Mixtures of the salts obtained with samples of 2,6-diaryl-substituted pyrylium perchlorates synthesized previously [3] gave no depression of the melting point. The structure of the compounds obtained is also confirmed by IR spectroscopy.

The study of the given method is continuing.

REFERENCES

1. M. Siemiatycki and M. Fugnitto, Bull. soc. chim. France, 538, 1961.
2. H. Stetter and A. Reichl, Ber., **93**, 1253, 1960.
3. V. V. Mezheritskii and G. N. Dorofeenko, ZhOrKh, **3**, 1533, 1967.

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Rostov-on-Don State University

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## STRUCTURE OF THE CONDENSATION PRODUCT OF 3,4-XYLENOL WITH CROTONALDEHYDE

S. P. Starkov and L. V. Glushkova

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UDC 547.814.1

In our recent paper [1], by an oversight no reference was made to the paper by L. P. Zalukaev and N. I. Poplavskaya [2] in which, using the condensation of crotonaldehyde with β-naphthol, which is similar in structure to 3,4-xylenol, the authors first established the formation of 4-(2-hydroxylnaphthyl)-2-methyl-5,6-benzochromane. Thus, our investigation on the structure of the product of the condensation of 3,4-xylenol with crotonaldehyde [1] is a confirmation of Zalukaev and Poplavskaya's conclusions [2].

REFERENCES

1. S. P. Starkov and L. V. Glushkova, KhGS [Chemistry of Heterocyclic Compounds], **4**, 16, 1968.
2. L. P. Zalukaev and N. I. Poplavskaya, ZhOKh, **29**, 238, 1959.

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Tambov State Pedagogical Institute

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## SYNTHESIS OF 1-ALKYL-, 1-ARALKYL-, and 1-ARYL-2-AMINOIMIDAZOLES

B. A. Tertov and V. V. Burykin

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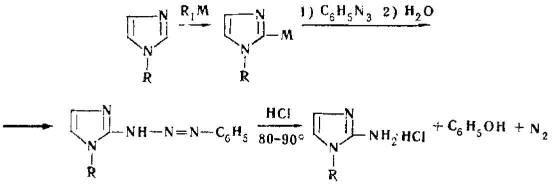
UDC 547.781.5

Several methods for the synthesis of 2-aminoimidazoles have been described [1-7].

We have established that the amino group can easily be introduced into position 2 of a N-substituted imidazole by treating the organosodium or organolithium compound of the corresponding imidazole with phenyl azide and subjecting the triazene formed to cleavage with mineral acid at 80-90° C.



$R = CH_3, C_2H_5, CH_2C_6H_5, C_6H_5$; $R_1 = C_6H_5, C_4H_9$; $M = Na, Li$

**2-Amino-1-methylimidazole hydrochloride monohydrate.** Obtained from 2-lithio-1-methylimidazole. Mp 83-84° C, which corresponds with that given in the literature [6,7]. Yield 70%.

**2-Amino-1-methylimidazole.** Bp 136-137° C (5 mm), mp 81.5-82.5° C. Found, %: C 49.66; H 7.51; N 43.38. Calculated for $C_4H_7N_3$, %: C 49.47; H 7.26; N 43.27.

**2-Amino-1-ethylimidazole.** Obtained from 1-ethyl-2-lithioimidazole. Bp 133-135° C (6 mm). Yield 51%. Found, %: C 53.84; H 8.47; N 37.70. Calculated for $C_5H_9N_3$, %: C 54.03; H 8.16; N 37.81.

**2-Amino-1-benzylimidazole hydrochloride.** Obtained from 1-benzyl-2-sodioimidazole. Mp 185-186° C. Yield 63.5%: Found, %: C 57.48; H 5.95; Cl 17.20; N 20.36. Calculated for $C_{10}H_{11}N_3 \cdot HCl$, %: C 57.28; H 5.77; Cl 16.91; N 20.04.

**2-Amino-1-benzylimidazole.** Mp 139-140° C (from water). Found, %: C 69.22; H 6.56; N 24.59. Calculated for $C_{10}H_{11}N_3$, %: C 69.34; H 6.40; N 24.26.

**2-Amino-1-phenylimidazole hydrochloride.** Obtained from 1-phenyl-2-sodioimidazole. Mp 206–206.5° C. Yield 43.5%. Found, %: C 55.23; H 4.75; Cl 18.20; N 21.80. Calculated for $C_9H_9N_3 \cdot HCl$, %: C 55.25; H 5.16; Cl 18.11; N 21.43.

**2-Amino-1-phenylimidazole.** Mp 125–126° C (from hexane). Found, %: C 67.70; H 5.83; N 26.45. Calculated for $C_9H_9N_3$, %: C 67.90; H 5.69; N 26.40.

REFERENCES

1. Heterocyclic Compounds, Vol. 5 [Russian translation], IL, Moscow, p. 179, 1961.
2. A. de Cat and A. van Dormael, Bull. soc. chim. Belges, **59**, 573, 1950.
3. A. Kreutzberger, J. Org. Chem., **27**, 886, 1962.
4. Th. Pyl, S. Melde, and H. Beyer, Lieb. Ann., **663**, 107, 1963.
5. Th. Pyl, H. Lahmer, and H. Beyer, Ber., **94**, 3217, 1961.
6. A. Lowson, J. Chem. Soc., 307, 1956.
7. B. T. Storey, W. W. Sullivan, and C. L. Moyer, J. Org. Chem., **29**, 3118, 1964.

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Rostov-on-Don State University

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# CYCLOHYDRAZINOCYANATION OF 2,2'-METHYLENE-BIS-CYCLOHEXANONE

V. A. Kaminskii, V. K. Gamov, and M. N. Tilichenko

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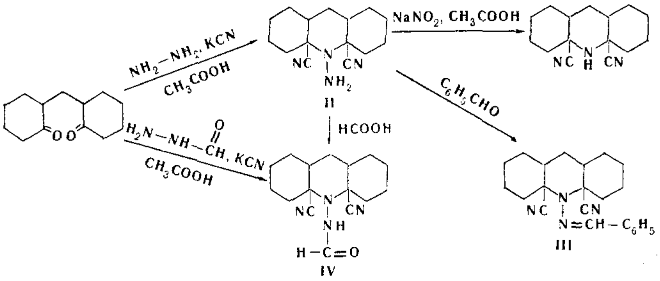
UDC 547.422+547.835

With 2,2'-methylene-bis-cyclohexanone (I) as an example, we have established that 1,5-diketones of this type are capable of undergoing a cyclohydrazinocyanation reaction analogous to the cycloaminocyanation reaction [1]. The reaction takes place exceptionally readily when the given ketone is mixed with KCN and hydrazine in dilute acetic acid; this gives a 90% yield of N-amino-11,14-dicyanoperhydroacridine (II). 1,5-Diketones of the aliphatic series have previously been subjected to reaction with hydrazine and hydrocyanic acid, but only in an aqueous medium, which gives the corresponding N-amino-2,6-dicyanopiperidines in low yields [2,3].



On reaction with benzaldehyde in propanol with the addition of acetic acid anhydride, **II** forms the N-benzylidene derivative **III**. The structure of **II** is shown definitively by the fact that on treatment with sodium nitrite in acetic acid it undergoes deamination and is converted into the known 11,14-dicyanoperhydroacridine [1].

On being boiled with 85% HCOOH, **II** gives the N-formyl derivative **IV**. The same compound is also formed by the reaction of **I**, KCN, and formhydrazide in dilute acetic acid.

**II**. Colorless crystals, mp 210–211° C (benzene–dioxane 1 : 2). Found, %: C 69.55; H 8.76; N 21.45. Calculated for $C_{15}H_{22}N_4$, %: C 69.77; H 8.53; N 21.70. IR spectrum (in $CCl_4$), cm$^{-1}$: 3380 (NH), 2220 (C≡N). **III**. Light yellow crystals, mp 225–228° C (ethanol). Found, %: C 76.22; H 7.69; N 16.31. Calculated for $C_{22}H_{26}N_4$, %: C 76.30; H 7.51; N 16.18. **IV**. Colorless crystals, mp 206–208° C (from 40% ethanol). Found, %: C 67.29; H 8.12; N 19.87. Calculated for $C_{16}H_{22}N_4$, %: C 67.13; H 7.69; N 19.58. IR spectrum (in KBr), cm$^{-1}$: 1690 (amide C=O), 2230 (C≡N).

REFERENCES

1. V. A. Kaminskii and M. N. Tilichenko, KhGS [Chemistry of Heterocyclic Compounds], **3**, 708, 1967.
2. C. G. Overberger and B. S. Marks, J. Am. Chem. Soc., **77**, 4097, 1955.
3. C. G. Overberger, G. Kesslin, and P.-T. Huang, J. Am. Chem. Soc., **81**, 3779, 1959.

27 June 1968

Far Eastern State University,
Vladivostok