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A simple and efficient approach to the synthesis of highly functionalized fused benzochromenes

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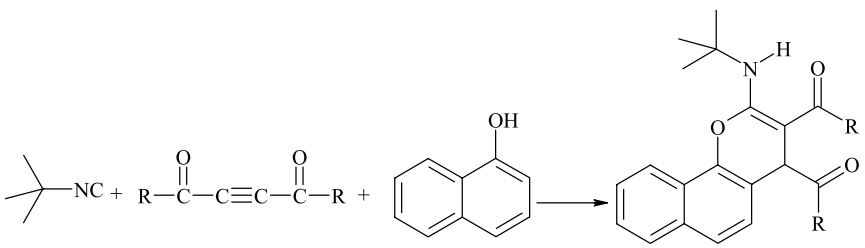
Abstract—Protonation of the reactive intermediates produced in the reaction between *tert*-butyl isocyanide and dimethyl acetylenedicarboxylate or dibenzoylacetylene, by 1-naphthol, 2-naphthol, 2,3-dihydroxynaphthalene, 2,7-dihydroxynaphthalene or 4-methyl-8-hydroxycoumarin leads to vinylnitrilium cations, which undergo carbon-centered Michael type addition with the conjugate base of the naphthols to produce highly functionalized benzochromenes.

1. Introduction

There has been considerable interest in chromenes and their benzo-derivatives, not least because of their value for a variety of industrial, biological, and chemical synthetic uses.[1] As a result, a large number of methods have appeared describing novel synthesis of these heterocycles.[2] 2-Amino-4*H*-benzochromenes have been of interest because of their biological activity[3] and a few methods have been reported for their synthesis.[4] As part of our current studies[5–8] on the development of new routes to heterocyclic systems, we now report an efficient synthetic route to polysubstituted benzochromenes using *tert*-butyl isocyanide and dibenzoylacetylene or dimethyl acetylenedicarboxylate (DMAD) in the presence of a naphthol such as 1-naphthol, 2-naphthol, 2,7-dihydroxynaphthalene, 2,3-dihydroxynaphthalene or 4-methyl-8-hydroxycoumarin.

2. Results and discussion

The reaction of DMAD with *tert*-butyl isocyanide in the presence of 1-naphthol, proceeded spontaneously at room temperature in dichloromethane, and produced dimethyl 2-*tert*-butylamino-4*H*-benzo[*h*]chromene-3,4-dicarboxylate (**3a**) (Scheme 1). The structure of **3a** was determined on the basis of its elemental analyses, mass spectrometry (MS), ^{1}H and ^{13}C NMR and IR spectral data. The MS spectrum of **3a** displayed molecular ion (M^+) peak at 369 *m*/*z*, which is consistent with 1:1:1 adduct of DMAD, *tert*-butyl isocyanide and 1-naphthol. The ^{1}H NMR spectrum of **3a** exhibited four singlets identified as *tert*-butyl (δ=1.75), methoxy (δ=3.65 and 3.74) and methine (δ=4.84) protons along with multiplets (δ=7.2–8.5) for the aromatic protons. The NH proton resonance at δ=9.18 disappeared after addition of D_2O to the $CDCl_3$ solution of **3a**. The proton-decoupled ^{13}C



1 **2**

1,3	R	%Yield of 3
a	OMe	94
b	Ph	70

Scheme 1.

Keywords: benzochromene; isocyanide; acetylenic compounds; addition reaction.

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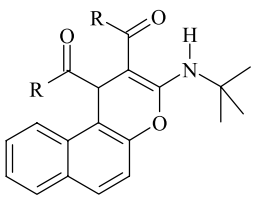
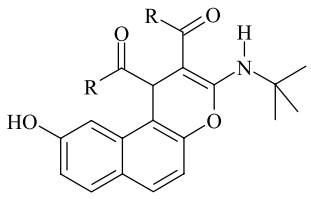
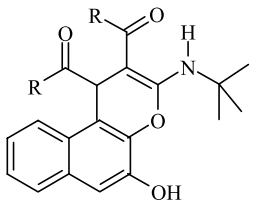


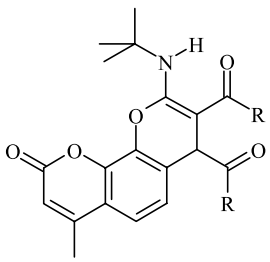
4	R	%Yield
a	OMe	93
b	Ph	75



5	R	%Yield
a	OMe	85
b	Ph	65



6	R	%Yield
a	OMe	90
b	Ph	57



7	R	%Yield
a	OMe	85
b	Ph	50

Scheme 2.

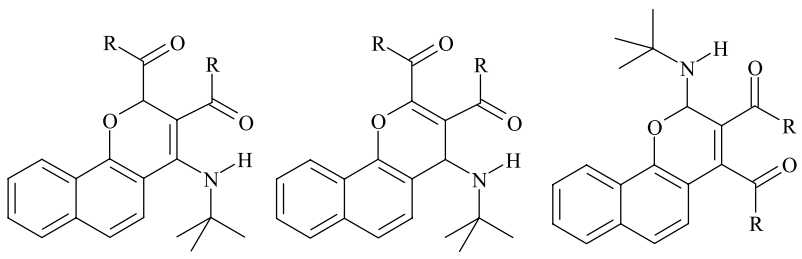
NMR spectrum of **3a** showed 19 distinct resonance in agreement with the proposed structure. The presence of oxo and amino groups at one end of the double bond strongly polarize the olefinic system. The α-carbon of these substituents appears at δ=161.3 and the β-carbon at δ=71.5. The carbonyl groups appear at δ=169.9 and 174.1. These signals along with the downfield shift of the NH group support an enaminocarbonyl structure with six-membered intramolecular hydrogen bonding.[9]

The reaction of dibenzoylacetylene, *tert*-butyl isocyanide, and 1-naphthol leads to product **3b**. The ^{1}H and ^{13}C NMR spectra of **3b** are similar to those for **3a** except for the ester moieties, which are replaced by benzoyl groups.

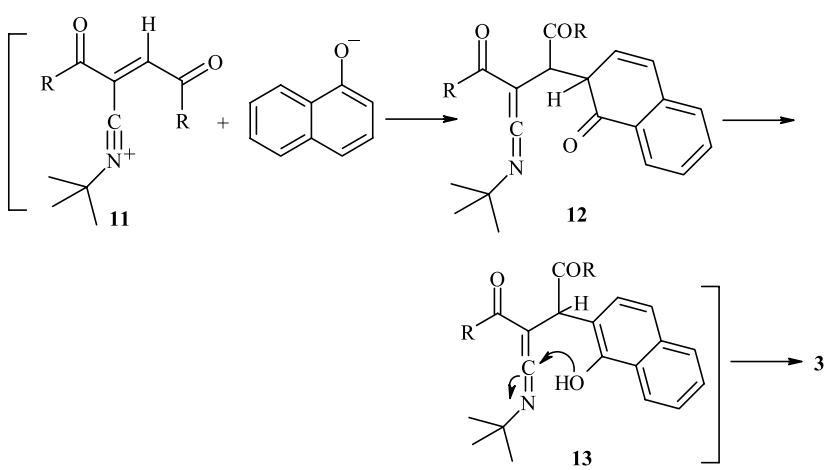
Using dibenzoylacetylene or DMAD with other hydroxy acids, such as 2-naphthol, 2,7-dihydroxynaphthalene, 2,3-dihydroxynaphthalene, or 4-methyl-8-hydroxycoumarin, in the presence of *tert*-butyl isocyanide, produced compounds **4**, **5**, **6**, and **7**, respectively (Scheme 2).

Structure **3** was distinguished from structures **8** and **10** (Scheme 3) on the basis of the ^{13}C NMR chemical shift of the methine group (δ=35.5–45.1 ppm). The methine group in **8** or **10** is expected to appear at about δ=60 ppm. Since the ^{1}H NMR signal of the saturated methine group exhibits a sharp singlet in different solvents, we exclude structures **9** and **10**, which are expected to show vicinal coupling for the HC–NH moiety. Moreover, the ^{1}H and ^{13}C chemical shifts are in better agreement with the enamino-ester **3**.

Although we have not yet established the mechanism of the reaction between *tert*-butyl isocyanide and electron deficient acetylenic compounds in the presence of 1-naphthol in an experimental manner, a possible explanation is proposed in Scheme 4.



Scheme 3.



Scheme 4.

On the basis of the well established chemistry of isocyanides[10–14] it is reasonable to assume that compound **3** results from nucleophilic addition of *tert*-butyl isocyanide to the acetylenic system and subsequent protonation of the 1:1 adduct by 1-naphthol. Then, the positively charged ion **11** is attacked by the enolate anion of 1-naphthol to produce the ketenimine **12**. Such an addition product may tautomerize and cyclize, under the reaction conditions employed, to produce **3**. Similar mechanistic scheme can be considered for formation of compounds **4**, **5**, **6**, or **7**.

In summary, we have found a simple and efficient method for the preparation of some highly functionalized benzochromene ring systems. The present method carries the advantage that not only is the reaction performed under neutral conditions, but also the starting materials and reagents can be mixed without any activation or modification.

3. Experimental

Melting points were measured on an Electrothermal 9100 apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. IR spectra were measured on a Shimadzu IR-460 spectrometer. ^{1}H and ^{13}C NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500.1 and 125.8 MHz, respectively. Dibenzoylacetylene was prepared by addition of ethynylmagnesium bromide to benzaldehyde[15] and subsequent oxidation[16] of the produced diol. Other chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and used without further purification.

3.1. General procedure

To a magnetically stirred solution of dibenzoylacetylene or DMAD (2 mmol) and the naphthol (2 mmol) in dichloromethane (20 mL) was added a mixture of *tert*-butyl isocyanide (0.17 g, 2 mmol) in dichloromethane (5 mL) at room temperature. The reaction mixture was then allowed to stir for 24 h. The solvent was removed under reduced pressure, and the residue was separated by silica gel (Merck 230–400 mesh) column chromatography using hexane–ethyl acetate mixture as eluent.

3.1.1. Dimethyl 2-*tert*-butylamino-4*H*-benzo[*h*]chromene-3,4-dicarboxylate (3a). White powder, 0.69 g, yield 94%, mp 120–122°C. IR (KBr) (ν_{max}/cm^{-1}): 3350, 1692, 1664, 1605. MS, *m*/*z* (%): 369 (M^+, 3), 310 (M^+-CO_2Me, 38), 254 [310−$(CH_3)_2CCH_2$, 100], 223 (254−OMe, 91). Anal. calcd for $C_{21}H_{23}NO_5$ (369.4): C, 68.28; H, 6.28; N, 3.79; found: C, 68.2; H, 6.3; N, 3.7%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.75 (s, 9H, *t*-Bu), 3.65 and 3.74 (2s, 6H, 2OMe), 4.84 (s, 1H, CH), 7.20–8.50 (m, 6H, 6CH), 9.18 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=30.37 ($3CH_3$), 41.53 (CH), 50.94, 52.32, and 52.34 ($2OCH_3$ and N–C), 71.50 (C), 114.10 (C), 121.02, 123.42, 124.19 (3CH), 125.54, and 125.80 (2C), 126.51, 127.82, and 133.69 (3CH), 144.15, and 161.25 (2C), 169.93, and 174.12 (2C=O).

3.1.2. (4-Benzoyl-2-*tert*-butylamino-4*H*-benzo[*h*]chromene-3-yl)-phenyl-methanone (3b). White powder, 0.65 g, yield 70%, mp 198–200°C. IR (KBr) (ν_{max}/cm^{-1}): 3390, 1668, 1636, 1610. MS, *m*/*z* (%): 461 (M^+, 4), 356 (M^+-COPh, 25), 300 [356−$(CH_3)_2CCH_2$, 39], 105 (COPh, 100). Anal. calcd for $C_{31}H_{27}NO_3$ (461.6): C, 80.67; H, 5.90; N, 3.03; found: C, 80.5; H, 6.0; N, 3.1%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.60 (s, 9H, *t*-Bu), 5.74 (s, 1H, CH), 6.80–8.50 (m, 16H, 16CH), 12.05 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=30.23 ($3CH_3$), 45.14 (CH), 52.90 (N–C), 84.78, and 116.31 (2C), 121.10 (CH), 123.76 (C), 124.30, 125.34, 126.28, 126.61, 126.81, 127.94, 128.37, 128.54, 128.60, 128.66, and 132.78 (15CH), 133.63, 136.28, 141.86, 144.72, and 163.52 (5C), 191.35, and 200.36 (2C=O).

3.1.3. Dimethyl 3-*tert*-butylamino-1*H*-benzo[*f*]chromene-1,2-dicarboxylate (4a). White powder, 0.68 g, yield 93%, mp 134–137°C. IR (KBr) (ν_{max}/cm^{-1}): 3230, 1728, 1652. MS *m*/*z* (%): 369 (M^+, 2), 310 (M^+-CO_2Me, 28), 254 [310−$(CH_3)_2CCH_2$, 80], 223 (254−OMe, 100). Anal. calcd for $C_{21}H_{23}NO_5$ (369.4): C, 68.28; H, 6.28; N, 3.79; found: C, 68.3; H, 6.3; N, 3.7%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.59 (s, 9H, *t*-Bu), 3.61 and 3.78 (2s, 6H, 2OMe), 5.42 (s, 1H, CH), 7.18–8.42 (m, 6H, 6CH), 8.89 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=30.60 ($3CH_3$), 38.29 (CH), 50.97, 52.23, and 52.52 ($2OCH_3$ and N–C), 72.58 (C), 114.13 (C), 116.56, 123.62, 125.06, 127.23, 128.37, and 129.43 (6CH), 131.04, and 131.21 (2C), 147.30, and 162.28 (2C), 169.71, and 173.42 (2C=O).

3.1.4. (1-Benzoyl-3-*tert*-butylamino-1*H*-benzo[*f*]chromene-2-yl)-phenyl-methanone (4b). White powder, 0.69 g, yield 75%, mp 208–210°C. IR (KBr) (ν_{max}/cm^{-1}): 3385, 1668, 1621. MS, *m*/*z* (%): 461 (M^+, 4), 356 (M^+-COPh, 10), 300 (356−$(CH_3)_2CCH_2$, 39), 105 (COPh, 100). Anal. calcd for $C_{31}H_{27}NO_3$ (461.6): C, 80.67; H, 5.90; N, 3.03; found: C, 80.4; H, 6.0; N, 3.1%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.60 (s, 9H, *t*-Bu), 6.38 (s, 1H, CH), 7.17–7.85 (m, 16H, 16CH), 11.49 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=30.31 ($3CH_3$), 41.14 (CH), 53.12 (N–*C*), 84.71 (C), 116.70 (CH), 116.88 (C), 122.18, 124.99, 127.34, 127.48, 128.34, 128.55, 128.75, 128.77, 129.25, and 129.57 (14CH), 130.66, and 131.19 (2C), 132.58 (CH), 135.92, 141.37, 148.24, and 164.02 (4C), 190.82, and 198.97 (2C=O).

3.1.5. Dimethyl 3-*tert*-butylamino-9-hydroxy-1*H*-benzo[*f*]chromene-1,2-dicarboxylate (5a). White powder, 0.65 g, yield 85%, mp 162–164°C. IR (KBr) (ν_{max}/cm^{-1}): 3400, 1699, 1669. MS, *m*/*z* (%): 385 (M^+, 1), 326 (M^+-CO_2Me, 79), 270 [326−$(CH_3)_2CCH_2$, 100], 239 (270−OMe, 94). Anal. calcd for $C_{21}H_{23}NO_6$ (385.4): C, 65.44; H, 6.02; N, 3.63; found: C, 65.5; H, 6.1; N, 3.7%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.75 (s, 9H, *t*-Bu), 3.65 and 3.74 (2s, 6H, 2OMe), 5.30 (s, 1H, CH), 7.20–8.50 (m, 7H, 6CH and OH), 8.90 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=30.50 ($3CH_3$), 38.69 (CH), 50.94, 52.36, and 52.53 ($2OCH_3$ and N–C), 72.32 (C), 106.03, 112.10, and 113.65 (3CH), 117.03 (C), 126.03 (CH), 129.25, and 130.44 (2C), 132.74 (CH), 147.69, 155.74, and 162.23 (3C), 169.90, and 174.08 (2C=O).

3.1.6. (1-Benzoyl-3-*tert*-butylamino-9-hydroxy-1*H*-benzo[*f*]chromene-2-yl)-phenyl-methanone (5b). White

powder, 0.62 g, yield 65%, mp 202–204°C. IR (KBr) (ν_{max}/cm^{-1}): 3370, 3160, 1672, 1625. MS, *m/z* (%): 477 (M^+, 2), 372 (M^+-COPh, 5), 316 [372−$(CH_3)_2CCH_2$, 12], 105 (COPh, 100). Anal. calcd for $C_{31}H_{27}NO_4$ (477.6): C, 77.97; H, 5.70; N, 2.93; found: C, 77.7; H, 5.7; N, 3.0%. ^{1}H NMR (500 MHz, DMSO-d_6, 25°C): δ=1.52 (s, 9H, *t*-Bu), 6.09 (s, 1H, CH), 6.95–7.61 (m, 13H, 13CH), 7.80 (d, $^3J_{H,H}$=8.8 Hz, 1H, CH), 7.85 (d, $^3J_{H,H}$=8.8 Hz, 1H, CH), 9.98 (s, 1H, OH), 11.32 (s, 1H, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): δ=30.34 ($3CH_3$), 41.06 (CH), 53.13 (N–C), 84.50 (C), 104.22, and 113.56 (2CH), 114.80 (C), 118.02 (CH), 125.76 (C), 127.43, 128.58, 129.04, 129.23, 129.80, 130.06, 131.03 (11CH), 132.36 (C), 133.45 (CH), 135.81, 141.40, 148.60, 157.42, and 163.80 (5C), 190.67, and 199.33 (2C=O).

3.1.7. Dimethyl 3-*tert*-butylamino-5-hydroxy-1*H*-benzo[*f*]chromene-1,2-dicarboxylate (6a). White powder, 0.65 g, yield 85%, mp 142–144°C. IR (KBr) (ν_{max}/cm^{-1}): 3230, 1722, 1646. MS, 385 (M^+, 2), 326 (M^+-CO_2Me, 39), 270 [326−$(CH_3)_2CCH_2$, 92], 239 (286−OMe, 100). Anal. calcd for $C_{21}H_{23}NO_6$ (385.4): C, 65.44; H, 6.02; N, 3.63; found: C, 65.2; H, 6.1; N, 3.5%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.60 (s, 9H, *t*-Bu), 3.55 and 3.81 (2s, 6H, 2OMe), 5.44 (s, 1H, CH), 6.46 (s, 1H, OH), 7.11–8.38 (m, 5H, 5CH), 8.86 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=30.38 ($3CH_3$), 38.36 (CH), 51.22, 52.40, and 52.48 ($2OCH_3$ and N–C), 72.64, and 110.38 (2C), 115.24, and 123.26 (2CH), 124.64 (C), 125.58, and 125.82 (2CH), 126.76 (C), 131.24 (CH), 138.91, 143.70, and 161.25 (3C), 169.88, and 173.92 (2C=O).

3.1.8. (1-Benzoyl-3-*tert*-butylamino-5-hydroxy-1*H*-benzo[*f*]chromene-2-yl)-phenyl-methanone (6b). White powder, 0.48 g, yield 50%, mp 187–189°C. IR (KBr) (ν_{max}/cm^{-1}): 3385, 3030, 1663, 1602. MS, *m/z* (%): 477 (M^+, 1), 372 (M^+-COPh, 10), 316 [372−$(CH_3)_2CCH_2$, 32], 105 (COPh, 100). Anal. calcd for $C_{31}H_{27}NO_4$ (477.6): C, 77.97; H, 5.70; N, 2.93; found: C, 77.8; H, 5.6; N, 2.8%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.62 (s, 9H, *t*-Bu), 5.29 (s, 1H, CH), 6.40 (s, 1H, OH), 6.87–7.57 (m, 15H, 15CH), 11.49 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=30.15 ($3CH_3$), 40.92 (CH), 53.36 (N–C), 84.64 (C), 110.96 (CH), 117.99 (C), 121.77, and 124.54 (2CH), 125.23 (C), 125.39, 127.11, 127.61, 128.44, 128.77, 128.82, and 129.75 (11CH), 131.54 (C), 132.90 (CH), 135.65, 139.82, 141.08, 144.21, 163.74 (5C), 190.85, and 199.23 (2C=O).

3.1.9. Dimethyl 2-*tert*-butylamino-7-methyl-9-oxo-4,9-dihydro-pyrano[3,2-*h*]chromene-3,4-dicarboxylate (7a). White powder, 0.73 g, yield 90%, mp 180–183°C. IR (KBr) (ν_{max}/cm^{-1}): 3140, 1726, 1666, 1611. MS, 401 (M^+, 2), 342 (M^+-CO_2Me, 39), 286 [342−$(CH_3)_2CCH_2$, 88], 255 (286−OMe, 100). Anal. calcd for $C_{21}H_{23}NO_7$ (401.4): C, 62.83; H, 5.78; N, 3.49; found: C, 62.7; H, 5.7; N, 3.3%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.69 (s, 9H, *t*-Bu), 2.51 (d, $^4J_{H,H}$=1.8 Hz, 3H, CH_3), 3.65 and 3.77 (2s, 6H, 2OMe), 5.38 (s, 1H, CH), 6.33 (q, $^4J_{H,H}$=1.8 Hz, 1H, CH), 7.10 (d, $^3J_{H,H}$=11.2 Hz, 1H, CH), 7.62 (d, $^3J_{H,H}$=11.2 Hz, 1H, CH), 8.85 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=18.69 (CH_3), 30.50 ($3CH_3$), 35.02 (CH), 50.84, 52.45, and 52.60 ($2OCH_3$ and N–C), 72.07, and 110.55 (2C), 112.14, and 113.44 (2CH), 116.50 (C), 124.77 (CH), 151.10, 152.13, 160.11, and 161.65 (4C), 169.43, 172.78, and 178.28 (3C=O).

3.1.10. 7,8-Dibenzoyl-9-*tert*-butylamino-4-methyl-7*H*-pyrano[3,2-*h*]chromene-2-one (7b). White powder, 0.56 g, yield 57%, mp 162–164°C. IR (KBr) (ν_{max}/cm^{-1}): 3340, 3050, 1726, 1660, 1610. MS, *m/z* (%): 388 (M^+-COPh, 5), 332 [388−$(CH_3)_2CCH_2$, 32], 105 (COPh, 100). Anal. calcd for $C_{31}H_{27}NO_5$ (493.6): C, 75.44; H, 5.51; N, 2.84; found: C, 75.3; H, 5.5; N, 2.8%. ^{1}H NMR (500 MHz, $CDCl_3$, 25°C): δ=1.59 (s, 9H, *t*-Bu), 2.33 (s, 3H, CH_3), 6.12 (s, 1H, CH), 6.18 (s, 1H, CH), 7.13–7.54 (m, 12H, 12CH), 11.65 (s, 1H, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$, 25°C): δ=18.58 (CH_3), 30.31 ($3CH_3$), 38.27 (CH), 53.30 (N–C), 84.16, and 112.07 (2C), 112.39, and 113.04 (2CH), 116.66 (C), 124.33, 126.61, 128.22, 128.56, 129.09, 129.31, and 132.90 (11CH), 135.39 (C), 140.81, 150.23, 152.07, 152.46, and 159.46 (5C), 163.56, 191.34, and 199.50 (3C=O).

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