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NORBORNENE-CONTAINING MANNICH BASES ON THE BASIS OF ALIPHATIC AMINES**G.E.Hajiyeva¹, E.H.Mammadbayli¹, S.I.Ibrahimli², I.A.Jafarov³**¹*Yu.Mamedaliyev Institute of Petrochemical Processes, NAS of Azerbaijan*²*Ganja State University*³*Azerbaijan State Pedagogical University**gulsum.mete@mail.ru*

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The novel norbornene-containing Mannich bases have been synthesized on the basis of norbornenylmethanol, aliphatic amines and formaldehyde. The physico-chemical properties of obtained products have been determined. The structure and composition of synthesized compounds have been proved by the methods, of elemental analysis, IR spectroscopy, NMR¹H and ¹³C.

Keywords: dicyclopentadiene, norbornenylmethanol, secondary amines, diethylamine, dipropylamine, dibutylamine, dipentylamine, dihexylamine, Mannich bases.

Introduction

Functionally substituted organic compounds containing also heteroatoms, such as sulfur and nitrogen, are used as additives to improve the quality of oils and fuels, including those used in the form of effective biologically active substances, medical preparations, pesticides etc. [1, 2]. Depending on the nature and structure of the heteroatom, the biological activity and reactivity of these compounds varies.

Among the biologically active compounds of a number of norbornene, their amine-containing derivatives are distinguished, the value of which is associated with the presence of two pharmacophore fragments – the nitrogen-containing group [3] and the carbon framework related to natural terpenes [4] – in their molecules. Since biologically active compounds, which combine several pharmacophore fragments, have more effective actions, the researchers try to expand the assortment of such compounds and study their physiological active properties [5].

The structure of bicyclo[2.2.1]heptane (norbornane) underlies many important natural compounds such as borneol, camphor etc. The norbornene fragment, like the norbornane and adamantane structures, form the basis of many amines that are drug substances. For amines with norbornan scaffolds, antiviral activity is characteristic [6].

The above-mentioned examples of the use

of nitrogen-containing compounds with the norbornan fragment do not completely reflect the areas of their practical application, it stimulates research on elucidating the features of the course of the compounds involved in this reaction. It is important for researchers to synthesize new generations of such compounds on the basis of available raw materials and to find more advantageous methods for their production [7–9]. One of the convenient and effective methods for obtaining nitrogen-containing organic compounds is the Mannich reaction [10, 11].

Discussion of obtained results

In this paper properties of previously unknown aminomethoxy derivatives of norbornene obtained by the one-pot Mannich reaction of norbornenylmethanol (III) with aliphatic amines (V–IX) are considered. In the preparation of the starting norbornenylmethanol dicyclopentadiene, isolated from the C₅ side fraction of the liquid pyrolysis products, is obtained from the EP-300 unit in Sumgayit city.

On the basis of dicyclopentadiene and allyl alcohol (II), norbornenylmethanol (III) was obtained. The reaction was carried out in an autoclave [12] at a reagent ratio of 1: 2.4 respectively, at a temperature of 170–180°C and a pressure of 4–5 atm, for 9 hours. At high temperature and pressure dicyclopentadiene monomerizes to cyclopentadiene, and as a result, the reaction proceeds according to scheme:

The yield of the obtained alcohol (III) is 74%.

After synthesizing amino-methoxy derivatives of norbornenyl methanol (X–XIV), aminomethoxy derivatives of norbornenyl methanol (III), formaldehyde (IV) and secondary amines [diethylamine (V), dipropylamine (VI), dibutylamine (VII), dipentylamine (VIII), dihexylamine (IX)]. The yield of the target products is 43–71%. Reactions proceed according to the scheme:

where R = C₂H₅ (V, X), C₃H₇ (VI, XI), C₄H₉ (VII, XII), C₅H₁₁ (VIII, XIII), C₆H₁₃ (IX, XIV).

It can be assumed that an N-oxymethyl derivative is formed intermediately:

After the initial attack of the carbon atom of the carbonyl group by the electron pair of the nitrogen atom, which is not separated by a pair of electrons, protonation and splitting of water follows, resulting in the formation of a cation. Attacking the positively charged carbon atom of

said cation with a carbanion formed from 5-(norborn-2-enyl)methanol (III) leads to the formation of Mannich bases (X–XIV).

The reactions were carried out in a solution of benzene at 78–80°C for 4–5 h with an equimolar ratio of the reagents. Compounds (X–XIV) are liquids with a characteristic odor, insoluble in water, readily soluble in organic solvents (ethanol, acetone, benzene, CCl₄, CHCl₃ etc.). The composition and structure of the obtained compounds (III, X–XIV) were confirmed by elemental analysis data, IR spectroscopy, ¹H NMR and ¹³C. In the IR spectra of 5-(norborn-2-enyl)methanol (III), a broad absorption band is observed in the 3500 cm^{−1} region characteristic of the hydroxyl group [13], and in the IR spectra of the aminomethoxy derivatives norborn-5-ene (X–XIV) this absorption band is absent. Absorption bands in the 1288–1100 cm^{−1} field are characteristic for stretching vibrations of the C–N bonds (X–XIV). In addition, absorption bands were observed in the regions 2857–2853 cm^{−1}, which are characteristic for the C–H (ν_{C–H}) bond of the CH₃ and CH₂ groups respectively. Valence vibrations of the C–O bond (ν_{C–O}) appear in the field of 1100–1048 cm^{−1} in the form of an intense absorption band. The ¹H and ¹³C NMR spectra of synthesized compounds confirm their indicated structure. The IR spectrum of compound (XII), NMR ¹H spectrum of compound (X) and ¹³C spectrum of compound (XI) are cited in Figure 1–3.

Experimental

The IR spectra of synthesized compounds were taken with the apparatus "SPEKTRUM BX" and "BRUKER" from ALPHA IR FURYE (Germany) in the range 4000–400 cm^{−1}. The ¹H and ¹³C NMR spectra were recorded on a BRUKER AM-300 spectrometer, at a frequency of 300 MHz in a C₆D₆ solvent, the internal standard was hexamethyldisiloxane.

The purity of the reaction products was determined by boiling point, elemental analysis and gas-liquid chromatography.

Elemental analysis was carried out using a "CARLOERBA" device – EA 1108.

Fig. 1. ^1H NMR spectrum of 5-(N,N-Diethylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (X).

Fig. 2. ^{13}C NMR spectrum of 5-(N,N-dipropylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (XI).

Fig. 3. IR spectrum of 5-(N,N-Dibutylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (XII).

GLC analysis was performed on a chromatograph LXM-8 MD, steel column (300×3 mm) with 5% PEGS (polyethylene glycol succinate) on Dichrome P, carrier gas – helium (40 cm³/min), detector cathrometer, column temperature 150⁰C, evaporator – 230⁰C. Refractive index (n_D^{20}) – on a refractometer of the brand "ABBEMAT" 350/500, density (ρ_4^{20}) – on the instrument "DMA" 4500 M.

The starting allyl alcohol was reactive. Before use, it was dried over MgSO₄ and distilled. On the basis of allyl alcohol and cyclopentadiene 5-(norborn-2-enyl)methanol (III) was prepared by the procedure of [12], which is the starting reagent in the further synthesis of norbornenyl-containing Mannich bases.

Diethylamine (V), dipropylamine (VI), dibutylamine (VII), dipentylamine (VIII) and dihexylamine (IX) there were used reactive. Before use, all five aliphatic secondary amines were distilled and determined their physico-chemical constants, which coincide with the literature data.

Ammoniac water, which is a medical preparation, was used as a 10% solution. Benzene was used as the solvent, which was purified and dried by the known method [14].

Paraformaldehyde is a product of formaldehyde polymerization, consisting of 8–100 monomers. When heated, it is depolymerized to formaldehyde. It was used as a reactive powder.

5-(Norborn-2-enyl)methanol (III) was prepared in an autoclave by the procedure of [12] for 9 hours at a temperature of 170–180⁰C, a pressure of 4–5 atm with a ratio of dicyclopentadiene and allylic alcohol (II) of 1:2.4 respectively. The yield of the desired product was 74%. The boiling point is 82–83⁰C (10 mm Hg), n_D^{20} 1.4970, ρ_4^{20} 1.027 g/cm³. IR spectrum, ν , cm⁻¹: 3500 (OH), 2880–2830 (C–H, CH₂), 1200–1100 (C–N). NMR ¹H, δ , ppm: 0.45 d.d (1H, C⁶H_{exo}, $J=4.1$ Hz), 1.19 t (1H, C⁷H_a, $J=7.9$ Hz), 1.44 t (1H, C⁶H_{endo}, $J=4.1$ Hz), 1.68 m (1H, C⁷H_s, $J=7.9$ Hz), 2.86 q (1H, C⁵H), 3.21–3.36 t (1H, C⁴H), 3.36–3.46 d.d (2H, C⁸H₂), 3.46 t (H, C¹H), 3.92 bp.s (H, OH), 6.00–6.08 m (2H, C²H, C³H). NMR ¹³C, δ , ppm: 28.83

(C⁶), 41.76 (C⁵), 42.26 (C¹), 43.60 (C⁴), 49.41 (C⁷), 65.76 (C⁸), 132.32 (C³), 137.78 (C²).

Aminomethoxy derivatives of norborn-5-ene (X–XIV). General methodology. To 0.2 moles of paraform (IV) diluted in 20 ml of benzene, 0.1 mole of norbornenyl methanol (III) diluted in 20 ml of benzene was added dropwise with stirring. Then, while stirring, a solution of 0.1 moles of secondary amine (V–IX) in 20 ml of benzene was added dropwise. Stirring was continued at 78–80⁰C for 4–5 hours. After cooling, the mixture was treated with an ammonia solution (10%). Washed with distilled water until neutral and dried over MgSO₄. The benzene was distilled off, the residue was distilled in a vacuum.

5-(N,N-Diethylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (X) was prepared from 12.4 g (0.1 mole) of norbornenylmethanol (III), 6 g (0.2 mole) of paraform (IV) and 14.6 g (0.2 mole) of diethylamine (V). Yield – 10.6 g (51%), b.p. – 102–104⁰C (4 mm Hg), n_D^{20} – 1.4672, ρ_4^{20} – 0.9395 g/cm³. IR spectrum, ν , cm⁻¹: 3057.66, 978.83, 932.86, 816.75 (C–H, CH=CH); 2961.78, 2858.75, 1460.29, 1359.48 (δ C–H, CH, CH₂, CH₃); 1629.96 (C=C); 1215.33 (C–N); 1057.02 (C–O–C); 715.65 (C–H, CH₂). NMR ¹H, δ , ppm: 1.042 t (6H, C^{13,15}H₃, $J=7.5$ Hz), 1.265–1.373 m (2H, C⁶H₂), 1.787 d.d.d (2H, C⁷H₂, $J=2.2, 2.1, 1.2$ Hz), 2.685–2.927 m (3H, C^{1,4,5}H), 3.295–3.424 m (4H, C^{12,14}H₂N), 3.424 d (2H, C⁸H₂O, $J=6.8$ Hz), 4.170 s (2H, C¹⁰H₂), 5.934–5.951 m (1H, C²H=), 6.105–6.116 m (1H, C³H=). NMR ¹³C, δ , ppm: 12.85 (C^{13,15}), 29.01 (C⁶), 38.87 (C⁵), 42.06 (C¹), 43.86 (C⁴), 45.11 (C⁷), 49.03 (C^{12,14}), 71.07 (C⁸), 84.14 (C¹⁰), 132.36 (C²), 136.64 (C³). Found, %: C 74.06, H 10.86, N 6.61. C₁₃H₂₃NO. Calculated, %: C 74.64, H 11.00, N 6.70.

5-(N,N-dipropylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (XI) was prepared from 10 g (0.08 mole) of norbornenylmethanol (III), 5 g (0.16 mole) of paraform (IV) and 8 g (0.08 mole) of dipropylamine (VI). Yield – 8.05 g (43%), b.p. – 119–122⁰C (4 mm Hg), n_D^{20} – 1.4670, ρ_4^{20} – 0.9228 g/cm³. IR spectrum, ν , cm⁻¹: 3059.09,

983.40, 962.53, 938.20, 903.37 37 (δ C–H, CH=CH); 2957.87, 2933.86, 2868.20, 1463.87, 1364.11, 1343.50 (C–H, CH, CH₂, CH₃); 1685.15 (C=C); 1202.44 (C–N); 1091.97, 1068.70, 1048.05 (C–O–C); 717.83 (mathematical oscillation C–H, CH₂). NMR ¹H, δ , ppm: 0.885–0.911 t (6H, C^{14,17}H₃), 1.268–1.465 m (7H, C^{13,16}H₂, C⁵H, C⁶H₂), 1.792 m (2H, C⁷H₂), 2.575 d.d (1H, C¹H, $J=0.1$, 1.2 Hz), 2.773 d.d (1H, C⁴H $J=0.1$, 1.2 Hz), 2.930–3.115 m (6H, C^{8,12,15}H₂), 4.078 s (2H, C¹⁰H₂), 5.942–6.116 m (2H, C^{2,3}H=). NMR ¹³C, δ , ppm: 11.30 (C^{14,17}), 21.16 (C^{13,16}), 29.01 (C⁶), 38.88 (C⁵), 42.06 (C¹), 43.87 (C⁴), 49.04 (C⁷), 53.73 (C^{12,15}), 71.11 (C⁸), 85.23 (C¹⁰), 132.37 (C²), 136.63 (C³). Found, %: C 75.22, H 11.96, N 5.17. C₁₅H₂₇NO. Calculated, %: C 75.95, H 11.39, N 5.91.

5-(N,N-Dibutylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (XII) was prepared from 12.4 g (0.1 mole) of norbornenylmethanol (III), 6 g (0.2 mole) of paraform (IV) and 12.9 g (0.1 mole) of dibutylamine (VII). Yield – 13 g (49%), b.p. – 135°C (2 mm Hg), n_D^{20} – 1.4660, ρ_4^{20} – 0.9100 g/cm³. IR spectrum, ν , cm^{–1}: 3059.11, 984.47, 960.39, 933.86, 904.21 (C–H, CH=CH); 2955.89, 2931.32, 2862.49, 2803.96, 1459.26, 1364.32, 1343.73 (C–H, CH₂, CH₃); 1685.27 (C=C); 1272.85, 1251.53, 1190.57 (C–O–C); 1070.08, 1048.83 (δ C–N); 717.92 (mathematical oscillation C–H, CH₂). NMR ¹H, δ , ppm: 0.922 t (6H, C^{15,19}H₃, $J=7.1$ Hz), 1.347–1.505 m (7H, C^{6,14,18}H₂, C⁵H), 1.791 d.d.d (2H, C⁷H₂, $J=2.3$, 2.1, 1.2 Hz), 2.474 d.d (1H, C¹H, $J=0.1$, 1.2 Hz), 2.636 d.d (1H, C⁴H, $J=0.1$, 1.2 Hz), 2.772–2.930 m (4H, C^{13,17}H₂), 3.001–3.117 m (6H, C^{8,12,16}H₂), 4.076 s (1H, C¹⁰H), 4.094 s (1H, C¹⁰H), 5.936–5.945 m (1H, C²H=), 6.108–6.118 m (1H, C³H=). NMR ¹³C, δ , ppm: 13.56 (C^{15,19}), 20.31 (C^{14,18}), 29.01 (C⁶), 30.27 (C^{13,17}), 38.88 (C⁵), 42.07 (C¹), 43.87 (C⁴), 49.05 (C⁷), 51.41 (C^{12,16}), 71.12 (C⁸), 85.11 (C¹⁰), 132.37 (C²), 136.64 (C³). Found, %: C 76.60, H 10.64, N 5.96. C₁₇H₃₁NO. Calculated, %: C 75.47, H 10.93, N 5.22.

5-(N,N-Dipentylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (XIII) was prepared from 12.4 g (0.1 mole) of norbornenylmethanol (III), 6 g (0.2 mole) of paraform (IV) and 15.7 g

(0.1 mole) of dipentylamine (VIII). Yield – 21 g (71%), b.p. – 185°C (13 mm Hg), n_D^{20} – 1.4680, ρ_4^{20} – 0.9057 g/cm³.

IR spectrum, ν , cm^{–1}: 3059.14, 984.35, 962.72, 936.78, 903.88 (C–H, CH=CH); 2956.51, 2931.00, 2862.51, 1462.17, 1376.94, 1343.69 (C–H, CH₂, CH₃); 1686.00 (C=C); 1252.93, 1224.92, 1192.25 (C–O–C); 1069.86, 1050.78 (δ C–N); 718.01 (mathematical oscillation C–H, CH₂). NMR ¹H, δ , ppm: 0.81–0.95 t (6H, C^{16,21}H₃, $J=7.3$ Hz), 1.00–1.60 m (12H, C^{13-15,18-20}H₂), 1.33–1.52 m (5H, C⁶H₂, C^{1,4,5}H), 1.79 d.d.d (2H, C⁷H₂, $J=2.2$, 2.1, 1.2 Hz), 3.0–3.34 m (6H, C⁸H₂O, C^{12,17}H₂N), 4.08 s (1H, OC¹⁰H₂N), 4.09 s (1H, OC¹⁰H₂N), 5.94–5.95 m (1H, C²H=), 6.11–6.12 m (1H, C³H=). NMR ¹³C, δ , ppm: 10.89 (C¹⁶), 13.63 (C²¹), 17.16 (C¹³), 22.47 (C²⁰), 27.24 (C¹⁴), 27.76 (C¹⁹), 33.16 (C¹³), 38.88 (C¹⁸), 42.06 (C⁶), 43.87 (C⁵), 49.05 (C¹), 51.69 (C⁴), 52.27 (C⁷), 58.65 (C¹⁷), 59.55 (C¹²), 71.23 (C⁸), 85.51 (C¹⁰), 132.37 (C²), 136.65 (C³). Found, %: C 77.36, H 12.27, N 4.03. C₁₉H₃₅NO. Calculated, %: C 77.82, H 11.95, N 4.78.

5-(N,N-Dihexylaminomethoxymethyl)bicyclo[2.2.1]hept-2-ene (XIV) was prepared from 12.4 g (0.1 mole) of norbornenylmethanol (III), 6 g (0.2 mole) of paraform (IV) and 15.7 g (0.1 mole) of dihexylamine (IX). Yield – 20.4 g (64%), b.p. – 192–194°C (6 mm Hg), n_D^{20} – 1.4645, ρ_4^{20} – 0.8854 g/cm³.

IR spectrum, ν , cm^{–1}: 3058.20, 987.49, 839.70 (C–H, CH=CH); 2927.88, 2857.86, 1459.83, 1361.95 (C–H, CH₂, CH₃); 1635.05 (C=C); 1221.39, 1172.05, 1063.83 (δ C–N); 1172.05 (C–O–C); 717.75 (mathematical oscil-

lation C–H, CH₂). NMR ¹H, δ, ppm: 0.91 t (6H, C^{17,23}H₃, *J*=7.1 Hz), 1.32–1.48 m (16H, C^{13-16,19-22}H₂), 1.79 d.d.d (2H, C⁷H₂, *J*=2.2, 2.1, 1.2 Hz), 2.47 d.d (1H, C¹H, *J*=0.1, 1.2 Hz), 2.63 d.d (1H, C⁴H, *J*=0.1, 1.2 Hz), 3.0–3.12 m (6H, C^{8,12,18}H₂), 4.08 s (1H, C¹⁰H₂), 4.09 s (1H, C¹⁰H₂), 5.94–6.11 m (2H, C^{2,3}H). NMR ¹³C, δ, ppm: 13.57 (C^{17,23}), 22.56 (C^{16,22}), 26.97 (C^{15,21}), 27.21 (C^{14,20}), 28.03 (C^{13,19}), 31.75 (C⁶), 38.89 (C⁵), 42.07 (C¹), 43.88 (C⁴), 49.04 (C⁷), 51.71 (C¹²), 51.76 (C¹⁸), 71.12 (C⁸), 85.16 (C¹⁰), 132.37 (C²), 136.63 (C³). Found, %: C 77.98, H 12.41, N 4.04. C₂₁H₃₉NO. Calculated, %: C 78.50, H 12.15, N 4.36.

Conclusion

Thus, previously not known norbornene-containing bases of Mannich based on norbornenylmethanol and aliphatic secondary amines and formaldehyde have been synthesized. Optimum conditions for obtaining the target products are found. The reaction was carried out at an equimolar ratio of the initial reagents at a temperature of 78–80°C in the presence of benzene for 4–5 hours, while the yield was 43–71%. Obtained liquid compounds with a characteristic odor, insoluble in water, readily soluble in organic solvents (ethanol, acetone, benzene, CCl₄, CHCl₃ etc.). The physicochemical constants of the synthesized compounds are determined. The structure of the compounds obtained is confirmed by the methods of elemental analysis, IR spectroscopy, NMR ¹H and ¹³C.

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ALİFATİK AMİNLƏR ƏSASINDA NORBORNEN TƏRKİBLİ MANNİX ƏSASLARI

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Norbornenilmetanol, alifatik aminlər və formaldehid əsasında yeni norbornen tərkibli Mannix əsasları sintez edilmişdir. Alınan maddələrin fiziki-kimyəvi xassələri öyrənilmişdir. Sintez olunmuş maddələrin tərkib və quruluşu element analizi, İQ, ¹H və ¹³C NMR spektroskopiyaya üsulları ilə təsdiq edilmişdir.

Açar sözlər: ditsiklopentadien, norbornenilmetanol, ikili aminlər, dietilamin, dipropilamin, dibutilamin, dipentilamin, diheksilamin, Mannix əsasları.

НОРБОРНЕНСОДЕРЖАЩИЕ ОСНОВАНИЯ МАННИХА НА ОСНОВЕ АЛИФАТИЧЕСКИХ АМИНОВ**Г.Э.Гаджиева, Э.Г.Мамедбейли, С.И.Ибрагимли, И.А.Джафаров**

На основе норборненилметанола, алифатических аминов и формальдегида синтезированы новые норборненсо-
держащие основания Манниха. Определены физико-химические характеристики целевых продуктов. Состав и
строение синтезированных соединений подтверждены методами элементного анализа, ИК-спектроскопии, ЯМР
 ^1H и ^{13}C .

Ключевые слова: дициклопентадиен, норборненилметанол, вторичные амины, диэтиламин, дипропиламин,
дибутиламин, дипентиламин, дигексиламин, основания Манниха.