Synthesis of 6,9a-Diaryl-1,2,3,4,9,9a-hexahydropyrimido[2,1-*c*][1,4]thiazines; A Correction of the Structure of 3,9-Diaryl-5,6,7,8-tetrahydro-2*H*-1,4,8-thiadiazecines

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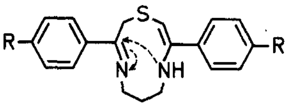
In a previous communication[1] we reported that the reaction of diphenacyl sulfides **2** (R = H, CH$_3$) with 1,2-diaminoethane (**3**, n = 2) gave rise to 3,8-diaryl-2,5,6,7-tetrahydro-1,4,7-thiadiazonines and that the analogous reactions of **2** with 1,3-diaminopropane (**3**, n = 3) gave 3,9-diaryl-5,6,7,8-tetrahydro-2*H*-1,4,8-thiadiazecines **1**.



1 R = H, CH$_3$

A subsequent study of the ^{13}C-N.M.R. spectra of the product of the latter reactions has shown that the correct structure is that of the previously unknown 6,9a-diaryl-1,2,3,4,9,9a-hexahydropyrimido[2,1-*c*][1,4]thiazines **4** (R = H, CH$_3$).

This study of the ^{13}C-N.M.R. data (Table) showed that, in addition to the expected 4 or 5 signals for sp^3 hybridised carbon atoms for **1**, R = H, CH$_3$, respectively a signal for an sp^3 hydrised carbon atom was present at $\delta = 71.340$ and

Table. ^{13}C-N.M.R. Data for 6,9a-Diaryl-1,2,3,4,9,9a-hexahydropyrimido[2,1-*c*][1,4]thiazines **4** (R = H, CH$_3$)

Chemical Shifts[a] δ [ppm]				Assignment
4 (R = H)[b]		**4** (R = CH$_3$)[c]		
—		21.173	(q, $J = 50$ Hz)	CH$_3$
25.225	(t, $J = 55$ Hz)	25.358	(t, $J = 57$ Hz)	C-3
40.562	(t, $J = 72$ Hz)	40.642	(t, $J = 74$ Hz)	C-2
41.831	(t, $J = 68$ Hz)	41.880	(t, $J = 74$ Hz)	C-4
44.977	(t, $J = 73$ Hz)	44.920	(t, $J = 75$ Hz)	C-9
71.340	(s)	71.240	(s)	C-9a
93.760	(d, $J = 111$ Hz)	92.817	(d, $J = 111$ Hz)	C-7
127.340 127.670 127.920 128.630 129.130	unresolved	127.476 127.600 129.150 129.708	unresolved	C$_{arom}$
140.380 142.945 146.224	(s)	137.024 137.365 137.613 140.062 146.262	(s)	C-6 and C$_{arom}$ devoid of H

[a] The multiplicity in the off-resonance proton decoupled spectra is given in brackets.

[b] Solution in CDCl$_3$.

[c] Solution in CDCl$_3$.

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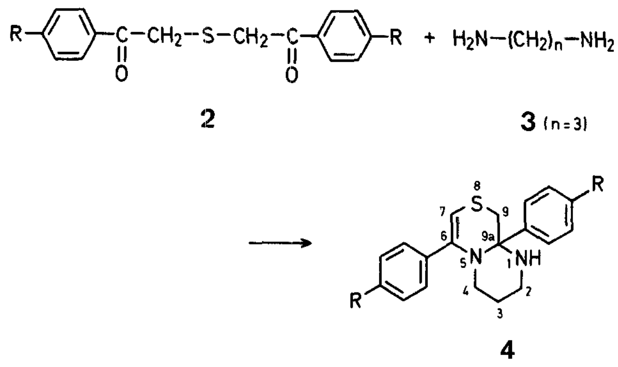

71.240 ppm, respectively, which remained unsplit in the off-resonance proton decoupled spectra. This signal is now assigned to C-9a in structure **4**. The downfield shift of this signal is probably due to the proximity of the two nitrogen atoms[2] in **4**. This observation together with the fact that the aryl carbon atoms linked to the ring system appear as separate signals as opposed to a single signal for the 3,8-diaryl-2,5,6,7-tetrahydro-1,4,7-thiadiazonines support the structure **4** and rule out structure **1**.

The formation of the bicyclic system **4** as opposed to the 10-membered monocyclic system **1** is a case of preferred[3] transannular HN→C N interaction at 1,6-positions.

6,9a-Diaryl-1,2,3,4,9,9a-hexahydropyrimido[2,1-*c*][1,4]thiazines 4 (R = H, CH_3):

For procedure, yield, m.p., microanalytical, and ^{1}H-N.M.R. data see details for product **5** (R = H, CH_3) in Ref. [1].

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[1] S. S. Sandhu, S. S. Tandon, H. Singh, *Synthesis* **1979**, 46.

[2] An sp^3 hybridised carbon atom flanked by three nitrogen atoms has been reported at 72.65 ppm, H. Singh, P. Singh, *Chem. Ind. (London)* **1978**, 126.

[3] Such observations have been recorded in the case of N→C O transannular interactions; W. L. F. Armarego, *Stereochemistry of Heterocyclic Compounds, Part 1, Nitrogen Heterocycles*, John Wiley & Sons, New York, 1977, p. 315.