

**From 1,2-Dialkoxyalkanes to 1,4-Dioxanes. A Transformation Mediated by NbCl₅ via
Multiple C–O Bond Cleavage at Room Temperature****

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SUPPLEMENTARY MATERIAL

Analytical and spectroscopical data for compound 2. Anal. Calcd for C₄H₁₀Cl₃O₃Nb: C, 15.7; H, 3.3; Nb, 30.4; Cl, 34.8. Found: C, 15.9; H, 3.2; Nb, 29.8; Cl, 34.0. IR (solid state): 2946w, 1466m, 1448m-s, 1277w-m, 1238w-m, 1188w, 1123w, 1071m, 1016s, 984s, 954vs [$\nu_{\text{Nb=O}}$], 927ms, 854vs, 818s cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ = 4.20 (s, 2H, CH₂), 3.98 ppm (s, 3H, CH₃). ¹³C NMR (400 MHz, CDCl₃): δ = 74.0 (CH₂), 63.5 ppm (CH₃).

NMR data for compound 4. ¹H NMR (400 MHz, CDCl₃): δ = 5.23 (t, 2H, ³J_{HH} = 5.49 Hz, CH₂ONb), 4.43 (t, 2H, ³J_{HH} = 5.49 Hz, CH₂OMe), 4.02 (s, 3H, Me) ppm. ¹³C NMR (400 MHz, CDCl₃): δ 78.7 (CH₂ONb), 78.3 (CH₂OMe), 65.2 (Me) ppm.