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SHORT COMMUNICATIONS

New Synthesis of 9,10-Dimethoxy-1-(3,4-dimethoxyphenyl)-3-(4-chlorophenyl)pyrazino[2,1-*a*]isoquinolinium Bromide

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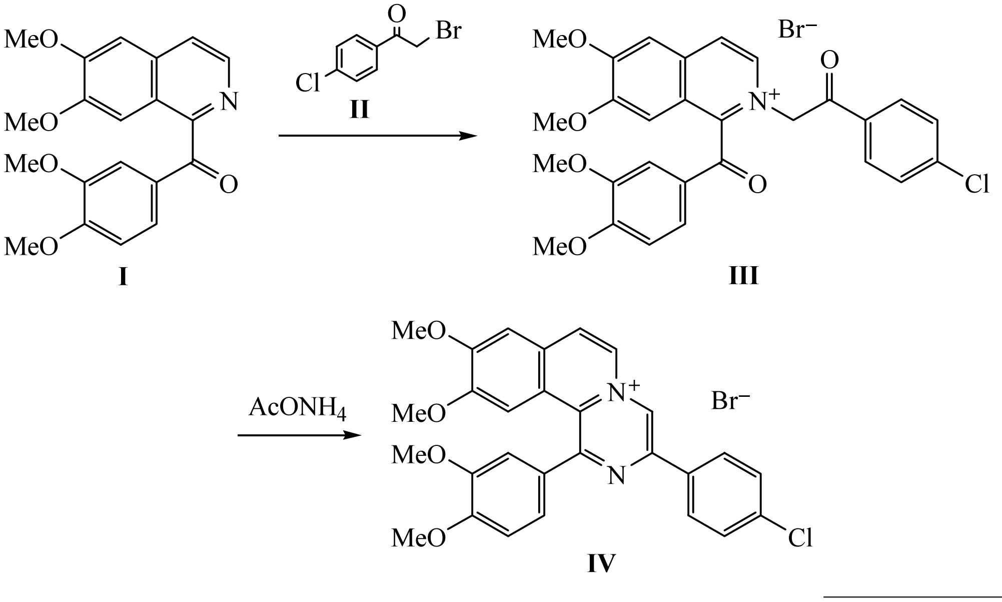
Papaverine and related alkaloids are now attract much attention with the aim at the synthesis of new heterocyclic systems [1, 2]. In this connection the modification of the nearest surrounding of the isoquinoline ring makes it possible to extend essentially the synthetic potential of papaverine alkaloids [3, 4].

We demonstrated that the pyrazinoisoquinolinium system **IV** underwent a ring closure at treating quaternary salt **III** with excess ammonium acetate in acetic acid. In its turn salt **III** was obtained in a quantitative yield by the reaction of papaveraldine (**I**) with *p*-chlorophenacyl bromide (**II**).



The known unique instances of building up pyrazionpyridinium systems were based on the reaction of 2-pyridylketimines with chloroacetyl chloride [5].

By an example of papaveraldine phenacyl salt we showed a new path to designing pyrazinoisoquinolinium systems.

***p*-Chlorophenacylpapaveraldinium bromide (III).** A mixture of 0.01 mol of papaveraldine (**I**) [3] and 0.011 mol of *p*-chlorophenacyl bromide (**II**) was boiled at stirring in acetone for 15 days. The precipitate was filtered off and recrystallized from methanol. Yield 99%, mp 185–186°C. ^{1}H NMR spectrum, δ, ppm: 3.75 s (3H, OCH_3), 3.82 s (3H, OCH_3), 3.89 s (3H, OCH_3), 4.16 s (3H, OCH_3), 6.43 s [2H, $CH_2C(O)$], 6.90 s (1H, H_{arom}), 6.89 d (1H, H_{arom}, *J* 8 Hz), 7.21 s (1H, H_{arom}), 7.41 s (1H, H_{arom}), 7.52 d (2H, C_6H_4Cl, *J* 8 Hz), 7.98 d (2H, C_6H_4Cl, *J* 8 Hz), 8.09 s (1H, H_{arom}), 8.65 d (1H, H_{arom}, *J* 7 Hz), 8.88 d (1H, H_{arom}, *J* 7 Hz). Found, %: C 57.35; H 4.35; N 2.45. $C_{28}H_{25}BrClNO_6$. Calculated, %: C 57.31; H 4.29; N 2.39.

9,10-Dimethoxy-1-(3,4-dimethoxyphenyl)-3-(4-chlorophenyl)pyrazino[2,1-*a*]isoquinolin-5-ium bromide (IV). A mixture of 1 g of salt **III** and 1 g of ammonium acetate was boiled in acetic acid for 7 h. On cooling the precipitated yellow crystals were filtered off, washed with acetic acid, and recrystallized from acetic acid. Yield 80%, mp 198–200°C. ^1H NMR spectrum, δ, ppm: 3.40 s (3H, OCH_3), 3.77 s (3H, OCH_3), 3.92 s (3H, OCH_3), 4.07 s (3H, OCH_3), 7,21 d (1H, H_{arom}, *J* 8 Hz), 7.33 m (3H, H_{arom}), 7.63 d (2H, C_6H_4Cl, *J* 8 Hz), 7.91 s (1H, H_{arom}), 8.39 d (2H, C_6H_4Cl, *J* 8 Hz), 8.64 d (1H, H_{arom}, *J* 7 Hz), 8.39 d (1H, H_{arom}, *J* 7 Hz), 10.32 s (1H, N^+–CH=C). Found, %: C 59.37; H 4.33; N 5.03. $C_{28}H_{24}BrClN_2O_4$. Calculated, %: C 59.22; H 4.26; N 4.93.

^1H NMR spectra (500 MHz) of 1% solutions of substances in DMSO-d_6 were registered on a spectrometer Bruker AM-500. Elemental analysis was carried out on an analyzer Perkin-Elmer 240. Melting point were measured on the Koeffler heating block. The reaction progress was monitored by TLC on Silufol UV-254 plates, eluent benzene–anhydrous ethanol, 9:1.

REFERENCES

1. Krivorotov, D.V., Vorob'ev, M.V., Polukeev, V.A., and Glibin, E.N., *Zh. Org. Khim.,* 2006, vol. 42, p. 594.
2. Mamyrbekova, Zh.A., Soldatova, S.A., Abelentsev, V.I., Solov'eva, T.I., Guryshev, V.N., and Soldatenkov, A.T., *Khim. Farm. Zh.,* 1994, vol. 28, p. 48.
3. Vaccher, C., Berthelot, P., Devergnies, M., Debaert, M. Bonte, J.P., and Viel, C., *J. Heterocycl. Chem.,* 1989, vol. 26, p. 811.
4. de Lera, A.R., Aubourg, S., Suau, R., and Castedo, L., *Heterocycles,* 1987, vol. 26, p. 675.
5. Batori, S. and Messmer, A., *J. Heterocycl. Chem.,* 1990, vol. 27, p. 1673.