Supporting Information

Room-Temperature Copper-Catalyzed α -Arylation of Malonates

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1. General considerations.

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All α -arylation reactions were performed in Rotaflo[®] (England) resealable screw cap Schlenk flask (approx. 20 mL volume) or screw-capped vials (approx. 10 mL) with Teflon septum-linner, in the presence of Teflon coated magnetic stirrer bar (3 mm $\times$ 8 mm). Toluene and tetrahydrofuran (THF) were distilled from sodium and sodium benzophenone ketyl under nitrogen, respectively.¹ Anhydrous 1,4-dioxane was purchased from Aldrich. 2-Picolinic acid **L10** was purchased from Aldrich without further purification. Commercial aryl iodides (liquid form only) were purified by passed through a short plug (0.5 cm width $\times$ 4 cm height) of neutral alumina or distillation. Malonates were distilled under nitrogen atmosphere and stored in a screw-capped vial. Cs_2CO_3 and K_3PO_4 were purchased from Aldrich and Fluka, respectively. Thin layer chromatography was performed on Merck precoated silica gel 60 F₂₅₄ plates. Silica gel (Merck, 230-400 mesh) was used for column chromatography. Room temperature stands for laboratory temperature (25 °C $\pm$ 1 °C). Melting points were recorded on an uncorrected Büchi Melting Point B-545 instrument. ¹H NMR spectra were recorded on a Bruker (400 MHz) or Varian (500 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in CDCl_3 (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ¹³C NMR spectra were referenced to CDCl_3 (δ 77.0 ppm, the middle peak). Coupling constants (J) were reported in Hertz (Hz). CDCl_3 was pre-dried by anhydrous K_2CO_3 powder. Mass spectra (EI-MS and ES-MS) were recorded on a HP 5989B Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Brüker APEX 47e FT-ICR mass spectrometer (ESIMS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m $\times$ 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP 6890 GC-FID system.

2. Preparation of phenolic ligand L5

2-(1,3-Dioxolan-2-yl)phenol.² A round bottom flask containing 118 mg of *p*-toluenesulfonic acid monohydrate was evacuated and backfilled with nitrogen (3 cycles). Toluene (47 mL) was added followed by salicylaldehyde (2.0 mL, 18.8 mmol) and ethylene glycol (4.2 mL, 75.2 mmol). The mixture was heated to reflux for 26 hours with continuous azeotropic removal of water and excess ethylene glycol by means of a Dean-Stark trap. The reaction mixture was allowed to reach room-temperature and washed with saturated aqueous sodium bicarbonate solution, brine and dried over anhydrous sodium sulphate. The crude mixture was concentrated by rotary evaporation. The crude product thus obtained was purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as the eluent to obtain the title compound as a pale yellow oil. Yield 31%; R_f = 0.15 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.75 (s, 1H), 7.28-7.22 (m, 1H), 6.91-6.88 (m, 2H), 5.93 (s, 1H), 4.16-4.14 (m, 2H), 4.09-4.07 (m, 2H).

3. General procedures Cu-catalyzed α -arylation of malonates

An oven-dried vial equipped with a Teflon-coated magnetic stirrer bar was charged sequentially with CuI (9.5 mg, 5.0 mol%), 2-picolinic acid **L10** (12.3 mg, 10.0 mol%), Cs₂CO₃ (0.98 g, 3.0 mmol), and aryl iodide (1.0 mmol), if a solid. The vial was evacuated and backfilled with nitrogen (3 cycles). Anhydrous 1,4-dioxane (1.0 mL) was added volumetrically followed by distilled diethyl malonate (304 μ L, 2.00 mmol) and aryl iodide, if a liquid (1.00 mmol). The vial was sealed and placed in a preheated oil bath at 70 °C or on the stir-plate at room temperature. After the designated time period, the completed reaction (judged by GC-MS and TLC analysis) was allowed to reach room temperature. The reaction mixtures were partitioned between ethyl acetate (20 mL $\times$ 3) and saturated aqueous NH₄Cl (10 mL). The organic portions were dried over Na₂SO₄, filtered and concentrated by rotary evaporation. The material thus obtained was purified by flash chromatography on silica gel to give the desired α -aryl malonate product. The pure product was then subjected to NMR and MS analyses.

4. Characterization data for coupling products

Diethyl 2-phenylmalonate (Table 1, entry 1).³ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a yellow oil. Yield 92%; $R_f = 0.4$ (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz) δ 7.42-7.33 (m, 5H), 4.62 (s, 1H), 4.25-4.18 (m, 4H), 1.26 (t, $J = 7.5$ Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 168.1, 132.8, 129.2, 128.5, 128.1, 61.7, 57.9, 14.0; MS(EI) 236.1 (M^+).

Diethyl 2-(2-methoxyphenyl)malonate (Table 1, entry 4).⁴ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a yellow oil. Yield 92%; $R_f = 0.3$ (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.34-7.28 (m, 2H), 6.97 (dt, $J = 1.0, 7.5$ Hz, 1H), 6.89 (d, $J = 8$ Hz, 1H), 5.11 (s, 1H), 4.26-4.20 (m, 4H), 3.82 (s, 3H), 1.27 (t, $J = 7.0$ Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.6, 156.9, 129.4, 129.2, 121.9, 120.6, 110.6, 61.5, 55.6, 51.2, 14.0; MS(EI) 266.1 (M^+).

Diethyl 2-(3-methoxyphenyl)malonate (Table 1, entry 5).⁵ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a pale yellow oil. Yield 79%; R_f = 0.25 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.28-7.25 (m, 1H), 6.98-6.96 (m, 2H), 6.87 (dq, J = 2.5, 8.5 Hz, 1H), 4.58 (s, 1H), 4.25-4.18 (m, 4H), 3.81 (s, 3H), 1.26 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.0, 159.6, 134.1, 129.5, 121.6, 114.8, 113.8, 61.8, 57.9, 55.2, 14.0; MS(EI) 266.1 (M^+).

Diethyl 2-(4-methoxyphenyl)malonate (Table 1, entry 6).⁶ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a colourless oil. Yield 80%; R_f = 0.25 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.32 (d, J = 8.0 Hz, 2H), 6.89 (d, J = 8.5 Hz, 2H), 4.56 (s, 1H), 4.24-4.17 (m, 4H), 3.80 (s, 3H), 1.26 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.4, 159.4, 130.3, 124.9, 114.0, 61.7, 57.1, 55.2, 14.0; MS(EI) 266.1 (M^+).

Diethyl 2-(2,4-dimethoxyphenyl)malonate (Table 1, entry 7).⁷ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a white solid. Yield 88%; R_f = 0.2 (ethyl acetate/hexane = 1:9); melting point: 58.5-59.5 °C; ^1H NMR (CDCl_3 , 500 MHz): δ 7.24 (s, 1H), 6.50 (dd, J = 2.5, 8.5 Hz, 1H), 4.46 (d, J = 2.0 Hz, 1H), 5.02 (s, 1H), 4.24-4.19 (m, 4H), 3.80 (d, J = 2.5 Hz, 6H), 1.26 (t, J = 7.0 Hz,

6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.8, 160.7, 157.9, 130.0, 114.3, 104.5, 98.6, 61.5, 55.6, 55.3, 50.6, 14.0; MS(EI) 296.1 (M^+).

Diethyl 2-(3,4,5-trimethoxyphenyl)malonate (Table 1, entry 8).⁸ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:5) as eluent to obtain the title compound as a yellow oil. Yield 81%; R_f = 0.2 (ethyl acetate/hexane = 1:5); ^1H NMR (CDCl_3 , 500 MHz): δ 6.62 (s, 2H), 4.51 (s, 1H), 4.26-4.18 (m, 4H), 3.85 (s, 6H), 3.83 (s, 3H), 1.27 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.0, 153.1, 137.9, 128.0, 106.4, 61.8, 60.7, 57.8, 56.1, 14.0; MS(EI) 326.1 (M^+).

Diethyl 2-(*o*-tolyl)malonate (Table 1, entry 9).⁹ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a pale yellow oil. Yield 64%; R_f = 0.4 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.40 (t, J = 3.0 Hz, 1H), 7.24-7.18 (m, 3H), 4.87 (s, 1H), 4.28-4.20 (m, 4H), 2.34 (s, 3H), 1.27 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.4, 136.5, 131.6, 130.5, 128.8, 128.1, 126.3, 61.7, 54.4, 19.7, 14.0; MS(EI) 250.1 (M^+).

Diethyl 2-(3,5-dimethylphenyl)malonate (Table 1, entry 10).⁷ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a yellow oil. Yield 88%; R_f = 0.4 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.00 (s, 2H), 6.97(s, 1H), 4.54 (s, 1H), 4.27-4.16 (m, 4H), 2.31 (s, 6H), 1.27 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.3, 138.1, 132.5, 129.9, 126.9, 61.7, 57.8, 21.3, 14.0; MS(EI) 264.1 (M^+).

Diethyl 2-(4-cyanophenyl)malonate (Table 1, entry 11).¹⁰ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:3) as eluent to obtain the title compound as a yellow oil. Yield 82%; R_f = 0.5 (ethyl acetate/hexane = 1:3); ^1H NMR (CDCl_3 , 500 MHz): δ 7.66 (d, J = 8.0 Hz, 2H), 7.54 (d, J = 8.0 Hz, 2H), 4.65 (s, 1H), 4.26-4.19 (m, 4H), 1.27 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.1, 137.7, 132.3, 130.2, 118.4, 112.2, 62.3, 57.7, 13.9; MS(EI) 261.1 (M^+).

Diethyl 2-(4-fluorophenyl)malonate (Table 1, entry 12).⁸ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a yellow

oil. Yield 73%; R_f = 0.5 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.40-7.37 (m, 2H), 7.06-7.02 (m, 2H), 4.59 (s, 1H), 4.25-4.17 (m, 4H), 1.26 (t, J = 7.5 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.0, 163.6, 161.6, 131.0, 130.9, 128.5, 115.6, 115.4, 61.9, 57.0, 13.9; MS(EI) 254.1 (M^+).

3-(1,3-Diethoxy-1,3-dioxopropan-2-yl)benzoate (Table 1, entry 13).⁷ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as a colourless oil. Yield 96%; R_f = 0.2 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 8.03 (tt, J = 1.5, 9.0 Hz, 2H), 7.64 (dd, J = 1.5, 8.0 Hz, 1H), 7.45 (t, J = 7.5 Hz, 1H), 4.67 (s, 1H), 4.37 (q, J = 7.5 Hz, 2H), 4.27-4.17 (m, 4H), 1.39 (t, J = 7.0 Hz, 3H), 1.26 (t, J = 7.5 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.8, 166.1, 133.6, 133.1, 130.8, 130.5, 129.4, 128.6, 62.0, 61.1, 57.7, 14.3, 14.0; MS(EI) 308.1 (M^+).

Diethyl 2-(naphthalen-1-yl)malonate (Table 1, entry 14).¹¹ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as an orange solid. Yield 95%; R_f = 0.4 (ethyl acetate/hexane = 1:9); melting point: 61.0-62.2 °C; ^1H NMR (CDCl_3 , 500 MHz): δ 7.98 (d, J = 9.0 Hz, 1H), 7.87 (q, J = 8.5 Hz, 2H), 7.59-7.48 (m, 4H), 5.44 (s, 1H), 4.29-4.23 (m, 4H), 1.27 (t, J = 7.5 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.4, 133.9, 131.6, 129.3, 129.0, 128.8, 127.0, 126.6, 125.8, 125.4, 122.8, 61.9, 54.4, 14.0; MS(EI) 286.1 (M^+).

Diethyl 2-(2-pyridyl)malonate (Table 1, entry 15).⁵ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:3) as eluent to obtain the title compound as an orange oil. Yield 90%; R_f = 0.3 (ethyl acetate/hexane = 1:3); ^1H NMR (CDCl_3 , 500 MHz): δ 8.55 (dt, J = 1.0, 5.0 Hz, 1H), 7.70 (dt, J = 2.0, 8.0 Hz, 1H), 7.48 (dd, J = 1.0, 9.5 Hz, 1H), 7.23 (dq, J = 1.0, 2.5 Hz, 1H), 4.92 (s, 1H), 4.28-4.20 (m, 4H), 1.25 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.4, 153.1, 149.3, 136.7, 123.7, 122.9, 61.9, 60.6, 13.9; MS(EI) 237.1 (M^+).

Diethyl 2-(3-pyridyl)malonate (Table 1, entry 16).⁷ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:3) as eluent to obtain the title compound as an orange oil. Yield 77%; R_f = 0.15 (ethyl acetate/hexane = 1:3); ^1H NMR (CDCl_3 , 500 MHz): δ 8.57 (d, J = 5.0 Hz, 2H), 7.84 (dt, J = 2.0, 8.0 Hz, 1H), 7.31 (q, J = 5.0 Hz, 1H), 4.62 (s, 1H), 4.27-4.17 (m, 4H), 1.26 (t, J = 7.0 Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.4, 150.3, 149.5, 136.9, 128.9, 123.5, 62.2, 55.4, 13.9; MS(EI) 237.1 (M^+).

Diethyl 2-(2-thiophenyl)malonate (Table 1, entry 17).¹² Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as an orange oil. Yield 68%; R_f = 0.3 (ethyl acetate/hexane = 1:9); ^1H NMR (CDCl_3 , 500 MHz): δ 7.31 (d, J = 5.0 Hz, 1H), 7.10 (d, J = 3.5 Hz, 1H), 7.0 (t, J = 4.5 Hz, 1H), 4.91 (s, 1H), 4.27-4.21 (m, 4H),

1.28 (t, $J = 7.5$ Hz, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.3, 133.4, 127.8, 126.5, 126.4, 62.1, 53.1, 13.9; MS(EI) 242.0 (M^+).

Dimethyl 2-phenylmalonate (Scheme 1).¹³ Purified by column chromatography on silica gel using ethyl acetate/hexane (1:9) as eluent to obtain the title compound as an off white solid. Yield 82%; $R_f = 0.3$ (ethyl acetate/hexane = 1:9); melting point: 49.6-50.8 °C; ^1H NMR (CDCl_3 , 500 MHz): δ 7.41-7.34 (m, 5H), 4.66 (s, 1H), 3.76 (s, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.5, 132.5, 129.2, 128.6, 128.3, 57.5, 52.8; MS(EI) 208.0 (M^+).

Scheme 2 compounds matched the spectroscopic data in Table 1 entries 1 and 15.

5. ^1H , ^{13}C NMR, and MS spectra

2-(1,3-dioxolan-2-yl)phenol

diethyl 2-phenylmalonate

STANDARD PROTON PARAMETERS

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 np 89400 drcs 1.0
 fb 4000 homo DEC2 n
 bs 32
 tpwr 57 dfrq2 0
 pw 5.5 dn2
 tof 0 dfr2 1
 nt 0 dfr2 0
 ct 8 dm2 n
 alock not used n
 gain FLAGS dm2 200
 il n homo2 1.0
 in n vxfie
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 pp nm proc not used
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 wp 112 wexp
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 ns 518.57
 rfp 3628.1
 th 3
 ins cdc ph 1.000

diethyl 2-phenylmalonate

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Misc Info :
Vial Number: 6

diethyl 2-(3-methoxyphenyl)malonate

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Sample Name: FAN061(6)
Misc Info :
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STANDARD CARBON PARAMETERS

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sw	5000.0	dres	1.0			
fb	28000	homo	n			
bs	8	DEC2				
tpwr	63	dfrq2	0			
dl	6.0	dn2	1			
tof	0	dpwr2	0			
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ct	2248	dmm2	c			
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FLAGS		dres2	1.0			
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hs	nn	proc	ft			
sp	-1888.3	fn	not used			
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vs	1204	werr				
sc	0	wexp				
wc	180	wbs				
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ins						
ai	cdc	ph				

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Sample Name: FAN072(2)
Misc Info :
Vial Number: 6

diethyl 2-o-tolylmalonate

STANDARD PROTON PARAMETERS

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wv 89480 dscs
fb 4800 homo DEC2 1.0
bs 32
tpwr 57 dfrq2 0
pw 5.5 dn2 1
tof 0 ddr2 1
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ct 8 dm2 200
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gain FLAGS dfrq2
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ns n proc not used
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wp 4451.9 werr f
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wc 180 wbs
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diethyl 2-o-tolylmalonate

STANDARD CARBON PARAMETERS

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np 130048 dseq
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ct 464 dm2 C
atlock not used n
gain not used n
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ins cdc ph
at 100.000

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diethyl 2-o-tolylmalonate

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Misc Info :
Vial Number: 1

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		dp		wfile			
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		nm	cdc	ph			

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Sample Name : FAN071(2)
Misc Info :
Vial Number : 4

diethyl 2-(4-cyanophenyl)malonate

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Operator :
Acquired : 27 Mar 2007 14:52 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name:
Misc Info :
Vial Number: 3

diethyl 2-(4-fluorophenyl)malonate

STANDARD PROTON PARAMETERS

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 tn H1 dmm
 at 1.892 dmf 200 C
 np 30272 dseq
 pw 8000.0 pres 1.0
 bs 499.742 homo DEC2
 bs 32
 tpwr 57 dfrq2 0
 pw 5.5 dn2
 d1 0 dpwr2 1
 nt 0 dot2 0
 nt 8 dm2 n
 clock 200 C
 gain not used dseq2 200
 gain not used dres2 1.0
 gain not used dres2 1.0
 f1 homo2 n
 in n
 dp n
 hs y
 proc y
 ft not used
 sp -292.5 math
 wp 4115.8
 vs 103 werr
 wc 180 wbs
 sc 0 wexp
 hzmm 24.54 wnt
 fs 53.57
 rfp 3620.1
 th 6.000
 ins cdc ph

diethyl 2-(4-fluorophenyl)malonate

STANDARD CARBON PARAMETERS

exp2	szpul	date	SAMPLE	DEC.	& VT
4	2007	499.742			
file	CDCl3	dn			
solvent	exp	dpwr			
ACQUISITION	exp	dpwr			
exp	37				
sfreq	125.671	dpwr			
tn	125.671	dmf			
at	1.300	dmf			
np	130048	dseq			
sw	50000.0	dres			
bs	2800.0	homo			
tpwr	63	dfrq2			
pw	6.0	dn2			
di	0	dpwr2			
nt	5000	dm2			
ct	976	dm2			
alock	not used	dm2			
gain	not used	dseq2			
flags	not used	dm2			
il	n	homo2			
in	n	homo2			
dp	n	homo2			
hs	n	homo2			
sp	-2437.6	fn			
wp	25761.0	math			
vs	487	worc			
vc	180	wexp			
h2mm	143.12	wbs			
is	500.00	wnt			
rf1	23272.6				
rfp	3675.5				
ins	100.000				
ai	cdc	ph

diethyl 2-(4-fluorophenyl)malonate

File : C:\msdchem\1\DATA\Fan\FAN064(2).D
Operator : FAN
Acquired : 13 Feb 2007 12:26 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name : FAN064(2)
Misc Info :
Vial Number : 2

STANDARD CARBON PARAMETERS

```

exp2 s2pu1
SAMPLE
date Apr 17 2007 dfrq DEC. & VT
solvent CDCl3 dn 499.742
file 37
ACQUISITION exp
pwr 37
sol 0
sfr 125.671 dm
tn 11696
at 1.300 dmf
np 130048 dseq
fb 360000 f1
fb 28000 homo DEC2
bs 8
tpwr 63 dfrq2 0
pw 6.0 dn2 1
pc 0 dn2 1
tof 0 dfr2 0
nt 5000 dm2 n
ct 2096 dm2 c
alock 10000
gain not used n
flags dfr2 1.0
il n homo2 n
in n lb PROCESSING n
ip n lf file 0.50
ns n
Display nm wfile
sp -2117.9 fn not used f
wp 27693.2 math
sc 75.0 werr
wc 180 wexp
hzmm 150.52 wbs
f1 22700.0 wnt
rfl 5625.8
th 100.000
at cdc ph
  
```

ethyl 3-(1,3-diethoxy-1,3-dioxopropan-2-yl)benzoate

File : C:\msdchem\1\DATA\Fan\FAN089(columned) .D
Operator :
Acquired : 13 Apr 2007 17:43 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name :
Misc Info :
Vial Number: 3

Average of 10.045 to 10.617 min.: FAN089(columned) D\data.ms

diethyl 2-(naphthalen-1-yl)malonate

STANDARD PROTON PARAMETERS									
exp2 szpul									
SAMPLE	DEC. & VT								
date	Apr 26 2007	dfrq	499.742						
solvent	DMSO-d6	dn	499.742						
acqfile	exp	dpwr	30						
acqfile	exp	dof	30						
sfreq	499.742	dm	nnn						
at	1.892	dnmf	205						
at	30272	dres	1.0						
np	8000	homo	DEC2						
sw	400	dpwr2	0						
th	57	dn2	1						
tpwr	5.5	dpwr2	0						
pd	0	dnf2	0						
dt	8	dnm2	200						
ct	8	dnf2	200						
alock	not used	dres2	1.0						
again	not used	ures2	1.0						
FLAGS	nn	n	PROCESSING						
1	1	n	n						
1	1	n	n						
hs	DISPLAY	nn	proc						
ds	499.2	math	not used						
sp	4974.8	math	not used						
sc	110	werr							
vc	180	wexp							
nmz	33.58	wint							
15	37.08	wnt							
rfl	5129.7	rfl							
rfl	3226.1	rfl							
ins	1.000	ins							
ins	1.000	ins

diethyl 2-(naphthalen-1-yl)malonate

STANDARD CARBON PARAMETERS

```

exp2 szpu1
date SAMPLE Apr 26 2007 dfrq DEC. & VT 459.742
solvent CDC13 dn
file ACQUISITION exp dpwr 37
sfrq 125.671 dfr 0
tn C13 dnm yvv
at 1.300 def 11696
np 130048 dscq
fb 500000 drcs 1.0
bs 28000 homo DEC2 n
tpwr 63 dfrq2 0
pw 6.0 dn2 1
tof 0 dfr2 0
nt 5000 dm2 n
ct 1752 dnm2 C
alock not used n dm2 18000
gain FLAGS not used drc2 1.0
il n homo2 n
in n lb PROCESSING 0.50
cp n vfile
ns DISPLAY mn proc ft
sp -2627.6 fn not used f
wp 27691.3 math
sc 30.8 werr
wc 180 wexp
hzmm 153.34 wbs
fz 2970.00 wnt
rfp 26675.8
th 26675.8
ins cdc ph
at 100.000
  
```

File : C:\msdchem\1\DATA\Fan\FAN073 (2) .D
Operator : FAN
Acquired : 26 Feb 2007 18:59 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name: FAN073 (2)
Misc Info :
Vial Number: 8

STANDARD CARBON PARAMETERS

exp2	s2pu1	SAMPLE	DEC. & VI
date	Feb 23 2007	dfrq	499.742
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION	dof	ddy	0
sfrq	125.871	dm	yyy
tn	513	dmm	w
at	1.300	dmr	11696
np	130048	dseq	
sw	50000.0	dres	1.0
fb	28000	homo	n
bs	8	DEC2	
tpwr	63	dfrq2	0
pw	6.0	dn2	i
dl	0	dpwr2	0
tof	0	dof2	n
nt	5000	dm2	c
ct	456	dmm2	10000
alock	not used	dseq2	
gain	not used	dres2	1.0
il	n	homo2	n
in	n	lb	PROCESSING
dp	y	wtfile	0.50
hs	nn	proc	ft
sp	-1952.3	fn	not used
wp	25030.1	math	f
vs	774	werr	
sc	0	wexp	
wc	180	wbs	
hzmm	139.06	wnt	
is	500.00		
rfl	22772.6		
rfl	9675.8		
th	10		
ins	100.000		
ai	cdc	ph

diethyl 2-(pyridin-2-yl)malonate

File : C:\msdchem\1\DATA\Fan\FAN065(2) .D
Operator : FAN
Acquired : 13 Feb 2007 13:10 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name: FAN065(2)
Misc Info :
Vial Number: 4

Scan 834 (7.853 min): FAN065(2).D\data.ms

diethyl 2-(pyridin-3-yl)malonate

STANDARD PROTON PARAMETERS

exp2 szpu1

date SAMPLE
Apr 2 2007 dfrq DEC. & VT 499.742

solvent CDCl3 dn 30

file ACQUISITION exp dpwr 30

trf 499.742 ddr 0

sfr 499.742 dnm 200

at 1.892 dmf 200

np 38272 dseq 1.0

pw 80950 dres 1.0

fw 34900 nmo DEC2

bs 32

tpwr 57 dfrq2 0

pw 5.5 dn2 1

tof 0 ddr2 0

nt 8 dm2 0

ct 8 dm2 0

alock not used n dmf2 200

gain FLAGS

il n homo2 1.0

in n y wtfile

ns nm proc ft

sp -362.9 math not used f

wp 4992.5 vert

sc 101 wexp

wc 180 wbs

h2mm 27.74 wnt

rs 33.57

rfl 3628.1

th 3628.1

ins cdc ph 6.000

diethyl 2-(pyridin-3-yl)malonate

STANDARD CARBON PARAMETERS

exp2	szpu1	date	Apr 2 2807	DEC. & VT	499.742
		solvent	CDCl3	dn	H1
		file	exp	dpr	37
		ACQUISITION	exp	dm	0
		tn	125.671	dm	yyv
		at	1.300	dmf	11696
		np	130048	dseq	
		sw	50000.0	dres	1.0
		fb	28000	homo	n
		bs	8		DEC2
		tpwr	63	dfrq2	0
		dv	6.0	dm2	
		dv	0	dfr2	1
		nt	0	dm2	0
		ct	5000	dm2	n
		alock	1624	dm2	c
		gain	not used	dseq2	10000
		FLAGS	not used	dres2	
		fl	n	homo2	1.0
		in	n		n
		ds	y	lb	PROCESSING 0.50
		ns	nn	wfile	
		sp	-1738.0	proc	ft
		wp	24873.0	fn	not used
		vs	1424	math	f
		sc	0	werr	
		wcmm	180	wexp	
		hzmm	138.16	wds	
		h1	508.16	wnt	
		rfl	2271.0		
		th	5675.8		
		ins	100.000		
		ai	cdc	ph

diethyl 2-(pyridin-3-yl)malonate

File : C:\msdchem\1\DATA\Fan\FAN085.D
Operator :
Acquired : 27 Mar 2007 14:30 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name :
Misc Info :
Vial Number: 2

diethyl 2-(thiophen-2-yl)malonate

diethyl 2-(thiophen-2-yl)malonate

STANDARD CARBON PARAMETERS

EXP2	S2PU1	SAMPLE	DEC. & VT
date	Apr 26 2007	dfrq	499.742
file	COU1	dpr	37
file	ACQUISITION	dof	0
tr	125.671	dm	yyv
tn	1.013	dmm	11896
np	130048	dseq	
sw	50000.0	dres	1.0
fb	28000	homo	DEC2
ts	63	dfrq2	0
pw	6.0	dn2	1
d1	0	dpwr2	0
tof	5000	dot2	0
ct	1488	dma	n
alock	not used	dmr2	10000
gain	not used	dseq2	
fl	1.0	dres2	1.0
fl	n	homop	PROCESSING
dp	n	lb	0.50
hs	nn	wtfile	
sp	1574.6	proc	not used
wp	24873.0	math	
vs	1433	werr	
sc	100	wexp	
hmm	136.18	wnt	
rf1	500.00	wnt	
rfl	22771.0		
rpp	9675.8		
lms	100.000		
al	cdc	ph

diethyl 2-(thiophen-2-yl)malonate

File : C:\msdchem\1\DATA\Fan\FAN092(2) .D
Operator :
Acquired : 17 Apr 2007 10:23 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name: FAN092(2)
Misc Info :
Vial Number: 4

dimethyl 2-phenylmalonate

STANDARD PROTON PARAMETERS

exp2	szpu1	SAMPLE	date	Mar 28 2007	DEC. & VT	dfrq	499.742
file	solvent	CDCl3	dn	50	HI		
file	ACQUISITION	exp	dpr	30	nnn		
file	exp	dof	dm	200	c		
sfrq	499.742	dm	dm	200	1.0		
tn	1.892	dmf	dseq	1.0	n		
np	30272	dres	homo	DEC2			
sw	8000.0	homo	homo	0			
fb	4000	homo	homo	0			
bs	32	homo	homo	0			
tpwr	57	homo	homo	0			
pw	5.5	homo	homo	0			
d1	0	homo	homo	0			
tof	0	homo	homo	0			
nt	8	homo	homo	0			
ct	8	homo	homo	0			
dm2	8	homo	homo	0			
dmf2	200	homo	homo	0			
dseq2	1.0	homo	homo	0			
dres2	1.0	homo	homo	0			
gain	not used	homo	homo	0			
alock	not used	homo	homo	0			
flags	not used	homo	homo	0			
il	n	homo	homo	0			
in	n	homo	homo	0			
dp	n	homo	homo	0			
hs	y	homo	homo	0			
DISPLAY	nn	homo	homo	0			
sp	-245.2	homo	homo	0			
wp	4346.5	homo	homo	0			
vs	116	homo	homo	0			
sc	0	homo	homo	0			
wc	180	homo	homo	0			
h2mm	24.15	homo	homo	0			
ts	33.57	homo	homo	0			
rfl	5130.2	homo	homo	0			
rffp	3628.1	homo	homo	0			
th	3	homo	homo	0			
ins	6.000	homo	homo	0			
nm	cdc	homo	homo	0			
ph		homo	homo	0

dimethyl 2-phenylmalonate

STANDARD CARBON PARAMETERS

exp2	s2pu1	SAMPLE	DEC. & VT
date	Mar 26 2007	dfrq	499.742
solvent	CDCl3	dn	H1
file	CDCl3	dwr	37
ACQUISITION	exp	dof	0
sfrq	125.671	dm	yyv
tr	1.300	dmm	11696
at	130048	dscq	1.0
np	50000.0	dres	1.0
fb	28000	homo	n
ds	8	DEC2	
tpwr	63	dfrq2	0
pw	6.0	dn2	1
dl	0	dpwr2	0
tof	5000	dof2	n
ct	408	dmm2	c
alock	not used	dseq2	10000
gain	not used	dres2	1.0
FLAGS		homo2	n
il	n	lb	PROCESSING
in	n	wtfile	0.50
dp	y	proc	ft
hs	nn	fn	not used
sp	-1927.9	math	f
wp	26332.5	werr	
vs	661	wexp	
sc	0	wbs	
wc	180	wnt	
hzmm	146.29		
is	500.00		
rfl	22771.0		
rffp	9675.8		
th	9		
ins	100.000		
ai	cdc		
ph

dimethyl 2-phenylmalonate

File : C:\msdchem\1\DATA\Fan\FAN081(2).D
Operator : Fan
Acquired : 21 Mar 2007 14:28 using AcqMethod METHOD_1.M
Instrument : 5973N
Sample Name: FAN081(2)
Misc Info :
Vial Number: 4

Scan 660 (6.858 min): FAN081(2).D\data.ms

6. X-ray crystallographic data

Table 1. Crystal data and structure refinement for bcgama10. (21/3-2007)

Identification code	gama10	
Empirical formula	$C_{15} H_{20} O_6$	
Formula weight	296.31	
Temperature	294(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	$a = 7.9220(9)$ Å	$= 101.847(2)^\circ$.
	$b = 8.5812(10)$ Å	$= 96.372(2)^\circ$.
	$c = 11.9716(14)$ Å	$= 97.702(2)^\circ$.
Volume	$781.22(16)$ Å ³	
Z	2	
Density (calculated)	1.260 Mg/m ³	
Absorption coefficient	0.097 mm ⁻¹	
F(000)	316	
Crystal size	0.50 x 0.50 x 0.48 mm ³	
Theta range for data collection	2.46 to 27.54°.	
Index ranges	$-10 \leq h \leq 10$, $-11 \leq k \leq 11$, $-15 \leq l \leq 15$	
Reflections collected	7413	
Independent reflections	3591 [R(int) = 0.0336]	
Completeness to theta = 27.54°	99.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000 and 0.801	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3591 / 0 / 194	
Goodness-of-fit on F ²	1.002	
Final R indices [I>2sigma(I)]	R1 = 0.0601, wR2 = 0.1570	
R indices (all data)	R1 = 0.0849, wR2 = 0.1778	
Largest diff. peak and hole	0.285 and -0.253 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for gamma10. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	6267(2)	1252(2)	8490(1)	76(1)
O(2)	4154(2)	2591(2)	8979(1)	73(1)
O(3)	7901(2)	2803(2)	6414(1)	71(1)
O(4)	8731(1)	4463(2)	8141(1)	61(1)
O(5)	4421(2)	5899(1)	6838(1)	60(1)
O(6)	807(2)	2307(2)	3659(1)	79(1)
C(1)	4401(2)	3131(2)	6605(1)	45(1)
C(2)	3764(2)	4395(2)	6201(1)	47(1)
C(3)	2565(2)	4073(2)	5221(1)	55(1)
C(4)	1981(2)	2491(2)	4629(1)	56(1)
C(5)	2588(2)	1219(2)	5001(2)	59(1)
C(6)	3791(2)	1573(2)	5985(2)	55(1)
C(7)	5765(2)	3508(2)	7649(1)	47(1)
C(8)	5461(2)	2309(2)	8411(1)	52(1)
C(9)	3665(3)	1462(3)	9696(2)	86(1)
C(10)	2090(4)	1801(3)	10130(2)	109(1)
C(11)	7568(2)	3516(2)	7309(1)	49(1)
C(12)	10509(2)	4611(3)	7907(2)	72(1)
C(13)	11571(3)	5820(3)	8857(2)	87(1)
C(14)	3870(3)	7245(2)	6455(2)	71(1)
C(15)	286(3)	732(3)	2972(2)	89(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for *gama10*.

O(1)-C(8)	1.190(2)
O(2)-C(8)	1.324(2)
O(2)-C(9)	1.463(2)
O(3)-C(11)	1.1950(19)
O(4)-C(11)	1.3283(18)
O(4)-C(12)	1.461(2)
O(5)-C(2)	1.3584(17)
O(5)-C(14)	1.429(2)
O(6)-C(4)	1.372(2)
O(6)-C(15)	1.412(3)
C(1)-C(6)	1.382(2)
C(1)-C(2)	1.405(2)
C(1)-C(7)	1.509(2)
C(2)-C(3)	1.379(2)
C(3)-C(4)	1.387(2)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.384(2)
C(5)-C(6)	1.384(2)
C(5)-H(5A)	0.9300
C(6)-H(6A)	0.9300
C(7)-C(8)	1.521(2)
C(7)-C(11)	1.528(2)
C(7)-H(7A)	0.9800
C(9)-C(10)	1.446(3)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(12)-C(13)	1.465(3)
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600

C(13)-H(13C)	0.9600
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(8)-O(2)-C(9)	116.02(15)
C(11)-O(4)-C(12)	115.86(13)
C(2)-O(5)-C(14)	118.04(13)
C(4)-O(6)-C(15)	117.27(16)
C(6)-C(1)-C(2)	117.69(14)
C(6)-C(1)-C(7)	122.47(13)
C(2)-C(1)-C(7)	119.77(12)
O(5)-C(2)-C(3)	124.44(13)
O(5)-C(2)-C(1)	115.07(13)
C(3)-C(2)-C(1)	120.48(13)
C(2)-C(3)-C(4)	119.92(15)
C(2)-C(3)-H(3A)	120.0
C(4)-C(3)-H(3A)	120.0
O(6)-C(4)-C(5)	123.81(16)
O(6)-C(4)-C(3)	115.15(15)
C(5)-C(4)-C(3)	121.03(15)
C(4)-C(5)-C(6)	117.95(15)
C(4)-C(5)-H(5A)	121.0
C(6)-C(5)-H(5A)	121.0
C(1)-C(6)-C(5)	122.92(15)
C(1)-C(6)-H(6A)	118.5
C(5)-C(6)-H(6A)	118.5
C(1)-C(7)-C(8)	111.31(12)
C(1)-C(7)-C(11)	111.62(12)
C(8)-C(7)-C(11)	109.28(12)
C(1)-C(7)-H(7A)	108.2
C(8)-C(7)-H(7A)	108.2
C(11)-C(7)-H(7A)	108.2

O(1)-C(8)-O(2)	123.92(16)
O(1)-C(8)-C(7)	125.52(15)
O(2)-C(8)-C(7)	110.55(13)
C(10)-C(9)-O(2)	109.13(19)
C(10)-C(9)-H(9A)	109.9
O(2)-C(9)-H(9A)	109.9
C(10)-C(9)-H(9B)	109.9
O(2)-C(9)-H(9B)	109.9
H(9A)-C(9)-H(9B)	108.3
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
O(3)-C(11)-O(4)	124.12(15)
O(3)-C(11)-C(7)	125.38(14)
O(4)-C(11)-C(7)	110.48(12)
O(4)-C(12)-C(13)	108.10(16)
O(4)-C(12)-H(12A)	110.1
C(13)-C(12)-H(12A)	110.1
O(4)-C(12)-H(12B)	110.1
C(13)-C(12)-H(12B)	110.1
H(12A)-C(12)-H(12B)	108.4
C(12)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
O(5)-C(14)-H(14A)	109.5
O(5)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
O(5)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5

O(6)-C(15)-H(15A)	109.5
O(6)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
O(6)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for gama10. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	84(1)	75(1)	93(1)	48(1)	26(1)	36(1)
O(2)	71(1)	92(1)	80(1)	51(1)	35(1)	32(1)
O(3)	62(1)	83(1)	66(1)	1(1)	18(1)	16(1)
O(4)	46(1)	83(1)	54(1)	13(1)	13(1)	8(1)
O(5)	81(1)	44(1)	64(1)	20(1)	15(1)	22(1)
O(6)	75(1)	93(1)	68(1)	18(1)	-7(1)	29(1)
C(1)	45(1)	46(1)	53(1)	21(1)	14(1)	13(1)
C(2)	50(1)	47(1)	53(1)	21(1)	22(1)	18(1)
C(3)	59(1)	62(1)	58(1)	29(1)	18(1)	29(1)
C(4)	49(1)	72(1)	54(1)	21(1)	10(1)	19(1)
C(5)	62(1)	51(1)	65(1)	16(1)	6(1)	8(1)
C(6)	60(1)	47(1)	65(1)	25(1)	7(1)	14(1)
C(7)	49(1)	45(1)	52(1)	18(1)	13(1)	14(1)
C(8)	50(1)	57(1)	54(1)	20(1)	8(1)	12(1)
C(9)	90(1)	104(1)	85(1)	57(1)	33(1)	20(1)
C(10)	114(2)	111(2)	116(2)	40(2)	57(2)	7(2)
C(11)	51(1)	50(1)	52(1)	18(1)	11(1)	14(1)
C(12)	46(1)	103(1)	69(1)	18(1)	17(1)	13(1)
C(13)	56(1)	119(2)	79(1)	17(1)	12(1)	-1(1)
C(14)	94(1)	49(1)	85(1)	31(1)	27(1)	29(1)
C(15)	79(1)	112(2)	67(1)	8(1)	-7(1)	18(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for gama10.

	x	y	z	U(eq)
H(3A)	2150	4916	4958	66
H(5A)	2200	158	4602	71
H(6A)	4207	726	6241	66
H(7A)	5707	4585	8100	56
H(9A)	3496	363	9243	103
H(9B)	4573	1574	10334	103
H(10A)	1734	1031	10571	164
H(10B)	1208	1729	9495	164
H(10C)	2282	2869	10612	164
H(12A)	10586	4943	7184	87
H(12B)	10909	3581	7847	87
H(13A)	12752	5917	8730	130
H(13B)	11467	5493	9570	130
H(13C)	11188	6843	8894	130
H(14A)	4466	8229	6961	106
H(14B)	2653	7193	6462	106
H(14C)	4121	7217	5686	106
H(15A)	-489	766	2305	133
H(15B)	-285	59	3411	133
H(15C)	1277	299	2733	133

Table 6. Torsion angles [$^{\circ}$] for gama10.

C(14)-O(5)-C(2)-C(3)	-1.7(2)
C(14)-O(5)-C(2)-C(1)	178.07(14)
C(6)-C(1)-C(2)-O(5)	-179.42(14)
C(7)-C(1)-C(2)-O(5)	-2.2(2)
C(6)-C(1)-C(2)-C(3)	0.3(2)
C(7)-C(1)-C(2)-C(3)	177.51(14)
O(5)-C(2)-C(3)-C(4)	179.72(14)
C(1)-C(2)-C(3)-C(4)	0.0(2)
C(15)-O(6)-C(4)-C(5)	-4.2(3)
C(15)-O(6)-C(4)-C(3)	174.69(17)
C(2)-C(3)-C(4)-O(6)	-179.23(15)
C(2)-C(3)-C(4)-C(5)	-0.3(3)
O(6)-C(4)-C(5)-C(6)	179.08(16)
C(3)-C(4)-C(5)-C(6)	0.3(3)
C(2)-C(1)-C(6)-C(5)	-0.4(2)
C(7)-C(1)-C(6)-C(5)	-177.48(15)
C(4)-C(5)-C(6)-C(1)	0.1(3)
C(6)-C(1)-C(7)-C(8)	-41.5(2)
C(2)-C(1)-C(7)-C(8)	141.46(14)
C(6)-C(1)-C(7)-C(11)	80.92(18)
C(2)-C(1)-C(7)-C(11)	-96.14(16)
C(9)-O(2)-C(8)-O(1)	-2.9(2)
C(9)-O(2)-C(8)-C(7)	176.70(14)
C(1)-C(7)-C(8)-O(1)	105.58(18)
C(11)-C(7)-C(8)-O(1)	-18.2(2)
C(1)-C(7)-C(8)-O(2)	-74.01(16)
C(11)-C(7)-C(8)-O(2)	162.25(13)
C(8)-O(2)-C(9)-C(10)	-172.34(18)
C(12)-O(4)-C(11)-O(3)	0.3(2)
C(12)-O(4)-C(11)-C(7)	-178.30(15)
C(1)-C(7)-C(11)-O(3)	-24.8(2)
C(8)-C(7)-C(11)-O(3)	98.79(19)
C(1)-C(7)-C(11)-O(4)	153.79(13)
C(8)-C(7)-C(11)-O(4)	-82.66(15)

C(11)-O(4)-C(12)-C(13)

174.20(17)

Symmetry transformations used to generate equivalent atoms:

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