B. A mixture of 0.20 g (1 mmole) of salt V and 0.13 g (1 mmole) of dimethyl sulfate in 25 ml of acetonitrile was boiled for 2 h, the solvent was then distilled off, and the residue was treated according to method A. The yield of compound VI was 0.015 g (8%), and of compound VII – 0.045 g (24%).

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AMINOMETHYLATION OF 1-R-TETRAZOLES

V. P. Karavai and P. N. Gaponik

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The reaction of tetrazole and a series of its 1-substituted derivatives with formaldehyde and alkylamines under mild conditions results in aminomethylation of the heterocyclic ring at the carbon atom with the formation of the corresponding Mannich bases. It was found that strong acids (HCl and CF_3COOH) have an activating action during the process. An ylide mechanism of the reaction was proposed from the data obtained.

Tetrazole and its 1H-derivatives have recently become relatively readily available compounds [1, 2] and, therefore, they are of interest as starting materials for the synthesis of various functionally substituted tetrazoles, which are used in medicine, biology, agriculture, and technology [3]. Very promising for this purpose are the reactions at the cyclic carbon atom, which have practically not been investigated until now [3-5], since it had been assumed that the electrophilic substitution of this hydrogen atom is impeded because of the deactivation of this position by annular nitrogen atoms [6].

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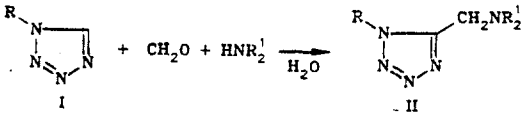
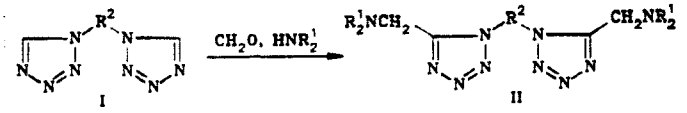
We have recently shown that under mild conditions 1-methyltetrazole enters the aminomethylation reaction as a CH-acid component [4]. It appeared to be expedient to examine this reaction in detail and explore the possibility of extending it to a wide range of 1-substituted tetrazoles in order to develop a general preparative method for the preparation of tetrazoles of the Mannich base type, which are difficultly available by other methods [7].



I, II a R=H, b R=CH_3, c R=C_2H_5, d R=CH_2CH_2OH, e R=$CH_2CH_2N_3$, f R=$CH{=}CH_2$, g R=$CH_2CH{=}CH_2$, h R=C_6H_5, i R=*p*-$NO_2C_6H_4$, j R=*p*-BrC_6H_4, k R=*m*-ClC_6H_4, l R=*p*-$CH_3OC_6H_4$, a-l $R^1=CH_3$; II m $R=R^1=C_2H_5$, n R=CH_3, $R_2^1=(-CH_2-)_5$

It was found that the optimal conditions for carrying out the reaction and for obtaining a good yield of the aminomethylation products, despite the use of derivatives of a single series and the same type of reaction centers, are determined to a considerable extent by the nature of the substituent in the tetrazole ring. Thus, alkyltetrazoles Ib, c are readily aminomethylated on heating in a neutral or a weakly acid aqueous solution with hydrochlorides of secondary alkylamines and a two- to threefold excess of formalin. The yield of the Mannich bases is 60-80%. The functionally substituted alkyltetrazoles Id-g, n, o, and also alkyl derivatives Ih-j, p, q require the removal of the reaction water for the process to proceed. Thus, it is highly recommended to apply azeotropic distillation using a mixture of aromatic hydrocarbons, such as benzene and nitrobenzene as the reaction medium. In some cases, in particular at a low solubility of the starting tetrazole Ip, q, the addition of acetic acid is effective. And, although the reaction generally proceeds under heterogeneous conditions, the yield of the end compounds reaches 70-80%. The Mannich bases of vinyl- and allyltetrazoles If, g, the multiple bonds of which are not affected during aminomethylation can be obtained under similar conditions in a yield of more than 75%. The unsubstituted tetrazole Ia is also unexpectedly aminomethylated readily and in high yield at the heterocyclic carbon atom. The occurrence of the reaction at the N–H bond is probably impeded, as in the case of 5-phenyltetrazole [8], by the high acidity of the compound (pK_a 4.89 [9]).

The reaction of binuclear derivatives – ethylenebis- and phenylenebis-1-H-tetrazoles (Io, q) proceeds simultaneously at two tetrazole groups with the formation of bis-Mannich bases IIo-q:



I, II $R^1=CH_3$, o $R^2=CH_2CH_2$, p $R^2=p$-C_6H_4, q $R^2=m$-C_6H_4

We did not succeed in obtaining the monosubstitution products by varying the ratios of the reagents and the synthesis conditions.

The unusualness of the aminomethylation reaction in the case of tetrazole and its derivatives consists in the entry of the electrophile into a strongly passivated π-deficient heterocyclic ring. The process possibly proceeds by an ylide mechanism, which makes it possible to explain the introduction of the substituent into the least-active position [10], and begins with attack by an electrophile, in this case the immonium cation III, on the most nucleophilic ring $N_{(4)}$ atom with the formation of a tetrazolium cation IV. As a result, the positive charge on the carbon atom further increases [11], resulting in a considerable increase of the mobility of the hydrogen atom at the 5-position of the heterocyclic ring [12]. After the elimination of a proton from the intermediate IV, the ylide V formed becomes stabilized due to interaction with electrophile II (or an intramolecular transfer of the aminoalkyl) with the formation of tetrazole II substituted at the carbon atom (see below).

The proposed mechanism of the aminomethylation process is confirmed by the reactions characteristic for tetrazolium salts that we have observed. Thus, for example, in a D_2O medium the exchange of the heterocyclic proton of 1-methyltetrazole (Ib) by deuterium in the presence of formaldehyde and dimethylamine hydrochloride is practically fully completed in 10-15 min at 90-100°C, while the addition of these reagents separately under the same conditions does not lead to a change in the integral intensity of the $C_{(5)}H$ signal in the PMR spectrum. It was also found that during the aminomethylation of 1-aryltetrazoles, particularly with electron acceptor substituents, evolution of nitrogen is observed. For example, in the reaction of nitro- and bromophenyltetrazoles Ii, j, the yield of nitrogen is 38-45%, while the Mannich bases are formed in a yield of less than 50%. Moreover, the corresponding para-substituted phenylcyanamides VIi, j were isolated from the reaction mixture and

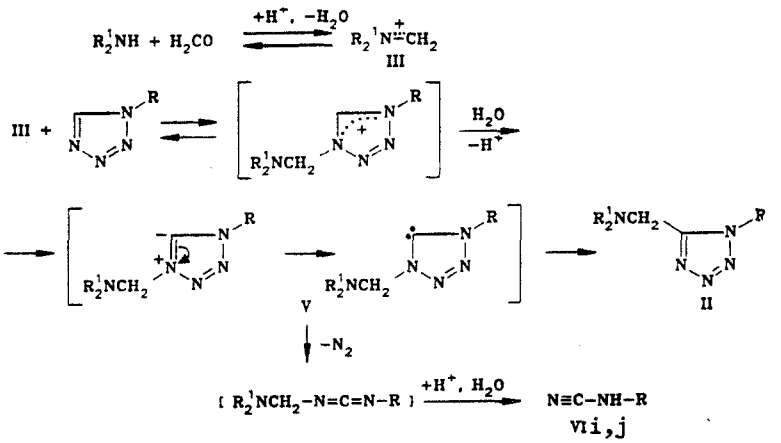
TABLE 1. Characteristics of Synthesized Compounds IIa-q

Compound	Empirical formula	Mp, °C* [Bp, °C, mm Hg]	PMR spectrum, δ, ppm ($CDCl_3$)			Yield, %		
			R^1	$>N-CH_2$	R	A	B	C
IIa	$C_4H_9N_5$	236...237 (dec.)	2,65**	4,32	—		78	
IIa'	$C_{10}H_{12}N_8O_7$	182...183	2,83**	4,65	8,58 (2H, s, arom. H)			
IIb	$C_5H_{11}N_5$	32...33 [113...115 (1,3)]	2,32; 2,08**	3,82 3,73	4,18 (3H, s, CH_3); 4,03 (3H, s, CH_3)	68	62	96
IIc	$C_6H_{13}N_5$	[107...109 (0,9)]	2,34	3,89	1,65 (3H, t, CH_3); 4,66 (2H, q, CH_2)	62		
IId	$C_6H_{13}N_5O$	Oil (150...151)***	2,31	3,80	4,58 (2H, t, CH_2OH); 4,86 (2H, t, N^1-CH_2); 5,89 (1H, br.s, OH)		73	
IIe	$C_6H_{12}N_8$	Oil (158...159)***	2,24	3,76	3,67 (2H, t, CH_2N_3); 4,61 (2H, t, N^1-CH_2)		70	
IIf	$C_6H_{11}N_5$	[83...85 (0,2)]	2,26	3,79	5,34; 6,14; 7,41 (3×1H, d.d, $CH=CH_2$, $J_1=15,6$; $J_2=9,0$; $J_3=1,2$ Hz)		76	
IIg	$C_7H_{13}N_5$	[95...97 (0,5)]	2,26	3,74	5,12...5,37 (4H, m, $-CH_2-$, $=CH_2$), 5,72...6,24 (1H, m; $=CH-$, $J_1=16,4$; $J_2=10,5$; $J_3=6,4$ Hz)		80	
IIh	$C_{10}H_{13}N_5$	47...48	2,38	3,73	7,50...7,80 (5H, m, Ph)		78	91
IIi	$C_{10}H_{12}N_6O_2$	150...152	2,35	3,74	8,07 (2H, d, $H^{2'}$); 8,45 (2H, d, $H^{3'}$)		36	89
IIj	$C_{10}H_{12}BrN_5$	97...98	2,30	3,65	7,58...7,80 (4H, m, C_6H_4)		46	
IIk	$C_{10}H_{12}ClN_5$	65...66	2,35	3,70	7,50...7,98 (4H, m, C_6H_4)			88
IIl	$C_{11}H_{15}N_5O$	50...51	2,31	3,65	3,89 (3H, s, OCH_3); 7,06 (2H, d, $H^{3'}$); 7,64 (2H, d, $H^{2'}$)			81
IIm	$C_8H_{17}N_5$	[118...120 (1,0)]	1,02 (6H, t, $2CH_3$); 2,49 (4H, q, $2CH_2$)	3,80	1,47 (3H, t, CH_3); 4,48 (2H, q, CH_2)	78		
IIn	$C_8H_{15}N_5$	40...42	1,40...1,60 (6H, m, $3CH_2$); 2,30...2,50 (4H, m, $2CH_2$)	3,83	4,18 (3H, s, CH_3)		76	86
IIo	$C_{10}H_{20}N_{10}$	150...151	2,21	3,54	5,06 (4H, s, C_2H_4)		72	
IIp	$C_{14}H_{20}N_{10}$	187...188	2,27	3,63	7,97 (4H, s, C_6H_4)		69	78
IIq	$C_{14}H_{20}N_{10}$	137...138	2,24	3,64	7,75...8,49 (4H, m, C_6H_4)		96	84

*Compounds IIa, a', i, o were recrystallized from ethanol, IIh, l, n from a 1:1 hexane–ether mixture, IIj, k, p, q from 2-propanol; n_D^{20}: IIc 1.4618, IIf 1.4801, IIg 1.4913, IIm 1.4662.

**In DMSO-D_6.

***Bases IId, e were analyzed in the form of iodomethylates; the melting points of iodomethylates are given (from ethanol).



identified. A similar cleavage with the elimination of the nitrogen molecule is a characteristic reaction of 1,4-disubstituted tetrazolium salts [12, 13].

Since tetrazoles undergo quaternization in acid media [11], it was expedient also to study the effect of preliminary protonation on the aminomethylation process, although for normal Mannich reactions the use of strong acids as solvents is not characteristic [14]. It was found that the reaction of tetrazoles I with formaldehyde and alkylamines in a hydrochloric (15-25%) or trifluoroacetic (85-100%) acids makes it possible to increase the yield of the desired end compounds substantially, reaching 90-95%. In this case, the mechanism of the process is possibly not different from that discussed above, except for the formation of the tetrazolium cation. The yield of the aminomethyl derivatives II increases as a result of the suppression of side reactions of the decomposition of the tetrazolium cations in acid media [12, 13]. The results obtained agree well with the occurrence of a maximum of the rate of deutero-exchange in 1-methyl-5-D-tetrazole in the H_0 values region of –1 to –2 [15].

It is convenient to use hydrochloric acid for the aminomethylation of water-soluble alkyltetrazoles, while for 1-aryltetrazoles better results are obtained when the reaction is carried out in CF_3COOH. In the latter case, the dependence of the reaction rate on the nature of the substituent in the phenyl ring is observed. Thus, while nitro-, bromo-, and phenylbistetrazoles Ii, j, q react practically completely in 1.5-2 h (yield ~90%), phenyltetrazole Ih requires 4-5 h, and anisyltetrazole Il, must be heated for 10-12 h to reach a yield of 80%. These data correlate with the results of the investigation of the deutero-exchange kinetics in 1-aryl-4-ethyltetrazolium salts in acid media [13]. The acceleration of the reaction when electron-acceptor groups are introduced is probably due to the possibility of additional stabilization of ylide V, while the negative charge is delocalized.

The presence of a qualitative correlation between the rate of aminomethylation of 1-aryltetrazoles and the electronic properties of the substituents, the rapid deutero-exchange of $C_{(5)}H$ in 1-alkyltetrazoles by the action of a formaldehyde–dimethylammonium chloride mixture and taking into account the data on the correlation of basicity (pK_{BH^+}) with the σ^H constants of the substituents and having a negative value of σ [11] together indicate that the limiting stage of the process is the splitting off of a proton from the tetrazolium cation IV.

The composition and structure of the compounds IIa-q obtained were established by IR and PMR spectroscopic methods and by elemental analysis. In the IR spectra of the aminomethyl derivatives, together with the characteristic absorption bands of substituents R, there are bands of the stretching-deformational vibrations of the tetrazole ring in the 980-1110 cm^{-1} region [3]. Instead of the signal of the $C_{(5)}H$ bond of the initial 1-substituted tetrazoles (3080-3150 cm^{-1}), a series of bands appears in the 2700-2970 cm^{-1} region corresponding to the stretching vibrations of the methylene and alkylamine groups.

The PMR spectra of the synthesized compounds IIb-q contain signals of alkylamino groups in the 1.00-2.50 ppm region and a singlet of methylene protons at 3.54-3.89 ppm. Proton signals of the functional groups at the cyclic nitrogen atom are also observed (Table 1). The exception is tetrazole IIa unsubstituted at the nitrogen atom, which is a weak-field shift (~0.6 ppm) of the proton signals of the dimethylaminomethyl fragment in the PMR spectrum. Comparison with the spectrum of the picrate of this compound IIa' under the same conditions indicates quaternization of the amino group by an acid N–H proton of the heterocyclic ring. This is also indicated by the presence of absorption in the 2200-2700 cm^{-1} range in the IR spectrum of compound IIa, which corresponds to the vibrations of the quaternary nitrogen atom [16]. The unusually high (for this series) melting point and the decreased solubility of tetrazole IIa in organic solvents are in agreement with the spectroscopic data.

All the tetrazoles IIa-q form hydrochlorides, which are generally very hygroscopic, and stable, well-crystallizable picrates, which can be used for the isolation and identification of the obtained compounds. Alkylating agents readily quaternize the synthesized bases at the amine nitrogen atom and form the corresponding ammonium salts in a practically quantitative yield.

EXPERIMENTAL

The IR spectra were obtained on a Specord IR-75 spectrophotometer in a thin layer and in KBr tablets. The PMR spectra were recorded on a Brucker WM-360 spectrometer (360 MHz) in $CDCl_3$ and DMSO-D_6, using HMDS as internal standard. The purity of the compounds obtained was verified by TLC on Silufol UV-254 plates with development of iodine vapors.

The elemental analysis data for C, H, and N correspond to the calculated values.

Tetrazoles Ia-e, g-q were obtained from the corresponding amines or their salts by the method described in [2]. 1-Vinyltetrazole If was synthesized by the reaction of tetrazole with vinyl acetate in the presence of mercury acetate [17].

Aminomethylation of 1-R-Tetrazoles. A. A mixture of 0.05 mole of tetrazole Ib, c, m, 0.05 mole of a dialkylamine hydrochloride, and 10 ml of a 37% formalin in 30 ml of water was boiled for 5 h. The solution was evaporated under vacuum, and the residue was crystallized from ethanol to obtain the hydrochloride. To isolate the base, 30 ml of a 20% solution of sodium hydroxide was added to the residue after evaporation of the reaction mixture, the organic layer was separated, the aqueous layer was extracted with ether (3 × 30 ml), and the extract was dried over potassium carbonate. After removal of the solvent, the base was distilled under vacuum to give compounds IIb, c, m.

B. A mixture of 0.02 mole of tetrazole (Ia, b, d-j, n), 0.02 mole of an amine hydrochloride, 0.9 g of paraform, and 2-3 drops of concentrated HCl was boiled in 20 ml of a 1:1 benzene–nitrobenzene mixture using an adapter for the azeotropic distillation of water. After 1.5-2 h the reaction mixture was cooled, the solvent was decanted, the residue was washed with ether, and 15 ml of a 20% NaOH solution was added. The amine that separated out was extracted with ether (3 × 20 ml), dried over potassium carbonate, and the solvent was distilled off. Bases IIb, f, g were distilled under vacuum, compounds IIh-j, n were recrystallized from a suitable solvent, while for identification amines IId, e were converted into the corresponding iodomethylates. To isolate the base IIa, the residue after decantation was dissolved in 50 ml of isopropanol, a solution of 0.8 g of NaOH in 6 ml of water was added, the mixture was brought to boiling and filtered hot. Tetrazole IIa crystallized from the solution on cooling.

In the reaction of binuclear tetrazoles IIo-q, double amounts of the reagents were used, and the synthesis of the bis-bases IIp, q was carried out in a 1:1 benzene–acetic acid mixture. After treatment with an alkali, the amines IIo-q were filtered and recrystallized.

Acidification of the alkaline solutions remaining after the extraction of bases IIi, j, gave arylcyanamides VIi, mp 182-184°C and VIj, mp 112-113°C (2-propanol–water, 1:1); according to the data in [18], mp 180 and 112°C, respectively.

C. A mixture of 0.02 mole (in the case of binuclear derivatives 0.01 mole) of a 1-substituted tetrazole, 0.02 mole of an amine hydrochloride and 1.5 g of paraform was boiled for 3 h in 20 ml of 20% HCl (Ib, m, n) or in 25 ml of CF_3COOH for 2 h (Ii-k, p, q) for 4.5 h (Ie) or for 11 h (Im). Finally, the solution was evaporated in vacuo and treated as described in methods **A** and **B**.

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VIBRATIONAL SPECTRA OF THE HYDRAZIDES OF (TETRAZOL-1-YL)- AND (TETRAZOL-2-YL)ACETIC ACIDS AND THEIR DEUTERATED ANALOGS

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The hydrazides of (tetrazol-1-yl)- and (tetrazol-2-yl)acetic acids and their deuterated analogs are investigated by means of IR spectroscopy. On the basis of the data obtained, assignments in the vibrational spectra of the isomers are made.

Establishing the mode of coordination of potentially polydentate, organic ligands, among which are the isomers of the hydrazides of tetrazolylacetic acids, is one of the major problems in coordination chemistry. The basic method for solving this problem is usually IR spectroscopy, but its application requires accurate knowledge of the positions of the vibrational bands of the fragments taking part in coordination.

Since calculations of the frequency and shape of the normal vibrations of various tetrazole derivatives [1-3], as well as of compounds containing the hydrazide fragment [4-6], have been described, the problem of assigning the bands in the IR spectra of the isomers of the hydrazides of tetrazolylacetic acids reduces to subtracting from the spectra the bands due to vibrations of the heterocyclic, hydrazide, and methylene groups. To help in this, it is necessary to investigate the vibrational spectra of deuterated analogs of the compounds to be studied.

The isomeric hydrazides of (tetrazol-1-yl)- and (tetrazol-2-yl)acetic acids (I and II) were obtained by the hydrazinolysis of the corresponding ethyl esters III and IV. In the PMR spectra, the proton signal from the tetrazole ring of isomer I was shifted to a weaker field compared to the signal in the spectrum of compound II. This is analogous to what was observed with the initial esters III and IV [7].

In the high-frequency region of the IR spectra of isomers I and II, three groups of bands are observed that arise from the stretching vibrations of the N–H bonds in the hydrazide group (3327-3223 cm^{-1}), the C–H bond in the tetrazole ring (3170 cm^{-1}), and the CH_2 group (3067-3037 cm^{-1}) (Fig. 1). On deuteration, the first group of bands is shifted in the spectra of compounds I-d_3 and I-d_4 to the 2530-2400 cm^{-1} region.

Deuteration of the hydrazide group makes it easy to subtract the eight vibrational bands of the tetrazole ring (R_1-R_8) from the spectrum of compound I. Their positions in the spectra of compounds I, I-d_3, and I-d_4 are virtually unchanged. Inasmuch as these vibrations preserve their relative identity with respect to frequency [1], the assignment of these bands is done. The assignment of the stretching vibrations of the ring are: two at 1483 and 1437 cm^{-1} (R_1 and R_2) arising basically from the stretching vibrations of the C=N and N=N bonds, and a band at 1257 cm^{-1} (R_3) arising from the vibrations of the C–N and N–N bonds. The ring breathing vibrations appear at 1176 cm^{-1} (R_4), then come three strong bands, 1108, 1032, and 972 cm^{-1}

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