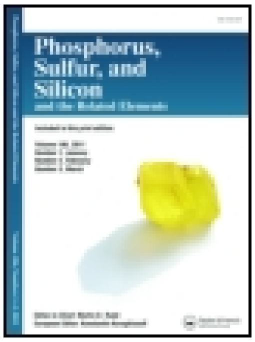

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Simple synthesis of (*E*) and (*Z*)-2-(arylmethylidene)-*N*-phenyl succinimides via Wittig olefination by using *PS-TPP* resin

Mohammad Bayat, Seyedeh Razieh Hosseini, and Elnaz Asmari

Department of Chemistry, Imam Khomeini International University, Qazvin, Iran

ABSTRACT

PS-Triphenylphosphine resin has been used in the *Wittig* reaction to prepare (*E*)- and (*Z*)-2-(arylmethylidene)-*N*-phenylsuccinimides. *PS-TP* phosphoranylidene-*N*-phenylsuccinicimide has been obtained from *PS-TPP* and *N*-phenylmaleimide. The *Wittig* reaction of stabilized *PS-TPP* ylide with an aromatic aldehyde produced the (*E*)-alkene as the major product, isolated in high yields by simple filtration of *PS*-resin, from the reaction mixture. The better stabilized *PS-TPP* succinic anhydride does not react in the same conditions.

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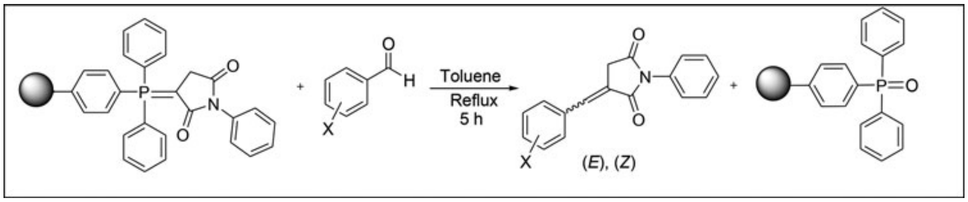
Phosphorus ylides; *PS-TPP*; *Wittig* olefination; *TPPSA*

GRAPHICAL ABSTRACT



Introduction

Phosphorus ylides are useful intermediates, which have been used in many reactions and are involved the synthesis of organic compounds.[1–7] Phosphorus ylides using triphenylphosphine (*TPP*) and maleic anhydride or maleimides were first reported by Hudson[8] and Hedaya,[9] but virtually no additional chemistry was reported until the early 2000s.[10]

Medicinal chemists in the pharmaceutical industry now routinely utilize solid-phase organic synthesis to prepare libraries of small organic molecules for screening.[11–14] Polymer-supported reagents (*PS*-reagents) have been in use since the 1960s, and have been the subject of several review articles.[15,16] Using *PS*-reagents is attractive and suitable for parallel synthesis because the reactions are often very clean and high yielding, and the workup involves simple filtration and evaporation of the solvent.

For some years it has been possible to perform some *Wittig* reactions with polymer-supported triphenylphosphine (*PS-TPP*) instead of triphenylphosphine (*TPP*). The treatment of *PS-TPP* with maleic anhydride or *N*-aryl maleimide afford cross-linked phosphorous ylides. These ylides undergo different chemical transformations. The application of *PS*-phosphonium ylides in the *Wittig* reaction enables the preparation of solutions of practically pure olefins free from phosphine oxide or phosphinium salt impurities, which remain connected to the polymer. These ylides react with aromatic aldehydes with the formation of olefins with high stereoselectivity.

We wish to report a simple synthetic route to stabilized *PS*-phosphorus ylides **2a** and **2b** using *PS-TPP* and maleic anhydride or *N*-aryl maleimide (Scheme 1), which undergo a smooth reaction with readily available aromatic aldehydes in boiling toluene to produce (*E*)- and (*Z*)-2-(arylmethylidene)-*N*-phenylsuccinimides (Scheme 2).

The methodology of *TPP* and *N*-aryl maleimide have been well described in the recent literature, but a limited number of papers have reported on Triphenylphosphoranylidenesuccinic anhydride (*TPPSA*) *Wittig* olefination reactions,[17–19] and to the best of our knowledge, there are no reports on the synthesis of these compounds by *PS-TPP via Wittig* reaction. Some (*E*)-2-(substituted aryl or alkyl methylidene)-*N*-alkyl or substituted aryl succinimide compounds were reported[20–27] and **4a-4b** are known[21] and other products presented in this work are novel.

Results and discussion

The reaction of *PS-TPPSI* **2a** and aromatic aldehyde **3** proceeds smoothly in toluene for 5 h without using any catalyst at reflux conditions, to produce a mixture of geometric isomers (*E*)-**4** and (*Z*)-**4** in excellent yield by a *Wittig* reaction (Scheme 2).

The structures of the products **4a-4k** were deduced from their IR, ^{1}H- and ^{13}C-NMR spectra. The ^{1}H- and ^{13}C-NMR spectra of the crude products indicated the formation of the title products. No product other than **4** could not be detected by ^{1}H-NMR spectroscopy.

CONTACT Mohammad Bayat bayat_mo@yahoo.com Department of Chemistry, Imam Khomeini International University, Qazvin, Iran.

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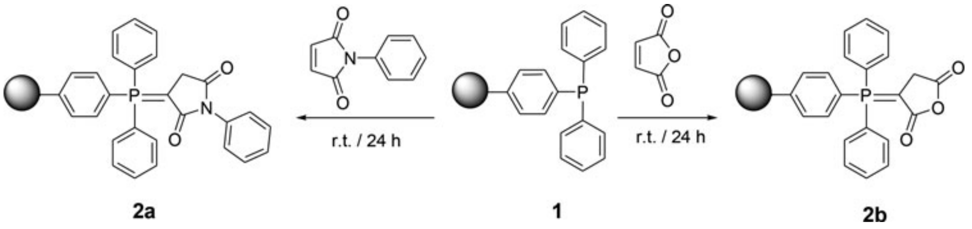
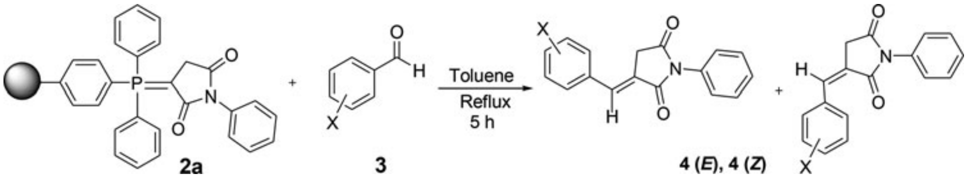


Scheme 1. Synthesis of PS-phosphorus ylides **2a** and **2b**.



Scheme 2. Wittig reaction using PS-phosphorus ylide **2a**.

In all of the reactions with aldehydes, the products are obtained as a mixture of (*E*)- and (*Z*)-isomers. As is usual for stabilized ylides, the (*E*)-isomer is dominant.[28] The isomeric ratio was calculated from the integral values for the methylene or vinylic H-atoms in the ^{1}H-NMR spectra of the mixture of (*E*)-**4** and (*Z*)-**4**. The ^{1}H-NMR spectra of the mixture of the isomers of (*E*)-**4c** revealed that they are formed in a 92: 8 ratio. The ^{1}H-NMR spectra of **4c** display signals at about 3.81 ppm for the two methylene H-atoms (*doublet*, $^4J_{HH} = 2.4$ Hz, CH_2) and 7.78 ppm for the vinylic H-atom ($^4J_{HH} = 2.4$ Hz,=CH), and signals at 3.71 ppm (*doublet*, $^4J_{HH} = 2.1$ Hz, CH_2) and 7.38 ppm ($^4J_{HH} = 2.1$ Hz,=CH) for (*Z*)-**4c** in agreement with the suggested structure. The ^{1}H-decoupled ^{13}C-NMR spectra of (*E*)-**4c** showed 15 distinct signals in agreement with the proposed structure. The characteristic signal of the CH_2 group was observed at 34.02 ppm. The ^{1}H- and ^{13}C-NMR spectra of compounds **4d**-**4k** were similar to that of **4c**, except for the signals of the substituted phenyl rings, which exhibited characteristic resonances with appropriate chemical shifts (see also *Exper. part*).

Polymer-supported triphenylphosphoranylidenesuccinic anhydride (*PS-TPPSA*) (**2b**) or triphenylphosphoranylidenesuccinic anhydride (*TPPSA*) **2c** both did not react in the Wittig olefination reaction successfully under the conditions mentioned above (Scheme 3). When **2b** and aromatic aldehydes are mixed, no changes were detected. Also, heating the mixture at 110 °C for up to 5 h does not yield an olefination product. Instead, triphenylphosphine is partly eliminated from the phosphorus ylide. These phosphorus ylides contain an anhydride group that can stabilize the negative charge from the carbanion (due to π-conjugation of the C=O group with neighboring the P=C moiety).

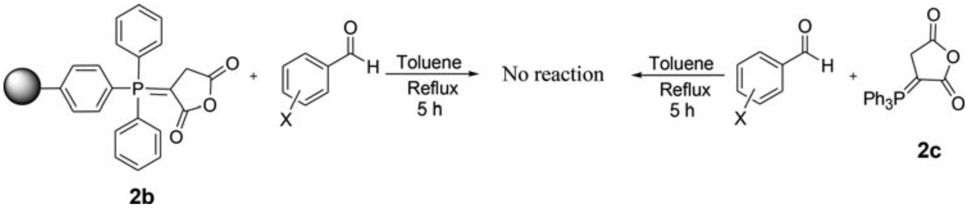
As shown in Table 1, aromatic aldehydes with electron-withdrawing or donating groups gave excellent yields of the corresponding product. The ratio of the major (*E*)- and minor (*Z*)-isomers of compounds **4a–4k** are shown in Table 1.

In conclusion, we have reported a simple method for the synthesis of (*E*)-2-(aryl methylidene)-*N*-phenyl succinimides from readily available aromatic aldehydes and *PS-TPPSI*. The present procedure has the advantages that, excess *PS-TPPSI* can be used to drive reactions to completion, impurities and excess reagents can be removed by simple washing of the solid-phase. The reactions are simple, high yields and moreover products can be obtained pure and free of triphenylphosphin oxide (*TPPO*).

Experimental

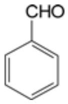
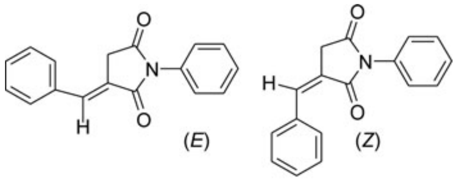
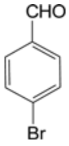
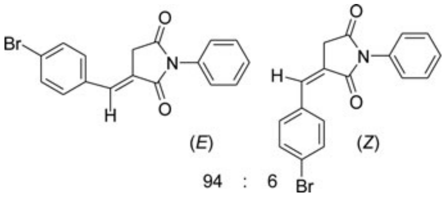
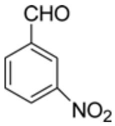
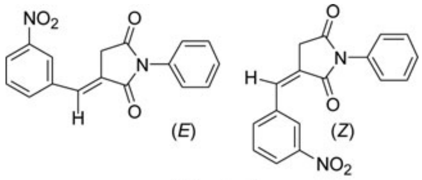
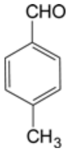
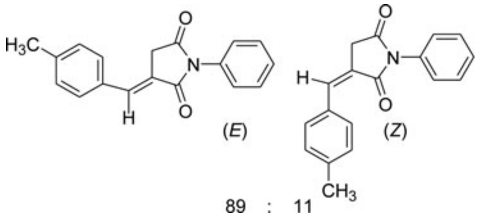
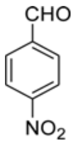
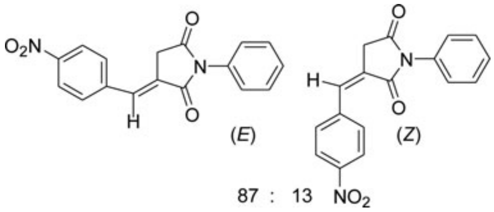
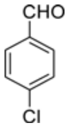
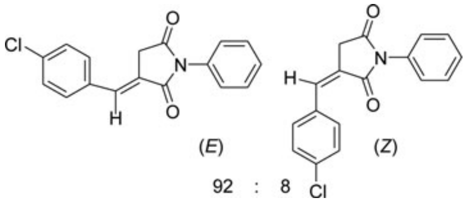
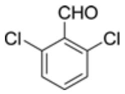
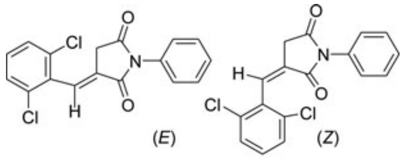
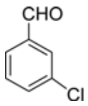
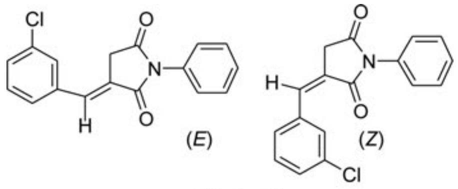
General. Triphenylphosphine resin (1% Cross-linked poly(styrene-codivinylbenzene) and contain 1–1.5 mmol TPP per gram of resin) was obtained from Akcros Chemical Co. Aromatic aldehydes and solvents used in this work were obtained from Fluka (Buchs, Switzerland) and used without further purification. NMR spectra: Bruker-DRX-300 Avance instrument; δ in ppm rel. to $MeSi_4$ as internal standard, J in Hz. M.p.: Gallenkamp electrothermal-9100 apparatus; not corrected. Mass spectra were recorded with an Agilent 5975C VL MSD with Triple-Axis Detector operating at an ionization potential of 70 eV. IR spectra (KBr): Bruker-Tensor-27 spectrometer; ν in cm^{-1}. The Supplemental Materials contains sample ^{1}H, ^{13}C NMR, IR and mass spectra for products 4 (Figures S1–S22).

Typical procedure for the preparation of PS-Triphenylphosphoranylidene-N-phenylsuccinicimide (**2a**). To a stirred solution of 0.173 g *N*-phenylmalimide (1 mmol) in dichloromethane (10 mL),[24] was added 0.8 g *PS-TPP* (1 mmol) in dichloromethane (4 mL) at room temperature. The reaction



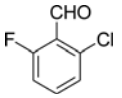
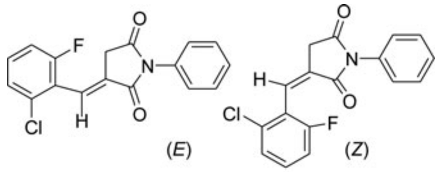
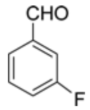
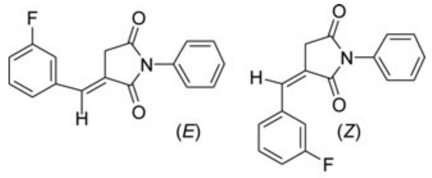
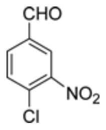
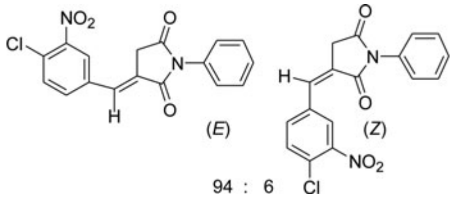
Scheme 3. Wittig olefination did not work for stabilized phosphorus ylides **2b** and **2c**.

Table 1. Synthesis of (E) and (Z)-2-(aryl methylidene)-N-phenyl succinimides **4a–4k**.

Entry	Aldehyde	Products and ratio	Yields (%)
A		 95 : 5	98
B		 94 : 6	88
C		 92 : 8	96
D		 89 : 11	99
E		 87 : 13	97
F		 92 : 8	87
g		 98 : 2	90
h		 92 : 8	90

(*Continued on next page*)

Table 1. (Continued).

Entry	Aldehyde	Products and ratio	Yields (%)
i		 (*E*) (*Z*) 99 : 1	96
j		 (*E*) (*Z*) 98 : 2	95
k		 (*E*) (*Z*) 94 : 6	94

mixture was stirred at room temperature for 24 h, the solution was filtered and *PS-TPPSI* **2a** dried.

Representative procedure for the preparation of compounds **4**. To a magnetically stirred suspension of **2a** (0.973 g, 1 mmol) in toluene (5 mL) was added benzaldehyde (0.106 g, 1 mmol) in dichloromethane (4 mL) at room temperature. The reaction mixture was heated at reflux for 5 h. After cooling the mixture, the solution was filtered and solvent was removed under reduced pressure. The solid products **4a–4k** were deduced from their ^{1}H- and ^{13}C-NMR spectra.

(E) and (Z)-2-(m-nitrophenyl methylidene)-N-phenyl succinimide (**4c**): Brown powder: (0.298 g, mp 142–144 °C, yield 96%); IR (KBr) ($\upsilon_{\max}$, cm^{-1}): 3069 (=CH$_{\text{str.}}$), 1708 (C=O), 1650 (C=C); Anal. calc. for $C_{17}H_{12}N_2O_4$ (308.08): C 66.23, H 3.92, N 9.09; found: C 66.1, H 4.1, N 8.9; ^{1}H NMR (300 MHz, CDCl$_3$): (*E*) Isomer: δ 3.81 (2H, d, $^4J_{\text{HH}}$ = 2.4 Hz, CH$_2$), 7.78 (1H, t, $^4J_{\text{HH}}$ = 2.4 Hz, CH), 7.40–7.71 (9H, m, arom); ^{13}C NMR (75.4 MHz, CDCl$_3$): δ 34.02 (CH$_2$), 124.20, 124.58, 126.37, 128.46, 128.62, 129.25, 131.73, 131.99, 132.02, 133.11, 141.38, 148.19 (aromatic carbons and C=C), 171.10, 172.13 (2C=O). (*Z*) Isomer: δ 3.71 (2H, d, $^4J_{\text{HH}}$ = 2.1 Hz, CH$_2$), 7.38 (1H, t, $^4J_{\text{HH}}$ = 2.1 Hz, CH), 7.40–7.71 (9H, m, arom).

(E) and (Z)-2-(p-methylphenyl methylidene)-N-phenyl succinimide (**4d**): Red powder: (0.339 g, mp 144–146 °C, yield 99%); IR (KBr) ($\upsilon_{\max}$, cm^{-1}): 3050 (=CH$_{\text{str.}}$), 1702 (C=O), 1652 (C=C); Anal. calc. for $C_{18}H_{15}NO_2$ (277.11): C 77.96, H 5.45, N 5.05; found: C 77.9, H 5.6, N 5.1; ^{1}H NMR (300 MHz, CDCl$_3$): (*E*) Isomer: δ 2.41 (3H, s, CH$_3$), 3.75 (2H, d, $^4J_{\text{HH}}$ = 2.4 Hz, CH$_2$), 7.78 (1H, t, $^4J_{\text{HH}}$ = 2.4 Hz, CH), 7.40–7.71 (9H, m, arom); ^{13}CNMR (75.4 MHz, CDCl$_3$): δ 21.56 (CH$_3$), 34.37 (CH$_2$), 126.49, 128.44, 128.56, 129.15, 129.73, 129.95, 132.05, 132.19, 133.12, 135.49 (aromatic carbons and C=C), 170.21, 173.21 (2C=O). (*Z*) Isomer: δ 2.39 (3H, s, CH$_3$), 3.70 (2H, d, $^4J_{\text{HH}}$ = 1.9 Hz, CH$_2$), 7.30 (1H, t, $^4J_{\text{HH}}$ = 1.9 Hz, CH), 7.40–7.71 (9H, m, arom).

(E) and (Z)-2-(p-nitrophenyl methylidene)-N-phenyl succinimide (**4e**): Brown powder: (0.301 g, mp 140–142 °C, yield 97%); IR (KBr) ($\upsilon_{\max}$, cm^{-1}): 3056 (=CH$_{\text{str.}}$), 1708 (C=O), 1596 (C=C); Anal. calc. for $C_{17}H_{12}N_2O_4$ (308.08): C 66.23, H 3.92, N 9.09; found: C 66.3, H 3.9, N 9.1; ^{1}H NMR (300 MH, CDCl$_3$): (*E*) Isomer: δ 3.79 (2H, d, $^4J_{\text{HH}}$ = 2.40 Hz, CH$_2$), 7.80 (1H, t, $^4J_{\text{HH}}$ = 2.40 Hz, CH), 7.40–7.71 (9H, m, arom); ^{13}CNMR (75.4 MHz, CDCl$_3$): δ 34.32 (CH$_2$), 124.36, 126.34, 128.46, 128.62, 129.28, 130.67, 131.73, 131.98, 132.03, 133.11 (aromatic carbons and C=C), 171.10, 172.13 (2C=O). (*Z*) Isomer: δ 3.72 (2H, d, $^4J_{\text{HH}}$ = 1.9 Hz, CH$_2$), 7.20 (1H, t, $^4J_{\text{HH}}$ = 1.9 Hz, CH), 7.40–7.71 (9H, m, arom).

(E) and (Z)-2-(p-chlorophenyl methylidene)-N-phenyl succinimide (**4f**): Red powder: (0.258 g, mp 226–228 °C, yield 87%); IR (KBr) ($\upsilon_{\max}$, cm^{-1}): 3050 (=CH$_{\text{str.}}$), 1706 (C=O), 1643 (C=C); Anal. calc. for $C_{17}H_{12}ClNO_2$ (297.06): C 68.58, H 4.06, N 4.70; found: C 68.7, H 4.0, N 4.6; ^{1}H NMR (300 MHz, CDCl$_3$): (*E*) Isomer: δ 3.73 (2H, d, $^4J_{\text{HH}}$ = 2.4 Hz, CH$_2$), 7.78 (1H, t, $^4J_{\text{HH}}$ = 2.4 Hz, CH), 7.32–7.43 (9H, m, arom); ^{13}C NMR (75.4 MHz, CDCl$_3$): δ 34.18 (CH$_2$), 126.44, 128.69, 128.73, 129.20, 129.53, 130.94, 131.38, 131.78, 131.96, 132.09 (aromatic carbons and C=C), 169.83, 172.75 (2C=O). (*Z*) Isomer: δ 3.65 (2H, d, $^4J_{\text{HH}}$ = 1.9 Hz, CH$_2$), 7.30 (1H, t, $^4J_{\text{HH}}$ = 1.9 Hz, CH), 7.40–7.71 (10H, m, arom).

(E) and (Z)-2-(1,6-dichlorophenyl methylidene)-N-phenyl succinimide (**4g**): Orange powder: (0.290 g, mp 143–145 °C, yield 90%); IR (KBr) ($\upsilon_{\max}$, cm^{-1}): 3065 (=CH$_{\text{str.}}$), 1716 (C=O), 1650 (C=C); Anal. calc. for $C_{17}H_{11}Cl_2NO_2$ (331.02): C 61.47, H 3.34, N 4.22; found: C 61.2, H 3.5, N 4.3; MS: m/z, (%) = 331 (M$^+$, 1), 313 (5), 285 (8), 244 (5), 222 (5), 201 (10), 173 (100), 145 (19), 111 (12), 75 (12); ^{1}H NMR (300 MHz, CDCl$_3$): (*E*) Isomer: δ 3.45 (2H, d, $^4J_{\text{HH}}$ = 2.4 Hz, CH$_2$), 7.76 (1H, t, $^4J_{\text{HH}}$ =

2.4 Hz, CH), 7.28–7.55 (8H, m, arom); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 33.53 (CH_2), 126.43, 128.45, 128.54, 128.61, 130.48, 130.54, 131.76, 131.97, 132.01, 134.05 (aromatic carbons and C=C), 168.28, 172.70 (2C=O). (*Z*) Isomer: δ 3.10 (2H, d, $^4J_{HH}$ = 1.8 Hz, CH_2), 7.59 (1H, t, $^4J_{HH}$ = 1.8 Hz, CH), 7.28–7.55 (8H, m, arom).

(E) and (Z)-2-(m-chlorophenyl methylidene)-N-phenyl succinimide (**4h**): White Powder, (0.267 g, mp 234–236 °C, yield 90%); IR (KBr) (υ_{max}, cm^{-1}): 3047 (=$CH_{str.}$), 1699 (C=O), 1600 (C=C); Anal. calc. for $C_{17}H_{12}ClNO_2$ (297.06): C 68.58, H 4.06, N 4.70; found: C 68.5, H 4.2, N 4.6; ^{1}H NMR (300 MHz, $CDCl_3$): (*E*) Isomer: δ 3.68 (2H, d, $^4J_{HH}$ = 2.0 Hz, CH_2), 7.54 (1H, t, $^4J_{HH}$ = 2.0 Hz, CH), 7.31–7.48 (9H, m, arom); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 34.16 (CH_2), 124.56, 126.42, 128.29, 128.74, 129.22, 129.89, 130.30, 130.44, 131.82, 133.83, 135.22, 135.77 (aromatic carbons and C=C), 169.69, 172.70 (2C=O). (*Z*) Isomer: δ 3.76 (2H, d, $^4J_{HH}$ = 2.4 Hz, CH_2), 7.70 (1H, t, $^4J_{HH}$ = 2.4 Hz, CH), 7.31–7.48 (9H, m, arom).

(E) and (Z)-2-(2-chloro-6-fluorophenyl methylidene)-N-phenyl succinimide (**4i**): Red powder: (0.302 g, yield 96%); IR (KBr) (υ_{max}, cm^{-1}): 3064 (=$CH_{str.}$), 1715 (C=O), 1641 (C=C); Anal. calc. for $C_{17}H_{12}ClFNO_2$ (315.05): C 64.67, H 3.51, N 4.44; found: C 64.9, H 3.8, N 4.3; ^{1}H NMR (300 MHz, $CDCl_3$): (*E*) Isomer: δ 3.51 (2H, d, $^4J_{HH}$ = 2.4 Hz, CH_2), 7.80 (1H, t, $^4J_{HH}$ = 2.4 Hz, CH), 7.10–7.58 (8H, m, arom); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 33.95 (CH_2), 118.07 (d, $^2J_{C\text{-}F}$ = 23.3 Hz, C_{ortho}), 125.86 (d, $^4J_{C\text{-}F}$ = 3.0 Hz, C_{para}), 133.36 (d, $^3J_{C\text{-}F}$ = 9.8 Hz, C_{meta}), 126.42, 126.80, 128.67, 129.16, 130.04, 131.89 (aromatic carbons and C=C), 161.53 (d, $^1J_{C\text{-}F}$ = 245 Hz, C_{ipso}), 168.67, 172.74 (2C=O). (*Z*) Isomer: δ 3.30 (2H, d, $^4J_{HH}$ = 2.0 Hz, CH_2), 7.30 (1H, t, $^4J_{HH}$ = 2.0 Hz, CH), 7.10–7.58 (8H, m, arom).

(E) and (Z)-2-(m-fluorophenyl methylidene)-N-phenyl succinimide (**4j**): Pale brown powder: (0.266 g, mp 176–178 °C, yield 95%); IR (KBr) (υ_{max}, cm^{-1}): 3056 (=$CH_{str.}$), 1707 (C=O), 1644 (C=C); Anal. calc. for $C_{17}H_{12}FNO_2$ (281.09): C 72.59, H 4.30, N 4.98; found: C 72.8, H 4.6, N 4.8; ^{1}H NMR (300 MHz, $CDCl_3$): (*E*) Isomer: δ 3.76 (2H, d, $^4J_{HH}$ = 2.4 Hz, CH_2), 7.70 (1H, t, $^4J_{HH}$ = 2.4 Hz, CH), 7.28–7.51 (9H, m, arom); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 34.15 (CH_2), 117.04 (d, $^2J_{C\text{-}F}$ = 22.6 Hz, C_{ortho}), 126.13 (d, $^4J_{C\text{-}F}$ = 3.0 Hz, C_{para}), 133.43 (d, $^3J_{C\text{-}F}$ = 8.2 Hz, C_{meta}), 126.42, 128.73, 129.22, 130.14, 131.83, 134.07 (aromatic carbons and C=C), 164.59 (d, $^1J_{C\text{-}F}$ = 245.0 Hz, C_{ipso}), 169.74, 172.70 (2C=O). (*Z*) Isomer: δ 3.69 (2H, d, $^4J_{HH}$ = 2.0 Hz, CH_2), 7.53 (1H, t, $^4J_{HH}$ = 2.0 Hz, CH), 7.28–7.51 (9H, m, arom).

(E) and (Z)-2-(4-chloro-3-nitrophenyl methylidene)-N-phenyl succinimide **(4k): Brown powder:** (0.322 g, mp 202–204 °C, yield 94%); IR (KBr) (υ_{max}, cm^{-1}): 3064 (=$CH_{str.}$), 1715 (C=O), 1642 (C=C); Anal. calc. for $C_{17}H_{12}ClNO_2$ (342.04): C 59.57, H 3.23, N 8.17; found: C 59.9, H 3.6, N 8.1; ^{1}H NMR (300 MHz, $CDCl_3$): (*E*) Isomer: δ 3.77 (2H, d, $^4J_{HH}$ = 2.4 Hz, CH_2), 7.53 (1H, t, $^4J_{HH}$ = 2.4 Hz, CH), 7.33–7.51 (8H, m, arom); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 33.53 (CH_2), 126.32, 126.38, 126.71, 128.52, 128.92, 129.29, 131.30, 131.61, 132.88, 133.12, 133.83, 133.93 (aromatic carbons and C=C), 168.10, 171.95 (2C=O). (*Z*) Isomer: δ 3.71 (2H, d, $^4J_{HH}$ = 2.0 Hz, CH_2), 7.53 (1H, t, $^4J_{HH}$ = 2.0 Hz, CH), 7.33–7.51 (8H, m, arom).

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