the reaction mixture stirred at room temperature. After 18 h the mixture was partitioned between ether and an ice-cold dilute $Na_2CO_3$ solution. The ether layer was washed with basic ($Na_2CO_3$) brine, dried, and evaporated to give crude **10** as an oil. This was dissolved in acetone and treated with 1 equiv of HCl to give 4.71 g (64.4%) of hydrochloride salt: mp 144–146 °C dec; IR (Nujol) 3270, 1620, 1595, 1582, 1040, and 745 $cm^{-1}$. Anal. Calcd for $C_{19}H_{23}NO_2S$·HCl: C, 62.36; H, 6.61; N, 3.83. Found: C, 62.21; H, 6.88; N, 3.65.

**4-(Dimethylamino)-2-(phenylthio)-*N*-(*p*-fluorophenyl)-crotonamide (11).** A solution of **4c** (3.86 g, 20 mmol) in ether was lithiated as described. After 1.25 h at 0 °C, a solution of 4-fluorophenyl isocyanate (3.29 g, 24 mmol) in 5 mL of ether was added dropwise. The bath was removed and the reaction mixture stirred at room temperature. After 18 h the reaction mixture was partitioned between ether and an ice-cold dilute $Na_2CO_3$ solution. The ether layer was washed with basic ($Na_2CO_3$) brine, dried, and evaporated to give an oily residue. Crystallization from ether–hexane gave 3.12 g (47.3%) of anilide **11**: mp 76–79 °C; NMR ($CDCl_3$) $\delta$ 2.29 (s, 6 H), 3.15–3.26 (d, 2 H), 6.06–6.28 (t, 1 H), 6.80–7.68 (m, 9 H), and 10.41 (br, 1 H, ex); IR ($CH_2Cl_2$) 1663, 1620, 1570, and 1212 $cm^{-1}$. Anal. Calcd for $C_{18}H_{19}FN_2OS$: C, 65.43; H, 5.80; N, 8.48. Found: C, 65.64; H, 5.90; N, 8.54.

**Acknowledgment.** We wish to acknowledge the support of these studies by Dr. Neville Finch and the carefully executed work of Ms. Ruth Behnke (NMR) and Mr. George Robertson (microanalyses).

**Registry No.**—**4c**, 63905-40-8; **8** HCl, 68213-00-3; **9** HCl, 68200-56-6; **10** HCl, 68200-57-7; dimethylamine, 124-40-3; 3-chloro-1-(phenylthio)-1-propene, 58749-54-5.

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# Synthetic Applications of *N*-Sulfonylamines: Reactions with Activated Dienes to Form Heterocycles

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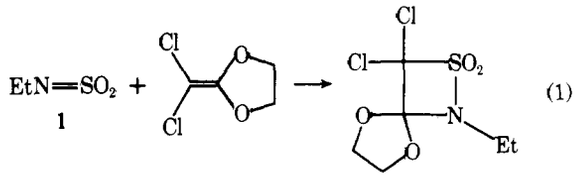
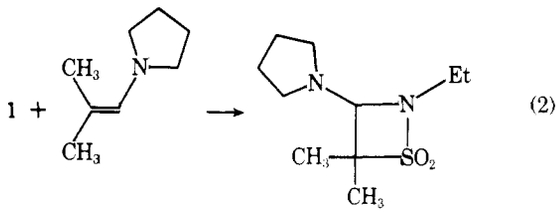
In 1967 Burgess reported the generation of a new class of heterocumulene, the *N*-sulfonylamines.[1] During the next few years he also described the generation and reactions of the related *N*-sulfonylamides[1,2] and *N*-sulfonylurethanes.[2,3] While the latter compounds were proven to be useful synthetically, the simple alkylsulfonylamines suffered from an overall lack of reactivity. Apart from interception by nucleophiles such as amines and anilines, the electrophilic reactivity of this species was limited to strongly nucleophilic olefins only. Thus Burgess was able to obtain 2 + 2 cycloadducts between *N*-ethylsulfonylamine (**1**)[4] and such reactive types as ketene acetals and enamines as shown (eq 1 and 2). Ethyl vinyl ether, however, failed to react.



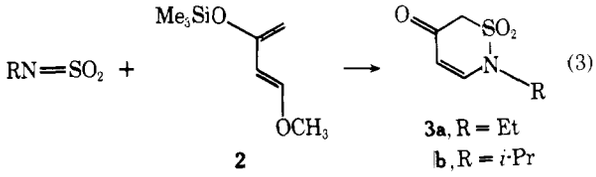

(1)




(2)

We now report that simple alkylsulfonylamines (e.g., **1**) react with certain activated dienes to form 1,2-thiazin-5(6*H*)-one 1,1-dioxides, a new heterocyclic ring system. This is an extension of the reactivity and synthetic utility of these unactivated heterocumulenes and represents the first example of their reaction with dienes in a formal 4 + 2 sense to afford six-membered heterocycles.

When a cold (−78 °C) solution of activated diene **2**[5] and a molar equivalent of triethylamine was treated with ethyl sulfamoyl chloride,[6] triethylamine hydrochloride immediately precipitated. Warming followed by filtration and concentration provided no characterizable products. However, when the reaction was worked up with aqueous acid, a single product, 2-ethyl-1,2-thiazin-5(6*H*)-one-1,1-dioxide (**3a**), was formed in 60% yield (eq 3). A similar experiment with *N*-isopropyl-

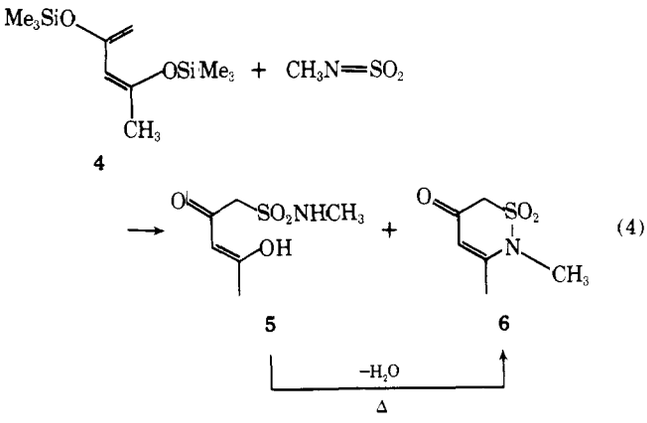



(3)

sulfonylamine gave the corresponding isopropyl derivative **3b**, in 71% yield. The assignment of these structures is based on spectral and analytical results, as well as analogy with Burgess' work.[1]

Although these products could arise from a concerted 4 + 2 cycloaddition, the failure to isolate products in the absence of an acidic workup certainly makes such an assumption suspect. Burgess investigated this question in the case of *N*-sulfonylurethanes and concluded that either a concerted or stepwise reaction was possible.[3b] To further probe this question, a second diene was examined.

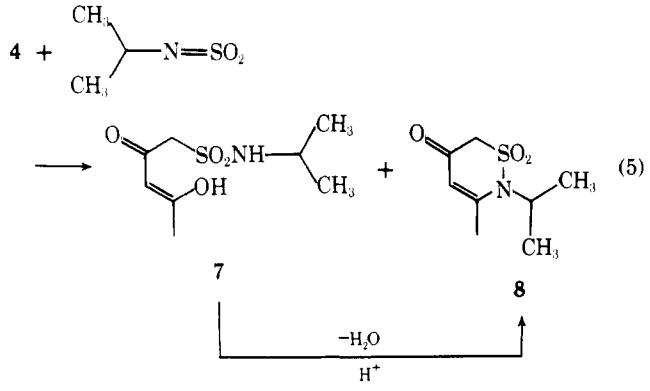
When *N*-sulfonylmethylamine was generated in the presence of activated diene **4**,[7] two products arose after workup with aqueous acid[8] (eq 4).




(4)

Spectral analysis suggested that they were the open chain diketosulfonamide **5** and its cyclization product **6** in a ratio of 3:2. Upon distillation, the former ring closed to afford 2,3-dimethyl-1,2-thiazin-5(6*H*)-one 1,1-dioxide (**6**) in 57% yield.

Repeating this sequence with *N*-isopropylsulfonylamine gave rise to a 6:1 ratio of diketosulfonamide **7** and its ring closed counterpart **8**. This mixture remained unchanged upon




distillation; however, refluxing it in toluene with a trace of acid catalyst did result in complete conversion to 2-isopropyl-3-methyl-1,2-thiazin-5(6*H*)-one 1,1-dioxide (**8**).

The isolation of diketosulfonamides **5** and **7** supports the conclusion that the heterocycles **6** and **8** are formed in a stepwise fashion. Since no intermediates were isolable in the reactions of activated diene **2**, a concerted mechanism for the formation of heterocycles **3a** and **3b** cannot be excluded. Nevertheless, the dependence of their isolation on an acidic workup strongly suggests that a two-step mechanism is operating.

Regardless of the particular path of reaction, the end results remain of significant. The reactivity limits of a heretofore relatively unreactive class of heterocumulene have been extended in such a manner as to allow synthesis of several examples of a new heterocyclic ring system. Further definition of these reactivity limits is in progress.

## Experimental Section

**General.** Melting points were determined on a Laboratory Devices Melt-temp apparatus and are uncorrected. Nuclear magnetic resonance spectra (NMR) were recorded on Varian T-60 and EM-360 spectrometers using tetramethylsilane as an internal standard. Combustion analyses were performed by Atlantic Microlabs. Dry column chromatography was accomplished on ICN activity 111/30 silica gel and elution was done with distilled solvents. Evaporative bulb-to-bulb distillations were performed with a Buchi Kugel-Rohr hot air oven. The boiling points reported for this technique are therefore the oven temperature at which distillation occurred. The drying process referred to in the workup procedure involved swirling the solution over an excess amount of anhydrous magnesium sulfate followed by filtration. Anhydrous THF was obtained by distillation from sodium metal (benzophenone indicator) and stored under argon in a Schlenk flask. The yields reported are of analytically pure material. No attempt has been made to maximize them.

**2-Ethyl-1,2-thiazin-5(6*H*)-one 1,1-Dioxide (3a).** A solution of 2.4 g (13.9 mmol) of activated diene **2** and 1.41 g (13.9 mmol) of triethylamine in 15 mL of anhydrous THF was maintained under a nitrogen atmosphere and cooled in a dry ice/acetone bath with vigorous stirring. A solution of 2 g (13.9 mmol) of *N*-ethylsulfamoyl chloride[6] in 10 mL of anhydrous THF was then added dropwise. After 2 h of stirring, the resulting suspension was allowed to come to room temperature and stirred an additional 1.5 h. The suspension was acidified with 15 mL of 10% hydrochloric acid, stirred 0.75 h, then extracted with three portions of ether. The extracts were combined, washed with brine, dried, and concentrated on a rotary evaporator. The resulting dark oil was chromatographed on a silica gel "dry column", using 40% ethyl acetate/cyclohexane as the eluant. After recovery of the product by extraction of the appropriate band with ethyl acetate, evaporative bulb-to-bulb distillation provided heterocycle **3a** as a yellow oil: bp 145 °C (0.05 mm); NMR ($CDCl_3$) $\delta$ 1.40 (t, $J$ = 7 Hz, 3 H), 3.76 (q $J$ = 7 Hz, 2 H), 4.15 (s, 2 H), 5.63 (d, $J$ = 9 Hz, 1 H), 7.20 (d, $J$ = 9 Hz, 1 H).

Anal. Calcd for $C_6H_9NO_3S$: C, 41.13; H, 5.18; N, 7.99. Found: C, 40.99; H, 5.24; N, 8.03.

**2-Isopropyl-1,2-thiazin-5(6*H*)-one 1,1-Dioxide (3b).** A solution of 3.44 g (0.02 mol) of activated diene **2** and 2.02 g (0.02 mol) of triethylamine in 20 mL of anhydrous THF was maintained under a nitrogen atmosphere and cooled in a dry ice/acetone bath with vigorous stirring. A solution of 3.15 g (0.02 mol) of *N*-isopropylsulfamoyl chloride[6] in 10 mL of anhydrous THF was added dropwise and the resulting suspension stirred 2 h. The reaction mixture was allowed to come to room temperature, stirred for 1 h, acidified with 20 mL of 10% hydrochloric acid, and stirred an additional 0.75 h. The mixture was extracted with three portions of ether, then the extracts were combined, washed with brine, dried, and concentrated on a rotary evaporator. Evaporative bulb-to-bulb distillation afforded 1.47 g (60%) of heterocycle **3b** as a colorless oil: bp 135 °C (0.1 mm); NMR ($CDCl_3$) $\delta$ 1.37 (d, $J$ = 7 Hz, 6 H), 4.13 (s, 2 H), 4.56 (m, $J$ = 7 Hz, 1 H), 5.58 (d, $J$ = 9 Hz, 1 H), 7.18 (d, $J$ = 9 Hz, 1 H).

Anal. Calcd for $C_7H_{11}NO_3S$: C, 44.44; H, 5.82; N, 7.41. Found: C, 44.65; H, 5.85; N, 7.43.

***N*-Methyl-2,4-dioxopentanesulfonamide (5) and 2,3-Dimethyl-1,2-thiazin-5(6*H*)-one 1,1-Dioxide (6).** A solution of 3.39 g (13.9 mmol) of activated diene **4** and 1.41 g (13.9 mmol) of triethylamine in 15 mL of anhydrous THF maintained under a nitrogen atmosphere was cooled in a dry ice/acetone bath with vigorous stirring. A solution of *N*-methylsulfamoyl chloride[6] in 10 mL of anhydrous THF was added dropwise and the resulting suspension stirred 1.75 h. After allowing the mixture to come to room temperature, stirring was continued for an additional 1 h. The mixture was then acidified with 15 mL of 10% hydrochloric acid, stirred 1 h, and extracted with three portions of ether. The extracts were combined, washed with brine, dried, and concentrated on a rotary evaporator. An NMR analysis of this crude mixture indicated the presence of what are assigned as **5** and **6** in a ratio of 3:2. Evaporative bulb-to-bulb distillation resulted in ring closure of **5** and provided a colorless oil which crystallized on standing. Recrystallization from isopropyl alcohol afforded 1.4 g (57%) of heterocycle **6** as white needles: mp 57–59 °C; NMR ($CDCl_3$) $\delta$ 2.20 (s, 3 H), 3.28 (s, 3 H), 4.06 (s, 2 H), 5.37 (s, 1 H).

Anal. Calcd for $C_6H_9NO_3S$: C, 41.13; H, 5.18; N, 7.99. Found: C, 41.09; H, 5.20; N, 7.95.

An attempt to isolate **5** by gravity column chromatography also resulted in ring closure. However, use of LC (column residence time = 30 min) gave rise to 0.5 g of **5** as an oil: NMR ($CDCl_3$) $\delta$ 2.11 (s, 3 H), 2.85 (d, $J$ = 3 Hz, 3 H), 3.93 (s, 2 H), 4.95 (broad s, 2 H), 5.76 (s, 1 H). This oil dehydratively ring closed to **6** slowly upon standing, or immediately with heating.

***N*-Isopropyl-2,4-dioxopentanesulfonamide (7).** A solution of 3.39 g (13.9 mmol) of activated diene **4** and 1.41 g (13.9 mmol) of triethylamine in 15 mL of anhydrous THF maintained under a nitrogen atmosphere was cooled in a dry ice/acetone bath with vigorous stirring. A solution of 2.19 g of *N*-isopropylsulfamoyl chloride[6] in 10 mL of anhydrous THF was added dropwise and stirring continued for 2 h. The cooling bath was removed, the stirring continued for 1.5 h, and the resulting mixture acidified with 15 mL of 10% hydrochloric acid. After an additional 1 h of stirring the mixture was extracted with three portions of ether and the extracts were combined, dried, and concentrated on a rotary evaporator. Evaporative bulb-to-bulb distillation gave 2.25 g (80%) of diketosulfonamide **7** as a yellow oil: bp 145 °C (0.05 mm); NMR ($CDCl_3$) $\delta$ 1.25 (d, $J$ = 6 Hz, 6 H), 2.12 (s, 3 H), 3.70 (m, $J$ = 6 Hz, 1 H), 3.95 (s, 2 H), 4.50 (broad s, 2 H), 5.77 (s, 1 H).

Anal. Calcd for $C_8H_{15}NO_4S$: C, 43.42; H, 6.83; N, 6.33. Found: C, 43.26; H, 6.79; N, 6.32.

**2-Isopropyl-3-methyl-1,2-thiazin-5(6*H*)-one 1,1-Dioxide (8).** An unpurified sample of diketosulfonamide **7** was prepared as above. This was dissolved in a convenient amount of toluene, a catalytic amount of methane sulfonic acid was added, and the resulting solution refluxed 6 h under a Dean-Stark trap. The solution was cooled and concentrated on a rotary evaporator and the crude product was chromatographed on a silica gel "dry column", using 40% ethyl acetate/cyclohexane as the eluant. Extraction of appropriate band with ethyl acetate afforded a solid. Recrystallization from toluene/methyl cyclohexane gave 1.11 g (39%) of heterocycle **8** as a crystalline solid: mp 98–101 °C; NMR ($CDCl_3$) $\delta$ 1.58 (d, $J$ = 7 Hz, 6 H), 2.25 (s, 3 H), 4.00 (s, 2 H), 4.40 (m, $J$ = 7 Hz, 1 H), 5.60 (s, 1 H).

Anal. Calcd for $C_8H_{13}NO_3S$: C, 47.27; H, 6.45; N, 6.89. Found: C, 47.29; H, 6.46; N, 6.88.

**Acknowledgment.** We wish to thank Professor Burgess for his helpful discussion during the inception of this work and for graciously providing us with detailed experimental results of a portion of his own research.

**Registry No.**—**2**, 59414-23-2; **3a**, 68225-95-6; **3b**, 68225-96-7; **4**, 68225-97-8; **5**, 68225-98-9; **6**, 68225-99-0; **7**, 68226-00-6; **8**, 68226-01-7; *N*-ethylsulfamoyl chloride, 16548-07-5; *N*-isopropylsulfamoyl chloride, 26118-67-2; *N*-methylsulfamoyl chloride, 10438-96-7; *N*-ethylsulfonylamine, 38336-91-3; *N*-isopropylsulfonylamine, 68226-02-8; *N*-methylsulfonylamine, 68226-03-9.

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(3) (a) G. M. Atkins and E. M. Burgess, *J. Am. Chem. Soc.*, **90**, 4744 (1968); (b) E. M. Burgess and W. M. Williams, *ibid.*, **94**, 4386 (1972); (c) E. M. Burgess and W. M. Williams, *J. Org. Chem.*, **38**, 1249 (1973).
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(8) Since *N*-sulfonylamines are extremely moisture sensitive, this effectively quenches the reaction. It is therefore unlikely that either **5** or **6** arise from reaction of the *N*-sulfonylamine with a partially hydrolyzed form of diene **4**.

# Carbon-13 Nuclear Magnetic Resonance Spectra of Substituted Pyridine *N*-Oxides[1]

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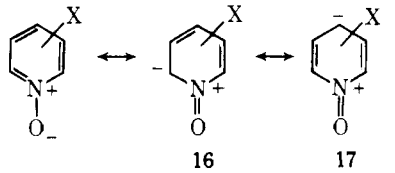
Pyridine *N*-oxides are an interesting class of organic compounds since the *N*-oxide functionality can activate the ring toward both electrophilic and nucleophilic attack.[2] We have studied the carbon-13 NMR spectra of substituted pyridine *N*-oxides in order to probe the ground state electron distribution in this molecular framework. In particular, we wished to assess the relative contributions of the substituent and the *N*-oxide functionality in determining this electron distribution.

## Results and Discussion

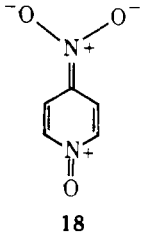
The carbon-13 chemical shifts for the pyridine *N*-oxides are given in Table I. In order to gain more insight into the effects of the *N*-oxide functionality, the carbon-13 chemical shifts of the substituted pyridine *N*-oxides were compared to those of the corresponding substituted pyridines.[3] Table I also contains the carbon-13 chemical shift difference, Δ, between the pyridine *N*-oxides and pyridines.

In general, the pyridine *N*-oxides show a large shielding effect (~10 ppm) at C-2, C-4, and C-6 relative to that observed in the corresponding pyridines. Surprisingly, the magnitude of the shielding effect is about the same at C-2, C-4, and C-6 for most cases, and is not greatly influenced by the nature or position of the substituent. Minor exceptions to this trend appear in Table I as reductions in the magnitude of the shielding effect. The only conspicuous exception to the trend is the 4-nitro substituted compound **7** in which there is an opposite deshielding effect at C-4. The shieldings observed at positions 2, 4, and 6 may be explained if resonance forms **16** and **17** make a substantial contribution to the pyridine



**16** **17**

*N*-oxide hybrid. These forms place significant electron density at the 2, 4, and 6 positions. Although other factors are known to be important, increasing electron density at carbon is associated with a shielding effect.[4] These observations apparently indicate that the oxygen of the *N*-oxide functionality is a strong electron donor to C-2, C-4, and C-6 in the molecular ground state and plays a dominant role in determining the electronic distribution at the ring carbon atoms. In some cases, the substituent may interact to perturb this effect. For example, in compound **7**, the strong electron-withdrawing res-



**18**

**Table I. Carbon-13 Chemical Shifts for Substituted Pyridine *N*-Oxides[a]**

| compd | X | $\delta_{C^{13}}$ C2 | C3 | C4 | C5 | C6 | X |
|---|---|---|---|---|---|---|---|
| 1 | H | 138.2 (−11.3)[b] | 125.3 (2.0) | 124.8 (−10.7) | 125.3 (2.0) | 138.2 (−11.3) | |
| 2 | 4-$CH_3$ | 137.2 (−11.8) | 125.6 (1.6) | 136.2 (−10.1) | 125.6 (1.6) | 137.2 (−11.8) | Me, 18.8 (−1.5) |
| 3 | 4-$N(CH_3)_2$ | 138.6 (−10.7) | 107.4 (1.2) | 150.5 (−3.3) | 107.4 (1.2) | 138.6 (−10.7) | Me, 39.6 (1.1) |
| 4 | 4-$OCH_3$ | 139.8 (−10.3) | 111.6 (2.5) | 158.1 (−6.7) | 111.6 (2.5) | 139.8 (−10.3) | Me, 55.9 (1.6) |
| 5 | 4-$COCH_3$ | 139.2 (−11.3) | 124.8 (4.1) | 132.0 (−10.2) | 124.8 (4.1) | 139.2 (−11.3) | Me, 25.9 (−0.2); CO, 193.4 (−3.4) |
| 6 | 4-Cl | 139.9 (−10.5) | 126.5 (2.7) | 133.7 (−10.0) | 126.5 (2.7) | 139.9 (−10.5) | |
| 7 | 4-$NO_2$ | 140.1 (−8.1) | 120.7 (0.4) | 142.0 (2.4) | 120.7 (0.4) | 140.1 (−8.1) | |
| 8 | 3-$COCH_3$ | 139.2 (−10.4) | 135.5 (3.5) | 125.9[c] (−9.2) | 124.4[c] (1.1) | 142.2 (−11.0) | Me, 26.5 (0.2); CO, 193.6 (−2.8) |
| 9 | 3-CN | 141.6[c] (−10.5) | 112.8 (3.1) | 129.4 (−9.5) | 126.7 (3.4) | 143.0[c] (−9.6) | CN, 113.6 (−2.5) |
| 10 | 3-Cl | 138.6[d] (−10.0) | 133.2 (1.3) | 125.6[c] (−9.9) | 125.8[c] (1.8) | 137.6[d] (−9.7) | |
| 11 | 3-Br | 140.4 (−10.3) | 120.1 (−0.5) | 128.9 (−9.4) | 125.8 (1.4) | 137.7 (−9.8) | |
| 12 | 2-$CH_3$ | 148.5 (−9.6) | 125.0 (2.1) | 126.1 (−9.8) | 123.1 (2.8) | 138.8 (−10.1) | Me, 17.2 (−6.9) |
| 13 | 2-$OCH_3$ | 157.9 (−6.1) | 108.1 (−2.6) | 130.0 (−8.2) | 117.1 (0.7) | 139.6 (−7.1) | Me, 56.9 (3.9) |
| 14 | 2-$COCH_3$ | 146.5 (−6.8) | 126.1[c] (4.9) | 126.4 (−10.1) | 127.8[c] (1.1) | 140.4 (−8.3) | Me, 30.2 (4.9); CO, 194.6 (−5.0) |
| 15 | 2-Cl | 141.1 (−10.1) | 125.4 (1.3) | 126.7 (−11.7) | 123.6 (1.6) | 140.0 (−9.5) | |

[a] Chemical shifts were converted to the $Me_4Si$ scale using the relationship $\delta_{Me_4Si} = \delta_{CDCl_3} + 76.91$. [b] Numbers in parentheses are the carbon-13 chemical shift differences, Δ, between the substitued pyridine *N*-oxides and the substituted pyridines: $\Delta = \delta_{^{13}C}$(substituted pyridine *N*-oxide) − $\delta_{^{13}C}$(substituted pyridine). A negative value, therefore, indicates greater shielding in the pyridine *N*-oxide. [c,d] Chemical shifts of these carbons may be reversed for the indicated pyridine *N*-oxide.

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