Radical reactions of [60]fullerene with β-enamino carbonyl compounds mediated by manganese(III) acetate†

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Manganese(III) acetate dihydrate-mediated reactions of [60]fullerene with β-enamino carbonyl compounds afforded [60]fullerene-fused pyrroline derivatives, of which the nitrogen atom is directly connected to the fullerene cage. A possible reaction mechanism is proposed.

Introduction

Chemical modification of [60]fullerene (C_{60}) to generate novel C_{60} derivatives with new structures and properties has attracted great attention over the last two decades for their potential applications in materials, biology and nanoscience.[1–3] Free radical reactions were one of the first investigated reactions of fullerenes[4,5] and are still attractive protocols to synthesize fullerene derivatives.[6–10] Over the past decades, manganese(III)-mediated free radical reactions have been explored extensively, have found widespread applications in organic synthesis, and have demonstrated remarkable advantages over traditional peroxide or light-initiated processes.[11–13] We have successfully applied manganese(III) acetate dihydrate ($Mn(OAc)_3\cdot 2H_2O$) to the free radical reactions of C_{60}.[14–18] The $Mn(OAc)_3\cdot 2H_2O$-mediated reactions of C_{60} with various active methylene compounds and aromatic methyl ketones afforded 1,4-adducts and 1,16-adducts of C_{60},[14,15] singly-bonded fullerene dimers,[14] C_{60}-fused dihydrofuran derivatives[16] and methanofullerenes.[14,16] The *in situ* generated ArC_{60}–H could be transformed to ArC_{60}–OAc by $Mn(OAc)_3\cdot 2H_2O$ in a one-pot procedure.[17] In our recent work, we found that $Mn(OAc)_3\cdot 2H_2O$-mediated reactions of C_{60} with carboxylic acids, carboxylic anhydrides, or malonic acids gave C_{60}-fused lactones, which underwent novel reductive ring opening by reacting with Grignard reagents.[18] An independent work on the $Mn(OAc)_3\cdot 2H_2O$-mediated reactions of C_{60} with malonate esters, β-keto esters and β-diketones in chlorobenzene and/or toluene was reported by Gao and co-workers.[19] In continuation of our interest in $Mn(OAc)_3\cdot 2H_2O$-mediated reactions of C_{60},[14–18] herein we report the radical reactions of C_{60} with β-enamino carbonyl compounds mediated by $Mn(OAc)_3\cdot 2H_2O$ to give C_{60}-fused pyrroline derivatives.

Results and discussion

β-Enamino carbonyl compounds have a vinyl C–H and an active N–H bond, while the enol forms of β-keto esters or β-diketones similarly possess a vinyl C–H and an active O–H bond (Fig. 1).

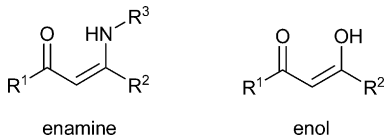

Fig. 1

We and Gao's group have already reported that the $Mn(OAc)_3\cdot 2H_2O$-mediated reactions of C_{60} with β-keto esters and β-diketones afforded C_{60}-fused dihydrofuran derivatives.[16,19] We were wondering if β-enamino carbonyl compounds could react with C_{60} in a similar way to β-keto esters and β-diketones. To our satisfaction, the $Mn(OAc)_3\cdot 2H_2O$-mediated reactions of C_{60} with β-enamino carbonyl compounds **1** in refluxing chlorobenzene gave the C_{60}-fused pyrroline derivatives **2** (Scheme 1).

To examine the scope and limitation of the used substrates, we synthesized various β-enamino carbonyl compounds **1a–1h** by the reactions of 1,3-dicarbonyl compounds such as 5,5-dimethyl-1,3-cyclohexanedione, acetoacetate esters and 2,4-pentanedione with both aromatic amines (aniline and 4-methylaniline) and aliphatic amines (benzyl amine and *n*-butylamine), and applied them to the $Mn(OAc)_3\cdot 2H_2O$-mediated reactions of C_{60}. The reaction times and isolated yields along with recovered C_{60} for the reactions of C_{60} with β-enamino carbonyl compounds **1a–1h** and $Mn(OAc)_3\cdot 2H_2O$ in a molar ratio of 1 : 2 : 2.5 in chlorobenzene at 140 °C are listed in Table 1.

As seen from Table 1, all examined substrates could react with C_{60}. Enaminones **1a** and **1b** derived from cyclic β-diketone and aromatic amine were most reactive and gave the highest yields (61–62%, 85–93% based on consumed C_{60}), while the β-enamino carbonyl compound **1h** derived from noncyclic β-diketone and aliphatic amine afforded the lowest yield and required a long reaction time. The exact reason for this phenomenon is not clear right now. Further extension of the reaction time for **1h** resulted in more consumption of C_{60}, but could not improve the product yield. It should be noted that the addition of a base such as 4-dimethylaminopyridine and the presence of air had a negligible effect on the reactions. Products **2a–2h** are not very stable, and tend to decompose in solution upon storage. Fullerene derivatives containing the enamine moiety were reported to photochemically react with oxygen,[20–22] thus the instability of compounds **2a–2h** were most likely due to their reactions with aerial oxygen during storage.

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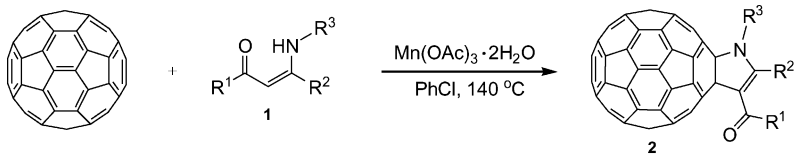
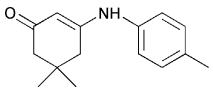
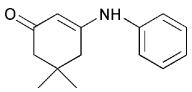
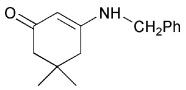
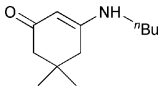
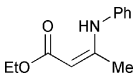
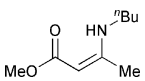
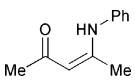
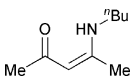


1a, **2a**: R^1, R^2 = $CH_2C(CH_3)_2CH_2$, R^3 = 4-CH_3Ph; **1b**, **2b**: R^1, R^2 = $CH_2C(CH_3)_2CH_2$, R^3 = Ph;
1c, **2c**: R^1, R^2 = $CH_2C(CH_3)_2CH_2$, R^3 = CH_2Ph; **1d**, **2d**: R^1, R^2 = $CH_2C(CH_3)_2CH_2$, R^3 = *n*-C_4H_9;
1e, **2e**: R^1 = OCH_2CH_3, R^2 = CH_3, R^3 = Ph; **1f**, **2f**: R^1 = OCH_3, R^2 = CH_3, R^3 = *n*-C_4H_9;
1g, **2g**: R^1 = R^2 = CH_3, R^3 = Ph; **1h**, **2h**: R^1 = R^2 = CH_3, R^3 = *n*-C_4H_9.

Scheme 1

Table 1 Reaction times, yields and recovered C_{60} for the reactions of C_{60} with **1a–1h** mediated by $Mn(OAc)_3 \cdot 2H_2O$ at 140 °C

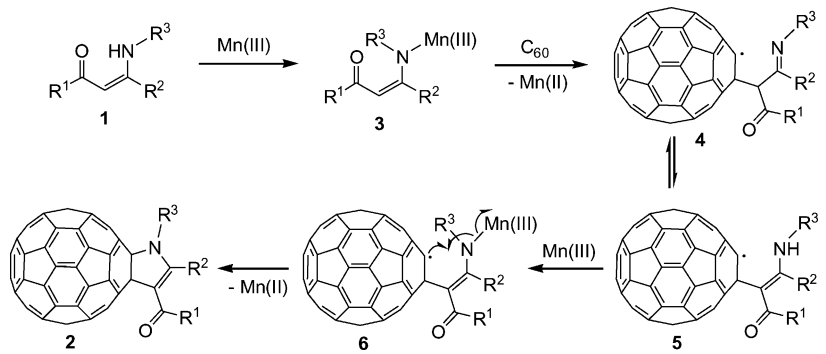
Entry	Substrate **1**	Product **2**	Reaction time/min	Yield (%)	Recovered C_{60} (%)
1		**2a**	15	61	28
2		**2b**	15	62	33
3		**2c**	45	38	35
4		**2d**	10	36	56
5		**2e**	45	34	53
6		**2f**	100	32	47
7		**2g**	15	32	52
8		**2h**	90	16	71

The structures of C_{60}-fused pyrroline derivatives **2a–2h** were fully established by their MS, ^{1}H NMR, ^{13}C NMR, FT-IR and UV-vis spectral data. The negative APCI mass spectra of **2a–2h** showed the correct molecular ion peaks. The ^{13}C NMR spectra of **2a–2h** clearly exhibited less than thirty peaks with two half-intensity ones in the range of 133–150 ppm for the sp^2-carbons of the fullerene cage, and two peaks at 88–91 and 72–74 ppm for the two sp^3-carbons of the fullerene skeleton, consistent with the C_s symmetry of their molecular structures.

A possible reaction mechanism similar to the $Mn(OAc)_3 \cdot 2H_2O$-mediated reactions of C_{60} with β-keto esters and β-diketones[16,19] is shown in Scheme 2 to elucidate the formation of **2a–2h**.

Chelation of $Mn(OAc)_3$ by the enamine nitrogen of **1** with the loss of acetic acid results in the formation of Mn(III)-complex **3**.[23,24] Homolytical addition of **3** to C_{60} gives fullerene radical **4**, which equilibrates to fullerene radical **5** with an enamine structure. Coordination of $Mn(OAc)_3$ by intermediate **5** generates Mn(III)-complex **6** that undergoes intramolecular cyclization with the loss of Mn(II) species to afford product **2**.

C_{60}-fused pyrroline derivatives have been synthesized by the reactions of C_{60} with nitrile ylides generated by photolysis of 2*H*-azirines[25,26] or from imidoyl chloride and triethylamine,[27,28] with a cyclic azomethine ylide formed from 2-phenyl-4,5-dihydrooxazol-5-one,[29] with isocyanides catalyzed by a base or Cu_2O,[30] and with



Scheme 2

N-(diphenylmethylene)glycinate esters under Bingel conditions.[31] However, none of them have a structure with a nitrogen atom bonded to a fullerene cage. Our present protocol is a unique way of preparing C_{60}-fused pyrroline derivatives with a nitrogen atom directly attached to a fullerene skeleton.

In summary, $Mn(OAc)_3 \cdot 2H_2O$ has been successfully utilized in the radical reactions of C_{60} with β-enamino carbonyl compounds to give C_{60}-fused pyrroline derivatives, of which the nitrogen atom is directly connected to the fullerene cage. Further application of $Mn(OAc)_3 \cdot 2H_2O$ and other inorganic compounds in fullerene chemistry is underway.

Experimental

General methods

^{1}H NMR and ^{13}C NMR spectra were recorded in CS_2–$CDCl_3$ at 300 MHz and 75 MHz, respectively, on a Bruker Avance 300 spectrometer. Negative APCI mass spectra were taken on a Thermo Finnigan LCQ Advantage MAX mass spectrometer. FT-IR spectra were recorded on a Shimadzu 8600 FT IR spectrometer. UV-vis spectra were obtained on a Shimadzu UV-2501PC spectrometer. C_{60} (>99.9%) was purchased from the Henan Tian'an Company. All other commercial available reagents are of analytical grade. Coupling constants are measured in Hz.

Typical procedure for the $Mn(OAc)_3 \cdot 2H_2O$-mediated reactions of C_{60} with β-enamino carbonyl compounds 1a–1h.

A mixture of C_{60} (36.0 mg, 0.05 mmol), β-enamino carbonyl compounds **1a–1h** (0.10 mmol) and $Mn(OAc)_3 \cdot 2H_2O$ (33.4 mg, 0.125 mmol) was dissolved in chlorobenzene (15 mL) and stirred in an oil bath preset at 140 °C for a desired time. After removal of the solvent *in vacuo*, the obtained residue was separated on a silica gel column with toluene or toluene–ethyl acetate as the eluent to give unreacted C_{60} and C_{60}-fused pyrroline derivatives **2a–2h**.

Spectral data of 2a. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 7.49 (d, $J = 8.1$, 2H), 7.32 (d, $J = 8.1$, 2H), 2.58 (s, 2H), 2.51 (s, 2H), 2.44 (s, 3H), 1.34 (s, 6H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with $Cr(acac)_3$ as relaxation reagent) (all 2C unless indicated) δ 189.75 (1C, *C*=O), 163.47 (1C, N*C*=C), 149.20, 147.68 (1C), 147.36, 146.95 (1C), 146.30, 145.84 (4C), 145.78, 145.76, 145.74, 145.22, 145.13, 144.98, 144.85, 144.69, 144.14, 143.75, 142.79, 142.58, 142.49, 142.41 (4C), 141.78 (4C), 141.75, 141.60, 139.82, 139.37 (1C, aryl *C*), 139.28, 136.90, 134.13, 134.00 (1C, aryl *C*), 130.53 (aryl *C*), 130.49 (aryl *C*), 106.45 (1C, NC=*C*), 91.33 (1C, sp^3-*C* of C_{60}), 71.96 (1C, sp^3-*C* of C_{60}), 51.44 (1C, OC*C*H$_2$), 38.21 (1C, NC*C*H$_2$), 33.85 (1C, *C*(CH$_3$)$_2$), 28.68 (C(*C*H$_3$)$_2$), 21.39 (1C, Ar*C*H$_3$); UV-vis ($CHCl_3$) λ_{max} nm (log ε) 256 (5.14), 313 (4.73), 428 (3.48), 690 (2.44); FT-IR ν/cm^{-1} (KBr) 2955, 2922, 2851, 1634, 1581, 1511, 1438, 1387, 1319, 1273, 1179, 1121, 1065, 1036, 806, 609, 575, 550, 527; MS (–APCI) *m*/*z* 947.

Spectral data of 2b. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 7.63 (d, $J = 7.3$, 2H), 7.54 (t, $J = 7.2$, 2H), 7.48 (t, $J = 7.2$, 1H), 2.59 (s, 2H), 2.53 (s, 2H), 1.35 (s, 6H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with $Cr(acac)_3$ as relaxation reagent) (all 2C unless indicated) δ 190.06 (1C, *C*=O), 163.28 (1C, N*C*=C), 149.07, 147.61 (1C), 147.24, 146.88 (1C), 146.22, 145.76 (4C), 145.71, 145.69, 145.66, 145.14, 144.94, 144.91, 144.77, 144.60, 143.99, 143.67, 142.71, 142.50, 142.40, 142.32 (4C), 141.68 (6C), 141.50, 139.77, 139.20, 136.85, 136.61 (1C, aryl *C*), 134.08, 130.66 (aryl *C*), 129.83 (aryl *C*), 129.19 (1C, aryl *C*), 106.67 (1C, NC=*C*), 91.20 (1C, sp^3-*C* of C_{60}), 71.95 (1C, sp^3-*C* of C_{60}), 51.44 (1C, CO*C*H$_2$), 38.11 (1C, NC*C*H$_2$), 33.86 (1C, *C*(CH$_3$)$_2$), 28.59 (C(*C*H$_3$)$_2$); UV-vis ($CHCl_3$) λ_{max} nm (log ε) 254 (5.13), 313 (4.72), 428 (3.46), 689 (2.41); FT-IR ν/cm^{-1} (KBr) 2953, 2925, 2866, 1633, 1581, 1517, 1493, 1431, 1386, 1320, 1273, 1179, 1120, 1065, 1002, 766, 701, 606, 574, 553, 527; MS (–APCI) *m*/*z* 933.

Spectral data of 2c. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 7.50 (d, $J = 7.4$, 2H), 7.39 (t, $J = 7.4$, 2H), 7.30 (t, $J = 7.2$, 1H), 5.47 (s, 2H), 2.78 (s, 2H), 2.53 (s, 2H), 1.37 (s, 6H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with $Cr(acac)_3$ as relaxation reagent) (all 2C unless indicated) δ 189.74 (1C, *C*=O), 164.13 (1C, N*C*=C), 149.89, 147.78 (1C), 147.44, 147.07 (1C), 146.41, 145.94 (6C), 145.88, 145.78, 145.30, 145.10, 144.95, 144.83, 144.67, 143.80, 143.59, 142.93, 142.68, 142.57, 142.50, 142.46, 141.94, 141.90, 141.86, 141.64, 139.91, 139.44, 137.20, 136.66 (1C, aryl *C*), 134.18, 129.14 (aryl *C*), 128.03 (1C, aryl *C*), 126.49 (aryl *C*), 105.75 (1C, NC=*C*), 89.94 (1C, sp^3-*C* of C_{60}), 72.05 (1C, sp^3-*C* of C_{60}), 51.30 (1C, CO*C*H$_2$), 47.76 (1C, Ph*C*H$_2$), 37.78 (1C, NC*C*H$_2$), 34.06 (1C, *C*(CH$_3$)$_2$), 28.84 (C(*C*H$_3$)$_2$); UV-vis ($CHCl_3$) λ_{max} nm (log ε) 256 (5.12), 314 (4.71), 428 (3.48), 689 (2.42); FT-IR ν/cm^{-1} (KBr) 2952, 2922, 2851, 1626, 1579, 1510, 1466, 1426, 1389, 1351, 1263,

1178, 1115, 1057, 735, 695, 604, 573, 550, 526; MS (–APCI) *m*/*z* 947.

Spectral data of 2d. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 4.20–4.12 (m, 2H), 2.86 (s, 2H), 2.50 (s, 2H), 2.08–1.95 (m, 2H), 1.60–1.41 (m, 2H), 1.41 (s, 6H), 1.04 (t, J = 7.3, 3H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with Cr(acac)$_3$ as relaxation reagent) (all 2C unless indicated) δ 189.12 (1C, *C*=O), 163.59 (1C, N*C*=C), 149.95, 147.87 (1C), 147.53, 147.11 (1C), 146.50, 146.01, 145.98, 145.96, 145.94, 145.89, 145.31, 145.16, 145.01, 144.93, 144.72, 143.97, 143.88, 143.01, 142.74, 142.65, 142.62, 142.55, 142.04, 141.97, 141.93, 141.81, 139.96, 139.62, 137.33, 134.14, 105.10 (1C, NC=*C*), 89.82 (1C, sp^3-*C* of C_{60}), 72.27 (1C, sp^3-*C* of C_{60}), 51.35 (1C, CO*C*H$_2$), 44.39 (1C, N*C*H$_2$), 37.83 (1C, NC*C*H$_2$), 34.02 (1C, NCH$_2$*C*H$_2$), 33.66 (1C, *C*(CH$_3$)$_2$), 29.00 (C(*C*H$_3$)$_2$), 20.79 (1C, *C*H$_2$CH$_3$), 14.08 (1C, CH$_2$*C*H$_3$); UV-vis (CHCl$_3$) λ_{max} nm (log ε) 255 (5.10), 314 (4.67), 428 (3.47), 690 (2.39); FT-IR ν/cm^{-1} (KBr) 2953, 2924, 2853, 1623, 1574, 1511, 1471, 1430, 1392, 1183, 1115, 1047, 1001, 573, 552, 526; MS (–APCI) *m*/*z* 913.

Spectral data of 2e. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 7.62 (d, J = 7.1, 2H), 7.52 (t, J = 7.3, 2H), 7.45 (t, J = 7.2, 1H), 4.30 (q, J = 7.2, 2H), 2.60 (s, 3H), 1.28 (t, J = 7.2, 3H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with Cr(acac)$_3$ as relaxation reagent) (all 2C unless indicated) δ 165.78 (1C, *C*OO), 159.57 (1C, N*C*=C), 149.51, 147.99, 147.90 (1C), 147.36 (1C), 146.49, 146.17, 146.11, 146.05, 145.98, 145.83, 145.32, 145.19, 145.11, 145.04, 144.81, 144.79, 144.14, 142.99, 142.84, 142.68 (4C), 142.65, 142.10, 142.01, 141.88, 141.80, 139.50, 139.17, 137.71, 136.77 (1C, aryl *C*), 134.79, 131.67 (aryl *C*), 129.94 (aryl *C*), 129.23 (1C, aryl *C*), 98.13 (1C, NC=*C*), 90.45 (1C, sp^3-*C* of C_{60}), 73.92 (1C, sp^3-*C* of C_{60}), 59.71 (1C, O*C*H$_2$CH$_3$), 15.37 (1C, C=C*C*H$_3$), 14.60 (1C, OCH$_2$*C*H$_3$); UV-vis (CHCl$_3$) λ_{max} nm (log ε) 256 (5.06), 309 (4.60), 427 (3.41), 693 (2.41); FT-IR ν/cm^{-1} (KBr) 2953, 2922, 2852, 1680, 1581, 1493, 1431, 1370, 1330, 1313, 1232, 1173, 1130, 1096, 1024, 763, 700, 575, 527; MS (–APCI) *m*/*z* 923.

Spectral data of 2f. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 4.17–4.11 (m, 2H), 3.76 (s, 3H), 2.88 (s, 3H), 2.04–1.93 (m, 2H), 1.55–1.45 (m, 2H), 1.01 (t, J = 7.3, 3H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with Cr(acac)$_3$ as relaxation reagent) (all 2C unless indicated) δ 165.78 (1C, *C*OO), 159.64 (1C, N*C*=C), 149.78, 147.64 (1C), 147.51, 147.08 (1C), 146.25, 145.84, 145.82, 145.79, 145.70, 145.61, 144.95, 144.92, 144.76, 144.60, 144.40, 144.03, 143.82, 142.89, 142.78, 142.58, 142.45, 142.43, 142.35, 141.86 (4C), 141.67, 141.56, 139.43, 139.01, 136.81, 134.30, 95.54 (1C, NC=*C*), 88.75 (1C, sp^3-*C* of C_{60}), 73.58 (1C, sp^3-*C* of C_{60}), 50.13 (1C, O*C*H$_3$), 44.01 (1C, N*C*H$_2$), 32.97 (1C, NCH$_2$*C*H$_2$), 20.53 (1C, *C*H$_2$CH$_3$), 13.86 (1C, *C*H$_3$), 13.84 (1C, *C*H$_3$); UV-vis (CHCl$_3$) λ_{max} nm (log ε) 256 (5.10), 309 (4.62), 427 (3.46), 690 (2.38); FT-IR ν/cm^{-1} (KBr) 2951, 2923, 2854, 1673, 1575, 1461, 1422, 1367, 1337, 1304, 1186, 1132, 1080, 1007, 932, 791, 774, 759, 574, 527; MS (–APCI) *m*/*z* 889.

Spectral data of 2g. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 7.65 (d, J = 7.0, 2H), 7.59–7.46 (m, 3H), 2.67 (s, 3H), 2.59 (s, 3H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with Cr(acac)$_3$ as relaxation reagent) (all 2C unless indicated) δ 190.70 (1C, *C*=O), 158.41 (1C, N*C*=C), 149.45, 148.35, 147.76 (1C), 147.12 (1C), 146.46, 146.03, 145.96, 145.92, 145.85, 145.79, 145.30 (4C), 145.06, 144.99, 144.93, 144.79, 143.96, 142.82, 142.72, 142.68, 142.50, 142.40, 141.92, 141.87, 141.65 (4C), 139.34, 138.86, 137.16 (1C, aryl *C*), 136.81, 134.36, 131.62 (aryl *C*), 129.91 (aryl *C*), 129.50 (1C, aryl *C*), 110.31 (1C, NC=*C*), 89.66 (1C, sp^3-*C* of C_{60}), 73.97 (1C, sp^3-*C* of C_{60}), 31.21 (1C, CO*C*H$_3$), 16.24 (1C, *C*H$_3$); UV-vis (CHCl$_3$) λ_{max} nm (log ε) 257 (5.08), 314 (4.69), 429 (3.48), 689 (2.36); FT-IR ν/cm^{-1} (KBr) 2921, 2851, 1616, 1557, 1513, 1490, 1426, 1379, 1357, 1334, 1170, 1127, 1071, 1025, 765, 701, 575, 552, 527; MS (–APCI) *m*/*z* 893.

Spectral data of 2h. ^{1}H NMR (300 MHz, CS_2–$CDCl_3$) δ 4.30–4.24 (m, 2H), 2.89 (s, 3H) 2.61 (s, 3H), 2.11–2.01 (m, 2H), 1.62–1.49 (m, 2H), 1.08 (t, J = 7.3, 3H); ^{13}C NMR (75 MHz, CS_2–$CDCl_3$, with Cr(acac)$_3$ as relaxation reagent) (all 2C unless indicated) δ 188.77 (1C, *C*=O), 156.91 (1C, N*C*=C), 149.55, 148.13, 147.50 (1C), 146.82 (1C), 146.24, 145.72 (4C), 145.63, 145.57 (4C), 144.97, 144.79, 144.67, 144.64, 144.61, 144.25, 143.65, 142.66, 142.50, 142.47, 142.32, 142.12, 141.79, 141.62, 141.44, 141.42, 139.30, 138.49, 136.89, 133.86, 108.35 (1C, NC=*C*), 87.94 (1C, sp^3-*C* of C_{60}), 74.05 (1C, sp^3-*C* of C_{60}), 44.06 (1C, N*C*H$_2$), 33.35 (1C, NCH$_2$*C*H$_2$), 31.01 (1C, CO*C*H$_3$), 20.65 (1C, *C*H$_2$CH$_3$), 14.73 (1C, *C*H$_3$), 13.86 (1C, *C*H$_3$); UV-vis (CHCl$_3$) λ_{max} nm (log ε) 256 (5.08), 314 (4.68), 429 (3.60), 687 (2.42); FT-IR ν/cm^{-1} (KBr) 2954, 2925, 2867, 1625, 1583, 1459, 1431, 1376, 1360, 1324, 1260, 1184, 1119, 1086, 1034, 976, 942, 575, 527; MS (–APCI) *m*/*z* 873.

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