Supporting Information

Umpolung Cyclization Reaction of N-Cinnamoylthioureas
in the Presence of DBU

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Contents

General -------------------------------------------------- S3
Single crystal X-Ray single crystallographic analysis of 2h ------------------ S4
Single crystal X-Ray single crystallographic analysis of 2i ------------------ S5
Single crystal X-Ray single crystallographic analysis of 2j ------------------ S6
Single crystal X-Ray single crystallographic analysis of 3i ------------------ S7
Single crystal X-Ray single crystallographic analysis of 3j ------------------ S8
Single crystal X-Ray single crystallographic analysis of 4j ------------------ S9
Single crystal X-Ray single crystallographic analysis of 5c ------------------ S10
1H and 13C NMR spectral chart of 1a-1j ----------------------------------- S11
1H and 13C NMR spectral chart of 2a-2j ----------------------------------- S21
1H and 13C NMR spectral chart of 3-5 ----------------------------------- S31
General.

NMR spectra were recorded in CDCl$_3$ solutions on Bruker 300 and 400 spectrometers for $^1$H- and $^{13}$C-NMR. Chemical shifts are reported in parts per million (ppm) relative to TMS as an internal standard. IR spectra were recorded on a JASCO FT/IR-230 spectrometer. High-resolution mass spectra (HRMS) were performed on an Orbitrap ThermoFisher Exactive ion trap mass spectrometer. X-ray single crystallographic analysis was conducted using a SMART APEX II (Bruker AXS) and APEX II ULTRA (Bruker AXS). Commercially available reagents and solvents were used without further purification.
Single crystal X-Ray crystallographic analysis of 2h (CCDC 1859352)

Colorless prism (0.20 x 0.10 x 0.05 mm$^3$), monoclinic space group $P2_1/c$, $a = 12.8458(5)$ Å, $b = 5.3427(2)$ Å, $c = 24.6144(10)$ Å, $\beta = 101.767(3)$ °, $V = 1653.82(11)$ Å$^3$, $Z = 4$, $\lambda$ (CuK$\alpha$) = 1.54178 Å, $\rho = 1.303$ g/cm$^3$, $\mu$ (CuK$\alpha$) = 1.776 mm$^{-1}$, 10898 reflections measured ($T = 173$ K, $3.514^{\circ} < \theta < 68.341^{\circ}$), nb of independent data collected: 3012, nb of independent data used for refinement: 2264 in the final least-squares refinement cycles on $F^2$, the model converged at $R_1 = 0.0495$, $wR_2 = 0.1324$ [$I > 2s(I)$], $R_1 = 0.0696$, $wR_2 = 0.1416$ (all data), and GOF = 1.007, H-atom parameters constrained.

Figure S1. Perspective view of 2h. Ellipsoids were drawn in 50% probability. Tortional angles: S-C1-N1-C2: 4.09 °, S-C1-N2-C2: 11.13 °, C1-N2-C1-O1: 5.29 °, C2-N1-C1-N1: 11.5 °.
Single crystal X-Ray structure analysis of 2i (CCDC 1859353)

Colorless prism (0.20 x 0.05 x 0.05 mm³), monoclinic space group P2₁/c, a = 13.0104(7) Å, b = 5.4301(3) Å, c = 24.2997(15) Å, β = 101.772(4) °, V = 1680.61(17) Å³, Z = 4, λ (CuKα) = 1.54178 Å, ρ = 1.282 g/cm³, μ (CuKα) = 1.747 mm⁻¹, 11611 reflections measured (T = 173 K, 3.470 ° < θ < 68.239 °), nb of independent data collected: 3051, nb of independent data used for refinement: 2600 in the final least-squares refinement cycles on F², the model converged at R₁ = 0.0468, wR₂ = 0.1307 [I > 2s(I)], R₁ = 0.0545, wR₂ = 0.1367 (all data), and GOF = 1.029, H-atom parameters constrained.

Figure S2. Perspective view of 2i. Ellipsoids were drawn in 50% probability.
Single crystal X-Ray structure analysis of 2j (CCDC 1859354)
Colorless prism (0.50 x 0.20 x 0.10 mm³), monoclinic space group P2₁/c, \( a = 13.613(2) \) Å, \( b = 5.3251(9) \) Å, \( c = 26.026(4) \) Å, \( \beta = 95.958(3) \) °, \( V = 1876.4(6) \) Å³, \( Z = 4 \), \( \lambda (\text{MoK} \alpha) = 0.71073 \) Å, \( \rho = 1.318 \) g/cm³, \( \mu (\text{MoK} \alpha) = 0.188 \) mm⁻¹, 10165 reflections measured (\( T = 173 \) K, \( 1.504 \) ° < \( \theta < 27.502 \) °), nb of independent data collected: 4244, nb of independent data used for refinement: 2649 in the final least-squares refinement cycles on \( F^2 \), the model converged at \( R_1 = 0.0521 \), \( wR_2 = 0.1235 \) [\( I > 2s(I) \)], \( R_1 = 0.0974 \), \( wR_2 = 0.1579 \) (all data), and GOF = 0.966, H-atom parameters constrained.

Figure S3. Perspective view of 2j. Ellipsoids were drawn in 50% probability.
Single crystal X-Ray structure analysis of 3i (CCDC 1859407)

Colorless prism (0.30 x 0.20 x 0.10 mm³), monoclinic space group P2₁/c, \(a = 12.4130(15)\) Å, \(b = 17.816(2)\) Å, \(c = 7.9085(10)\) Å, \(\beta = 106.841(2)\) °, \(V = 1674.0(4)\) Å³, \(Z = 4\), \(\lambda (\text{MoK} \alpha) = 0.71073\) Å, \(\rho = 1.287\) g/cm³, \(\mu (\text{MoK} \alpha) = 0.199\) mm⁻¹, 9554 reflections measured (T = 173 K, 2.5558 ° < \(\theta\) < 27.5219 °), nb of independent data collected: 3838, nb of independent data used for refinement: 2379 in the final least-squares refinement cycles on \(F^2\), the model converged at \(R_1 = 0.0512, \ wR_2 = 0.1170\ [l > 2s(l)], \ R_1 = 0.0924, \ wR_2 = 0.1447\) (all data), and GOF = 0.929, H-atom parameters constrained.

Figure S4. Perspective view of 3i. Ellipsoids were drawn in 50% probability.
**Single crystal X-Ray structure analysis of 3j (CCDC 1859355)**

Colorless prism (0.50 x 0.50 x 0.10 mm³), triclinic space group $P-1$, $a = 8.887(2)$ Å, $b = 9.348(2)$ Å, $c = 12.182(3)$ Å, $\alpha = 80.025(3)\, ^\circ$, $\beta = 72.125(3)\, ^\circ$, $\gamma = 88.273(3)\, ^\circ$, $V = 948.3(4)$ Å³, $Z = 2$, $\lambda$(MoKα) = 0.71073 Å, $\rho = 1.304$ g/cm³, $\mu$(MoKα) = 0.186 mm⁻¹, 5472 reflections measured ($T = 173$ K, $2.2128\, ^\circ < \theta < 27.5491\, ^\circ$), nb of independent data collected: 4141, nb of independent data used for refinement: 3585 in the final least-squares refinement cycles on $F^2$, the model converged at $R_1 = 0.0365$ $wR_2 = 0.0914$ [$I > 2s(I)$], $R_1 = 0.0429$, $wR_2 = 0.0954$ (all data), and GOF = 1.065, H-atom parameters constrained.

**Figure S5.** Perspective view of 3j. Ellipsoids were drawn in 50% probability.
**Single crystal X-Ray structure analysis of 4g (CCDC 1859357)**

Colorless prism (0.50 x 0.50 x 0.10 mm³), monoclinic space group C2/c, \(a = 34.665(4)\) Å, \(b = 6.9265(9)\) Å, \(c = 14.4146(18)\) Å, \(\beta = 113.1370(10)\) °, \(V = 3182.7(7)\) Å³, \(Z = 8\), \(\lambda (\text{MoK}) = 0.71073\) Å, \(\rho = 1.296\) g/cm³, \(\mu (\text{MoK}) = 0.207\) mm⁻¹, 17622 reflections measured (\(T = 173\) K, \(2.5558° < \theta < 27.5219°\)), nb of independent data collected: 3651, nb of independent data used for refinement: 3146 in the final least-squares refinement cycles on \(F^2\), the model converged at \(R_1 = 0.0310, wR_2 = 0.0789 [I > 2s(I)]\), \(R_1 = 0.0380, wR_2 = 0.0871\) (all data), and \(\text{GOF} = 1.042\), H-atom parameters constrained.

![Figure S6. Perspective view of 4g. Ellipsoids were drawn in 50% probability.](image)
Single crystal X-Ray structure analysis of 5c (CCDC 1859359)
Colorless prism (0.40 x 0.30 x 0.10 mm³), triclinic space group P-1, a = 8.7745(14) Å, b = 9.4352(16) Å, c = 20.409(3) Å, α = 93.504(2) °, β = 97.479(2) °, γ = 90.918(2) °, V = 1671.6(5) Å³, Z = 4, λ (MoKα) = 0.71073 Å, ρ = 1.218 g/cm³, μ (MoKα) = 0.200 mm⁻¹, 9602 reflections measured (T = 173 K, 2.1632 ° < θ < 23.5028 °), nb of independent data collected: 7319, nb of independent data used for refinement: 4764 in the final least-squares refinement cycles on F², the model converged at R₁ = 0.0538, wR₂ = 0.1302 [I > 2s(I)], R₁ = 0.0851, wR₂ = 0.1602 (all data), and GOF = 0.999, H-atom parameters constrained.

Figure S7. Perspective view of 5c. Ellipsoids were drawn in 50% probability.
### Table

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### Figure S8

1H and 13C NMR spectra of 1a
Figure S9. $^1$H and $^{13}$C NMR spectra of 1b
Figure S10. $^1$H and $^{13}$C NMR spectra of 1c

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X_Points = 32768
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Scans = 128
Temp_Get = 300[K]
Filter_Factor = 32
**Figure S11.** $^1$H and $^{13}$C NMR spectra of 1d
Figure S12. $^1$H and $^{13}$C NMR spectra of 1e
Figure S13. $^1$H and $^{13}$C NMR spectra of $^{1}$f
Figure S14. $^1$H and $^{13}$C NMR spectra of 1g

--- PROCESSING PARAMETERS ---

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Figure S15. $^1$H and $^{13}$C NMR spectra of 1h
Figure S16. $^1$H and $^{13}$C NMR spectra of 1i.
Figure S17. $^1$H and $^{13}$C NMR spectra of 1j
Figure S18. $^1$H and $^{13}$C NMR spectra of 2a
Figure S19. $^1$H and $^{13}$C NMR spectra of 2b
Figure S20. $^1$H and $^{13}$C NMR spectra of 2c.
Figure S21. $^1$H and $^{13}$C NMR spectra of 2d.
Figure S22. $^1$H and $^{13}$C NMR spectra of 2e.
Figure S23. \(^1\)H and \(^{13}\)C NMR spectra of 2f.
Figure S24. $^1$H and $^{13}$C NMR spectra of 2g

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Figure S25. $^1$H and $^{13}$C NMR spectra of $2h$
Figure S26. $^1$H and $^{13}$C NMR spectra of 2i
Figure S27. $^1$H and $^{13}$C NMR spectra of 2j.

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X: parts per Million: $^1$H

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X: parts per Million: $^{13}$C
Figure S28. $^1$H and $^{13}$C NMR spectra of 3a
Figure S29. $^1$H and $^{13}$C NMR spectra of 3b
Figure S30. \(^1\)H and \(^{13}\)C NMR spectra of 3c.
Figure S31. $^1$H and $^{13}$C NMR spectra of 3d.

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Figure S32. ¹H and ¹³C NMR spectra of 3e

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Figure S34. $^1$H and $^{13}$C NMR spectra of 3g

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Figure S35. $^1$H and $^{13}$C NMR spectra of 3h
Figure S36. $^1$H and $^{13}$C NMR spectra of 3i.
Figure S37. $^1$H and $^{13}$C NMR spectra of 3j
Figure S38. $^1$H and $^{13}$C NMR spectra of 4d
Figure S39. $^1$H and $^{13}$C NMR spectra of 4f
Figure S40. $^1$H and $^{13}$C NMR spectra of 4g
Figure S4. $^1$H and $^{13}$C NMR spectra of 4h
Figure S42. $^1$H and $^{13}$C NMR spectra of 4i

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*X_Offset = 1.50115 kHz*
*X_Points = 16384*
*X_Sweep = 3.61271676 kHz*
*Scans = 8*
*Temp_Get = 300 K*
*Filter_Factor = 32*

*Filename = 23744rei-C_1-7.jdf*
*Author = root*
*Experiment = zgpg30*
*Sample_Id = Parameter file, TOPSPINVer*
*Solvent = CHLOROFORM-D*
*Creation_Time = 30-MAR-2017 16:08:33*
*Revision_Time = 30-MAR-2017 16:09:27*
*Current_Time = 30-MAR-2017 16:09:32*
*Comment = Parameter file, TOPSPINVer*
*Data_Format = 1D COMPLEX*
*Dim_Size = 32768*
*Dim_Title = 13C*
*Dim_Units = [ppm]*
*Dimensions = X*
*Spectrometer = BRUKER_DMX_NMR*
*Field_Strength = 7.05156322 T (300 MHz)*
*X_Domain = 13C*
*X_Freq = 75.50116822 MHz*
*X_Freq_Flip = TRUE*
*X_Offset = 8.30421504 kHz*
*X_Points = 32768*
*X_Sweep = 18.11594203 kHz*
*Scans = 128*
*Temp_Get = 300 K*
*Filter_Factor = 8*
Figure S43. $^1$H and $^{13}$C NMR spectra of 4j.

--- PROCESSING PARAMETERS ---

$^1$H NMR:
- dc_balance(0, FALSE)
- sexp(2.0 Hz, 0.0 s)
- trapezoid(0%, 0%, 80%, 100%)
- zerofill(1)
- fft(1, TRUE, TRUE)

$^{13}$C NMR:
- dc_balance(0, FALSE)
- sexp(0.2 Hz, 0.0 s)
- trapezoid(0%, 0%, 80%, 100%)
- zerofill(1)
- fft(1, TRUE, TRUE)

Filename = 4-0589rei-H_1-3.jdf
Author = nmr
Experiment = zg30
Sample_Id = green
Solvent = CHLOROFORM-D
Creation_Time = 14-SEP-2018 20:37:18
Revision_Time = 14-SEP-2018 20:42:07
Comment = green
Data_Format = 1D COMPLEX
Dim_Size = 32768
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39910925 [T] (400 [MHz])
X_Domain = 1H
X_Freq = 400.1820009 [MHz]
X_Freq_Flip = TRUE
X_Offset = 2.0009 [kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 4.82625483 [kHz]
Scans = 8
Temp_Get = 296.76 [K]
Filter_Factor = 4144

Filename = 24203rei-C_1-5.jdf
Author = root
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPINVer
Solvent = CHLOROFORM-D
Creation_Time = 30-MAR-2017 16:52:27
Revision_Time = 30-MAR-2017 16:54:49
Current_Time = 30-MAR-2017 16:54:53
Comment = Parameter file, TOPSPINVer
Data_Format = 1D COMPLEX
Dim_Size = 32768
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 7.05156322 [T] (300 [MHz])
X_Domain = 13C
X_Freq = 75.50116822 [MHz]
X_Freq_Flip = TRUE
X_Offset = 8.30421504 [kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 18.11594203 [kHz]
Scans = 128
Temp_Get = 300 [K]
Filter_Factor = 8
Figure S44. $^1$H and $^{13}$C NMR spectra of 5b

--- PROCESSING PARAMETERS ---
- dc_balance[ 0, FALSE ]
- expm[ 2.0[Hz], 0.0[s] ]
- trapezoid[ 0.15, 0.15, 80%, 100% ]
- zerofill[ 1 ]
- ppm

--- PROCESSING PARAMETERS ---
- dc_balance[ 0, FALSE ]
- expm[ 2.0[Hz], 0.0[s] ]
- trapezoid[ 0.15, 0.15, 80%, 100% ]
- zerofill[ 1 ]
- ppm
Figure S45. $^1$H and $^{13}$C NMR spectra of 5c