Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Acetyl-3-methyl-4*H*-1,4-benzothiazine: a redetermination at 100 K

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.120
Data-to-parameter ratio = 15.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

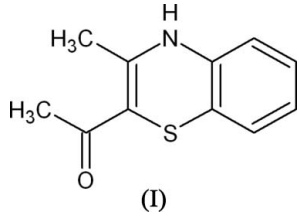
Received 13 March 2006
Accepted 23 March 2006

The structure of the title compound, $C_{11}H_{11}NOS$, has been reported previously at room temperature by Ferguson & Ruhl [*Cryst. Struct. Commun.* (1982), **11**, 1033–1038]. We report here a redetermination, at 100 (2) K, with improved precision. The structure displays N—H···O hydrogen bonding.

Comment

1,4-Benzothiazine and its derivatives have attracted intense interest in recent years because of their diverse pharmacological properties (Akkurt, Öztürk Yıldırım *et al.*, 2005; Akkurt, Türktekin *et al.*, 2005; Wang *et al.*, 2005; Aravindan *et al.*, 2003); some of these compounds exhibit antimicrobial (Habib *et al.*, 1997; Sastry *et al.*, 1990; Rao *et al.*, 1982, 1983), antibacterial (Desai & Mehta, 1997; Miky *et al.*, 1997), antifungal (Chaffman & Brogden, 1985), anti-oxidant (Vladimirov *et al.*, 1991), antiviral (Ergen *et al.*, 1996), anti-HIV-1 (Invidiata *et al.*, 1996) and antihypertensive activities (Kando & Hashimoto, 1993; Keita *et al.*, 2000).

The antimicrobial activities of the title compound, (I), have been studied. The compound is active against *Staphylococcus aureus* [MIC (minimum inhibitory concentration) 39.06 mg ml^{-1}], *Acinetobacter haemolyticus* (MIC 39.06 mg ml^{-1}), *Pseudomonas aeruginosa* (MIC 9.76 mg ml^{-1}) and *Candida albicans* (MIC 39.06 mg ml^{-1}), but it is not active against *Escherichia coli*, *Klebsiella pneumoniae* and *Salmonella typhimirium*. Antifungal activity was determined against *Candida albicans*, and the compound showed better activity (MIC 39.06) than fluconazole did.



(I)

The crystal structure of the title compound, (I), has been reported previously at room temperature (Ferguson & Ruhl, 1982). We have redetermined this crystal structure at 100 K. The present work is of significantly improved precision and we were able to determine the positions of the H atoms. The precision of the unit-cell dimensions was improved by an order of magnitude. The unit-cell volume decreased by *ca* 37 Å^3, consistent with the determination at low temperature. In general, the molecular geometric parameters are slightly different; the bond lengths are shorter in the low-temperature structure. In addition, intermolecular N—H···O interactions

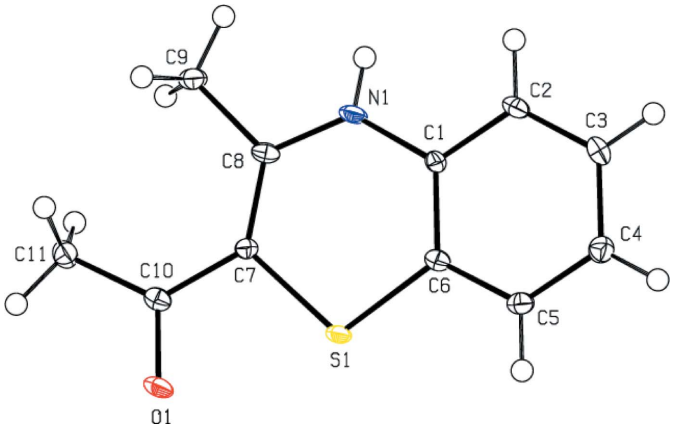


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids. H atoms are represented by circles of arbitrary size.

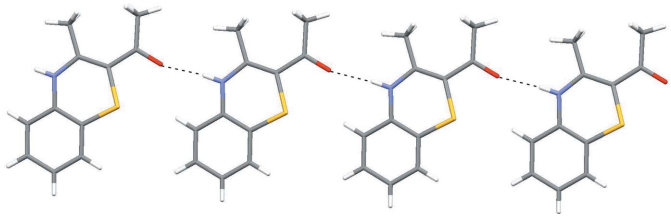


Figure 2
A view of the hydrogen bonding (dashed lines) in compound (I).

connect molecules into a one-dimensional chain along the *b* axis (Table 1 and Fig. 2). Excluding H atoms, the molecule is essentially planar, as seen in Fig. 3. The six-membered rings are planar, as indicated by the torsion angles C1—C2—C3—C4 = −1.0 (3)°, C7—S1—C6—C1 = −7.4 (2)° and C1—N1—C8—C7 = −4.9 (3)°. The remaining parts are also planar [S1—C7—C10—O1 = 3.8 (2)°, S1—C7—C10—C11 = −173.90 (17)° and C10—C7—C8—C9 = −0.5 (4)°.

Experimental

Compound (I) was prepared by the reaction of 2-aminothiophenol (0.25 g, 2 mmol) and acetylacetone (0.2 g, 2 mmol) in ethanol (50 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. Elemental analysis calculated: C 64.36, H 5.40, N 6.82%; found: C 64.71, H 5.55, N 6.78%. IR (cm^{-1}): 1633 (C═N), 1790 (C═O), 2363 (S—*R*), 1495, 1458 (benzene ring).

Crystal data

$C_{11}H_{11}NOS$
M_r = 205.27
Monoclinic, $P2_1/c$
a = 7.3606 (15) Å
b = 7.4968 (15) Å
c = 17.375 (4) Å
β = 97.78 (3)°
V = 949.9 (4) Å^3
Z = 4
D_x = 1.435 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2288 reflections
θ = 2.4–27.5°
μ = 0.30 mm^{-1}
T = 100 (2) K
Prism, orange
0.4 × 0.2 × 0.2 mm

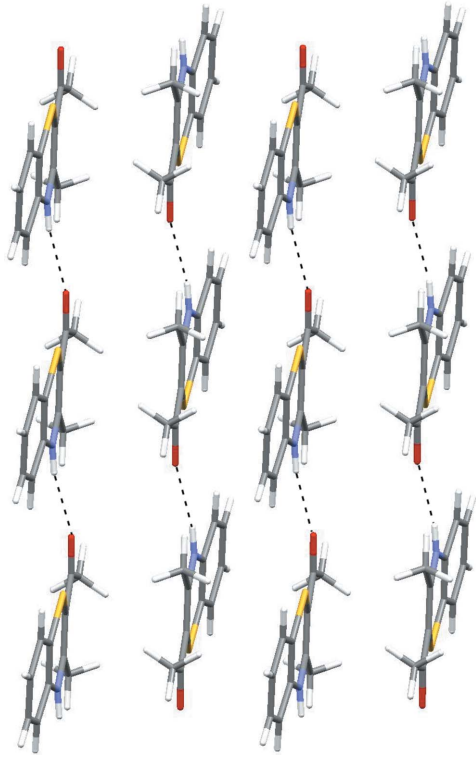


Figure 3
The crystal packing of (I). Hydrogen bonds are shown as dashed lines.

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.845, T_{max} = 0.941
6163 measured reflections
2085 independent reflections
1908 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°
h = −9 → 9
k = −9 → 9
l = −21 → 22

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.120
S = 1.12
2085 reflections
132 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.4415P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.826 (15)	2.042 (17)	2.854 (2)	166.99 (16)

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

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was

allowed to rotate freely about its C—C bond. The position of the amine H atom was refined freely along with an isotropic displacement parameter. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *MERCURY* (Version 1.4; Bruno *et al.*, 2002; Taylor & Macrae, 2001); software used to prepare material for publication: *SHELXTL* (Bruker, 1999).

The authors are grateful to the research funds of TUBITAK TBAG-2307 (103 T041) for financial support.

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