On the Preparation and Rearrangement of Some Vinylic Sulphoxides

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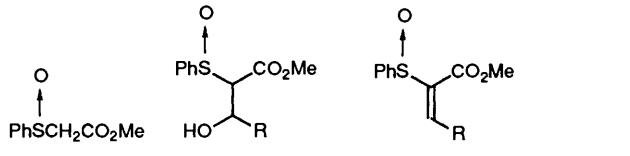
The condensation of methyl benzenesulphinylacetate **1** with a series of aldehydes has been explored using different catalysts. With zinc chloride, the enolate of **1** produces the conjugated ester directly. A base-catalysed rearrangement of these conjugated esters to the corresponding γ-hydroxy unsaturated ester can be effected. Alternatively, the use of magnesium methoxide as catalyst during the condensation of the aldehydes with the reagent **1** produces these hydroxy esters directly.

Use of standard methods for reacting the enolate anion of methyl benzenesulphinylacetate (MBSA) and similar derivatives with aldehydes proceeds to yield the aldol-type of product **2** in excellent yields.[1] Methods for dehydrating this system to give the corresponding conjugated ester failed.[2] A problem with the dehydration step is the sensitivity of the sulphoxide group to degradation by acidic reagents, for example, onset of Pummerer type reactions. However, under controlled conditions we have established that dehydration of the intermediate alcohols **2** can be effected in high yields.[3] This paper describes further applications of this method, the use of other catalysts and the rearrangement behaviour of the product esters.

It was argued that use of a mild Lewis acid catalyst during the reaction of the aldehyde with the anion of MBSA would aid the dehydration. Of several common Lewis acids tried, it was found that anhydrous zinc chloride was the most efficient. Thus treatment of the preformed sodium enolate of MBSA with anhydrous zinc chloride in tetrahydrofuran, followed by the addition of the aldehyde afforded, as the major product, the corresponding conjugated ester. Examples of reactions studied are detailed in Table 1. The importance of these α-benzenesulphinyl-conjugated esters has been acknowledged;[4] although the method only produces these derivatives in modest yields they have previously only been available by a multi-step process.[5]

In all cases only one isomer appeared to be formed and was isolated. The geometry about the formed double bond was explored using the Eu(fod)$_3$ shift reagent. Addition of this to the benzaldehyde product **3** resulted in a preferential shift of the vinylic proton; the downfield shift of the *ortho*-protons was less marked. Since it is known that the sulphoxide oxygen co-ordinates to the europium ion more strongly than the ester carbonyl group,[6] the large shift of the vinylic hydrogen atom indicates that this is *syn* to the sulphoxide group and hence implies the *E* geometry of the double bond. A similar pattern of shifts was observed with the crotonaldehyde condensate **6**. The



1 **2**

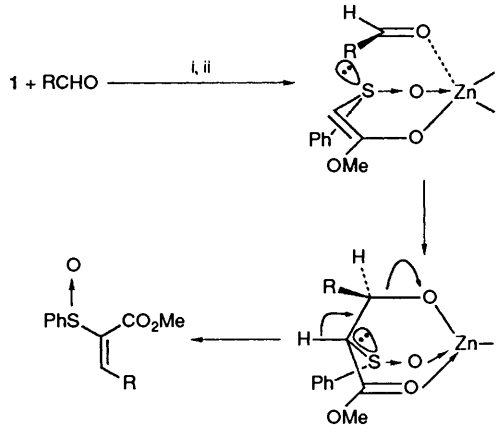
3 R = Ph **7** R = Me
4 R = *n*-C_5H_{11} **8** R = $CHMe_2$
5 R = *n*-C_4H_9 **9** R = CH_2CH_2Ph
6 R = *t*-CH=CHMe

role of the zinc ion in the dehydration step can possibly be explained by assuming initial co-ordination of both the sulphoxide group and the ester enolate ion to the metal ion and that the aldehydic oxygen approaches the zinc ion during reaction to form a tight complex (Scheme 1) in which the alkyl

Table 1 Condensation of MBSA with aldehydes (RCHO) to give unsaturated esters[a]

R	Yield (%)	Product
Ph	20	**3**
n-C_5H_{11}	38	**4**
n-C_4H_9	35	**5**
MeCH=CH	39	**6**
Me	34	**7**
Pr^i	12	**8**
$PhCH_2CH_2$	48	**9**

[a] See Experimental for reaction conditions. Yields are isolated, pure yields.



Scheme 1 *Reagents:* i, NaH; ii, $ZnCl_2$

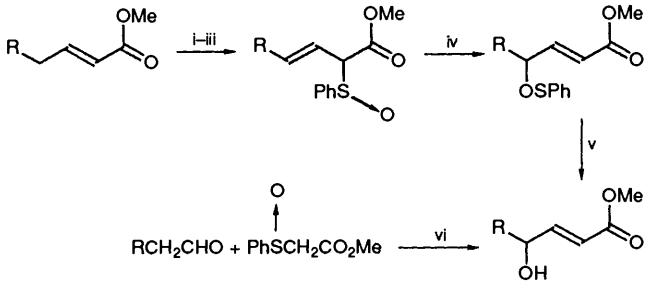
group is placed away from the bulky sulphoxide group. Elimination of the β-hydroxy group is assisted by the zinc ion, to generate the olefin. This explanation is supported by the observation that, since the arrangement about the zinc atom is sterically cluttered, bulky aldehydes, such as isobutyraldehyde only give very low yields of product. Attempted reaction with simple ketones under these conditions also failed.

Under the zinc chloride conditions, formation of the intermediate β-hydroxy ester **2** was not usually observed. Only in the case of acetaldehyde was a small amount of this intermediate **2** (R = Me) isolated. One side product consistently isolated from these condensations was a small amount of diphenyl disulphide (generally < 10%), presumably produced by some decomposition of the MBSA during the reaction.

Of the other Lewis catalysts tried, Hauser's base, bromo-

magnesium–diisopropylamide,[7] produced mixtures of the β-hydroxyesters and the conjugated esters—short reaction times (1–2 h) giving mainly the former and long reaction times (36 h) favouring the latter, although, in all cases absolute yields were less than those using zinc chloride.

The direct method of preparing the α-benzenesulphinyl-αβ-unsaturated esters **3** to **9**, listed in Table 1, allowed us to examine the [2,3]-sigmatropic rearrangements first observed by Mislow,[8] exploited by Evans[9] in the preparation of allylic alcohols, developed by Montellano and Hsu[10] and used in the synthesis of the verrucarins by Trost.[11] In these, a five-step process was carried out, the key stage being the [2,3]-sigmatropic rearrangement of the allylic sulphoxide into the allylic sulphenic ester, followed by reduction of the S–O bond to liberate the alcohol (Scheme 2).



Scheme 2 *Reagents:* i, base; ii, $PhSSO_2Ph$; iii, oxidn.; iv, heat; v, thiophile; vi, this work

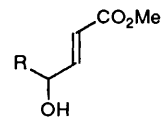
The sigmatropic rearrangement is aided by the fact that, at the same time, the double bond moves into the thermodynamically more favourable conjugated system; the rearrangement can thus be carried out under very mild conditions.

Since conjugated esters bearing γ-hydrogen atoms can be equilibrated with their βγ-isomers, we envisaged that treatment of the conjugated adducts with mild base should promote the rearrangement. Initially use of a thiophile was employed to aid reduction of the intermediate sulphenate ester. Thus heating the ester **9** with triethylamine in the presence of 1 equiv. of trimethyl phosphite did effect the desired transformation (Scheme 2) to give the alcohol **11**. In this case, the alcohol was contaminated by the presence of some of the phosphate ester **10**. This complication was in agreement with the observations of Martellano and Hsu[10] who noted that, in an analogous rearrangement, the use of conventional thiophiles gave complications whilst satisfactory results were obtained by warming the sulphoxide in a phosphate buffer at pH 7.

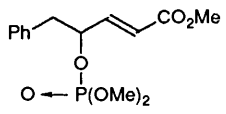
As a consequence, we resorted to use of weak nucleophiles under mildly basic conditions and the use of aqueous pyridine in tetrahydrofuran proved to be the most effective. Thus, heating the conjugated ester **9** under these conditions at reflux for 3 h smoothly gave the α-hydroxy ester **11** in 94% yield. Some diphenyl disulphide, a side product from the cleavage of the intermediate sulphenate ester, was also isolated. The generality of this process was illustrated by rearranging the sulphoxide **4** under similar conditions to give the corresponding alcohol **12** in 52% yield.

One further product was also sought from the rearrangement reaction of compound **9**: that arising from collapse of the intermediate sulphenate ester by elimination of the thiol to give the γ-ketone **14**, itself prepared by oxidation of the alcohol **11** with pyridinium chlorochromate. However, this type of fragmentation was not observed, even in the presence of stronger bases. Presumably cleavage of the weak sulphur–oxygen bond of the sulphenic ester intermediate is always preferred.

Rearrangement of the extended conjugated ester **6**, obtained



11 R = CH_2Ph
12 R = n-C_4H_9
13 R = $CH{=}CH_2$



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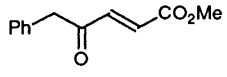

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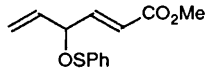

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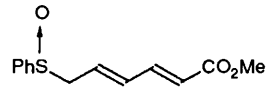

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from MBSA by condensation with crotonaldehyde, was next attempted. It was reasoned that this might rearrange to the intermediate sulphenic ester **15**, which could then either collapse to give the alcohol **13** or, alternatively, undergo a further [2,3]-sigmatropic rearrangement to the sulphoxide **16**. In the event, use of aqueous pyridine left the starting sulphoxide **6** unchanged, pointing to the fact that a stronger base was required to effect the initial double-bond shift. With 1,5-diazabicyclo[4.3.0]non-5-ene (DBN) a clean rearrangement occurred from which the new sulphoxide **16** was isolated in 72% yield.

Two mechanisms may be postulated for the rearrangement, either the double [2,3]-sigmatropic process described above or a one-step [1,5]-shift, involving the direct migration of the benzenesulphinyl group to the γ-position. Although we have no conclusive evidence for either mechanism, steric factors would favour the two-step process, the transition state leading to the [1,5]-shift being less easy to accommodate (but not impossible) than those involved in the two-step process. However attempts to trap the intermediate sulphenate ester **15** with thiophiles, such as trimethyl phosphite failed, the only product isolated being the rearranged sulphoxide **16**, again in high yield.

The geometry of the product dienoic ester was of interest. Use of the $Eu(fod)_3$ NMR shift reagent to resolve the complex coupling pattern of the vinylic protons established a *trans* relationship (2*E*,4*E*) about both double bonds; a [1,5]-sigmatropic process would initially result in formation of the 2*E*,4*Z*-isomer, although subsequent *cis–trans* isomerisation might occur under the conditions of the reaction.

One further experiment to study the rearrangement of **6** to **16** was made. Since [2,3]-sigmatropic shifts are reversible,[8] the sulphoxide **16** would be in thermal equilibrium with the sulphenic ester intermediate **15**. However, prolonged heating of the sulphoxide in the presence of trimethyl phosphite did not lead to generation of the alcohol **13**. Use of more vigorous conditions only led to general decomposition products.

The observation tha MBSA could be condensed with aldehydes to give the conjugated esters, *e.g.* **9**, and that the latter could be rearranged under mild base conditions to the rearranged alcohols, *e.g.* **11**, opens up the possibility of effecting the overall procedure in a one-pot process.

Earlier work[12] had established that MBSA could be condensed with unsaturated ketones in the presence of magnesium methoxide, a process developed into a new route for making phenols.[13] Use of magnesium methoxide in methanol was thus used in the reactions with aldehydes. Reaction of 3-phenylpropionaldehyde with MBSA using

magnesium methoxide as catalyst in dry methanol afforded, directly, the rearranged alcohol **11**. Although the isolated yield was only modest (25%) in this case, reaction of MBSA with hexaldehyde under these conditions gave the rearranged alcohol **12** in 58% yield. The reaction thus represents an easy route to γ-hydroxy αβ-unsaturated esters; structures which exist in a wide range of natural products.[14] The method provides a useful addition to existing methodology.[15]

Experimental

M.p.s were determined on a Kofler block and are uncorrected. IR spectra were recorded on a Perkin-Elmer 297 spectrophotometer either on solutions in chloroform, Nujol mulls or, for liquids, as films. ^{1}H NMR spectra were recorded on a Varian 360A (60 MHz) or JEOL FX90Q (90 MHz) spectrometer and are quoted in ppm relative to tetramethylsilane as internal reference. All *J*-values are in Hz. Mass spectra were recorded on an AEI-Kratos MS 9/50 instrument. Microanalytical determinations were performed by the University of Leeds Microanalytical Laboratory.

TLC was carried out on glass plates precoated with Merck Kieselgel 60GF$_{254}$ and column chromatography was generally carried out on MN Kieselgel 60 (Camlab) packed and run under pressure. Solvents were dried and distilled before use using standard methods.[16] Light petroleum refers to the fraction of boiling range 60–80 °C and ether refers to diethyl ether. Solutions of organic compounds from extractions were dried over anhydrous sodium sulphate before being filtered and evaporated under reduced pressure on a rotary evaporator. Dry nitrogen was used as the atmosphere for reactions. Methyl benzenesulphinylacetate (MBSA) was prepared by oxidation of methyl benzenethioacetate with 3-chloroperbenzoic acid and had m.p. 48–50 °C (lit.,[17] 48–50 °C).

Condensation of MBSA with Aldehydes.—General method. MBSA (198 mg, 1 mmol) in tetrahydrofuran (THF) (3 cm^3) was added dropwise to a stirred suspension of sodium hydride (43 mg, 55% w/w dispersion in oil; 1 mmol) in THF (4 cm^3) at 0 °C and the reaction mixture warmed to room temperature for 30 min before re-cooling to 0 °C, followed by the slow addition of anhydrous zinc chloride (1.0–1.2 mmol) in THF (3 cm^3). After a further 30 min at 0 °C the aldehyde (1 mmol) in THF (3 cm^3) was added. The reaction mixture was warmed to room temperature and stirred for 15–18 h, before finally heating to reflux for 30 min, during which time a precipitate formed. Ethyl acetate and saturated aqueous ammonium chloride were added to the mixture and the organic layer was separated; the aqueous layer was extracted with more ethyl acetate. The organic layers were combined, washed with water, dried and evaporated under reduced pressure to afford the products, which were purified either by preparative TLC or by column chromatography. In this manner the following compounds were prepared.

Methyl (E)-3-*phenyl*-2-(*phenylsulphinyl*)*prop*-2-*enoate* **3**. Yield, 20%, isolated as an oil; ν_{max}/cm^{-1} 1740, 1620, 1280 and 1050; δ 3.6 (3 H, s, MeO), 7.25–7.75 (11 H, m) (Found: M$^+$, 286.06589. $C_{16}H_{14}O_3S$ requires *M*, 286.06635).

Methyl (E)-2-(*phenylsulphinyl*)*oct*-2-*enoate* **4**. Yield, 38%, isolated as an oil; ν_{max}/cm^{-1} 1730, 1630, 1440, 1250 and 1040; δ 0.92 (3 H, t, *J* 7, Me), 1.18–1.70 (6 H, m), 2.75 (2 H, q, *J* 7, CH_2), 2.65 (3 H, s, MeO), 7.18 (1 H, m, vinylic H), 7.4–7.75 (5 H, m, aromatic H) (Found: C, 64.0; H, 7.3; S, 11.4%. $C_{15}H_{20}O_3S$ requires C, 64.25; H, 7.2; S, 11.4%).

Methyl (E)-2-(*phenylsulphinyl*)*hept*-2-*enoate* **5**. Yield, 35%, isolated as an oil; ν_{max}/cm^{-1} 1725, 1630, 1480 and 1030; δ 0.92 (3 H, t, *J* 7, Me), 1.18–1.78 (4 H, m), 2.75 (2 H, m) 3.65 (3 H, s, MeO), 7.17 (1 H, m, vinylic H), 7.33–7.80 (5 H, m, aromatic H) (Found: C, 63.1; H, 6.8; S, 12.0%; M$^+$, 266.09762. $C_{14}H_{18}O_3S$ requires C, 63.1; H, 6.8; S, 12.0%, *M*, 266.09766).

Methyl (2E,4E)-2-(*phenylsulphinyl*)*hexa*-2,4-*dienoate* **6**. Yield, 39%, m.p. 64–65 °C, isolated as an oil; ν_{max}/cm^{-1} 1720, 1635, 1480 and 1040; δ 1.98 (3 H, dd, *J* 7, 1, Me), 3.68 (3 H, s, MeO), 6.52 (1 H, dq, *J* 16, 7, vinylic H), 7.15 (1 H, dd, *J* 12, 1, vinylic H), 7.32–7.78 (6 H, m, aromatic and vinylic H) (Found: C, 62.5; H, 5.75; S, 12.55%. $C_{13}H_{14}O_3S$ requires C, 62.4; H, 5.6; S, 12.8%).

Methyl (E)-2-(*phenylsulphinyl*)*but*-2-*enoate* **7**. Yield, 34%, isolated as an oil; ν_{max}/cm^{-1} 1730, 1640, 1270 and 1040; δ 2.30 (3 H, d, *J* 7, Me), 3.67 (3 H, s, MeO), 7.44 (6 H, m, aromatic and vinylic H) (Found: M$^+$, 224.05098. $C_{11}H_{12}O_3S$ requires *M*, 224.05071). In this experiment a small quantity of the alcohol **2** (R = Me) was isolated as an oil (9%). This diastereoisomeric mixture showed, ν_{max} 3600–3100, 1740 and 1030; δ 1.2–1.5 (3 H, m, Me), 3.0–3.8 (5 H, m), 4.38–4.78 (1 H, br s, exchanged with D_2O, OH), 7.40–7.90 (5 H, m, aromatic H).

Methyl (E)-4-*methyl*-2-(*phenylsulphinyl*)*pent*-2-*enoate* **8**. Yield, 12%, isolated as an oil; ν_{max}/cm^{-1} 1730, 1630 and 1050; δ 1.16, 1.17 (2 × 3 H, d, *J* 7, Me_2), 3.40–3.60 (1 H, m), 3.68 (3 H, s, MeO), 6.95 (1 H, d, *J* 7, vinylic H), 7.40–7.78 (5 H, m, aromatic H) (Found: M$^+$, 242.08181; $C_{13}H_{16}O_3S$ requires *M*, 252.08201).

Methyl (E)-5-*phenyl*-2-(*phenylsulphinyl*)*pent*-2-*enoate* **9**. Yield, 48%, isolated as an oil; ν_{max}/cm^{-1} 1725, 1635 and 1050; δ 2.8–3.3 (4 H, m), 3.70 (3 H, s, MeO), 7.2–7.4 (6 H, m, vinylic and aromatic H) (Found: C, 68.5; H, 5.6; S, 10.0. $C_{18}H_{18}O_3S$ requires C, 68.8; H, 5.8; S, 10.2%).

Rearrangement of 2-(*Phenylsulphinyl*)*alkenoates.—General method.* The conjugated ester (3 mmol) was dissolved in pyridine (10 cm^3) and water (1 cm^3) and the solution stirred at room temperature for 18 h before adding THF (10 cm^3) and heating the mixture to reflux for 3 h. The bulk of the solvent was removed under reduced pressure and the residue partitioned between ether (50 cm^3) and saturated aqueous ammonium chloride (50 cm^3). The layers were separated, the aqueous phase was re-extracted with ether and the organic layers were combined, dried and evaporated. The residue was chromatographed through silica gel to afford the corresponding allylic alcohol. In this manner the following alcohols were prepared.

Methyl (E)-4-*hydroxy*-5-*phenylpent*-2-*enoate* **11**. Yield, 94% from the sulphoxide **9**; ν_{max}/cm^{-1} 3400, 1720, 1655; δ 2.0 (1 H, s, exchanged by D_2O), 2.9 (2 H, m), 3.75 (3 H, s, MeO), 4.40 (1 H, m), 5.94 (1 H, dd, *J* 16, 1, 2-H), 6.90 (1 H, dd, *J* 16, 6, 3-H), 7.3 (5 H, m, aromatic H) (Found: C, 70.0; H, 6.83. $C_{12}H_{14}O_3$ requires C, 69.9; H, 6.8%).

Methyl (E)-4-*hydroxyoct*-2-*enoate* **12**. Yield, 52% yield from the sulphoxide **4**; ν_{max}/cm^{-1} 3500, 1720, 1660 and 1260; δ 0.75–1.05 (3 H, m), 1.1–1.8 (6 H, m), 2.74 (1 H, s, exchanged by D_2O, OH), 3.75 (3 H, s, MeO), 4.05 (1 H, m), 6.02 (1 H, br d, *J* 17, 2-H), 6.94 (1 H, dd, *J* 17, 6, 3-H) (Found: C, 62.7; H, 9.6. $C_9H_{16}O_3$ requires C, 62.8; H, 9.4%).

Use of Trimethyl Phosphite in the Rearrangement of **9**.—To the sulphoxide **9** (78 mg, 0.25 mmol) in ether (6 cm^3) were added triethylamine (0.12 cm^3, 0.84 mmol) and trimethyl phosphite (0.12 g, 1 mmol) and the solution was heated at reflux for 14 h before being cooled, diluted with ether and washed with aqueous sodium hydrogen carbonate, dilute HCl and brine, The solvent was removed and the residue chromatographed through silica gel, using 19:1 dichloromethane–ethyl acetate, to give, initially, the alcohol **11** (19 mg, 36%), identical to the material described above, and a slightly more polar material, shown to be the *phosphate ester* **10** (25 mg, 32%), ν_{max}/cm^{-1} 1720, 1660, 1270, 1040 and 750. The ester was characterised by hydrolysis

with potassium hydroxide (2 mol dm^{-3}) at room temperature for 14 d to give the alcohol **11**.

Preparation of Methyl (E)-4-*Oxo*-5-*phenylpent*-2-*enoate* **14** (R = $PhCH_2$).—The allylic alcohol **11** (0.5 g, 2.4 mmol) in dichloromethane (20 cm^3) was added to pyridinium chlorochromate (1.04 g, 4.8 mmol) and powdered 3 Å molecular sieve (2 g) and the mixture stirred at room temperature for 1 h. Ether (50 cm^3) was added, the mixture was filtered through silica gel, and the filtrate was evaporated to give the *ketone* (0.465 g, 94%) as an oil, ν_{max}/cm^{-1} 1720, 1690, 1620 and 1600; δ 3.75 (3 H, s, MeO), 3.9 (2 H, s, CH_2), 6.7 (1 H, d, *J* 16, vinylic H), 7.0 (1 H, d, *J* 16, vinylic H), 7.3 (5 H, m, aromatic H).

Rearrangement of the Dienoic Ester **6**.—To a solution of the sulphoxide (100 mg, 0.4 mmol) in THF (20 cm^3) at −15 °C was added diazabicyclononene (12 mg, 0.1 mmol) in THF (3 cm^3) and the solution left for 16 h at room temperature before removing the solvent under reduced pressure and partitioning the residue between ethyl acetate and brine. The organic extract was dried and the residue chromatographed through silica gel to give *methyl* (2E,4E)-4-(*phenylsulphinyl*)*hexa*-2,4-*dienoate* **16** (45 mg, 45%) as a crystalline solid, m.p. 74–75 °C; ν_{max} 1740, 1650 and 1040, δ 3.7 (3 H, s, MeO), 3.5–3.9 (2 H, m, CH_2), 5.8 (1 H, d, *J* 17, vinylic H), 5.62–6.0 (1 H, m, vinylic H), 6.2 (1 H, dd, *J* 11, 17, vinylic H), 7.2 (1 H, dd, *J* 11, 17, vinylic H), 7.55 (5 H, m, aromatic H) (Found: C, 62.3; H, 5.45; S, 13.0. $C_{13}H_{14}O_3S$ requires C, 62.4; H, 5.6; S, 13.0%).

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