# SYNTHESIS OF DERIVATIVES OF CONDENSED IMIDAZOLE SYSTEMS FROM 2-HALOIMIDAZOLES AND 8-HALOXANTHINES

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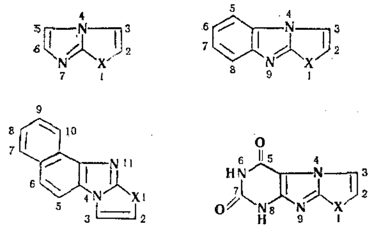
By methods known in the literature for closing imidazole [1] and thiazole [2] rings, we have performed the synthesis of derivatives of a number of condensed imidazole systems. By the reaction of 2-bromo-4,5-diphenylimidazole, 2-chlorobenzimidazole, and 2-chloronaphth[1,2-d]imidazole with α-halo ketones we have obtained the corresponding N-acylmethyl-2-haloimidazoles (I-IV). By heating I-IV and also 8-bromo-7-phenacyltheophylline (V) [3] with ammonia and amines we have obtained VI-IX. The reaction of I-V with thiourea or sodium hydrosulfide and subsequent cyclization of the intermediate mercapto compounds under the action of HBr, $H_2SO_4$, or $POCl_3$ has given X-XIII.



VI-IX X=NH; X-XIII X=S

**2-Bromo-1-phenacyl-4,5-diphenylimidazole (I).** Mp 180-181° C (methanol). Found, %: C 66.05; H 4.04; Br 19.57; N 6.99. Calculated for $C_{23}H_{17}BrN_2O$, %: C 66.19; H 4.11; Br 19.15; N 6.71. **2-Chloro-2-phenacylbenzimidazole (II).** Mp 168-170° C (methanol). Found, %: C 66.29; H 3.89; Cl 13.54; N 10.38. Calculated for $C_{15}H_{11}ClN_2O$, %: C 66.55; H 4.10; Cl 13.10; N 10.35. **3-Acetonyl-2-chloronaphth[1,2-d]imidazole (III).** Mp 133-134° C (aqueous ethanol). Found, %: C 64.75; H 4.18; Cl 14.08; N 10.97. Calculated for $C_{14}H_{11}ClN_2O$, %: C 64.99; H 4.29; Cl 13.71; N 10.83. **3-Phenacyl-2-chloronaphth[1,2-d]imidazole (IV).** Mp 200-201° C (aqueous methanol). Found, %: C 71.55; H 4.27; Cl 11.06; N 8.83. Calculated for $C_{19}H_{13}ClN_2O$, %: C 71.14; H 4.08; Cl 11.05; N 8.73. **1,2,5,6-Tetraphenylimidazo[1,2-a]imidazole (VI).** Mp 252-253° C (aqueous methanol). Found, %: C 84.37; H 5.30; N 10.16. Calculated for $C_{29}H_{21}N_3$, %: C 84.64; H 5.14; N 10.21. **2-Phenylimidazo[1,2-a]benzimidazole (VII).** Mp 285-287° C (decomp., acetic acid). Found, %: C 77.32; H 4.86; N 18.14. Calculated for $C_{15}H_{11}N_3$, %: C 77.23; H 4.75; N 18.02. **1-(p-Methoxyphenyl)-2-phenylnaphth[1,2-d]imidazo[3,2-b]imidazole (VIII).** Mp 266-267° C (ethanol). Found, %: C 80.34; H 4.99; N 10.61. Calculated for $C_{26}H_{19}N_3O$, %: C 80.18; H 4.92; N 10.79. **6,8-Dimethyl-2-phenylimidazo[1,2-f]xanthine (IX).** Decomp. p. > 320° C (acetic acid) [4]. **2,5,6-Triphenylimidazo[2,1-b]thiazole (X).** Mp 175-177° C (dioxane). Found, %: C 78.36; H 4.67; S 8.97. Calculated for $C_{23}H_{16}N_2S$, %: C 78.38; H 4.58; S 9.09. **2-Phenylthiazolo[3,2-a]benzimidazole (XI).** Mp 166-167° C (aqueous ethanol). Found, %: C 72.14; H 3.91; N 10.91. S 12.73. Calculated for $C_{15}H_{10}N_2S$, %: C 71.97; H 4.03; N 11.19; S 12.81. **2-Methylnaphth[1,2-d]imidazo[3,2-b]thiazole (XII).** Mp 184-185° C (decomp., ethanol). Found, %: C 70.54; H 4.25; N 12.04; S 13.66. Calculated for $C_{14}H_{10}N_2S$, %: C 70.56; H 4.23; N 11.75; S 13.46. **6,8-Dimethyl-2-phenylthiazolo[3,2-f]xanthine (XIII).** Mp 260-261.5° C (dimethylformamide). Found, %: C 57.44; H 4.01; N 17.83; S 10.40. Calculated for $C_{15}H_{12}N_4O_2S$, %: C 57.67; H 3.87; N 17.94; S 10.26.

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# THE EXISTENCE OF A TRANSANNULAR BOND BETWEEN THE SILICON ATOM AND THE NITRO GROUP IN THE MOLECULE OF 5-NITRO-2,2,5-TRIMETHYL-2-SILA-1,3-DIOXANE

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In the PMR spectra of 2,2-dimethyl-2-sila-1,3-dioxane (I) and its derivatives II-III (see table), the resonance of the methylene protons has a singlet nature right down to the freezing temperature of solutions in methylene chloride (~70° C). In contrast to this, the PMR spectrum of 2,2,5-trimethyl-5-nitro-2-sila-1,3-dioxane (IV) contains a well-defined AB multiplet of $CH_2$ protons and two singlet signals of the protons of the trimethylsilyl groups even at room temperature. On this basis, it may be concluded that the molecules I-III undergo rapid conformational transitions while IV exists as a single conformer.