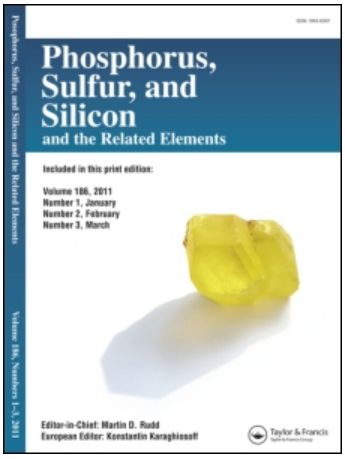

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

4-Aminophenyl Diphenylphosphinite (APDPP) as a Heterogeneous and Acid Scavenger Reagent for Thiocyantion or Isothiocyanation of Alcohols and Protected Alcohols

Nasser Iranpoor[a]; Habib Firouzabadi[a]; Mohammad Gholinejad[a]
[a] Chemistry Department, College of Sciences, Shiraz University, Shiraz, Iran

To cite this Article Iranpoor, Nasser , Firouzabadi, Habib and Gholinejad, Mohammad(2009) '4-Aminophenyl Diphenylphosphinite (APDPP) as a Heterogeneous and Acid Scavenger Reagent for Thiocyantion or Isothiocyanation of Alcohols and Protected Alcohols', Phosphorus, Sulfur, and Silicon and the Related Elements, 184: 8, 2010 — 2019
To link to this Article: DOI: 10.1080/10426500802417950
URL: http://dx.doi.org/10.1080/10426500802417950

Phosphorus, Sulfur, and Silicon, 184:2010–2019, 2009

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500802417950

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4-Aminophenyl Diphenylphosphinite (APDPP) as a Heterogeneous and Acid Scavenger Reagent for Thiocyantion or Isothiocyanation of Alcohols and Protected Alcohols

Nasser Iranpoor, Habib Firouzabadi, and Mohammad Gholinejad
Chemistry Department, College of Sciences, Shiraz University, Shiraz, Iran

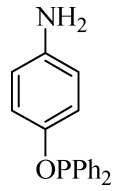
4-Aminophenyl diphenylphosphinite (APDPP) as a heterogeneous phosphinite reagent is used for the efficient conversion of alcohols, trimethylsilyl- and tetrahydropyranyl ethers, α-hydroxy phosphonates, and α-trimethylsilyloxyphosphonates to their corresponding thiocyanates or isothiocyanates in the presence of Br_2 and NH_4SCN.

Keywords Alcohol; 4-aminophenyl diphenylphosphinite; isothiocyanate; tetrahydropyranyl ether; thiocyanate; trimethylsilyl ether

INTRODUCTION

Conversion of alcohols to alkyl halides and alkyl thiocyanates or isothiocyanates by the use of phosphines has been widely studied.[1–6] Apart from these reactions, the use of in situ generated $PPh_3(SCN)_2$ for this transformation is also reported.[7,8] In these reactions, the separation of the soluble phosphine oxide byproduct is tedious and time-consuming work. Some attempts to solve this problem, such as the use of polymer supported phosphines,[9–12] phosphines tagged crown ethers,[13] (*p*-dimethyl aminophenyl)-diphenylphosphine (DAPDP),[14] 1,2-bis(diphenylphosphineo)ethane,[15] 4-diphenyl phosphinyl benzoic acid 2-trimethylsilanyl ether ester (DPPBE),[16] and perfluorinated phosphines[17] are reported in the literature. Polymer supported

Received 16 April 2008; accepted 18 August 2008.
We are grateful to Shiraz University Research Council for the partial support of this work and for a grant from Organization of Management and Programming of Iran.
Address correspondence to Nasser Iranpoor and Habib Firouzabadi, Chemistry Department, College of Sciences, Shiraz University, Shiraz 71454, Iran. E-mail: iranpoor@chem.susc.ac.ir; firouzabadi@chem.susc.ac.ir



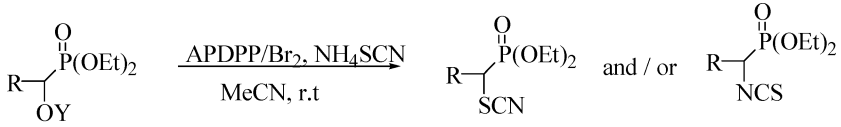
(I)

FIGURE 1 The structure of APDPP.

phosphines are the most widely used heterogeneous reagents for replacement of triphenylphosphine in organic synthesis. We have also recently prepared and applied $(SiO_2)_nPCl_{3-n}$, (Silphos) as an inorganic, silica-based, polymeric phosphorane reagent in different organic transformations.[18-19] However, apart from their costs, the polymeric reagents usually have the problems of lower efficiency compared to the monomeric ones and also difficulty of scaling-up the reactions. In this article, we report the use of 4-aminophenyl diphenylphosphinite (APDPP) (Figure 1) as a heterogeneous and acid scavenger reagent for the conversion of alcohols, and trimethylsilyl and tetrahydropyranyl ethers to their corresponding thiocyanates or isothiocyanates in the presence of molecular bromine and NH_4SCN in acetonitrile at room temperature (Scheme 1). α-Hydroxyphosphonates and α-trimethylsilyloxyphosphonates were also successfully subjected to this transformation (Scheme 2).

Y: H, $SiMe_3$,THP
R: benzyl, *p*-methoxybenzyl, *p*-nitrobenzyl, 1-phenyl-3-propyl, 2-octyl, 1,1-diphenylmethyl, 1-adamantyl, *o*-nitrobenzyl

SCHEME 1



Y: H, $SiMe_3$
R: benzyl, *p*-methoxybenzyl, *p*-chlorobenzyl

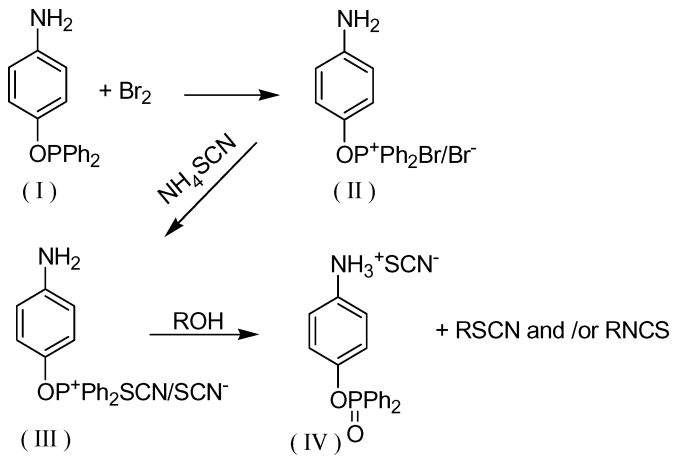
SCHEME 2

RESULTS AND DISCUSSION

There are different reagent systems reported in the literature for the preparation of thiocyanates as important and valuable sulfur-containing compounds.[20–24] Most of the reported methods involve the use of homogeneous phosphorous-containing reagents and usually suffer from the tedious work-up procedure and isolation of the produced byproduct phosphorous oxide compounds. Recently, we have introduced 4-aminophenphyl diphenylphosphinite (APDPP) as a heterogeneous phosphinite reagent for the conversion of alcohols and protected alcohols to their corresponding halides in the presence of molecular halogens or *N*-halosuccinimides.[25]

Now we report on the applicability of this heterogeneous phosphorane (APDPP) for the efficient conversion of hydroxyl functionality and its trimethylsilyl and tetrahydropyranyl derivatives to thiocyanate or isothiocyanate functionalities in the presence of Br_2 and NH_4SCN. Optimization of the reaction conditions showed that the stoichiometric ratio of 1:1.6:1.6:3.2 for the mixture of benzyl alcohol/APDPP/Br_2/NH_4SCN in CH_3CN at room temperature is suitable for conversion of benzyl alcohol to its corresponding thiocyanate. These reaction conditions were then applied for the conversion of structurally different alcohols and α-hydroxyphosphinates to their thiocyanates or isothiocyanates. The results of this study are shown in Table I.

The suggested mechanism is similar to that which has been proposed for Ph_3P (Scheme 3).[7,8]



SCHEME 3

$$ROH \xrightarrow[MeCN,\ r.t]{APDPP/Br_2,\ NH_4SCN} RSCN \text{ and/or} RNCS$$

TABLE I Conversion of Alcohols and α-Hydroxyphosphinates into Thiocyanates or Isothiocyanates[a,b]

Entry	R	Time (h)	Ratio of RSCN/RNCS	Isolated yield %
1	Benzyl	1	100/0	94
2	*p*-methoxybenzyl	0.75	92/8	82
3	*p*-nitrobenzyl	5	100/0	80
4	1-phenyl-3-propyl	1	100/0	90
5	2-octyl	3	54/46	89 (as mixture)
6	1,1-diphenylmethyl	3	0/100	90
7	1-adamantyl	8	15/85	85 (as mixture)
8	benzyl-$\underset{\vert}{C}HPO(OEt)_2$	5	85/15	63
9	*p*-methoxybenzyl-$\underset{\vert}{C}HPO(OEt)_2$	5	80/20	60

[a]Optimized ratio of ROH/APDPP/Br_2/NH_4SCN is 1:1.6:1.6:3.2.

[b]All the products are known compounds and identified by comparison of their spectral data with those prepared according to the literature.[6–9,28,29]

The conversion of alcohols into thiocyanates or isothiocyates could occur through the reaction between APDPP (I) with molecular bromine to produce $APDPP^+Br/Br^-$ (II), followed by the reaction with NH_4SCN to produce the reactive heterogeneous $APDPP^+SCN/SCN^-$ (III) as the thiocyataing agent. Attack of alcohol to the positively charged phosphonium ion in $APDPP^+SCN/SCN^-$ (III) makes the hydroxyl functionality a good leaving group in which its nucleophilic substitution with thiocyanate or isothiocyanate ions produces the product. In support of this mechanism, the precipitated ammonium thiocyanate salt of the corresponding phosphinate ester (IV) was easily isolated by filtration and washed with water to remove its contaminated ammonium bromide. After drying in vacuum oven, its IR spectrum as KBr disk showed the ammonium group as a broad strong band centered at 3105 cm^{-1}. The absorption bands for SCN and P=O groups also appear as strong bands at 2056 and 1400 cm^{-1}, respectively. The formation of (IV) shows the acid scavenger nature of this phosphinite reagent.

We then examined the possibility of this transformation for trimethylsilyl and also THP ethers. The optimized stoichiometric ratios of $ROSiMe_3$/APDPP/Br_2/NH_4SCN and ROTHP/APDPP/Br_2/NH_4SCN were found to be 1:1.2:1.2:2.4 and 1:2:2:4, respectively, in CH_3CN at room temperature. These optimized ratios were employed for the

$$ROY \xrightarrow[MeCN,\ r.t]{APDPP/Br_2,\ NH_4SCN} RSCN \text{ and/or } RNCS$$

TABLE II Conversion of Trimethylsilyl Ethers, α-Trimethylsilyloxyphosphonates, and Tetrahydropyranyl Ethers into Thiocyanates and Isothiocynates[a,b]

Entry	R	Y	Time	RSCN/ RNCS	Isolated yield (%)
1	benzyl	$SiMe_3$	immediately	100/0	92
2	*p*-methoxybenzyl	$SiMe_3$	immediately	92/8	86
3	*o*-nitrobenzyl	$SiMe_3$	5 min	100/0	90
4	2-octyl	$SiMe_3$	5 min	52/48	89 (as a mixture)
5	1,1-diphenylmethyl	$SiMe_3$	5 min	0/100	90
6	benzyl	THP	7 h	100/0	83
7	1-phenyl-3-propyl	THP	8 h	100/0	80
8	1,1-diphenylmethyl	THP	24 h	0/100	70
9	benzyl-$\underset{\vert}{C}HPO(OEt)_2$	$SiMe_3$	45 min	92/8	85
10	*p*-methoxybenzyl-$\underset{\vert}{C}HPO(OEt)_2$	$SiMe_3$	30 min	88/12	77
11	*p*-chlorobenzyl-$\underset{\vert}{C}HPO(OEt)_2$	$SiMe_3$	1 h	94/6	80

[a]The molar ratio of ROTHP/APDPP/Br_2/NH_4SCN is 1:2:2:4 and α-trimethylsilyloxyphosphonates or $ROSiMe_3$/APDPP/Br_2/NH_4SCN is 1:1.2:1.2:2.4.

[b]All the products are known compounds and identified by comparison of their spectral data with those prepared according to the literature.[6–9,28]

conversion of trimethylsilyl ethers, tetrahydrpyranyl ethers, and also α-trimethylsilyloxyphosphinate to their corresponding thiocyanates or isothiocyanates in good to high yields. The results of this study are shown in Table II.

The formation of thiocyanates or isothiocyanates in these reactions is a structure-dependent reaction that has been previously observed by us and the other groups.[5–7] It has been observed that increasing the steric hindrance of the substrate increases the formation of isothiocyates. The results of Tables I and II show that the reaction of primary alcohols produces thiocyanates as the sole product along with trace formation of isothiocyanates as the minor products (0–8%). For 2-octanol as a secondary alcohol, both thiocyanate and isothiocyanate were obtained in nearly equal amounts. Also, benzhydrol as a bulky alcohol gave its isothiocyanate as the sole product of the reaction. For 1-adamananol as a tertiary alcohol, its corresponding isothiocyanate and thiocyanate were produced with the ratio 85:15.

$$ROY \xrightarrow[MeCN,\ r.t]{APDPP/Br_2,\ NH_4SCN} RSCN \text{ and/or } RNCS$$

TABLE III Selective Reaction of Trimethyl Silyl Ethers in the Presence of Hydroxyl Compounds

Entry	Binary mixture	R	Time (min)	GC yields (%)[a]
1	benzyl	H	1	0
		$SiMe_3$		100
2	*p*-methoxybenzyl	H	1	0
		$SiMe_3$		100
3		H	5	0
	o-nitrobenzyl	$SiMe_3$		100
4	2-octyl	H	5	10
		$SiMe_3$		90
5	1,1-diphenylmethyl	H	5	5
		$SiMe_3$		95
6	benzyl-$\underset{\vert}{C}HPO(OEt)_2$	H	45	5
		$SiMe_3$		95

[a]The yield was determined by GC analysis using internal standard.

We have also observed that with APDPP/Br_2/NH_4SCN, the conversion of trimethylsilyl ethers to their thiocyanates or isothiocyanates is a highly selective reaction in the presence of their alcohols. We have shown this selectivity by a series of competitive reactions in binary mixtures as summarized in Table III.

The reason for the higher reactivity of trimethylsilyl ethers compared to alcohols is not clear to us. However, we may suggest that the more pronounced H-bonding interaction between alcohol molecules and the amino group of APDPP(III) makes them less reactive compared to silyl ethers in this transformation.

EXPERIMENTAL

Chemicals were either prepared in our laboratory or obtained from Fluka (Switzerland) and Merck chemical companies. Infrared spectra were recorded on a Perkin-Elmer 781 spectrometer. Nuclear magnetic resonance spectra were recorded on an Avance DPX-250 MHz spectrometer. GC analyses were recorded on a Shimadzu GC-14A.

Typical Procedure for the Conversion of Benzyl Alcohol to Benzyl Thiocyanate by APDPP/Br_2/NH_4SCN

To a flask containing a stirring mixture of APDPP (0.46 g, 1.6 mmol) and Br_2 (0.08 mL, 1.6 mmol), NH_4SCN (0.24 g, 3.2 mmol) in dry CH_3CN

(7 mL) was added at room temperature and stirred for 2 h until all bromine was consumed. Then benzyl alcohol (0.1 mL, 1 mmol) was added to this mixture. TLC monitoring or GC analysis showed the completion of the reaction after 1 h. The reaction mixture was filtered to remove the produced heterogeneous phosphinate ester and ammonium bromide. The solvent was then evaporated, and the residue was washed with 25 mL of diethyl ether. Diethyl ether was then evaporated and for further purification, the product was chromatographed on a column of silica-gel using *n*-hexane-ethyl acetate (5:1) as eluent. Benzyl thiocyanate was obtained in 94% yield (0.14 g), mp = 38–39°C (Lit.[8], mp 39–40°C), ^{1}H NMR ($CDCl_3$/TMS): $\delta = 4.25$ (2H, s), 7.21–7.31 (5H, m); ^{13}C NMR ($CDCl_3$/TMS): $\delta = 134.8$, 129.5, 129.4, 129.3, 112.4, 38.7. This reaction was also successfully scaled up to 8 folds. Isolated yield for the scaled up reaction was found to be 91%.

Typical Procedure for the Conversion of Benzyl Trimethylsilyl Ether to Benzylthiocyanate by APDPP/Br_2/NH_4SCN

NH_4SCN (0.18 g, 2.4 mmol) was added to a flask containing APDPP (0.35 g, 1.2 mmol) and Br_2 (0.06 ml, 1.2 mmol) in CH_3CN (7 mL) at room temperature. After 2 h, the orange color of bromine changed to pale yellow or disappeared. To this reaction mixture, benzyltrimethylsilyl ether (0.18 g, 1 mmol) was added at room temperature. GC analysis or TLC monitoring showed the completion of the reaction after 1 min. For isolation of the produced phosphinite ester byproduct, the mixture was filtered. After evaporation of the solvent, the residue was washed with diethyl ether (25 mL). The crude product was chromatographed on a column of silica-gel using *n*-hexane-ethyl acetate (5:1) as eluent. The corresponding thiocyanate was obtained in 92% yields (0.13 g). The product was identified by comparison with a known sample.[8]

Typical Procedure for the Conversion of Benzyltetrahydropyranyl Ether to Benzylthiocyanate by APDPP/Br_2/NH_4SCN

To a flask containing a stirring mixture of APDPP (2 mmol, 0.58 g) and Br_2 (2 mmol, 0.1 ml) in CH_3CN (7 mL), NH_4SCN (0.3 g, 4 mmol) was added at room temperature. After 2 h, benzyltetrahydropyranyl ether (0.18 g, 1 mmol) was added to the reaction mixture. TLC and GC analysis of the reaction showed the completion of the reaction after 7 h. The reaction mixture was filtrated to remove the produced phosphinate ester. After evaporation of the solvent, the residue was washed with 25 mL of diethyl ether. In order to purify the obtained product, it

was passed through a short column of silica-gel using *n*-hexane-ethyl acetate (5:1) as eluent. Benzyl thiocyanate was obtained in 83% yield (0.12 g).

Typical Procedure for the Conversion of Diethyl α-Hydroxybenzylphosphinate to Diethyl α-Thiocyanatobenzylphosphinate Using APDPP/Br_2/NH_4SCN

To a flask containing APDPP (0.46 g, 1.6 mmol) and Br_2 (0.1 ml, 2 mmol) in CH_3CN (7 mL), NH_4SCN (0.24 g, 3.2 mmol) was added at room temperature and stirred for 2 h. To this reaction mixture, diethyl α-hydroxybenzylphosphinate (0.244 g, 1 mmol) was added. TLC and GC analysis showed the completion of reaction after 5 h. The mixture was filtered, and after evaporation of solvent, the residue was washed with diethyl ether (25 mL). After evaporation, the residue was chromatographed over a short column of silica-gel (2 cm thick) using *n*-hexane-ethyl acetate as an eluent. α-Thiocyanatobenzylphosphinate was obtained in 63% yield, 0.18 g. The product was identified by comparison of its spectral data with the literature.[6a]

Typical Procedure for the Conversion of Diethyl α-Trimethylsilyloxyphosphonates to Diethyl α-Thiocyanobenzylphosphinate by APDPP/Br_2/NH_4SCN

To a flask containing a stirring mixture of APDPP (0.35 g, 1.2 mmol) and Br_2 (0.06 g, 1.2 mmol) in CH_3CN (7 mL), NH_4SCN (0.18 g, 2.4 mmol) was added at room temperature and stirred for 2 h. Then α-trimethylsilyloxyphosphonates (0.31 g, 1 mmol) was added to this reaction mixture. TLC and GC analysis of the reaction showed the completion of the reaction after 45 min. The reaction mixture was then filtered. After evaporation of the solvent, the residue was washed with 25 mL of diethyl ether. For further purification, the residue was chromatographed on a column of silica-gel using *n*-hexane-ethyl acetate (5:2) as eluent. α-Thiocyanobenzylphosphinate was obtained in 85% yield (0.24 g) and identified by comparison with a known sample.[6a] ^{1}H NMR ($CDCl_3$/TMS): δ (ppm) = 1.12 (t, 3 H, $3J_{HH}$= 7.15 Hz, OCH_2CH_3), 1.40 (t, 3 H, $3J_{HH}$= 7.15 Hz, OCH_2CH_3), 3.85–3.96 (m, 1 H, OCH_2CH_3), 4.09–4.23 (m, 1 H, OCH_2CH_3), 4.33–4.42 (m, 2 H, OCH_2CH_3), 4.65 (d, 1 H, $2J_{PH}$= 17.8 Hz, CH), 7.47–7.60 (m, 5 H, C_6H_5). ^{13}C NMR ($CDCl_3$/TMS): δ(ppm) = 16.34 (d, $3J_{CP}$= 5.7 Hz, OCH_2CH_3), 16.47 (d, $3J_{CP}$= 5.7 Hz, OCH_2CH_3), 47.83 (d, $1J_{CP}$= 148.1 Hz, CH), 64.07 (d, $2J_{CP}$= 7 Hz, OCH_2CH_3), 64.69 (d, $2J_{CP}$= 7 Hz, OCH_2CH_3), 114.54

(SCN), 130.08–130.43, 133.01 (C_6H_5). ^{31}P NMR ($CDCl_3$): δ‘(ppm) = 17.17.

Diethyl α-Thiocyanato-4-methoxybenzylphosphonate

^1H NMR ($CDCl_3$/TMS): δ = 1.34 (t, 3 H, $3J_{HH}$= 7.15 Hz, OCH_2CH_3), 1.56 (t, 3 H, $3J_{HH}$= 7.15 Hz, OCH_2CH_3), 3.98 (s, 3 H, 4-OCH_3), 4.09–4.39 (m, 4 H, OCH_2CH_3), 4.62 (d, 1 H, $2J_{PH}$= 17.6 Hz, CH), 7.03 (d, 2 H, $3J_{HH}$= 8.6 Hz, C_6H_4), 7.45 (d, 2 H, $3J_{HH}$= 8.6 Hz, C_6H_4). ^{13}C NMR ($CDCl_3$/TMS): δ = 16.64 (d, $3J_{CP}$= 5.6 Hz, OCH_2CH_3), 16.83 (d, $3J_{CP}$= 5.6 Hz, OCH_2CH_3), 47.64 (d, $1J_{CP}$= 146.8 Hz, CH), 56.70 (4-OCH_3), 64.38 (d, $2J_{CP}$= 7.15 Hz, OCH_2CH_3), 64.82 (d, $2J_{CP}$= 7.15 Hz, OCH_2CH_3), 114.63 (SCN), 115.11, 129.18, 131.11, 160.78 (C_6H_4). ^{31}P NMR ($CDCl_3$): δ(ppm) = 17.43.

Diethyl α-Thiocyanato-4-chlorobenzylphosphonate

^1H NMR ($CDCl_3$/TMS): δ= 1.37 (t, 3 H, $3J_{HH}$= 7.15 Hz, OCH_2CH_3), 1.49 (t, 3 H, $3J_{HH}$= 7.15 Hz, OCH_2CH_3), 3.91–4.06 (m, 1 H,OCH_2CH_3), 4.12–4.22 (m, 1 H, OCH_2CH_3), 4.28–4.39 (m, 2 H,OCH_2CH_3), 4.71 (d, 1 H, $2J_{PH}$= 18.3 Hz, CH), 7.39 (d, 2 H, $2J_{HH}$= 8.6 Hz, C_6H_4), 7.78 (d, 2 H, $2J_{HH}$= 8.6 Hz, C_6H_4).^{13}C NMR ($CDCl_3$/TMS): δ= 16.66 (d, $3J_{CP}$= 5.6 Hz, OCH_2CH_3), 16.91 (d, $3J_{CP}$= 5.6 Hz, OCH_2CH_3), 47.54 (d, $1J_{CP}$= 145.1 Hz, CH), 64.52 (d, $2J_{CP}$= 7.15 Hz, OCH_2CH_3), 65.11 (d, $2J_{CP}$= 7.15 Hz, OCH_2CH_3), 114.01 (SCN), 130.1, 131.21, 131.45, 136.04 (C_6H_4). ^{31}P NMR ($CDCl_3$): δ(ppm) = 16.58.

***p*-MeO-PhCH$_2$SCN**: ^1H NMR ($CDCl_3$/TMS): δ = 3.2 (3H, s), 3.9 (2H, s), 6.88–7.26 (4H, m); ^{13}C NMR ($CDCl_3$/TMS): δ = 159.5, 132.8, 127.5, 115.9, 111.2, 55.3, 38.8.

PhCH$_2$CH$_2$CH$_2$SCN: ^1H NMR ($CDCl_3$/TMS): δ= 2.0 (2 H, q), 2.7 (2 H, t), 2.9 (2 H, t), 7.1–7.5 (5 H, m);^{13}C NMR ($CDCl_3$/TMS): δ= 140.3, 129.1, 128.9, 126.8, 112.6, 34.2, 32.5, 31.6.

CH$_3$(CH$_2$)$_5$CH(SCN)CH$_3$: ^1H NMR ($CDCl_3$/TMS): δ= 0.8 (3 H, t), 0.75 (2 H, m), 0.95 (3 H, d), 1.1 (6 H, m), 1.7 (2 H, q), 2.5 (1 H, m);^{13}C NMR ($CDCl_3$/TMS): δ= 111.9, 46.1, 37.3, 32.0, 29.1, 27.3, 22.9, 22.3, 14.4.

Ph$_2$CHNCS: mp = 56–58°C, (Lit.[26], mp 57–58°C); ^1H NMR ($CDCl_3$/TMS): δ= 6.98 (1 H, s), 6.98-7.38 (10 H, m).^{13}C NMR ($CDCl_3$/TMS): δ= 150.0, 139.6, 129.3, 128.7, 127.9, 65.0.

***p*-NO$_2$-PhCH$_2$SCN**: mp = 87–88°C, (Lit.,[27] mp 85–86°C); ^1H NMR ($CDCl_3$/TMS): δ= 4.22 (2 H, S), 7.52 (2 H, m), 8.23(2 H, m);^{13}C NMR ($CDCl_3$/TMS): δ= 146.4, 140.1, 128.3, 122.7, 109.3, 35.32.

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