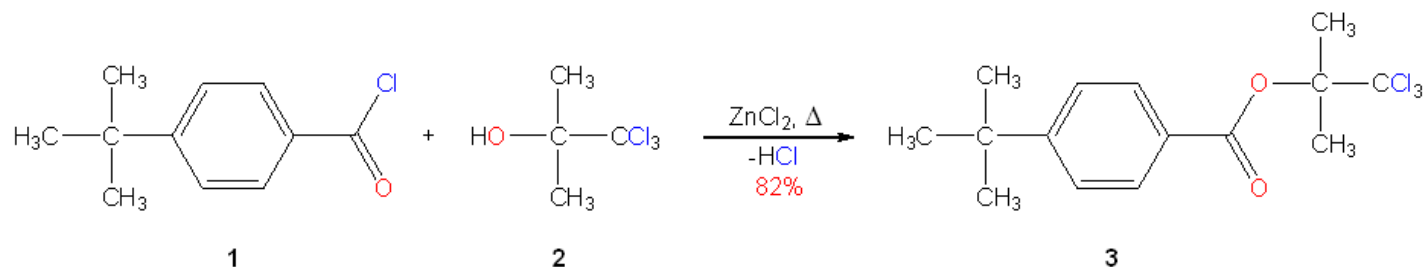


2,2,2-Trichloro-1,1-dimethylethyl 4-*tert*-butylbenzoate**Lajos Kovács^{1,*}, Péter Forgó², Zoltán Kele¹ and Éva Klement¹**¹Department of Medicinal Chemistry²Department of Organic Chemistry, University of Szeged, Dóm tér 8, H-6720 Szeged, Hungary. Fax: +36-62-54 59 71*E-mail: kovacs@ovrisc.mdche.u-szeged.hu

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In our previous work we have demonstrated the utility of the 2,2,2-trichloro-1,1-dimethylethyl (b, b, b-trichloro-*tert*-butyl, Tcb) group for the protection of carboxylic acids [1]. Herein we would like to describe the preparation of one of their representatives, the title aromatic ester. The procedure applied by us is based on the work by Gupta and Srivastava [2].

Experimental: Freshly prepared anhydrous zinc chloride (0.41 g, 3.0 mmol) was chilled in an ice-bath (0–5 °C), then 4-*tert*-butylbenzoyl chloride (2.73 mL, 2.95 g, 15.0 mmol) and anhydrous b, b, b-trichloro-*tert*-butanol (1.775 g, 10.0 mmol, prepared by *in vacuo* drying of the commercial hemihydrate over phosphorus pentoxide) were added, each in one portion. The flask was removed from the ice-bath and gently shaken to initiate the evolution of hydrogen chloride. When the gas evolution had subsided the reaction mixture was heated at 100 °C for 2 h. The mixture was diluted with diethyl ether (100 mL), washed with cold satd. NaHCO₃ solution (3 x 50 mL) and brine (50 mL), respectively. The organic phase was dried (MgSO₄) and evaporated. The oily residue solidified upon standing (2.756 g, 82%, TLC: single spot). An analytical sample was obtained by recrystallization from methanol (14 mL).

Mp. 78.7–79.8 °C.

TLC: light petroleum-ethyl acetate 7:3, R_f 0.76.Anal. calcd. for C₁₅H₁₉Cl₃O₂ (337.669): C, 53.35; H, 5.67; Cl, 31.50%; found C, 53.48; H, 5.78; Cl, 31.29%.IR (n, cm⁻¹, CHCl₃, KBr): 705m, 789m, 806m, 856w, 1016w, 1119s, 1193m, 1284s, 1370w, 1387w, 1410w, 1462w, 1606w, 1722s (ν_{C=O}), 2871w, 2905w, 2967m.¹H NMR (CDCl₃, 500 MHz, ppm): 1.34 (s, 9H, *tert*-Bu), 2.03 (s, 6H, (CH₃)₂C), 7.48 (d, 2H, *J*=8.5 Hz, arom. CHs), 8.00 (d, 2H, *J*=8.5 Hz, arom. CHs).¹³C NMR (CDCl₃, 125 MHz, ppm, assignment based on HMBC and HMQC experiments): 21.42 ((CH₃)₂CCCl₃), 31.09 ((CH₃)₃C), 35.11 ((CH₃)₃C), 88.97 (CCl₃), 107.12 ((CH₃)₂CCCl₃), 125.47 (arom. CHs), 127.89 (arom. quaternary C), 129.74 (arom. CHs), 156.99 (arom. quaternary C), 164.46 (COO).CI-MS (isobutane, m/z): 341, 339, 337 ([M+H]⁺, isotopic peaks); 305, 303 ([M-Cl]⁺, isotopic peaks). The calculated peak distributions correspond to the expected structure.**References:**

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Sample availability: sample available from the authors.

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