Parallel Kinetic Resolution of D-Labelled 2-Aryl-Propionic and Butanoic Acids Using *Quasi*-Enantiomeric Combinations of Oxazolidin-2-Ones

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ABSTRACT The parallel kinetic resolution of racemic 2-aryl-2-deuterio-propionic and butanoic acids using an equimolar combination of *quasi*-enantiomeric oxazolidin-2-ones is discussed. The levels of diastereoselectivity were high leading to enantiomerically pure D-labeled products in good yield. *Chirality 22:193–205, 2010.*

KEY WORDS: parallel kinetic resolution; kinetic resolution; *quasi*-enantiomers; 2-phenylpropionic acid; resolution; oxazolidin-2-ones

INTRODUCTION

The synthesis of enantiomerically pure profens[1–7] such as ibuprofen[8–10] and naproxen[11–14] is well documented. Since 2005, we have been interested in the mutual and parallel kinetic resolutions[15–21] of profen-based pentafluorophenyl active esters (based on carbon skeleton of 2-phenylpropionic acid) using a combination of oxazolidin-2-ones as a strategy for the separation of their enantiomers. For example, treatment of the active ester, pentafluorophenyl 2-phenylpropionate (*rac*)-**2**, with an equimolar amount of lithiated oxazolidin-2-ones {derived from the deprotonation of an equimolar mixture of oxazolidin-2-ones (*R*)-**1** and (*S*)-**1** [≡ (*rac*)-**1**] with *n*-BuLi}, gave the corresponding adducts (*S*,*R*)-*syn*- and (*R*,*S*)-*syn*-**3** [≡ (*rac*)-*syn*-**3**] as the major diastereoisomer in 70% yield with >94% *d.e.* (Scheme 1). The corresponding oxazolidin-2-one *anti*-**3** can be synthesised using Evans' diasteeoselective alkylation method; for further information see. Ref. 22–24 From this study, it was apparent that the (*R*)-enantiomer of oxazolidin-2-one **1** recognized the (*S*)-enantiomer of the active ester **2** to give oxazolidin-2-one (*S*,*R*)-*syn*-**3**, and the complementary enantiomeric oxazolidin-2-one (*S*)-**1** recognized the remaining (*R*)-enantiomer of the active ester **2** to give the enantiomeric adduct (*R*,*S*)-*syn*-**3** in an equal and opposite stereochemical sense (Scheme 1). We have also demonstrated that an isotopomeric mixture of *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-[D_2]-**1** can be used to resolve this active ester (*rac*)-**2** to give an inseparable mixture of labeled and unlabeled oxazolidin-2-ones (*S*,*R*)-*syn*-**3** and (*R*,*S*)-[D_2]-*syn*-**3** in a combined 70% yield with comparable diastereoselection to its unlabeled variant (>94% *d.e.*) (Scheme 1).[22]

We now report an extension of this study to the parallel kinetic resolution of a series of deuterium labeled 2-phenyl/aryl propionic and butanoic acids [D_1]-**4**-**7**[25] using a *quasi*-enantiomeric combination of isotopomeric and designer oxazolidin-2-ones. For this study, we had to synthesize a series of D-labeled pentafluorophenyl active esters [D_1]-**2** and [D_1]-**8**-**10** (Scheme 2). Addition of DCC to a stirred solution of D-labeled carboxylic acids [D_1]-**4**-**7** and pentafluorophenol in dichloromethane, gave the corresponding pentafluorophenyl active esters [D_1]-**2** and [D_1]-**8**-**10** in good yields (>85%) with high levels of deuterium incorporation (>95% D-incorporation) (Scheme 2).

With these active esters in hand, we next probed their parallel kinetic resolution using an isotopomeric pair of *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-[D_2]-**1** (Scheme 3). Treatment of an equimolar amount of oxazolidin-2-ones (*R*)-**1** and (*S*)-[D_2]-**1** with *n*-BuLi in THF at −78°C, followed by the addition of active esters [D_1]-**2** and [D_1]-**8**-**10**, gave after 2 h, an inseparable isotopomeric mixture of the corresponding oxazolidin-2-one adducts (*S*,*R*)-*syn*- and (*R*,*R*)-*anti*-[D_1]-**3**, [D_1]-**11**, [D_1]-**12** and [D_1]-**13**, and (*R*,*S*)-*syn*- and (*S*,*S*)-*anti*-[D_3]-**3**, [D_3]-**11**, [D_3]-**12** and [D_3]-**13**, respectively in good yields (71%–78%) with high levels of diastereoselectivity (94%–96% *d.e.*) (Scheme 3). From this study, there appears to be no primary or secondary kinetic isotope effect as the levels of deuterium incorporation remained unchanged during the course of this reaction. The D-labeled moiety served primarily as a stereochemical marker for enantiomeric recognition.

We first chose to investigate the optical resolution of these active esters [D_1]-**2** and [D_1]-**8**-**10** using a combination of separable oxazolidin-2-ones based on Evans' and Seebach's oxazolidin-2-ones (*R*)-**1**[26] and (*S*)-**14**[27,28], respectively (Scheme 4). Treatment of an equimolar combination of oxazolidin-2-ones (*R*)-**1** and (*S*)-**14** with *n*-BuLi in

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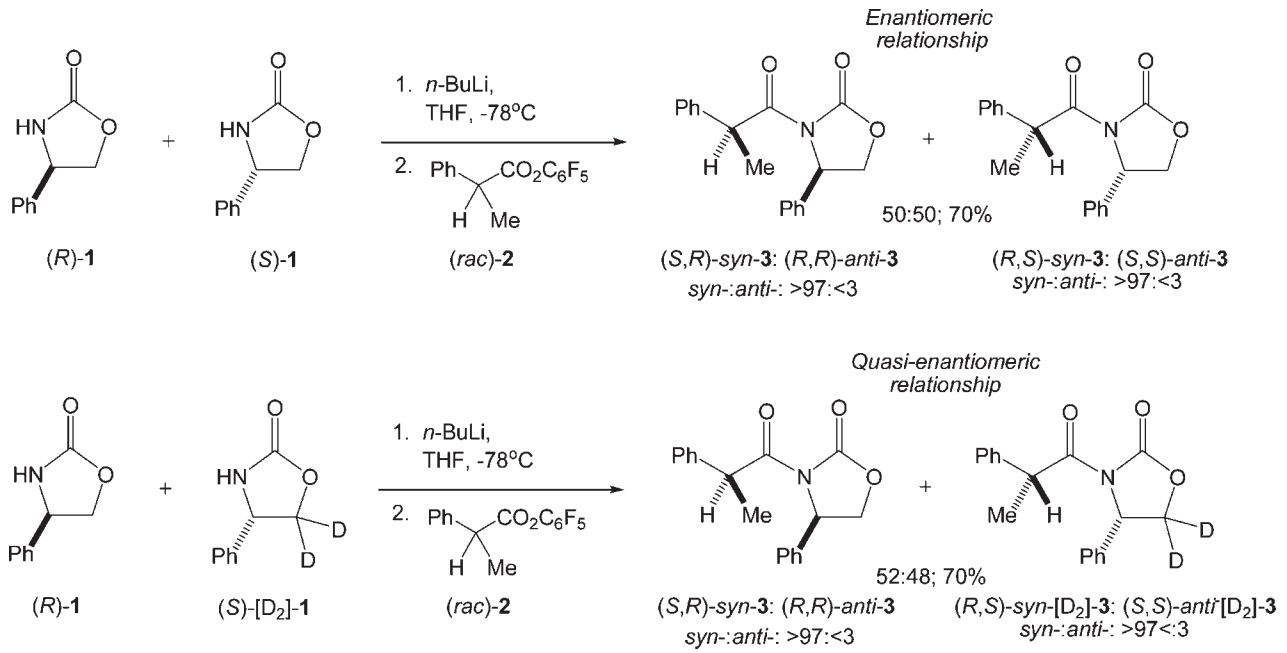
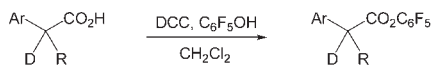


Scheme 1. Mutual/parallel kinetic resolution of active ester (*rac*)-**2** using (*quasi*-)enantiomeric oxazolidin-2-ones (*R*)-**1**/(*S*)-**1** and (*R*)-**1**/(*S*)-[D_2]-**1**.

THF at −78°C, followed by the addition of the D-labeled active esters [D_1]-**2** and [D_1]-**8-10**, gave after 2 h, a mixture of oxazolidin-2-one adducts (*S,R*)-[D_1]-*syn*-**3** (in 66% yield with 88% *d.e.*) and (*R,S*)-[D_1]-*syn*-**15** (in 54% yield with 94% *d.e.*) (for [D_1]-**2**), (*S,R*)-[D_1]-*syn*-**11** (in 69% yield with 98% *d.e.*) and (*R,S*)-[D_1]-*syn*-**16** (in 63% yield with 98% *d.e.*) (for [D_1]-**8**), (*S,R*)-[D_1]-*syn*-**12** (in 64% yield with 90% *d.e.*) and (*R,S*)-[D_1]-*syn*-**17** (in 57% yield with 98% *d.e.*) (for [D_1]-**9**), and (*S,R*)-[D_1]-*syn*-**13** (in 69% yield with 90% *d.e.*) and (*R,S*)-[D_1]-*syn*-**18** (in 64% yield with 94% *d.e.*) (for [D_1]-**10**) in high yields with excellent levels of diastereocontrol (Scheme 4). All products were easily separable by column chromatography [eluting with light petroleum ether (b.p. 40-60°C)/diether ether (1:1)] {ΔR_F [light petroleum ether (b.p. 40-60°C)/diether ether (1:1)] = ∼0.25} with the exception of those [(*R,S*)-*syn*-[D_1]- and (*S,S*)-*anti*-[D_1]-**15-18**] derived from Seebach's oxazolidin-2-one (*S*)-**14** (Scheme 4).

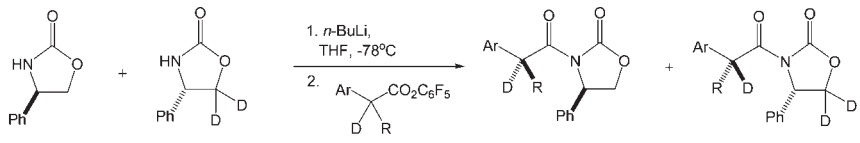
In an attempt to improve separability, we next turned our attention to probing the parallel kinetic resolution of these D-labeled active esters [D_1]-**2** and [D_1]-**8-10** using a combination of designer oxazolidin-2-ones based on Fox's[29] and Evans'[26] oxazolidin-2-ones (*R*)-**19** and (*S*)-**1**, respectively, which were known to lead to separable diastereoisomeric adducts. Deprotonation of the oxazolidin-2-ones (*R*)-**19** and (*S*)-**1** using *n*-BuLi in THF at −78°C, followed by the addition of active esters [D_1]-**2** and [D_1]-**8-10**, gave after 2 h, the corresponding oxazolidin-2-ones (*S,R*)-[D_1]-*syn*-**20** (in 78% with 94% *d.e.*) and (*R,S*)-[D_1]-*syn*-**3** (in 69% yield with 94% *d.e.*) (for [D_1]-**2**), (*S,R*)-[D_1]-*syn*-**21** (in 67% yield with 98% *d.e.*) and (*R,S*)-[D_1]-*syn*-**11** (in 68% yield with 98% *d.e.*) (for [D_1]-**8**), (*S,R*)-[D_1]-*syn*-**22** (in 71% with 90% *d.e.*) and (*R,S*)-[D_1]-*syn*-**12** (in 76% yield with 92% *d.e.*) (for [D_1]-**9**), and (*S,R*)-[D_1]-*syn*-**23** (in 77% with 94% *d.e.*) and (*R,S*)-[D_1]-*syn*-**13** (in 76% yield with 94% *d.e.*) (for [D_1]-**10**), respectively, in high yield with excellent levels of diastereocontrol (Scheme 5).

With these adducts at hand, we next investigated the hydrolysis of two pairs of *quasi*-enantiomeric combinations of oxazolidin-2-ones (*S,R*)-*syn*-[D_1]-**3** and (*R,S*)-*syn*-[D_1]-**15**, and (*S,R*)-*syn*-[D_1]-**20** and (*R,S*)-*syn*-[D_1]-**3** using LiOH monohydrate (mediated by hydrogen peroxide) in THF/H_2O (3:1) (Scheme 6). Simple treatment of the oxazolidin-2-ones (*S,R*)-*syn*-[D_1]-**3**, (*R,S*)-*syn*-[D_1]-**15**, (*S,R*)-*syn*-[D_1]-**20** and (*R,S*)-*syn*-[D_1]-**3** with LiOH/H_2O_2 in THF/H_2O (3:1), and stirring the resulting solution for 12 h, gave the corresponding enantiomerically enriched 2-deuterio-2-phenylpropionic acids [D_1]-**4** in good yields (Scheme 6). The enantiomeric excess of 2-phenylpropionic acid **4** was determined by derivatisation with enantiomerically pure 1-phenylethanol using a DCC/DMAP coupling procedure; see experimental details for a representative procedure. The levels of deuterium incorporation for the carboxylic



Entry	Carboxylic acid	[D]:[H]	Ar	R	Active ester	[D]:[H]	Yield
1	[D_1]-**4**	97:3	Ph	Me	[D_1]-**2**	97:3	85%
2	[D_1]-**5**	96:4	Ph	Et	[D_1]-**8**	96:4	86%
3	[D_1]-**6**	96:4	4-MeC_6H_4-	Me	[D_1]-**9**	96:4	89%
4	[D_1]-**7**	95:5	4-*i*-BuC_6H_4-	Me	[D_1]-**10**	95:5	87%

Scheme 2. Synthesis of active esters [D_1]-**2**, [D_1]-**8**, [D_1]-**9** and [D_1]-**10**.



Active ester	Ar	R	Oxazolidinone adducts derived from (*R*)-**1**	D.e.	Oxazolidinone adducts derived from (*S*)-[D_2]-**1**	D.e.	[D_3]:[D_2] [D_1]:[H]	[D_3]:[D_1]	Yield
[D_1]-**2**	Ph	Me	(*S,R*)-*syn*-[D_1]-**3**: (*R,R*)-*anti*-[D_1]-**3** 97:3	94%	(*R,S*)-*syn*-[D_3]-**3**: (*S,S*)-*anti*-[D_3]-**3** 97:3	94%	97:3	52:48	71%
[D_1]-**8**	Ph	Et	(*S,R*)-*syn*-[D_1]-**11**: (*R,R*)-*anti*-[D_1]-**11** 98:2	96%	(*R,S*)-*syn*-[D_3]-**11**: (*S,S*)-*anti*-[D_3]-**11** 98:2	96%	95:5	53:47	78%
[D_1]-**9**	4-MeC_6H_4-	Me	(*S,R*)-*syn*-[D_1]-**12**: (*R,R*)-*anti*-[D_1]-**12** 98:2	96%	(*R,S*)-*syn*-[D_3]-**12**: (*S,S*)-*anti*-[D_3]-**12** 98:2	96%	97:3	51:49	73%
[D_1]-**10**	4-*i*-BuC_6H_4-	Me	(*S,R*)-*syn*-[D_1]-**13**: (*R,R*)-*anti*-[D_1]-**13** 97:3	94%	(*R,S*)-*syn*-[D_3]-**13**: (*S,S*)-*anti*-[D_3]-**13** 97:3	94%	95:5	53:47	73%

Scheme 3. Parallel kinetic resolution of active esters (*rac*)-[D_1]-**2** and [D_1]-**8-10** using *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-[D_2]-**1**.

acids (*e.g.*, [D_1]-**4**) remained unchanged (±2%) throughout the resolution sequence.

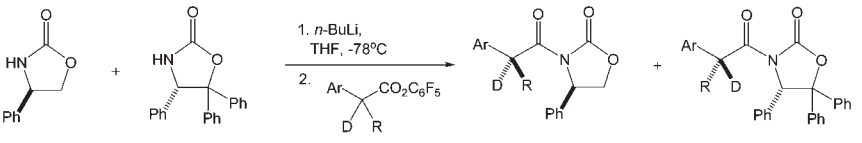
EXPERIMENTAL

General

All solvents were distilled before use. All reactions were carried out under nitrogen using oven-dried glassware. Flash column chromatography was carried out using Merck Kieselgel 60 (230–400 mesh). Thin layer chromatography (TLC) was carried out on commercially available pre-coated plates (Merck Kieselgel 60F_{254} silica). Proton and carbon NMR spectra were recorded on a Bruker 400 MHz Fourier transform spectrometer using an internal deuterium lock. Chemical shifts are quoted in parts per million downfield from tetramethylsilane. Carbon NMR spectra were recorded with broad proton decoupling. Infrared spectra were recorded on a Shimadzu 8300 FTIR spectrometer. Optical rotations were measured using an automatic AA-10 Optical Activity polarimeter. All isotopically labeled oxazolidin-2-one adducts have been given an L superscript and unlabeled derivatives a U superscript.

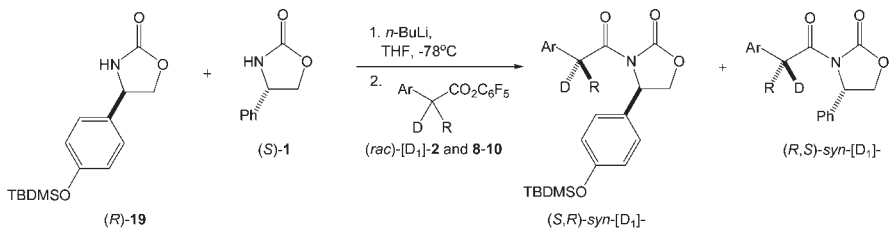
Pentafluorophenyl 2-deuterio-2-phenyl-propionate (rac)-[D_1]-2

N,N'-Dicyclohexylcarbodiimide (DCC) (0.78 g, 3.82 mmol) was slowly added to a stirred solution of 2-deuterio-2-phenylpropionic acid (*rac*)-[D_1]-**2** (0.51 g, 3.38 mmol, [D]:[H] = 97:3) in dichloromethane (5 mL) at room temperature. The resulting solution was stirred for 15 minutes. A solution of pentafluorophenol (0.62 g, 3.40 mmol) in dichloromethane (5 mL) was added slowly and the solution was stirred for a further 12 h. The resulting white precipitate (dicyclohexylurea) was removed through filtration (using a sintered funnel). The reaction was quenched with water (20 mL) and extracted with dichloromethane (3 × 10 mL). The combined organic layers were dried (over $MgSO_4$) and evaporated under reduced pres-



Active ester	Ar	R	Oxazolidinone adducts derived from (*R*)-**1**	D.e.	[D]:[H]	Yield	Oxazolidinone adducts derived from (*S*)-**14**	D.e.	[D]:[H]	Yield
[D_1]-**2**	Ph	Me	(*S,R*)-*syn*-[D_1]-**3**: (*R,R*)-*anti*-[D_1]-**3** 94:6	88%	97:3	66%	(*R,S*)-*syn*-[D_1]-**15**: (*S,S*)-*anti*-[D_1]-**15** 97:3	94%	97:3	54%
[D_1]-**8**	Ph	Et	(*S,R*)-*syn*-[D_1]-**11**: (*R,R*)-*anti*-[D_1]-**11** 99:1	98%	95:5	69%	(*R,S*)-*syn*-[D_1]-**16**: (*S,S*)-*anti*-[D_1]-**16** 99:1	98%	95:5	63%
[D_1]-**9**	4-MeC_6H_4-	Me	(*S,R*)-*syn*-[D_1]-**12**: (*R,R*)-*anti*-[D_1]-**12** 95:5	90%	97:3	64%	(*R,S*)-*syn*-[D_1]-**17**: (*S,S*)-*anti*-[D_1]-**17** 99:1	98%	97:3	57%
[D_1]-**10**	4-*i*-BuC_6H_4-	Me	(*S,R*)-*syn*-[D_1]-**13**: (*R,R*)-*anti*-[D_1]-**13** 95:5	90%	95:5	69%	(*R,S*)-*syn*-[D_1]-**18**: (*S,S*)-*anti*-[D_1]-**18** 97:3	94%	95:5	64%

Scheme 4. Parallel kinetic resolution of active esters (*rac*)-[D_1]-**2** and [D_1]-**8-10** using *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-**14**.



Active ester	Ar	R	Oxazolidinone adducts derived from (*R*)-**19**	D.e.	[D]:[H]	Yield	Oxazolidinone adducts derived from (*S*)-**1**	D.e.	[D]:[H]	Yield
[D_1]-**2**	Ph	Me	(*S,R*)-*syn*-[D_1]-**20**: (*R,R*)-*anti*-[D_1]-**20** 97:3	94%	95:5	78%	(*R,S*)-*syn*-[D_1]-**3**: (*S,S*)-*anti*-[D_1]-**3** 97:3	94%	95:5	69%
[D_1]-**8**	Ph	Et	(*S,R*)-*syn*-[D_1]-**21**: (*R,R*)-*anti*-[D_1]-**21** 99:1	98%	95:5	67%	(*R,S*)-*syn*-[D_1]-**11**: (*S,S*)-*anti*-[D_1]-**11** 99:1	98%	95:5	68%
[D_1]-**9**	4-MeC_6H_4-	Me	(*S,R*)-*syn*-[D_1]-**22**: (*R,R*)-*anti*-[D_1]-**22** 95:5	90%	96:4	71%	(*R,S*)-*syn*-[D_1]-**12**: (*S,S*)-*anti*-[D_1]-**12** 96:4	92%	96:4	76%
[D_1]-**10**	4-*i*-BuC_6H_4-	Me	(*S,R*)-*syn*-[D_1]-**23**: (*R,R*)-*anti*-[D_1]-**23** 97:3	94%	95:5	77%	(*R,S*)-*syn*-[D_1]-**13**: (*S,S*)-*anti*-[D_1]-**13** 97:3	94%	95:5	76%

Scheme 5. Parallel kinetic resolution of active esters (*rac*)-[D_1]-**2** and [D_1]-**8-10** using *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**19** and (*S*)-**1**.

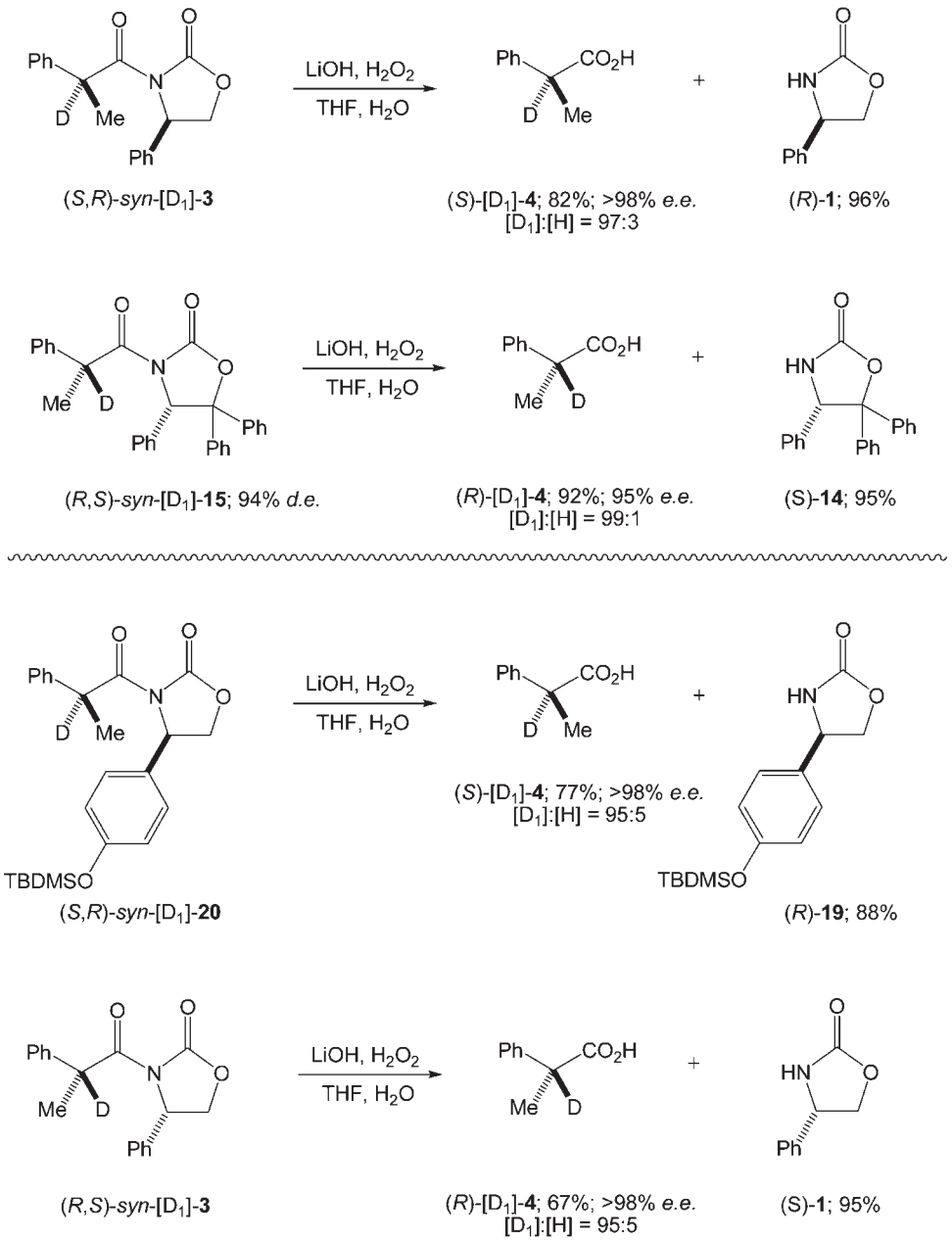
sure. The residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (9:1) to give pentafluorophenyl 2-deuterio-2-phenyl-propionate (*rac*)-[D_1]-**2** (0.91 g, 85%, [D]:[H] = 97:3) as a colourless oil; R_F [light petroleum (40–60°C):diethyl ether (1:1)] 0.88; $\nu_{max}(CH_2Cl_2)cm^{-1}$ 2397 (C-D) and 1781 (C=O); δ_H (400 MHz; $CDCl_3$) 7.41-7.25 (5 H, m, 5 × CH; Ph), and 1.63 (3 H, s, CH_3CD); δ_C (100 MHz; $CDCl_3$) 170.7 (C=O), 141.4 (142.7 × and 140.20, 2 C, ddt, $^1J_{C,F}$ = 251.3 Hz, $^2J_{C,F}$ = 12.2 Hz and $^3J_{C,F}$ = 3.8 Hz, C(2)-F), 139.7 (141.00 and 138.48, 1 C, dtt, $^1J_{C,F}$ = 253.2 Hz, $^2J_{C,F}$ = 13.4 Hz and $^3J_{C,F}$ = 4.2 Hz, C(4)-F), 139.0 (*i*-C; Ph), 138.1 (139.35 and 136.88, 2 C, dtdd, $^1J_{C,F}$ = 249.1 Hz, $^2J_{C,F}$ = 14.5 Hz, $^3J_{C,F}$ = 5.7 Hz and $^4J_{C,F}$ = 3.1 Hz, C(3)-F), 129.0,[2] 127.8[2] and 127.5[1] (5 × CH; Ph), 125.4 (1 C, tdt, $^2J_{C,F}$ = 14.5 Hz, $^4J_{C,F}$ = 4.2 Hz and $^3J_{C,F}$ = 2.0 Hz, *i*-CO; OC_6F_5), 45.1 (unlabelled Ph*C*HCH$_3$), 44.7 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ph*C*DCH$_3$) and 18.5 (PhCD*C*H$_3$) (Found M^+, 317.0579; $C_{15}H_8DF_5O_2$ requires M^+, 317.0580); negative isotopic shift at 44.7 ppm (Ph*C*DCH$_3$) was 0.3591 ppm (35.9 Hz at 100.6 MHz).

*Pentafluorophenyl 2-deuterio-2-phenylbutanoate (***rac***)-[D_1]-8*

In the same way as the active ester (*rac*)-[D_1]-**2**, 2-deuterio-2-phenylbutanoic acid (*rac*)-[D_1]-**5** (0.53 g, 3.18 mmol, [D]:[H] = 96:4), DCC (0.73 g, 3.52 mmol) and pentafluorophenol (0.59 g, 3.20 mmol) in dichloromethane (40 mL), gave after purification by flash column chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C): diethyl ether (9:1), pentafluorophenyl 2-deuterio-2-phenylbutanoate (*rac*)-[D_1]-**8** (0.91 g, 86%, [D]:[H] = 96:4) as a colourless liquid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.80; $\nu_{max}(CHCl_3)cm^{-1}$ 2399 (C-D) and 1772 (C=O); δ_H (400 MHz; $CDCl_3$) 7.41-7.27 (5 H, m, 5 × CH; Ph), 2.27 (1 H, dq, 13.9 and 7.5, CH_AH$_B$CH$_3$), 1.97 (1 H, dq, 13.9 and 7.5, CH$_A$H$_B$CH$_3$) and 1.01 (3 H, t, *J* 7.5, CH$_2$CH_3); δ_C (100 MHz; $CDCl_3$) 170.1 (OC=O), 141.1 (142.36 and 139.88, 2 C, dddt, $^1J_{C,F}$ = 251.3 Hz, $^2J_{C,F}$ = 11.9 Hz, $^3J_{C,F}$ = 3.4 Hz and $^4J_{C,F}$ = 3.4 Hz, C(2)-F), 139.4 (140.65 and 138.14, 1 C, dtt, $^1J_{C,F}$ = 252.8 Hz, $^2J_{C,F}$ = 13.9 Hz and $^3J_{C,F}$ = 3.8 Hz, C(4)-F), 137.8 (139.04 and 136.55, 2 C, dtdd, $^1J_{C,F}$ = 254.3 Hz, $^2J_{C,F}$ = 14.2 Hz, $^3J_{C,F}$ = 4.9 Hz and $^4J_{C,F}$ = 2.6 Hz, C(3)-F), 137.2 (*i*-C; Ph), 128.8,[2] 127.9[2] and 127.8[1] (5 × CH; Ph), 125.3 (1 C, tdt, $^2J_{C,F}$ = 14.2 Hz, $^4J_{C,F}$ = 4.4 Hz and $^3J_{C,F}$ = 2.2 Hz, *i*-CO; OC_6F_5), 52.7 (unlabelled PhCH), 52.3 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.6 Hz, PhCD), 26.6 (*C*H$_2$CH$_3$) and 11.8 (CH$_2$*C*H$_3$) (Found M^+, 331.0732; $C_{16}H_{10}DF_5O_2$ requires M^+, 331.0736); negative isotopic shift at 52.3 ppm (Ph*C*D) was 0.3973 ppm (39.7 Hz at 100.6 MHz).

*Pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (***rac***)-[D_1]-9*

In the same way as the active ester (*rac*)-[D_1]-**2**, 2-deuterio-2-(4-methylphenyl)propionic acid (*rac*)-[D_1]-**6** (0.72 g, 4.35 mmol, [D]:[H] = 96:6), DCC (1.01 g, 4.91 mmol) and pentafluorophenol (0.81 g, 4.42 mmol) in dichloromethane (20 mL), gave after purification by flash column chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C): diethyl ether (9:1), pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (*rac*)-[D_1]-**9** (1.29 g, 89%, [D]:[H] = 96:4) as a colourless oil; R_F [light petroleum ether (b.p. 40–60°C): diethyl ether (1:1)] 0.83; ν_{max}(film)cm^{-1} 2399 (C=O) and 1780 (C=O); δ_H (400 MHz; $CDCl_3$) 7.25 (2 H, dd, *J* 8.2 and 1.8, 2 × CH; Ar), 7.19 (2 H, dd, *J* 8.2 and 1.8, 2 × CH; Ar), 2.35 (3 H, s, CH_3; Ar) and 1.62 (3 H, s, CH_3CD); δ_C (100 MHz; $CDCl_3$) 170.8 (C=O), 141.2 (142.50 and 139.95, 2 C, ddt, $^1J_{C,F}$ = 251.6 Hz, $^2J_{C,F}$ = 11.9 Hz and $^3J_{C,F}$ = 4.6 Hz, C(2)-F), 139.5 (140.74 and 138.23, 1 C, dtt, $^1J_{C,F}$ = 252.8 Hz, $^2J_{C,F}$ = 13.4 Hz and $^3J_{C,F}$ = 3.8 Hz), C(4)-F), 137.9 (139.14 and 136.63, 2 C, dtdd, $^1J_{C,F}$ = 252.8 Hz, $^2J_{C,F}$ = 12.1 Hz, $^3J_{C,F}$ = 5.3 Hz and $^4J_{C,F}$ = 3.1 Hz, C(3)-F), 137.6 and 135.8 (2 × *i*-C; Ar),



Scheme 6. Synthesis of enantiomerically pure 2-deuterio-2-phenylpropionic acids (*S*)-[D_1]- and (*R*)-[D_1]-**4**.

129.7[2] and 127.4[2] (4 × CH; Ar), 125.2 (1 C, tdt, $^2J_{C,F}$ = 14.3 Hz, $^4J_{C,F}$ = 4.6 Hz and $^3J_{C,F}$ = 2.3 Hz, *i*-CO; OC_6F_5), 44.8 (1 C, s, unlabelled ArCH), 44.4 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.4 Hz, ArCD), 21.1 (CH_3; Ar) and 18.6 (ArCD*C*H_3) (Found M^+, 331.0739; $C_{16}H_{10}DF_5O_2$ requires M^+, 331.0736); negative isotopic shift at 44.4 ppm (Ph*C*D) was 0.3590 ppm (35.9 Hz at 100.6 MHz).

*Pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (***rac***)-[D_1]-10*

In the same way as the active ester (*rac*)-[D_1]-**2**, 2-deuterio-2-(4-isobutylphenyl)propionic acid (*rac*)-[D_1]-**7** (0.65 g, 3.13 mmol, [D]:[H] = 95:5), DCC (0.71 g, 3.44 mmol) and pentafluorophenol (0.59 g, 3.20 mmol) in dichloromethane (100 mL), gave after purification by flash column chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C): diethyl ether (9:1), pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (*rac*)-[D_1]-**10** (1.02 g, 87%, [D]:[H] = 95:5) as a colourless liquid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.80; $\nu_{max}(CHCl_3)cm^{-1}$ 2399 (C-D) and 1781 (C=O); δ_H(400 MHz; $CDCl_3$) 7.20 (2 H, dt, *J* 8.2 and 2.2, 2 × CH; Ar), 7.07 (2 H, dt, *J* 8.2 and 2.2, 2 × CH; Ar), 2.40 (2 H, d, *J* 7.2, CH_2CH), 1.86-1.72 (1 H, m, C*H*$(CH_3)_2$), 1.55 (3 H, s, ArCDCH_3) and 0.83 (6 H, d, *J* 6.7, $(CH_3)_2$CH); δ_C (100 MHz; $CDCl_3$) 170.8 (C=O), 141.4 (*i*-C; Ar), 141.2 (142.50

and 140.01, 2 C, ddt, $^1J_{C,F}$ = 251.3 Hz, $^2J_{C,F}$ = 11.9 Hz and $^3J_{C,F}$ = 4.2 Hz, C(2)-F), 139.4 (140.74 and 138.23, 1 C, dtt, $^1J_{C,F}$ = 253.2 Hz, $^2J_{C,F}$ = 13.8 Hz and $^3J_{C,F}$ = 3.8 Hz, C(4)-F), 137.8 (139.14 and 136.64, 2 C, dtdd, $^1J_{C,F}$ = 254.7 Hz, $^2J_{C,F}$ = 14.5 Hz, $^3J_{C,F}$ = 5.3 and $^4J_{C,F}$ = 3.0 Hz, C(3)-F), 135.9 (*i*-C; Ar), 129.7[2] and 127.2[2] (4 × CH; Ar), 124.8 (1 C, tdt, $^2J_{C,F}$ = 14.2 Hz, $^4J_{C,F}$ = 4.6 Hz and $^3J_{C,F}$ = 2.3 Hz, *i*-CO; OC_6F_5), 45.1 (CH_2; Ar), 44.7 (unlabelled Ar*C*HCH$_3$), 44.4 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ar*C*DCH$_3$), 30.1 (*C*HCH$_2$), 22.3 (CH(*C*H$_3$)$_2$) and 18.4 (ArCD*C*H$_3$) (Found M^+, 337.1203; $C_{19}H_{16}DF_5O_2$ requires M^+, 373.1206); negative isotopic shift at 44.39 ppm (Ar*C*D) was 0.3629 ppm (36.2 Hz at 100.6 MHz).

*Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-phenylpropionate (*rac*)-[D_1]-2 using 4-phenyl-oxazolidin-2-one (R)-1 and 4-phenyl-5,5-dideuterio-oxazolidin-2-one (S)-[D_2]-1*

n-BuLi (0.19 mL, 2.5 M in hexane, 0.47 mmol) was added to a stirred solution of 4-phenyl-oxazolidin-2-one (*R*)-**1** (32 mg, 0.20 mmol) and 4-phenyl-5,5-dideuterio-oxazolidin-2-one (*S*)-[D_2]-**1** (33 mg, 0.20 mmol) in THF (5 mL) at −78°C. After stirring for 1 h, a solution of pentafluorophenyl 2-deuterio-2-phenylpropionate (*rac*)-[D_1]-**2** (0.152 g, 0.48 mmol, [D]:[H] = 97:3) in THF (1 mL) was added. The resulting mixture was stirred for 2 h at −78°C. The reaction was quenched with water (10 mL). The organic layer was extracted with diethyl ether (2 × 10 mL), dried (over $MgSO_4$) and evaporated under reduced pressure to give a mixture of diastereoisomeric oxazolidin-2-ones **3** [ratio 97:3: *syn-*:*anti-*]. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give an inseparable mixture of (2*S*,4*R*)-3-[2-deuterio-2-phenylpropionyl]-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**3** and (2*R*,4*S*)-3-[2-deuterio-2-phenylpropionyl]-4-phenyl-5,5-dideuterio-oxazolidin-2-one (*R*,*S*)-*syn*-[D_3]-**3** (84 mg, 71%; ratio (*S*,*R*)-*syn*-[D_1]-**3**: (*R*,*S*)-*syn*-[D_3]-**3** = 52:48) as a white solid; R_F [light petroleum ether (b.p. 40–60°C): diethyl ether: (1:1)] 0.33; m.p. 121–123°C; (Ref. 22 unlabelled (*rac*)-*syn*-**3**; m.p. 124-125 °C; unlabelled (*R*,*S*)-**3**; m.p. 128-130°C); $[\alpha]_D^{20}$ = +3.90 (*c* 1.2, $CHCl_3$) {Ref. 30 (*S*,*R*)-*syn*-**3**; $[\alpha]_D^{20}$ = +92.5 (*c* 4.9, $CHCl_3$)}; ν_{max} ($CHCl_3$)cm^{-1} 2400 (C-D), 1780 (OC=O) and 1706 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.22-7.11 (12 H, m, 12 × CH; 2 × Ph^{U+L}), 7.06-7.01 (4 H, m, 4 × CH; Ph^{U+L}), 6.87-6.82 (4 H, m, 4 × CH; Ph^{U+L}), 5.38 (1 H, dd *J* 9.0 and 5.1, CHN^U), 5.37 (1 H, s, CHN^L), 4.57 (1 H, t, *J* 9.0, C*H*$_A$H$_B$O^U), 4.02 (1 H, dd, *J* 9.0 and 5.1, CH$_A$*H*$_B$O^U) and 1.32 (6 H, s, PhCDC*H*$_3^{U+L}$); negative isotopic shift at 5.37 ppm was 0.0106 ppm (1.06 Hz at 400 MHz); δ_C(100 MHz; $CDCl_3$) 173.7 (2 C; NC=O^{U+L}), 153.1 (2 C; OC=O^{U+L}), 139.7 and 138.2 (4 × *i*-C; 2 × Ph^{U+L}), 128.9,[4] 128.5,[6] 128.2,[4] 127.1[2] and 125.9[4] (20 × CH; 2 × Ph^{U+L}), 69.6 (CH_2O^U), 68.9 (1 C, quintet, $^1J_{C,D}$ = 21.4 Hz, CD_2O^L), 57.7 (CHN^U), 57.6 (CHN^L), 43.8 (unlabelled Ph*C*HCH$_3$), 43.4 (2 C, t [1:1:1], $^1J_{C,D}$ = 21.4 Hz, Ph*C*DCH$_3^{U+L}$) and 18.5 (2 × PhCD*C*H$_3^{U+L}$); negative isotopic shifts at 68.9405 ppm was 0.6188 ppm (61.88 Hz at 100 MHz), 57.6039 ppm was 0.1681 ppm (16.81 Hz at 400 MHz) and at 43.4482 ppm was 0.3590 ppm (35.90 Hz at 400 MHz). By mass spectrometry, this mixture of oxazolidin-2-ones (*S*,*R*)-*syn*-[D_1]-**3** and (*R*,*S*)-*syn*-[D_3]-**3** gave a 52:48 ratio of (*S*,*R*)-*syn*-[D_1]-**3**: (*R*,*S*)-*syn*-[D_3]-**3**. For (*S*,*R*)-*syn*-[D_1]-**3**; found MNH_4^+, 314.1607; $C_{18}H_{20}DN_2O_3$ requires MNH_4^+, 314.1609, and for (*R*,*S*)-*syn*-[D_3]-**3**; found MNH_4^+, 316.1733; $C_{18}H_{18}D_3N_2O_3$ requires MNH_4^+, 316.1735.

*Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-phenylbutanoate (*rac*)-[D_1]-8 using 4-phenyl-oxazolidin-2-one (R)-1 and 4-phenyl-5,5-dideuterio-oxazolidin-2-one (S)-[D_2]-1*

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.19 mL, 2.5 M in hexane, 0.47 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (33 mg, 0.20 mmol), 4-phenyl-5,5-dideuterio-oxazolidin-2-one (*S*)-[D_2]-**1** (34 mg, 0.20 mmol), and pentafluorophenyl 2-deuterio-2-phenylbutanoate (*rac*)-[D_1]-**8** (0.161 g, 0.48 mmol, [D]:[H] = 96:4), gave an inseparable mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-[D_1]-**11** and (*R*,*S*)-[D_3]-**11** (ratio 98:2: *syn-*:*anti-*). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give inseparable mixture of (2*S*,4*R*)-3-[2-deuterio-2-phenylbutanoyl]-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**11** and (2*R*,4*S*)-3-[2-deuterio-2-phenylbutanoyl]-4-phenyl-5,5-dideuterio-oxazolidin-2-one (*R*,*S*)-*syn*-[D_3]-**11** (97 mg, 78 %; ratio (*S*,*R*)-*syn*-[D_1]-**11**: (*R*,*S*)-*syn*-[D_3]-**11** = 53:47) as a white solid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.47; m.p. 70–72°C; $[\alpha]_D^{20}$ = +2.88 (*c* 3.2, $CHCl_3$); {Ref. 30 (*S*,*R*)-*syn*-**11**; $[\alpha]_D^{20}$ = +77.4 (*c* 4.0, $CHCl_3$)}; ν_{max}(film)cm^{-1} 2401 (C-D), 1781 (OC=O) and 1711 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.22-7.10 (12 H, m, 12 × CH; 2 × Ph^{U+L}), 7.08-7.04 (4 H, m, 4 × CH; Ph^{U+L}), 6.82 (4 H, dt, *J* 7.2 and 1.5, 4 × CH; Ph^{U+L}), 5.39 (1 H, dd, *J* 8.9 and 5.0, CHN^U), 5.39 (1 H, s, CHN^L), 4.55 (1 H, t, *J* 8.9, C*H*$_A$H$_B$O^U), 3.99 (1 H, dd, *J* 8.9 and 5.0, CH$_A$*H*$_B$O^U), 1.97 (2 H, dq, 13.6 and 7.3, C*H*$_A$H$_B$CH$_3^{U+L}$), 1.66 (2 H, dq, 13.6 and 7.3, CH$_A$*H*$_B$CH$_3^{U+L}$) and 0.82 (6 H, t, *J* 7.3, C*H*$_3$CH$_2^{U+L}$); negative isotopic shift at 5.39 ppm was 0.0101 ppm (1.01 Hz at 400 MHz); δ_C(100 MHz; $CDCl_3$) 173.0 (2 C; NC=O^{U+L}), 153.1 (2 C; OC=O^{U+L}), 138.2 (2 × *i*-C; Ph_A), 137.9 (2 × *i*-C; Ph_B), 128.8,[4] 128.6,[4] 128.4,[6] 127.1[2] and 125.6[4] (20 × CH; 2 × Ph^{U+L}), 69.5 (CH_2O^U), 68.8 (1 C, quintet, $^1J_{C,D}$ = 20.6, CD_2O^L), 57.6 (CHN^U), 57.5 (CHN^L), 51.1 (unlabelled Ph*C*HCH$_3$), 50.7 (2 C, t [1:1:1], $^1J_{C,D}$ = 20.6 Hz, Ph*C*DCH$_3^{U+L}$), 26.1 (2 × *C*H$_2$CH$_3^{U+L}$) and 11.9 (2 × CH$_2$*C*H$_3^{U+L}$); negative isotopic shift at 68.8 ppm was 0.6570 ppm (65.70 Hz at 100 MHz), 57.5 ppm was 0.1674 ppm (16.74 Hz at 100 MHz) and 50.7 ppm was 0.3973 ppm (39.73 Hz at 100 MHz). By mass spectrometry, this mixture of oxazolidin-2-ones (*S*,*R*)-*syn*-[D_1]-**11** and (*R*,*S*)-*syn*-[D_3]-**11** gave a 53:47 ratio of (*S*,*R*)-*syn*-[D_1]-**11**:(*R*,*S*)-*syn*-[D_3]-**11**. For (*S*,*R*)-*syn*-[D_1]-**11**; found MNH_4^+, 328.1769; $C_{19}H_{22}DN_2O_3$ requires MNH_4^+, 328.1766, and for (*R*,*S*)-*syn*-[D_3]-**11**; found MNH_4^+, 330.1890; $C_{19}H_{20}D_3N_2O_3$ requires MNH_4^+, 330.1891.

Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (rac)-[D_1]-9 using 4-phenyl-oxazolidin-2-one (R)-1 and 4-phenyl-5,5-dideuterio-oxazolidin-2-one (S)-[D_2]-1

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.24 mL, 2.5 M in hexane, 0.60 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (39 mg, 0.24 mmol), 4-phenyl-5,5-dideuterio-oxazolidin-2-one (*S*)-[D_2]-**1** (40 mg, 0.24 mmol), and pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (*rac*)-[D_1]-**9** (0.20 g, 0.60 mmol, [D]:[H] = 96:4), gave an inseparable mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-[D_1]-**12** and (*R*,*S*)-[D_3]-**12** (ratio 98:2: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give an inseparable mixture of (2*S*,4*R*)-3-[2-deuterio-2-(4-methylphenyl)propionate]-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**12** and (2*R*,4*S*)-3-[2-deuterio-2-(4-methylphenyl)propionate]-4-phenyl-5,5-dideuterio-oxazolidin-2-one (*R*,*S*)-*syn*-[D_3]-**12** (0.109 g, 73%; ratio (*S*,*R*)-*syn*-[D_1]-**12**: (*R*,*S*)-*syn*-[D_3]-**12** = 51:49) as a white solid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.40; m.p. 117–119°C {Ref. 22 unlabelled (*rac*)-**12**; m.p. 102–104°C; Ref. [22] (*S*,*R*)-*syn*-**12**; m.p. 107–109°C}; $[\alpha]_D^{20}$ = +5.6 (*c* 3.2, $CHCl_3$) {Ref. 30 (*S*,*R*)-*syn*-**12**; $[\alpha]_D^{20}$ = +121.6 (*c* 0.6, $CHCl_3$) $\nu_{max}(CHCl_3)cm^{-1}$ 2401 (C-D), 1781 (OC=O) and 1713 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.31-7.21 (6 H, m, 6 × CH; Ph^{U+L}), 7.06 (4 H, br d, *J* 7.9, 4 × CH, Ar^{U+L}), 7.08 (4 H, dt, *J* 7.9 and 2.0, 4 × CH; Ar^{U+L}), 6.96 (4 H, dt, *J* 7.6 and 1.8, 4 × CH; Ph^{U+L}), 5.46 (1 H, dd *J* 8.9 and 5.0, CHN^U), 5.45 (1 H, s, CDN^L), 4.62 (1 H, t, *J* 8.9, $CH_AH_BO^U$), 4.08 (1 H, dd, *J* 8.9 and 5.2, $CH_AH_BO^U$), 2.34 (6 H, s, 2 × CH_3; Ar^{U+L}) and 1.39 (6 H, s, $ArCDCH_3^{U+L}$); negative isotopic shift at 5.45 ppm was 0.01025 ppm (1.25 Hz at 400 MHz); δ_C(100.6 MHz; $CDCl_3$) 173.8 (2 C; $NC=O^{U+L}$), 153.0 (2 C; $OC=O^{U+L}$), 138.2 (2 × *i*-C; Ar^{U+L}), 136.7 (2 × *i*-C; Ar^{U+L}), 136.6 (2 × *i*-C; Ph^{U+L}), 129.0[4] and 127.9[4] (8 × CH; Ar^{U+L}), 128.7[4], 128.3[2] and 125.7[4] (10 × CH; Ph^{U+L}), 69.5 (CH_2O^U), 68.8 (1 C, quintet, $^1J_{C,D}$ = 22.9 Hz, CD_2O^L), 57.7 (CHN^U), 57.5 (CHN^L), 43.3 (unlabelled Ar*C*HCH$_3$), 43.0 (2 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ar*C*DCH_3^{U+L}), 22.5 (2 C; CH_3; Ar^{U+L}) and 18.5 (2 C; ArCD*C*H_3^{U+L}) negative isotopic shifts at 68.8259 was 0.6494 ppm (64.94 Hz at 100 MHz), 57.4969ppm was 0.1680 ppm (16.80 Hz at 100 MHz) and at 43.6039 ppm was 0.3667 ppm (36.67 Hz at 400 MHz). By mass spectrometry, this mixture of oxazolidin-2-ones (*S*,*R*)-*syn*-[D_1]-**12** and (*R*,*S*)-*syn*-[D_3]-**12** gave a 51:49 ratio of (*S*,*R*)-*syn*-[D_1]-**12**:(*R*,*S*)-*syn*-[D_3]-**12**. For (*S*,*R*)-*syn*-[D_1]-**12**; found MNH_4^+, 328.1766; $C_{19}H_{22}DN_2O_3$ requires MNH_4^+, 328.1766, and for (*R*,*S*)-*syn*-[D_3]-**12**; found MNH_4^+, 330.1892; $C_{19}H_{20}D_3N_2O_3$ requires MNH_4^+, 330.1891.

Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (rac)-[D_1]-10 using 4-phenyl-oxazolidin-2-one (R)-1 and 4-phenyl-5,5-dideuterio-oxazolidin-2-one (S)-[D_2]-1

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.13 mL, 2.5 M in hexane, 0.325 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (21 mg, 0.13 mmol), 4-phenyl-5,5-dideuterio-oxazolidin-2-one (*S*)-[D_2]-**1** (21 mg, 0.13 mmol), and pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (*rac*)-[D_1]-**10** (0.119 g, 0.32 mmol, [D]:[H] = 95:5), gave an inseparable mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-**13** and (*R*,*S*)-[D_2]-**13** (ratio 97:3: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give an inseparable mixture of (2*S*,4*R*)-3-[2-deuterio-2-(4-isobutylphenyl)propionate]-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**13** and (2*R*,4*S*)-3-[2-deuterio-2-(4-isobutylphenyl)propionate]-4-phenyl-5,5-dideuterio-oxazolidin-2-one (*R*,*S*)-*syn*-[D_3]-**13** (67 mg, 73%; ratio (*S*,*R*)-*syn*-[D_1]-**13**: (*R*,*S*)-*syn*-[D_3]-**13** = 53:47) as a white solid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1) 0.40; m.p. 69–71°C (Ref. 22 unlabelled (*rac*)-**13**; m.p. 69–71°C; Ref. 22 (*S*,*R*)-*syn*-**13**; m.p. 97–99°C}; $[\alpha]_D^{20}$ = +12.1 (*c* 3.0, $CHCl_3$) {Ref. 30 (*S*,*R*)-*syn*-**13**; $[\alpha]_D^{20}$ = +118.7 (*c* 6.0, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2401 (C-D), 1779 (OC=O) and 1706 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.20-7.08 (6 H, m, 6 × CH; Ph and/or Ar^{U+L}), 6.96-6.91 (8 H, m, 8 × CH, Ph and Ar^{U+L}), 6.83 (4 H, dt, *J* 7.9 and 1.9, 4 × CH; Ar^{U+L}), 5.37 (1 H, dd *J* 9.1 and 5.1, CHN^U), 5.36 (1 H, s, CHN^L), 4.54 (1 H, t, *J* 9.1, $CH_AH_BO^U$), 3.98 (1 H, dd, *J* 9.1 and 5.1, $CH_AH_BO^U$), 2.37 (4 H, dd, *J* 7.4 and 2.2, CH_2^{U+L}), 1.89-1.79 (2 H, appears as a nonet, *J* 6.7, ($CH(CH_3)_2^{U+L}$), 1.31 (6 H, s, $ArCDCH_3^{U+L}$), 0.84 (6 H, d, *J* 6.7, $CH_3^ACH\ ^BCH_3$) and 0.82 (6 H, d, *J* 6.7, $CH_3^ACH^BCH_3^{U+L}$); negative isotopic shift at 5.36 ppm was 0.01025 ppm (1.025 Hz at 400 MHz); δ_C(100.6 MHz; $CDCl_3$) 173.8 (2 C; $NC=O^{U+L}$), 153.1 (2 C; $OC=O^{U+L}$), 140.5 (2 × *i*-C; Ar^{U+L}), 138.2 (2 × *i*-C; Ar^{U+L}), 136.8 (2 × *i*-C; Ph^{U+L}), 129.1[4] and 127.8[4] (8 × CH; Ar^{U+L}), 128.8,[2] 128.4[4] and 125.6[4] (10 × CH; Ph), 69.5 (CH_2O^U), 68.8 (1 C, quintet, $^1J_{C,D}$ = 23.7 Hz, CD_2O^L), 57.6 (CHN^U), 57.5 (CHN^L), 45.1 (2 C; $CH(CH_3)_2^{U+L}$), 43.2 (unlabelled Ar*C*HCH$_3$), 42.8 (2 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ar*C*DCH_3^{U+L}), 30.1 (2 C; CH_2^{U+L}), 22.3 (2 C, s, $CH_3^ACH\ ^BCH_3^{U+L}$), 22.2 (2 C, s, $CH_3^ACH\ ^BCH_3^{U+L}$) and 18.3 (2 C, ArCD*C*H_3^{U+L}); negative isotopic shifts at 68.9 was 0.6341 ppm (63.41 Hz at 100 MHz), 57.4892 ppm was 0.1757 ppm (17.57 Hz at 100 MHz) and at 42.8752 ppm was 0.3667 ppm (36.67 Hz at 400 MHz). By mass spectrometry, this mixture of oxazolidin-2-ones (*S*,*R*)-*syn*-[D_1]-**13** and (*R*,*S*)-*syn*-[D_3]-**13** gave a 53:47 ratio of (*S*,*R*)-*syn*-[D_1]-**13**:(*R*,*S*)-*syn*-[D_3]-**13**. For (*S*,*R*)-*syn*-[D_1]-**13**; found MNH_4^+, 370.2234; $C_{22}H_{28}DN_2O_3$ requires MNH_4^+, 370.2235, and for (*R*,*S*)-*syn*-[D_3]-**13**; found MNH_4^+, 372.2357; $C_{22}H_{26}D_3N_2O_3$ requires MNH_4^+, 372.2361.

Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-phenylpropionate (rac)-[D_1]-1 using a quasi-enantiomeric combination of oxazolidin-2-one (R)-1 and oxazolidin-2-one (S)-14

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.48 mL, 2.5 M in hexane, 1.20 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (79 mg, 0.49 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**14** (0.157 g, 0.49 mmol) and pentafluorophenyl 2-deuterio-2-phenylpropionate (*rac*)-[D_1]-**2** (0.38 g, 1.21 mmol, [D]:[H] = 97:3), gave a mixture of two diaster-

eoisomeric oxazolidin-2-ones (*S,R*)-*syn*-**3** (ratio 94:6: *syn*-:*anti*-) and (*R,S*)-*syn*-**15** (ratio 97:3: *syn*-:*anti*-) in a ratio of 55:45. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give the (2*S,*4*R*)-3-(2-deuterio-2-phenylpropionyl)-4-phenyl-oxazolidin-2-one (*S,R*)-*syn*-[D_1]-**3** (96 mg, 66%) as a white solid; m.p. 121–123°C (Ref. 31 unlabelled (*R,S*)-*syn*-**3**; m.p. 124–126°C); R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.42; $\nu_{max}(CHCl_3)cm^{-1}$ 2397 (C-D), 1780 (OC=O) and 1701 (NC=O); $[\alpha]_D^{20}$ = +71.6 (*c* 6.4, $CHCl_3$) {unlabelled (*S,R*)-*syn*-**3**; $[\alpha]_D^{20}$ = +92.5 (*c* 4.9, $CHCl_3$)} {Ref. 30 unlabelled (*S,R*)-*syn*-**3**; $[\alpha]_D^{20}$ = +92.5 (*c* 4.9, $CHCl_3$)}; δ_H(400 MHz; $CDCl_3$) 7.29-7.21 (6 H, m, 6 × CH; 2 × Ph), 7.14-7.10 (2 H, m, 2 × CH; Ph), 6.96-6.92 (2 H, m, 2 × CH; Ph), 5.45 (1 H, dd *J* 9.0 and 5.1, CHN), 4.63 (1 H, t, *J* 9.0, C$H_A$$H_B$O), 4.08 (1 H, dd, *J* 9.0 and 5.1, C$H_A$$H_B$O) and 1.40 (3 H, s, PhCDCH_3); δ_C (100 MHz; $CDCl_3$) 174.1 (NC=O), 153.6 (OC=O), 140.2 and 138.7 (2 × *i*-C; 2 × Ph), 129.3,[2] 128.9,[3] 128.6,[2] 127.5[1] and 126.3[2] (10 × CH; 2 × Ph), 70.0 (CH_2O), 58.2 (CHN), 44.2 (unlabelled Ph*C*HCH$_3$), 43.8 (1 C, t [1:1:1], $^1J_{C,D}$ 20.3, Ph*C*DCH$_3$) and 19.0 (PhCD*C*H$_3$) (Found MH^+, 297.1346; $C_{18}H_{17}DNO_3$ requires MH^+, 297.1344); negative isotopic shift at 43.8 ppm (Ph*C*D) was 0.366 ppm (36.6 Hz at 100.6 MHz) and (2*R,*4*S*)-3-(2-deuterio-2-phenylpropionyl)-4,5,5-triphenyl-oxazolidin-2-one (*R,S*)-*syn*-[D_1]-**15** (0.12 g, 54 %) as a white powder; R_F [light petroleum ether (b.p. 40-60°C):diethyl ether (1:1)] 0.58; m.p. 156-158°C {Ref. 32 unlabelled m.p. 154-156°C}; $[\alpha]_D^{20}$ = −226.4 (*c* 4.2, $CHCl_3$) {Ref. 32 unlabelled (*R,S*)-*syn*-**15**; $[\alpha]_D^{20}$ = −255.1 (*c* 3.4, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2389 (C-D), 1779 (OC=O) and 1704 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.66 (2 H, br d, *J* 7.7, 2 × CH; Ph), 7.50-7.36 (4 H, m, 4 × CH; Ph), 7.18-7.11 (2 H, m, 2 × CH; Ph), 7.11-7.07 (2 H, m, 2 × CH; Ph), 7.01-6.89 (8 H, m, 8 × CH; Ph), 6.67 (2 H, br d, *J* 7.7, 2 × CH; Ph), 6.30 (1 H, s, CHN) and 1.37 (3 H, s, PhCDCH_3); δ_C(100 MHz; $CDCl_3$) 173.2 (NC=O), 152.0 (OC=O), 141.8, 137.9 and 134.9 (3 × *i*-C; 3 × Ph – oxazolidin-2-one), 139.4 (*i*-C; Ph), 128.9,[2] 128.8,[1] 128.4,[2] 128.2,[2] 127.9,[4] 127.5,[2] 127.4,[1] 127.3,[1] 126.9,[1] 126.1[2] and 126.0[2] (20 × CH; 4 × Ph), 88.5 (CPh_2O), 65.9 (CHN), 43.9 (unlabelled Ph*C*HCH$_3$), 43.5 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.6 Hz, PhCD*C*H$_3$) (Found MH^+, 449.1968; $C_{30}H_{25}DNO_3^+$ requires 449.1970; and found MNH_4^+, 465.2155; $C_{30}H_{28}DN_2O_3^+$ requires MNH_4^+, 465.2157); negative isotopic shift at 43.5 ppm (Ph*C*D) was 0.3743 ppm (37.4 Hz at 100.6 MHz).

*Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-phenylbutanoate (***rac***)-[D_1]-8 using a quasi-enantiomeric combination of oxazolidin-2-one (***R***)-1 and oxazolidin-2-one (***S***)-14*

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.36 mL, 2.5 M in hexane, 0.90 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (62 mg, 0.38 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**14** (0.12 g, 0.38 mmol) and pentafluorophenyl 2-deuterio-2-phenylbutanoate (*rac*)-[D_1]-**8** (0.30 g, 0.91 mmol, [D]:[H] = 96:4), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-*syn*-**11** (ratio 99:1: *syn*-:*anti*-) and (*R,S*)-*syn*-**16** (ratio 99:1: *syn*-:*anti*-) in a ratio of 52:48. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give (2*S,*4*R*)-3-(2-deuterio-2-phenylbutanoyl)-4-phenyl-oxazolidin-2-one (*S,R*)-*syn*-[D_1]-**11** (81 mg, 69%) as a white solid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.29; m.p. 80–82°C {Ref. 32 unlabelled (*S,R*)-*syn*-**11**; m.p. 82–84°C}; $[\alpha]_D^{20}$ = +53.2 (*c* 4.6, $CHCl_3$) {Ref. 30 (*S,R*)-*syn*-**11**; $[\alpha]_D^{20}$ = +77.4 (*c* 4.0, $CHCl_3$); Ref. 22 (*R,S*)-*syn*-[D_2]-**11**; $[\alpha]_D^{20}$ = −55.2 (*c* 4.6, $CHCl_3$)}; $\nu_{max}(CH_2Cl_2)cm^{-1}$ 2399 (C-D), 1772 (OC=O) and 1700 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.19-7.09 (6 H, m, 6 × CH; Ph), 7.04-7.02 (2 H, m, 2 × CH; Ph), 6.81 (2 H, dt, *J* 8.4 and 1.8, 2 × CH; Ph), 5.38 (1 H, dd, *J* 8.9 and 5.0, CHN), 4.53 (1 H, t, *J* 8.9, C$H_A$$H_B$O), 3.98 (1 H, dd, *J* 8.9 and 5.0, C$H_A$$H_B$O), 1.95 (1 H, dq, *J* 13.5 and 7.4, C$H_A$$H_BCH_3$), 1.63 (1 H, dq, *J* 13.5 and 7.4, C$H_A$$H_BCH_3$) and 0.79 (3 H, t, *J* 7.4, CH_3CH$_2$); δ_C(100 MHz; $CDCl_3$) 173.0 (NC=O), 153.1 (OC=O), 138.2 and 138.0 (2 × *i*-C; 2 × Ph), 128.8,[2] 128.6,[2] 128.3,[3] 127.1[1] and 125.6[2] (10 × CH; 2 × Ph), 69.4 (CH_2O), 57.7 (CHN), 50.7 (1 C, t [1:1:1], $^1J_{C,D}$ = 21.3 Hz, PhCDEt), 26.2 (*C*H_2CH$_3$) and 11.9 (CH_2*C*H$_3$) (Found MNH_4^+, 328.1766; $C_{19}H_{22}DN_2O_3$ requires MNH_4^+, 328.1766); negative isotopic shift at 50.7 ppm (Ph*C*D) was 0.3973 ppm (39.7 Hz at 100.6 MHz); and (2*R,*4*S*)-3-(2-deuterio-2-phenylbutanoyl)-4,5,5-triphenyl-oxazolidin-2-one (*R,S*)-*syn*-[D_1]-**16** (0.11 g, 63 %) as a white powder; R_F [light petroleum ether (b.p. 40-60°C): diethyl ether (1:1)] 0.52; m.p. 121-122°C {Ref. 32 unlabelled (*R,S*)-*syn*-**16**; m.p. 150–153°C}; $[\alpha]_D^{20}$ = −231.4 (*c* 5.6, $CHCl_3$) {Ref. 32 unlabelled (*R,S*)-*syn*-**16**; $[\alpha]_D^{20}$ = −195.2 (*c* 3.4, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2399 (C-D), 1779 (OC=O) and 1707 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.55 (2 H, br d, *J* 7.5, 2 × CH; Ph), 7.39-7.29 (3 H, m, 3 × CH; Ph), 7.16-7.07 (5 H, m, 5 × CH; 2 × Ph), 6.90-6.80 (8 H, m, 8 × CH; 3 × Ph), 6.57-6.55 (2 H, d, *J* 7.5, 2 × CH; Ph), 6.20 (1 H, s, CHN), 1.90 (1 H, dq, *J* 13.5 and 7.3, C$H_A$$H_BCH_3$), 1.63 (1 H, dq, *J* 13.5 and 7.3, C$H_A$$H_BCH_3$) and 0.68 (3 H, t, *J* 7.3, CH_3CH$_2$); δ_C(100 MHz; $CDCl_3$) 172.8 (NC=O), 152.1 (OC=O), 141.7, 137.9 and 135.0 (3 × *i*-C; 3 × Ph), 137.6 (*i*-C; PhCHEt), 128.9,[3] 128.8,[2] 128.3,[2] 127.9,[2] 127.8,[1] 127.5,[2] 127.4,[1] 127.3,[2] 127.1,[1] 126.3[2] and 126.2[2] (20 × CH; 4 × Ph), 88.6 (CPh_2O), 65.9 (CHN), 50.7 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.9 Hz, Ar*C*DEt), 26.6 (*C*H_2CH$_3$) and 11.8 (CH_2*C*H$_3$) (Found MNH_4^+, 480.2386; $C_{31}H_{30}DN_2O_3$ requires MNH_4^+, 480.2392); negative isotopic shift at 50.7 ppm (Ph*C*D) was 0.4125 ppm (41.2 Hz at 100.6 MHz).

*Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (***rac***)-[D_1]-9 using a quasi-enantiomeric combination of oxazolidin-2-one (***R***)-1 and oxazolidin-2-one (***S***)-14*

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.37 mL, 2.5 M in hexane, 0.92 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (62 mg, 0.38 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**14** (0.12 g, 0.38 mmol) and pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (*rac*)-[D_1]-**9** (0.307 g, 0.92 mmol, [D]:[H] = 96:4), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-*syn*-**12** (ratio 95:5: *syn*-:*anti*-) and (*R,S*)-*syn*-**17** (ratio 99:1: *syn*-:*anti*-) in a

ratio of 53:47. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give (2*S*,4*R*)-3-[2-deuterio-2-(4-methylphenyl)propionyl]-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**12** (75 mg, 64%) as a white solid; R_F [light petroleum ether (b.p. 40-60 °C):diethyl ether (1:1)] 0.30; m.p. 110–112°C {Ref. 32 unlabelled (*S*,*R*)-*syn*-**12**; m.p. 105-110 °C; for unlabelled (*R*,*S*)-*syn*-**12**; m.p. 105–110°C}; $\nu_{max}(CHCl_3)cm^{-1}$ 2397 (C-D), 1772 (OC=O) and 1700 (NC=O); $[\alpha]_D^{20}$ = +90.4 (*c* 4.6, $CHCl_3$) {Ref. 30 (*S*,*R*)-*syn*-**12**; $[\alpha]_D^{20}$ = +121.6 (*c* 0.6, $CHCl_3$)} {for unlabelled (*R*,*S*)-*syn*-**12**; $[\alpha]_D^{20}$ = −116.5 (*c* 0.8, $CHCl_3$)}; δ_H (400 MHz, $CDCl_3$) 7.28-7.21 (3 H, m, 3 × CH; Ph), 7.06 (2 H, br d, *J* 8.1, 2 × CH; Ar), 7.01 (2 H, dt, *J* 8.1 and 2.2, 2 × CH; Ar), 6.96 (2 H, dt, *J* 7.7 and 1.8, 2 × CH; Ph), 5.47 (1 H, dd, *J* 9.1 and 5.1, CHN), 4.64 (1 H, t, *J* 9.1, C*H*$_A$H$_B$O), 4.06 (1 H, dd, *J* 9.1 and 5.1, CH$_A$*H*$_B$O), 2.34 (3 H, s, CH_3; Ar) and 1.39 (3 H, s, ArCDC*H*$_3$); δ_C (100 MHz, $CDCl_3$) 173.5 (NC=O), 152.7 (OC=O), 137.9 (*i*-CMe; Ar), 136.4 (*i*-C; Ar), 136.2 (*i*-C; Ph), 129.8,[2] 128.4,[2] 128.0,[1] 127.6[2] and 125.5[2] (9 × CH; Ph and Ar), 69.1 (CH_2O), 57.3 (CHN), 42.6 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.2 Hz, Ar*C*DCH$_3$), 20.7 (CH_3; Ar) and 18.1 (ArCD*C*H$_3$) (Found MNH_4^+, 328.1768; $C_{19}H_{22}DN_2O_3^+$ requires MNH_4^+, 328.1766); negative isotopic shift at 42.6 ppm (Ar*C*D) was 0.3666 ppm (36.6 Hz at 100.6 MHz); and (2*R*,4*S*)-3-[2-deuterio-2-(4-methylphenyl)-propionyl]-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-[D_1]-**17** (0.101 g, 57 %) as a white powder; R_F [light petroleum ether (b.p. 40-60°C): diethyl ether (1:1)] 0.57; m.p. 87–89°C {Ref. 32 unlabelled (*R*,*S*)-*syn*-**17**; m.p. 119-121°C}; $[\alpha]_D^{20}$ = −213.2 (*c* 5.4, $CHCl_3$) {Ref. 32 unlabelled (*R*,*S*)-*syn*-**17**; $[\alpha]_D^{20}$ = −258.6 (*c* 2.4, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2399 (C-D), 1780 (OC=O) and 1703 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.65 (2 H, br d, *J* 7.5, 2 × CH; Ph), 7.48-7.38 (3 H, m, 3 × CH; Ph), 7.04-6.89 (12 H, m, 12 × CH; Ph and Ar), 6.69 (2 H, br d, *J* 7.5, Ph), 6.29 (1 H, s, CHN), 2.32 (3 H, s, CH_3Ar) and 1.36 (3 H, s, ArCDC*H*$_3$); δ_C(100 MHz; $CDCl_3$) 173.5 (NC=O), 151.9 (OC=O), 141.8, 138.0 and 135.0 (3 × *i*-C; 3 × Ph), 136.7 and 136.5 (2 × *i*-C; Ar), 129.1,[2] 128.9,[2] 128.8,[1] 128.2,[2] 127.8,[3] 127.5,[2] 127.4,[2] 127.3,[1] 126.2[2] and 126.1[2] (19 × CH; 3 × Ph and Ar), 88.5 (CPh_2O), 65.9 (CHN), 44.6 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.6 Hz, Ar*C*DCH$_3$), 21.0 (CH_3Ar) and 19.0 (ArCH*C*H$_3$) (Found MH^+, 463.2124; $C_{31}H_{27}DNO_3$ requires MH^+, 463.2126); negative isotopic shift at 44.6 ppm (Ar*C*D) was 0.3629 ppm (36.3 Hz at 100.6 MHz).

Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (rac)-[D_1]-**10** *using a* quasi-*enantiomeric combination of oxazolidin-2-ones* (R)-**1** *and oxazolidin-2-one* (S)-**14**

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.37 mL, 2.5 M in hexane, 0.925 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (64 mg, 0.39 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**14** (0.122 g, 0.39 mmol) and pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (*rac*)-[D_1]-**10** (0.346 g, 0.92 mmol, [D]:[H] = 95:5), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-*syn*-**13** (ratio 95:5: *syn*-:*anti*-) and (*R*,*S*)-*syn*-**18** (ratio 97:3: *syn*-:*anti*-) in a ratio of 52:48. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give (2*S*,4*R*)-3-[2-deuterio-2-(4-isobutylphenyl)propionyl]-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**13** (95 mg, 69%) as a white solid; m.p. 78–80°C (Ref. [32] unlabelled (*S*,*R*)-*syn*-**13**; m.p. 86–88°C); R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.37; $[\alpha]_D^{25}$ = +98.9 (*c* 4.6, $CHCl_3$) {Ref. 32 (*S*,*R*)-*syn*-**13**; $[\alpha]_D^{25}$ = +118.7 (*c* 6.0, $CHCl_3$)} {for unlabelled (*R*,*S*)-*syn*-**13**; lit. $[\alpha]_D^{25}$ = −114.6 (*c* 4.2, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2399 (C-D), 1780 (OC=O) and 1705 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.15-7.07 (3 H, m, 3 × CH; Ph), 7.00 (4 H, m, 4 × CH, Ph and Ar), 6.80 (2 H, dt, *J* 7.9 and 1.9, 2 × CH; Ar), 5.36 (1 H, dd *J* 9.0 and 5.2, CHN), 4.53 (1 H, t, *J* 9.0, C*H*$_A$H$_B$O), 3.97 (1 H, dd, *J* 9.0 and 5.2, CH$_A$*H*$_B$O), 2.43 (2 H, dd, *J* 7.3 and 2.2, CH_2Ar), 1.82-1.74 (1 H, m, (C*H*$(CH_3)_2$), 1.32 (3 H, s, ArCDC*H*$_3$), 0.82 (3 H, d, *J* 6.6, C*H*$_3^A$CHCH$_3^B$) and 0.80 (3 H, d, *J* 6.6, CH$_3^A$CHC*H*$_3^B$); δ_C(100.6 MHz; $CDCl_3$) 173.8 (NC=O), 153.1 (OC=O), 140.5 (*i*-C; Ar), 138.2 (*i*-C; Ar), 136.8 (*i*-C; Ph), 129.3[2] and 128.7[2] (4 × CH; Ar), 128.4,[1] 127.8[2] and 125.7[2] (5 × CH; Ph), 69.5 (CH_2O), 57.6 (CHN), 44.9 (*C*H$(CH_3)_2$), 42.8 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.6 Hz, Ar*C*DCH$_3$), 30.1 (*C*H$_2$Ar), 22.5[2] (*C*H$_3)_2$CH) and 18.4 (ArCD*C*H$_3$) (Found MNH_4^+, 370.2232; $C_{22}H_{28}DN_2O_3$ requires MNH_4^+, 370.2235); negative isotopic shift at 42.8 ppm (Ar*C*D) was 0.3665 ppm (36.6 Hz at 100.6 MHz); and (2*R*,4*S*)-3-[2-deuterio-2-(4-isobutylphenyl)propionyl]-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-[D_1]-**18** (0.126 g, 64 %) as a white powder; R_F [light petroleum ether (b.p. 40-60°C): diethyl ether (1:1)] 0.63; m.p. 57–59°C {Ref. 32 unlabelled m.p. = 62-64°C}; $[\alpha]_D^{20}$ = −228.7 (*c* 5.0, $CHCl_3$) {Ref. 32 unlabelled (*R*,*S*)-*syn*-**18**; $[\alpha]_D^{20}$ = −306.7 (*c* 4.4, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2399 (C-D), 1780 (OC=O) and 1704 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.66 (2 H, br d, *J* 7.3, 2 × CH; Ph), 7.49-7.37 (3 H, m, 3 × CH; Ph), 7.28 (2 H, br d, *J* 8.1, 2 × CH; Ar), 7.16 (2 H, br d, *J* 8.1, 2 × CH; Ar), 7.07-6.85 (8 H, m, 8 × CH; 3 × Ph), 6.66 (2 H, br d, *J* 7.3, 2 × CH; Ph), 6.29 (1 H, s, CHN), 2.44 (2 H, dd, *J* 7.2 and 1.6, CH_2Ar), 1.91-1.80 (1 H, appears as a septet, *J* 6.8, $(CH_3)_2$C*H*), 1.36 (3 H, s, ArCDC*H*$_3$), 0.92 (3 H, d, *J* 6.6, C*H*$_3^A$CHCH$_3^B$) and 0.91 (3 H, d, *J* 6.6, CH$_3^A$CHC*H*$_3^B$); δ_C(100 MHz; $CDCl_3$) 173.5 (NC=O), 152.1 (OC=O), 141.8, 138.1 and 135.0 (3 × *i*-C; 3 × Ph), 140.5 (*i*-*C*CH$_2$; Ar), 136.6 (*i*-C; Ar), 129.2,[2] 128.9,[3] 128.9,[1] 128.1,[2] 127.9,[1] 127.9,[2] 127.6,[2] 127.4,[1] 127.3,[1] 126.3[2] and 126.2[2] (19 × CH; 19 × Ph and Ar), 88.6 (CPh_2O), 66.0 (CHN), 45.0 $(CH_3)_2$*C*H), 43.1 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.1 Hz, Ar*C*DCH$_3$), 30.2 (CH_2Ar), 22.5 (*C*H$_3^A$CHCH$_3^B$), 22.4 (CH$_3^A$CH*C*H$_3^B$) and 18.9 (ArCD*C*H$_3$) (Found MNH_4^+, 522.2859; $C_{34}H_{36}DN_2O_3$ requires MNH_4^+, 522.2861); negative isotopic shift at 43.1 ppm (Ar*C*D) was 0.3781 ppm (37.8 Hz at 100.6 MHz).

Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-phenylpropionate (rac)-[D_1]-**2** *using 4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one* (R)-**19** *and 4-phenyl-oxazolidin-2-one* (S)-**1**

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.38 mL, 2.5 M in hexane, 0.95 mmol), 4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*R*)-**19** (0.117 g, 0.40 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**1** (65 mg, 0.40 mmol), and pentafluorophenyl 2-deuterio-2-phenylpropio-

nate (*rac*)-[D_1]-**2** (0.303 g, 0.95 mmol, [D]:[H] = 97:3), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-*syn*-**20** (ratio 97:3: *syn-*:*anti-*) and (*R,S*)-*syn*-**2** (ratio 97:3: *syn-*:*anti-*) in a ratio of 53:47. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give (2*S,*4*R*)-3-[2-deuterio-2-phenylpropionyl]-4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*S,R*)-*syn*-[D_1]-**20** (0.133 g, 78%) as a white solid; R_F [light petroleum ether (b.p. 40–60°C): diethyl ether (1:1)] 0.50; m.p. 80–81°C (Ref. 30 for unlabelled (*S,R*)-*syn*-**20**; m.p. 96–98°C); $[\alpha]_D^{20}$ = +97.2 (*c* 5.0, $CHCl_3$); {Ref. 30 (*R,S*)-**20**; $[\alpha]_D^{20}$ = −95.2 (*c* 2.0, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2400 (C-D), 1776 (OC=O) and 1706 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.19-7.15 (3 H, m, 3 × CH; Ph), 7.05-7.00 (2 H, m, 2 × CH; Ph), 6.87 (2 H, dt, *J* 8.4 and 2.4, 2 × CH; Ar), 6.63 (2 H, dt, *J* 8.4 and 2.4, 2 × CH; Ar), 5.35 (1 H, dd, *J* 9.0 and 5.0, CHN), 4.54 (1 H, t, *J* 9.0, CH_AH_BO), 4.04 (1 H, dd, *J* 9.0 and 5.0, CH_AH_BO), 1.34 (3H, s, PhCDCH_3), 0.94 (9 H, s, 3 × CH_3C; *t*-Bu) and 0.14 (6 H, s, 2 × CH_3Si); δ_C(100 MHz; $CDCl_3$) 173.6 (NC=O), 155.7 (*i*-C; Ar), 153.0 (OC=O), 139.8 (*i*-C; Ph), 130.9 (*i*-C; Ar), 128.4[2], 128.0[2] and 127.0[1] (5 × CH; Ph), 127.3[2] and 120.2[2] (4 × CH; Ar), 69.7 (CH_2O), 57.3 (CHN), 43.8 (unlabelled Ph*C*HCH$_3$), 43.4 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ph*C*DCH$_3$), 25.6[3] (3 × *C*H$_3$C; *t*-Bu), 18.4 (PhCH*C*H$_3$), 18.1 (CH$_3$*C*; *t*-Bu), −4.5 (*C*H$_3^A$SiCH$_3^B$) and −4.5 (CH$_3^A$Si*C*H$_3^B$); negative isotopic shift at 43.4405 ppm was 0.3367 ppm (33.67 Hz at 100 MHz) (Found MH^+, 427.2157; $C_{24}H_{31}DNO_4Si$ requires MH^+, 427.2158); and (2*R,*4*S*)-3-[2-deuterio-2-phenylpropionyl]-4-phenyl-oxazolidin-2-one (*R,S*)-*syn*-[D_1]-**3** (82 mg, 69 %) as a white solid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.33; m.p. 121–123°C (Ref. 31 (*R,S*)-*syn*-**3**; m.p. 124–126°C); $[\alpha]_D^{23}$ = −79.1 (*c* 5.6, $CHCl_3$) {for unlabelled (*R,S*)-*syn*-**3**; $[\alpha]_D^{23}$ = −91.9 (*c* 4.9, $CHCl_3$); {Ref. 30 unlabelled (*S,R*)-*syn*-**3**, $[\alpha]_D^{20}$ = +92.5 (*c* 4.9, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2401 (C-D), 1781 (OC=O) and 1713 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.20-7.11 (6 H, m, 6 × CH; 2 × Ph), 7.04-7.00 (2 H, m, 2 × CH; Ph), 6.86-6.82 (2 H, m, 2 × CH; Ph), 5.37 (1 H, dd *J* 9.0 and 5.1, CHN), 4.53 (1 H, t, *J* 9.0, CH_AH_BO), 3.99 (1 H, dd, *J* 9.0 and 5.1, CH_AH_BO) and 1.31 (3 H, s, PhCDCH_3); δ_C(100 MHz; $CDCl_3$) 173.6 (C=O), 153.0 (C=O), 139.7 and 138.2 (2 × *i*-C; 2 × Ph), 128.8,[2] 128.6,[3] 128.0,[2] 127.0[1] and 125.8[2] (10 × CH; 2 × Ph), 69.5 (CH_2O), 57.7 (NCH), 43.7 (unlabelled Ph*C*HCH$_3$), 43.4 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.6 Hz, Ph*C*DCH$_3$) and 18.4 (PhCH*C*H$_3$); negative isotopic shift at 43.3871 ppm was 0.3666 ppm (36.66 Hz at 100 MHz) (Found MNH_4^+, 314.1608; $C_{18}H_{20}DN_2O_3$ requires MNH_4^+, 314.1609).

*Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-phenylbutanoate (*rac*)-[D_1]-8 using 4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*R*)-19 and 4-phenyl-oxazolidin-2-one (*S*)-1*

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.37 mL, 2.5 M in hexane, 0.92 mmol), 4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*R*)-**19** (0.11 g, 0.38 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**1** (62 mg, 0.38 mmol), and pentafluorophenyl 2-deuterio-2-phenylbutanoate (*rac*)-[D_1]-**8** (0.30 g, 0.91 mmol, [D]:[H] = 96:4), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-*syn*-**21** (ratio 99:1: *syn-*:*anti-*) and (*R,S*)-*syn*-**11** (ratio 99:1: *syn-*:*anti-*) in a ratio of 53:47. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give (2*S,*4*R*)-3-[2-deuterio-2-phenylbutanoyl]-4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*S,R*)-*syn*-[D_1]-**21** (0.11 g, 67%) as a colourless oil; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.57; $[\alpha]_D^{20}$ = +87.6 (*c* 4.0, $CHCl_3$) {Ref. 30 unlabelled (*R,S*)-*syn*-**21**; $[\alpha]_D^{20}$ = −89.4 (*c* 4.4, $CHCl_3$)}; $\nu_{max}(CH_2Cl_2)cm^{-1}$ 2401 (C-D), 1778 (OC=O) and 1702 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.19-7.16 (3 H, m, 3 × CH; Ph), 7.07-7.04 (2 H, m, 2 × CH; Ph), 6.75 (2 H, dt, *J* 8.5 and 2.5, 2 × CH; Ar), 6.61 (2 H, dt, *J* 8.5 and 2.5, 2 × CH; Ar), 5.37 (1 H, dd, *J* 9.0 and 5.0, CHN), 4.56 (1 H, t, *J* 9.0, CH_AH_BO), 4.03 (1H, dd, *J* 9.0 and 5.0, CH_AH_BO), 2.00 (1 H, dq, *J* 13.8 and 7.3, CH_AH_BCH$_3$), 1.66 (1 H, dq, *J* 13.8 and 7.3, CH_AH_BCH$_3$), 0.94 (9 H, s, 3 × CH_3; *t*-Bu), 0.83 (3 H, t, *J* 7.3, CH_3CH$_2$), 0.14 (6 H, s, 2 × SiCH$_3$); δ_C (100 MHz; $CDCl_3$) 173.0 (NC=O), 155.7 (OC=O), 153.2 (*i*-CO; Ar), 138.1 (*i*-C; Ph) 130.9 (*i*-C; Ar), 128.6,[2] 128.4,[2] 127.1,[2] 127.0[1] and 120.2[2] (9 × CH; Ph and Ar), 69.6 (CH_2O), 57.3 (CHN), 51.1 (unlabelled PhCH), 50.7 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, PhCD), 26.2 (*C*H$_2$CH$_3$), 25.6[3] (3 × *C*H$_3$C; *t*-Bu), 18.1 (CH$_3$*C*; *t*-Bu), 11.9 (CH$_2$*C*H$_3$) and −4.5[2] (2 × SiCH$_3$); negative isotopic shift at 50.7742 ppm was 0.3972 ppm (39.72 Hz at 100 MHz) (Found MH^+, 441.2312; $C_{25}H_{33}DNO_4Si$ requires MH^+, 441.2314); and (2*R,*4*S*)-3-[2-deuterio-2-phenylbutanoyl]-4-phenyl-oxazolidin-2-one (*R,S*)-*syn*-[D_1]-**11** (80 mg, 68 %) as a white solid; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.43; m.p. 80–82°C (Ref. 32 unlabelled (*S,R*)-*syn*-**11**; m.p. 82–84°C); $[\alpha]_D^{20}$ = −59.4 (*c* 3.2, $CHCl_3$); {Ref. 30 (*S,R*)-*syn*-**11**; $[\alpha]_D^{20}$ = +77.4 (*c* 4.0, $CHCl_3$); Ref. 22 (*R,S*)-*syn*-[D_2]-**11**; $[\alpha]_D^{20}$ = −54.2 (*c* 4.6, $CHCl_3$)}; $\nu_{max}(CH_2Cl_2)cm^{-1}$ 2401 (C-D), 1782 (OC=O) and 1708 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.18-7.08 (6 H, m, 6 × CH; Ph), 7.06-7.01 (2 H, m, 2 × CH; Ph), 6.82-6.78 (2 H, m, 2 × CH; Ph), 5.38 (1 H, dd, *J* 8.9 and 5.0, CHN), 4.57 (1 H, t, *J* 8.9, CH_AH_BO), 3.98 (1 H, dd, *J* 8.9 and 5.0, CH_AH_BO), 2.00-1.90 (1 H, dq, *J* 13.5 and 7.3, CH_AH_BCH$_3$), 1.68-1.58 (1 H, dq, *J* 13.5 and 7.3, CH_AH_BCH$_3$) and 0.79 (3 H, t, *J* 7.5, CH_3CH$_2$); δ_C(100 MHz; $CDCl_3$) 173.1 (NC=O), 153.1 (OC=O), 138.2 and 137.9 (2 × *i*-C; 2 × Ph), 128.8,[2] 128.7,[2] 128.4,[1] 128.3,[2] 127.1,[1] and 125.6[2] (10 × CH; 2 × Ph), 69.4 (CH_2O), 57.7 (CHN), 51.1 (unlabelled Ph*C*H), 50.7 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ph*C*D), 26.1 (*C*H$_2$CH$_3$) and 11.9 (CH$_2$*C*H$_3$); negative isotopic shift at 50.7055 ppm was 0.4049 ppm (40.49 Hz at 100 MHz) (Found MNH_4^+, 328.1765; $C_{19}H_{22}DN_2O_3$ requires MNH_4^+, 328.1766).

*Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (*rac*)-[D_1]-9 using 4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*R*)-19 and 4-phenyl-oxazolidin-2-one (*S*)-1*

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.53 mL, 2.5 M in hexane, 1.325 mmol), 4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*R*)-**19** (0.162 g,

0.55 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**1** (90 mg, 0.55 mmol), and pentafluorophenyl 2-deuterio-2-(4-methylphenyl)propionate (*rac*)-[D_1]-**9** (0.438 g, 1.32 mmol, [D]:[H] = 96:4), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-*syn*-[D_1]-**22** (ratio 95:5: *syn*-:*anti*-) and (*R,S*)-*syn*-[D_1]-**12** (ratio 96:4: *syn*-:*anti*-) in a ratio of 53:47. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give (2*S,4R*)-3-[2-deuterio-2-(4-methylphenyl)propionate]-4-[4-(*tert*-butyldimethylsilyloxy)-phenyl]-oxazolidin-2-one (*S,R*)-*syn*-[D_1]-**22** (0.172 g, 71 %) as a colourless oil; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.53; $[\alpha]_D^{20}$ = +121.1 (*c* 4.2, $CHCl_3$) {Ref. 30 (*R,S*)-*syn*-**22**; $[\alpha]_D^{20}$ = −120.3 (*c* 6.0, $CHCl_3$)}; $\nu_{max}(CH_2Cl_2)cm^{-1}$ 2401 (C-D), 1777 (OC=O) and 1702 (NC=O); δ_H(400 MHz; $CDCl_3$) 6.95 (2 H, br d, *J* 8.0, 2 × CH; Ar_A), 6.91 (2 H, dt, *J* 8.0 and 1.8, 2 × CH; Ar_A), 6.78 (2 H, dt, *J* 8.5 and 2.5, 2 × CH; Ar_B), 6.62 (2 H, dt, *J* 8.5 and 2.5, 2 × CH; Ar_B), 5.35 (1 H, dd, *J* 9.0 and 5.0, CHN), 4.56 (1 H, t, *J* 9.0, CH_AH$_B$O), 4.05 (1H, dd, *J* 9.0 and 5.0, CH$_A$$H_B$O), 2.27 (3 H, s, CH_3; Ar), 1.32 (3 H, s, ArCDCH_3), 0.93 (9 H, s, 3 × CH_3; *t*-Bu) and 0.15 (6 H, s, 2 × $SiCH_3$); δ_C (100 MHz; $CDCl_3$) 173.8 (NC=O), 155.8 (OC=O), 153.1 (*i*-CO; Ar_B), 136.9, 136.6 and 130.9 (3 × *i*-C; Ar_A and Ar_B), 129.1, 128.0, 127.3 and 120.2 (4 × CH; Ar_A and Ar_B), 69.7 (CH_2O), 57.3 (CHN), 43.4 (unlabelled Ar*C*HCH$_3$), 43.0 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ar*C*DCH$_3$), 25.6[3] (3 × CH_3; *t*-Bu), 21.0 (CH_3; Ar), 18.5 (ArCD*C*H$_3$), 18.2 (CH_3*C*; *t*-Bu) and −4.5[2] (2 × $SiCH_3$); negative isotopic shift at 43.0433 ppm was 0.3667 ppm (36.67 Hz at 100 MHz) (Found MH^+, 441.2315; $C_{25}H_{33}DNO_4Si$ requires MH^+, 441.2314); and (2*R,4S*)-3-[2-deuterio-2-(4-methylphenyl)propionyl]-4-phenyl-oxazolidin-2-one (*R,S*)-*syn*-[D_1]-**12** (0.129 g, 76 %) as a white solid; R_F [light petroleum ether (b.p. 40-60 °C):diethyl ether (1:1)] 0.31; m.p. 118–120°C; {Ref. [32] (*R,S*)-*syn*-**12**; m.p. 105-110°C}; ν_{max} ($CHCl_3$) cm^{-1} 2400 (C-D), 1781 (OC=O) and 1706 (NC=O); $[\alpha]_D^{20}$ = −101.4 (*c* 2.6, $CHCl_3$) {Ref. 32 (*S,R*)-*syn*-**12**; $[\alpha]_D^{20}$ = +121.6 (*c* 0.6, $CHCl_3$)}; δ_H (400 MHz, $CDCl_3$) 7.22-7.12 (3 H, m, 3 × CH; Ph), 6.97 (2 H, br d, *J* 8.2, 2 × CH; Ar), 6.91 (2 H, dt, *J* 8.2 and 2.2, 2 × CH; Ar), 6.86 (2 H, dt, *J* 6.9 and 1.8, 2 × CH; Ph), 5.36 (1 H, dd, *J* 9.0 and 5.1, CHN), 4.54 (1 H, t, *J* 9.0, CH_AH$_B$O), 3.99 (1 H, dd, *J* 9.0 and 5.1, CH$_A$$H_B$O), 2.25 (3 H, s, CH_3; Ar) and 1.32 (3 H, s, ArCDCH_3); δ_C (100 MHz, $CDCl_3$) 173.9 (NC=O), 153.1 (OC=O), 138.3 (*i*-CMe; Ar), 136.8 (*i*-C; Ar), 136.7 (*i*-C; Ph), 129.1,[2] 128.8,[2] 128.5,[1] 128.0[2] and 125.8[2] (9 × CH; Ph and Ar), 69.6 (CH_2O), 57.8 (CHN), 43.4 (unlabelled Ar*C*HCH$_3$), 43.0 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.6 Hz, Ar*C*DCH$_3$), 21.0 (CH_3; Ar) and 18.6 (ArCH*C*H$_3$); negative isotopic shift at 43.0815 ppm was 0.3667 ppm (36.67 Hz at 100 MHz). (Found MNH_4^+, 328.1766; $C_{19}H_{22}DN_2O_3^+$ requires MNH_4^+, 328.1766).

*Parallel kinetic resolution of pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (***rac***)-[D_1]-10 using 4-[4-(***tert***-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (***R***)-19 and 4-phenyl-oxazolidin-2-one (***S***)-1*

In the same way as oxazolidin-2-one **3**, *n*-butyl lithium (0.49 mL, 2.5 M in hexane, 1.225 mmol), 4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*R*)-**19** (0.146 g, 0.50 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**1** (82 mg, 0.50 mmol), and pentafluorophenyl 2-deuterio-2-(4-isobutylphenyl)propionate (*rac*)-[D_1]-**10** (0.45 g, 1.21 mmol, [D]:[H] = 95:5), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-*syn*-**23** (ratio 97:3: *syn*-:*anti*-) and (*R,S*)-*syn*-**13** (ratio 97:3: *syn*-:*anti*-) in a ratio of 53:47. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (b.p. 40–60°C):diethyl ether (7:3) to give (2*S,4R*)-3-[2-deuterio-2-(4-isobutylphenyl)propionate]-4-[4-(*tert*-butyldimethylsilyloxy)phenyl]-oxazolidin-2-one (*S,R*)-*syn*-[D_1]-**23** (0.185 g, 77%) as a colourless oil; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.50; (Ref. 30 unlabelled (*R,S*)-*syn*-**23**; m.p. 68–70°C); $[a]_D^{20}$ = +134.5 (*c* 6.2, $CHCl_3$) {Ref. 30 (*R,S*)-*syn*-**23**; $[\alpha]_D^{20}$ = −129.6 (*c* 3.4, $CHCl_3$)}; $\nu_{max}(CH_2Cl_2)cm^{-1}$ 2400 (C-D), 1776 (OC=O) and 1702 (NC=O); δ_H (400 MHz; $CDCl_3$) 6.96 (4 H, br s, 4 × CH; Ar_A), 6.73 (2 H, dt, *J* 8.4 and 2.4, 2 × CH; Ar_B), 6.60 (2 H, dt, *J* 8.4 and 2.4, 2 × CH; Ar_B), 5.37 (1 H, dd, *J* 9.0 and 5.1, CHN), 4.56 (1 H, t, *J* 9.0, CH_AH$_B$O), 4.03 (1H, dd, *J* 9.0 and 5.1, CH$_A$$H_B$O), 2.40 (2 H, d, *J* 6.9, CH_2Ar), 1.83 (1 H, appears as a nonet, *J* 6.8, $(CH_3)_2$C*H*), 1.34 (3 H, s, ArCDCH_3), 0.95 (9 H, s, 3 × CH_3; *t*-Bu), 0.91 (3 H, d, *J* 6.8, CH_3^ACHCH_3^B), 0.89 (3 H, d, *J* 6.8 , CH_3^ACHCH_3^B) and 0.15 (6 H, s, 2 × $SiCH_3$); δ_C (100 MHz; $CDCl_3$) 173.8 (NC=O), 155.6 (OC=O), 153.1 (*i*-CO; Ar_A), 140.4, 136.9 and 130.9 (3 × *i*-C; Ar_A and Ar_B), 129.1, 127.8, 127.1 and 120.2 (4 × CH; Ar_A and Ar_B), 69.6 (CH_2O), 57.2 (CHN), 45.0 (CH_2Ar), 43.3 (Ar*C*HCH$_3$), 42.8 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ar*C*DCH$_3$), 30.1 (*C*H$_3$)$_2$CH), 25.5[3] (3 × *C*H$_3$C; *t*-Bu), 22.3 and 22.2 (2 × CH_3; *i*-Bu), 18.2 (ArCH*C*H$_3$), 18.1 (CH_3*C*; *t*-Bu) and −4.5[2] (2 × $SiCH_3$); negative isotopic shift at 42.8523 ppm was 0.3743 ppm (37.43 Hz at 100 MHz) (Found MH^+, 483.2788; $C_{28}H_{39}DNO_4Si$ requires MH^+, 483.2784); and (2*R,4S*)-3-[2-deuterio-(4-isobutylphenyl)propionyl]-4-phenyl-oxazolidin-2-one (*R,S*)-*syn*-[D_1]-**13** (0.134 g, 76 %) as a colourless oil; R_F [light petroleum ether (b.p. 40–60°C):diethyl ether (1:1)] 0.34; {Ref. 32 (*S,R*)-*syn*-**13**; m.p. 86–88°C}; $[\alpha]_D^{25}$ = −113.2 (*c* 3.8, $CHCl_3$) {Ref. 32 (*S,R*)-*syn*-**13**; $[\alpha]_D^{25}$ = +118.7 (*c* 6.0, $CHCl_3$); for unlabelled (*R,S*)-*syn*-**13**; $[\alpha]_D^{25}$ = −114.6 (*c* 4.2, $CHCl_3$)}; $\nu_{max}(CHCl_3)cm^{-1}$ 2400 (C-D), 1780 (OC=O) and 1707 (NC=O); δ_H(400 MHz; $CDCl_3$) 7.20-7.08 (3 H, m, 3 × CH; Ph), 6.97-6.90 (4 H, m, 4 × CH, Ph and Ar), 6.82 (2 H, dt, *J* 7.8 and 2.0, 2 × CH; Ar), 5.37 (1 H, dd *J* 9.1 and 5.1, CHN), 4.54 (1 H, t, *J* 9.1, CH_AH$_B$O), 3.96 (1 H, dd, *J* 9.1 and 5.1, CH$_A$$H_B$O), 2.35 (2 H, dd, *J* 7.4 and 2.2, CH_2Ar), 1.83-1.71 (1 H, appears as a nonet, *J* 6.8, (C*H*(CH$_3$)$_2$), 1.31 (3 H, s, ArCDCH_3), 0.84 (3 H, d, *J* 6.6, CH_3^ACHCH_3^B) and 0.82 (3 H, d, *J* 6.6, CH_3^ACHCH_3^B); δ_C(100.6 MHz; $CDCl_3$) 173.8 (NC=O), 153.1 (OC=O), 140.5 (*i*-C; Ar), 138.2 (*i*-C; Ar), 136.8 (*i*-C; Ph), 129.2[2] and 128.7[2] (4 × CH; Ar), 128.4,[1] 127.8[2] and 125.7[2] (5 × CH; Ph), 69.5 (CH_2O), 57.6 (CHN), 45.0 (*C*H(CH$_3$)$_2$), 43.2 (unlabelled Ar*C*HCH$_3$), 42.8 (1 C, t [1:1:1], $^1J_{C,D}$ = 19.8 Hz, Ar*C*DCH$_3$), 30.2 (*C*H$_2$Ar), 22.4 (*C*H$_3^A$CHCH$_3^B$), 22.3 (CH$_3^A$CH*C*H$_3^B$) and 18.2 (ArCD*C*H$_3$); negative isotopic shift at 42.8829 ppm was 0.3590 ppm (35.90 Hz at 100 MHz) (Found MH^+, 353.1970; $C_{22}H_{25}DNO_3$ requires MH^+, 353.1971).

Hydrolysis of oxazolidin-2-one (S,R)-syn-[D$_1$]-3; synthesis of 2-deuterio-2-phenylpropionic acid (S)-[D$_1$]-4

Lithium hydroxide monohydrate (32 mg, 0.76 mmol) was slowly added to a stirred solution of oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**3** (0.18 g, 0.60 mmol) and hydrogen peroxide (0.22 mL, 3.53 M in H_2O, 0.79 mmol) in THF/water (1:1; 5 mL). The reaction mixture was stirred at room temperature for 12 h. The reaction was quenched with water (10 mL) and extracted with dichloromethane (3 × 10 mL). The combined organic layers were dried (over $MgSO_4$) and evaporated under reduced pressure to give the oxazolidin-2-one (*R*)-**1** (0.116 g, 96 %) as a white solid. After acidic extraction of the aqueous layer using HCl (1 M, 10 mL) and dichloromethane (3 × 10 mL), gave 2-deuterio-2-phenylpropionic acid (*S*)-**4** (75 mg, 82%) ([D]:[H] = 97:3) as a colourless liquid; >98% *e.e.*, $[\alpha]_D^{24}$ = + 71.8 (*c* 0.15, $CHCl_3$); {unlabelled (*S*)-**4**; Ref. 31 $[a]_D^{20}$ = +71.2 (*c* 0.66, $CHCl_3$)}; ν_{max} (film)cm^{-1} 2306 (CD) and 1704 (C=O); δ_H (270 MHz; $CDCl_3$) 7.35-7.20 (5 H, m, 5 × CH; Ph) and 1.50 (3 H, s, CH_3); δ_C (100 MHz; $CDCl_3$) 180.6 (C=O), 140.0 (*i*-C; Ph), 129.0,[2] 128.0[2] and 127.7[1] (5 × CH; Ph), 45.7 (1 C, s, unlabelled Ph*C*HCH$_3$), 45.3 (1 C, t [1:1:1], $^1J_{C,D}$ = 20.1 Hz, Ph*C*DCH$_3$) and 18.3 (CD*C*H$_3$); (Found MNH_4^+, 169.1081; $C_9H_{13}DNO_2$ requires 169.1082). Isotope shift at C(2) was 0.27 ppm (27.1 Hz at 100.613 MHz).

Hydrolysis of oxazolidin-2-one (R,S)-syn-[D$_1$]-15; synthesis of 2-deuterio-2-phenylpropionic acid (R)-[D$_1$]-4

In the same way as 2-deuterio-2-phenylpropionic acid (*S*)-[D_1]-**4**, oxazolidin-2-one (*R*,*S*)-*syn*-[D_1]-**15** (61 mg, 0.14 mmol), lithium hydroxide monohydrate (11 mg, 0.27 mmol) and hydrogen peroxide (80 μL, 3.53 M in H_2O, 0.27 mmol,) in THF/water (1:1; 5 mL), gave after purification by aqueous extraction as 2-deuterio-2-phenylpropionic acid (*R*)-[D_1]-**4**[33] (19 mg, 92%) ([D]:[H] = 99:1) as a colourless liquid; >95% *e.e.*, $[\alpha]_D^{22}$ = −64.9 (*c* 3.6, $CHCl_3$) {unlabelled (*R*)-**4**; Ref. 30 $[\alpha]_D^{22}$ = −71.4 (*c* 0.7, $CHCl_3$)} which was spectroscopically identical to those previously obtained. The combined organic layers were dried (over $MgSO_4$) and evaporated under reduced pressure and purified by column chromatography to give the 4,5,5-triphenyloxazolidin-2-one (*S*)-**14** (41 mg, 95%) as a white solid.

Hydrolysis of oxazolidin-2-one (S,R)-syn-[D$_1$]-20; synthesis of 2-deuterio-2-phenylpropionic acid (S)-[D$_1$]-4

In the same way as 2-deuterio-2-phenylpropionic acid (*S*)-[D_1]-**4**, oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**20** (73 mg, 0.17 mmol), lithium hydroxide monohydrate (7 mg, 0.17 mmol) and hydrogen peroxide (50 μL, 3.53 M in H_2O, 0.17 mmol) in THF/water (1:1; 5 mL), gave after purification by aqueous extraction as 2-deuterio-2-phenylpropionic acid (*S*)-[D_1]-**4** (20 mg, 77%) ([D]:[H] = 95:5) as a colourless liquid; >98% *e.e.*, $[\alpha]_D^{22}$ = +68.0 (*c* 1.7, $CHCl_3$) {unlabelled (*S*)-**4**; Ref. 31 $[\alpha]_D^{22}$ = +71.2 (*c* 0.66, $CHCl_3$)} which was spectroscopically identical to those previously obtained. The combined organic layers were dried (over $MgSO_4$) and evaporated under reduced pressure and purified by column chromatography to give the oxazolidin-2-one (*R*)-**19** (44 mg, 88%) as a white solid.

Hydrolysis of oxazolidin-2-one (R,S)-syn-[D$_1$]-3; synthesis of 2-deuterio-2-phenylpropionic acid (R)-[D$_1$]-4

In the same way as 2-deuterio-2-phenylpropionic acid (*S*)-[D_1]-**4**, oxazolidin-2-one (*R*,*S*)-*syn*-[D_1]-**3** (44 mg, 0.15 mmol), lithium hydroxide monohydrate (12 mg, 0.30 mmol) and hydrogen peroxide (80 μL, 3.53 M in H_2O, 0.30 mmol) in THF/water (1:1; 5 mL), gave after purification by aqueous extraction as 2-deuterio-2-phenylpropionic acid (*R*)-[D_1]-**4** (15 mg, 67%) ([D]:[H] = 95:5) as a colourless liquid; >98% *e.e.*, $[\alpha]_D^{22}$ = −63.6 (*c* 3.0, $CHCl_3$); which was spectroscopically identical to those previously obtained. The combined organic layers were dried (over $MgSO_4$) and evaporated under reduced pressure and purified by column chromatography to give the oxazolidin-2-one (*S*)-**1** (23 mg, 95%) as a white solid.

Representative procedure for determining the enantiomeric excess of 2-deuterio-2-phenylpropionic acid 4

(*S*)-1-Phenyl-ethanol (12 mg, 99 μmol) was added to a stirred solution of 2-deuterio-2-phenyl-propionic acid (*S*)-[D_1]-**4** (15 mg, 99 μmol) {derived from the hydrolysis of oxazolidin-2-one (*S*,*R*)-*syn*-[D_1]-**20**}, DMAP (3 mg, 20 μmol) and DCC (23 mg, 0.11 mmol) in CH_2Cl_2 (5 ml) at room temperature. The solution was stirred for 12 h. The resulting DCU was filtered off, and the remaining CH_2Cl_2 layer was washed with brine (10 mL), dried (over $MgSO_4$) and evaporated under reduced pressure. By ^{1}H NMR spectroscopy (400 MHz) a single diastereoisomeric ester [(*S*,*S*)-*anti*-][34] was detected. The enantiomeric excess of the original 2-deuterio-2-phenyl-propionic acid (*S*)-[D_1]-**4** was >98% *e.e.*

In the same way as above, (*R*)-1-phenyl-ethanol (33 mg, 0.27 mmol) was added to a stirred solution of 2-deuterio-2-phenyl-propionic acid (*R*)-[D_1]-**4** (41 mg, 0.27 mmol) {derived from the hydrolysis of oxazolidin-2-one (*R*,*S*)-*syn*-[D_1]-**15**}, DMAP (7 mg, 50 μmol) and DCC (62 mg, 0.30 mmol) in CH_2Cl_2 (5 ml) at room temperature. The solution was stirred for 12 h. The resulting DCU was filtered off, and the remaining CH_2Cl_2 layer was washed with brine (10 mL), dried (over $MgSO_4$) and evaporated under reduced pressure. By ^{1}H NMR spectroscopy (400 MHz) a single diastereoisomeric ester [(*R*,*R*)-*anti*-][34] was detected. The enantiomeric excess of the original 2-deuterio-2-phenyl-propionic acid (*R*)-[D_1]-**4** was >98% *e.e.*

CONCLUSIONS

In conclusion, we have reported the efficient parallel kinetic resolution of a series of structurally related pentafluorophenyl active esters {*e.g.*, (*rac*)-[D_1]-**2**} using three combinations of designer oxazolidin-2-ones (*R*)-**1** and (*S*)-[D_2]-**1**, (*R*)-**1** and (*S*)-**14**, and (*S*)-**1** and (*R*)-**19**. The levels of diastereocontrol were found to be excellent favoring the formation of the corresponding *syn*-oxazolidin-2-one

adducts **3**, **15** and **20** in high yields with excellent levels of diastereoselectivity. The preferred combination of *quasi*-enantiomeric oxazolidin-2-ones for the efficient resolution of pentafluorophenyl 2-deuterio-2-phenylpropionate (*rac*)-[D_1]-**2** was found to involve the oxazolidin-2-ones (*S*)-**1** and (*R*)-**19**. These oxazolidin-2-ones were shown to be efficient *quasi*-enantiomers for the parallel kinetic resolution and separation of a variety of 2-deuterio-2-aryl-propionic and butanoic acids in good yield with high levels of deuterium incorporation.

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