

Comprehensive Organic Chemistry Experiments for the Laboratory Classroom

Edited by Carlos A M Afonso, Nuno R Candeias,
Dulce Pereira Simão, Alexandre F Trindade,
Jaime A S Coelho, Bin Tan
and Robert Franzén

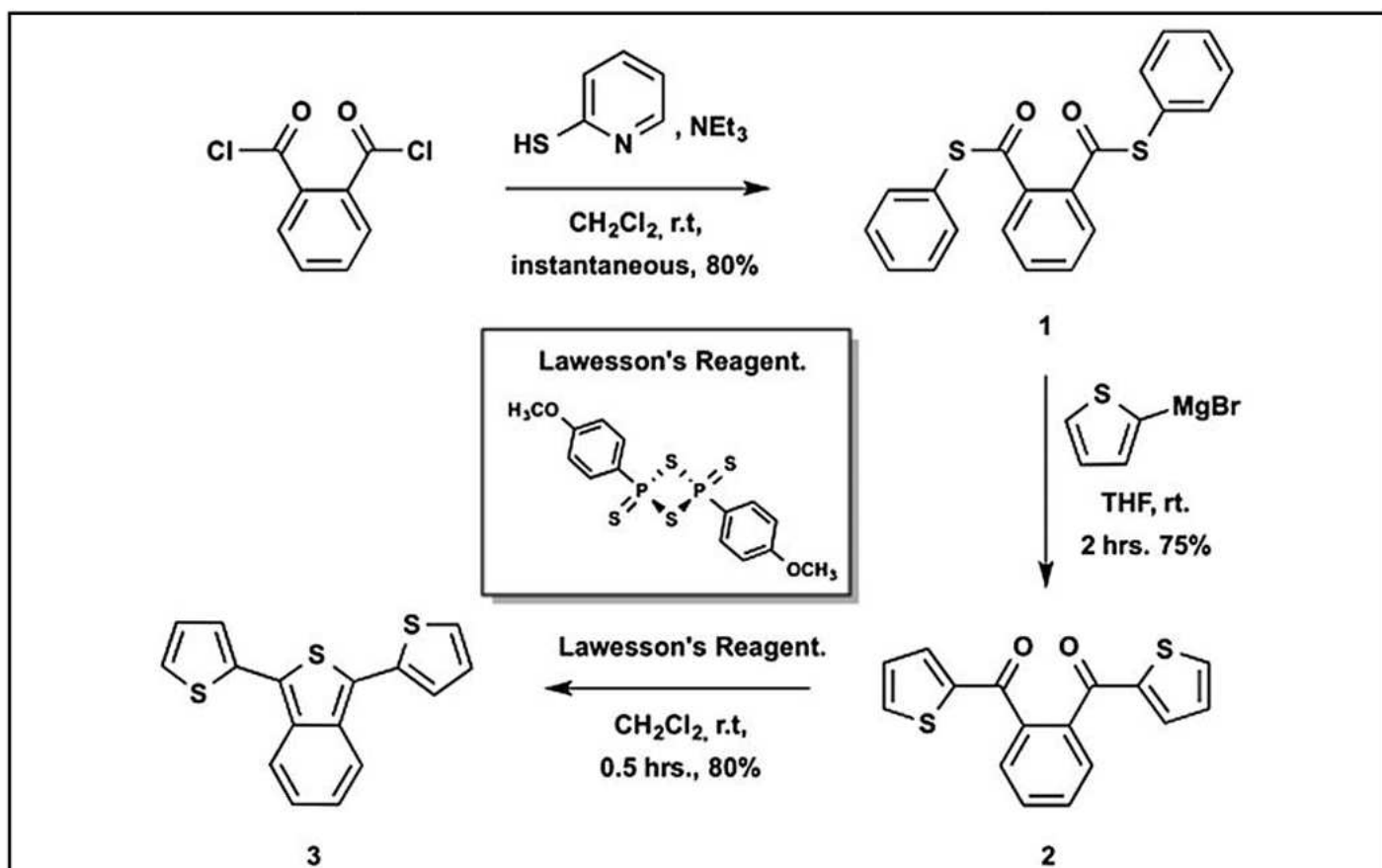


17.7. Synthesis of 1,3-Dithienylbenzo[*c*]thiophene

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Number of sessions	Hazard level	Difficulty level	Level of study
3 (4 h + 6 h + 6 h)	High	High	Intermediate

Class names Acyl chlorides, ketones, thiophenes, benzo[*c*]thiophenes, thioesters

Concepts involved This experiment provides students with an example of an *S*-heterocyclization reaction (thionation) involving Lawesson's reagent, in a challenging three-step convergent synthetic procedure. Hence, students face three different types of reaction: a thioesterification in the first step, providing a good example of acyl leaving group reactivity; a Grignard reaction in the formation of a *o*-diketone, showing a C–C bond formation reaction; and finally the *S*-heterocyclization of the previous product to afford the product 1,3-dithienylbenzo[*c*]thiophene

Chemicals needed 2-Mercaptopyridine, phthaloyl dichloride, triethylamine, 2-bromothiophene, 2,4-bis(4-methoxyphenyl)-2,4-dithioxo-1,3,2,4-dithiadiphosphetane (Lawesson's reagent), iodine, tetrahydrofuran, dichloromethane, chloroform, ethanol, *n*-hexane, hydrochloric acid, sodium hydroxide, sodium bicarbonate, anhydrous sodium sulfate

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