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Synthesis of 1,3-Diketones from 3-(4-R-Phenyl)propionic Acids

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Abstract—Self-acylation of 3-(4-R-phenyl)propionic acids (R = H, Br, 1-adamantyl) in trifluoroacetic anhydride catalyzed by trifluoromethanesulfonic acid provides a simple and efficient synthesis of 1,3-diketones. Indan-1-ones formed in the first step undergo acylation to give the corresponding 2-(3-phenyl-1-oxopropyl)indan-1-ones as the major products. One-pot synthesis of polysubstituted pyrazoles directly from 3-(4-R-phenyl)propionic acids is described.

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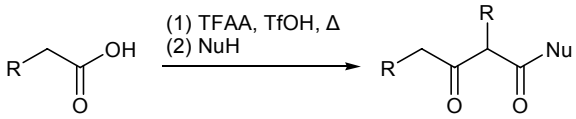
1,3-Diketones constitute one of the most important classes of organic compounds; they are widely used as key building blocks in organic synthesis and exhibit various kinds of biological activity, including antitumor, antiviral, anti-inflammatory, antioxidant, and antimicrobial; they also show a broad spectrum of ionophoric properties [1–3].

1,3-Diketones are traditionally synthesized by the Claisen condensation which in a general case includes C-acylation of the α-position of a ketone taken as enolate, enamine, or silyl enol ether. Acid halides, esters (including formates and oxalates), and anhydrides, dialkyl carbonates, methoxymagnesium methyl carbonate, *N*-acyl imidazoles, acyl cyanides, and acylbenzotriazoles are commonly used as acylating agents [4]. Despite numerous modifications of this method [1, 2], in particular recent ones [5–9], none of them ensure direct synthesis of β-diketones from acids and ketones with simultaneous activation of both carbonyl and methylene components immediately during the reaction.

In this article we describe a simple and efficient method for the synthesis of 1,3-diketones via intra- and intermolecular self-acylation of β-phenylpropionic acids in the system trifluoroacetic anhydride–trifluoromethanesulfonic acid (TFAA–TfOH). We previously found [10] that self-acylation of aliphatic carboxylic acids in trifluoroacetic anhydride, catalyzed by trifluoromethanesulfonic acid, provides a convenient synthetic approach to 2,4-dialkyl-substituted acetoacetic acids and their derivatives (Scheme 1). Trifluoroacetic anhydride used as a reaction medium and activator readily converts carboxylic acids into mixed anhydrides, acyl trifluoroacetates, which are efficient acylating agents [11], while the presence of trifluoromethanesulfonic acid favors enolization and enhances the acylating power of acyl trifluoroacetates.

Scheme 1.

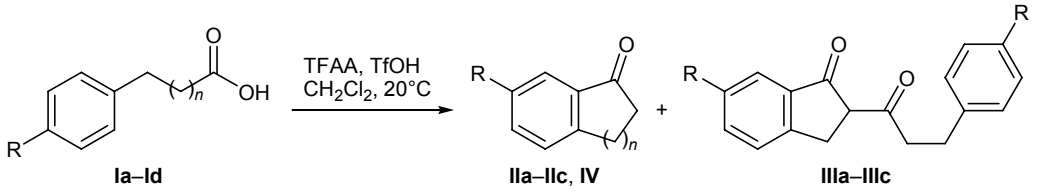


R = 1-adamantyl, *t*-Bu, *i*-Pr; NuH = H_2O, ROH, R′R″NH.

In this work we studied the transformations of ω-phenylalkanoic acids **Ia**–**Id** in the acylating system TFAA–TfOH (see table). Unlike phenylacetic acid which underwent polymerization under these conditions [10], β-phenylpropionic acid **Ia** in a mixture of TFAA and methylene chloride in the presence of 0.25 equiv of TfOH at room temperature was converted into 2-(3-phenyl-1-oxopropyl)indan-1-one (**IIIa**) in ~55% yield (Scheme 2). Diketone **IIIa** is the product of acylation of intermediate indan-1-one (**IIa**). Compound **IIa** was isolated from the reaction mixture in a small amount (<2%), and 40% of initial acid **Ia** was recovered (see table, run no. 2). The yield of **IIIa** increased to 80% when 0.5 equiv of TfOH was used (run no. 3). By contrast, γ-phenylbutyric acid (**Id**) under analogous conditions (run nos. 12, 13) was quantitatively converted into tetralone (**IV**).

Acid-catalyzed intramolecular cyclization of 3-arylpropanoic and 4-arylbutanoic acids into indan-1-ones

Scheme 2.



R = H (**a**, **d**), Br (**b**), 1-Ad (**c**); **Ia**–**Ic**, **IIa**–**IIc**, n = 1; **Id**, **IV**, n = 2.

and tetrahydronaphthalen-1-ones is well known [12–16]; however, in no case further acylation with formation of β-diketones was observed. As far as we know, both intra- and intermolecular acylation with formation of indan-1-one together with an appreciable amount of diketone **IIIa** was reported only for activated 3-phenylpropanoyl perchlorate (prepared from 3-phenylpropanoyl chloride and silver perchlorate) in nitromethane solution [17].

Acyl trifluoroacetates generated *in situ* from carboxylic acids and TFAA were used to acylate arenes in the presence of acid catalysts (H_3PO_4 or TfOH) [18–20]. For example, the system TFAA–TfOH was applied to prepare aryl alkyl ketones from aromatic compounds and carboxylic acids, but no formation of 1,3-diketones was observed [20]. The difference between our results and those reported in [20] may be rationalized by the fact that in the latter case considerably larger amount of superacid (TfOH) was used (4 equiv against 0.25–0.5 equiv in our experiments), so that the acylation of ketones slowed down. In fact, increase of the amount of TfOH in the reaction with 3-phenylpropionic acid (**Ia**) led to sharp decrease of the yield of diketone **IIIa** and increase of the yield of indan-1-one (**IIa**), which attained 94% in the presence of 1.5 equiv of TfOH (see table; run nos. 4, 5).

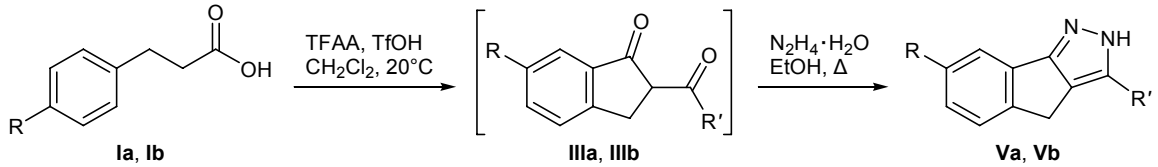
The reaction of 3-(4-bromophenyl)propionic acid (**Ib**) with TFAA in the presence of 0.5 equiv of TfOH was chemoselective, and the only product was diketone **IIIb** (~67%, run no. 7); the highest yield of **IIIb** was obtained using 1 equiv of TfOH (~78%, run no. 8). Raising the amount of TfOH to 3 equiv made indanone **IIb** the major product (67%, run no. 10); simultaneously, the amount of unreacted acid **Ib** increased (13 and 24%, respectively). From 3-[4-(1-adamantyl)phenyl]propionic acid (**Ic**) we obtained 48% of ketone **IIc** and 35% of diketone **IIIc** (run no. 11). We believe that the lower yield of diketone **IIIc** is related to the poor solubility of 6-(1-adamantyl)indan-1-one (**IIc**) in the reaction medium. Obviously, the intra- and intermolecular acylation of 3-phenylpropionic acids is sensitive to both the nature of substituent in the phenyl

Self-acylation of ω-phenylalkanoic acids **Ia**–**Id**

Run no.	Initial acid no.	Molar ratio **I**–TFAA–TfOH	Reaction time, h	Product, yield,[a] %
1	**Ia**	1:6:0	2	**IIIa**, <2
2	**Ia**	1:6:0.25	2	**IIa**, <2; **IIIa** 55 (50)
3	**Ia**	1:6:0.5	2	**IIa**, 23 (20); **IIIa**, 80 (75)
4	**Ia**	1:6:1.0	2	**IIa**, 81; **IIIa**, 16
5	**Ia**	1:6:1.5	0.5	**IIa**, 96 (94); **IIIa**, <1
6	**Ib**	1:6:0.25	2	**IIIb**, 27
7	**Ib**	1:6:0.5	2	**IIIb**, 67 (47)
8	**Ib**	1:6:1.0	2	**IIb**, 9 (8); **IIIb**, 78 (70)
9	**Ib**	1:6:1.5	2	**IIb**, 28; **IIIb**, 58
10	**Ib**	1:6:3.0	2	**IIb**, 67 (62); **IIIb**, <1
11	**Ic**	1:6:0.5	3	**IIc** (48); **IIIc** (35)
12	**Id**	1:6:0.25	1.5	**IV**, 96 (89)
13	**Id**	1:6:0.5	1.5	**IV**, 89

[a] The yields were determined on the basis of the ^{1}H NMR data; the yields of the products isolated by silica gel chromatography are given in parentheses.

Scheme 3.



Ia, **Ib** **IIIa**, **IIIb** **Va**, **Vb**

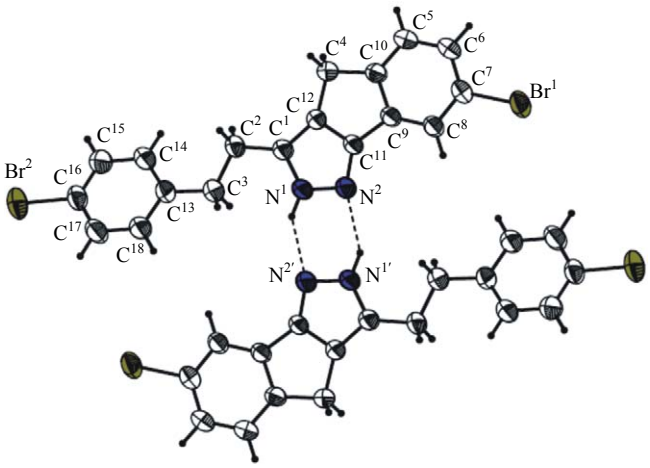
R = H, R′ = $PhCH_2CH_2$ (**a**); R = Br, R′ = 4-$BrC_6H_4CH_2CH_2$ (**b**).

fragment and the amount of trifluoromethanesulfonic acid added.

1,3-Diketones are very popular intermediate products in the synthesis of various heterocyclic compounds. For example, the most widely used method for the synthesis of pyrazoles is based on the reaction of 1,3-diketones with hydrazine derivatives [21, 22]. With a view to extend the synthetic potential of the described reaction, we tried to obtain pyrazoles **Va** and **Vb** from 3-phenylpropionic acids **Ia** and **Ib** in a one-pot process. For this purpose, the reaction mixtures containing diketones **IIIa** and **IIIb** (run nos. 3, 8) were evaporated under reduced pressure, the residue was dissolved in ethanol, hydrazine hydrate was added to the solution, and the mixture was heated under reflux. As a result, we isolated pyrazoles **Va** and **Vb** in 69 and 62% yield, respectively (Scheme 3).

The structure of pyrazole **Vb** was determined by X-ray analysis (CCDC 950017) [23]. Molecules **Vb** in crystal form centrosymmetric dimers through intermolecular hydrogen bonds N^1–$H^1\cdots N^{2'}$ (see figure).

The structure of the newly synthesized compounds was confirmed by their ^1H and ^{13}C NMR spectra and



Structure of centrosymmetric dimer formed by molecules of 7-bromo-3-[2-(4-bromophenyl)ethyl]-1,4-dihydroindeno-[1,2-*c*]pyrazole (**Vb**) in crystal according to the X-ray diffraction data.

elemental compositions. Diketones **IIIa**–**IIIc** in $CDCl_3$ solution exist mainly in the enol form, as follows from the presence in their ^{13}C NMR spectra of a strong signal at δ_C ~110 ppm, which belongs to the α-olefinic quaternary carbon atom in the C(OH)=C–C(O) fragment. The minor diketone tautomer characteristically displays a multiplet signal at δ ~3.9 ppm in the ^1H NMR spectrum due to the C(O)CHC(O) proton, and the corresponding carbon signal is located at δ_C ~61 ppm in the ^{13}C NMR spectrum.

To conclude, we have shown for the first time that self-acylation of 3-phenylpropionic acids in the system trifluoroacetic anhydride–trifluoromethanesulfonic acid–methylene chloride gives β-diketones as the major products. The described reaction makes it possible to synthesize substituted pyrazoles directly from 3-phenylpropionic acid via a one-pot process where diketones are formed as intermediate products. Studies on the synthetic potential of this procedure will be continued.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance-400 spectrometer at 400 and 100 MHz, respectively, using $CDCl_3$ as solvent; the chemical shifts were determined relative to the solvent signals. The X-ray diffraction data for compound **Vb** were acquired on an Enraf Nonius CAD4 diffractometer at 295 K (CuK_α radiation, graphite monochromator, ω-scanning). Analytical thin-layer chromatography was carried out on Merck DC Alufolien Kieselgel 60 F_{254} plates; spots were detected under UV light (λ 254 nm). Silica gel Merck Kieselgel 40/60 was used for preparative column chromatography. Trifluoroacetic anhydride was distilled over P_2O_5 prior to use.

Self-acylation of 3-(4-R-phenyl)propionic acids Ia–Ic (*general procedure*). A solution of acid **Ia–Ic** and trifluoroacetic anhydride in 1 mL of methylene chloride was stirred for 15 min, a required amount of trifluoromethanesulfonic acid was added, and the mixture was stirred for a time indicated in table (TLC). The mixture was evaporated, the residue was treated

with water (3 mL) and dissolved in methylene chloride (15 mL), the solution was washed with a 5% solution of $NaHCO_3$ (2×3 mL) and water (2×3 mL) and dried over $MgSO_4$, the solvent was distilled off under reduced pressure, and the residue was purified by chromatography on silica gel using hexane–methylene chloride as eluent.

Indan-1-one (IIa) was synthesized by reaction of 150 mg (1 mmol) of acid **Ia** with 0.85 mL (6 mmol) of TFAA in the presence of 0.133 mL (1.5 mmol) of TfOH. Yield 124 mg (94%), mp 37–40°C; published data [17]: mp 39–42°C.

6-Bromoindan-1-one (IIb) was synthesized by reaction of 229 mg (1 mmol) of acid **Ib** with 0.85 mL (6 mmol) of TFAA in the presence of 0.265 mL (3.0 mmol) of TfOH. Yield 130 mg (62%), mp 111–115°C; published data [24]: mp 109–110°C.

6-(1-Adamantyl)indan-1-one (IIc) was synthesized from 284 mg (1 mmol) of acid **Ic** using 0.85 mL (6 mmol) of TFAA and 0.044 mL (0.5 mmol) of TfOH. Yield 128 mg (48%), white crystals, mp 164–165°C. ^{1}H NMR spectrum, δ, ppm: 1.60–1.80 m (6H, Ad), 1.91 br.s (6H, Ad), 2.09 br.s (3H, Ad), 2.66 m (2H, CH_2), 3.07 m (2H, CH_2), 7.40 d (1H, H_{arom}, J = 7.8 Hz), 7.61 d (1H, H_{arom}, J = 7.8 Hz), 7.73 d (1H, H_{arom}, J = 2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 25.28 (CH_2), 28.78 (CH), 36.24 (C), 36.58 (CH_2), 43.13 (CH_2), 119.76 (CH_{arom}), 126.19 (CH_{arom}), 131.90 (CH_{arom}), 136.94 (C_{arom}), 150.96 (C_{arom}), 152.58 (C_{arom}), 207.35 (CO). Found, %: C 85.32; H 8.04. $C_{19}H_{22}O$. Calculated, %: C 85.67; H 8.32.

2-(3-Phenyl-1-oxopropyl)indan-1-one (IIIa) was synthesized from 150 mg (1 mmol) of acid **Ia** using 0.85 mL (6 mmol) of TFAA and 0.044 mL (0.5 mmol) of TfOH. Yield 75% (100 mg), red powder, mp 65–66°C; published data [17]: mp 68–69°C. ^{1}H NMR spectrum, δ, ppm (ketone–enol ratio 20:80): enol: 2.73 t (2H, CH_2, J = 8 Hz), 3.04 t (2H, CH_2, J = 8 Hz), 3.40 s (2H, CH_2), 7.15–7.32 m (5H, H_{arom}), 7.33–7.55 m (3H, H_{arom}), 7.80 d (1H, H_{arom}, J = 7.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 29.91 (CH_2), 31.64 (CH_2), 36.79 (CH_2), 110.50 [**C**=C(OH)], 123.02 (CH_{arom}), 125.60 (CH_{arom}), 126.25 (CH_{arom}), 127.21 (CH_{arom}), 128.29 (CH_{arom}), 128.45 (CH_{arom}), 132.65 (C_{arom}), 138.05 (C_{arom}), 140.64 (C_{arom}), 147.44 (C_{arom}), 179.70 [C=**C**(OH)], 191.06 (CO).

6-Bromo-2-[3-(4-bromophenyl)-1-oxopropyl]-indan-1-one (IIIb) was synthesized from 229 mg (1 mmol) of acid **Ib** using 0.85 mL (6 mmol) of TFAA and 0.088 mL (1 mmol) of TfOH. Yield 148 mg (70%), red powder, mp 147–148°C. ^{1}H NMR spectrum, δ, ppm (ketone–enol ratio 15:85); enol: 2.72 t (2H, CH_2, J = 7.2 Hz), 2.99 t (2H, CH_2, J = 7.2 Hz), 3.36 s (2H, CH_2), 7.10 d (2H, H_{arom}, J = 8 Hz), 7.31 d (1H, H_{arom}, J = 7.6 Hz), 7.40 d (2H, H_{arom}, J = 8 Hz), 7.63 d (1H, H_{arom}, J = 7.6 Hz), 7.91 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 29.46 (CH_2), 30.40 (CH_2), 36.45 (CH_2), 110.53 [**C**=C(OH)], 119.77 (C_{arom}), 121.09 (C_{arom}), 125.66 (CH_{arom}), 126.74 (CH_{arom}), 129.72 (CH_{arom}), 131.20 (CH_{arom}), 135.00 (CH_{arom}), 139.11 (C_{arom}), 139.47 (C_{arom}), 145.16 (C_{arom}), 180.72 [C=**C**(OH)], 187.95 (CO). Found, %: C 51.01; H 3.23. $C_{18}H_{14}Br_2O_2$. Calculated, %: C 51.22; H 3.34.

6-(1-Adamantyl)-2-{3-[4-(1-adamantyl)phenyl]-1-oxopropyl}indan-1-one (IIIc) was synthesized from 284 mg (1 mmol) of acid **Ic** using 0.85 mL (6 mmol) of TFAA and 0.044 mL (0.5 mmol) of TfOH. Yield 155 mg (35%), grey powder, mp 177–178°C. ^{1}H NMR spectrum, δ, ppm (ketone–enol ratio 5:95); enol: 1.70–1.90 m (24H, Ad), 2.05–2.15 m (6H, Ad), 2.72 t (2H, CH_2), 3.01 t (2H, CH_2), 3.35 s (2H, CH_2), 7.19 d (2H, H_{arom}, J = 8 Hz), 7.28 d (2H, H_{arom}, J = 8 Hz), 7.39 d (1H, H_{arom}, J = 7.9 Hz), 7.58 d (1H, H_{arom}, J = 7.9 Hz), 7.82 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 28.48 (CH), 28.53 (CH), 29.02 (CH_2), 30.93 (CH_2), 35.49 (C), 35.97 (C), 36.28 (CH_2), 36.37 (CH_2), 110.51 [**C**=C(OH)], 119.02 (CH_{arom}), 124.60 (CH_{arom}), 124.87 (CH_{arom}), 127.64 (CH_{arom}), 129.70 (CH_{arom}), 137.34 (C_{arom}), 137.73 (C_{arom}), 144.75 (C_{arom}), 149.04 (C_{arom}), 150.63 (C_{arom}), 178.24 [C=**C**(OH)], 192.00 (CO). Found, %: C 84.44; H 8.33. $C_{18}H_{14}Br_2O_2$. Calculated, %: C 84.12; H 8.65.

1,2,3,4-Tetrahydronaphthalen-1-one (IV) was synthesized from 164 mg (1 mmol) of acid **Id** using 0.85 mL (6 mmol) of TFAA and 0.133 mL (1.5 mmol) of TfOH. Yield 130 mg (89%), oily substance.

Pyrazoles Va and Vb (*general procedure***).** A solution of acid **Ia** or **Ib** in a mixture of trifluoroacetic anhydride and 1 mL of methylene chloride was stirred for 15 min, a required amount of trifluoromethanesulfonic acid was added, and the mixture was kept for 2 h and evaporated under reduced pressure. The residue was dissolved in 5 mL of ethanol, 0.05 mL (1 mmol) of hydrazine hydrate was added, and the mixture was heated for 2 h under reflux. The solvent was distilled off, the oily residue was dissolved in 15 mL of methylene chloride, the solution was washed with a 5% solution of $NaHCO_3$ and water, dried over $MgSO_4$, and evaporated, and the residue was purified by chromatography on silica gel using methylene chloride–methanol (50:1) as eluent.

3-(2-Phenylethyl)-1,4-dihydroindeno[1,2-*c*]pyrazole (Va) was synthesized from 150 mg (1 mmol) of acid **Ia**, 0.85 mL (6 mmol) of TFAA, 0.044 mL (0.5 mmol) of TfOH, and 0.05 mL (1 mmol) of hydrazine hydrate. Yield 90 mg (69%), brown powder, mp 108–110°C. ^{1}H NMR spectrum, δ, ppm: 3.04 m (4H, CH_2), 3.42 s (2H, CH_2), 7.14 m (2H, H_{arom}), 7.17–7.40 m (4H, H_{arom}), 7.44 d (1H, H_{arom}, J = 6.4 Hz), 7.71 d (1H, H_{arom}, J = 6.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 27.54 (CH_2), 28.36 (CH_2), 34.78 (CH_2), 119.91 (CH), 121.64 (C), 122.73 (CH), 125.83 (CH), 126.28 (CH), 126.43 (2CH), 128.36 (CH), 128.54 (CH), 134.72 (C), 137.93 (C), 140.81 (C), 148.67 (C). Found, %: C 82.67; H 6.35; N 10.37. $C_{18}H_{16}N_2$. Calculated, %: C 83.05; H 6.19; N 10.76.

7-Bromo-3-[2-(4-bromophenyl)ethyl]-1,4-dihydroindeno[1,2-*c*]pyrazole (Vb) was synthesized from 229 mg (1 mmol) of acid **Ib**, 0.85 mL (6 mmol) of TFAA, 0.088 mL (1 mmol) of TfOH, and 0.05 mL (1 mmol) of hydrazine hydrate. Yield 150 mg (72%), red crystals, mp 172–174°C. ^{1}H NMR spectrum, δ, ppm: 2.92–3.08 m (4H, CH_2), 3.36 s (2H, CH_2), 7.01 d (2H, H_{arom}, J = 8 Hz), 7.45–7.28 m (3H, H_{arom}), 7.82 s (1H, H_{arom}), 8.50 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 26.86 (CH_2), 27.70 (CH_2), 33.68 (CH_2), 119.81 (C), 120.46 (C), 122.73 (CH), 126.78 (CH), 129.00 (CH), 129.64 (2CH), 131.15 (C), 131.23 (2CH), 135.94 (C), 138.98 (C), 146.76 (C). Found, %: C 52.27; H 3.30; N 6.46. $C_{18}H_{14}Br_2N_2$. Calculated, %: C 51.71; H 3.37; N 6.70.

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