Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

SmI₂-mediated ketyl-arene coupling of β-arylthio ketones: a facile and diastereoselective synthesis of thiochroman derivatives

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**General Information.** THF was distilled from sodium/benzophenone. Metallic samarium and other solvents were obtained from commercial sources, and used without further purification unless otherwise stated. All the cascade reactions were carried out under a nitrogen atmosphere in oven-dried flasks. All melting points are uncorrected. The $^1$H and $^{13}$C NMR spectra were recorded in CDCl$_3$ on a 400 or 600 MHz instrument with TMS as internal standard. Recorded shifts are reported in parts per million (δ) downfield from TMS. Data are represented as follows: Chemical shift, multiplicity ($s$ = singlet, $d$ = doublet, $t$ = triplet, $q$ = quartet, $m$ = multiplet, $b$ = broad), coupling constant ($J$, Hz) and integration. TLC was carried out with 0.2 mm thick silica gel plates (GF254). Visualization was accomplished by UV light. The columns were hand packed with silica gel 60 (300–400 mesh). Unknown products were additionally confirmed by high-resolution mass spectra (HRMS) using a TOF-MS instrument with an ESI or APCI source.
NMR Spectra for new substrates 1
NMR Spectra for products 2

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NOE effect for 2S

1) Irradiation of $H^5$ and NOE with $H^1$

2) Irradiation of $H^2$ and NOE with $H^6$ or $H^7$, $CH_3$
NMR spectra for products 3

![NMR Spectra](image-url)
NMR Spectra and X-Ray Crystal Structure of product 4j
X-Ray Crystal Structure of 4j' :

The thermal ellipsoid plot for the crystal structure of 4j' (50% ellipsoid contour percent probability level):

For more details please see the CIF file. The crystal data of 4j' has already been deposited at Cambridge Crystallographic Data Center, UK, and the CCDC reference number is 1470852.