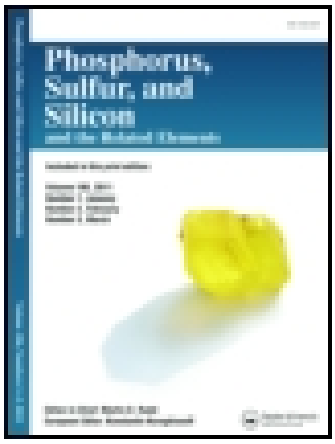

Phosphorus, Sulfur, and Silicon and the Related Elements

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Benzoylation of 2(3H)-benzothiazolones in the Presence of 8 Equivalents of Zinc Chloride in N,N-dimethylformamide

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BENZOYLATION OF 2(3H)-BENZOTHIAZOLONES IN THE PRESENCE OF 8 EQUIVALENTS OF ZINC CHLORIDE IN N,N-DIMETHYLFORMAMIDE

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The use of the mixture of aluminum chloride-N,N-dimethylformamide[1,2] ($AlCl_3$-DMF, 11:1) reagent in the Friedl-Crafts C acylation reaction of 2(3H)-benzothiazolones was previously reported.[3] These acylations reactions were found to proceed with high regioselectivity. The precise position of acylation was uniquivocally assigned by x-ray single-crystal defraction in the case of 6-benzoyl-2(3H)-benzothiazolone.[4] The assignment of the position of acylation was extended to other compounds by the use of high-field ***^{1}H-NMR****.[3]*

Keywords: 2(3H)-benzothiazolones; 6-benzoyl-2(3H)-benzothiazolones; acylation reaction; benzoyl chloride; benzoylation; zinc chloride

6-Benzoyl-2(3H)-benzothiazolones are obtained by the reaction of 2(3H)-benzothiazolones with benzoic acid in polyphosporic acid as solvent and catalyst or with benzoyl chloride in the mixture $AlCl_3$-DMF.[3]

We conducted the reaction of 2(3H)-benzothiazolones (**1a–b**) with benzoyl chloride in the presence of **8** equivalents of zinc chloride in N,N-dimethylformamide as solvent[5] at 140°C for 2 h and obtained the corresponding 6-benzoyl-2(3H)-benzothiazolones (**2a–b**) with high yields (Scheme 1).

The benzoylation of 2(3H)-benzothiazolones with the mixture of zinc chloride-DMF was realized for the first time.

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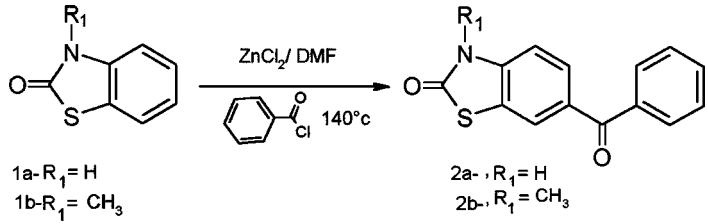

SCHEME 1

EXPERIMENTAL

Melting points were determined using an electrothermal melting point apparatus and are uncorrected. The **IR** spectra were recorded on a Shimadzu 40-spectrometer and the **^{1}H-NMR**. Spectra were recorded using a Bruker 80 MHz spectrometer. Chemical shifts are reported in ppm with tetramethylsilane as internal standard and were compatible with the compounds obtained by the $AlCl_3$-DMF method.

6-Benzoyl-2(3H)-benzothiazolone (2a)

Dimethyl formamide (0.037 mol, 2.8 ml) was slowly added to zinc chloride (0.104 mol, 14.18 g). The mixture was stirred and maintained at 75°C and 2(3H)-benzothiazolone **(1a)** (0.013 mol, 2 g) and benzoyl chloride (0.0195 mol, 2.73 ml) were added. The reaction mixture was warmed at 140°C under stirring for 2 h. After cooling the complex was decomposed by addition of ice water. The resulting residue was stirred for 1 h, filtered, washed with water, and recrystallized from ethanol to give the title compound (yield: 82%): m.p. 216–217°C; **IR** (KBr): 3220, 1680, 1630 cm^{-1}; **^{1}H-NMR**. (DMSO-D_6, δ in ppm) 7.23 (d, 1 H, J_o = 8.40 Hz); 7.60 (m, 6H); 8.00 (d, 1H, j_m = 1.7 Hz); 12.10 (s, 1H, amide).

3-Methyl-6-benzoyl-2(3H) benzothiazolone (2b)

Dimethyl formamide (0.037 mol, 2.8 ml) was slowly added to zinc chloride (0.104 mol, 14.18 g). The mixture was stirred and maintained at 75°C and 3-methyl-2(3H)-benzothiazolone **(1b)** (0.013 mol, 2.16 g) and benzoyl chloride (0.0195 mol, 2.73 ml) were added. The reaction mixture was warmed at 140°C under stirring for 2 h. After cooling the complex was decomposed by addition of ice water. The resulting residue was stirred for 1 h, filtered, washed with water, and recrystallized from ethanol to give the title compound (yield: 86%): m.p. 147–148°C; **IR** (KBr): 1670, 1630 cm^{-1}; **^{1}H-NMR**. (DMSO-D_6, δ in ppm) 3.46 (s, N—CH_3); 7.35 (d, 1H, J = 8.70 Hz); 7.50 (m, 7H).

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