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Parallel kinetic resolution of racemic oxazolidinones using quasi-enantiomeric active esters

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Abstract—Racemic Evans' oxazolidinones were efficiently resolved using a combination of quasi-enantiomeric profens. The levels of stereocontrol were high, leading to products with predictable configurations.

1. Introduction

The separation of enantiomeric substrates using a parallel kinetic resolution (PKR) is becoming a more popular method.[1] In recent years, attention has been focussed on the use of traditional chiral auxiliaries as complementary quasi-enantiomeric resolving agents.[2] In particular, Davies[3] has demonstrated this philosophy with resolution of a racemic enone *rac*-**3** using an equimolar combination of quasi-enantiomeric lithium amides (*S*)-**1** and (*R*)-**2** (Scheme 1). The levels of enantiomer recognition were excellent leading to separable β-amino esters *syn*,*syn*,*anti*-**4** and *syn*,*syn*,*anti*-**5** in good yield with superb levels of diastereoisomeric control (Scheme 1).

Moreover, Fox[4] has shown the use of a pair of quasi-enantiomeric oxazolidinones (*S*)-**6** and (*R*)-**7** as resolving components for the resolution of a racemic mixed anhydride *rac*-**8** to give the corresponding oxazolidinone adducts **9** and **10** with near perfect levels of stereocontrol (Scheme 2). These adducts were efficiently separated using a Vedejs' post-modification strategy[5] by the treatment of oxazolidinones **9** and **10** with TBAF to give the more separable oxazolidinones **9** and **11** (Scheme 2).

Over the last few years, we have employed this PKR strategy[6] for the resolution of active esters, such as *rac*-**14**,[7] using a pair of quasi-enantiomeric oxazolidinones (*R*)-**12** and (*S*)-**13** to give two separable diastereoisomerically pure oxazolidinones *syn*,*anti*-**15** and *syn*,*anti*-**16** in moderate yield and with high levels of diastereocontrol (Scheme 3).[8]

2. Results and discussion

Herein, we report[9] an extension of our methodology for the complementary resolution of racemic Evans'[10] oxazolidinones, such as *rac*-**A**, using an equimolar combination of quasi-enantiomeric profens [e.g., (*R*)-**B** and (*S*)-**C**] to give the corresponding oxazolidinone adducts (*R*,*S*)-**D** and (*S*,*R*)-**E** (Scheme 4). Simple separation and hydrolysis of each adduct should lead to both individual enantiomers of the original oxazolidinone (*S*)- and (*R*)-**A**, respectively (Scheme 4).

With this aim in mind, we first probed the kinetic resolution of a series of structurally related racemic oxazolidinones *rac*-**12**, *rac*-**13**, *rac*-**20**, *rac*-**21** and *rac*-**22** using two quasi-enantiomeric pentafluorophenyl active esters (*R*)-**14** and (*S*)-**19** (Schemes 5–7). We initially chose to use this combination of pentafluorophenyl active esters (*R*)-**14** and (*S*)-**19** for synthetic ease, and the resulting oxazolidinone adducts were also known to be separable

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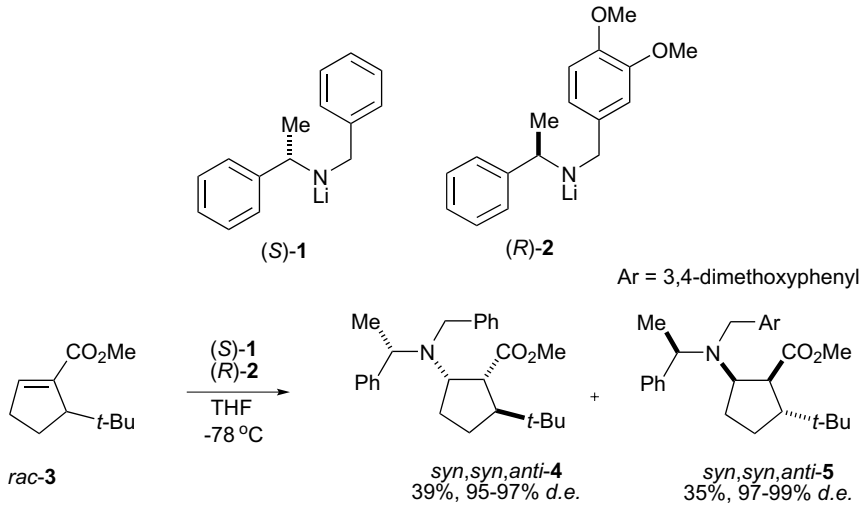
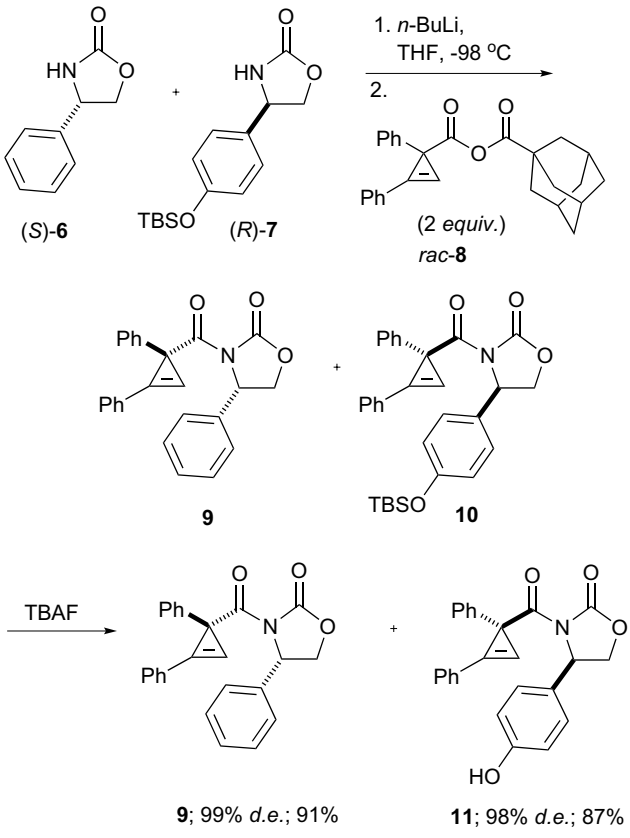


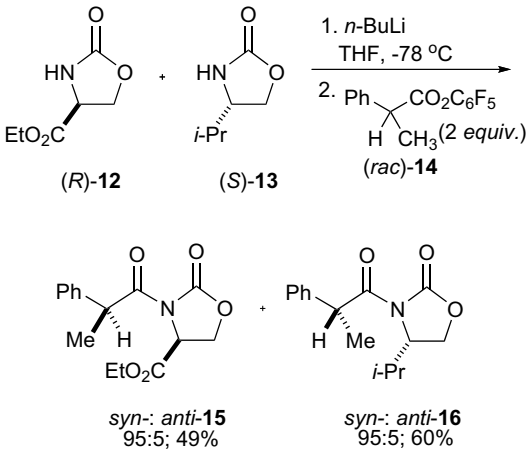
Scheme 1. Parallel kinetic resolution of enone (*rac*)-**3** using quasi-enantiomeric lithium amides **1** and **2**.

(Scheme 5).[9] The addition of pentafluorophenol to a stirred solution of DCC and carboxylic acids, (*R*)-**17** or (*S*)-**18**, in dichloromethane gave the corresponding enantiomerically pure active esters (*R*)-**14** and (*S*)-**19** in 85% and 84% yields, respectively (Scheme 5).

For this kinetic resolution study, we chose to use 2 equiv of racemic oxazolidinone, as this would mirror our standard parallel kinetic resolution conditions.[8] The addition of



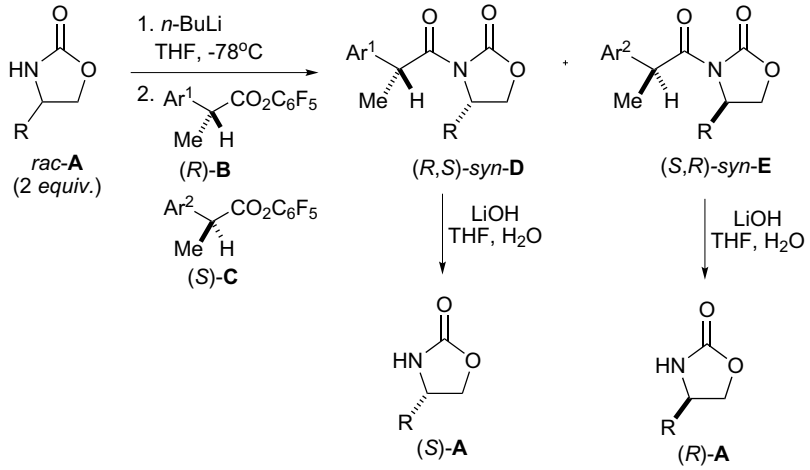
Scheme 2. Parallel kinetic resolution of mixed anhydride (*rac*)-**8** using quasi-enantiomeric oxazolidinones **6** and **7**.



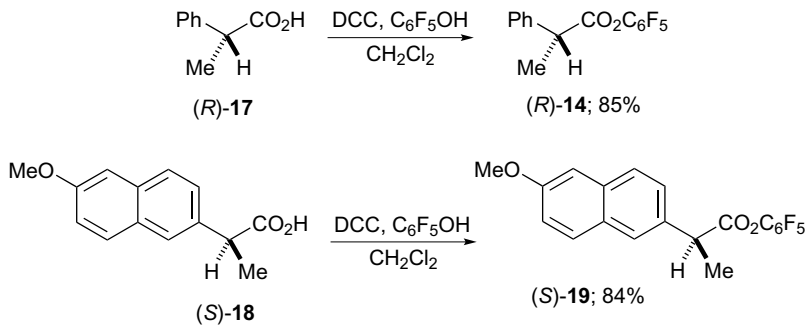
Scheme 3. Parallel kinetic resolution of active ester (*rac*)-**14** using quasi-enantiomeric oxazolidinones **12** and **13**.

n-BuLi to a stirred solution of oxazolidinones *rac*-**12**, *rac*-**13**, *rac*-**20**, *rac*-**21** and *rac*-**22** in THF at −78 °C, followed by the addition of either enantiomerically pure active esters (*R*)-**14** or (*S*)-**19**, gave the corresponding oxazolidinone adducts *syn*- and *anti*-**15**, *syn*- and *anti*-**16**, *syn*- and *anti*-**23**, *syn*- and *anti*-**24**, and *syn*,*syn*- and *anti*,*syn*-**25** [derived from (*R*)-**14**—Scheme 6] and *syn*- and *anti*-**26**, *syn*- and *anti*-**27**, *syn*- and *anti*-**28**, *syn*- and *anti*-**29**, and *syn*,*syn*- and *anti*,*syn*-**30** [derived from (*S*)-**19**—Scheme 7], respectively. These active esters (*R*)-**14** and (*S*)-**19** proved to be moderately diastereoselective favouring the formation of the corresponding *syn*-oxazolidinone adduct (from 34% to 64% de). Higher levels of diastereocontrol were preferred for oxazolidinones, which contained a sterically demanding group at its C(4) position; for example, when using oxazolidinones *rac*-**13** and *rac*-**20**, when $R = i$-Pr and Ph, respectively (Schemes 6 and 7). The stereochemistry of these adducts was assigned by comparison with known derivatives.[8,9]

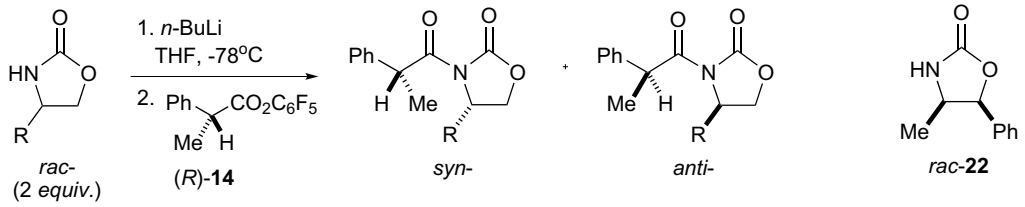
With this information in hand, we next probed the parallel kinetic resolution of this series of oxazolidinones *rac*-**12**,



Scheme 4. Proposed parallel kinetic resolution of oxazolidinone (*rac*)-**A** using active esters (*R*)-**B** and (*S*)-**C**.



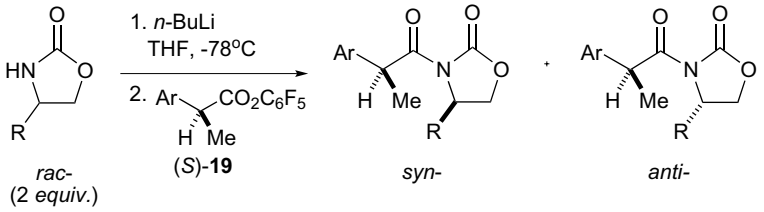
Scheme 5. Synthesis of active esters (*R*)-**14** and (*S*)-**19**.



Oxazolidinone	*rac*-**12**; R = CO_2Et	*rac*-**13**; R = *i*-Pr	*rac*-**20**; R = Ph	*rac*-**21**; R = CH_2Ph	*rac*-**22**
Products	*syn*-**15**: *anti*-**15**	*syn*-**16**: *anti*-**16**	*syn*-**23**: *anti*-**23**	*syn*-**24**: *anti*-**24**	*syn*,*syn*-**25**: *anti*,*syn*-**25**
Ratio	78:22	74:26	80:20	67:33	74:26
Yield	59%	60%	61%	71%	74%

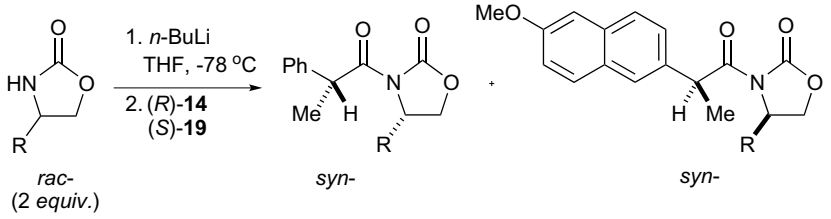
Scheme 6. Kinetic resolution of oxazolidinones (*rac*)-**12**, **13** and **20**–**22** using active ester (*R*)-**14**.

rac-**13**, *rac*-**20**, *rac*-**21** and *rac*-**22** using an equimolar combination of quasi-enantiomeric active esters (*R*)-**14** and (*S*)-**19** (Scheme 8). The addition of an equimolar mixture of active esters (*R*)-**14** and (*S*)-**19** to a stirred solution of the lithiated racemic oxazolidinones derived from *rac*-**12**, *rac*-**13**, *rac*-**20**, *rac*-**21** and *rac*-**22** (2 equiv) in THF at −78 °C, gave the corresponding oxazolidinone adducts *syn*-**15**, *syn*-**16**, *syn*-**23**, *syn*-**24**, and *syn*,*syn*-**25** [derived from (*R*)-**14**] and the complementary *syn*-**26**, *syn*-**27**, *syn*-**28**, *syn*-**29** and *syn*,*syn*-**30** [derived from (*S*)-**19**] as the major diastereoisomers, respectively (Scheme 8). These oxazolidinones were efficiently separated by column chromatography to give the diastereoisomerically pure adducts (Scheme 8).[11] The levels of diastereocontrol (and consequently enantiomer selection) were near perfect for the oxazolidinones *rac*-**12**, *rac*-**13** and *rac*-**20** (Scheme 8). Whereas, for those oxazolidinones, such as *rac*-**21** and *rac*-**22**, which were sterically less demanding at their C(4)-position, lower levels of



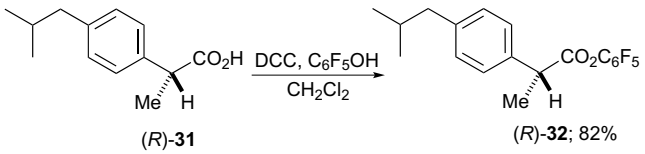
Oxazolidinone	*rac*-**12**; R = CO_2Et	*rac*-**13**; R = *i*-Pr	*rac*-**20**; R = Ph	*rac*-**21**; R = Bn	*rac*-**22**
Products	*syn*-**26**: *anti*-**26**	*syn*-**27**: *anti*-**27**	*syn*-**28**: *anti*-**28**	*syn*-**29**: *anti*-**29**	*syn,syn*-**30**: *anti,syn*-**30**
Ratio	79:21	82:18	79:21	73:27	67:33
Yield	78%	69%	67%	79%	51%

Scheme 7. Kinetic resolution of oxazolidinones (*rac*)-**12**, **13** and **20**–**22** using active ester (*S*)-**19**.



Entry	Oxazolidinone	Products [derived from (*R*)-**14**]	Products [derived from (*S*)-**19**]
1	*rac*-**12**; R = CO_2Et	*syn*-**15**:*anti*-**15**; 95:5; 55%	*syn*-**26**:*anti*-**26**; 95:5; 59%
2	*rac*-**13**; R = *i*-Pr	*syn*-**16**:*anti*-**16**; 95:5; 57%	*syn*-**27**:*anti*-**27**; 95:5; 59%
3	*rac*-**20**; R = Ph	*syn*-**23**:*anti*-**23**; 95:5; 65%	*syn*-**28**:*anti*-**28**; 95:5; 61%
4	*rac*-**21**; R = Bn	*syn*-**24**:*anti*-**24**; 72:28; 47%	*syn*-**29**:*anti*-**29**; 70:30; 42%
5	*rac*-**22**	*syn*-**25**:*anti*-**25**; 82:18; 44%	*syn*-**30**:*anti*-**30**; 85:15; 49%

Scheme 8. The parallel kinetic resolution of oxazolidinones (*rac*)-**12**, **13** and **20**–**22** using quasi-enantiomeric oxazolidinones (*R*)-**14** and (*S*)-**19**.

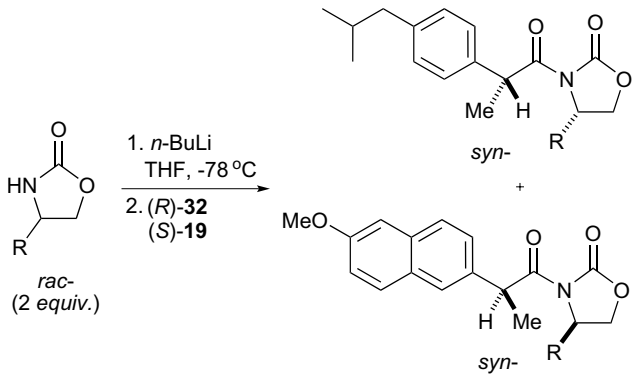


Scheme 9. Synthesis of active ester (*R*)-**32**.

complementary enantiomer selection were obtained (Scheme 8).

Our attention next turned to the use of naproxen (*S*)-**19** and ibuprofen (*R*)-**31** as complementary quasi-enantiomeric resolving agents for the resolution of racemic oxazolidinones (Schemes 9 and 10). We chose to focus on this particular combination of quasi-enantiomeric active esters (*S*)-**19** and (*R*)-**32**, as these would have greater adducts separation (Scheme 10).[12] The required active ester, (*R*)-**32**, was synthesised in 82% yield by the addition of DCC to a stirred solution of ibuprofen (*R*)-**31** and pentafluorophenol in dichloromethane (Scheme 9). The treatment of a solution of oxazolidinones *rac*-**12**, *rac*-**13** and *rac*-**20** (2 equiv) in THF at −78 °C with *n*-BuLi, followed by the addition of a stirred solution of active esters (*R*)-**32** and (*S*)-**19** in THF, gave the required pair of complementary quasi-enantiomeric adducts *syn*-**33** and *syn*-**26** [derived from *rac*-**12**] *syn*-**34** and *syn*-**27** [derived from *rac*-**12**], and *syn*-**35** and *syn*-**28** [derived from *rac*-**20**], respectively with high levels of stereocontrol (Scheme 10). These adducts were efficiently separated by column chromatography to give the corresponding diastereoisomerically pure adducts in good yields (Scheme 10).

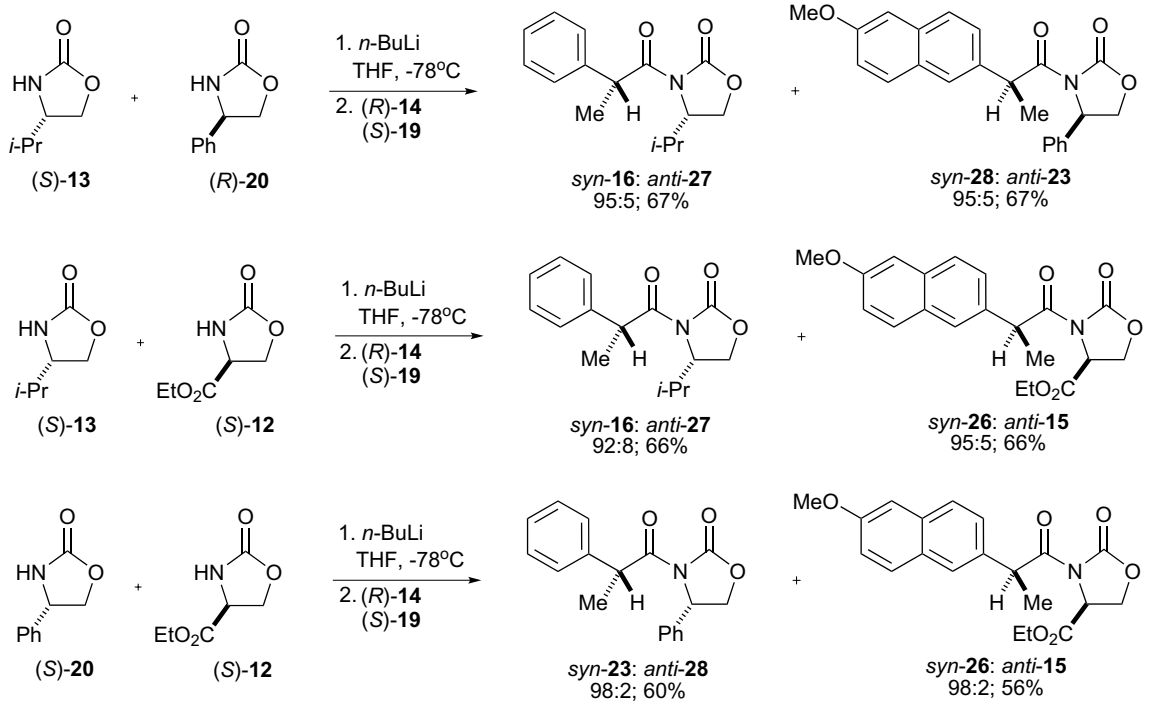
In an attempt to gain a greater understanding of this recognition process, we next focussed our attention on the mutual kinetic separation of an equimolar mixture of two complementary oxazolidinones, such as (*S*)-**13** and (*R*)-**20**, using two quasi-enantiomeric active esters (*R*)-**14** and (*S*)-**19** (Scheme 11). The addition of an equimolar solution



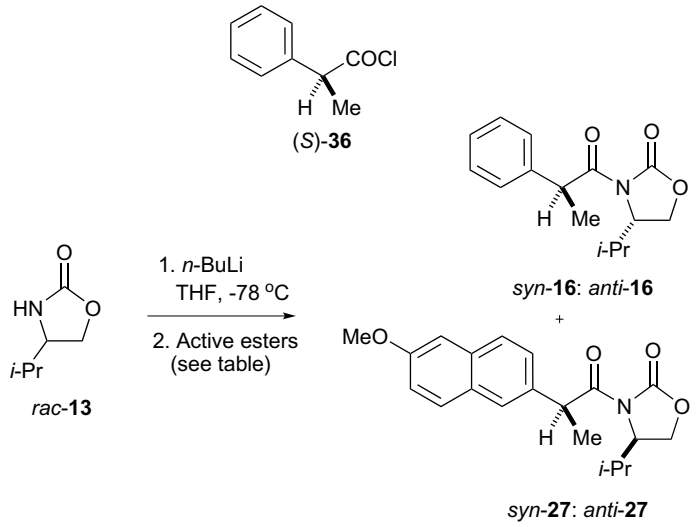
Entry	Oxazolidinone	Products [derived from (*R*)-**32**]	Products [derived from (*S*)-**19**]
1	*rac*-**12**; R = CO_2Et	*syn*-**33**:*anti*-**33**; 96:4; 41%	*syn*-**26**:*anti*-**26**; 96:4; 41%
2	*rac*-**13**; R = *i*-Pr	*syn*-**34**:*anti*-**34**; 95:5; 70%	*syn*-**27**:*anti*-**27**; 95:5; 74%
3	*rac*-**20**; R = Ph	*syn*-**35**:*anti*-**35**; 97:3; 54%	*syn*-**28**:*anti*-**28**; 95:5; 48%

Scheme 10. Parallel kinetic resolution of oxazolidinone (*rac*)-**12**, **13** and **20** using quasi-enantiomeric active esters (*S*)-**19** and (*R*)-**32**.

of active esters (*R*)-**14** and (*S*)-**19** in THF to a stirred solution of lithiated oxazolidinones in THF at −78 °C [derived from the treatment of (*S*)-**13** and (*R*)-**20** with *n*-BuLi (2 equiv)], gave the corresponding oxazolidinone adducts *syn*-**16** and *syn*-**28** in good yield and with excellent levels of mutual recognition (Scheme 11).[13] From this study, it was evident that oxazolidinone (*S*)-**13** preferentially recognised the (*R*)-enantiomer of the active ester **14** (to give *syn*-**16**), whereas the complementary oxazolidinone (*R*)-**20** recognised the (*S*)-enantiomer of enantiomer of the active ester **19** (to give *syn*-**28**), and vice versa (Scheme 11). The relative amounts of the mismatched adducts *anti*-**23** and *anti*-**27** were determined by ^{1}H NMR spectroscopy (by comparison with known adducts). For the remaining oxa-



Scheme 11. Mutual kinetic separation of oxazolidinones **12**, **13** and **20** with active esters (*R*)-**14** and (*S*)-**19**.

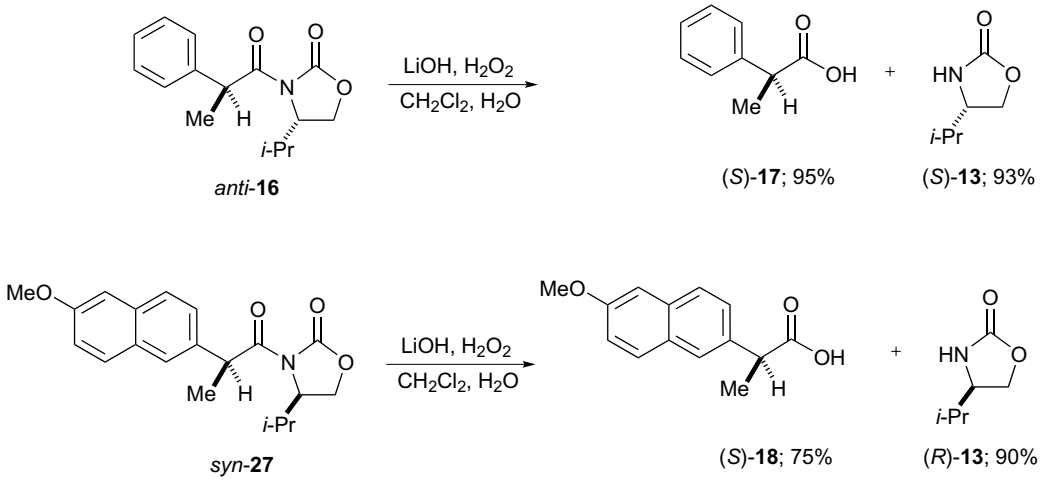


Entry	Active ester and/or acid chloride	Products	
1	(*S*)-**19** then (*S*)-**36**	*syn*-:*anti*-**16**; 12:88; 84%	*syn*-:*anti*-**27**; 85:15; 84%
2	*rac*-**36**	*syn*-:*anti*-**16**; 30:70; 60%[13]	—
3	*rac*-**19**	—	*syn*-:*anti*-**27**; 98:2; 58%
4	(*S*)-**36** then (*S*)-**19**	*syn*-:*anti*-**16** 40:60; 55%	*syn*-:*anti*-**27**; 92:8; 59%

Scheme 12. Sequential resolution of oxazolidinone (*rac*)-**13** with acid chloride (*S*)-**36** and active ester (*S*)-**19**.

zolidinone combinations (*S*)-**13** and (*R*)-**12**, and (*S*)-**20** and (*R*)-**12**, these gave the corresponding oxazolidinone adducts *syn*-**16** and *syn*-**28**, and *syn*-**23** and *syn*-**26**, respectively, with similarly high levels of mutual kinetic separation (Scheme 11).

In an attempt to probe this mutual recognition process, we next investigated the sequential resolution of racemic oxazolidinone *rac*-**13** using two complementary carbonyl derivatives which had differing degrees of electrophilicity and enantiomer selection (Scheme 12). We chose to use the more electrophilic acid chloride (*S*)-**36** and the less electrophilic active ester (*S*)-**19**, as these are known to favour the (*S*)-[14] and (*R*)-enantiomers[8,9] of **13**, respectively (Scheme 12). The treatment of the lithiated oxazolidinone *rac*-**13-Li** [derived from *rac*-**13** and *n*-BuLi], followed by the sequential addition of the active ester (*S*)-**19** and acid chloride (*S*)-**36**, gave the corresponding oxazolidinone adducts *anti*-**16** and *syn*-**27** in good yield with modest to good levels of diastereocontrol (Scheme 12, entry 1). The relative reaction rates and the resulting diastereocontrol were evidently different; the oxazolidinone *anti*-**16** was formed with significantly higher diastereoselectivity relative to its mutual kinetic resolution,[14] whereas, its complementary



Scheme 13. Hydrolysis of oxazolidinones adducts *syn*-**16** and *syn*-**27**.

partner, oxazolidinone *syn*-**27** was formed with lower diastereocontrol (Scheme 12, entries 2 and 3). This is understandable as the formation of *syn*-**27** and *anti*-**16** must proceed via kinetic resolution of racemic *rac*-**13** and scalemic oxazolidinone (*S*)-**13** [due to the (*R*)-enantiomer being partially removed from *rac*-**13** by the active ester (*S*)-**19**], respectively. By comparison, the addition of acid chloride (*S*)-**36** to the lithiated oxazolidinone [derived from *n*-BuLi addition to oxazolidinone *rac*-**13**], followed by the active ester (*S*)-**19**, gave the corresponding adducts *anti*-**16** and *syn*-**27** in good yield. The levels of diastereocontrol for the oxazolidinone *anti*-**16** and *syn*-**27** were slightly lower than their mutual kinetic resolutions (Scheme 12, entry 4 vs entries 2 and 3). This particular reagent combination was evidently less diastereoselective, and illustrated that this outcome was governed more by the less diastereoselective acid chloride (*S*)-**36** than its complementary active ester (*S*)-**19** (Scheme 12).

Lithium hydroxide mediated hydrolysis of these oxazolidinone adducts *anti*-**16** and *syn*-**27**, gave the corresponding enantiomerically pure (*S*)- and (*R*)-enantiomers of oxazolidinone **13**, respectively, in good yields (Scheme 13). The complementary resolving profens (*S*)-**17** and (*S*)-**18** were re-isolated in excellent yield with no loss of enantiomeric purity (Scheme 13). This has been shown to be the case for a wide variety of structurally related profen-based oxazolidinone adducts and this has been reported elsewhere.[14]

3. Conclusion

In conclusion, we have reported an efficient parallel kinetic resolution of racemic oxazolidinones using an equimolar combination of quasi-enantiomeric profens. This methodology[8,9] is efficient for a variety of structurally related oxazolidinones [e.g., *rac*-**20**] and quasi-enantiomeric profens [e.g., (*R*)-**14** and (*S*)-**19**], and is predictable leading to the separable, diastereoisomerically pure, *syn*-adducts **23** and **28** in good yield.

4. Experimental

4.1. General

All solvents were distilled before use. All reactions were carried out under nitrogen using an 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 and 270 MHz Fourier transform spectrometers 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 rotation was measured using an automatic AA-10 Optical Activity Ltd polarimeter.

4.2. (−)-Pentafluorophenyl 2-phenylpropionate (*R*)-14

2-Phenylpropionic acid (*R*)-**17** (3.0 g, 20.0 mmol) $\{[\alpha]_D^{24} = -71.7$ (*c* 2.0, $CHCl_3)\}$ was added to a stirred solution of *N*,*N*′-dicyclohexylcarbodiimide (DCC) (4.53 g, 22.0 mmol) in dichloromethane (100 ml). The resulting solution was stirred for 10 min. A solution of pentafluorophenol (4.90 g, 26.7 mmol) in dichloromethane (20 ml) was slowly added, and the resulting solution was stirred for 12 h. The resulting precipitate (*N*,*N*′-dicyclohexylurea) was filtered off (using suction filtration). Water (100 ml) was added and the solution extracted with dichloromethane (3×100 ml) and dried over $MgSO_4$. The combined organic layers were evaporated under reduced pressure. The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (9:1) to give pentafluorophenyl 2-phenylpropionate (*R*)-**14** (5.37 g, 85%) as a colourless oil; R_F [light petroleum (40–60 °C)/diethyl ether (1:1)] 0.75; $[\alpha]_D^{24} = -75.0$ (*c* 3.3, $CHCl_3$); $\nu_{max}(CHCl_3)/cm^{-1}$ 1779 (C=O); δ_H (270 MHz; $CDCl_3$) 7.41–7.29 (5H, m, $5 \times$CH; Ph), 4.07 (1H, q, *J* 7.2, PhC*H*CH_3) and 1.64 (3H, d, 6.9, PhCHCH_3); δ_C (100 MHz; $CDCl_3$) 170.6 (OC=O), 141.1 (142.40 and 139.90, 2C, ddt, $^1J_{C,F} = 251.3$ Hz, $^2J_{C,F} = 12.2$ Hz and $^3J_{C,F} = 3.8$ Hz, C(2)–F), 139.4 (140.70 and 138.18, 1C, dtt, $^1J_{C,F} = 253.2$ Hz, $^2J_{C,F} = 13.4$ Hz and $^3J_{C,F} = 4.2$ Hz, C(4)–F), 138.7 (*i*-C; Ph), 137.8 (139.05 and 136.58, 2C, 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), 128.9, 127.8 and 127.5 ($3 \times$CH; Ph), 125.2 (1C, tdt, $^2J_{C,F} = 14.2$ Hz, $^4J_{C,F} = 4.2$ Hz and $^3J_{C,F} = 2.0$ Hz, *i*-CO; OC_6F_5), 45.1 (PhCHCH_3) and 18.5 (PhC*H*CH_3); δ_F (378 MHz; $CDCl_3$) −152.6 (2F, d, $^3J_{F,F}$ 17.0, F_{ortho}), −157.9 (1F, t, $^3J_{F,F}$ 21.7, F_{para}) and −162.3 (2F, dd, $^3J_{F,F}$ 21.7 and 17.0, F_{meta}) (Found M^+, 316.0514. $C_{15}H_9F_5O_2$ requires M^+, 316.0517).

4.3. (+)-Pentafluorophenyl-2-(6-methoxynaphthalene-2-yl)-propionate (*S*)-19

In the same way as the active ester (*R*)-**14**, (*S*)-(+)-6-methoxy-(2-naphthyl)propionic acid (*S*)-**18** (5.0 g, 21.7 mmol) $\{[\alpha]_D^{20} = +64.8$ (*c* 3.4, $CHCl_3)\}$, DCC (4.93 g, 23.9 mmol) and pentafluorophenol (4.0 g, 21.7 mmol) gave pentafluorophenyl-2-(6-methoxynaphthalene-2-yl)propionate (*S*)-**19** (7.24 g, 84%) as a white powder; mp = 78–80 °C; R_F [light petroleum (40–60 °C)/diethyl ether (1:1)] 0.65; $[\alpha]_D^{20} = +93.6$ (*c* 5.6, $CHCl_3$); $\nu_{max}(CHCl_3)/cm^{-1}$ 1781 (C=O); δ_H (250 MHz; $CDCl_3$) 7.76–7.13 (6H, m, $6 \times$CH; Ar), 4.38 (1H, q, *J* 7.2, C*H*CH_3) 3.91 (3H, s, CH_3) and 1.71 (3H, d, *J* 7.2, CH_3CH); δ_C (100 MHz; $CDCl_3$) 170.7 (C=O), 157.9 (*i*-CO; Ar), 141.0 (142.32 and 139.82.67, 2C, ddt, $^1J_{C,F} = 249.8$ Hz, $^2J_{C,F} = 12.2$ Hz and $^3J_{C,F} = 4.6$ Hz, C(2)–F), 139.3 (140.63 and 138.11, 1C, dtt, $^1J_{C,F} = 252.1$ Hz, $^2J_{C,F} = 13.0$ Hz and $^3J_{C,F} = 4.5$ Hz, C(4)–F), 137.8 (139.04 and 136.54, 2C, dtdd, $^1J_{C,F} = 250.6$ Hz, $^2J_{C,F} = 13.8$ Hz, $^3J_{C,F} = 5.3$ and $^4J_{C,F} = 3.0$ Hz, C(3)–F) , 133.9, 133.7 and 128.9 ($3 \times$*i*-C; Ar), 129.3, 127.5, 126.2, 125.7, 119.3 and 105.6 ($6 \times$CH; Ar), 125.2 (1C, m, *i*-CO; OC_6F_5), 55.3 (OCH_3), 45.9 (Ar*C*H) and 18.5 (CH*C*H_3); δ_F (378 MHz; $CDCl_3$) −152.5 (2F, d, $^3J_{F,F}$ 17.0, F_{ortho}), −157.9 (1F, t, $^3J_{F,F}$ 21.6, F_{para}) and −162.3 (2F, dd, $^3J_{F,F}$

21.6 and 17.0, F_{meta}) (Found M^+, 396.0783; $C_{20}H_{13}F_5O_3^+$ requires 396.0779).

4.4. Kinetic resolution of racemic oxazolidinones using (−)-pentafluorophenyl 2-phenylpropionate (*R*)-14

4.4.1. (4*R*)-Isopropyl-3-(2*R*-phenylpropionyl)-oxazolidin-2-one *anti*-16 and (4*S*)-isopropyl-3-(2*R*-phenylpropionyl)-oxazolidin-2-one *syn*-16. *n*-BuLi (0.61 ml, 2.5 M in hexane, 1.54 mmol) was added to a stirred solution of (±)-4-isopropyl-oxazolidin-2-one *rac*-**13** (0.20 g, 1.54 mmol) in THF (5 ml) at −78 °C. After stirring for 1 h, a solution of (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.24 g, 0.77 mmol) 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 separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**16** [*syn*-/*anti*- 74:26]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (*R*,*R*)-*anti*-**16** (32 mg, 16%); R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.64; $[\alpha]_D^{20} = -109.6$ (*c* 11.6, $CHCl_3$); {(*S*,*S*)-*anti*-**16**; $[\alpha]_D^{20} = +128.9$ (*c* 3.5, $CHCl_3$);[14] and lit.[15] $[\alpha]_D^{20} = +100.6$ (*c* 1.11, $CHCl_3$)}, ν_{max} (film) cm^{-1} 1774 (C=O) and 1701 (C=O); δ_H (250 MHz; $CDCl_3$) 7.38–7.20 (5H, m, 5 × CH; Ph), 5.15 (1H, q, *J* 7.0, PhC*H*CH_3), 4.39–4.33 (1H, m, *i*-PrCHN), 4.18–4.08 (2H, m, CH_2O), 2.50–2.38 (1H, m, C*H*$(CH_3)_2$), 1.52 (3H, d, *J* 7.0, PhCHC*H*$_3$), 0.92 (3H, d, *J* 7.0, CH_3^ACHCH_3^B) and 0.91 (3H, d, *J* 6.9, CH_3^ACHCH_3^B); δ_C (62.9 MHz; $CDCl_3$) 174.7 (NC=O), 153.6 (OC=O), 140.4 (*i*-C; Ph), 128.6, 128.2 and 127.2 (3 × CH; Ph), 63.2 (CH_2O), 59.1 (*i*-PrCHN), 43.1 (PhC*H*CH_3), 28.6 (*C*H$(CH_3)_2$), 19.7 (CH_3), 18.1 (CH_3) and 14.8 (PhCHC*H*$_3$) (Found MH^+ 262.1434; $C_{15}H_{20}NO_3^+$ requires 262.1443); *m*/*z* 262 (30, MH^+), 130 (48, M−C_9H_8O) and 105 (100, M−$C_7H_{11}NO_3$); and the oxazolidinone (*R*,*S*)-*syn*-**16** (88 mg, 44%); R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.43; $[\alpha]_D^{20} = -19.4$ (*c* 3.0, $CHCl_3$); {lit.[14] $[\alpha]_D^{20} = -19.8$ (*c* 3.3, $CHCl_3$); lit.[16] $[\alpha]_D^{20} = -19.2$ (*c* 1.15, $CHCl_3$)}; {for (*S*,*R*)-**16**; $[\alpha]_D^{20} = +18.3$ (*c* 6.0, $CHCl_3$)}; ν_{max}($CHCl_3$)/cm^{-1} 1774 (C=O) and 1703 (C=O); δ_H (250 MHz; $CDCl_3$) 7.39–7.19 (5H, m, 5 × CH; Ph), 5.14 (1H, q, *J* 6.9, PhC*H*CH_3), 4.49 (1H, m, *i*-PrCHN), 4.24 (1H, t, *J* 8.9, CH_AH$_B$O), 4.10 (1H, dd, *J* 8.9 and 3.5, CH_AH_BO), 2.24–2.12 (1H, m, C*H*$(CH_3)_2$), 1.47 (3H, d, *J* 6.9, PhC*H*CH_3), 0.79 (3H, d, *J* 7.0, CH_3^ACHCH_3^B) and 0.46 (3H, d, *J* 6.9, ^{A}CH_3CH^BCH_3); δ_C (62.9 MHz; $CDCl_3$) 174.5 (NC=O), 153.5 (OC=O), 140.5 (*i*-C; Ph), 128.6, 128.1 and 127.2 (3 × CH; Ph), 62.9 (CH_2O), 58.1 (*i*-PrCHN), 43.3 (PhC*H*CH_3), 27.9 (*C*H$(CH_3)_2$), 18.7 (CH_3), 17.8 (CH_3) and 14.1 (PhC*H*CH_3) (Found MH^+ 262.1432; $C_{15}H_{20}NO_3^+$ requires 262.1443); *m*/*z* 262 (30%, MH^+), 130 (48, M−C_9H_8O) and 105 (100, M−$C_7H_{11}NO_3$).

4.4.2. Synthesis of (4*R*)-4-phenyl-3-(2*R*-phenylpropionyl)-oxazolidin-2-one *anti*-23 and (4*S*)-4-phenyl-3-(2*R*-phenylpropionyl)-oxazolidin-2-one *syn*-23. In the same way as oxazolidinone **16**, *n*-BuLi (0.49 ml, 2.5 M in hexane, 1.22 mmol), (±)-4-phenyl oxazolidin-2-one *rac*-**20** (0.20 g, 1.22 mmol) and (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.19 g, 0.61 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**23** [*syn*-/*anti*- 80:20]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (*R*,*R*)-*anti*-**23** (21 mg, 12%) as a white solid; mp 158–160 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.58; $[\alpha]_D^{20} = -179.1$ (*c* 3.0, $CHCl_3$); {lit.[14] $[\alpha]_D^{20} = -180.5$ (*c* 1.52, $CHCl_3$); lit.[17] $[\alpha]_D^{27} = -163.2$ (*c* 0.1, $CHCl_3$)}; ν_{max}($CHCl_3$)/cm^{-1} 1780 (C=O) and 1700 (C=O); δ_H (270 MHz; $CDCl_3$) 7.39–7.26 (10H, m, 10 × CH; 2 × Ph), 5.32 (1H, dd, *J* 8.8 and 3.2, PhCHN), 5.11 (1H, q, *J* 7.2, PhC*H*CH_3), 4.55 (1H, t, *J* 8.8, CH_AH$_B$O), 4.21 (1H, dd, *J* 8.8 and 3.2, CH_AH_BO) and 1.40 (3H, d, *J* 7.2, PhCHC*H*$_3$); δ_C (62.9 MHz; $CDCl_3$) 174.1 (NC=O), 152.9 (OC=O), 140.2 (*i*-C; Ph_A), 139.4 (*i*-C; Ph_B), 129.3[2], 128.7[1], 128.6[2], 128.2[2], 127.3[1] and 125.8[2] (10 × CH; Ph_A and Ph_B), 69.7 (CH_2O), 58.1 (PhCHN), 43.2 (PhC*H*CH_3) and 19.4 (PhCHC*H*$_3$) (Found MH^+, 296.1282; $C_{18}H_{18}NO_3^+$ requires 296.1287); and the oxazolidinone (*R*,*S*)-*syn*-**23** (88 mg, 49%) as a white solid; mp 128–130 °C {for (*S*,*R*)-*syn*-**23**; mp = 140–142 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.42; ν_{max}($CHCl_3$)/cm^{-1} 1778 (C=O) and 1701 (C=O); $[\alpha]_D^{20} = -83.4$ (*c* 5.0, $CHCl_3$); {lit.[14] (*S*,*R*)-**23**; $[\alpha]_D^{20} = +88.5$ (*c* 4.0, $CHCl_3$); lit.[17] (*S*,*R*)-**23** $[\alpha]_D^{27} = +143.4$ (*c* 0.5, $CHCl_3$)}; δ_H (270 MHz; $CDCl_3$) 7.29–7.21 (10H, m, 10 × CH; 2 × Ph), 5.45 (1H, dd *J* 9.0 and 5.1, PhCHN), 5.09 (1H, q, *J* 6.9, PhC*H*CH_3), 4.63 (1H, t, *J* 9.0, CH_AH$_B$O), 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 (62.9 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], 128.7[1], 128.5[2], 128.2[2], 127.1[1] and 125.9[2] (10 × CH; Ph_A and Ph_B), 69.6 (CH_2O), 57.9 (PhCHN), 43.9 (Ph*C*HCH$_3$) and 18.6 (PhCH*C*H$_3$) (Found MH^+, 296.1286; $C_{15}H_{18}NO_3^+$ requires 296.1287); *m*/*z* 295.1 (10%, M^+), 132.1 (100, Ph(CH$_3$)C=C=O$^+$), 105.1 (25, $PhCH_2^+$) and 77.1 (20, Ph^+).

4.4.3. (4*R*)-Benzyl-3-(2*R*-phenylpropionyl)-oxazolidine-2-one *anti*-24 and (4*S*)-benzyl-3-(2*R*-phenylpropionyl)-oxazolidine-2-one *syn*-24. In the same way as oxazolidinone **16**, *n*-BuLi (0.45 ml, 2.5 M in hexane, 1.12 mmol), (±)-4-benzyl-oxazolidin-2-one *rac*-**21** (0.20 g, 1.12 mmol) and (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.17 g, 0.56 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**24** [*syn*-/*anti*- 67:33]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (*R*,*R*)-*anti*-**24** (40 mg, 23%) as an oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.66; $[\alpha]_D^{20} = -104.0$ (*c* 4.0, $CHCl_3$) {lit.[14] (*S*,*S*)-**24**; $[\alpha]_D^{20} = +130.4$ (*c* 1.8, $CHCl_3$); lit.[15] $[\alpha]_D^{20} = +107.1$ (*c* 1.01, $CHCl_3$)}; ν_{max}($CHCl_3$)/cm^{-1} 1780 (C=O) and 1699 (C=O); δ_H (270 MHz; $CDCl_3$) 7.39–7.21 (10H, m, 10 × CH; 2 × Ph), 5.12 (1H, q, *J* 7.0, PhC*H*CH_3), 4.61–4.54 (1H, m, PhCHN), 4.12–4.10 (2H, m, CH_2O), 3.35 (1H, dd, *J* 13.1 and 3.2, CH_AH$_B$Ph), 2.80 (1H, dd, *J* 13.1 and 9.8, CH_AH_BPh) and 1.55 (3H, d, *J* 7.0, PhCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$)

174.7 (NC=O), 152.9 (OC=O), 140.3 (*i*-C; Ph_A), 135.4 (*i*-C; Ph_B), 129.5[2], 129.0[2], 128.7[2], 128.1[2], 127.4[1] and 127.3[1] (10 × CH; Ph_A and Ph_B), 65.9 (CH_2O), 55.8 (BnCHN), 43.2 (Ph*C*HCH$_3$), 38.0 (CH_2Ph) and 19.5 (PhCH*C*H$_3$) (Found MH^+ 310.1442. $C_{19}H_{20}NO_3^+$ requires 310.1443); *m*/*z* 310 (80%, MH^+), 178 (18, M−C_9H_8O), 132 (100, M−$C_{10}H_{12}NO_2$) and 105 (18, M−$C_{11}H_{11}NO_3$); and the oxazolidinone (*R*,*S*)-*syn*-**24** (85 mg, 48%) as a viscous oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.43; $[\alpha]_D^{20} = +1.46$ (*c* 9.8, $CHCl_3$) [lit.[14] $[\alpha]_D^{20} = +2.8$ (*c* 5.5, $CHCl_3$); lit.[14,18] $[\alpha]_D^{20} = +2.2$ (*c* 2.8, $CHCl_3$); lit.[15] $[\alpha]_D^{27} = +16.1$ (*c* 0.96, $CHCl_3$)]; ν_{max} ($CHCl_3$); cm^{-1} 1775 (C=O) and 1700 (C=O); δ_H (270 MHz; $CDCl_3$) 7.45–6.94 (10H, m, 10 × CH; Ph_A and Ph_B), 5.11 (1H, q, *J* 6.9, PhC*H*CH$_3$), 4.79–4.70 (1H, m, BnCHN), 4.18 (1H, t, *J* 8.5, C*H*$_A$H$_B$O), 4.07 (1H, dd *J* 8.5 and 3.2, CH$_A$*H*$_B$O), 3.08 (1H, dd *J* 13.5 and 3.2, C*H*$_A$H$_B$Ph), 2.58 (1H, dd, *J* 13.5 and 8.8, CH$_A$*H*$_B$Ph) and 1.52 (3H, d, *J* 6.9, PhC*H*CH$_3$); δ_C (100 MHz; $CDCl_3$) 174.5 (NC=O), 153.0 (OC=O), 140.2 (*i*-C; Ph_A), 135.0 (*i*-C; Ph_B), 129.4[2], 128.8[2], 128.6[2], 128.3[2], 127.3[1] and 127.2[1] (10 × CH; Ph_A and Ph_B), 65.8 (CH_2O), 54.9 (BnCHN), 43.2 (Ph*C*HCH$_3$), 37.4 (CH_2) and 19.2 (PhCH*C*H$_3$) (Found MH^+ 310.1438. $C_{19}H_{20}NO_3^+$ requires 310.1443); *m*/*z* 310 (80%, MH^+), 178 (15, M−C_9H_8O), 132 (100, M−$C_{10}H_{12}NO_2$) and 105 (15, M−$C_{11}H_{11}NO_3$).

4.4.4. (4*R*,5*S*)-4-Methyl-5-phenyl-3-(2*R*-phenylpropionyl)-oxazolidin-2-one *anti*-25 and (4*S*,5*R*)-4-methyl-5-phenyl-3-(2*R*-phenylpropionyl)-oxazolidin-2-one *syn*-25. In the same way as oxazolidinone **16**, *n*-BuLi (0.54 ml, 2.5 M in hexane, 1.13 mmol), (±)-4-methyl-5-phenyl oxazolidin-2-one *rac*-**22** (0.20 g, 1.13 mmol) and (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.17 g, 0.56 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**24** [*syn*-/*anti*- 74:26]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (4*R*,5*S*,2*R*)-*anti*-**25** (33 mg, 19%) as a white solid; mp 89–92 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.76; $[\alpha]_D^{20} = -39.2$ (*c* 4.0, $CHCl_3$); {for (4*S*5*R*,2*S*)-*anti*-**25**; $[\alpha]_D^{20} = +35.1$ (*c* 3.6, $CHCl_3$)} {lit.[14] $[\alpha]_D^{20} = -42.7$ (*c* 3, $CHCl_3$)}; ν_{max}($CHCl_3$)/cm^{-1} 1778 (C=O) and 1697 (C=O); δ_H (250 MHz; $CDCl_3$) 7.44–7.24 (10H, m, 10 × CH; 2 × Ph), 5.49 (1H, d, *J* 7.1, OCHPh), 5.14 (1H, q, *J* 7.1, PhC*H*CH$_3$), 4.68 (1H, m, CH$_3$C*H*N), 1.51 (3H, d, *J* 7.1, PhCHC*H*$_3$) and 0.94 (3H, d, *J* 6.6, C*H*$_3$CHN); δ_C (62.9 MHz; $CDCl_3$) 174.5 (NC=O), 152.6 (OC=O), 140.5 (*i*-C; Ph_A; PhCHCH$_3$), 133.3 (*i*-C; Ph_B; PhCHO), 129.2, 129.1, 128.7, 128.2, 127.3 and 125.6 (6 × CH; Ph_A and Ph_B), 78.7 (OCHPh), 55.5 (CH$_3$*C*HN), 43.4 (Ph*C*HCH$_3$), 19.3 (PhCH*C*H$_3$) and 14.6 (*C*H$_3$CHN) (Found MH^+ 310.1430. $C_{19}H_{20}NO_3^+$ requires 310.1443); *m*/*z* 310 (31%, MH^+), 178 (9, M−C_9H_8O) and 105 (100, M−$C_{11}H_{11}NO_3$); and the oxazolidinone (4*S*5*R*,2*R*)-*syn*-**25** (95 mg, 55%) as a white solid; mp 112–114 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.63; $[\alpha]_D^{20} = -106.9$ (*c* 3.2, $CHCl_3$); [lit.[14] (4*R*5*S*,2*S*)-**25**; $[\alpha]_D^{20} = +105.9$ (*c* 2.6, $CHCl_3$)]; ν_{max}($CHCl_3$)/cm^{-1} 1774 (C=O) and 1701 (C=O); δ_H (250 MHz; $CDCl_3$) 7.40–7.17 (10H, m, 10 × CH; Ph_A and Ph_B), 5.64 (1H, d, *J* 7.2, OCHPh), 5.08 (1H, q, *J* 7.1, PhC*H*CH$_3$), 4.82 (1H, m, BnCHN), 1.51 (3H, d, *J* 7.1, PhCHC*H*$_3$) and 0.74 (3H, d, *J* 6.6, C*H*$_3$CHN); δ_C (62.9 MHz; $CDCl_3$) 174.3 (NC=O), 152.5 (OC=O), 140.3 (*i*-C; Ph_A; PhCHCH$_3$), 133.5 (*i*-C; Ph_B; PhCHO), 128.9, 128.8, 128.6, 128.1, 127.1 and 125.7 (6 × CH; Ph_A and Ph_B), 78.8 (OCHPh), 54.7 (BnCHN), 43.6 (Ph*C*HCH$_3$), 19.4 (PhCHC*H*$_3$) and 14.1 (*C*H$_3$CHN) (Found MH^+ 310.1460. $C_{19}H_{20}NO_3^+$ requires 310.1443); *m*/*z* 310 (28%, MH^+), 178 (8, M−C_9H_8O) and 105 (100, M−$C_{11}H_{11}NO_3$).

4.4.5. Synthesis of ethyl (4*S*,2*R*)-2-oxa-3-(2′-phenylpropionyl)-oxazolidin-4-carboxylate *anti*-15 and ethyl (4*R*,2*R*)-2-oxa-3-(2′-phenylpropionyl)-oxazolidin-4-carboxylate *syn*-15. In the same way as oxazolidinone **16**, *n*-BuLi (0.50 ml, 2.5 M in hexane, 1.25 mmol), (±)-ethyl oxazolidin-2-one 4-carboxylate *rac*-**12** (0.20 g, 1.25 mmol) and (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.197 g, 0.62 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**15** [*syn*-/*anti*- 78:22]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (4*S*,2*R*)-*anti*-**15**(23 mg, 13%) as an oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.42; $[\alpha]_D^{20} = -129.5$ (*c* 2.2, $CHCl_3$); [lit.[14] $[\alpha]_D^{20} = -135.8$ (*c* 4.5, $CHCl_3$)]; ν_{max}($CHCl_3$)/cm^{-1} 1794 (C=O), 1747 (C=O) and 1705 (C=O); δ_H (270 MHz; $CDCl_3$) 7.33–7.20 (5H, m, 5 × CH; Ph), 5.10 (1H, q, *J* 7.0, PhC*H*CH$_3$), 4.77 (1H, dd, *J* 9.4 and 3.7, EtO$_2$CCHN), 4.38 (1H, t, *J* 9.4, C*H*$_A$H$_B$O), 4.31–4.21 (3H, m, CH$_A$*H*$_B$O and C*H*$_2$CH$_3$), 1.50 (3H, d, *J* 7.0, PhCHC*H*$_3$) and 1.30 (3H, t, *J* 7.2, C*H*$_3$CH$_2$); δ_C (62.9 MHz; $CDCl_3$) 174.5 (NC=O), 168.7 (CC=O), 152.1 (OC=O), 140.0 (*i*-C; Ph), 128.7, 128.3 and 127.4 (3 × CH; Ph), 64.3 (CH_2O), 62.6 (CH_2O), 55.9 (EtO$_2$CCHN), 43.0 (Ph*C*HCH$_3$), 19.3 (PhCH*C*H$_3$) and 14.1 (*C*H$_3$CH$_2$) (Found MH^+, 292.1195; $C_{15}H_{18}NO_5^+$ requires 292.1185) and the oxazolidinone (4*R*,2*R*)-*syn*-**15** (84 mg, 46%) as a white powder; mp 97–99 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.30; $[\alpha]_D^{20} = -24.8$ (*c* 5.3, $CHCl_3$) [lit.[14] (4*S*,2*S*)-*syn*-**15** $[\alpha]_D^{20} = +17.2$ (*c* 2.2, $CHCl_3$)]; ν_{max}($CHCl_3$)/cm^{-1} 1793 (C=O), 1747 (C=O) and 1705 (C=O); δ_H (270 MHz; $CDCl_3$) 7.40–7.20 (5H, m, 5 × CH; Ph), 5.03 (1H, q, *J* 7.0, PhC*H*CH$_3$), 4.94 (1H, dd, *J* 9.3 and 4.9, EtO$_2$CCHN), 4.52 (1H, t, *J* 9.3, C*H*$_A$H$_B$O), 4.23 (1H, dd, *J* 9.3 and 4.9, CH$_A$*H*$_B$O), 4.11 (2H, q, *J* 7.2, C*H*$_2$CH$_3$), 1.48 (3H, d, *J* 7.0, PhCHC*H*$_3$) and 1.11 (3H, t, *J* 7.2, C*H*$_3$CH$_2$); δ_C (62.9 MHz; $CDCl_3$) 174.3 (NC=O), 168.1 (CC=O), 152.0 (OC=O), 139.8 (*i*-C; Ph), 128.5, 128.2 and 127.2 (3 × CH; Ph), 64.3 (CH_2O), 62.4 (CH_2O), 55.7 (EtO$_2$CCHN), 43.2 (Ph*C*HCH$_3$), 19.4 (PhCH*C*H$_3$) and 13.9 (*C*H$_3$CH$_2$) (Found MH^+, 292.1195; $C_{15}H_{18}NO_5^+$ requires 292.1185).

4.5. Kinetic resolution of racemic oxazolidinones using (+)-pentafluorophenyl 6-methoxy-(2-naphthyl)propionate (*S*)-19

4.5.1. Synthesis of (4*S*,2*S*)-3-[2-(6-methoxynaphth-2-yl)-propionyl]-4-phenyl-oxazolidin-2-one *anti*-28 and (4*R*,2*S*)-3-[2-(6-methoxynaphth-2-yl)-propionyl]-4-phenyl-oxazolidin-2-one *syn*-28. In the same way as oxazolidinone **16**, *n*-BuLi (0.49 ml, 2.5 M in hexane, 1.22 mmol), (±)-4-phenyl-oxazolidin-2-one *rac*-**20** (0.20 g, 1.22 mmol) and (+)-pentafluoro-

phenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.24 g, 0.61 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *anti*- and *syn*-**28** [*anti*-/*syn*-21:79]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (*S*,*S*)-*anti*-**28** (32 mg, 14%) as a white solid; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.45; $[\alpha]_D^{20} = +218.4$ (*c* 2.0, $CHCl_3$); mp 120–121 °C; $\nu_{max}(CHCl_3)/cm^{-1}$ 1782 (NC=O) and 1705 (OC=O); δ_H (270 MHz; $CDCl_3$) 7.75 (1H, s, CH; Ar), 7.69 (2H, dd, *J* 8.6 and 2.5, 2 × CH; Ar and Ph), 7.49–7.30 (6H, m, 6 × CH; Ar and Ph), 7.15–7.10 (2H, m, 2 × CH, Ar and Ph), 5.31 (1H, dd, *J* 8.6 and 3.2, PhCHN), and 5.27 (1H, q, *J* 6.9, ArC*H*CH_3), 4.47 (1H, t, *J* 8.6, CH_AH_BO), 4.17 (1H, dd, *J* 8.6 and 3.2, CH_AH_BO), 3.90 (3H, s, CH_3O) and 1.48 (3H, d, *J* 6.9, CH_3CH); δ_C (100.6 MHz; $CDCl_3$) 174.1 (NC=O), 157.6 (OC=O), 153.2 (*i*-CO; Ar), 139.3, 135.3, 133.7 and 128.8 (4 × *i*-C; Ar and Ph), 129.2, 127.1, 126.8, 126.7, 118.9 and 105.5 (6 × CH; Ar), 128.8,[2] 128.6[1] and 125.7[2] (5 × CH; Ph), 69.6 (CH_2O), 58.0 (PhCHN), 55.2 (CH_3O), 43.0 (Ar*C*HCH$_3$) and 19.3 (ArCH*C*H$_3$) (Found MH^+, 376.1545; $C_{23}H_{22}NO_4$ requires 376.1543); and the oxazolidinone (*R*,*S*)-*syn*-**28** (0.121 g, 53%) as a white solid; mp 156–158 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.33; $[\alpha]_D^{27} = +194.3$ (*c* 1.6, $CHCl_3$); {lit.[9] +166.2, *c* 1.49, $CHCl_3$}; $\nu_{max}(CHCl_3)/cm^{-1}$ 1780 (NC=O), and 1699 (OC=O); δ_H (270 MHz; $CDCl_3$) 7.60 (1H, d, *J* 8.4, CH; Ar), 7.51 (1H, d, *J* 8.4, 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 or 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* 8.9, CH_AH_BO), 4.03 (1H, dd, *J* 8.9 and 5.2, CH_AH_BO), 3.92 (3H, s, CH_3O) and 1.44 (3H, d, *J* 6.9, ArCHCH_3); δ_C (100 MHz; $CDCl_3$) 173.6 (NC=O), 157.6 (OC=O), 153.0 (*i*-CO; Ar), 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 (Ar*C*HCH$_3$) and 18.7 (ArCH*C*H$_3$) (Found MH^+, 376.1553; $C_{23}H_{22}NO_4$ requires 376.1543).

4.5.2. Synthesis of (4*S*,2*S*)-4-isopropyl-3-[2-(6-methoxynaphth-2-yl)propionyl]-oxazolidin-2-one *anti*-27 and (4*R*,2*S*)-4-isopropyl-3-[2-(6-methoxynaphth-2-yl)propionyl]-oxazolidin-2-one *syn*-27. In the same way as oxazolidinone **16**, *n*-BuLi (0.62 ml, 2.5 M in hexane, 1.55 mmol), (±)-4-isopropyl-oxazolidin-2-one *rac*-**27** (0.20 g, 1.55 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.30 g, 0.77 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**27** [*syn*-/*anti*- 82:18]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (*S*,*S*)-*anti*-**27** (34 mg, 13%) as a white solid; mp 120–121 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.51; $[\alpha]_D^{20} = +167.5$ (*c* 1.4, $CHCl_3$); {$[\alpha]_D^{20} = +156.4$ (*c* 0.62, $CHCl_3$)};[19] $\nu_{max}(CHCl_3)/cm^{-1}$ 1778 (NC=O) and 1701 (OC=O); δ_H (270 MHz; $CDCl_3$) 7.70 (1H, s, CH; Ar), 7.68 (2H, dd, *J* 8.4 and 2.7, 2 × CH; Ar), 7.46 (1H, dd, *J* 8.7 and 1.6, CH; Ar), 7.14–7.09 (2H, m, 2 × CH; Ar), 5.28 (1H, q, *J* 6.9, ArCH), 4.36–4.31 (1H, dt, *J* 9.1 and 3.2, CHN), 4.10 (1H, dd, *J* 9.1 and 3.2, CH_AH_BO), 4.05 (1H, t, *J* 9.1, CH_AH_BO), 3.88 (3H, s, CH_3O), 2.50–2.39 (1H, m, C*H*$(CH_3)_2$), 1.57 (3H, d, *J* 7.2, ArC*H*CH_3) and 0.90 (6H, ~d, *J* 6.9, 2 × CH_3, CH_3CHCH_3); δ_C (100 MHz; $CDCl_3$) 174.7 (NC=O), 157.6 (OC=O), 153.7 (*i*-CO; Ar), 135.4, 133.8 and 128.8 (3 × *i*-CC; Ar), 129.3, 127.0, 126.8, 126.6, 118.8 and 105.5 (6 × CH; Ar), 63.0 (CH_2O), 59.0 (*i*-PrCHN), 55.2 (OCH_3), 42.8 (Ar*C*HCH$_3$), 28.5 (*C*H$(CH_3)_2$), 19.6 (CH_3; *i*-Pr), 17.9 (CH_3; *i*-Pr), and 14.6 (ArCH*C*H$_3$) (Found MH^+, 342.1707; $C_{20}H_{24}NO_4$ requires 342.1700); and the oxazolidinone (*R*,*S*)-*syn*-**27** (0.15 g, 56%) as an oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1) 0.34; $[\alpha]_D^{22} = +59.6$ (*c* 3.3, $CHCl_3$); $\nu_{max}(CHCl_3)/cm^{-1}$ 1778 (NC=O) and 1701 (OC=O); δ_H (270 MHz; $CDCl_3$) 7.72 (1H, s, CH; Ar), 7.67 (2H, br d, *J* 8.4, 2 × CH; Ar), 7.45 (1H, dd, *J* 8.4 and 1.6, CH; Ar), 7.13–7.09 (2H, m, 2 × CH; Ar), 5.26 (1H, q, *J* 6.9, ArC*H*CH_3), 4.52–4.46 (1H, dt, *J* 8.9 and 3.3, CHN), 4.21 (1H, t, *J* 8.9, CH_AH_BO), 4.06 (1H, dd, *J* 8.9 and 3.3, CH_AH_BO), 3.88 (3H, s, CH_3O), 2.25–2.13 (1H, m, C*H*$(CH_3)_2$), 1.53 (3H, d, *J* 6.9, ArCHCH_3), 0.75 (3H, d, *J* 6.9, CH_3^ACHCH_3^B) and 0.38 (3H, d, *J* 6.9, CH_3^ACHCH_3^B); δ_C (100 MHz; $CDCl_3$) 174.6 (NC=O), 157.6 (OC=O), 153.3 (*i*-CO; Ar), 135.7, 133.7 and 128.9 (3 × *i*-CC; Ar), 129.4, 127.0, 126.7, 126.6, 118.8 and 105.5 (6 × CH; Ar), 62.9 (CH_2O), 58.1 (*i*-PrCHN), 55.3 (OCH_3), 43.2 (Ar*C*HCH$_3$), 27.9 (*C*H$(CH_3)_2$), 18.7 (CH_3; *i*-Pr), 17.7 (CH_3; *i*-Pr) and 14.0 (ArCH*C*H$_3$) (Found MH^+, 342.1701; $C_{20}H_{24}NO_4$ requires 342.1700).

4.5.3. Synthesis of (4*S*,2*S*)-4-benzyl-3-[2-(6-methoxynaphth-2-yl)-propionyl]-oxazolidin-2-one *anti*-29 and (4*R*,2*S*)-4-benzyl-3-[2-(6-methoxynaphth-2-yl)-propionyl]-oxazolidin-2-one *syn*-29. In the same way as oxazolidinone **16**, *n*-BuLi (0.44 ml, 2.5 M in hexane, 1.12 mmol), (±)-4-benzyl-oxazolidin-2-one *rac*-**21** (0.20 g, 1.12 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.22 g, 0.56 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**29** [*syn*-/*anti*- 73:27]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (*S*,*S*)-*anti*-**29** (45 mg, 21%) as a white solid; mp 77–79 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.42; $[\alpha]_D^{30} = +135.6$ (*c* 0.73, $CHCl_3$); $\nu_{max}(CHCl_3)/cm^{-1}$ 1780 (C=O) and 1697 (C=O); δ_H (270 MHz; $CDCl_3$) 7.74 (1H, s, CH; Ar), 7.69 (2H, d, *J* 8.5, 2 × CH; Ar), 7.48 (1H, dd, *J* 8.4 and 1.7, CH; Ar), 7.37–7.09 (7H, m, 7 × CH, Ar and Ph), 5.26 (1H, q, *J* 7.2, ArC*H*CH_3), 4.62–4.54 (1H, m, BnCHN), 4.08 (1H, dd, *J* 9.1 and 2.4, CH_AH_BO), 3.97 (1H, t, *J* 9.1, CH_AH_BO), 3.89 (3H, s, CH_3O), 3.36 (1H, dd, *J* 13.1 and 3.2, CH_AH_BPh), 2.82 (1H, dd, *J* 13.1 and 3.2, CH_AH_BPh) and 1.62 (3H, d, *J* 6.9, ArC*H*CH_3); δ_C (100 MHz; $CDCl_3$) 174.7 (NC=O), 157.6 (OC=O), 152.9 (*i*-CO; Ar), 135.3, 135.4, 133.8 and 129.8 (4 × *i*-C; Ar and Ph), 129.3, 127.1, 126.7, 126.6, 118.9 and 105.5 (6 × CH; Ar), 128.9,[2] 128.8[2] and 127.3[1] (5 × CH; Ph), 65.8 (CH_2O), 55.8 (BnNCH), 55.2 (CH_3O), 42.9 (Ar*C*HCH$_3$), 37.9 (*C*H_2Ph) and 19.4 (Ar*C*HCH_3) (Found MH^+, 390.1702; $C_{24}H_{24}NO_4$ requires 390.1700) and the oxazolidinone *syn*-(*S*,*R*)-**29** (0.126 g, 58%) as a white solid; mp 61–63 °C; R_F [light petroleum

(bp 40–60 °C)/diethyl ether (1:1)] 0.35; $[\alpha]_D^{23} = +29.2$ (*c* 1.2, $CHCl_3$); {$[\alpha]_D^{30} = +22.8$ (*c* 0.91, $CHCl_3$)};[18,19] $\nu_{max}(CHCl_3)$/cm^{-1} 1778 (C=O) and 1699 (C=O); δ_H (270 MHz; $CDCl_3$) 7.82 (1H, s, CH; Ar), 7.73 (2H, d, *J* 8.4, 2 × CH; Ar and/or Ph), 7.54 (1H, dd, *J* 8.4 and 1.5, CH; Ar), 7.17–7.02 (5H, m, 5 × CH; Ar and Ph), 6.88 (2H, d, *J* 7.1, 2 × CH; Ar and/or Ph), 5.26 (1H, q, *J* 6.9, ArC*H*CH_3), 4.79–4.71 (1H, m, BnCHN), 4.16 (1H, t, *J* 8.9, CH_AH_BO), 4.04 (1H, dd, *J* 8.9 and 3.1, CH_AH_BO), 3.91 (3H, s, CH_3O), 3.06 (1H, dd, *J* 13.6 and 3.5, CH_AH_BPh), 2.55 (1H, dd, *J* 13.6 and 8.7, CH_AH_BPh) and 1.60 (3H, d, *J* 6.9, CH_3CH); δ_C (100 MHz; $CDCl_3$) 174.5 (NC=O), 157.7 (OC=O), 152.9 (*i*-CO; Ar), 135.2, 134.8, 133.7 and 129.7 (4 × *i*-C; Ar and Ph), 129.3, 127.2, 126.6, 125.9, 118.9 and 105.5 (6 × CH; Ar), 128.9[2], 128.7[2] and 128.5[1] (5 × CH; Ph), 65.8 (CH_2O), 55.2 (CH_3O), 54.8 (BnCHN), 43.0 (Ar*C*HCH$_3$), 37.3 (*C*H_2Ph) and 19.0 (ArCH*C*H$_3$) (Found MH^+, 390.1706; $C_{24}H_{24}NO_4$ requires 390.1700).

4.5.4. Synthesis of (4*S*,5*R*,2*S*)-3-[2-(6-methoxynaphth-2-yl)-propionyl]-4-methyl-5-phenyl-oxazolidin-2-one *anti*-30 and (4*R*,5*S*,2*S*)-3-[2-(6-methoxynaphth-2-yl)]-4-methyl-5-phenyl-oxazolidin-2-one *syn*-30. In the same way as oxazolidinone **16**, *n*-BuLi (0.45 ml, 2.5 M in hexane, 1.12 mmol), (±)-4-methyl-5-phenyl-oxazolidin-2-one *rac*-**22** (0.20 g, 1.12 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.22 g, 0.56 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**30** [*syn*-/*anti*- 67:33]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (4*S*,5*R*,2*S*)-*anti*-**30** (37 mg, 17%) as a white solid; mp 140–142 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.65; $[\alpha]_D^{23} = +88.9$ (*c* 1.2, $CHCl_3$); $\nu_{max}(CHCl_3)$/cm^{-1} 1776 (C=O) and 1697 (C=O); δ_H (270 MHz; $CDCl_3$) 7.76 (1H, s, CH; Ar), 7.71 (2H, dd, *J* 8.7 and 2.5, 2 × CH; Ar), 7.49 (1H, dd, *J* 8.4 and 1.7, CH; Ar), 7.40–7.22 (5H, m, 5 × CH; Ar and Ph), 7.25–7.11 (2H, m, 2 × CH; Ar and Ph), 5.43 (1H, d, *J* 7.1, PhCHO), 5.27 (1H, q, *J* 6.9, ArC*H*CH_3), 4.71–4.61 (1H, m, CH_3C*H*N), 3.90 (3H, s, CH_3O), 1.57 (3H, d, *J* 6.9, ArCHC*H*$_3$) and 0.94 (3H, *J* 6.4, C*H*$_3$CHN); δ_C (100 MHz; $CDCl_3$) 174.5 (NC=O), 157.5 (OC=O), 152.6 (*i*-CO; Ar), 135.6, 133.7, 133.2 and 129.0 (4 × *i*-C; Ar and Ph), 129.3, 127.1, 126.7, 126.6, 118.9 and 105.5 (6 × CH; Ar), 128.8[1], 128.7[2] and 125.6[2] (5 × CH; Ph), 78.6 (PhCHO), 55.4 (CH_3*C*HN), 55.3 (OCH_3), 43.2 (Ar*C*HCH$_3$), 19.2 (ArCH*C*H$_3$) and 14.5 (*C*H$_3$CHN); (Found MH^+, 390.1704; $C_{24}H_{24}NO_4$ requires 390.1700) and the oxazolidinone (4*R*,5*S*,2*S*)-*syn*-**30** (74 mg, 34%) as a white solid; mp 147–149 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.55; $[\alpha]_D^{23} = +142.9$ (*c* 1.4, $CHCl_3$); $\nu_{max}(CHCl_3)$/cm^{-1} 1782 (C=O) and 1701 (C=O); δ_H (270 MHz; $CDCl_3$) 7.74 (1H, s, CH; Ar), 7.70 (2H, br d, *J* 8.4, 2 × CH; Ar and/or Ph), 7.47 (1H, dd, *J* 8.4 and 1.5, CH; Ar), 7.30–7.27 (3H, m, 3 × CH; Ar and Ph), 7.15–7.11 (4H, m, 4 × CH; Ar and Ph), 5.62 (1H, d, *J* 7.4, PhCHO), 5.20 (1H, q, *J* 6.9, ArC*H*CH_3), 4.88–4.78 (1H, m, CH_3C*H*N), 3.90 (3H, s, CH_3O), 1.57 (3H, d, *J* 6.9, ArC*H*CH_3) and 0.71 (3H, d, *J* 6.4, C*H*$_3$CHN); δ_C (100 MHz; $CDCl_3$) 174.3 (NC=O), 157.6 (OC=O), 152.5 (*i*-CO; Ar), 135.4, 133.6, 133.3 and 128.9 (4 × *i*-C; Ar and Ph), 129.3, 127.1, 126.7, 126.7, 118.8 and 105.5 (6 × CH; Ar), 128.7[1], 128.5[2] and 125.6[2] (5 × CH; Ph), 78.7 (PhCHO), 55.3 (OCH_3), 54.6 (CH_3*C*HN), 43.5 (Ar*C*HCH$_3$), 19.3 (ArCH*C*H$_3$) and 14.1 (*C*H$_3$CHN) (Found MH^+, 390.1699; $C_{24}H_{24}NO_4$ requires 390.1700).

4.5.5. Synthesis of ethyl (4*R*,2*S*)-3-[2-(6-methoxynaphth-2-yl)-propionyl]-2-oxazolidine-4-carboxylate *anti*-26 and ethyl (4*S*,2*S*)-3-[2-(6-methoxynaphth-2-yl)]-2-oxazolidinone-4-carboxylate *syn*-26. In the same way as oxazolidinone **16**, *n*-BuLi (0.50 ml, 2.5 M in hexane, 1.25 mmol), (±)-ethyl-oxazolidin-2-one 4-carboxylate *rac*-**12** (0.20 g, 1.25 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.24 g, 0.62 mmol) in THF gave a separable mixture of two diastereoisomeric oxazolidinones *syn*- and *anti*-**26** [*syn*-/*anti*- 79:21]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinone (*S*,*R*)-*anti*-**26** (39 mg, 17%) as an oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.28; $[\alpha]_D^{23} = +146.8$ (*c* 0.92, $CHCl_3$); $\nu_{max}(CHCl_3)$/cm^{-1} 1791 (C=O), 1751 (C=O) and 1705 (C=O); δ_H (270 MHz; $CDCl_3$) 7.72 (1H, s, CH; Ar), 7.67 (2H, dd, *J* 8.4 and 2.6, 2 × CH; Ar), 7.46 (1H, dd, *J* 8.6 and 1.9, CH; Ar), 7.11 (1H, dd, *J* 8.4 and 2.6, CH; Ar), 7.07 (1H, s, CH; Ar), 5.24 (1H, q, *J* 6.9, ArC*H*CH_3), 4.76 (1H, dd, *J* 9.1 and 3.7, EtO_2CCHN), 4.37–4.20 (4H, m, 2 × CH_2O), 3.88 (3H, s, CH_3O), 1.58 (3H, d, *J* 6.9, C*H*$_3$CH) and 1.31 (3H, t, *J* 6.9, C*H*$_3$CH$_2$); δ_C (100 MHz; $CDCl_3$) 174.5 (NC=O), 168.6 (CC=O), 157.7 (OC=O), 152.0 (*i*-CO; Ar), 135.0, 133.8 and 128.8 (3 × *i*-C; Ar), 129.3, 127.1, 126.8, 126.7, 119.0 and 105.5 (6 × CH; Ar and Ph), 64.2 (CH_2O), 62.5 (CH_2O), 55.8 (CHN), 55.3 (CH_3O), 42.8 (Ar*C*HCH$_3$), 19.1 (ArCH*C*H$_3$) and 14.0 (*C*H$_3$CH$_2$) (Found MH^+, 372.1445, $C_{20}H_{22}NO_6$ requires 372.1442); and the oxazolidinone (*S*,*S*)-*syn*-**26** (0.14 g, 61%) as a white solid; mp 155–157 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.18; $[\alpha]_D^{25} = +55.7$ (*c* 3.0, $CHCl_3$); $\nu_{max}(CHCl_3)$/cm^{-1} 1789 (C=O), 1745 (C=O) and 1705 (C=O); δ_H (270 MHz; $CDCl_3$) 7.74 (1H, s, CH; Ar), 7.67 (2H, dd, *J* 8.4 and 2.6, 2 × CH; Ar), 7.44 (1H, dd *J* 8.6 and 1.8, CH; Ar), 7.13 (1H, dd, *J* 8.4 and 2.6, 2 × CH; Ar), 7.09 (1H, s, CH; Ar), 5.16 (1H, q, *J* 6.9, ArC*H*CH_3), 4.94 (1H, dd, *J* 9.7 and 4.9, EtO_2CCHN), 4.49 (1H, t, *J* 9.7, CH_AH_BCH), 4.20 (1H, dd, *J* 9.7 and 4.9, CH_AH_BCH), 4.07 (2H, q, *J* 7.2, CH_2CH$_3$), 3.88 (3H, s, CH_3O), 1.55 (3H, d, *J* 7.2, CH_3CH) and 1.03 (3H, t, *J* 7.2, C*H*$_3$CH$_2$); δ_C (100 MHz; $CDCl_3$) 174.3 (NC=O), 167.9 (CC=O), 157.6 (OC=O), 151.9 (*i*-CO; Ar), 134.8, 133.7, 128.9 (3 × *i*-C; Ar), 129.1, 127.1, 126.9, 126.8, 118.7 and 105.6 (6 × CH; Ar), 64.1 (CH_2O), 63.6 (CH_2O), 55.6 (CHN), 55.2 (CH_3O), 43.1 (Ar*C*HCH$_3$), 19.2 (ArCH*C*H$_3$) and 13.7 (*C*H$_3$CH$_2$) (Found MNH_4^+, 389.1703; $C_{20}H_{25}N_2O_6$ requires 389.1707).

4.5.6. Parallel kinetic resolution of racemic ethyl-oxazolidin-2-one 4-carboxylate *rac*-12 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.50 ml, 2.5 M in hexane, 0.125 mmol), (±)-ethyl-oxazolidin-2-one 4-carboxylate *rac*-**12** (0.20 g, 0.125 mmol), (−)-pentafluorophenyl 2-

phenylpropionate (*R*)-**14** (0.20 g, 0.625 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.25 g, 0.625 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**15** [*syn*-/*anti*- 95:5] and *syn*- and *anti*-**26** [*syn*-/*anti*- 95:5]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give oxazolidinones *syn*-**15** (0.10 g, 55%) and *syn*-**26** (0.139 g, 59%).

4.5.7. Parallel kinetic resolution of racemic 4-isopropyl-oxazolidin-2-one *rac*-13 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.62 ml, 2.5 M in hexane, 1.54 mmol), (±)-4-isopropyl-oxazolidin-2-one *rac*-**13** (0.20 g, 1.54 mmol), (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.24 g, 0.77 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.31 g, 0.77 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**16** [*syn*-/*anti*- 95:5] and *syn*- and *anti*-**27** [*syn*-/*anti*- 95:5]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give oxazolidinones *syn*-**16** (0.115 g, 57%) and *syn*-**27** (0.155 g, 59%).

4.5.8. Parallel kinetic resolution of racemic 4-phenyl-oxazolidin-2-one *rac*-20 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.48 ml, 2.5 M in hexane, 0.122 mmol), (±)-4-phenyl-oxazolidin-2-one *rac*-**20** (0.20 g, 0.122 mmol), (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.19 g, 0.61 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.24 g, 0.61 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**23** [*syn*-/*anti*- 95:5] and *syn*- and *anti*-**28** [*syn*-/*anti*- 95:5]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give oxazolidinones *syn*-**23** (0.180 g, 65%) and *syn*-**28** (0.139 g, 61%).

4.5.9. Parallel kinetic resolution of racemic 4-benzyl-oxazolidin-2-one *rac*-21 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.45 ml, 2.5 M in hexane, 1.12 mmol), (±)-4-benzyl-oxazolidin-2-one *rac*-**21** (0.20 g, 1.12 mmol), (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.17 g, 0.56 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.22 g, 0.56 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**24** [*syn*-/*anti*- 78:28] and *syn*- and *anti*-**29** [*syn*-/*anti*- 70:30]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *anti*-**24** (22 mg, 13%), a partially separable mixture (~86 mg) of *syn*-**24** (~58 mg, 34%) and *anti*-**29** (~28 mg, 13%), and *syn*-**29** (63 mg, 29%).

4.5.10. Parallel kinetic resolution of racemic 4-methyl-5-phenyl-oxazolidin-2-one *rac*-22 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.45 ml, 2.5 M in hexane, 1.12 mmol), (±)-4-methyl-5-phenyl-oxazolidin-2-one *rac*-**22** (0.20 g, 1.22 mmol), (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.17 g, 0.56 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.22 g, 0.56 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**25** [*syn*-/*anti*- 82:18] and *syn*- and *anti*-**30** [*syn*-/*anti*- 85:15]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *anti*-**25** (0.13 mg, 8%), an inseparable mixture (~77 mg) of *syn*-**25** (~62 mg, 36%) and *anti*-**30** (~15 mg, 7%), and *syn*-**30** (91 mg, 42%).

4.5.11. Pentafluorophenyl 2-(4-isobutylphenyl)propionate (*R*)-32. 2-(4-Isopropylphenyl)propionic acid (*R*)-**31** (0.50 g, 2.42 mmol) {$[\alpha]_D^{20} = -54.3$ (*c* 2.0, EtOH)} was added to a stirred solution of *N*,*N*′-dicyclohexylcarbodiimide (DCC) (0.50 g, 2.67 mmol) in dichloromethane (10 ml). The resulting solution was stirred for 10 min. A solution of pentafluorophenol (0.45 g, 0.25 ml, 2.42 mmol) in dichloromethane (5 ml) was slowly added, and the resulting solution was stirred for 12 h. The resulting precipitate (*N*,*N*′-dicyclohexylurea) was filtered off (using suction filtration). Water (10 ml) was added and the solution was extracted with dichloromethane (3 × 20 ml) and dried over $MgSO_4$. The combined organic layers were evaporated under reduced pressure. The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (9:1) to give pentafluorophenyl 2-(4-isopropylphenyl)propionate (*R*)-**32** (0.74 g, 82%) as a liquid; R_F [light petroleum (bp 40–60 °C)/diethyl ether (9:1)] 0.63; $[\alpha]_D^{25} = -91.4$ (*c* 5.0, $CHCl_3$); {for (*S*)-**32**; $[\alpha]_D^{25} = +91.7$ (*c* 29.6, $CHCl_3$)}; $\nu_{max}(CHCl_3)/cm^{-1}$ 1782 (C=O); δ_H (270 MHz; $CDCl_3$) 7.26 (2H, dt, *J* 8.2 and 2.2, 2 × CH; Ar), 7.14 (2H, dt, *J* 8.2 and 2.2, 2 × CH; Ar), 4.04 (1H, q, *J* 7.2, ArC*H*CH_3), 2.46 (2H, d, *J* 7.2, CH_2Ar), 1.92–1.80 (1H, m, C*H*$(CH_3)_2$), 1.62 (3H, d, *J* 7.2, ArCH*C*H_3), 0.99 (3H, d, *J* 6.7, $(CH_3)_ACH(CH_3)_B$) and 0.88 (3H, d, *J* 6.7, $(CH_3)_BCH(CH_3)_A$); δ_C (100 MHz; $CDCl_3$) 170.3 (OC=O), 140.8 (*i*-C; Ar), 141.2 (142.92 and 139.42, 2C, ddt, $^1J_{C,F} = 251.3$ Hz, $^2J_{C,F} = 11.9$ Hz and $^3J_{C,F} = 4.2$ Hz, C(2)–F), 138.9 (140.18 and 137.66, 1C, dtt, $^1J_{C,F} = 253.2$ Hz, $^2J_{C,F} = 13.8$ Hz and $^3J_{C,F} = 3.8$ Hz, C(4)–F), 137.3 (138.61 and 136.08, 2C, 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), 135.5 (*i*-C; Ar), 129.1 and 126.7 (2 × CH; Ar), 124.7 (1C, 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), 44.5 (CH_2Ar), 44.4 (Ar*C*HCH$_3$), 29.7 (*C*HCH_2), 21.9 (CH(*C*H$_3$)$_2$) and 18.0 (ArCH*C*H$_3$); δ_F (378 MHz; $CDCl_3$) −152.6 (2F, d, $^3J_{F,F}$ 18.5, F_{ortho}), −158.1 (1F, t, $^3J_{F,F}$ 20.8, F_{para}) and −162.4 (2F, dd, $^3J_{F,F}$ 20.8 and 18.5, F_{meta}) (Found M^+, 372.1144; $C_{19}H_{17}F_5O_2$ requires 372.1143).

4.5.12. Parallel kinetic resolution of racemic ethyl-oxazolidin-2-one 4-carboxylate *rac*-12 using a combination of quasi-enantiomeric oxazolidinones (*R*)-32 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.50 ml, 2.5 M in hexane, 1.25 mmol), (±)-ethyl-oxazolidin-2-one 4-carboxylate *rac*-**12** (0.20 g, 1.25 mmol), (−)-pentafluorophenyl 2-(4-isobutylphenyl)propionate (*R*)-**32** (0.23 g, 0.625 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)pro-

pionate (*S*)-**19** (0.25 g, 0.625 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**26** [*syn*-/*anti*- 96:4] and *syn*- and *anti*-**33** [*syn*-/*anti*- 96:4]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *syn*-**26** (0.106 g, 46%) and *syn*-**33** (0.89 mg, 41%).

Characterisation data for: *Ethyl 3-[2-(4-isobutylphenyl)-propionyl]-2-oxo-oxazolidine-4-carboxylate (4S,2R)-anti*-**33**: Oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.53; $[\alpha]_D^{25} = -125.4$ (*c* 1.2, $CHCl_3$); $\nu_{max}(CHCl_3)/cm^{-1}$ 1791 (C=O), 1751 (C=O) and 1701 (C=O); δ_H (270 MHz; $CDCl_3$) 7.23 (2H, d, *J* 8.0, 2 × CH; Ar), 7.10 (2H, d, *J* 8.0, 2 × CH; Ar), 5.09 (1H, q, *J* 6.9, ArC*H*CH_3), 4.78 (1H, dd, *J* 9.4 and 3.7, EtO_2CCHN), 4.41 (1H, t, *J* 9.0, CH_AH_BO), 4.33–4.23 (3H, m, 3 × CH, CH_AH_BO and CH_2CH_3), 2.42 (2H, d, *J* 7.2, CH_2), 1.87–1.77 (1H, m, C*H*$(CH_3)_2$), 1.49 (3H, d, *J* 6.9, ArCHCH_3), 1.30 (3H, t, *J* 7.2, CH_3CH_2) and 0.88 (6H, d, *J* 6.7, $CH_3^ACHCH_3^B$); δ_C (100.6 MHz; $CDCl_3$) 174.7 (NC=O), 168.6 (CC=O), 152.0 (OC=O), 140.8 (*i*-C; Ar), 137.1 (*i*-C; Ar), 129.4 and 127.9 (2 × CH; Ar), 64.2 (CH_2O), 62.5 (CH_2O; ester), 55.9 (EtO_2CCHN), 45.1 ($CH(CH_3)_2$), 42.5 (Ar*C*HCH_3), 30.2 (CH_2), 22.4 ($CH_3^ACHCH_3^B$), 19.2 (ArCH*C*H_3) and 14.0 (CH_3CH_2) (Found MNH_4^+, 365.2069; $C_{19}H_{29}N_2O_5$ requires 365.2171). *Ethyl 3-[2-(4-isobutylphenyl)-propionyl]-2-oxo-oxazolidine-4-carboxylate (4R,2R)-syn*-**33**: Oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.35; $[\alpha]_D^{25} = -25.5$ (*c* 5.0, $CHCl_3$); {for (4*S*,2*S*)-**33**; $[\alpha]_D^{25} = +29.8$ (*c* 0.95, $CHCl_3$)};[18] $\nu_{max}(CHCl_3)/cm^{-1}$ 1791 (C=O), 1747 (C=O) and 1699 (C=O); δ_H (270 MHz; $CDCl_3$) 7.23 (2H, d, *J* 8.1, 2 × CH; Ar), 7.10 (2H, d, *J* 8.1, 2 × CH; Ar), 5.01 (1H, q, *J* 6.9, ArC*H*CH_3), 4.93 (1H, dd, *J* 9.4 and 4.7, EtO_2CCHN), 4.51 (1H, t, *J* 9.4, CH_AH_BO), 4.24 (1H, dd, *J* 9.4 and 4.7, CH_AH_BO), 4.10 (2H, q, *J* 7.2, CH_2CH_3), 2.42 (2H, d, *J* 7.2, CH_2), 1.87–1.77 (1H, m, C*H*$(CH_3)_2$), 1.46 (3H, d, *J* 6.9, ArCHCH_3), 1.10 (3H, t, *J* 7.2, CH_3CH_2) and 0.88 (6H, d, *J* 6.7, $CH_3^ACHCH_3^B$); δ_C (100.6 MHz; $CDCl_3$) 174.5 (NC=O), 168.0 (OC=O), 152.0 (*i*-C; Ar), 140.5 (*i*-C; Ar), 136.9 (*i*-C; Ph), 129.2 and 127.9 (2 × CH; Ar), 64.2 (CH_2O), 62.3 (CH_2O; ester), 55.7 (EtO_2CCHN), 45.1 ($CH(CH_3)_2$), 42.7 (Ar*C*HCH_3), 30.1 (CH_2), 22.4 ($CH_3^ACHCH_3^B$), 19.3 (ArCH*C*H_3) and 13.8 (CH_3CH_2) (Found MNH_4^+, 365.2073; $C_{19}H_{29}N_2O_5$ requires 365.2071).

4.5.13. Parallel kinetic resolution of racemic 4-isopropyl-oxazolidin-2-one *rac*-13 using a combination of quasi-enantiomeric oxazolidinones (*R*)-32 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.61 ml, 2.5 M in hexane, 1.54 mmol), (±)-4-isopropyl-oxazolidin-2-one *rac*-**13** (0.20 g, 1.54 mmol), (−)-pentafluorophenyl 2-(4-isobutylphenyl)propionate (*R*)-**32** (0.28 g, 0.77 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.30 g, 0.77 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**27** [*syn*-/*anti*- 95:5] and *syn*- and *anti*-**34** [*syn*-/*anti*- 95:5]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *syn*-**27** (0.19 g, 74%) and *syn*-**34** (0.17 g, 70%).

Characterisation data for: *3-[2-(4-Isobutylphenyl)-propionyl]-4-isopropyl-oxazolidin-2-one (4R,2R)-anti*-**34**: Oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.77; $[\alpha]_D^{20} = -87.5$ (*c* 12.0, $CHCl_3$); [(4*S*,2*S*)-*anti*-**34**; $[\alpha]_D^{20} = +117.3$ (*c* 1.3, $CHCl_3$)]; $\nu_{max}(CHCl_3)/cm^{-1}$ 1776 (C=O) and 1692 (C=O); δ_H (250 MHz; $CDCl_3$) 7.23 (2H, d, *J* 8.2, 2 × CH; Ar), 7.07 (2H, d, *J* 8.2, 2 × CH; Ar), 5.11 (1H, q, *J* 7.2, ArC*H*CH_3), 4.37–4.32 (1H, dt, *J* 7.2 and 3.8, *i*-PrC*H*N), 4.15–4.07 (2H, m, CH_2O), 2.46–2.39 (2H, m, CH_2), 1.87–1.77 (1H, m, C*H*$(CH_3)_2$), 1.49 (3H, d, *J* 7.2, ArCHCH_3) and 0.92–8.86 (12H, m, 2 × $CH(CH_3)_2$); δ_C (100.6 MHz; $CDCl_3$) 174.9 (NC=O), 153.6 (OC=O), 140.6 (*i*-C; Ar), 137.5 (*i*-C; Ar), 129.3 and 127.8 (2 × CH; Ar), 63.1 (CH_2O), 59.0 (*i*-PrC*H*N), 45.1 ($CH(CH_3)_2$), 42.6 (Ar*C*HCH_3), 30.2 (CH_2), 28.6 ($CH(CH_3)_2$), 22.7 ($CH_3^ACHCH_3^B$; *i*-BuC_6H_4–), 22.4 ($CH_3^ACHCH_3^B$; *i*-BuC_6H_4–), 19.7 ($CH_3^ACHCH_3^B$; oxazolidinone), 18.0 ($CH_3^ACHCH_3^B$; oxazolidinone) and 14.7 (ArCH*C*H_3) (Found MH$^+$, 318.20062; $C_{19}H_{28}NO_3$ requires 318.2064). *3-[2-(4-Isobutylphenyl)-propionyl]-4-isopropyl-oxazolidin-2-one (4S,2R)-syn*-**34**: Oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1) 0.55; $[\alpha]_D^{22} = -33.0$ (*c* 1.2, $CHCl_3$) {for (4*R*,2*S*)-*syn*-**34** $[\alpha]_D^{22} = +34.2$ (*c* 2.0, $CHCl_3$)}; $\nu_{max}(CHCl_3)/cm^{-1}$ 1778 (C=O) and 1699 (C=O); δ_H (400 MHz; $CDCl_3$) 7.23 (2H, d, *J* 8.2, 2 × CH; Ar), 7.03 (2H, d, *J* 8.2, 2 × CH; Ar), 5.11 (1H, q, *J* 6.9, ArC*H*CH_3), 4.50–4.44 (1H, dt, *J* 8.80 and 3.48, *i*-PrC*H*N), 4.21 (1H, t, *J* 8.6, CH_AH_BO), 4.07 (1H, dd, *J* 8.6 and 3.5, CH_AH_BO), 2.40 (2H, d, *J* 7.2, CH_2), 2.19–2.07 (1H, m, C*H*$(CH_3)_2$), 1.87–1.75 (1H, m, C*H*$(CH_3)_2$), 1.44 (3H, d, *J* 6.9, ArCHCH_3), 0.85 (3H, d, *J* 6.7, CH_3; $CH_3^ACHCH_3^B$), 0.84 (3H, d, *J* 6.7, CH_3; $CH_3^ACHCH_3^B$), 0.76 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$) and 0.38 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$); δ_C (100.6 MHz; $CDCl_3$) 174.8 (NC=O), 153.5 (OC=O), 140.6 (*i*-C; Ar), 137.6 (*i*-C; Ar), 129.3 and 127.8 (2 × CH; Ar), 62.8 (CH_2O), 58.0 (*i*-PrC*H*N), 45.0 ($CH(CH_3)_2$), 42.9 (Ar*C*HCH_3), 30.2 (CH_2), 27.8 ($CH(CH_3)_2$; oxazolidinone), 22.7 ($CH_3^ACHCH_3^B$; *i*-BuC_6H_4–), 22.3 ($CH_3^ACHCH_3^B$; *i*-BuC_6H_4–), 18.5 ($CH_3^ACHCH_3^B$; oxazolidinone), 17.7 ($CH_3^ACHCH_3^B$; oxazolidinone) and 14.0 (ArCH*C*H_3) (Found M, 317.1979; $C_{29}H_{27}NO_3$ requires 317.1985).

4.5.14. Parallel kinetic resolution of racemic 4-phenyl-oxazolidin-2-one *rac*-20 using a combination of quasi-enantiomeric oxazolidinones (*R*)-32 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.48 ml, 2.5 M in hexane, 1.22 mmol), (±)-4-phenyl-oxazolidin-2-one *rac*-**20** (0.20 g, 1.22 mmol), (−)-pentafluorophenyl 2-(4-isobutylphenyl)-propionate (*R*)-**32** (0.22 g, 0.61 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.24 g, 0.61 mmol) in THF gave a separable pair of diastereoisomeric oxazolidinones *syn*- and *anti*-**28** [*syn*-/*anti*- 95:5] and *syn*- and *anti*-**35** [*syn*-/*anti*- 97:3]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *syn*-**28** (0.11 g, 48%) and *syn*-**35** (0.11 g, 54%).

Characterisation data for: *3-[2-(4-Isobutylphenyl)propionyl]-4-phenyl-oxazolidin-2-one* (*4R,2R*)-*anti*-**35**: White solid; mp 155–158 °C; R_F [light petroleum (bp 40–60 °C)/

diethyl ether (1:1)] 0.62; $[\alpha]_D^{25} = -151.3$ (c 1.3, $CHCl_3$) {for (*S*,*S*)-*anti*-**35**; $[\alpha]_D^{25} = +144.5$ (c 7.2, $CHCl_3$)}; $\nu_{max}(CHCl_3)/cm^{-1}$ 1780 (C=O) and 1701 (C=O); δ_H (270 MHz; $CDCl_3$) 7.28–7.15 (3H, m, 3 × CH; Ph and/or Ar), 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, PhCHN), 5.09 (1H, q, J 6.9; ArCHCH$_3$), 4.63 (1H, t, J 9.0, CH_AH$_B$O), 4.06 (1H, dd, J 9.0 and 5.2, CH$_A$$H_B$O), 2.43 (2H, d, J 7.4, CH$_2$), 1.89–1.79 (1H, m, CH(CH$_3$)$_2$), 1.38 (3H, d, J 6.9, ArCHCH_3) and 0.90 (6H, d, J 6.7, 2 × CH$_3$, CH_3^ACHCH_3^B); δ_C (100.6 MHz; $CDCl_3$) 174.3 (NC=O), 153.3 (OC=O), 140.7 (*i*-C; Ar), 139.4 (*i*-C; Ar), 137.4 (*i*-C; Ph), 129.3 and 127.0 (2 × CH; Ar), 129.2, 128.7 and 125.8 (3 × CH; Ph), 69.7 (CH$_2$O), 58.1 (PhCHN), 45.1 (*C*H(CH$_3$)$_2$), 42.7 (Ar*C*HCH$_3$), 30.2 (CH$_2$), 22.4 (2C, s, CH_3^ACHCH_3^B) and 19.4 (ArCH*C*H$_3$) (Found MH$^+$, 352.1909; $C_{22}H_{26}NO_3$ requires 352.1907). *3-[2′-(4-Isobutylphenyl)propionyl]-4-phenyl-oxazolidin-2-one* (*4S*,*2R*)-*syn*-**35**: White solid; mp 86–88 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.41; $[\alpha]_D^{25} = -114.6$ (c 4.2, $CHCl_3$); {lit.[9] $[\alpha]_D^{25} = -99.1$ (c 0.4, $CHCl_3$)}; [(4*R*,2*S*)-**35**; $[\alpha]_D^{25} = +123.8$ (c 1.0, $CHCl_3$)]; $\nu_{max}(CHCl_3)/cm^{-1}$ 1779 (C=O) and 1705 (C=O); δ_H (270 MHz; $CDCl_3$) 7.28–7.15 (3H, m, 3 × CH; Ph and/or Ar), 7.00 (4H, m, 4 × CH, Ph and Ar), 6.90 (2H, dt, J 7.9 and 1.9, 2 × CH; Ar), 5.44 (1H, dd J 9.2 and 5.2, CHN), 5.09 (1H, q, J 6.9; ArCH), 4.63 (1H, t, J 9.0, CH_AH$_B$O), 4.06 (1H, dd, J 9.0 and 5.2, CH$_A$$H_B$O), 2.43 (2H, d, J 7.4, CH$_2$), 1.89–1.79 (1H, m, CH(CH$_3$)$_2$), 1.38 (3H, d, J 6.9, CH_3CH) and 0.90 (6H, d, J 6.7, 2 × CH$_3$, 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 and 127.0 (2 × CH; Ar), 129.2, 128.7 and 125.8 (3 × CH; Ph), 69.7 (CH$_2$O), 58.1 (CHN), 45.1 (*C*H(CH$_3$)$_2$), 42.7 (ArCH), 30.2 (CH$_2$), 22.4 (2C, s, CH_3^ACHCH_3^B) and 19.4 (*C*H$_3$CH$_2$) (Found MH$^+$, 352.1909; $C_{22}H_{26}NO_3$ requires 352.1907); m/z 351.1 (10% M$^+$), 188.1 (Ar(CH$_3$)C=C=O$^+$), 161.1 (10, Ar$^+$CHCH$_3$), 145.1 (145, ArCH$_2^+$) and 77.1 (10, Ph$^+$).

4.5.15. Mutual kinetic separation of 4-isopropyl-oxazolidin-2-one (*S*)-13 and 4-phenyl-oxazolidin-2-one (*R*)-20 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (1.23 ml, 2.5 M in hexane, 3.08 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13**(0.20 g, 1.54 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**20** (0.25 g, 1.54 mmol), (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.48 g, 1.54 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.61 g, 1.54 mmol) in THF gave a separable mixture of oxazolidinones *syn*-**16** and *anti*-**27** [*syn*-/*anti*- 95:5], and *syn*-**28** and *anti*-**23** [*syn*-/*anti*- 95:5]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give oxazolidinones *syn*-**16** (0.40 g, 67%) and *syn*-**28** (0.57 g, 67%).

4.5.16. Mutual kinetic separation of 4-isopropyl-oxazolidin-2-one (*S*)-13 and ethyl-oxazolidin-2-one 4-carboxylate (*S*)-12 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.30 ml, 2.5 M in hexane, 0.752 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (48 mg, 0.376 mmol), ethyl-oxazolidin-2-one 4-carboxylate (*S*)-**12** (60 mg, 0.37 mmol), (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.12 g, 0.376 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.149 g, 0.376 mmol) in THF gave a separable mixture of oxazolidinones *syn*-**16** and *anti*-**27** [*syn*-/*anti*- 92:8], and *syn*-**26** and *anti*-**15** [*syn*-/*anti*- 95:5]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *syn*-**16** (96 mg, 65%) and *syn*-**26** (93 mg, 66%).

4.5.17. Mutual kinetic separation of 4-phenyl-oxazolidin-2-one (*S*)-20 and ethyl-oxazolidin-2-one 4-carboxylate (*S*)-12 using a combination of quasi-enantiomeric oxazolidinones (*R*)-14 and (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.32 ml, 2.5 M in hexane, 0.79 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**20** (64 mg, 0.395 mmol), ethyl-oxazolidin-2-one 4-carboxylate (*S*)-**12** (62 mg, 0.395 mmol), (−)-pentafluorophenyl 2-phenylpropionate (*R*)-**14** (0.125 g, 0.395 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.156 g, 0.395 mmol) in THF gave a separable mixture of oxazolidinones *syn*-**23** and *anti*-**28** [*syn*-/*anti*- 98:2], and *syn*-**26** and *anti*-**15** [*syn*-/*anti*- 98:2]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *syn*-**23** (70 mg, 60%) and *syn*-**26** (82 mg, 56%).

4.5.18. Synthesis of (+)-2-phenylpropionyl chloride (*S*)-36. Oxalyl chloride (0.93 g, 7.33 mmol) was added to a stirred solution of (*S*)-2-phenylpropionic acid (1.0 g, 6.66 mmol) {$[\alpha]_D^{20} = +72.0$ (c 1.4, $CHCl_3$)} in dry toluene (10 ml) at room temperature. The resulting solution was stirred for 12 h. The organic solvent was removed under reduced pressure to give 2-phenylpropionyl chloride (*S*)-**36** (0.95 g, 85%) as an oil; $[\alpha]_D^{25} = +73.2$ (c 4.0, $CHCl_3$) {lit.[20] (*S*)-**36**; $[\alpha]_D^{25} = +74.2$ (c 2.8, $CHCl_3$); lit.[21] (*R*)-**36**; $[\alpha]_D^{25} = -72.6$, $CHCl_3$}; 1782 (C=O), δ_H (270 MHz, $CDCl_3$) 7.36–7.28 (5H, m, 5 × CH; Ar), 4.11 (1H, q, J 7.1, PhCHCH$_3$) and 1.59 (3H, d, J 7.1, PhCHCH_3); δ_C (100 MHz, $CDCl_3$) 175.9 (C=O), 140.2 (*i*-C; Ph), 129.9, 129.3 and 129.2 (3 × CH; Ph), 57.9 (Ph*C*HCH$_3$) and 18.6 (PhCH*C*H$_3$) (Found M(^{35}Cl), 168.0337; C_9H_9ClO requires 168.0336).

4.5.19. Sequential kinetic resolution of 4-isopropyl-oxazolidin-2-one *rac*-13 using pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-19 and then 2-phenylpropionyl chloride (*S*)-36. In the same way as oxazolidinone **16**, *n*-BuLi (0.62 ml, 2.5 M in hexane, 1.55 mmol), 4-isopropyl-oxazolidin-2-one *rac*-**13** (0.20 g, 1.55 mmol) and (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.31 g, 0.77 mmol) in THF, and after 1 h followed by 2-phenylpropionyl chloride (*S*)-**36** (0.13 g, 0.77 mmol), gave a separable mixture of oxazolidinones *syn*-**16** and *anti*-**16** [*syn*-/*anti*- 12:88], and *syn*-**27** and *anti*-**27** [*syn*-/*anti*- 85:15]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *anti*- and *syn*-**16** (0.17 g, 84%) and *anti*- and *syn*-**27** (0.22 g, 84%).

4.5.20. Sequential kinetic resolution of 4-isopropyl-oxazolidin-2-one *rac*-13 using 2-phenylpropionyl chloride (*S*)-36 and then pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.62 ml, 2.5 M in hexane, 1.55 mmol), 4-isopropyl-oxazolidin-2-one *rac*-**13** (0.20 g, 1.55 mmol) and 2-phenylpropionyl chloride (*S*)-**36** (0.13 g, 0.77 mmol) in THF, and after 1 h followed by (+)-pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate (*S*)-**19** (0.31 g, 0.77 mmol), gave a separable mixture of oxazolidinones *syn*-**16** and *anti*-**16** [*syn*-/*anti*- 40:60], and *syn*-**27** and *anti*-**27** [*syn*-/*anti*- 92:8]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *anti*- and *syn*-**16** (0.11 g, 55%), and *anti*- and *syn*-**27** (0.15 g, 59%).

4.5.21. Pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate *rac*-19. In the same way as active ester (*R*)-**32**, racemic 2-(6-methoxynaphth-2-yl)propionic acid (2.13 g, 9.25 mmol), pentafluorophenol (1.7 g, 9.25 mmol) and DCC (2.1 g, 10.18 mmol) in CH_2Cl_2 (30 ml) gave after purification by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (9:1), the pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate *rac*-**19** as a white solid; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.65; mp 51–53 °C; $\nu_{max}(CHCl_3)/cm^{-1}$ 1781 (C=O); δ_H (400 MHz; $CDCl_3$) 7.78 (1H, d, *J* 8.5, CH; Ar), 7.76 (1H, d, *J* 1.8, CH; Ar), 7.75 (1H, d, *J* 8.8, CH; Ar), 7.46 (1H, dd, *J* 8.5 and 1.8, CH; Ar), 7.19 (1H, dd, *J* 8.8 and 2.6, CH; Ar) 7.15 (1H, d, *J* 2.6, CH; Ar), 4.38 (1H, q, *J* 7.2, ArC*H*CH_3) 3.91 (3H, s, OCH_3) and 1.71 (3H, d, *J* 7.2, ArCHCH_3); δ_C (100 MHz; $CDCl_3$) 170.7 (C=O), 157.9 (*i*-CO; Ar), 141.0 (142.32 and 139.8, 2C, ddt, $^1J_{C,F} = 249.8$ Hz, $^2J_{C,F} = 12.2$ Hz and $^3J_{C,F} = 4.6$ Hz, C(2)–F), 139.3 (140.63 and 138.11, 1C, dtt, $^1J_{C,F} = 252.1$ Hz, $^2J_{C,F} = 13.0$ Hz and $^3J_{C,F} = 4.5$ Hz, C(4)–F), 137.8 (139.04 and 136.54, 2C, dtdd, $^1J_{C,F} = 250.6$ Hz, $^2J_{C,F} = 13.8$ Hz, $^3J_{C,F} = 5.3$ and $^4J_{C,F} = 3.0$ Hz, C(3)–F) , 133.9, 133.7 and 128.9 (3 × *i*-C; Ar), 129.3, 127.5, 126.2, 125.7, 119.3 and 105.6 (6 × CH; Ar), 125.2 (1C, m, *i*-CO; OC_6F_5), 55.3 (OCH_3), 45.9 (Ar*C*HCH$_3$) and 18.5 (ArCH*C*H$_3$); δ_F (378 MHz; $CDCl_3$) −152.5 (2F, d, $^3J_{F,F}$ 17.0, F_{ortho}), −157.9 (1F, t, $^3J_{F,F}$ 21.6, F_{para}) and −162.3 (2F, dd, $^3J_{F,F}$ 21.6 and 17.0, F_{meta}) (Found M^+, 396.0783; $C_{20}H_{13}F_5O_3^+$ requires 396.0779).

4.5.22. Mutual kinetic resolution of 4-isopropyl-oxazolidin-2-one *rac*-13 using pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate *rac*-19. In the same way as oxazolidinone **16**, *n*-BuLi (0.72 ml, 2.5 M in hexane, 1.79 mmol), 4-isopropyl-oxazolidin-2-one *rac*-**13** (0.21 g, 1.63 mmol) and pentafluorophenyl 2-(6-methoxynaphth-2-yl)propionate *rac*-**19** (0.64 g, 1.63 mmol) in THF gave a separable mixture of oxazolidinones *rac-syn*-**27** and *rac-anti*-**27** [*syn*-/*anti*- >98:2]. The crude mixture was purified by flash column chromatography on silica gel eluting with light petroleum (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidinones *anti*-**27** (60 mg, 10%) as a white solid; mp 122–124 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.51; $\nu_{max}(CHCl_3)/cm^{-1}$ 1778 (NC=O) and 1701 (OC=O); δ_H (270 MHz; $CDCl_3$) 7.70 (1H, s, CH; Ar), 7.68 (2H, dd, *J* 8.4 and 2.7, 2 × CH; Ar), 7.46 (1H, dd, *J* 8.7 and 1.6, CH; Ar), 7.14–7.09 (2H, m, 2 × CH; Ar), 5.28 (1H, q, *J* 6.9, ArC*H*CH_3), 4.36–4.31 (1H, dt, *J* 9.1 and 3.2, *i*-PrCHN), 4.10 (1H, dd, *J* 9.1 and 3.2, CH_AH_BO), 4.05 (1H, t, *J* 9.1, CH_AH_BO), 3.88 (3H, s, CH_3O), 2.50–2.39 (1H, m, C*H*$(CH_3)_2$), 1.57 (3H, d, *J* 7.2, ArCHCH_3), 0.86 (3H, d, *J* 7.2, $CH_3^ACHCH_3^B$) and 0.85 (3H, d, *J* 7.2, $CH_3^ACHCH_3^B$); δ_C (100 MHz; $CDCl_3$) 174.7 (NC=O), 157.6 (OC=O), 153.7 (*i*-CO; Ar), 135.4, 133.8 and 128.8 (3 × *i*-CC; Ar), 129.3, 127.0, 126.8, 126.6, 118.8 and 105.5 (6 × CH; Ar), 63.0 (CH_2O), 59.0 (*i*-PrCHN), 55.2 (OCH_3), 42.8 (Ar*C*HCH$_3$), 28.5 (*C*H$(CH_3)_2$), 19.6 (CH_3^A; *i*-Pr), 17.9 (CH_3^B; *i*-Pr), and 14.6 (ArCH*C*H$_3$) (Found MH^+, 342.1707; $C_{20}H_{24}NO_4$ requires 342.1700); and *syn*-**27** (0.29 g, 48%) as a white solid; mp 92–94 °C; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.34; $\nu_{max}(CHCl_3)/cm^{-1}$ 1778 (NC=O) and 1701 (OC=O); δ_H (270 MHz; $CDCl_3$) 7.69 (3H, m, 3 × CH; Ar), 7.45 (1H, dd, *J* 8.4 and 1.6, 1 × CH; Ar), 7.13–7.09 (2H, m, 2 × CH; Ar), 5.26 (1H, q, *J* 6.9, ArC*H*CH_3), 4.52–4.46 (1H, dt, *J* 8.9 and 3.2, *i*-PrCHN), 4.21 (1H, t, *J* 8.9, CH_AH_BO), 4.06 (1H, dd, *J* 8.9 and 3.2, CH_AH_BO), 3.88 (3H, s, CH_3O), 2.25–2.13 (1H, m, C*H*$(CH_3)_2$), 1.53 (3H, d, *J* 6.9, ArCHCH_3), 0.75 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$) and 0.38 (3H, d, *J* 6.9, $CH_3^ACHCH_3^B$); δ_C (100 MHz; $CDCl_3$) 174.6 (NC=O), 157.6 (OC=O), 153.5 (*i*-CO; Ar), 135.7, 133.7 and 128.9 (3 × *i*-CC; Ar), 129.4, 127.0, 126.7, 126.6, 118.8 and 105.5 (6 × CH; Ar), 62.9 (CH_2O), 58.1 (*i*-PrCHN), 55.3 (OCH_3), 43.2 (Ar*C*HCH$_3$), 27.9 (*C*H$(CH_3)_2$), 18.7 (CH_3^A; *i*-Pr), 17.7 (CH_3^B; *i*-Pr) and 14.0 (ArCH*C*H$_3$) (Found MH^+, 342.1701; $C_{20}H_{24}NO_4$ requires 342.1700).

4.5.23. Hydrolysis of oxazolidione adducts *anti*-16 (+)-2-phenylpropionic acid (*S*)-17. Lithium hydroxide monohydrate (16 mg, 0.38 mmol) was slowly added to a stirred solution of oxazolidinone (*S*,*S*)-*anti*-**16** (0.10 g, 0.38 mmol) and hydrogen peroxide (13 mg, 0.38 mmol, 30%/w) 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 recovered oxazolidinone (*S*)-**13** (46 mg, 95%) as a white solid $[\alpha]_D^{20} = +13.7$ (*c* 3.8, $CHCl_3$), {lit.[22] $[\alpha]_D^{20} = +13.3$ (*c* 6.8, $CHCl_3$)}; and (+)-2-phenylpropionic acid (*S*)-**17** (53 mg, 93%) as an oil; R_F [light petroleum (bp 40–60 °C)/diethyl ether (1:9)] 0.5; $[\alpha]_D^{20} = +71.5$ (*c* 2.0, $CHCl_3$), {lit.[23] $[\alpha]_D^{20} = +72.0$}; $\nu_{max}(CHCl_3)/cm^{-1}$ 1706 (C=O); δ_H (270 MHz; $CDCl_3$) 7.45–6.98 (5H, m, 5 × CH; Ph), 3.75 (1H, q, *J* 7.2, PhCH) and 1.5 (3H, d, *J* 7.2, CH_3CH); δ_C (67.9 MHz; $CDCl_3$) 181.4 (C=O), 139.9 (*i*-C; Ph), 128.9, 127.8 and 127.6 (3 × CH; Ph), 45.6 (PhCH) and 18.3 (CH_3) (Found MH^+ 151.0753. $C_9H_{11}NO_2^+$ requires 151.0759); *m*/*z* 151 (30%, MH) and 105 (100, M−CH_2O_2).

4.5.24. Hydrolysis of oxazolidione adducts *syn*-27 (+)-2-(6-methoxynaphth-2-yl)propionic acid (*S*)-18. Lithium hydroxide monohydrate (29 mg, 0.70 mmol) was slowly added to a stirred solution of oxazolidinone *syn*-**27** (0.12 g, 0.35 mmol) and hydrogen peroxide (24 mg, 0.2 ml, 0.70 mmol, 30%/w) in THF/water (3:1; 4 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 recovered oxazolidinone (*R*)-**13** (41 mg, 90%) as a white solid; R_F [diethyl ether] 0.35; $[\alpha]_D^{20} = -14.0$ (*c* 2.4, $CHCl_3$), {lit.[24] $[\alpha]_D^{20} = -15.5$ (*c* 7.0, $CHCl_3$)}; and (+)-2-(6-methoxynaphth-2-yl)propionic acid (*S*)-**18** (60 mg, 74%) as a white solid; R_F [diethyl ether] 0.56; mp 151–153 °C; $[\alpha]_D^{20} = +65.0$ (*c* 1.0, $CHCl_3$), {lit.[25] $[\alpha]_D^{21} = +65.3$ (*c* 1.0, $CHCl_3$); authentic sample from Aldrich Chemical Company; $[\alpha]_D^{20} = +64.8$ (*c* 3.4, $CHCl_3$); mp 152–154 °C}; $\nu_{max}(CHCl_3)/cm^{-1}$ 1710 (C=O); δ_H (400 MHz; $CDCl_3$) 7.73–7.68 (3H, m, 3 × CH; Ph), 7.41 (1H, dd, *J* 8.4 and 1.8, CH; Ar), 7.16–7.10 (2H, m, 2 × CH; Ph), 3.91 (3H, s, CH_3O), 3.88 (1H, q, *J* 7.0, ArC*H*CH_3) and 1.59 (3H, d, *J* 7.0, ArCHC*H*$_3$); δ_C (100 MHz; $CDCl_3$) 180.2 (C=O), 157.9 (*i*-CO; Ar), 134.8, 133.8 and 128.9 (3 × *i*-C; Ar), 129.3, 127.2, 126.2, 126.1, 119.0 and 105.6 (6 × CH; Ar), 55.3 (CH_3O), 45.2 (Ar*C*HCH$_3$) and 18.1 (ArCH*C*H$_3$); *m*/*z* 230 (60%, M^+) and 185 (100, $ArCHCH_3^+$).

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11. The yields were based on the amount of each active ester used.
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