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A Convenient Procedure for Preparation of 1-(1-Aminoalkyl)-1-cyclopropanols from *N*-Benzyl α-Amino Acid Esters

I. L. Lysenko and O. G. Kulinkovich

Byelorussian State University, pr. Skoriny 4, Minsk, 220050 Belarus

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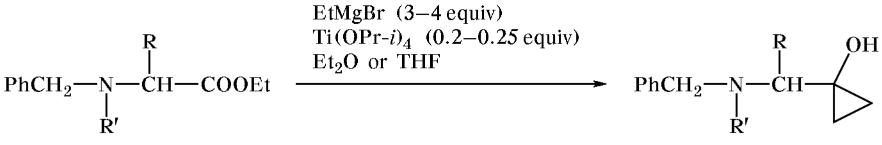
Abstract—The reaction of *N*-benzyl α-amino acid ethyl esters with ethylmagnesium bromide in the presence of a catalytic amount of titanium tetraisopropoxide leads to formation of the corresponding 1-aminoalkyl-1-cyclopropanols in high yields. Hydrogenation of the latter over palladium catalyst ensures selective removal of one or two protecting benzyl groups from the nitrogen atom.

1-Aminoalkyl-substituted cyclopropanols and their derivatives exhibit various kinds of biological activity. For example, an opioid χ-agonist Bremazocine [1] containing an 1-aminomethylcyclopropanol moiety is capable of increasing the concentration of corticosterol in blood plasma [2], and 2-amino-2-(1-hydroxycyclopropyl)acetic acid (Cleonine) is a component of the natural antibiotic Cleomycin [3]. 1-Aminoalkyl-1-cyclopropanols are usually synthesized by the action of primary or secondary amines on oxaspiropentane [4] or via transformations of cyclopropanone derivatives [5]. In the present communication we describe an efficient procedure for preparation of 1-(1-aminoalkyl)-1-cyclopropanols from N-substituted α-amino acid esters and ethylmagnesium bromide in the presence of titanium tetraisopropoxide [6]. This reaction was successfully used previously in the synthesis of substituted 1-alkylcyclopropanols containing alkoxide and acetal moieties [7], as well as of those possessing halogen atoms in the alkyl group [8]. There are published data on intramolecular cyclopropanation of *N*-allyl α-amino acid esters [9].

N,*N*-Dibenzylglycine ethyl ester (**Ia**) and ethyl esters derived from chiral (**Ib**–**Id**) and racemic amino acids (**Ie**–**Ig**) were brought into reaction with an ether solution of 3 equiv of ethylmagnesium bromide containing 0.2 equiv of titanium tetraisopropoxide [6]. As a result, the corresponding 1-(1-dibenzylaminoalkyl)-1-cyclopropanols (**IIa**–**IIg**) were formed in high yield (Scheme 1). When the reaction was performed with a smaller amount of ethylmagnesium bromide or catalyst, the yield of the target product diminished because of reduced conversion or formation of diethylcarbinols via addition of ethylmagnesium bromide at the ester group. The yields of compounds **IIa** and **IIe** from *N*,*N*-dibenzylglycine and *N*,*N*-dibenzyl-α-alanine isopropyl esters were lower by 10–15%, and a part of the initial ester remained unchanged.

We have developed a convenient (from the preparative viewpoint) procedure for isolation of 1-(1-dibenzylaminoalkyl)-1-cyclopropanols **IIa**–**IIg**, which allowed us to obtain sufficiently pure products without chromatographic purification. When the reaction was complete, the mixture was treated with 2 M hydrochloric acid at 0°C, 5 equiv of the acid per 1 equiv of the initial ester being taken. As a result, 1-(1-dibenzylaminoalkyl)-1-cyclopropanols **IIa**–**IIg** in the form of the corresponding hydrochlorides (which are insoluble in diethyl ether and water) were separated as an oily material which crystallized in some cases. Solid hydrochlorides of **IIa**, **IIc**–**IIe**, and **IIg** were filtered off and washed with a small amount of 1 M hydrochloric acid and with ether. Free bases **IIa**, **IIc**–**IIe**, and **IIg** were isolated by treatment of the corresponding hydrochlorides with a solution of sodium hydrogen carbonate, followed by extraction with methylene chloride; the extract was dried and evaporated. The yield of 1-(1-dibenzylaminoalkyl)-1-cyclopropanols **IIa**, **IIc**–**IIe**, and **IIg** with a purity of no less than 95% (according to the ^{1}H NMR data) was about 80%. Noncrystallizable hydrochlorides of **IIb**, **IId**, and **IIf** were dissolved in methylene chloride and were then treated in a similar way. In this case products **IIb**–**IIg** contained more than 90% of the main substance (^{1}H NMR). 1-(1-Dibenzylaminoalkyl)-1-cyclopropanols **IIa**–**IIg** are viscous oily

Scheme 1.



Ia–Ii **IIa–IIi**

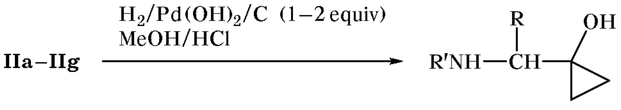
Ia, **IIa**, R = H, R′ = PhCH$_2$; (*S*)-**Ib**, (*S*)-**IIb**, R = CH$_3$, R′ = PhCH$_2$; (*S*)-**Ic**, (*S*)-**IIc**, R = *iso*-C$_3$H$_7$, R′ = PhCH$_2$; (*S*)-**Id**, (*S*)-**IId**, R = *iso*-C$_4$H$_9$, R′ = PhCH$_2$; *rac*-**Ie**, *rac*-**IIe**, R = C$_2$H$_5$, R′ = PhCH$_2$; *rac*-**If**, *rac*-**IIf**, R = CH$_3$, R′ = PhCH$_2$; *rac*-**Ig**, *rac*-**IIg**, R = *iso*-C$_3$H$_7$, R′ = PhCH$_2$; *rac*-**Ih**, *rac*-**IIh**, RR′ = (CH$_2$)$_3$; *rac*-**Ii**, *rac*-**IIi**, RR′ = (CH$_2$)$_4$.

substances which turn dark on exposure to air. Their yields and physical properties are summarized in Table 1. Stretching vibrations of the hydroxy group in the IR spectra of compounds **IIa**–**IIg** were observed in the region 3300–3600 cm^{-1}.

However, we failed to apply the same procedure to the synthesis of cyclopropanols from *N*-benzylproline ethyl ester (**Ih**) and *N*-benzylpipecolic acid ethyl ester (**Ii**). Under similar conditions we obtained complex mixtures of products, presumably due to adsorption of the catalyst on abundant precipitate liberated during the process. We succeeded in avoiding separation of the solid product by carrying out the reaction in boiling tetrahydrofuran with 4 equiv of ethylmagnesium bromide and 0.25 equiv of titanium tetraisopropoxide. Compounds **IIh** and **IIi** were isolated by column chromatography on silica gel using benzene–ether as eluent; these are viscous yellowish oily substances which solidify on cooling. The IR spectra of **IIh** and **IIi** contain characteristic absorption bands of the hydroxy group at 3400–3500 cm^{-1}. The yields, ^{1}H NMR spectral data, and elemental analyses of cyclopropanols **IIh** and **IIi** are given in Table 1.

1-(1-Dibenzylaminoalkyl)-1-cyclopropanols **IIa**–**IIe** were deprotected by catalytic hydrogenation over palladium hydroxide on charcoal [10]. The reaction was complete after absorption of 2 equiv of hydrogen, and 1-(1-aminoalkyl)-1-cyclopropanols **IIIa**–**IIIe** were formed in quantitative yield (Scheme 2). In the hydrogenation of crude hydrochlorides of 1-(1-dibenzylaminoalkyl)-1-cyclopropanols **IIa**–**IIg**, which was performed under the same conditions, only 1 equiv of hydrogen was absorbed. Removal of the solvent, followed by treatment with alkali, gave 1-(1-benzylaminoalkyl)-1-cyclopropanols **IVa**–**IVg** as the only product. The structure of aminoalkylcyclopropanols **III** and **IV** was proved by ^{1}H NMR spectroscopy (signals of protons of the cyclopropane ring were observed in the region δ 0.2–0.8 ppm), IR spectroscopy (broad absorption bands of the OH and NH groups in the region 3200–3500 cm^{-1}), and elemental analysis (Table 2).

Scheme 2.



IIIa–IIIe, IVa–IVg

IIIa, R = R′ = H; (*S*)-**IIIb**, R = CH$_3$, R′ = H; (*S*)-**IIIc**, R = *iso*-C$_3$H$_7$, R′ = H; (*S*)-**IIId**, R = *iso*-C$_4$H$_9$, R′ = H; *rac*-**IIIe**, R = C$_2$H$_5$, R′ = H; **IVa**, R = H, R′ = PhCH$_2$; (*S*)-**IVb**, R = CH$_3$, R′ = PhCH$_2$; (*S*)-**IVc**, R = *iso*-C$_3$H$_7$, R′ = PhCH$_2$; (*S*)-**IVd**, R = *iso*-C$_4$H$_9$, R′ = PhCH$_2$; *rac*-**IVe**, R = C$_2$H$_5$, R′ = PhCH$_2$; *rac*-**IVf**, R = CH$_3$, R′ = PhCH$_2$; *rac*-**IVg**, R = *iso*-C$_3$H$_7$, R′ = PhCH$_2$.

Thus, the cyclopropanation of *N*,*N*-dibenzyl α-amino acid esters **Ia**–**Ig** by the action of ethylmagnesium bromide in the presence of titanium tetraisopropoxide with subsequent removal of the benzyl protection is an efficient method for preparation of both racemic and optically active 1-(1-aminoalkyl)-1-cyclopropanols **IIIa**–**IIIe**. The procedure is also applicable to the synthesis of cyclopropanols from heterocyclic amino acids.

EXPERIMENTAL

The ^{1}H NMR spectra of compounds **II**–**IV** as solutions in CDCl$_3$ were recorded on a Bruker AC-200 spectrometer at 200 MHz. The IR spectra of compounds **IIIa** and **IVa** were measured on a Specord 75IR instrument from solutions in chloroform; compounds **IIa**–**IIi**, **IIIb**–**IIIe**, **IVa**, **IVb**, **IVd**, and **IVe** were examined as thin films using a Perkin–Elmer 1000 FTIR spectrometer. Diethyl ether and tetrahydrofuran were dried and distilled over metallic sodium. *N*,*N*-Dibenzyl amino acid ethyl esters **Ia**–**Ig** [11], *N*-benzylproline ethyl ester (**Ih**) [12], and *N*-benzyl-

Table 1. Yields, spectral parameters, and elemental analyses of 1-(1-aminoalkyl)-1-cyclopropanols **IIa**–**IIi**

Comp. no.	Yield, %	$[\alpha]_D^{17}$,[a] deg	^{1}H NMR spectrum, δ, ppm (*J*, Hz)	Found, %		Formula	Calculated, %	
				C	H		C	H
IIa	90	–	0.32–0.42 m (2H), 0.72–0.82 m (2H), 2.63 s (2H), 3.40 br.s (1H), 3.72 s (4H), 7.20–7.40 m (10H)	80.76	8.87	$C_{18}H_{21}NO$	80.86	7.92
IIb	88	–40	0.28–0.40 m (1H), 0.64–0.72 m (3H), 0.84 d (3H, 7), 3.26 q (1H, 7), 3.36 d (2H, 14), 4.00 d (2H, 14), 7.14–7.44 m (10H)	81.30	8.33	$C_{19}H_{23}NO$	81.10	8.24
IIc	92	–14	0.22–0.38 m (1H), 0.48–0.64 m (1H), 0.72–1.00 m (2H), 1.02 d (3H, 7), 1.16 d (3H, 7), 1.78 d (1H, 10), 2.20–2.48 m (1H), 3.5 br.s (1H), 3.88 d (2H, 14), 4.12 d (2H, 14), 7.14–7.48 m (10H)	81.45	8.88	$C_{21}H_{27}NO$	81.51	8.79
IId	95	23	0.46–0.82 m (4H), 0.84 d (3H, 6), 0.92 d (3H, 6), 0.94–1.10 m (1H), 1.50– 1.80 m (2H), 2.82 d.d (1H, 7. 6), 3.5 br.s (1H), 3.57 d (2H, 14), 3.97 d (2H, 14), 7.18–7.36 m (10H)	81.73	8.98	$C_{22}H_{29}NO$	81.69	9.04
IIe	85	–	0.46–0.70 m (3H), 0.72–0.82 m (1H), 1.02 t (3H, 7.5), 1.12–1.36 m (1H), 1.64–1.90 m (1H), 2.68 t (1H, 6.5), 3.18–3.40 s (1H), 3.64 d (2H, 14), 4.00 d (2H, 14), 7.16–7.38 m (10H)	81.20	8.62	$C_{20}H_{25}NO$	81.31	8.53
IIf	90	–	0.28–0.40 m (1H), 0.64–0.72 m (3H), 0.84 d (3H, 7), 3.26 q (1H, 7), 3.36 d (2H, 14), 4.00 d (2H, 14), 17.14–7.44 m (10H)	80.98	8.27	$C_{19}H_{23}NO$	81.10	8.24
IIg	92	–	0.22–0.38 m (1H), 0.48–0.64 m (1H), 0.72–1.00 m (2H), 1.02 d (3H, 7), 1.16 d (3H, 7), 1.78 d (1H, 10), 2.20–2.48 m (1H), 3.5 br.s (1H), 3.88 d (2H, 14), 4.12 d (2H, 14), 7.14–7.48 m (10H)	81.60	8.67	$C_{21}H_{27}NO$	81.51	8.79
IIh[b]	45	–	0.28–0.42 m (1H), 0.42–0.54 m (1H), 0.74–0.92 m (2H), 1.60–2.20 m (5H), 2.20–2.36 m (1H), 2.96–3.08 m (1H), 3.30 d (1H, 13), 3.54 br.s (1H), 4.50 d (1H, 13), 7.20–7.42 m (5H)	77.59	8.70	$C_{14}H_{19}NO$	77.38	8.81
IIi[b]	60	–	0.12–0.20 m (1H), 0.32–0.46 m (1H), 0.48–0.86 m (1H), 0.86–1.08 m (1H),1.15–1.64 m (4H), 1.72–1.98 m (4H), 3.84–3.96 m (1H), 2.90 d (1H, 13), 3.24 br.s (1H), 4.74 d (1H, 14), 7.20–7.42 m (5H)	77.95	9.25	$C_{15}H_{21}NO$	77.88	9.15

[a] *c* = 10, Et_2O.

[b] The reaction was carried out in boiling tetrahydrofuran using 4 equiv of EtMgBr and 0.25 equiv of $Ti(OPr\text{-}i)_4$.

Table 2. Yields, spectral parameters, and elemental analyses of 1-(1-aminoalkyl)-1-cyclopropanols **IIIa**–**IIIe** and **IVa**–**IVg**

Comp. no.	Yield, %	$[\alpha]_D^{17}$, deg	^{1}H NMR spectrum, δ, ppm (*J*, Hz)	Found, %		Formula	Calculated, %	
				C	H		C	H
IIIa	95	–	0.42–0.52 m (2H), 0.72–0.82 m (2H), 2.76 s (2H), 3.46 br.s (3H)	55.02	10.47	C_4H_9NO	55.15	10.41
IIIb	95	–5 (*c* = 4, MeOH)	0.32–0.42 m (2H), 0.60–0.70 m (2H), 1.06 d (3H, 7), 2.38 q (1H, 7), 2.68 br.s (3H)	59.24	11.09	$C_5H_{11}NO$	59.37	10.96
IIIc[a]	95	18 (*c* = 4, MeOH)	0.20–0.38 m (1H), 0.50–6.04 m (2H), 0.82–1.00 m (1H), 0.96 d (3H, 7), 1.02 d (3H, 7), 1.78 d (1H, 9), 1.80–2.04 m (1H), 2.80 br.s (3H)	65.25	11.76	$C_7H_{15}NO$	65.07	11.70
IIId	98	–7 (*c* = 3.7, MeOH)	0.32–0.50 m (2H), 0.6–0.7 m (1H), 0.7–0.8 m (1H), 0.86 d (3H, 7), 0.92 d (3H, 7), 1.40 t (2H, 7), 1.58–1.82 m (1H), 2.28 t (1H, 7), 2.44 br.s (3H)	66.86	12.10	$C_8H_{17}NO$	67.09	11.96
IIIe	95	–	0.36–0.60 m (2H), 0.66–0.92 m (2H), 1.00 t (3H, 7.5), 1.40–1.64 m (1H), 1.64–1.86 m (1H), 2.23 d.d (1H, 7.5, 6.5), 3.4 br.s (3H)	62.81	11.45	$C_6H_{13}NO$	62.57	11.38
IVa[b]	90	–	0.36–0.48 m (2H), 0.70–0.82 m (2H), 2.70 s (2H), 3.22 br.s (2H), 3.80 s (2H), 7.22–7.38 m (5H)	74.69	8.48	$C_{11}H_{15}NO$	74.54	8.53
IVb	98	–14.2 (*c* = 10, Et_2O)	0.26–0.38 m (1H), 0.38–0. 50 m (1H), 0. 70–0.82 m (2H), 1.10 d (3H, 6. 5), 2.28 q (1H, 6.5), 2.40–3.00 s (2H), 3.82 d (2H, 13), 3.90 d (2H, 13), 7.20–7.40 m (5H)	75.25	8.89	$C_{12}H_{17}NO$	75.35	8.96
IVc	90	–34.4 (*c* = 5, CH_2Cl_2)	0.34–0.58 m (2H), 0.66–0.78 m (1H), 0.86–1.00 m (1H), 1.04 d (3H, 7), 1.06 d (3H, 7), 1.84 d (1H, 7), 1.98 m (1H), 2.40 br.s (2H), 3.78 d (1H, 13), 4.08 d (1H, 13), 7.20–7.40 m (5H)	76.70	9.70	$C_{14}H_{21}NO$	76.67	9.65
IVd	95	–22 (*c* = 10, Et_2O)	0.24–0.40 m (1H), 0.40–0.54 m (1H), 0.70–0.86 m (5H), 0.92 d (3H, 6.5), 1.36–1.76 m (3H), 2.11 d.d (1H, 8.5, 5), 2.4 br.s (2H), 3.77 d (1H, 14), 4.03 d (1H, 14), 7.2–7.36 m (5H)	77.15	10.05	$C_{15}H_{23}NO$	77.21	9.93
IVe	90	–	0.32–0.46 m (2H), 0.70–0.90 m (2H), 0.96 t (3H, 7.5), 1.46–1.86 m (2H), 1.98 d.d (1H, 8, 6), 3.80 d (2H, 14) 4.00 d (2H, 14), 7.20–7.40 m (5H)	75.94	9.25	$C_{13}H_{19}NO$	76.06	9.33

Table 2. (Contd.)

Comp. no.	Yield, %	$[\alpha]_D^{17}$, deg	^{1}H NMR spectrum, δ, ppm (*J*, Hz)	Found, %		Formula	Calculated, %	
				C	H		C	H
IVf	93	–	0.26–0.38 m (1H), 0.38–0. 50 m (1H), 0. 70–0.82 m (2H), 1.10 d (3H, 6. 5), 2.28 q (1H, 6.5), 2.40–3.00 s (2H), 3.82 d (2H, 13), 3.90 d (2H, 13), 7.20–7.40 m (5H)	75.12	9.05	$C_{12}H_{17}NO$	75.35	8.96
IVg	90	–	0.34–0.58 m (2H), 0.66–0.78 m (1H), 0.86–1.00 m (1H), 1.04 d (3H, 7), 1.06 d (3H, 7), 1.84 d (1H, 7), 1.98 m (1H), 2.40 br.s (2H), 3.78 d (1H, 13), 4.08 d (1H, 13), 7.20–7.40 m (5H)	76.82	9.58	$C_{14}H_{21}NO$	76.67	9.65

[a] mp 54–56°C (from hexane–ether).

[b] mp 47–48°C (from hexane–ether).

pipecolic acid ethyl ester (**Ii**) [13] were synthesized by known methods.

1-(1-Dibenzylaminoalkyl)-1-cyclopropanols IIa–IIg. To a solution of 5 mmol of ester **Ia**–**Ig** and 0.28 ml (1 mmol) of titanium tetraisopropoxide in 15 ml of diethyl ether we added with stirring at room temperature 15 ml of a 1 M solution of ethylmagnesium bromide (over a period of 40–60 min). The mixture was left overnight, cooled to 0°C, treated with 12 ml of 2 M hydrochloric acid, and stirred for 2–4 h at room temperature. When oily amino alcohol hydrochlorides (compounds **IIb** and **IIf**) separated from the mixture, the layers were allowed to settle down, and the ether layer was decanted. The oily material was dissolved in 10 ml of methylene chloride, and the aqueous phase was additionally treated with 2×10 ml of methylene chloride. The methylene chloride extracts containing crude hydrochlorides of **IIb** and **IIf** were combined, washed with 5 ml of a saturated aqueous solution of sodium chloride and 5 ml of a saturated aqueous solution of sodium hydrogen carbonate and dried over anhydrous sodium sulfate. Removal of the solvent under reduced pressure gave aminoalkylcyclopropanols **IIb** and **IIf**. Oily hydrochlorides of amino alcohols **IIa**, **IIc**–**IIe**, and **IIg** crystallized; the precipitate was filtered off, washed on a filter with 5 ml of 1 M hydrochloric acid and 5 ml of diethyl ether, and dried in air. The solid hydrochlorides were treated with 5 ml of aqueous sodium hydrogen carbonate, free bases **IIa**, **IIc**–**IIe**, and **IIg** were extracted into methylene chloride, the extract was dried over anhydrous sodium sulfate, and the solvent was distilled off. The yields, spectral parameters, and elemental analyses of 1-(1-dibenzylaminoalkyl)-1-cyclopropanols **IIa**–**IIg** are given in Table 1.

1-(1-Benzyltetrahydro-1*H*-2-pyrrolyl)-1-cyclopropanol (IIh) and 1-(1-benzyl-2-piperidinyl)-1-cyclopropanol (IIi). A solution of 5 mmol of *N*-benzyl amino acid ester **Ih** or **Ii** and 0.35 ml (1.25 mmol) of titanium tetraisopropoxide in 15 ml of tetrahydrofuran was heated to the boiling point, and 20 ml of a 1 M solution of ethylmagnesium bromide in tetrahydrofuran was added with stirring over a period of 40–60 min. The mixture was refluxed for an additional 1 h while stirring. The solvent was distilled off under reduced pressure, and the residue was treated with 18 ml of 2 M hydrochloric acid on cooling to 0°C. The mixture was stirred for 2 h, the organic phase was separated, and the aqueous layer was saturated with solid sodium chloride and extracted with methylene chloride (3×10 ml). The combined organic extracts were washed with 5 ml of a saturated solution of sodium hydrogen carbonate, dried over magnesium sulfate, and evaporated. The residue was subjected to column chromatography on silica gel using benzene–diethyl ether as eluent. The yields, spectral parameters, and elemental analyses of compounds **IIh** and **IIi** are given in Table 1.

1-(1-Aminoalkyl)-1-cyclopropanols IIIa–IIIe were obtained by hydrogenation of 5–20 mmol of 1-(1-dibenzylaminoalkyl)-1-cyclopropanols **IIa**–**IIe** in the presence of $Pd(OH)_2/C$, following the procedure described in [10]. Products **IIIa**–**IIIe** were quickly distilled under reduced pressure (oil pump). The yields, spectral parameters, and elemental analyses of aminoalkylcyclopropanols **IIIa**–**IIIe** are summarized in Table 2.

1-(1-Benzylaminoalkyl)-1-cyclopropanols IVa–IVg. Methanol, 10–100 ml, was added to 5–50 mmol of crude hydrochloride of **IIa**–**IIg**, obtained after removal of the solvent from the methylene chloride extract (see above). The catalyst (20% of palladium hydroxide on charcoal, 10 wt % of the initial hydrochloride) was added, and the mixture was stirred under hydrogen (atmospheric pressure). When hydrogen was no longer absorbed, the catalyst was filtered off and washed with methanol on a filter. The filtrate was evaporated, and the residue was neutralized with aqueous ammonia and extracted with ether. The extract was dried over anhydrous sodium sulfate and evaporated to leave products **IVb**–**IVg** as yellowish oily substances which turned dark on storage; amino alcohol **IVa** was isolated as a low-melting crystalline substance. Table 2 lists the yields, spectral parameters, and elemental analyses of 1-(1-benzylaminoalkyl)-1-cyclopropanols **IVa**–**IVg**.

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