Pergamon

Tetrahedron Letters 41 (2000) 567–568

TETRAHEDRON LETTERS

One-pot, four-component synthesis of dialkyl [indane-1,3-dione-2-ylidene]alkoxysuccinates

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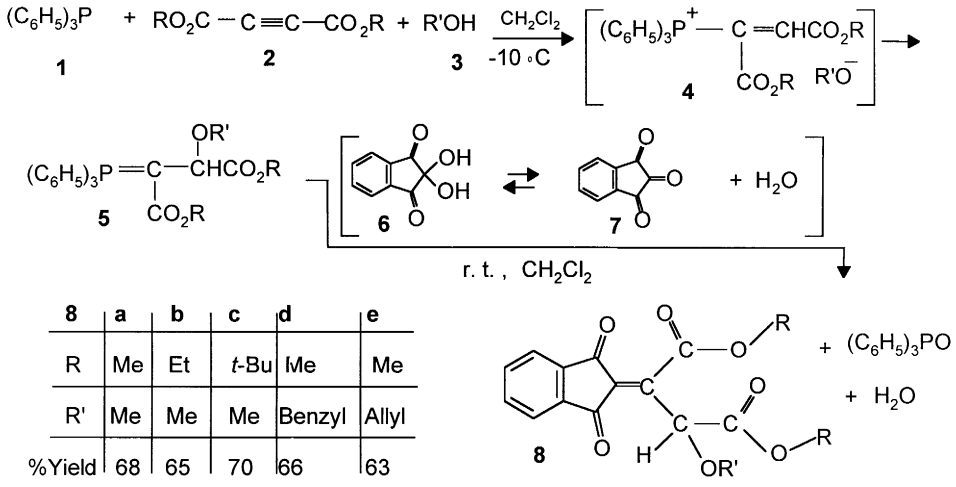
Received 21 July 1999; accepted 2 November 1999

Abstract

A one-pot synthesis of dialkyl [indane-1,3-dione-2-ylidene]alkoxysuccinates in fairly good yields by the reaction of alcohols, dialkyl acetylenedicarboxylates, triphenylphosphine and ninhydrin is reported.

Keywords: intermolecular Wittig reaction; triphenylphosphine; acetylenic esters; dialkyl alkoxysuccinates.

Quite recently, we have established a method for heterocyclic synthesis using a novel approach employing vinylphosphonium salts.[1–3] In this note, we wish to describe the preparation of dialkyl [indane-1,3-dione-2-ylidene]alkoxysuccinates **8** in fairly good yields (Scheme 1).



8	a	b	c	d	e
R	Me	Et	*t*-Bu	Me	Me
R'	Me	Me	Me	Benzyl	Allyl
%Yield	68	65	70	66	63

Scheme 1.

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0040-4039/00/$ - see front matter
PII: S0040-4039(99)02066-3

Reactions are known in which an α,β-unsaturated carbonyl compound is produced from a phosphorane and a carbonyl compound such as an aldehyde or ketone.[4–8] Thus, compounds **8** may be regarded as the products of intermolecular Wittig reactions. Such addition–olefination products may result from an initial addition of triphenylphosphine **1** to the acetylenic ester **2** and concomitant protonation of the 1:1 adduct,[9] followed by attack of the alkoxide on the vinylphosphonium cation to form phosphorane **5**. Attack of the stabilized ylide **5** on the highly electron deficient carbonyl group of indane-1,2,3-trione **7** [10] in a normal intermolecular Wittig reaction would lead to the dialkyl [indane-1,3-dione-2-ylidene]alkoxysuccinates **8**.

In summary, we have developed a convenient, one-pot method for preparing dialkyl [indane-1,3-dione-2-ylidene]alkoxysuccinates **8** utilising in situ generation of the phosphorane **5**. Other aspects of this process are under investigation.

Experimental. Typical process for the preparation of **8**: To a magnetically stirred solution of triphenylphosphine (0.262 g, 1 mmol) and **3** (1 mmol) in CH_2Cl_2 (5 ml) was added dropwise a mixture of **2** (1 mmol) in CH_2Cl_2 (5 ml) at −10°C over 15 min. The mixture was allowed to warm up to room temperature and powdered ninhydrin **6** (0.18 g, 1 mmol) was added and stirred for 24 h. The solvent was removed under reduced pressure and the viscous residue was purified by flash column chromatography (silica gel; hexane–ethyl acetate). The solvent was removed under reduced pressure and the products were obtained as white crystals (**8a**, m.p. 115.5–117.5°C; **8c**, m.p. 130.9–132.9°C) and viscous colourless oils (**8b**, **8d**–**e**). Analytical and spectroscopic data (^{1}H (500 MHz) and ^{13}C (125 MHz) NMR, MS, IR, UV) are in good agreement with the proposed structures. Spectral data for **8c**: UV (ethanol, 95%), λ_{max}/nm (log ε): 204.8 (3.69), 247.2 (3.79), 280.0 (3.99). IR (KBr) (ν_{max}, cm^{-1}): 2978, 2930, 1738, 1700. ^{1}H NMR ($CDCl_3$): δ 1.43 and 1.55 (18H, 2s, $2OC(CH_3)_3$), 3.49 (3H, s, OCH_3), 5.77 (1H, s, OCH), 7.59 (1H, t, *J*=7.6 Hz, CH(arom.)), 7.75 (1H, t, *J*=7.6 Hz, CH(arom.)), 7.83 (1H, d, J=7.6 Hz, CH(arom.)), 8.29 (1H, d, J=7.6 Hz, CH(arom.)). ^{13}C NMR ($CDCl_3$): δ 28.18, 28.50, 52.52, 82.87, 83.39, 91.95, 114.59, 125.20, 125.51, 128.71, 132.20, 136.05, 139.04, 140.32, 147.36, 161.48, 167.26, 190.71. MS, *m/z*: 402(M^+), 371, 347, 315, 291, 271, 258, 245, 228, 57 (100%), 41. Anal. calcd for $C_{22}H_{26}O_7$(402.45): C, 65.66; H, 6.51%. Found: C, 65.23; H, 6.55%.

Acknowledgements

This work was supported by the Zanjan University Research Council.

References

1. Yavari, I.; Ramazani, A. *Synth. Commun*. **1997**, 1385–1390.
2. Yavari, I.; Esmaili, A. A.; Ramazani, A.; Bolbol-Amiri, A. R. *Chemical Monthly* **1997**, *128*, 927–931.
3. Yavari, I.; Ramazani, A.; Esmaili, A. A. *J.Chem. Res.(S)* **1997**, 208–209.
4. McCombie, S. W.; Luchaco, C. A. *Tetrahedron Lett*. **1997**, *38*, 5775–5776.
5. Barco, A.; Benetti, S.; Risi, C. D.; Marchetti, P.; Pollini, G. P.; Zanirato, V. *Tetrahedron Lett*. **1998**, *39*, 7591–7594.
6. Bhatia, G. S.; Lowe, R. F.; Stoodley, R. J. *Tetrahedron Lett*. **1998**, *39*, 9245–9248.
7. Wei, X.; Taylor, R. J. K. *Tetrahedron Lett*. **1998**, *39*, 3815–3818.
8. Barco, A.; Benetti, S.; Risi, C. D.; Marchetti, P.; Pollini, G. P.; Zanirato, V. *Tetrahedron Lett*. **1998**, *39*, 1973–1976.
9. We have also used EtOH, *i*-PrOH and *n*-BuOH in this reaction, but yields of corresponding products **8** were low. The weak acidity of these alcohols may be the factor in the reduction of yields.
10. We have also used less reactive aldehydes and ketones in this reaction, but no products were observed even at reflux temperature (toluene as solvent) after 24 h. TLC indicated that the solution contained ylide **5** and the starting aldehyde or ketone.