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One-Pot Synthesis of Cage Alcohols

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Abstract—An efficient one-pot procedure has been developed for the synthesis of cage alcohols with hydroxy groups in the bridgehead positions. The procedure includes initial nitroxylation with nitric acid or a mixture of nitric acid with acetic acid and subsequent hydrolysis in the presence of urea.

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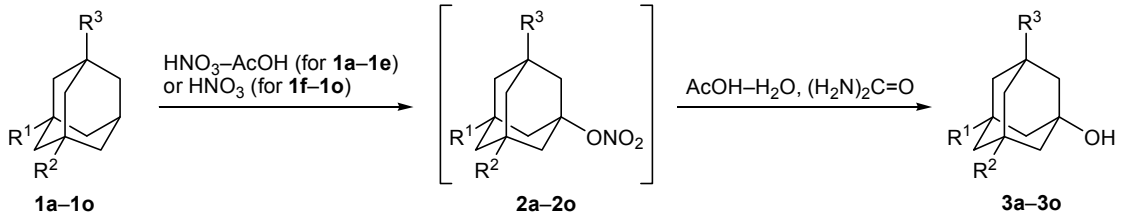
Hydroxy derivatives of adamantane are key starting materials in the synthesis of biologically active compounds [1–10] and those possessing practically important properties [11–18]. Hydroxy adamantanes are generally synthesized by oxidation of cage hydrocarbons in the presence of transition metal compounds [19–35], hydrolysis of halogen derivatives [36–38], ethers [39–43], and amines [44], and decarboxylation of adamantane-1-carboxylic acids [45–47]. Despite a large number of known methods for their synthesis, many procedures are not free from such disadvantages as the use of difficultly accessible catalysts, low selectivity, and poor yield. Therefore, there is a need of a general method for the synthesis of cage hydroxy derivatives directly from the corresponding hydrocarbons.

We have developed a one-pot procedure for the synthesis of substituted adamantan-1-ols via successive nitroxylation of initial cage compound with fuming nitric acid or a mixture of nitric acid and acetic acid and subsequent hydrolysis of intermediate adamantyl nitrate in aqueous acetic acid in the presence of urea [48]. In the first step, the reaction of substrate **1a**–**1o** with fuming nitric acid gives the corresponding adamantyl nitrate **2a**–**2o** (Scheme 1). Optimal conditions for the nitroxylation of cage compounds, including those containing electron-withdrawing substituents in the bridgehead positions, were proposed in [49–54]; the necessity of adding acetic acid at the stage of nitroxylation of adamantane and its homologs **1a**–**1e** was also justified therein.

The chemical nature and ratio of the nitroxylation products are determined by transformations of cage nitric acid esters in nitric acid. Assuming that generation of a cationic center in nitric acid medium is possible, it is reasonable to expect that addition of water as external nucleophile would force the equilibrium to shift toward formation of products resulting

Scheme 1.

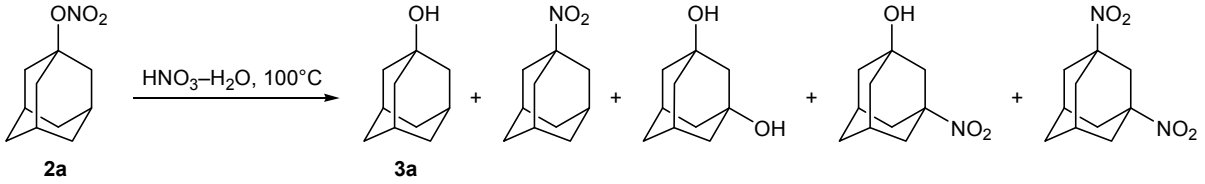


1a–1o **2a–2o** **3a–3o**

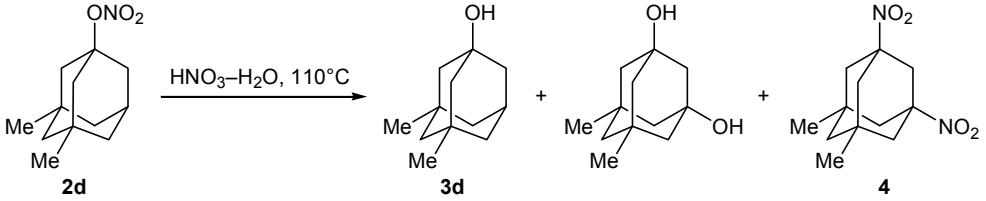
$R^1 = R^2 = R^3 = H$ (**a**); $R^1 = Me$, $R^2 = R^3 = H$ (**b**); $R^1 = Et$, $R^2 = R^3 = H$ (**c**); $R^1 = R^2 = Me$, $R^3 = H$ (**d**); $R^1 = R^2 = R^3 = Me$ (**e**); $R^1 = CH_2COOH$, $R^2 = R^3 = H$ (**f**); $R^1 = CH_2COOH$, $R^2 = Me$, $R^3 = H$ (**g**); $R^1 = CH_2COOH$, $R^2 = Et$, $R^3 = H$ (**h**); $R^1 = CH_2COOH$, $R^2 = R^3 = Me$ (**i**); $R^1 = COOH$, $R^2 = R^3 = H$ (**j**); $R^1 = COOH$, $R^2 = Me$, $R^3 = H$ (**k**); $R^1 = COOH$, $R^2 = Et$, $R^3 = H$ (**l**); $R^1 = COOH$, $R^2 = R^3 = Me$ (**m**); $R^1 = CF_3$, $R^2 = R^3 = H$ (**n**); $R^1 = C(NO_2)_3$, $R^2 = R^3 = H$ (**o**).

† Deceased.

Scheme 2.



Scheme 3.



from the reaction of the cation with predominant and most nucleophilic component of the reaction mixture. Addition of water to the reaction mixture and subsequent heating lead to the formation of hydrolysis products (alcohols) [55]. However, this process is accompanied by side oxidation and nitration. For example, according to the GC/MS data, the hydrolysis of adamantan-1-yl nitrate (**2a**) in dilute nitric acid gives a mixture of adamantan-1-ol (**3a**, 81.7%), 1-nitroadamantane (8.6%), adamantane-1,3-diol (6.0%), 3-nitroadamantan-1-ol (2.8%), and 1,3-dinitroadamantane (0.7%) (Scheme 2). In addition, traces of 5-nitroadamantan-2-one and *cis*- and *trans*-1,4-dinitroadamantanes were detected. These findings indicate that the hydrolysis is accompanied by partial substitution of tertiary hydroxy group by nitro group.

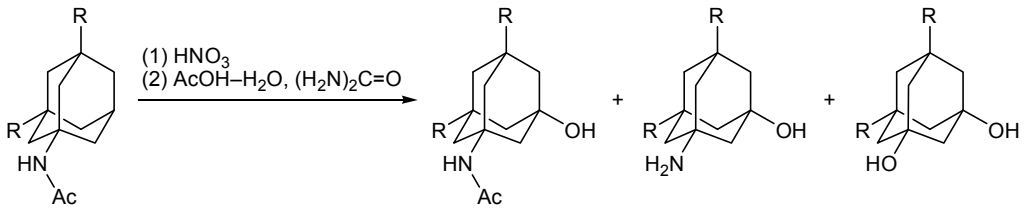
In the hydrolysis of 3,5-dimethyladamantan-1-yl nitrate (**2d**) at 110°C (4 h), apart from hydroxy derivatives, 6% of 5,7-dimethyl-1,3-dinitroadamantane (**4**) was isolated, whose structure was confirmed by ^{1}H and ^{13}C NMR and mass spectra (Scheme 3).

Obviously, high selectivity in the hydrolysis stage can be achieved by binding nitrogen oxides liberated at the nitroxylation stage and generated by thermal decomposition of nitric acid [56]. In fact, when the hydrolysis was carried out in the presence of urea, the yields of alcohols **3a**–**3e** were fairly high (71–91%).

The nitroxylation of cage substrates containing electron-withdrawing substituents was more selective than the reaction with parent hydrocarbons [49]. No nitro compounds were detected in the reaction mixtures, and the fraction of the corresponding alcohols was insignificant. Compounds **1f**–**1o** were converted to hydroxy derivatives by treatment with fuming nitric acid, followed by addition of urea in aqueous acetic acid under reflux. Kinetic studies showed that carboxymethyl, carboxy, trifluoromethyl, and nitromethyl groups strongly inhibited nitroxylation [49]. The effects of carboxy and trinitromethyl groups determined harsh conditions in the synthesis of nitrates. Carboxymethyl group deactivates the adamantane skeleton to a lesser extent. As might be expected, the rate of hydrolysis of nitroxy derivatives **2f**–**2o** was not high, and an appreciable yield of alcohols **3f**–**3o** was attained only after prolonged heating. Compounds **3f**–**3o** were thus isolated in 65–90% yield.

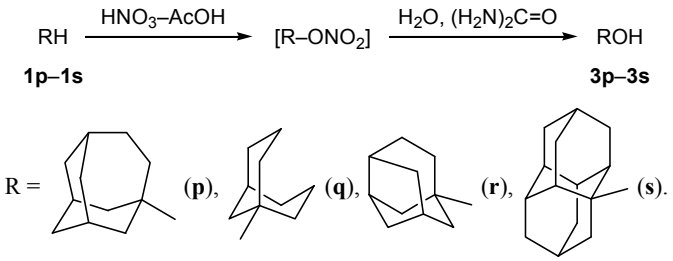
Alcohols **6a** and **6b** were formed from acetylaminoadamantanes **5a** and **5b** in low yields. The reaction was accompanied by hydrolysis of the amide moiety with formation of amino alcohols **7a** and **7b** (isolated as

Scheme 4.



R = H (**a**), Me (**b**).

Scheme 5.



hydrochlorides) and dihydroxy derivatives **8a** and **8b** (Scheme 4).

The procedure proposed for the synthesis of hydroxy adamantanes is fairly general and is suitable for compounds with various substituents in the bridgehead positions, as well as for related cage compounds such as homoadamantane (**1p**), bicyclo[3.3.1]nonane (**1q**), protoadamantane (**1r**), and diamantane (**1s**). The corresponding hydroxy derivatives **3p**–**3s** were isolated in 36–86% yield, and their physical constants and spectral parameters were in agreement with published data [42, 57, 58]. Diamantane (**1s**) reacted with nitric acid exclusively at the medial position, while in the reaction with homoadamantane (**1p**) up to 5% of isomeric homoadamantan-1-ol was formed (according to the GC/MS data).

In summary, we have developed an efficient one-pot procedure for the synthesis of cage alcohols with high yields directly from hydrocarbon precursors with the use of accessible reagents via successive nitroxylation and hydrolysis.

EXPERIMENTAL

The mass spectra (electron impact, 70 eV) were recorded on a Thermo Finnigan DSQ GC/MS instrument [ZB5MS quartz capillary column, 30×0.32 mm, film thickness 0.25 μm; carrier gas helium, flow rate 1.5 mL/min; injector temperature 300°C; oven temperature programming from 80°C (1 min) to 340°C at a rate of 20 deg/min]. The ^{1}H and ^{13}C NMR spectra were recorded on a Jeol JNM ECX-400 spectrometer (400 MHz for ^{1}H). The IR spectra were measured in KBr on a Shimadzu IR Affinity-1 spectrometer. The elemental analyses were obtained with a EuroVector 3000 EA analyzer using L-cystine as standard.

Alcohols 3a-3e and 3p-3s (*general procedure***).** Compound **1a**–**1e** or **1p**–**1s**, 15 g, was dispersed in 25 mL of glacial acetic acid, and 10 equiv of fuming nitric acid was added dropwise with stirring at 15–20°C. The mixture was stirred for 1 h at 20°C, and a solution of 5 g of urea in 120 mL of 15% aqueous acetic acid was slowly added dropwise with vigorous stirring, maintaining the temperature no higher than 25°C. The mixture was then slowly heated to 100°C, kept for 1 h at that temperature, and cooled to 50°C, and 40% aqueous sodium hydroxide was added to pH 10. The mixture was cooled to room temperature, and the precipitate was filtered off and recrystallized.

Adamantan-1-ol (3a). Yield 91%, mp 280–282°C (from toluene) [59].

3-Methyladamantan-1-ol (3b). Yield 82%, mp 130–131°C (from hexane) [51].

3-Ethyladamantan-1-ol (3c). Yield 71%, mp 65–66°C (from hexane) [51].

3,5-Dimethyladamantan-1-ol (3d). Yield 79%, mp 96–97°C (from hexane) [51].

3,5,7-Trimethyladamantan-1-ol (3e). Yield 79%, mp 120–121°C (from hexane) [51].

Homoadamantan-3-ol (3p). Yield 73%, mp 270–272°C (from hexane) [42]; according to the GLC data, the reaction gave a mixture of homoadamantan-3- and -1-ols at a ratio of 25:1.

Protoadamantan-6-ol (3q). Yield 86%, mp 234–236°C [57].

Bicyclo[3.3.1]nonan-1-ol (3r). Yield 85%, mp 186–188°C [42].

Diamantan-1-ol (3s). Yield 36%, mp 289–291°C [58].

5,7-Dimethyl-1,3-dinitroadamantane (4) was isolated by column chromatography (eluent CCl_4) when the hydroxylation of **1d** was carried out in the absence of urea (110°C, 4 h). Yield 6%, mp 161–163°C [60]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.09 s (6H, CH_3), 1.28–1.31 m (2H, CH_2), 1.92–1.96 m (8H, CH_2), 2.61–2.64 m (2H, CH_2). ^{13}C NMR spectrum, δ_C, ppm: 28.70 (CH_3), 34.95 (CH_2), 41.90 (CH_2), 44.60 (CH_2), 47.30, 86.30. Mass spectrum, *m*/*z* (I_{rel}, %): 208 (5), 162 (3), 161 (28), 133 (6), 29 (100).

Alcohols 3f–3o (*general procedure*). Compound **1f–1o**, 5 g, was added with stirring to 35 (**1f–1i**), 50 (**1j–1m**), or 70 equiv (**1n**, **1o**) of fuming nitric acid at 15–20°C, and the mixture was stirred for 3–5 h at 20°C. A solution of 3 g of urea in 100 mL of 20% aqueous acetic acid was slowly added dropwise with vigorous stirring to the resulting solution, maintaining the temperature no higher than 25°C. The mixture was then slowly heated to 90°C, kept for 5 h at that temperature, cooled to 50°C, and treated with 40% aqueous sodium hydroxide to pH 5. The mixture was cooled to room temperature, and the precipitate was filtered off and recrystallized.

(3-Hydroxyadamantan-1-yl)acetic acid (3f). Yield 80%, mp 126–128°C; published data [61]: mp 127–128°C.

(3-Hydroxy-5-methyladamantan-1-yl)acetic acid (3g). Yield 72%, mp 97–99°C; published data [61]: mp 98–100°C.

(3-Ethyl-5-hydroxyadamantan-1-yl)acetic acid (3h). Yield 75%, mp 205–206°C (from acetone); published data [47]: mp 205–207°C.

(3-Hydroxy-5,7-dimethyladamantan-1-yl)acetic acid (3i). Yield 84%, mp 128–130°C; published data [61]: mp 126–127°C.

3-Hydroxyadamantane-1-carboxylic acid (3j). Yield 90%, mp 203–205°C; published data [62]: mp 202–203°C.

3-Hydroxy-5-methyladamantane-1-carboxylic acid (3k). Yield 65%, mp 163–165°C; published data [63]: mp 163–164°C.

3-Ethyl-5-hydroxyadamantane-1-carboxylic acid (3l). Yield 79%, mp 163–164°C (from acetone–hexane). IR spectrum, ν, cm^{-1}: 3310 (O–H), 2920, 2850 (C–H), 1700 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 0.71 t (3H, CH_3, J = 6.84 Hz), 0.99–2.25 m (15H, CH_2, CH), 4.38 s (1H, OH), 12.02 s (1H, COOH). Found, %: C 69.47; H 9.06. $C_{13}H_{20}O_3$. Calculated, %: C 69.64; H 8.93.

3-Hydroxy-5,7-dimethyladamantane-1-carboxylic acid (3m). Yield 85%, mp 228–230°C (from acetone–hexane); published data [64]: mp 235–236°C.

3-Trifluoromethyladamantan-1-ol (3n). Yield 85%, mp 78–80°C (from hexane); published data [65]: mp 82°C.

3-Trinitromethyladamantan-1-ol (3o). Yield 92%, mp 160–162°C (from acetone); published data [66]: mp 167°C.

***N*-(3-Hydroxyadamantan-1-yl)acetamide (6a).** Yield 13%, mp 228–230°C (from acetone); published data [67]: mp 220°C. Diol **8a** and **7a** hydrochloride were also isolated.

***N*-(3-Hydroxy-5,7-dimethyladamantan-1-yl)-acetamide (6b).** Yield 17%, mp 187–189°C (from benzene) [68]. Diol **8b** and **7b** hydrochloride were also isolated.

3-Aminodamantan-1-ol (7a) hydrochloride. Yield 46%, mp >300°C (from *i*-PrOH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.35–1.46 m (6H, CH_2), 1.48–1.67 m (6H, CH_2), 2.14 s (2H, CH), 4.57 s (1H, OH), 8.22 br.s (3H, NH_3^+). ^{13}C NMR spectrum, δ_C, ppm: 30.14 (CH), 34.57 (CH_2), 39.41 (CH_2), 43.95 (CH_2), 48.37 (CH_2), 53.58, 67.57. Found, %: C 59.00; H 8.93. $C_{10}H_{18}ClNO$. Calculated, %: C 58.96; H 8.91.

3-Amino-5,7-dimethyladamantan-1-ol (7b) hydrochloride. Yield 66%, mp >340°C (from *i*-PrOH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 0.83 s (6H, CH_3), 0.97–1.00 m (2H, CH_2), 1.18 s (4H, CH_2), 1.30–1.38 m (4H, CH_2), 1.57 s (2H, CH_2), 4.77 s (1H, OH), 8.27 br.s (3H, NH_3^+). ^{13}C NMR spectrum, δ_C, ppm: 29.39 (CH_3), 30.02 (C), 45.52 (CH_2), 46.88 (CH_2), 49.13 (CH_2), 50.28 (CH_2), 54.29, 68.72. Found, %: C 62.79; H 9.61. $C_{12}H_{21}ClNO$. Calculated, %: C 62.75; H 9.58.

Adamantane-1,3-diol (8a). Yield 11%, mp 312–315°C (from dioxane); published data [11]: mp 315°C.

5,7-Dimethyladamantane-1,3-diol (8b). Yield 9%, mp 216–218°C (from $CHCl_3$); published data [51]: mp 217–218°C.

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