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Organic Chemistry

Synthesis of some Derivatives of N-(1-Adamantyl)Carbonyl-N'-Benzyliden-*o*-Phenylendiamine

Shota Samsoniya[*], Davit Zurabishvili[**], Tinatin Bukia[**], Giorgi Buzaladze[**], Medea Lomidze[**], Elizbar Elizbarashvili[§], Uli Kazmaier[§§]

[*] *Academy Member, Department of Chemistry, I.Javakhishvili Tbilisi State University, Tbilisi*
[**] *Faculty of Exact and Natural Sciences, Scientific-Research Institute of Organic Chemistry, Division of Alicyclic compounds, I.Javakhishvili Tbilisi State University, Tbilisi*
[§] *Faculty of Chemical Technology and Metallurgy, Technical University of Georgia, Tbilisi*
[§§] *Institute of Organic Chemistry, University of Saarland, Saarbrucken, Germany*

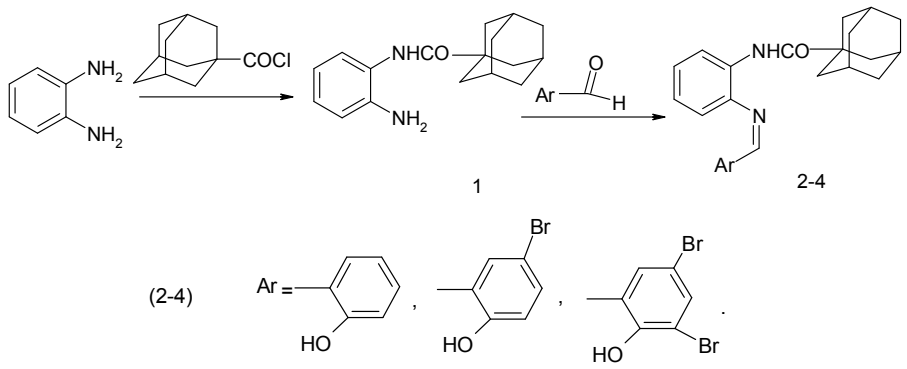
ABSTRACT. N-(1-adamantyl)carbonyl-*o*-phenylenediamine and 4-methoxy-2-aminophenyl-N-(1-adamantyl)carboxamide are synthesized. Condensation reaction of the synthesized compounds with aldehydes of salicyl-, 5-bromo salicyl- and 3,5-dibromo salicylic is studied. As a result N-(1-adamantyl)carbonyl-N'-(2-hydroxybenzylidene)-, N-(1-adamantyl)carbonyl-N'-(2-hydroxy-5-bromobenzylidene)-, N-(1-adamantyl)carbonyl-N'-(2-hydroxy-3,5-dibromo benzylidene)-, 4-methoxy-N-(1-adamantyl)carbonyl-N'-(2-hydroxybenzylidene)-, 4-methoxy- N-(1-adamantyl)carbonyl-N'-(2-hydroxy-5-bromo benzylidene)-, 4-methoxy- N-(1-adamantyl)carbonyl-N'-(2-hydroxy-3,5-dibromobenzylidene)-*o*-phenylenediamines are obtained. Structures of the compounds are confirmed by IR, UV and NMR spectra.

***Key words**: adamantane, adamantoyllation, carboxamides, arylidenimines, o-phenylendiamines.*

Organic compounds with adamantane fragment possess antiviral, antibacterial, anticancer, anticataleptic, immunotropic, neuro-psychotropic and other properties. They amplify energy of the human body and significantly improve emotional and physical state in patients [1-7].

Introduction of adamantane fragment into the molecule of a preparation changes or partly increases its biological activity, in most of the cases reduces toxicity, which can be explained by spatial structure of the compound, hydrophobia and lipophilicity. Also suitable conditions of transportation into the biological membranes, prolongation effect of preparations, high immunotropicity and others can be considered [1-3, 7].

Considering high biological activities of N-(1-adamantyl)carboxamides and arylidenimines [1,4,7], we aimed to synthesize new derivatives of *o*-phenylenediamine in the molecule with adamantane-1-carboxamide (Ad–CONH) and arylidenimine (Ar–CH=N) groups.



Scheme 1. Derivatives of the N-(1-adamantyl)carbonyl-N'-benzylidene-*o*-phenylendiamines.

N-(1-adamantyl)carbonyl-N'-benzylidene-*o*-phenylendiamines were obtained according to Scheme 1.

N-(1-adamantyl)carbonyl-*o*-phenylenediamine (1) at first was obtained by Sasaki and coauthors [8] acting with adamantane-1-carbonylchloride on the *o*-phenylenediamine at the existence of triethylamine in the absolute ether medium. The adamantoyllation reaction of *o*-phenylenediamine was carried out by the method of Schotten-Baumann [9]. By condensation of aminoamide (1) with salicyl-, 5-bromo salicyl- and 3,5-dibromo salicyl aldehydes for 2-5 hr heating in the alcohol area N-(1-adamantyl)carbonyl- N'-benzylidene-*o*-phenylendiamines (2-4) were obtained with 68-89% yield.

Derivatives of 4-methoxy-N-(1-adamantyl)carbonyl-N'-benzylidene-*o*-phenylendiamines were obtained according to Scheme 2.

Experimental part

The reaction and monitoring the purity of compounds were carried out on the plate „Silicagel on TLC PET-foils". IR spectra were recorded with devices: Thermo Nicolet Avatar 370, Varian 660-IR and FT-IR in Nujol, hexachlorobutadiene and KBr. UV spectrum was recorded with the spectrophotometer " Specord Agilent 8453 UV/VIS; ^{1}H and ^{13}C nuclear magnetic resonance spectra – with the spectrometer Bruker at 400 and 100 Hz, respectively. Melting point was determined by Boetius apparatus PHMKO5.

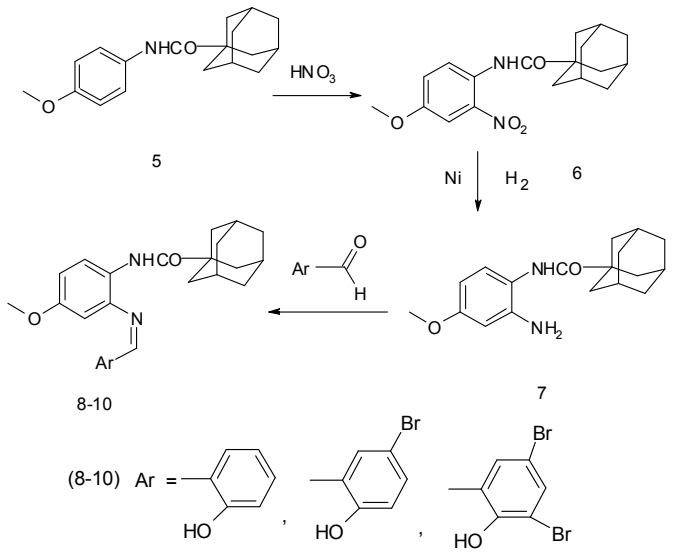
N-(1-adamantyl)carbonyl-*o*-phenylenediamine (1). 5.4 g (29 mmol) adamantane-1-carboxylic acid and 6 ml (60 mmol) thionyl chloride were heated at 50 °C in the water bath for 1 hr. After finishing the reaction the excess of thionyl chloride was expelled by vacuum, residual was treated with absolute ether and evaporated in the rotation evaporator, dilute in the absolute ether (30 ml). Ether solution of adamantane-1-carbonyl chloride obtained is used for adamantoyllation.

200 ml toluene and 20 ml 5% NaOH aqueous solution were added to 3.78g (35 mmol) *o*-phenylendiamine. Then under cooling reaction mixture with ice water it was added drop by drop to already prepared 30 ml ether solution of adamantane-1-carbonyl chloride, mixed for 30 min. Then the reaction mixture was mixed at the room temperature for 3 hrs and hold overnight. Reaction mixture was decomposed in the ice water, isolated sediment was filtered through the Schott funnel, washed with water, dried and 7.1g (87.65%) white crystals of compound (1) were obtained, mp 216-218 °C (chloroform), (229-231°C methanol [8]). R_f 0.88 (acetone: CCl_4, 1:2).

IR spectrum (KBr), ν, cm^{-1} : 1643 (C=O), 2846.6-2900.6 (C-HAd), 3010, 3050 (C-HAr), 3270.0 (NH), 3378.8, 3471.0 (NH_2).

UV spectrum (EtOH), $\lambda_{max.}$ nm (Abs): 209.00 (0.694), 289.00 (0.140).

^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.71 (6H, m, HAd), 1.92- 1.93 (6H, m, HAd), 2.02 (3H, m, HAd); 4.63 (2H, s, NH_2); 6.56 (1H, td, J_1 =8.0, J_2 =1.2, HAr), 6.74 (1H, dd, J_1 =8.0, J_2 =1.2, HAr), 6.92 (1H, td, J_1 = 8.0, J_2=1.6, HAr), 7.01 (1H, dd, J_1= 8.0, J_2 = 1.6, HAr); 8.64 (1H, s, NH).



Scheme 2. Derivatives of the 4-methoxy-N-(1-adamantyl)carbonyl-N'-benzylidene-*o*-phenylendiamines.

^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 27.62 (3C–Ad), 35.99(3C–Ad), 38.54 (3C–Ad), 40.40 (1C–Ad); 116.04, 116.29, 123.71, 125.98, 126.49, 142.84, 175.89 (C=O).

N-(1-adamantyl)carbonyl-N'-(2-hydroxybenzylidene)-*o*-phenylendiamine (2). Mixture of 2g (7.4 mmol) N-(1-adamantyl)carbonyl-*o*-phenylendiamine and 1 ml (9.3 mmol) salicyl aldehyde was heated in 20 ml of absolute ethanol for 5 hrs. Reaction mixture was cooled at the room temperature. Crystallized product was filtered, washed with a small amount of absolute hexane and dried. 2.77 g (68%) yellow crystals were obtained; mp154-156°C (ethanol). R_f 0.61 (light petroleum:ethylacetate, 3:1).

IR spectrum (KBr), ν, cm^{-1}: 1612.3 (C=N); 1666.3 (C=O); 2850.3, 2908.0 (C-H Ad), 3010.0, 3050.0 (C-HAr); 3100.0 (NH); 3425.1 (OH).

UV spectrum (EtOH), $\lambda_{max.}$, nm, (Abs): 218.00 (0.55), 231.00 (0.495), 264.00 (0.456), 345.00(0.283).

^{1}H NMR spectrum δ, ppm (*J*, Hz): 1.66-1.76 (6H, m, H Ad), 1.98- 1.99 (6H, m, H Ad), 2.10 (3H, m, H Ad); 7.01 (1H, td, J_1=7.6, J_2=1.0, H Ar), 7.07 (1H, d, *J*=8, H Ar), 7.13-7.15 (2H, m, H Ar), 7.28-7.32 (1H, m, H Ar), 7.42-7.47 (2H, m, HAr); 8.14 (1H, s, N=CH); 8.48 (1H, d, *J*=8, H Ar);8.65 (1H, s, NHCO);12.43 (1H, s, OH).

^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.12(3C-Ad), 36.38 (3C-Ad), 39.21 (3C-Ad), 42.01 (1C-Ad), 117.37, 117.87, 119.22,119.55, 120.65, 124.11, 128.05, 132.28, 132.79, 133.12, 133.81, 160.88 (N=CH), 164.14 (C-OH), 176.19 (C=O).

The synthesis of compounds described below (3, 4 and 8-10) was carried out in the same way as that of compound 2.

N-(1-adamantyl)carbonyl-N'-(2-hydroxy-5-bromobenzylidene)-*o*-phenylendiamine (3). 1.49g(89%) lemon-yellow crystals were obtained; mp182-183°C (ethanol). R_f 0.60 (light petroleum:ethylacetate, 3:1).

IR spectrum (HCBD, Nujol), ν,cm^{-1}: 1612.3 (C=N); 1658.6 (C=O), 2846.6, 2900.6 (C-H Ad), 3031.7, 3077.9 (C-H Ar), 3116.7-3270.8 (NH, OH)

UV spectrum (EtOH), $\lambda_{max.}$, nm, (Abs): 224.00 (0.655), 348.00 (0.261); 357.00 (0.268);

^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.70 (6H,m, H Ad), 1.91 (6H,m, H Ad), 2.02 (3H,m, H Ad); 6.96 (1H, d, *J*= 8.8, H Ar), 7.22 (1H, td, J_1=1.6, J_2= 7.6, H Ar), 7.28 (1H, td, J_1=1.6, J_2=7.6, H Ar), 7.40 (1H, dd, J_1=1.6, J_2=8.0, H Ar), 7.54 (1H, dd, J_1=2.8, J_2=8.8, H Ar), 7.79 (1H, dd, , J_1=1.2, J_2=8.0, H Ar), 7.99 (1H, d, *J*=2.8, H Ar); 8.89 (CH=N), 9.09 (NHCO), 12.15 (OH).

^{13}C NMR spectrum (DMSO-d_6), δ,ppm: 27.63 (3C-Ad), 34.79 (1C-Ad), 36.08 (3C-Ad), 40.75 (3C –Ad);

110.71 (C-Br), 118.22 (2C-Ar), 120.54 (2C-Ar), 121.20 (2C-Ar), 134.19, 142.70, 144.88, 158.05, 161.09 (C=N), 161.87 (C-OH), 179.96 (C=O).

N-(1-adamantyl)carbonyl-2-(2-hydroxy-3,5-dibromobenzylidene)-*o*-phenylendiamine (4). 1.25 g (77%) orange crystals were obtained, mp 178-180°C (ethanol); R_f 0.55 (light petroleum:ethylacetate, 3:1).

IR spectrum (HCBD, Nujol), ν, cm^{-1}: 1650.85 (C=O), 2908, 2850 (C-HAd),3062.6 (C-HAr), 3111.6, 3263.1, 3324.0 (OH, NH).

UV spectrum (EtOH), $\lambda_{max.}$, nm, (Abs): 220.00 (0.702);

^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.76 (6H, m, HAd), 1,99-1, 96 (6H, m, HAd), 2.10 (3H, m, H Ad), 7.17 (1H, dd, ,J_1=3.6, J_2= 5.8, HAr), 7.39 (1H, dd, J_1=3.6, J_2= 5.8, HAr), 7.55 (1H, d, *J* =2.4, HAr), 7.80 (1H, d, *J* =2.4, HAr), 8.02 (1H, CH=N), 8.40 (1H, d, *J* =8.0, HAr), 8.56 (1H, s, NHCO), 13.48 (1H, s, C-OH).

^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.11 (3C-Ad), 36.44 (3C-Ad), 39.35 (3C-Ad), 41.31 (1C-Ad), 110.93 (C-Br), 112.26 (C-Br), 117.80, 121.51, 124.46, 125.57, 126.02, 129.01, 130.79, 133.78, 138.68, 161.52 (C-OH, C=N), 177.17 (C=O).

4-methoxyphenyl-N-(1-adamantyl) carboxamide (5) was synthesized with *p*-anisidine adamantoyllation using the method of Schotten-Baumann [10]. By nitration of the synthesized compound with 57% -nitric acid in the acetic acid medium 4-methoxy-2-nitrophenyl-N-(1-adamantyl)carboxamide (6) was obtained [11,12]. By catalytic reduction of the nitro-compound (6) with molecular hydrogen in the ethyl acetate, at the existence of Raney nickel, 4-methoxy-2-aminophenyl-N-(1-adamantyl)carboxamide (7) was isolated [13]. Condensation reaction of amino amide (7) with aldehydes of salicyl-, 5-bromo salicyl- and 3,5-dibromo salicyl heating for 3-5 hrs in the alcohol medium. Corresponding derivatives of 4-methoxy-N-(1-adamantyl)carbonyl-N'-benzylidene-*o*-phenylendiamine (8-10) were obtained.

4-methoxy-2-nitrophenyl-N-(1-adamantyl)carboxamide (6). 5 ml acetic anhydride was added to the 5.7g (0.02 mol) of 4-methoxyphenyl-N-(1-adamantyl)carboxamide (5) solved in 20 ml acetic acid. Under mixing and cooling with ice 5 ml (0.06 mol) 57%-HNO_3 was added drop by drop. Then the product was mixed at the room temperature for 50 min. Reaction mixture was decomposed in the icy water. Isolated sediment was filtered and washed with water to neutral reaction. 6.53g (99%) yellow crystals were obtained; mp 133-134 °C (ethanol) (according to [10] it is134-135°C).

IR spectrum, (HCBD, Nujol), ν, cm^{-1}: 1249.0, 1288.0 (C-O-C); 1350.0, 1581.0 (NO_2); 1650.0 (C=O); 2846.0, 2908.0 (C-HAd, CH_3); 3090 (C-HAr); 3278 (NH).

^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*,Hz): 1.70 (6H, m, HAd), 1.85 (6H, m, HAd), 2.02 (3H, m, HAd), 3.83 (3H, s, CH_3), 7.30 (1H, dd, J_1=9.0, J_2 =3.0, HAr), 7.49 (1H, d, *J*= 3.0, HAr), 7.65 (1H, d, *J*= 9.0, HAr); 9.62 (1H, s, NHCO).

^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 27.5, 35.9, 38.2, 40.5, 55.9, 108.8,120.3, 125.0, 127.3,142.8,155.7,175.6 (C=O).

4-methoxy-2-aminophenyl-N-(1-adamantyl) carboxamide (7). Hydration of 4-methoxy-2-nitrophenyl-N-(1-adamantyl)carboxamide (6) was carried out in ethyl acetate with molecular hydrogen at the existence of Raney nickel. After finishing the reaction the suspension was filtered; the mass rest on the filter was washed in acetone; after removal of the solver 2.07 g (90%) pinkish sediment was obtained mp 210-211°C (ethanol) [13].

IR spectrum, (HCBD, Nujol), ν, cm^{-1}: 1280 (C-O-C), 1635 (C=O); 2846, 2900 (C-HAd, CH_3); 3001, 3054 (C-HAr);3286, 3402 (NH, NH_2).

UV spectrum (EtOH), $\lambda_{max.}$, nm, (Abs): 211 (0.85), 291 (0.13).

^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.69 (6H, m, HAd), 1.900 (6H, m, HAd), 1.999 (3H, m, HAd); 3.65 (3H, s, CH_3), 4.64 (2H, s, NH_2), 6.14 (1H, dd, J_1 = 8.4 , J_2= 0.8, HAr), 6.31 (1H, d, *J* = 0.8, HAr), 6.82 (1H, d, *J* =8.4, HAr), 8.51 (1H, s, NHCO).

^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 27.63, 36.02, 38.58, 40.28, 54.71, 100.78, 101.82, 116.88, 127.79, 144.35, 157.85.

4-methoxy-N-(1-adamantyl)carbonyl-N'-(2-hydroxybenzylidene)-*o*-phenylendiamine (8). 0.68 g (70%) lemon crystals were obtained. mp163-164°C (ethanol). R_f 0.49 (light petroleum:ethylacetate, 3:1).

IR spectrum (HCBD, Nujol), ν, cm^{-1}: 1604.56 (C=N), 1658.56 (C=O), 2846.56, 2908.27 (C-HAd), 3008, 3062 (C-HAr), 3155.13, 3300.69, 3432.84 (NH, OH).

UV spectrum, $\lambda_{max.}$, nm (Abs): 213.00 (0.650), 266.00 (0.438),347.00 (0.187).

^{1}H NMR spectrum (DMSO-d_6), δ,ppm (*J*,Hz):1.68 (6H, m, HAd), 1.88, 1.87 (6H, m, HAd), 1. 99 (3H, m, H Ad),3.81 (3H, s, CH_3), 6.85 (1H, dd, J_1=2.8, J_2 =8.8, H Ar), 6.96 (1H, s, HAr), 6.98-6.99 (2H, m, HAr), 7.37-7.43 (2H, m, HAr), 7.66 (1H, dd, J_1=1.6, J_2 =8.0, HAr), 8.83 (1H, s, CH=N), 8.88 (1H, s, NHCO), 12.70 (1H, s, OH).

^{13}C NMR spectrum (DMSO-d_6), δ,ppm: 27.57 (3C-Ad), 35.99(3C-Ad), 38.46(3C-Ad), 40.41(1C-Ad), 55.40 (1C–CH_3), 112.28, 116.57, 118.95, 119.51, 125.16, 126.98, 132.17, 133.25, 143.61, 157.33, 160.17 (C=N), 163.23 (C-OH), 175.67 (C=O).

4-methoxy-N-(1-adamantyl)carbonyl-N'-(2-hydroxy-5-bromo benzylidene)-*o*-phenylendiamine (9). 0.91 g (81%) lemon crystals were obtained, mp 200-202°C (ethanol). R_f 0.45 (light petroleum:ethylacetate, 3:1).

IR spectrum (HCBD, Nujol), ν, cm^{-1} : 1612.28 (C=N), 1658.56 (C=O), 2846, 2908 (C-HAd), 3000.84, 3070.27 (C-HAr), 3409.70 (NH, OH).

UV spectrum (EtOH), $\lambda_{max.}$, nm (Abs): 221.00 (0.516), 347.00 (0.187);

^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*,Hz):1.75 (6H, m, HAd), 1.96 (6H, m, HAd), 2.09 (3H, m, HAd); 3.83 (3H, s, CH_3); 6.68 (1H, d, *J*=2.4, HAr), 6.87 (1H, dd, J_1=2.4, J_2=9, HAr), 6.97 (1H, d, *J*=8.8, HAr), 7.50 (1H, dd, J_1=2.4, J_2 =8.8, HAr), 7.57 (1H, d, *J*=2.4, H Ar); 7.79 (1H, s, CH=N); 8.24 (1H, d, *J*=9.2, HAr); 8.55 (1H, s, NHCO); 12.505 (1H, s, OH).

^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 28.12, 36.40, 39.22, 41.76, 55.68 (CH_3), 104.006, 111.011, 113.00, 119.38, 120.52, 122.94, 125.64, 134.68, 139.32, 136.38, 156.59 (C-O), 159.91 (C=N), 162.65 (C-OH), 176 (C=O).

4-methoxy-N-(1-adamantyl)carbonyl-N'-(2-hydroxy-3,5-dibromo benzylidene)-*o*-phenylendiamine (10). 0.83 g (89%) lemon crystals were obtained. mp237-238°C (ethanol). R_f 0.40 (light petroleum: ethylacetate, 3:1).

IR spectrum (HCBD, Nujol), ν, cm^{-1}: 1604.56 (C=N), 1658.56 (C=O), 2846.56, 2900.56, (C-HAd), 3000.84, 3062.56 (C-HAr), 3378.84 (NH, OH).

UV spectrum (EtOH), $\lambda_{max.}$, nm (Abs): 207.00 (0.765), 348.00 (0.148);

^{1}H NMR spectrum (DMSO-d_6), δ,ppm (*J*, Hz): 1.70 (6H,m, HAd), 1.91 (6H, m, HAd), 1.99 (3H, m, HAd); 3.82 (3H, s, CH_3); 6.91 (1H, dd, J_1=2.8, J_2=8.8 , HAr), 7.08 (1H, d, *J*=2.8, HAr), 7.28 (1H, d, *J*=8.8, HAr), 7.87 (1H, d, *J*=2.4, HAr), 7.93 (1H, d, *J*=2.4, HAr), 8.95(1H, s,NHCO), 8.93 (1H, s, CH=N); 14.49 (1H, s, OH);

^{13}C NMR spectrum (DMSO-d_6), δ,ppm: 27.61, 36.04, 38.01, 38.38,48.89 (C-CH_3),73.98, 89.13, 94.04, 104.40, 106.81, 113.13,116.46,123.48,168.81 (C=N), 170.47 (C-OH), 179.91 (C=O).

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ორგანული ქიმია

N-(1-ადამანტილ)კარბონილ- N'-ბენზილიდენ-*o*-ფენილენდიამინის ზოგიერთი წარმოებულის სინთეზი

შ. სამსონია[*], დ. ზურაბიშვილი[**], თ. ბუკია[**], გ. ბუზალაძე[**], მ. ლომიძე[**], ე. ელიზბარაშვილი[§], უ. კაცმაიერი[§§].

[*] *აკადემიკოსი, ი. ჯავახიშვილის სახ. თბილისის სახელმწიფო უნივერსიტეტი, ქიმიის დეპარტამენტი, თბილისი*

[**] *ი.ჯავახიშვილის სახ. თბილისის სახელმწიფო უნივერსიტეტი, ზუსტ და საბუნებისმეტყველო მეცნიერებათა ფაკულტეტი, ორგანული ქიმიის სამეცნიერო-კვლევითი ინსტიტუტის ალიციკლური ნაერთების განყოფილება, თბილისი*

[§] *საქართველოს ტექნიკური უნივერსიტეტი, ქიმიური ტექნოლოგიისა და მეტალურგიის ფაკულტეტი, თბილისი*

[§§] *ზაარლანდის უნივერსიტეტი, ორგანული ქიმიის ინსტიტუტი, საარბრუკენი, გერმანია.*

სინთეზირებულია N-(1-ადამანტილ)კარბონილ-*o*-ფენილენდიამინი და 4-მეთოქსი-2-ამინოფენილ–N–(1–ადამანტილ)კარბოქსამიდი. შესწავლილია მათი კონდენსაციის რეაქცია სალიცილის-, 5-ბრომსალიცილის- და 3,5-დიბრომსალიცილის ალდეჰიდებთან. მიღებულია: N-(1-ადამანტილ)კარბონილ-N'-(2-ჰიდროქსიბენზილიდენ)-, N-(1-ადამანტილ)კარბონილ-N'-(2-ჰიდროქსი-5-ბრომბენზილიდენ)-, N-(1-ადამანტილ)კარბონილ-N'-(2-ჰიდროქსი-3,5-დიბრომბენზილიდენ)-, 4-მეთოქსი-N-(1-ადამანტილ)კარბონილ-N'-(2-ჰიდროქსიბენზილიდენ)-, 4-მეთოქსი-N-(1-ადამანტილ)კარბონილ-N'-(2-ჰიდროქსი–5-ბრომბენზილიდენ)-, 4-მეთოქსი-N-(1-ადამანტილ)კარბონილ-N'-(2-ჰიდროქსი–3,5-დიბრომბენზილიდენ)-*o*-ფენილენ დიამინები. სინთეზირებული ნაერთების აგებულება დადასტურებულია იწ, უი და ბმრ სპექტრული მონაცემებით.

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