Supporting Information

A Direct Greener Route towards the Synthesis of 2-Aroyl-3,5diarylthiophenes from 1,5-Diketones

Rengasamy Chithiravel,^a Kandasamy Rajaguru,^a Shanmugam Muthusubramanian,^{*a} and

Nattamai Bhuvanesh^b

 ^a Department of Organic Chemistry, School of Chemistry, Madurai Kamaraj University, Madurai - 625 021, India
^b X-ray Diffraction Laboratory, Department of Chemistry, Texas A & M University, College Station, Texas 77842, USA

*E-mail: <u>muthumanian2001@yahoo.com</u>

Table of contents	Page No
Experimental section	S3
¹ H & ¹³ C NMR spectra of compound 3a	S4
¹ H & ¹³ C NMR spectra of compound 3b	S6
¹ H & ¹³ C NMR spectra of compound 3c	S8
¹ H & ¹³ C NMR spectra of compound 3d	S10
¹ H & ¹³ C NMR spectra of compound 3e	S12
¹ H & ¹³ C NMR spectra of compound 3m	S14
¹ H & ¹³ C NMR spectra of compound 3n	S16
¹ H & ¹³ C NMR spectra of compound 3o	S18
¹ H & ¹³ C NMR spectra of compound 4a	S20
¹ H & ¹³ C NMR spectra of compound 4b	S22
¹ H & ¹³ C NMR spectra of compound 4c	S24
¹ H & ¹³ C NMR spectra of compound 4d	S26
¹ H & ¹³ C NMR spectra of compound 4e	S28
¹ H & ¹³ C NMR spectra of compound 4f	S30
¹ H & ¹³ C NMR spectra of compound 4g	S32
¹ H & ¹³ C NMR spectra of compound 4h	S34
¹ H & ¹³ C NMR spectra of compound 4i	S36
¹ H & ¹³ C NMR spectra of compound 4j	S38
¹ H & ¹³ C NMR spectra of compound 4k	S40
¹ H & ¹³ C NMR spectra of compound 4 I	S42
¹ H & ¹³ C NMR spectra of compound 4m	S44
¹ H & ¹³ C NMR spectra of compound 4n	S46
¹ H & ¹³ C NMR spectra of compound 4o	S48

General procedure

Synthesis of 1,3,5-triarylpentan-1,5-diones (3): A mixture of chalcone **1** (1.0 equiv.), aryl methyl ketone **2** (1.0 equiv.) and powdered sodium hydroxide (2.0 equiv.) was crushed together for twenty minutes using a pestle and mortar. The mixture got solidified and the completion of the reaction was monitored by TLC chromatography using petroleum ether : ethyl acetate mixture (4:1) as eluent. After the completion of the reaction, the reaction mass was washed with water to remove the sodium hydroxide to give the corresponding 1,3,5-triarylpentan-1,5-diones. The product was further purified by recrystallization from ethanol to give colorless crystals.

Synthesis of 3,5-diaryl-2-aroylthiophene 4: A mixture of 1,3,5-triarylpentane-1,5-dione **3** (1.0 equiv.), morpholine (2.0 equiv.) and elemental sulfur (1.1 equiv.) was taken in a 10 ml quartz vial and placed in the microwave oven. The vial was sealed and subjected to microwave irradiation at 110 °C for 5 min. The reaction was monitored by TLC chromatography using petroleum ether : ethyl acetate mixture (4:1) as eluent. After the completion of the reaction, the reaction was cooled to room temperature and ice cooled water was added. The precipitate obtained was filtered, dried in vacuum and recrystallized from ethanol to afford **4**.



Figure 1. ¹H NMR Spectrum of compound **3a**



Figure 2. ¹³C Spectrum of compound **3a**



Figure 3. ¹H NMR Spectrum of compound **3b**



Figure 4. ¹³C Spectrum of compound **3b**



Figure 5. ¹H NMR Spectrum of compound **3**c



Figure 6. ¹³C Spectrum of compound **3**c



Figure 7. ¹H NMR Spectrum of compound **3d**



Figure 8. ¹³C Spectrum of compound **3d**



Figure 9. ¹H NMR Spectrum of compound **3e**



Figure 10. ¹³C Spectrum of compound **3e**



Figure 11. ¹H NMR Spectrum of compound **3m**



Figure 12. ¹³C Spectrum of compound **3m**



Figure 13. ¹H NMR Spectrum of compound **3n**



Figure 14. ¹³C Spectrum of compound **3n**



Figure 15. ¹H NMR Spectrum of compound **30**



Figure 16. ¹³C Spectrum of compound **30**



Figure 17. ¹H NMR Spectrum of compound **4a**



Figure 18. ¹³C Spectrum of compound **4a**



Figure 19. ¹H NMR Spectrum of compound **4b**



Figure 20. ¹³C Spectrum of compound **4b**



Figure 21. ¹H NMR Spectrum of compound **4**c



Figure 22. ¹³C Spectrum of compound **4c**



Figure 23. ¹H NMR Spectrum of compound **4d**



Figure 24. ¹³C Spectrum of compound **4d**



Figure 25. ¹H NMR Spectrum of compound **4e**



Figure 26. ¹³C Spectrum of compound **4e**



Figure 27. ¹H NMR Spectrum of compound **4f**



Figure 28. ¹³C Spectrum of compound **4f**



Figure 29. ¹H NMR Spectrum of compound **4g**



Figure 30. ¹³C Spectrum of compound **4g**



Figure 31. ¹H NMR Spectrum of compound **4h**



Figure 32. ¹³C Spectrum of compound **4h**



Figure 33. ¹H NMR Spectrum of compound **4i**



Figure 34. ¹³C Spectrum of compound **4i**



Figure 35. ¹H NMR Spectrum of compound **4**j



Figure 36. ¹³C Spectrum of compound **4**j



Figure 37. ¹H NMR Spectrum of compound **4**k



Figure 38. ¹³C Spectrum of compound **4**k



Figure 39. ¹H NMR Spectrum of compound **4**I



Figure 40. ¹³C Spectrum of compound **4**l



Figure 41. ¹H NMR Spectrum of compound **4m**



Figure 42. ¹³C Spectrum of compound **4m**



Figure 43. ¹H NMR Spectrum of compound **4n**



Figure 44. ¹³C Spectrum of compound **4n**



Figure 45. ¹H NMR Spectrum of compound **40**



Figure 46. ¹³C Spectrum of compound **40**