tracted with an ether–hexane mixture (3:1) and the combined extracts were dried over anhydrous magnesium sulfate. Preparative thin layer chromatography on silica gel afforded 165 mg (52%) of pure (±)-*ar*-turmerone (**1**): ν_{max} ($CHCl_3$) 1685 (C=O), 1620 (C=CH–), 1515 ($-C_6H_4-$), 819 cm^{-1} ($p-C_6H_4-$); nmr (CCl_4) δ 7.00 (s, 4 H, $p-CH_3C_6H_4-$), 5.90 [m, 1 H, $-CH=C(CH_3)_2$], 3.20 [m, 1 H, $C_7H_7\mathbf{CH}(CH_3)-$], 2.50 (m, 2 H, $-CH_2CO-$), 2.25 (s, 3 H, $p-\mathbf{CH_3}C_6H_5-$), 2.08 [s, 3 H, $-COCH=C(\mathbf{CH_3})_2$, methyl cis to carbonyl], 1.81 [s, 3 H, $-COCH=C(CH_3)_2$, methyl trans to carbonyl], 1.20 [d, 3 H, $C_7H_7CH(\mathbf{CH_3})-$]; *m/e* 216. The analytical sample was obtained as a colorless oil by preparative tlc followed by molecular distillation, bp (bath) 90° (0.07 mm).

Anal. Calcd for $C_{15}H_{20}O$: C, 83.28; H, 9.32. Found: C, 83.42; H, 9.32.

Acknowledgment.—Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

Registry No.—**1**, 38142-58-4; **2**, 4202-14-6; **4**, 40601-28-3; **5**, 40601-29-4.

An Improved Synthesis of 2-Methoxypropene[1]

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Received March 13, 1973

The advantages of the use of 2-methoxypropene[2] (**1**), 1-methoxycyclohexene (**2**), and 4-methoxy-5,6-dihydro-2*H*-pyran (**3**), over dihydropyran (**4**), for the protection of alcohol functions have been discussed.[3] The reaction of **1** with allylic alcohols to form allyl vinyl ethers which rearrange on heating to γ,δ-unsaturated ketones has been described.[4] Because we were interested in another use for **1**, we sought to improve the tedious methods for preparation described.[5]

The improved method described herein involves adding acetone dimethyl ketal to a solution of succinic anhydride and benzoic acid[6] in pyridine and diethylene glycol dimethyl ether (diglyme) at 110–120°. The desired **1** distills as formed in excellent yield. An 8-mol run can be completed in 2–2.5 hr. When acetic anhydride[4] is used in place of succinic anhydride, methyl acetate codistills with **1** and an aqueous alkaline hydrolysis of the mixture is necessary to obtain pure **1**.

The method using succinic anhydride is mainly valuable when a low-boiling vinyl ether is desired. In the case of the formation of α-methoxystyrene from acetophenone dimethyl ketal the method using succinic anhydride requires an aqueous work-up and hence has no advantage over that using acetic anhydride, but the example is given to indicate the generality of the method.

Experimental Section

2-Methoxypropene (**1**).—To a stirred solution at 110–120° of 820 g (8.2 mol) of succinic anhydride and 24 g (0.2 mol) of benzoic acid in 640 g (8 mol) of pyridine and 600 ml of diglyme in a 3-l. three-necked round-bottomed flask fitted with a pressure-equalizing addition funnel, thermometer, and an efficient fractionating column[7] was added 832 g (8 mol) of acetone dimethyl ketal over 1.5 hr. Shortly after the ketal addition was commenced **1** distilled. After about 2 hr 547 g (95%) of **1** was obtained as a colorless liquid, bp 37°. This product, nmr (CCl_4, TMS δ 0.0) 3.80 (s, 2, $=CH_2$), 3.48 (s, 3, CH_3O-), 1.75 (s, 3, CH_3C), had a strong ir band (20% in CCl_4) at 6.08 μ (1640 cm^{-1}) for an olefin and no bands at 3.00 (3350 cm^{-1}, methanol), or near 5.8 μ (1750 cm^{-1}, acetone).

That the amount of pyridine used can be greatly decreased was shown by a similar experiment in which 208 g (2.0 mol) of acetone dimethyl ketal was added during 20 min to a solution at 110–120° of 220 g (2.2 mol) of succinic anhydride and 12 g (0.1 mol) of benzoic acid in 250 ml of diglyme and 16 g (0.2 mol) of pyridine. The yield of pure **1** obtained in 70 min was 130 g (90%).

α-Methoxystyrene (**2**).—To a solution at 110–120° of 33 g of succinic anhydride and 1.2 g of benzoic acid in 30 ml of pyridine and 35 ml of diglyme was added 46 g of acetophenone dimethyl ketal during 15 min. After a further 15 min the mixture was cooled and added to 200 ml of 2 *N* potassium hydroxide. The neutral product was extracted with ether and worked up in a conventional way to yield 36.0 g (97%) of **2**, bp 114° (50 mm).[8]

Registry No.—**1**, 116-11-0; **2**, 4747-13-1; acetone dimethyl ketal, 77-76-9; acetophenone dimethyl ketal, 4316-35-2.

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[1] This work was supported by Grant 12554 of the National Science Foundation.

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[6] The reaction takes place much more slowly if benzoic acid is omitted.

[7] We used a 1.75 × 60 cm column packed with stainless steel heligrad packing. However, a 1.75 × 30 cm column packed with 0.25-in. glass helices worked almost as well.

[8] S. Winstein and L. L. Ingraham, *J. Amer. Chem. Soc.*, **77**, 1738 (1955), gave bp 85–89° (20 mm).

Improved Synthesis of Deuterated Olefins from the Wittig Reaction

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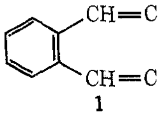
Received March 13, 1973

The utility of the Wittig reaction[1] for the synthesis of deuterated alkenes has been plagued by the occurrence of extensive deuterium scrambling and exchange with the reaction medium. Atkinson and coworkers[2] found that *n*-propyl- or *n*-butyllithium should be used as a base rather than the anion of dimethyl sulfoxide[3] in order to minimize deuterium exchange *via* enolization of the carbonyl compound. However, work-up procedures are tedious and yields are characteristically low.

In the course of some spectroscopic studies, we required a sample of *o*-divinylbenzene-d_4 (**1**). Survey of the literature revealed a synthesis[4] from Ph_3PCD_3Br



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