

# 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

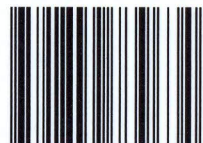


This expansive and practical textbook contains organic chemistry experiments for teaching in the laboratory at the undergraduate level covering a range of functional group transformations and key organic reactions. The editorial team have collected contributions from around the world and standardized them for publication. Each experiment will explore a modern chemistry scenario, such as: sustainable chemistry; application in the pharmaceutical industry; catalysis and material sciences, to name a few. All the experiments will be complemented with a set of questions to challenge the students and a section for the instructors, concerning the results obtained and advice on getting the best outcome from the experiment. A section covering practical aspects with tips and advice for the instructors, together with the results obtained in the laboratory by students, has been compiled for each experiment.

Targeted at professors and lecturers in chemistry, this useful text will provide up to date experiments putting the science into context for the students.

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## 7.9. An Efficient Methodology for the Synthesis of the 3-Styryl Coumarin

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Number of sessions (duration of each session)	Hazard level	Difficulty level	Level of study
3 (4 h each)	Moderate	Medium	Advanced
<b>Class names</b> $\alpha,\beta$ -Unsaturated carbonyls, aryl halides, styryls			
<b>Concepts involved</b> This experiment gives a useful method for the synthesis of 3-styrylcoumarin, using an effective $\alpha$ -unsaturated carbonyl halogenation and high yield palladium coupling reactions			
<b>Chemicals needed</b> Coumarin (2H-chromen-2-one), Oxone® (potassium peroxydisulfate), hydrobromic acid, triethylamine, dichloromethane, dimethylformamide, hexane, <i>n</i> -propanol, anhydrous sodium sulfate, calcium chloride, silica gel (230–400 mesh), potassium vinyltrifluoroborate, [1,1'-bis(diphenylphosphino)ferrocene]dichloropalladium(II)·CH <sub>2</sub> Cl <sub>2</sub> , bromobenzene or/and iodobenzene, tetrakis(triphenylphosphine)palladium(0), silver acetate			
<b>Equipment and experimental techniques involved</b> Apparatus for reaction with temperature and magnetic stirring, liquid–liquid extraction apparatus, flash column chromatography, NMR spectrometer. UV-vis spectrophotometer			
<b>Keywords</b> Heck reaction, liquid–liquid extraction, NMR, Suzuki reaction, $\alpha,\beta$ -unsaturated carbonyl halogenation, UV			

### Background

Coumarins (or benzopyranones), whether naturally occurring or synthetic, have attracted the interest of the scientific community due to their broad pharmacological activities,<sup>1,2</sup> such as antiprotozoal,<sup>3</sup> anticancer,<sup>4</sup> antibacterial,<sup>5</sup> among others. Depending on the nature and substitution pattern, coumarins show exceptional optical properties,<sup>6</sup> as they constitute the largest class of fluorescent dyes,<sup>7</sup> widely used as emission layers in organic light-emitting diodes (OLED),<sup>8</sup> optical brighteners,<sup>9</sup> nonlinear optical chromophores,<sup>10</sup> fluorescent whiteners,<sup>11</sup> fluorescent labels and probes for physiological measurement<sup>12</sup> and, more recently, in labelling<sup>13</sup> and caging.<sup>14</sup> Developments from the last decade show that the introduction of appropriate substituents into the coumarin ring contribute to structures with improved photophysical and spectroscopic properties.<sup>15</sup> Many articles dealing with their synthesis, reactivity and spectral profile

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