## SYNTHESIS OF DERIVATIVES OF A NEW HETEROCYCLIC SYSTEM - 1,2,4-DITHIAZEPINE

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Prior to our research 1.2.4-dithiazepine and its substituted derivatives were unknown. We have shown that compounds of this series (I and II) can be obtained by the action of half-molar amounts of bromine on solutions of acetylthioacetic acid arylamides in chloroform at low temperature. The reaction evidently proceeds via the following scheme:

$$\begin{array}{c} O & S \\ CH_3CCH_2CNHC_6H_4R & Br_2 & CH_3CCH_2CNHC_6H_4R & CH_3CCH_2C=NHC_6H_4R \\ \hline \\ O & SH \\ CH_3CCH=CNHC_6H_4R & CH_3CCH_2C=NHC_6H_4R \\ \hline \\ -HBr & S \\ RC_6H_4NHC=CHCCH_3 \\ \hline \\ CH_3CCH & CH_3 \\ \hline \\ RC_6H_4NHC=CHCCH_3 \\ \hline \\ CH_3CCH & CH_3 \\ \hline \\ CH_3CCH$$

## EXPERIMENTAL

 $\odot$ -Acetonylidene-4-phenyl-5-methyl-1,2,4-dithiazepin-7-one (I). This compound, with mp 197-198° (two recrystallizations from absolute alcohol; it began to decompose at 199-201° with the evolution of gas bubbles) and M 309.8 (cryoscopically in dioxane) at 280 (ebullioscopically in dichloroethane) (calculated M 291.39), was obtained in 84% yield. PMR spectrum (in CF<sub>3</sub>COOH),  $\delta$ , ppm: 8.15, 7.15 (5H, m, phenyl ring); 5.65 (2H, s, =CH); 2.15 (6H, s, CH<sub>3</sub>). IR spectrum (in KBr): 3060, 3030, and 3010 (phenyl ring); 2930 (CH<sub>3</sub>); 1615 cm<sup>-1</sup> (C = O). UV spectrum (in ethanol):  $\lambda_{max}$  343 nm (log  $\epsilon$  4.80).

3-Acetonylidene-4-(p-tolyl)-5-methyl-1.2,4-dithiazepin-7-one. This compound, with mp 222-224° (dec., twice from absolute ethanol), was obtained in 67% yield.

The presence of a disulfide bond in I was confirmed by its conversion by means of cyanide ion in dimethylformamide to a cyclic monosulfite = 2-acetonylidene-3-phenyl-4-methyl-1,3-thiazin-6-one (III) = with mp 181-182° (recrystallized twice from ethanol) and M 259 (mass spectroscopically) (M calculated 259), in 74% yield. IR spectrum (KBr): 1665, 1610 (C = O); 1590 cm<sup>-1</sup> (C = C). UV spectrum (in ethanol),  $\lambda_{max}$ , nm (log  $\epsilon$ ): 223 (4.26), 240 (4.04), 288 (3.93), 351 (4.65), 356 (4.64).

The results of elementary analysis of I, II, and III were in agreement with the calculated values.

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