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Synthesis of Amino Alcohols of the Cyclododecane and Decahydro-1,4-ethanonaphthalene Series

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Abstract—2-Aminocyclododecan-1-ol and 6(3)-aminodecahydro-1,4-ethanonaphthalen-5(2)-ols (mixture of isomers) were synthesized in two steps via oxidative hydroxybromination of cyclododecene and 1,2,3,4,4a,5,6,8a(1,4,4a,5,6,7,8,8a)-octahydro-1,4-ethanonaphthalines with the system hydrogen peroxide–HBr. In the first step, oxidation of hydrogen bromide with hydrogen peroxide generated intermediate dioxidanium bromide which added to the C=C double bond of the unsaturated substrate to give the corresponding α-bromo alcohols. In the second step, substitution of the bromine atom by amino group in the presence of an alkali afforded α-amino alcohols.

Keywords: cyclododecene, tricyclododecene, aminocyclododecanol, hydrobromic acid, hydrogen peroxide, primary and secondary amines, aminooctrahydro-1,4-ethanonaphthol

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Preparation of polyfunctional C_5–C_{12}-alicyclic compounds is of great interest due to broad spectrum of their application in petrochemical and organic synthesis. These compounds are important not only per se but also as intermediate products in the synthesis of biologically active and pharmacological agents [1–9], corrosion inhibitors, and antioxidants [9, 10]. Among them, of particular importance are macrocyclic and bridged amino alcohols and their derivatives [11–14]. Introduction of hydrophilic amino and hydroxy groups into a hydrophobic carbon macrocycle could significantly change their biological and pharmacological activity [9, 12]. A number of synthetic analogs of various natural biologically active compounds, as well as of antiviral and antibacterial agents [9, 11, 12], have been designed on the basis of macrocyclic amino alcohols [3].

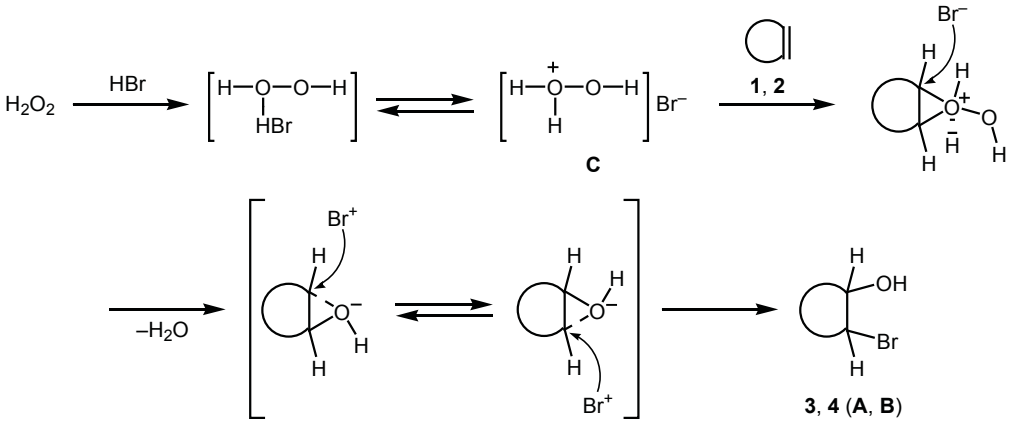
The most common method for the synthesis of various amino alcohols is ring opening of oxiranes by the action of nitrogen nucleophiles [4, 9–15]. Published data on macrocyclic and bridged amino alcohols are few in number [16–19], and the available information refers mainly to heterocyclic derivatives [7, 12].

The present study was aimed as synthesizing macrocyclic and bridged polycyclic amino alcohols from products of oxidative hydroxybromination of unsaturated cyclic C_{12}-hydrocarbons, namely cyclododecene and 1,2,3,4,4a,5,6,8a-octahydro-1,4-ethanonaphthalene, using the system H_2O_2–HBr.

We previously reported the results of studies on the synthesis of vicinal amino alcohols based on cyclohexene and bicyclo[2.2.1]hept-2-ene derivatives [16, 20] via addition of intermediate oxidizing species generated *in situ* to the C=C double bond and subsequent substitution of halogen by the action of a primary or secondary amine. In continuation of these studies, in this work we tried to synthesize amino alcohols from macro- and polycyclic hydrocarbons.

The starting materials were cyclododecene (**1**) and a mixture of isomeric 1,2,3,4,4a,5,6,8a- (**2A**) and 1,4,4a,5,6,7,8,8a-octahydro-1,4-ethanonaphthalenes (**2B**) which was obtained by partial hydrogenation of 1,4,4a,5,6,8a-hexahydro-1,4-ethanonaphthalene over nickel–kieselguhr catalyst according to [21]. We have found that both cyclododecene (**1**) and isomer mixture **2A**/**2B** reacted with the system H_2O_2–HBr under mild conditions (20–50°C) to give the corresponding α-bromo alcohols **3** and **4** (**A**/**B**) via addition of a metastable complex generated *in situ* from hydrogen peroxide and hydrogen bromide. The yield of **3** and **4** increased as the temperature rose from 20 to 50°C and reached 59.3–84.6%. Further raising the temperature to

Scheme 1.



1, Cyclododecene; **2**, 1,2,3,4,4a,5,6,8a- and 1,4,4a,5,6,7,8,8a-octahydro-1,4-ethanonaphthalenes (mixture of isomers); **3**, 2-bromocyclododean-1-ol; **4**, 6-bromodecahydro-1,4-ethanonaphthalen-5-ol (**A**) and 3-bromodecahydro-1,4-ethanonaphthalen-2-ol (**B**) (mixture of isomers).

75°C promoted decomposition of hydrogen peroxide and intermediate complex with liberation of molecular oxygen and/or bromine, respectively.

Scheme 1 shows a plausible mechanism for the formation of cyclic α-bromo alcohols from cyclic olefins in the system H_2O_2–HBr. The attack of hydrogen bromide molecule on the oxygen atom of hydrogen peroxide give intermediate complex **C** containing electrophilic oxygen. The addition of this complex to C=C double bond of the substrate is followed by elimination of water and energetically favorable charge redistribution with the generation of active hydroxy bromide intermediate. Fast addition of electrophilic oxygen to the double bond gives carbocation which then reacts with bromide ion to produce hydroxy bromide. Presumably, the addition of electrophilic oxygen is controlled by structural conformational directionality in a way similar to nucleophilic oxirane ring opening according to the Fürst–Plattner rule [12, 13].

By reacting bromo alcohols **3** and **4** with primary and secondary amines we obtained amino alcohols **5a**–**5d** and **6a**–**6d** (Scheme 2).

EXPERIMENTAL

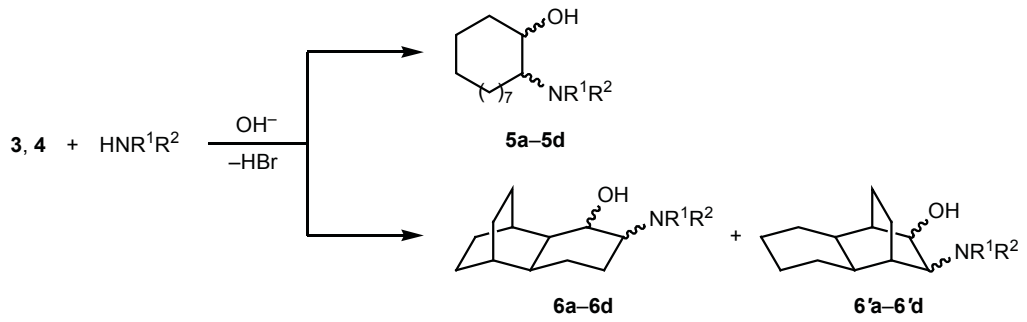
The IR spectra were recorded in the range 400–4000 cm^{-1} on a Bruker Alpha FT-IR spectrometer from samples dispersed in mineral oil or pressed with KBr. The ^1H and ^{13}C NMR spectra were measured on a Bruker 300 instrument at 300.18 and 75 MHz, respectively, using chloroform-*d* as solvent and reference (δ 7.25, δ_C 77.00 ppm). Elemental analysis was performed on a Leco TruSpec Micro analyzer (USA). GLC analyses were run on a Tsvet-500 chromatograph equipped with a thermal conductivity detector and a 2000×4-mm column packed with 10% poly(ethylene glycol succinate) on Chromosorb W; carrier gas He, flow rate 40 cm^3/min; oven temperature 150°C.

Butylamine, diethylamine, piperidine, morpholine, hydrogen peroxide, aqueous HBr, and cyclododecene (1:1 mixture of *cis* and *trans* isomers) were commercial products purchased from Alfa Aesar or Johnson Matthey PLC; a mixture of 1,2,3,4,4a,5,6,8a-octahydro-1,4-ethanonaphthalene (**2A**) and 1,4,4a,5,6,7,8,8a-octahydro-1,4-ethanonaphthalene (**2B**) (92.5:7.5) was obtained by partial hydrogenation of 1,4,4a,5,6,8a-hexahydro-1,4-ethanonaphthalene over nickel–kieselguhr according to the procedure reported in [5, 16].

General procedure for the hydroxybromination of C_{12}-cycloolefins [16, 19]**.** A flask was charged at a required temperature with 0.15–0.2 mol of 8–15% aqueous HBr and 0.1 mol of substrate **1** or **2**, and 0.2–0.25 mol of 26–30% aqueous hydrogen peroxide was added at a rate of 10 g/h from a dropping funnel with vigorous stirring (200–250 rpm). The mixture was stirred for 5–6 h until the oxidant was consumed completely according to the data of permanganometric and iodometric titration. The organic layer was separated, the aqueous layer was extracted with toluene (2× 100 mL), the extracts were combined with the organic phase, treated with a 10% aqueous solution of Na_2CO_3, and dried over magnesium sulfate, and the solvent was removed under reduced pressure.

2-Bromocyclododecan-1-ol (3) was synthesized from 4.98 g (30 mmol) of cyclododecene. Yield 6.49 g (82.3%), *cis*/*trans* ratio 35:65, bp 207–209°C

Scheme 2.



5, **6**, $R^1 = R^2 =$ Et (**a**); $R^1 =$ H, $R^2 =$ Bu (**b**); R^1R^2N = piperidin-1-yl (**c**), morpholin-4-yl (**d**).

(0.204 kPa). IR spectrum, ν, cm^{-1}: 3495 (OH), 2860–2855 (CH_2, sym.), 1460 (δCH_2, asym.), 1360, 1345 (δCH), 1128 (δOH), 765, 750 (C–Br) [22]. ^{1}H NMR spectrum, δ, ppm: 1.31–1.73 m (20H, CH_2), 3.57 t (1H, C**H**OH, *J* = 7.9 Hz), 3.58 br.s (1H, OH), 3.67 t (1H, CHBr, *J* = 7.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 78.2 (C^1), 47.4 (C^2), 32.8 (C^3), 30.8 (C^{12}), 24.8 (C^6–C^{10}), 23.8 (C^5), 23.6 (C^{11}), 21 (C^4) [23]. Found, %: C 54.25; H 8.23; Br 30.18. $C_{12}H_{23}BrO$. Calculated, %: C 54.75; H 8.74; Br 30.42.

6-Bromodecahydro-1,4-ethanonaphthalen-5-ol (4A) and 3-bromodecahydro-1,4-ethanonaphthalen-2-ol (4B) were synthesized from 5.67 g (35 mmol) of isomer mixture **A**/**B**. Yield 7.14 g (78.8%), ratio **4A**/**4B** 92.6:7.4, mp 101–103°C. IR spectrum, ν, cm^{-1}: 3495 (OH), 2860–2855 (CH_2, sym.), 1460 (δCH_2, asym.), 1360, 1345 (δCH), 1095 (δOH), 765, 750 (C–Br). ^{1}H NMR spectrum, δ, ppm: 1.30–1.78 m (16H, CH_2), 3.49 d (1H, CHBr, *J* = 7.3 Hz), 3.59 d (1H, **H**COH, *J* = 7.2 Hz), 3.65 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 75.6 (C^6), 57 (C^4), 42.8 (C^9), 41.8 (C^5), 31.3 (C^7), 27.9 (C^{10}), 26.5 (C^7), 25.0 (C^3), 24.5 (C^1, C^{11}), 23.8 (C^2, C^{12}). Found, %: C 54.87; H 6.98; Br 30.68. $C_{12}H_{19}BrO$. Calculated, %: C 55.38; H 7.69; Br 30.77.

General procedure for the reaction of compounds 3 and 4 with amines. A 40% solution of potassium hydroxide in propan-2-ol or a solution of K_2CO_3 in chloroform, 30–50 mL, was added to 6.57 g (25 mmol) of compound **3** or 6.48 g (25 mmol) of **4**. The mixture was heated to 50–60°C, and 0.15–0.25 mmol of the corresponding amine was added dropwise with stirring over a period of 1–1.5 h. The mixture was stirred for 3–6 h at that temperature until the initial compound (**3** or **4**) disappeared completely according to the GLC and TL data. Liquid products were washed until neutral reaction (litmus), dried over $MgSO_4$, and distilled under reduced pressure. Crystalline products were filtered off, washed with propan-2-ol, dried, and recrystallized from propan-2-ol–water (5:1).

2-(Diethylamino)cyclododecan-1-ol (5a) was synthesized from 6.57 g (25 mmol) of **3** and 3.3 g (45 mmol) of diethylamine. Yield 5.5 g (86.3%), *cis*/*trans* 45:55, mp 71–73°C. IR spectrum, ν, cm^{-1}: 3496 (OH), 3340, 3236 (NH), 2960 (CH_3, sym.), 2855 (CH_2, sym.), 1295 (C–N), 1460 (δCH_2, asym.), 1096 (δOH). ^{1}H NMR spectrum, δ, ppm: 1.05 t [6H, $N(CH_2C\mathbf{H}_3)_2$, *J* = 8.2 Hz], 1.26–1.58 m (20H, CH_2), 2.42 d [4H, $N(C\mathbf{H}_2)_2$, *J* = 8.1 Hz], 2.70 t (1H, CHN, *J* = 8.0 Hz), 3.46 d (1H, C**H**OH, *J* = 8.1 Hz), 3.58 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 74.2 (C^1), 70.6 (C^2), 50.3 (NCH_2), 30.2 (C^{12}), 28.7 (C^3), 24.6 (C^5–C^{11}), 13.8 (CH_3). Found, %: C 74.88; H 12.56; N 5.24. $C_{16}H_{33}NO$. Calculated, %: C 75.29; H 12.94; N 5.49.

2-(Butylamino)cyclododecan-1-ol (5b) was synthesized from 6.57 g (25 mmol) of **3** and 3.65 g (50 mmol) of butan-1-amine. Yield 4.7 g (73.8%), c*is*/*trans* 45:55, mp 90–93°C. IR spectrum, ν, cm^{-1}: 3496 (OH), 3240 (NH), 1665 (C–N), 1460 (δCH_2, asym.), 1450 (δCH), 1110 (δOH). ^{1}H NMR spectrum, δ, ppm: 0.94 t (3H, CH_3, *J* = 6.8 Hz), 1.27–1.46 m (24H, CH_2), 2.10 br.s (1H, NH), 2.57 d (2H, NCH_2, *J* = 7.2 Hz), 2.71 d (1H, CHN, *J* = 7.9 Hz), 3.47 t (1H, C**H**OH, *J* = 8.1 Hz), 3.63 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 76.4 (C^1), 63.8 (C^2), 30.8 (C^3), 29.8 (C^{12}), 24.6 (C^5–C^{10}), 24.3 (C^{11}), 24 (C^4), 50.4 (C^1), 24 (CH_2), 11.7 (CH_3). Found, %: C 74.85; H 12.12; N 5.21. $C_{16}H_{33}NO$. Calculated, %: C 75.29; H 12.41; N 5.49.

2-(Piperidin-1-yl)cyclododecan-1-ol (5c) was synthesized from 6.57 g (25 mmol) of **3** and 4.25 g (50 mmol) of piperidine. Yield 4.90 g (73.5%), *cis*/*trans* 45:55, mp 95–97°C. IR spectrum, ν, cm^{-1}: 3496 (OH), 3240 (NH), 1665 (C–N), 1462 (δCH_2, asym.), 1450 (δCH), 1110 (δOH). ^{1}H NMR spectrum, δ, ppm: 1.26–1.63 m (26H, CH_2), 2.49 t [4H, $N(CH_2)_2$,

J = 7.3 Hz], 2.72 d (1H, CHN, J = 7.2 Hz), 3.46 t (1H, C**H**OH, J = 8.2, 7.1 Hz), 3.61 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 74.3 (C^1), 70.9 (C^2), 30.2 (C^{12}), 28.7 (C^3), 24.6 (C^5–C^{11}), 20.4 (C^4), piperidin: 54.9 (NCH_2), 26.4 ($C^{3'}$, $C^{5'}$), 24.7 ($C^{4'}$). Found, %: C 75.97; H 12.13; N 5.22. $C_{17}H_{33}NO$. Calculated, %: C 76.40; H 12.36; N 5.27.

2-(Morpholin-4-yl)cyclododecan-1-ol (5d) was synthesized from 6.57 g (25 mmol) of **3** and 4.35 g (50 mmol) of morpholine. Yield 4.81 g (71.6%), *cis*/*trans* 45:55, mp 110–113°C. IR spectrum, ν, cm^{-1}: 3494 (OH), 3256 (NH), 3240 (NH), 1525 (C–N), 1460 (δCH_2, asym.), 1340, 1125 (C–O–C), 1111, 1082 (δOH). ^{1}H NMR spectrum, δ, ppm: 1.23–1.56 m (20H, CH_2), 2.65 t [4H, N$(CH_2)_2$, J = 7.2 Hz], 2.72 d (1H, CHN, J = 8.0, 7.1 Hz), 3.47 d (1H, C**H**OH, J = 8.3, 7.3 Hz), 3.56 br.s (1H, OH), 3.62 t [4H, O$(CH_2)_2$, J = 7.3 Hz]. Found, %: C 71.68; H 11.22; N 5.0. $C_{16}H_{31}NO_2$. Calculated, %: C 71.38; H 11.52; N 5.2.

6-(Diethylamino)decahydro-1,4-ethanonaphthalen-5-ol (6a) and 3-(diethylamino)decahydro-1,4-ethanonaphthalen-2-ol (6′a) (mixture of regioisomers at a ratio of 88:12) were synthesized from 6.48 g (25 mmol) of **4** and 3.65 g (50 mmol) of diethylamine. Yield 5.4 g (86.1%), mp 117–119°C. IR spectrum, ν, cm^{-1}: 3496 (OH), 3321 (NH), 3256 (NH), 2960 (CH_3), 2862–2856 (CH_2, sym.), 1658 (CN), 1460 (δCH_2, asym.), 1128, 1110 (δOH). ^{1}H NMR spectrum, δ, ppm: 1.05 t (6H, CH_3, J = 8.1 Hz), 1.31–1.63 m (16H, CH_2), 2.44 d [4H, N$(CH_2)_2$, J = 8.2, 7.0 Hz], 2.68 t (1H, CHN, J = 9.8 Hz), 3.42 d (1H, C**H**OH, J = 8.2, 7.1 Hz), 3.63 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 72.3 (C^5), 68.7 (C^6), 50.7 (C^4, C^7), 42.4 (C^9), 30.7 (C^{10}), 29.5 (C^{11}), 24.7 (C^8), 24.5 (C^1, C^2, C^{12}), 24.3 (C^7), 28.7 (C^1, C^2), 19.5 (C^3, C^4). Found, %: C 75.71; H 11.07; N 5.34. $C_{16}H_{29}NO$. Calculated, %: C 76.49; H 11.55; N 5.58.

6-(Butylamino)decahydro-1,4-ethanonaphthalen-5-ol (6b) and 3-(butylamino)decahydro-1,4-ethanonaphthalen-2-ol (6′b) (mixture of regioisomers at a ratio of 92.6:7.4) were synthesized from 6.48 g (25 mmol) of **4** and 3.65 g (50 mmol) of butan-1-amine. Yield 5.17 g (82.5%), mp 123–126°C. IR spectrum, ν, cm^{-1}: 3496 (OH), 3321 (N–H), 3258 (N–H), 2960 (CH_3, sym.), 2865–2856 (CH_2, sym.), 1528 (δN–H), 1460 (CH_2, asym.), 1128, 1111 (δOH). ^{1}H NMR spectrum, δ, ppm: 0.93 t (3H, CH_3, J = 8.1 Hz), 1.29–1.68 m (20H, CH_2), 2.3 s (1H, NH), 2.57 d (1H, C**H**NH, J = 9.7, 7.3 Hz), 2.60 d (2H, NCH_2, J = 9.4, 7.3 Hz), 3.43 d (1H, C**H**OH, J = 9.6, 7.2 Hz), 3.63 br.s (1H, OH). Found, %: C 75.92; H 11.12; N 5.21. $C_{16}H_{29}NO$. Calculated, %: C 76.49; H 11.55; N 5.58.

6-(Piperidin-1-yl)decahydro-1,4-ethanonaphthalen-5-ol (6c) and 3-(piperidin-1-yl)decahydro-1,4-ethanonaphthalen-2-ol (6′c) (mixture of regioisomers at a ratio of 92.6:7.4) were synthesized from 6.48 g (25 mmol) of **4** and 4.25 g (50 mmol) of piperidine. Yield 5.48 g (83.4%), mp 143–145°C. IR spectrum, ν, cm^{-1}: 3496 (OH), 3321, 3258 (CN), 2865–2856 (CH_2, sym.), 1528 (CN), 1460 (δCH_2, asym.), 1128, 1111 (δOH). ^{1}H NMR spectrum, δ, ppm: 1.30–1.63 m (22H, CH_2), 2.48 t [4H, N$(CH_2)_2$, J = 7.2 Hz], 2.69 d [1H, C**H**N$(CH_2)_2$, J = 9.8, 7.2 Hz], 3.42 d (1H, C**H**OH, J = 9.8, 7.3 Hz), 3.63 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 76.5 (C^6), 70.8 (C^5), 40.2 (C^9), 34.8 (C^4), 29.8 (C^3), 28.8 (C^{10}), 24.7 (C^1, C^2), 24.5 (C^7, C^8, C^{11}, C^{12}), 54.8 (NCH_2), 26.4 ($C^{3'}$, $C^{5'}$), 24.7 ($C^{4'}$). Found, %: C 76.93; H 9.94; N 5.0. $C_{17}H_{29}NO$. Calculated, %: C 77.57; H 11.03; N 5.32.

6-(Morpholin-4-yl)decahydro-1,4-ethanonaphthalen-5-ol (6d) and 3-(morpholin-4-yl)decahydro-1,4-ethanonaphthalen-2-ol (6′d) (mixture of regioisomers at a ratio of 85:15) were synthesized from 6.47 g (25 mmol) of **4** and 4.35 g (50 mmol) of morpholine. Yield 5.1 g (77.0%), mp 157–159°C. IR spectrum, ν, cm^{-1}: 3495 (OH), 3320, 3256 (CN), 1526 (CN), 1462 (δCH_2, asym.), 1240, 1123 (C–O–C), 1110, 1082 (δOH). ^{1}H NMR spectrum, δ, ppm: 1.28–1.62 m (16H, CH_2), 2.70 d (1H, C**H**N$(CH_2)_2$, J = 9.8, 7.2 Hz], 2.69 t [4H, N$(CH_2)_2$, J = 7.2 Hz], 3.42 d (1H, C**H**OH, J = 97.2 Hz), 3.58 br.s (1H, OH), 3.63 t [4H, O$(CH_2)_2$, J = 7.2 Hz]. ^{13}C NMR spectrum, δ_C, ppm: 76.2 (C^6), 70.8 (C^5), 40.3 (C^9), 34.9 (C^4), 28.9 (C^3, C^{10}), 24.7 (C^{12}), 24.6 (C^1, C^2, C^7, C^8, C^{11}), 67.4 (OCH_2), 52.5 (NCH_2). Found, %: C 71.92; H 10.06; N 5.12. $C_{16}H_{27}NO_2$. Calculated, %: C 72.45; H 10.19; N 5.28.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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