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Synthesis of Nonracemic Polysubstituted Cyclohexanes through Cascade Reactions of (2*R*,3*S*)-2-Acetyl-3-aryl-4-nitrobutanoates, Adducts of Ni(II)-Catalyzed Michael Addition

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Abstract—Stereoselective synthesis of ethyl-4,6-diaryl-2-hydroxy-2-methyl-5-nitro-3-formylcyclohexanecarboxylates was described. The formation of the asymmetric site of the required configuration is achieved by an enantioselective Michael addition of ethyl acetoacetate to nitroalkenes in the presence of a chiral Ni(II) complex with (*R,R*)-*N,N'*-dibenzylcyclohexane-1,2-diamine. Further reaction of the products obtained with cinnamic aldehyde led to the formation of polysubstituted cyclohexanes.

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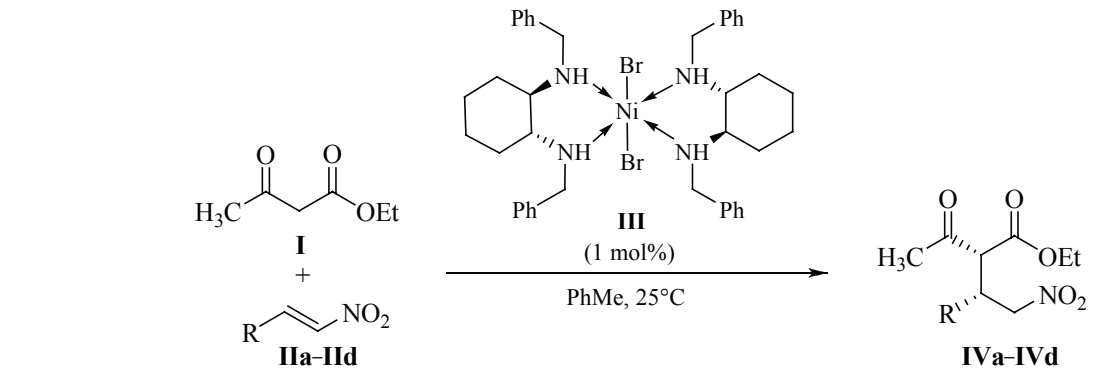
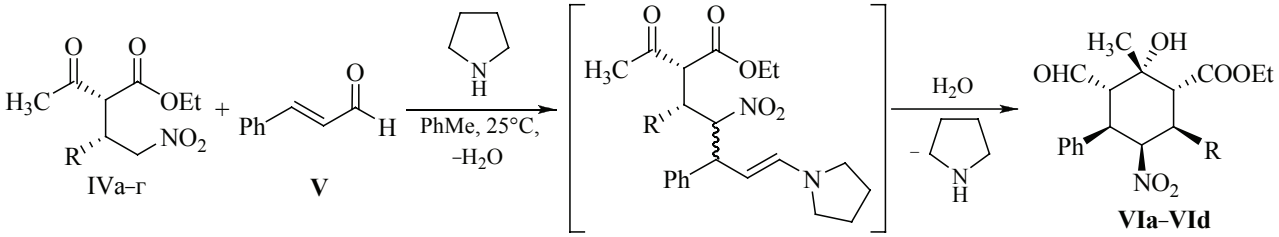
The utilization of cascade reactions in the asymmetric synthesis of polysubstituted alicyclic compounds with several stereocenters is lately a vigorously growing synthetic strategy for the preparation of biologically active compounds and chiral building blocks [1]. Several examples were described of the synthesis of functionally substituted chiral cyclohexane derivatives via cascade reactions of adducts of alkyl acetoacetate and nitroalkenes with the use of iminium-enamine activation [2]. The key target in this case is the building of a chiral center of the desired configuration governing the stereodirection of the subsequent processes. In this event the organic catalysis is applied with chiral thiourea derivatives [2].

In [3] the enantioselective Michael addition of β-ketoesters to nitroalkenes catalyzed by chiral Ni(II) complexes was described for the first time. Ni(II) complexes with diamine ligands were successfully used in reactions of 1,3-dicarbonyl compounds with various unsaturated nitro derivatives [4]. We formerly studied the catalytic activity of complexes of various metals in the asymmetric Michael reaction [5]. Synthetic approaches were developed to the preparation of (*R*)-rolipram, (*R*)-phenotropil, and 4-amino-(3*R*)-methyl-butanoic acid [6], underlain by the addition of diethyl malonate to nitroalkenes catalyzed by the Ni(II) complex with *N,N'*-dibenzylcyclohexane-1,2-diamine.

The approach we developed to the stereoselective synthesis of polyfunctional derivatives of cyclohexane **VIa–VId** is based on the use of successive reactions of Ni-catalyzed Michael addition of ethyl acetoacetate (**I**) to nitroalkenes **IIa–IId** with the subsequent cascade transformation (Michael addition–aldol condensation) of the obtained chiral adducts **IVa–IVd** with cinnamic aldehyde (**V**) under the conditions of pyrrolidine catalysis.

The addition of ethyl acetoacetate (**I**) to nitroalkenes **IIa–IId** in the presence of complex **III** affords adducts **IVa–IVd** with the prevailing formation of (3*S*)-enantiomers [3] and *dr* ~1 : 1. By the subsequent recrystallization from the mixture toluene–petroleum ether we isolated individual diastereomers **IVa–IVd** (*de* 86–96%). The ratio of diastereomers was determined by the analysis of the ^{1}H NMR spectra of adducts **IVa–IVd**, where the chemical shifts of the protons of methyl groups of two diastereomers differed by 0.23–0.27 ppm. The spectra contain two triplets of the methyl protons of the ester groups with the triplet of the main diastereomer shifted upfield, and two singlets of the methyl protons of the acetyl groups with the singlet of the main diastereomer shifted downfield. The assignment of the signals of diastereomers of compounds





R = Ph (**a**), 4-ClC_6H_4 (**b**), 4-$MeOC_6H_4$ (**c**), 4-$NO_2C_6H_4$ (**d**).

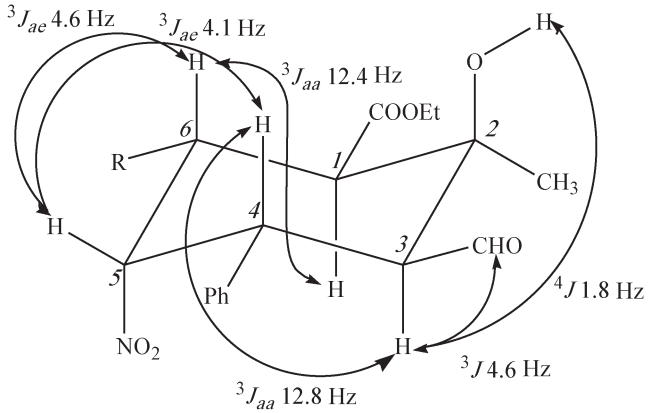
IVa–IVd and establishing their absolute configuration were performed based on the published data [3, 7]. The subsequent cascade reaction catalyzed by pyrrolidine consists in the Michael addition of the obtained adducts **IVa–IVd** to cinnamic aldehyde (**V**) followed by the intramolecular aldol condenation. The reaction results in the corresponding cyclohexanes **VIa–VId** as confirmed by the NMR spectra ^1H, ^{13}C, DEPT, and HMQC. Thus in the spectrum of the 2D experiment HMQC ^1H–^{13}C of compound **VIa** the cross-peak 4.30/41.6 ppm corresponds to the linked C–H atoms at the aryl group R, the cross-peak 4.00/44.3 ppm belongs to the linked C–H atoms at the phenyl group, the cross-peak 3.94/61.3 ppm is due to the correlation of the bound C–H atoms of the methylene group of the ester fragment, the cross-peaks 3.83/49.6 and 3.50/54.7 ppm correspond to the methane groups at the ester and formyl substituents, respectively. The spectrum contains also the cross-peak 5.02/93.1 ppm corresponding to the linked C–H atoms at the nitro group.

The relative configuration of compounds **VIa–VId** corresponds to the data on the stereochemistry of the reaction of methyl 2-acetyl-3-aryl-4-nitrobutanoates with the cinnamic aldehyde under similar conditions [2]. It is confirmed by the values of the vicinal spin-spin coupling constants in the ^1H NMR spectrum of compound **VId**.

The absolute configuration of cyclohexanes **VIa–VId** was established from the measured angles of the optical rotation and the published values for the methyl (1*S*,2*R*,3*R*,4*S*,5*R*,6*R*)-2-hydroxy-2-methyl-5-nitro-4,6-diphenyl-3-formylcyclohexanecarboxylate prepared from the methyl (3*R*)-2-acetyl-4-nitro-3-phenylbutanoate under analogous conditions [2].

EXPERIMENTAL

Mass spectra were measured on an instrument Thermo Finnigan Trace DSQ, capillary column BPX-5 (30 × 0.32 mm), ionizing electrons energy 70 eV. The optical rota-



R = 4-NO_2-C_6H_4.

tion was measured on an automatic polarimeter Rudolph Research Analytical Autopol V Plus. NMR spectra ^{1}H, ^{13}C, DEPT, and HMQC were registered on a spectrometer JEOL JNM-ECX400 [399.78 (^{1}H) and 100.53 MHz (^{13}C)] in $CDCl_3$. The signals of deuterated solvent served as internal reference. Elemental analysis was carried out on a CHNS-analyzer Euro Vector EA-3000.

Chiral catalyst **III** was prepared by procedure [3].

Ethyl (2*R*,3*S*)-2-acetyl-3-aryl-4-nitrobutanoates IVa–IVd. To a solution of 1 equiv. of nitroalkene **IIa–IId** and 2 equiv. of ethyl acetoacetate (**I**) in 30 mL of toluene was added 1 mol% of nickel complex **III**, and the mixture was maintained for 24 h at room temperature. Toluene and excess etyl acetoacetate were distilled off in a vacuum, the residue was dissolved in 20 mL of chloroform and was filtered through a silica gel bed. Chloroform was distilled off, the residue was recrystallized from a mixture of toluene–petroleum ether, 2 : 1, at –20°C.

Ethyl (2*R*,3*S*)-2-acetyl-4-nitro-3-phenylbutanoate (IVa). Yield 3.5 g (35%), *de* 98% (^{1}H NMR data), mp 72–75°C, $[\alpha]_D^{20}$ +146.6° (*c* 0.01 g mL^{-1}, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 0.99 t (3H, $CH_2\underline{CH}_3$, $^3J_{HH}$ 7.1 Hz), 2.29 s (3H, $COCH_3$), 3.95 q (2H, $\underline{CH}_2CH_3$, $^3J_{HH}$ 7.1 Hz), 4.11 d (1H, $\underline{CH}COOEt$, $^3J_{HH}$ 10.1 Hz), 4.16–4.22 m (1H, $\underline{CH}Ph$), 4.74 d (2H, $\underline{CH}_2NO_2$, $^3J_{HH}$ 6.1 Hz), 7.18–7.20 m ($2H_{arom}$), 7.24–7.32 m ($3H_{arom}$). ^{13}C NMR spectrum, δ, ppm: 13.77 ($CH_2\underline{CH}_3$), 30.22 ($CO\underline{CH}_3$), 42.39 ($\underline{CH}Ph$), 62.06 ($\underline{CH}COMe$), 62.09 ($\underline{CH}_2Me$), 77.98 (CH_2NO_2), 128.05 (C^o), 128.39 (C^p), 129.05 (C^m), 136.50 (C^i), 166.95 (COOEt), 201.28 (COMe). Mass spectrum, *m/z* (I_{rel}, %): 233 (4) $[M-NO_2]^+$, 189 (80), 159 (48), 145 (65), 115 (48), 91 (33), 77 (11), 43 (100). Found, %: C 60.89; H 6.41; N 4.78. $C_{14}H_{17}NO_5$. Calculated, %: C 60.21; H 6.14; N 5.01. m 279.29.

Ethyl (2*R*,3*S*)-2-acetyl-4-nitro-3-(4-chlorophenyl)butanoate (IVb). Yield 2.9 g (29%), *de* 92% (^{1}H NMR data), mp 79–82°C, $[\alpha]_D^{20}$ +115.7° (*c* 0.01 g mL^{-1}, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.04 t (3H, $CH_2\underline{CH}_3$, $^3J_{HH}$ 7.3 Hz), 2.29 s (3H, $COCH_3$), 3.98 q (2H, $\underline{CH}_2CH_3$, $^3J_{HH}$ 7.3 Hz), 4.07 d (1H, $\underline{CH}COOEt$, $^3J_{HH}$ 10.1 Hz), 4.14–4.22 m (1H, $C\underline{H}Ar$), 4.70 d (1H, CH_2NO_2, $^2J_{HH}$ 1.4 Hz), 4.72 s (1H, CH_2NO_2), 7.14 d (2H, H^o, $^3J_{HH}$ 8.5 Hz), 7.28 d (2H, H^m, $^3J_{HH}$ 8.5 Hz). ^{13}C NMR spectrum, δ, ppm: 13.83 ($CH_2\underline{CH}_3$), 30.15 ($CO\underline{CH}_3$), 41.75 ($\underline{CH}Ar$), 61.84 ($\underline{CH}COMe$), 62.24 ($\underline{CH}_2Me$), 77.76 (CH_2NO_2), 129.25 (C^o), 129.47 (C^m), 134.34 (C^i), 135.08 (C^i), 166.72 (COOEt), 200.88 (COMe). Mass spectrum, *m/z* (I_{rel}, %): 313 (1) $[M]^+$, 225 (78), 193 (46), 179 (42), 165 (12), 138 (24), 115 (25), 103 (12), 75 (7), 43 (100). Found, %: C 53.98; H 5.45; N 4.19. $C_{14}H_{16}ClNO_5$. Calculated, %: C 53.60; H 5.14; N 4.46. M 313.74.

Ethyl (2*R*,3*S*)-2-acetyl-3-(4-methoxyphenyl)-4-nitrobutanoate (IVc). Yield 4.1 g (41%), *de* 86% (^{1}H NMR data), mp 82–85°C, $[\alpha]_D^{20}$ +91.9° (*c* 0.01 g mL^{-1}, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.03 t (3H, $CH_2\underline{CH}_3$, $^3J_{HH}$ 7.3 Hz), 2.29 s (3H, $COCH_3$), 3.76 s (3H, OCH_3), 3.97 q (2H, $\underline{CH}_2CH_3$, $^3J_{HH}$ 7.1 Hz), 4.06 d (1H, $C\underline{H}COOEt$, $^3J_{HH}$ 10.1 Hz), 4.11–4.18 m (1H, $C\underline{H}Ar$), 4.70 d (1H, CH_2NO_2, $^2J_{HH}$ 2.1 Hz), 4.71 s (1H, CH_2NO_2), 6.82 d (2H, H^m, $^3J_{HH}$ 8.7 Hz), 7.11 d (2H, H^o, $^3J_{HH}$ 8.7 Hz). ^{13}C NMR spectrum, δ, ppm: 13.85 ($CH_2\underline{CH}_3$), 30.17 ($CO\underline{CH}_3$), 41.74 ($\underline{CH}Ar$), 55.30 ($O\underline{CH}_3$), 62.02 ($\underline{CH}_2Me$), 62.26 ($\underline{CH}COMe$), 78.20 (CH_2NO_2), 114.39 (C^m), 128.30 (C^i), 129.17 (C^o), 159.47 (C^i), 166.01 (COOEt), 201.36 (COMe). Found, %: C 58.65; H 6.32; N 4.24. $C_{15}H_{19}NO_6$. Calculated, %: C 58.25; H 6.19; N 4.53.

Ethyl (2*R*,3*S*)-2-acetyl-4-nitro-3-(4-nitrophenyl) butanoate (IVd). Yield 0.58 g (35%), *de* 96% (^{1}H NMR data), mp 102–106°C, $[\alpha]_D^{20}$ +102.1° (*c* 0.01 g mL^{-1}, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.05 t (3H, $CH_2\underline{CH}_3$, $^3J_{HH}$ 7.1 Hz), 2.28 s (3H, $COCH_3$), 3.99 q (1H, $\underline{CH}_2CH_3$, $^3J_{HH}$ 7.1 Hz), 4.004 q (1H, $\underline{CH}_2CH_3$, $^3J_{HH}$ 7.1 Hz), 4.13 d (1H, $\underline{CH}COOEt$, $^3J_{HH}$ 10.1 Hz), 4.29–4.35 m (1H, $C\underline{H}Ar$), 4.76 s (1H, CH_2NO_2), 4.78 d (1H, CH_2NO_2, $^2J_{HH}$ 2.1 Hz), 7.41 d (2H, H^o, $^3J_{HH}$ 8.7 Hz), 8.18 d (2H, H^m, $^3J_{HH}$ 8.7 Hz). ^{13}C NMR spectrum, δ, ppm: 13.88 ($CH_2\underline{CH}_3$), 30.18 ($CO\underline{CH}_3$), 41.94 ($\underline{CH}Ar$), 61.39 ($\underline{CH}COMe$), 62.50 ($\underline{CH}_2Me$), 77.29 (CH_2NO_2), 124.22 (C^m), 129.06 (C^o), 144.06 (C^i), 147.85 (C^i), 166.38 (COOEt), 200.32 (COMe). Found, %: C 54.38; H 5.30; N 8.21. $C_{14}H_{16}N_2O_7$. Calculated, %: C 51.85; H 4.97; N 8.64.

Ethyl (1*R*,2*S*,3*S*,4*R*,5*S*,6*S*)-4,6-diaryl-2-hydroxy-2-methyl-5-nitro-3-formylcyclohexanecarboxylates VIa–VId. To a solution of 1 equiv. of compound **IVa–IVd** in 10 mL of toluene was added 1.1 equiv. of cinnamic aldehyde (**V**) and 1 equiv. of pyrrolidine, the mixture was stirred at room temperature for 24 h. The separated precipitate was filtered off and recrystallized from methanol.

Ethyl (1*R*,2*S*,3*S*,4*R*,5*S*,6*S*)-2-hydroxy-2-methyl-5-nitro-4,6-diphenyl-3-formylcyclohexanecarboxylate (VIa). Yield 0.95 g (33%), mp 220–222°C (decomp.), $[\alpha]_D^{20}$ –13.8° (*c* 0.011 g mL^{-1}, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 0.89 t (3H,

$COOCH_2\underline{CH_3}$, $^3J_{HH}$ 7.1 Hz), 1.47 s (3H, CH_3), 3.52 d.d.d (1H, $\underline{CH}CHO$, $^3J_{HH}$ 12.6, $^3J_{HH}$ 3.0, $^4J_{HH}$ 1.6 Hz), 3.81–3.84 m (2H, $\underline{CH}COOEt$, OH), 3.88–4.02 m (3H, $COO\underline{CH_2}CH_3$, $\underline{CH}Ph$), 4.30 d.d (1H, $\underline{CH}Ph$, $^3J_{HH}$ 12.6, $^3J_{HH}$ 4.6 Hz), 5.03 t (1H, $CHNO_2$, $^3J_{HH}$ 4.6 Hz), 7.14–7.30 m ($10H_{arom}$), 9.51 d (1H, CHO, $^3J_{HH}$ 4.6 Hz). ^{13}C NMR spectrum, δ, ppm: 13.71 ($COOCH_2\underline{CH_3}$), 27.32 (CH_3), 41.80 (CHPh), 44.41 (CHPh), 49.82 ($\underline{CH}COOEt$), 54.80 ($\underline{CH}CHO$), 61.42 ($COO\underline{CH_2}CH_3$), 72.11 [$\underline{C}(CH_3)OH$], 93.33 ($CHNO_2$), 128.22 (C^o), 128.24 (C^o), 128.65 (C^p), 128.68 (C^p), 129.01 (C^m), 129.42 (C^m), 135.41 (C^i), 135.50 (C^i), 174.30 (COOEt), 202.9 (CHO). Found, %: C 66.58; H 6.04; N 3.84. $C_{23}H_{25}NO_6$. Calculated, %: C 67.14; H 6.12; N 3.40.

Ethyl (1*R*,2*S*,3*S*,4*R*,5*S*,6*S*)-2-hydroxy-2-methyl-5-nitro-4-phenyl-3-formyl-6-(4-chlorophenyl)cyclohexanecarboxylate (VIb). Yield 0.9 g (32%), mp 228–230°C (decomp.), $[\alpha]_D^{20}$ –10.18° (*c* 0.011 g mL^{-1}, $CHCl_3$). ^1H NMR spectrum, δ, ppm: 0.96 t (3H, $COOCH_2\underline{CH_3}$, $^3J_{HH}$ 7.1 Hz), 1.47 s (3H, CH_3), 3.47–3.51 d.d.d (1H, $\underline{CH}CHO$, $^3J_{HH}$ 12.6, $^3J_{HH}$ 4.4, $^4J_{HH}$ 1.6 Hz), 3.81–3.84 m (2H, $\underline{CH}COOEt$, OH), 3.88–4.02 m (3H, $COO\underline{CH_2}CH_3$, $\underline{CH}Ph$), 4.28 d.d (1H, CHAr, $^3J_{HH}$ 12.6, $^3J_{HH}$ 6.5 Hz), 5.00 t (1H, $CHNO_2$, $^3J_{HH}$ 4.3 Hz), 7.13–7.15 m ($4H_{arom}$), 7.24–7.30 m ($5H_{arom}$), 9.50 d (1H, CHO, $^3J_{HH}$ 4.4 Hz). ^{13}C NMR spectrum, δ, ppm: 13.80 ($COOCH_2\underline{CH_3}$), 27.20 (CH_3), 41.70 (CHAr), 43.71 (CHPh), 49.70 (CHCOOEt), 54.73 ($\underline{CH}CHO$), 61.62 ($COO\underline{CH_2}CH_3$), 72.10 [$\underline{C}(CH_3)OH$], 93.10 ($CHNO_2$), 128.20 (C^m), 128.70 (C^p), 129.20 (C^o), 129.40 (C^m) 129.50 (C^o), 134.10 (C^i), 134.62 (C^i), 135.21 (C^i), 173.91 (COOEt), 202.80 (CHO). Found, %: C 62.28; H 5.31; N 3.44. $C_{23}H_{24}ClNO_6$. Calculated, %: C 61.95; H 5.43; N 3.14.

Ethyl (1*R*,2*S*,3*S*,4*R*,5*S*,6*S*)-2-hydroxy-2-methyl-6-(4-methoxyphenyl)-5-nitro-4-phenyl-3-formylcyclohexanecarboxylate (VIc). Yield 0.93 g (33%), mp 165–166°C, $[\alpha]_D^{20}$ –7.45° (*c* 0.011 g mL^{-1}, $CHCl_3$). ^1H NMR spectrum, δ, ppm: 0.93 t (3H, $COOCH_2\underline{CH_3}$, $^3J_{HH}$ 7.1 Hz), 1.46 s (3H, CH_3), 3.46–3.51 m (1H, $\underline{CH}CHO$), 3.74 c (3H, OCH_3), 3.76–3.81 m (2H, $\underline{CH}COOEt$, OH), 3.91–3.98 m (3H, $COO\underline{CH_2}CH_3$, CHPh), 4.27 d.d (1H, $C\underline{H}Ar$, $^3J_{HH}$ 12.3, $^3J_{HH}$ 4.6 Hz), 5.00 t (1H, $CHNO_2$, $^3J_{HH}$ 4.3 Hz), 6.79 d (2H, CH^m, $^3J_{HH}$ 8.7 Hz), 7.09–7.15 m ($4H_{arom}$), 7.22–7.30 m ($3H_{arom}$), 9.50 d (1H, CHO, $^3J_{HH}$ 4.6 Hz). ^{13}C NMR spectrum, δ, ppm: 13.82 ($COOCH_2\underline{CH_3}$), 27.32 (CH_3), 41.71 ($\underline{C}HAr$), 43.62 ($\underline{C}HPh$), 50.00 (CHCOOEt), 54.71 ($\underline{CH}CHO$), 55.30 (OCH_3), 61.40 ($COO\underline{CH_2}CH_3$), 72.20 [$\underline{C}(CH_3)OH$], 93.51 ($CHNO_2$), 114.31 (C^m), 127.52 (C^i), 128.21 (C^m), 128.62 (C^p), 129.31 (C^m), 129.41 (CH^o), 135.41 (C^i), 159.61 (C^i), 174.42 (COOEt), 203.10 (CHO). Found, %: C 65.58; H 6.02; N 3.58. $C_{24}H_{27}NO_7$. Calculated, %: C 65.29; H 6.16; N 3.17.

Ethyl (1*R*,2*S*,3*S*,4*R*,5*S*,6*S*)-2-hydroxy-2-methyl-5-nitro-6-(4-nitrophenyl)-4-phenyl-3-formylcyclohexanecarboxylate (VId). Yield 0.08 g (20%), mp 242–245°C, $[\alpha]_D^{20}$ –11.1° (*c* 0.011 g mL^{-1}, $CHCl_3$). ^1H NMR spectrum, δ, ppm: 0.97 t (3H, $COOCH_2\underline{CH_3}$, $^3J_{HH}$ 7.3 Hz), 1.50 s (3H, CH_3), 3.52 d.d.d (1H, $\underline{CH}CHO$, $^3J_{HH}$ 12.8, $^3J_{HH}$ 4.5, $^4J_{HH}$ 1.8 Hz), 3.59 d (1H, OH, $^3J_{HH}$ 1.8 Hz), 3.85 d (1H, CHCOOEt, $^3J_{HH}$ 12.4 Hz), 3.98 q (1H, $COO\underline{CH_2}CH_3$, $^3J_{HH}$ 7.3 Hz), 3.98 q (1H, $COO\underline{CH_2}CH_3$, $^3J_{HH}$ 7.3 Hz), 4.16 d.d (1H, CHPh, $^3J_{HH}$ 12.8, $^3J_{HH}$ 4.1 Hz), 4.30 d.d (1H, CHAr, $^3J_{HH}$ 12.4, $^3J_{HH}$ 4.5 Hz), 5.02 t (1H, $CHNO_2$, $^3J_{HH}$ 4.5 Hz), 7.14–7.16 m (2H, CH^o), 7.27–7.32 m ($3H_{arom}$), 7.41 d (2H, H^o, $^3J_{HH}$ 8.7 Hz), 8.16 d (2H, H^m, $^3J_{HH}$ 8.7 Hz), 9.50 d (1H, CHO, $^3J_{HH}$ 4.6 Hz). ^{13}C NMR spectrum, δ, ppm: 13.93 ($COOCH_2\underline{CH_3}$), 27.17 (CH_3), 41.72 ($\underline{C}HPh$), 44.09 ($\underline{C}HAr$), 49.58 (CHCOOEt), 54.60 ($\underline{CH}CHO$), 61.80 ($COO\underline{CH_2}CH_3$), 72.10 [$\underline{C}(CH_3)OH$], 92.62 ($CHNO_2$), 124.22 (CH^m), 128.20 (C^o), 128.93 (C^p), 129.29 (C^o), 129.56 (C^m), 134.77 (C^i), 142.89 (C^i), 148.06 (C^i), 173.51 (COOEt), 202.40 (CHO). Found, %: C 60.96; H 5.16; N 6.53. $C_{23}H_{24}N_2O_8$. Calculated, %: C 60.52; H 5.30; N 6.14.

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