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THE BEHAVIOUR OF INTRAMOLECULAR CHARGE TRANSFER COMPLEXES UNDER ELECTRON IMPACT

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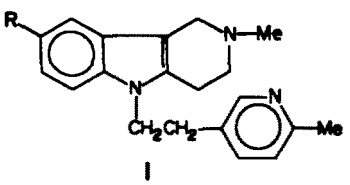
Abstract—Mass spectra of the three series of intramolecular charge transfer complexes: pyridylethylated tetrahydro-γ-carbolines and indoles as well as dinitrobenzoyl derivatives of N-(aminoalkyl)indoles, show stabilities of molecular ions to increase with electron donor strength of substituents in the indole nucleus. The results obtained demonstrate that stabilities of molecular autocomplexes can be estimated from their mass spectra.

During recent years, a new branch of the chemistry of charge transfer complexes (CTC) has developed. Its objective is intramolecular CT complexes (or molecular autocomplexes), which contain interacting donor and acceptor moieties separated by polymethylene bridges that break conjugation chains (see, e.g. Refs. 1, 2).

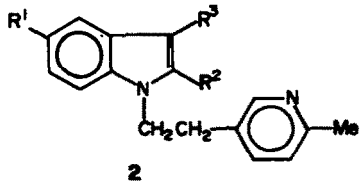
Numerous roles played by indole derivatives in biological systems and the demand for studying their metabolism and mechanisms of biological action have prompted us to undertake a study of fine structural features of indole intramolecular CTC. In addition to the standard techniques for studying CTC, the UV and proton NMR spectroscopy, molecular autocomplexes which contain the donor and acceptor counterparts in a definite stoichiometric ratio, invite the use of mass spectrometry.

With compounds 1, the interaction between the donor (indole) and acceptor (pyridine) counterparts only gives rise to observable effects in the NMR spectra of the protonated forms (protonation strengthens electron withdrawing action of the pyridine nucleus). Thus, the NMR spectrum of **1b** (CS_2) contains an unresolved multiplet at about 7.2–7.6 ppm corresponding to the benzene and pyridine ring protons (the benzene ring protons of model compound **4b** give signals in the same region) and a signal at 8.6 ppm (the pyridine ring α-proton). A solution of **1b** in trifluoroacetic acid furnishes a radically different spectrum (Fig. 1). The multiplet is replaced with a well-resolved spectrum comprising distinct peaks from both benzene and pyridine ring protons. The benzene protons give rise to a doublet at δ 6.2 ppm



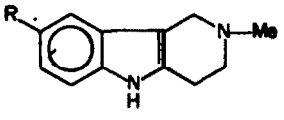
1

1a: R = H
1b: R = Me
1c: R = OMe
1d: R = Br



2

2a: R¹ = R² = H, R³ = Et
2b: R¹ = H, R² = Me, R³ = Pr
2c: R¹ = R² = H, R³ = Me
2d: R¹ = H, R² = Me, R³ = Ph
2e: R¹ = R² = R³ = Me
2f: R¹ = OMe, R² = R³ = Me
2g: R¹ = Me, R² + R³ = $(CH_2)_4$



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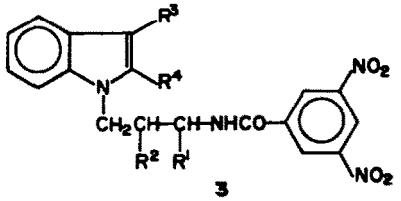
4a: R = H
4b: R = Me
4c: R = OMe
4d: R = Br



3

3a: R¹ = R² = R⁴ = H, R³ = Me
3b: R¹ = R² = H, R³ + R⁴ = $(CH_2)_4$
3c: R¹ = Me, R² = H, R³ + R⁴ = $(CH_2)_4$
3d: R¹ = H, R² = Me, R³ = R⁴ = Me
3e: R¹ = R⁴ = Me, R² = H, R³ = COOEt
3f: R¹ = R² = H, R³ = R⁴ = COOMe

Proof for the existence of compounds **1–3** *in the form of CTC.* We have studied the UV and NMR spectra of compounds **1**, **2**, and **3** in order to make certain that these species occur in the conformations favouring spatial interactions of the CTC type, with the interacting moieties lying above each other.

(8-H, $J_{8,7}$ 8 Hz), a quartet at 6.8 ppm (7-H, $J_{7,8}$ 8 Hz, $J_{7,5}$ 2 Hz), and a singlet broadened by a meta-interaction at about 7.0 ppm (5-H). While with model compound **4b**, the benzene ring proton signals retain their positions on going from CS_2 to trifluoroacetic acid, compound **1b** shows appreciable solvent induced

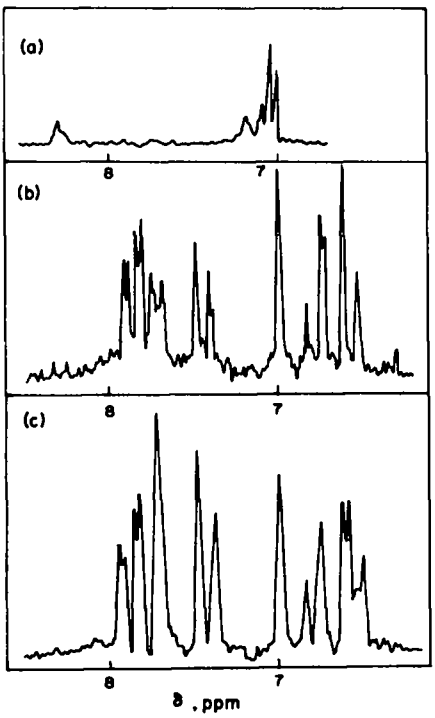


Fig. 1. The NMR spectra of compound 1b. (a) in CS_2; (b) in CF_3COOH; (c) in CF_3COOD.

shifts which amount to $\Delta\delta$ 0.2 and 0.8 ppm for 7-H and 8-H, respectively. The doublet at 7.5 ppm corresponds to the pyridine ring β-proton ($J_{\beta\gamma}$ 8 Hz), and the quartet at δ of 7.9 ppm to the γ-proton ($J_{\gamma\beta}$ 8 Hz, $J_{\gamma\alpha}$ 2 Hz). The α-H signal appears at unusually high field (7.8 ppm, quartet), while the corresponding signal from the model compound, 2-methyl-5-ethylpyridine, occurs at 9.3 ppm (trifluoroacetic acid). The quartet nature of the signal shows the α-proton to be coupled not only with the γ one ($J_{\alpha\gamma}$ 2 Hz), but also with the $\overset{+}{N}$–H pyridine ring proton, the latter due to high basisity of pyridine and low rates of proton exchange under the conditions of spectral measurements. The additional splitting disappears in deuterated trifluoroacetic acid.

The differences between the NMR spectra of model structures and of compounds 1 provide evidence of the formation of intramolecular complexes. The temperature dependence of the NMR spectrum of 1b in trifluoroacetic acid supports this conclusion. The temperature rise from 20 to 50° weakens the complex and shifts the α-H signal downfield by 0.2 ppm. After cooling, the spectrum regains its initial appearance.

Similar effects are observed in the spectra of compounds 2.

Unfortunately, low solubility of compounds 3 in most organic solvents blocks the application of the NMR technique. Even high resolution spectra (Brucker-360) of solutions of 3 in deuterated dimethylsulfoxide do not show signal shifts from the model compounds (namely, the corresponding nonacylated N-(aminoalkyl)indoles and N-methyl-3,5-dinitrobenzamide). It may well be that dissolution of the complexes in that strong solvent leads to effective solvation of both parts of the molecule and destroys the complex, the more so that DMSO is noted[13] for its ability to cause dissociation of CTC.

The UV spectra of compounds 3 have proved more instructive. Though the crystalline products are strongly coloured, the colouration ranging from yellow to red, the CT bands can not be detected in the spectra of their chloroform solutions because of masking effects by the indole and aromatic ring chromophores. To overcome this difficulty, we have resolved the spectra into Gauss' components. The CT bands have thus been assigned and the effects of the pyrrole ring substituents on the positions of their maxima have been determined (Table 1).

As seen from Table 1, the CT bands undergo bathochromic shifts with increase in substituent donor action in the series: **3d > 3b > 3a > 3e > 3f**. This implies facilitation of spatial charge transfer in that direction, i.e. strengthening of CTC-type interactions.

Thus, both NMR and UV spectral data show that compounds 1, 2, and 3 exist in the form of intramolecular CTC.

Mass-spectral study. Electron impact stabilities of the molecular ions from 9-/2-(2 - methylpyridyl - 5)ethyl/-1,2,3,4 - tetrahydro - γ - carbolines (1a–d) (W_M, Table 2) increase with electron donor strength of the substituent R. As seen from Table 2, a pronounced stability increase occurs on going from 1a (W_M 12.3) to 1b–d. With 1c and 1d, the relative increase is about 35%, whereas the introduction of the Me group into the benzene ring gives a 55% stability gain. A lower W_M value for compound 1c

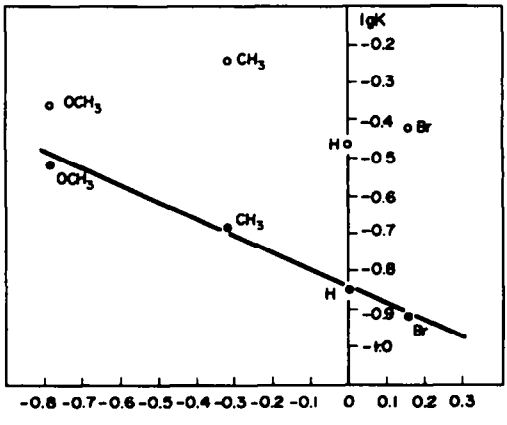


Fig. 2. Correlation of ions F_1 and F_2 formation rates (from the molecular ions 1a–d) with δ^+-constants. O, Formation of ions F_1 ($lgK_1 = lgI_{F_1}/I_M$); ●, Formation of ions F_2 ($lgK_2 = lgI_{F_2}/I_M$).

Table 1. Parameters of the CT bands in electron spectra of compounds 3

Compound	3d	3b	3a	3e	3f
λ_{max}, nm	421	381	378	325	321
lg ε	2.10	2.40	2.13	2.41	3.36

Table 2. Mass-spectra of the compounds 1

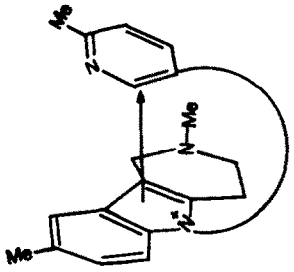
Compound	m/e (intensity, % of the maximal ion)	W_M†
1a	81(5.8) 83(11.6) 85(9.3) 95(5.3) 97(10.6) 111(5.8) 119(6.0) 120(16.6) 128(9.3) 129 (14.6) 143(85.9) 144(9.6) 155(5.3) 156 (100.0) 157(11.1) 183(5.8) 197(6.8) 199 (10.3) 246(7.3) 262(25.0) 263(6.3) 290 (5.0) 304(47.0) 305(84.0) 306(14.0)	12.3
1b	157(72.6) 158(8.7) 170(100.0) 171(13.4) 213(13.4) 261(5.1) 276(36.3) 277(8.1) 318 (35.0) 319(57.8) 320(12.0)	19.1
1c	81(9.0) 83(12.0) 84(5.0) 95(7.0) 97(10.0) 111(6.0) 119(5.1) 120(7.0) 143(11.0) 161 (9.0) 172(34.0) 186(100.0) 187(7.0) 229 (9.0) 238(6.0) 292(34.0) 334(40.0) 335 (96.0) 336(14.0)	16.7
1d	119(14.7) 120(59.0) 121(7.7) 154(14.0) 155 (27.0) 156(5.1) 221(88.3) 222(5.8) 223(86.1) 234(100.0) 235(7.7) 236(95.6) 237(6.7) 261 (5.7) 263(5.1) 277(9.0) 279(8.0) 340(26.6) 341(5.7) 342(25.9) 343(5.4) 382(39.0) 383 (72.2) 384(48.6) 385(66.3) 387(7.7)	16.7

†W_M is the stability of the molecular ion under electron impact ($W_M = I_M/\Sigma I$, where I_M is intensity of $[M]^+$ and ΣI is full ion current).

compared with 1b is due to increase in the number of fragmentation pathways.

The observed W_M variations do not conform to the known patterns of stabilities of aromatic systems; they, however, fit the suggestion of the CTC nature of the compounds studied. In fact, we have shown earlier[14] that substitutions in the benzene ring of tetrahydro-γ-carbolines 4 lead to lowering rather than an increase of the W_M values because of the increased number of fragmentation pathways. An unusual increase of W_M with donor strength of substituents in the benzene ring observed in compounds 1 is characteristic for molecular autocomplexes. Enhancement of electron donating ability leads to the greater extents of charge transfer and hence to stabilization of molecular ions.

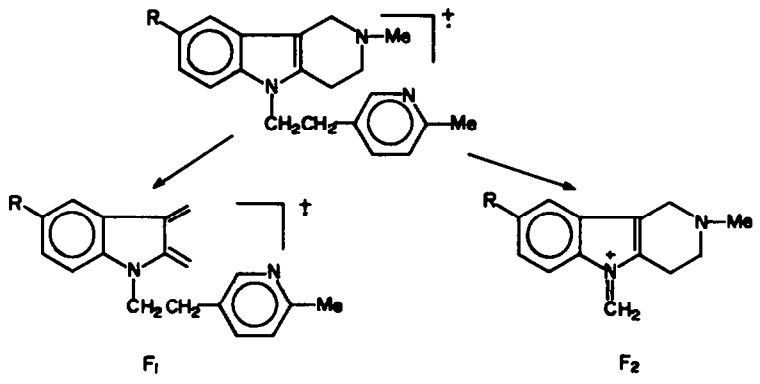
We have already mentioned that protonation of the pyridine nucleus in 1 increases its electron withdrawing action and (according to the NMR data) favours the formation of CTC. Forming of CTC involves electron density redistribution[15] that leads to localization of a partial positive charge in the indole nucleus. As we believed that the molecular ion of unprotonated species, after electron impact has similar charge distributions, it means that the molecular ions may be compared with the protonated forms of the parent molecules.



In fact, the ions F_1 formed by the retrodienic fragmentation of compound 1 are several times less intense than (with unsubstituted at the N atom) tetrahydro-γ-carbolines 4 whole positive charge is mainly localized on the amine nitrogen of the piperidine ring. We have calculated the reaction constants ρ and correlation coefficients r for the formation of F_1 and F_2 from molecular ions 1a–d. With F_1, there is no correlation with the σ^+-constants (ρ 0.470, r −0.1194, Fig. 2, Table 3). With

Table 3. Dates of $\lg K_1$ and $\lg K_2$ and σ^+-constants for the compounds 1a–d

Compound	R	σ^+	I_M	I_{F_1}	I_{F_2}	$K_1=I_{F_1}/I_M$	$K_2=I_{F_2}/I_M$	$\lg K_1$	$\lg K_2$
1c	OCH_3	−0.778	16.7	7.1	4.9	0.4251	0.2934	−0.3715	−0.5327
1b	CH_3	−0.311	19.1	11.0	4.1	0.5759	0.2147	−0.2396	−0.6682
1a	H	0.000	12.3	4.1	1.7	0.3333	0.1382	−0.4772	−0.8595
1d	Br	+0.150	16.7	6.1	2.1	0.3653	0.1257	−0.4373	−0.9007



the F_2 ion, the reaction constant ρ is −0.4126 and the correlation coefficient r is 0.986. The calculation results point to localization of the molecular ion charge mainly on the pyrrole ring nitrogen. Such a situation may develop as a result of electron transfer from the pyrrole to the pyridine ring, the initial charge carrier. Pyridine thus acts as electron acceptor (an obvious analogy to what occurs in the protonated form of compounds 1).

The mass spectra of compounds 1 thus confirm the conclusions made on the basis of the NMR data.

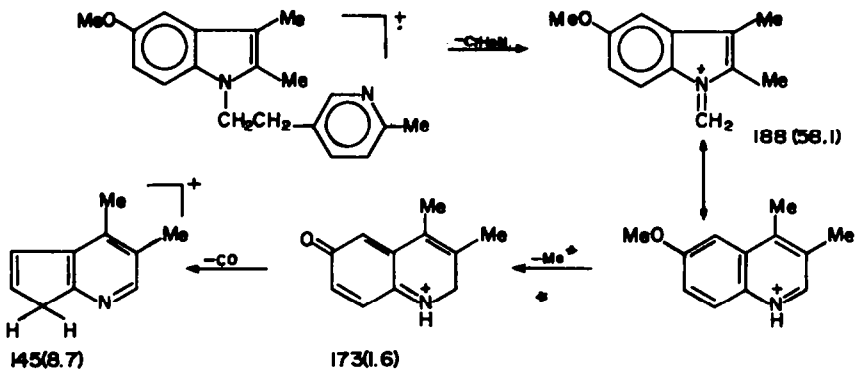
Pyridylethylated indoles 2a–g (Table 4) show a similar behaviour under electron impact conditions. Again, the W_M values (molecular ion stabilities) increase with electron donor strength of the indole moiety. Realization of additional fragmentation pathways for compounds 2a and 2b (fragmentations of the Et and Pr substituents) explains the observed lowering of W_M in these species.

Fragmentations of the molecular ions of compounds 2a–g proceed as with pyridylethylated derivatives in general; there are no specific features to be mentioned. The predominant electron impact induced dissociation involves rupture of the C–C α–β bond of the pyridylethyl chain giving rise to the base peak.

Compound 2b having the 3-Pr substituent in the indole ring gives ions *m/e* 263 and 157 formed by the loss of the Et radical from the molecular and the base ions, respectively. The presence of the 5-OMe substituent explains certain peculiarities observed in the spectrum of 2f. The possible fragmentation pathways are shown in the scheme (see page 2935).

Table 4. Mass-spectra of the compounds 2

Compound	m/e (intensity, % of maximal ion)	W_M
2a	57(11.2) 77(1.0) 86(1.1) 91(1.3) 105(1.3) 106 (1.0) 115(2.1) 128(1.1) 130(5.8) 143(18.0) 158(100.0) 159(10.0) 249(1.2) 264(19.0)	6.6
2b	57(2.1) 77(1.2) 91(1.0) 105(1.0) 106(1.3) 115 (1.7) 128(1.0) 130(2.7) 143(3.0) 144(11.4) 156 (6.5) 157(17.5) 158(5.5) 186(100.0) 187(13.3) 263(45.6) 264(8.7) 292(40.0)	8.6
2c	42(1.1) 77(1.2) 105(1.0) 106(1.4) 115(3.8) 128(1.3) 130(1.3) 143(6.6) 144(100.0) 145 (9.0) 250(19.9)	11.6
2d	57(1.3) 77(1.0) 105(1.7) 106(2.1) 115(2.1) 128(1.0) 130(1.1) 142(1.2) 143(1.1) 178(2.6) 205(2.8) 218(3.4) 219(2.1) 220(100.0) 221 (14.7) 326(25.0)	13.5
2e	42(1.0) 43(1.2) 57(1.3) 91(1.0) 105(1.1) 106 (2.0) 128(1.0) 130(1.3) 142(1.1) 143(1.0) 156 (2.2) 157(4.8) 158(1.1) 170(1.2) 172(100.0) 173(11.0) 278(24.6)	14.8
2f	57(1.2) 105(1.1) 106(2.1) 128(1.0) 130(1.0) 144(1.3) 145(15.0) 173(2.7) 188(100.0) 189 (10.0) 294(25.6)	14.9
2g	42(1.3) 43(2.1) 57(1.1) 58(1.0) 77(1.5) 105 (1.0) 128(2.1) 170(6.4) 198(100.0) 199(12.0) 304(28.1)	15.0



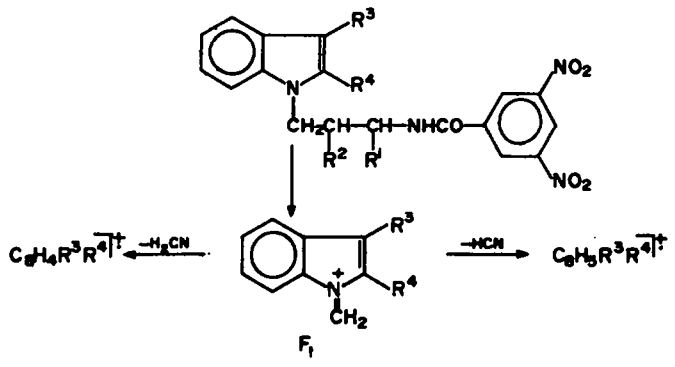
In order to demonstrate generality of the pattern observed for series 1 and 2 (increase of stabilities of molecular ions with strength of the parent CTC) we have also studied compounds 3. The latter species should have certain advantages over those already discussed. With the dinitrobenzoyl acceptor (3) instead of the pyridine one (1 and 2), intramolecular CTC should have higher stabilities. The substituent effects should also increase, as indole derivatives are known[16] to participate in the formation of CTC mostly through the C_2–C_3 bond, while transfer of substituent effects across the indole bond system (from the benzene to pyrrole ring) should considerably weaken their action.[17]

As expected, the W_M values have been found to increase with electron donor strength of the 2- and 3-substituents along the series: 3f < 3e < 3a < 3d < 3c < 3b (Table 5). Compounds 3b and 3c have higher W_M values compared with 3d, though the $-(CH_2)_4-$ group is known to be a weaker donor than two methyl groups. That, however, is easy to explain, for cyclic systems are more stable to electron impact than open chains.[18]

On the whole, the W_M values for compounds 3 are higher than those characterizing compounds 1. This provides yet another evidence of direct relationship between stabilities of CTC and respective molecular ions. In the absence of CT interactions, molecular ions 3

Table 5. Mass-spectra of the compounds 3

Compound	m/e (intensity, % of maximal ion)	W_M
3a	45(11.9) 75(11.0) 77(6.9) 78(14.7) 86(6.3) 103(6.1) 130(20.9) 131(11.3) 143(6.6) 144 (72.5) 145(37.0) 158(10.5) 171(9.5) 195(7.8) 212(8.3) 382(100.0)	19.3
3b	75(5.1) 143(9.4) 156(9.0) 157(11.1) 167(5.9) 168(8.9) 170(19.0) 171(5.9) 183(5.4) 184(30.3) 185(9.8) 211(5.4) 422(100.0)	29.5
3c	75(5.9) 143(9.2) 156(9.7) 167(5.1) 168(8.2) 170(12.8) 171(5.9) 182(5.1) 184(84.8) 185(21.6) 195(5.0) 218(5.6) 419(5.3) 436(100.0)	27.1
3d	75(1.2) 77(2.1) 78(1.3) 115(1.0) 130(1.5) 143 (6.4) 144(9.9) 145(6.9) 158(100.0) 159(21.2) 410(90.0)	22.5
3e	55(8.3) 75(14.3) 77(14.1) 105(37.4) 115(6.6) 122(15.2) 143(6.7) 144(42.9) 145(6.0) 149(7.8) 156(4.0) 157(8.3) 158(17.8) 166(6.7) 171(98.3) 172(11.0) 174(5.2) 184(15.9) 188(22.6) 195 (20.9) 198(4.3) 203(7.1) 216(12.2) 217(13.4) 218(3.1) 220(4.3) 230(9.3) 266(7.2) 405(6.4) 423(9.8) 468(100.0) 469(25.3)	16.7
3f	45(30.6) 75(12.8) 77(12.3) 105(30.3) 122(17.1) 129(2.8) 143(12.0) 149(8.0) 166(6.8) 170(8.6) 184(4.8) 188(30.9) 189(2.6) 195(17.5) 198(9.8) 201(6.5) 202(8.9) 214(9.7) 215(5.5) 216(11.7) 228(100.0) 229(13.4) 233(5.7) 241(9.2) 246 (12.8) 247(27.5) 252(4.6) 260(2.8) 393(6.8) 453(5.5) 484(70.8) 485(17.5)	13.2



should have lower W_M values, because the number of fragmentation pathways increases considerably on going from 1 to 3. In fact, substitution of the dinitrobenzamide grouping for the pyridine one, elongation of the alkyl chain by one CH_2 unit, and in some cases branching of these chains would provide additional possibilities for fragmentation.

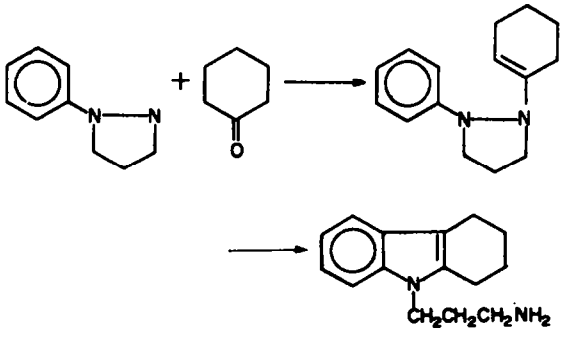
There is nothing specific about fragmentation mechanisms of complexes 3 that can be considered as evidence for or against the formation of intramolecular CTC. All spectra are dominated by ions F_1 formed in the "β-cleavage" of the aliphatic C–C bond. Dinitrobenzoyl ions (*m/e* 195) and other fragment ions have very low intensities, whereas the *summary intensity* of molecular ions and ions F_1 range from 40 to 60%.

To sum up, the results obtained for three series of molecular autocomplexes of the indole family show that stability of molecular ions toward electron impact increases with electron donor power of the electron excess moiety (that is, with strength of CTC).

EXPERIMENTAL

Preparations. The compounds of series 1 and 2 were synthesized by interaction of tetrahydro-γ-carbolines or respective indoles with 2-methyl-5-vinylpyridine.[3]

The compounds 3 were obtained by acylation of N-(aminoalkyl)indoles with 3,5-dinitrobenzoyl chloride. The preparation of N-(aminoalkyl)indoles was described earlier.[4-6] The reaction involves a Fisher-like rearrangement of 1-aryl-2-vinylpyrazolidines formed in the condensation of 1-arylpyrazolidines[7-10] with ketones.



Later, Eberle *et al.*[11,12] reported a similar synthesis of N-(aminoalkyl)indoles.

The mass spectra of compounds 1 were measured on an MX-1303 instrument equipped with a modified direct inlet system and operated at: ionization energy 35 eV, emission current 150 mA, accelerating voltage 2 kV, temp. 100–120°. The mass spectra of 2 and 3 were obtained on a Jeol JMS-01-SG-2 spectrometer at ionization energy 75 eV and temp. of 100–120°. The proton NMR spectra were recorded with Varian T-60 and XL-100 instruments. The UV spectra were obtained on a Cary-15 spectrophotometer.

Expansion of the observed spectra into Gauss' components was performed using a mini-computer "Electronica EKVM TZ-16" by the first derivative method. The parameters of the CT band were determined from the linear section of the $d \ln \varepsilon/d\nu = f(\nu)$ curve by the parabolic regression method.

A part of this work has been presented on The III[rd] All-Union Conference on Charge Transfer Complexes and Ion-Radical Salts (8 III Vsesoyuznoe Soveshchanie po Kompleksam s Perenosom Zaryada i Ion-Radikal'nym Solyam) (Riga)[19] and The First Moscow Conference on Organic Chemistry and Technology (Pervoe moscowskoe Soveshechanie po organicheskoi Khimii i Tekhnologii).[20]

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