Amine-Promoted, Organocatalytic Aziridination of Enones

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General: All reactions were conducted in oven – (160 °C) or flame-dried glassware under an inert atmosphere of dry N₂ unless otherwise stated. Melting points were obtained using a Reichert hot plate microscope and are uncorrected. Infrared (IR) spectra were recorded on a Mattson Satellite FTIR spectrometer, ν_{max} in cm⁻¹. ¹H NMR spectra were recorded on a Bruker AV 400, DRX 400 or AV 500 (400 or 500 MHz) spectrometer. Chemical shifts are reported in ppm with the solvent resonance resulting from incomplete deuteration as the internal standard (CDCl₃: 7.26). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet or combinations thereof), integration, coupling constants, and assignment. ¹³C NMR spectra were recorded on a Bruker AV 400, DRX 400 or AV 500 (100 or 125 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm with the solvent resonance as the internal standard (¹³CDCl₃: 77.16). Enantiomer ratios were determined by chiral HPLC analysis: Chiral Technologies Chiralpak AD-H column (4.6 mm × 250 mm). High resolution mass spectrometry was performed on a Micromass AutoSpec Premier. Optical rotation values were recorded on an Optical Activity Autopolarimeter.

Reagents: Solvents were purified and dried using standard methods as required: THF and diethyl ether by distillation from sodium benzophenone ketyl, dichloromethane by distillation from calcium hydride. DMF was dried by stirring with MgSO₄ for 3 h, filtered, stirred with 4 Å MS for 12 h, and then distilled under reduced pressure. All other solvents were used as supplied unless otherwise stated. Silica gel chromatography was performed with silica gel, particle size 40–63 nm obtained from VWR International Ltd.

All reagents were purchased from Aldrich Chemical Company, Fisher Chemicals, or Lancaster Synthesis and purified when appropriate prior to use.¹ All amines used in this study were purified prior to use using standard techniques.¹ All chalcone derivatives in this study were purified by recrystallisation from hot ethanol prior to use.

Acetophenone: Aldrich

***tert*-Butyl acrylate:** Aldrich

Chalcone: Lancaster (recrystallised from hot ethanol prior to use)

Dioxane: Aldrich

Diphenylphosphinyl chloride: Acros

3-Furaldehyde: Aldrich

Hydroxylamine hydrochloride: Lancaster

Substrates: The aromatic chalcone substrates were either bought or could be made utilising a base catalysed Claisen-Schmidt condensation reaction between the acetophenone derivative and corresponding aldehyde (see below).² The alkyl chalcone derivatives were prepared using a Wittig reaction between phenacyl triphenylphosphonium bromide and the corresponding aldehyde (see below) according to the procedure of Pedersen.³

Representative example for the preparation of chalcone **6**:²

Acetophenone (2.79 mL, 23.9 mmol) was added to a mixture of MeOH (57.5 mL) and H₂O (11.5 mL), and finely ground KOH (2.69 g, 47.9 mmol) was added. The solution was allowed to stir at rt for 10 min, then 3-furaldehyde (2.00 mL, 23.9 mmol) was added dropwise over 5 min. The dark brown solution was allowed to stir at rt for 2 h, after which tlc analysis showed the reaction was complete (as the reaction progresses a fine precipitate formed). Water (80 mL) and saturated NH₄Cl solution (50 mL) were added and the mixture was extracted with ether (3 × 100 mL). The ether was washed with brine (50 mL), dried (Na₂SO₄) and concentrated in vacuo. The crude chalcone was purified by recrystallisation from hot ethanol to give the chalcone **6** (2.38 g, 12.0 mmol, 50%) as a light brown solid. ¹H NMR (400 MHz, CDCl₃): δ 8.01–7.98 (m, 2H, PhH), 7.22 (d, 2H, *J* = 15.4 Hz, CH=CH, ArH), 7.60–7.56 (m, 1H, PhH), 7.51–7.48 (m, 3H, PhH, ArH), 7.25 (d, 1H, *J* = 15.4 Hz, CH=CH), 6.71 (s, 1H, ArH). ¹³C NMR (100 MHz, CDCl₃) δ 190.5, 145.6, 144.6, 138.2, 134.9, 132.8, 128.7, 128.5, 123.3, 122.1, 107.5.

Representative example for the preparation of (*E*)-1-phenylhept-2-en-1-one **7**:³

Sodium hydroxide (2.0 M, 2.5 mL) was added to a solution of phenacyl triphenylphosphonium bromide (1.00 g, 2.17 mmol) in CH₂Cl₂ (5 mL) and the resulting mixture allowed to stir at rt for 14 h. The two phases were separated and the aqueous layer was extracted with CH₂Cl₂ (10 mL). The CH₂Cl₂ extracts were combined, and to this solution was added valeraldehyde (1.39 mL, 13.0 mmol) dropwise over 1 h. The solution was allowed to stir at rt for 1 h, then concentrated in vacuo. The crude chalcone derivative was purified by column chromatography (100% petroleum ether to 10% EtOAc in petroleum ether), which gave (*E*)-1-phenylhept-2-en-1-one **7** (229 mg, 1.22 mmol, 56%) as a clear oil. ¹H NMR (400 MHz,

¹ Purification of Laboratory Chemicals; W. L. F. Armarego, D. D. Perrin, **1998**.

² A slight modification of the reported procedure was performed, see: a) B. A. Bhat, K. L. Dhar, S. C. Puri, A. K. Saxena, M. Shanmugavel, G. N. Qazi, *Bio. Med. Chem. Lett.* **2005**, *15*, 3177–3180; b) E. P. Kohler, H. M. Chadwell, *Org. Synth.* **1922**, *2*, 1–2; **1942**, *Coll. Vol. 1*, 78–79.

³ F. D. Therkelson, A.-L. L. Hansen, E. B. Pedersen, C. Nielsen, *Org. Biomol. Chem.* **2003**, *1*, 2908–2918.

CDCl₃): δ 7.93–7.91 (m, 2H, PhH), 7.56–7.53 (m, 1H, PhH), 7.48–7.44 (m, 2H, PhH), 7.07 (dt, 1H, J = 15.3, 6.9 Hz, CH₂CH=CH), 6.87 (dd, 1H, J = 15.3, 1.3 Hz, CH₂CH=CH), 2.33 (tdd, 2H, J = 7.3, 6.9, 1.3 Hz, CH₂CH=CH), 1.51 (tt, 2H, J = 7.3, 7.3 Hz, CH₃CH₂CH₂), 1.38 (apparent sx, 2H, J = 7.3 Hz, CH₃CH₂CH₂), 0.93 (t, 3H, J = 7.3 Hz, CH₃). ¹³C NMR (100 MHz, CDCl₃) δ 191.1, 150.2, 138.1, 132.6, 128.6, 125.9, 32.6, 30.3, 22.4, 13.9.

Preparation of *O*-diphenylphosphinyl hydroxylamine **4**, DppONH₂

There are numerous methods for the preparation of DppONH₂, however we have found the most useful and straightforward to be a method reported by Colvin *et al* in 1982,⁴ which has since been used successfully in 1998.⁵

Aqueous NaOH (1.94 g, 48.6 mmol, in 7.00 mL H₂O, 7.1M) was added to a stirred solution of hydroxylamine hydrochloride (3.96 g, 57.0 mmol) in H₂O (8.5 mL). Dioxane (30 mL) was added and the solution cooled using an ice/salt bath. Diphenylphosphinyl chloride (5.00 g, 21.1 mL) in dioxane (25 mL) was added in one portion with vigorous stirring. Stirring continued for 4 min as copious precipitation ensued. Water (30 mL) was added and the slurry was filtered, washing with cold water (30 mL). The white tacky solid was dried under vacuum in a desiccator (3 h), then purified by stirring as a slurry with aqueous NaOH (50 mL, 0.25M) at 0 °C for 30 min. The product was filtered, washing with cold water (30 mL), then dried under vacuum in a desiccator to give *O*-diphenylphosphinyl hydroxylamine **4** (3.00 g, 12.9 mmol, 61%) as a white powdery solid: m.p. >135 °C, gradual decomp. (lit. >130 °C, gradual decomp.⁶). ¹H NMR (400 MHz, CDCl₃): δ 7.87–7.82 (m, 4H, PhH), 7.57–7.54 (m, 2H, PhH), 7.50–7.47 (m, 4H, PhH), 5.9 (br s, 2H, NH₂). ³¹P NMR (162 MHz, CDCl₃) δ 37.6. m/z 234 (M^+ + H, 100%), 201 (M^+ – ONH₂, 25%).

In situ hydrazinium salt formation and aziridination of chalcones

A note regarding the general procedure: The representative procedure below is for N-methylmorpholine (NMM) as amine and MeCN used as solvent. The same procedure applies for N-methylpyrrolidine (NMP) as amine and other solvents.

General procedure for the aziridination using DppONH₂

NMM (28 μ L, 0.25 mmol) was added dropwise over 1 min to a solution of DppONH₂ (59 mg, 0.25 mmol) in MeCN (4 mL) at rt under N₂. The white mixture was allowed to stir for 30 min, then NaOH (19 mg, 0.48 mmol) and chalcone (50 mg, 0.24 mmol) were added sequentially. The mixture was allowed to stir at rt for 10 h (tlc indicated reaction was complete), then quenched by the addition of saturated NH₄Cl solution (2 mL). The solution was extracted with CH₂Cl₂ (3 $\times$ 10 mL), the organics combined, dried (Na₂SO₄) and concentrated in vacuo. Purification by column chromatography (100% petroleum ether to 10% EtOAc in petroleum ether) gave the aziridine (47.2 mg, 0.211 mmol, 88%) as a white solid: m.p. 99–101 °C (lit. 100–101 °C⁷). Data matches previously reported values. ¹H NMR (400 MHz, CDCl₃): δ 8.00 (d, 1H, J = 8.8 Hz, PhH), 8.00 (d, 1H, J = 8.8 Hz, PhH), 7.61 (td, 1H, J = 7.4, 1.2 Hz, PhH), 7.49 (t, 2H, J = 7.4 Hz, PhH), 7.39–7.30 (m, 5H, PhH), 3.52 (dd, 1H, J = 7.9, 2.1 Hz, CHNH), 3.18 (dd, 1H, J = 9.1, 2.1 Hz, CHNH), 2.68 (br dd, 1H, J = 9.1, 7.9 Hz, NH). ¹³C

⁴ E. W. Colvin, G. W. Kirby, A. C. Wilson, *Tetrahedron Lett.* **1982**, 23, 3835–3836.

⁵ M. G. Stocksdales, S. Ramurthy, M. J. Miller, *J. Org. Chem.* **1998**, 63, 1221–1225.

⁶ M. J. P. Harger, *J. Chem. Soc., Perkin Trans. 1*, **1981**, 3284–3288.

⁷ J. Xu, P. Jiao, *J. Chem. Soc., Perkin Trans. 1* **2002**, 1491–1493.

NMR (100 MHz, CDCl₃) δ 195.8, 138.4, 136.0, 133.9, 128.9, 128.6, 128.4, 128.0, 126.3, 44.2, 43.6.

Investigation of amine and solvent on the aziridination process

After the initial success of performing in situ amination of NMM and subsequent aziridination of chalcone, we set out to investigate the effect of amine and solvent on the reaction. A range of amines (aromatic, secondary and tertiary) were studied in the reaction and the results are shown in Table 4. Entry 1 shows the original result with NMM; this promoter is effective in several solvents, with CH₂Cl₂ and DMF giving comparable conversion to MeCN (entries 2 and 5). Pyridines and secondary amines are not effective amines for this reaction, with DMAP, pyridine, morpholine and diisopropylamine giving no aziridine after 24 hours (entries 18–24). With other tertiary amines, DABCO was found to be inferior to NMM under our in situ reaction conditions (entry 6). Quinuclidine gave poor results in MeCN, but reasonable conversion in CH₂Cl₂ and DMF (entries 7–9). N-Methylpiperidine was a poor promoter in MeCN (entry 10); N-methylpyrrolidine (NMP) gave equally good results as NMM (entries 11, 12 and 15) while triethylamine worked surprisingly well (entries 16 and 17). Overall, these results indicated NMM and NMP as the best promoters. We do not currently have a convincing rationale for the high solvent dependency on rates, but it may be associated with the relative solubilities of the intermediate hydrazinium salts.

Table 4. Investigation of the effect of amine and solvent on the aziridination method^a

Entry	Amine	Solvent; Time (h)	Conv (%) ^b	Yield (%) ^c
1	NMM	MeCN; 9	>95	88
2	NMM	CH ₂ Cl ₂ ; 3	90	88
3	NMM	THF; 5	-	86
4	NMM	Toluene; 24	~42	-
5	NMM	DMF; 6	88	82
6	DABCO	MeCN; 24	65	-
7	Quinuclidine	MeCN; 24	<2	-
8	Quinuclidine	CH ₂ Cl ₂ ; 10	89	80
9	Quinuclidine	DMF; 24	70	65
10	N-methylpiperidine	MeCN; 24	<2	-
11	NMP	MeCN; 8	>98	75
12	NMP	CH ₂ Cl ₂ ; 2	>95	94
13	NMP	THF; 10	62	58
14	NMP	Toluene; 24	>98	89
15	NMP	DMF; 2	>98	88
16	Triethylamine	MeCN; 24	80	57
17	Triethylamine	CH ₂ Cl ₂ ; 24	70	50
18	DMAP	MeCN; 24	<2	-
19	Pyridine	MeCN; 24	<2	-
20	Pyridine	CH ₂ Cl ₂ ; 24	~14 ^d	-
21	Morpholine	MeCN; 24	<2	-
22	Diisopropylamine	MeCN; 24	<2	-
23	Morpholine	CH ₂ Cl ₂ ; 24	<2	-
24	Diisopropylamine	CH ₂ Cl ₂ ; 24	<2	-

^a Reactions performed on 0.12 mmol scale. ^b Based on ¹H NMR analysis of the crude reaction mixture. ^c Isolated yields after column chromatography. ^d Product was identified in the crude ¹H NMR as reduction of the alkene, presumably by diimide formation.

Scope of the aziridination procedure

(3-(3-Nitrophenyl)aziridin-2-yl)(phenyl)methanone **Table 1, entry 1**

The general procedure for aziridination using DppONH₂, NMM and MeCN was followed. The reaction turned a deep purple colour and gave the aziridine (49 mg, 0.18 mmol, 75%) as a white solid: m.p. 120–122 °C. IR (neat) ν 1661, 1527, 1351, 1259 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 8.24 (s, 1H, PhH), 8.17 (d, 1H, *J* = 8.2 Hz, PhH), 8.00 (d, 2H, *J* = 7.3 Hz, PhH), 7.72 (d, 1H, *J* = 7.7 Hz, PhH), 7.64 (t, 1H, *J* = 7.4 Hz, PhH), 7.56–7.49 (m, 3H, PhH), 3.53 (dd, 1H, *J* = 7.7, 2.0 Hz, CHNH), 3.27 (dd, 1H, *J* = 8.9, 2.0 Hz, CHNH), 2.79–2.74 (br m, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 194.9, 148.5, 140.7, 135.6, 134.2, 132.4, 129.5, 128.9, 128.4, 122.8, 121.3, 43.9, 42.1. HRMS (CI, NH₃): *m/z* [M⁺ + H] calcd for C₁₅H₁₃N₂O₃: 269.0926; found: 269.0932.

4-(3-Benzoylaziridin-2-yl)benzonitrile **Table 1, entry 2**

The general procedure for aziridination using DppONH₂, NMM and MeCN was followed. The reaction turned an orange/red colour and gave the aziridine (33 mg, 0.13 mmol, 62%) as a slight yellow solid: m.p. 74–75 °C (lit. 113.5–113.7 °C⁸). Data matches previously reported values: ¹H NMR (400 MHz, CDCl₃): δ 7.98 (d, 2H, *J* = 7.3 Hz, PhH), 7.67–7.61 (m, 3H, PhH), 7.53–7.47 (m, 4H, PhH), 3.49 (br d, 1H, *J* = 5.7 Hz, CHNH), 3.20 (br d, 1H, *J* = 7.2 Hz, CHNH), 2.79–2.70 (br m, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 194.9, 143.8, 135.5, 134.2, 132.4, 128.9, 128.3, 127.0, 118.7, 111.5, 44.1, 42.5.

Phenyl(3-*p*-tolylaziridin-2-yl)methanone **Table 1, entry 3**

The general procedure for aziridination using DppONH₂, NMM and MeCN was followed. The reaction turned a slight yellow colour and gave the aziridine (44 mg, 0.185 mmol, 78%) as a white solid: m.p. 107–108 °C (lit. 106–107 °C⁷). Data matches previously reported values: ¹H NMR (400 MHz, CDCl₃): δ 8.03–8.00 (m, 2H, PhH), 7.65–7.62 (m, 1H, PhH), 7.53–7.50 (m, 2H, PhH), 7.30–7.28 (m, 2H, PhH), 7.21–7.19 (m, 2H, PhH), 3.52 (br s, 1H, CHNH), 3.18 (br s, 1H, CHNH), 2.68 (br, 1H, NH), 2.39 (s, 3H, CH₃). ¹³C NMR (100 MHz, CDCl₃) δ 195.8, 137.7, 136.0, 135.4, 133.8, 129.3, 128.8, 128.3, 126.1, 119.0, 44.2, 43.6, 21.2.

(3-(4-Methoxyphenyl)aziridin-2-yl)(phenyl)methanone **Table 1, entry 4**

The general procedure for aziridination using DppONH₂, NMP and CH₂Cl₂ was followed. The reaction turned a yellow colour and gave the aziridine (47.9 mg, 0.19 mmol, 79%) as a yellow oil. Data matches previously reported values: ¹H NMR (400 MHz, CDCl₃): δ 8.00–7.98 (m, 2H, PhH), 7.63–7.59 (m, 1H, PhH), 7.51–7.47 (m, 2H, PhH), 7.29 (d, 2H, *J* = 8.7 Hz, ArH), 6.90 (d, 2H, *J* = 8.7 Hz, ArH), 3.82 (s, 3H, CH₃), 3.48–3.47 (br m, 1H, CHNH), 3.15–3.13 (br m, 1H, CHNH), 2.71–2.58 (br m, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 195.9, 159.5, 136.0, 133.9, 130.5, 128.9, 128.4, 127.4, 114.1, 55.4, 44.3, 43.4.

⁸ Prepared by a two-step asymmetric aziridination of chalcones using rare-earth metal catalyzed Michael addition of hydroxylamines, see: X. L. Jin, H. Sugihara, K. Daikai, H. Tateishi, Y. Z. Jin, H. Furuno, J. Inanaga, *Tetrahedron* **2002**, 58, 8321–8329.

(4-Chlorophenyl)(3-phenylaziridin-2-yl)methanone **Table 1, entry 5**

The general procedure for aziridination using DppONH₂, NMM and MeCN was followed. The reaction turned a slight yellow colour and gave the aziridine (55.5 mg, 0.215 mmol, 90%) as a white solid: m.p. 76–78 °C (lit. 76–78 °C⁷). Data matches previously reported values: ¹H NMR (400 MHz, CDCl₃): δ 7.95–7.92 (m, 2H, PhH), 7.48–7.45 (m, 2H, PhH), 7.38–7.30 (m, 5H, PhH), 3.45 (br d, 1H, *J* = 4.8 Hz, CHNH), 3.19 (br d, 1H, *J* = 5.2 Hz, CHNH), 2.70–2.63 (br m, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 194.6, 140.4, 138.1, 134.2, 129.7, 129.2, 128.6, 128.0, 126.2, 44.1, 43.7.

(3-Phenylaziridin-2-yl)(*p*-tolyl)methanone **Table 1, entry 6**

The general procedure for aziridination using DppONH₂, NMM and MeCN was followed. The reaction turned a slight yellow colour and gave the aziridine (48.5 mg, 0.204 mmol, 86%) as a slight yellow solid: m.p. 89–90 °C (lit. 89–90 °C⁷). Data matches previously reported values: ¹H NMR (400 MHz, CDCl₃): δ 7.96–7.92 (m, 2H, PhH), 7.40–7.29 (m, 7H, PhH), 3.52 (br s, 1H, CHNH), 3.19 (br s, 1H, CHNH), 2.69 (br, 1H, NH), 2.45 (s, 3H, CH₃). ¹³C NMR (100 MHz, CDCl₃) δ 195.2, 144.9, 138.5, 133.5, 129.5, 129.3, 128.6, 128.5, 127.9, 126.2, 44.0, 43.4, 21.8.

(4-Methoxyphenyl)(3-phenylaziridin-2-yl)methanone **Table 1, entry 7**

The general procedure for aziridination using DppONH₂, NMM and MeCN was followed. The reaction turned a slight yellow colour and gave the aziridine (58.5 mg, 0.231 mmol, 97%) as a white solid: m.p. 50–51 °C (lit. 71–72 °C⁷). Data matches previously reported values: ¹H NMR (400 MHz, CDCl₃): δ 8.00–7.97 (m, 2H, PhH), 7.37–7.29 (m, 5H, PhH), 6.97–6.94 (m, 2H, PhH), 3.90 (s, 3H, OCH₃), 3.49 (d, 1H, *J* = 4.4 Hz, CHNH), 3.18 (d, 1H, *J* = 5.2 Hz, CHNH), 2.70–2.64 (br m, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 194.0, 164.2, 138.6, 130.7, 129.0, 128.6, 127.8, 126.2, 114.1, 55.6, 43.8, 43.1.

(3-(Naphthalen-1-yl)aziridin-2-yl)(phenyl)methanone **Table 1, entry 8**

The general procedure for aziridination using DppONH₂, NMP and CH₂Cl₂ was followed. The reaction turned an orange colour and gave the aziridine (43.9 mg, 0.16 mmol, 67%) as a slight yellow solid: m.p. 148–150 °C. IR (neat) ν 1664, 1450, 1261 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 8.07 (d, 2H, *J* = 7.4 Hz, ArH), 8.01 (d, 1H, *J* = 8.3 Hz, ArH), 7.91 (d, 1H, *J* = 8.1 Hz, ArH), 7.83 (d, 1H, *J* = 8.1 Hz, ArH), 7.77 (d, 1H, *J* = 7.1 Hz, ArH), 7.63 (t, 1H, *J* = 7.4 Hz, ArH), 7.53–7.42 (m, 5H, ArH), 3.78 (br s, 1H, CHNH), 3.54 (br s, 1H, CHNH), 2.73 (br s, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 196.3, 135.9, 134.0, 133.5, 131.9, 129.0, 128.9, 128.5, 128.2, 126.5, 126.1, 126.0, 125.7, 124.0, 122.8, 43.1, 41.9. HRMS (CI, NH₃): *m/z* [M⁺ + H] calcd for C₁₉H₁₆NO: 274.1232; found: 274.1240.

(3-(Furan-3-yl)aziridin-2-yl)(phenyl)methanone **Table 1, entry 9**

The general procedure for aziridination using DppONH₂, NMM and MeCN was followed. The reaction turned a deep brown colour and gave the aziridine (46.1 mg, 0.22 mmol, 90%) as a light brown solid: m.p. 76–77 °C. IR (neat) ν 1668, 1598, 1450, 1259, 1012 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 8.01 (d, 2H, *J* = 7.3 Hz, PhH), 7.65–7.61 (m, 1H, PhH), 7.53–7.49 (m, 3H, PhH, ArH), 7.41–7.40 (m, 1H, ArH), 6.42 (d, 1H, *J* = 0.9 Hz, ArH), 3.52 (br s, 1H, CHNH), 3.07 (br s, 1H, CHNH), 2.54 (br s, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 196.0, 143.6, 140.7, 136.0, 134.0, 128.9, 128.4, 124.1, 108.5, 42.7, 36.7. HRMS (CI, NH₃): *m/z* [*M*⁺ + *H*] calcd for C₁₃H₁₂NO₂: 214.0868; found: 214.0875.

(3-Butylaziridin-2-yl)(phenyl)methanone **Table 1, entry 10**

The general procedure for aziridination using DppONH₂, NMM and CH₂Cl₂ was followed. The aziridine (40.6 mg, 0.200 mmol, 83%) was isolated as a clear oil. Data matches previously reported values.⁹ ¹H NMR (400 MHz, CDCl₃): δ 8.04–8.01 (m, 2H, PhH), 7.64–7.60 (m, 1H, PhH), 7.53–7.50 (m, 2H, PhH), 3.26 (s, 1H, CHNH), 2.18–2.15 (m, 1H, CHNH), 2.06 (br s, 1H, NH), 1.62–1.57 (m, 2H, CH₂CHNH), 1.55–1.33 (m, 4H, CH₃CH₂CH₂), 0.92 (t, 3H, *J* = 7.2 Hz, CH₃). ¹³C NMR (100 MHz, CDCl₃) δ 197.4, 136.2, 133.7, 128.8, 128.2, 43.4, 39.9, 33.1, 29.4, 22.5, 14.0. HRMS (CI, NH₃): *m/z* [*M*⁺ + *H*] calcd for C₁₃H₁₈NO: 204.1388; found: 204.1385.

(3-Isobutylaziridin-2-yl)(phenyl)methanone **Table 1, entry 11**

The general procedure for aziridination using DppONH₂, NMM and CH₂Cl₂ was followed. The aziridine (38.1 mg, 0.187 mmol, 78%) was isolated as a clear oil. Data matches previously reported values.¹⁰ ¹H NMR (400 MHz, CDCl₃): δ 8.03–8.01 (m, 2H, PhH), 7.64–7.60 (m, 1H, PhH), 7.53–7.49 (m, 2H, PhH), 3.24 (d, 1H, *J* = 2.2 Hz, CHNH), 2.19 (td, 1H, *J* = 6.2, 2.2 Hz, CHNH), 2.00 (br, 1H, NH), 1.88–1.77 (m, 1H, CHCH₂), 1.55–1.45 (m, 2H, CH₂CHNH), 0.97 (d, 6H, *J* = 6.6 Hz, CH(CH₃)₂). ¹³C NMR (100 MHz, CDCl₃) δ 197.4, 136.3, 133.8, 128.9, 128.3, 42.5, 42.3, 40.0, 27.4, 22.9, 22.6.

(3-Phenethylaziridin-2-yl)(phenyl)methanone **Table 1, entry 12**

The general procedure for aziridination using DppONH₂, NMM and CH₂Cl₂ was followed. The aziridine (37.4 mg, 0.149 mmol, 62%) was isolated as a clear oil. IR (neat) ν 3267, 2925, 2856, 1668, 1451, 1259 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 7.87–7.83 (m, 2H, PhH), 7.63–7.60 (m, 1H, PhH), 7.50–7.46 (m, 2H, PhH), 7.28–7.19 (m, 5H, PhH), 3.21 (d, 1H, *J* = 2.2 Hz, CHNH), 2.93–2.74 (m, 2H, CH₂), 2.20 (td, 1H, *J* = 6.0, 2.2 Hz, CHNH), 2.03–1.87 (m, 3H, CH₂, NH). ¹³C NMR (100 MHz, CDCl₃) δ 197.3, 141.3, 136.1, 133.8, 128.9, 128.7, 128.5, 128.3, 126.2, 42.8, 40.0, 34.9, 33.5. HRMS (ES): *m/z* [*M*⁺ + *H*] calcd for C₁₇H₁₈NO: 252.1388; found: 252.1400; *m/z* [*M*⁺ + *Na*] calcd for C₁₇H₁₇NONa: 274.1208; found: 274.1216.

⁹ I. Coldham, A. J. Collis, R. J. Mould, R. E. Rathmell, *J. Chem. Soc., Perkin Trans. I* **1995**, 2739–2745.

¹⁰ N. Yamagiwa, H. Qin, S. Matsunaga, M. Shibasaki, *J. Am. Chem. Soc.* **2005**, 127, 13419–13427.

tert-Butyl aziridine-2-carboxylate **Table 1, entry 13**

The general procedure for aziridination using DppONH₂, NMM and CH₂Cl₂ was followed. The aziridine (27.5 mg, 0.192 mmol, 80%) was isolated as a clear oil (care should be taken as the aziridine is volatile). IR (neat) ν 2980, 1724, 1457, 1239, 1158 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 2.45 (dd, 1H, *J* = 5.4, 3.0 Hz, CHNH), 1.94 (dd, 1H, *J* = 3.0, 1.5 Hz, CHNH), 1.81 (d, 1H, *J* = 5.4 Hz, CHNH), 1.50 (s, 9H, C(CH₃)₃), 1.35 (br, 1H, NH). ¹³C NMR (100 MHz, CDCl₃) δ 172.1, 82.2, 29.9, 28.1, 27.0. HRMS (CI, NH₃): *m/z* [M⁺ + H] calcd for C₇H₁₄NO₂: 144.1025; found: 144.1027.

tert-Butyl 3-phenylaziridine-2-carboxylate **Table 1, entry 14**

The general procedure for aziridination using DppONH₂, NMM and CH₂Cl₂ was followed. The aziridine (8.5 mg, 0.039 mmol, 32%) was isolated as a clear oil. IR (neat) ν 3283, 2980, 2934, 1721, 1230, 1157 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 7.34–7.27 (m, 5H, PhH), 3.17 (br s, 1H, CHNH), 2.50 (br s, 1H, CHNH), 1.82 (br, 1H, NH), 1.50 (s, 9H, C(CH₃)₃). ¹³C NMR (100 MHz, CDCl₃) δ 170.9, 138.3, 128.6, 127.8, 126.3, 82.5, 40.6, 40.1, 28.2. HRMS (CI, NH₃): *m/z* [M⁺ + H] calcd for C₁₃H₁₈NO₂: 220.1338; found: 220.1342.

Sub-stoichiometric amine in the aziridination of chalcones

General procedure for the aziridination using DppONH₂ and sub-stoichiometric amine.

NMP (3.1 μ L, 0.030 mmol, 25 mol %) was added to a solution of DppONH₂ (7.25 mg, 0.0315 mmol) in DMF (2 mL) at rt. The clear solution was allowed to stir for 30 min, then NaOH (9.6 mg, 0.24 mmol) and chalcone (25 mg, 0.12 mmol) were added sequentially. The reaction was allowed to stir at rt for 1 h, then DppONH₂ (7.25 mg, 0.0315 mmol) was added. This was repeated a further twice, after which tlc indicated the reaction was nearly complete. The reaction mixture was quenched by the addition of saturated NH₄Cl solution (2 mL). The solution was extracted with CH₂Cl₂ (3 $\times$ 10 mL), the organics combined, dried (Na₂SO₄) and concentrated in vacuo. Purification by column chromatography (100% petroleum ether to 10% EtOAc in petroleum ether) gave the aziridine (17.6 mg, 0.079 mmol, 66%) as a white solid. Data matches previously reported values.

In situ hydrazinium salt formation and enantioselective aziridination of chalcone

Quinine (41 mg, 0.13 mmol) was added in one portion to a solution of DppONH₂ (29 mg, 0.13 mmol) in CH₂Cl₂ (2 mL) at rt. The white mixture was allowed to stir for 30 min, then *isopropanol* (20 μ L, 0.25 mmol), NaH (60% dispersion in mineral oil, 10 mg, 0.25 mmol) and chalcone (25 mg, 0.12 mmol) were added sequentially. The mixture was allowed to stir at rt for 18 h (tlc indicated reaction was complete), then quenched by the addition of saturated NH₄Cl solution (2 mL). The solution was extracted with CH₂Cl₂ (3 $\times$ 10 mL), the organics combined, dried (Na₂SO₄) and concentrated in vacuo. Purification by column chromatography (100% petroleum ether to 10% EtOAc in petroleum ether) gave the aziridine (17.2 mg, 0.08 mmol, 64%) as a white solid. Data matches previously reported values. [α]_D²² –139 (*c* 0.66, CHCl₃) for a sample of 56% ee (lit. [α]_D²² +251.3 (*c* 0.85, CHCl₃) for a sample of 95% ee¹¹).

Optical purity determined by chiral HPLC analysis in comparison with authentic racemic material.

Conditions: 93:7 hexane:IPA, 0.8 mL/min, 254 nm, AD-H.

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	13.157	BB	0.2519	7882.54980	489.60324	49.7964
2	15.018	BB	0.2986	7946.99219	415.94998	50.2036

authentic racemic

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	13.105	BB	0.2534	9707.99023	598.07959	21.7625
2	14.940	BB	0.3009	3.49009e4	1807.81433	78.2375

56% ee

Investigation into the enantioselective aziridination of chalcone using quinine

Table 5. Enantioselective aziridination of chalcone using quinine^a

Entry	Solvent	Base	Yield (%) ^b	ee (%) ^c
1	CH ₂ Cl ₂	NaH/ <i>i</i> -PrOH	64	56
2	CH ₂ Cl ₂	NaOH	59	46
3	Toluene	NaH/ <i>i</i> -PrOH	64	41
4	Toluene	NaOH	43	7
5	THF	NaH/ <i>i</i> -PrOH	0	-
6	THF	NaOH	0	-
7	DMF	NaOH	36	0

^a Reactions performed on 0.12 mmol scale. ^b Isolated yields after column chromatography. ^c Determined by chiral HPLC, for conditions see above.

cb-234

Av-400C

Current Data Parameters
 NAME Oct13-2006
 EXPNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061013
 Time 17.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 512
 DM 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.40 usec
 PL1 -3.00 dB
 SFO1 400.2024012 MHz

F2 - Processing parameters
 SI 16384
 SF 400.2000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

cb-148

Av-400A

Current Data Parameters
 NAME Jul20-2006
 EXPNO 160
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060721
 Time 3.33
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.803094 Hz
 AQ 0.6226420 sec
 RG 287
 DM 19.000 usec
 DE 6.00 usec
 TE 294.8 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 1.00 dB
 SFO1 100.6942501 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 19.79 dB
 PL13 19.79 dB
 SFO2 400.4119220 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6831750 MHz
 WDW EM
 SSB 0
 LB 2.50 Hz
 GB 0
 PC 1.40

cb-051

Av-400A **Table 1, entry 2**

Current Data Parameters
 NAME Oct17-2006
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061017
 Time 8.55
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 406
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.65 usec
 PL1 0.00 dB
 SFO1 400.4124025 MHz

F2 - Processing parameters
 SI 16384
 SF 400.4100000 MHz
 WDW EM
 SSB 0
 LB 0.55 Hz
 GB 0
 PC 1.00

cb-060

Table 1, entry 3

Av-400A

Current Data Parameters
 NAME Oct17-2006
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061017
 Time 8.59
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 406
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 1
 D10 1

----- CHANNEL f1 -----
 NUC1 1H
 P1 10.65 usec
 PL1 0.00 dB
 SFO1 400.4124025 MHz

F2 - Processing parameters
 SI 16384
 SF 400.4100000 MHz
 WDW EM
 SSB 0
 LB 0.55 Hz
 GB 0
 PC 1.00

C Baxter CB-060 in CDCl3 ; ¹³C{¹H} spectrum using Av500 ; April-2006/21

Av-500

Current Data Parameters
 NAME April-2006
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060411
 Time 14.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2000
 DS 2
 SWH 35714.285 Hz
 FIDRES 1.089913 Hz
 AQ 0.4588030 sec
 RG 128
 DW 14.000 usec
 DE 6.00 usec
 TE 294.8 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 D10 1

----- CHANNEL f1 -----
 NUC1 13C
 P1 9.00 usec
 PL1 1.50 dB
 SFO1 125.7703648 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCDP2 100.00 usec
 PL2 1.00 dB
 PL12 20.37 dB
 PL13 20.37 dB
 SFO2 500.1325006 MHz

F2 - Processing parameters
 SI 16384
 SF 125.7577890 MHz
 WDW RM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.00

cb-201

Av-400A

Current Data Parameters
 NAME Sep01-2006
 EXPNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060901
 Time 17.46
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 181
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.25 usec
 PL1 0.00 dB
 SFO1 400.4124025 MHz

F2 - Processing parameters
 SI 16384
 SF 400.4100000 MHz
 WDW EM
 SSB 0
 LB 0.55 Hz
 GB 0
 PC 1.00

cb-201

Av-400A

Current Data Parameters
 NAME Sep01-2006
 EXPNO 41
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060902
 Time 4.57
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.803094 Hz
 AQ 0.6226420 sec
 RG 287
 DM 19.000 usec
 DE 8.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 1.00 dB
 SFO1 100.6942501 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 19.79 dB
 PL13 19.79 dB
 SFO2 400.4119220 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6831750 MHz
 WDW EM
 SSB 0
 LB 2.50 Hz
 GB 0
 PC 1.40

cb-100

Av-400C

Current Data Parameters
 NAME Oct13-2006
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061013
 Time 17.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 362
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.40 usec
 PL1 -3.00 dB
 SFO1 400.2024012 MHz

F2 - Processing parameters
 SI 16384
 SF 400.2000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

cb-100

Av-400A

Current Data Parameters
 NAME Aug07-2006
 EXPNO 71
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060807
 Time 22.59
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.803094 Hz
 AQ 0.6226420 sec
 RG 287
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 0.00 dB
 SFO1 100.6261501 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FPCPD2 100.00 usec
 PL2 0.00 dB
 PL12 19.79 dB
 PL13 19.79 dB
 SFO2 400.4119220 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6261501 MHz
 WDW EM
 SSB 0
 LB 2.50 Hz
 GB 0
 PC 1.40

cb-183

Table 1, entry 9

Av-400A

Current Data Parameters
 NAME Aug03-2006
 EXPNO 130
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060803
 Time 17.53
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 406
 DW 62.400 usec
 DE 6.00 usec
 TE 294.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.25 usec
 PL1 0.00 dB
 SFO1 400.4124025 MHz

F2 - Processing parameters
 SI 16384
 SF 400.4100000 MHz
 WDW EM
 SSB 0
 LB 0.55 Hz
 GB 0
 PC 1.00

cb-183

Av-400A

Current Data Parameters
 NAME Aug03-2006
 EXPNO 131
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060804
 Time 21.59
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.803094 Hz
 AQ 0.6226420 sec
 RG 287
 DW 19.000 usec
 DE 8.00 usec
 TE 294.5 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TD0 1

===== CHANNEL F1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 1.00 dB
 SFO1 100.6261501 MHz

===== CHANNEL F2 =====
 CDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 19.79 dB
 PL13 19.79 dB
 SFO2 400.4119220 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6261500 MHz
 WDW EM
 SSB 0
 LB 2.50 Hz
 GB 0
 PC 1.40

cb-184

Table 1, entry 10

Av-400B

Current Data Parameters
 NAME Aug04-2006
 EXPNO 100
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060804
 Time 13.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 161
 DW 62.400 usec
 DE 6.00 usec
 TE 296.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 -3.00 dB
 SFO1 400.3224019 MHz

F2 - Processing parameters
 SI 16384
 SF 400.3200000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

cb-184

Av-400B

Current Data Parameters
 NAME Aug04-2006
 EXPNO 101
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060804
 Time 18.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 216
 DS 4
 SWH 28409.092 Hz
 FIDRES 0.866977 Hz
 AQ 0.5767668 sec
 RG 203
 DM 17.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL F1 =====
 NUC1 13C
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 100.6716167 MHz

===== CHANNEL F2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.06 dB
 PL13 18.00 dB
 SFO2 400.3220016 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6605440 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.20

cb-217

Table 1, entry 11

Av-400A

Current Data Parameters
 NAME Sep12-2006
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060912
 Time 16.44
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 256
 DW 62.400 usec
 DE 6.00 usec
 TE 295.5 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.25 usec
 PL1 0.00 dB
 SFO1 400.4124025 MHz

F2 - Processing parameters
 SI 16384
 SF 400.4100000 MHz
 WDW EM
 SSB 0
 LB 0.55 Hz
 GB 0
 PC 1.00

cb-217

Av-400A

Current Data Parameters
 NAME Sep12-2006
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060912
 Time 22.13
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.803094 Hz
 AQ 0.6226420 sec
 RG 287
 DW 19.000 usec
 DE 6.00 usec
 TE 294.5 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 1.00 dB
 SFO1 100.6942501 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 19.79 dB
 PL13 19.79 dB
 SFO2 400.4119220 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6831750 MHz
 WDW EM
 SSB 0
 LB 2.50 Hz
 GB 0
 PC 1.40

cb-224

Av-400A

Current Data Parameters
 NAME Sep19-2006
 EXPNO 80
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060919
 Time 16.45
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 362
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.25 usec
 PL1 0.00 dB
 SFO1 400.4124025 MHz

F2 - Processing parameters
 SI 16384
 SF 400.4100000 MHz
 WDW EM
 SSB 0
 LB 0.55 Hz
 GB 0
 PC 1.00

cb-224

Av-400A

Current Data Parameters
 NAME Sep19-2006
 EXPNO 81
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060920
 Time 1.59
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2500
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.803094 Hz
 AQ 0.6226420 sec
 RG 287
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TDO 1

===== CHANNEL F1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 1.00 dB
 SFO1 100.6261501 MHz

===== CHANNEL F2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 19.79 dB
 PL13 19.79 dB
 SFO2 400.4119220 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6261500 MHz
 WDW EM
 SSB 0
 LB 2.50 Hz
 GB 0
 PC 1.40

cb-200

Table 1, entry 13

Av-400A

Current Data Parameters
 NAME Aug31-2006
 EXPNO 80
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060831
 Time 17.41
 INSTRUM spect
 PROBN 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 256
 DW 62.400 usec
 DE 6.00 usec
 TE 294.3 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.25 usec
 PL1 0.00 dB
 SFO1 400.4124025 MHz

F2 - Processing parameters
 SI 16384
 SF 400.4100000 MHz
 WDW EM
 SSB 0
 LB 0.55 Hz
 GB 0
 PC 1.00

cb-200

Av-400A

Current Data Parameters
 NAME Aug31-2006
 EXPNO 81
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060901
 Time 3.05
 INSTRUM spect
 PROBN 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.803094 Hz
 AQ 0.6226420 sec
 RG 287
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TDO 1

===== CHANNEL F1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 1.00 dB
 SFO1 100.6942501 MHz

===== CHANNEL F2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 19.79 dB
 PL13 19.79 dB
 SFO2 400.4119220 MHz

F2 - Processing parameters
 SI 16384
 SF 100.6831750 MHz
 WDW EM
 SSB 0
 LB 2.50 Hz
 GB 0
 PC 1.40

cb-203

Av-400A

Current Data Parameters
NAME Sep05-2006
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060905
Time 17.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 362
DW 62.400 usec
DE 6.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.25 usec
PL1 0.00 dB
SFO1 400.4124025 MHz

F2 - Processing parameters
SI 16384
SF 400.4100000 MHz
WDW EM
SSB 0
LB 0.55 Hz
GB 0
PC 1.00

cb-203

Av-400A

Current Data Parameters
NAME Sep05-2006
EXPNO 61
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060906
Time 2.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 2000
DS 4
SWH 26315.789 Hz
FIDRES 0.803094 Hz
AQ 0.6226420 sec
RG 287
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 1.00 dB
SFO1 100.6942501 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.79 dB
PL13 19.79 dB
SFO2 400.4119220 MHz

F2 - Processing parameters
SI 16384
SF 100.6831750 MHz
WDW EM
SSB 0
LB 2.50 Hz
GB 0
PC 1.40