Structure of 1,2,3,4,9,9a-hexahydro-6,9a-diphenyl-1H-pyrimido[2,1-c][1,4]thiazine

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Abstract

The title compound C\textsubscript{18}H\textsubscript{20}N\textsubscript{2}S is monoclinic, with \(a = 10.256(2)\), \(b = 7.470(4)\), \(c = 21.377(4)\) Å, \(\beta = 101.52(2)^\circ\), \(z = 4\) and space group \(P2_1/n\). The structure was solved by direct methods, and refined by weighted full-matrix least squares. The refinement, based on 1373 reflections with \(I \geq 2.5 \sigma (I)\), converged to a final \(R\) of 0.062 (\(R_w = 0.060\)). The conformation of the thiazine ring is a distorted half chair, and that of the pyrimidine ring is a distorted chair. All nonhydrogen intermolecular distances are greater than 3.5 Å.

Introduction

The condensation reaction of 2,2′-thiadiacetophenone with 1,3-diaminopropane probably proceeds via formation of a ten membered ring system, which on transannular interaction of the N atom with the \(-\text{C}==\text{N}-\) group results in the formation of pyrimido [2,1-c][1,4]thiazine(I). The formation of this bicyclic product was observed also when a similar reaction was carried out with 1,2-diaminoethane and 1,2-diaminopropane, which has been confirmed by crystal structure analyses of 2,3,8,8a-tetrahydro-2-methyl-5,8a-diphenyl-1H-imidazo [2,1-c][1,4]thiazine(II) (Sandhu and Hundal, 1982a) and 2,3,8,8a-tetrahydro-5,8a-diphenyl-1H-imidazo[2,1-c][1,4]thiazine(III) (Sandhu and Hundal, 1982b) abbreviated as DMIT and DIT in the present paper. The structural features of [1,4] thiazines have been very well characterized (Andreetti \textit{et al.}, 1980, and references therein), but studies on pyrimido[2,1-c][1,4]thiazines appear to be
limited only to their characterization by spectroscopic techniques. This fact, and in order to study the effect of fusing rings onto the conformation of the [1,4]thiazine ring system, has prompted us to solve the crystal structure of the title compound.

**Experimental**

Crystals of the title compound were grown from ethyl acetate solution. Preliminary unit-cell parameter and space-group determination was done by photographic methods. A crystal of size 0.1, 0.3, 0.3 mm was used for data collection on Syntex P2\(_1\) single crystal X-ray diffractometer; graphite monochromatized Mo K\(_\alpha\) (\(\lambda = 0.71073\) Å) were used. The final unit-cell parameters were obtained from a least-squares treatment of 25 reflections with 17 \(\leq \theta \leq 21^\circ\).

**Crystal data**

C\(_{18}H_{20}N_2S\), \(M_r = 308.44\), \(D_m = 1.27(1)\), \(D_c = 1.27\) g cm\(^{-1}\), \(a = 10.256(2)\) Å, \(b = 7.470(4)\) Å, \(c = 21.377(4)\) Å, \(\beta = 101.52(2)^\circ\), \(V_c = 1605(1)\) Å\(^3\), \(Z = 4\), space group \(P2_1/m\), \(\mu = 1.9\) cm\(^{-1}\), \(F(000) = 656\).

A total of 1800 reflections \((h = -7\) to 11, \(k = 0\) to 8 and \(l = 0\) to 23) was measured, using the \(\omega - 2\theta\) scan mode. Three standard reflections measured every hour showed no noticeable intensity variations. Data were corrected for Lorentz and polarization effects but not for absorption. A total of 1373 reflections with \(I \geq 2.5\sigma(I)\) were used for the structure determination and refinement. The structure was solved by direct methods using SHELX 76 (Sheldrick, 1976), and refined by least squares with anisotropic temperature factors for non-H atoms. The function minimized was \(\sum w(|F_o| - |F_c|)^2\), where \(w = 1.00/[\sigma^2(F_o) + 0.0001|F_o|^2]\); \(\sigma(F_o)\) was based on counting statistics. All H atoms were located by geometrical considerations (C—H 1.08 Å, \(\angle\) H—C—H 109.5° for \(sp^3\) carbon and 120° for \(sp^2\) carbon; N(7) was assumed to be \(sp^3\)). Phenyl groups were refined as rigid groups (C—C 1.395 Å; C—H 1.08 Å, \(\angle\) C—C—C 120°). All H atoms were assigned fixed \(U_{iso}\) value of 0.07 Å\(^2\). The final \(R\) was 0.062 and \(R_w\) was 0.060. The maximum shift per esd was 0.051, and the final difference Fourier map showed highest and lowest residual peaks 0.3 e Å\(^{-3}\) and \(-0.43\) e Å\(^{-3}\). Complex neutral atom scattering factors were used (Cromer and Mann, 1968; Cromer and Liberman, 1970). Mean plane calculations were computed with PARST (Nardelli, 1983), and all calculations were carried out on a ESPL semimicro SM-32 computer.

**Results and discussion**

The atomic parameters, bond length and angles are listed in Tables 1 and 2. Figure 2 shows the crystallographic labelling scheme used in the structure.