accomplished in this way. The mole percentages of these materials were determined from the corrected areas of the peaks. The *meta* isomer could not be resolved despite the use of various conditions and column packings. Analysis for the *meta* isomer was carried out by a previously reported[2] standard base-line technique on a Beckman IR-9 infrared spectrophotometer at 12.90 μ. The mean of several trials was subtracted from the *meta* + *para* value obtained by vpc to give the isomer distributions. A summary of several runs is given in Table V.

TABLE V

PRODUCT ISOMER DISTRIBUTIONS IN THE CHLORINATION OF ETHYLBENZENE IN ACETIC ACID

Run	*ortho*	*meta*	*para*
1	52.1	0.53	47.4
2	53.5	0.48	46.0
3	51.2	0.58	48.3
Mean	52.3	0.53	47.2

A similar procedure was used on the products from the chlorination of toluene in acetic acid at 25°. The results, shown in Table II, are in agreement with reported values.[5] The infrared analysis for *m*-chlorotoluene was made at 12.96 μ.

Registry No.—I, 100-41-4; acetic acid, 64-19-7; *o*-chloroethylbenzene, 89-96-3; *m*-chloroethylbenzene, 620-16-6; *p*-chloroethylbenzene, 622-98-0.

Regeneration of Carbonyl Compounds from Oximes Using Iron Pentacarbonyl and Boron Trifluoride

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Received March 10, 1967

The parent carbonyl compound may be regenerated from an oxime by treatment of the latter in aqueous solution with sodium bisulphite,[1] levulinic or pyruvic acids,[2] or formaldehyde and concentrated hydrochloric acid.[3] However, there may be instances when it is desirable to effect this transformation under anhydrous conditions. We have found that treatment of an oxime with an equimolar quantity of iron pentacarbonyl and a catalytic amount of boron trifluoride in refluxing butyl ether results in the formation of the carbonyl compound in 55–81% yield. The examples listed in Table I indicate that the reaction is applicable to the oximes of aldehydes and ketones of widely differing character; the limitations, if any, of the reaction are not yet apparent.

The mechanism of the reaction, and hence the function of the boron trifluoride, are not yet known. Reactive of santonin oxime ($C_{15}H_{19}NO_3$) with iron pentacarbonyl alone gave a complex $C_{15}H_{19}NO_3\cdot Fe(CO)_3$;[4] the other oximes failed to react. No reaction took place when the oximes were treated with a catalytic amount of boron trifluoride in butyl ether. No carbonyl compound was regenerated by treatment of fluorenone phenylhydrazone or of N-2,6-trichloro-*p*-benzoquinoneimine with iron pentacarbonyl and boron trifluoride. Hence it seems likely that the oxygen atom of the regenerated carbonyl group comes from the oxygen atom of the oxime.

TABLE I

YIELDS OF CARBONYL COMPOUNDS FROM OXIMES

Parent carbonyl compd	Reflux time, hr	Isolation procedure	Yield, %
Cyclohexanone	20	. . .	81
4-Methyl-4-trichloromethyl-cyclohexadien-1-one	10	b	72
Fluorenone	16	a	69
Cholest-4-en-3-one[a]	16	d	67
Santonin	18	c	67
O-Methylpodocarpinal	17	c	55

[a] Oxime mp 152–152.5, prepared according to C. W. Shoppee, G. Kreiger, and R. N. Mirrington, *J. Chem. Soc.*, 1050 (1962).

Experimental Section

A mixture of the oxime (2–35 mmoles) and iron pentacarbonyl (1.1 mole/mole of oxime) in dry butyl ether (50–100 ml) containing boron trifluoride etherate (about 5% w/w of oxime) was refluxed with stirring under nitrogen. The solution was cooled and filtered and the solvent removed at 30 mm (except when the volatile cyclohexanone was formed; this was isolated as the 2,4-dinitrophenylhydrazone by treating the filtrate with 2,4-dinitrophenylhydrazine in the usual manner). The residue of crude carbonyl compound was purified by trituration with petroleum ether (bp 30–60°), hexane, or methylene chloride (procedures a, b, or c, respectively) or by chromatography on Florisil using acetone as eluent (procedure d). The purity of the products recorded in Table I was indicated by melting points agreeing with values in the literature and by the absence of more than one spot on thin layer chromatograms.

Registry No.—Boron trifluoride, 7637-07-2.

Acknowledgments.—We thank Mr. M. J. Davis and Mr. W. Zehetner for the gift of oximes and the National Research Council for financial support.

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The Preparation of Esters of 4-Alkyl-2,4-pentadienoic Acids by the Phosphonate Modification of the Wittig Reaction

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Received March 8, 1967

This note describes conditions for the rapid and convenient preparation of the ethyl esters of several 4-alkyl-2,4-pentadienoic acids from 2-alkylacroleins (**1**) and triethyl phosphonoacetate (**2**). Subsequent saponification of the esters gives the corresponding carboxylic acids and this route has been found to be useful for the preparation of several 4-alkyl-2,4-pentadienoic acids (**5**).