ORGANIC LETTERS

2001
Vol. 3, No. 25
4095−4098

Generation, Observation, and Free Radical Reactivity of Aliphatic Bisketenes: The Solution to a Long-Standing Problem

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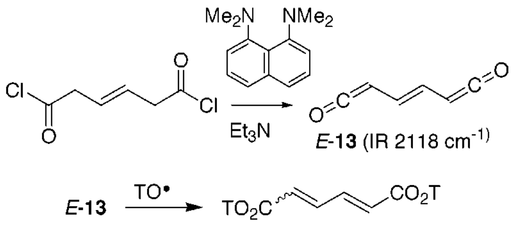
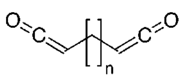
Received October 4, 2001

ABSTRACT

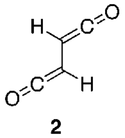


Bisketenes $O{=}C{=}CH(CH_2)_nCH{=}C{=}O$ (1b,c,d, n = 4, 3, 6) and (E)-$O{=}C{=}CHCH{=}CHCH{=}C{=}O$ (E-13) were generated in solution by dehydrochlorination of bis(acyl chlorides) and by photochemical Wolff rearrangements and identified by their characteristic IR signals. The bisketenes react with aminoxyl radicals to give tetraaddition products for 1b and conjugate 1,6-diaddition for E-13.

In 1923 Staudinger and Kreis[1a] reported on their systematic efforts to prepare α,ω-bisketenes **1** beginning with $(CH{=}C{=}O)_2$ (**2**) for $n = 0$, which may be termed a 1,2-bisketene. Although carbon suboxide, C_3O_2 (**3**), had been reported in 1906,[1b,c] their efforts to prepare representatives of type **1** were unsuccessful.



1



2

$O{=}C{=}C{=}C{=}O$

3

There have been many further unsuccessful efforts aimed at the preparation of bisketenes **1**, which are attractive targets because of their multiple functionality and their potential utility for polymer formation. Bisketenes **1** are difficult to obtain because of their propensity for self-reaction and their sensitivity to air and moisture.[2,3] In one successful study, dehydrochlorination of **4** gave **5** with the ketenyl groups held apart, as a unstable crystalline solid which polymerized on warming.[3d] Reaction of **6a** with Et_3N in the presence of chloral gave the cycloadduct **7**,[3e] while **6b** with Et_3N gave the lactone **8** (Scheme 1) which on heating with KOH forms cycloheptanone.[3a,b] Bisketenes were not directly observed in these latter procedures, and their intermediacy is questionable. Bis(acyl chlorides) **6** with Et_3N also gave dimeric bislactones which were converted to macrocyclic diketones.[3c]

Wolff rearrangements of bis(diazo ketones) have also been examined as a route to bisketenes,[4] although the intermediates have usually been trapped in nucleophilic solvents or with

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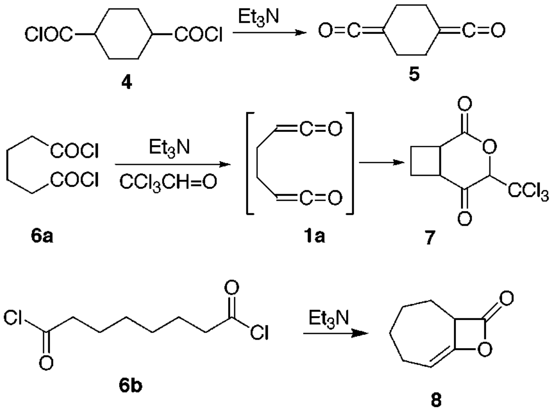
[1] (a) Staudinger, H.; Kreis, W. *Helv. Chim. Acta* **1923**, *6*, 321−326. (b) Diels, O.; Wolf, B. *Chem. Ber.* **1906**, *39*, 689−697. (c) Staudinger, H.; Bereza, S. *Chem. Ber.* **1908**, *41*, 4461−4465.

[2] (a) Tidwell, T. T. *Ketenes*; Wiley: New York, 1995. (b) Allen, A. D.; Ma, J.; McAllister, M. A.; Tidwell, T. T. *Acc. Chem. Res.* **1995**, *28*, 265−271.

[3] (a) Sauer, J. C. *J. Am. Chem. Soc.* **1947**, *69*, 2444−2448. (b) Blomquist, A. T.; Spencer, R. D. *J. Am. Chem. Soc.* **1948**, *70*, 30−33. (c) Gudmundsdottir, A. D.; Lewis, T. J.; Randall, L. H.; Scheffer, J. R.; Rettig, S. J.; Trotter, J.; Wu, C.-H. *J. Am. Chem. Soc.* **1996**, *118*, 6167−6184. (d) Hatchard, W. R.; Schneider, A. K. *J. Am. Chem. Soc.* **1957**, *79*, 6261−6263. (e) Borrman, D.; Wegler, R. *Chem. Ber.* **1966**, *99*, 1245−1251. (f) Baldwin, J. E. *J. Org. Chem.* **1963**, *28*, 3112−3114.

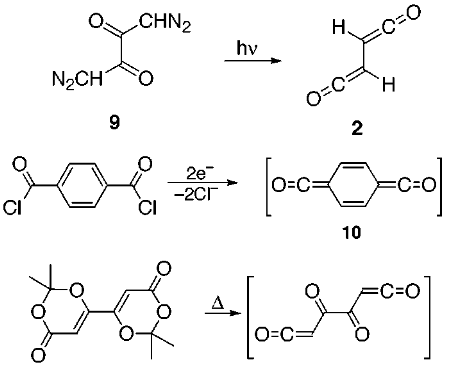
10.1021/ol016853t CCC: $20.00

Scheme 1



other reagents without direct observation of discrete bisketenes. However, photolysis of **9** in an argon matrix at 10 K showed[4b] the formation of **2** (Scheme 2), with an IR band at 2125 cm^{-1}. Other bis(diazo ketones) gave intramolecular reaction of ketenyl groups with the remaining diazo ketone moiety[4c] and polymer formation.[4d]

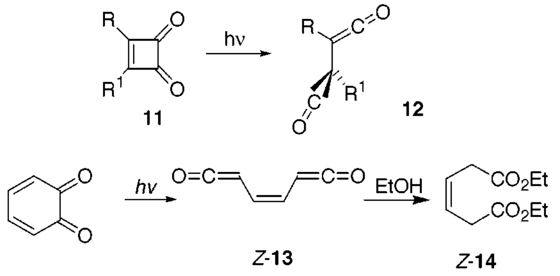
Scheme 2



Reductive dechlorination of terephthalyl chloride led to a polymeric material suggested to arise from bisketene **10** (Scheme 2).[5a] The formation of **10** has been directly observed in an argon matrix at 10 K, as identified by the IR band at 2082 cm^{-1}.[5b] Bis(dioxinone) thermolysis (Scheme 2)[5c,d] has also been examined, but the putative bisketene intermediate was captured with alcohol and not directly observed.

The thermal and photochemical ring opening of cyclobut-1-ene-3,4-diones **11** has been quite useful for generation of a variety of 1,2-bisketenes **12** (Scheme 3),[6,7] but the 1,2-bisketenes formed are generally thermodynamically unstable relative to their precursors and so can usually only be observed by using fast reaction or matrix isolation techniques. In some cases (e.g., R, R^1 = Ph; R = Ph, R^1 = H) the ring closure of the bisketenes is sufficiently slow that the direct observation of these species in solution at room temperature using conventional techniques is feasible.[7a] When R is a ketenyl-stabilizing silyl substituent, the bisketenes are often more stable than the ring-closed forms and can even be isolated in pure form.[2,7b] Theoretical studies[2b] led to the unexpected conclusion that 1,2-bisketenes exist in the twisted conformation **12**, and this was confirmed by X-ray analysis.[7d]

Scheme 3



The photolysis of *o*-quinones has been reported to form (*Z*)-1,6-dioxo-1,3,5-hexadienes,[8] as in the photolysis of the parent *o*-benzoquinone to give *Z*-**13**, as identified by its IR band at 2125 cm^{-1} in Nujol at −170 °C,[8a] or at 2120 cm^{-1} in 2-methyl-THF/THF at 77 K.[8b] Reaction of *Z*-**13** with EtOH gave the diester *Z*-**14** (Scheme 3).[8a]

Despite these efforts over almost 80 years, the examples of nonsilylated bisketenes observed in solution are few, and include **3**, cyclic compounds such as **5**, and 1,2-bisketenes **12**. We have now succeeded in the generation and observation of simple unstabilized bisketenes **1** and *E*-**13** as observable intermediates, using both Wolff rearrangements, as we recently showed for monoketenes,[9a−c] and dehydrochlorination of bis(acyl chlorides), as recently improved,[9d] with 1,8-bis(dimethylamino) naphthalene **15** as base. The

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(4) (a) Horner, L.; Spietschka, E. *Chem. Ber.* **1952**, *85*, 225−229. (b) Maier, G.; Reisenauer, H. P.; Sayrac, T. *Chem. Ber.* **1982**, *115*, 2192−2201. (c) Nakatani, K.; Takada, K.; Odagaki, Y.; Isoe, S. *J. Chem. Soc., Chem. Commun.* **1993**, 556−557. (d) Fahr, E. *Liebigs Ann.* **1960**, *638*, 1−32. (e) Ernest, I.; Hofman, J. *Chem. Listy* **1951**, *45*, 261−264. (f) Doyle, M. P.; Hu, W.; Phillips, I. M.; Wee, A. G. H. *Org. Lett.* **2000**, *2*, 1777−1779. (g) Font, J.; Valls, J.; Serratosa, F. *Tetrahedron* **1974**, *30*, 455−458. (h) Eaton, P. E.; Leipzig, B. D. *J. Am. Chem. Soc.* **1983**, *105*, 1655−1658.

(5) (a) Utley, J. H. P.; Gao, Y.; Lines, R. *J. Chem. Soc., Chem. Commun.* **1993**, 1540−1542. (b) Marquardt, R.; Sander, W.; Laue, T.; Hopf, H. *Liebigs Ann.* **1995**, 1643−1648. (c) Stachel, H. D. *Arch. Pharm.* **1962**, *295*, 735−744. (d) Stachel, H. D. *Angew. Chem.* **1957**, *69*, 507.

(6) (a) Mallory, F. B.; Roberts, J. D. *J. Am. Chem. Soc.* **1961**, *83*, 393−397. (b) Staab, H.; Ipaktschi, J. *Chem. Ber.* **1968**, *101*, 1457−1472. (c) Chapman, O. L.; McIntosh, C. L.; Barber, L. L. *J. Chem. Soc., Chem. Commun.* **1971**, 1162−1163. (d) Obata, N.; Takizawa, T. *J. Chem. Soc., Chem. Commun.* **1971**, 587−588.

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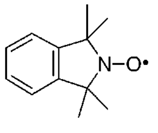
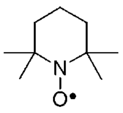
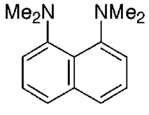
reactions of **1** and *E*-**13** with the aminoxyl free radicals tetramethylisoindolinyloxyl (TMIO, IO$^\bullet$) and tetramethylpiperidinyloxyl (TEMPO, TO$^\bullet$) are also reported. We have previously shown that monoketenes react with aminoxyl radicals by initial attack at the carbonyl carbon, forming enolic radicals which react further to form diadducts,[9a,b,e,f] and have recently extended this methodology to aryl bisketenes.[9g]


TMIO (IO•)

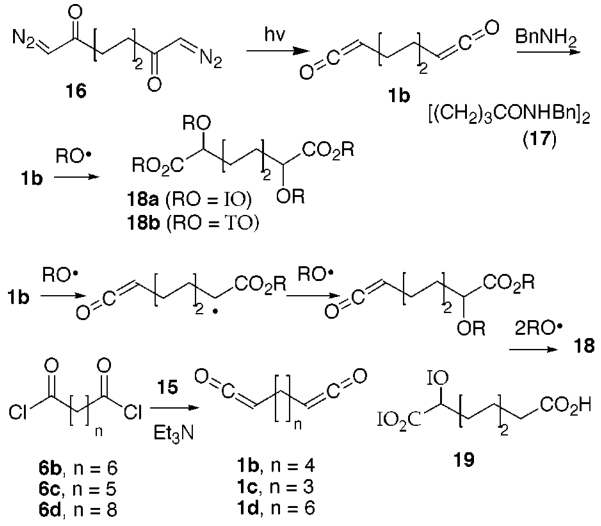

TEMPO (TO•)



15

Photolysis ofthe bis(diazo ketone) **16**[4d,e] with 254 nm light in hexane at room temperature showed progressive diminution of the diazo ketone IR absorption at 2106 cm^{-1} with a concomitant increase in absorption at 2120 cm^{-1} assigned to the bisketene **1b**.[10] Addition of benzylamine (BnNH$_2$) to the bisketene after photolysis gave the bisamide **17** (43%),[11] confirming formation of **1b**. Reaction of **1b** from **16** with TMIO[10] or TEMPO also gave the adducts **18a**,**b** which would form in a stepwise fashion by successive addition of two aminoxyl radicals to one ketenyl group (Scheme 4), followed by addition to the second ketenyl group. The reaction with TMIO is advantageous because of the better chromatographic behavior of the adducts formed,[9c] and the TMIO moiety has no CH$_2$ groups allowing better resolution of the ^{1}H NMR spectrum. Both **17** and **18** are formed as mixtures of diastereomers, and in **18a** all eight methyl signals of the diastreromers were resolved in the ^{1}H NMR spectrum. The isolated yield of **18a** was however only 11% because of the difficulty experienced during chromatography. The IR of **16** at 2100 cm^{-1} in toluene disappeared upon irradiation and that of **1b** appeared at 2112 cm^{-1}. The difference in the position of the IR absorption of **1b** in hexane and in toluene reflects a significant solvent effect.

Scheme 4



Reaction of suberyl chloride (**6b**) with **15** and a catalytic amount of Et$_3$N in toluene by the procedure of Lectka[9d] gave an IR band at 2114 cm^{-1} ascribed to the bisketene **1b**. Reaction of **6b** with **15** in the presence of TMIO or TEMPO and Et$_3$N gave **18a**[12] and **18b**,[13] respectively, as mixtures of *meso* and *dl* isomers (Scheme 4). In the reaction with TMIO the acid **19** was also isolated in 19% yield and evidently arose from adventitious moisture.[12]

Reactions of pimelyl chloride (**6c**) and sebacyl chloride (**6d**) with **15** led to IR bands at 2114 and 2113 cm^{-1}, respectively, due to bisketenes **1c** and **1d**, respectively. Addition of TEMPO to the bisketenes gave the tetraadducts corresponding to **18b**, but these were not completely purified (Supporting Information).

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(9) (a) Allen, A. D.; Cheng, B.; Fenwick, M. H.; Huang, W.; Missiha, S.; Tahmassebi, D.; Tidwell, T. T. *Org. Lett*. **1999**, *1*, 693−696. (b) Allen, A. D.; Cheng, B.; Fenwick, M. H.; Givehchi, B.; Henry-Riyad, H.; Nikolaev, V. A.; Shikhova, E. A.; Tahmassebi, D.; Tidwell, T. T.; Wang, S. *J. Org. Chem*. **2001**, *66*, 2611−2617. (c) Allen, A. D.; Fenwick, M. H.; Henry-Riyad, H.; Tidwell, T. T. *J. Org. Chem*. **2001**, *66*, 5759−5765. (d) Taggi, A. E.; Hafez, A. M.; Wack, H.; Young, B.; Drury, W. J., III; Lectka, T. *J. Am. Chem. Soc*. **2000**, *122*, 7831−7832. (e) Tidwell. T. T.; Fenwick, M. H. *Eur. J. Org. Chem*. **2001**, 3415−3419. (f) Allen, A. D.; Porter, J.; Tahmassebi, D.; Tidwell, T. T. *J. Org. Chem*. **2001**, *66*, 7420−7426. (g) Allen, A. D.; Rangwala, H.; Saidi, K.; Tidwell, T. T.; Wang, J. *Russ. Chem. Bull*. **2001**, in press.

(10) A solution of bis(diazo ketone) **16** (14 mg, 0.070 mmol) in 25 mL of hexane was irradiated with 254 nm light for 2 min, resulting in the complete disappearance of the IR absorption of **16** at 2105 cm^{-1} and formation of a signal at 2120 cm^{-1} due to **1b**. Similar irradiation in toluene showed an IR band of **16** at 2100.5 cm^{-1} and of **1b** at 2112 cm^{-1}. Immediately after irradiation in hexane, TMIO (56 mg, 0.29 mmol) was added and the solution heated at 60 °C overnight. The solvent was evaporated, giving 56 mg of crude product. Chromatography (1/4 EtOAc/hexane) gave **18a** (6 mg, 0.007 mmol, 11%) which was rinsed with hexane to give **18a** (2 mg, 0.003 mmol, 4%), mp 187 °C (dec). ^{1}H NMR (CDCl$_3$) δ 1.40 (s, 6), 1.45 (s, 6), 1.468 (s, 6), 1.473 (s, 6), 1.50 (s, 6) 1.54 (s, 6), 1.55 (s, 6), 1.62 (s, 6), 1.74 (m, 4), 2.0 (m, 4), 4.64 (t, 2, $J = 6.0$ Hz), 7.1 (m, 8), 7.25 (m, 8). ^{13}C NMR (CDCl$_3$) δ 25.6, 25.87, 25.9, 26.0, 29.0, 30.2, 31.6, 33.1, 68.4, 68.6, 82.9, 121.6, 121.7, 121.9, 127.4, 127.6, 127.8, 144.0, 144.8, 145.5, 173.2, 173.3. Line broadening ascribed to restricted rotation was observed in some of the ^{1}H and ^{13}C NMR signals. IR (CDCl$_3$) 1779 cm^{-1}. FABMS *m*/*z* 899 (MH$^+$, 2), 174 (100).

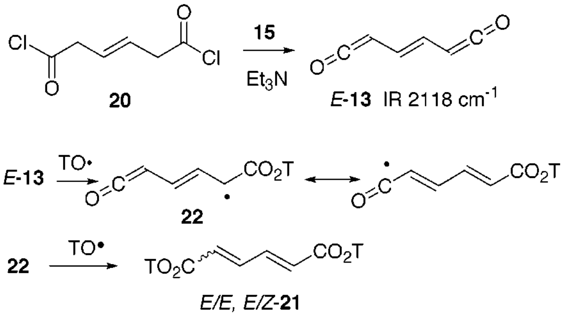
(11) A solution of **16** (15 mg) in 50 mL of hexane was photolyzed with 254 nm light for 2 min, and then benzylamine (20 μL) was added. A white precipitate formed, which was recrystallized from CHCl$_3$ giving **17** (12 mg, 0.034 mmol, 43%) as white crystals, mp 174−174.5 °C. ^{1}H NMR (CDCl$_3$) δ 1.55−1.7 (m, 8), 2.25 (t, 4); 4.40 (s, 2), 4.44 (s, 2), 5.74 (bs, 2), 7.2−7.4 (m, 10). ^{13}C NMR (CDCl$_3$) δ 25.6, 28.9, 36.7, 43.8, 127.7, 128.1, 128.9, 138.6, 173.0. EIMS *m*/*z* 352 (5), 106 (100), 91 (60). HREIMS *m*/*z* calcd for $C_{22}H_{28}N_2O_4$ 352.2151, found 352.2161.

(12) Suberyl chloride (**6b**, 26 mg, 0.14 mmol) in 2 mL of toluene with TMIO (99 mg, 0.52 mmol), **15** (59 mg, 0.27 mmol), and Et$_3$N (4 mg, 0.04 mmol) was stirred overnight and then heated at 60 °C for 7 h. The solution was filtered, extracted with HCl, and dried, and the solvent was evaporated giving 100 mg of crude product. Chromatography (1/4 EtOAc/hexane) gave impure **18a** and then (1/1 EtOAc/hexane) **19** (14 mg, 0.026 mmol, 19%). ^{1}H NMR (CDCl$_3$) δ 1.30−2.05 (m, 32), 2.39 (2, t, $J = 7.4$ Hz), 4.58 (t, 1, $J = 6.1$ Hz), 7.1 (m, 4), 7.2 (m, 4). ^{13}C NMR (CDCl$_3$) δ 24.8, 25.5, 25.7, 26.0, 29.5, 30.3, 31.6, 33.1, 33.9, 68.5, 68.6, 83.0, 121.5, 121.7, 121.8, 127.3, 127.5, 127.7, 144.0, 144.8, 145.4, 173.2, 178.0. IR (CDCl$_3$) 3518, 3200−2800 (br), 1779, 1709 cm^{-1}. ESMS *m*/*z* 559 (MNa$^+$), 537 (MH$^+$).

(13) To suberyl chloride (**6b**, 100 mg, 0.47 mmol) in 15 mL of toluene at 0 °C and TEMPO (326 mg, 2.1 mmol) were added **15** (230 mg, 1.1 mmol) and Et$_3$N (15 μL), and the solution was stirred overnight at room temperature. The solution was filtered, extracted with HCl, dried, and concentrated. The soluble product was dissolved in pentane which was concentrated and chromatographed (25% EtOAc/hexane) giving a mixture of *meso*,*dl*-**18b** as a clear oil (88 mg, 0.12 mmol, 23%). ^{1}H NMR (CDCl$_3$) δ 0.8−2.0 (m, 78), 4.43 (m, 2). ^{13}C NMR (CDCl$_3$) δ 13.1, 16.0, 16.1, 19.5, 19.6, 19.7, 21.7, 23.1, 23.2, 24.2, 28.4, 28.5, 28.7, 30.9, 31.0, 31.2, 31.3, 31.4, 32.6, 33.5, 37.9, 38.2, 38.3, 38.6, 39.4, 52.4, 58.5, 58.7, 59.0, 59.2, 59.6, 82.4, 82.5, 170.6, 170.7.IR (CDCl$_3$) 1769 cm^{-1}. HREIMS *mz* calcd for $C_{44}H_{83}N_4O_6$ (MH$^+$) 763.6313, found 763.6378.

Reaction of (*E*)-3-hexenedioyl chloride (**20**) with **15** rapidly formed (*E*)-1,6-dioxo-1,3,5-hexatriene (*E*-**13**),[14] as identified by the IR band at 2118 cm^{-1} (Scheme 5). Reaction of preformed *E*-**13** with MeOH gave dimethyl (*E*)-hex-3-enedioate (81%),[15] while reaction with TEMPO gave the bis(TEMPO) adducts *E/E*- and *E/Z*-**21** in a 9/1 ratio and 36% yield, from which *E/E*-**21** could be isolated[16] The formation of **21** would occur through the intermediate radical **22**, and such an α,*ω*-diaddition to a bisketene was previously observed to occur in a polar addition with Br_2.[7e] The rapid formation of the ketene IR band and the reactions with MeOH and TEMPO are strong evidence for the intermediacy of *E*-**13**.

Scheme 5



In summary, representative unstabilized bisketenes of different structural types have now been been prepared by convenient procedures as observable intermediates and utilized in synthetic and mechanistic investigations. The way is now open for the preparation of further examples and for the utilization of their multifunctional reactivity.

Acknowledgment. This Letter is dedicated to Professor Manfred Christl on the occasion of his 60th birthday and in recognition of his contributions to ketene chemistry. Financial support by the Natural Sciences and Engineering Research Council of Canada and the donation of the tetramethylisoindolinyloxyl by Prof. Steve Bottle (Queensland Institute of Technology) are gratefully acknowledged.

Supporting Information Available: NMR and IR spectra and experimental data. This material is available free of charge via the Internet at http://pubs.acs.org.

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[14] Addition of (*E*)-3-hexenedioyl chloride (**25**, 42 mg, 0.24 mmol) to **15** (161 mg, 0.75 mmol) in 2 mL of toluene at 0 °C gave an immediate orange color. Triethylamine (14 mg, 0.14 mmol) was added, the solution was stirred for 5 min, 1.5 mL of methanol was added, and the solution was stirred 2 h at room temperature. The solution was filtered, concentrated, acidified with 1 N HCl, and extracted with ether. The ether was dried, concentrated, and subjected to Kugelrohr distillation yielding dimethyl (*E*)-hex-3-enedioate[15] as a colorless oil (35 mg, 0.20 mmol, 81%). ^{1}H NMR ($CDCl_3$) δ 3.09−3.11 (m, 4, CH_2), 3.68 (s, 6, 2CH_3), 5.67−5.74 (m, 2, C*H*).

(15) Hoppen, S.; Bäurle, S.; Koert, U. *Chem. Eur. J.* **2000**, *6*, 2382−2396.

(16) (*E*)-3-Hexenedioyl chloride (**20**, 34 mg, 0.19 mmol) was added to **15** (122 mg, 0.57 mmol) and TEMPO (420 mg, 2.69 mmol) in 2 mL of toluene at 0 °C, followed by Et_3N (2.2 mg, 0.022 mmol). The solution was stirred overnight at room temperature, filtered, and concentrated, and excess TEMPO was sublimed. Chromatography (10% EtOAc/hexane) gave *E*,*E*- and *E*,*Z*-**21** as a 9/1 mixture (28 mg, 0.068 mmol, 36%). Recrystallization from MeOH gave *E*,*E*-**21** as a white solid (mp 218−221 °C). ^{1}H NMR ($CDCl_3$) δ 1.06 (s, 12, 4 CH_3), 1.18 (s, 12, 4 CH_3), 1.4−1.8 (m, 12), 6.27 (bd, 2, $TO_2CCH{=}CH$, $J = 12.0$ Hz), 7.35−7.50 (m, 2, $TO_2CCH{=}CH$). ^{13}C NMR ($CDCl_3$) δ 6.7, 20.4, 31.7, 38.8, 60.1, 126.9, 141.1, 166.0. IR ($CDCl_3$) 1737, 1612 cm^{-1}. EIMS *m/z* 421 (MH^+, 5), 405 ($M^+ - CH_3$, 15), 156 (TO^+, 100), 140 (T^+, 92). HREIMS *m/z* calcd for $C_{24}H_{41}N_2O_4$ 421.3046, found 421.3066 (IR and MS taken as a mixture of *E*,*E*- and *E*,*Z*-**21**). *E*,*Z*-**21**: ^{1}H NMR ($CDCl_3$) δ 1.06 (s, 12, 4 CH_3), 1.18 (s, 12, 4 CH_3), 1.4−1.8 (m, 12H), 6.01 (bd, 1, $TO_2CCH{=}CH\{Z\}$), $J = 10.0$ Hz), 6.17 (bd, 1H, $TO_2CCH{=}CH\{E\}$), $J = 15.0$ Hz), 6.70 (dd, 1, $J = 11.6$, 10.0 Hz, $TO_2CCH{=}CH\{Z\}$), 8.44 (dd, 1, $TO_2CCH{=}CH\{E\}$, $J = 13.9$, 12.4 Hz). ^{13}C NMR ($CDCl_3$) δ 20.5, 31.1, 37.9, 59.3, 124.3, 128.0, 128.7, 138.4, 138.7, 140.7, 165.5, 166.0, 166.7. The ^{1}H NMR data agreed with the corresponding signals reported[17] for (*E*,Z)-$MeO_2C(CH{=}CH)_2CO_2Me$.

(17) Martin, M.-E.; Planchenault, D.; Huet, F. *Tetrahedron* **1995**, *51*, 4985−4990.