Synthesis and Properties of Alkyl 2-[2-(Diarylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2-enoates

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Abstract—Ring opening of 3-[(diarylmethylidene)hydrazinylidene]-5-*tert*-butylfuran-2(3*H*)-ones by the action of alcohols afforded previously unknown alkyl 2-[2-(diarylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2-enoates which were found to exist in solution as mixtures of tautomers.

Keywords: 2,4-dioxobutanoic acid esters, ring opening of 3-hydrazinylidenefuran-2(3*H*)-ones, primary and secondary alcohols, tautomers

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3-Hydrazinylidene(imino)furan-2(3H)-ones constitute a promising class of furan derivatives due to their synthetic accessibility [1-5] and high reactivity [6-16]. We recently reported the reaction of 5-tert-butyl-3-[(9H-fluoren-9-ylidene)hydrazinylidene]furan-2(3H)one with NH nucleophiles, which afforded [2-(9H-fluoren-9-ylidene)hydrazinyl]-5,5-dimethyl-4-oxo-2-hex-2-enamides [17]. Furthermore, previous studies have shown that 3-imino(hydrazinylidene)furan-2(3H)-ones undergo ring opening in reactions with OH nucleophiles to give alkyl aroylpyruvates [10, 18]. There are no published data on chemical transformations of structurally related 3-[(arylmethylidene)hydrazinylidene]-5-tert-butyl-3H-furan-2-ones in reactions with alcohols. Then, the present work was aimed at studying this reaction and the properties of the products.

Reactions of 3-[(arylmethylidene)hydrazinylidene]-5-*tert*-butyl-3*H*-furan-2-ones **1a** and **1b** with alcohols in the absence of a catalyst gave alkyl 2-[2-(diarylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2enoates **2a–2i** in low yields. The yield was considerably improved when a catalytic amount of triethylamine was added to the reaction mixture (Scheme 1). Compounds **2a–2i** are pale yellow crystalline solids soluble in toluene, diethyl ether, and acetonitrile and insoluble in propan-2-ol, ethanol, and water.

We failed to obtain esters **2** from 5-*tert*-butyl-3-[(1,2-diphenyl-2-oxoethylidene)hydrazinylidene]furan-2(3*H*)-one (**1c**) or 5-*tert*-butyl-3-[(9*H*-fluoren-9-ylidene)hydrazinylidene]furan-2(3*H*)-one (1d). Instead, the corresponding ketone azines **3a** and **3b** were isolated (Scheme 2). Similar rearrangement of the target product into azine was described in [19]. We also found that compounds **1a–1d** did not react with sterically hindered 2-methylpropan-2-ol, and only unchanged initial compounds were isolated.

The IR spectra of 2a-2i in mineral oil showed absorption bands in the region 1735–1740 cm⁻¹ due to stretching vibrations of the ester carbonyl group and at 1553–1621 cm⁻¹ due to ketone carbonyl involved in intramolecular hydrogen bond N–H…O=C, as well as C=C and C=N bonds.

According to the ¹H NMR data, compounds 2a-2din DMSO- d_6 solution exist as equilibrium mixtures of enehydrazine (A, B) and hydrazone tautomers (C, D). Enehydrazino ketone structures A and B characteristically displayed a singlet from the C³H=C proton at δ 5.47–5.52 and 5.52–5.58 ppm and a broadened singlet of the NH proton at δ 12.01–12.02 and 12.10– 12.12 ppm, respectively. The downfield position of the NH singlet is rationalized by intramolecular hydrogen bonding with the C⁴=O carbonyl group. The overall concentration of tautomers A and B ranges from 55 to 60%, and the equilibrium is shifted toward structure A (30-35%), which does not contradict published data [5, 19]. Ketohydrazones C (E) and D (Z) are characterized by a two-proton singlet for methylene protons on C^3 at δ 4.02–4.04 and 4.01–4.02 ppm, respectively.





1, $R^1 = Ph$, $R^2 = 4-MeOC_6H_4$ (a); $R^1 = R^2 = Ph$ (b); 2, $R^1 = Ph$, $R^2 = 4-MeOC_6H_4$, $R^3 = Me$ (a), Et (b), Bu (c), MeCH₂C(Me)₂CH₂ (d); $R^1 = R^2 = Ph$, $R^3 = Me$ (e), Et (f), *i*-Pr (g), Bu (h), MeCH₂C(Me)₂CH₂ (i).

Structure C predominates over D, presumably due to steric and energy factors.

The ¹H NMR spectra of **2e–2i** in DMSO- d_6 differed from the spectra of **2a–2d**, since they contained signals from only two tautomers, enchydrazine **A** and hydrazone **C**. In particular, tautomer **A** gave rise to olefinic proton signal at δ 5.54–5.60 ppm and NH signal at δ 12.04–12.10 ppm, and structure **C** was characterized by a two-proton singlet at δ 4.01–4.05 ppm for C³H₂ methylene group. The fraction of tautomer **A** was estimated at 65–98% on the basis of the ¹H NMR signal intensities, presumably due to additional stabilization of that structure by intramolecular hydrogen bond C⁴=O···H–N.

The assigned structures were also confirmed by the mass spectra of **2a** and **2e**, which showed the corresponding molecular and fragment ion peaks.

EXPERIMENTAL

The progress of reactions was monitored, and the purity of the isolated compounds was checked, by TLC on Silufol plates using diethyl ether-benzene-acetone (10:9:1) as eluent; spots were visualized by treatment with iodine vapor. The IR spectra were recorded on an FSM-1202 spectrometer with Fourier transform from samples dispersed in mineral oil. The ¹H NMR spectra were recorded on a Bruker Avance III HD spectrometer at 400 MHz using DMSO- d_6 as solvent. The mass spectra (electron impact, 70 eV) were obtained with a Finnigan MAT INCOS 50 instrument. Elemental analysis was performed on a Leco CHNS-932 analyzer. The melting (decomposition) points were measured with a PTP-2 melting point apparatus. Compounds 1a-1d were synthesized according to the procedures described in [17, 20-21].



1c, 3a, $R^1 = Ph$, $R^2 = COPh$; 1d, 3b, $R^1R^2 = 1,1'$ -biphenyl-2,2'-diyl.

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Scheme 2.

General procedure for the synthesis of compounds 2a–2i. A mixture of 0.001 mol of compound 1a or 1b, 20 mL of ethanol, and 0.2 mmol (20 mol %) of triethylamine was refluxed for 5 min until a yellow solution was formed. The mixture was cooled, and the precipitate was filtered off and purified by recrystallization from an appropriate solvent.

Methyl 2-{2-[(4-methoxyphenyl)(phenyl)methylidene|hydrazinyl}-5,5-dimethyl-4-oxohex-2-enoate (2a). Yield 0.33 g (85%), pale yellow crystals, mp 145– 147°C (from EtOH). IR spectrum, v, cm^{-1} : 1740 (C=O), 1613, 1553 (C=C, C=N). ¹H NMR spectrum, δ , ppm: A (35%): 0.98 s (9H, *t*-Bu), 3.76 s (3H, OCH₃), 3.90 s (3H, OCH₃), 5.52 s (1H, CH), 6.93–7.66 m (9H, H_{arom}), 12.02 br.s (1H, NH); **B** (25%): 1.01 s (9H, t-Bu), 3.79 s (3H, OCH₃), 3.87 s (3H, OCH₃), 5.58 s (1H, CH), 6.93-7.66 m (9H, H_{arom}), 12.12 br.s (1H, NH); C (22%): 1.07 s (9H, *t*-Bu), 3.67 s (3H, OCH₃), 3.83 s (3H, OCH₃), 4.04 s (2H, CH₂), 6.93-7.66 m (9H, H_{arom}); **D** (18%): 1.04 s (9H, *t*-Bu), 3.71 s (3H, OCH₃), 3.90 s (3H, OCH₃), 4.02 s (2H, CH₂), 6.93-7.66 m (9H, H_{arom}). Mass spectrum, *m/z* (*I*_{rel}, %): 394 $(24) [M]^+, 337 (100) [M - t-Bu]^+, 335 (11) [M - t-Bu]^+$ $COOCH_3]^+$, 309 (33) $[M - t-BuCO]^+$, 210 (78) [Ph₂C=N]⁺, 77 (25) [Ph]⁺, 57 (31) [*t*-Bu]⁺. Found, %: C 70.05; H 6.63; N 7.09. C₂₃H₂₆N₂O₄. Calculated, %: C 70.03; H 6.64; N 7.10. M 394.47.

Ethyl 2-{2-[(4-methoxyphenyl)(phenyl)methylidene|hydrazinyl}-5,5-dimethyl-4-oxohex-2-enoate (2b). Yield 0.38 g (94%), pale yellow crystals, mp 127–129°C (from EtOH). IR spectrum, v, cm^{-1} : 1737 (C=O), 1621, 1558 (C=C, C=H, C=O_{chel}). ¹H NMR spectrum, δ , ppm: A (32%): 0.99 s (9H, *t*-Bu), 1.36 t (3H, CH₂CH₃), 3.76 s (3H, OCH₃), 4.38 q (2H, CH₂CH₃), 5.48 s (1H, CH), 6.92–7.65 m (9H, H_{arom}), 12.02 br.s (1H, NH); **B** (25%): 1.01 s (9H, *t*-Bu), 1.15 t (3H, CH₂CH₃), 3.80 s (3H, OCH₃), 4.14 q (2H, CH₂CH₃), 5.53 s (1H, CH), 6.92–7.65 m (9H, H_{arom}), 12.12 br.s (1H, NH); C (23%): 1.08 s (9H, t-Bu), 1.33 t (3H, CH₂CH₃), 3.88 s (3H, OCH₃), 4.03 s (2H, CH₂), 4.38 q (2H, CH₂CH₃), 6.92–7.65 m (9H, H_{arom}); **D** (20%): 1.05 s (9H, *t*-Bu), 1.19 t (3H, CH₂CH₃), 3.82 s (3H, OCH₃), 4.01 s (2H, CH₂), 4.18 q (2H, CH₂CH₃), 6.92-7.65 m (9H, H_{arom}). Found, %: C 70.59; H 6.90; N 6.84. C₂₄H₂₈N₂O₄. Calculated, %: C 70.57; H 6.91; N 6.86. *M* 408.50.

Butyl 2-{2-[(4-methoxyphenyl)(phenyl)methylidene]hydrazinyl}-5,5-dimethyl-4-oxohex-2-enoate (2c). Yield 0.38 g (87%), pale yellow crystals, mp 107– 109°C (from EtOH). IR spectrum, v, cm⁻¹: 1738 (C=O), 1618, 1561 (C=C, C=N, C=O_{chel}). ¹H NMR spectrum, δ, ppm: A (30%): 0.81–0.91 m (3H, CH₂CH₃), 0.99 s (9H, *t*-Bu), 1.20–1.74 m (4H, CH₂CH₂), 3.76 s (3H, OCH₃), 4.33 t (2H, OCH₂), 5.47 s (1H, CH), 6.91-7.68 m (9H, H_{arom}), 12.01 br.s (1H, NH); **B** (25%): 0.81–0.91 m (3H, CH₂CH₃), 1.08 s (9H, t-Bu), 1.20–1.74 m (4H, CH₂CH₂), 3.80 s (3H, OCH₃), 4.09 t (2H, OCH₂), 5.53 s (1H, CH), 6.91-7.68 m (9H, H_{arom}), 12.10 br.s (1H, NH); C (24%): 0.81–0.91 m (3H, CH₂CH₃), 1.01 s (9H, *t*-Bu), 1.20–1.74 m (4H, CH₂CH₂), 3.88 s (3H, OCH₃), 4.02 s (2H, CH₂), 4.14 t (2H, OCH₂), 6.91–7.68 m (9H, H_{arom}); **D** (21%): 0.81–0.91 m (3H, CH₂CH₃), 1.05 s (9H, t-Bu), 1.20–1.74 m (4H, CH₂CH₂), 3.81 s (3H, OCH₃), 4.01 s (2H, CH₂), 4.33 t (2H, OCH₂), 6.91-7.68 m (9H, H_{arom}). Found, %: C 71.52; H 7.35; N 6.45. C₂₆H₃₂N₂O₄. Calculated, %: C 71.53; H 7.39; N 6.42. M 436.55.

2,2-Dimethylbutyl 2-{2-[(4-methoxyphenyl)-(phenyl)methylidene]hydrazinyl}-5,5-dimethyl-4oxohex-2-enoate (2d). Yield 0.39 g (83%), pale yellow crystals, mp 88–90°C (from EtOH). IR spectrum, v, cm⁻¹: 1739 (C=O), 1617, 1561 (C=C, C=N, C=O_{chel}). ¹H NMR spectrum, δ , ppm: A (31%): 0.78–0.86 m (3H, CH₃), 0.99 s (9H, *t*-Bu), 1.14–1.40 m (6H, CH₃), 1.48–1.77 m (2H, CH₂CH₃), 3.76 s (3H, OCH₃), 4.32 t (2H, OCH₂), 5.47 s (1H, CH), 6.91–7.68 m (9H, H_{arom}), 12.01 br.s (1H, NH); **B** (25%): 0.78–0.86 m (3H, CH₃), 1.07 s (9H, t-Bu), 1.14–1.40 m (6H, CH₃), 1.48–1.77 m (2H, CH₂CH₃), 3.88 s (3H, OCH₃), 4.09 t (2H, OCH₂), 5.52 s (1H, CH), 6.91-7.68 m (9H, H_{arom}), 12.10 br.s (1H, NH); C (23%): 0.78–0.86 m (3H, CH₃), 1.01 s (9H, *t*-Bu), 1.14–1.40 m (6H, CH₃), 1.48–1.77 m (2H, CH₂CH₃), 3.80 s (3H, OCH₃), 4.02 s (2H, CH₂), 4.32 t (2H, OCH₂), 6.91–7.68 m (9H, H_{arom}); **D** (21%): 0.78– 0.86 m (3H, CH₃), 1.05 s (9H, *t*-Bu), 1.14–1.40 m (6H, CH₃), 1.48–1.77 m (2H, CH₂CH₃), 3.81 s (3H, OCH₃), 4.01 s (2H, CH₂), 4.13 t (2H, OCH₂), 6.91–7.68 m (9H, H_{arom}). Found, %: C 71.52; H 7.35; N 6.45. C₂₈H₃₈N₂O₄. Calculated, %: C 72.39; H 7.81; N 6.03. *M* 436.55.

Methyl 2-[2-(diphenylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2-enoate (2e). Yield 0.35 g (96%), pale yellow crystals, mp 152–154°C (from EtOH). IR spectrum, v, cm⁻¹: 1739 (C=O), 1619, 1560 (C=C, C=N, C=O_{chel}). ¹H NMR spectrum, δ , ppm: A (88%): 1.01 s (9H, *t*-Bu), 3.93 s (3H, OCH₃), 5.60 s (1H, CH), 7.29–7.68 m (10H, H_{arom}), 12.05 s (1H, NH); C (12%): 1.08 s (9H, *t*-Bu), 3.70 s (3H, OCH₃), 4.05 s (2H, CH₂), 7.29–7.68 m (10H, H_{arom}). Mass spectrum, *m/z* (*I*_{rel}, %): 364 (12) [*M*]⁺, 307 (100) [*M* – *t*-Bu]⁺, 305 (6) [*M* – COOCH₃]⁺, 279 (16) [M - t-BuCO]⁺, 181 (9) [Ph₂C=N]⁺, 77 (33) [Ph]⁺, 57 (23) [t-Bu]⁺. Found, %: C 72.52; H 6.60; N 7.68; O 13.15. C₂₂H₂₄N₂O₃. Calculated, %: C 72.51; H 6.64; N 7.69; O 13.17. *M* 364.44.

Ethyl 2-[2-(diphenylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2-enoate (2f). Yield 0.37 g (98%), pale yellow crystals, mp 142–144°C (from EtOH). IR spectrum, v, cm⁻¹: 1739 (C=O), 1615, 1554 (C=C, C=N, C=O_{chel}). ¹H NMR spectrum, δ , ppm: A (65%): 1.00 s (9H, *t*-Bu), 1.35 t (3H, CH₂CH₃), 4.39 q (2H, CH₂CH₃), 5.54 s (1H, CH), 7.27–7.69 m (10H, H_{arom}), 12.04 br.s (1H, NH); C (35%): 1.07 s (9H, *t*-Bu), 1.16 t (3H, CH₂CH₃), 4.01 s (2H, CH₂), 4.15 q (2H, CH₂CH₃), 7.27–7.69 m (10H, H_{arom}). Found, %: C 72.95; H 6.90; N 7.43; O 12.69. C₂₃H₂₆N₂O₃. Calculated, %: C 72.99; H 6.92; N 7.40; O 12.68. *M* 378.47.

Propan-2-yl 2-[2-(diphenylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2-enoate (2g). Yield 0.29 g (75%), pale yellow crystals, mp 128–130°C (from *i*-PrOH). IR spectrum, v, cm⁻¹: 1735 (C=O), 1617, 1556 (C=C, C=N, C=O_{chel}). ¹H NMR spectrum, δ, ppm: **A** (95%): 1.01 s (9H, *t*-Bu), 1.37 d [6H, CH(CH₃)₂], 5.17–5.27 m [1H, CH(CH₃)₂], 5.55 s (1H, CH), 7.28–7.69 m (10H, H_{arom}), 12.10 s (1H, NH); **C** (5%): 1.04 s (9H, *t*-Bu), 1.32 d [6H, CH(CH₃)₂], 7.28–7.69 m (10H, H_{arom}). Found, %: C 73.46; H 7.19; N 7.13; O 12.22. C₂₄H₂₈N₂O₃. Calculated, %: C 73.44; H 7.19; N 7.14; O 12.23. *M* 392.50.

Butyl 2-[2-(diphenylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2-enoate (2h). Yield 0.34 g (83%), pale yellow crystals, mp 121–123°C (from EtOH). IR spectrum, v, cm⁻¹: 1737 (C=O), 1619, 1558 (C=C, C=N, C=O_{chel}). ¹H NMR spectrum, δ , ppm: A (98%): 0.90 t (3H, CH₃), 1.01 s (9H, *t*-Bu), 1.36– 1.45 m (2H, CH₂CH₃), 1.69–1.75 m (2H, CH₂CH₂CH₃), 4.36 t (2H, OCH₂), 5.56 s (1H, CH), 7.28–7.70 m (10H, H_{arom}), 12.06 s (1H, NH); C (2%): 0.84 t (3H, CH₃), 1.08 s (9H, *t*-Bu), 1.21–1.29 m (2H, CH₂CH₃), 1.49–1.57 m (2H, CH₂CH₂CH₃), 4.04 s (2H, CH₂), 4.12 t (2H, OCH₂), 7.28–7.70 m (10H, H_{arom}). Found, %: C 73.82; H 7.46; N 6.91; O 11.82. C₂₅H₃₀N₂O₃. Calculated, %: C 73.86; H 7.44; N 6.89; O 11.81. *M* 406.53.

2,2-Dimethylbutyl 2-[2-(diphenylmethylidene)hydrazinyl]-5,5-dimethyl-4-oxohex-2-enoate (2i). Yield 0.33 g (77%), pale yellow crystals, mp 119– 120°C (from EtOH). IR spectrum, v, cm⁻¹: 1738 (C=O), 1619, 1559 (C=C, C=N, C=O_{chel}). ¹H NMR spectrum, δ , ppm: A (90%): 0.80–0.87 m (3H, CH₃), 1.01 s (9H, *t*-Bu), 1.26–1.40 m (6H, CH₃), 1.70–1.77 m (2H, CH₂CH₃), 4.35 t (2H, OCH₂), 5.56 s (1H, CH), 7.28–7.70 m (10H, H_{arom}), 12.06 s (1H, NH); C (10%): 0.80–0.87 m (3H, CH₃), 1.08 s (9H, *t*-Bu), 1.26–1.40 m (6H, CH₃), 1.51–1.58 m (2H, CH₂CH₃), 4.04 s (2H, CH₂), 4.12 t (2H, OCH₂), 7.28–7.70 m (10H, H_{arom}). Found, %: C 74.65; H 7.88; N 6.42; O 11.03. C₂₇H₃₄N₂O₃. Calculated, %: C 74.62; H 7.89; N 6.45; O 11.04. *M* 434.58.

CONFLICT OF INTEREST

The authors declare the absence of conflict of interest.

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