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Synthesis of 2-(Dibenzosuberen-5-yl)-1,3-dicarbonyl Compounds

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Abstract—Reaction of dibenzosuberen-5-ol with 1,3-dicarbonyl compounds catalyzed with scandium(III), copper(II), or indium(III) triflate (5 mol %) afforded 2-(dibenzosuberen-5-yl)-1,3-dicarbonyl compounds in 63–86% yield.

Keywords: dibenzosuberenol, 1,3-dicarbonyl compounds, Lewis acid, dehydrative coupling

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Many compounds containing dibenzosuberene fragment (5*H*-dibenzo[*a,d*]cycloheptene) exhibit various types of biological activity and find application as medicines [1, 2]. Some antidepressants like protriptyline [3] and its structural analogs, nitrogen-containing compounds with dibenzosuberene fragment, are well known and are used as muscle relaxants and histamine antagonists. Thus, 2-(dibenzosuberen-5-yl)-indane-1,3-dione inhibits some enzymes [4–6] and can be used as a starting compound for the synthesis of various derivatives due to the presence of 1,3-dicarbonyl fragment, which possesses a great synthetic potential [7, 8]. Apart the above mentioned derivative of 1,3-indanone only few representatives of 1,3-dioxo compounds like (dibenzosuberenyl)malonic acid [9] and *N*-[2-(dibenzosuberen-5-yl)acetoacetyl]-4-benzyl-oxazolidin-2-one [10] are known.

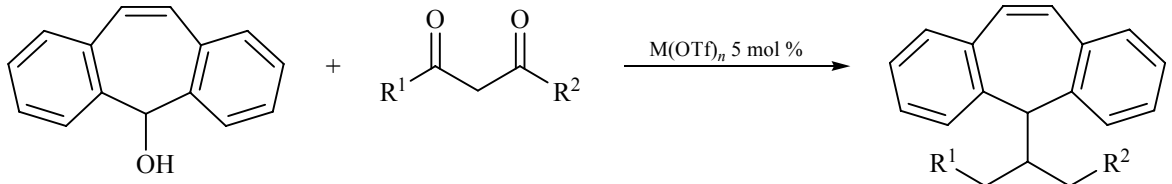
Taking into account the practical importance of the compounds we elaborated a convenient preparative method of dibenzosuberene fragment introduction into 1,3-dicarbonyl compounds. One of the simplest methods of 1,3-dicarbonyl compounds alkylation is the reaction of dehydrative coupling with propargylic, allylic, and benzylic alcohols, which form easily stabilized carbocations under catalysis with Brønsted or Lewis acids [11, 12]. In extension of our investigations [13, 14] an attempt to expand this procedure to dibenzosuberen-5-ol was undertaken. From the multitude of catalysts suitable for carbocation generation, scandium(III), indium(III), and copper(II) triflates were chosen.

The optimization of the reaction conditions was carried out for the reaction of dibenzosuberen-5-ol **I** with acetylacetone **IIa** taken in stoichiometric amounts in 1,2-dichloroethane in the presence of 5 mol % of Lewis acid. The reagents consumption was monitored by GC-MS. Unlike allylic alcohols [15] in this case the reaction proceeded extremely slowly at room temperature; however, an increase in the temperature to 40°C resulted in the maximal conversion of the reagents already in 30 min. The specific indication of the reaction completion was the change in the color of the reaction mixture from crimson-red caused by the presence in the solution of 5*H*-dibenzo[*a,d*]cyclohepten-5-yl cation to light-yellow. The reaction product was isolated by flash chromatography. The maximum yield of compound **IIIa** (84%) was attained under catalysis with $Sc(OTf)_3$; an efficiency of two other catalysts, $In(OTf)_3$ and $Cu(OTf)_2$, was quite similar: the product was prepared in 82 and 79% yield, respectively.

It was established that in 1,2-dichloroethane the reaction of dibenzosuberen-5-ol **I** with 1,3-dicarbonyl compounds **IIa–IIk** of various types proceeded smoothly with the formation of **IIIa–IIIk** in 63–86% yield. In the case of cyclic 1,3-dicarbonyl compounds **IIi–IIk** the reaction was performed in nitromethane (Scheme 1).

The reaction products **IIIa**, **IIIc–IIIk** were crystalline compounds melting without decomposition; compound **IIIb** was colorless oily substance. Derivatives of barbituric and thiobarbituric acids **IIIj** and **IIIk** as distinct from the other prepared compounds occurred to be practically insoluble in chloroform and well soluble only in DMSO.

Scheme 1.



I **IIa–IIk** **IIIa–IIIk**

$R^1 = R^2$ = Me (**a**); R^1 = Ph, R^2 = Me (**b**); $R^1 = R^2$ = Ph (**c**); R^1 = Ph, $R^2 = CF_3$ (**d**); R^1 = 2-thienyl, $R^2 = CF_3$ (**e**); R^1 = Me, R^2 = OEt (**f**); R^1 = Ph, R^2 = OEt (**g**); $R^1 = R^2$ = OEt (**h**); R^1, R^2 = $–CH_2C(Me_2)CH_2–$ (**i**), –NHC(O)NH– (**j**); –NHC(S)NH– (**k**); M = Sc, In (n = 3); M = Cu (n = 2).

The specific feature of ^{1}H NMR spectra of the prepared compounds was nonequivalence of the protons of the ethylene bridge of dibenzosuberenyl fragment, when the 1,3-dicarbonyl fragment was unsymmetrically substituted. Thus, in the spectrum of acetylacetone derivative **IIIa** a singlet signal of the ethylene bridge protons was observed at 7.04 ppm; in the spectrum of benzoylacetone derivative **IIIb** these protons resonated as two doublets at 6.98 and 7.12 ppm (with coupling constants of 11.9 and 11.4 Hz respectively).

The developed approach towards dibenzosuberenyl fragment introduction into 1,3-dicarbonyl compounds under the catalysis with Lewis acids is a convenient alternative to the described before acid-catalyzed reaction of this type [9, 10].

EXPERIMENTAL

^{1}H and ^{13}C NMR spectra were registered on an ECA400 instrument (JEOL) in $CDCl_3$ (Aldrich) or in $(CD_3)_2SO$ (Deutero GmbH); tetramethylsilane by Aldrich was used as a reference. IR spectra were recorded on an IR Prestige spectrometer (Shimadzu). The reaction progress was monitored by GC-MS on a GC-2010 spectrometer (Shimadzu) equipped with a mass-selective detector QP-2010 Plus Shimadzu (column Supelko SLB-5ms, *h* 30 m, ramp from 60 to 265°C at a rate 30 deg/min). Melting points were determined in open capillaries on a Stuart SMP30 instrument. In the work commercially available dibenzosuberen-5-ol and 1,3-diketones were used; indium, scandium, and copper triflates were prepared by the known method [16].

3-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)pentane-2,4-dione (IIIa). A solution of dibenzosuberenol (0.15 g, 0.72 mmol) and acetylacetone (0.072 g, 0.72 mmol) in 2 mL of 1,2-dichloroethane was added to a mixture of 1,2-dichloroethane (3 mL) and $Sc(OTf)_3$ (0.018 g, 0.036 mmol). The reaction mixture was stirred at 40°C for 30 min then treated with 10 mL of 2 M HCl in a separatory funnel. The organic layer was separated; the aqueous layer was extracted with chloroform (3 ×10 mL). The combined organic fractions were evaporated; the residue was purified by chromatography (eluent hexane–ethyl acetate, 10 : 1). Yield 0.18 g (84%), colorless crystals, mp 167°C. IR spectrum (KBr), ν, cm^{-1}: 3045, 3026, 3001 (=C–H); 2966, 2908 (C–H); 1734, 1699 (C=O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.83 (6H, CH_3), 4.75 d (1H, CH, *J* 11.4 Hz), 4.87 (1H, CH, *J* 11.4 Hz), 7.04 s (2H, CH), 7.21–7.25 m (2H, CH), 7.28–7.37 m (2H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 30.3, 53.7, 65.8, 127.1, 129.1, 129.8, 130.5, 131.3, 134.1, 136.4, 202.4. Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 290 (3) $[M]^+$, 202 (5), 191 (100), 165 (9).

2-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)-1-phenylbutane-1,3-dione (IIIb) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and benzoylacetone (0.116 g, 0.72 mmol). Yield 0.22 g (86%), colorless oily substance. IR spectrum (film), ν, cm^{-1}: 3064, 3020 (=C–H); 2935 (C–H); 1730, 1712, 1680, 1670 (C=O); 1597, 1490 (C=C). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.88 (3H, CH_3), 5.15 d (1H, CH, *J* 11 Hz), 5.66 d (1H, CH, *J* 11.4 Hz), 6.98 d (1H, CH, *J* 11.9 Hz), 7.04–7.15 m (3H, CH), 7.20–7.28 m (2H, CH), 7.29–7.37 m (4H, CH), 7.39–7.49 m (3H, CH), 7.65–7.72 m (2H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 29.3, 53.8, 60.5, 126.8, 127.3, 128.4, 128.6, 129.0, 129.2, 129.6, 129.8, 130.6, 131.1, 131.4, 131.5, 133.4, 133.9, 134.6, 136.5, 136.9, 137.2, 194.5, 202.0. Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 352 (2) $[M]^+$, 191 (100), 165 (7).

2-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)-1,3-diphenylpropane-1,3-dione (IIIc) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol

(0.15 g, 0.72 mmol), and dibenzoylmethane (0.161 g, 0.72 mmol). Yield 0.21 g (71%), colorless crystals, mp 157°C. IR spectrum (KBr), ν, cm^{-1}: 3059, 3018 (=C–H); 1699, 1656 (C=O); 1595 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.35 d (1H, CH, *J* 11.4 Hz), 6.53 (1H, CH, *J* 11.4 Hz), 7.02–7.10 m (2H, CH), 7.08 s (2H, CH), 7.12–7.16 m (2H, CH), 7.19–7.30 m (6H, CH), 7.3–7.45 m (4H, CH), 7.64–7.70 m (4H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 54.8, 55.0, 126.8, 128.3, 128.3, 129.0, 129.4, 131.3, 131.6, 133.0, 134.2, 136.9, 137.3, 194.0.

2-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)-1-phenyl-4,4,4-trifluorobutane-1,3-dione (IIId) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and 1-phenyl-4,4,4-trifluorobutane-1,3-dione (0.161 g, 0.72 mmol); reaction time 1 h. Yield 0.23 g (79%), colorless crystals, mp 123–125°C (hexane). IR spectrum (KBr), ν, cm^{-1}: 3068, 3049, 3026 (=C–H); 1724, 1680 (C=O); 1595, 1492 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.10 d (1H, CH, *J* 10.9 Hz), 6.16 d (1H, CH, *J* 11.5 Hz), 6.89 d (1H, CH, *J* 11.9 Hz), 6.92–7.01 m (2H, CH), 7.10 d (1H, CH, *J* 11.9 Hz), 7.13–7.18 m (1H, CH), 7.20–7.37 m (6H, CH), 7.41–7.50 m (2H, CH), 7.52–7.58 m (2H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 52.3, 54.9, 115.0 q (CF_3, *J* 292 Hz), 127.2, 127.5, 128.2, 128.3, 129.0, 129.4, 129.4, 129.8, 130.7, 131.1, 131.4, 131.4, 133.6, 133.9, 134.5, 134.8, 135.7, 136.3, 184.0 q (*J* 36 Hz), 191.9. Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 406 (2) $[M]^+$, 191 (100), 165 (7), 105 (6).

2-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)-1-(thiophen-2-yl)-4,4,4-trifluorobutane-1,3-dione (IIIe) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and 1-(thiophen-2-yl)-4,4,4-trifluorobutane-1,3-dione (0.16 g, 0.72 mmol); reaction time 1 h. Yield 0.24 g (77%), colorless crystals, mp 133–134°C (hexane). IR spectrum (KBr), ν, cm^{-1}: 3095, 3064, 3049, 3020 (=C–H); 1762, 1654 (C=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.13 d (1H, CH, *J* 11.3 Hz), 5.94 (1H, CH, *J* 11.3 Hz), 6.97–7.01 m (2H, CH), 7.03–7.14 m (3H, CH), 7.17–7.21 m (1H, CH), 7.24–7.29 m (2H, CH), 7.31–7.37 m (2H, CH), 7.43–7.49 m (1H, CH), 7.54–7.57 m (2H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 53.9, 54.8, 115.1 q (*J* 292 Hz), 127.4, 127.5, 128.0, 129.2, 129.4, 129.5, 129.8, 130.8, 131.1, 131.4, 131.4, 133.2, 133.9, 134.3, 135.0, 135.5, 136.0, 143.3, 183.0, 183.7 q (*J* 36 Hz).

Ethyl 2-(5*H*-dibenzo[*a*,*d*]cyclohepten-5-yl)-3-oxobutanoate (IIIf) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and ethyl acetoacetate (0.094 g, 0.72 mmol). Yield 0.15 g (63%), colorless crystals, mp 68–70°C. IR spectrum (KBr), ν, cm^{-1}: 3066, 3055, 3026 (=C–H); 2976, 2931 (C–H); 1743, 1726, 1708 (C=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.95 t (3H, CH_3, *J* 7.4 Hz), 1.86 s (3H, CH_3), 3.83 q (2H, CH_2, *J* 7.3 Hz), 4.48 d (1H, CH, *J* 11.5 Hz), 4.85 (1H, CH, *J* 11.5 Hz), 7.01 d (1H, CH, *J* 11.4 Hz), 7.05 d (1H, CH, *J* 11.9 Hz), 7.20–7.24 m (2H, CH), 7.28–7.32 m (2H, CH), 7.35–7.42 m (2H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.8, 30.4, 53.0, 57.5, 61.1, 127.0, 127.1, 128.6, 129.0, 129.4, 129.6, 130.8, 130.9, 131.6, 134.0, 134.3, 136.6, 167.6, 201.8. Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 320 (4) $[M]^+$, 191 (100), 165 (7).

Ethyl 2-(5*H*-dibenzo[*a*,*d*]cyclohepten-5-yl)-3-oxo-3-phenylpropanoate (IIIg) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and ethyl benzoylacetate (0.14 g, 0.72 mmol); reaction time 1 h. Yield 0.18 g (67%), colorless crystals, mp 112–113°C (hexane). IR spectrum (KBr), ν, cm^{-1}: 3066, 3055, 3020 (=C–H); 2978, 2953, 2937 (C–H); 1722, 1687 (C=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.89 t (3H, CH_3, *J* 7 Hz), 3.77 q (2H, CH_2, *J* 7.3 Hz), 5.13 d (1H, CH, *J* 11 Hz), 5.38 (1H, CH, *J* 11.3 Hz), 6.93 d (1H, CH, *J* 11.9 Hz), 7.06–7.12 m (3H, CH), 7.22–7.27 m (2H, CH), 7.29–7.37 m (4H, CH), 7.41–7.50 m (3H, CH), 7.70–7.75 m (2H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.8, 52.6, 53.2, 61.3, 126.8, 127.1, 128.4, 128.5, 128.9, 128.9, 129.4, 129.5, 130.9, 131.0, 131.4, 131.7, 133.3, 134.0, 134.8, 136.8, 136.9, 137.1, 167.8, 193.6. Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 382 (2) $[M]^+$, 191 (100), 165 (5), 105 (6).

Diethyl 2-(5*H*-dibenzo[*a*,*d*]cyclohepten-5-yl)-propanedioate (IIIh) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and diethyl malonate (0.12 g, 0.72 mmol); reaction time 1 h. Yield 0.16 g (64%), colorless crystals, mp 87–88°C (hexane). IR spectrum (KBr), ν, cm^{-1}: 3064, 3030 (=C–H); 2972, 2929, 2906 (C–H); 1753 (C=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.95 t (6H, CH_3, *J* 6.8 Hz), 3.85 q (4H, CH_2, *J* 6.8 Hz), 4.22 d (1H, CH, *J* 11.5 Hz), 4.83 (1H, CH, *J* 11.9 Hz), 7.01 s (2H, CH), 7.20–7.25 m (2H, CH), 7.27–7.33 m (4H, CH), 7.38–7.43 m (2H, CH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.8, 50.6, 53.4, 61.1, 127.0, 128.6, 129.5, 130.8, 131.4, 134.4, 136.7, 167.7. Mass spectrum (EI, 70 eV), *m/z* (I_{rel}, %): 350 (5) $[M]^+$, 202 (4), 191 (100), 165 (6).

2-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)-5,5-dimethylcyclohexane-1,3-dione (IIIi) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and dimedone (0.1 g, 0.72 mmol) in nitromethane; reaction time 1 h. Yield 0.19 g (79%), colorless crystals, mp 192–195°C (hexane). IR spectrum (KBr), ν, cm^{-1}: 3066, 3049, 3022 (=C–H); 2954, 2929, 2868 (C–H); 1718, 1703, 1687 (C=O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.87 s (3H, CH_3), 1.04 s (3H, CH_3), 2.33 d (2H, CH_2, *J* 12.4 Hz), 2.57 d (2H, CH_2, *J* 13.3 Hz), 4.19 d (1H, CH, *J* 11 Hz), 4.65 d (1H, CH, *J* 11 Hz), 7.00 s (1H, CH), 7.21–7.25 m (2H, CH), 7.29–7.33 m (6H, CH). ^{13}C NMR spectrum ($(CD_3)_2SO$), δ_C, ppm: 27.9, 28.9, 30.7, 53.3, 53.9, 65.2, 127.1, 128.8, 129.8, 129.8, 131.1, 133.8, 136.3, 203.7.

5-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (IIIj) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and barbituric acid (0.09 g, 0.72 mmol) in nitromethane; reaction time 3 h. Yield 0.18 g (81%), pale brown crystalline powder, mp 200°C. IR spectrum (KBr), ν, cm^{-1}: 3194, 3076 br (N–H); 1755, 1726, 1701, 1676 (C=O). ^1H NMR spectrum [$(CD_3)_2SO$], δ_C, ppm: 3.46 d (1H, CH, *J* 11 Hz), 4.70 d (1H, CH, *J* 11 Hz), 7.09 s (2H, CH), 7.25–7.30 m (4H, CH), 7.31–7.36 m (2H, CH), 7.36–7.42 m (2H, CH), 10.87 s (2H, NH). ^{13}C NMR spectrum [$(CD_3)_2SO$], δ_C, ppm: 49.2, 55.6, 126.9, 128.4, 129.2, 129.7, 130.8, 134.0, 136.0, 151.2, 168.5.

5-(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)-2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione (IIIk) was prepared similarly from $Sc(OTf)_3$ (0.018 g, 0.036 mmol), dibenzosuberenol (0.15 g, 0.72 mmol), and thiobarbituric acid (0.1 g, 0.72 mmol) in nitromethane; reaction time 3 h. Yield 0.17 g (73%), pale yellow crystalline powder, mp 180°C. IR spectrum (KBr), ν, cm^{-1}: 3169 br (N–H); 1734, 1705 (C=O). ^1H NMR spectrum [$(CD_3)_2SO$], δ, ppm: 3.55 d (1H, CH, *J* 11.5 Hz), 4.61 d (1H, CH, *J* 11.4 Hz), 7.10 s (2H, CH), 7.24–7.33 m (6H, CH), 7.37–7.44 m (2H, CH), 11.97 s (2H, NH). ^{13}C NMR spectrum [$(CD_3)_2SO$], δ_C, ppm: 49.5, 55.4, 127.0, 128.5, 129.2, 129.7, 130.8, 134.1, 135.7, 166.7, 179.8.

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