**Enantioselective Friedel-Crafts Reaction of Indoles with Carbonyl Compounds
Catalyzed by Bifunctional Cinchona Alkaloids**

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Supporting Information

General Information. ^1H and ^{13}C NMR spectra were recorded on a Varian instrument (400 MHz and 100 MHz, respectively) and internally referenced to tetramethylsilane signal or residual protio solvent signals. Data for ^1H NMR are recorded as follows: chemical shift (δ , ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet), integration, coupling constant (Hz). Data for ^{13}C NMR are reported in terms of chemical shift (δ , ppm). Infrared spectra were recorded on a Perkin Elmer FT-IR Spectrometer and are reported in frequency of absorption. Low resolution mass spectra for all the new compounds were performed by 70SE CI+, and were recorded and exact mass spectra on a 70-VSE-B high resolution mass spectrometer. Specific rotations were measured on a Jasco Digital Polarimeter.

High performance liquid chromatography (HPLC) analysis was performed on a Hewlett-Packard 1100 Series instrument equipped with a quaternary pump, using a Daicel Chiralcel OJ, OD Column (250 x 4.6 mm) or Chiralpak AD, AS Column (250 x 4.6 mm). UV absorption was monitored at 220 nm or at 254 nm.

Materials: α -Ketoesters **3a**, **3b** were prepared according to literature procedures.¹ α -ketoesters **3c-g** and aldehydes **5d-f** were commercially available and purified by flash chromatography (silica gel 60, 0.040-0.063 mm, purchased from EM SCIENCE Inc.) before they were used for the Friedel-Crafts reaction. Aldehyde **5a** was used as ~50% solution in toluene, which was commercially available. Indoles **2**, aldehydes **5b-c** and quinidine (**QD**), cinchonine (**CN**) were commercially available and used without any further purification. C6'-OH cinchona alkaloids **Q-1a-d** and **QD-1a-d** were prepared following procedures reported from these laboratories.²

General procedure for asymmetric addition of indoles to α -ketoesters and aldehydes:

Table 1: Highly Enantioselective Friedel-Crafts Addition of Indoles **2** to α -Ketoesters **3** with Q-1d and QD-1d (in parentheses) ^a

$\text{Indole } \mathbf{2} \text{ (0.4 mmol)} + \text{R}'\text{-C(=O)CO}_2\text{Et } \mathbf{3} \text{ (0.2 mmol)} \xrightarrow{\text{Q-1d (QD-1d) (10mol\%)}} \text{Product } \mathbf{4}$

entry	R		R'		T/ °C	t/h	Yield/% ^b	ee/% ^c
1	H	2A	Bu ^t -	3a	23	63(62)	89(96)	87(88)
2	6-MeO	2B	Bu ^t -	3a	23	40(40)	86(79)	84(88)
3	H	2A	Ph-	3b	23	44(43)	79(80)	88(90)
4	H	2A	4-CN-Ph-	3c	23	56(55)	88(77)	97(97)
5	4-MeO	2C	4-CN-Ph-	3c	23	64(64)	79(79)	99(99)
6	5-Cl	2D	4-CN-Ph-	3c	23	71(70)	85(96)	96(94)
7	6-MeO	2B	4-CN-Ph-	3c	23	58(59)	97(97)	97(96)
8	6-Br	2E	4-CN-Ph-	3c	23	61(61)	67(80)	95(95)
9	7-Me	2F	4-CN-Ph-	3c	23	66(66)	96(85)	97(97)
10	H	2A	4-NO ₂ -Ph-	3d	23	72(71)	88(95)	98(95)
11	H	2A	4-Cl-Ph-	3e	23	(24)	(13 ^g)	(95)
12 ^d	H	2A	4-Cl-Ph-	3e	23	72(88)	95(93)	95(95)
13 ^e	H	2A	4-Cl-Ph-	3e	70	24(24)	97(96)	89(86)
14 ^d	H	2A	Ph-	3f	23	88(88)	63(71)	94(93)
15 ^e	H	2A	Ph-	3f	70	36(36)	92(91)	86(86)
16 ^f	6-MeO	2B	Ph-	3f	70	28(29)	92(72)	84(82)
17 ^e	6-Cl	2G	Ph-	3f	70	51(51)	96(87)	84(81)
18 ^e	H	2A	4-MeO-Ph-	3g	70	56	52	83

^a Unless noted, reactions were carried with 0.2 mmol of **3**, 0.4 mmol of **2** in 0.4 mL Et₂O with 10 mol% Q-1d, the results in parentheses were obtained with QD-1d to give opposite enantiomer, see Supporting Information for details; ^b Isolated yield; ^c Determined by HPLC analysis; ^d The reaction were carried out in ether (10 M); ^e The reaction were carried out in TBME(10 M); ^f The reaction were carried out in TBME(4 M); ^g Conversion is determined by ¹HNMR analysis.

General procedure for Friedel-Crafts addition of indoles **2 to α -ketoester **3**:**

At room temperature to the solution of pyruvates **3a-d** (0.2 mmol) in Et₂O (0.4 mL) was added Q-**1d** or QD-**1d** (10 mol %) and followed by addition of indoles (0.4 mmol). Then the mixture was let stand at room temperature for 40-72 hours (as indicated in Table 1, entries 1-10). The reaction mixture was subjected to flash chromatography to afford the desired product **4**. For entries 12 and 14 in Table 1, 0.02 mL of Et₂O was added to the reaction mixture. For entries 13, 15, 17, 18, these reactions were carried out at 70°C in 0.02 mL of *t*-butyl methyl ether (TBME). For entries 16, the reaction was carried out at 70°C in 0.05 mL of *t*-butyl methyl ether (TBME).

Table 2: Asymmetric Friedel-Crafts Addition of Indoles **2** to Aldehydes **5** with Q-**1d** and QD-**1d** (in parentheses)

Entry	R		R'		T/°C	t/h	6/7 [a]	yield/% [b]	ee/% [c]
1	H	2A	EtO ₂ C-	5a^d	r.t.	6	>95/5	95(85)	93(93)
2	6-Br	2E	EtO ₂ C-	5a^d	r.t.	4	>95/5	96(95)	90(90)
3	6-OMe	2B	EtO ₂ C-	5a^d	r.t.	4	>95/5	94(93)	90(82)
4	H	2A	2-NO ₂ -Ph-	5b	r.t.	72	>95/5	96(95)	90(89)
5	H	2A	4-NO ₂ -Ph-	5c	r.t.	72	>95/5	90(90)	88(83)
6	H	2A	4-CF ₃ -Ph-	5d	r.t.	72	>95/5	85(83)	88(88)
7	H	2A	4-Cl-Ph-	5e	70	40	7/1	75	83
8	H	2A	Ph-	5f	70	48	2/1	60	82

[a] The ratio was determined by ¹HNMR analysis of crude product; [b] Isolated yield;

[c] Determined by HPLC analysis; [d] ~50 wt% in toluene.

General procedure for Friedel-Crafts addition of indoles **2 to aldehyde **5**:**

At room temperature to a solution of the aldehyde (**5a-d**) (0.2 mmol) in TBME (0.02 mL) was added Q-**1d** or QD-**1d** (10 mol %) and followed by the addition of the indoles (0.4 mmol). Then the mixture was allowed to stand at room temperature for 4-72 hours (as

indicated in Table 2, entries 1-6) or at 70 °C for 40-48 h (entries 7-8, Table 2). The reaction mixture was subjected to flash chromatography to afford the desired product **6**.

Data for products **4** and **6**:

4Aa

(-)-4Aa was obtained in 89 % yield as a foam after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/3) and 87% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 75:25, 1.0 ml/min, λ 220 nm, $t(\text{minor})$ = 14.2 min, $t(\text{major})$ = 7.7 min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 63 hours. $[\alpha]_{\text{D}}^{25}$ = -1.8 (c 1.0, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 8.14 (br, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.44-7.42 (m, 1H), 7.34-7.32 (m, 1H), 7.19 (t, J = 7.2 Hz, 1H), 7.11 (t, J = 7.2 Hz, 1H), 4.39-4.31 (m, 1H), 4.17-4.09 (m, 1H), 4.07 (s, 1H), 1.29 (s, 9H), 1.21 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.5, 136.8, 124.4, 123.9, 122.2, 120.2, 119.8, 115.7, 111.3, 93.8, 77.2, 68.9, 63.0, 30.7, 27.5, 13.8; IR (CHCl_3) ν 3408 (br), 3058, 2970, 2242, 1731, 1619, 1543, 1458, 1255, 1033; HRMS (CI) m/z calcd. for ($\text{C}_{18}\text{H}_{21}\text{NO}_3 + \text{H}^+$): 300.1599, found: 300.1592.

(+)- 4Aa was obtained as a foam in 96% yield and 88% ee from a reaction catalyzed by **QD-1d** (10 mol %) at room temperature for 62 hours.

4Ba

(+)-4Ba was obtained in 79 % yield as a foam after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/4) and 88% ee as determined by HPLC analysis [Chiralcel OD, Hexanes:IPA, 90:10, 1.0 ml/min, λ 220 nm, $t(\text{minor})$ = 14.6 min, $t(\text{major})$ = 16.5 min] from a reaction catalyzed by **QD-1d** (10 mol%) at room temperature for 40 hours. $[\alpha]_{\text{D}}^{25}$ = +3.6 (c 1.08, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 8.07 (s, 1H), 7.62 (d, J = 8.8 Hz, 1H), 7.22 (d, J = 2.4 Hz, 1H), 6.76 (dd, J = 8.8 Hz, 2.4 Hz, 1H), 6.68 (d, J = 2.4 Hz, 1H), 4.35-4.29 (m, 1H), 4.14-4.08 (m, 1H), 4.09 (s, 1H), 3.76 (s, 3H), 1.25 (s, 9H), 1.21-1.16 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.5,

156.3, 137.6, 122.7, 120.7, 118.7, 115.7, 109.9, 94.5, 93.7, 77.2, 68.9, 63.0, 55.4, 30.6, 27.5, 13.8; IR (CHCl₃) ν 3405 (br), 2970, 2869, 2246, 1731, 1630, 1547, 1456, 1254, 1032.

(-)- **4Ba** was obtained as a foam in 86% yield and 84% ee from a reaction catalyzed by **Q-1d** (10 mol %) at room temperature for 40 hours.

(+)-**4Ab** This product was obtained in 79 % yield as a foam after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/3) and 88% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 70:30, 1.0 ml/min, λ 220 nm, t (minor) = 17.9 min, t (major) = 14.2

min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 44 hours. $[\alpha]_D^{25} = +14.2$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.20 (br, 1H), 7.83 (d, J = 8.0 Hz, 1H), 7.53-7.50 (m, 3H), 7.37-7.31 (m, 4H), 7.21 (t, J = 7.2 Hz, 1H), 7.14 (t, J = 7.2 Hz, 1H), 4.40-4.32 (m, 1H), 4.26 (s, 1H), 4.27-4.19 (m, 1H), 1.23 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 172.0, 136.8, 131.9, 128.8, 128.3, 124.4, 124.1, 122.3, 122.0, 120.1, 115.2, 111.4, 87.3, 84.8, 69.4, 63.4, 13.9; IR (CHCl₃) ν 3403 (br), 3060, 2981, 2226, 1729, 1596, 1458, 1242.

(-)- **4Ab** was obtained as a foam in 80% yield and 90% ee from a reaction catalyzed by **QD-1d** (10 mol %) at room temperature for 43 hours.

(+)-**4Ac** was obtained as a white powder in 88 % yield after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/2) and 97% ee as determined by HPLC analysis [Chiralcel OD, Hexanes:IPA, 80:20, 1.0 ml/min, λ

220 nm, t (minor) = 12.5 min, t (major) = 15.6 min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 56 hours. $[\alpha]_D^{25} = +30.0$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.17 (br, 1H), 7.74 (d, J = 8.8 Hz, 2H), 7.63 (d, J = 8.8 Hz, 2H), 7.38 (d, J = 7.6 Hz, 1H), 7.35 (d, J = 7.6 Hz, 1H), 7.20 (t, J = 7.6 Hz, 1H), 7.12 (d, J = 2.4 Hz,

1H), 7.04 (t, $J = 7.6$ Hz, 1H), 4.33 (q, $J = 7.2$ Hz, 2H), 4.28 (s, 1H), 1.26 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 173.5, 146.4, 136.7, 131.8, 127.9, 125.0, 123.6, 122.6, 120.5, 120.1, 118.7, 116.8, 111.7, 111.4, 77.3, 63.2, 14.0; IR (CHCl_3) ν 3406 (br), 2983, 2230 (CN), 1727, 1605, 1458, 1241; HRMS (CI) m/z calcd. for ($\text{C}_{19}\text{H}_{16}\text{N}_2\text{O}_3 + \text{H}^+$): 321.1239, found: 321.1243.

(-)-**4Ac** was obtained as a white powder in 77% yield and 97% ee from a reaction catalyzed by QD-**1d** (10 mol %) at room temperature for 55 hours.

(-)-**4Cc** was obtained in 79 % yield as a white powder after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/2) and 99% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 75:25, 1.0 ml/min, λ 220 nm, $t(\text{minor}) = 11.5$ min, $t(\text{major}) = 13.2$

min] from a reaction catalyzed by Q-**1d** (10 mol%) at room temperature for 64 hours. $[\alpha]_{\text{D}}^{25} = -95.8$ (c 1.0, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 8.06 (br, 1H), 7.86 (d, $J = 8.8$ Hz, 2H), 7.67 (d, $J = 8.8$ Hz, 2H), 7.16 (t, $J = 8.0$ Hz, 1H), 7.02 (d, $J = 8.0$ Hz, 1H), 6.55 (d, $J = 8.0$ Hz, 1H), 6.54 (d, $J = 2.4$ Hz, 1H), 5.05 (s, 1H), 4.31-4.24 (m, 2H), 3.86 (s, 3H), 1.25 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.9, 152.0, 146.4, 138.4, 131.7, 127.4, 123.7, 123.0, 118.9, 118.8, 115.4, 111.6, 105.4, 100.5, 62.2, 55.1, 14.1; IR (CHCl_3) ν 3397 (br), 2981, 2228, 1732, 1619, 1587, 1508, 1236, 1091; HRMS (CI) m/z calcd. for ($\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_4 + \text{H}^+$): 351.1345, found: 351.1349.

(+)-**4Cc** was obtained as a white powder in 79% yield and 99% ee from a reaction catalyzed by QD-**1d** (10 mol %) at room temperature for 64 hours.

(-)-(R)-**4Dc** was obtained in 85 % yield as a white powder after flash chromatography (elution solvent: Ethyl Acetate/Hexane = 1/2) and 96% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 80:20, 1.0 ml/min, λ 220 nm, $t(\text{minor}) = 15.3$ min, $t(\text{major}) = 13.0$ min] from a reaction

catalyzed by **Q-1d** (10 mol%) at room temperature for 71 hours. $[\alpha]_D^{25} = -1.4$ (c 1.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.19 (br, 1H), 7.70 (d, $J = 8.0$ Hz, 2H), 7.64 (d, $J = 8.0$ Hz, 2H), 7.38 (s, 1H), 7.30 (d, $J = 9.2$ Hz, 1H), 7.17-7.15 (m, 2H), 4.33 (q, $J = 7.2$ Hz, 2H), 4.31 (s, 1H), 1.27 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 173.3, 146.1, 135.1, 131.9, 127.8, 126.2, 126.0, 124.8, 123.2, 120.2, 118.6, 116.7, 112.4, 112.0, 77.1, 63.5, 14.0; IR (CHCl₃) ν 3413 (br), 2983, 2231, 1729, 1606, 1569, 1464, 1239; HRMS (CI) m/z calcd. for (C₁₉H₁₅N₂O₃Cl + H⁺): 355.0849, found: 355.0855.

(+)-(S)-4Dc was obtained as a white powder in 96% yield and 94% ee from a reaction catalyzed by **QD-1d** (10.0 mol %) at room temperature for 70 hours. **The absolute configuration of (+)-4Dc was determined to be S by X-ray analysis.**

(+)-4Bc was obtained in 97 % yield as a thick oil after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/2) and 97% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 65:35, 1.0 ml/min, λ 220 nm, $t(\text{minor}) = 13.4$ min, $t(\text{major}) = 16.0$ min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 58 hours. $[\alpha]_D^{25} = +25.1$ (c 1.3, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.03 (br, 1H), 7.73 (d, $J = 8.4$ Hz, 2H), 7.62 (d, $J = 8.4$ Hz, 2H), 7.21 (d, $J = 8.4$ Hz, 1H), 7.00 (d, $J = 2.0$ Hz, 1H), 6.85 (d, $J = 2.0$ Hz, 1H), 6.70 (dd, $J = 2.0$ Hz, 8.4 Hz, 1H), 4.32 (q, $J = 7.2$ Hz, 2H), 4.25 (s, 1H), 3.82 (s, 3H), 1.26 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 173.5, 156.6, 146.5, 137.5, 131.7, 127.9, 122.4, 121.2, 119.4, 118.7, 116.8, 111.6, 110.1, 94.6, 77.3, 63.2, 55.5, 14.0; IR (CHCl₃) ν 3405 (br), 2982, 2229, 1727, 1629, 1544, 1457, 1257; HRMS (CI) m/z calcd. for (C₂₀H₁₈N₂O₄ + H⁺): 351.1345, found: 351.1352.

(-)-4Bc was obtained as a thick oil in 97% yield and 96% ee from a reaction catalyzed by **QD-1d** (10 mol %) at room temperature for 59 hours.

(+)-4Ec was obtained in 67 % yield as a white powder after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/2) and 95% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 70:30, 0.9 ml/min, λ 254 nm, $t(\text{minor})$ = 11.4 min, $t(\text{major})$ = 13.4

min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 61 hours. $[\alpha]_D^{25}$ = + 13.8 (c 1.0, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 8.18 (br, 1H), 7.70 (d, J = 8.4 Hz, 2H), 7.63 (d, J = 8.4 Hz, 2H), 7.54 (s, 1H), 7.22 (d, J = 8.4 Hz, 1H), 7.13 (d, J = 8.4 Hz, 1H), 7.11 (br, 1H), 4.32 (q, J = 7.2 Hz, 2H), 4.31 (s, 1H), 1.26 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 173.3, 146.2, 137.5, 131.9, 127.8, 124.1, 124.0, 123.5, 121.9, 118.6, 117.1, 116.3, 114.3, 111.9, 77.1, 63.4, 14.0; IR (CHCl_3) ν 3411 (br), 2982, 2230, 1728, 1607, 1539, 1454, 1252; HRMS (CI) m/z calcd. for ($\text{C}_{19}\text{H}_{15}\text{N}_2\text{O}_3\text{Br} + \text{H}^+$): 399.0344, found: 399.0335.

(-)-4Ec This product was obtained as a white powder in 80% yield and 95% ee from a reaction catalyzed by **QD-1d** (10 mol %) at room temperature for 61 hours.

(+)-4Fc was obtained in 96 % yield as a thick oil after flash chromatography (elution gradient: Ethyl Acetate/Hexanes = 1/2) and 97% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 75:25, 1.0 ml/min, λ 220 nm, $t(\text{minor})$ = 14.7 min, $t(\text{major})$ = 16.8 min] from a reaction catalyzed by

Q-1d (10 mol%) at room temperature for 66 hours. $[\alpha]_D^{25}$ = + 32.9 (c 1.3, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 8.11 (br, 1H), 7.74 (d, J = 6.8 Hz, 2H), 7.62 (d, J = 6.8 Hz, 2H), 7.19 (d, J = 8.0 Hz, 1H), 7.12 (d, J = 2.4 Hz, 1H), 7.01 (d, J = 6.8 Hz, 1H), 6.96 (t, J = 8.0 Hz, 1H), 4.32 (q, J = 6.8 Hz, 2H), 4.26 (s, 1H), 2.49 (s, 3H), 1.26 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 173.6, 146.5, 136.3, 131.8, 128.0, 124.6, 123.3, 123.1, 120.5, 120.4, 118.8, 118.3, 117.4, 111.7, 63.2, 16.5, 14.0; IR (CHCl_3) ν 3405 (br), 2981, 2230, 1727, 1605, 1498, 1248; HRMS (CI) m/z calcd. for ($\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_3 + \text{H}^+$): 335.1395, found: 335.1391.

(-)-**4Fc** was obtained as a thick oil in 85% yield and 97% ee from a reaction catalyzed by QD-**1d** (10 mol %) at room temperature for 66 hours.

(+)-**4Ad** was obtained as a yellow powder in 88 % yield after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/3) and 98% ee as determined by HPLC analysis [Chiralcel AS, Hexanes:IPA, 80:20, 1.0 ml/min, λ 220 nm, $t(\text{minor})$ = 16.7 min, $t(\text{major})$ = 22.7

min] from a reaction catalyzed by Q-**1d** (10 mol%) at room temperature for 72 hours. $[\alpha]_D^{25}$ = + 30 (c 1.28, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 8.20 (br, 1H), 8.18 (d, J = 9.2 Hz, 2H), 7.80 (d, J = 9.2 Hz, 2H), 7.36 (t, J = 8.4 Hz, 2H), 7.20 (t, J = 7.6 Hz, 1H), 7.11-7.10 (m, 1H), 7.03 (t, J = 7.2 Hz, 1H), 4.33 (q, J = 7.2 Hz, 2H), 4.32 (s, 1H), 1.26 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 173.4, 148.3, 147.5, 136.7, 128.2, 125.0, 123.5, 123.1, 122.7, 120.5, 120.2, 116.9, 111.4, 77.2, 63.3, 13.4; IR (CHCl_3) ν 3410 (br), 2983, 1727, 1596, 1520, 1349, 1243; HRMS (CI) m/z calcd. for ($\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_5 + \text{H}^+$): 341.1137, found: 321.1143.

(-)-**4Ad** was obtained as a yellow powder in 95% yield and 95% ee from a reaction catalyzed by QD-**1d** (10 mol %) at room temperature for 71 hours.

(-)-**4Ae** was obtained as a white powder in 93 % yield after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/3) and 95% ee as determined by HPLC analysis [Chiralpak OD + OD, Hexanes:IPA, 85:15, 0.9 ml/min, λ 220 nm, $t(\text{minor})$ = 29.7 min, $t(\text{major})$ = 26.7 min] from a reaction

catalyzed by QD-**1d** (10 mol%) at room temperature for 88 hours. $[\alpha]_D^{25}$ = - 5.9 (c 1.2, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 8.15 (br, 1H), 7.56-7.54 (m, 2H), 7.41 (d, J = 8.4 Hz, 1H), 7.33-7.30 (m, 3H), 7.18 (t, J = 7.2 Hz, 1H), 7.04 (t, J = 7.2 Hz, 1H), 7.00 (d, J = 2.4 Hz, 1H), 4.32-4.29 (m, 3H), 1.24 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 175.0, 140.5, 137.4, 134.6, 129.2, 128.9, 126.0, 124.5, 123.1, 121.5, 120.7, 118.2, 111.9,

77.8, 63.7, 14.7; IR (CHCl₃) ν 3407 (br), 3059, 2982, 1722, 1543, 1489, 1458, 1242, 1092;

(+)-**4Ae** was obtained as a white powder in 95% yield and 95% ee from a reaction catalyzed by **Q-1d** (10 mol %) at room temperature for 72 hours.

(-)-**4Ae** was obtained as a white powder in 96% yield and 86% ee from a reaction catalyzed by **QD-1d** (10 mol %) at 70 °C for 24 hours.

(+)-**4Ae** was obtained as a white powder in 97% yield and 89% ee from a reaction catalyzed by **Q-1d** (10 mol %) at 70 °C for 24 hours.

(-)-**4Af** was obtained as a white powder in 63 % yield after flash chromatography (elution solvent: Ethyl Acetate/Hexanes = 1/3) and 94% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 80:20, 1.0 ml/min, λ 220 nm, t (minor) = 9.8 min, t (major) = 14.7 min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 88 hours. $[\alpha]_D^{25} = -11.5$ (c 0.95, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.15 (br, 1H), 7.63-7.61 (m, 2H), 7.44 (d, J = 8.4 Hz, 1H), 7.36-7.34 (m, 3H), 7.30 (d, J = 7.6 Hz, 1H), 7.17 (t, J = 7.6 Hz, 1H), 7.04 (t, J = 7.6 Hz, 1H), 6.98 (d, J = 2.4 Hz, 1H), 4.32-4.29 (m, 3H), 1.23 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 175.5, 141.9, 137.4, 128.7, 128.6, 127.6, 126.2, 124.8, 122.9, 121.6, 120.5, 118.4, 111.9, 78.2, 63.5, 14.7; IR (CHCl₃) ν 3408 (br), 3058, 2981, 1720, 1543, 1493, 1458, 1241, 1095;

(+)- **4Af** was obtained as a white powder in 71% yield and 93% ee from a reaction catalyzed by **QD-1d** (10.0 mol %) at room temperature for 88 hours.

(-)-**4Bf** was obtained in 92 % yield after flash chromatography (elution solvent: Ethyl Acetate/Hexane = 1/2) and 84% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 70:30, 1.0 ml/min, λ 220

nm, t(minor) = 18.7 min, t(major) = 12.6 min] from a reaction catalyzed by **Q-1d** (10 mol%) at 70 °C. for 28 hours. $[\alpha]_D^{25} = -6.8$ (c 0.5, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.05 (s, 1H), 7.59-7.56 (m, 2H), 7.34-7.26 (m, 4H), 6.85 (d, *J* = 3.2 Hz, 1H), 6.72 (d, *J* = 1.6 Hz, 1H), 6.67 (dd, *J* = 8.4 Hz, 2.4 Hz, 1H), 4.29-4.24 (m, 2H), 4.26 (s, 1H), 3.77 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 174.8, 156.4, 141.3, 137.5, 128.0, 127.9, 126.9, 122.9, 121.5, 119.8, 117.6, 109.8, 94.4, 77.5, 62.8, 55.5, 14.0; IR (CHCl₃) ν 3405 (br), 2981, 2834, 1718, 1634, 1558, 1576, 1258; HRMS (EI) *m/z* calcd. for (C₁₉H₁₉NO₄): 325.1314, found: 325.1306.

(+)- **4Bf** was obtained in 72% yield and 82% ee from a reaction catalyzed by **QD-1d** (10.0 mol %) at 70 °C for 29 hours.

(-)-**4Gf** was obtained in 96 % yield after flash chromatography (elution solvent: Ethyl Acetate/Hexane = 1/3) and 84% ee as determined by HPLC analysis [Chiralpak AD, Hexanes:IPA, 70:30, 1.0 ml/min, λ 220 nm, t(minor) = 15.4 min, t(major) = 9.5 min] from a reaction catalyzed by **Q-1d** (10 mol%) at 70

°C for 51 hours. $[\alpha]_D^{25} = -14$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.22 (br, 1H), 7.55-7.53 (m, 2H), 7.35-7.30 (m, 4H), 7.24 (d, *J* = 1.6 Hz, 1H), 6.98-6.96 (m, 2H), 4.31 (s, 1H), 4.30-4.26 (m, 2H), 1.21 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 174.5, 141.0, 137.0, 128.2, 128.1, 126.8, 124.7, 124.1, 121.8, 120.6, 117.9, 111.1, 77.4, 62.9, 14.0; IR (CHCl₃) ν 3419 (br.), 2981, 1718, 1449, 1255, 1095; HRMS (EI) *m/z* calcd. for (C₁₈H₁₆NO₃): 329.0819, found:): 329.0819.

(+)- **4Gf** was obtained in 87% yield and 81% ee from a reaction catalyzed by **QD-1d** (10.0 mol %) at 70 °C for 51 hours.

(-)-**4Ag** was obtained in 52 % yield after flash chromatography (elution solvent: Ethyl Acetate/Hexane = 1/3) and 83% ee as determined by HPLC analysis

[Chiralpak AD, Hexanes:IPA, 60:40, 1.0 ml/min, λ 220 nm, t(minor) = 14.5 min,

t(major) = 10.1 min] from a reaction catalyzed by **Q-1d** (10 mol%) at 70 °C for 56 hours. $[\alpha]_D^{25} = -8.4$ (c 0.7, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.11 (br, 1H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.17 (t, *J* = 8.0 Hz, 1H), 7.10 (d, *J* = 2.4 Hz, 1H), 7.04 (t, *J* = 8.0 Hz, 1H), 6.86 (t, *J* = 8.4 Hz, 2H), 4.30-4.28 (m, 2H), 4.21 (s, 1H), 3.81 (s, 3H), 1.25-1.21 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 175.7, 159.9, 137.4, 134.2, 128.9, 126.3, 124.6, 122.9, 121.8, 120.5, 118.8, 114.0, 111.8, 77.9, 63.4, 55.9, 14.7; IR (CHCl₃) ν 3408 (br.) 2981, 1722, 1609, 1510, 1459, 1249, 1033.

(-)-**6Aa** was obtained as a colorless oil in 95 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/2) and 93 % ee as determined by HPLC analysis [Daicel chiralcel OD, hexanes:IPA, 90:10, 1.0 ml/min, λ 220 nm, t (major) = 35.8 min, t (minor) = 45.2 min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 6 hours. $[\alpha]_D^{25} = -95.5$ (c 0.8, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.22 (br, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.34 (t, *J* = 8.0 Hz, 1H), 7.26-7.06 (m, 3H), 5.47 (d, *J* = 6.0 Hz, 1H), 4.43-4.24 (m, 1H), 4.22-4.10 (m, 1H), 3.33 (d, *J* = 6.0 Hz, 1H), 1.28-1.16 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 174.7, 137.1, 126.0, 123.9, 123.2, 120.8, 120.1, 114.5, 112.0, 68.0, 62.7, 14.8; HRMS (CI) *m/z* calcd. for (C₁₂H₁₃NO₃+Na⁺): 242.0793, found: 242.0789; IR (CHCl₃) ν 3406, 1733, 1458, 1201, 1078, 1048, 745 cm⁻¹.

(+)- **6Aa** was obtained in 85% yield and 93% ee from a reaction catalyzed by **QD-1d** (10.0 mol %) at r.t. for 6 hours.

(-)-**6Ea** was obtained as a colorless oil in 96 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/2) and 90 % ee as determined by HPLC analysis [Daicel chiralcel OD + OD, hexanes:IPA, 90:10, 1.0 ml/min, λ 220 nm, t (major) = 48.84 min, t (minor) = 45.83 min] from a reaction catalyzed by **Q-1d** (10 mol%) at room temperature for 4 hours. $[\alpha]_D^{25} = -76.7$ (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 8.29 (br, 1H), 7.56 (d, *J* = 8.8 Hz, 1H), 7.48 (d, *J* = 1.6 Hz, 1H), 7.32 (dd,

$J = 8.8$ Hz, 1.6 Hz, 1H), 7.18 (d, $J = 2.4$ Hz, 1H), 5.43 (d, $J = 4.8$ Hz, 1H), 4.34 - 4.243 (m, 1H), 4.22 - 4.12 (m, 1H), 3.39 (d, $J = 4.8$ Hz, 1H), 1.28 - 1.16 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 174.5 , 137.9 , 124.9 , 124.1 , 121.5 , 116.8 , 115.0 , 114.7 , 67.8 , 62.9 , 14.8 ; HRMS (CI) m/z calcd. for $(\text{C}_{12}\text{H}_{12}\text{BrNO}_3 + \text{Na}^+)$: 319.9898 , found: 319.9891 ; IR (CHCl_3) ν 3410 , 1734 , 1456 , 1222 , 1051 , 805 , 758 cm^{-1} .

(+)- **6Ea** was obtained in 95% yield and 90% ee from a reaction catalyzed by QD-**1d** (10 mol %) at room temperature for 4 hours.

(-)-**6Ba** was obtained as a colorless oil in 94 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/2) and 90 % ee as determined by HPLC analysis [Daicel chiralcel OD, hexanes:IPA, 80:20, 1.0 ml/min, λ 220 nm, t

(major) = 16.00 min, t (minor) = 14.59 min] from a reaction catalyzed by Q-**1d** (10 mol%) at room temperature for 4 hours. $[\alpha]_{\text{D}}^{25} = -85.1$ (c 0.8 , CHCl_3) ; ^1H NMR (400 MHz, CDCl_3) δ 8.13 (br, 1H), 7.57 (d, $J = 9.2$ Hz, 1H), 7.10 - 7.06 (m, 1H), 6.83 - 6.78 (m, 2H), 5.42 (d, $J = 6.4$ Hz, 1H), 4.34 - 4.24 (m, 1H), 4.22 - 4.12 (m, 1H), 3.82 (s, 3H), 3.33 (d, $J = 6.4$ Hz, 1H), 1.25 - 1.12 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 174.3 , 156.9 , 137.5 , 122.3 , 120.3 , 119.9 , 114.0 , 110.4 , 94.9 , 67.6 , 62.3 , 55.8 , 14.3 ; HRMS (CI) m/z calcd. for $(\text{C}_{13}\text{H}_{15}\text{NO}_4 + \text{H}^+)$: 250.1079 , found: 250.1071 ; IR (CHCl_3) ν 3403 , 2931 , 1732 , 1633 , 1456 , 1201 , 1079 , 808 , 760 cm^{-1} .

(+)- **6Ba** was obtained in 93% yield and 82% ee from a reaction catalyzed by QD-**1d** (10.0 mol %) at room temperature for 4 hours.

(-)-**6Ab** was obtained as a yellow solid in 96 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/4) and 90 % ee as determined by HPLC analysis [Daicel chiralcel OD, hexanes:IPA, 80:20, 1.0 ml/min, λ 220 nm, t (major) = 14.6 min, t (minor) = 18.6 min] from a reaction catalyzed by Q-**1d** (10 mol%)

at r.t. for 72 hours. m. p. 215 - 217 $^{\circ}\text{C}$; $[\alpha]_{\text{D}}^{25} = -100.0$ (c 0.9 , Acetone) ; ^1H NMR (400

MHz, Acetone- d^6) δ 10.13 (br, 1H), 8.09 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.72 (t, J = 8.0 Hz, 1H), 7.58-7.48 (m, 2 H), 7.35 (d, J = 8.0 Hz, 1H), 7.07 (dt, J = 1.2 Hz, 6.4 Hz, 1H), 6.98-6.92 (m, 2 H), 6.79-6.72 (m, 1H), 4.87 (d, J = 4.8, 1H); ^{13}C NMR (100 MHz, Acetone- d^6) δ 148.9, 139.8, 137.2, 132.8, 128.9, 128.1, 126.5, 124.1, 136.6, 121.8, 119.7, 119.2, 118.0, 111.6, 64.8; HRMS (CI) m/z calcd. for ($\text{C}_{15}\text{H}_{12}\text{N}_2\text{O}_3\text{-OH}$): 251.0821, found: 251.0812; IR (CHCl_3) ν 3409, 1608, 1520, 1341, 1058, 745 cm^{-1} .

(+)- **6Ab** was obtained in 95% yield and 89% ee from a reaction catalyzed by QD-**1d** (10.0 mol %) at room temperature for 72 hours.

(+)-**6Ac** was obtained as a yellow solid in 90 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/4) and 88 % ee as determined by HPLC analysis [Daicel chiralcel OD, hexanes:IPA, 90:10, 1.0 ml/min, λ 220 nm, t

(major) = 85.6 min, t (minor) = 101.4 min] from a reaction catalyzed by Q-**1d** (10 mol%) at r.t. for 72 hours. m. p. 162-163 $^{\circ}\text{C}$; $[\alpha]_{\text{D}}^{25}$ = 53.8 (c 0.8, Acetone) ; ^1H NMR (400 MHz, Acetone- d^6) δ 10.20 (br, 1H), 8.19 (d, J = 8.4 Hz, 2H), 7.79 (d, J = 8.4 Hz, 2H), 7.53 (d, J = 8.0 Hz, 1H), 7.37 (d, J = 8.0 Hz, 1H), 7.23-7.18 (m, 1H), 7.07 (t, J = 7.2 Hz, 1H), 6.94 (t, J = 7.2 Hz, 1H), 6.25 (d, J = 4.0 Hz, 1H), 4.91 (d, J = 4.0, 1H); ^{13}C NMR (100 MHz, Acetone- d^6) δ 153.5, 147.0, 137.4, 127.6 (2C), 126.1, 123.4, 123.3 (2C), 121.8, 119.8, 119.2, 119.0, 111.6, 69.1; HRMS (CI) m/z calcd. for ($\text{C}_{15}\text{H}_{12}\text{N}_2\text{O}_3\text{-OH}$): 251.0821, found: 251.0815; IR (CHCl_3) ν 3370, 2924, 1593, 1514, 1343, 1071, 1000 cm^{-1} .

(-)- **6Ac** was obtained in 90% yield and 83% ee from a reaction catalyzed by QD-**1d** (10 mol %) at room temperature for 72 hours.

(+)-**6Ad** was obtained as a white solid in 85 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/4) and 88 % ee as determined by HPLC analysis [Daicel chiralcel OD, hexanes:IPA, 90:10, 1.0 ml/min, λ 220 nm, t

(major) = 42.0 min, t (minor) = 45.7 min] from a reaction catalyzed by Q-**1d** (10 mol%)

at r.t. for 72 hours. m. p. 111-112 °C; $[\alpha]_D^{25} = +25.0$ (c 0.8, CHCl₃) ; ¹H NMR (400 MHz, CDCl₃) δ 8.11 (br, 1H), 7.66-7.54 (m, 5H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.22 (t, *J* = 8.0 Hz, 1H), 7.11 (t, *J* = 8.0 Hz, 1H), 6.96 (s, 1H), 6.21 (d, *J* = 3.6 Hz, 1H), 2.30 (d, *J* = 3.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 148.0, 137.3, 127.3 (2C), 126.2, 125.94, 125.91, 125.87, 125.84, 123.6, 123.4, 120.8, 120.1, 119.7, 112.1, 70.3; HRMS (CI) *m/z* calcd. for (C₁₆H₁₂F₃NO-OH): 274.0844, found: 274.0833; IR (CHCl₃) ν 3399, 3225, 1606, 1328, 1118, 747 cm⁻¹.

(-)- **6Ad** was obtained in 83% yield and 88% ee from a reaction catalyzed by QD-**1d** (10.0 mol %) at room temperature for 72 hours.

(+)- **6Ae** was obtained as a yellow solid in 75 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/4) and 83 % ee as determined by HPLC analysis [Daicel chiralcel OJ, hexanes:IPA, 80:20, 1.0 ml/min, λ 220 nm, *t* (major) = 35.87 min, *t* (minor) = 41.39 min] from a reaction catalyzed by Q-**1d** (10 mol%) at 70 °C for 40 hours. m. p. 88-90 °C; $[\alpha]_D^{25} = +25.6$ (c 0.8, CHCl₃) ; ¹H NMR (400 MHz, Acetone-d₆) δ

8.10 (br, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.22 (t, *J* = 8.0 Hz, 1H), 7.10 (t, *J* = 8.0 Hz, 1H), 6.95 (d, *J* = 2.0 Hz, 1H), 6.14 (d, *J* = 2.8 Hz, 1H), 2.26 (d, *J* = 2.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.6, 137.3, 133.7, 129.1 (2C), 128.5 (2C), 126.2, 123.5, 123.3, 120.7, 120.2, 120.0, 112.0, 70.3; HRMS (CI) *m/z* calcd. for (C₁₅H₁₂ClNO-OH): 240.0580, found: 240.0575; IR (CHCl₃) ν 3408, 1684, 1490, 1457, 1090, 1013, 744 cm⁻¹.

(+)- **6Af** was obtained as a colorless oil in 60 % yield after flash chromatography (elution solvent: ethyl acetate/hexanes = 1/4) and 82 % ee as determined by HPLC analysis [Daicel chiralcel OJ + OJ, hexanes:IPA, 70:30, 1.0 ml/min, λ 220 nm, *t* (major) = 76.81 min, *t*

(minor) = 71.31 min] from a reaction catalyzed by Q-**1d** (10 mol%) at 70 °C for 48 hours. $[\alpha]_D^{25} = +9.5$ (c 0.7, CHCl₃) ; ¹H NMR (400 MHz, CDCl₃) δ 8.07 (br, 1H), 7.62 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 7.6 Hz, 2H), 7.43-7.27 (m, 4H), 7.23 (t, *J* = 8.0 Hz, 1H),

7.10 (t, $J = 8.0$ Hz, 1H), 6.98-6.94 (m, 1H), 6.19 (d, $J = 3.6$ Hz, 1H), 2.23 (d, $J = 3.6$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.2, 137.3, 129.0 (2C), 128.1, 127.2 (2C), 126.5, 123.4, 123.1, 120.6, 120.5, 120.3, 111.9, 71.0; HRMS (CI) m/z calcd. for ($\text{C}_{15}\text{H}_{13}\text{NO-OH}$): 206.0970, found: 206.0960; IR (CHCl_3) ν 3415, 3058, 1601, 1456, 1217, 1093, 744 cm^{-1} .

X-Ray analysis of (+)-**4Dc** obtained with QD-**1d**:

Experimental Section:

A colorless block crystal with dimensions 0.10 x 0.08 x 0.02 mm was mounted on a glass fiber using very small amount of paratone oil.

Data were collected using a Bruker SMART CCD (charge coupled device) based diffractometer equipped with an Oxford Cryostream low-temperature apparatus operating at 193 K. Data were measured using omega scans of 0.3 ° per frame for 30 seconds, such that a hemisphere was collected. A total of 1271 frames were collected with a maximum resolution of 0.76 Å. The first 50 frames were recollected at the end of data collection to monitor for decay. Cell parameters were retrieved using SMART³ software and refined using SAINT on all observed reflections. Data reduction was performed using the SAINT software⁴ which corrects for Lp and decay. Absorption corrections were applied using SADABS⁸ multiscan technique, supplied by George Sheldrick. The structures are solved by the direct method using the SHELXS-97⁵ program and refined by least squares method on F^2 , SHELXL-97,⁶ incorporated in SHELXTL-PC V 6.10.⁷

The structure was solved in the space group $P4_32_12$ (# 96) by analysis of systematic absences. All non-hydrogen atoms are refined anisotropically. Hydrogens were found by difference Fourier methods and refined isotropically. The Flack⁹ parameter is used to determine chirality of the crystal studied, the value should be near zero, a value of one is the other enantiomer and a value of 0.5 is racemic. The Flack parameter was refined to -0.05(7), confirming the absolute stereochemistry. The crystal used for the diffraction study showed no decomposition during data collection. All drawing are done at 50% ellipsoids.

Acknowledgement. The CCD based x-ray diffractometer at Harvard University was purchased through NIH grant (1S10RR11937-01).

^a Obtained with graphite monochromated Mo K α ($\lambda = 0.71073 \text{ \AA}$) radiation.

^b $R1 = \sum ||F_o| - |F_c|| / \sum |F_o|$. ^c $wR2 = \{\sum [w(F_o^2 - F_c^2)^2] / \{\sum [w(F_o^2)^2]\}^{1/2}$.

The following are 50% thermal ellipsoidal drawings of the molecule in the asymmetric cell with various amount of labeling.

As shown above, the absolute configuration of (+)-**4Dc** which was obtained with QD-**1d** is *S*.

This is a drawing of the packing along the a-axis with dotted lines showing hydrogen bonding scheme.

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HPLC analysis for products **4** and **6**:

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 75:25, 1.0 mL/min, λ 220 nm

Racemic **4Aa**

(+)-**4Aa** 88% ee
Product obtained with QD-1d

(-)-**4Aa** 87% ee
Product obtained with Q-1d

HPLC Conditions: Daicel chiralcel OD, Hexane:IPA, 90:10, 1.0 mL/min, λ 220 nm

Racemic **4Ba**

(+)-**4Ba** 88% ee
Product obtained with QD-1d

(-)-**4Ba** 84% ee
Product obtained with Q-1d

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 70:30, 1.0 mL/min, λ 220 nm

Racemic 4Ab

(-)-4Ab 90% ee
Product obtained with QD-1d

HPLC Conditions: Daicel chiralcel OD, Hexane:IPA, 80:20, 1.0 mL/min, λ 220 nm

Racemic **4Ac**

(-)-**4Ac** 97% ee
 Product obtained with QD-1d

(+)-**4Ac** 97% ee
 Product obtained with Q-1d

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 75:25, 1.0 mL/min, λ 220 nm

Racemic 4Cc

(+)-4Cc 99% ee
Product obtained with QD-1d

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 80:20, 1.0 mL/min, λ 220 nm

Racemic 4Dc

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area [mAU]	Height [mAU]	Area %
1	13.020	1	VV	1.55053e4	350.00443	49.8950
2	15.287	1	VB	1.55705e4	300.93158	50.1050

Totals : 3.10758e4 650.93600

(+)-(S)-4Dc 94% ee
Product obtained with QD-1d

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area [mAU]	Height [mAU]	Area %
1	13.060	1	BV	709.91632	15.54268	2.9457
2	15.282	1	VB	2.33902e4	446.37289	97.0543

Totals : 2.41001e4 461.91558

(-)-(R)-4Dc 96% ee
Product obtained with Q-1d

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area [mAU]	Height [mAU]	Area %
1	13.001	1	VV	2.65380e4	597.75262	98.2324
2	15.285	1	VB	477.52225	8.84364	1.7676

Totals : 2.70155e4 606.59626

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 65:35, 1.0 mL/min, λ 254 nm

Racemic 4Bc

(-)-4Bc 96% ee
Product obtained with QD-1d

(+)-4Bc 97% ee
Product obtained with Q-1d

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 70:30, 0.9 mL/min, λ 254 nm

Racemic **4Ec**

(-)-**4Ec** 95% ee
Product obtained with QD-1d

(+)-**4Ec** 95% ee
Product obtained with Q-1d

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 75:25, 1.0 mL/min, λ 220 nm

Racemic **4Fc**

(-)-**4Fc** 97% ee
Product obtained with QD-1d

(+)-**4Fc** 97% ee
Product obtained with Q-1d

HPLC Conditions: Daicel chiralpak AS, Hexane:IPA, 80:20, 1.0 mL/min, λ 220 nm

Racemic 4Ad

(-)-4Ad 95% ee
Product obtained with QD-1d

(+)-4Ad 98% ee
Product obtained with Q-1d

HPLC Conditions: Daicel chiralcel OD + OD, Hexane:IPA, 85:15, 0.9 mL/min, λ 220 nm

Racemic **4Ae**

(-)-**4Ae** 95% ee
 Product obtained with QD-1d at r.t.

(+)-**4Ae** 95% ee
 Product obtained with Q-1d at r.t.

HPLC Conditions: Daicel chiralcel OD, Hexane:IPA, 90:10, 1.0 mL/min, λ 220 nm

Racemic 4Ae

=====
Area Percent Report
=====
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VMD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height [mAU]	Area %
1	19.318	BB	0.8327	3794.42339	67.88111	49.9594
2	22.161	BB	0.9481	3800.73560	60.88723	50.0402

Totals : 7595.35889 128.76834

=====
Area Percent Report
=====
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VMD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height [mAU]	Area %
1	19.241	BB	0.8407	7727.28467	137.78299	92.9104
2	22.161	BB	0.9097	589.46044	5.56800	7.0896

Totals : 8316.92511 147.35098

*** End of Report ***

=====
Area Percent Report
=====
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VMD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height [mAU]	Area %
1	19.543	BB	0.8353	377.81934	6.77764	5.4417
2	22.171	BB	0.9726	6565.25146	103.14462	94.5583

Totals : 6943.07080 109.92227

*** End of Report ***

(-)-4Ae 86% ee

Product obtained with QD-1d at 70 °C

(+)-4Ae 89% ee

Product obtained with Q-1d at 70 °C

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 80:20, 1.0 mL/min, λ 220 nm

Racemic **4Af**

(+)-**4Af** 93% ee
Product obtained with QD-1d at r.t

(-)-**4Af** 94% ee
Product obtained with Q-1d at r.t

(+)-4Af 86% ee

Product obtained with QD-1d at 70 °C

(-)-4Af 86% ee

Product obtained with Q-1d at 70 °C

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 70:30, 1.0 mL/min, λ 220 nm

Racemic **4Bf**

(+)-**4Bf** 82% ee
Product obtained with QD-**1d** at 70 °C

(-)-**4Bf** 84% ee
Product obtained with Q-**1d** at 70 °C

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 70:30, 1.0 mL/min, λ 220 nm

Racemic **4Gf**

(+)-**4Gf** 81% ee
Product obtained with QD-**1d** at 70 °C

(-)-**4Gf** 84% ee
Product obtained with Q-**1d** at 70 °C

HPLC Conditions: Daicel chiralpak AD, Hexane:IPA, 60:40, 1.0 mL/min, λ 220 nm

Racemic 4Ag

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	%s	Height [mAU]	Area %
1	10.029	1	VV	4.14143e4	1677.34155	49.8148	
2	14.167	1	VV	4.17223e4	987.61316	50.1852	
Totals :				8.31367e4	2664.95471		

(-)-4Ag 83% ee
Product obtained with Q-1d at 70 °C

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	%s	Height [mAU]	Area %
1	10.063	1	VB	1.22149e4	509.49905	91.5297	
2	14.557	1	BB	1130.37781	31.59361	8.4703	
Totals :				1.33452e4	541.09266		

HPLC Conditions: Daicel chiralpak OD, Hexane:IPA, 90:10 1.0 mL/min, λ 220 nm

Racemic 6Aa

(-)-6Aa 93% ee
Product obtained with Q-1d

(+)-6Aa 93% ee
Product obtained with QD-1d

HPLC Conditions: Daicel chiralpak OD +OD, Hexane:IPA, 90:10 1.0 mL/min, λ 220 nm

Racemic 6Ea

Area Percent Report

Sorted By	Retention Time
Multiplier	1.0000
Dilution	1.0000
Sample Amount	1.00000 [ng/ul] (not used in calc.)

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	*s	Height [mAU]	Area %
1	47.687	1	VV	8352.37109		68.24383	45.6839
2	51.369	1	VBA	9930.61133		67.45737	54.3161

Totals : 1.82830e4 135.70119

(-)-6Ea 90% ee
Product obtained with Q-1d

Area Percent Report

Sorted By	Retention Time
Multiplier	1.0000
Dilution	1.0000
Sample Amount	1.00000 [ng/ul] (not used in calc.)

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	*s	Height [mAU]	Area %
1	45.829	1	BV	2577.25024		28.03049	4.9821
2	48.844	1	VV	4.91529e4		496.33813	95.0179

Totals : 5.17301e4 525.16862

(+)-6Ea 90% ee
Product obtained with QD-1d

Area Percent Report

Sorted By	Retention Time
Multiplier	1.0000
Dilution	1.0000
Sample Amount	1.00000 [ng/ul] (not used in calc.)

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	*s	Height [mAU]	Area %
1	45.160	1	BV	4.59719e4		489.77731	94.8908
2	49.367	1	VV	2475.24902		24.94793	5.1092

Totals : 4.84472e4 514.72524

HPLC Conditions: Daicel chiralpak OD, Hexane:IPA, 80:20 1.0 mL/min, λ 220 nm

Racemic 6Ba

(+)-6Ba 82% ee
Product obtained with QD-1d

(-)-6Ba 90% ee
Product obtained with Q-1d

HPLC Conditions: Daicel chiralpak OD, Hexane:IPA, 80:20 1.0 mL/min, λ 220 nm

Racemic 6Ab

(-)-6Ab 90% ee
Product obtained with Q-1d

(+)-6Ab 89% ee
Product obtained with QD-1d

HPLC Conditions: Daicel chiralpak OD, Hexane:IPA, 90:10 1.0 mL/min, λ 220 nm

Racemic 6Ac

(+)-6Ac 88% ee
Product obtained with Q-1d

(-)-6Ac 83% ee
Product obtained with QD-1d

HPLC Conditions: Daicel chiralpak OD, Hexane:IPA, 90:10 1.0 mL/min, λ 220 nm

Racemic 6Ad

(+)-6Ad 88% ee
Product obtained with Q-1d

(-)-6Ad 88% ee
Product obtained with QD-1d

HPLC Conditions: Daicel chiralpak OJ, Hexane:IPA, 80:20 1.0 mL/min, λ 220 nm

Racemic **6Ae**

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=215 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	*s	Height [mAU]	Area %
1	36.023	1	VV	1.60313e4		160.71773	49.9059
2	40.987	1	VB	1.60917e4		134.50078	50.0941

Totals : 3.21230e4 295.21851

(+)-**6Ae** 83% ee
Product obtained with **Q-1d**

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	35.874	BB	1.5031	1.59155e4		153.89485	91.5671
2	41.387	BB	1.5308	1465.74561		13.17564	8.4329

Totals : 1.73813e4 167.07049

HPLC Conditions: Daicel chiralpak OJ + OJ, Hexane:IPA, 70:30 1.0 mL/min, λ 220 nm

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	*s	Height [mAU]	Area %
1	69.894	1	BV	1.38800e4		82.08083	49.2903
2	75.615	1	VB	1.42797e4		75.11284	50.7097

Totals : 2.81597e4 157.19367

(+)-6Af 82% ee
Product obtained with Q-1d

Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=215 nm

Peak #	RetTime [min]	Sig	Type	Area mAU	*s	Height [mAU]	Area %
1	71.308	1	BV	1090.67786		6.55105	8.9447
2	76.810	1	VB	1.11029e4		57.88432	91.0553

Totals : 1.21936e4 64.43537

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: ji-14-45-c

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

288 repetitions

OBSERVE C13, 100.5180368 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pu]

Solvent: CDCl₃

Ambient temperature

File: j1-14-41-h

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

64 repetitions

OBSERVE H1, 399.7532410 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 45 sec

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: 1j-14-41-c
INOVA-500 "cosy"

Pulse 53.1 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
352 repetitions
OBSERVE C13, 100.5180384 MHz
DECOUPLE H1, 399.7552472 MHz
Power 37 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: li-13-104-h

INOVA-500 "cosy"

Pulse 45.4 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532392 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-13-105-c

INOVA-500 "cosy"

Pulse 51.6 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

448 repetitions

OBSERVE C13, 100.5180391 MHz

DECOUPLE H1, 399.7552472 MHz

Power 39 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-14-21-h

INOVA-500 "cosy"

Pulse 45.4 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532380 MHz

DATA PROCESSING

Ft size 16384

Total time 0 min, 26 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-14-21-c

INOVA-500 "cosy"

Pulse 51.6 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

608 repetitions

OBSERVE C13, 100.5180338 MHz

DECOUPLE H1, 399.7552472 MHz

Power 39 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-14-18-h

INOVA-500 "cosy"

Pulse 45.4 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532380 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

4Dc

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: jf-14-18-c
INOVA-500 "cosy"

Pulse 51.6 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
1152 repetitions
OBSERVE C13, 100.5180330 MHz
DECOUPLE H1, 399.7552472 MHz
Power 39 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 21 min, 22 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 1i-14-12-c

INOVA-500 "cosy"

Pulse 51.6 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

288 repetitions

OBSERVE C13, 100.5180391 MHz

DECOUPLE H1, 399.7552472 MHz

Power 39 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pu]

Solvent: CDCl₃

Ambient temperature

File: J1-14-15-h-2

INOVA-500 "cosy"

Pulse 45.4 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532380 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-14-15-c

INOVA-500 "cosy"

Pulse 51.6 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

576 repetitions

OBSERVE C13, 100.5180361 MHz

DECOUPLE H1, 399.7552472 MHz

Power 39 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 11-14-24-h

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532410 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

4Fc

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-14-34-h

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532410 MHz

DATA PROCESSING

Ft size 16384

Total time 0 min, 26 sec

4Ad

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-14-34-c

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

512 repetitions

OBSERVE C13, 100.5180384 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: wyq-1-99-2H

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532447 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

4Ae

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: wq-1-99-2C

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

588 repetitions

OBSERVE C13, 100.5180368 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 33 hr, 33 min, 39 sec

wyq-1-98-H

Pulse Sequence: s2pul

4Af

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: wyq-1-98-C

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

1784 repetitions

OBSERVE C13, 100.5180376 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 33 hr, 33 min, 39 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: j1-15-20-h

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532471 MHz

DATA PROCESSING

Ft size 16384

Total time 0 min, 26 sec

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: j1-15-20-c
INOVA-500 "cosy"

Pulse 53.1 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
1024 repetitions
OBSERVE C13, 100.5180384 MHz
DECOUPLE H1, 399.7552472 MHz
Power 37 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: ji-15-23-h

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532447 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

4Gf

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: ji-15-23-c

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

1024 repetitions

OBSERVE C13, 100.5180368 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: WYQ-1-139-2-H
INOVA-500 "gamble"

Pulse 46.1 degrees
Acq. time 1.638 sec
Width 5000.0 Hz
32 repetitions
OBSERVE H1, 399.7532349 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 52 sec

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: wyq-1-139-2-C
INOVA-500 "gamble"

Pulse 53.1 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
1416 repetitions
OBSERVE C13, 100.5180359 MHz
DECOUPLE H1, 399.7552490 MHz
Power 37 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 21 min, 40 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: WYQ-1-117-H

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532416 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: WYQ-1-117-C

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

1952 repetitions

OBSERVE C13, 100.5180353 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 3 hr, 21 min, 20 sec

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: wq-1-133-C
INOVA-500 "cosy"

Pulse 53.1 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
1280 repetitions
OBSERVE C13, 100.5180345 MHz
DECOUPLE H1, 399.7552472 MHz
Power 37 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 3 hr, 21 min, 20 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: WYQ-1-131-C

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

2857 repetitions

OBSERVE C13, 100.5180353 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 3356 hr, 7 min, 38 sec

Pulse Sequence: s2pu]
Solvent: Acetone
Ambient temperature
File: wyq-1-2-NO2-benzaldehyde-product-H
INOVA-500 "cosy"

Pulse 46.1 degrees
Acq. time 1.638 sec
Wdth 5000.0 Hz
32 repetitions
OBSERVE H1, 399.7553301 MHz
DATA PROCESSING
Ft size 16384
Total time 0 min, 52 sec

wyq-1-4-NO2-benzaldehyde-product-H

Pulse Sequence: s2pul

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: WYQ-1-70-CF3

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532441 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: wyq-1-69-CF₃-C

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

873 repetitions

OBSERVE C13, 100.5180353 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 20 min, 37 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: WYQ-1-137-2-Et3N

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

32 repetitions

OBSERVE H1, 399.7532453 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 52 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: wq-1-138-1-C

INOVA-500 "cosy"

Pulse 53.1 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

2996 repetitions

OBSERVE C13, 100.5180368 MHz

DECOUPLE H1, 399.7552472 MHz

Power 37 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 335 hr, 36 min, 44 sec

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: wyq-i-136-product-H

INOVA-500 "cosy"

Pulse 46.1 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

32 repetitions

OBSERVE H1, 399.7532447 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 52 sec

6Af

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: wyq-1-136-product-C
NOVA-500 "cosy"

Pulse 53.1 degrees
Acq. time 1.159 sec
Width 25000.0 Hz
3036 repetitions
RESERVE C13, 100.5180353 MHz
DECOUPLE H1, 399.7552472 MHz
Power 37 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 size 65536
Total time 3 hr, 21 min, 20 sec