A solvent- and catalyst-free domino reaction for the efficient synthesis of 3-aryltriazolidine-2-thiones under microwave irradiation

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Supporting Information

Structure determination using NMR spectroscopic data of 3e

The structure of 4-hydroxy-3-(p-tolyl)thiazolidine-2-thione 3e was deduced from one- and two-dimensional NMR spectroscopic data. The structural elucidation using NMR spectroscopy is discussed below.

In the $^1$H NMR spectrum of 3e, the H-5 hydrogens appear as a broad singlet 5.78 ppm and it shows a C-H COSY correlation with the carbon signal at 92.4 ppm. Further, the H-5 have a H-H COCY correlation with a hydrogen at 3.86 ppm ($J = 12.1, 6.1$ Hz) assignable to H-4a. Moreover, the H-5 protons show a HMB correlation with C-2 at 196.8 ppm. The diastereotopic protons, H-4 appear as doublets of doublets at 3.86 ppm ($J = 12.1, 6.1$ Hz) and 3.29 ($J = 12.3, 1.5$ Hz) which show HMBCs with C-2 and C-4 at 196.8 and 92.4 ppm respectively. The H-H COSY correlation reveals that one of the diastereotopic H-4a protons couples with H-5 proton and the protons, H-
4a and H-4b couple with each other. The hydrogens, H-4a and H-4b show a C-H COSY correlation with the carbon signal at 36.7 ppm.

The aromatic protons appear as a multiplet at 7.72-7.41 ppm. The methyl hydrogens appear as a singlet at 2.39 ppm which show HMBCs with C-3’ and C-4’ at 127.8 and 136.7 ppm respectively. The hemi aminalic OH appears as a broad singlet at 3.63 ppm. The >C=S carbon appears at 196.8 ppm, which is a characteristic carbon signal for the 4-hydroxy-3-aryltiazolidine-2-thione system. Finally the structure of the product was also confirmed by ESI mass analysis.
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Fig. 1 $^1$H NMR Spectrum 3a(DMSO-$d_6$)

Fig. 2 $^{13}$C NMR Spectrum of 3a(DMSO-$d_6$)
Fig. 3 ESI mass spectrum of 3a

Molecular weight: 229.29
Exact Mass: 229.00

Fig. 4 $^1$H NMR Spectrum 3b (DMSO-d$_6$)
Fig. 5 $^{13}$C NMR Spectrum of 3b (DMSO-d$_6$)

Molecular weight: 245.74
Exact Mass: 244.97

Fig. 6 ESI mass spectrum of 3b
Fig. 7. $^1$H NMR Spectrum of 3c (CDCl$_3$)

Fig. 8. $^{13}$C NMR Spectrum of 3c (CDCl$_3$ + DMSO-d$_6$)
Fig. 9 ESI mass spectrum of 3c

Molecular weight: 290.19
Exact Mass: 288.92

Fig. 10 $^1$H NMR Spectrum 3d(DMSO-d$_6$)
Fig. 11. $^{13}$C NMR Spectrum of 3d (DMSO-d$_6$)

Molecular weight: 211.30
Exact Mass: 211.01

Fig. 12. ESI mass spectrum of 3d
Fig. 1. H NMR Spectrum of 3e(CDCl₃)

Fig. 13. H NMR Spectrum of 3e(CDCl₃)

Fig. 14. C NMR Spectrum of 3e(CDCl₃)
Fig. 15 DEPT Spectrum of 3c (CDCl₃)

Fig. 16 H, H-COSY Spectrum of 3c (CDCl₃)
Fig. 17 HMBC Spectrum of 3c (CDCl<sub>3</sub>)

Molecular weight: 225.32

Exact Mass: 225.03

Fig. 18 ESI mass spectrum of 3e
Fig. 19 $^1$H NMR Spectrum 3f (CDCl$_3$ + DMSO-$d_6$)

Fig. 20 $^{13}$C NMR Spectrum of 3f (CDCl$_3$ + DMSO-$d_6$)
Fig. 21 ESI mass spectrum of 3f

Molecular weight: 239.35
Exact Mass: 239.04

Fig. 22 $^1$H NMR Spectrum 3g (DMSO-d$_6$)
Fig. 23 $^{13}$C NMR Spectrum of 3g (DMSO-d$_6$)

Molecular weight: 253.38
Exact Mass: 253.06

Fig. 24 ESI mass spectrum of 3g
Fig. 25 $^1$H NMR Spectrum of 3h (CDCl$_3$ + DMSO-d$_6$)

Fig. 26 $^{13}$C NMR Spectrum of 3h (DMSO-d$_6$)
Fig. 27 ESI mass spectrum of 3h

Molecular weight: 241.32
Exact Mass: 241.02

Fig. 28 $^1$H NMR Spectrum 3i (DMSO-d$_6$)
Fig. 29 $^{13}$C NMR Spectrum of 3i (DMSO-$d_6$)

![NMR Spectrum Image]

Fig. 30 ESI mass spectrum of 3i

![Mass Spectrum Image]

Molecular weight: 229.29
Exact Mass: 229.00
Fig. 31 $^1$H NMR Spectrum 3j (CDCl$_3$ + DMSO-d$_6$)

Fig. 32 $^{13}$C NMR Spectrum of 3j (DMSO-d$_6$)
Fig. 33 ESI mass spectrum of 3j

Molecular weight: 290.19

Exact Mass: 288.92

Fig. 34 $^1$H NMR Spectrum 3k (CDCl$_3$)
Fig. 35 $^{13}$C NMR Spectrum of 3k (CDCl$_3$)

Molecular weight: 225.32
Exact Mass: 225.03
Fig. 37 $^1$H NMR Spectrum of 31 (CDCl$_3$ + DMSO-d$_6$)

Fig. 38 $^{13}$C NMR Spectrum of 31 (CDCl$_3$ + DMSO-d$_6$)
Molecular weight: 241.32
Exact Mass: 241.02

**Fig. 39** ESI mass spectrum of 3l

**Fig. 40** $^1$H NMR Spectrum 3m (CDCl$_3$ + DMSO-d$_6$)
Fig. 41 $^{13}$C NMR Spectrum of 3m(CDCl$_3$ + DMSO-d$_6$)

Fig. 42 $^1$H NMR Spectrum 5a(CDCl$_3$)
Fig. 43 $^{13}$C NMR Spectrum of 5a (CDCl$_3$)

Fig. 44 $^1$H NMR Spectrum 5b (CDCl$_3$)
**Fig. 45** C NMR Spectrum of 5b (CDCl$_3$)

**Fig. 46** H NMR Spectrum 5c (CDCl$_3$)
**Fig. 47** $^{13}$C NMR Spectrum of $5c$ (CDCl$_3$)

Molecular weight: 207.25

Exact Mass: 207.04

**Fig. 48** ESI mass spectrum of $5c$

Molecular weight: 207.25

Exact Mass: 207.04
**Fig. 49** $^1$H NMR Spectrum of 6a (CDCl$_3$)

**Fig. 50** $^{13}$C NMR Spectrum of 6a (CDCl$_3$)
Fig. 51: ESI mass spectrum of 6a

Molecular weight: 193.28
Exact Mass: 193.00

Fig. 52: 'H NMR Spectrum 6b (CDCl₃)
Fig. 53: $^{13}$C NMR Spectrum of 6b (CDCl$_3$)

Molecular weight: 211.27

Exact Mass: 210.99

Fig. 54: ESI mass spectrum of 6b
Fig. 55 $^1$H NMR Spectrum 6c(CDCl$_3$)

Fig. 56 $^{13}$C NMR Spectrum of 6c(CDCl$_3$)
Fig. 57 ESI mass spectrum of 6c

Fig. 58 $^1$H NMR Spectrum 6d (CDCl$_3$)
Fig. 59 $^{13}$C NMR Spectrum of 6d (CDCl$_3$)

Molecular weight: 211.27

Exact Mass: 210.99

Fig. 60 ESI mass spectrum of 6d
Fig. 61 $^1$H NMR Spectrum of 6e(CDCl$_3$)

Fig. 62 $^{13}$C NMR Spectrum of 6e(CDCl$_3$)