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Parallel kinetic resolution of active esters using a *quasi*-enantiomeric combination of (*R*)-4-phenyl-oxazolidin-2-one and (*S*)-4,5,5-triphenyl-oxazolidin-2-one

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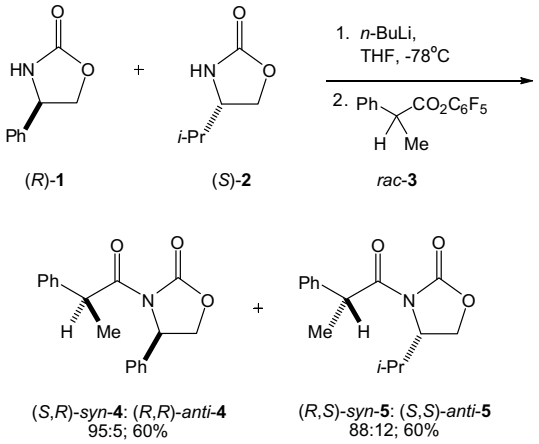
ABSTRACT

The parallel kinetic resolution of a series of racemic pentafluorophenyl 2-(4-aryl/phenyl)-propionates and -butanoates using a *quasi*-enantiomeric combination of (*R*)-4-phenyl-oxazolidin-2-one and (*S*)-4,5,5-triphenyl-oxazolidin-2-one is discussed. The levels of diastereoselectivity were excellent (86–98% de) leading to separable *quasi*-enantiomeric oxazolidin-2-one adducts in good yield. This methodology was used to resolve 2-phenyl propionic acid.

1. Introduction

The continuing development of novel synthetic methods for the efficient separation of enantiomers is paramount.[1] One particular strategy that has recently attracted some attention has been the parallel kinetic separation of enantiomers using a combination of *quasi*-enantiomeric[2] auxiliary components.[3,4] We have been interested in the philosophy of this approach[5] through the use of *quasi*-enantiomeric combinations of Evans' oxazolidin-2-ones, such as (*R*)-**1** and (*S*)-**2**, as mutual resolving components (Scheme 1).[6]



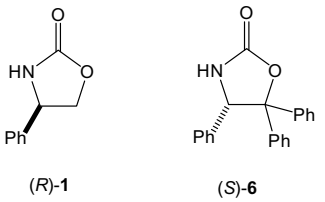
Scheme 1. Parallel kinetic resolution of active ester *rac*-**3** using *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-**2**.

Within this area, we have recently reported the parallel kinetic resolution of an active ester, pentafluorophenyl 2-phenylpropionate *rac*-**3**, using a combination of (lithiated) Evans' oxazolidin-2-ones (*R*)-**1** and (*S*)-**2** to give the oxazolidin-2-one adducts (*S*,*R*)-*syn*-**4** (in 60% yield) and (*R*,*S*)-*syn*-**5** (in 60% yield) with 90% and 76% diastereoisomeric excesses, respectively (Scheme 1).[6]

2. Results and discussion

From this study, it was evident that the levels of stereocontrol and mutual recognition were higher for the phenyl-glycine derived oxazolidin-2-one (*R*)-**1** than for its *quasi*-enantiomeric partner, the valine-derived oxazolidin-2-one (*S*)-**2** (90% de vs 76% de) (Scheme 1). In order to facilitate higher enantiomeric recognition, a better surrogate than oxazolidin-2-one (*S*)-**2** for the (*S*)-enantiomer of 4-phenyl-oxazolidin-2-one **1** was sought (Scheme 1).[6]

In an attempt to address this issue of complementarity, we chose to focus our attention on the use of a Davies[7]/Seebach[8] inspired designer 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** due to its structural similarity to the original Evans 4-phenyl-oxazolidin-2-one **1**[9] (Scheme 2). We herein report our study on the use of 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6**[10] as a surrogate for the



Scheme 2. *Quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-**6**.

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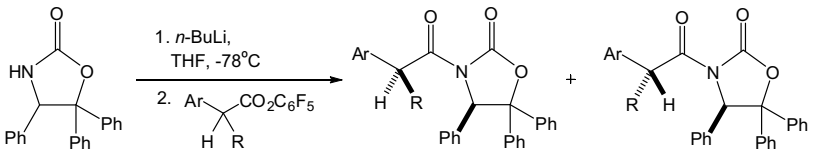


Entry	Active ester	Ar	R	Oxazolidinone adducts	D.e.	Yield
1	*rac*-**3**	Ph	Me	*rac-syn*-**7**: *rac-anti*-**7** 89:11	78%	53%
2	*rac*-**8**	Ph	Et	*rac-syn*-**9**: *rac-anti*-**9** 97:3	94%	83%
3	*rac*-**10**	Ph	*i*-Pr	*rac-syn*-**11**: *rac-anti*-**11** 78:22	56%	18%
4	*rac*-**12**	4-MeC_6H_4-	Me	*rac-syn*-**13**: *rac-anti*-**13** 93:7	86%	60%
5	*rac*-**14**	4-ClC_6H_4-	Me	*rac-syn*-**15**: *rac-anti*-**15** 92:8	84%	58%
6	*rac*-**16**	4-*i*-BuC_6H_4-	Me	*rac-syn*-**17**: *rac-anti*-**17** 95:5	90%	77%

Scheme 3. Mutual kinetic resolution of active esters *rac*-**3**, **8**, **10**, **12**, **14** and **16** using oxazolidin-2-one *rac*-**6**.

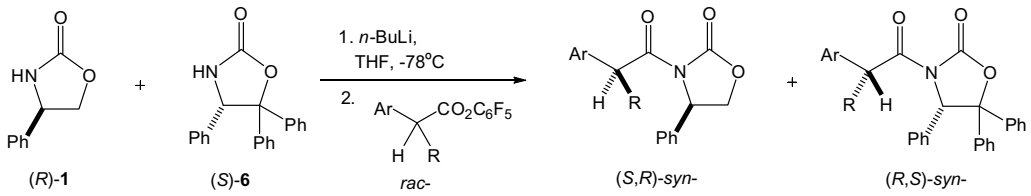
(*S*)-enantiomer of 4-phenyl-oxazolidin-2-one **1** for the efficient parallel kinetic resolution of a series of structurally related active esters (Scheme 2).

With this oxazolidin-2-one *rac*-**6** in hand, we first investigated its mutual kinetic resolution with pentafluorophenyl 2-phenylpropionate *rac*-**3** in order to determine its relative stereoselection (Scheme 3). Deprotonation of oxazolidin-2-one *rac*-**6** in THF at −78 °C using *n*-BuLi, followed by the addition of pentafluorophenyl 2-phenylpropionate *rac*-**3**, gave after 2 h at −78 °C an inseparable diastereoisomeric mixture of oxazolidin-2-ones (*SR*,*RS*)-*syn*- and (*RS*,*RS*)-*anti*-**7** (ratio 89:11)[10,11] in a combined 53% yield (Scheme 3). Interestingly, the levels of mutual recognition between the 4,5,5-triphenyl-oxazolidin-2-one (*R*)-**6** and the corresponding (*S*)-enantiomer of active ester **3** (and its mirror image combination) were noticeably lower than that of its parent (mutual kinetic) resolution involving 4-phenyl-oxazolidin-2-one **1** (78% de for *rac*-**6** vs 94% de for *rac*-**1**).[6,12]

We next focused our attention on the mutual kinetic resolution of a series of structurally related active esters *rac*-**8**, *rac*-**10**, *rac*-**12**, *rac*-**14** and *rac*-**16** (derived from the DCC coupling of pentafluorophenol with the corresponding carboxylic acids)[12] using 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** as our mutual resolving component under standard conditions (Scheme 3: entries 2–6). Deprotonation of 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** using *n*-BuLi in THF at −78 °C, followed by the addition of active esters *rac*-**8**, *rac*-**10**, *rac*-**12**, *rac*-**14** and *rac*-**16** in THF, gave after 2 h the corresponding oxazolidin-2-one adducts *rac-syn*- and *rac-anti*-**9**, **11**, **13**, **15** and **17** in 18–83% yields with 56–94% diastereoisomeric excesses (Scheme 3: entries 2–6). The levels of diastereocontrol were found to be excellent (ranging from 56% to 94% diastereoisomeric excess) favouring the formation of the corresponding *syn*-diastereoisomeric oxazolidin-2-one. The sterically demanding active ester *rac*-**8** was found to give the highest level of diastereoselectivity (94% de) (Scheme 3: entry 2). However, there appears to be a sterically demanding threshold as the more sterically encumbered active ester *rac*-**10** gave lower levels of stereocontrol. The levels of diastereocontrol were found to be high for a series of active esters *rac*-**12**, **14** and **16** derived from a variety of structurally related 2-arylpropionic acids (Scheme 3: entry 1 vs entries 4–6).[13]

With this information at hand, we next attempted the parallel kinetic resolution of pentafluorophenyl 2-phenylpropionate *rac*-**3** using an equimolar combination of *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-**6** (Scheme 4). Treatment of an equimolar amount of oxazolidin-2-ones (*R*)-**1** and (*S*)-**6** with *n*-BuLi in THF at −78 °C, followed by the addition of pentafluorophenyl phenylpropionate *rac*-**3**, gave after 2 h at −78 °C the corresponding *syn*-oxazolidin-2-one adducts (*S*,*R*)-*syn*-**4** and (*R*,*S*)-*syn*-**7** in 58% and 60% yields with 94% and 96% diastereoisomeric excesses, respectively (Scheme 4: entry 1).[14] The levels of enantiomer selection between (*R*)-**1** and (*S*)-**3** [to give (*S*,*R*)-**4**], and (*S*)-**6** and (*R*)-**3** [to give (*R*,*S*)-**7**] were excellent. For 4-phenyl-oxazolidin-2-one (*R*)-**1**, the levels of molecular recognition were comparable for both its parallel and mutual kinetic resolutions. In comparison, 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** gave significantly higher levels of diastereocontrol for its parallel kinetic resolution (of *rac*-**3**) than its mutual kinetic resolution (PKR: 96% de vs MKR: 78% de) (Scheme 3: entry 1 vs Scheme 4: entry 1). The levels of diastereoselection for oxazolidin-2-one (*S*)-**6** were also found to be dependent on the amount of the complementary oxazolidin-2-one (*R*)-**1** used. For example, using non-equimolar amounts of oxazolidin-2-ones (*R*)-**1** and (*S*)-**6** (in a relative ratio of 1:3 and 3:1) gave the corresponding oxazolidin-2-one adducts *rac-syn*-**4** and *rac-syn*-**7** in 53% yield with >96% de and 45% yield with 86% de [for (*R*)-**1**:(*S*)-**6**—ratio 1:3], respectively, and in 65% yield with 52% de and 74% yield with >96% de [for (*R*)-**1**:(*S*)-**6**—ratio 3:1], respectively. Interestingly, in both cases for oxazolidin-2-one (*S*)-**6**, the levels of enantiomeric recognition were greater than its original mutual recognition process. Whereas, using a supra- and sub-stoichiometric amount of oxazolidin-2-one (*R*)-**1** gave lower and higher levels of diastereocontrol, respectively.

We next turned our attention to the parallel kinetic resolution of a series of structurally related active esters *rac*-**8**, *rac*-**10**, *rac*-**12**, *rac*-**14** and *rac*-**16** using a *quasi*-enantiomeric combination of oxazolidin-2-ones (*R*)-**1** and (*S*)-**6** (Scheme 4: entries 2–6). Deprotonation of an equimolar combination of oxazolidin-2-ones (*R*)-**1** and (*S*)-**6** with *n*-BuLi in THF at −78 °C, followed by the addition of active esters *rac*-**8**, *rac*-**10**, *rac*-**12**, *rac*-**14** and *rac*-**16**, gave after 2 h at −78 °C the corresponding oxazolidin-2-one adducts (*S*,*R*)-



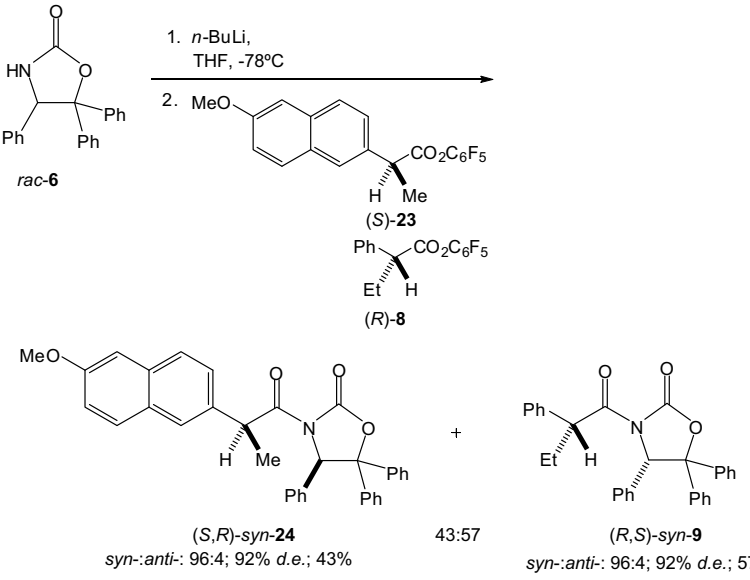
Entry	Active ester	Ar	R	Oxazolidinone adducts derived from (*R*)-**1**	D.e.	Yield	Oxazolidinone adducts derived from (*S*)-**6**	D.e.	Yield	Ratio
1	*rac*-**3**	Ph	Me	(*S*,*R*)-*syn*-**4**: (*R*,*R*)-*anti*-**4** 97:3	94%	58%	(*R*,*S*)-*syn*-**7**: (*S*,*S*)-*anti*-**7** 98:2	96%	60%	**4**: **7** 52:48
2	*rac*-**8**	Ph	Et	(*S*,*R*)-*syn*-**18**: (*R*,*R*)-*anti*-**18** 99:1	98%	63%	(*R*,*S*)-*syn*-**9**: (*S*,*S*)-*anti*-**9** 97:3	94%	59%	**18**: **9** 50:50
3	*rac*-**10**	Ph	*i*-Pr	(*S*,*R*)-*syn*-**19**: (*R*,*R*)-*anti*-**19** 77:23	54%	79%	(*R*,*S*)-*syn*-**11**: (*S*,*S*)-*anti*-**11** 88:12	76%	43%	**19**: **11** 69:31
4	*rac*-**12**	4-MeC_6H_4-	Me	(*S*,*R*)-*syn*-**20**: (*R*,*R*)-*anti*-**20** 99:1	98%	45%	(*R*,*S*)-*syn*-**13**: (*S*,*S*)-*anti*-**13** 98:2	96%	51%	**20**: **13** 50:50
5	*rac*-**14**	4-ClC_6H_4-	Me	(*S*,*R*)-*syn*-**21**: (*R*,*R*)-*anti*-**21** 93:7	86%	71%	(*R*,*S*)-*syn*-**15**: (*S*,*S*)-*anti*-**15** 96:4	92%	60%	**21**: **15** 52:48
6	*rac*-**16**	4-*i*-BuC_6H_4-	Me	(*S*,*R*)-*syn*-**22**: (*R*,*R*)-*anti*-**22** 97:3	94%	60%	(*R*,*S*)-*syn*-**17**: (*S*,*S*)-*anti*-**17** 98:2	96%	55%	**22**: **17** 56:44

Scheme 4. Parallel kinetic resolution of active esters *rac*-**3**, **8**, **10**, **12**, **14** and **16** using a combination of *quasi*-enantiomeric oxazolidin-2-ones (*R*)-**1** and (*S*)-**6**.

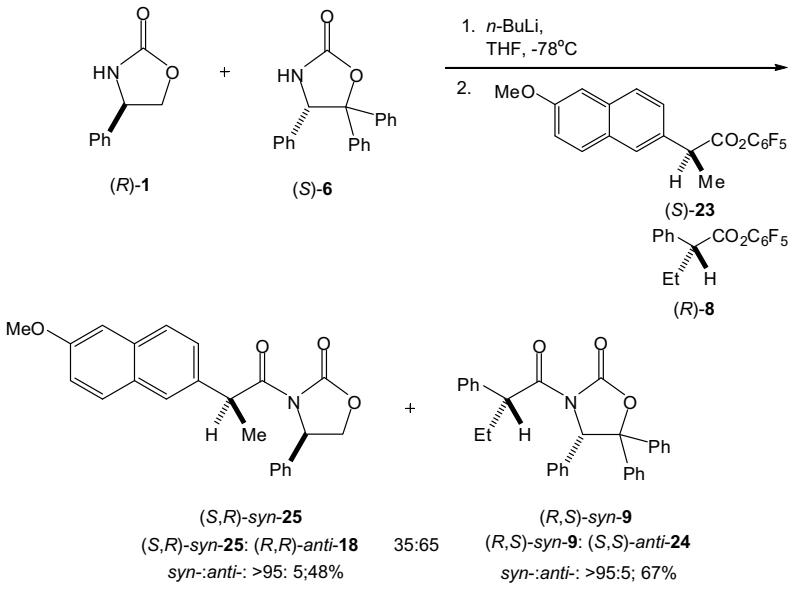
syn-**18** and (*R*,*S*)-*syn*-**9** (in 63% and 59% yields for *rac*-**8**), (*S*,*R*)-*syn*-**19** and (*R*,*S*)-*syn*-**11** (in 79% and 43% yields for *rac*-**10**), (*S*,*R*)-*syn*-**20** and (*R*,*S*)-*syn*-**13** (in 45% and 51% yields for *rac*-**12**), (*S*,*R*)-*syn*-**21** and (*R*,*S*)-*syn*-**15** (in 71% and 60% yields for *rac*-**14**) and (*S*,*R*)-*syn*-**22** and (*R*,*S*)-*syn*-**17** (in 60% and 55% yields for *rac*-**16**) with excellent diastereoselectivities (54–98% de) (Scheme 4: entries 2–6). Interestingly, in agreement with the previous study, the levels of diastereoselection were found to be higher for the 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**1** [in the presence of oxazolidin-2-one (*R*)-**1**] than their corresponding mutual kinetic resolutions (Scheme 3 vs Scheme 4).

These reactions proceeded efficiently to give access to easily separable *syn*-oxazolidin-2-one adducts, such as (*S*,*R*)-*syn*-**18** and (*R*,*S*)-*syn*-**9** [derived from Evans' and Seebach's oxazolidin-2-ones (*R*)-**1** and (*S*)-**6**, respectively, and the corresponding active ester *rac*-**8**]; ΔR_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] = 0.30.

We next focussed our attention on the complementary parallel kinetic resolution of 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** using a *quasi*-enantiomeric combination of separable active esters[15] (*S*)-**23** and (*R*)-**8** (Scheme 5). Treatment of 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** with *n*-BuLi in THF at −78 °C, followed by the addition



Scheme 5. Parallel kinetic resolution of oxazolidin-2-one *rac*-**6** using active esters (*S*)-**23** and (*R*)-**8**.

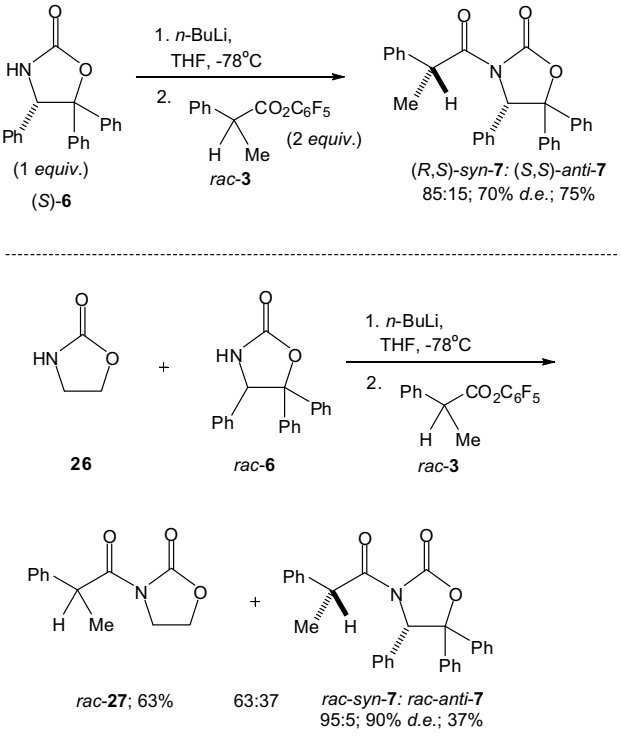


Scheme 6. Mutual kinetic separation of oxazolidin-2-ones (*R*)-**1** and (*S*)-**6** using active esters (*S*)-**23** and (*R*)-**8**.

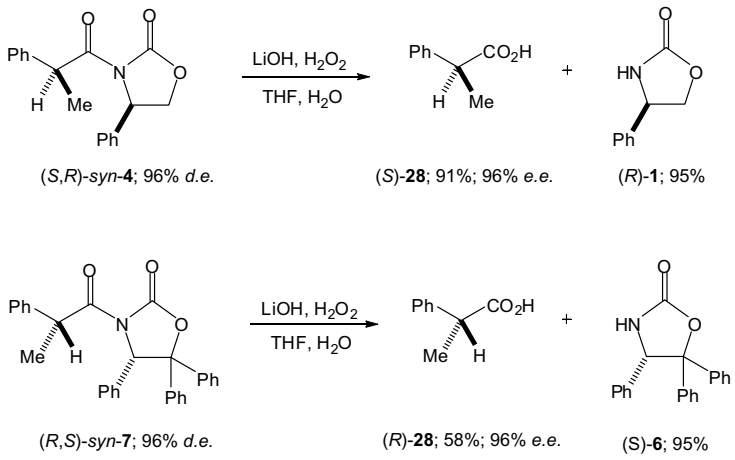
of an equimolar amount of active esters (*S*)-**23** and (*R*)-**8** in THF, gave after 2 h at −78 °C the corresponding oxazolidin-2-one adducts (*S*,*R*)-*syn*-**24** and (*R*,*S*)-*syn*-**9** in 43% and 57% yields, respectively, with an equal 92% diastereoisomeric excess (Scheme 5). These adducts were efficiently separated/isolated by column chromatography due to their difference in polarity {ΔR_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] = 0.14}. The levels of molecular recognition were also found to be similar to those of their parent mutual kinetic resolutions (as shown in Scheme 3).

With this information at hand, we next investigated the configurational stability of these active esters by probing their mutual kinetic separation. Treatment of a pair of *quasi*-enantiomeric active esters (*S*)-**23** and (*R*)-**8** with a *quasi*-enantiomeric combination of lithiated oxazolidin-2-ones [formed by *n*-BuLi deprotonation of (*R*)-**1** and (*S*)-**6**], gave the corresponding oxazolidin-2-one adducts (*S*,*R*)-*syn*-**25** and (*R*,*S*)-*syn*-**9** in 48% and 67% yields, respectively, with high levels of enantiomeric (and substrate) recognition (Scheme 6). From this study, it appears that these active esters (*S*)-**23** and (*R*)-**8** were configurationally stable under these reaction conditions as the corresponding diastereoisomeric adducts (*R*,*R*)-*anti*-**25** and (*S*,*S*)-*anti*-**9** were absent (as determined by 400 MHz ^{1}H NMR spectroscopy) (Scheme 6). The levels of enantiomer selection between (*R*)-**1** and (*S*)-**23** [to give (*S*,*R*)-*syn*-**25**], and (*S*)-**6** and (*R*)-**8** [to give (*R*,*S*)-*syn*-**9**] were excellent. Interestingly, these levels of enantiomeric recognition were noticeably higher than those that were suggested from the parent mutual kinetic resolution of Seebach's oxazolidin-2-one *rac*-**6** with the active ester *rac*-**3** [to give the oxazolidin-2-one adduct *rac*-*syn*-**7** in 53% with 78% de (Scheme 3: entry 1)].

In an attempt to probe this apparent anomaly, we next investigated the kinetic resolution of active ester *rac*-**3** (2 equiv) using Seebach's oxazolidin-2-one (*S*)-**6** (1 equiv) (Scheme 7). Treatment of oxazolidin-2-one (*S*)-**6** with *n*-BuLi in THF at −78 °C, followed by the addition of active ester *rac*-**3** (2 equiv), gave an inseparable diastereoisomeric mixture of oxazolidin-2-one adducts (*R*,*S*)-*syn*- and (*S*,*R*)-*anti*-**7** in 75% yield (ratio 85:15) (Scheme 7).[16] The levels of diastereoisomeric control were found to be surprisingly similar for both the kinetic (70% de) and mutual resolutions (78% de) of the same racemic active ester **3** (Schemes 3 and 7). However, these levels of diastereocontrol could be improved to >90% de by the addition of the simplest achiral oxazolidin-2-one surrogate **26** (Scheme 7). Deprotonation of an equimolar amount of oxazolidin-2-ones **26** and *rac*-**6** with *n*-BuLi (2.2 equiv) at −78 °C in THF, followed by the addition of active ester *rac*-**3**, gave a separable mixture of the oxazolidin-2-ones **27** (in 63% yield) and *rac*-*syn*-**7** (in 37% yield with 90% de) (Scheme 7). Interestingly, the mutual kinetic resolution of oxazolidin-2-one *rac*-**6** with active ester *rac*-**3** appeared to mimic the outcome derived from its traditional kinetic resolution, whereas using the achiral oxazolidin-2-one **26** (as a complementary surrogate) allowed the reaction to proceed



Scheme 7. Kinetic and mutual resolutions of oxazolidin-2-ones (*S*)- and *rac*-**6** using active ester *rac*-**3**.



Scheme 8. Hydrolysis of oxazolidin-2-one adducts (*S*,*R*)-*syn*-**4** and (*R*,*S*)-*syn*-**7**.

via a more diastereoselective mutual kinetic resolution pathway. From this study, it appears that hetero-aggregation between the lithiated oxazolidin-2-ones (derived from *rac*-**6** and **26**) is in-part responsible for this increase in diastereoselection.

Access to both enantiomers of 2-phenylpropionic acid (*S*)- and (*R*)-**28** was achieved through hydrolysis [LiOH monohydrate/hydrogen peroxide in THF/H_2O (3:1)] of a pair of *quasi*-enantiomeric adducts [e.g., (*S*,*R*)-*syn*-**4** and (*R*,*S*)-*syn*-**7**] in 91% and 58% yields, respectively (Scheme 8).[17]

3. Conclusion

In conclusion, we have reported the efficient parallel kinetic resolution of a series of active esters (e.g., *rac*-**3**) using an equimolar *quasi*-enantiomer combination of 4-phenyl-oxazolidin-2-one (*R*)-**1** and 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6**. The levels of diastereocontrol were found to be excellent, favouring the formation of the corresponding *syn*-oxazolidin-2-one adducts (*S*,*R*)-*syn*-**4** and (*R*,*S*)-*syn*-**7** in good yields with excellent levels of diastereoselectivity. This combination of oxazolidin-2-ones was shown to be efficient *quasi*-enantiomers for the parallel kinetic resolution and separation of a variety of structurally related 2-aryl propionic and butanoic acids.

4. Experimental

4.1. 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 Ltd polarimeter. The active esters, pentafluorophenyl 2-phenylpropionate *rac*-**3**, pentafluorophenyl 2-phenylbutanoate *rac*-**8**, pentafluorophenyl 2-(4-methylphenyl)propionate *rac*-**12** and pentafluorophenyl 2-(4-isobutylphenyl)propionate *rac*-**16**, have been reported elsewhere.[12]

4.2. Synthesis of pentafluorophenyl 2-phenyl-3-methylbutanoate *rac*-10

Pentafluorophenol (0.5 g, 2.72 mmol) in dichloromethane (5 mL) was added to *N*,*N*′-dicyclohexylcarbodiimide (0.62 g, 3.00 mmol) in dichloromethane (5 mL), and the resulting solution was stirred for 5 min. A solution of racemic 2-phenyl-3-methyl butanoic acid (0.48 g, 2.72 mmol) in dichloromethane (20 mL) was added in four equal portions over 1 h. The solution was stirred for 12 h and the resulting precipitate (*N*,*N*′-dicyclohexylurea) was filtered off (using suction filtration). Brine (30 mL) was added and the solution was extracted into dichloromethane (3 × 50 mL). The combined organic layers were dried over $MgSO_4$ and evaporated under reduced pressure. The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (9:1) to give pentafluorophenyl 2-phenyl-3-methyl butanoate *rac*-**10** (0.69 g, 74%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (9:1)] 0.77; mp 40–42 °C; ν_{max} (film) cm^{-1} 1776 (C=O); δ_H (400 MHz; $CDCl_3$) 7.39–7.31 (5H, m, 5 × CH; Ph), 3.51 (1H, d, *J* 10.3, PhC*H*i-Pr), 2.51–2.40 (1H, m, C*H*$(CH_3)_2$), 1.15 (3H, d, *J* 6.6, CH_3^ACHCH_3^B) and 0.79 (3H, d, *J* 6.6, CH_3^ACHCH_3^B); δ_C (100 MHz; $CDCl_3$) 169.9 (C=O), 141.1 (142.37 and 139.88, 2 C, ddt, $^1J_{C,F}$ = 251.4 Hz, $^2J_{C,F}$ = 12.3 Hz and $^3J_{C,F}$ = 3.8 Hz, C(2)–F), 139.4 (140.65 and 138.14, 1C, dtt, $^1J_{C,F}$ = 252.9 Hz, $^2J_{C,F}$ = 13.8 Hz and $^3J_{C,F}$ = 4.6 Hz, C(4)–F), 137.8 (139.05 and 136.58, 2C, dtdd, $^1J_{C,F}$ = 249.1 Hz, $^2J_{C,F}$ = 13.1 Hz, $^3J_{C,F}$ = 5.4 Hz and $^4J_{C,F}$ = 3.1 Hz, C(3)–F), 136.4 (*i*-C; Ph), 128.8[2], 128.5[2] and 127.9[1] (5 × CH; Ph), 125.1 (1 C, tdt, $^2J_{C,F}$ = 14.6 Hz, $^4J_{C,F}$ = 4.6 Hz and $^3J_{C,F}$ = 2.3 Hz, *i*-CO; OC_6F_5), 59.2 (PhC*H*i-Pr), 31.8 (*C*H$(CH_3)_2$), 21.2 (CH_3^ACHCH_3^B) and 20.0 (CH_3^ACHCH_3^B); δ_F (378 MHz; $CDCl_3$) −152.3 (2 F, dt, $_3J_{F,F}$ 17.3 and $^4J_{F,F}$ 4.8, F_{ortho}), −158.0 (1F, t, $^3J_{F,F}$ 21.9, F_{para}) and −162.4 (2F, td, $^3J_{F,F}$ 21.9 and $^4J_{F,F}$ 4.8 F_{meta}) (Found M^+ 344.0829; $C_{17}H_{13}F_5O_2^+$ requires M^+ 344.0830); *m*/*z* 344 (10%, M^+), 133 [65, $(PhCHC_3H_7)^+$] and 91 [100, $(PhCH_2)^+$].

4.3. Synthesis of pentafluorophenyl 2-(4-chlorophenyl)-propionate *rac*-12

In the same way as the active ester *rac*-**10**, 4-chlorophenylpropionic acid (1.49 g, 8.11 mmol), *N*,*N*′-dicyclohexylcarbodiimide (1.84 g, 8.92 mmol) and pentafluorophenol (1.49 g, 8.11 mmol) gave the pentafluorophenyl 2-(4-chlorophenyl)propionate *rac*-**12** (2.61 g, 92%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82; mp 37–40 °C; ν_{max} (film) cm^{-1}

1782 (C=O); δ_H (400 MHz; $CDCl_3$) 7.35 (2H, dt, *J* 8.6 and 2.2, 2 × CH; Ar), 7.29 (2H, dt, *J* 8.6 and 2.2, 2 × CH; Ar), 4.04 (1H, q, *J* 7.1, ArC*H*CH_3) and 1.63 (3H, d, *J* 7.1, ArCHCH_3); δ_C (100 MHz; $CDCl_3$) 170.2 (C=O), 141.1 (142.29 and 139.78, 2C, ddt, $^1J_{C,F}$ = 250.6 Hz, $^2J_{C,F}$ = 12.2 Hz and $^3J_{C,F}$ = 3.8 Hz, C(2)–F), 139.5 (140.73 and 138.21, 1C, dtt, $^1J_{C,F}$ = 252.1 Hz, $^2J_{C,F}$ = 13.7 Hz and $^3J_{C,F}$ = 3.8 Hz, C(4)–F), 137.8 (139.05 and 136.54, 2C, dtdd, $^1J_{C,F}$ = 250.9 Hz, $^2J_{C,F}$ = 14.5 Hz, $^3J_{C,F}$ = 5.3 Hz and $^4J_{C,F}$ = 3.1 Hz, C(3)–F), 137.1 (*i*-CCl; Ar), 133.7 (*i*-C; Ar), 129.1[2] and 128.8[2] (4 × CH; Ar), 125.0 (1 C, tdt, $^2J_{C,F}$ = 14.5 Hz, $^4J_{C,F}$ = 5.3 Hz and $^3J_{C,F}$ = 3.0 Hz, *i*-CO; OC_6F_5), 44.4 (ArCH) and 18.5 (CH_3CH); (Found M(^{35}Cl)$^+$, 350.0124; $C_{15}H_8ClF_5O_2$ requires M(^{35}Cl)$^+$, 350.0127).

4.4. Mutual kinetic resolution of pentafluorophenyl 2-phenylpropionate *rac*-3 using 4,5,5-triphenyl oxazolidin-2-one *rac*-6

n-BuLi (0.28 mL, 2.5 M in hexane, 0.70 mmol) was added to a stirred solution of 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (0.20 g, 0.63 mmol) in THF at −78 °C. After stirring for 1 h, a solution of pentafluorophenyl 2-phenylpropionate *rac*-**3** (0.20 g, 0.63 mmol) in THF (1 mL) was added. The resulting mixture was stirred for 2 h at −78 °C. The reaction mixture was quenched with water (10 mL). The organic layer was extracted with dichloromethane (3 × 10 mL), dried over $MgSO_4$ and evaporated under reduced pressure to give a mixture of diastereoisomeric oxazolidin-2-ones **7** [ratio 89:11: *syn*-:*anti*-]. The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the (2*RS*,4*SR*)-3-(2-phenylpropionyl)-4,5,5-triphenyl-oxazolidin-2-one *rac-syn*-**7** (0.15 g, 53%, 78% de) as a white powder; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.58; mp 160–165 °C; ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1704 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.63 (2H, br d, *J* 7.7, 2 × CH; Ph), 7.46–7.36 (4H, m, 4 × CH; Ph), 7.19 (2H, dd, *J* 5.0, and 2.0, 2 × CH; Ph), 7.11–7.07 (2H, m, 2 × CH; Ph), 7.01–6.86 (8H, m, 8 × CH; Ph), 6.65 (2H, br d, *J* 7.7, 2 × CH; Ph), 6.25 (1H, s, CHN), 4.98 (1H, q, *J* 7.0, PhC*H*CH_3) and 1.35 (3H, d, *J* 7.0, PhCHCH_3); δ_C (100 MHz; $CDCl_3$) 173.2 (NC=O), 152.0 (OC=O), 141.8, 138.0 and 135.0 (3 × *i*-C; 3 × Ph–oxazolidin-2-one), 139.5 (*i*-C; Ph), 128.9[2], 128.8[1], 128.4[2], 128.3[2], 127.9[3], 127.6[2], 127.5[1], 127.4[2], 127.0[1], 126.2[2] and 126.1[2] (20 × CH; 4 × Ph), 88.5 (CPh_2O), 66.0 (CHN), 44.0 (PhCHCH$_3$) and 19.0 (PhCH*C*H$_3$) (Found M$^+$, 447.1835; $C_{30}H_{25}NO_3$ requires M$^+$, 447.1829); *m*/*z* 447 (10%, M$^+$), 315 (5, M− Ph(CH_3)C=C=O$^+$), 256 (15, PhCHCPh_2^+), 183 (20, Ph_2C=OH$^+$), 105 (100, PhCH=NH$^+$) and 77 (20, Ph$^+$).

4.5. Mutual kinetic resolution of pentafluorophenyl 2-phenylbutanoate *rac*-8 using 4,5,5-triphenyl oxazolidin-2-one *rac*-6

In the same way as oxazolidin-2-one **7**, *n*-butyl lithium (0.14 mL, 2.5 M in hexane, 0.35 mmol), 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-phenylbutanoate *rac*-**8** (0.105 g, 0.32 mmol) gave a diastereoisomeric mixture of oxazolidin-2-ones *rac-syn*- and *rac-anti*-**9** (ratio 97:3: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the (2*RS*,4*SR*)-3-(2-phenylbutanoyl)-4,5,5-triphenyl-oxazolidin-2-one *rac-syn*-**9** (0.121 g, 83%, 94% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.61; mp 158–160 °C; ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1707 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.57 (2H, br d, *J* 7.5, 2 × CH; Ph), 7.41–7.30 (3H, m, 3 × CH; Ph), 7.16–7.05 (5H, m, 5 × CH; Ph), 6.94–6.79 (8H, m, 8 × CH; 3 × Ph), 6.57 (2H, br d, *J* 7.5, Ph), 6.20 (1H, s, CHN), 4.72 (1H, t, *J* 7.3, ArC*H*Et), 1.97–1.85 (1H, m, CH_AH$_B$CH$_3$), 1.69–1.57 (1H, m, CH$_A$H$_B$CH$_3$) and 0.70 (3H, t, *J* 7.3, CH_3CH$_A$H$_B$); δ_C (100 MHz; $CDCl_3$) 172.8 (NC=O), 152.1 (OC=O), 141.7, 138.0 and 135.0 (3 × *i*-C; 3 × Ph), 137.7 (*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), 51.1 (ArCHEt), 26.7 (CH_2CH_3) and 11.8 (CH_2CH_3) (Found MH$^+$, 462.2067; $C_{31}H_{28}NO_3$ requires MH$^+$, 462.2064).

4.6. Mutual kinetic resolution of pentafluorophenyl 2-phenyl-3-methylbutanoate *rac*-10 using 4,5,5-triphenyl oxazolidin-2-one *rac*-6

In the same way as oxazolidin-2-one **7**, *n*-butyl lithium (0.14 mL, 2.5 M in hexane, 0.35 mmol), 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate *rac*-**10** (0.11 g, 0.32 mmol) gave a diastereoisomeric mixture of oxazolidin-2-ones *rac-syn*- and *rac-anti*-**11** (ratio 78:22: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give an inseparable mixture of (2*RS*,4*SR*)-3-(2-phenyl-3-methylbutanoyl)-4,5,5-triphenyl-oxazolidin-2-one *rac-syn*-**11** and (2*RS*,4*RS*)-3-(2-phenyl-3-methylbutanoyl)-4,5,5-triphenyl-oxazolidin-2-one *rac-anti*-**11** (27 mg, 18%, ratio 78:22, 56% de in favour of *syn*-) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.71; mp 166–172 °C; ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1702 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.57 (2H, br d, *J* 7.7, 2 × CH; Ph)^{2s}, 7.40–7.30 (8H, m, 4 × CH; Ph)$^{3s+5a}$, 7.19–7.00 (10H, m, 10 × CH; Ph),$^{5s+5a}$ 6.95–6.70 (18H, m, 16 × CH; Ph),$^{8s+10a}$ 6.53 (2H, br d, *J* 7.7, 2 × CH; Ph),^{2s} 6.19 (1H, s, PhCHN),^{s} 5.98 (1H, s, PhCHN),^{a} 4.70 (1H, d, *J* 10.3, PhC*H**i*-Pr),^{a} 4.55 (1H, br d, *J* 10.1, PhC*H**i*-Pr),^{s} 2.35–2.19 (2H, m, 2 × C*H*$(CH_3)_2$),$^{s+a}$ 0.77 (6H, d, *J* 6.9, 2 × CH_3^ACHCH_3^B),$^{3s+3a}$ 0.58 (3H, d, *J* 6.9, CH_3^ACHCH_3^B)^{3a} and 0.55 (3H, d, *J* 6.9, CH_3^ACHCH_3^B)^{3s}; δ_C (100 MHz; $CDCl_3$) 173.7 (NC=O),^{a} 172.8 (NC=O),^{s} 152.6 (OC=O),^{a} 152.3 (OC=O),^{s} 141.6, 137.8, 136.9 and 134.9 (4 × *i*-C; 4 × Ph),^{4s} 141.3, 137.6, 136.6 and 136.0 (4 × *i*-C; 4 × Ph),^{4a} 129.3^{2s}, 128.9^{1s}, 128.8^{2s}, 128.2^{2s}, 127.9^{2s}, 127.7,^{1s} 127.5^{2s}, 127.4^{1s}, 127.1^{1s}, 127.0^{2s}, 126.3^{2s} and 126.4^{2s} (20 × CH; 4 × Ph),^{20s} 129.3,^{2a} 128.9,^{1a} 128.6^{2a}, 128.5^{1a}, 128.3^{2a}, 128.2^{2a}, 128.0^{1a}, 127.6,^{2a} 127.5^{2a}, 127.1,^{1a} 126.4^{2a} and 125.9^{2a} (20 × CH; 4 × Ph),^{20a} 88.7 (CPh_2O),^{1a} 88.6 (CPh_2O),^{1s} 66.6 (CHN),^{1a} 65.8 (CHN),^{1s} 56.7 (PhCH*i*-Pr),^{1s} 56.1 (PhCH*i*-Pr),^{1a} 31.6 (*C*H$(CH_3)_2$)^{1a}, 31.5 (*C*H$(CH_3)_2$)^{1s}, 21.4^{3a}, 21.3^{3s}, 20.0^{3a} and 20.0^{3s} (4 × CH_3)$^{6s+6a}$ (Found MNH_4^+, 493.2488; $C_{32}H_{33}N_2O_3$ requires MNH_4^+, 493.2486).

4.7. Mutual kinetic resolution of pentafluorophenyl 2-(4-methylphenyl)propionate *rac*-12 using 4,5,5-triphenyl oxazolidin-2-one *rac*-6

In the same way as oxazolidin-2-one **7**, *n*-butyl lithium (0.14 mL, 2.5 M in hexane, 0.35 mmol), 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-(4-methylphenyl)propionate *rac*-**12** (0.105 g, 0.32 mmol) gave a diastereoisomeric mixture of oxazolidin-2-ones *rac-syn*- and *rac-anti*-**13** (ratio 93:7: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the (2*RS*,4*SR*)-3-[2-(4-methylphenyl)propionyl]-4,5,5-triphenyl-oxazolidin-2-one *rac-syn*-**13** (88 mg, 60%, 86% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.57; mp 153–158 °C; ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1703 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.64 (2H, br d, *J* 7.7, 2 × CH; Ar), 7.48–7.38 (3H, m, 3 × CH; Ph), 7.04–6.89 (12H, m, 12 × CH; Ph), 6.69 (2H,

br d, *J* 7.7, Ar), 6.26 (1H, s, CHN), 4.95 (1H, q, *J* 7.0, ArC*H*CH_3), 2.32 (3H, s, CH_3Ar) and 1.35 (3H, d, *J* 7.0, ArCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 173.4 (NC=O), 152.0 (OC=O), 141.8, 138.0 and 135.1 (3 × *i*-C; 3 × Ph), 136.6 and 136.5 (2 × *i*-C; 2 × Ar), 129.1[2], 128.9[2], 128.9[1], 128.1[2], 127.9[3], 127.5[2], 127.4[2], 127.3[1], 126.3[2] and 126.1[2] (19 × CH; 3 × Ph and Ar), 88.5 (CPh_2O), 66.0 (CHN), 44.6 (ArC*H*CH_3), 21.1 (CH_3Ar) and 19.1 (ArCH*C*H_3) (Found MH^+, 462.2062; $C_{31}H_{28}NO_3$ requires MH^+, 462.2064).

4.8. Mutual kinetic resolution of pentafluorophenyl 2-(4-chlorophenyl)propionate *rac*-14 using 4,5,5-triphenyl oxazolidin-2-one *rac*-6

In the same way as oxazolidin-2-one **7**, *n*-butyl lithium (0.14 mL, 2.5 M in hexane, 0.35 mmol), 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propionate *rac*-**14** (0.11 g, 0.32 mmol) gave a diastereoisomeric mixture of oxazolidin-2-ones *rac-syn-* and *rac-anti-***15** (ratio 92:8: *syn-*:*anti-*). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the (2*RS*,4*SR*)-3-[2-(4-chlorophenyl)propionyl]-4,5,5-triphenyl-oxazolidin-2-one *rac-syn-***15** (88 mg, 58%, 84% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.68; mp 135–138 °C; ν_{max} ($CHCl_3$) cm^{-1} 1781 (OC=O) and 1708 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.58 (2H, br d, *J* 7.7, 2 × CH; Ph), 7.40–7.30 (3H, m, 3 × CH; Ph), 7.10 (2H, dt, *J* 8.2 and 1.9, 2 × CH; Ar), 6.94 (2H, dt, *J* 8.2 and 1.9, 2 × CH; Ar), 6.99–6.82 (8H, m, 8 × CH; Ph), 6.60 (2H, br d, *J* 7.2, Ph), 6.19 (1H, s, CHN), 4.90 (1H, q, *J* 7.0, ArC*H*CH_3), 1.28 (3H, d, *J* 7.0, ArCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 172.8 (NC=O), 151.9 (OC=O), 141.8, 138.0, 137.9, 134.9 and 132.8 (5 × *i*-C; 3 × Ph and 2 × Ar), 129.6[2], 128.9[2], 128.9[1], 128.5[2], 128.0[1], 127.9[2], 127.6[2], 127.4[1], 127.4[2], 126.1[2] and 126.0[2] (19 × CH; 3 × Ph and Ar), 88.6 (CPh_2O), 65.9 (CHN), 43.3 (ArC*H*CH$_3$) and 18.9 (ArCH*C*H_3) (Found $M(^{35}Cl)NH_4^+$, 499.1786; $C_{30}H_{28}ClN_2O_3$ requires $M(^{35}Cl)NH_4^+$, 499.1783).

4.9. Mutual kinetic resolution of pentafluorophenyl 2-(4-isobutylphenyl)propionate *rac*-16 using 4,5,5-triphenyl oxazolidin-2-one *rac*-6

In the same way as oxazolidin-2-one **7**, *n*-butyl lithium (0.14 mL, 2.5 M in hexane, 0.35 mmol), 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propionate *rac*-**16** (0.118 g, 0.32 mmol), gave a diastereoisomeric mixture of oxazolidin-2-ones *rac-syn-* and *rac-anti-***17** (ratio 95:5: *syn-*:*anti-*). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the (2*RS*,4*SR*)-3-[2-(4-isobutylphenyl)propionyl]-4,5,5-triphenyl-oxazolidin-2-one *rac-syn-***17** (0.125 g, 77%, 90% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.73; mp 158–162 °C; ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1704 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.66 (2H, br d, *J* 7.3, 2 × CH; Ar), 7.49–7.38 (3H, m, 3 × CH; Ph), 7.06–6.87 (12H, m, 12 × CH; 3 × Ph), 6.66 (2H, br d, *J* 7.3, Ar), 6.28 (1H, s, CHN), 5.00 (1H, q, *J* 7.0, ArC*H*CH_3), 2.44 (2H, dd, *J* 7.2 and 1.6 CH_2Ar), 1.91–1.79 (1H, m (appears as a septet $J \sim 6.8$), $(CH_3)_2CH$), 1.37 (3H, d, *J* 7.0, ArCHC*H*$_3$), 0.92 (3H, d, *J* 6.6, CH_3^ACHCH_3^B) and 0.91 (3H, d, *J* 6.6, CH_3^ACHCH_3^B); δ_C (100 MHz; $CDCl_3$) 173.5 (NC=O), 152.0 (OC=O), 141.8, 138.1 and 135.0 (3 × *i*-C; 3 × Ph), 140.5 (*i*-*C*H_2; Ar), 136.6 (*i*-C; Ar), 129.1[2], 128.9[2], 128.9[1], 128.0[2], 127.9[1], 127.8[2], 127.5[2], 127.4[3], 126.3[2], and 126.2[2] (19 × CH; 19 × Ph and Ar), 88.5 (CPh_2O), 66.0 (CHN), 45.0 ($(CH_3)_2$CH), 43.4 (ArC*H*CH_3), 30.2 (CH_2Ar), 22.4 (CH_3^ACHCH_3^B), 22.3 (CH_3^ACHCH_3^B) and 18.9 (ArCH*C*H_3) (Found MNH_4^+, 521.2796; $C_{34}H_{37}N_2O_3$ requires MNH_4^+, 521.2799).

4.10. Parallel kinetic resolution of pentafluorophenyl 2-phenylpropionate *rac*-3 using a *quasi*-enantiomeric combination of 4-phenyloxazolidin-2-one (*R*)-1 and 4,5,5-triphenyl oxazolidin-2-one (*S*)-6

In the same way as oxazolidin-2-one *rac-syn-***7**, *n*-butyl lithium (0.28 mL, 2.5 M in hexane, 0.70 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (50 mg, 0.32 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-phenylpropionate *rac*-**3** (0.20 g, 0.63 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-**4** (ratio 97:3: *syn-*:*anti-*) and (*R*,*S*)-**7** (ratio 98:2: *syn-*:*anti-*). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the (2*S*,4*R*)-3-(2-phenylpropionyl)-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn-***4** (54 mg, 58%) as a white solid; mp 140–142 °C; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.23; ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1701 (NC=O); $[\alpha]_D^{20} = +92.5$ (*c* 4.9, $CHCl_3$); {lit.[18] $[\alpha]_D^{20} = +88.5$ (*c* 4.0, $CHCl_3$)}; δ_H (400 MHz; $CDCl_3$) 7.29–7.21 (10H, m, 10 × CH; 2 × Ph), 5.45 (1H, dd *J* 9.0 and 5.1, CHN), 5.09 (1H, q, *J* 6.9, PhC*H*CH_3), 4.63 (1H, t, *J* 9.0, CH_AH_BO), 4.08 (1H, dd, *J* 9.0 and 5.1, CH_AH_BO) and 1.39 (3H, d, *J* 6.9, PhCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 173.7 (NC=O), 153.2 (OC=O), 139.9 (*i*-C; Ph^A), 138.3 (*i*-C; Ph^B), 128.9,[2] 128.5,[3] 128.2,[2] 127.1[1] and 125.9[2] (10 × CH; 2 × Ph), 69.6 (CH_2O), 57.9 (CHN), 43.9 (PhC*H*CH$_3$) and 18.6 (PhCH*C*H_3) (Found MH^+, 296.1286; $C_{15}H_{18}NO_3^+$ requires 296.1287); and (2*R*,4*S*)-3-(2-phenylpropionyl)-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn-***7** (0.85 mg, 60%, 96% de) as a white powder; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 154–156 °C; $[\alpha]_D^{20} = -255.1$ (*c* 3.4, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (NC=O) and 1704 (OC=O); δ_H (400 MHz; $CDCl_3$) 7.63 (2H, br d, *J* 7.7, 2 × CH; Ph), 7.46–7.36 (4H, m, 4 × CH; Ph), 7.19 (2H, br dd, *J* 5.0, and 2.0, 2 × CH; Ph), 7.11–7.07 (2H, m, 2 × CH; Ph), 7.01–6.86 (8H, m, 8 × CH; Ph), 6.65 (2H, br d, *J* 7.7, 2 × CH; Ph), 6.25 (1H, s, CHN), 4.98 (1H, q, *J* 7.0, PhC*H*CH_3) and 1.35 (3H, d, *J* 7.0, PhCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 173.2 (OC=O), 152.0 (NC=O), 141.8, 138.0 and 135.0 (3 × *i*-C; 3 × Ph–oxazolidin-2-one), 139.5 (*i*-C; Ph), 128.9[2], 128.8[1], 128.4[2], 128.3[2], 127.9[3], 127.6[2], 127.5[1], 127.4[2], 127.0[1], 126.2[2] and 126.1[2] (20 × CH; 4 × Ph), 88.5 (CPh_2O), 66.0 (CHN), 44.0 (PhC*H*CH$_3$) and 19.0 (PhCH*C*H_3) (Found MH^+, 448.1908; $C_{30}H_{26}NO_3$ requires 448.1907); *m*/*z* 447 (10%, M^+), 315 (5, $M^+ - Ph(CH_3)C{=}C{=}O$), 256 (15, PhCHCPh_2^+), 183 (20, $Ph_2C{=}OH^+$), 105 (100, PhCH=NH^+) and 77 (20, Ph^+).

4.11. Parallel kinetic resolution of pentafluorophenyl 2-phenylbutanoate *rac*-8 using a *quasi*-enantiomeric combination of 4-phenyloxazolidin-2-one (*R*)-1 and 4,5,5-triphenyl oxazolidin-2-one (*S*)-6

In the same way as oxazolidin-2-one *rac-syn-***7**, *n*-butyl lithium (0.28 mL, 2.5 M in hexane, 0.70 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (50 mg, 0.32 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-phenylbutanoate *rac*-**8** (0.209 g, 0.63 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-**18** (ratio 99:1: *syn-*:*anti-*) and (*R*,*S*)-**9** (ratio 97:3: *syn-*:*anti-*). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*S*,4*R*)-3-(2-phenylbutanoyl)-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn-***18** (62 mg, 63%) as a white solid; mp 82–84 °C; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.33; $[\alpha]_D^{20} = +77.4$ (*c* 4.0, $CHCl_3$); ν_{max} (CH_2Cl_2) cm^{-1} 1772 (OC=O) and 1700 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.17–7.09 (6H, m, 6 × CH; Ph), 7.04–7.02 (2H, m, 2 × CH; Ph), 6.81–6.79 (2H, m, 2 × CH; Ph), 5.38 (1H, dd, *J* 8.8 and 5.0, CHN), 4.82 (1H, t, *J* 7.5, PhC*H*Et), 4.55 (1H, t, *J* 8.8, CH_AH_BO), 3.98

(1H, dd, *J* 8.8 and 5.0, CH_AH_BO), 2.01–1.90 (1H, ddq, *J* 13.5, 7.3 and 7.5, $CH_AH_BCH_3$), 1.68–1.57 (1H, ddq, *J* 13.5, 7.3 and 7.5, $CH_AH_BCH_3$) and 0.84 (3H, t, *J* 7.5, CH_3CH_2); δ_C (100 MHz; $CDCl_3$) 173.0 (NC=O), 153.1 (OC=O), 138.2 (*i*-C; Ph^A), 138.0 (*i*-CC; Ph^B), 128.8[2], 128.7[2], 128.4[1], 128.3[2], 127.1[1] and 125.6[2] (10 × CH; Ph^A and Ph^B), 69.4 (CH_2O), 57.7 (CHN), 51.1 (PhCH), 26.2 (CH_2CH_3) and 11.9 (CH_2CH_3) (Found MH^+, 310.1437; $C_{19}H_{20}NO_3$ requires MH^+, 310.1443); and (2*R*,4*S*)-3-(2-phenylbutanoyl)-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-**9** (86 mg, 59%, 94% de) as a white powder; R_f [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 150–153 °C; $[\alpha]_D^{20} = -195.2$ (*c* 3.4, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (NC=O) and 1707 (OC=O); δ_H (400 MHz; $CDCl_3$) 7.57 (2H, br d, *J* 7.5, 2 × CH; Ph), 7.41–7.30 (3H, m, 3 × CH; Ph), 7.16–7.05 (5H, m, 5 × CH; Ph), 6.94–6.79 (8H, m, 8 × CH; 3 × Ph), 6.57 (2H, br d, *J* 7.5, Ph), 6.20 (1H, s, CHN), 4.72 (1H, t, *J* 7.3, ArC*H*Et), 19.7–1.85 (1H, m, $CH_AH_BCH_3$), 1.69–1.57 (1H, m, $CH_AH_BCH_3$) and 0.70 (3H, t, *J* 7.3, $CH_3CH_AH_B$); δ_C (100 MHz; $CDCl_3$) 172.8 (OC=O), 152.1 (NC=O), 141.7, 138.0 and 135.0 (3 × *i*-C; 3 × Ph), 137.7 (*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), 51.1 (ArCHEt), 26.7 (CH_2CH_3) and 11.8 (CH_2CH_3) (Found MH^+, 462.2062; $C_{31}H_{28}NO_3$ requires MH^+, 462.2064); *m*/*z* 416 (20%, M^+), 315 (10, MH−ArCHCH$_3$C=NH$^+$), 256 (40, PhCHCPh$_2^+$), 183 (50, Ph$_2$C=OH$^+$), 146 (40, ArCHCH$_3$C=NH$^+$), 119 (70, PhCHEt$^+$), 91 (100, PhCH$_2^+$), and 77 (20, Ph$^+$).

4.12. Parallel kinetic resolution of pentafluorophenyl 2-phenyl-3-methylbutanoate *rac*-10 using a *quasi*-enantiomeric combination of 4-phenyloxazolidin-2-one (*R*)-1 and 4,5,5-triphenyl oxazolidin-2-one (*S*)-6

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (0.28 mL, 2.5 M in hexane, 0.70 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (50 mg, 0.32 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate *rac*-**10** (0.22 g, 0.63 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-**19** (ratio 77:23: *syn*-:*anti*-) and (*R*,*S*)-**11** (ratio 88:12: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*S*,4*R*)-3-(2-phenyl-3-methylbutanoyl)-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-**19** (80 mg, 79%, 54% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.40; mp 80–92 °C; $[\alpha]_D^{25} = +1.3$ (*c* 3.0, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1781 (OC=O) and 1780 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.40–7.10 (18H, m, 8 × CH; 2 × Ph^s and 10 × CH; 2 × Ph^a), 6.77 (2H, d, *J* 7.3, 2 × CH; Ph^s), 5.46 (1H, dd, *J* 8.9 and 4.7, PhCHN^s), 5.33 (1H, dd, *J* 8.8 and 3.7, PhCHN^a), 4.76 (1H, d, *J* 10.7, PhCH*i*-Pr^a), 4.66 (1H, d, *J* 10.7, PhCH*i*-Pr^s), 4.64 (1H, t, *J* 8.8, $CH_AH_BO^s$), 4.48 (1H, t, *J* 8.8, $CH_AH_BO^a$), 4.21 (1H, dd, *J* 8.8 and 3.7, CH_AH_BO), 4.05 (1H, dd, *J* 8.8 and 4.7, CH_AH_BO), 2.40–2.30 (2H, m, 2 × C*H*(CH$_3$)$_2$),$^{s+a}$ 1.04 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$),^{s} 0.76 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$)^a, 0.58 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$)^s and 0.55 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$)^a; δ_C (100 MHz; $CDCl_3$) 173.8 (NC=O),^{a} 172.9 (NC=O),^{s} 153.4 (OC=O),^{a} 153.2 (OC=O),^{s} 139.4 (*i*-C; Ph),^{a} 138.2 (*i*-C; Ph),^{s} 137.9 (*i*-C; Ph),^{a} 137.1 (*i*-C; Ph),^{s} 129.3,[2a] 128.9,[1a] 128.6,[1a] 128.3,[2a] 127.3[2a] and 125.8[2a] (10 × CH; 2 × Ph),[10a] 129.2,[2s] 128.8,[2s] 128.3,[2s] 128.3,[1s] 127.2[1s] and 125.3[2s] (10 × CH; 2 × Ph),[10s] 69.4 (CH_2O),^{s} 69.3 (CH_2O),^{a} 57.9 (PhCHN),^{a} 57.6 (PhCHN),^{s} 56.9 (PhCH*i*-Pr),^{s} 55.9 (PhCH*i*-Pr),^{a} 32.8 (*C*H(CH$_3$)$_2$),^{a} 30.7 (*C*H(CH$_3$)$_2$),^{s} 21.6 ($CH_3^ACHCH_3^B$),^{s} 21.2 ($CH_3^ACHCH_3^B$),^{a} 20.1 ($CH_3^ACHCH_3^B$),^{a} and 20.0 ($CH_3^ACHCH_3^B$)^s (Found MNH_4^+, 341.1864; $C_{20}H_{25}N_2O_3^+$ requires MNH_4^+, 341.1860); and (2*R*,4*S*)-3-(2-phenyl-3-methylbutanoyl)-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-**11** (65 mg, 43%, 76% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.71; mp 170–178 °C; $[\alpha]_D^{25} = -270.9$ (*c* 2.6, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1711 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.57 (2H, br d, *J* 7.7, 2 × CH; Ph)^{2s}, 7.40–7.30 (8H, m, 4 × CH; Ph)$^{3s+5a}$, 7.19–7.00 (10H, m, 10 × CH; Ph),$^{5s+5a}$ 6.95–6.70 (18H, m, 16 × CH; Ph),$^{8s+10a}$ 6.53 (2H, br d, *J* 7.7, 2 × CH; Ph),^{2s} 6.19 (1H, s, PhCHN),^{s} 5.98 (1H, s, PhCHN),^{a} 4.70 (1H, d, *J* 10.3, PhC*H*i-Pr),^{a} 4.55 (1H, br d, *J* 10.1, PhC*H*i-Pr),^{s} 2.35–2.19 (2H, m, 2 × C*H*(CH$_3$)$_2$),$^{s+a}$ 0.77 (6H, d, *J* 6.9, 2 × $CH_3^ACHCH_3^B$),$^{3s+3a}$ 0.58 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$)^{3a} and 0.55 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$)^{3s}; δ_C (100 MHz; $CDCl_3$) 173.7 (NC=O),^{a} 172.8 (NC=O),^{s} 152.6 (OC=O),^{a} 152.3 (OC=O),^{s} 141.6, 137.8, 136.9 and 134.9 (4 × *i*-C; 4 × Ph),^{4s} 141.3, 137.6, 136.6 and 136.0 (4 × *i*-C; 4 × Ph),^{4a} 129.3[2s], 128.9[1s], 128.8[2s], 128.2[2s], 127.9[2s], 127.7,[1s] 127.5[2s], 127.4[1s], 127.1[1s], 127.0[2s], 126.3[2s] and 126.4[2s] (20 × CH; 4 × Ph),[20s] 129.3,[2a] 128.9,[1a] 128.6[2a], 128.5[1a], 128.3[2a], 128.2[2a], 128.0[1a], 127.6,[2a] 127.5[2a], 127.1,[1a] 126.4[2a] and 125.9[2a] (20 × CH; 4 × Ph),[20a] 88.7 (CPh_2O),[1a] 88.6 (CPh_2O),[1s] 66.6 (CHN),[1a] 65.8 (CHN),[1s] 56.7 (PhCH*i*-Pr),[1s] 56.1 (PhCH*i*-Pr),[1a] 31.6 (CH(CH$_3$)$_2$)[1a], 31.5 (*C*H(CH$_3$)$_2$)[1s], 21.4[3a], 21.3[3s], 20.0[3a] and 20.0[3s] (4 × CH_3)[6s+6a] (Found MNH_4^+, 493.2490; $C_{32}H_{33}N_2O_3$ requires MNH_4^+, 493.2486).

4.13. Parallel kinetic resolution of pentafluorophenyl 2-(4-methylphenyl)propionate *rac*-12 using a *quasi*-enantiomeric combination of 4-phenyloxazolidin-2-one (*R*)-1 and 4,5,5-triphenyl oxazolidin-2-one (*S*)-6

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (0.28 mL, 2.5 M in hexane, 0.70 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (50 mg, 0.32 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-(4-methylphenyl)propionate *rac*-**12** (0.209 g, 0.63 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-**20** (ratio 99:1: *syn*-:*anti*-) and (*R*,*S*)-**13** (ratio 98:2: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*S*,4*R*)-3-[(4-methylphenyl)propionyl]-4-phenyl-oxazolidin-2-one (*S*,*R*)-*syn*-**20** (44 mg, 45%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.36; mp 105–110 °C {for (*R*,*S*)-*syn*-**20**; mp 105–110 °C}; ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1700 (NC=O); $[\alpha]_D^{20} = +121.6$ (*c* 0.6, $CHCl_3$) {for (*R*,*S*)-*syn*-**20**; $[\alpha]_D^{20} = -116.5$ (*c* 0.8, $CHCl_3$)}; δ_H (400 MHz, $CDCl_3$) 7.21–7.12 (3H, m, 3 × CH; Ph), 6.96 (2H, d, *J* 8.2, 2 × CH; Ar), 6.90 (2H, d, *J* 8.2, 2 × CH; Ar), 6.86 (2H, d, *J* 6.9, 2 × CH; Ph), 5.36 (1H, dd, *J* 9.1 and 5.1, CHN), 5.01 (1H, q, *J* 6.9, ArC*H*CH$_3$), 4.54 (1H, t, *J* 9.1, CH_AH_BO), 3.99 (1H, dd, *J* 9.1 and 5.1, CH_AH_BO), 2.24 (3H, s, CH_3; Ar) and 1.32 (3H, d, *J* 6.9, ArCHC*H*$_3$); δ_C (100 MHz, $CDCl_3$) 173.5 (NC=O), 154.9 (OC=O), 138.4 (*i*-CMe; Ar), 136.8 (*i*-C; Ar), 136.4 (*i*-C; Ph), 129.1[2], 128.6[2], 128.4[1], 127.6[2] and 125.7[2] (9 × CH; Ph and Ar), 69.6 (CH_2O), 57.8 (CHN), 43.2 (ArCHCH$_3$), 21.0 (CH_3; Ar) and 18.7 (ArCHCH$_3$) (Found MNH_4^+, 327.1701; $C_{19}H_{23}N_2O_3^+$ requires MNH_4^+, 327.1709); and (2*R*,4*S*)-3-[2-(4-methylphenyl)propionyl]-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-**13** (75 mg, 51%, 96% de) as a white powder; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.58; mp 119–121 °C; $[\alpha]_D^{20} = -258.6$ (*c* 2.4, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1703 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.64 (2H, br d, *J* 7.7, 2 × CH; Ar), 7.48–7.38 (3H, m, 3 × CH; Ph), 7.04–6.89 (12H, m, 12 × CH; Ph), 6.69 (2H, br d, *J* 7.7, Ar), 6.26 (1H, s, CHN), 4.95 (1H, q, *J* 7.0, ArC*H*CH$_3$), 2.32 (3H, s, CH_3Ar) and 1.35 (3H, d, *J* 7.0, ArCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 173.4 (NC=O), 152.0 (OC=O), 141.8, 138.0 and 135.1 (3 × *i*-C; 3 × Ph), 136.6 and 136.5 (2 × *i*-C; 3 × Ar), 129.1[2], 128.9[2], 128.9[1], 128.1[2], 127.9[3], 127.5[2], 127.4[2], 127.3[1], 126.3[2] and 126.1[2] (19 × CH; 3 × Ph and Ar), 88.5 (CPh_2O), 66.0 (CHN), 44.6 (ArCHCH$_3$), 21.1 (CH_3Ar) and 19.1 (ArCHC*H*$_3$) (Found MH^+, 462.2068; $C_{31}H_{28}NO_3$ requires MH^+, 462.2064); *m*/*z* 461 (15%, M^+), 315 (5, MH^+−ArCHCH$_3$CO), 256 (40, PhCHCPh$_2^+$), 183 (10, Ph$_2$ C=OH$^+$), 146 (80, ArCHCH$_3$C=NH$^+$), 119 (100, ArCHCH$_3^+$) and 77 (20, Ph$^+$).

4.14. Parallel kinetic resolution of pentafluorophenyl 2-(4-chlorophenyl)propionate *rac*-14 using a *quasi*-enantiomeric combination of 4-phenyloxazolidin-2-one (*R*)-1 and 4,5,5-triphenyl oxazolidin-2-one (*S*)-6

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (0.28 mL, 2.5 M in hexane, 0.70 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (50 mg, 0.32 mmol), 4,5,5-triphenyl oxazolidin-2-one (*S*)-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propionate *rac*-**14** (0.22 g, 0.63 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-**21** (ratio 93:7: *syn*-:*anti*-) and (*R,S*)-**15** (ratio 96:4: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*S*,4*R*)-3-[(4-chlorophenyl)propionyl]-4-phenyl-oxazolidin-2-one (*S,R*)-*syn*-**21** (74 mg, 71%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.27; mp 142–145 °C {for (*R,S*)-*syn*-**21**; mp 142–144 °C}; ν_{max} ($CHCl_3$) cm^{-1} 1782 (OC=O) and 1700 (NC=O); $[\alpha]_D^{20} = +144.4$ (*c* 1.6, $CHCl_3$) {for (*R,S*)-*syn*-**21**; $[\alpha]_D^{20} = -142.4$ (*c* 1.5, $CHCl_3$)}; δ_H (400 MHz, $CDCl_3$) 7.32–7.22 (3H, m, 3 × CH; Ph), 7.18 (2H, dt, *J* 8.5 and 2.2, 2 × CH; Ar), 7.01 (2H, dt, *J* 8.5 and 2.2, 2 × CH; Ar), 6.95 (2H, dt, *J* 6.8 and 1.5, 2 × CH; Ph), 5.45 (1H, dd, *J* 9.0 and 4.8, CHN), 5.06 (1H, q, *J* 6.8, ArC*H*CH_3), 4.65 (1H, t, *J* 9.0, CH_AH_BO), 4.13 (1H, dd, *J* 9.0 and 4.8, CH_AH_BO) and 1.37 (3H, d, *J* 6.8, ArCHCH_3); δ_C (100 MHz, $CDCl_3$) 173.8 (NC=O), 152.8 (OC=O), 138.2 (*i*-CC; Ar), 133.2 (*i*-C; Ar), 132.8 (*i*-CCl; Ar), 129.7[2], 128.8[2], 128.6[3] and 125.6[2] (9 × CH; 2 × Ar), 69.4 (CH_2O), 57.9 (CHN), 43.8 (ArCHCH_3) and 18.9 (ArCH*C*H_3) (Found M(^{35}Cl)NH_4^+, 347.1154; $C_{18}H_{20}ClN_2O_3$ requires M(^{35}Cl)NH_4^+, 347.1157); and (2*R*,4*S*)-3-[(4-chlorophenyl)propionyl]-4,5,5-triphenyl oxazolidin-2-one (*R,S*)-*syn*-**15** (91 mg, 60%, 92% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.60; mp 100–102 °C; $[\alpha]_D^{20} = -296.2$ (*c* 3.4, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1706 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.58 (2H, br d, *J* 7.7, 2 × CH; Ph), 7.40–7.30 (3H, m, 3 × CH; Ph), 7.10 (2H, dt, *J* 8.2 and 1.9, 2 × CH; Ar), 6.94 (2H, dt, *J* 8.2 and 1.9, 2 × CH; Ar), 6.99–6.82 (8H, m, 8 × CH; Ph), 6.60 (2H, br d, *J* 7.2, Ph), 6.19 (1H, s, CHN), 4.90 (1H, q, *J* 7.0, ArC*H*CH_3), 1.28 (3H, d, *J* 7.0, ArCHCH_3); δ_C (100 MHz; $CDCl_3$) 172.8 (NC=O), 151.9 (OC=O), 141.8, 138.0, 137.9, 134.9 and 132.8 (5 × *i*-C; 3 × Ph and 2 × Ar), 129.6[2], 128.9[2], 128.9[1], 128.5[2], 128.0[1], 127.9[2], 127.6[2], 127.4[1], 127.4[2], 126.1[2] and 126.0[2] (19 × CH; 3 × Ph and Ar), 88.6 (CPh_2O), 65.9 (CHN), 43.3 (ArCHCH_3) and 18.9 (ArCH*C*H_3) (Found M(^{35}Cl)NH_4^+, 499.1786; $C_{30}H_{28}ClN_2O_3$ requires M(^{35}Cl)NH_4^+, 499.1783) (Found M(^{35}Cl)H^+, 482.1517; $C_{30}H_{25}ClNO_3$ requires M(^{35}Cl)H^+, 482.1525).

4.15. Parallel kinetic resolution of pentafluorophenyl 2-(4-isobutylphenyl)propionate *rac*-16 using a *quasi*-enantiomeric combination of 4-phenyloxazolidin-2-one (*R*)-1 and 4,5,5-triphenyl oxazolidin-2-one (*S*)-6

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (0.28 mL, 2.5 M in hexane, 0.70 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (50 mg, 0.32 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** (0.10 g, 0.32 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propionate *rac*-**16** (0.235 g, 0.63 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-**22** (ratio 97:3: *syn*-:*anti*-) and (*R,S*)-**17** (ratio 98:2: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*S*,4*R*)-3-[2-(4-isobutylphenyl)propionyl]-4-phenyl-oxazolidin-2-one (*S,R*)-*syn*-**22** (67 mg, 60%) as a white solid; mp 86–88 °C; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.39; $[\alpha]_D^{25} = +118.7$ (*c* 6.0, $CHCl_3$) {for (*R,S*)-*syn*-**22**; lit. $[\alpha]_D^{25} = -114.6$ (*c* 4.2, $CHCl_3$);[19] ν_{max} ($CHCl_3$) cm^{-1} 1779 (OC=O) and 1705 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.28–7.15 (3H, m, 3 × CH; Ph), 7.00 (4H, m, 4 × CH, Ph and Ar), 6.90 (2H, dt, *J* 7.9 and 1.9, 2 × CH; Ar), 5.44 (1H, dd *J* 9.2 and 5.2, CHN), 5.09 (1H, q, *J* 6.9, ArC*H*CH_3), 4.63 (1H, t, *J* 9.0, CH_AH_BO), 4.06 (1H, dd, *J* 9.0 and 5.2, CH_AH_BO), 2.43 (2H, d, *J* 7.4, CH_2Ar), 1.89–1.79 (1H, m, (C*H*$(CH_3)_2$), 1.38 (3H, d, *J* 6.9, ArCHCH_3), 0.90 (3H, d, *J* 6.6, CH_3^ACHCH_3^B) and 0.89 (3H, d, *J* 6.6, CH_3^ACHCH_3^B); δ_C (100.6 MHz; $CDCl_3$) 174.3 (NC=O), 153.3 (OC=O), 140.7 (*i*-C; Ar), 139.4 (*i*-C; Ar), 137.4 (*i*-C; Ph), 129.3[2] and 127.0[2] (4 × CH; Ar), 129.2,[1] 128.7[2] and 125.8[2] (5 × CH; Ph), 69.7 (CH_2O), 58.1 (CHN), 45.1 (C*H*$(CH_3)_2$), 42.7 (ArC*H*CH_3), 30.2 (CH_2Ar), 22.4[2] ($(CH_3)_2CH$) and 19.4 (CH_3CH_2) (Found MH^+, 352.1909; $C_{22}H_{26}NO_3$ requires MH^+, 352.1907); *m*/*z* 351.1 (10% M^+), 188.1 (10, Ar(CH_3)C=C=O^+), 161.1 (10, Ar^+CHCH_3), 145.1 (100, $ArCH_2^+$) and 77.1 (10, Ph^+) (Found MNH_4^+, 369.2171; $C_{22}H_{29}N_2O_3$ requires MNH_4^+, 369.2173); and (2*R*,4*S*)-3-[(4-isobutylphenyl)propionyl]-4,5,5-triphenyl-oxazolidin-2-one (*R,S*)-*syn*-**17** (88 g, 55%, 96% de) as a white powder; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 62–64 °C; $[\alpha]_D^{25} = -306.7$ (*c* 4.4, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1704 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.66 (2H, br d, *J* 7.3, 2 × CH; Ar), 7.49–7.38 (3H, m, 3 × CH; Ph), 7.06–6.87 (12H, m, 12 × CH; 3 × Ph), 6.66 (2H, br d, *J* 7.3, Ar), 6.28 (1H, s, CHN), 5.00 (1H, q, *J* 7.0, ArC*H*CH_3), 2.44 (2H, dd, *J* 7.2 and 1.6 CH_2Ar), 1.91–1.79 (1H, m (appears as a septet *J* ~ 6.8), $(CH_3)_2$C*H*), 1.37 (3H, d, *J* 7.0, ArCHCH_3), 0.92 (3H, d, *J* 6.6, CH_3^ACHCH_3^B) and 0.91 (3H, d, *J* 6.6, CH_3^ACHCH_3^B); δ_C (100 MHz; $CDCl_3$) 173.5 (NC=O), 152.0 (OC=O), 141.8, 138.1 and 135.0 (3 × *i*-C; 3 × Ph), 140.5 (*i*-CCH_2; Ar), 136.6 (*i*-C; Ar), 129.1[2], 128.9[2], 128.9[1], 128.0[2], 127.9[1], 127.8[2], 127.5[2], 127.4[3], 126.3[2], and 126.2[2] (19 × CH; 19 × Ph and Ar), 88.5 (CPh_2O), 66.0 (CHN), 45.0 ($(CH_3)_2CH$), 43.4 (ArC*H*CH_3), 30.2 (CH_2Ar), 22.4 (CH_3^ACHCH_3^B), 22.3 (CH_3^AC*H*CH_3^B) and 18.9 (ArCH*C*H_3) (Found MNH_4^+, 521.2800; $C_{34}H_{37}N_2O_3$ requires MNH_4^+, 521.2799); *m*/*z* 503 (40%, M^+), 459 (10, M^+-CO_2), 315 (10, MH−ArCHCH_3C=NH^+), 256 (90, PhCHCPh_2^+), 188 (100, ArCH-CH_3C=NH^+), 161 (90, ArCHCH_3^+) and 77 (20, Ph^+).

4.16. Parallel kinetic resolution of 4,5,5-triphenyl oxazolidin-2-one *rac*-6 using a *quasi*-enantiomeric combination of pentafluorophenyl 2-(6-methoxynaphthalen-2-yl)propionate (*S*)-23 and pentafluorophenyl 2-phenylbutanoate (*R*)-8

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (60 μL, 2.5 M in hexane, 0.15 mmol), 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (42 mg, 0.13 mmol), pentafluorophenyl 2-(6-methoxynaphthalen-2-yl)propionate (*S*)-**23** (26 mg, 67 μmol) and pentafluorophenyl 2-phenylbutanoate (*R*)-**8** (22 mg, 67 μmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S,R*)-**24** (ratio 96:4: *syn*-:*anti*-) and (*R,S*)-**9** (ratio 96:4: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*S*,4*R*)-3-[2-(6-methoxynaphth-2-yl)-propionyl]-4,5,5-triphenyl-oxazolidin-2-one (*S,R*)-*syn*-**24** (15 mg, 43%, 92% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.53; $[\alpha]_D^{25} = +302.5$ (*c* 1.2, $CHCl_3$) mp 106–110 °C; ν_{max} ($CHCl_3$) cm^{-1} 1781 (OC=O) and 1710 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.60–7.51 (3H, m, 3 × CH; Ar and Ph), 7.41–7.33 (4H, m, 4 × CH; Ar and/or Ph), 7.23 (1H, br s, CH; Ar), 7.20 (1H, dd, *J* 8.4 and 1.8, CH; Ar and/or Ph), 7.05–7.01 (2H, m, 2 × CH; Ar and/or Ph), 6.90 (1H, d, *J* 7.9, CH; Ar or Ph), 6.88–6.81 (5H, m, 5 × CH; Ar and Ph), 6.70 (2H, t, *J* 7.9, 2 × CH; Ar), 6.58 (2H, d, *J* 7.3, 2 × CH; Ar), 6.22 (1H, s, PhCH), 5.05 (1H, br q, *J* 7.0, ArCHCH_3), 3.85 (3H, s, CH_3O) and 1.36 (3H, d, *J* 7.0, ArCHCH_3); δ_C (100 MHz; $CDCl_3$) 173.2 (NC=O), 157.6 (*i*-CO; Ar), 151.8 (OC=O), 141.8, 138.0, 135.0, 134.9 133.6 and 128.7 (6 × *i*-C; Ar and Ph), 129.4, 127.0, 126.3, 126.2, 118.6 and 105.5 (6 × CH; Ar), 128.9,[2] 128.8,[1] 127.9,[2] 127.5,[2] 127.4,[2] 127.4,[1] 127.3,[1] 126.2[2] and 126.1[2]

(15 × CH; 3 × Ph), 88.5 (Ph_2CO), 66.0 (PhCHN), 55.3 (CH_3O), 44.0 (ArCHCH$_3$) and 19.1 (ArCHCH$_3$) (Found M^+, 527.2085; $C_{35}H_{29}NO_4$ requires M^+, 527.2091); and (2*R*,4*S*)-3-(2-phenyl-butanoyl)-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-**9** (18 mg, 57%, 92% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.66; which was found to be spectroscopically identical to that obtained previously.

4.17. Parallel kinetic separation of 4-phenyl-oxazolidin-2-one (*R*)-1 and 4,5,5-triphenyl oxazolidin-2-one (*S*)-6 using a *quasi*-enantiomeric combination of pentafluorophenyl 2-(6-methoxynaphthalen-2-yl)propionate (*S*)-23 and pentafluorophenyl 2-phenylbutanoate (*R*)-8

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (0.24 mL, 2.5 M in hexane, 0.60 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**1** (0.43 g, 0.27 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** (0.86 g, 0.27 mmol), pentafluorophenyl 2-(6-methoxynaphthalen-2-yl)propionate (*S*)-**23** (0.11 g, 0.27 mmol) and pentafluorophenyl 2-phenylbutanoate (*R*)-**8** (90 mg, 0.27 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (*S*,*R*)-**25** (ratio >95:5: *syn*-:*anti*-) and (*R*,*S*)-**9** (ratio >95:5: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2 S,4 R)-3-[2-(6-methoxynaphth-2-yl)-propionyl]-4-oxazolidin-2-one (*S*,*R*)-*syn*-**25** (49 mg, 48%) as a white solid; R_f [light petroleum (40–60 °C)/diethyl ether (1:1)] 0.33; mp 168–170 °C; $[\alpha]_D^{20} = +207.5$ (*c* 0.8, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1699 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.60 (1H, d, *J* 8.4, 1 × CH; Ar), 7.51 (1H, d, *J* 8.4, 1 × CH; Ar), 7.33 (1H, s, CH; Ar), 7.29–7.09 (6H, m, 6 × CH; Ar and Ph), 6.90 (2H, d, *J* 7.1, 2 × CH; Ar and Ph), 5.46 (1H, dd, *J* 8.9 and 5.2, PhCHN), 5.20 (1H, q, *J* 6.9, ArC*H*CH$_3$), 4.60 (1H, t, *J* 9.1, CH_AH_BO), 4.03 (1H, dd *J* 8.9 and 5.2, CH$_A$*H*$_B$O), 3.92 (3H, s, CH_3O) and 1.44 (3H, d, *J* 6.9, ArCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 173.6 (NC=O), 157.6 (*i*-CO; Ar), 153.0 (OC=O), 138.2, 135.1, 133.6 and 128.8 (4 × *i*-C; Ar and Ph), 129.4, 127.0, 126.4, 126.3, 118.7 and 105.5 (6 × CH; Ar), 128.8^2, 127.2^1 and 125.9^2 (5 × CH; Ph), 69.5 (CH_2O), 57.8 (PhCHN), 55.3 (CH_3O), 43.8 (ArC*H*CH$_3$) and 18.7 (ArCHC*H*$_3$) (Found MH^+, 376.1553; $C_{23}H_{22}NO_4$ requires MH^+, 376.1543); and (2*R*,4*S*)-3-(2-phenyl-butanoyl)-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-**9** (84 mg, 67%, >90% de) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.66; which was found to be spectroscopically identical to that obtained previously.

4.18. Kinetic resolution of pentafluorophenyl 2-phenyl-propionate *rac*-3 using 4,5,5-triphenyl oxazolidin-2-one (*S*)-6

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (0.32 mL, 2.5 M in hexane, 0.79 mmol), 4,5,5-triphenyl-oxazolidin-2-one (*S*)-**6** (0.249 g, 0.79 mmol) and pentafluorophenyl 2-phenyl-propionate *rac*-**3** (0.50 g, 1.60 mmol), gave after purification by column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) an inseparable diastereoisomeric mixture of oxazolidin-2-ones (*R*,*S*)-*syn*- and (*S*,*S*)-*anti*-**7** (ratio 85:15: *syn*-:*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*R*,4*S*)-3-(2-phenylpropionyl)-4,5,5-triphenyl-oxazolidin-2-one (*R*,*S*)-*syn*-**7** (0.265 g, 75%, 70% de) as a white powder; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.60; mp 154–156 °C; $[\alpha]_D^{20} = -255.1$ (*c* 3.4, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1704 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.63 (2H, br d, *J* 7.7, 2 × CH; Ph), 7.46–7.36 (4H, m, 4 × CH; Ph), 7.19 (2H, br dd, *J* 5.0, and 2.0, 2 × CH; Ph), 7.11–7.07 (2H, m, 2 × CH; Ph), 7.01–6.86 (8H, m, 8 × CH; Ph), 6.65 (2H, br d, *J* 7.7, 2 × CH; Ph), 6.25 (1H, s, CHN), 4.98 (1H, q, *J* 7.0, PhC*H*CH$_3$) and 1.35 (3H, d, *J* 7.0, PhCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 173.2 (NC=O), 152.0 (OC=O), 141.8, 138.0 and 135.0 (3 × *i*-C; 3 × Ph–oxazolidin-2-one), 139.5 (*i*-C; Ph), 128.9^2, 128.8^1, 128.4^2, 128.3^2, 127.9^3, 127.6^2, 127.5^1, 127.4^2, 127.0^1, 126.2^2 and 126.1^2 (20 × CH; 4 × Ph), 88.5 (CPh_2O), 66.0 (CHN), 44.0 (PhC*H*CH$_3$) and 19.0 (PhCHC*H*$_3$) (Found MNa^+, 448.1912; $C_{24}H_{31}NO_4SiNa$ requires MNa^+, 448.1915); *m*/*z* 447 (10%, M^+), 315 (5, M^+−Ph(CH$_3$)C=C=O), 256 (15, PhCHCPh$_2^+$), 183 (20, $Ph_2C{=}OH^+$), 105 (100, PhCH=NH^+) and 77 (20, Ph^+); and the pentafluorophenyl 2-phenylpropionate (*S*)-**3** (0.18 g, 36%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.83; $[\alpha]_D^{20} = +47.21$ (*c* 7.6, $CHCl_3$); ~63% ee {lit.[20] (*S*)-**3**; $[\alpha]_D^{20} = +74.5$ (*c* 4.9, $CHCl_3$); lit.[19] (*R*)-**3**; $[\alpha]_D^{20} = -75.0$ (*c* 3.3, $CHCl_3$)}.

4.19. Mutual kinetic resolution of 4,5,5-triphenyl oxazolidin-2-one *rac*-6 and oxazolidin-2-one 26 using pentafluorophenyl 2-phenylpropionate *rac*-3

In the same way as oxazolidin-2-one *rac-syn*-**7**, *n*-butyl lithium (0.41 mL, 2.5 M in hexane, 1.26 mmol), 4,5,5-triphenyl-oxazolidin-2-one *rac*-**6** (0.181 g, 0.57 mmol), oxazolidin-2-one **26** (50 mg, 0.57 mmol) and pentafluorophenyl 2-phenylpropionate *rac*-**6** (0.363 g, 1.15 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones *rac-syn*- and *anti*-**7** (ratio 95:5: *syn*-:*anti*-) and *rac*-**27** (ratio **7**:**27**: 39:61). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give (2*RS*,4*SR*)-3-(2-phenylpropionyl)-4,5,5-triphenyl-oxazolidin-2-one (*RS*,*SR*)-*rac-syn*-**7** (95 mg, 37%, 90% de) as a white powder; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.83; which was spectroscopically identical to that obtained previously; and 3-(2-phenylpropionyl)-oxazolidin-2-one *rac*-**27** (79 mg, 63%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.14; ν_{max} ($CHCl_3$) cm^{-1} 1772 (OC=O) and 1700 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.37 (2H, dt, *J* 7.1 and 1.5, 2 × CH; Ph), 7.31 (2H, br ddd, *J* 7.1, 1.5 and 1.0, 2 × CH; Ph), 7.27–7.22 (1H, br tt, *J* 7.1 and 1.5, CH; Ph), 5.11 (1H, q, *J* 7.0, C*H*CH$_3$), 4.42–4.25 (2H, m, CH_2O), 4.11–4.02 (1H, ABq, CH_AH_BN), 3.97–3.89 (1H, ABq, CH$_A$*H*$_B$N) and 1.50 (3H, d, *J* 7.0, CH_3); δ_C (100 MHz; $CDCl_3$) 174.4 (NC=O), 152.9 (OC=O), 140.2 (*i*-C; Ph), 128.4^2, 129.0^2 and 127.0^1 (5 × CH; Ph), 61.6 (CH_2O), 42.7 (CH_2N), 42.6 (*C*HCH$_3$) and 19.2 (CH_3) (Found M^+, 219.0888; $C_{12}H_{13}NO_3$ requires M^+, 219.0890); *m*/*z* 219 (3%, M^+), 132 (10, PhCH$_3$C=C=O^+), 105 (55, PhCH$_3$CH$^+$), 104 (50, PhCHCH$_2^+$), 77 (95, $C_6H_5^+$) and 43 (100, $NHCO^+$).

4.20. Synthesis of 2-phenylpropionic acid (*S*)-28: hydrolysis of oxazolidin-2-one (*S*,*R*)-*syn*-4

Lithium hydroxide monohydrate (0.34 g, 8.02 mmol) was slowly added to a stirred solution of oxazolidin-2-one (*R*,*S*)-*syn*-**4** (1.34 g, 4.01 mmol) and hydrogen peroxide (2.27 mL, 8.02 mmol, 40%/w) in THF/water (3:1; 32 mL). The reaction mixture was stirred at room temperature for 12 h. The reaction mixture was quenched with water (10 mL) and extracted with dichloromethane (3 × 10 mL). The combined organic layers were dried over $MgSO_4$ and were evaporated under reduced pressure to give the recovered oxazolidin-2-one (*R*)-**1** (0.62 g, 95%) as a white solid; $[\alpha]_D^{20} = -48.3$ (*c* 2.0, $CHCl_3$), {for (*S*)-**1**; lit.[18] $[\alpha]_D^{20} = +49.5$ (*c* 2.1, $CHCl_3$; for (*R*)-**1**; lit.[21] $[\alpha]_D^{26} = -40.9$ (*c* 3.0, $CHCl_3$) and $[\alpha]_D^{26} = -55.5$ (*c* 3.8, EtOH)}; and 2-phenylpropionic acid (+)-(*S*)-**28** (0.55 g, 91%) as colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:9)] 0.5; $[\alpha]_D^{20} = +71.7$ (*c* 1.0, $CHCl_3$), {lit.[18] $[\alpha]_D^{22} = +71.2$ (*c* 0.66, $CHCl_3$)}; ν_{max} ($CHCl_3$) cm^{-1} 1706 (C=O); δ_H (400 MHz; $CDCl_3$) 7.45–6.98 (5H, m, 5 × CH; Ph), 3.75 (1H, q, *J* 7.2, PhC*H*CH$_3$) and 1.5 (3H, d, *J* 7.2, PhCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 180.4 (C=O),

139.7 (*i*-C; Ph), 128.7[2], 127.6[2] and 127.4[1] (5 × CH; Ph), 45.3 (PhC*H*CH$_3$) and 18.1 (PhCHC*H*$_3$) (Found MH$^+$, 151.0753. $C_9H_{11}NO_2^+$) requires MH$^+$, 151.0759).

4.21. Synthesis of 2-phenylpropionic acid (*R*)-28: hydrolysis of oxazolidin-2-one (*R*,*S*)-*syn*-7

In the same way as 2-phenylpropionic acid (*S*)-**28**, lithium hydroxide monohydrate (13 mg, 0.30 mmol), hydrogen peroxide (10 mg, 80 μL, 0.30 mmol, 40%/w) and oxazolidin-2-one *syn*-(*R*,*S*)-**7** (67 mg, 0.15 mmol) in THF/water (3:1; 4 mL), gave after an acidic extraction, (−)-2-phenylpropionic acid (*R*)-**28** (13 mg, 58%) as colourless oil; >95% ee;[17] $[\alpha]_D^{20} = -71.8$ (*c* 2.0, $CHCl_3$) {lit.[18] $[\alpha]_D^{22} = -71.2$ (*c* 0.66, $CHCl_3$)}, which was spectroscopically identical to that reported previously; and the 4,5,5-triphenyloxazolidin-2-one (*S*)-**6** (44 mg, 95%) as a white solid; $[\alpha]_D^{20} = -210.0$ (*c* 0.4, EtOH) {lit.[18] $[\alpha]_D^{20} = -213.3$ (*c* 0.5, EtOH)}; which was spectroscopically identical to those previously obtained.

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14. Interestingly, the characteristic CHN peak for the oxazolidin-2-ones (*S*,*R*)-*syn*-4 and (*R*,*S*)-*syn*-**7** appears at 5.45 ppm and 6.25 ppm (by ^{1}H NMR spectroscopy) and 57.9 ppm and 66.0 ppm (by ^{13}C NMR spectroscopy), respectively.
15. ΔR_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] = 0.12.
16. The (*S*)-enantiomer of active ester **3** was recovered with 72% ee {$[\alpha]_D^{20} = +47.2$ (*c* 7.6, $CHCl_3$); {lit.[16] (*S*)-**3**; $[\alpha]_D^{20} = +74.5$ (*c* 4.9, $CHCl_3$); lit.[19] (*R*)-**3**; $[\alpha]_D^{20} = -75.0$ (*c* 3.3, $CHCl_3$)}. This was determined by derivatisation of the parent carboxylic acid [(formed by LiOH·H_2O-mediated hydrolysis of (*S*)-**3**] using the method outlined in Ref. 17.
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