effects and absorption, the latter by empirical ψ scan (μ =

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* The cell given in the *Abstract* can be transformed to the standard cell with all angles acute by the transformation (100/0$\bar{1}$0/00$\bar{1}$).

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