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Stereoselective synthesis of substituted arylsulfonylated 1,3-butadienes and 2-propenoates by sulfonylation of acetylenic ester

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Stereoselective synthesis of substituted arylsulfonylated 1,3-butadienes and 2-propenoates by sulfonylation of acetylenic ester

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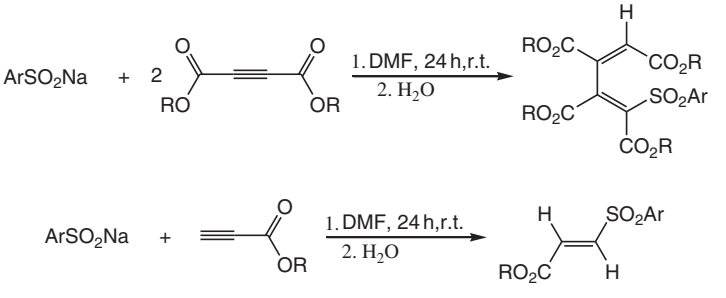
Protonation of the reactive intermediates produced in the reaction between sodium arylsulfinates and two equiv. of dialkyl acetylenedicarboxylates in DMF, by H_2O lead to substituted (1*E*,3*E*)-1-(arylsulfonyl)-1,3-butadiene-1,2,3,4-tetracarboxylates in moderate yields. A regioselective method for the synthesis of alkyl (*E*)-3-(arylsulfonyl)-2-propenoates is described. These reactions provide a useful synthetic route to highly functionalized 1,3-butadienes and 2-propenoates.



Keywords: sodium arylsulfinate; dialkyl acetylenedicarboxylate; alkyl propiolate; 1,3-butadiene; 2-propenoate

1. Introduction

Carbon–carbon double bands are among the most versatile functional groups in organic chemistry. Many reactions have been established for the synthesis of C=C bonds (*1–4*). Among them, preparation of polysubstituted conjugated dienes is an important transformation, because such dienes are useful intermediates and important structural constituents in the synthesis of natural products and optical materials (*5–8*). The introduction of heterosubstituents has a significant influence on the reactivity, regioselectivity, and stereochemistry of the diene and also adds versatility in further reactions of the cycloadducts (*9–14*). Consequently, these dienes are becoming well established as useful intermediates in the organic synthesis. In the past several decades, much effort has been

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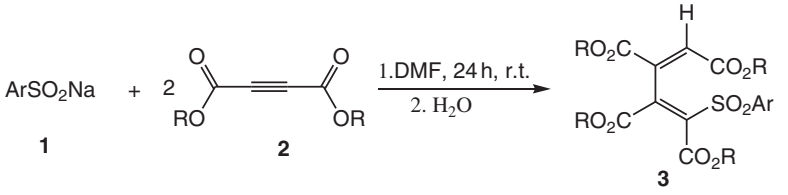
devoted to introduce the sulfone functionality into organic molecules because of the interesting effects of functionality on their structure stability, reactivity, and biological activity of resulting compounds (*15–18*). Therefore, unsaturated sulfones have emerged as effective synthetic targets in the recent years (*19*).

2. Results and discussion

As a part of our ongoing project on the development of new methods for the synthesis of substituted (1*E*,3*E*)-1-(arylsulfonyl)-1,3-butadiene-1,2,3,4-tetracarboxylates via multicomponent reactions, we have focused on the utility of arylsulfinate salts (*20*, *21*). When sodium arylsulfinate was treated with two equiv. of dialkyl acetylenedicarboxylate in DMF at ambient temperature, functionalized 1,3-butadiene was produced in good yields (Scheme 1).



3	Ar	R	Ylied %
a	4-MeC_6H_4	Me	88
b	4-MeC_6H_4	Et	85
c	C_6H_5	Me	90
d	C_6H_5	Et	81

Scheme 1. Synthesis of substituted 1,3-butadienes from sodium arylsulfinates and dialkyl acetylenedicarboxylates.

The ^{1}H and ^{13}C NMR spectra of ylides **3a–3d** are consistent with the presence of one isomer. The mass spectra of the products displayed molecular ion peaks at appropriate*m*/*z*values. The ^{1}H NMR spectra of compound **3a** exhibited five single sharp lines readily recognized as arising from methyl (δ 2.40 ppm) and methoxy (δ 3.45, 3.72, 3.84, and 3.93 ppm) groups, supporting the IR absorption at 1733 cm^{-1}. A singlet (δ 6.90 ppm) is observed for the olefinic H-atom, and the 4-methylphenyl moiety gave rise to characteristic signals in the aromatic region of the spectrum. The ^{1}H-decoupled ^{13}C NMR spectrum of **3a** showed four carbomethoxy groups at δ 162.0, 162.2, 163.4, and 163.7 ppm. The structure of **3a** was unambiguously confirmed by an X-ray crystallographic analysis, which disclose the 1*E*,3*E*-form of the generated C=C double bonds as two conformers (Figure 1). The *S*-cis conformation incorporates a van der Waals repulsion between the (4-methylphenyl)sulfonyl on C-1 and methylcarboxylate on C-4.

The nucleophilic reactions of sodium arylsulfinates **1** with alkyl propiolates **4** at room temperature gave substituted alkyl (*E*)-3-(arylsulfonyl)-2-propiolates **5a–5d**, stereoselectively, in moderate yields. The results are listed in Scheme 2 and no *Z*-isomers of product **5** were detected. The ^{1}H NMR (400 MHz) spectrum of **5a** displayed a triplet at δ 1.31 and quartet 4.25 characteristic of ethoxy groups, while the olefinic protons were observed as two separate doublets at δ 6.85 and δ 7.34 (^3J = 15.2 Hz). The values of the coupling constants for the ethylenic protons indicated that the 2-propenoates **5** existed exclusively in the *E*-configuration.

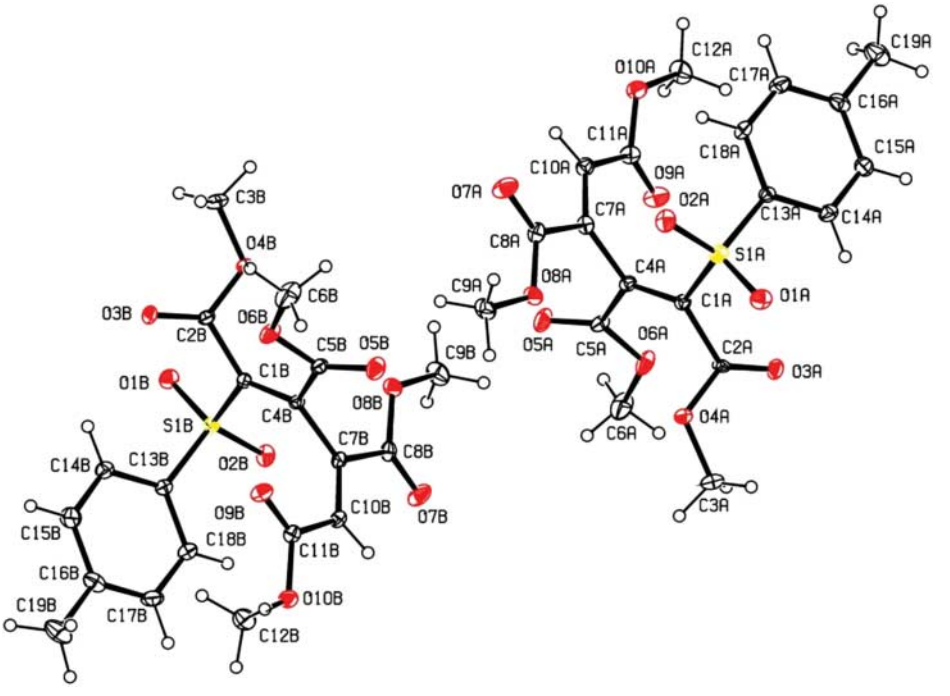
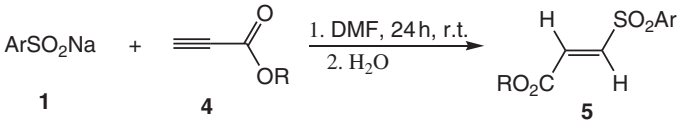


Figure 1. X-ray crystal structure of compound **3a**.

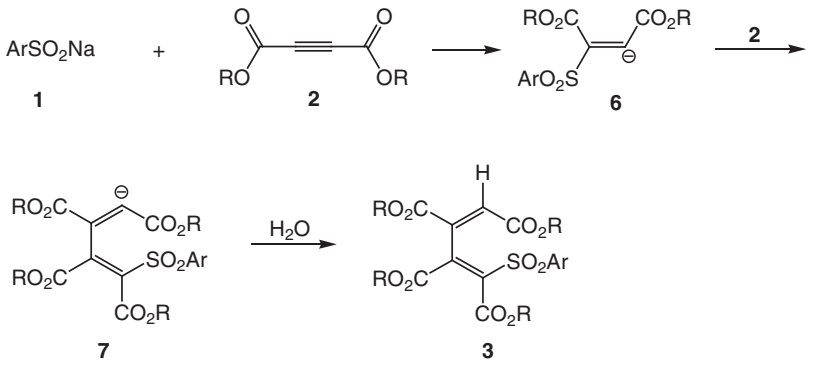


5	Ar	R	Ylied %
a	Ph	Et	72
b	Ph	Me	68
c	4-MeC_6H_4	Me	70
d	4-MeC_6H_4	Et	65

Scheme 2. Synthesis of substituted 2-propenoates from sodium arylsulfinates and alkyl propiolates.

Mechanistically, it is conceivable that the reaction between **1** and **2** involves the initial formation of intermediate **6**, which undergoes further reaction with a second molecule of **2** to produce **7**. Protonation of this carbanion by H_2O leads to product **3** (Scheme 3).

In summary, we have demonstrated a highly efficient and stereselective synthesis for a series of substituted 1,3-butadienes and 2-propenoates, which are potential candidates for cycloaddition reactions. Further investigations aimed at defining the scope and limitations of the reaction are in progress.



Scheme 3. Proposed mechanism pathway.

3. Experimental

Compounds **1** and dialkyl actylenedicarboxylate were obtained commercially and used without further purification. M.p.: Electrothermal-9100 apparatus. IR spectra: Shimadzu-IR-460 spectrometer; in cm^{-1}. ^1H- and ^{13}C-NMR spectra: Bruker DRX-400 Avance instrument at 400.1 and 100.6 MHz, respectively; δ in ppm and J in Hz. MS: Finnigan-MAT-8430EI-MS mass spectrometer; at 70 eV; in m/z (rel%). Elemental analyses: Vario EL III CHNOS elemental analyzer.

3.1. *General procedure for the preparation of substituted (1E,3E)-1-(arylsulfonyl)-1, 3-butadiene-1,2,3,4-tetracarboxylates 3*

A solution of sodium arylsulfinate (1 mmol) in DMF (5 mL) was cooled to −5°C. Then, dialkyl acetylenedicarboxylate (2 mmol) in DMF (2 mL) was added dropwise, and the mixture was stirred for 24 h at room temperature. The mixture was poured onto H_2O (15 mL), extracted with AcOEt (30 mL), dried ($MgSO_4$), and the solvent was removed under reduced pressure. The residue was purified by a silica gel column chromatography using hexane/AcOEt (5:1) as an eluent to give 1,3-butadienes **3a–3d**.

3.2. *General procedure for the preparation of substituted alkyl (E)-3-(arylsulfonyl)-2-propenoate 5*

A solution of sodium arylsulfinate (1 mmol) in DMF (3 mL) was cooled to −5°C. Then, dialkyl acetylenedicarboxylate (1 mmol) in DMF (2 mL) was added dropwise, and the mixture was stirred for 24 h at room temperature. The mixture was poured onto H_2O (15 mL), extracted with AcOEt (30 mL), dried ($MgSO_4$), and the solvent was removed under reduced pressure. The residue was purified by a silica gel column chromatography using hexane/AcOEt (7:1) as an eluent to give 1,3-butadienes **5a–5d**.

3.2.1. *Tetramethyl (1E,3E)-1-[(4-methylphenyl)sulfonyl]-1,3-butadiene-1,2,3, 4-tetracarboxylate **(3a)***

Colorless solid; m.p. 150°C; yield: 0.38 g (88%). IR (KBr) (ν_{max}, cm^{-1}): 2985, 1733, 1446, 1341, 1251, 1138, 1019, 723. Anal. Calcd for $C_{19}H_{20}O_{10}S$ (440.41): C, 51.82; H, 4.58; S, 7.28. Found:

C, 51.02; H, 4.44; N, 7.59%. MS: m/z (%) = 440 (3, M^+), 409 (10), 381 (25), 285 (100), 257 (53), 139 (82), 91 (55), 77 (32). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 2.40 (3H, s, Me); 3.45 (3H, s, MeO); 3.72 (3H, s, MeO); 3.84 (3H, s, MeO); 3.93 (3H, s, MeO); 6.90 (1H, s, CH); 7.30 (2H, d, ^3J = 8.1 Hz, 2 CH); 7.41 (2H, d, ^3J = 8.1 Hz, 2 CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 21.5 (Me); 52.4 (MeO); 53.5 (MeO); 53.8 (MeO); 53.9 (MeO); 128.8 (2 CH); 129.4 (2 CH); 130.4 (CH); 136.2 (C); 136.8 (C); 138.1 (C); 143.9 (C); 145.7 (C); 162.0 (C=O); 162.2 (C=O); 163.4 (C=O); 163.7 (C=O) ppm.

3.2.2. *Tetraethyl (1E,3E)-1-[(4-methylphenyl)sulfonyl]-1,3-butadiene-1,2,3,4-tetracarboxylate* ***(3b)***

Yellow oil; yield: 0.42 g (85%). IR (KBr) (ν_{max}, cm^{-1}): 2984, 1730, 1603, 1457, 1372, 1264, 1153, 1029, 809, 709. Anal. Calcd for $C_{23}H_{28}O_{10}S$ (496.52): C, 55.64; H, 5.68; S, 6.46%. Found: C, 56.10; H, 5.87; S, 6.38%. MS: m/z (%) = 496 (1, M^+), 341 (100), 313 (95), 267 (65), 239 (45), 155 (63), 139 (74), 91 (95), 77 (25). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 1.07 (3H, t, ^3J = 7.2 Hz, Me); 1.18 (3H, t, ^3J = 7.2 Hz, Me); 1.28–1.35 (6H, m, 2 Me); 2.42 (3H, s, Me); 3.84 (2H, q, ^3J = 7.2 Hz, CH_2O); 4.18 (2H, q, ^3J = 7.2 Hz, CH_2O); 4.28–4.46 (4H, m, 2 CH_2O); 6.90 (1H, s, CH); 7.28 (2H, d, ^3J = 8.2 Hz, 2 CH); 7.40 (2H, d, ^3J = 8.2 Hz, 2 CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 13.6 (Me); 13.7 (Me); 13.9 (Me); 14.0 (Me); 21.6 (Me); 61.4 (CH_2O); 62.3 (CH_2O); 62.6 (CH_2O); 62.7 (CH_2O); 128.9 (2 CH); 129.7 (2 CH); 130.5 (CH); 136.2 (C); 136.9 (C); 138.1 (C); 143.8 (C); 145.6 (C); 162.0 (C=O); 162.2 (C=O); 163.5 (C=O); 163.6 (C=O) ppm.

3.2.3. *Tetramethyl (1E,3E)-1-(phenylsulfonyl)-1,3-butadiene-1,2,3,4-tetracarboxylate* ***(3c)***

Colorless solid; m.p. 103–105°C; yield: 0.38 g (90%). IR (KBr) (ν_{max}, cm^{-1}): 2980, 1737, 1443, 1328, 1257, 1157, 1021, 727. Anal. Calcd for $C_{18}H_{18}O_{10}S$ (426.39): C, 50.70; H, 4.25; S, 7.52. Found: C, 51.14; H, 4.54; S, 7.64%. MS: m/z(%) = 426 (2, M^+), 395 (7), 367 (35), 285 (100), 257 (45), 125 (70), 109 (20), 77 (85), 59 (36). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 3.46 (3H, s, MeO); 3.73 (3H, s, MeO); 3.85 (3H, s, MeO); 3.93 (3H, s, MeO); 6.92 (1H, s, CH); 7.52 (2H, t, ^3J = 7.6 Hz, 2 CH); 7.64 (1H, t, ^3J = 7.2 Hz, CH); 7.89 (2H, d, ^3J = 8.0 Hz, 2 CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 52.2 (MeO); 53.3 (MeO); 53.5 (MeO); 53.6 (MeO); 128.9 (2 CH); 129.0 (2 CH); 130.3 (CH); 134.3 (CH); 137.2 (C); 138.0 (C); 139.1 (C); 143.9 (C); 162.4 (C=O); 162.5 (C=O); 163.8 (C=O); 164.0 (C=O) ppm.

3.2.4. *Tetraethyl (1E,3E)-1-(phenylsulfonyl)-1,3-butadiene-1,2,3,4-tetracarboxylate* ***(3d)***

Yellow oil; yield: 0.24 g (81%). IR (KBr) (ν_{max}, cm^{-1}): 2980, 1725, 1609, 1438, 1368, 1269, 1149, 1033, 825. Anal. Calcd for $C_{22}H_{26}O_{10}S$ (482.49): C, 54.77; H, 5.43; S, 6.64%. Found: C, 55.12; H, 5.14; S, 6.39%. MS: m/z(%) = 482 (5, M^+), 341 (100), 313 (85), 253 (70), 239 (36), 141 (58), 125 (18), 77 (25). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 1.09 (3H, t, ^3J = 7.1 Hz, Me); 1.20 (3H, t, ^3J = 7.1 Hz, Me); 1.31–1.39 (6H, m, 2 Me); 3.84 (2H, q, ^3J = 7.1 Hz, CH_2O); 4.18 (2H, q, ^3J = 7.1 Hz, CH_2O); 4.30 (2H, q, ^3J = 7.1 Hz, CH_2O); 4.37 (2H, q, ^3J = 7.1 Hz, CH_2O); 6.92 (1H, s, CH); 7.49 (2H, t, ^3J = 7.4 Hz, 2 CH); 7.65 (1H, t, ^3J = 7.3 Hz, CH); 8.05 (1H, d, ^3J = 7.8 Hz, CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 13.6 (Me), 13.7 (Me); 13.9 (Me); 14.0 (Me); 61.5 (CH_2O); 62.2 (CH_2O); 62.5 (CH_2O); 62.7 (CH_2O); 128.7 (2 CH); 129.0 (2 CH); 130.3 (CH); 134.3 (CH); 137.5 (C); 138.0 (C); 139.2 (C); 143.5 (C); 161.9 (C=O); 162.1 (C=O); 163.5 (C=O); 163.6 (C=O) ppm.

3.2.5. *Ethyl (E)-3-(phenylsulfonyl)-2-propenoate* ***(5a)***

Colorless oil; yield: 0.17 g (72%). IR (KBr) (ν_{max}, cm^{-1}): 2971, 1706, 1425, 1317, 1221, 1142, 908, 627. Anal. Calcd for $C_{11}H_{12}O_4S$ (240.27): C, 54.99; H, 5.03; S, 13.34. Found: C, 54.68; H, 5.14; S, 13.53%. MS: m/z(%) = 240 (3, M^+), 195 (40), 141 (76), 77 (100). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 1.31 (3H, t,^3J = 7.6 Hz, Me); 4.25 (2H, q, ^3J = 7.6 Hz, CH_2O); 6.85 (1H, d, ^3J = 15.2 Hz, CH); 7.34 (1H, d, ^3J = 15.2 Hz, CH); 7.60 (2H, t, ^3J = 7.2 Hz, 2 CH); 7.70 (1H, t, ^3J = 7.2 Hz, CH); 7.93 (2H, d, ^3J = 7.6 Hz, 2 CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 14.0 (Me); 62.0 (CH_2O); 128.3 (2 CH); 129.6 (2 CH); 131.0 (CH); 134.4 (CH); 138.4 (C); 143.1 (CH); 163.4 (C=O) ppm.

3.2.6. *Methyl (E)-3-(phenylsulfonyl)-2-propenoate* ***(5b)***

Colorless oil; yield: 0.15 g (68%). IR (KBr) (ν_{max}, cm^{-1}): 2915, 1712, 1403, 1321, 1239, 1162, 825. Anal. Calcd for $C_{10}H_{10}O_4S$ (226.24): C, 53.09; H, 4.45; S, 14.17. Found: C, 53.28; H, 4.58; S, 14.33%. MS: m/z (%) = 226 (5, M^+), 195 (35), 141 (80), 77 (100). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 3.85 (3H, s, MeO); 6.84 (1H, d, ^3J = 15.1 Hz, CH); 7.37 (1H, d, ^3J = 15.1 Hz, CH); 7.60 (2H, t, ^3J = 7.2 Hz, 2 CH); 7.68 (1H, t, ^3J = 7.2 Hz, CH); 7.92 (2H, d, ^3J = 7.5 Hz, 2 CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 52.7 (MeO); 128.3 (2 CH); 129.5 (2 CH); 131.0 (CH); 134.3 (CH); 138.5 (C); 143.1 (CH); 163.2 (C=O) ppm.

3.2.7. *Methyl (E)-3-[(4-methylphenyl)sulfonyl]-2-propenoate* ***(5c)***

Colorless oil; yield: 0.16 g (70%). IR (KBr) (ν_{max}, cm^{-1}): 2912, 1710, 1429, 1309, 1218, 1136, 795. Anal. Calcd for $C_{11}H_{12}O_4S$ (240.27): C, 54.99; H, 5.03; S, 13.34. Found: C, 54.67; H, 5.14; S, 13.52%. MS: m/z (%) = 240 (4, M^+), 209 (73), 155 (85), 141 (82), 91 (90), 77 (100). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 2.48 (3H, s, Me); 3.81 (3H, s, MeO); 6.82 (1H, d, ^3J = 15.1 Hz, CH); 7.30 (1H, d, ^3J = 15.1 Hz, CH); 7.40 (2H, d, ^3J = 8.0 Hz, 2 CH); 7.82 (2H, d, ^3J = 8.0 Hz, 2 CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 21.7 (Me); 52.8 (MeO); 128.4 (2 CH); 129.9 (CH); 130.3 (2 CH); 135.3 (C); 143.7 (CH); 145.7 (C); 164.0 (C=O) ppm.

3.2.8. *Ethyl (E)-3-[(4-methylphenyl)sulfonyl]-2-propenoate* ***(5d)***

Colorless oil; yield: 0.16 g (65%). IR (KBr) (ν_{max}, cm^{-1}): 2925, 1711, 1462, 1321, 1218, 1172, 826. Anal. Calcd for $C_{12}H_{14}O_4S$ (254.29): C, 56.68; H, 5.55; S, 12.61. Found: C, 56.39; H, 5.44; S, 12.53%. MS: m/z (%) = 254 (5, M^+), 240 (30), 209 (66), 155 (70), 141 (85), 91 (100), 77 (80). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 1.29 (3H, t, ^3J = 7.6 Hz, Me); 2.46 (3H, s, Me); 4.25 (2H, q, ^3J = 7.6 Hz, CH_2O); 6.80 (1H, d, ^3J = 15.1 Hz, CH); 7.32 (1H, d, ^3J = 15.1 Hz, CH); 7.36 (2H, d, ^3J = 8.0 Hz, 2 CH); 7.82 (2H, d, ^3J = 8.0 Hz, 2 CH) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 14.2 (Me); 21.6 (Me); 62.3 (CH_2O); 128.4 (2 CH); 129.8 (CH); 130.2 (2 CH); 135.4 (C); 143.7 (CH); 145.9 (C); 164.1 (C=O) ppm.

CCDC 892482 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/data_request/cif.

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