Catalytic Preparation of Aziridines with an Iron Lewis Acid

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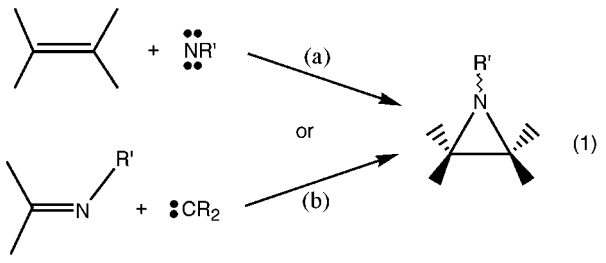
The iron Lewis acid, $[(\eta^5\text{-}C_5H_5)Fe(CO)_2(THF)]^+[BF_4]^-$, was found to be an effective catalyst for the preparation of aziridines. This new method provides a facile, one-step route to predominantly *cis*-aziridines, with yields up to 95%, from compounds with a diazo functionality and a variety of substituted *N*-benzylidene imines with *N*-aryl or *N*-alkyl groups. The reaction mechanism is believed to proceed through an electrophilic iminium ion intermediate. To support this idea, the iron Lewis acid−imine complex $[(\eta^5\text{-}C_5H_5)Fe(CO)_2(PhCH{=}NPh)]^+[BF_4]^-$ was prepared, characterized, and reacted with different diazo compounds to provide the resultant *cis*-aziridines. Alternatively, it may be possible that the aziridines were derived from an electrophilic carbenoid intermediate, as is often proposed. Thus, the iron carbene $[(\eta^5\text{-}C_5H_5)Fe(CO)_2(CHPh)]^+[SO_3CF_3]^-$ was prepared and treated with *N*-benzylideneaniline; however, the resultant aziridine was not formed.

Introduction

The iron Lewis acid, $[(\eta^5\text{-}C_5H_5)Fe(CO)_2(THF)]^+[BF_4]^-$ (**1**), has been shown to be an effective catalyst for inducing reactions such as Diels−Alder,[1] cyclopropanation,[2] and epoxidation,[3] yet the catalytic potential of **1** has not been fully explored. Building upon these precedents, the use of the catalyst has now been extended to the synthesis of aziridines. After the recent proliferation of reactions within the epoxide family, the aziridine functionality has become more attractive as another versatile intermediate in organic synthesis. Many biologically important moieties have been derived from aziridines, such as amino acids, β-lactam antibiotics, and alkaloids.[4] Several noncatalytic methods are available for the preparation of aziridines,[5] including ring closure of 1,2-amino alcohols,[6] ring opening of epoxides by sodium azide followed by reaction with triphenylphosphine,[7] and also the addition of α-halo ester enolates to *N*-trimethylsilyl imines.[8]

Two contrasting pathways exist for the catalytic preparation of this three-membered ring (eq 1). One methodology (pathway a) involves the transfer, mostly by copper salts, of a nitrenoid intermediate from the precursor [*N*-(*p*-toluenesulfonyl)imino]phenyliodinane (PhI=NTs) to an olefin substrate;[9] however, transfers mediated by (salen)manganese(III) complexes have also been reported.[10] Chloramine-T was also used as a nitrogen source in a catalytic transfer to a variety of alkenes.[11] In contrast, another methodology (pathway b) involves the transfer of a carbenoid intermediate from a diazo compound to an imine substrate.[12] Both pathways have received much attention, but the focus of this paper will be on the latter pathway. Recently, catalytic synthesis of aziridines with common Lewis acids was reported; however, an alternative mechanism was suggested that does not proceed through a carbenoid intermediate but rather through an iminium ion intermediate.[13] Herein, we confirm that an iron Lewis acid-activated iminium ion undergoes attack by diazo nucleophiles to produce predominantly *cis*-aziridines.



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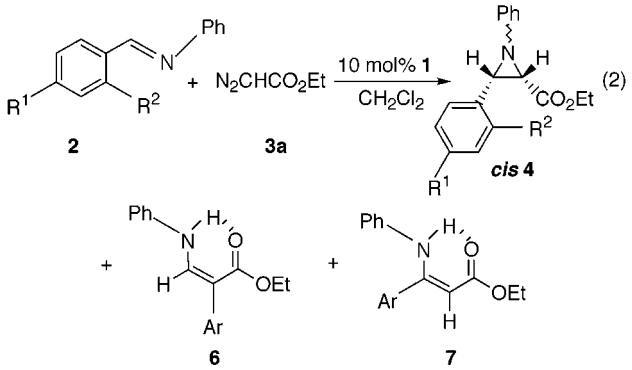
Results and Discussion

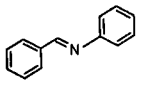
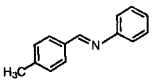
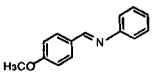
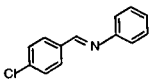
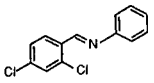
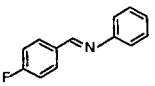
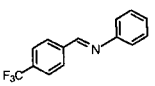
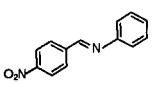
The iron Lewis acid (**1**) in catalytic concentrations was observed to induce a reaction between ethyl diazoacetate (EDA, **3a**) and different imines to form aziridines **4**, (eq 2). In the presence of 10 mol % of **1**, EDA in excess was



a) $R^1 = R^2 = H$ b) $R^1 = CH_3$, $R^2 = H$ c) $R^1 = OCH_3$, $R^2 = H$
d) $R^1 = Cl$, $R^2 = H$ e) $R^1 = R^2 = Cl$ f) $R^1 = F$, $R^2 = H$
g) $R^1 = CF_3$, $R^2 = H$ h) $R^1 = NO_2$, $R^2 = H$

found to consume all of the imine compound. In the crude reaction mixture, different β-amino-α,β-unsaturated ester byproducts (AUEs, **6** and **7**) and diethyl maleate and fumarate were also observed. When EDA and *N*-benzylideneaniline (**2a**) were reacted without the catalyst **1** under similar conditions, neither the aziridine nor any byproducts were formed. The catalyst **1** and the imine **2a** were mixed; however, no catalytic transformation occurred. When the iron Lewis acid **1** was treated with EDA at 40 °C for 12 h, the isolated reaction mixture contained 98% diethyl maleate and fumarate in a 5:1 ratio.[2b]

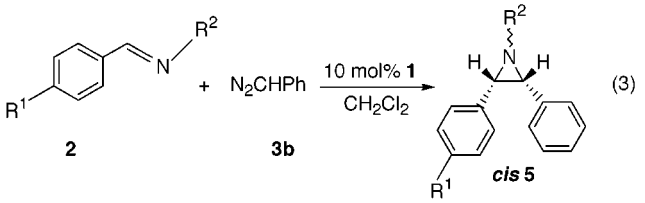
Table 1. Yields of Aziridines Prepared from Various Imines and EDA[a]

Entry	Imine (2)	eq. EDA (3a)	*cis*:*trans*	% Yield 4[b,c,d]
1		1	all *cis*	40
2		1.25	-----	0[e]
3		1	-----	0[e]
4		1.25	all *cis*	50
5		1.25	1 : 3	38
6		1.25	all *cis*	44
7		1.25	all *cis*	53
8		2	4 : 1	78

[a] All reactions were carried out in CH_2Cl_2 at rt for 18 h. [b] Isolated yields based upon the imine. [c] A mixture of AUE byproducts was isolated with yields ranging from 10-40%, additionally, 5-10% of the imine decomposed to the corresponding aldehyde and aniline. [d] Diethyl maleate and fumarate were isolated from the crude in a 5-10% yield. [e] In addition to the AUE byproducts, other unidentified compounds were also formed.

Different imine substrates were prepared in order to determine the effects that various substitutions would make upon the overall yield of aziridine and its cis-to-trans isomeric ratio. These results are summarized in Table 1. The unsubstituted imine **2a** provided a moderate 40% yield of *cis*-aziridine with no *trans* isomer, along with the AUE byproducts (entry 1). In most cases, the *cis*-aziridine was produced exclusively, without formation of the *trans* isomer. However, when chlorine was substituted at the *ortho* and *para* positions, the *trans* isomer was more prevalent, possibly due to a steric interaction (entry 5). Electron-donating groups were substituted at the *para* position on the benzylidene portion of the imine, and it was found that the aziridines were not the preferred product, but rather the AUE byproducts were formed in addition to other unidentified compounds (entries 2 and 3). Electron-withdrawing groups were substituted at the *para* position, and it was found that the yield of the aziridines increased while the yield of the AUE byproducts decreased. The *p*-nitroimine **2h** provided the greatest yield of aziridine, 78%, but it was a 4:1 mixture of *cis* and *trans* (entry 8).

In an effort to increase the yield of the aziridine product while broadening the scope of this catalytic reaction, a more nucleophilic diazo compound was employed. It was found that phenyl diazomethane (PDM, **3b**) is an excellent reagent for this transformation (eq 3). Utilizing the same reaction conditions, 10 mol % of

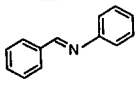
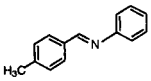
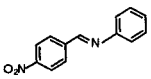
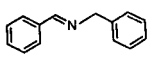
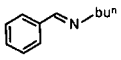


a) $R^1 = H$, $R^2 = Ph$ b) $R^1 = CH_3$, $R^2 = Ph$ h) $R^1 = NO_2$, $R^2 = Ph$
i) $R^1 = H$, $R^2 = Bn$ j) $R^1 = H$, $R^2 = bu^n$

1 and the drop-by-drop addition of PDM at room temperature over several hours, the yield of *cis*-aziridine (**5**) dramatically increased without production of the *trans* isomer in all cases (Table 2). Imine **2b** with a *p*-methyl substituent on the benzylidene moiety was found to provide a nearly quantitative (95%) transformation to the *cis*-aziridine (entry 2). Thus, PDM provides high yields of *cis*-aziridines when electron-withdrawing or electron-donating groups are substituted at the *para* position of the benzylidene moiety on the imine. Interestingly, the AUE byproducts observed when EDA was used as the diazo functionality source were not observed in the cases involving PDM.

Two plausible reaction mechanisms may be envisaged that are analogous to those already proposed in the literature. The most commonly postulated mechanism for reactions of this type involves a carbenoid intermedi-

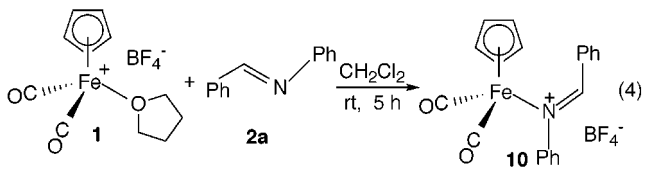
Table 2. Yields of Aziridines Prepared from Various Imines and PDM[a]

Entry	Imine (2)	eq. PDM (3b)	*cis*:*trans*	%Yield 5[b]
1		1.5	all *cis*	95
2		1.5	all *cis*	95
3		1.5	all *cis*	81
4		1.5	all *cis*	75
5		1.5	all *cis*	72

[a]All reactions were carried out in CH_2Cl_2 at rt for 18 h. [b]Isolated yields based upon the imine.

ate due to the appearance of carbene coupling products of the diazo compound.[14] In an effort to ascertain whether an aziridine may be derived through a pathway involving the reaction of an iron carbene and an imine, $[(\eta^5\text{-}C_5H_5)Fe(CHPh)(CO)_2]^+[SO_3CF_3]^-$ (**8**) was prepared[15] and treated with imine **2a**. The resultant *cis*-1,2,3-triphenylaziridine (**5a**) was not formed. However, in accord with a mechanism postulated by Brookhart and Templeton,[13] we propose a mechanism that proceeds through an iminium ion intermediate (Scheme 1). In this mechanism, **1** dissociates the THF ligand to form the active catalytic species, $[CpFe(CO)_2]^+$ (**9**). The imine coordinates to the iron Lewis acid, producing an electrophilic iminium ion (**10**). In turn, a nucleophilic diazo compound (**3**) may attack to produce intermediate **11**, which undergoes ring closure to form the aziridine **4** or **5** and regenerates the active catalyst (**9**). The AUE byproducts are formed as a result of 1,2-aryl (**6**) or hydrogen (**7**) migration within intermediate **11** to yield an imine byproduct only when EDA is used. Apparently, these imine byproducts tautomerize to enamines, the observed byproducts **6** and **7**.

To provide evidence for this mechanism, we attempted to prepare, isolate, and characterize the intermediate of this pathway, iminium ion **10**. It was prepared by mixing **1** with an excess of imine **2a** in a CH_2Cl_2 solution and then stirring at room temperature for 5 h, during which time the solution changed from red to orange. Extraction and recrystallization of the crude provided an atmosphere-sensitive, tan powder (eq 4). The resulting product



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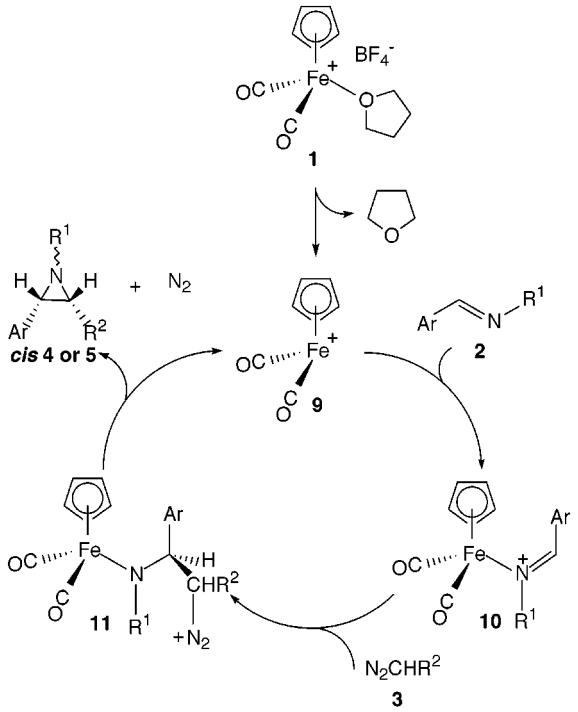
provided ^{1}H and ^{13}C NMR spectra consistent with the structure of the imine-bound iron Lewis acid complex

Scheme 1



(**10**). In the ^{1}H NMR spectrum, the shift of the benzylic proton was moved downfield from $\delta = 8.48$ to $\delta = 8.73$ ppm by coordination to the iron Lewis acid. The ^{13}C spectrum showed a downfield shift for the benzylic carbon as well, from 160.2 to 186.4 ppm, a deshielding effect due to a σ-donation by nitrogen to the positive metal center.[16] Additionally, the IR spectrum was consistent with an η^1-bound imine dicarbonyl iron Lewis acid, where the C=N stretching frequency was found to decrease from 1628 to 1603 cm^{-1} upon coordination to the iron Lewis acid.[16]

Further evidence was provided for the mechanism depicted in Scheme 1 by treatment of the iminium ion complex **10** with an excess of EDA. The crude was worked up as usual and found to contain the *cis*-aziridine **4a** in addition to the AUE byproducts **6a** and **7a**. By integration of the proton NMR, the ratio of aziridine **4a** to the byproducts (**6a** + **7a**) was 2.3:1, approximately equal to the 2:1 product ratio for the catalytic reaction, and the ratio of **6a** to **7a** was 5:1, again the same product distribution. Furthermore, intermediate **10** was dissolved in $CDCl_3$ in an NMR tube and treated with PDM. The resultant *cis*-1,2,3-triphenylaziridine (**5a**) was observed to form within a couple minutes.

Formation of the carbene coupling products of ethyl diazoacetate and phenyldiazomethane suggests that the pathway to the aziridines may also proceed through an iron carbene. However, the failure of the iron carbene **8** to produce the aziridine **5a** when treated with imine **2a** does not support a carbene mechanism. Also, the preparation and subsequent reactions of the activated iminium ion intermediate **10** to produce the *cis*-aziridines and byproducts in the same proportion as in the catalytic reactions provide additional new evidence that the pathway to aziridines does not involve an iron carbene, despite formation of the dimers. The appearance of the

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(16) Similar organometallic−imine complexes have been previously reported; see: Faller, J. W.; Ma, Y.; Smart, C. J.; DiVerdi, M. J. *J. Organomet. Chem.* **1991**, *420*, 237−252.

dimer byproducts in small quantities can be attributed mainly to the excess of the diazo compound.

In summary, we have extended the chemistry of $[(\eta^5\text{-}C_5H_5)Fe(CO)_2(THF)]^+[BF_4]^-$ (**1**) to include catalysis of aziridine preparation. For the first time, **1** in catalytic quantities was shown to induce reactions between a variety of substituted aryl imines and different diazo compounds to produce aziridines with yields up to 95%, in most cases with exclusive formation of the *cis* isomer. Furthermore, this catalyst is a relatively mild Lewis acid[1] and thus may be more tolerant of sensitive functionalities. The reaction mechanism is believed to proceed through an electrophilic iminium ion intermediate. Our current research is directed toward creating chiral variants of this catalyst in order to prepare enantiomerically enriched aziridines.

Experimental Section

General Procedures. All operations were performed under a dry nitrogen atmosphere using standard Schlenk techniques. All reaction vessels were flamed under vacuum and filled with nitrogen prior to use. HPLC grade dichloromethane (EM Science) was distilled under nitrogen from phosphorus pentoxide. Reagent grade benzene (EM Science) and toluene (Mallinckrodt) were distilled under nitrogen from sodium. Reagent grade ether (EM Science) and THF (EM Science) were distilled under nitrogen from the sodium−benzophenone ketyl. Technical grade pentane (Phillips) was mixed with concentrated sulfuric acid, washed with sodium bicarbonate, washed with distilled water, dried over sodium sulfate, and distilled from sodium. HPLC grade methanol (EM Science) was distilled under nitrogen from magnesium iodide. HPLC grade ethyl acetate (Spectrum) was dried over 4A molecular sieves prior to use. Untreated reagent grade absolute ethanol (EM Science) was used for the recrystallization of the imines. Untreated reagent grade hexanes (EM Science) were used for column chromatography.

The iron Lewis acid catalyst $[(\eta^5\text{-}C_5H_5)Fe(CO)_2(THF)]^+[BF_4]^-$ (**1**)[17a] was synthesized as first reported by our group,[17c] by protonation of the known methyl complex $(\eta^5\text{-}C_5H_5)Fe(CO)_2(CH_3)$.[18] Phenyldiazomethane (**3b**) was prepared using literature procedures.[19] The iron carbene $[(\eta^5\text{-}C_5H_5)Fe(CHPh)(CO)_2]^+[SO_3CF_3]^-$ was also prepared as our group first reported.[15] Benzaldehyde, benzophenone, 4-fluorobenzaldehyde, *N*-(4-methoxybenzylidene)aniline (**2c**), tosyl hydrazide, chlorotrimethylsilane, and trimethylsilyl trifluoromethanesulfonate were purchased from Lancaster Synthesis. Ethyl diazoacetate (**3a**, 90% solution in CH_2Cl_2), sodium (lump in kerosene), 4-tolualdehyde, 4-chlorobenzaldehyde, 2,4-dichlorobenzaldehyde, 4-nitrobenzaldehyde, *n*-butylamine, *N*-benzylidenebenzylamine (**2i**), cyclopentadienyliron dicarbonyl dimer, iodomethane, and tetrafluoroboric acid−diethyl ether complex were purchased from Aldrich Chemical Co. Aniline, α,α,α-trifluoro-4-tolualdehyde, and phosphorus pentoxide were purchased from Fisher Scientific. Flash chromatography was performed using EM Science silica gel 60, 70−230 mesh. Thin-layer chromatography was performed using EM Science F_{254} silica gel 60. Previously reported compounds were identified by ^{1}H NMR spectrometry using a 250 MHz or a 300 MHz spectrometer and have literature references provided. IR spectra were obtained from solid samples in KBr disks. New compounds were additionally characterized by ^{13}C NMR spectrometry at 62.9 MHz, GC/MS (EI) at either 70 or 15 eV, and elemental analysis.

General Preparation of Imines. A 100-mL round-bottom flask was charged with 30 mL of toluene or benzene, followed by approximately 25 mmol of the appropriate aryl aldehyde and then 25 mmol of the appropriate primary amine compound. The solution was magnetically stirred at room temperature for 6 h. The solvent was removed under reduced pressure, leaving behind an off-white solid which was recrystallized from absolute ethanol. Residual ethanol was removed by warming the crystals in a flask with a water bath until the crystals melted under reduced pressure. Upon cooling, the liquid rapidly crystallized into slightly yellow crystals which were then used without further purification. When the imine product was a liquid, the solvent was removed under reduced pressure, leaving behind a yellowish oil. The crude product was distilled under reduced pressure (0.5 mmHg) using a short-path condenser which provided a clean fraction when the temperature reached the appropriate level. The clear distillate was collected and used without further purification.

***N*-Benzylideneaniline (2a):**[20] ^{1}H NMR ($CDCl_3$) δ 8.46 (s, 1H), 7.93−7.89 (m, 2H), 7.49−7.37 (m, 5H), 7.25−7.20 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 160.2, 152.0, 136.1, 131.3, 129.0, 128.7, 128.6, 125.8, 120.8; IR (KBr) 3055, 2890, 1952, 1628 (C=N), 1585, 1485, 1450 cm^{-1}.

***N*-(4-Methylbenzylidene)aniline (2b):**[21] ^{1}H NMR ($CDCl_3$) δ 8.42 (s, 1H), 7.82−7.78 (d, J = 8.1 Hz, 2H), 7.43−7.36 (m, 2H), 7.30−7.19 (m, 5H), 2.42 (s, 3H).

***N*-(4-Chlorobenzylidene)aniline (2d):**[21] ^{1}H NMR ($CDCl_3$) δ 8.42 (s, 1H), 7.86−7.83 (d, J = 8.25 Hz, 2H), 7.47−7.37 (m, 4H), 7.28−7.20 (m, 3H).

***N*-(2,4-Dichlorobenzylidene)aniline (2e):**[22] ^{1}H NMR ($CDCl_3$) δ 8.86 (s, 1H), 8.23−8.20 (d, J = 8.5 Hz, 1H), 7.45−7.24 (m, 7H).

***N*-(4-Fluorobenzylidene)aniline (2f):**[21] ^{1}H NMR ($CDCl_3$) δ 8.42 (s, 1H), 7.94−7.88 (m, 2H), 7.44−7.38 (m, 2H), 7.28−7.13 (m, 5H).

***N*-(4-Trifluoromethylbenzylidene)aniline (2g):**[21] ^{1}H NMR ($CDCl_3$) δ 8.50 (s, 1H), 8.04−8.01 (d, J = 8.1 Hz, 2H), 7.75−7.72 (d, J = 8.175 Hz, 2H), 7.46−7.40 (m, 2H), 7.31−7.23 (m, 3H).

***N*-(4-Nitrobenzylidene)aniline (2h):**[21] ^{1}H NMR ($CDCl_3$) δ 8.56 (s, 1H), 8.35−8.31 (d, J = 8.75 Hz, 2H), 8.10−8.06 (m, 2H), 7.47−7.41 (m, 2H), 7.33−7.25 (m, 3H).

***N*-Benzylidenebutylamine (2j):**[23] ^{1}H NMR ($CDCl_3$) δ 8.27 (s, 1H), 7.75−7.69 (m, 2H), 7.43−7.38 (m, 3H), 3.65−3.59 (m, 2H), 1.75−1.64 (m, 2H), 1.44−1.32 (m, 2H), 0.98−0.92 (t, J = 7.3 Hz, 3H).

General Preparation of Aziridines. A 100-mL round-bottom flask was charged with approximately 0.20 mmol of $[(\eta^5\text{-}C_5H_5)Fe(CO)_2(THF)]^+[BF_4]^-$, followed by 3 mL of CH_2Cl_2 and then 10 equiv of the appropriate imine. A diazo solution was created by diluting 15 equiv of the appropriate diazo compound to 5 mL with CH_2Cl_2. The diazo solution was added drop-by-drop over 12 h via syringe pump to the imine−catalyst solution, which was then allowed to magnetically stir for an additional hour. To the reaction, 5 mL of THF was added, and the mixture was stirred for an additional hour. The reaction mixture was filtered through a silica gel plug, and the products were eluted, first with 50 mL of ether and then 30 mL of ethyl acetate. The solvent was removed under reduced pressure, and the products were separated on a silica gel column using pentane or hexanes with 0−10% ethyl acetate gradient elution. The collected fractions were analyzed by TLC, and the fractions with similar components were recombined. The solvent was removed under reduced pressure, and the remaining oils or solids were analyzed by ^{1}H NMR.

***cis*-2-Ethoxycarbonyl-1,3-diphenylaziridine (4a).**[13] An EDA solution was prepared by diluting EDA (0.23 mL, 2.01

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mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (67.4 mg, 0.201 mmol) and **2a** (371 mg, 2.01 mmol) in 3 mL of CH_2Cl_2, providing 150 mg of *cis*-aziridine, a 40% isolated yield: ^{1}H NMR ($CDCl_3$) δ 7.54−7.49 (m, 2H), 7.40−7.25 (m, 5H), 7.08−7.03 (m, 3H), 4.10−3.95 (m, 2H), 3.62−3.55 (d, J = 6.75 Hz, 1H), 3.23−3.18 (d, J = 6.75 Hz, 1H), 1.03−0.95 (t, J = 7.12 Hz, 3H). In addition, **6a** and **7a** were formed and isolated as a mixture (114 mg, 21% yield) in a 5:1 ratio. For **6a**:[13] ^{1}H NMR ($CDCl_3$) δ 10.37−10.31 (bd, J = 12.75 Hz, 1H), 7.45−7.39 (d, J = 12.75 Hz, 1H), 7.37−7.22 (m, 7H), 7.04−6.96 (m, 3H), 4.31−4.19 (q, J = 7.0 Hz, 2H), 1.35−1.27 (t, J = 7.0 Hz, 3H). For **7a**:[13] ^{1}H NMR ($CDCl_3$) δ 10.31 (bs, 1H), 7.40−6.60 (m, 10H), 4.99 (s, 1H), 4.22−4.10 (q, J = 7.0 Hz, 2H), 1.28−1.20 (t, J = 7.0 Hz, 3H).

***cis*-(2-(4-Chlorophenyl)-3-ethoxycarbonyl)-1-phenylaziridine (4d).**[12a] An EDA solution was prepared by diluting EDA (0.15 mL, 1.32 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (35.5 mg, 0.106 mmol) and **2d** (228 mg, 1.06 mmol) in 5 mL of CH_2Cl_2, providing 160 mg of *cis*-aziridine, a 50% isolated yield: ^{1}H NMR ($CDCl_3$) δ 7.49−7.40 (m, 2H), 7.35−7.20 (m, 4H), 7.08−6.99 (m, 3H), 4.11−3.90 (m, 2H), 3.55−3.50 (d, J = 6.7 Hz, 1H), 3.21−3.16 (d, J = 6.7 Hz, 1H), 1.09−0.99 (t, J = 7.1 Hz, 3H). In addition, **6d** and **7d**[24] were formed and isolated as a mixture (70 mg, 22% yield) in a 5:1 ratio. For **6d**: ^{1}H NMR ($CDCl_3$) δ 10.38−10.33 (bd, J = 12.75 Hz, 1H), 7.40−6.95 (m, 10H), 4.30−4.19 (q, J = 7.0 Hz, 2H), 1.34−1.24 (t, J = 7.0 Hz, 3H).

2-(2,4-Dichlorophenyl)-3-ethoxycarbonyl-1-phenylaziridine (4e).[25] An EDA solution was prepared by diluting EDA (0.21 mL, 1.79 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (48.1 mg, 0.143 mmol) and **2e** (358 mg, 1.43 mmol) in 6 mL of CH_2Cl_2, providing 57 mg of *cis*- and 126 mg of *trans*-aziridine, a 1:2.2 ratio and a 38% overall isolated yield. Spectrometric and analytical data for the *cis* isomer: ^{1}H NMR ($CDCl_3$) δ 7.72−7.67 (d, J = 8.3 Hz, 1H), 7.38−7.22 (m, 4H), 7.11−7.00 (m, 3H), 4.15−3.95 (m, 2H), 3.71−3.67 (d, J = 6.67 Hz, 1H), 3.32−3.28 (d, J = 6.5 Hz, 1H), 1.10−1.01 (t, J = 7.12 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.2, 152.0, 134.3, 134.2, 131.6, 131.3, 129.3, 128.6, 126.9, 123.8, 119.9, 61.2, 45.2, 45.0, 13.9. MS (15 eV) *m*/*z*: 337 (10), 335 (15), [M$^+$]; 306 (10), [M$^+$ − Et]; 300 (24), [M$^+$ − Cl]; 292 (10), 290 (16), [M$^+$ − OEt]; 266 (11), 264 (67), 262 (100), [M$^+$ − CO_2Et]; 245 (24), 243 (39), [M$^+$ − NHPh]; 227 (27), [M$^+$ − CO_2Et, − Cl]; 104 (61), [M$^+$ − CO_2Et, − $CHPhCl_2$]. Anal. Calcd for $C_{17}H_{15}Cl_2NO_2$: C, 60.73; H, 4.50; N, 4.17. Found: C, 60.62; H, 4.50; N, 3.91. Spectrometric data for the *trans* isomer: ^{1}H NMR ($CDCl_3$) δ 7.43−6.90 (m, 8H), 4.19−4.00 (m, 2H), 4.06−4.05 (d, J = 2.3 Hz, 1H), 3.11−3.10 (d, J = 2.5 Hz, 1H), 1.15−1.09 (t, J = 7.13 Hz, 3H). MS (70 eV) *m*/*z*: 337 (9), 335 (13), [M$^+$]; 300 (17), [M$^+$ − Cl]; 292 (14), 290 (21), [M$^+$ − OEt]; 266 (13), 264 (67), 262 (100), [M$^+$ − CO_2Et]; 245 (21), 243 (33), [M$^+$ − NHPh]; 229 (27), 227 (72), [M$^+$ − CO_2Et, − Cl]; 104 (84), [M$^+$ − CO_2Et, − $CHPhCl_2$]. An analytically pure sample of the *trans* isomer was not obtained; therefore the CHN analysis was not accomplished. In addition, **6e** and **7e**[24] were formed and isolated as a mixture (102 mg, 21% yield) in a 4:1 ratio. For **6e**: ^{1}H NMR ($CDCl_3$) δ 10.32−10.26 (bd, J = 12.5 Hz, 1H), 7.43−6.90 (m, 9H), 4.25−4.12 (q, J = 7.0 Hz, 2H), 1.30−1.20 (t, J = 7.0 Hz, 3H).

***cis*-(2-Ethoxycarbonyl-3-(4-fluorophenyl))-1-phenylaziridine (4f).**[25] An EDA solution was prepared by diluting EDA (0.17 mL, 1.44 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (38.7 mg, 0.115 mmol) and **2f** (230 mg, 1.15 mmol) in 5 mL of CH_2Cl_2, providing 144 mg of *cis*-aziridine, a 44% isolated yield: ^{1}H NMR ($CDCl_3$) δ 7.53−7.47 (m, 2H), 7.31−7.23 (m, 2H), 7.08−7.00 (m, 5H), 4.13−3.93 (m, 2H), 3.57−3.54 (d, J = 6.70 Hz, 1H), 3.20−3.17 (d, J = 6.68 Hz, 1H), 1.07−1.01 (t, J = 7.14 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, (d, 164.5, 160.6), 152.2, 130.5, (d, 129.5, 129.4), 129.3, 123.6, 119.9, (d, 115.2, 114.9), 61.1, 46.5, 45.6, 14.0. MS (70 eV) *m*/*z*: 285 (11), [M$^+$]; 256 (11), [M$^+$ − Et]; 240 (23), [M$^+$ − OEt]; 212 (100), [M$^+$ − CO_2Et]; 193 (30), [M$^+$ − NHPh or − CO_2Et, − F]; 104 (72), [M$^+$ − CO_2Et, − CHPhF]. Anal. Calcd for $C_{17}H_{16}FNO_2$: C, 71.56; H, 5.65; N, 4.91. Found: C, 71.52; H, 5.63; N, 4.81. In addition, **6f** and **7f**[24] were formed and isolated as a mixture (45 mg, 14% yield) in a 10:1 ratio. For **6f**: ^{1}H NMR ($CDCl_3$) δ 10.34−10.28 (bd, J = 12.75 Hz, 1H), 7.40−7.34 (d, J = 12.75 Hz, 1H), 7.40−7.21 (m, 4H), 7.10−6.90 (m, 5H), 4.29−4.19 (q, J = 7.0 Hz, 2H), 1.34−1.24 (t, J = 7.0 Hz, 3H).

***cis*-(2-Ethoxycarbonyl-3-(4-trifluoromethylphenyl))-1-phenylaziridine (4g).**[25] An EDA solution was prepared by diluting EDA (0.22 mL, 1.87 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (50.2 mg, 0.149 mmol) and **2g** (372 mg, 1.49 mmol) in 5 mL of CH_2Cl_2, providing 267 mg of *cis*-aziridine, a 53% isolated yield, obtained as an off-white crystalline compound: mp = 87.5−89 °C; ^{1}H NMR ($CDCl_3$) δ 7.68−7.55 (m, 4H), 7.33−7.20 (m, 2H), 7.10−7.00 (m, 3H), 4.13−3.90 (m, 2H), 3.63−3.60 (d, J = 6.73 Hz, 1H), 3.26−3.23 (d, J = 6.75 Hz, 1H), 1.05−0.98 (t, J = 7.12 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.2, 152.0, 138.9, 129.3, 128.2, 123.8, 119.9, 61.2, 46.5, 45.7, 13.9, [130.7, 126.3, 122.0, 117.7, q, ^1J(C,F) = 1080 Hz, CF_3], [131.0, 130.5, 130.0, 129.5, q, ^2J(C,F) = 128 Hz], [125.1, 125.0, 124.9, m]. MS (70 eV) *m*/*z*: 335 (8), [M$^+$]; 290 (9), [M$^+$ − OEt]; 262 (63), [M$^+$ − CO_2Et]; 243 (11), [M$^+$ − NHPh]; 104 (100), [M$^+$ − CO_2Et, − $CHPhCF_3$]. Anal. Calcd for $C_{18}H_{16}F_3NO_2$: C, 64.47; H, 4.81; N, 4.18. Found: C, 64.34; H, 4.75; N, 4.06. In addition, **6g** and **7g**[24] were formed and isolated as a mixture (100 mg, 20% yield) in a 4:1 ratio. For **6g**: ^{1}H NMR ($CDCl_3$) δ 10.50−10.45 (bd, J = 12.75 Hz, 1H), 7.70−7.00 (m, 10H), 4.34−4.22 (q, J = 7.0 Hz, 2H), 1.35−1.30 (t, J = 7.0 Hz, 3H).

2-Ethoxycarbonyl-3-(4-nitrophenyl)-1-phenylaziridine (4h).[13] An EDA solution was created by diluting EDA (0.18 mL, 1.51 mmol) to 4 mL with CH_2Cl_2 and then was reacted with a solution of **1** (25.4 mg, 0.076 mmol) and **2h** (171 mg, 0.76 mmol) in 4 mL of CH_2Cl_2, providing an isolated mixture of *cis*- and *trans*-aziridines in a 4:1 ratio and 78% overall yield (182 mg), based upon the imine. For the *cis* isomer: ^{1}H NMR ($CDCl_3$) δ 8.23−8.16 (d, J = 8.9 Hz, 2H), 7.73−7.66 (d, J = 8.9 Hz, 2H), 7.32−7.21 (m, 2H), 7.10−6.98 (m, 3H), 4.13−3.95 (m, 2H), 3.66−3.62 (d, J = 6.7 Hz, 1H), 3.30−3.26 (d, J = 6.7 Hz, 1H), 1.08−1.00 (t, J = 7.1 Hz, 3H). For the *trans* isomer: ^{1}H NMR ($CDCl_3$) δ 8.23−8.19 (d, J = 8.8 Hz, 2H), 7.55−7.51 (d, J = 8.8 Hz, 2H), 7.33−6.87 (m, 5H), 4.15−3.95 (m, 2H), 3.89−3.88 (d, J = 2.3 Hz, 1H), 3.24−3.23 (d, J = 2.3 Hz, 1H), 1.19−1.12 (t, J = 7.15 Hz, 3H). In addition, **6h** and **7h** were formed and isolated as a mixture (52 mg, 22% yield) in a 3:1 ratio. For **6h**:[13] ^{1}H NMR ($CDCl_3$) δ 10.60−10.54 (bd, J = 13.0 Hz, 1H), 8.20−8.14 (d, J = 8.75 Hz, 2H), 7.55−7.50 (d, J = 13.0 Hz, 1H), 7.52−7.48 (d, J = 8.75 Hz, 2H), 7.38−7.30 (m, 2H), 7.11−7.03 (m, 3H), 4.34−4.25 (q, J = 7.0 Hz, 2H), 1.36−1.28 (t, J = 7.0 Hz, 3H). For **7h**:[13] ^{1}H NMR ($CDCl_3$) δ 10.23 (bs, 1H), 8.15−8.11 (d, J = 8.75 Hz, 2H), 7.53−7.49 (d, J = 8.75 Hz, 2H), 7.13−7.07 (m, 2H), 6.98−6.90 (m, 1H), 6.67−6.63 (d, J = 7.75 Hz, 2H), 5.03 (s, 1H), 4.27−4.17 (q, J = 7.0 Hz, 2H), 1.35−1.29 (t, J = 7.0 Hz, 3H).

***cis*-1,2,3-Triphenylaziridine (5a).**[26] A PDM solution was prepared by diluting 2.6 M PDM (0.91 mL, 2.13 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (50.0 mg, 0.149 mmol) and **2a** (275 mg, 1.49 mmol) in 5 mL of CH_2Cl_2, providing 383 mg of *cis*-aziridine, a 95% isolated yield: ^{1}H NMR ($CDCl_3$) δ 7.35−7.03 (m, 15H), 3.67 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 154.7, 136.0, 129.2, 127.9 (2C), 127.0, 122.7, 119.9, 49.1.

***cis*-2-(4-Methylphenyl)-1,3-diphenylaziridine (5b).**[25] A PDM solution was prepared by diluting 2.7 M PDM (1.05 mL, 2.55 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a

(24) These compounds (**7d**, **7e**, **7f**, and **7g**) were isolated as an inseparable mixture along with **6d**, **6e**, **6f**, and **6g**, respectively, and were the minor component of these fractions. In addition, their ^{1}H NMR peaks were mostly buried under the peaks for byproducts **6**; therefore, the ^{1}H NMR peak assignments were not accomplished.

(25) To our knowledge, these compounds are new and previously unreported; therefore, they have been more thoroughly characterized.

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solution of **1** (57.2 mg, 0.170 mmol) and **2b** (339 mg, 1.70 mmol) in 5 mL of CH_2Cl_2, providing 461 mg of *cis*-aziridine, a 95% isolated yield. An analytically pure sample was obtained from a second separation using 25% CH_2Cl_2 in pentane on a silica gel column: ^{1}H NMR ($CDCl_3$) δ 7.35−7.00 (m, 14H), 3.64 (s, 2H), 2.28 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 154.8, 136.5, 136.2, 133.0, 129.2, 128.6, 127.9 (2C), 127.7, 126.9, 122.7, 119.9, 49.1, 49.0, 21.0. MS (70 eV) *m*/*z*: 285 (52), [M^+]; 284 (77), [$M^+ - H$]; 270 (18); 181 (44); 167 (100); 165 (85); 104 (96), [$M^+ -$ Ph, − $CHPhCH_3$]. Anal. Calcd for $C_{21}H_{19}N$: C, 88.38; H, 6.71; N, 4.91. Found: C, 88.36; H, 6.70; N, 4.94.

***cis*-2-(4-Nitrophenyl)-1,3-diphenylaziridine (5h).**[25] A PDM solution was prepared by diluting 2.6 M PDM (0.89 mL, 2.08 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (48.8 mg, 0.145 mmol) and **2h** (335 mg, 1.45 mmol) in 5 mL of CH_2Cl_2, providing 372 mg of *cis*-aziridine, an 81% isolated yield: ^{1}H NMR ($CDCl_3$) δ 8.06−8.03 (d, J = 8.73 Hz, 2H), 7.46−7.05 (m, 12H), 3.80−3.77 (d, J = 6.5 Hz, 1H), 3.71−3.69 (d, J = 6.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 153.6, 147.2, 143.7, 134.8, 129.3, 128.5, 128.2, 127.7, 127.5, 123.3, 123.1, 119.8, 49.6, 48.2. MS (70 eV) *m*/*z*: 316 (33), [M^+]; 315 (55), [$M^+ - H$]; 269 (26); 212 (37); 167 (48); 165 (100); 152 (30); 104 (41), [$M^+ -$ Ph, − $CHPhNO_2$]. Anal. Calcd for $C_{20}H_{16}N_2O_2$: C, 75.93; H, 5.10; N, 8.86. Found: C, 75.93; H, 5.05; N, 8.63.

***cis*-1-Benzyl-2,3-diphenylaziridine (5i).**[27] A PDM solution was prepared by diluting 2.7 M PDM (0.80 mL, 1.94 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (43.5 mg, 0.130 mmol) and **2i** (0.246 mL, 1.30 mmol) in 5 mL of CH_2Cl_2, providing 277 mg of *cis*-aziridine, a 75% isolated yield: ^{1}H NMR ($CDCl_3$) δ 7.55−7.05 (m, 15H), 3.90 (s, 2H), 3.08 (s, 2H).

***cis*-1-*n*-Butyl-2,3-diphenylaziridine (5j).**[27] A PDM solution was prepared by diluting 2.6 M PDM (0.86 mL, 2.00 mmol) to 5 mL with CH_2Cl_2 and then was reacted with a solution of **1** (47.0 mg, 0.140 mmol) and **2i** (0.253 mL, 1.40 mmol) in 5 mL of CH_2Cl_2, providing 253 mg of *cis*-aziridine, a 72% isolated yield: ^{1}H NMR ($CDCl_3$) δ 7.22−7.01 (m, 10H), 2.85 (s, 2H), 2.69−2.62 (t, J = 6.9 Hz, 2H), 1.75−1.60 (m, 2H), 1.60−1.41 (m, 2H), 1.00−0.92 (t, J = 7.3 Hz, 3H).

Reaction of the Imine 2a with the Iron Carbene 8. An excess of **2a** (450 mg, 2.47 mmol) was added to **8** in 10 mL of CH_2Cl_2 at −78 °C; the solution was allowed to come to room temperature over 4 h, at which time 5 mL of THF was added, and was then stirred for an additional 15 min. The crude was filtered through a silica gel plug and eluted first with 50 mL of ether and then 30 mL of EtOAc. The solvent was removed under reduced pressure to provide 0.929 g of an oil. The ^{1}H NMR spectrum ($CDCl_3$) showed much unreacted imine; however, the expected *cis*-1,2,3-triphenylaziridine with a singlet at 3.66 ppm was not present.

Iron Lewis Acid−Imine Complex [(η^5-C_5H_5)Fe(CO)$_2$(PhCH=NPh)]$^+$[BF_4]$^-$ (10).[25] A 100-mL round-bottom flask was charged with **1** (500 mg, 1.46 mmol) and then 10 mL of CH_2Cl_2. Imine **2a** (2.69 g, 14.6 mmol) was added to this solution. This solution was allowed to stir for 5 h at room temperature, during which time the solution turned from the bright-red color of the iron Lewis acid to a yellow-orange color. The solvent was removed under reduced pressure. The uncoordinated imine was extracted with 20 mL of ether (2 times); the supernatant solution was removed by transferring it through a cannula, leaving behind a brown tar. The residue was recrystallized two times by dissolving it in 1 mL of CH_2Cl_2 and then adding 20 mL of ether at −78 °C to cause precipitation. The precipitate was vacuum pumped for 24 h, providing a tan powder which was found to turn to a gum when exposed to air: ^{1}H NMR ($CDCl_3$) δ 8.70 (s, 1H), 7.70−7.10 (m, 10 H), 5.10 (s, 5H); ^{13}C NMR ($CDCl_3$) δ 208.8, 186.4, 159.6, 135.1, 132.3, 130.1, 129.6, 128.2, 121.0, 120.3, 87.5; IR (KBr) 3120, 3063, 2059, 2002, 1603 (C=N), 1493, 1424, 1066 cm^{-1}.

Reaction of [(η^5-C_5H_5)Fe(CO)$_2$(PhCH=NPh)]$^+$[BF_4]$^-$ (10) with EDA. A 100-mL round-bottom flask was charged with **10** (95%, 155 mg, 0.330 mmol), 4 mL of CH_2Cl_2, and then 1.5 equiv of EDA (0.058 mL, 0.495 mmol). The solution was allowed to stir for 3 h at room temperature, at which time 2 mL of THF was added, and then was stirred for an additional 15 min. The crude was filtered through a silica gel plug and eluted with 125 mL of EtOAc. The solvent was removed under reduced pressure, providing a brown oil which looked to still the contain iron Lewis acid. The oil was dissolved in 10 mL of CH_3CN and stirred for 15 min. The solvent was removed under reduced pressure, resulting in a brown oil that was eluted through a silica column with 10% EtOAc in pentane to provide a yellow oil. ^{1}H NMR ($CDCl_3$) showed a broad doublet (J = 12.8 Hz) at 10.4 ppm with an area of 1.20 due to **6a**; a singlet at 5.0 ppm with an area of 0.23 due to **7a**; and two doublets (J = 6.7 Hz) at 3.6 and 3.2 ppm with areas of 3.27 and 3.21, respectively, due to each proton of the aziridine ring.

Reaction of [(η^5-C_5H_5)Fe(CO)$_2$(PhCH=NPh)]$^+$[BF_4]$^-$ (10) with PDM. Approximately 5 mg of **10** was placed into an NMR tube and dissolved in 0.75 mL of $CDCl_3$, followed by 1 drop of PDM. The resulting proton spectrum after 10 min showed a singlet at 3.65 ppm, indicative of the formation of *cis*-1,2,3-triphenylaziridine.

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