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Dedicated to Full Member of the Russian Academy of Sciences B.A. Trofimov on the 65th Anniversary of His Birth

Tetrazoles: XLVI.* 3*H*-1,3,4-Benzo- and Pyrido[6,7-*b*][1,3,4]triazepines from 5-Aryltetrazoles. Synthesis and Chemical Properties

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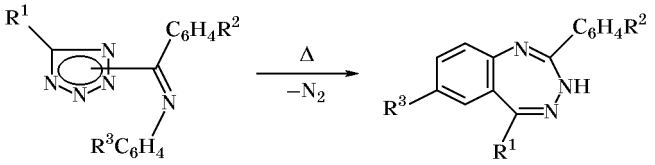
Abstract—2,5-Disubstituted 3*H*-1,3,4-benzotriazepines were synthesized by heating of *N*-imidoyltetrazoles in toluene at 110°C. Alkylation of these compounds, as well as of 3*H*-pyrido[6,7-*b*][1,3,4]triazepines with alkyl halides in THF in the presence of potassium *tert*-butoxide gave the corresponding 1-alkyl derivatives in good yield and with high regioselectivity.

Despite a keen interest in 3*H*-1,3,4-benzotriazepines, methods of preparation and chemical properties of these compounds have been studied insufficiently [2, 3]. Among known methods for building up a triazepine ring, the most accessible is likely to be thermal transformation of *N*-imidoyltetrazoles [4–8]. This procedure is general and is applicable to the synthesis of triazepine systems fused with both benzene and pyridine ring [9]. While continuing studies in this line, we have synthesized a series of hitherto unknown 3*H*-1,3,4-benzotriazepines and examined alkylation of these compounds and also of structurally related 3*H*-pyrido[6,7-*b*][1,3,4]triazepines.

3*H*-1,3,4-Benzotriazepines were prepared by the procedure reported by us previously [4]. Initial *N*-imidoyltetrazoles were subjected to thermolysis in toluene at 110°C, and the corresponding products **Ia**–**Ie** were obtained in 47–72% yield (Scheme 1).

Scheme 1.

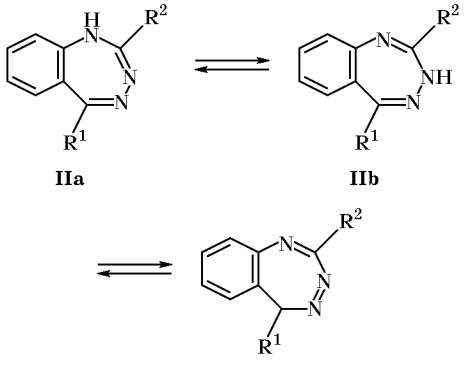


Ia–Ie

R^1 = Ph, R^2 = H, R^3 = OEt (**a**); R^1 = 4-$MeOC_6H_4$, R^2 = R^3 = H (**b**); R^1 = Ph, R^2 = 4-Br, R^3 = Cl (**c**); R^1 = 4-BrC_6H_4, R^2 = H, R^3 = Me (**d**); R^1 = 2-pyridyl, R^2 = R^3 = H (**e**).

Alkylation of 1,3,4-triazepines may be regarded as an efficient way of their functionalization; however, only a few published data on this reaction are available [2, 3, 10]. Obviously, successful research in this line requires consideration of such problems as tautomerism of 1,3,4-triazepines and dual reactivity of heteroanions derived therefrom by deprotonation. Theoretically, 1,3,4-triazepines can exist as tautomers **IIa**–**IIc** (Scheme 2).

Scheme 2.



IIa **IIb**

IIc

Tautomeric properties of 1,3,4-triazepines almost were not studied. According to crystallographic data

* For communication XLV, see [1].

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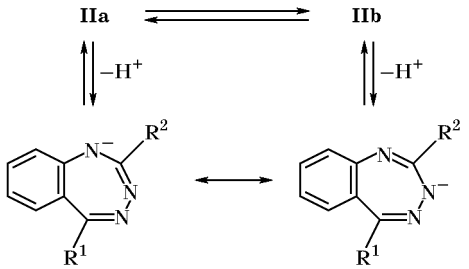
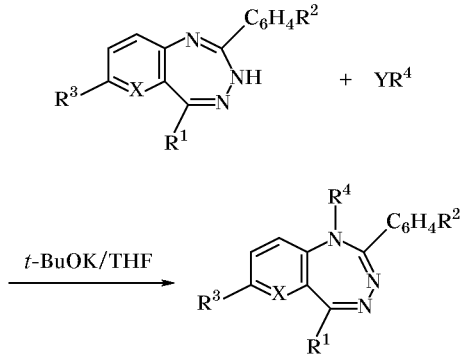
for some 2,5-disubstituted 1,3,4-triazepines, these compounds in crystal exist as 3*H*-tautomers **IIb** [11, 12]. Fusco *et al.* [13] noted that 4,5-dihydro-1,3,4-triazepines in solution have 1*H*-structure **IIa**. Some indirect data suggest that the tautomeric equilibrium **IIa** ⇄ **IIc** is displaced toward 1*H*- and 3*H*-tautomers, for no C^5-substituted 1,3,4-triazepines were synthesized by direct alkylation or arylation. Deprotonation of **IIa** and **IIb** gives the corresponding anions which may be represented as shown in Scheme 3.

Scheme 3.



Such anions could react with alkylating agents to afford mixtures of N^1- and N^3-substituted isomers. Factors affecting the regioselectivity of these reactions have not been discussed so far. Taking into account the lack of information on the alkylation of 1,3,4-triazepines, there were some difficulties while choosing the optimal conditions. For example, our numerous attempts to effect alkylation of 1,3,4-triazepines with alkyl iodides and dimethyl sulfate in the presence of sodium hydroxide or organic bases (such as triethylamine and pyridine), were unsuccessful. We also failed to accomplish the alkylation under conditions of phase-transfer catalysis in the system methylene chloride (or chloroform)–aqueous sodium hydroxide in the presence of tetrabutylammonium bromide. A probable reason is that 1,3,4-triazepines are very weak NH acids [2] which could not be deprotonated by the action of sodium hydroxide and organic bases. In fact, by successive treatment of 3*H*-1,3,4-benzo- and pyrido[6,7-*b*][1,3,4]triazepines with such a strong base as potassium *tert*-butoxide and alkyl halides in tetrahydrofuran we succeeded in obtaining *N*-alkyltriazepines **IIIa**–**IIIj** in high yields (Scheme 4).

Scheme 4.

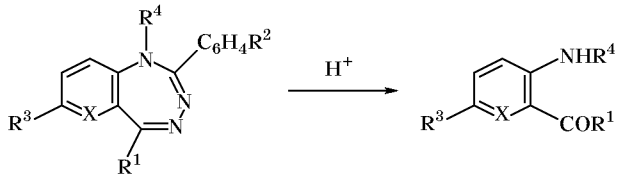


IIIa–**IIIj**

III, X = CH; R^1 = Ph, R^2 = R^3 = H, R^4 = Me (**a**); R^1 = Ph, R^2 = R = H, R^4 = Et (**b**); R^1 = Ph, R^2 = R^3 = H, R^4 = Bu (**c**); R^1 = Ph, R^2 = 4-Br, R^3 = H, R^4 = Me (**d**); R^1 = Ph, R^2 = 4-NO_2, R^3 = H, R^4 = Me (**e**); R^1 = Ph, R^2 = 4-Br, R^3 = Cl, R^4 = Me (**f**); R^1 = 4-pyridyl, R^2 = R^3 = H, R^4 = Me (**g**); X = N; R^1 = Ph, R^2 = R^3 = H, R^4 = Me (**h**); R^1 = Ph, R^2 = 4-Me, R^3 = H, R^4 = Me (**i**); R^1 = Ph, R^2 = R^3 = H, R^4 = Et (**j**); Y = Hlg.

The alkylation occurred with high regioselectivity. According to the NMR data, in all cases only the corresponding 1-alkyl derivatives **IIIa**–**IIIj** were formed. Their structure was proved by the transformation of some products into the corresponding amino ketones **IV** (Scheme 5).

Scheme 5.



IIIb, IIId, IIIe, IIIh **IV**

+ $R^2C_6H_4COOH$

Thus we have shown that neither the nature of alkylating agent nor substituent structure in the substrate affect the direction of alkylation of ambident triazepine anions. Because of the lack of any data on the electronic structure of such anions, it is difficult to advance a rigorous explanation for the observed reaction pattern. Presumably, the N^1 atom in the triazpine anion possesses the largest negative charge which determines the site of preferential electrophilic attack by alkylating agent.

EXPERIMENTAL

The IR spectra were obtained on a UR-20 spectrometer in KBr. The ^{1}H NMR spectra were recorded on a Bruker AC-200 instrument. The purity of the products was checked by TLC on Silufol UV-254 plates using carbon tetrachloride–ethyl acetate (3:2) as eluent.

7-Ethoxy-2,5-diphenyl-3*H*-1,3,4-benzotriazepine (Ia). A solution of 0.01 mol *N*-(*p*-ethoxyphenyl)-benzimidoyl chloride in 10 ml of chloroform was added dropwise over a period of 30 min to a mixture of 0.01 mol of 5-phenyltetrazole, 0.001 mol of tetrabutylammonium bromide, 10 ml of a 10% aqueous solution of sodium hydroxide, and 30 ml of chloroform under stirring at 20°C. The mixture was stirred for 4 h at 20°C, and the organic phase was separated, washed with a 1% aqueous solution of NaOH and with water (2 × 10 ml), and dried over magnesium sulfate. The solvent was removed under reduced pressure, 20 ml of toluene was added to the residue, the mixture was heated for 3 h at 110°C, and the solvent was distilled off under reduced pressure. Yield 2.29 g (70%), mp 243–245°C (from acetonitrile). IR spectrum, ν, cm^{-1}: 905, 925, 940, 950, 960, 995, 1005, 1035, 1050, 1080, 1095, 1115, 1165, 1185, 1215, 1245, 1265, 1295, 1325, 1400, 1415, 1450, 1460, 1480, 1500, 1510, 1565, 1570, 1585, 1595, 1630, 2865, 2895, 2910, 2935, 2945, 2990, 3035, 3070, 3300. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.31 t (3H, CH_3), 3.91 q (2H, CH_2), 6.45–7.96 m (13H, H_{arom}, and 1H, NH). Found, %: C 77.38; H 5.61; N 12.30. $C_{22}H_{19}N_3O$. Calculated, %: C 77.40; H 5.61; N 12.31.

5-(4-Methoxyphenyl)-2-phenyl-3*H*-1,3,4-benzotriazepine (Ib). Yield 47%, mp 238–239°C (from butyl acetate). IR spectrum, ν, cm^{-1}: 920, 935, 950, 965, 985, 1010, 1040, 1080, 1090, 1115, 1175, 1185, 1235, 1270, 1285, 1300, 1320, 1330, 1420, 1455, 1510, 1520, 1555, 1580, 1600, 1615, 1685, 2840, 2860, 2905, 2940, 2965, 3010, 3070, 3085, 3335. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.81 s (3H, OCH_3), 6.9–8.2 m (13H, H_{arom}), 9.2 s (1H, NH). Found, %: C 77.05; H 5.23; N 12.72. $C_{21}H_{17}N_3O$. Calculated, %: C 77.04; H 5.23; N 12.84.

2-(4-Bromophenyl)-7-chloro-5-phenyl-3*H*-1,3,4-benzotriazepine (Ic). Yield 72%, mp 224–225°C (from DMF–ethanol, 1 : 1). IR spectrum, ν, cm^{-1}: 900, 935, 945, 990, 1015, 1035, 1080, 1105, 1130, 1175, 1190, 1225, 1240, 1260, 1270, 1285, 1300, 1325, 1390, 1400, 1445, 1495, 1550, 1560, 1590, 1615, 2865, 2935, 3040, 3065, 3315. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.8–8.3 m (12H, H_{arom}). Found, %: C 58.45; H 3.15; N 10.25. $C_{20}H_{13}BrClN_3$. Calculated, %: C 58.49; H 3.19; Br 19.46; Cl 8.63; N 10.23.

5-(4-Bromophenyl)-7-methyl-2-phenyl-3*H*-1,3,4-benzotriazepine (Id). Yield 65%, mp 252–253°C (from DMF–2-propanol, 1 : 1). IR spectrum, ν, cm^{-1}: 905, 925, 940, 955, 975, 990, 1015, 1035, 1075, 1090, 1115, 1135, 1160, 1185, 1225, 1235, 1330, 1365, 1395, 1435, 1450, 1490, 1505, 1545, 1565, 1570, 1595, 1625, 2865, 2935, 3040, 3070, 3345. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.25 s (3H, CH_3), 7.05–7.95 m (12H, H_{arom}), 8.1 s (1H, NH). Found, %: C 64.63; H 4.22; N 10.90. $C_{21}H_{16}BrN_3$. Calculated, %: C 64.63; H 4.13; Br 20.47; N 10.77.

5-(2-Pyridyl)-2-phenyl-3*H*-1,3,4-benzotriazepine (Ie). Yield 61%, mp 248–249°C (from acetonitrile). IR spectrum, ν, cm^{-1}: 905, 920, 935, 955, 965, 990, 1000, 1005, 1035, 1050, 1080, 1095, 1120, 1165, 1185, 1230, 1250, 1290, 1300, 1305, 1335, 1415, 1435, 1460, 1475, 1500, 1565, 1570, 1600, 1615, 2865, 2935, 3070, 3345. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.02–8.57 m (13H, H_{arom}, 1H, NH). Found, %: C 76.50; H 4.73; N 18.76. $C_{19}H_{14}N_4$. Calculated, %: C 76.49; H 4.73; N 18.78.

1-Methyl-2,5-diphenyl-1*H*-1,3,4-benzotriazepine (IIIa). Potassium *tert*-butoxide, 2 mmol, was added to a solution of 1.7 mmol of 2,5-diphenyl-3*H*-1,3,4-benzotriazepine in 30 ml of anhydrous tetrahydrofuran. The mixture was stirred for 30 min at 20°C, 2.5 mmol of methyl iodide was added, and the mixture was stirred for 2 h at 20°C and diluted with 150 ml of water. The precipitate was filtered off. Yield 0.42 g (80%), mp 173–175°C (from aqueous DMF). IR spectrum, ν, cm^{-1}: 930, 945, 965, 975, 990, 1005, 1025, 1045, 1080, 1090, 1130, 1160, 1170, 1195, 1270, 1280, 1300, 1320, 1325, 1355, 1425, 1450, 1470, 1490, 1520, 1550, 1565, 1590, 1605, 1645, 2820, 2865, 2890, 2930, 2965, 3005, 3030, 3070. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.06 s (3H, CH_3), 7.09–7.42 m (14H, H_{arom}). Found, %: C 81.18; H 5.7; N 13.48. $C_{21}H_{17}N_3$. Calculated, %: C 81.03; H 5.47; N 13.50.

1-Ethyl-2,5-diphenyl-1*H*-1,3,4-benzotriazepine (IIIb). Yield 57%, mp 204–206°C (from aqueous DMF). IR spectrum, ν, cm^{-1}: 925, 950, 965, 975, 990, 1005, 1030, 1055, 1080, 1100, 1105, 1135, 1160, 1180, 1250, 1285, 1325, 1365, 1385, 1410, 1445, 1475, 1495, 1545, 1565, 1590, 1605, 1640, 2860, 2895, 2935, 2945, 2990, 3040, 3075. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.05 t (3H, CH_3), 3.35 m (1H, NCH_2), 3.70 m (1H, NCH_2), 7.32–7.62 m (14H, H_{arom}). Found, %: C 81.13; H 5.98; N 12.98. $C_{22}H_{19}N_3$. Calculated, %: C 81.23; H 5.85; N 12.92.

1-Butyl-2,5-diphenyl-1*H*-1,3,4-benzotriazepine (IIIc). Yield 48%, mp 77–80°C (from petroleum ether). IR spectrum, ν, cm^{-1}: 925, 950, 975, 990, 1005, 1030, 1070, 1085, 1110, 1140, 1175, 1230, 1260, 1280, 1305, 1330, 1350, 1375, 1450, 1470, 1490, 1545, 1595, 1605, 2885, 2940, 2965, 3045,

3070. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 0,74 t (3H, CH_3), 1.2–1.5 m (4H, CH_2CH_2), 3.23 m (1H, NCH_2), 3.7 m (1H, NCH_2), 7.05–7.6 m (14H, H_{arom}). Found, %: C 81.62; H 6.55; N 11.93. $C_{24}H_{23}N_3$. Calculated, %: C 81.59; H 6.51; N 11.90.

2-(4-Bromophenyl)-1-methyl-5-phenyl-1*H*-1,3,4-benzotriazepine (IIId). Yield 91%, mp 204–205°C (from aqueous DMF). IR spectrum, ν, cm^{-1}: 925, 945, 960, 975, 990, 1010, 1045, 1075, 1095, 1115, 1135, 1165, 1175, 1200, 1275, 1285, 1330, 1395, 1430, 1450, 1475, 1490, 1545, 1595, 1605, 1645, 2835, 2930, 2960, 3040, 3070. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.3 s (3H, CH_3), 7.05–7.6 m (13H, H_{arom}). Found, %: C 64.79; H 4.09; N 10.77. $C_{21}H_{16}BrN_3$. Calculated, %: C 64.63; H 4.13; Br 20.47; N 10.77.

1-Methyl-2-(4-nitrophenyl)-5-phenyl-1*H*-1,3,4-benzotriazepine (IIIe). Yield 59%, mp 222–224°C (from aqueous DMF). IR spectrum, ν, cm^{-1}: 920, 950, 980, 990, 1015, 1050, 1085, 1095, 1115, 1135, 1160, 1175, 1205, 1225, 1275, 1325, 1340, 1355, 1410, 1450, 1485, 1520, 1545, 1555, 1595, 1605, 1645, 2840, 2860, 2935, 2985, 3035, 3070. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.07 s (3H, CH_3), 7.07–8.3 m (13H, H_{arom}). Found, %: C 70.79; H 4.37; N 15.91. $C_{21}H_{16}N_4O_2$. Calculated, %: C 70.78; H 4.49; N 15.72.

2-(4-Bromophenyl)-7-chloro-1-methyl-5-phenyl-1*H*-1,3,4-benzotriazepine (IIIf). Yield 75%, mp 186–187°C (from ethyl acetate). IR spectrum, ν, cm^{-1}: 950, 990, 1020, 1040, 1060, 1100, 1180, 1200, 1280, 1320, 1400, 1470, 1490, 1570, 1590, 2820, 2880, 2890. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.03 s (3H, CH_3), 7.47–7.69 m (12H, H_{arom}). Found, %: C 59.42; H 3.66; N 9.93. $C_{21}H_{15}BrClN_3$. Calculated, %: C 59.36; H 3.55; Br 18.84; Cl 8.36; N 9.89.

1-Methyl-2-phenyl-5-(4-pyridyl)-1*H*-1,3,4-benzotriazepine (IIIg). Yield 30%, mp 190–193°C (from aqueous DMF). IR spectrum, ν, cm^{-1}: 920, 955, 975, 990, 1000, 1030, 1050, 1070, 1095, 1130, 1140, 1155, 1175, 1205, 1220, 1285, 1325, 1335, 1385, 1415, 1450, 1465, 1490, 1550, 1575, 1590, 1600, 1690, 2845, 2940, 2985, 3035, 3075. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.05 s (3H, CH_3), 7.07–8.7 m (13H, H_{arom}). Found, %: C 76.95; H 5.17; N 17.87. $C_{20}H_{16}N_4$. Calculated, %: C 76.92; H 5.13; N 17.94.

1-Methyl-2,5-diphenylpyrido[6,7-*b*][1,3,4]triazepine (IIIh). The product was purified by column chromatography on silica gel using CCl_4–ethyl acetate (3:2) as eluent. Yield 63%, mp 198–200°C. IR spectrum, ν, cm^{-1}: 925, 950, 975, 990, 1005, 1025, 1045, 1060, 1085, 1095, 1140, 1145, 1165, 1180, 1195, 1240, 1265, 1285, 1315, 1330, 1440, 1450, 1475, 1495, 1550, 1560, 1575, 1595, 2835, 2870, 2930, 2960, 3005, 3035, 3075. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 3.15 s (3H, CH_3), 7.39–8.45 m (13H, H_{arom}). Found, %: C 76.90; H 5.15; N 17.92. $C_{20}H_{16}N_4$. Calculated, %: C 76.92; H 5.13; N 17.95.

1-Methyl-5-phenyl-2-(4-tolyl)pyrido[6,7-*b*]-[1,3,4]triazepine (IIIi). The product was purified by column chromatography on silica gel using CCl_4–ethyl acetate (3:2) as eluent. Yield 31%, mp 159–161°C. IR spectrum, ν, cm^{-1}: 915, 940, 955, 990, 1025, 1045, 1065, 1095, 1125, 1145, 1180, 1195, 1220, 1240, 1265, 1290, 1300, 1330, 1385, 1445, 1475, 1495, 1520, 1550, 1570, 1595, 1615, 2865, 2940, 2980, 3010, 3035, 3070, 3100. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 2.38 s (3H, CH_3), 3.14 s (3H, NCH_3), 7.26–8.44 m (12H, H_{arom}). Found, %: C 77.27; H 5.62; N 17.28. $C_{21}H_{18}N_4$. Calculated, %: C 77.3; H 5.5; N 17.2.

1-Ethyl-2,5-diphenylpyrido[6,7-*b*][1,3,4]triazepine (IIIj). The product was purified by column chromatography on silica gel using CCl_4–ethyl acetate (3:2) as eluent. Yield 40%, mp 158–160°C. IR spectrum, ν, cm^{-1}: 930, 960, 990, 1005, 1030, 1050, 1065, 1080, 1095, 1110, 1150, 1175, 1235, 1255, 1280, 1335, 1375, 1390, 1450, 1500, 1545, 1555, 1575, 1595, 2865, 2910, 2940, 2990, 3045, 3075. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.09 t (3H, CH_3), 3.4 m (1H, CH_2), 3.83 m (1H, CH_2), 7.37–8.46 m (13H, H_{arom}). Found, %: C 77.26; H 5.55; N 17.15. $C_{21}H_{18}N_4$. Calculated, %: C 77.28; H 5.56; N 17.17.

Acid hydrolysis of 1-methyl-2,5-diphenylpyrido-[6,7-*b*][1,3,4]triazepine (IIIh). A mixture of 1.2 mmol of triazepine **IIIh** and 10 ml of 17% hydrochloric acid was heated for 2 h at 100°C. The mixture was cooled to 5°C, and the precipitate of benzoic acid, 0.058 g (40%), mp 122°C, was filtered off. The filtrate was made alkaline (pH 10–12) by adding 10% aqueous sodium hydroxide, and the precipitate was filtered off, washed with 10 ml of water, and dried in air to obtain 0.204 g (80%) of compound **IV**, mp 93–94°C (from hexane). IR spectrum, ν, cm^{-1}: 920, 945, 980, 1000, 1035, 1050, 1070, 1110, 1135, 1155, 1175, 1235, 1255, 1300, 1320, 1345, 1355, 1390, 1435, 1450, 1465, 1480, 1515, 1565, 1585, 1605, 1635, 2835, 2875, 2930, 3070, 3360. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.95 d (3H, CH_3), 7.22–8.02 m (8H, H_{arom}, 1H, NH). Found, %: C 73.55; H 5.65; N 13.12. $C_{13}H_{12}N_2O$. Calculated, %: C 73.58; H 5.66; N 13.20.

Acid hydrolysis of triazepines **IIIb**, **IIId**, and **IIIe** was performed in a similar way. The properties of the resulting 2-ethylaminobenzophenone and 2-methylaminobenzophenone were in agreement with published data [14].

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