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Citation:

A Desilylation and a One-Pot Desilylation-Oxidation of Aliphatic *tert*-Butyldimethylsilyl Ethers Using Catalytic Quantities of PdCl₂(CH₃CN)₂ Noel S. Wilson and Brian A. Keay pp 2918 – 2919.

Tables:

Table 1. Times and Yields for the Desilylation and Oxidation of TBDMS Ethers

| starting material | time for desilylation (h) | alcohol (% yield) | time for oxidation (h) | aldehyde or ketone (% yield) ^b |
|----------------------|---------------------------------|----------------------|------------------------------|---|
| 1.8 | 14 | 9 (91)** | 6 | 10 (86) |
| 2. 11 | 14 | 12 (80)a | 6 | 13 (76) |
| 3. 14 | 16 | 15 (73)a | 4 | 16 (79) |
| 4. 17 | 12 | 18 (82)a | 10 | 19 (69) |
| 5. 20 | 18 | $21 (86)^a$ | 22 | 22 (80) |
| 6. 23 | 13 | 24 (80) ^c | 7 | 25 (75) |
| 7. 26 | 16 | 27 (78)c | 22 | 28 (78) |
| 8. 29 | 14 | $30 (78)^a$ | 20 | 31 (70) |
| 9.32 | 20 | 33 (82)c | 7d | 34 (10) |

 $[^]a$ Isolated yields using acetone, water (5 equiv), 75 °C, 6 h. b Isolated yields using DMF:acetone (1:1), water (5 equiv), 120 °C, 9 h, and then add 10 mol % PPh3, 2-bromomesitylene (1.1 equiv). c GC yields using the procedure in footnote b above. d Addition of another 5 mol % catalyst did not affect the yield.

Table 2. Compatibility of the Desilylation-Oxidation Conditions with Other Protecting Groups of Alcohols

| entry | starting material | alcohol (% yield)# | aldehyde (% yield)ª |
|-------|------------------------|-----------------------|------------------------|
| 1 | $35, R = SiEt_3$ | 36 (56) | 37 (40) |
| 2 | 38, $R = Si(i-Pr)_3$ | 39 (80) | 40 (70) |
| 3 | 41, $R = Si(t-Bu)Ph_2$ | 42 (81) | 43 (78) |
| 4 | 44, $R = MOM$ | $45 (78)^b$ | 46 (60) |
| 5 | 47, R = Bn | 48 (80) | 49 (66) |
| 6 | 50, R = THP | 51 (61)b | 52 (-)c |
| 7 | 53, $R = Ac$ | 54 (65) ^b | 55 (55) |

^a Isolated yields. ^b Diol was present by GC. ^cNMR indicated decomposition has occurred.

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