

precipitate of hydrazine was filtered off, and recrystallized from EtOH. Yield 33%, mp 200°–201° C (201°–203° C [3]). 6-Ethoxy-2-hydrazinebenzothiazole was obtained in 30% yield, mp 174°–175° C (175°–176° C [3]).

4- α -Naphthylthiosemicarbazide* mp 145°–147° C (MeOH), colorless prisms, yield 33%. Found: N 19.84; S 14.73%. Calculated for C₁₁H₁₁N₃S: N 19.34; S 14.74%. 2-Hydrazinonaphtho[1,2]thiazole hydrobromide was crystallized from boiling water, using active charcoal. Yield 90%. The hydrobromide was suspended in dimethylformamide, and addition of alkali liberated the hydrazine base, mp 239°–242° C, readily soluble in dimethylformamide and dioxane, less soluble in EtOH. Found: N 19.36; S 15.33%. Calculated for C₁₁H₉N₃S: N 19.54; S 14.89%. It reacted with benzaldehyde to give the hydrazone, mp 195°–196° C (MeOH). Found: N 14.20; S 10.61%. Calculated for C₁₈H₁₃N₃S: N 13.85; 10.57%.

4- β -Naphthylthiosemicarbazide*. Mp 170°–172° C (MeOH), prisms, yield 21–24%. Found: N 19.43; S 15.21%. Calculated for C₁₁H₁₁N₃S: N 19.34; S 14.74%.

*Prepared as described in [5].

2-Hydrazinonaphtho[2,1]-thiazole hydrobromide prepared from it was crystallized from water, (yield 66%). The hydrazine base was separated similarly to 2-hydrazinonaphtho[1,2]thiazole, mp 219°–220° C. 2-Hydrazinonaphtho[2,1]thiazole reacted with CH₃CHO to give a hydrazone, mp 243°–245° C (241°–243° C [4]).

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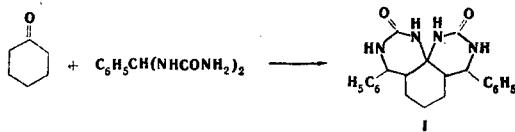
6,6'-SPIROBISHEXAHYDROPYRIMIDINES

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Reaction of cyclohexanone with benzalbisurea (BBU) in ethanol in the presence of HCl gave a compound of the formula C₂₂H₂₄N₄O₂[1]. From its IR, UV, and PMR spectra, and from information in the literature regarding the action of aliphatic ketones on urea [2–4], it can be inferred that it was 2,2'-dioxo-4,4'-diphenyl-5,5'-trimethylene-6,6'-spirobishexahydropyrimidine (I). Compound I was colorless, slightly soluble in organic solvents, mp 335° C (decomp). Found: C 70.5; 70.5; H 6.54; 6.62; N 14.7; 14.9%. Calculated for C₂₂H₂₄N₄O₂: C 70.2; H 6.42; N 14.9%.



IR spectrum (in KBr), ν cm⁻¹: NH 3200, 3400; CH₂ 2860; 2920; C=O 1680 and 1660 (same intensity); absorption bands characteristic of C=C and C=N lacking. UV spectrum (in AcOH) λ_{\max} , m μ (lg ϵ): 258 (2.77); 264 (2.70); 284–302 (hump) (2.30). PMR spectrum (in CF₃COOH, internal standard TMS): CH arom δ 7.0 (singlet); NH δ 4.5 (multiplet); CH₂ and CH δ 2.0–1.0 (multiplet).

Under similar conditions condensation of 2-methylcyclohexanone and acetone with BBM gave respectively 2,2'-dioxo-4,4'-diphenyl-5-methyl-5,5'-trimethylene-6,6'-spirobishexahydropyrimidine (II), mp >350° C, and 2,2'-dioxo-4,4'-diphenyl-6,6'-spirobishexahydropyrimidine III, mp 312°–315° C (decomp). Compound III was also prepared from dibenzalacetone and urea using the method of [3].

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