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Magnesium iodide promoted defluorinative reactions of 2,2-difluorocyclopropyl aryl ketones with aryl imines: A new, general synthesis of 2-alkylideneazetidines

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ABSTRACT

Upon treatment with anhydrous MgI_2, 2,2-difluorocyclopropyl aryl ketones undergo a ring-opening process leading to complete loss of fluorine. When carried out in the presence of aryl imines, the reaction leads to a novel synthesis of 2-alkylideneazetidenes. A fluorine-free allenyl ketone is proposed as an intermediate in these reactions.

1. Introduction

Recently we reported the acid catalyzed, regioselective ring-opening of fluorinated cyclopropyl ketones (i.e., **1** in Scheme 1) to give 3,3-difluoro-4-bromobutan-1-ones (**2**) [1]. Such reactions as this exemplify the synthetic exploitation of the inherent zwitterionic charge affinity of cyclopropyl ketone systems, as shown in Fig. 1.

In following up this work, we wished to determine whether it might be possible to devise a way to intercept this putative dipole in a condensation reaction using a carbonyl or imine reactant, leading essentially to an overall cycloaddition process.

There are a number of reports of such chemistry involving *non-fluorine*-substituted cyclopropyl ketones in Lewis acid catalyzed ring-opening condensation reactions with aldehydes or imines to prepare five-membered ring heterocycles. One good example is Carreira's MgI_2-catalyzed condensation of spiro[cyclopropan-1,3′-oxindol] **3** with *N*-aryl- and alkylsulfonyl aldimines (Scheme 2) [2]. Another example is Oshima's stepwise enolate formation via iodide ring-opening of phenyl cyclopropyl ketones, followed by aldol reaction and subsequent cyclization to form tetrahydrofurans (Scheme 3) [3]. Lastly there is the report by Olsson of the three component, magnesium iodide promoted synthesis of substituted pyrrolidines from cyclopropyl ketones (Scheme 4) [4].

Our intended strategy was to mimic the Olsson conditions using 2,2-difluorocyclopropyl ketones, with the expectation that either difluoropyrrolidines or fluoro-dihydropyrroles would be formed (Scheme 5). However, in the event, a completely unexpected and unprecedented reaction occurred, leading to products that were completely fluorine-free.

2. Results

When phenyl 2,2-difluoro-3-phenylcyclopropyl ketone (**4a**) was allowed to react with imine **5a** under Olsson conditions, the only isolable product was azetidin-2-ylidine compound (**6a**) in a modest 32% yield (Scheme 6). The structure of **6a** was consistent with the ^1H and ^{13}C NMR spectral data shown in Fig. 2.

Reactions of **4a** with other bis-aromatic imines led to similar results, as shown in Table 1 (column 7), and X-ray crystal structure characterization was carried out on product **6i** (Fig. 3).

In considering the possible mechanism for the reaction, which probably required multiple iodide involvement in the reaction, optimization experiments were carried out utilizing excess MgI_2, with the reaction proceeding to give considerably enhanced yields when using four equivalents of MgI_2 (final column, Table 1). It was also found that best results were obtained using freshly prepared anhydrous MgI_2 via the reaction of Mg powder with I_2 in refluxing anhydrous diethyl ether.

Only diaryl imines were utilized in this study, largely because of ease of preparation and stability. The one ketone substrate not

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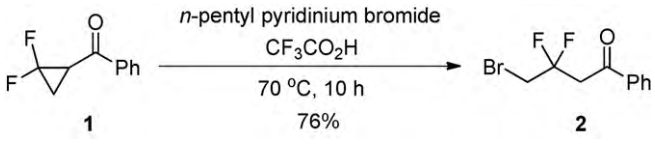


Scheme 1.

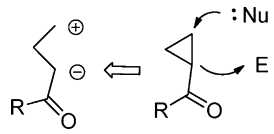
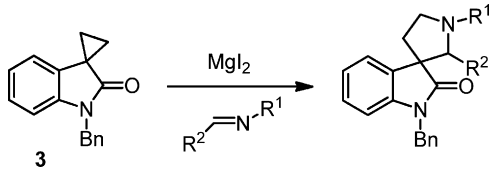
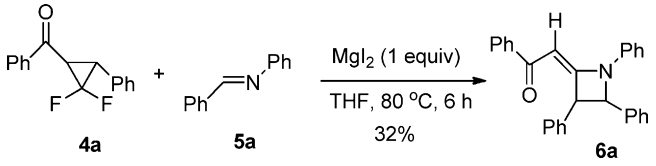


Fig. 1. Inherent zwitterionic charge affinity of cyclopropyl ketones.



Scheme 2.



Scheme 6.

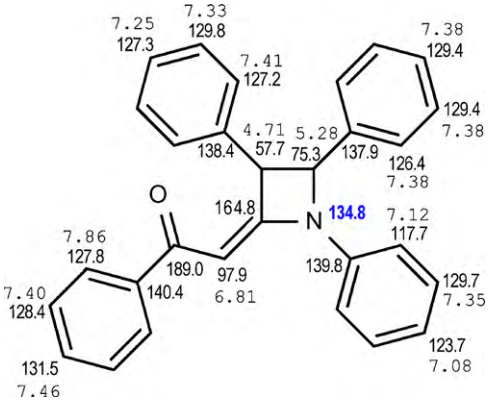


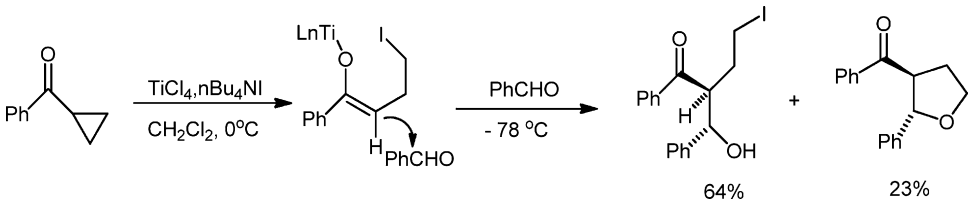
Fig. 2. ^{1}H and ^{13}C NMR data for azetidinylidene product **6a**.

bearing a phenyl substituent on the cyclopropane ring (**4c**) underwent the reaction similarly, but with a reduced yield (Scheme 7).

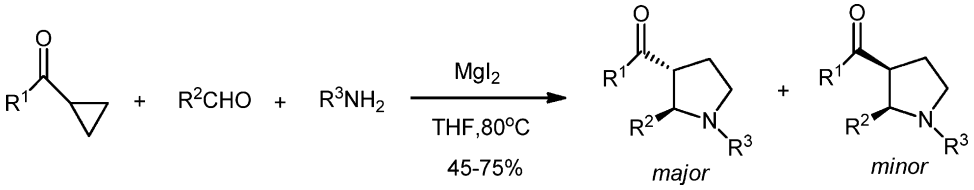
Lastly, in an experiment designed to provide mechanistic insight, the reaction was carried out under otherwise identical conditions, but in the absence of an imine, with the result given in Scheme 8. The structure of iodofuran product **7** was confirmed by comparison of its ^{1}H and ^{13}C NMR spectral data (Fig. 4) with those reported for the compound [5]. Iodofuran **7** could also be detected as a very minor product in many of the other reactions of **4a** described in Table 1, although it was never isolated.

3. Discussion

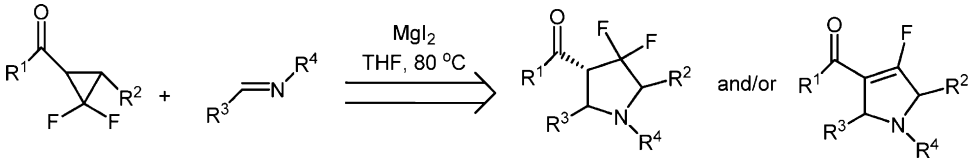
A reasonable mechanism for the observed results requires iodide ion to act both as a nucleophile and as a reducing agent during the course of the reaction. Also, an allenyl ketone (**10**) is a likely intermediate in the reaction. The action of iodide ion as a nucleophile in the initial Mg^{2+}-catalyzed ring-opening process to form enolate intermediate **8**, which then undergoes elimination of one of the fluorines to form iodide intermediate **9** is consistent with the similar ring-opening results depicted in Schemes 2–4. Unique to our system, and distinctly different from the ultimate regiochemistry of imine attack that is observed in these earlier



Scheme 3.

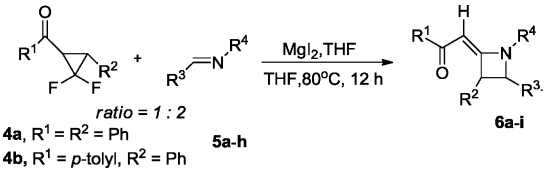


Scheme 4.



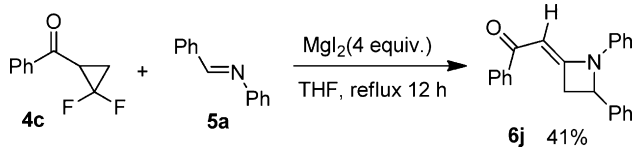
Scheme 5.

Table 1
MgI_2-induced reactions of 2,2-difluorocyclopropyl ketones **4** with imines **5**.

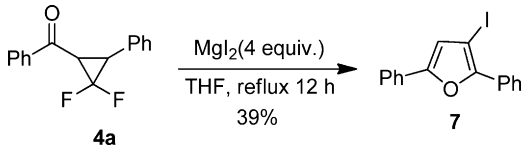


Entry	Ketone	Imine	R^3	R^4	Product	Yield (%)[a] 1 equiv. MgI_2	Yield (%)[a] 4 equiv. MgI_2
1	**4a**	**5a**	C_6H_5	C_6H_5	**6a**	32	57
2	**4a**	**5b**	4-MeC_6H_4	C_6H_5	**6b**	13	59
3	**4a**	**5c**	4-$MeOC_6H_4$	C_6H_5	**6c**	33	60
4	**4a**	**5d**	4-ClC_6H_4	C_6H_5	**6d**	27	60
5	**4a**	**5e**	4-BrC_6H_4	C_6H_5	**6e**	21	64
6	**4a**	**5f**	4-FC_6H_5	C_6H_5	**6f**	23	61
7	**4a**	**5g**	C_6H_5	4-MeC_6H_4	**6g**	30	60
8	**4a**	**5h**	C_6H_5	4-ClC_6H_4	**6h**	33	66
9	**4b**	**5a**	C_6H_5	C_6H_5	**6i**	35	49

[a] Isolated yields.



Scheme 7.



Scheme 8.

results is the proposed reductive β-elimination of intermediate **9** by iodide ion to eliminate the second fluorine and form allene intermediate **10**. Molecular iodine would be formed in this step, and this is reflected by the increasingly red color of the reaction mixture, which color is removed by washing with $NaHSO_3$ during work-up of the reaction.

In the absence of imine, the allenyl ketone apparently undergoes electrophilic attack at the central allenic carbon of **10** by I_2, either synchronous with cyclization or via intermediate **11** with subsequent cyclization to form intermediate **12**, which deprotonates to form iodofuran product **7**.

In the presence of imine, allenyl ketone intermediate **10** undergoes Michael attack by the nucleophilic nitrogen of the imine to form enolate, zwitterionic intermediate **13**, which can undergo cyclization to form the much more thermodynamically-stable of the two potential azetidinylidene products, **6a** (Scheme 9).

Alternatively, intermediate **9** might itself undergo Michael addition of imine, followed by fluoride loss to form intermediate **14**, which upon reductive elimination of iodine would lead to intermediate **13** and subsequent cyclization to product **6a** (Scheme 10).

The results were unexpected mainly because fluoride is generally observed to be a poor leaving group, and in this reaction two fluorides have been lost during the course of the reaction. Perhaps that is why these reactions proceed at best with only modest yields. Thus, it would be interesting to see whether the dichloro analogues of **4** might well produce better yields in this reaction. In fact, 2,2-dichlorocyclopropyl ketones are even less well known than the difluoro ones, and although they have been observed to undergo ring-opening reactions in the presence of nucleophiles, apparently by elimination-addition mechanisms (Scheme 11) [6,7], no reactions under Lewis acid catalysis have been reported.

Compounds with the structural features present in our observed products (**6a–j**) could well be of pharmaceutical/medicinal interest, but they have thus far only infrequently been mentioned in the literature. There are a few papers related to the synthesis of 2-methyleneazetidines, where they have been

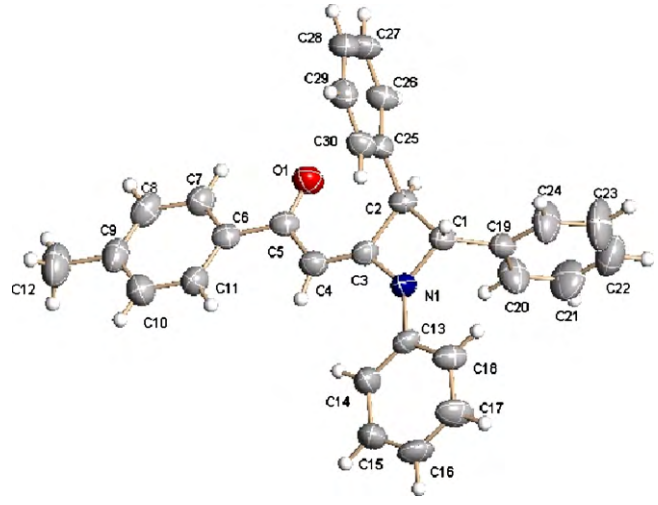


Fig. 3. Ortep drawing of azetidinylidine product **6i**.

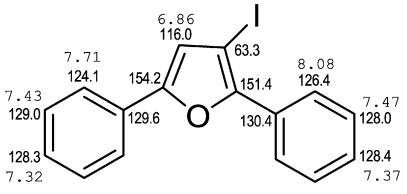
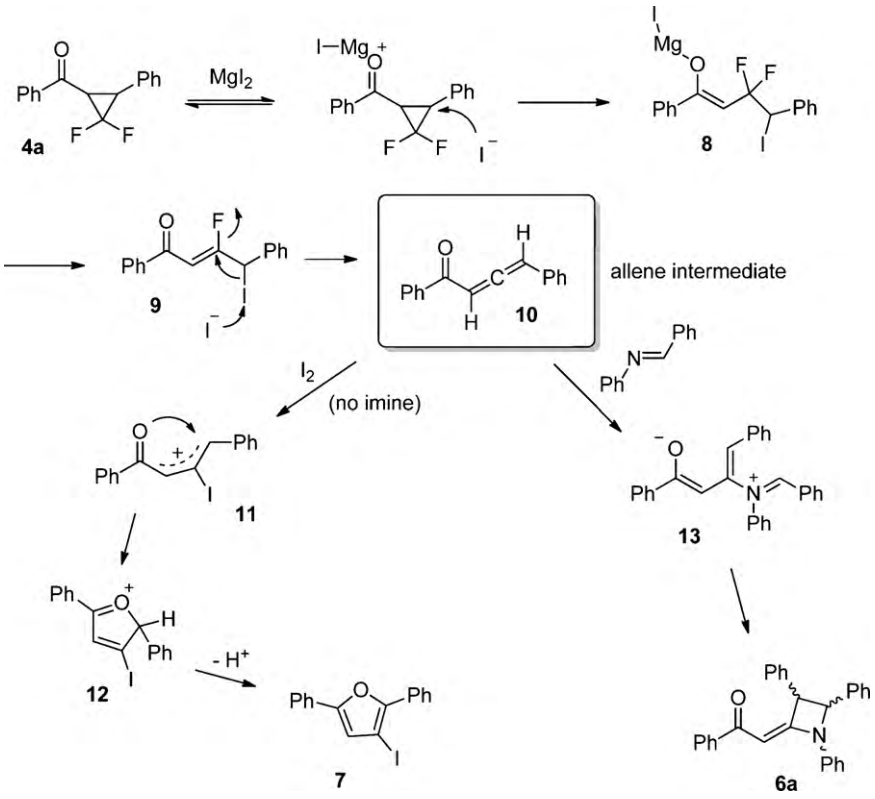
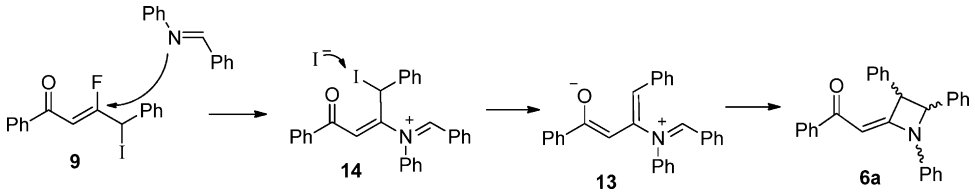


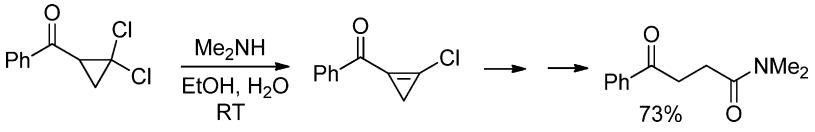
Fig. 4. ^1H and ^{13}C NMR data for iodofuran 7.



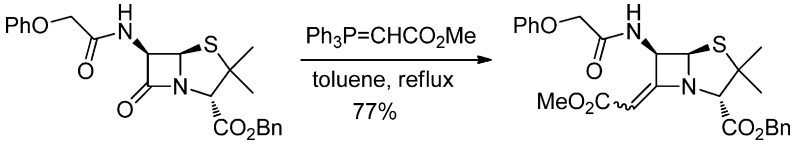
Scheme 9. Proposed mechanism.



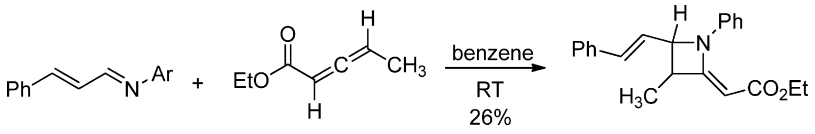
Scheme 10. Alternative, allene-free mechanism for formation of azetidene products.



Scheme 11.



Scheme 12.



Scheme 13.

prepared either by methylenation of β-lactams using dimethyltitanocene [8,9], or by cyclization reactions [10–12]. 2-Alkylideneazetidine compounds with a conjugated enone structure like those in **6** are even less frequently encountered. They have been prepared from β-lactams through reactions with stabilized Wittig reagents, as in making the penicillin derivative shown in Scheme 12 [13,14], and they have also been prepared by the [2 + 2] cycloaddition of a conjugated aldimine with an allenyl ketone (Scheme 13) [15], a reaction that resembles closely our own proposed mechanistic process.

4. Conclusions

Thus, an attempt to synthesize fluorine substituted five-membered-ring heterocycles via Lewis acid catalyzed ring-opening reactions of aldimines with 2,2-difluorocyclopropyl ketones led, instead, to complete loss of fluorine from these compounds with the formation of novel 2-alkylideneazetidinyl derivatives. The observed reactions likely proceed via [2 + 2] cycloaddition of the imines to an allenyl ketone intermediate, and they should constitute a good, general method for synthesis of this potentially interesting molecular system.

5. Experimental

5.1. Materials and general information

Unless otherwise specified, proton and carbon NMR spectra were obtained in $CDCl_3$ at 300 and 75.5 and 282 MHz, respectively, and chemical shifts are reported upfield relative to TMS. All 2,2-difluorocyclopropyl ketones have been previously reported and were prepared by the reaction of difluorocarbene reagent, trimethylsilyl 2-fluorosulfonyl-2,2-difluoroacetate (TFDA), with the respective α,β-unsaturated ketones [1,16]. All imines were prepared in the usual manner from corresponding aldehydes and anilines in ethanol under reflux.

5.2. Magnesium (II) iodide

Under nitrogen, in a dry 100 mL of three-necked flask were added magnesium powder (2.43 g, 100 mmol) and anhydrous ethyl ether (50 mL). Iodine (5 g, 20 mmol) was added portwise to keep a gentle reflux. After addition, the refluxing was continued until the color of iodine disappeared. The hot mixture was filtered under nitrogen and the solution was cooled to −20 °C. The precipitated white solid was collected and dried in a vacuum container.

5.3. Typical procedure for reaction of 2,2-difluorocyclopropyl ketones with imines

Under nitrogen, into a 50 mL three-necked flask were added phenyl cyclopropyl ketone (0.258 g, 1 mmol), *N*-benzylideneaniline (181 mg, 1 mmol) and anhydrous tetrahydrofuran (10 mL). Magnesium iodide (1.016 g, 4 mmol) was added to the solution in one portion with stirring. The mixture was heated to reflux and stirred for 12 h, during which time the reaction became increasing red in color. After cooling to room temperature, 5 mL of saturated sodium thiosulfate solution was added, and the reaction mixture was extracted with diethyl ether (3× 20 mL). The combined organic layers were dried over sodium sulfate, filtered, and the solvent removed with a rotary evaporator. The residue was purified by silica flash column chromatography.

5.3.1. (E)-1-Phenyl-2-(1,3,4-triphenylazetidin-2-ylidene)ethanone, 6a

57%; yellow solid, mp 162 °C, ^{1}H NMR, δ 4.70 (t, *J* = 1.5 Hz, 1H), 5.27 (d, *J* = 2.1 Hz, 1H), 6,80 (d, *J* = 1.5 Hz, 1H), 7.05–7.12 (m, 3H), 7.24–7.45 (m, 15H), 7.83–7.86 (m, 2H); ^{13}C NMR, δ 57.8, 75.3, 91.8, 117.7, 123.7, 126.4, 127.2, 127.4, 127.8, 128.4, 128.9, 129.0, 129.4, 129.7, 131.5, 137.8, 138.4, 139.8, 140.5, 164.7, 189.0; HRMS (EI) calcd for $C_{29}H_{23}NO$, 401.1780, found 401.1781. Anal. Calcd for $C_{29}H_{23}NO$: C, 86.75; H, 5.77; N, 3.49. Found: C, 86.59; H, 5.46; N, 3.30.

5.3.2. (E)-1-Phenyl-2-(1,3-diphenyl-4-(4-methylphenyl)azetidin-2-ylidene)ethanone, 6b

59%; yellow solid, mp 153 °C, ^{1}H NMR, δ 2.35 (s, 3H), 4.69 (t, *J* = 1.5 Hz, 1H), 5.24 (d, *J* = 2.4 Hz, 1H), 6,79 (d, *J* = 1.5 Hz, 1H), 7.04–7.45 (m, 17H), 7.84–7.87 (m, 2H); ^{13}C NMR, δ 57.9, 75.3, 91.7, 117.8, 123.6, 126.4, 127.2, 127.3, 127.8, 128.4, 128.9, 129.7, 130.1, 131.5, 134.8, 138.5, 138.8, 139.8, 140.6, 164.8, 189.0; HRMS (EI) calcd for $C_{30}H_{25}NO$, 415.1936, found 415.1942. Anal. Calcd for $C_{30}H_{25}NO$: C, 86.71; H, 6.06; N, 3.37. Found: C, 86.81; H, 6.25; N, 3.17.

5.3.3. (E)-1-Phenyl-2-(1,3-diphenyl-4-(4-methoxyphenyl)azetidin-2-ylidene)ethanone, 6c

60%; yellow solid, mp 153 °C, ^{1}H NMR, δ 3.81 (s, 3H), 4.70 (t, *J* = 1.5 Hz, 1H), 5.23 (d, *J* = 2.4 Hz, 1H), 6.78 (d, *J* = 1.5 Hz, 1H), 6.88–6.91(m, 2H), 7.07–7.13 (m, 3H), 7.22–7.43 (m, 12H), 7.84–7.87 (m, 2H); ^{13}C NMR, δ 55.5, 57.9, 75.1, 91.6, 114.8, 117.8, 123.6, 127.2, 127.3, 127.8, 127.9, 128.4, 128.9, 129.6, 129.8, 131.5, 138.4, 139.7, 140.6, 160.1, 164.8, 189.0; HRMS (EI) calcd for $C_{30}H_{25}NO_2$, 431.1885, found 431.1849. Anal. Calcd for $C_{30}H_{25}NO_2$: C, 83.50; H, 5.84; N, 3.25. Found: C, 83.18; H, 5.72; N, 3.18.

5.3.4. (E)-1-Phenyl-2-(1,3-diphenyl-4-(4-chlorophenyl)azetidin-2-ylidene)ethanone, 6d

60%; solid, mp 168 °C, ^{1}H NMR, δ 4.66 (t, *J* = 1.8 Hz, 1H), 5.25 (d, *J* = 2.1 Hz, 1H), 6.79 (d, *J* = 1.5 Hz, 1H), 7.06–7.11 (m, 3H), 7.24–7.45 (m, 14H), 7.83–7.86 (m, 2H); ^{13}C NMR, δ 57.7, 74.5, 92.0, 117.6, 123.8, 127.2, 127.5, 127.8, 128.5, 129.0, 129.7, 129.8, 131.6, 134.8, 136.3, 138.1, 139.5, 140.3, 164.4, 189.1; HRMS (EI) calcd for $C_{29}H_{22}ClNO$, 435.1390, found 435.1407. Anal. Calcd for $C_{29}H_{22}ClNO$: C, 79.90; H, 5.09; N, 3.21. Found: C, 79.97; H, 5.12; N, 3.03.

5.3.5. (E)-1-Phenyl-2-(1,3-diphenyl-4-(4-bromophenyl)azetidin-2-ylidene)ethanone, 6e

64%; solid; ^{1}H NMR, δ 4.66 (t, *J* = 1.5 Hz, 1H), 5.23 (d, *J* = 2.4 Hz, 1H), 6,79 (d, *J* = 1.5 Hz, 1H), 7.06–7.11 (m, 3H), 7.24–7.45 (m, 14H), 7.83–7.86 (m, 2H); ^{13}C NMR, δ 57.7, 74.6, 92.0, 117.6, 122.9, 123.9, 127.2, 127.5, 127.8, 128.1, 129.0, 129.8, 131.6, 132.6, 136.8, 138.1, 139.5, 140.0, 164.3, 189.1; HRMS (EI) calcd for $C_{29}H_{22}BrNO$, 479.0885, found 479.0909. Anal. Calcd for $C_{29}H_{22}BrNO$: C, 72.50; H, 4.62; N, 2.92. Found: C, 72.43; H, 4.67; N, 2.85.

5.3.6. (E)-1-Phenyl-2-(1,3-diphenyl-4-(4-fluorophenyl)azetidin-2-ylidene)ethanone, 6f

61%; solid; ^{1}H NMR, δ 4.66 (t, *J* = 1.5 Hz, 1H), 5.23 (d, *J* = 2.4 Hz, 1H), 6,79 (d, *J* = 1.5 Hz, 1H), 7.06–7.11 (m, 3H), 7.22–7.52 (m, 14H), 7.83–7.85 (m, 2H); ^{13}C NMR, δ 57.8, 74.5, 91.9, 116.3, 116.6, 117.7, 123.8, 127.1, 127.5, 127.8, 128.1, 128.2, 128.4, 129.0, 129.7, 131.6, 133.6, 138.1, 139.5, 140.4, 164.4, 189.0; HRMS (EI) calcd for $C_{29}H_{22}FNO$, 419.1685, found 419.1667. Anal. Calcd for $C_{29}H_{22}FNO$: C, 83.03; H, 5.29; N, 3.34. Found: C, 82.88; H, 5.42; N, 3.23.

5.3.7. (E)-1-Phenyl-2-(1-(4-methylphenyl)-1,3-diphenylazetidin-2-ylidene)ethanone, 6g

59%; yellow solid, mp 153 °C, ^{1}H NMR, δ 2.32 (s, 3H), 4.71 (t, *J* = 1.2 Hz, 1H), 5.27 (d, *J* = 2.4 Hz, 1H), 6.76 (d, *J* = 1.2 Hz, 1H), 7.02–7.04 (m, 2H), 7.14–7.17 (m, 2H), 7.25–7.46 (m, 13H), 7.85–7.88 (m, 2H); ^{13}C NMR, δ 57.8, 75.3, 91.3, 117.8, 126.4, 127.2, 127.4, 123.8, 128.4, 128.9, 128.9, 129.4, 130.2, 131.4, 133.5, 137.3, 137.9, 138.4,

140.6, 164.8, 188.9; HRMS (EI) calcd for $C_{30}H_{25}NO$, 415.1936, found 415.1940. Anal. Calcd for $C_{30}H_{25}NO$: C, 86.71; H, 6.06; N, 3.37. Found: C, 86.62; H, 6.15; N, 3.35.

5.3.8. (E)-1-Phenyl-2-(1-(4-chlorophenyl)-1,3-diphenylazetidin-2-ylidene)ethanone, **6h**

66%; solid, mp 168 °C; ^{1}H NMR, δ 4.71 (t, J = 1.2 Hz, 1H), 5.23 (d, J = 2.4 Hz, 1H), 6,73 (d, J = 1.2 Hz, 1H), 7.00–7.03 (m, 2H), 7.24–7.46 (m, 15H), 7.82–7.85 (m, 2H); ^{13}C NMR, δ 57.8, 75.4, 92.2, 118.8, 126.4, 127.2, 127.5, 127.8, 128.5, 128.8, 129.0, 129.0, 129.5, 129.8, 131.6, 137.4, 138.1, 138.3, 140.3, 164.3, 188.9; HRMS (EI) calcd for $C_{29}H_{22}ClNO$, 435.1390, found 435.1398. Anal. Calcd for $C_{29}H_{22}ClNO$: C, 79.90; H, 5.09; N, 3.21. Found: C, 79.81; H, 5.06; N, 3.17.

5.3.9. (E)-1-(4-Methylphenyl)-2-(1,3,4-triphenylazetidin-2-ylidene)ethanone, **6i**

49%; solid, mp 146 °C; ^{1}H NMR, δ 2.37 (s,3H), 4.69 (m, 1H), 5.25 (d, J = 2.4 Hz, 1H), 6,78 (d, J = 1.5 Hz, 1H), 7.03–7.40 (m, 17H), 7.74–7.77 (m, 2H); ^{13}C NMR, δ 21.8, 57.7, 75.2, 91.8, 117.6, 123.5, 126.4, 127.2, 127.3, 127.9, 128.9, 129.1, 129.4, 129.6, 137.8, 137.9, 138.4, 139.8, 141.9, 164.3, 188.7; HRMS (EI) calcd for $C_{30}H_{26}NO[M+H]^+$, 416.2006, found 416.1994. Anal. Calcd for $C_{30}H_{26}ClNO$: C, 86.71; H, 6.06; N, 3.37. Found: C, 86.31; H, 6.17; N, 3.23.

5.3.10. (E)-1-Phenyl-2-(1,4-diphenylazetidin-2-ylidene)ethanone, **6j**

41%; solid, mp 131 °C, ^{1}H NMR, δ 3.40(dddd, J = 1.5 Hz, 2.7 Hz, 16.5 Hz, 1H), 3.95 (dddd, J = 1.5 Hz, 5.7 Hz, 16.5 Hz, 1H), 5.54 (dd, J = 2.7 Hz, 5.7 Hz, 1H), 6.68 (t, J = 1.5 Hz, 1H), 7.00–7.06 (m, 3H), 7.28–7.50 (m, 10H), 7.93–7.96 (m, 2H); ^{13}C NMR, δ 21.8, 57.7, 75.2, 91.8, 117.6, 123.5, 126.4, 127.2, 127.3, 127.9, 128.9, 129.1, 129.4, 129.6, 137.8, 137.9, 138.4, 139.8, 141.9, 164.3, 188.7; HRMS (EI) calcd for $C_{23}H_{20}NO[M+H]^+$, 326.1539, found 326.1542. Anal. Calcd for $C_{30}H_{26}ClNO$: C, 84.89; H, 5.89; N, 4.30. Found: C, 84.91; H, 6.06; N, 4.23.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jfluchem.2010.06.021.

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