acetonitrile (1:10) made 0.2 M in Bu_4NClO_4. A constant potential of −1.6 V vs. SCE was applied to the resulting solution at room temperature until 2 Faraday/mol had been taken up. After electrolysis, the reduction mixture was taken up in ether and stripped of solvent. The infrared spectrum of the crude product mixture showed no absorption in the OH stretch region. The components of the mixture were isolated by preparative gas–liquid chromatography (5 ft × 0.25 in. 20% SE-30 on 100-mesh Chromosorb P, 150 °C). The first fraction exhibited spectral properties identical with those of an authentic sample of **21**.[11] The second fraction was characterized by high-resolution mass spectrometry (Du Pont CEC 21-110 instrument) as an acetonitrile adduct of **2**. The absence of hydrodimer was established by the absence of a peak upon injection of the crude mixture at the retention volume of an authentic sample of the analogous hydrodimer **16**.

Acknowledgment. We are grateful to the National Science Foundation and to the Robert A. Welch Foundation for financial support of this research. M.A.F. is grateful for support as an Alfred P. Sloan Research Fellow and as a Camille and Henry Dreyfus Teacher–Scholar.

Registry No. **2**, 36319-95-6; **3**, 1073-14-9; **14**, 80451-50-9; **15**, 85293-79-4; **16**, 85293-80-7; **17**, 80451-51-0; **18**, 85293-81-8; **19**, 85293-82-9; **20**, 66808-99-9; **21**, 85293-83-0.

Conversion of Aldehydes into Geminal Diacetates[1]

Kanwarpal S. Kochhar, Balkrishna S. Bal, R. P. Deshpande,
S. N. Rajadhyaksha, and Harold W. Pinnick*

Departments of Chemistry, Bucknell University, Lewisburg, Pennsylvania 17837, and University of Georgia, Athens, Georgia 30602

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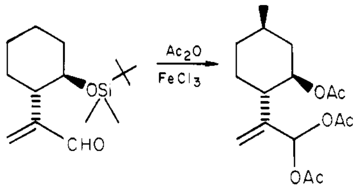
Aldehydes react with anhydrides under acid catalysis to give geminal diacetates, but the yields are usually poor.[2,3] There is keen interest in the development of an efficient method for preparing these compounds because the derived enol acetate is a particularly useful product,[4] especially when the aldehyde is α,β-unsaturated so that the enol acetate is an acetoxybutadiene.[5] This elimination reaction has been accomplished with either weak base[4,5b] or acid catalysis.[6]

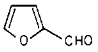
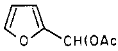
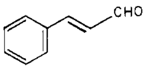
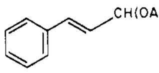
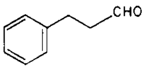
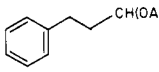
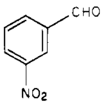
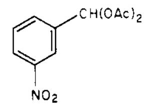
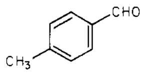
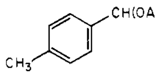
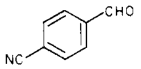
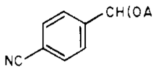
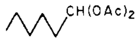
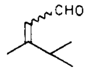
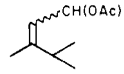
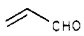
During a study aimed at the synthesis of α-methylene lactones,[7] a new preparation of a geminal diacetate was discovered (eq 1).[8] This provides an interesting protecting group for aldehydes[9] because this functionality should have moderate stability to acid and yet should be removable under very mildly basic conditions. This nicely complements the commonly used acetal aldehyde protecting groups.[9]



(1)

In order to examine the feasibility of these systems as protecting groups and synthetic intermediates, we explored the scope of this reaction by examining a variety of aldehydes (see Table I). Both aromatic and aliphatic aldehydes give good, although unmaximized, yields of the corresponding geminal diacetates. For example, benzaldehyde gives α,α-diacetoxytoluene in 79% yield, and 1,1-diacetoxyhexane is obtained in 65% yield from hexanal. In addition, aromatic aldehydes bearing either electron-donating or -withdrawing groups work with equal facility (the yields from benzaldehyde, *m*-nitrobenzaldehyde, *p*-tolualdehyde, and *p*-cyanobenzaldehyde all lie in the range 79–93%). Furthermore, all of these reactions are remarkably fast. All systems were complete in less than 30 min in contrast to the results of Miller.[3] Finally, α,β-unsaturated aldehydes also react. Elimination of acetic acid from these systems bearing a γ hydrogen leads to 1-acetoxybutadienes.[4,5]

Table I. Geminal Diacetates from Aldehydes

aldehyde	diacetate	% yield[a,b]	acetal CH chemical shift, ppm
C_6H_5CHO	$C_6H_5CH(OAc)_2$	79[c]	7.67
CH_3CH_2CHO	$CH_3CH_2CH(OAc)_2$	57	6.72
		60[c]	7.70
		60[c]	7.33[d]
		89	6.85
		93[c]	7.73
		93[c]	7.63
		85[c]	7.32
		63	6.65
		70	7.35
	$CH_2{=}CHCH(OAc)_2$	64	7.03

[a] The percent yields are not maximized. In most cases, the value given is from a single experiment. [b] All yields refer to pure isolated products. [c] Solids. [d] This value is approximate because of overlapping aromatic signals.

The stability of these geminal diacetates has been studied. Thus, they are stable when stirred with either methanol overnight or 10% aqueous HCl in methanol for 30 min. Furthermore, neither 10% aqueous sodium carbonate and ether for 70 min nor sodium bicarbonate in aqueous THF for 4 h has any effect. Complete conversion

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* Address correspondence to this author at Bucknell University.

[1] Protecting Groups in Organic Synthesis. 8. For part 7, see: Barot, B. C.; Pinnick, H. W. *J. Org. Chem.* **1981**, *46*, 2981.

[2] Thiele, J.; Winter, E. *Justus Liebigs Ann. Chem.* **1960**, *311*, 355.

[3] A recent paper outlines the use of phosphorus trichloride as a catalyst for this reaction with good results: Michie, J. K.; Miller, J. A. *Synthesis* **1981**, 824. This paper contains an excellent historical account of this reaction.

[4] For example, see: (a) Wichterle, O.; Hudlicky, M. *Czech. Collect. Chem. Commun.* **1947**, *12*, 564. (b) Georgieff, K. K.; Dupre, A. *Can. J. Chem.* **1960**, *38*, 1070. (c) Blanc, P. Y. *Helv. Chim. Acta* **1961**, *44*, 1.

[5] These are especially useful in Diels–Alder reactions. For example: see Snider, B. B.; Amin, S. G. *Synth. Commun.* **1978**, *8*, 117.

[6] (a) Hagemeyer, H. J.; Hull, D. C. *Ind. Eng. Chem.* **1949**, *41*, 2920. (b) McDonald, E.; Suksamrarn, A.; Wylie, R. D. *J. Chem. Soc., Perkin Trans. 1* **1979**, 1893.

[7] Bal, B. S.; Pinnick, H. W. *Heterocycles* **1981**, *16*, 2091.

[8] Acetic anhydride and ferric chloride convert *tert*-butyldimethylsilyl ethers into acetate esters: Ganem, B.; Small, V. R., Jr. *J. Org. Chem.* **1974**, *39*, 3728.

[9] For other protecting groups for aldehydes, see: Greene, Theodora W. "Protective Groups in Organic Synthesis"; Wiley: New York, 1981.

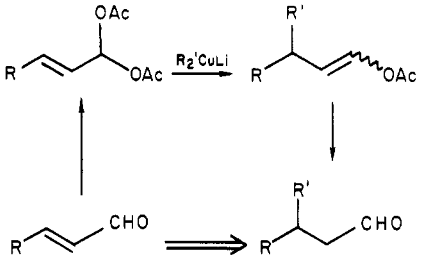
0022-3263/83/1948-1765$01.50/0

to the corresponding aldehyde is accomplished with either sodium hydroxide or potassium carbonate in aqueous THF overnight. Consequently, removal of this potential protecting group is possible under very mild conditions. Stability toward aqueous acid makes this protecting group complimentary to acetals.[9]

Another possible application of geminal diacetates derived from α,β-unsaturated aldehydes is in connection with the interesting S_N2' coupling reaction which allylic acetates undergo with organocuprates (eq 2).[10] With geminal di-

(2)

acetates, β-substituted aldehydes would be expected after hydrolysis (eq 3). This reaction sequence allows conjugate



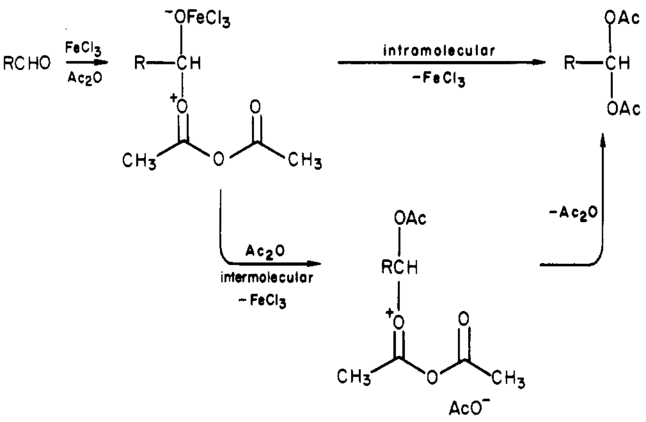
(3)

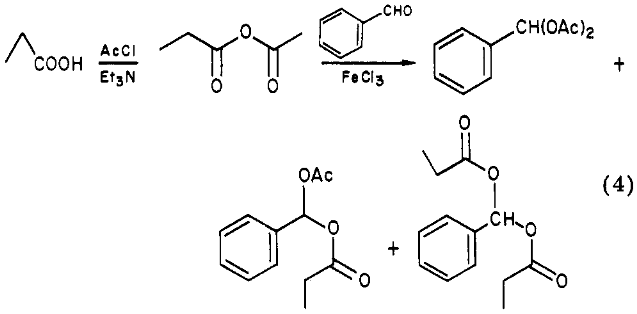
addition to unsaturated aldehydes—not always an easy reaction because of the high electrophilicity of the aldehyde carbonyl.[11,12]

The mechanism of the title reaction could involve either intermolecular or intramolecular transfer of the second acetate group after initial attack by acetic anhydride (Scheme I).

In order to test this aspect of the conversion, we prepared acetic propionic anhydride from acetyl chloride and propionic acid, and the geminal acylation reaction was run with this compound. Analysis of the products by GC/MS showed a nearly 1:2:1 ratio of geminal diacetate, mixed acetate/propionate, and geminal dipropionate, respectively (eq 4). This supports the intermolecular pathway of Scheme I.

Scheme I





(4)

Experimental Section

All proton NMR spectra were recorded on either a Varian EM-360 or a JEOL FX-90Q instrument. The carbon-13 NMR spectra were run on the latter spectrometer. All chemical shifts are reported in parts per million from tetramethylsilane as an internal standard. Mass spectra were obtained by using a Finnigan 4021-C GC/MS. Chemically induced (CI) mass spectra were obtained by using methane as the carrier gas. Infrared spectra were run on a Perkin-Elmer Model 137 or 297 spectrophotometer with polystyrene for calibration (1601 cm^{-1}). Melting points were taken on a Mel-Temp apparatus and are uncorrected. All distillations were carried out using a Büchi Kugelrohr apparatus.

Benzaldehyde. To 6 mL of freshly distilled acetic anhydride at 0 °C was added 2.12 g (20.0 mmol) of benzaldehyde. The resulting solution was stirred for 15 min, and 0.1 g[13] of anhydrous ferric chloride was added. After another 20 min of stirring, the reaction mixture was poured into 50 mL of hexane and 10 mL of water. The aqueous layer was extracted with three 30-mL portions of hexane, and the combined organic extracts were washed with three 30-mL portions of water. The organic phase was dried and concentrated to give 3.2 g (79%) of white crystalline solid, pure by GC: mp 44–46 °C (lit.[3] mp 44–46 °C); IR (Nujol) 1750, 1380, 1245, 1210, 1065, 1015, 960, 758, 695 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.67 (s, 1 H), 7.4 (s, 5 H), 2.1 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 168.645, 135.652, 129.693, 128.610, 126.659, 89.767, 20.748; GC/MS (EI) *m/e* (relative intensity) 208 (P, 2), 165 (9), 149 (6), 123 (3), 107 (40), 105 (70), 77 (28), 51 (13), 43 (100).

Propionaldehyde. From 6 mL of acetic anhydride and 1.5 g (25.9 mmol) of propionaldehyde as above was obtained 2.37 g (57.3%) of colorless liquid pure by GC: bp 115–118 °C (8 mm); IR (NaCl) 3000, 1760, 1460, 1430, 1245, 1205, 1115, 1062, 1015, 965 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 6.72 (t, J = 6 Hz, 1 H), 2.1 (s, 6 H), 1.4–2.0 (m, 7 H), 1.0 (t, J = 7 Hz, 3 H); ^{13}C NMR ($CDCl_3$) 168.916, 91.392, 26.599, 20.640, 7.638; GC/MS (CI), *m/e* (relative intensity) 161 (P + 1, 4), 145 (10), 131 (2), 119 (10), 103 (49), 101 (100), 89 (23), 61 (40), 59 (28).

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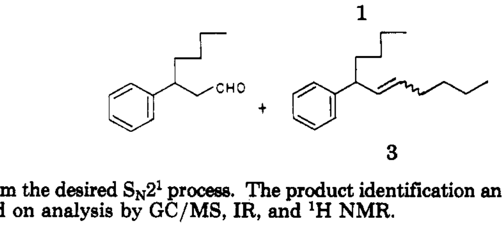
[10] (a) This reaction has been studied extensively. For example, see: Posner, G. H. *Org. React.* **1975**, *22*, 281. Goering, H. L.; Singleton, V. D., Jr. *J. Am. Chem. Soc.* **1976**, *98*, 7854. Kreft, A. *Tetrahedron Lett.* **1977**, 1035. See also: Yamamoto, Y.; Yamamoto, S.; Yatagai, H.; Maruyama, K. *J. Am. Chem. Soc.* **1980**, *102*, 2318.

[11] Posner, G. H. *Org. React.* **1972**, *19*, 24. El Bouz, M.; Roux-Schmitt, M.-C.; Wartski, L. *J. Chem. Soc., Chem. Commun.* **1979**, 779. The Clive research group has found that "$(CH_3)_5Cu_3Li_2$" will transfer a methyl group to the β-position of α,β-unsaturated aldehydes: Clive, D. L. J.; Farina, V.; Beaulieu, P. *J. Chem. Soc., Chem. Commun.* **1981**, 643. Clive, D. L. J.; Farina, V.; Beaulieu, P. L. *J. Org. Chem.* **1982**, *47*, 2572. Normant recently has studied the addition of cuprates to α,β-unsaturated aldehydes: Chuit, C.; Foulon, J. P.; Normant, J. F. *Tetrahedron* **1981**, *37*, 1385.

[12] In the event, the geminal diacetate derived from cinnamaldehyde reacts with excess lithium di-*n*-butylcuprate (prepared as outlined in: Posner, G. H. *Org. React.* **1975**, *22*, 297) to give a 1:6:3 mixture of the expected enol acetate, the aldehyde hydrolysis product, and the compound resulting from an initial S_N2 coupling reaction followed by an S_N2' reaction with the intermediate allylic acetate. Thus, 70% of the products result from the desired S_N2' process. The product identification and ratio are based on analysis by GC/MS, IR, and ^{1}H NMR.



1

3

[13] The amount of ferric chloride can be varied from 0.1 to 0.2 g without significantly changing the percent yield. Commercially available anhydrous ferric chloride is adequate for this reaction.

Furfural. From excess acetic anhydride and 1.20 g (12.5 mmol) of furfural as usual was obtained 1.5 g (60%) of colorless liquid which solidified to a white crystalline solid, pure by GC: bp 120–125 °C (15 mm) [lit.[3] bp 132–136 °C (18 mm)]; IR (Nujol) 1755, 1500, 1470, 1245, 1205, 1075, 1060, 1015, 965, 935, 925, 902, 880, 830, 750 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.70 (s, 1 H), 7.45 (br s, 1 H), 6.45 (m, 2 H), 2.1 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 168.374, 148.113, 143.678, 110.461, 109.703, 83.591, 20.586; GC/MS (EI), m/e (relative intensity) 198 (P, 3), 155 (11), 139 (6), 113 (3), 97 (60), 95 (59), 69 (3), 43 (100), 39 (27).

Cinnamaldehyde. Cinnamaldehyde (1.80 g, 13.6 mmol) and excess acetic anhydride as usual gave 1.95 g (60%) of a white solid which was recrystallized from ether–hexane: mp 84–86 °C (lit.[3] mp 84–87 °C); IR (NaCl) 1755, 1660, 1615, 1490, 1470, 1245, 1195, 1120, 1060, 1005, 940, 748, 690 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.1–7.6 (m, 6 H), 6.87 (d, J = 16 Hz, 1 H), 5.93 (dd, J = 16, 7 Hz, 1 H), 2.1 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 168.591, 135.598, 135.219, 128.826, 128.664, 127.039, 121.838, 89.767, 20.857; GC/MS (EI) m/e (relative intensity) 234 (P, 1), 192 (1), 174 (1), 133 (25), 131 (56), 115 (7), 104 (12), 77 (12), 55 (11), 43 (100).

3-Phenylpropionaldehyde. From 1.50 g (11.2 mmol) of the aldehyde and excess acetic anhydride according to the usual procedure was obtained 2.36 g (89.3%) of colorless liquid, pure by GC: bp 185–190 °C (0.7 mm); IR (NaCl) 3020, 2930, 1755, 1600, 1500, 1460, 1245, 1205, 1105, 1010, 945, 745, 695 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.22 (s, 5 H), 6.85 (t, J = 6 Hz, 1 H), 2.7 (m, 2 H), 2.2 (m, 2 H), 2.1 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 168.753, 140.636, 128.501, 128.285, 126.118, 90.146, 34.671, 29.687, 20.532; GC/MS (EI), m/e (relative intensity) 176 (P − HOAc, 6), 134 (22), 116 (32), 105 (31), 91 (28), 78 (11), 65 (8), 43 (100); GC/MS (CI), m/e (relative intensity) 237 (P + 1, 1), 195 (1), 176 (1), 163 (1), 145 (4), 117 (100), 103 (5), 89 (5), 61 (15).

3-Nitrobenzaldehyde. The aldehyde (1.00 g, 6.62 mmol) was combined with excess acetic anhydride and run in the usual way to yield 1.55 g (92.5%) of a white solid, pure by GC: mp 64–66 °C; IR (Nujol) 1755, 1535, 1455, 1232, 1200, 1090, 1055, 1010, 985, 905, 815, 735 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.73 (s, 1 H), 7.4–8.5 (m, 4 H), 2.12 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 168.645, 148.438, 137.765, 132.944, 129.910, 124.547, 121.892, 88.412, 20.694; GC/MS (EI) m/e (relative intensity) 210 (P − acetyl, 2), 194 (3), 150 (18), 134 (21), 105 (10), 77 (12), 51 (13), 43 (100); GC/MS (CI), m/e (relative intensity) 294 (P + 41, 1), 282 (P + 29, 7), 224 (1), 212 (2), 194 (33), 180 (4), 164 (5), 152 (75), 136 (5), 122 (10), 103 (45), 89 (13), 61 (100).

4-Tolualdehyde. 4-Tolualdehyde (2.00 g, 16.6 mmol) was allowed to react with excess acetic anhydride in the usual way to give 3.45 g (93%) of GC-pure white solid: mp 78–80 °C (lit.[3] mp 68–70 °C); IR (Nujol) 1765, 1750, 1230, 1205, 1070, 1005, 960, 930, 815 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.10 (s, 6 H), 2.35 (s, 3 H), 7.30 (AB q, J_{AB} = 8 Hz, J_{AA} = 14 Hz, 4 H), 7.63 (s 1 H); ^{13}C NMR ($CDCl_3$) δ 20.748, 21.182, 89.821, 126.605, 129.260, 132.835, 139.661, 168.645; GC/MS (EI), m/e (relative intensity) 222 (P, 0.5), 179 (2), 163 (2), 121 (22), 119 (39), 91 (20), 43 (100).

4-Cyanobenzaldehyde. The aldehyde (0.50 g, 3.44 mmol) was allowed to react in the usual way to give 0.72 g (84.7%) of white solid pure by GC: mp 98–102 °C; IR (Nujol) 2190, 1750, 1450, 1235, 1195, 1065, 1010, 960, 830 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.67 (s, 4 H), 7.32 (s, 1 H), 2.15 (s, 6 H); ^{13}C NMR ($CDCl_3$) 168.536, 140.311, 132.456, 127.580, 118.154, 113.658, 88.575, 20.694; GC/MS (EI) m/e (relative intensity) 234 (P + 1, 1), 190 (5), 173 (2), 130 (37), 102 (14), 76 (5), 51 (7), 43 (100); GC/MS (CI), m/e (relative intensity) 274 (P + 41, 5), 262 (P + 29, 11), 234 (P + 1, 100), 219 (1), 190 (2), 174 (13), 160 (10), 145 (4), 132 (96), 118 (2), 103 (18), 89 (15), 69 (8), 61 (50).

Hexanal. The usual procedure gave a colorless liquid pure by GC: bp 128–129 °C (2 mm); IR (NaCl) 2950, 2860, 1750, 1460, 1370, 1230, 1110, 1090, 1040, 990, 950 cm^{-1}; ^{1}H NMR (CCl_4) δ 6.65 (t, J = 5 Hz, 1 H), 2.1 (s, 6 H), 1.2–1.8 (m, 8 H), 0.9 (t, J = 5 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 169.078, 90.742, 33.263, 31.475, 23.132, 22.536, 20.694, 13.922; GC/MS (CI), m/e (relative intensity) 243 (P + 41, 1), 203 (P + 1, 2), 161 (9), 143 (100), 131 (6), 103 (23), 89 (21), 83 (34), 61 (30).

Acrolein. The usual procedure gave a colorless liquid, pure by GC: bp 98–105 °C (2 mm); IR (NaCl) 3080, 2980, 2930, 1750, 1425, 1360, 1230, 1200, 1120, 1000 cm^{-1}; ^{1}H NMR (CCl_4) δ 2.1 (s, 6 H), 5.2–6.3 (m, 3 H), 7.03 (d, J = 5 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 168.536, 131.535, 120.321, 89.171, 20.694; GC/MS (CI), m/e (relative intensity) 159 (P + 1, 1), 145 (11), 117 (5), 103 (23), 99 (100), 89 (20), 61 (14), 57 (24).

Reaction of Benzaldehyde with Acetic Propionic Anhydride. Acetic propionic anhydride (prepared from acetyl chloride, triethylamine, and propionic acid) and benzaldehyde were allowed to react in the usual way to give a product which was analyzed by GC/MS as a 1:2:1 ratio of three components: A, 25% α,α-diacetoxytoluene; **B**, 50% α-acetoxy-α-propionoxytoluene; **C**, 25% α,α-dipropionoxytoluene. The mass spectra (EI) were as follows. A: m/e (relative intensity) 208 (P, 1), 165 (4), 149 (3), 123 (2), 105 (40), 77 (27), 60 (3), 51 (17), 43 (100). B: m/e (relative intensity) 222 (P, 1), 179 (2), 165 (4), 149 (3), 105 (48), 77 (32), 57 (57), 51 (22), 43 (100). C: m/e (relative intensity) 236 (P, 1), 179 (4), 163 (3), 105 (37), 77 (26), 57 (100), 51 (18).

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Registry No. Benzaldehyde, 100-52-7; propionaldehyde, 123-38-6; furfural, 98-01-1; cinnamaldehyde, 104-55-2; 3-phenylpropionaldehyde, 104-53-0; 3-nitrobenzaldehyde, 99-61-6; 4-tolualdehyde, 104-87-0; 4-cyanobenzaldehyde, 105-07-7; hexanal, 66-25-1; 3,4-dimethyl-2-pentenal, 57398-52-4; acrolein, 107-02-8; α,α-diacetoxytoluene, 581-55-5; 1,1-propanediol diacetate, 33931-80-5; 2-furanylmethanediol diacetate, 613-75-2; 3-phenyl-2-propene-1,1-diol diacetate, 64847-78-5; 3-phenyl-1,1-propanediol diacetate, 85337-09-3; (3-nitrophenyl)methanediol diacetate, 29949-19-7; (4-methylphenyl)methanediol diacetate, 2929-93-3; (4-cyanophenyl)methanediol diacetate, 36735-42-9; 1,1-hexanediol diacetate, 64847-81-0; 3,4-dimethylpent-2-ene-1,1-diol diacetate, 85337-10-6; 2-propene-1,1-diol diacetate, 869-29-4; acetic anhydride, 108-24-7; acetic propionic anhydride, 13080-96-1; α-acetoxy-α-propionoxytoluene, 85337-11-7; α,α-dipropionoxytoluene, 55696-47-4; ferric chloride, 7705-08-0.

Synthesis of Functionalized Aliphatic Aldehydes via a Copper-Catalyzed Grignard Coupling Reaction

Robert A. Volkmann,* Jeffery T. Davis, and
Clifford N. Meltz

Central Research, Pfizer Inc., Groton, Connecticut 06340

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Aldehydo esters have found widespread use in the preparation of prostaglandins,[1] leukotrienes,[2] insect pheromones,[3] and a variety of other natural products.[4] A number of procedures are available for their synthesis.[5] Those most commonly employed involve the ozonolysis of

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