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New synthesis of diphenyl-*N*-(substituted)ketenimines from diaminophosphonium diazaylides

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Abstract

Diaminophosphonium diazaylides **2** react under mild conditions with diphenylacetyl chloride, to afford diphenyl-*N*-(substituted)ketenimines **4** or, depending on the case, their transformation products: either the tautomer **8**, or the dimer **9**. The general reaction seems to proceed firstly via an elimination step on the acid chloride followed then by an aza-Wittig reaction between the resulting ketene **7** and the diaminophosphonium monoazaylide **6**.

Keywords: Diaminophosphonium diazaylide; Diphenylacetyl chloride; Ketenimine; Aza-Wittig reaction

1. Introduction

In recent work, we have shown that phosphonium diylides **1** are excellent tools in organic synthesis [1]. Indeed, these reagents, thanks to their reinforced carbanionic activity, are more reactive than the corresponding triphenylphosphonium monoylides and offer a general method for the synthesis of numerous α,β-unsaturated functions [1] (Scheme 1).

The corresponding nitrogen compounds, diaminophosphonium diazaylides **2**, until now mainly used in coordination chemistry [2], have been less studied in the field of organic synthesis. As a first application example we have found that these reagents react as equivalents of the synthon RNH^-, thus allowing the synthesis of primary or secondary amines [3]. The results reported here show another example of application for nitrogen diazaylides **2**: the synthesis of diphenyl-*N*-(substituted)ketenimines **4**.

2. Results

We have studied the reaction between various diazaylides **2**, regardless of their stabilization, and diphenylacetyl chloride (Scheme 2, Table 1).

Non-stabilized and semi-stabilized diazaylides **2** are synthesized from the corresponding phosphonium salts **3** [3,4]. After the in situ addition, at −50°C, of one equivalent of diphenylacetyl chloride the non-stabilized diazaylide **2a** (R = *n*-Bu) immediately disappears to instantaneously give the corresponding phosphinic amide **5a** as sole organophosphorus product, as shown by the ^{31}P-NMR spectrum of the reaction mixture (Table 1). Simultaneously the formation of the diphenyl-*N*-*n*-butylketenimine **4a** can be observed: the IR spectrum of the reaction mixture exhibits a very strong absorption at 2010 cm^{-1}, characteristic of such heterocumulenes. The yield in ketenimine **4a**, which then reaches 100% is determined by ^{1}H-NMR titration. It should be noted that this method corroborates the yield, first determined by ^{31}P-NMR, for the phosphinic amide **5a**.

Non-stabilized diazaylide **2b** for which the substituent on nitrogen is a more bulky group (R = *i*-Pr) also reacts quickly with diphenylacetyl chloride to give quantitatively the corresponding ketenimine **4b**. With a still more bulky group (R = *t*-Bu), the reaction is

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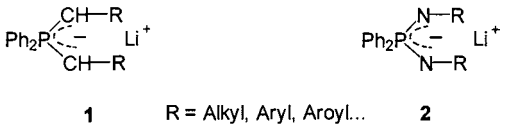


Scheme 1. Lithium dialkyldiphenylphosphonium diylides **1** and nitrogen analogs **2**.

slower, but a yield of 70% in ketenimine can be obtained after 5 h at 65°C. At this time, the transformation ratio of the starting diazaylide **2c** reaches 100%, but only 60% of the phosphinic amide **5c** is formed (^{31}P-NMR). Moreover, after the work-up of the reaction mixture about 30% of the diaminophosphonium monoazaylide **6c** is isolated together with a significant amount of ketene **7**. This result is interesting from a mechanistic point of view: in a first step, the diazaylide deprotonates the diphenylacetyl chloride leading to the concomitant formation of the corresponding ketene **7** and monoazaylide **6c**; the second step consists of an in situ aza-Wittig reaction between the two species giving the ketenimine **4c** and the phosphinic amide **5c** (Scheme 2).

Starting from the diazaylide **2d** ($R = CH_2Ph$), the formation of a high yield in ketenimine **4d** was also immediately observed. It is interesting to note that after some time at 20°C, the product evolves continuously to the major formation of the tautomer **8**, which is thermodynamically more stable (Scheme 3).

As in the previous cases, the *N*-allylsubstituted diazaylide **2e** reacts at low temperature with diphenylacetyl chloride immediately resulting in a good amount of the corresponding ketenimine **4e**. However, this compound is quickly consumed, as shown by the disappearance of the IR absorption at 2015 cm^{-1}, to give quantitatively the symmetric dimer **9** (Scheme 4).

The identification of dimer **9** was based on the spectroscopic data which allowed us to quickly discard the two possible dissymetric structures. Between the two possible remaining forms the diazetidine **9′** was also discarded on the basis of the ^{13}C-NMR and by comparison with the results of the literature concerning a structure close to cyclobutane **9** [5].

With the semi-stabilized diazaylide **2f** ($R = Ph$) the reaction is slow, but affords the diphenylketene **7** together with the intermediate monoazaylide **6f** (isolated yield: 75%). This result further corroborates the proposed mechanism.

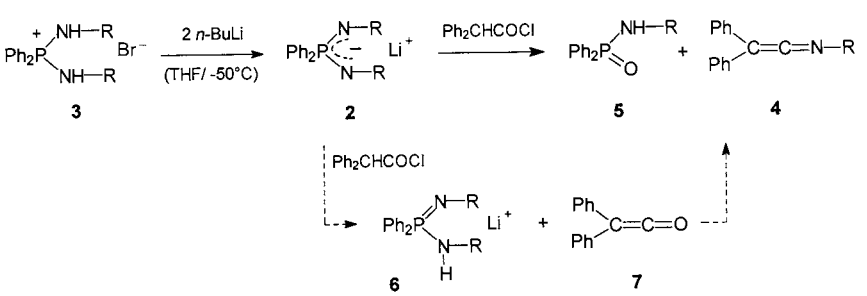
Finally, with the stabilized diazaylide **2g** ($R = C(O)Ph$), the reaction is also slow and the maximum yield in ketenimine **4g**, determined on the basis of the phosphinic amide formed, only reaches 25%. The ketenimine can be detected on the IR spectra only at the beginning of the reaction, the corresponding signal disappearing after 2 h. This observation is in accordance with the results of the literature mentioning that *N*-aroyldiphenylketenimines are very unstable (they are characterized only by IR spectra) [6]. It should be noted that the formation of the intermediates **6g** and **7** can also be observed.

The mechanism proposed in Scheme 2 contains the illustration for the three categories of diazaylides **2** (stabilized, non- and semi-stabilized). Accordingly, this mechanism seems to be general, although we did not observed the intermediate **6** and **7** in the case where the reaction is particularly quick.

The reactivity of *n*-butylaminotriphenylphosphonium monoazaylide **10** was also tested towards diphenylacetyl chloride (Scheme 5). The ketenimine **4a** is formed but this method was not developed because of the presence of two phosphorus by-products, **11** and **12**, and because of the lower reactivity of **10** in comparison with the corresponding diazaylide **2a** (60% yield after 1 h at 20°C with the monoazaylide, and 100% after 5 min at −50°C with the diazaylide).

3. Conclusions

In conclusion, the study of the reactivity of diaminophosphonium diazaylides **2** affords a new method for the synthesis, under mild conditions, of diphenyl-*N*-(substituted)ketenimines, which are a family of not very stable compounds, and of the cyclic dimer of the *N*-allyldiphenylketenimine. These results represent an additional example of the potential of phosphonium diylides in organic synthesis.



Scheme 2. Synthesis of diphenyl-*N*-(substituted)ketenimines **4** by reaction of diphenylacetyl chloride with various diaminophosphonium diazaylides **2**.

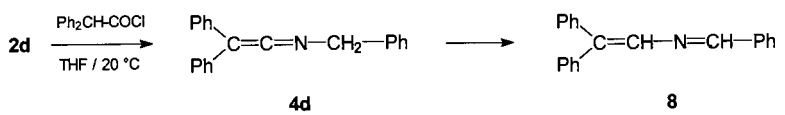
Table 1
Synthesis of diphenyl-*N*-(substituted)ketenimines (**4**) by reaction of diaminophosphonium diazaylides **2** with diphenylacetyl chloride

Diazaylide	R	Reaction conditions (°C)	**2** tr (%) [a]	**4**		**5** yield (%) [d]	Other products
				IR (cm^{-1}) [b] C=C=N	Yield (%) [c]		
2a	*n*-Bu	5 min/−50	100	2010 vs	100	100	
2b	*i*-Pr	1 h/20	100	2000 vs	80	85	
		12 h/20	100	2000 vs	100	100	
2c	*t*-Bu	12 h/20	20	2005 s	15	20	
		5 h/65	100	2005 vs	70	60	**6c**: 30%
		22 h/65	100	2005 vs	90	95	
2d	CH_2Ph	5 min/−50	100	2010 vs	60	80	
		2 h/20	100	2010 vs	65	100	
		30 h/20	100	2010 s	25	100	**8**: 60% [c]
2e	$CH_2CH{=}CH_2$	5 min/−50	100	2015 vs	70 [e]	60	
		1 h/0	100	2015 w	– [f]	80	
		6 h/20	100	–	0	95	**9**: 100% [e]
2f	Ph	2 h/20	100	1995 m	25 [g]	20	**6f**: 75%
2g	C(O)Ph	1 h/20	– [f]	2020 m	10 [g]	10	
		24 h/20	80	–	0 (25 [g])	25	**6g**: 50%

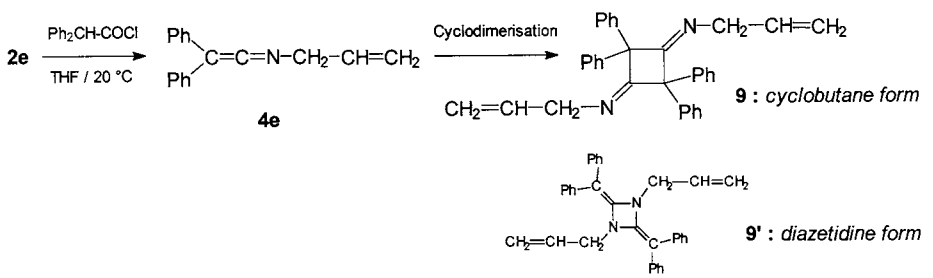
[a] tr, transformation ratio of **2** determined by ^{31}P-NMR.
[b] vs, very strong; s, strong; m, medium; w, weak.
[c] Yield determined after concentration of a sample of the reaction mixture by ^{1}H-NMR titration with *p*-iodoanisole as internal standard (isolated yields are given in Section 4).
[d] Yield determined by ^{1}H-NMR titration and/or by ^{31}P-NMR.
[e] Yield determined by ^{1}H-NMR titration on the basis of the N-CH_2 signal.
[f] Undetermined.
[g] Determined from the phosphinic amide yield.

4. Experimental

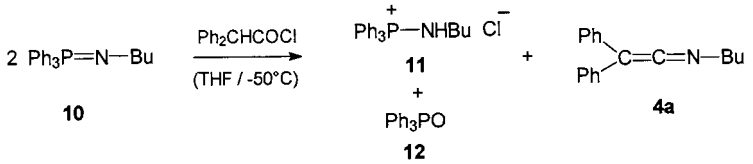
Melting points were determined using a Wild Leitz 350 and are given uncorrected. ^{1}H-, ^{31}P- and ^{13}C-NMR were recorded on a Bruker AC-200 spectrometer (200.132, 81.0 and 50.323 MHz, respectively). IR spectra were obtained with a Perkin–Elmer 377. Mass spectra were measured with a Jeol JMS DX-300 spectrometer. All solvents were distilled from drying agents prior to use. The reactions were performed under nitrogen using Schlenk techniques. *n*-Butyllithium commercial solutions in hexane (Aldrich) were titrated before use [7]. Diaminophosphonium salts were prepared according to the literature [4]. Commercial diphenylacetyl chloride (Lancaster) and 4-iodoanisole (Aldrich) were used without purification.



Scheme 3. In situ formation of compound **8**.



Scheme 4. In situ cyclodimerisation of the diphenyl-*N*-allylketenimine **4e**.



Scheme 5. Synthesis of the ketenimine **4a** by reaction of two equivalents of *n*-butylaminotriphenylphosphonium monoazaylide **10** with diphenylacetyl chloride.

4.1. Synthesis of diphenyl-N-(substituted)ketenimines 4

4.1.1. General procedure

To 5 mmol of diaminodiphenylphosphonium bromide **3** in THF (20 ml) was added dropwise 10 mmol of *n*-BuLi (4.35 ml, 2.3 M in hexane) at −50°C. Then *p*-iodoanisole (1.17 g, 5 mmol) was added for the ^{1}H-NMR titration. After 30 min at this temperature, diphenylacetyl chloride (1.15 g, 5 mmol) was added resulting in a yellow coloration of the solution. Depending on the diazaylide **2**, the reaction mixture is then allowed to warm up to room temperature or refluxed in THF (see Table 1). When the reaction (monitored by ^{31}P- and ^{1}H-NMR) is stopped the THF is evaporated and water is added (20 ml). The aqueous phase is then extracted twice with methylene chloride (2 × 30 ml) and the combined organic layers are dried on $MgSO_4$, filtered and concentrated under vacuum (at a temperature below 10°C). Then the work-up depends on the ketenimine.

4.1.2. Diphenyl-N-n-butylketenimine (4a)

From bis-*n*-butylaminodiphenylphosphonium bromide **3a** (2.05 g, 5 mmol). After concentration of the organic layer, diphenyl-*N*-*n*-butylketenimine **4a** is distilled under reduced pressure. Yellow liquid; reaction yield: 100%; isolated yield: 80%.

B.p.$_{0.5}$ 90°C {lit. [8] 150–153 (16 mm)}. ^{1}H-NMR ($CDCl_3$): $\delta = 1.03$ (d, $J = 7.3$ Hz, 3H, CH_3), 1.40 (m, $J = 7.3$ Hz, 2H, CH_2CH$_3$), 1.80 (m, $J = 7.5$ Hz, 2H, CH_2CH$_2$CH$_3$), 3.68 (t, $J = 7.3$ Hz, 2H, CH_2N), 7.33–7.42 (m, 10H, 2 C$_6H_5$). ^{13}C{^{1}H}-NMR ($CDCl_3$): $\delta =$ 13.72 (s, *C*H$_3$), 20.21 (s, *C*H$_2$CH$_3$), 32.44 (s, *C*H$_2$CH$_2$CH$_3$), 52.93 (s, *C*H$_2$N), 78.60 (s, *C*=C=N), 125.89 (s, *p*-*C*, C$_6$H$_5$), 127.64 (s, *o*-*C*, C$_6$H$_5$), 128.75 (s, *m*-*C*, C$_6$H$_5$), 135.20 (s, *i*-*C*, C$_6$H$_5$), 185.40 (s, C=*C*=N). MS (EI): m/z (%) = 249 (41, M^+), 193 (100),165 (64), 57(4). Anal. Calc. for $C_{18}H_{19}N$ (249.35); C, 86.70; H, 7.68; N, 5.62. Found: C, 87.21; H, 7.53; N, 5.62%.

4.1.3. Diphenyl-N-i-propylketenimine (4b)

From bis-*i*-propylaminodiphenylphosphonium bromide **3b** (1.90 g, 5 mmol). After concentration of the organic layer, petroleum ether (15 ml) is added to the residue affording the phosphinic amide **5b** as a white precipitate. After filtration (**5b** is isolated in a 95% yield: 1.22 g) the organic layer is concentrated to give the ketenimine **4** as a yellow solid. Reaction yield: 100%; isolated yield: 65%.

M.p. 49°C (recrystallization in petroleum ether (b.p. 45–60°C) at −30°C) {lit. [9] m.p. 45–46°C}. ^{1}H-NMR ($CDCl_3$) [10]: $\delta = 1.38$ (d, $J = 6.4$ Hz, 6H, CH_3), 3.94 (m, $J = 6.4$ Hz, 1H, C*H*N), 7.17–7.36 (m, 10H, 2C$_6H_5$). ^{13}C{^{1}H} ($CDCl_3$): $\delta = 23.72$ (s, 2C, *C*H$_3$), 55.11 (s, *C*HN), 79.10 (*C*=C=N), 125.82 (s, *p*-*C*, C$_6$H$_5$), 127.50 (s, *o*-*C*, C$_6$H$_5$), 128.11 (s, *m*-*C*, C$_6$H$_5$), 135.21 (s, *i*-*C*, C$_6$H$_5$), 184.0 (s, C=*C*=N). MS (EI): m/z (%) = 235 (36, M^+), 193 (100), 166 (99). Anal. Calc. for $C_{17}H_{17}N$ (235.33); C, 86.76; H, 7.28; N, 5.95. Found: C, 86.79; H, 7.23; N, 6.03%.

4.1.4. Diphenyl-N-t-butylketenimine (4c)

From bis-*t*-butylaminodiphenylphosphonium bromide **3c** (2.04 g, 5 mmol). The work-up is performed and the reaction stopped after 5 h at 65°C. After concentration of the organic layer, the residue is purified by chromatography on basic alumina to give the corresponding ketenimine **4c** (yield: 50%). (The diaminophosphonium monoazaylide **6c** is also obtained, not completely pure, with a yield around 30%.)

M.p. 49°C (lit. [10] m.p. 50°C). ^{1}H-NMR ($CDCl_3$) [10]: $\delta = 1.48$ (s, 9H, *t*-C$_4H_9$), 7.04–7.36 (m, 10H, 2C$_6H_5$). ^{13}C{^{1}H}-NMR ($CDCl_3$): $\delta = 29.75$ (s, 3C, *C*H$_3$), 54.25 (s, N–*C*), 78.15 (*C*=C=N), 125.79 (s, *p*-*C*, C$_6$H$_5$), 127.55 (s, *o*-*C*, C$_6$H$_5$), 128.21 (s, *m*-*C*, C$_6$H$_5$), 135.30 (s, *i*-*C*, C$_6$H$_5$), 183.55 (s, C=*C*=N). MS (EI): m/z (%) = 249 (4, M^+), 193 (100), 165 (54), 57 (38).

4.1.5. Diphenyl-N-benzylketenimine (4d) and N-(2,2-diphenylethenyl)benzenimine (8)

From bis-benzylaminodiphenylphosphonium bromide **3d** (2.38 g, 5 mmol). The work up is performed and the reaction stopped after 30 h at 20°C, when the main part of the ketenimine is transformed in the tautomer **8**. After concentration of the organic layer, petroleum ether (20 ml) is added to the residue, affording a mixture of phosphinic amide **5d** and tautomer **8** as a yellow precipitate. After filtration the petroleum ether is eliminated under vacuum to give the ketenimine **4d** as a yellow oil [11]. Yield: 25%. ^{1}H-NMR ($CDCl_3$) [11]: $\delta = 4.77$ (s, 2H, CH_2), 7.10–7.42 (m, 15H, 3C$_6H_5$). MS (EI): m/z (%) = 283 (25, M^+), 192 (35), 165 (38), 91 (100).

The yellow precipitate is chromatographed on basic alumina ((a) AcOEt/Hexane:10/90 to obtain **8**, and (b) MeOH to recover **5d**). Compound **8** is recrystallized in petroleum ether as a yellow solid (yield: 45%).

M.p. 129°C (petroleum ether: b.p. 45–60°C) (lit. [12] m.p. 131.5–133.5°C). IR (KBr): $\nu = 3420$, 1620 (C=N), 1550, 1480, 1440, 1380 cm^{-1}. ^{1}H-NMR ($CDCl_3$): $\delta =$ 7.33–7.54 (m, 13H, 2 C_6H_5 and *m*- and *p*- from CH–C_6H_5), 7.77–7.80 (m, 3H, 2H *o*- from CH–C_6H_5 and C*H*=CPh_2), 8.42 (s, 1H, C*H*–N). ^{13}C{^{1}H}-NMR ($CDCl_3$): $\delta = 127.5$ and 127.53 (2 s, *p*-*C*, Ph_2C), 127.77 (s, *o*-*C*, CH–C_6H_5), 128.32 and 128.68 (2s, *o*-*C*, Ph_2C), 128.81 and 128.87 (2s, *m*-*C*, Ph_2C), 130.94 (s, *p*-*C*, CH–C_6H_5), 131.74 (s, *m*-*C*, CH–C_6H_5), 136.71 (s, *i*-*C*, CH–C_6H_5), 138.74 and 140.96 (2s, *i*-*C*, Ph_2C), 140.0 (*C*H=CPh_2), 141.75 (CH=*C*Ph_2), 161.33 (*C*H=N). MS (EI): m/z (%) = 283 (62, M^+), 206 (100), 178 (32), 165 (24).

4.1.6. 2,2,4,4-Tetraphenyl cyclobutane-1,3-bis-N-allylimine (9)

From bis-allylaminodiphenylphosphonium bromide **3e** (1.88 g, 5 mmol). After concentration of the organic layer the residue is distillated under reduced pressure to give **9**. Yellow oil; yield: 85%.

B.p.$_{0.3}$ 142°C. IR (NaCl/film): $\nu = 3060$, 3037, 2962, 2940, 1740, 1667, 1617, 1510, 1467, 1208, 945 cm^{-1}. ^{1}H-NMR ($CDCl_3$): $\delta = 3.14$ (dt, $^3J = 6.9$ Hz, $^4J = 1.1$ Hz, 4H, CH_2N), 5.15–5.26 (m, 4H, CH=CH_2), 5.65–5.87 (m, 2H, C*H*=CH_2), 7.26–7.42 (m, 20H, 4C_6H_5). ^{13}C{^{1}H}-NMR ($CDCl_3$): $\delta = 44$ (*C*H_2N), 51.84 (*C*Ph_2), 120.45 (CH=*C*H_2), 122.08 (*C*=N), 127.10 (*m*-*C*, C_6H_5), 128 (*p*-*C*, C_6H_5), 128.91 (*o*-*C*, C_6H_5), 131.92 (*C*H=CH_2), 139.80 (*i*-*C*, C_6H_5). MS (EI): m/z (%) = 233 (12), 192 (100), 165 (90). FAB$^+$ (NBA) $m/z = 467$ ($M + 1$).

4.1.7. Triphenylketenimine (4f) and bis-phenylamino-diphenylphosphonium monoazaylide (6f)

From bis-phenylaminodiphenylphosphonium bromide **3f** (2.39 g, 5 mmol). After concentration of the organic layer the intermediate monoylide **6f** is precipitated by addition of petroleum ether (40 ml). After filtration the organic layer is concentrated to give **4f** as a yellow solid (yield: 20%).

M.p. 52°C (recrystallization in petroleum ether (b.p. 45–60°C), 3 days at −20°){lit. [13] m.p. 55°C}. ^{1}H-NMR ($CDCl_3$): $\delta = 7.22$–7.44 (m, 15H, 3C_6H_5). ^{13}C{^{1}H}-NMR ($CDCl_3$): $\delta = 78.01$ (*C*=C=N), 124.02 and 126.54 (2s, *o*-*C* and *m*-*C*, N–C_6H_5), 127.85 (*p*-*C*, N–C_6H_5), 127.88 (*o*-*C*, C–Ph_2), 128.89 (*m*-*C*, C–Ph_2), 129.59 (*p*-*C*, C–Ph_2), 134.0 (*i*-*C*, C–Ph_2), 140.70 (*i*-*C*, N–C_6H_5), 190.61 (C=*C*=N). MS (EI): m/z (%) = 249 (84, M^+), 192 (4), 165 (100). Anal. Calc. for $C_{18}H_{19}N$ (269.34); C, 89.18; H, 5.61; N, 5.20. Found: C, 88.89; H, 5.22; N, 4.98%.

6f is recrystallized in THF (yield: 75%).

M.p. 178–180°C (THF). IR (KBr): $\nu = 3400$, 3020, 2920, 2820, 1600, 1580, 1480, 1430, 1310–1300 (P=N), 1280, 1230 cm^{-1}. ^{31}P{^{1}H}-NMR ($CDCl_3$): $\delta = -1.6$ ppm. ^{1}H-NMR ($CDCl_3$): $\delta = 6.83$–7.12 (m, 10H, 2C_6H_5), 7.42–7.52 (m, 6H, Ph_2P), 7.88–7.99 (m, 4H, Ph_2P). ^{13}C{^{1}H}-NMR (DMSOD): $\delta = 125.13$ (*p*-C, N–C_6H_5), 126.03 (d, $^2J_{PC} = 11.3$ Hz, *o*-*C*, Ph_2P), 133.65 (d, $^3J_{PC} = 14.1$ Hz, *m*-*C*, Ph_2P), 133.79 (*m*-C, N–C_6H_5), 136.33 (d, $^1J_{PC}$, *i*-*C*, Ph_2P), 136.99 (*p*-*C*, Ph_2P), 137.08 (d, $^3J_{PC} = 9.8$ Hz, *o*-C, N–C_6H_5), 149.75 (*i*-*C*, N–C_6H_5). MS (FAB: GT): $m/z = 369$ ($M + 1$).

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