Supplementary data

Fluoride-induced chemiluminescent decomposition of 1,2-dioxetanes bearing a phenyl moiety substituted with a methyl having an electron-withdrawing group

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Synthesis of dihydrofurans (2)

4-tert-Butyl-5-(3-cyanomethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (**2a**): A solution of 4-*tert*-butyl-5-(3-bromomethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (3.81 g, 11.8 mmol), NaCN (804 mg, 16.4 mmol) and tetra-*n*-butylammonium hydrogen sulfate (804 mg, 2.37 mmol) in THF-H_2O (5:1) (36 mL) was stirred at refluxing temperature for 28 h. After cooling, the reaction mixture was poured into sat. aq. NaCl and then extracted with ethyl acetate (AcOEt). The organic layer was washed twice with sat. aq. NaCl, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was chromatographed on silica gel and eluted with AcOEt-hexane (1:9) to give 2.43 g of 4-*tert*-butyl-5-(3-cyanomethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (**2a**) as an oil in 76.4 % yield.

2a: ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.04 (s, 9H), 1.34 (s, 6H), 3.76 (s, 2H), 3.88 (s, 2H), 7.26-7.38 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 23.5, 27.4, 32.5, 32.6, 47.3, 83.2, 117.6, 126.3, 127.4, 128.6, 129.2, 129.4, 129.6, 137.1, 148.8; IR (liquid film) 2957, 2869, 2251, 1482 cm^{-1}; Mass (m/z, %) 269 (M$^+$, 11), 254 (100), 198 (8), 171 (9), 144 (42).

4-tert-Butyl-5-(3-methoxycarbonylmethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (**2b**): $NaHCO_3$ (512 mg, 6.09 mmol) was added to a solution of 4-*tert*-butyl-5-(3-carboxymethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (1.17 g, 4.06 mmol), which was easily prepared from **2a** by hydrolysis, in DMF (10 mL) at room temperature under N_2 atmosphere. After stirring for 20 min, methyl iodide (0.51 mL, 8.19 mmol) was added to the solution and was stirred overnight. The reaction mixture was poured into sat. aq. NH_4Cl and then extracted with AcOEt. The organic layer was washed twice with sat. aq. NaCl, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was chromatographed on silica gel and eluted with AcOEt-hexane (1:4) to give 1.1 g of 4-*tert*-butyl-5-(3-methoxycarbonylmethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (**2b**) as an oil in 89.7 % yield.

2b: ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.04 (s 9H), 1.33 (s, 6H), 3.62 (s, 2H), 3.67 (s, 3H), 3.87 (s, 2H), 7.18-7.30 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 27.4, 32.4, 32.6, 41.1, 47.2, 51.9, 83.1, 125.8, 128.0, 128.5, 128.8, 130.7, 133.4, 136.3, 149.7, 171.6; IR (liquid film) 2955, 2870, 1742 cm^{-1}; Mass (m/z, %) 302 (M$^+$, 18), 287 (100), 177 (46), 171 (29).

4-tert-Butyl-5-(3-benzoylmethylphenyl)-3,3-dimethyl-2,3-dihydrofuran (**2c**): A solution of 4-*tert*-butyl-5-(3-carboxymethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (304 mg, 1.05 mmol) in THF (2 mL) was added dropwise over 10 min to a solution of *n*-butyllithium (1.62 mol/L in hexane, 1.35 mL, 2.19 mmol) in THF (1.5 mL) at -78℃ under N_2 atmosphere and was stirred for 30 min. Benzoyl chloride (0.13 mL, 1.12 mmol) was added to the solution and was stirred for 1 h. Then H_2O (10 mL) was added to the solution. The reaction mixture was poured into 1N HCl and then extracted with AcOEt. The organic layer was washed twice with sat. aq. NaCl, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was chromatographed on silica gel and eluted with AcOEt-hexane (1:9) to give 96 mg of 4-*tert*-butyl-5-(3-benzoylmethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (**2c**) as an oil in 26.2 % yield.

2c: ^{1}H-NMR (500 MHz, $CDCl_3$) δ_H 0.99 (s, 9H), 1.32 (s, 6H), 3.86 (s, 2H), 4.27 (s, 2H), 7.17 (d, $J = 7.8$ Hz, 1H), 7.21-7.22 (m, 2H), 7.28 (t, $J = 7.3$ Hz, 1H), 7.43 (t, $J = 7.8$ Hz, 2H), 7.53 (t, $J = 6.9$ Hz, 1H), 7.98 (d, $J = 8.2$ Hz, 2H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C 27.4, 32.3, 32.5, 45.5, 47.1, 83.1, 125.8, 128.2, 128.3, 128.6, 128.6, 129.0, 131.0, 133.0, 134.1, 136.5, 136.5, 149.7, 197.4; IR (liquid film) 2957, 2869, 1683 cm^{-1}; Mass (m/z, %) 348 (M^+, 22), 333 (100), 308 (9), 277 (17), 223 (38), 105 (67).

4-tert-Butyl-5-[3-cyano(methoxycarbonyl)methyl]phenyl-3,3-dimethyl-2,3-dihydrofuran (**2d**): A solution of 4-*tert*-butyl-5-(3-cyanomethyl)phenyl-3,3-dimethyl-2,3-dihydrofuran (511 mg, 1.90 mmol) and dimethyl carbonate (0.24 mL, 2.85 mmol) in toluene (4 mL) was added dropwise over 5 min to a suspension of NaH (4.03 mmol) in toluene (4 mL) under N_2 atmosphere. After stirring for 1 h, the reaction mixture was poured into 1N HCl and then extracted with AcOEt. The organic layer was washed twice with sat. aq. NaCl, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was chromatographed on silica gel and eluted with AcOEt-hexane (1:9) to give 582 mg of 4-*tert*-butyl-5-[3-cyano(methoxycarbonyl)-methyl]phenyl-3,3-dimethyl-2,3-dihydrofuran (**2d**) as an oil in 93.7 % yield.

2d: ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.03 (s, 9H), 1.34 (s, 6H), 3.79 (s, 3H), 3.88 (s, 2H), 4.73 (s, 1H), 7.33-7.44 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 27.4, 32.5, 32.6, 43.4, 47.3, 53.9, 83.2, 115.3, 126.7, 127.4, 128.8, 129.2, 129.5, 130.7, 137.3, 148.6, 165.1; IR (liquid film) 2957, 2869, 2252, 1653 cm^{-1}; Mass (m/z, %) 327 (M$^+$, 13), 312 (100), 202 (42).

Synthesis of bicyclic dioxetanes (**1a-1d**); **general procedure**: A dihydrofuran (**2**) (200 mg) and a catalytic amount of tetraphenylporphin (TPP) (1 mg) were irradiated with a 940-W Na lamp in CH_2Cl_2 (10 mL) under an oxygen atmosphere at 0℃ for 1 h. After irradiation, the photolysate was concentrated *in vacuo* and the residue was chromatographed on silica gel. Elution with hexane-CH_2Cl_2 (1:1) gave the corresponding dioxetane (**1**) in the yields as follows: 88.9 % for 5-*tert*-butyl-1-(3-cyanomethyl)phenyl-4,4-dimethyl-2,6,7-trioxabicyclo-[3.2.0]heptane (**1a**), 85.6 % for 5-*tert*-butyl-1-(3-methoxycarbonylmethyl)phenyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]heptane (**1b**), 90.2 % for 5-*tert*-butyl-1-(3-benzoylmethyl)phenyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]heptane (**1c**), and 93.7 % for 5-*tert*-butyl-1-[3-cyano(methoxycarbonyl)methyl]phenyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]-heptane (**1d**).

5-tert-Butyl-1-(3-cyanomethyl)phenyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]heptane (**1a**): pale yellow granules melted at 99.8-100.6℃ (from hexane-CH_2Cl_2). ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 0.98 (s, 9H), 1.16 (s, 3H), 1.39 (s, 3H), 3.79 (s, 2H), 3.83 (d, J = 8.2 Hz, 1H), 4.59 (broad d, J = 8.2 Hz, 1H), 7.39 (d, J = 7.6 Hz, 1H), 7.44 (t, J = 7.6 Hz, 1H), 7.59 (broad s, 1H), 7.62 (d, J = 7.6 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 18.5, 23.7, 25.2, 26.8, 36.7, 45.7, 80.3, 104.9, 116.2, 117.4, 127.8, 128.1, 128.7, 128.9, 129.7, 137.1; IR (KBr) 2966, 2902, 2251 cm^{-1}; Mass (m/z, %) 302 (M$^+$+1, 2), 269 (7), 244 (40), 218 (52), 162 (48), 144 (89), 85 (66), 57 (100); HRMS (TOF)* [M+Na]$^+$: 324.1587 cald for $C_{18}H_{23}NO_3Na$ 324.1576. (* Acc TOF Mass JEOL

JMS-T100LC). Anal. Calcd. for $C_{18}H_{23}NO_3$: C, 71.73; H, 7.69; N, 4.65. Found: C, 71.86; H, 8.08; N, 4.73.

5-tert-Butyl-1-(3-methoxycarbonylmethyl)phenyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]-heptane **(1b)**: pale yellow oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 0.97 (s 9H), 1.16 (s, 3H), 1.38 (s, 3H), 3.66 (s, 2H), 3.67 (s, 3H), 3.82 (d, J = 8.1 Hz, 1H), 4.59 (broad d, J = 8.1 Hz, 1H), 7.30 (d with fine coupling, J = 7.6 Hz, 1H), 7.35 (dd, J = 7.8 and 7.6 Hz, 1H), 7.53-7.55 (m, 2H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C 18.5, 25.1, 26.7, 36.7, 41.1, 45.6, 52.0, 80.2, 104.8, 116.5, 127.1, 128.1, 129.3, 130.3, 133.7, 136.2, 171.6; IR (liquid film): 2962, 2896, 1740 cm^{-1}; Mass (m/z, %) 334 (M$^+$, trace), 302 (trace), 277 (18), 195 (13), 177 (100); HRMS (TOF) [M+Na]$^+$: 357.1686 cald for $C_{19}H_{26}NO_5Na$ 357.1678.

5-tert-Butyl-1-(3-benzoylmethyl)phenyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]heptane **(1c)**: pale yellow granules melted at 110.0-110.9□ (from hexane-CH_2Cl_2). ^{1}H-NMR (500 MHz, $CDCl_3$) δ_H 0.92 (s, 9H), 1.14 (s, 3H), 1.35 (s, 3H), 3.81 (d, J = 8.2 Hz, 1H), 4.32 (s, 2H), 4.57 (d, J = 8.2 Hz, 1H), 7.30 (d, J = 7.6 Hz, 1H), 7.36 (dd, J = 8.2 and 7.6 Hz, 1H), 7.44 (dd, J = 8.3 and 7.3 Hz, 2H), 7.51-7.57 (m, 3H), 7.99 (d with fine coupling, J = 8.3 Hz, 2H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C 18.5, 25.1, 26.7, 36.6, 45.5, 45.6, 80.2, 104.9, 116.6, 126.9, 128.2, 128.6, 128.6, 129.5, 130.5, 133.2, 134.3, 136.3, 136.5, 197.2; IR (KBr) 3001, 2968, 2905, 1689 cm^{-1}; Mass (m/z, %) 348 (M$^+$-32, 1), 323 (20), 241 (21),223 (100), 118 (8), 105 (24); HRMS (TOF) [M+Na]$^+$: 403.1894 cald for $C_{24}H_{28}O_4Na$ 403.1885. (* Acc TOF Mass JEOL JMS-T100LC)

5-tert-Butyl-1-[3-cyano(methoxycarbonyl)methyl]phenyl-4,4-dimethyl-2,6,7-trioxabicyclo-[3.2.0]heptane **(1d)**: pale yellow oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 0.96 (s, 9H), 1.17 (s, 6H), 1.38 (s, 1.5H), 1.39 (s, 1.5H), 3.78 (s, 1.5H), 3.79 (s, 1.5H), 3.85 (d, J = 8.3 Hz, 1H), 4.59 (d, J

= 8.3 Hz, 1H), 4.78 (s, 1H), 7.45-7.55 (m, 2H), 7.66-7.75 (m, 2H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C 18.4, 25.1, 26.7, 36.7, 43.4, 45.6, 54.0, 80.3, 80.3, 105.0, 115.2, 115.2, 116.0, 128.2, 128.2, 128.9, 128.9, 129.0, 129.0, 129.3, 129.3, 129.6, 137.4, 137.4, 165.1, 165.1; IR (liquid film) 2962, 2254, 1753 cm^{-1}; Mass (m/z, %) 360 (M^++1, 1), 327 (4), 302 (26), 287 (29), 276 (63), 220 (63), 202 (100); HRMS (TOF) $[M+Na]^+$: 382.1626 cald for $C_{20}H_{25}NO_5Na$ 382.1630.

5-tert-Butyl-1-(4-cyanomethyl)phenyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]heptane (**8**):

A dioxetane (**8**) was synthesized by the singlet oxygenation of the corresponding dihydrofuran similarly to the case of **1**.

8: pale yellow granules melted at 69.3-70.2□ (from hexane-CH_2Cl_2). ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 0.97 (s, 9H), 1.16 (s, 3H), 1.38 (s, 3H), 3.79 (s, 2H), 3.83 (d, J = 8.1 Hz, 1H), 4.58 (d, J = 8.1 Hz, 1H), 7.37 (d, J = 8.4 Hz, 2H), 7.60 (d, J = 8.4 Hz, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 18.4, 23.4, 25.0, 26.7, 36.6, 45.6, 80.2, 104.9, 116.3, 117.5, 127.5, 129.1, 131.3, 136.0; IR (KBr) 2969, 2894, 2251, 1615 cm^{-1}; Mass (m/z, %) 302 (M^++1, 1), 269 (2), 244 (56), 216 (19), 177 (21), 162 (24), 144 (100); HRMS (TOF)* $[M+Na]^+$: 324.1584 cald for $C_{18}H_{23}NO_3Na$ 324.1576.

Thermolysis of bicyclic dioxetanes (1a-1d); general procedure: A solution of 5-*tert*-butyl-4,4-dimethyl-2,6,7-trioxabicyclo[3.2.0]heptane bearing a substituted phenyl (**1**) (30 mg) in *p*-xylene (5 mL) was stirred under N_2 atmosphere for 2 h at 140 □. After cooling, the reaction mixture was concentrated *in vacuo*. ^{1}H-NMR Spectral analysis showed that the residue included the desired ester (**3**) exclusively. Chromatographic purification [silica gel / AcOEt-hexane (1:4)] of the residue gave the corresponding 2,2,4,4-tetramethyl- 3-oxopentyl benzoate (**3**).

3-Cyanomethylbenzoic acid 2,2,4,4-tetramethyl-3-oxopentyl ester (**3a**): colorless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.29 (s, 9H), 1.39 (s, 6H), 3.79 (s, 2H), 4.41 (s, 2H), 7.46 (dd, *J* = 8.1 and 7.7 Hz, 1H), 7.55 (d, *J* = 7.7 Hz, 1H), 7.93~7.95 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 23.5, 23.7, 28.2, 45.9, 49.2, 72.4, 117.1, 128.9, 129.1, 129.3, 130.3, 131.0, 132.2, 165.3, 215.7; IR (liquid film) 2972, 2250, 1721, 1685 cm^{-1}; Mass (m/z, %) 302 (M^++1, trace), 244 (35), 218 (43), 216 (31), 162 (48), 144 (100).

3-Methoxycarbonylmethylbenzoic acid 2,2,4,4-tetramethyl-3-oxopentyl ester (**3b**): colorless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.28 (s, 9H), 1.38 (s, 6H), 3.66 (s, 2H), 3.70 (s, 3H), 4.41 (s, 2H), 7.39 (dd, *J* = 7.8 and 7.6 Hz, 1H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.86~7.89 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 23.8, 28.2, 40.9, 45.9, 49.2, 52.2, 72.1, 128.2, 128.6, 130.3, 130.3, 133.8, 134.3, 165.9, 171.3, 215.7; IR (liquid film) 2958, 1741, 1722, 1686 cm^{-1}; Mass (m/z, %) 334 (M^+, trace), 277 (18), 195 (16), 177 (100).

3-Benzoylmethylbenzoic acid 2,2,4,4-tetramethyl-3-oxopentyl ester (**3c**): colorless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.27 (s, 9H), 1.37 (s, 6H), 4.33 (s, 2H), 4.40 (s, 2H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.46~7.49 (m, 3H), 7.58 (t with fine coupling, *J* = 7.4 Hz, 1H), 7.86~7.89 (m, 2H), 7.98~8.02 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ_C 23.8, 28.2, 45.1, 45.9, 49.2, 72.1, 128.0, 128.4, 128.6, 128.7, 130.3, 130.6, 133.3, 134.2, 134.8, 136.3, 165.9, 196.7, 215.8; IR (liquid film) 2973, 1721, 1684 cm^{-1}; Mass (m/z, %) 381 (M^++1, trace), 323 (17), 241 (22), 223 (100), 118 (9), 105 (35).

3-Cyano(methoxycarbonyl)methylbenzoic acid 2,2,4,4-tetramethyl-3-oxopentyl ester (**3d**): colorless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.29 (s, 9H), 1.40 (s, 3H), 1.40 (s, 3H), 3.82 (s, 3H), 4.41 (s, 2H), 4.79 (s, 1H), 7.51 (t, *J* = 7.8 Hz, 1H), 7.68 (d with fine coupling, *J* = 7.8 Hz, 1H), 8.01 (d with fine coupling, *J* = 7.8 Hz, 1H), 8.06 (s with fine coupling, 1H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C 23.7, 23.7, 28.2, 43.3, 45.9, 49.2, 54.1, 72.5, 114.9, 129.0, 129.5, 130.3, 131.3, 132.2, 164.8, 165.0, 215.7; IR (liquid film) 2960, 2252, 1753, 1723, 1685 cm^{-1}; Mass (m/z, %) 360 (M^++1, trace), 302 (28), 276 (66), 274 (20), 220 (74), 202 (100).

Chemiluminescence measurement; **general procedure**: Chemiluminescence were measured using a Hitachi FP-750 spectrometer and/or Hamamatsu Photonics PMA-11 multi-channel detector.

Freshly prepared solution (2 mL) of TBAF (1.0 x 10^{-1} mol/L) in DMSO was transferred to a quartz cell (10 x 10 x 50 mm) and the latter placed into the spectrometer, which was thermostated with stirring at 25□. After 3-5 min, a solution of the dioxetane in DMSO (1.0 x 10^{-3} mol/L, 1 mL) was added by means of a syringe with immediate starting of measurement. The intensity of the light emission time-course was recorded and processed according to first-order kinetics. The total light emission was estimated by comparing it with that of an adamantylidene dioxetane, whose chemiluminescent efficiency Φ^{CL} has been reported to be 0.29 and was used here as standard (ref. 10).

Isolation of ketoesters (3) from the spent reaction mixture after chemiluminescent decomposition of dioxetanes (1); **general procedure**: A solution of TBAF (1 M in THF, 0.1 mL) in DMSO (0.9 mL) was added to a solution of the dioxetane (**1**) (20 mg) in DMSO (3 mL) at room temperature under N_2 atmosphere. After stirring, for 1-3 min in the case of **1a-1c**, and for 1 h for **1d**, H_2O (1 mL) was added to the solution, and then the reaction mixture was poured into sat. aq. NH_4Cl and extracted with AcOEt. The organic layer was washed twice with sat. aq. NaCl, dried over $MgSO_4$, and concentrated *in vacuo*. ^{1}H-NMR spectral analysis showed that the residue was comprised of the corresponding ketoester (**3**) and little of other products. The residue was purified by chromatograpy [silica gel / AcOEt-hexane (1:4)] to give the corresponding ketoester (**3**).

***4-[1-cyano-1-(methoxycarbonyl)ethyl]benzoic acid 2,2,4,4-tetramethyl-3-oxopentyl ester* (6d)**: TBAF (1M in THF, 0.5 mL, 0.5 mmol) was added to a solution of 3-[cyano(methoxycarbonyl)methyl]benzoic acid 2,2,4,4-tetramethyl-3-oxopentyl ester (**3d**) (82.9 mg, 0.231 mmol) in DMSO (2 mL) at room temperature under N_2 atmosphere. After 5 min, MeI (0.05 mL, 0.803 mmol) was added to the solution and stirred for 5 min, and then the reaction mixture was poured into sat. aq. NH_4Cl and extracted with AcOEt. The organic layer was washed twice with sat. aq. NaCl, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was chromatographed on silica gel and eluted with AcOEt-hexane (1:4) to give 66.0 mg of 3-(1-cyano-1-methoxycarbonylethyl)benzoic acid 2,2,4,4-tetramethyl-3-oxopentyl ester (**6d**) as on oil in 76.6 % yield.

6d: ^{1}H-NMR (400 MHz, $CDCl_3$) δ_H 1.29 (s, 9H), 1.40 (s, 3H), 1.40 (s, 3H), 1.98 (s, 3H), 3.80 (s, 3H), 4.41 (s, 2H), 7.50 (t, J = 7.8 Hz, 1H), 7.75 (d with fine coupling, J = 7.8 Hz, 1H), 7.99 (d with fine coupling, J = 7.8 Hz, 1H), 8.13 (s with fine coupling, 1H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C 23.6, 23.7, 24.8, 28.1, 45.9, 47.9, 49.1, 54.1, 72.6, 118.9, 126.8, 129.4, 130.0, 130.4, 131.2, 136.1, 165.2, 168.0, 215.8; IR (liquid film) 2962, 2246, 1751, 1724, 1686, cm^{-1}; Mass (m/z, %) 374 (M^++1, trace), 316 (25), 290 (56), 288 (15), 234 (71), 216 (100).