



Efficient parallel resolution of pentafluorophenyl active esters using quasi-enantiomeric combinations of oxazolidin-2-ones

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ABSTRACT

The parallel resolution of racemic pentafluorophenyl 2-aryl/phenylpropanoates and butanoates using an equimolar combination of quasi-enantiomeric Evans oxazolidin-2-ones is discussed. The levels of diastereoselectivity were excellent (>90% de) leading to separable quasi-enantiomeric oxazolidin-2-ones in good yield. This methodology was used to resolve a series of structurally related 2-aryl/phenylpropanoic and butanoic acids.

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1. Introduction

Since 1997, there has been a steady increase in the number of reports on the use of parallel kinetic resolutions as a synthetic strategy for the separation of enantiomers.^{1–3} Within this area, Davies has demonstrated⁴ the parallel resolution of racemic enone (*rac*)-**3** using a quasi-enantiomeric combination of lithium amides (*S*)-**1** and (*R*)-**2** to give two complementary diastereoisomerically pure β -amino esters *syn,syn,anti*-**4** and *syn,syn,anti*-**5** in 39% and 35% yields with 95–97% and 97–99% diastereoisomeric excesses (Scheme 1).⁵ The levels of mutual recognition between (*S*)-**1** and (*S*)-**3**, and (*R*)-**2** and (*R*)-**3** were excellent (>20:1) leading to separable β -amino esters **4** and **5** in good yields with >95% de (Scheme 1).⁵

Since 2005, we have been interested in the philosophy of this approach for the (mutual) resolution of α -substituted carboxylic acids (such as pentafluorophenyl active esters) and masked α -amino acids (such as oxazolidin-2-ones).^{6–8} We have reported the efficient parallel kinetic resolution of racemic oxazolidin-2-ones, such as 4-phenyl-oxazolidinone (*rac*)-**8**, using a pair of quasi-enantiomeric active esters (*S*)-**6** and (*R*)-**7** to give two separable oxazolidin-2-ones (*S,R*)-*syn*-**9** and (*R,S*)-*syn*-**10** in 48% and 54% yields with 90% and 94% diastereoisomeric excesses (Scheme 2).⁹ From this study, it was apparent that the (*R*)-enantiomer of oxazolidin-2-one **8** recognised the (*S*)-enantiomer of active ester (*S*)-**6**, whereas, the remaining (*S*)-enantiomer of oxazolidin-2-one **8** recognised the complementary active ester (*R*)-**7** (Scheme 2).¹⁰

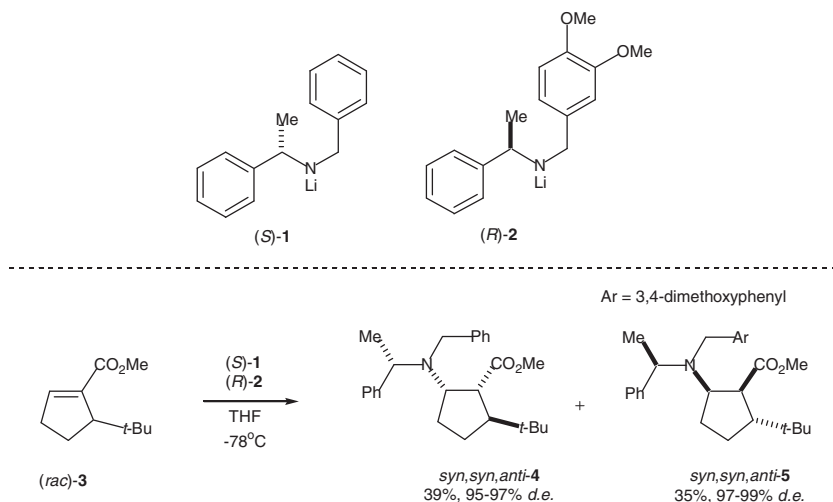
2. Results and discussion

Herein we report an extension to this methodology for the complementary resolution of racemic α -substituted carboxylic acids, such as (*rac*)-**C**, using the corresponding pentafluorophenyl active ester (*rac*)-**D**, and an equimolar combination of Evans' oxazolidin-2-ones, such as (*R*)-**A** and (*S*)-**B**, as parallel resolving components to give the oxazolidin-2-one adducts (*S,R*)-*syn*-**E** and (*R,S*)-*syn*-**F** (Scheme 3). Simple separation of these adducts, by column chromatography, followed by hydrolysis, would lead to both individual enantiomers of the original α -substituted carboxylic acids (*S*)- and (*R*)-**C**, respectively (Scheme 3).

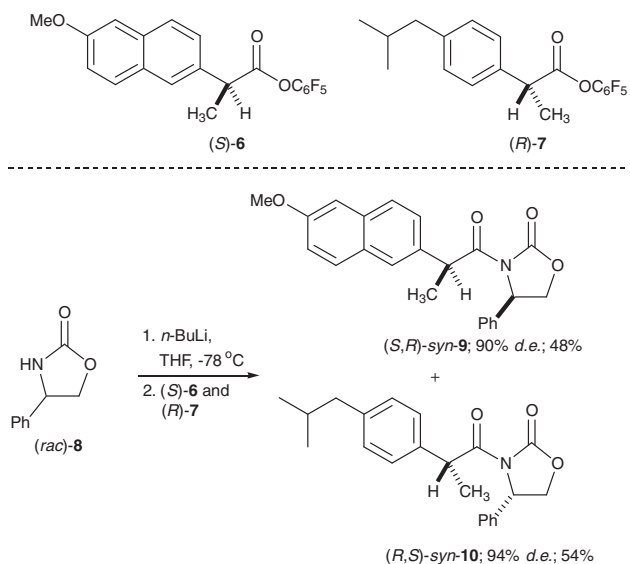
With this aim in mind, we first studied the mutual kinetic resolution of a series of structurally related racemic Evans' oxazolidinones (*rac*)-**8**, (*rac*)-(4*RS*,5*SR*)-*syn*-**11** and (*rac*)-**12–14** and pentafluorophenyl active esters (*rac*)-**6–7** and (*rac*)-**15–19** to get a measure of their stereochemical recognition (as shown in Schemes 4 and 5). Deprotonation of each racemic oxazolidin-2-one, (*rac*)-(4*RS*,5*SR*)-*syn*-**11**, (*rac*)-**12**, (*rac*)-**13**, (*rac*)-**8** and (*rac*)-**14**, in THF at -78 °C, followed by the addition of the active esters (*rac*)-**15**, (*rac*)-**16**, (*rac*)-**17**, (*rac*)-**18**, (*rac*)-**7**, (*rac*)-**19** and (*rac*)-**6** gave, after stirring for 2 h at -78 °C, the corresponding oxazolidin-2-one adducts **9**, **10** and **20–52** in moderate to good yields (25%→75%) with good to excellent levels of diastereocontrol (14% de→96% de) (Scheme 6). From these results, it was evident that the oxazolidin-2-ones (*rac*)-**8**, (*rac*)-**13** and (*rac*)-**14** gave higher levels of diastereocontrol for active esters (*rac*)-**6–7**, (*rac*)-**15** and (*rac*)-**18–19** [derived from the corresponding 2-(4-substituted-aryl)propanoic and 2-(4-substituted-aryl)butanoic acids] than the less sterically demanding oxazolidin-2-ones (*rac*)-(4*RS*,5*SR*)-*syn*-**11** and (*rac*)-**12** (Scheme 6). Oxazolidin-2-ones that contained a sterically demanding sp^3 -hybridised C(4)-substituent [e.g., *i*-Pr in (*rac*)-**13**] appeared to give higher levels of mutual recognition and

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



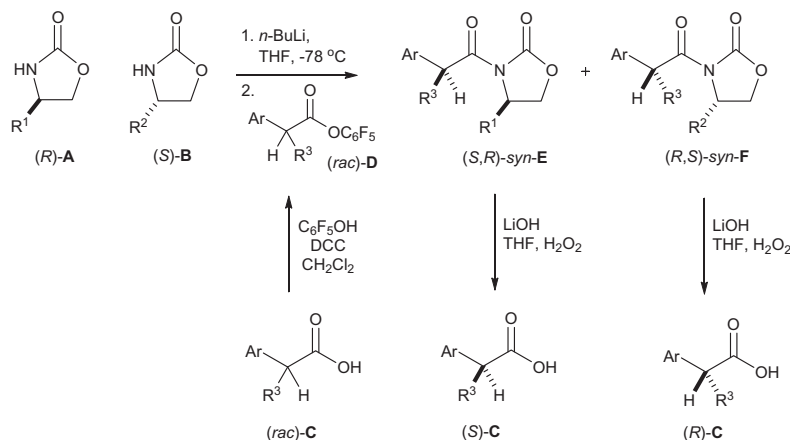
Scheme 2. Parallel kinetic resolution of oxazolidin-2-one (*rac*)-**8** using a *quasi*-enantiomeric combination of active esters (*S*)-**6** and (*R*)-**7**.

favoured formation of the corresponding *syn*-oxazolidin-2-one adducts (with 36%→92% de). The less sterically demanding C(4)-

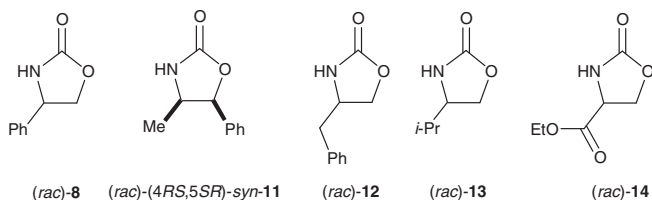
substituted oxazolidin-2-ones [e.g., Me and CH₂Ph in (*rac*)-(4*RS*,5*SR*)-**11** and (*rac*)-**12**, respectively] gave significantly lower levels of diastereocontrol (38%→58% de) (Scheme 6). However, those oxazolidin-2-ones which contained an *sp*²-hybridised C(4)-substituent [e.g., Ph and CO₂Et in (*rac*)-**8** and (*rac*)-**14**, respectively],¹¹ gave higher levels of diastereoselection (88%–96% de).

For a series of pentafluorophenyl 4-substituted aryl propanoates (*rac*)-**6–7** and (*rac*)-**18–19**, these gave similar levels of diastereoselection to their parent ester (*rac*)-**15** with the exception of pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19**, which was marginally less diastereoselective (Scheme 6). Increasing the sterically demanding nature of the C(2)-substituent of the active ester, from a methyl group [in (*rac*)-**15**] to an ethyl group [in (*rac*)-**16**], generally increased the levels of diastereocontrol (Scheme 6). However, there appears to be a steric threshold at this C(2)-position, as increasing the size of the ethyl group [in (*rac*)-**16**] to a larger isopropyl group [in (*rac*)-**17**] significantly lowered the levels of diastereocontrol (Scheme 6). Herein it was evident that the oxazolidin-2-one, 4-phenyloxazolidin-2-one (*rac*)-**8** gave the highest levels of mutual recognition for a wide range of structurally related pentafluorophenyl 2-(aryl/or phenyl) propanoates and butanoates. The relative levels of diastereoselection for these oxazolidin-2-ones were found to be: (*rac*)-**8** > (*rac*)-**14** > (*rac*)-**13** ≫ (*rac*)-**12** > (*rac*)-(4*RS*,5*SR*)-**11**.

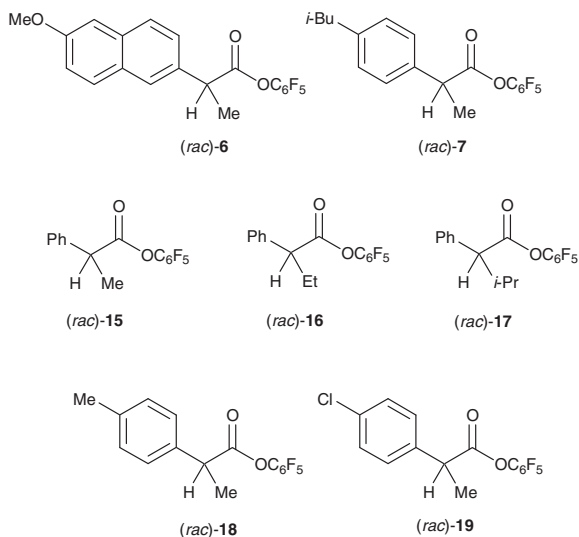
With this information in hand, we next studied the parallel kinetic resolution of this series of active esters (*rac*)-**15**, (*rac*)-**16**,



Scheme 3. Proposed parallel kinetic resolution of carboxylic acid (*rac*)-**C** using a *quasi*-enantiomeric combination of oxazolidin-2-ones (*R*)-**A** and (*S*)-**B**.



Scheme 4. Oxazolidin-2-ones (*rac*)-**8**, (*rac*)-(4*RS*,5*SR*)-*syn*-**11** and (*rac*)-**12–14**.



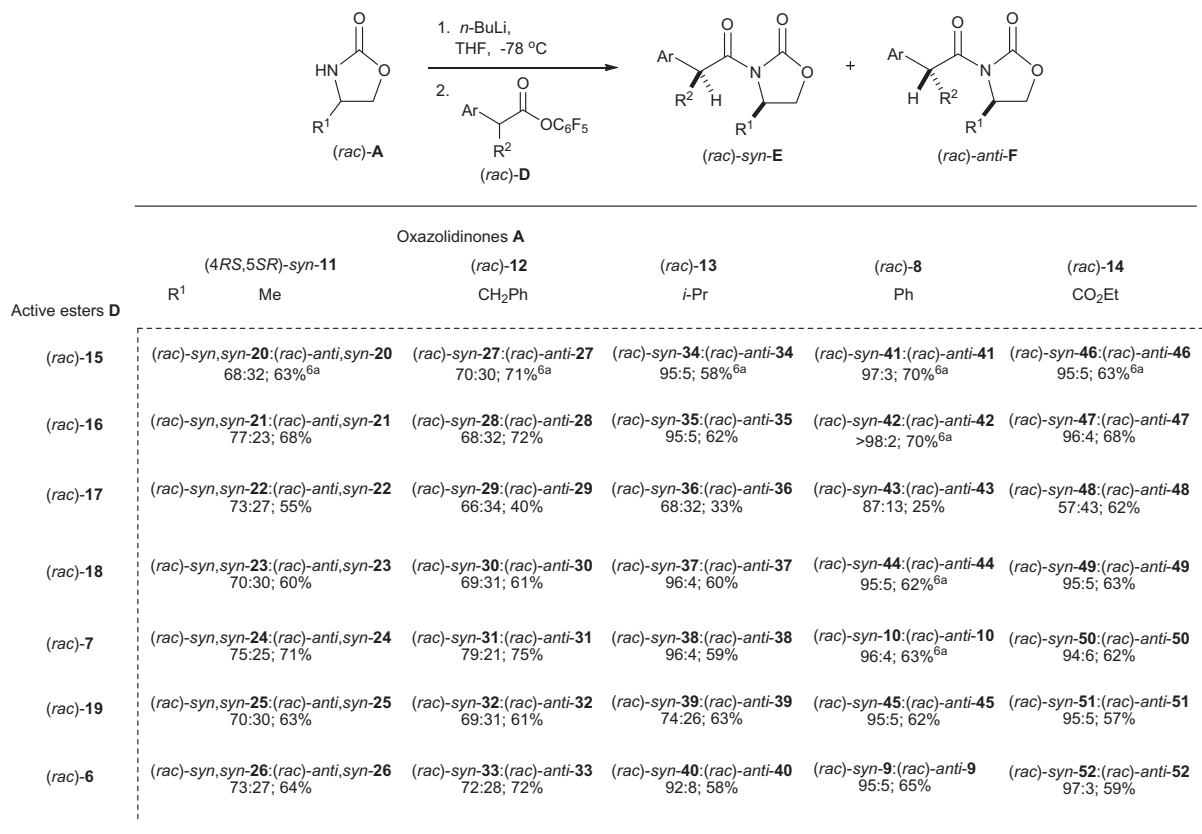
Scheme 5. Active esters (*rac*)-**6**, (*rac*)-**7** and (*rac*)-**15–19**.

(*rac*)-**17**, (*rac*)-**18**, (*rac*)-**7**, (*rac*)-**19** and (*rac*)-**6** using three combinations of the more diastereoselective oxazolidin-2-ones, (*S*)-**13** and (*R*)-**8** (in Scheme 7), (*S*)-**13** and (*S*)-**14** (in Scheme 8), and (*S*)-**8** and (*S*)-**14** (in Scheme 9). Treatment of each pair of oxazolidin-2-ones, (*S*)-**13** and (*R*)-**8** (in Scheme 7), (*S*)-**13** and (*S*)-**14** (in Scheme 8), and (*S*)-**8** and (*S*)-**14**, with *n*-BuLi in THF at $-78\text{ }^{\circ}\text{C}$, followed by the addition of the active esters (*rac*)-**15–18**, (*rac*)-**7**, (*rac*)-**19** and (*rac*)-**6**, gave the corresponding oxazolidin-2-one adducts in moderate to good yields and with good to excellent levels of diastereocontrol (Schemes 7–9).

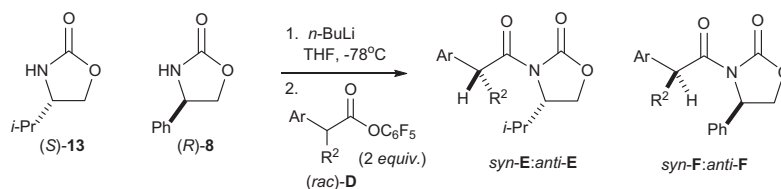
The levels of diastereoselection were found to be comparable to those obtained from their corresponding mutual kinetic resolutions (as shown in Scheme 6). The levels of diastereoselection were found to be the highest for the pair of *quasi*-enantiomeric oxazolidin-2-ones, (*S*)-**8** and (*S*)-**14**, which contained sp^2 -hybridised C(4)-substituents (in Scheme 9). The oxazolidin-2-ones, (*S*)-**13** and (*R*)-**8** (in Scheme 7), were marginally more diastereoselective than the remaining pair of oxazolidin-2-ones, (*S*)-**13** and (*S*)-**14** (in Scheme 8).

These oxazolidin-2-one adducts were separated efficiently by flash chromatography; the *anti*-adducts were found to have higher retention factors (R_F) than their corresponding *syn*-adducts. There was also an increased separability for adducts derived from the oxazolidin-2-ones (*R*)-**8** and (*S*)-**13** that contained hydrophobic (Ph and *i*-Pr) groups and adducts derived from the more polar oxazolidin-2-one (*S*)-**14** that contained a hydrophilic ethoxycarbonyl group [ΔR_F [light petroleum (bp $40\text{--}60\text{ }^{\circ}\text{C}$)/diethyl ether] ~ 0.1].

Access to enantiomerically pure 2-phenylpropanoic acids (*S*)- and (*R*)-**53** was achieved by LiOH/H₂O₂ mediated hydrolysis of oxazolidin-2-ones (*S,R*)-*syn*-**41**, (*R,S*)-*syn*-**34** and (*S,S*)-*syn*-**46** (Scheme 10). Treatment of adducts (*S,R*)-*syn*-**41**, (*R,S*)-*syn*-**34** and

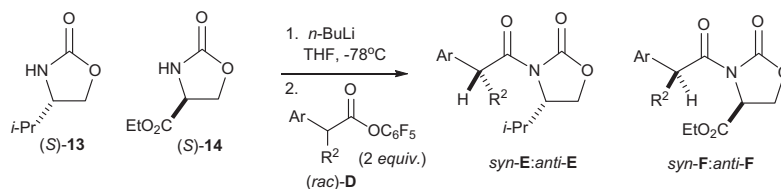


Scheme 6. Mutual kinetic resolution of active esters (*rac*)-**6**, (*rac*)-**7** and (*rac*)-**15–19** using oxazolidin-2-ones (*rac*)-**8**, (*rac*)-(4*RS*,5*SR*)-*syn*-**11**, (*rac*)-**12**, (*rac*)-**13** and (*rac*)-**14**.



Entry	Active esters (<i>rac</i>)-D	Product Oxazolidin-2-ones
1	(<i>rac</i>)-15	(<i>R,S</i>)- <i>syn</i> -34:(<i>S,S</i>)- <i>anti</i> -34 95:5; 60% ^{6a} (<i>S,R</i>)- <i>syn</i> -41:(<i>R,R</i>)- <i>anti</i> -41 95:5; 60% ^{6a}
2	(<i>rac</i>)-16	(<i>R,S</i>)- <i>syn</i> -35:(<i>S,S</i>)- <i>anti</i> -35 95:5; 67% (<i>S,R</i>)- <i>syn</i> -42:(<i>R,R</i>)- <i>anti</i> -42 95:5; 64%
3	(<i>rac</i>)-17	(<i>R,S</i>)- <i>syn</i> -36:(<i>S,S</i>)- <i>anti</i> -36 79:21; 58% (<i>S,R</i>)- <i>syn</i> -43:(<i>R,R</i>)- <i>anti</i> -43 84:16; 60%
4	(<i>rac</i>)-18	(<i>R,S</i>)- <i>syn</i> -37:(<i>S,S</i>)- <i>anti</i> -37 98:2; 61% (<i>S,R</i>)- <i>syn</i> -44:(<i>R,R</i>)- <i>anti</i> -44 98:2; 63%
5	(<i>rac</i>)-7	(<i>R,S</i>)- <i>syn</i> -38:(<i>S,S</i>)- <i>anti</i> -38 95:5; 68% (<i>S,R</i>)- <i>syn</i> -10:(<i>R,R</i>)- <i>anti</i> -10 95:5; 62%
6	(<i>rac</i>)-19	(<i>R,S</i>)- <i>syn</i> -39:(<i>S,S</i>)- <i>anti</i> -39 98:2; 64% (<i>S,R</i>)- <i>syn</i> -45:(<i>R,R</i>)- <i>anti</i> -45 98:2; 61%
7	(<i>rac</i>)-6	(<i>R,S</i>)- <i>syn</i> -40:(<i>S,S</i>)- <i>anti</i> -40 96:4; 65% (<i>S,R</i>)- <i>syn</i> -9:(<i>R,R</i>)- <i>anti</i> -9 96:4; 40%

Scheme 7. Parallel kinetic resolution of active esters (*rac*)-6, (*rac*)-7 and (*rac*)-15–19 using oxazolidin-2-ones (*S*)-13 and (*R*)-8.

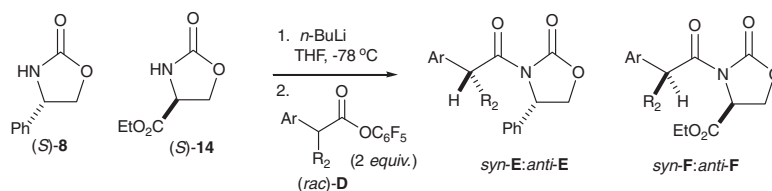


Entry	Active esters (<i>rac</i>)-D	Product Oxazolidin-2-ones
1	(<i>rac</i>)-15	(<i>R,S</i>)- <i>syn</i> -34:(<i>S,S</i>)- <i>anti</i> -34 95:5; 60% ^{6a} (<i>S,S</i>)- <i>syn</i> -46:(<i>R,S</i>)- <i>anti</i> -46 95:5; 49% ^{6a}
2	(<i>rac</i>)-16	(<i>R,S</i>)- <i>syn</i> -35:(<i>S,S</i>)- <i>anti</i> -35 96:4; 62% (<i>S,S</i>)- <i>syn</i> -47:(<i>R,S</i>)- <i>anti</i> -47 95:5; 65%
3	(<i>rac</i>)-17	(<i>R,S</i>)- <i>syn</i> -36:(<i>S,S</i>)- <i>anti</i> -36 68:32; 61% (<i>S,S</i>)- <i>syn</i> -48:(<i>R,S</i>)- <i>anti</i> -48 89:11; 64%
4	(<i>rac</i>)-18	(<i>R,S</i>)- <i>syn</i> -37:(<i>S,S</i>)- <i>anti</i> -37 95:5; 61% (<i>S,S</i>)- <i>syn</i> -49:(<i>R,S</i>)- <i>anti</i> -49 95:5; 68%
5	(<i>rac</i>)-7	(<i>R,S</i>)- <i>syn</i> -38:(<i>S,S</i>)- <i>anti</i> -38 95:5; 65% (<i>S,S</i>)- <i>syn</i> -50:(<i>R,S</i>)- <i>anti</i> -50 95:5; 63%
6	(<i>rac</i>)-19	(<i>R,S</i>)- <i>syn</i> -39:(<i>S,S</i>)- <i>anti</i> -39 90:10; 62% (<i>S,S</i>)- <i>syn</i> -51:(<i>R,S</i>)- <i>anti</i> -51 95:5; 65%
7	(<i>rac</i>)-6	(<i>R,S</i>)- <i>syn</i> -40:(<i>S,S</i>)- <i>anti</i> -40 93:7; 61% (<i>S,S</i>)- <i>syn</i> -52:(<i>R,S</i>)- <i>anti</i> -52 96:4; 48%

Scheme 8. Parallel kinetic resolution of active esters (*rac*)-6, (*rac*)-7 and (*rac*)-15–19 using oxazolidin-2-ones (*S*)-13 and (*S*)-14.

(*S,S*)-*syn*-46 with LiOH/H₂O₂ in THF/H₂O (3:1), and stirring the resulting solution for 12 h, gave the enantiomerically pure 2-phenylpropanoic acids (*S*)-53, (*R*)-53 and (*S*)-53 in 90%, 85% and 87% yields, respectively (Scheme 10). In addition, the hydrolysis

of the remaining oxazolidin-2-ones (*S,R*)-*syn*-42–43, (*S,R*)-*syn*-44–45 and (*S,R*)-*syn*-9–10 under our standard conditions gave access to the corresponding enantiomerically pure 2-phenylbutanoic acids (*S*)-54 (in 91% yield) and (*S*)-55 (in 83% yield), and the



Entry	Active esters (<i>rac</i>)-D	Product Oxazolidin-2-ones
1	(<i>rac</i>)-15	(<i>R,S</i>)- <i>syn</i> -41:(<i>S,S</i>)- <i>anti</i> -41 95:5; 70% (<i>S,S</i>)- <i>syn</i> -46:(<i>R,S</i>)- <i>anti</i> -46 98:2; 71%
2	(<i>rac</i>)-16	(<i>R,S</i>)- <i>syn</i> -42:(<i>S,S</i>)- <i>anti</i> -42 95:5; 65% (<i>S,S</i>)- <i>syn</i> -47:(<i>R,S</i>)- <i>anti</i> -47 95:5; 69%
3	(<i>rac</i>)-17	(<i>R,S</i>)- <i>syn</i> -43:(<i>S,S</i>)- <i>anti</i> -43 86:14; 64% (<i>S,S</i>)- <i>syn</i> -48:(<i>R,S</i>)- <i>anti</i> -48 92:8; 62%
4	(<i>rac</i>)-18	(<i>R,S</i>)- <i>syn</i> -44:(<i>S,S</i>)- <i>anti</i> -44 98:2; 60% (<i>S,S</i>)- <i>syn</i> -49:(<i>R,S</i>)- <i>anti</i> -49 95:5; 61%
5	(<i>rac</i>)-7	(<i>R,S</i>)- <i>syn</i> -10:(<i>S,S</i>)- <i>anti</i> -10 95:5; 69% (<i>S,S</i>)- <i>syn</i> -50:(<i>R,S</i>)- <i>anti</i> -50 95:5; 63%
6	(<i>rac</i>)-19	(<i>R,S</i>)- <i>syn</i> -45:(<i>S,S</i>)- <i>anti</i> -45 95:5; 61% (<i>S,S</i>)- <i>syn</i> -51:(<i>R,S</i>)- <i>anti</i> -51 95:5; 58%
7	(<i>rac</i>)-6	(<i>R,S</i>)- <i>syn</i> -9:(<i>S,S</i>)- <i>anti</i> -9 95:5; 74% (<i>S,S</i>)- <i>syn</i> -52:(<i>R,S</i>)- <i>anti</i> -52 98:2; 50%

Scheme 9. Parallel kinetic resolution of active esters (*rac*)-6, (*rac*)-7 and (*rac*)-15–19 using oxazolidin-2-ones (*S*)-8 and (*S*)-14.

4-substituted-aryl propanoic acids (*S*)-56 (in 89% yield), (*S*)-57 (in 92% yield), (*S*)-58 (in 85% yield) and (*S*)-59 (in 90% yield) in good yield and with high levels of enantiomeric purity (Scheme 10).¹²

3. Conclusion

In conclusion, we have reported an efficient parallel kinetic resolution of a series of structurally related active esters, such as pentafluorophenyl 2-phenylpropanoate (*rac*)-15 using a combination of *quasi*-enantiomeric Evans' oxazolidinones. This methodology appears to be efficient for a variety of structurally related oxazolidinones [e.g., (*S*)-8 and (*S*)-14] and gives the separable diastereoisomerically pure *syn*-oxazolidin-2-one adducts 41 and 46 in good yield. Our reaction type is complementary to Evans' original *anti*-alkylation methodology¹³ (for prostereogenic phenylacetyl oxazolidin-2-ones) as this method favours the formation of related oxazolidin-2-one *anti*-adducts with near perfect diastereocontrol.

4. Experimental

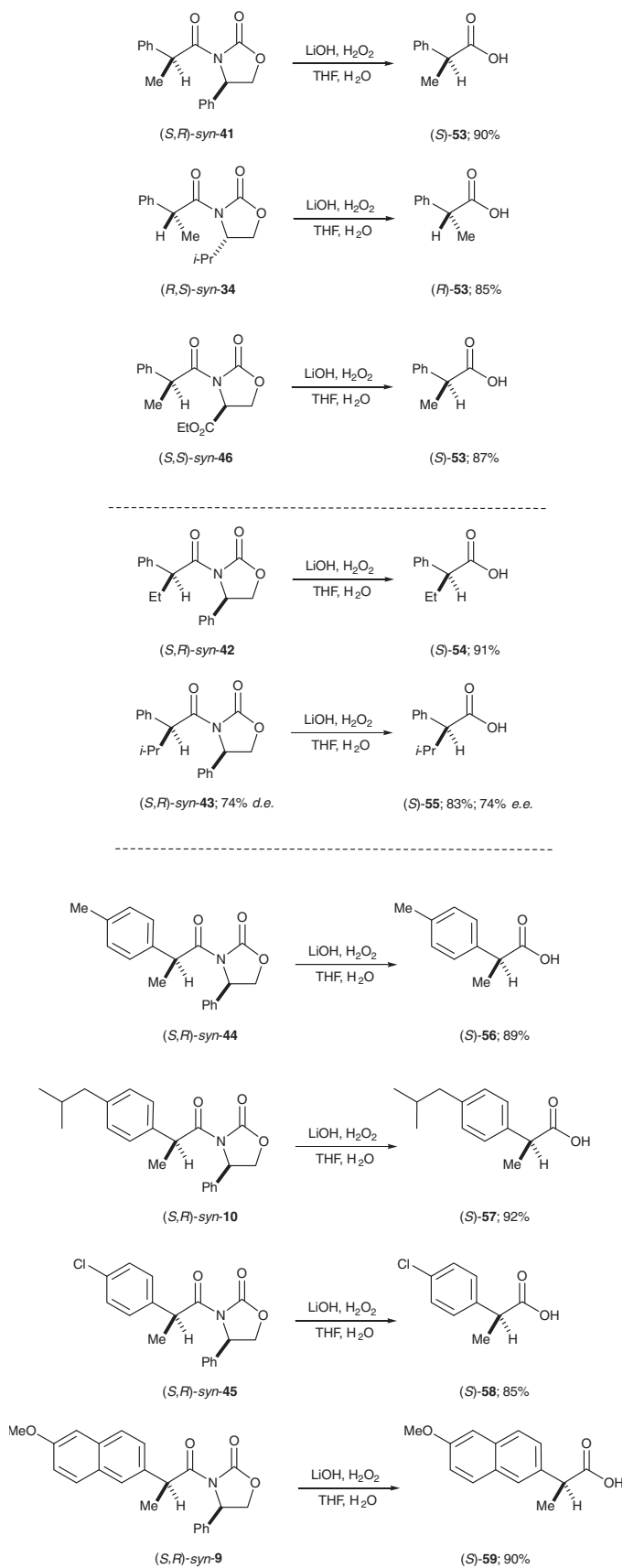
4.1. General

All solvents were distilled before use. All the 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 60F₂₅₄ 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.

4.2. Pentafluorophenyl 2-(6-methoxynaphthalen-2-yl)propanoate (*rac*)-6

2-(6-Methoxy-naphthalen-2-yl)-propanoic acid (*rac*)-59 (5.0 g, 21.7 mmol) was added to a stirred solution of *N,N'*-dicyclohexylcarbodiimide (DCC) (4.90 g, 23.9 mmol) in dichloromethane (20 mL). The solution was stirred for 2 min. Pentafluorophenol (4.00 g, 21.7 mmol) in dichloromethane (50 mL) was added and the resulting solution was stirred for 12 h. The resulting precipitate (*N,N'*-dicyclohexylurea) was filtered off (using suction filtration). Water (50 mL) was added and the solution was extracted into dichloromethane (3 × 100 mL). The combined organic layers were dried (over MgSO₄) 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-(6-methoxy-naphthalen-2-yl)-propanoate (*rac*)-6 (8.61 g, 70%) as a white solid; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (9:1)] 0.65; mp 51–53 °C; ν_{\max} (CHCl₃) cm⁻¹ 1781 (C=O); δ_{H} (400 MHz; CDCl₃) 7.76–7.77 (1H, d, *J* 8.6, CH; Ar), 7.75 (1H, br s, CH; Ar), 7.74 (1H, d, *J* 8.6, CH; Ar), 7.45 (1H, dd, *J* 8.6 and 1.8, CH; Ar), 7.18 (1H, dd, *J* 8.6 and 2.5, CH, Ar), 7.14 (1H, br t, *J* 2.5, CH; Ar), 4.38 (1H, q, *J* 7.2, ArCHCH₃), 3.91 (3H, s, OCH₃) and 1.71 (3H, d, *J* 7.2, ArCHCH₃); δ_{C} (100 MHz; CDCl₃) 170.7 (C=O), 157.9 (*i*-CO; Ar), 141.0 (142.32 and 139.8, 2C, ddt, ¹*J*_{C,F} = 249.8, ²*J*_{C,F} = 12.2 and ³*J*_{C,F} = 4.6, C(2)-F), 139.3 (140.63 and 138.11, 1C, dtt, ¹*J*_{C,F} = 252.1, ²*J*_{C,F} = 13.0 and ³*J*_{C,F} = 4.5, C(4)-F), 137.8 (139.04 and 136.54, 2C, dtdd, ¹*J*_{C,F} = 250.6, ²*J*_{C,F} = 13.8, ³*J*_{C,F} = 5.3 and ⁴*J*_{C,F} = 3.0, 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; OC₆F₅), 55.3 (OCH₃), 45.9 (ArCHCH₃) and 18.5 (ArCHCH₃); δ_{F} (378 MHz; CDCl₃),



Scheme 10. Hydrolysis of oxazolidin-2-ones (*S,R*)-syn-41, (*R,S*)-syn-34, (*S,S*)-syn-46, (*S,R*)-syn-42, (*S,R*)-syn-43, (*S,R*)-syn-44, (*S,R*)-syn-10, (*S,R*)-syn-45 and (*S,R*)-syn-9.

–152.5 (2F, d, $^3J_{\text{F,F}} = 17.0$, F_{ortho}), –157.9 (1F, t, $^3J_{\text{F,F}} = 21.6$, F_{para}) and –162.3 (2F, dd, $^3J_{\text{F,F}} = 21.6$ and 17.0, F_{meta}) (Found M^+ , 396.0783; $\text{C}_{20}\text{H}_{13}\text{F}_5\text{O}_3^+$ requires 396.0779).

4.3. Pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-7

This has been reported elsewhere.¹⁴

4.4. Pentafluorophenyl 2-phenylpropanoate (*rac*)-15

This has been reported elsewhere.¹⁴

4.5. Pentafluorophenyl 2-phenylbutanoate (*rac*)-16

This has been reported elsewhere.¹⁴

4.6. Pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-17

At first, DCC (1.65 g, 7.28 mmol) was slowly added to a solution of pentafluorophenol (1.34 g, 7.28 mmol) in dichloromethane (10 mL). The resulting solution was stirred for 5 min. 2-Phenyl-3-methyl butanoic acid (*rac*)-55 (1.3 g, 7.28 mmol) in dichloromethane (10 mL) was slowly added (in four portions) to this solution (over 2 h). The solution was stirred for 12 h and the resulting precipitate (*N,N'*-dicyclohexylurea) was filtered off (using suction filtration). Water (30 mL) was added and the solution was extracted into dichloromethane (3 × 100 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*)-17 (2.05 g, 82%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (9:1)] 0.77; mp 45–48 °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, PhCHi-Pr), 2.51–2.40 (1H, m, $\text{CH}(\text{CH}_3)_2$), 1.15 (3H, d, J 6.6, $\text{CH}_3^{\text{A}}\text{CHCH}_3^{\text{B}}$) and 0.79 (3H, d, J 6.6, $\text{CH}_3^{\text{A}}\text{CHCH}_3^{\text{B}}$); δ_{C} (100 MHz; CDCl_3) 169.9 (OC=O), 141.1 (142.37 and 139.88, 2C, ddt, $^1J_{\text{C,F}} = 251.4$, $^2J_{\text{C,F}} = 12.3$ and $^3J_{\text{C,F}} = 3.8$, C(2)-F), 139.4 (140.65 and 138.14, 1C, dtd, $^1J_{\text{C,F}} = 252.9$, $^2J_{\text{C,F}} = 13.8$ and $^3J_{\text{C,F}} = 4.6$, C(4)-F), 137.8 (139.05 and 136.58, 2C, dtdd, $^1J_{\text{C,F}} = 249.1$, $^2J_{\text{C,F}} = 13.1$, $^3J_{\text{C,F}} = 5.4$ and $^4J_{\text{C,F}} = 3.1$, C(3)-F), 136.4 (*i*-C; Ph), 128.8², 128.5² and 127.9¹ (5 × CH; Ph), 125.1 (1C, tdt, $^2J_{\text{C,F}} = 14.6$, $^4J_{\text{C,F}} = 4.6$ and $^3J_{\text{C,F}} = 2.3$, *i*-CO; OC_6F_5), 59.2 (PhCHi-Pr), 31.8 ($\text{CH}(\text{CH}_3)_2$), 21.2 ($\text{CH}_3^{\text{A}}\text{CHCH}_3^{\text{B}}$) and 20.0 ($\text{CH}_3^{\text{A}}\text{CHCH}_3^{\text{B}}$); δ_{F} (378 MHz; CDCl_3) –152.3 (2F, dt, $^3J_{\text{F,F}} = 17.3$ and $^4J_{\text{F,F}} = 4.8$, F_{ortho}), –158.0 (1F, t, $^3J_{\text{F,F}} = 21.9$, F_{para}) and –162.4 (2F, td, $^3J_{\text{F,F}} = 21.9$ and $^4J_{\text{F,F}} = 4.8$, F_{meta}) (Found M^+ 344.0829; $\text{C}_{17}\text{H}_{13}\text{F}_5\text{O}_2^+$ requires M^+ 344.0830); m/z 344 (10%, M^+), 133 [65, (PhCHC₃H₇)⁺] and 91 [100, (PhCH₂)⁺].

4.7. Pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-18

In the same way as active ester (*rac*)-6, 2-(4-methylphenyl)propanoic acid (*rac*)-56 (3.0 g, 18.2 mmol), DCC (4.14 g, 19.2 mmol) and pentafluorophenol (3.36 g, 18.2 mmol) gave, pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-18 (5.10 g, 85%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (9:1)] 0.65; ν_{max} (film) cm^{-1} 1785 (C=O); δ_{H} (400 MHz; CDCl_3) 7.24 (2H, dt, J 8.2 and 2.1, 2 × CH; Ar), 7.18 (2H, dt, J 8.2 and 2.1, 2 × CH; Ar), 4.03 (1H, q, J 7.2, ArCHCH₃), 2.34 (3H, s, CH₃; Ar) and 1.62 (3H, d, J 7.2, ArCHCH₃); δ_{C} (100 MHz; CDCl_3) 170.6 (OC=O), 141.1 (142.51 and 139.89, 2C, dtd, $^1J_{\text{C,F}} = 251.6$, $^2J_{\text{C,F}} = 11.9$ and $^3J_{\text{C,F}} = 4.6$, C(2)-F), 139.4 (140.63 and 138.12, 1C, dtd, $^1J_{\text{C,F}} = 252.8$, $^2J_{\text{C,F}} = 13.4$ and $^3J_{\text{C,F}} = 3.8$),

C(4)-F), 137.8 (139.07 and 136.56, 2C, dtdd, $^1J_{C,F} = 252.8$, $^2J_{C,F} = 12.1$, $^3J_{C,F} = 5.3$ and $^4J_{C,F} = 3.1$, C(3)-F), 137.4 and 135.8 (2 × *i*-C; Ar), 129.5 and 127.2 (2 × CH; Ar), 125.2 (1C, tdt, $^2J_{C,F} = 14.3$, $^4J_{C,F} = 4.6$ and $^3J_{C,F} = 2.3$, *i*-CO; OC₆F₅), 44.6 (ArCHCH₃), 20.8 (CH₃; Ar) and 18.4 (ArCHCH₃); δ_F (378 MHz; CDCl₃) -152.5 (2F, d, $^3J_{F,F} = 18.5$, *F*_{ortho}), -158.0 (1F, t, $^3J_{F,F} = 20.9$, *F*_{para}) and -162.4 (2F, dd, $^3J_{F,F} = 20.9$ and 18.5, *F*_{meta}) (Found M⁺ 330.0671; C₁₆H₁₁F₅O₂⁺ requires M⁺, 330.0674).

4.8. Pentafluorophenyl-2-(4-chlorophenyl)propanoate (*rac*)-19

In the same way as active ester (*rac*)-6, 4-chlorophenylpropanoic acid (*rac*)-58 (5.00 g, 27.1 mmol), *N,N'*-dicyclohexylcarbodiimide (6.14 g, 29.8 mmol) and pentafluorophenol (4.98 g, 27.1 mmol), gave the active ester (*rac*)-19 (8.73 g, 92%) as a colourless liquid; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (9:1)] 0.62; ν_{\max} (film) cm⁻¹ 1782 (C=O); δ_H (400 MHz; CDCl₃) 7.35 (2H, dt, *J* 8.8 and 2.2, 2 × CH; Ar), 7.29 (2H, dt, *J* 8.8 and 2.2, 2 × CH; Ar), 4.04 (1H, q, *J* 7.1, ArCHCH₃) and 1.63 (3H, d, *J* 7.1, ArCHCH₃); δ_C (100 MHz; CDCl₃) 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), 129.1² and 128.8² (4 × CH; Ar), 125.0 (1C, tdt, $^2J_{C,F} = 14.5$ Hz, $^4J_{C,F} = 5.3$ Hz and $^3J_{C,F} = 3.0$ Hz, *i*-CO; OC₆F₅), 44.4 (ArCH) and 18.5 (CH₃CH); δ_F (378 MHz; CDCl₃) -152.6 (2F, d, $^3J_{F,F} = 18.5$, *F*_{ortho}), -157.6 (1F, t, $^3J_{F,F} = 20.9$, *F*_{para}) and -162.0 (2F, dd, $^3J_{F,F} = 20.9$ and 18.5, *F*_{meta}) (Found M(³⁵Cl)⁺, 350.0124; C₁₅H₈ClF₅O₂ requires M(³⁵Cl)⁺, 350.0127).

4.9. Mutual kinetic resolutions of oxazolidin-2-ones (*rac*)-8, (4*RS*,5*SR*)-11, (*rac*)-12, (*rac*)-13 and (*rac*)-14 using active esters (*rac*)-6, (*rac*)-7, (*rac*)-15, (*rac*)-16, (*rac*)-17, (*rac*)-18 and (*rac*)-19

For these compounds (oxazolidin-2-ones (*rac*)-8, (4*RS*,5*SR*)-11, (*rac*)-12, (*rac*)-13 and (*rac*)-14 using active esters (*rac*)-6, (*rac*)-7, (*rac*)-15, (*rac*)-16, (*rac*)-17, (*rac*)-18 and (*rac*)-19) the mutual kinetic resolutions are given below.

4.10. Synthesis of 4-methyl-5-phenyl-3-(2-phenylpropanoyl)-oxazolidin-2-one (*rac*)-anti,syn-20 and 4-methyl-5-phenyl-3-(2-phenylpropanoyl)oxazolidin-2-one (*rac*)-syn,syn-20

At first, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol) was added to a stirred solution of 4-methyl-5-phenyl-oxazolidin-2-one (*rac*)-(4*RS*,5*SR*)-11 (0.24 g, 1.36 mmol) in THF at -78 °C. After stirring for 1 h, a solution of pentafluorophenyl 2-phenylpropanoate (*rac*)-15 (0.47 g, 1.50 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 MgSO₄) and evaporated under reduced pressure to give a mixture of diastereoisomeric oxazolidin-2-ones **20** [ratio 68:32 *syn,syn*:-*anti,syn*]. 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 oxazolidin-2-one (*rac*)-anti,syn-20 (88 mg, 22%) as a colourless viscous oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.76; ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl₃) 7.43–7.38 (3H, m, 3 × CH; Ph), 7.37–7.30 (4H, m, 4 × CH; 2 × Ph), 7.28–7.22 (3H, m, 3 × CH; Ph), 5.49 (1H, d, *J* 7.1, OCHPh), 5.14 (1H, q, *J* 7.1, PhCHCH₃), 4.68 (1H, m, CH₃CHN), 1.51 (3H, d, *J* 7.1, PhCHCH₃) and 0.94 (3H, d, *J* 6.6, CH₃CHN); δ_C (100 MHz; CDCl₃) 174.1 (NC=O), 152.4 (OC=O), 140.3 (*i*-C; Ph; PhCHCH₃), 133.0 (*i*-C;

Ph; PhCHO), 129.4,⁵ 127.9,² 127.0¹ and 125.4² (10 × CH; 2 × Ph), 78.4 (OCHPh), 55.1 (CH₃CHN), 43.1 (PhCHCH₃), 19.0 (CH₃CHN) and 14.3 (PhCHCH₃) (Found MH⁺ 310.1430. C₁₉H₂₀NO₃⁺ requires MH⁺, 310.1443); and the oxazolidin-2-one (*rac*)-syn,syn-20 (184 mg, 43%) as white crystalline solid; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 92–95 °C; ν_{\max} (CHCl₃) cm⁻¹ 1774 (OC=O) and 1701 (NC=O); δ_H (400 MHz; CDCl₃) 7.42–7.37 (2H, m, 2 × CH; Ph), 7.36–7.30 (5H, m, 5 × CH; 2 × Ph), 7.28–7.24 (1H, m, CH; Ph), 7.21–7.16 (2H, m, 2 × CH; Ph), 5.64 (1H, d, *J* 7.2, OCHPh), 5.08 (1H, q, *J* 7.1, PhCHCH₃), 4.82 (1H, m, CH₃CHN), 1.51 (3H, d, *J* 7.1, PhCHCH₃) and 0.74 (3H, d, *J* 6.6, CH₃CHN); δ_C (100 MHz; CDCl₃) 174.0 (NC=O), 152.3 (OC=O), 140.1 (*i*-C; Ph; PhCHCH₃), 133.3 (*i*-C; Ph; PhCHO), 128.5,¹ 128.4,⁴ 127.9,² 126.9¹ and 125.5² (10 × CH; 2 × Ph), 78.8 (OCHPh), 54.4 (CH₃CHN), 43.3 (PhCHCH₃), 19.3 (CH₃CHN) and 14.0 (PhCHCH₃) (Found MH⁺ 310.1460. C₁₉H₂₀NO₃⁺ requires 310.1443).

4.11. Synthesis of 4-benzyl-3-(2-phenylpropanoyl)oxazolidin-2-one (*rac*)-anti-27 and 4-benzyl-3-(2-phenylpropanoyl)oxazolidin-2-one (*rac*)-syn-27

In the same way as the oxazolidin-2-one (*rac*)-20, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-benzyl-oxazolidin-2-one (*rac*)-12 (0.24 g, 1.36 mmol) and pentafluorophenyl 2-phenylpropanoate (*rac*)-15 (0.47 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-27 (ratio 70:30 *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 oxazolidin-2-one (*rac*)-anti-27 (89 mg, 21%) as a white crystalline solid; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.66; mp 64–67 °C; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1699 (NC=O); δ_H (400 MHz; CDCl₃) 7.39–7.21 (10H, m, 10 × CH; 2 × Ph), 5.12 (1H, q, *J* 7.0, PhCHCH₃), 4.61–4.55 (1H, m, BnCHN), 4.10 (1H, dd, *J* 9.2 and 2.4, CH_AH_BO), 4.01 (1H, t, *J* 9.2, CH_AH_BO), 3.35 (1H, dd, *J* 13.1 and 3.2, CH_AH_BPh), 2.80 (1H, dd, *J* 13.1 and 9.8, CH_AH_BPh) and 1.55 (3H, d, *J* 7.0, PhCHCH₃); δ_C (100 MHz; CDCl₃) 174.5 (NC=O), 152.7 (OC=O), 140.2 (*i*-C; Ph), 135.3 (*i*-C; Ph), 129.3,² 128.8,² 128.5,² 128.0,² 127.2¹ and 127.1¹ (10 × CH; 2 × Ph), 65.7 (CH₂O), 55.8 (BnCHN), 42.8 (PhCHCH₃), 37.8 (CH₂Ph) and 19.3 (PhCHCH₃) (Found MH⁺ 310.1442. C₁₉H₂₀NO₃⁺ requires 310.1443); and the oxazolidin-2-one (*rac*)-syn-27 (212 mg, 50%) as a colourless oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.43; ν_{\max} (CHCl₃) cm⁻¹ 1775 (OC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl₃) 7.49–7.45 (2H, m, 2 × CH; Ph), 7.40–7.34 (2H, m, 2 × CH; Ph), 7.31–7.28 (1H, m, CH; Ph), 7.23–7.18 (3H, m, 3 × CH; Ph), 6.98–6.94 (2H, m, 2 × CH; Ph), 5.11 (1H, q, *J* 6.9, PhCHCH₃), 4.78–4.72 (1H, m, BnCHN), 4.18 (1H, t, *J* 8.5, CH_AH_BO), 4.07 (1H, dd, *J* 8.5 and 3.2, CH_AH_BO), 3.08 (1H, dd, *J* 13.5 and 3.2, CH_AH_BPh), 2.58 (1H, dd, *J* 13.5 and 8.8, CH_AH_BPh) and 1.52 (3H, d, *J* 6.9, PhCHCH₃); δ_C (100 MHz; CDCl₃) 174.1 (NC=O), 152.7 (OC=O), 149.9 (*i*-C; Ph_A), 134.7 (*i*-C; Ph_B), 129.2,² 128.5,² 128.4,² 128.0,² 127.0¹ and 126.9¹ (10 × CH; 2 × Ph), 65.5 (CH₂O), 54.6 (BnCHN), 42.9 (PhCHCH₃), 37.0 (CH₂Ph) and 18.9 (PhCHCH₃) (Found MH⁺ 310.1438. C₁₉H₂₀NO₃⁺ requires 310.1443).

4.12. Synthesis of 4-isopropyl-3-(2-phenylpropanoyl)oxazolidin-2-one (*rac*)-anti-34 and 4-isopropyl-3-(2-phenylpropanoyl)oxazolidin-2-one (*rac*)-syn-34

In the same way as the oxazolidin-2-one (*rac*)-20, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-isopropyl-oxazolidin-2-one (*rac*)-13 (0.175 g, 1.36 mmol) and pentafluorophenyl 2-phenylpropanoate (*rac*)-15 (0.47 g, 1.50 mmol), gave the oxazolidin-2-ones

syn- and *anti*-**34** (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 oxazolidin-2-one (*rac*)-*anti*-**34** (10 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.64; ν_{\max} (film) cm^{-1} 1774 (OC=O) and 1701 (NC=O); δ_H (400 MHz; CDCl_3) 7.38–7.21 (5H, m, 5 \times CH; Ph), 5.15 (1H, q, J 7.0, PhCHCH₃), 4.39–4.33 (1H, dt, J 9.1 and 3.2, *i*-PrCHN), 4.10 (1H, dd, J 9.1 and 3.2, CH_AH_BO), 4.02 (1H, t, J 9.1, CH_AH_BO), 2.46–2.38 (1H, m, CH(CH₃)₂), 1.51 (3H, d, J 7.0, PhCHCH₃), 0.91 (3H, d, J 7.0, CH₃^ACHCH₃^B) and 0.90 (3H, d, J 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl_3) 174.3 (NC=O), 153.4 (OC=O), 140.1 (*i*-C; Ph), 128.3,² 127.9² and 126.9¹ (5 \times CH; Ph), 62.8 (CH₂O), 58.7 (*i*-PrCHN), 42.7 (PhCHCH₃), 28.3 (CH(CH₃)₂), 19.5 (CH₃^ACHCH₃^B), 17.7 (CH₃^ACHCH₃^B) and 14.5 (PhCHCH₃) (Found MH^+ 262.1434; C₁₅H₂₀NO₃⁺ requires 262.1443); the oxazolidin-2-one (*rac*)-*syn*-**34** (196 mg, 55%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.43; mp 44–47 °C; ν_{\max} (CHCl₃) cm^{-1} 1774 (OC=O) and 1703 (NC=O); δ_H (400 MHz; CDCl_3) 7.37–7.32 (3H, m, 4 \times CH; Ph), 7.23–7.18 (2H, m, 2 \times CH; Ph), 5.13 (1H, q, J 6.9, PhCHCH₃), 4.47 (1H, dt, J 8.9 and 3.5, *i*-PrCHN), 4.22 (1H, t, J 8.9, CH_AH_BO), 4.09 (1H, dd, J 8.9 and 3.5, CH_AH_BO), 2.21–2.12 (1H, m, CH(CH₃)₂), 1.46 (3H, d, J 6.9, PhCHCH₃), 0.79 (3H, d, J 7.0, CH₃^ACHCH₃^B) and 0.44 (3H, d, J 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl_3) 174.4 (NC=O), 153.4 (OC=O), 140.4 (*i*-C; Ph), 128.4,² 127.9² and 127.0¹ (5 \times CH; Ph), 62.8 (CH₂O), 57.9 (*i*-PrCHN), 43.2 (PhCHCH₃), 27.8 (CH(CH₃)₂), 18.8 (CH₃^ACHCH₃^B), 17.7 (CH₃^ACHCH₃^B) and 13.9 (PhCHCH₃) (Found MH^+ 262.1432; C₁₅H₂₀NO₃⁺ requires 262.1443).

4.13. Synthesis of 4-phenyl-3-(2-phenylpropanoyl)oxazolidin-2-one (*rac*)-*anti*-**41** and 4-phenyl-3-(2-phenylpropanoyl)oxazolidin-2-one (*rac*)-*syn*-**41**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-phenyl oxazolidin-2-one (*rac*)-**8** (0.22 g, 1.36 mmol) and pentafluorophenyl 2-phenylpropanoate (*rac*)-**13** (0.47 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**41** (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 oxazolidin-2-one (*rac*)-*anti*-**41** (8 mg, 2%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.58; mp 106–108 °C; ν_{\max} (CHCl₃) cm^{-1} 1780 (OC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl_3) 7.35–7.26 (6H, m, 6 \times CH; Ph), 7.26–7.17 (4H, m, 4 \times CH; Ph), 5.28 (1H, dd, J 8.8 and 3.2, PhCHN), 5.06 (1H, q, J 7.2, PhCHCH₃), 4.47 (1H, t, J 8.8, CH_AH_BO), 4.14 (1H, dd, J 8.8 and 3.2, CH_AH_BO) and 1.35 (3H, d, J 7.2, PhCHCH₃); δ_C (100 MHz; CDCl_3) 174.0 (NC=O), 153.2 (OC=O), 140.1 (*i*-C; Ph), 139.1 (*i*-C; Ph), 129.2,² 128.7,¹ 128.6,² 128.2,² 127.2¹ and 125.8² (10 \times CH; 2 \times Ph), 69.7 (CH₂O), 58.1 (PhCHN), 43.2 (PhCHCH₃) and 19.4 (PhCHCH₃) (Found MH^+ , 296.1282; C₁₈H₁₈NO₃⁺ requires 296.1287); and the oxazolidin-2-one (*rac*)-*syn*-**41** (0.27 g, 68%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; mp 124–125 °C; ν_{\max} (CHCl₃) cm^{-1} 1778 (OC=O) and 1701 (NC=O); δ_H (400 MHz; CDCl_3) 7.23–7.10 (10H, m, 10 \times CH; 2 \times Ph), 5.37 (1H, dd J 9.0 and 5.1, PhCHN), 5.02 (1H, q, J 6.9, PhCHCH₃), 4.55 (1H, t, J 9.0, CH_AH_BO), 3.99 (1H, dd, J 9.0 and 5.1, CH_AH_BO) and 1.34 (3H, d, J 6.9, PhCHCH₃); δ_C (100 MHz; CDCl_3) 173.6 (NC=O), 153.1 (OC=O), 139.7 (*i*-C; Ph), 138.2 (*i*-C; Ph), 128.8,² 128.4,³ 128.1,² 127.0¹ and 125.8² (10 \times CH; 2 \times Ph), 69.5 (CH₂O), 57.8 (PhCHN), 43.9 (PhCHCH₃) and 18.6 (PhCHCH₃) (Found MH^+ , 296.1286; C₁₅H₁₈NO₃⁺ requires 296.1287).

4.14. Synthesis of ethyl 2-oxa-3-(2-phenylpropanoyl)oxazolidin-4-carboxylate (*rac*)-*anti*-**46** and ethyl 2-oxa-3-(2-phenylpropanoyl)oxazolidin-4-carboxylate (*rac*)-*syn*-**46**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), ethyl oxazolidin-2-one 4-carboxylate (*rac*)-**14** (0.40 g, 1.36 mmol) and pentafluorophenyl 2-phenylpropanoate (*rac*)-**15** (0.21 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**46** (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 oxazolidin-2-one (*rac*)-*anti*-**46** (12 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; ν_{\max} (CHCl₃) cm^{-1} 1794 (OC=O), 1747 (CC=O) and 1705 (NC=O); δ_H (400 MHz; CDCl_3) 7.33–7.20 (5H, m, 5 \times CH; Ph), 5.10 (1H, q, J 7.0, PhCHCH₃), 4.77 (1H, dd, J 9.2 and 3.5, EtO₂CCHN), 4.38 (1H, t, J 9.2, CH_AH_BO), 4.29 (1H, q, J 7.2, CH₃CH_AH_BO), 4.28 (1H, q, J 7.2, CH₃CH_AH_BO), 4.26 (1H, dd, J 9.2 and 3.5, CH_AH_BO), 1.50 (3H, d, J 7.0, PhCHCH₃) and 1.30 (3H, t, J 7.2, CH₃CH₂O); δ_C (100 MHz; CDCl_3) 174.5 (NC=O), 168.7 (CC=O), 152.1 (OC=O), 140.0 (*i*-C; Ph), 128.7,² 128.3² and 127.4¹ (5 \times CH; Ph), 64.3 (CH₂O), 62.6 (CH₂O), 55.9 (EtO₂CCHN), 43.0 (PhCHCH₃), 19.3 (PhCHCH₃) and 14.1 (CH₃CH₂O) (Found MH^+ , 292.1195; C₁₅H₁₈NO₅⁺ requires 292.1185); and the oxazolidin-2-one (*rac*)-*syn*-**46** (236 mg, 60%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.30; ν_{\max} (CHCl₃) cm^{-1} 1793 (OC=O), 1747 (CC=O) and 1705 (NC=O); δ_H (400 MHz; CDCl_3) 7.40–7.20 (5H, m, 5 \times CH; Ph), 5.03 (1H, q, J 7.0, PhCHCH₃), 4.94 (1H, dd, J 9.3 and 4.9, EtO₂CCHN), 4.52 (1H, t, J 9.3, CH_AH_BO), 4.23 (1H, dd, J 9.3 and 4.9, CH_AH_BO), 4.11 (2H, q, J 7.2, CH₃CH₂O), 1.48 (3H, d, J 7.0, PhCHCH₃) and 1.11 (3H, t, J 7.2, CH₃CH₂O); δ_C (100 MHz; CDCl_3) 174.1 (NC=O), 167.9 (CC=O), 151.8 (OC=O), 139.6 (*i*-C; Ph), 128.4,² 128.1² and 127.1¹ (5 \times CH; Ph), 64.1 (CH₂O), 62.3 (CH₂O), 55.6 (EtO₂CCHN), 43.0 (PhCHCH₃), 19.2 (PhCHCH₃) and 13.7 (CH₃CH₂O) (Found MH^+ , 292.1195; C₁₅H₁₈NO₅⁺ requires 292.1185).

4.15. Synthesis of 4-methyl-5-phenyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*anti*,*syn*-**21** and 4-methyl-5-phenyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*syn*,*syn*-**21**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-(4*RS*,5*SR*)-**11** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** (0.49 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **21** (ratio: 77:23 *syn*,*syn*:*syn*,*anti*-). The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*,*syn*-**21** (65 mg, 15%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.75; ν_{\max} (CHCl₃) cm^{-1} 1782 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl_3) 7.44–7.24 (10H, m, 10 \times CH; 2 \times Ph), 5.49 (1H, d, J 7.2, PhCHO), 4.97 (1H, t, J 7.5, PhCH₂Et), 4.69 (1H, m (appears as a br quintet, J 7.2), CH₃CHN), 2.14 (1H, ddq, J 13.4, 7.5 and 7.3, CH_AH_BCH₃), 1.84 (1H, ddq, J 13.4, 7.5 and 7.3, CH_AH_BCH₃), 0.92 (3H, d, J 6.7, CH₃CHN) and 0.89 (3H, t, J 7.3, CH₂CH₃); δ_C (100 MHz; CDCl_3) 174.4 (NC=O), 153.1 (OC=O), 139.2 (*i*-C; Ph), 133.6 (*i*-C; Ph), 128.6,¹ 128.6,⁴ 128.5,² 127.3¹ and 125.6² (10 \times CH; Ph_A and Ph_B), 78.9 (PhCHO), 55.7 (CH₃CHN), 50.8 (PhCH₂Et), 27.7 (PhCHCH₂CH₃), 14.9 (CH₃CHN) and 12.4 (CH₂CH₃) (Found MH^+ , 324.1585; C₂₀H₂₂NO₃ requires 324.1600); and the oxazolidin-2-one (*rac*)-*syn*,*syn*-**21** (0.23 g, 53%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; ν_{\max} (CHCl₃) cm^{-1} 1778 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl_3) 7.40–7.18 (10H, m, 10 \times CH; 2 \times Ph), 5.65 (1H,

d, *J* 7.5, PhCHO), 4.89–4.77 (2H, m, 2 × PhCH₂Et and CH₃CHN), 2.11 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 1.84 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 0.91 (3H, t, *J* 7.4, CH₂CH₃) and 0.71 (3H, d, *J* 6.6, CH₃CHN); δ_C (100 MHz; CDCl₃) 173.6 (NC=O), 152.5 (OC=O), 138.4 (*i*-C; Ph_A), 133.3 (*i*-C; Ph_B), 128.6,¹ 128.5,⁴ 128.4,² 127.1¹ and 125.6² (10 × CH; Ph_A and Ph_B), 78.6 (PhCHO), 54.6 (CH₃CHN), 50.9 (PhCH₂Et), 27.1 (PhCH₂CH₂CH₃), 14.0 (CH₃CHN) and 12.0 (CH₂CH₃) (Found MH⁺, 324.1583; C₂₀H₂₂NO₃ requires 324.1600).

4.16. Synthesis of 4-benzyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*anti*-28 and 4-benzyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*syn*-28

In the same way as the oxazolidin-2-one (*rac*)-20, *n*-BuLi (1.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-12 (0.24 g, 1.36 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-16 (0.49 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones 28 (ratio: 68:32 *syn*–*anti*). The crude residue was purified by flash column chromatography on silica gel eluting light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-28 (0.10 g, 22%) as a colourless oil; *R*_F [light petroleum (40–60 °C)/diethyl ether (1:1)] 0.70; *v*_{max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1691 (NC=O); δ_H (400 MHz; CDCl₃) 7.41–7.22 (10H, m, 10 × CH; 2 × Ph), 4.95 (1H, t, *J* 7.5, PhCH₂Et), 4.64–4.55 (1H, m, BnCHN), 4.12–4.00 (2H, m, CH₂O), 3.37 (1H, dd, *J* 13.3 and 3.3, CH_AH_BPh), 2.79 (1H, dd, *J* 13.3 and 9.8, CH_AH_BPh), 2.17 (1H, ddq, *J* 13.4, 7.5 and 7.3, CH_AH_BCH₃), 1.88 (1H, ddq, *J* 13.4, 7.5 and 7.3, CH_AH_BCH₃) and 0.93 (3H, t, *J* 7.3, CH₂CH₃); δ_C (100 MHz; CDCl₃) 174.2 (NC=O), 153.0 (OC=O), 138.7 (*i*-C; Ph_A), 135.4 (*i*-C; Ph_B), 129.5,² 129.0,² 128.7,² 128.6,² 127.5¹ and 127.4¹ (10 × CH; Ph_A and Ph_B), 65.8 (CH₂O), 55.8 (BnCHN), 50.4 (PhCH₂Et), 38.1 (CH₂Ph), 27.5 (PhCH₂CH₂CH₃) and 12.1 (CH₂CH₃) (Found MH⁺, 324.1612; C₂₀H₂₂NO₃ requires 324.1600); and the oxazolidin-2-one (*rac*)-*syn*-28 (0.22 g, 50%) as a colourless oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; *v*_{max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1691 (NC=O); δ_H (400 MHz; CDCl₃) 7.46–6.91 (10H, m, 10 × CH; 2 × Ph), 4.91 (1H, t, *J* 7.6, PhCH₂Et), 4.81–4.71 (1H, m, BnCHN), 4.19 (1H, br t, *J* 8.8, CH_AH_BO), 4.08 (1H, dd, *J* 8.8 and 3.1, CH_AH_BO), 3.05 (1H, dd, *J* 13.5 and 3.3, CH_AH_BPh), 2.59 (1H, dd, 13.5 and 8.7, CH_AH_BPh), 2.15 (1H, ddq, *J* 13.4, 7.5 and 7.3, CH_AH_BCH₃), 1.86 (1H, ddq, *J* 13.4, 7.5 and 7.3, CH_AH_BCH₃) and 0.90 (3H, t, *J* 7.4, CH₂CH₃); δ_C (100 MHz; CDCl₃) 174.0 (NC=O), 153.0 (OC=O), 138.5 (*i*-C; Ph_A), 134.9 (*i*-C; Ph_B), 129.5,² 128.9,² 128.6,² 128.3,² 127.3¹ and 127.2¹ (10 × CH; Ph_A and Ph_B), 65.7 (CH₂O), 54.9 (BnCHN), 50.6 (PhCH₂Et), 37.4 (CH₂Ph), 27.0 (PhCH₂CH₂CH₃) and 12.1 (CH₂CH₃) (Found MH⁺, 324.1585; C₁₆H₂₂NO₃ requires 324.1600).

4.17. Synthesis of 4-isopropyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*anti*-35 and 4-isopropyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*syn*-35

In the same way as oxazolidin-2-one (*rac*)-20, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-13 (0.17 g, 1.36 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-16 (0.49 g, 1.50 mmol), gave a mixture of two separable diastereoisomeric oxazolidin-2-ones 35 (ratio: 95:5 *syn*–*anti*). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-35 (11 mg, 3%) as a colourless oil; *R*_F [light petroleum (40–60 °C)/diethyl ether (1:1)] 0.63; *v*_{max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl₃) 7.40–7.35 (2H, m, 2 × CH; Ph), 7.28–7.19 (3H, m, 3 × CH; Ph), 4.98 (1H, t, *J* 7.5, PhCH₂Et), 4.40–4.34 (1H, m, *i*-PrCHN), 4.17–4.07 (2H, m, CH₂O), 2.49–2.38 (1H, m, CH(CH₃)₂), 2.15 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 1.82 (1H, ddq, *J*

13.4, 7.5 and 7.4, CH_AH_BCH₃), 0.85 (3H, d, *J* 7.0, CH₃^ACHCH₃^B) 0.84 (3H, d, *J* 6.9, CH₃^ACHCH₃^B) and 0.82 (3H, t, *J* 7.4, CH₃CH₂); δ_C (100 MHz; CDCl₃) 174.1 (NC=O), 153.6 (OC=O), 138.6 (*i*-C; Ph), 128.6,² 128.4² and 127.1¹ (5 × CH; Ph), 62.8 (CH₂O), 58.3 (*i*-PrCHN), 50.1 (PhCH₂Et), 28.4 (CH(CH₃)₂), 27.7 (CH₂CH₃), 17.9 (CH₃^ACHCH₃^B) 14.5 (CH₃^ACHCH₃^B) and 12.0 (CH₂CH₃) (Found MH⁺, 276.1612; C₁₆H₂₂NO₃ requires 276.1600); and the oxazolidin-2-one (*rac*)-*syn*-35 (0.22 g, 59%) as an oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.53; *v*_{max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl₃) 7.39–7.36 (2H, m, 2 × CH; Ph), 7.32–7.21 (3H, m, 3 × CH; Ph), 4.94 (1H, t, *J* 7.5, PhCH₂Et), 4.52–4.47 (1H, m, *i*-PrCHN), 4.24 (1H, t, *J* 9.0, CH_AH_BO), 4.1 (1H, dd, *J* 9.0 and 3.4, CH_AH_BO), 2.21–2.15 (1H, m, CH(CH₃)₂), 2.10 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 1.81 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 0.87 (3H, t, *J* 7.4, CH₃CH₂), 0.78 (3H, d, *J* 7.0, CH₃^ACHCH₃^B) and 0.42 (3H, d, *J* 7.0, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl₃) 174.0 (NC=O), 153.5 (OC=O), 138.9 (*i*-C; Ph), 128.6,² 128.5² and 127.2¹ (5 × CH; Ph), 62.9 (*i*-PrCHN), 58.1 (CH₂O), 50.7 (PhCH₂Et), 27.9 (CH(CH₃)₂), 26.4 (CH₂CH₃), 17.8 (CH₃^ACHCH₃^B), 14.0 (CH₃^ACHCH₃^B) and 12.0 (CH₂CH₃) (Found MH⁺, 276.1587; C₁₆H₂₂NO₃ requires 276.1600).

4.18. Synthesis of 4-phenyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*anti*-42 and 4-phenyl-3-(2-phenylbutanoyl)oxazolidin-2-one (*rac*)-*syn*-42

In the same way as the oxazolidin-2-one (*rac*)-20, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-8 (0.22 g, 1.36 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-16 (0.49 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones 42 (ratio: >98:2 *syn*–*anti*). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-42 (5 mg, 1%) as a colourless oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; *v*_{max} (CHCl₃) cm⁻¹ 1780 (OC=O), 1703 (NC=O) and 1600 (Ph); δ_H (400 MHz; CDCl₃) 7.44–7.21 (10H, m, 10 × CH; 2 × Ph), 5.34 (1H, dd, *J* 8.7 and 3.4, PhCHN), 4.96 (1H, t, *J* 7.5, PhCH₂Et), 4.54 (1H, br t, *J* 8.7, CH_AH_BO), 4.20 (1H, dd, *J* 8.7 and 3.4, CH_AH_BO), 2.01 (1H, ddq, *J* 13.5, 7.5 and 7.4, CH_AH_BCH₃), 1.74 (1H, ddq, *J* 13.5, 7.5 and 7.4, CH_AH_BCH₃) and 0.76 (3H, t, *J* 7.4, CH₂CH₃); δ_C (100 MHz; CDCl₃) 173.7 (NC=O), 153.4 (OC=O), 139.5 (*i*-C; Ph), 138.6 (*i*-C; Ph), 129.1,² 128.8,² 128.7,¹ 128.5,² 127.3¹ and 125.8² (10 × CH; Ph_A and Ph_B), 69.4 (CH₂O), 58.1 (PhCHN), 50.4 (PhCH₂Et), 27.7 (CH₂Ph) and 12.0 (CH₂CH₃) (Found MH⁺, 310.1430; C₁₉H₂₀NO₃ requires 310.1443); and the oxazolidin-2-one (*rac*)-*syn*-42 (0.29 g, 69%) as a white solid; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.50; mp 58–62 °C; *v*_{max} (film)cm⁻¹ 1780 (OC=O) and 1703 (NC=O); δ_H (400 MHz; CDCl₃) 7.27–7.19 (6H, m, 6 × CH; 2 × Ph), 7.14–7.12 (2H, m, 2 × CH; Ph), 6.91–6.89 (2H, m, 2 × CH; Ph), 5.46 (1H, dd, *J* 8.9 and 5.0, PhCHN), 4.90 (1H, t, *J* 7.5, PhCH₂Et), 4.63 (1H, t, *J* 8.9, CH_AH_BO), 4.07 (1H, dd, *J* 8.9 and 5.0, CH_AH_BO), 2.04 (1H, ddq, *J* 13.5, 7.5 and 7.4, CH_AH_BCH₃), 1.71 (1H, ddq, *J* 13.5, 7.5 and 7.4, CH_AH_BCH₃) and 0.87 (3H, t, *J* 7.4, CH₂CH₃); δ_C (100 MHz; CDCl₃) 173.2 (NC=O), 153.2 (OC=O), 138.4 (*i*-C; Ph_A), 138.1 (*i*-C; Ph_B), 128.9,² 128.8,² 128.4,¹ 128.3,² 127.1¹ and 125.6² (10 × CH; Ph_A and Ph_B), 69.6 (CH₂O), 57.8 (PhCHN), 51.2 (PhCH₂Et), 26.3 (CH₂Ph) and 12.0 (CH₂CH₃) (Found MH⁺, 310.1437; C₁₉H₂₀NO₃ requires 310.1443).

4.19. Synthesis of ethyl 2-oxa-3-(2-phenylbutanoyl)oxazolidin-4-carboxylate (*rac*)-*anti*-47 and ethyl 2-oxa-3-(2-phenylbutanoyl)oxazolidin-4-carboxylate (*rac*)-*syn*-47

In the same way as the oxazolidin-2-one (*rac*)-20, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-14

(0.21 g, 1.36 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** (0.49 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **47** (ratio: 96:4 *syn*–*anti*). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**47** (11 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.48; ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1747 (CC=O) and 1705 (NC=O); δ_H (400 MHz; CDCl₃) 7.32–7.27 (2H, m, 2 × CH; Ph), 7.25–7.14 (3H, m, 3 × CH; Ph), 4.94 (1H, t, *J* 7.5, PhCH₂Et), 4.79 (1H, dd, *J* 9.4 and 3.5, EtO₂CCHN), 4.40 (1H, t, *J* 9.3, CH_AH_BO), 4.29 (2H, q, *J* 7.1, OCH₂CH₃), 4.24 (1H, dd, *J* 9.3 and 3.5, CH_AH_BO), 2.21–2.08 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 1.87–1.74 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 1.30 (3H, t, *J* 7.1, OCH₂CH₃) and 0.91 (3H, t, *J* 7.4, CH₂CH₃; PhCH₂Et); δ_C (100.6 MHz; CDCl₃) 174.1 (NC=O), 168.6 (CC=O), 152.2 (OC=O), 138.5 (*i*-C; Ph_A), 128.8,² 128.6² and 127.5¹ (5 × CH; Ph), 64.2 (CH₂O), 62.6 (CH₂O), 55.9 (EtO₂CCHN), 50.2 (PhCH₂Et), 27.5 (PhCH₂CH₂CH₃), 14.1 (OCH₂CH₃) and 12.0 (CH₂CH₃; PhCH₂Et) (Found M⁺, 305.1258; C₁₆H₁₉NO₅ requires 305.1258); and the oxazolidin-2-one (*rac*)-*syn*-**47** (0.27 g, 65%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.38; ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1747 (CC=O) and 1701 (NC=O); δ_H (400 MHz; CDCl₃) 7.36–7.23 (5H, m, 5 × CH; Ph), 4.94 (1H, dd, *J* 9.4 and 4.7, EtO₂CCHN), 4.82 (1H, t, *J* 7.5, PhCH₂Et), 4.51 (1H, t, *J* 9.4, CH_AH_BO), 4.23 (1H, dd, *J* 9.4 and 4.7, CH_AH_BO), 4.07 (2H, q, *J* 7.1, OCH₂CH₃), 2.14–2.01 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 1.86–1.75 (1H, ddq, *J* 13.4, 7.5 and 7.4, CH_AH_BCH₃), 1.06 (3H, t, *J* 7.1, OCH₂CH₃) and 0.87 (3H, t, *J* 7.4, CH₂CH₃; PhCH₂Et); δ_C (100.6 MHz; CDCl₃) 173.7 (NC=O), 168.1 (CC=O), 152.1 (OC=O), 138.0 (*i*-C; Ph), 128.9,² 128.6² and 127.3¹ (5 × CH; Ph), 64.2 (CH₂O), 62.3 (CH₂O), 55.7 (EtO₂CCHN), 50.5 (PhCH₂Et), 27.3 (PhCH₂CH₂CH₃), 13.8 (OCH₂CH₃) and 12.1 (CH₂CH₃; PhCH₂Et) (Found M⁺, 305.1256; C₁₆H₁₉NO₅ requires 305.1258).

4.20. Synthesis of 4-methyl-5-phenyl-3-[2-phenyl-3-methylbutanoyl]oxazolidin-2-one (*rac*)-*anti*,*syn*-**22** and 4-methyl-5-phenyl-3-[2-phenyl-3-methylbutanoyl]oxazolidin-2-one (*rac*)-*syn*,*syn*-**22**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-methyl-5-phenyl-oxazolidin-2-one (*rac*)-(4*RS*,5*SR*)-**11** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.52 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*,*syn*- and *anti*,*syn*-**22** (ratio 73:27:*syn*,*syn*–*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 oxazolidin-2-one (*rac*)-*anti*,*syn*-**22** (68 mg, 15%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.71; mp 98–100 °C; ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl₃) 7.35–7.16 (10H, m, 10 × CH; 2 × Ph), 5.41 (1H, d, *J* 7.2, CHOPh), 4.74 (1H, d, *J* 10.6, PhCH_i-Pr), 4.63 (1H, dq, *J* 6.7 and 6.6, CH₃CHN), 2.42 (1H, m, CH(CH₃)₂), 0.97 (3H, d, *J* 6.7, CH₃CHN), 0.87 (3H, d, *J* 6.6, CH₃CHCH₃^B) and 0.59 (3H, d, *J* 6.8, CH₃CHCH₃^B); δ_C (100 MHz; CDCl₃) 174.1 (NC=O), 152.7 (OC=O), 138.2 (*i*-C; Ph), 133.3 (*i*-C; Ph), 129.1,² 128.7,¹ 128.6,² 128.5,² 127.4¹ and 125.6² (10 × CH; 2 × Ph), 78.4 (OCHPh), 55.9 (CH₃CHN), 55.2 (PhCH_i-Pr), 32.4 (CH(CH₃)₂), 21.4 (CCH₃CHCH₃^B), 20.1 (CH₃CHCH₃^B) and 14.6 (CH₃CHN); (Found MNH₄⁺, 355.2018; C₂₁H₂₇N₂O₃⁺ requires MNH₄⁺ 355.2016); and the oxazolidin-2-one (*rac*)-*syn*,*syn*-**22** (0.185 g, 40%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.66; mp 100–103 °C; ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1701 (NC=O); δ_H (400 MHz; CDCl₃) 7.34–7.13 (10H, m, 10 × CH; 2 × Ph), 5.58 (1H, d, *J* 7.5, CH, OCHPh), 4.74 (1H, dq, *J* 6.7 and 6.6,

CH₃CHN), 4.59 (1H, d, *J* 10.6, PhCH_i-Pr), 2.38 (1H, m, CH(CH₃)₂), 1.00 (3H, d, *J* 6.7, CH₃CHN), 0.64 (3H, d, *J* 6.8, CH₃CHCH₃^B) and 0.59 (3H, d, *J* 6.8, CH₃CHCH₃^B); δ_C (100 MHz; CDCl₃) 173.7 (NC=O), 152.6 (OC=O), 137.7 (*i*-C; Ph), 133.3 (*i*-C; Ph), 129.1,² 128.7,¹ 128.6,² 128.4,² 127.2¹ and 125.6² (10 × CH; 2 × Ph), 78.6 (OCHPh), 56.7 (CH₃CHN), 54.8 (PhCH_i-Pr), 31.9 (CH(CH₃)₂), 21.7 (CH₃CHCH₃^B), 20.2 (CH₃CHCH₃^B) and 14.0 (CH₃CHN) (Found MH⁺, 338.1740; C₂₁H₂₄NO₃⁺ requires MH⁺ 338.1751).

4.21. Synthesis of 4-benzyl-3-[2-phenyl-3-methylbutanoyl]oxazolidin-2-one (*rac*)-*anti*,*syn*-**29** and 4-benzyl-3-[2-phenyl-3-methylbutanoyl]oxazolidin-2-one (*rac*)-*syn*,*syn*-**29**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-benzyl-oxazolidin-2-one (*rac*)-**12** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.51 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**29** (ratio: 66:34 *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 oxazolidin-2-one (*rac*)-*anti*-**29** (62 mg, 14%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 87–89 °C; ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1710 (NC=O); δ_H (400 MHz; CDCl₃) 7.42–7.39 (2H, dt, *J* 8.1 and 1.2, 2 × CH; Ph), 7.35–7.20 (8H, m, 8 × CH; 2 × Ph), 4.78 (1H, d, *J* 10.6, PhCH_i-Pr), 4.61–4.52 (1H, m, *i*-PrCHN), 4.07 (1H, br dd, *J* 9.0 and 2.6, CH_AH_BO), 4.00 (1H, t, *J* 9.0, CH_AH_BO), 3.39 (1H, dd, *J* 13.3 and 3.3, CH_AH_BPh), 2.73 (1H, dd, *J* 13.3 and 10.1, CH_AH_BPh), 2.53–2.46 (1H, m, CH(CH₃)₂), 1.09 (3H, d, *J* 6.4, CH₃CHCH₃^B) and 0.72 (3H, d, *J* 6.8, CH₃CHCH₃^B); δ_C (100 MHz; CDCl₃) 174.2 (NC=O), 153.1 (OC=O), 138.0 (*i*-C; Ph), 135.4 (*i*-C; Ph), 129.4², 129.2², 129.0², 128.5², 127.4¹ and 127.3¹ (10 × CH; 2 × Ph), 65.6 (CH₂O), 55.9 (BnCHN), 55.8 (PhCH_i-Pr), 38.1 (PhCH₂), 32.5 (CH(CH₃)₂), 21.5 (CH₃CHCH₃^B) and 20.2 (CH₃CHCH₃^B) (Found MNH₄⁺, 355.2019; C₂₁H₂₇N₂O₃⁺ requires MNH₄⁺ 355.2016); and the oxazolidin-2-one (*rac*)-*syn*-**29** (0.12 g, 26%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.40; ν_{\max} (CHCl₃) cm⁻¹ 1773 (OC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl₃) 7.39 (2H, dt, *J* 7.0 and 1.7, 2 × CH; Ph), 7.28 (2H, tt, *J* 7.0 and 1.2, 2 × CH; Ph), 7.22 (1H, *J* 7.2 and 1.5, 1 × CH; Ph), 7.12–7.05 (3H, m, 3 × CH; Ph), 6.80 (2H, dt, *J* 6.2 and 1.8, 2 × CH; Ph), 4.73–4.66 (1H, m, BnCHN), 4.65 (1H, d, *J* 10.8, PhCH_i-Pr), 4.13 (1H, t, *J* 9.0, CH_AH_BO), 4.01 (1H, dd, *J* 9.0 and 2.8, CH_AH_BO), 2.89 (1H, dd, *J* 13.6 and 3.3, CH_AH_BPh), 2.53 (1H, dd, *J* 13.6 and 8.3, CH_AH_BPh), 2.49–2.39 (1H, m, CH(CH₃)₂), 0.99 (3H, d, *J* 6.4, CH₃CHCH₃^B) and 0.65 (3H, d, *J* 6.8, CH₃CHCH₃^B); δ_C (100 MHz; CDCl₃) 173.9 (NC=O), 153.0 (OC=O), 137.8 (*i*-C; Ph), 134.8 (*i*-C; Ph), 129.4,² 129.3,² 128.8,² 128.5,² 127.4¹ and 127.2¹ (10 × CH; 2 × Ph), 65.6 (CH₂O), 56.4 (BnCHN), 54.8 (PhCH_i-Pr), 37.1 (PhCH₂), 31.7 (CH(CH₃)₂), 21.7 (CH₃CHCH₃^B) and 20.1 (CH₃CHCH₃^B) (Found MNH₄⁺, 355.2018; C₂₁H₂₇N₂O₃⁺ requires MNH₄⁺ 355.2016).

4.22. Synthesis of 4-isopropyl-3-[2-phenyl-3-methylbutanoyl]oxazolidin-2-one (*rac*)-*anti*-**36** and 4-isopropyl-3-[2-phenyl-3-methylbutanoyl]oxazolidin-2-one (*rac*)-*syn*-**36**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-isopropyl-oxazolidin-2-one (*rac*)-**13** (0.17 g, 1.36 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.51 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**36** (ratio 68:32:*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 oxazolidin-2-ones (*rac*)-*anti*- and (*rac*)-*syn*-**36** (0.13 g, 33%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82.

Oxazolidin-2-one (*rac*)-*anti*-**36**; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82; ν_{\max} (CHCl₃) cm⁻¹ 1770 (OC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.43–7.21 (5H, m, 5 × CH; Ph), 4.77 (1H, d, *J* 10.5, PhCHI-Pr), 4.49 (1H, m, *i*-PrCHN), 4.23 (1H, t, *J* 8.7, CH_AH_BO), 4.10 (1H, dd, *J* 8.7 and 3.2, CH_AH_BO), 2.53–2.41 (1H, m, CH(CH₃)₂; PhCHI-Pr), 2.16–2.07 (1H, m, CH(CH₃)₂; oxazolidin-2-one), 1.05, 0.76, 0.71 and 0.25 (4 × 3H, d, *J* ~6.9, 2 × CH(CH₃)₂); δ_C (100 MHz, CDCl₃) 174.2 (NC=O), 153.3 (OC=O), 138.3 (*i*-C; Ph), 129.3², 128.6² and 127.3¹ (5 × CH; Ph), 62.6 (CH₂O), 58.8 (*i*-PrCHN), 55.9 (PhCHI-Pr), 32.7 (CH(CH₃)₂; oxazolidin-2-one), 28.3 (CH(CH₃)₂; PhCHI-Pr), 21.4, 20.2, 18.2 and 14.6 (4C, 4 × CH₃; 2 × CH(CH₃)₂) (Found MH⁺, 290.1751; C₁₇H₂₄NO₃⁺ requires MH⁺ 290.1751; and found MNH₄⁺, 307.2015; C₁₇H₂₇N₂O₃⁺ requires MNH₄⁺ 307.2016).

Oxazolidin-2-one (*rac*)-*syn*-**36**; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82; ν_{\max} (CHCl₃) cm⁻¹ 1772 (OC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.43–7.22 (5H, m, 5 × CH; Ph), 4.81 (1H, d, *J* 10.5, PhCHI-Pr), 4.37 (1H, m, *i*-PrCHN), 4.15–4.08 (2H, m, CH₂O), 2.53–2.43 (2H, m, 2 × CH(CH₃)₂), 1.05, 0.92, 0.88 and 0.71 (4 × 3H, d, *J* 6.9, 2 × CH(CH₃)₂); δ_C (100 MHz, CDCl₃) 174.2 (NC=O), 153.2 (OC=O), 138.3 (*i*-C; Ph), 129.4², 128.5² and 127.3¹ (5 × CH; Ph), 62.8 (CH₂O), 58.8 (*i*-PrCHN), 55.4 (PhCHI-Pr), 32.7 (CH(CH₃)₂; oxazolidin-2-one), 28.4 (CH(CH₃)₂; PhCHI-Pr), 21.4, 20.2, 18.2 and 14.6 (4C, 4 × CH₃; 2 × CH(CH₃)₂) (Found MH⁺, 290.1751; C₁₇H₂₄NO₃⁺ requires MH⁺ 290.1751; and found MNH₄⁺, 307.2015; C₁₇H₂₇N₂O₃⁺ requires MNH₄⁺ 307.2016).

4.23. Synthesis of 4-phenyl-3-[2-phenyl-3-methylbutanoyl]-oxazolidin-2-one (*rac*)-*anti*-**43** and 4-phenyl-3-[2-phenyl-3-methylbutanoyl]oxazolidin-2-one (*rac*)-*syn*-**43**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-phenyl-oxazolidin-2-one (*rac*)-**8** (0.22 g, 1.36 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.51 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**43** (ratio: 87:13 *syn*:-*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum (40–60 °C)/diethyl ether (7:3) to give an inseparable mixture of oxazolidin-2-ones (*rac*)-*anti*- and (*rac*)-*syn*-**43** (0.11 g, 25%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.64.

Oxazolidin-2-one (*rac*)-*anti*-**43**; R_f [light petroleum (bp 40–60 °C)/diethyl ether (1:1)] 0.64; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.37–7.04 (10H, m, 10 × CH; 2 × Ph), 5.27 (1H, dd, *J* 8.8 and 3.2, PhCHN), 4.71 (1H, d, *J* 10.7, PhCHI-Pr), 4.48 (1H, t, *J* 8.8, CH_AH_BO), 4.14 (1H, dd, *J* 8.8 and 3.2, CH_AH_BO), 2.33–2.23 (1H, m, CH(CH₃)₂), 0.74 (3H, d, *J* 6.9, CH₃^ACHCH₃^B) and 0.55 (3H, d, *J* 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz, CDCl₃) 174.0 (NC=O), 153.3 (OC=O), 139.6 (*i*-C; Ph), 137.3 (*i*-C; Ph), 129.3², 128.9¹, 128.6¹, 128.3² and 125.8² (10 × CH; 2 × Ph), 69.4 (CH₂O), 58.1 (PhCHN), 55.9 (PhCHI-Pr), 32.8 (CH(CH₃)₂), 21.3 (CH₃^ACHCH₃^B) and 20.2 (CH₃^ACHCH₃^B) (Found MNH₄⁺, 341.1864; C₂₀H₂₅N₂O₃⁺ requires MNH₄⁺ 341.1860).

Oxazolidin-2-one (*R,S*)-*syn*-**43** (derived from a stereospecific synthesis); R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.61; ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.20–7.01 (8H, m, 8 × CH; 2 × Ph), 6.73 (2H, d, *J* 7.3, 2 × CH; Ph), 5.40 (1H, dd, *J* 8.7 and 3.6, PhCHN), 4.66 (1H, d, *J* 10.7, PhCHI-Pr), 4.56 (1H, t, *J* 8.8, CH_AH_BO), 3.97 (1H, dd, *J* 8.8 and 3.6, CH_AH_BO), 2.36–2.23 (1H, m, CH(CH₃)₂), 0.97 (3H, d, *J* 6.9, CH₃^ACHCH₃^B) and 0.58 (3H, d, *J* 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz, CDCl₃) 174.0 (NC=O), 153.2 (OC=O), 139.7 (*i*-C; Ph), 137.8 (*i*-C; Ph), 129.2², 128.9¹, 128.6¹, 128.3² and 125.9² (10 × CH; 2 × Ph), 69.5 (CH₂O), 58.2 (PhCHN), 55.5 (PhCHI-Pr), 32.8 (CH(CH₃)₂), 21.3 (CH₃^ACHCH₃^B) and 20.2 (CH₃^ACHCH₃^B) (Found MNH₄⁺, 341.1864; C₂₀H₂₅N₂O₃⁺ requires MNH₄⁺ 341.1860).

4.24. Synthesis of ethyl 2-oxa-3-[2-phenyl-3-methylbutanoyl]-oxazolidin-4-carboxylate (*rac*)-*anti*-**48** and ethyl 2-oxa-3-[2-phenyl-3-methylbutanoyl]oxazolidin-4-carboxylate (*rac*)-*syn*-**48**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), ethyl oxazolidin-2-one 4-carboxylate (*rac*)-**14** (0.21 g, 1.36 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.51 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**48** (ratio: 57:43 *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 oxazolidin-2-one (*rac*)-*anti*-**48** (0.11 g, 25%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1751 (CC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.43–7.30 (5H, m, 5 × CH; Ph), 4.71 (1H, dd, *J* 9.1 and 3.7, EtO₂CCHN), 4.70 (1H, d, *J* 10.4, PhCHI-Pr), 4.34 (1H, t, *J* 9.1, CH_AH_BO), 4.26 (3H, m, CH_ACH_BO and OCH₂CH₃), 2.45–2.35 (1H, m, CH(CH₃)₂), 1.24 (3H, t, *J* 9.1, OCH₂CH₃), 1.02 (3H, d, *J* 6.9, CH₃^ACHCH₃^B) and 0.64 (3H, d, *J* 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz, CDCl₃) 174.1 (NC=O), 168.5 (EtOC=O), 152.3 (OC=O), 137.7 (*i*-C; Ph), 129.2², 128.5² and 127.4¹ (5 × CH; Ph), 63.9 (CH₂O), 62.5 (CH₂O), 55.8 (EtO₂CCHN), 55.7 (PhCHI-Pr), 32.9 (CH(CH₃)₂), 21.3 (CH₃^ACHCH₃^B), 20.2 (CH₃^ACHCH₃^B) and 13.6 (OCH₂CH₃) (Found MNH₄⁺, 337.1759; C₁₇H₂₅N₂O₅⁺ requires MNH₄⁺ 337.1758); and the oxazolidin-2-one (*rac*)-*syn*-**48** (0.16 g, 37%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; mp 60–63 °C; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1755 (CC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.35–7.15 (5H, m, 5 × CH; Ph), 4.87 (1H, dd, *J* 9.1 and 3.8, EtO₂CCHN), 4.61 (1H, d, *J* 10.5, PhCHI-Pr), 4.45 (1H, t, *J* 9.1, CH_AH_BO), 4.16 (1H, dd, *J* 9.1 and 3.8, CH_AH_BO), 3.99–3.89 (1H, m, OCH₂CH₃), 2.43–2.37 (1H, m, CH(CH₃)₂), 0.98 (3H, t, *J* 6.8, OCH₂CH₃), 0.91 (3H, d, *J* 6.9, CH₃^ACHCH₃^B) and 0.64 (3H, d, *J* 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz, CDCl₃) 173.9 (NC=O), 168.5 (EtOC=O), 153.0 (OC=O), 137.5 (*i*-C; Ph), 129.3², 128.6² and 127.7¹ (5 × CH; Ph), 63.7 (CH₂O), 62.5 (CH₂O), 55.9 (EtO₂CCHN), 55.3 (PhCHI-Pr), 32.6 (CH(CH₃)₂), 21.2 (CH₃^ACHCH₃^B) and 20.3 (CH₃^ACHCH₃^B) and 13.9 (OCH₂CH₃) (Found MNH₄⁺, 337.1755; C₁₇H₂₅N₂O₅⁺ requires MNH₄⁺ 337.1758).

4.25. Synthesis of 4-methyl-5-phenyl-3-[2-(4-methylphenyl)propanoyl]oxazolidin-2-one (*rac*)-*anti*,*syn*-**23** and 4-methyl-5-phenyl-3-[2-(4-methylphenyl)propanoyl]oxazolidin-2-one (*rac*)-*syn*,*syn*-**23**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-methyl-5-phenyl-oxazolidin-2-one (4*RS*,5*SR*)-(*rac*)-**11** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** (0.49 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*,*syn*- and *anti*,*syn*-**23** (ratio 70:30 *syn*,*syn*:-*anti*,*syn*-). 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 oxazolidin-2-one (*rac*)-*anti*,*syn*-**23** (83 mg, 19%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.71; δ_H (400 MHz, CDCl₃) 7.35–7.28 (3H, m, 3 × CH; Ph), 7.21–7.19 (2H, m, 2 × CH; Ph), 7.20 (2H, dt, *J* 7.9 and 2.1, 2 × CH; Ar), 7.08 (2H, dt, *J* 7.9 and 2.1, 2 × CH; Ar), 5.46 (1H, d, *J* 6.9, OCHPh; oxazolidin-2-one), 5.04 (1H, q, *J* 7.1, ArCHCH₃), 4.60 (1H, m, CH₃CHN), 2.29 (3H, s, CH₃; Ar), 1.46 (3H, d, *J* 7.1, ArCHCH₃) and 0.89 (3H, d, *J* 6.9, CHCH₃; oxazolidin-2-one); δ_C (100 MHz, CDCl₃) 174.8 (NC=O), 152.9 (OC=O), 137.7 (*i*-C; Ar), 137.1 (*i*-C, Ar), 133.9 (*i*-C; Ph), 129.7² and 128.3² (4 × CH; Ar), 128.6³ and 125.5² (5 × CH; Ph), 79.1 (PhCHO), 55.0 (CH₃CHN), 43.6 (ArCHCH₃), 21.4 (CH₃; Ar), 19.8 (ArCHCH₃) and 14.5 (CH₃CHN; oxazolidin-2-one) (Found

MNH_4^+ 341.1862; $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_3^+$ requires MNH_4^+ , 341.1860); and the oxazolidin-2-one (*rac*)-*syn*,*syn*-**23** (0.18 g, 41%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.47; δ_H (400 MHz, CDCl_3) 7.31–7.23 (3H, m, 3 × CH; Ph), 7.18–7.23 (2H, dt, J 8.1 and 2.1, 2 × CH; Ar), 7.18 (2H, m, 2 × CH; Ph), 7.13–7.11 (2H, dt, J 8.1 and 2.0, 2 × CH; Ar), 5.57 (1H, d, J 7.1, OCH-Ph; oxazolidin-2-one), 4.96 (1H, q, J 6.9, ArCHCH₃), 4.75 (1H, m, CH₃CHN), 2.25 (3H, s, CH₃; Ar), 1.42 (3H, d, J 6.9, ArCHCH₃) and 0.89 (3H, d, J 7.1, CH₃CHN; oxazolidin-2-one); δ_C (100 MHz, CDCl_3) 174.7 (NC=O), 152.8 (OC=O), 137.6 (*i*-C; Ar), 137.2 (*i*-C, Ar), 133.8 (*i*-C; Ph), 129.6² and 128.2² (4 × CH; Ar), 129.1³ and 126.2² (5 × CH; Ph), 79.2 (PhCHO), 55.1 (CH₃CHN), 43.4 (ArCHCH₃), 21.2 (CH₃; Ar), 19.9 (ArCHCH₃) and 14.3 (CH₃CHN; oxazolidin-2-one) (Found MNH_4^+ 341.1858; $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_3^+$ requires MNH_4^+ , 341.1860).

4.26. Synthesis of 4-benzyl-3-[2-(4-methylphenyl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**30** and 4-benzyl-3-[2-(4-methylphenyl)propanoyl]oxazolidin-2-one (*rac*)-*syn*-**30**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-benzyl-oxazolidin-2-one (*rac*)-**12** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**17** (0.49 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**30** (ratio: 69:31 *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 oxazolidin-2-one (*rac*)-*anti*-**30** (83 mg, 19%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.50; ν_{max} (CHCl_3) cm^{-1} 1781 (OC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl_3) 7.27–7.13 (7H, m, 7 × CH; Ar and Ph), 7.04 (2H, dt, J 8.2 and 2.1, 2 × CH; Ar), 5.00 (1H, q, J 7.0, ArCHCH₃), 4.54–4.45 (1H, m, BnCHN), 4.02 (1H, dd, J 9.0 and 2.2, CH_AH_BO), 3.96 (1H, t, J 9.0, CH_AH_BO), 3.27 (1H, dd, J 13.1 and 3.1, CH_AH_BPh), 2.71 (1H, dd, J 13.1 and 9.6, CH_AH_BPh), 2.23 (3H, s, CH₃; Ar) and 1.45 (3H, d, J 7.0, ArCHCH₃); δ_C (100 MHz; CDCl_3) 174.7 (NC=O), 152.8 (OC=O), 137.0 (*i*-C; Ar), 136.8 (*i*-CCH₃; Ar), 135.3 (*i*-C; Ph), 129.3², 128.9² and 127.3¹ (5 × CH; Ph), 129.2² and 127.9² (4 × CH; Ar), 65.8 (CH₂O), 55.7 (BnCHN), 42.6 (ArCHCH₃), 37.8 (CH₂Ph), 21.1 (CH₃; Ar) and 19.4 (ArCHCH₃) (Found MNH_4^+ 341.1860; $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_3^+$ requires MNH_4^+ , 341.1860); and the oxazolidin-2-one (*rac*)-*syn*-**30** (0.185 g, 42%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.29; ν_{max} (CHCl_3) cm^{-1} 1781 (OC=O) and 1700 (NC=O); mp 94–96 °C; δ_H (400 MHz; CDCl_3) 7.23 (2H, dt, J 8.2 and 2.1, 2 × CH; Ar), 7.13–7.09 (3H, m, 3 × CH; Ph), 7.07 (2H, dt, J 8.2 and 2.1, 2 × CH; Ar), 6.89–6.87 (2H, m, 2 × CH; Ph), 4.99 (1H, q, J 7.0, ArCHCH₃), 4.68–4.62 (1H, m, BnCHN), 4.08 (1H, t, J 8.6, CH_AH_BO), 3.97 (1H, dd, J 8.6 and 3.1, CH_AH_BO), 3.01 (1H, dd, J 13.1 and 3.5, CH_AH_BPh), 2.49 (1H, dd, J 13.1 and 8.8, CH_AH_BPh), 2.26 (3H, s, CH₃; Ar) and 1.41 (3H, d, J 7.0, ArCHCH₃); δ_C (100 MHz; CDCl_3) 174.5 (NC=O), 152.8 (OC=O), 137.0 (*i*-C; Ar), 136.8 (*i*-CCH₃; Ar), 134.9 (*i*-C; Ph), 129.4², 128.4² and 127.1¹ (5 × CH; Ph), 129.3² and 128.0² (4 × CH; Ar), 65.6 (CH₂O), 54.8 (BnCHN), 42.7 (ArCHCH₃), 37.2 (CH₂Ph), 21.0 (CH₃; Ar) and 19.0 (ArCHCH₃) (Found MNH_4^+ 341.1863; $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_3^+$ requires MNH_4^+ , 341.1860).

4.27. Synthesis of 4-isopropyl-3-[2-(4-methylphenyl)propanoyl]oxazolidin-2-one (*rac*)-*anti*-**37** and 4-isopropyl-3-[2-(4-methylphenyl)propanoyl]oxazolidin-2-one (*rac*)-*syn*-**37**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-isopropyl-oxazolidin-2-one (*rac*)-**13** (0.17 g, 1.36 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** (0.49 g, 1.50 mmol), gave

the oxazolidin-2-ones *syn*- and *anti*-**37** (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 the oxazolidin-2-one (*rac*)-*anti*-**37** (11 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; ν_{max} (CHCl_3) cm^{-1} 1780 (OC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl_3) 7.16 (2H, dt, J 8.1 and 2.1, 2 × CH; Ar), 7.04 (2H, dt, J 8.1 and 2.1, 2 × CH; Ar), 5.15 (1H, q, J 7.0, ArCHCH₃), 4.29–4.25 (1H, m, *i*-PrCHN), 4.09–4.01 (2H, m, CH₂O), 2.41–2.31 (1H, m, CH(CH₃)₂), 2.24 (3H, s, CH₃; Ar), 1.42 (3H, d, J 7.0, ArCHCH₃), 1.13 (3H, d, J 6.9, CH₃^ACHCH₃^B) and 0.84 (3H, d, J 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl_3) 174.7 (NC=O), 153.3 (OC=O), 137.2 (*i*-C; Ar), 136.8 (*i*-CCH₃; Ar), 129.2² and 127.9² (4 × CH; Ar), 63.0 (CH₂O), 59.0 (*i*-PrCHN), 42.6 (ArCHCH₃), 28.4 (CH(CH₃)₂), 21.9 (CH₃; Ar), 19.1 (ArCHCH₃), 18.0 (CH₃^ACHCH₃^B) and 14.6 (CH₃^ACHCH₃^B) (Found MNH_4^+ 293.1857; $\text{C}_{16}\text{H}_{25}\text{N}_2\text{O}_3^+$ requires MNH_4^+ , 293.1860); and the oxazolidin-2-one (*rac*)-*syn*-**37** (0.21 g, 57%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.50; ν_{max} (CHCl_3) cm^{-1} 1780 (OC=O) and 1700 (NC=O); mp 62–64 °C; δ_H (400 MHz; CDCl_3) 7.17 (2H, dt, J 8.1 and 2.1, 2 × CH; Ar), 7.01 (2H, dt, J 8.1 and 2.1, 2 × CH; Ar), 5.00 (1H, q, J 6.8, ArCHCH₃), 4.42–4.38 (1H, m, *i*-PrCHN), 4.14 (1H, t, J 9.1, CH_AH_BO), 4.01 (1H, dd, J 9.1 and 3.4, CH_AH_BO), 2.21 (3H, s, CH₃; Ar), 2.15–2.05 (1H, m, CH(CH₃)₂), 1.36 (3H, d, J 6.8, ArCHCH₃), 0.71 (3H, d, J 6.9, CH₃^ACHCH₃^B) and 0.39 (3H, d, J 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl_3) 174.5 (NC=O), 153.3 (OC=O), 137.3 (*i*-C; Ar), 136.5 (*i*-CCH₃; Ar), 129.0² and 127.7² (4 × CH; Ar), 62.9 (CH₂O), 57.9 (*i*-PrCHN), 42.7 (ArCHCH₃), 27.7 (CH(CH₃)₂), 20.9 (CH₃; Ar), 18.6 (ArCHCH₃), 18.0 (CH₃^ACHCH₃^B) and 14.6 (CH₃^ACHCH₃^B) (Found MNH_4^+ 293.1858; $\text{C}_{16}\text{H}_{25}\text{N}_2\text{O}_3^+$ requires MNH_4^+ , 293.1860).

4.28. Synthesis of 4-phenyl-3-[2-(4-methylphenyl)propanoyl]oxazolidin-2-one (*rac*)-*anti*,*syn*-**44** and 4-phenyl-3-[2-(4-methylphenyl)propanoyl]oxazolidin-2-one (*rac*)-*syn*,*syn*-**44**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-phenyl-oxazolidin-2-one (*rac*)-**8** (0.22 g, 1.36 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** (0.49 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**44** (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 oxazolidin-2-one (*rac*)-*anti*-**44** (13 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.47; mp 124–127 °C; ν_{max} (CHCl_3) cm^{-1} 1781 (OC=O) and 1705 (NC=O); δ_H (400 MHz, CDCl_3) 7.35–7.23 (5H, m, 5 × CH; Ph), 7.17 (2H, dt, J 8.1 and 2.1, 2 × CH; Ar), 6.95 (2H, dt, J 8.1 and 2.1, 2 × CH; Ar), 5.25 (1H, dd, J 8.6 and 3.1, PhCHN), 5.01 (1H, q, J 7.1, ArCHCH₃), 4.48 (1H, t, J 8.6, CH_AH_BO), 4.14 (1H, dd, J 8.6 and 3.1, CH_AH_BO), 2.25 (3H, s, CH₃; Ar) and 1.32 (3H, d, J 7.1, ArCHCH₃); δ_C (100 MHz, CDCl_3) 173.7 (NC=O), 155.0 (OC=O), 138.2 (*i*-C; Ar), 136.8 (*i*-CCH₃; Ar), 136.6 (*i*-C; Ph), 129.2² and 127.9² (4 × CH; Ar), 128.7², 128.4¹, and 125.8² (5 × CH; Ph), 69.4 (CH₂O), 57.7 (PhCHN), 43.3 (ArCHCH₃), 21.0 (CH₃; Ar) and 18.6 (ArCHCH₃) (Found MNH_4^+ 327.1710; $\text{C}_{19}\text{H}_{23}\text{N}_2\text{O}_3^+$ requires MNH_4^+ , 327.1700); and the oxazolidin-2-one (*rac*)-*syn*-**44** (0.25 g, 59%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.29; mp 120–123 °C; ν_{max} (CHCl_3) cm^{-1} 1780 (OC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl_3) 7.21–7.12 (3H, m, 3 × CH; Ph), 6.96 (2H, dt, J 8.2 and 2.1, 2 × CH; Ar), 6.90 (2H, dt, J 8.2 and 2.1, 2 × CH; Ar), 6.86 (2H, m, 2 × CH; Ph), 5.36 (1H, dd, J 9.1 and 5.1, PhCHN), 5.01 (1H, q, J 6.9, ArCHCH₃), 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, CH₃; Ar) and 1.32 (3H, d, J 6.9, ArCHCH₃); δ_C (100 MHz, CDCl_3) 173.5 (NC=O), 154.9 (OC=O), 138.4 (*i*-CCH₃; Ar), 136.8 (*i*-C; Ar), 136.4 (*i*-C; Ph), 129.1²

and 127.6² (4 × CH; Ar), 128.6², 128.4¹ and 125.7² (5 × CH; Ph), 69.6 (CH₂O), 57.8 (PhCHN), 43.2 (ArCHCH₃), 21.0 (CH₃; Ar) and 18.7 (ArCHCH₃) (Found MNH₄⁺ 327.1700; C₁₉H₂₃N₂O₃⁺ requires MNH₄⁺, 327.1700).

4.29. Synthesis of ethyl 2-oxa-3-[2-(4-methylphenyl)propanoyl]oxazolidin-4-carboxylate (*rac*)-*anti*-49 and ethyl 2-oxa-3-[2-(4-methylphenyl)propanoyl]oxazolidin-4-carboxylate (*rac*)-*syn*-49

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**14** (0.21 g, 1.36 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** (0.49 g, 1.50 mmol), gave the oxazolidin-2-ones *syn*- and *anti*-**49** (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 oxazolidin-2-one (*rac*)-*anti*-**49** (12 mg, 3%) as a colourless oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.40; *v*_{max} (CHCl₃) cm⁻¹ 1794 (OC=O), 1747 (CC=O) and 1700 (NC=O); *δ*_H (400 MHz; CDCl₃) 7.18 (2H, dt, *J* 8.1 and 2.1, 2 × CH; Ar), 7.06 (2H, dt, *J* 8.1 and 2.1, 2 × CH; Ar), 5.01 (1H, q, *J* 7.0, ArCHCH₃), 4.70 (1H, dd, *J* 9.1 and 3.5, EtO₂CCHN), 4.34 (1H, t, *J* 9.1, CH_AH_BO), 4.26–4.17 (3H, m, CH_AH_BO and OCH₂CH₃), 2.26 (3H, s, CH₃; Ar), 1.42 (3H, d, *J* 7.0, ArCHCH₃) and 1.24 (3H, t, *J* 7.2, OCH₂CH₃); *δ*_C (100 MHz; CDCl₃) 174.5 (NC=O), 168.5 (CC=O), 151.0 (OC=O), 137.0 (*i*-C; Ar), 136.8 (*i*-CCH₃; Ar), 129.3² and 128.0² (2 × CH; Ar), 65.8 (CH₂O; oxazolidin-2-one), 62.6 (OCH₂CH₃), 55.8 (EtO₂CCHN), 42.4 (ArCHCH₃), 21.0 (CH₃; Ar), 19.1 (ArCHCH₃) and 14.0 (OCH₂CH₃) (Found MNH₄⁺ 323.1596; C₁₆H₂₃N₂O₅⁺ requires MNH₄⁺, 323.1601); and the oxazolidin-2-one (*rac*)-*syn*-**49** (0.25 g, 60%) as a colourless oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.20; *v*_{max} (CHCl₃) cm⁻¹ 1794 (OC=O), 1746 (CC=O) and 1700 (NC=O); *δ*_H (400 MHz; CDCl₃) 7.16 (2H, dt, *J* 8.2 and 2.1, 2 × CH; Ar), 7.03 (2H, dt, *J* 8.2 and 2.1, 2 × CH; Ar), 4.90 (1H, q, *J* 7.0, ArCHCH₃), 4.84 (1H, dd, *J* 9.5 and 4.7, EtO₂CCHN), 4.41 (1H, t, *J* 9.5, CH_AH_BO), 4.13 (1H, dd, *J* 9.5 and 4.7, CH_AH_BO), 4.05 (2H, q, *J* 7.2, OCH₂CH₃), 2.23 (3H, CH₃; Ar), 1.38 (3H, d, *J* 7.0, ArCHCH₃) and 1.06 (3H, t, *J* 7.2, CH₃CH₂O); *δ*_C (100 MHz; CDCl₃) 174.1 (NC=O), 167.9 (CC=O), 151.7 (OC=O), 136.6 (*i*-CCH₃; Ar), 136.6 (*i*-C; Ar), 129.0² and 127.9² (2 × CH; Ar), 64.0 (CH₂O; oxazolidin-2-one), 62.1 (CH₃CH₂O), 55.5 (EtO₂CCHN), 42.6 (ArCHCH₃), 20.9 (CH₃; Ar), 19.1 (ArCHCH₃) and 13.6 (CH₃CH₂O) (Found MNH₄⁺ 323.1607; C₁₆H₂₃N₂O₅⁺ requires MNH₄⁺, 323.1601).

4.30. Synthesis of 3-[2-(4-isobutylphenyl)propanoyl]-4-methyl-5-phenyl-oxazolidin-2-one (*rac*)-*anti*,*syn*-**24** and 3-[2-(4-isobutylphenyl)propanoyl]-4-methyl-5-phenyl-oxazolidin-2-one (*rac*)-*syn*,*syn*-**24**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-(4*R*S,5*R*)-**11** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.55 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **24** (ratio: 75:25 *syn*,*syn*–*anti*,*syn*). The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*,*syn*-**24** (93 mg, 19%) as a colourless oil; *R*_F [light petroleum ether (40–60 °C)/diethyl ether (1:1)] 0.77; *v*_{max} (CHCl₃) cm⁻¹ 1776 (OC=O) and 1692 (NC=O); *δ*_H (400 MHz; CDCl₃) 7.42–7.24 (7H, m, 7 × CH; Ph and Ar), 7.10 (2H, br d, *J* 8.2, 2 × CH; Ar), 5.48 (1H, d, *J* 7.2, PhCHO), 5.12 (1H, q, *J* 7.0, ArCHCH₃), 4.67 (1H, m, CH₃CHN), 2.44 (2H, d, *J* 7.2, CH₂Ar), 1.90–1.80 (1H, m, CH(CH₃)₂), 1.47 (3H, d, *J* 7.0, ArCHCH₃), 0.91 (3H, d, *J* 6.9, CH₃;

CH₃CHN) and 0.89 (6H, d, *J* 6.9, (CH₃)₂CH); *δ*_C (100.6 MHz; CDCl₃) 174.7 (NC=O), 152.6 (OC=O), 140.7 (*i*-C; Ar), 137.7 (*i*-C; Ar), 133.3 (*i*-C; Ph), 129.4² and 127.8² (4 × CH; Ar), 128.7³ and 125.6² (5 × CH; Ph), 78.7 (PhCHO), 55.4 (*i*-PrCHN), 45.1 (CH(CH₃)₂), 42.9 (ArCHCH₃), 30.2 (CH₂Ar), 22.5 (2 × CH₃^ACHCH₃^B; *i*-BuC₆H₄-), 19.3 (ArCHCH₃) and 14.5 (CH₃CHN) (Found M⁺, 365.1988; C₂₃H₂₇NO₃ requires M⁺, 365.1985); and the oxazolidin-2-one (*rac*)-*syn*,*syn*-**24** (0.26 g, 52%) as a colourless oil; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; *v*_{max} (CHCl₃) cm⁻¹ 1770 (OC=O) and 1699 (NC=O); *δ*_H (400 MHz; CDCl₃) 7.38–7.16 (7H, m, 7 × CH; Ph and Ar), 7.08 (2H, dt, *J* 8.2 and 2.1, 2 × CH; Ar), 5.63 (1H, d, *J* 7.4, PhCHO), 5.05 (1H, q, *J* 7.1, ArCHCH₃), 4.81 (1H, m, *i*-PrCHN), 2.43 (2H, d, *J* 7.2, CH₂Ar), 1.89–1.79 (1H, m, CH(CH₃)₂), 1.48 (3H, d, *J* 7.1, ArCHCH₃), 0.88 (3H, d, *J* 6.7, CH₃^ACHCH₃^B), 0.87 (3H, d, *J* 6.7, CH₃^ACHCH₃^B) and 0.72 (3H, d, *J* 6.7, CH₃CHN); *δ*_C (100.6 MHz; CDCl₃) 174.6 (NC=O), 152.6 (OC=O), 140.5 (*i*-C; Ar), 137.5 (*i*-C; Ar), 133.6 (*i*-C; Ph), 129.3² and 127.7² (4 × CH; Ar), 128.8¹, 128.7² and 125.8² (5 × CH; Ph), 78.8 (PhCHO), 54.7 (CH₃CHN), 45.1 (CH(CH₃)₂), 42.2 (ArCHCH₃), 30.1 (ArCH₂), 22.5² (2C; CH₃^ACHCH₃^B; *i*-BuC₆H₄-), 19.4 (ArCHCH₃) and 14.2 (CH₃CHN) (Found M⁺, 365.1986; C₂₃H₂₇NO₃ requires M⁺, 365.1985).

4.31. Synthesis of 4-benzyl-3-[2-(4-isobutylphenyl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**31** and 4-benzyl-3-[2-(4-isobutylphenyl)propanoyl]-oxazolidin-2-one (*rac*)-*syn*-**31**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.60 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**12** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.55 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **31** (ratio: 79:21 *syn*–*anti*). The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**31** (78 mg, 15%) as a white crystalline solid; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.68; mp 79–80 °C; *v*_{max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1696 (NC=O); *δ*_H (400 MHz; CDCl₃) 7.36–7.18 (7H, m, 7 × CH; Ar and Ph), 7.08 (2H, dt, *J* 8.2 and 2.1, 2 × CH; Ar), 5.11 (1H, q, *J* 6.9, ArCHCH₃), 4.62–4.54 (1H, m, BnCHN), 4.12–4.00 (2H, m, CH₂O), 3.34 (1H, dd, *J* 13.2 and 3.3, CH_AH_BPh), 2.79 (1H, dd, *J* 13.2 and 9.8, CH_AH_BPh), 2.43 (2H, d, *J* 7.2, ArCH₂), 1.89–1.79 (1H, m, CH(CH₃)₂), 1.54 (3H, d, *J* 6.9, ArCHCH₃) and 1.54 (6H, d, *J* ~6.7, 2 × CH₃, CH(CH₃)₂); *δ*_C (100.6 MHz; CDCl₃) 174.9 (NC=O), 153.0 (OC=O), 140.7 (*i*-C; Ar), 137.5 (*i*-C; Ar), 135.4 (*i*-C; Ph), 129.5², 129.0² and 127.4¹ (5 × CH; Ph), 129.3² and 127.9² (4 × CH; Ar); 65.9 (CH₂O), 55.9 (BnCHN), 45.1 (CH(CH₃)₂), 42.7 (ArCHCH₃), 38.0 (PhCH₂), 30.2 (ArCH₂), 22.4 (2C; CH(CH₃)₂) and 19.5 (ArCHCH₃) (Found MH⁺, 366.2061; C₂₃H₂₈NO₃ requires MH⁺, 366.2164); and the oxazolidin-2-one (*rac*)-*syn*-**31** (0.30 g, 60%) as a white crystalline solid; *R*_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.37; mp 93–95 °C; *v*_{max} (CHCl₃) cm⁻¹ 1768 (OC=O) and 1698 (NC=O); *δ*_H (400 MHz; CDCl₃) 7.23 (2H, br d, *J* 8.2, 2 × CH; Ar), 7.24–6.93 (7H, m, 7 × CH; Ph and Ar), 5.10 (1H, q, *J* 6.9, ArCHCH₃), 4.79–4.70 (1H, m, BnCHN), 4.17 (1H, t, *J* 8.9, CH_AH_BO), 4.06 (1H, dd, *J* 8.9 and 3.2, CH_AH_BO), 3.06 (1H, dd, *J* 13.4 and 3.2, CH_AH_BPh), 2.59 (1H, dd, *J* 13.4 and 8.7, CH_AH_BPh), 2.46 (2H, d, *J* 7.2, ArCH₂), 1.91–1.82 (1H, m, CH(CH₃)₂), 1.51 (3H, d, *J* 6.9, ArCHCH₃), 0.91 (3H, d, *J* 6.7, CH₃^ACHCH₃^B) and 0.90 (3H, d, *J* 6.7, CH₃^ACHCH₃^B); *δ*_C (100.6 MHz; CDCl₃) 174.7 (NC=O), 153.0 (OC=O), 140.7 (*i*-C; Ar), 137.4 (*i*-C; Ar), 135.0 (*i*-C; Ph), 129.5², 128.0² and 127.2¹ (5 × CH; Ph), 129.4² and 127.9² (4 × CH; Ar), 65.7 (CH₂O), 54.9 (BnCHN), 45.1 (CH(CH₃)₂), 42.8 (ArCHCH₃), 37.4 (PhCH₂), 30.2 (ArCH₂), 22.5 (CH₃^ACHCH₃^B), 22.4 (CH₃^ACHCH₃^B) and 19.2 (ArCHCH₃) (Found MH⁺, 366.2065; C₂₃H₂₈NO₃ requires MH⁺, 366.2064).

4.32. Synthesis of 3-[2-(4-isobutylphenyl)propanoyl]-4-isopropyl-oxazolidin-2-one (*rac*)-*anti*-38 and 3-[2-(4-isobutylphenyl)propanoyl]-4-isopropyl-oxazolidin-2-one (*rac*)-*syn*-38

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.60 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**13** (0.17 g, 1.36 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.55 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **38** (ratio: 96:4 *syn*:-*anti*-). The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**38** (16 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.77; ν_{\max} (CHCl₃) cm⁻¹ 1776 (OC=O) and 1692 (NC=O); δ_H (400 MHz; CDCl₃) 7.23 (2H, br d, *J* 8.2, 2 × CH; Ar), 7.07 (2H, br d, *J* 8.2, 2 × CH; Ar), 5.11 (1H, q, *J* 7.2, ArCHCH₃), 4.37–4.32 (1H, m, *i*-PrCHN), 4.15–4.07 (2H, m, CH₂O), 2.46–2.39 (2H, m, CH₂Ar), 1.87–1.77 (1H, m, CH(CH₃)₂), 1.49 (3H, d, *J* 7.2, ArCHCH₃), 0.91 (3H, d, *J* 6.9, CH₃; CH(CH₃)₂), 0.90 (3H, d, *J* 6.9, CH₃; CH(CH₃)₂) and 0.88 (6H, d, *J* 6.9, 2 × CH₃; CH(CH₃)₂); δ_C (100.6 MHz; CDCl₃) 174.9 (NC=O), 153.6 (OC=O), 140.6 (*i*-C; Ar), 137.5 (*i*-C; Ar), 129.3² and 127.8² (4 × CH; Ar), 63.1 (CH₂O), 59.0 (*i*-PrCHN), 45.1 (CH(CH₃)₂), 42.6 (ArCHCH₃), 30.2 (CH₂Ar), 28.6 (CH(CH₃)₂), 22.7 (CH₃CHCH₃^B; *i*-BuC₆H₄-), 22.4 (CH₃CHCH₃^B; *i*-BuC₆H₄-), 19.7 (CH₃CHCH₃^B; oxazolidin-2-one), 18.0 (CH₃CHCH₃^B; oxazolidin-2-one) and 14.7 (ArCHCH₃) (Found MH⁺, 318.20062; C₁₉H₂₈NO₃ requires MH⁺, 318.2064); and the oxazolidin-2-one (*rac*)-*syn*-**38** (0.24 g, 56%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1699 (NC=O); δ_H (400 MHz; CDCl₃) 7.23 (2H, br d, *J* 8.2, 2 × CH; Ar), 7.03 (2H, br d, *J* 8.2, 2 × CH; Ar), 5.11 (1H, q, *J* 6.9, ArCHCH₃), 4.50–4.44 (1H, m, *i*-PrCHN), 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, CH₂Ar), 2.19–2.07 (1H, m, CH(CH₃)₂; oxazolidin-2-one), 1.87–1.75 (1H, m, CH(CH₃)₂; ArCHCH₃), 1.44 (3H, d, *J* 6.9, ArCHCH₃), 0.85 (6H, d, *J* ~6.7, 2 × CH₃; CH(CH₃)₂), 0.76 (3H, d, *J* 6.9, CH₃CHCH₃^B) and 0.38 (3H, d, *J* 6.9, CH₃CHCH₃^B); δ_C (100.6 MHz; CDCl₃) 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 (CH₂O), 58.0 (*i*-PrCHN), 45.0 (CH(CH₃)₂), 42.9 (ArCHCH₃), 30.2 (CH₂Ar), 27.8 (CH(CH₃)₂; oxazolidin-2-one), 22.7 (CH₃CHCH₃^B; *i*-BuC₆H₄-), 22.3 (CH₃CHCH₃^B; *i*-BuC₆H₄-), 18.5 (CH₃CHCH₃^B; oxazolidin-2-one), 17.7 (CH₃CHCH₃^B; oxazolidin-2-one) and 14.0 (ArCHCH₃) (Found M⁺, 317.1979; C₂₉H₂₇NO₃ requires M⁺, 317.1985).

4.33. Synthesis of 3-[2-(4-isobutylphenyl)propanoyl]-4-phenyl-oxazolidin-2-one (*rac*)-*anti*-10 and 3-[2-(4-isobutylphenyl)propanoyl]-4-phenyl-oxazolidin-2-one (*rac*)-*syn*-10

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**8** (0.22 g, 1.36 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.55 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **10** (ratio: 96:4 *syn*:-*anti*-). The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether, (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**10** (14 mg, 3%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.62; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1701 (NC=O); mp 150–154 °C; δ_H (400 MHz; CDCl₃) 7.39–7.23 (7H, m, 7 × CH; Ar and Ph), 7.07 (2H, dt, *J* 8.2 and 2.1, 2 × CH; Ar), 5.33 (1H, dd, *J* 8.4 and 3.2, PhCHN), 5.10 (1H, q, *J* 7.1, ArCHCH₃), 4.55 (1H, t, *J* 8.9, CH_AH_BO), 4.20 (1H, dd, *J* 8.9 and 3.2, CH_AH_BO), 2.42 (2H, d, *J* 7.2, ArCH₂), 1.88–1.78 (1H, m, CH(CH₃)₂), 1.39 (3H, d, *J* 7.1, ArCHCH₃), 0.89 (3H, d, *J* 6.7, CH₃CHCH₃^B) and 0.88 (3H, d, *J* 6.7, CH₃CHCH₃^B); δ_C

(100.6 MHz; CDCl₃) 173.9 (NC=O), 153.2 (OC=O), 140.6 (*i*-C; Ar), 138.3 (*i*-C; Ar), 137.0 (*i*-C; Ph), 129.3² and 128.0² (4 × CH; Ar), 128.8², 128.5¹ and 125.8² (5 × CH; Ph), 69.6 (CH₂O), 57.8 (PhCHN), 45.1 (CH(CH₃)₂), 43.3 (ArCHCH₃), 30.2 (ArCH₂), 22.4 (2C, CH(CH₃)₂) and 18.5 (ArCHCH₃) (Found MH⁺, 352.1913; C₂₂H₂₆NO₃ requires MH⁺, 352.1907); and the oxazolidin-2-one (*rac*)-*syn*-**10** (0.29 g, 60%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.41; ν_{\max} (CHCl₃) cm⁻¹ 1779 (OC=O) and 1705 (NC=O); mp 67–71 °C; δ_H (400 MHz; CDCl₃) 7.28–7.15 (3H, m, 3 × CH; Ph and/or Ar), 7.03–7.00 (4H, m, 4 × CH; Ph and Ar), 6.90 (2H, dt, *J* 7.9 and 2.1, 2 × CH; Ar), 5.44 (1H, dd, *J* 9.2 and 5.2, PhCHN), 5.09 (1H, q, *J* 6.9; ArCHCH₃), 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, ArCH₂), 1.89–1.79 (1H, m, CH(CH₃)₂), 1.38 (3H, d, *J* 6.9, ArCHCH₃), 0.91 (3H, d, *J* 6.7, CH₃CHCH₃^B) and 0.91 (3H, d, *J* 6.7, CH₃CHCH₃^B); δ_C (100.6 MHz; CDCl₃) 174.3 (NC=O), 153.3 (OC=O), 140.7 (*i*-C; Ar), 139.4 (*i*-C; Ar), 137.4 (*i*-C; Ph), 129.3² and 127.0² (4 × CH; Ar), 129.2², 128.7¹ and 125.8² (5 × CH; Ph), 69.7 (CH₂O), 58.1 (PhCHN), 45.1 (CH(CH₃)₂), 42.7 (ArCHCH₃), 30.2 (ArCH₂), 22.4 (2C, CH(CH₃)₂) and 19.4 (ArCHCH₃) (Found MH⁺, 352.1909; C₂₂H₂₆NO₃ requires MH⁺, 352.1907).

4.34. Synthesis of ethyl 3-[2-(4-isobutylphenyl)propanoyl] oxazolidin-2-one 4-carboxylate (*rac*)-*anti*-50 and ethyl 3-[2-(4-isobutylphenyl)propanoyl] oxazolidin-2-one 4-carboxylate (*rac*)-*syn*-50

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**14** (0.21 g, 1.36 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.55 g, 1.36 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **50** (ratio: 94:6 *syn*:-*anti*-). The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**50** (19 mg, 4%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.53; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1751 (CC=O) and 1701 (NC=O); δ_H (400 MHz; CDCl₃) 7.23 (2H, dt, *J* 8.0 and 2.1, 2 × CH; Ar), 7.10 (2H, dt, *J* 8.0 and 2.1, 2 × CH; Ar), 5.09 (1H, q, *J* 6.9, ArCHCH₃), 4.78 (1H, dd, *J* 9.4 and 3.7, EtO₂CCHN), 4.41 (1H, t, *J* 9.0, CH_AH_BO), 4.33–4.23 (3H, m, 3 × CH, CH_AH_BO and CH₂CH₃), 2.42 (2H, d, *J* 7.2, ArCH₂), 1.87–1.77 (1H, m, CH(CH₃)₂), 1.49 (3H, d, *J* 6.9, ArCHCH₃), 1.30 (3H, t, *J* 7.2, CH₂CH₃) and 0.88 (6H, d, *J* ~6.7, CH(CH₃)₂); δ_C (100.6 MHz; CDCl₃) 174.7 (NC=O), 168.6 (CC=O), 152.0 (OC=O), 140.8 (*i*-C; Ar), 137.1 (*i*-C; Ar), 129.4² and 127.9² (4 × CH; Ar), 64.2 (CH₂O), 62.5 (CH₂O; ester), 55.9 (EtO₂CCHN), 45.1 (CH(CH₃)₂), 42.5 (ArCHCH₃), 30.2 (ArCH₂), 22.4 (CH(CH₃)₂), 19.2 (ArCHCH₃) and 14.0 (CH₂CH₃) (Found MNH₄⁺, 365.2069; C₁₉H₂₉N₂O₅ requires MNH₄⁺, 365.2171); and the oxazolidin-2-one (*rac*)-*syn*-**50** (0.275 g, 58%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.35; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1747 (CC=O) and 1699 (NC=O); δ_H (400 MHz; CDCl₃) 7.23 (2H, dt, *J* 8.1 and 2.1, 2 × CH; Ar), 7.10 (2H, dt, *J* 8.1 and 2.1, 2 × CH; Ar), 5.01 (1H, q, *J* 6.9, ArCHCH₃), 4.93 (1H, dd, *J* 9.4 and 4.7, EtO₂CCHN), 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, CH₂CH₃), 2.42 (2H, d, *J* 7.2, ArCH₂), 1.87–1.77 (1H, m, CH(CH₃)₂), 1.46 (3H, d, *J* 6.9, ArCHCH₃), 1.10 (3H, t, *J* 7.2, CH₂CH₃) and 0.88 (6H, d, *J* 6.7, 2 × CH₃; CH(CH₃)₂); δ_C (100.6 MHz; CDCl₃) 174.5 (NC=O), 168.0 (OC=O), 152.0 (*i*-C; Ar), 140.5 (*i*-C; Ar), 136.9 (*i*-C; Ph), 129.2² and 127.9² (4 × CH; Ar), 64.2 (CH₂O), 62.3 (CH₂O; ester), 55.7 (EtO₂CCHN), 45.1 (CH(CH₃)₂), 42.7 (ArCHCH₃), 30.1 (ArCH₂), 22.4 (CH(CH₃)₂), 19.3 (ArCHCH₃) and 13.8 (CH₂CH₃) (Found MNH₄⁺, 365.2073; C₁₉H₂₉N₂O₅ requires MNH₄⁺, 365.2071).

4.35. Synthesis of 4-methyl-5-phenyl 3-[(4-chlorophenyl)propanoyl]-oxazolidin-2-one (*rac*)-(2*RS*,4*RS*,5*SR*)-*anti*,*syn*-25 and 3-[(4-chlorophenyl)propanoyl]-4-methyl-5-phenyl-oxazolidin-2-one (*rac*)-(2*SR*,4*RS*,5*SR*)-*syn*,*syn*-25

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (4*RS*,5*SR*)-(*rac*)-**11** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.53 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **25** (ratio: 70:30 *syn*,*syn*-:*anti*,*syn*-). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*,*syn*-**25** (88 mg, 19%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)] 0.58; mp 89–91 °C; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1702 (CC=O); δ_H (400 MHz, CDCl₃) 7.36–7.28 (3H, m, 3 × CH; Ph), 7.25–7.23 (4H, ABq, *J* 3.4, 4 × CH; Ar), 7.22–7.19 (2H, m, 2 × CH; Ph), 5.42 (1H, d, *J* 7.1, OCHPh), 5.04 (1H, q, *J* 7.0, ArCHCH₃) 4.60 (1H, m (appears as a quintet, *J* 6.6), CH₃CHN), 1.42 (3H, d, *J* 7.0, ArCHCH₃) and 0.86 (3H, d, *J* 6.6, CH₃CHN); δ_C (100 MHz, CDCl₃) 173.9 (NC=O), 152.5 (OC=O), 138.8 (*i*-CC; Ar), 133.1 (*i*-CCl; Ar), 133.0 (*i*-C; Ph), 129.5² and 128.8² (4 × CH; Ar), 128.8,¹ 128.7² and 125.6² (5 × CH; Ph), 78.7 (OCHPh), 55.4 (CH₃CHN), 42.7 (ArCHCH₃), 19.2 (ArCHCH₃) and 14.5 (CH₃CHN) (Found MNH₄(³⁵Cl)⁺, 361.1307. C₁₉H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 361.1313); and the oxazolidin-2-one (*rac*)-*syn*,*syn*-**25** (0.205 g, 44%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.23; mp 65–67 °C ν_{\max} (CHCl₃) cm⁻¹ 1779 (OC=O) and 1699 (NC=O); δ_H (400 MHz, CDCl₃) 7.32–7.27 (3H, m, 3 × CH; Ph), 7.24–7.21 (4H, ABq, *J* 1.3, 4 × CH, Ar), 7.15–7.11 (2H, m, 2 × CH, Ph), 5.60 (1H, d, *J* 7.3, OCHPh) 4.97 (1H, q, *J* 7.0, ArCHCH₃), 4.66 (1H, dq, *J* 7.3 and 6.6, CH₃CHN), 1.41 (3H, d, *J* 7.0, ArCHCH₃) and 0.91 (3H, d, *J* 6.6, CH₃CHN); δ_C (100 MHz, CDCl₃) 173.8 (NC=O), 152.4 (C=O), 138.6 (*i*-CC; Ar), 133.2 (*i*-C; Ph), 132.9 (*i*-CCl; Ar), 129.4² and 128.7² (2 × CH; Ar), 128.8,¹ 128.6² and 125.6² (5 × CH; Ph), 78.8 (OCHPh), 54.7 (CH₃CHN), 43.0 (ArCHCH₃), 19.3 (ArCHCH₃) and 14.1 (CH₃CHN) (Found MNH₄(³⁵Cl)⁺, 361.1311. C₁₉H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 361.1313).

4.36. Synthesis of 4-benzyl-3-[(4-chlorophenyl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**32** and 4-benzyl-3-[(4-chlorophenyl)propanoyl]-oxazolidin-2-one (*rac*)-*syn*-**32**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**12** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.53 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **32** (ratio: 69:31 *syn*-:*anti*-). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**32** (88 mg, 19%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)] 0.61; mp 70–73 °C; ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1698 (NC=O); δ_H (400 MHz, CDCl₃) 7.35–7.25 (7H, m, 7 × CH; 2 × Ph), 7.20 (2H, dd, *J* 8.2 and 1.5, 2 × CH; Ph), 5.07 (1H, q, *J* 7.0, ArCHCH₃), 4.64–4.56 (1H, m, BnCHN), 4.11 (1H, dd (ABq), *J* 8.7 and 2.6, CH_AH_BO), 4.07 (1H, t, *J* 8.7, CH_AH_BO), 3.32 (1H, dd, *J* 13.2 and 3.3, CH_AH_BPh), 2.78 (1H, dd, *J* 13.2 and 9.5, CH_AH_BPh) and 1.50 (3H, d, *J* 7.0, ArCHCH₃); δ_C (100 MHz, CDCl₃) 174.2 (NC=O), 152.8 (OC=O), 138.6 (*i*-CC; Ar), 135.2 (*i*-C; Ph), 133.1 (*i*-CCl; Ar), 129.5² and 128.9² (2 × CH; Ar), 129.4,² 128.7² and 127.4¹ (5 × CH; Ph), 65.9 (CH₂O), 55.7 (BnCHN), 42.5 (ArCHCH₃), 37.8 (PhCH₂) and 19.4 (ArCHCH₃) (Found MNH₄(³⁵Cl)⁺, 361.1315. C₁₉H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 361.1313); and the

oxazolidin-2-one (*rac*)-*syn*-**32** (0.196 g, 42%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)] 0.43; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1708 (NC=O); δ_H (400 MHz, CDCl₃) 7.37 (2H, dt, *J* 8.2 and 2.2, 2 × CH; Ar), 7.32 (2H, dt, *J* 8.2 and 2.2, 2 × CH; Ar), 7.23–7.20 (3H, m, 3 × CH; Ph), 6.98 (2H, dd, *J* 6.2 and 2.1, 2 × CH; Ph), 5.08 (1H, q, *J* 7.0, ArCHCH₃), 4.75–3.99 (1H, m, BnCHN), 4.20 (1H, t, *J* 8.9, CH_AH_BO), 4.09 (1H, dd, *J* 8.9 and 3.2, CH_AH_BO), 3.08 (1H, dd, *J* 13.6 and 3.5, CH_AH_BPh), 2.58 (1H, dd, *J* 13.6 and 9.0, CH_AH_BPh) and 1.50 (3H, d, *J* 7.0, ArCHCH₃); δ_C (100 MHz, CDCl₃) 174.0 (NC=O), 152.8 (OC=O), 138.6 (*i*-CC; Ar), 134.7 (*i*-C; Ph), 133.1 (*i*-CCl; Ar), 129.6² and 128.8² (2 × CH; Ar), 129.3,² 128.7² and 127.5¹ (5 × CH; Ph), 65.9 (CH₂O), 54.9 (BnCHN), 42.5 (ArCHCH₃), 37.3 (PhCH₂) and 19.0 (ArCHCH₃) (Found MNH₄(³⁵Cl)⁺, 361.1310. C₁₉H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 361.1313).

4.37. Synthesis of 4-isopropyl-3-[(4-chlorophenyl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**39** and 4-isopropyl-3-[(4-chlorophenyl)propanoyl]-oxazolidin-2-one (*rac*)-*syn*-**39**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**13** (0.17 g, 1.36 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.53 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones (*rac*)-**13** (ratio: 74:26 *syn*-:*anti*-). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**39** (68 mg, 17%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.56; mp 62–64 °C; ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1702 (NC=O); δ_H (400 MHz; CDCl₃) 7.26 (2H, dt, *J* 8.2 and 2.1, 2 × CH; Ar), 7.15 (2H, dt, *J* 8.2 and 2.1, 2 × CH; Ar), 5.00 (1H, q, *J* 7.1, ArCHCH₃), 4.27–4.24 (1H, m, *i*-PrCHN), 4.00–3.96 (2H, m, CH₂O), 2.40–2.30 (1H, m, CH(CH₃)₂), 1.31 (3H, d, *J* 7.0, ArCHCH₃), 1.15 (3H, d, *J* 6.9, CH₃^ACHCH₃^B) and 0.90 (3H, d, *J* 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl₃) 174.0 (NC=O), 153.4 (OC=O), 138.9 (*i*-CC; Ar), 132.9 (*i*-CCl; Ar), 129.4² and 128.8² (4 × CH; Ar), 62.9 (CH₂O), 58.1 (*i*-PrCHN), 42.6 (ArCHCH₃), 27.9 (CH(CH₃)₂), 18.6 (ArCHCH₃), 17.7 (CH₃^ACHCH₃^B) and 14.1 (CH₃^ACHCH₃^B) (Found MNH₄(³⁵Cl)⁺ 313.1310; C₁₅H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 313.1313); the oxazolidin-2-one (*rac*)-*syn*-**39** (0.185 g, 46%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.32; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl₃) 7.25–7.18 (4H, m; 4 × CH; Ar), 5.00 (1H, q, *J* 6.9, ArCHCH₃), 4.43–4.38 (1H, m, *i*-PrCHN), 4.18 (1H, t, *J* 9.1, CH_AH_BO), 4.05 (1H, dd, *J* 9.1 and 3.3, CH_AH_BO), 2.16–2.06 (1H, m, CH(CH₃)₂), 1.37 (3H, d, *J* 6.9, ArCHCH₃), 0.73 (3H, d, *J* 6.9, CH₃^ACHCH₃^B) and 0.43 (3H, d, *J* 6.9, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl₃) 174.1 (NC=O), 153.4 (OC=O), 138.7 (*i*-C; Ar), 132.7 (*i*-CCl; Ar), 129.3² and 128.7² (4 × CH; Ar), 62.6 (CH₂O), 57.9 (*i*-PrCHN), 42.5 (ArCHCH₃), 27.7 (CH(CH₃)₂), 18.6 (ArCHCH₃), 17.9 (CH₃^ACHCH₃^B) and 14.3 (CH₃^ACHCH₃^B) (Found MNH₄(³⁵Cl)⁺ 313.1311; C₁₅H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 313.1313).

4.38. Synthesis of 4-phenyl-3-[(4-chlorophenyl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**45** and 4-phenyl-3-[(4-chlorophenyl)propanoyl]-oxazolidin-2-one (*rac*)-*syn*-**45**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**8** (0.22 g, 1.36 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.53 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **45** (ratio: 95:5 *syn*-:*anti*-). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)

to give the oxazolidin-2-one (*rac*)-*anti*-**45** (18 mg, 4%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.65; mp 74–76 °C; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.31–7.15 (5H, m, 5 × CH; Ph), 7.14 (2H, dt, *J* 8.2 and 1.2, 2 × CH; Ar), 6.98 (2H, dt, *J* 8.1 and 1.2, 2 × CH; Ar), 5.26 (1H, dd, *J* 8.6 and 3.2, PhCHN), 5.16 (1H, q, *J* 7.1, ArCHCH₃), 4.51 (1H, t, *J* 8.6, CH_AH_BO), 4.16 (1H, dd, *J* 8.6 and 3.1, CH_AH_BO) and 1.56 (3H, d, *J* 7.1, ArCHCH₃); δ_C (100 MHz, CDCl₃) 173.9 (NC=O), 152.9 (OC=O), 138.1 (*i*-C; Ar), 133.0 (*i*-C; Ph), 130.9 (*i*-CCl; Ar), 129.5², 128.9², 128.6³ and 125.8² (9 × CH; Ar and Ph) (Found MNH₄(³⁵Cl)⁺ 347.1154; C₁₈H₂₀ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 347.1157); the oxazolidin-2-one (*rac*)-*syn*-**45** (0.26 g, 58%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.35; mp 116–117 °C; ν_{\max} (CHCl₃) cm⁻¹ 1782 (OC=O) and 1700 (NC=O); δ_H (400 MHz, CDCl₃) 7.33–7.23 (3H, m, 3 × CH; Ph), 7.19 (2H, dt, *J* 8.4 and 2.0, 2 × CH; Ar), 7.02 (2H, dt, *J* 8.4 and 2.0, 2 × CH; Ar), 6.96 (2H, d, *J* 6.8 and 1.7, 2 × CH; Ph), 5.45 (1H, dd, *J* 9.0 and 4.8, PhCHN), 4.99 (1H, q, *J* 7.0, ArCHCH₃), 4.58 (1H, t, *J* 9.0, CH_AH_BO), 4.50 (1H, dd, *J* 9.0 and 4.8, CH_AH_BO) and 1.30 (3H, d, *J* 7.0, ArCHCH₃); δ_C (100 MHz, CDCl₃) 173.8 (NC=O), 152.8 (OC=O), 138.2 (*i*-CC; Ar), 133.2 (*i*-C; Ph), 132.8 (*i*-CCl; Ar), 129.7², 128.8², 128.6¹, 128.6² and 125.6² (9 × CH; Ph and Ar), 69.4 (CH₂O), 57.9 (PhCHN), 43.8 (ArCHCH₃) and 18.9 (ArCHCH₃) (Found MNH₄(³⁵Cl)⁺ 347.1154; C₁₈H₂₀ClN₂O₃ requires MNH₄(³⁵Cl)⁺, 347.1157).

4.39. Synthesis of ethyl 2-oxa-3-[(4-chlorophenyl)propanoyl]-oxazolidin-4-carboxylate (*rac*)-*anti*-**51** and ethyl 2-oxa-3-[(4-chlorophenyl)propanoyl]-oxazolidin-4-carboxylate (*rac*)-*syn*-**51**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**14** (0.21 g, 1.36 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.53 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **51** (ratio 95:5 *syn*:*anti*). The crude residue was purified by flash column chromatography on a silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**51** (13 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.31; ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1748 (CC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl₃) 7.30–7.28 (4H, m, 4 × CH; Ar), 5.01 (1H, q, *J* 6.8, ArCHCH₃), 4.72 (1H, dd, *J* 9.3 and 3.6, EtO₂CCHN), 4.38 (1H, t, *J* 9.3, CH_AH_BO), 4.24–4.19 (3H, m, CH_AH_BO and OCH₂CH₃), 1.43 (3H, d, *J* 6.8, ArCHCH₃) and 1.23 (3H, t, *J* 7.1, OCH₂CH₃); δ_C (100 MHz; CDCl₃) 173.7 (NC=O), 167.8 (EtOC=O), 151.8 (OC=O), 138.1 (*i*-CC; Ar), 133.0 (*i*-CCl; Ar), 129.3² and 128.0² (4 × CH; Ar), 65.8 (CH₂O; oxazolidin-2-one), 64.2 (OCH₂CH₃), 55.5 (EtO₂CCHN), 42.6 (ArCHCH₃), 19.1 (ArCHCH₃) and 13.8 (OCH₂CH₃) (Found MNH₄(³⁵Cl)⁺, 343.1059; C₁₅H₂₀ClN₂O₅ requires MNH₄(³⁵Cl)⁺, 343.1055); and the oxazolidin-2-one (*rac*)-*syn*-**51** (0.239 g, 54%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.20; ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1745 (CC=O) and 1700 (NC=O); δ_H (400 MHz; CDCl₃) 7.30–7.27 (4H, s, 4 × CH; Ar), 4.93 (1H, q, *J* 7.2, PhCHCH₃), 4.87 (1H, dd, *J* 9.6 and 4.8, EtO₂CCHN), 4.47 (1H, t, *J* 9.6, CH_AH_BO), 4.18 (1H, dd, *J* 9.6 and 4.8, CH_AH_BO), 4.07 (1H, dq, *J* 15.0 and 7.2, OCH_AH_BCH₃), 4.05 (1H, dq, *J* 15.0 and 7.2, OCH_AH_BCH₃), 1.39 (3H, d, *J* 7.2, ArCHCH₃) and 1.09 (3H, t, *J* 7.2, OCH₂CH₃); δ_C (100 MHz; CDCl₃) 173.8 (NC=O), 167.8 (EtOC=O), 151.6 (OC=O), 138.3 (*i*-C; Ar), 132.9 (*i*-CCl; Ar), 129.6² and 128.3² (4 × CH; Ar), 65.7 (CH₂O; oxazolidin-2-one), 64.3 (OCH₂CH₃), 55.5 (EtO₂CCHN), 42.5 (ArCHCH₃), 19.3 (ArCHCH₃) and 13.7 (OCH₂CH₃) (Found MNH₄(³⁵Cl)⁺, 343.1057; C₁₅H₂₀ClN₂O₅ requires MNH₄(³⁵Cl)⁺, 343.1055).

4.40. Synthesis of 4-methyl-5-phenyl-3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*,*syn*-**26** and 3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-4-methyl-5-phenyl-oxazolidin-2-one (*rac*)-*syn*,*syn*-**26**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-methyl-5-phenyl-oxazolidin-2-one (4*RS*,5*SR*)-(*rac*)-**11** (0.24 g, 1.36 mmol) and pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** (0.59 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **26** (ratio: 73:27 *syn*,*syn*:*anti*,*syn*). The crude residue was purified by flash column chromatography on silica gel eluting with [light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)] to give the oxazolidin-2-one (*rac*)-*anti*,*syn*-**26** (91 mg, 17%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.65; ν_{\max} (CHCl₃) cm⁻¹ 1776 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl₃) 7.76 (1H, d, *J* 1.8, CH; Ar), 7.72 (2H, dd, *J* 8.7 and 2.5, 2 × CH; Ar), 7.49 (1H, dd, *J* 8.4 and 1.8, CH; Ar), 7.41–7.32 (3H, m, 3 × CH; Ph), 7.27–7.23 (2H, m, 2 × CH; Ph), 7.14 (1H, dd, *J* 8.4 and 1.8, CH; Ar) and 7.11 (1H, br s, CH; Ar), 5.43 (1H, d, *J* 7.1, PhCHO), 5.27 (1H, q, *J* 6.9, ArCHCH₃), 4.71–4.61 (1H, m, CH₃CHN), 3.90 (3H, s, OCH₃), 1.57 (3H, d, *J* 6.9, ArCHCH₃) and 0.94 (3H, *J* 6.4, CH₃CHN); δ_C (100 MHz; CDCl₃) 174.5 (NC=O), 157.5 (*i*-CO), 152.6 (OC=O), 135.6, 133.7, 133.2 and 129.3 (4 × *i*-C; Ar and Ph), 129.0, 127.1, 126.7, 126.6, 118.9 and 105.5 (6 × CH; Ar), 128.8¹, 128.7² and 125.6² (5 × CH; Ph), 78.6 (PhCHO), 55.4 (CH₃CHN), 55.3 (OCH₃), 43.2 (ArCHCH₃), 19.2 (ArCHCH₃) and 14.5 (CH₃CHN) (Found MNH₄⁺, 407.1964; C₂₄H₂₇N₂O₄ requires MNH₄⁺ 407.1965); and oxazolidin-2-one (*rac*)-*syn*,*syn*-**26** (0.25 g, 47%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; ν_{\max} (CHCl₃) cm⁻¹ 1782 (OC=O) and 1701 (NC=O); δ_H (400 MHz; CDCl₃) 7.72 (1H, d, *J* 1.5, CH; Ar), 7.68 (2H, d, *J* 8.6, 2 × CH; Ar), 7.45 (1H, dd, *J* 8.6 and 1.8, CH; Ar), 7.31–7.27 (3H, m, 3 × CH; Ph), 7.16–7.07 (3H, m, 3 × CH; Ar and Ph), 7.05 (1H, br s, CH; Ar), 5.62 (1H, d, *J* 7.4, PhCHO), 5.20 (1H, q, *J* 6.9, ArCHCH₃), 4.88–4.78 (1H, m, CH₃CHN), 3.90 (3H, s, CH₃O), 1.57 (3H, d, *J* 6.9, ArCHCH₃) and 0.71 (3H, d, *J* 6.4, CH₃CHN); δ_C (100 MHz; CDCl₃) 174.3 (NC=O), 157.6 (*i*-CO; Ar), 152.5 (OC=O), 135.4, 133.6, 133.3 and 129.3 (4 × *i*-C; Ar and Ph), 128.9, 127.1, 126.7, 126.7, 118.8 and 105.5 (6 × CH; Ar), 128.7¹, 128.5² and 125.6² (5 × CH; Ph), 78.7 (PhCHO), 55.3 (OCH₃), 54.6 (CH₃CHN), 43.5 (ArCHCH₃), 19.3 (ArCHCH₃) and 14.1 (CH₃CHN) (Found MNH₄⁺, 407.1968; C₂₄H₂₇N₂O₄ requires MNH₄⁺ 407.1965).

4.41. Synthesis of 4-benzyl-3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**33** and 4-benzyl-3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-oxazolidin-2-one (*rac*)-*syn*-**33**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-benzyl-oxazolidin-2-one (*rac*)-**12** (0.24 g, 1.36 mmol) and the pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** (0.59 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones (*rac*)-**33** (ratio: 72:28 *syn*:*anti*). The crude residue was purified by flash column chromatography on silica gel eluting with [light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)] to give the oxazolidin-2-one (*rac*)-*anti*-**33** (0.106 g, 20%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1697 (NC=O); δ_H (400 MHz; CDCl₃) 7.74 (1H, br s, CH; Ar), 7.70 (2H, d, *J* 8.2, 2 × CH; Ar), 7.48 (1H, dd, *J* 8.4 and 1.8, CH; Ar), 7.37–7.21 (5H, m, 5 × CH; Ph), 7.15 (1H, dd, *J* 8.4 and 1.8, CH; Ar), 7.08 (1H, br s, CH; Ar), 5.26 (1H, q, *J* 6.9, ArCHCH₃), 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, OCH₃), 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, ArCHCH₃);

δ_C (100 MHz; $CDCl_3$) 174.7 (NC=O), 157.6 (*i*-CO; Ar), 152.9 (OC=O; Ar), 135.3, 135.4, 133.8 and 129.8 ($4 \times i$ -C; Ar and Ph), 129.3, 127.1, 126.7, 126.6, 118.9 and 105.5 ($6 \times$ CH; Ar), 128.9,² 128.8² and 127.3¹ ($5 \times$ CH; Ph), 65.8 (CH_2O), 55.8 ($BnNCH$), 55.2 (CH_3O), 42.9 (ArCHCH₃), 37.9 (CH_2Ph) and 19.4 (ArCHCH₃) (Found MNH_4^+ , 407.1960; $C_{24}H_{27}N_2O_4$ requires MNH_4^+ 407.1965); and the oxazolidin-2-one (*rac*)-*syn*-**33** (0.274 g, 52%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.35; mp 134–136 °C; ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1699 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.82 (1H, d, *J* 1.5, CH; Ar), 7.73 (2H, d, *J* 8.6, $2 \times$ CH; Ar), 7.54 (1H, dd, *J* 8.6 and 1.5; Ar), 7.15–7.10 (3H, m, $3 \times$ CH; Ph), 7.13 (1H, br s, CH; Ar), 7.05–7.02 (1H, br d, *J* 8.6, CH; Ar), 6.88 (2H, br d, *J* 7.1, $2 \times$ CH; Ph), 5.26 (1H, q, *J* 6.9, ArCHCH₃), 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, OCH_3), 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, ArCHCH₃); δ_C (100 MHz; $CDCl_3$) 174.5 (NC=O), 157.7 (*i*-CO; Ar), 152.9 (OC=O), 135.2, 134.8, 133.7 and 129.7 ($4 \times i$ -C; Ar and Ph), 129.3, 127.2, 126.6, 125.9, 118.9 and 105.5 ($6 \times$ CH; Ar), 128.9,² 128.7² and 128.5¹ ($5 \times$ CH; Ph), 65.8 (CH_2O), 55.2 (CH_3O), 54.8 ($BnCHN$), 43.0 (ArCHCH₃), 37.3 (CH_2Ph) and 19.0 (ArCHCH₃) (Found MNH_4^+ , 407.1971. $C_{24}H_{27}ClN_2O_4$ requires MNH_4^+ , 407.1965).

4.42. Synthesis of 4-isopropyl-3-[2-(6-methoxy-naphthalene-2-yl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**40** and 4-isopropyl-3-[2-(6-methoxy-naphthalene-2-yl)propanoyl]-oxazolidin-2-one (*rac*)-*syn*-**40**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**13** (0.17 g, 1.36 mmol) and the pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** (0.59 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **40** (ratio: 92:8 *syn*-:*anti*-). The crude residue was purified by flash column chromatography on silica gel eluting with light petroleum ether (bp 40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*rac*)-*anti*-**40** (19 mg, 4%) as a white crystalline solid; R_F [light petroleum ether (40–60 °C)/diethyl ether (1:1)] 0.51; mp 122–124 °C; ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1701 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.70 (1H, s, CH; Ar), 7.68 (2H, dd, *J* 8.4 and 2.7, $2 \times$ CH; Ar- OCH_3), 7.46 (1H, dd, *J* 8.7 and 1.6, Ar), 7.13 (1H, dd, *J* 8.7 and 1.7, CH; Ar), 7.09 (1H, s, CH; Ar), 5.28 (1H, q, *J* 6.9, ArCHCH₃), 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, $CH(CH_3)_2$), 1.57 (3H, d, *J* 6.9, ArCHCH₃), 0.91 (3H, d, *J* 6.9, $CH_3^A CHCH_3^B$) and 0.90 (3H, d, *J* 6.9, $CH_3^A CHCH_3^B$); δ_C (100 MHz; $CDCl_3$) 174.7 (NC=O), 157.6 (*i*-C-O; Ar), 153.7 (OC=O), 135.4, 133.8 and 128.8 ($3 \times i$ -C; Ar), 129.3, 127.0, 126.8, 126.6, 118.8 and 105.5 ($6 \times$ CH; Ar), 63.0 (CH_2O), 59.0 (*i*-PrCHN), 55.2 (OCH_3), 42.8 (ArCHCH₃), 28.5 ($CH(CH_3)_2$), 19.6 ($CH_3^A CHCH_3^B$), 17.9 ($CH_3^A CHCH_3^B$) and 14.6 (ArCHCH₃) (Found MH^+ , 342.1707; $C_{20}H_{24}NO_4$ requires MH^+ , 342.1700); and the oxazolidin-2-one (*rac*)-*syn*-**40** (0.25 g, 54%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.34; mp 92–94 °C; ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1701 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.72 (1H, br d, *J* 1.7, CH; Ar), 7.69 (2H, dd, *J* 8.6 and 2.5, $2 \times$ CH; Ar), 7.45 (1H, dd, *J* 8.6 and 1.8, CH; Ar), 7.13 (1H, dd, *J* 8.6 and 1.8, CH; Ar), 7.09 (1H, s, CH; Ar), 5.26 (1H, q, *J* 6.9, ArCHCH₃), 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, $CH(CH_3)_2$), 1.53 (3H, d, *J* 6.9, ArCHCH₃), 0.75 (3H, d, *J* 6.9, $CH_3^A CHCH_3^B$) and 0.38 (3H, d, *J* 6.9, $CH_3^A CHCH_3^B$); δ_C (100 MHz; $CDCl_3$) 174.6 (NC=O), 157.6 (*i*-C-O; Ar), 153.5 (OC=O), 135.7, 133.7 and 128.9 ($3 \times i$ -C; Ar), 129.4, 127.0, 126.7, 126.6, 118.8 and 105.5 ($6 \times$ CH; Ar), 62.9 (CH_2O), 58.1

(*i*-PrCHN), 55.3 (OCH_3), 43.2 (ArCHCH₃), 27.9 ($CH(CH_3)_2$), 18.7 ($CH_3^A CHCH_3^B$), 17.7 ($CH_3^A CHCH_3^B$) and 14.0 (ArCHCH₃) (Found MH^+ , 342.1701; $C_{20}H_{24}NO_4$ requires MH^+ , 342.1700).

4.43. Synthesis of 4-phenyl-3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-oxazolidin-2-one (*rac*)-*anti*-**9** and 4-phenyl-3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-oxazolidin-2-one (*rac*)-*syn*-**9**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), 4-phenyl-oxazolidin-2-one (*rac*)-**8** (0.22 g, 1.36 mmol) and the pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** (0.59 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **9** (ratio: 95:5 *syn*-:*anti*-). The crude residue was purified by flash column chromatography on silica gel eluting with [light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)] to give oxazolidin-2-one (*rac*)-*anti*-**9** (16 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.45; ν_{max} ($CHCl_3$) cm^{-1} 1782 (OC=O) and 1705 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.75 (1H, s, CH; Ar), 7.69 (2H, dd, *J* 8.6 and 2.6, $2 \times$ CH; Ar), 7.49–7.30 (6H, m, $6 \times$ CH; Ar and Ph), 7.15–7.10 (2H, m, $2 \times$ CH; Ar and Ph), 5.31 (1H, dd, *J* 8.6 and 3.3, CH_AH_BO), 5.27 (1H, q, *J* 7.0, ArCHCH₃), 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* 7.0, ArCHCH₃); δ_C (100.6 MHz; $CDCl_3$) 174.1 (NC=O), 157.6 (*i*-OC; Ar), 153.2 (OC=O), 139.3, 135.3, 133.7 and 128.8 ($4 \times i$ -C; Ar), 129.2, 127.1, 126.8, 126.7, 118.9 and 105.5 ($6 \times$ CH; Ar), 128.8,² 128.6¹ and 125.7² ($5 \times$ CH; Ph), 69.6 (CH_2O), 58.0 (PhCHN), 55.2 (CH_3O), 43.0 (ArCHCH₃) and 19.3 (ArCHCH₃) (Found MH^+ , 376.1545; $C_{23}H_{22}NO_4$ requires MH^+ , 376.1543); and the oxazolidin-2-one (*rac*)-*syn*-**9** (0.32 g, 62%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.33; mp 137–139 °C; 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, CH; Ar), 7.51 (1H, br d, *J* 8.4, CH; Ar), 7.33 (1H, s, CH; Ar), 7.29–7.24 (3H, m, $3 \times$ CH; Ph), 7.15–7.10 (2H, m, $2 \times$ CH; Ph), 6.91 (2H, br d, *J* 7.0, $2 \times$ CH; Ar), 5.46 (1H, dd, *J* 8.9 and 5.2, PhCHN), 5.20 (1H, q, *J* 6.9, ArCHCH₃), 4.60 (1H, t, *J* 9.1, 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, ArCHCH₃); 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 ($4 \times i$ -C; Ar and Ph), 129.4, 127.0, 126.4, 126.3, 118.7 and 105.5 ($6 \times$ CH; Ar), 128.8,² 127.2¹ and 125.9² ($5 \times$ CH; Ph), 69.5 (CH_2O), 57.8 (PhCHN), 55.3 (CH_3O), 43.8 (ArCHCH₃) and 18.7 (ArCHCH₃) (Found MH^+ , 376.1553; $C_{23}H_{22}NO_4$ requires MH^+ , 376.1543).

4.44. Synthesis of ethyl 3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-oxazolidin-2-one 4-carboxylate (*rac*)-*anti*-**52** and ethyl 3-[2-(6-methoxynaphthalene-2-yl)propanoyl]-oxazolidin-2-one 4-carboxylate (*rac*)-*syn*-**52**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-BuLi (0.6 mL, 2.5 M in hexane, 1.50 mmol), oxazolidin-2-one (*rac*)-**14** (0.21 g, 1.36 mmol) and the pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** (0.59 g, 1.50 mmol), gave a separable mixture of two diastereoisomeric oxazolidin-2-ones **52** (ratio: 97:3 *syn*-:*anti*-). The crude residue was purified by flash column chromatography on a silica gel [light petroleum ether (bp 40–60 °C)/diethyl ether (7:3)] to give the oxazolidin-2-one (*rac*)-*anti*-**52** (9 mg, 2%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.28; ν_{max} ($CHCl_3$) cm^{-1} 1791 (OC=O), 1751 (CC=O) and 1705 (NC=O); δ_H (400 MHz; $CDCl_3$) 7.72 (1H, s, CH; Ar), 7.67 (2H, dd, *J* 8.4 and 2.6, $2 \times$ CH; Ar), 7.44 (1H, dd, *J* 8.4 and 2.6, 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, ArCHCH₃),

4.76 (1H, dd, *J* 9.1 and 3.7, EtO₂CCHN), 4.37–4.20 (4H, m, 2 × CH₂O), 3.88 (3H, s, OCH₃), 1.58 (3H, d, *J* 6.9, ArCHCH₃) and 1.31 (3H, t, *J* 6.9, OCH₂CH₃); δ_C (100 MHz; CDCl₃) 174.5 (NC=O), 168.6 (EtOC=O), 157.7 (*i*-CO; Ar), 152.0 (OC=O), 135.0, 133.8 and 129.3 (3 × *i*-C; Ar), 128.8, 127.1, 126.8, 126.7, 119.0 and 105.5 (6 × CH; Ar), 64.2 (CH₂O), 62.5 (CH₂O), 55.8 (EtO₂CCHN), 55.3 (OCH₃), 42.8 (ArCHCH₃), 19.1 (ArCHCH₃) and 14.0 (OCH₂CH₃) (Found MH⁺, 372.1445, C₂₀H₂₂NO₆ requires MH⁺, 372.1442); and the oxazolidin-2-one (*rac*)-**syn-52** (0.29 g, 57%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.18; mp 118–121 °C; ν_{max} (CHCl₃) cm⁻¹ 1789 (OC=O), 1745 (CC=O) and 1705 (NC=O); δ_H (400 MHz; CDCl₃) 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, ArCHCH₃), 4.94 (1H, dd, *J* 9.7 and 4.9, EtO₂CCHN), 4.49 (1H, t, *J* 9.7, CH_AH_BO), 4.20 (1H, dd, *J* 9.7 and 4.9, CH_AH_BO), 4.07 (2H, q, *J* 7.2, OCH₂CH₃), 3.88 (3H, s, OCH₃), 1.55 (3H, d, *J* 7.2, ArCHCH₃) and 1.03 (3H, t, *J* 7.2, OCH₂CH₃); δ_C (100 MHz; CDCl₃) 174.3 (NC=O), 167.9 (EtOC=O; ester), 157.6 (OC=O), 151.9 (*i*-CO; Ar), 134.8, 133.7 and 128.9 (3 × *i*-C; Ar), 129.1, 127.1, 126.9, 126.8, 118.7 and 105.6 (6 × CH; Ar), 64.1 (CH₂O), 63.6 (CH₂O), 55.6 (EtO₂CCHN), 55.2 (OCH₃), 43.10 (ArCHCH₃), 19.2 (ArCHCH₃) and 13.7 (OCH₂CH₃) (Found MNH₄⁺, 389.1703; C₂₀H₂₅N₂O₆ requires MNH₄⁺, 389.1707).

4.45. Parallel kinetic resolutions of active esters (*rac*)-**6**, (*rac*)-**7**, (*rac*)-**15**, (*rac*)-**16**, (*rac*)-**17**, (*rac*)-**18** and (*rac*)-**19** using a quasi-enantiomeric combination of oxazolidin-2-ones (*S*)-**13** and (*R*)-**8**

See Ref. 15.

4.46. Parallel kinetic resolution of pentafluorophenyl 2-phenylpropanoate (*rac*)-**15** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-phenyl oxazolidin-2-one (*R*)-**8**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-phenyl-oxazolidin-2-one (*R*)-**8** (0.106 g, 0.65 mmol) and pentafluorophenyl 2-phenylpropanoate (*rac*)-**15** (0.458 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**34** (ratio 95:5:*syn*-:*anti*-) and oxazolidin-2-ones (*S,R*)-*syn*- and (*R,R*)-*anti*-**41** (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 oxazolidin-2-one (*S,S*)-*anti*-**34** (5 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.64; ν_{max} (film) cm⁻¹ 1774 (OC=O) and 1701 (NC=O); [α]_D²⁰ = +128.9 (c 3.5, CHCl₃) (Found MH⁺ 262.1434; C₁₅H₂₀NO₃⁺ requires 262.1443); the oxazolidin-2-one (*R,S*)-*syn*-**34** (96 mg, 57%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.43; ν_{max} (CHCl₃) cm⁻¹ 1774 (OC=O) and 1703 (NC=O); [α]_D²⁰ = -19.8 (c 3.3, CHCl₃) (Found MH⁺ 262.1432; C₁₅H₂₀NO₃⁺ requires 262.1443); the oxazolidin-2-one (*R,R*)-*anti*-**41** (5 mg, 3%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.58; mp 158–160 °C; ν_{max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O); [α]_D²⁰ = -165.2 (c 2.0, CHCl₃) (Found MH⁺, 296.1282; C₁₈H₁₈NO₃⁺ requires 296.1287); the oxazolidin-2-one (*S,R*)-*syn*-**41** (0.11 g, 57%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; mp 140–142 °C; ν_{max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1701 (NC=O); [α]_D²⁰ = +88.5 (c 4.0, CHCl₃) (Found MH⁺, 296.1286; C₁₅H₁₈NO₃⁺ requires 296.1287). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)]—(*S,S*)-*anti*-**34** (R_F 0.64); (*R,S*)-*syn*-**34** (R_F 0.43); (*R,R*)-*anti*-**41** (R_F 0.58) and (*S,R*)-*syn*-**41** (R_F 0.42).

4.47. Parallel kinetic resolution of pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-phenyl oxazolidin-2-one (*R*)-**8**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-phenyl oxazolidin-2-one (*R*)-**8** (0.106 g, 0.65 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** (0.478 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**35** (ratio 95:5:*syn*-:*anti*-) and oxazolidin-2-ones (*S,R*)-*syn*- and (*R,R*)-*anti*-**42** (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 oxazolidin-2-one (*S,S*)-*anti*-**35** (6 mg, 3%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 65–67 °C; [α]_D²⁰ = +117.6 (c 0.66, CHCl₃); ν_{max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O) (Found MH⁺, 276.1612; C₁₆H₂₂NO₃ requires 276.1600); and the oxazolidin-2-one (*R,S*)-*syn*-**35** (114 mg, 64%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.53; [α]_D²⁰ = -24.6 (c 5.0, CHCl₃) {for (*S,R*)-*syn*-**35**; [α]_D²⁰ = +22.4 (c 6.9, CHCl₃)}; ν_{max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O) (Found MH⁺, 276.1587; C₁₆H₂₂NO₃ requires 276.1600); and the oxazolidin-2-one (*R,R*)-*anti*-**42** (6 mg, 3%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; mp 136–140 °C; [α]_D²⁰ = -160.0 (c 0.74, CHCl₃); {for (*S,S*)-*anti*-**42**; [α]_D²⁰ = +150.4 (c 4.9, CHCl₃)}; ν_{max} (CHCl₃) cm⁻¹ 1780 (OC=O), 1703 (NC=O) and 1600 (Ph) (Found MH⁺, 310.1430; C₁₉H₂₀NO₃ requires 310.1443); and the oxazolidin-2-one (*S,R*)-*syn*-**42** (0.122 g, 61%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.50; mp 82–84 °C; [α]_D²⁰ = +77.4 (c 4.0, CHCl₃) {for (*R,S*)-*syn*-**42**; [α]_D²⁰ = -95.6 (c 3.0, CHCl₃)}; ν_{max} (film) cm⁻¹ 1780 (OC=O) and 1703 (NC=O) (Found MH⁺, 310.1437; C₁₉H₂₀NO₃ requires 310.1443). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)]—(*S,S*)-*anti*-**35** (R_F 0.63); (*R,S*)-*syn*-**35** (R_F 0.53); (*R,R*)-*anti*-**42** (R_F 0.55) and (*S,R*)-*syn*-**42** (R_F 0.50).

4.48. Parallel kinetic resolution of pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-phenyl-oxazolidin-2-one (*R*)-**8**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-phenyl oxazolidin-2-one (*R*)-**8** (0.106 g, 0.65 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.499 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**36** (ratio 79:21:*syn*-:*anti*-) and oxazolidin-2-ones (*S,R*)-*syn*- and (*R,R*)-*anti*-**43** (ratio 84:16:*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 oxazolidin-2-ones (*S,S*)-*anti*-**36** and (*R,S*)-*syn*-**36** (0.109 g, 58%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] ~0.82; characterisation for (*S,S*)-*anti*-**36**; colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82; ν_{max} (CHCl₃) cm⁻¹ 1770 (OC=O) and 1700 (NC=O) (Found MH⁺, 290.1751; C₁₇H₂₄NO₃⁺ requires MH⁺ 290.1751); the oxazolidin-2-one (*R,S*)-**36** as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82; ν_{max} (CHCl₃) cm⁻¹ 1772 (OC=O) and 1700 (NC=O) (Found MH⁺, 290.1751; C₁₇H₂₄NO₃⁺ requires MH⁺ 290.1751); and an inseparable mixture of oxazolidin-2-ones (*R,R*)-*anti*-**43** and (*S,R*)-*syn*-**43** (0.127 g, 60%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] ~0.55; ν_{max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O) (Found MNH₄⁺, 341.1860);

$C_{20}H_{25}N_2O_3^+$ requires MNH_4^+ 341.1860). Characterisation data for oxazolidin-2-one (*S,R*)-*syn*-**43**; colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; $[\alpha]_D^{20} = +78.4$ (c 0.5, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1781 (OC=O) and 1700 (NC=O) (Found MNH_4^+ , 341.1860; $C_{20}H_{25}N_2O_3^+$ requires MNH_4^+ 341.1860). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] – (*S,S*)-*anti*-**36** (R_F 0.82); (*R,S*)-*syn*-**36** (R_F 0.82); (*R,R*)-*anti*-**43** (R_F 0.62) and (*S,R*)-*syn*-**43** (R_F 0.55).

4.49. Parallel kinetic resolution of pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-phenyl-oxazolidin-2-one (*R*)-**8**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-phenyl oxazolidin-2-one (*R*)-**8** (0.106 g, 0.65 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** (0.479 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**37** (ratio 98:2:*syn*–:*anti*–) and oxazolidin-2-ones (*S,R*)-*syn*- and (*R,R*)-*anti*-**44** (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 oxazolidin-2-one (*S,S*)-*anti*-**37** (2 mg, 1%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; $[\alpha]_D^{23} = +115.5$ (c 0.7, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1701 (NC=O); (Found MNH_4^+ , 293.1857; $C_{16}H_{25}N_2O_3^+$ requires MNH_4^+ , 293.1860); the oxazolidin-2-one (*R,S*)-*syn*-**37** (0.107 g, 60%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.50; $[\alpha]_D^{23} = -26.7$ (c 1.8, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1700 (NC=O) (Found MNH_4^+ , 293.1858; $C_{16}H_{25}N_2O_3^+$ requires MNH_4^+ , 293.1860); the oxazolidin-2-one (*R,R*)-*anti*-**44** (6 mg, 3%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.47; mp 124–126 °C; $[\alpha]_D^{23} = -179.1$ (c 3.0, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1779 (OC=O) and 1699 (NC=O); (Found MNH_4^+ , 341.1860; $C_{20}H_{25}N_2O_3^+$ requires MNH_4^+ 341.1860); and the oxazolidin-2-one (*S,R*)-*syn*-**44** (0.12 g, 60%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.29; mp 120–122 °C; $[\alpha]_D^{23} = +121.6$ (c 0.6, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1702 (NC=O); (Found MNH_4^+ , 341.1860; $C_{20}H_{25}N_2O_3^+$ requires MNH_4^+ 341.1860). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] – (*S,S*)-*anti*-**37** (R_F 0.63); (*R,S*)-*syn*-**37** (R_F 0.50); (*R,R*)-*anti*-**44** (R_F 0.47) and (*S,S*)-*syn*-**44** (R_F 0.29).

4.50. Parallel kinetic resolution of pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-phenyl oxazolidin-2-one (*R*)-**8**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-phenyl oxazolidin-2-one (*R*)-**8** (0.106 g, 0.65 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.539 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**38** (ratio 95:5:*syn*–:*anti*–) and oxazolidin-2-ones (*S,R*)-*syn*- and (*R,R*)-*anti*-**10** (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 oxazolidin-2-one (*S,S*)-*anti*-**38** (8 mg, 4%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.77; ν_{max} ($CHCl_3$) cm^{-1} 1776 (OC=O) and 1692 (NC=O); $[\alpha]_D^{25} = +117.3$ (c 1.3, $CHCl_3$) (Found MH^+ , 318.20062; $C_{19}H_{28}NO_3$ requires

318.2064); the oxazolidin-2-one (*R,S*)-*syn*-**38** (0.133 g, 64%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; ν_{max} ($CHCl_3$) cm^{-1} 1778 (OC=O) and 1699 (NC=O); $[\alpha]_D^{25} = -33.0$ (c 1.2, $CHCl_3$) (Found M , 317.1979; $C_{29}H_{27}NO_3$ requires 317.1985); the oxazolidin-2-one (*R,R*)-*anti*-**10** (7 mg, 3%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.62; ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1701 (NC=O); mp 155–158 °C; $[\alpha]_D^{25} = -145.7$ (c 3.0, $CHCl_3$) (Found MH^+ , 352.1913; $C_{22}H_{26}NO_3$ requires 352.1907); the oxazolidin-2-one (*S,R*)-*syn*-**10** (0.134 g, 59%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.41; ν_{max} ($CHCl_3$) cm^{-1} 1779 (OC=O) and 1705 (NC=O); mp 86–88 °C; $[\alpha]_D^{25} = +118.7$ (c 6.0, $CHCl_3$) (Found MH^+ , 352.1909; $C_{22}H_{26}NO_3$ requires 352.1907). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] – (*S,S*)-*anti*-**38** (R_F 0.77); (*R,S*)-*syn*-**38** (R_F 0.55); (*R,R*)-*anti*-**10** (R_F 0.62) and (*S,R*)-*syn*-**10** (R_F 0.41).

4.51. Parallel kinetic resolution of pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-phenyl-oxazolidin-2-one (*R*)-**8**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-phenyl oxazolidin-2-one (*R*)-**8** (0.106 g, 0.65 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.508 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**39** (ratio 98:2:*syn*–:*anti*–) and oxazolidin-2-ones (*S,R*)-*syn*- and (*R,R*)-*anti*-**45** (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 oxazolidin-2-one (*S,S*)-*anti*-**39** (4 mg, 2%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.56; ν_{max} ($CHCl_3$) cm^{-1} 1781 (OC=O) and 1702 (NC=O); $[\alpha]_D^{23} = +101.5$ (c 5.8, $CHCl_3$); (Found $MNH_4^{+35}Cl$) 313.1310; $C_{15}H_{22}ClN_2O_3$ requires $MNH_4^{+35}Cl$ 313.1313); the oxazolidin-2-one (*R,S*)-*syn*-**39** (0.119 g, 62%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.32; ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1700 (NC=O); mp 63–65 °C; $[\alpha]_D^{23} = -32.4$ (c 1.9, $CHCl_3$) (Found $MNH_4^{+35}Cl$) 313.1311; $C_{15}H_{22}ClN_2O_3$ requires $MNH_4^{+35}Cl$ 313.1313); the oxazolidin-2-one (*R,R*)-*anti*-**45** (5 mg, 2%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; $[\alpha]_D^{23} = -156.3$ (c 1.2, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1780 (OC=O) and 1700 (NC=O); (Found $MNH_4^{+35}Cl$) 347.1154; $C_{18}H_{20}ClN_2O_3$ requires $MNH_4^{+35}Cl$ 347.1157); and the oxazolidin-2-one (*S,R*)-*syn*-**45** (0.126 g, 59%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.27; mp 142–145 °C; $[\alpha]_D^{23} = +144.4$ (c 1.6, $CHCl_3$); ν_{max} ($CHCl_3$) cm^{-1} 1782 (OC=O) and 1700 (NC=O) (Found $MNH_4^{+35}Cl$) 347.1154; $C_{18}H_{20}ClN_2O_3$ requires $MNH_4^{+35}Cl$ 347.1157); R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] – (*S,S*)-*anti*-**39** (R_F 0.56); (*R,S*)-*syn*-**39** (R_F 0.32); (*R,R*)-*anti*-**45** (R_F 0.42) and (*S,R*)-*syn*-**45** (R_F 0.27).

4.52. Parallel kinetic resolution of pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-phenyl-oxazolidin-2-one (*R*)-**8**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-phenyl oxazolidin-2-one (*R*)-**8** (0.106 g, 0.65 mmol) and pentafluorophenyl

2-(6-methoxynaphthalene-2-yl)-propanoate (*rac*)-**6** (0.574 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**40** (ratio 96:4:*syn*:-*anti*-) and oxazolidin-2-ones (*S,R*)-*syn*- and (*R,R*)-*anti*-**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 the (*S,S*)-*anti*-**40** (6 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.51; ν_{\max} (CHCl₃) cm⁻¹ 1776 (OC=O) and 1700 (NC=O); $[\alpha]_D^{23} = +194.3$ (c 1.6, CHCl₃) (Found MH⁺, 342.1707; C₂₀H₂₄NO₄⁺ requires MH⁺, 342.1700); (*R,S*)-*syn*-**40** (0.138 g, 62%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.34; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1701 (NC=O); $[\alpha]_D^{23} = -59.6$ (c 3.3, CHCl₃) (Found MH⁺, 342.1701; C₂₀H₂₄NO₄⁺ requires MH⁺, 342.1700); (*R,R*)-*anti*-**9** (5 mg, 2%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.45; ν_{\max} (CHCl₃) cm⁻¹ 1779 (OC=O) and 1699 (NC=O); $[\alpha]_D^{23} = -164.2$ (c 1.3, CHCl₃) (Found MH⁺, 376.1545; C₂₃H₂₂NO₄⁺ requires MH⁺, 376.1543); and (*S,R*)-*syn*-**9** (94 mg, 38%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.33; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O); $[\alpha]_D^{23} = +166.2$ (c 1.5, CHCl₃) (Found MH⁺, 376.1553; C₂₃H₂₂NO₄⁺ requires MH⁺, 376.1543). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] – (*S,S*)-*anti*-**40** (R_F 0.51); (*R,S*)-*syn*-**40** (R_F 0.34); (*R,R*)-*anti*-**9** (R_F 0.45) and (*S,R*)-*syn*-**9** (R_F 0.33).

4.53. Parallel kinetic resolutions of active esters (*rac*)-**6**, (*rac*)-**7**, (*rac*)-**15**, (*rac*)-**16**, (*rac*)-**17**, (*rac*)-**18** and (*rac*)-**19** using a quasi-enantiomeric combination of oxazolidin-2-ones (*S*)-**13** and (*S*)-**14**

See Ref. 15.

4.54. Parallel kinetic resolution of pentafluorophenyl 2-phenylpropanoate (*rac*)-**15** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-phenylpropanoate (*rac*)-**15** (0.458 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**34** (ratio 95:5:*syn*:-*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**48** (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 oxazolidin-2-one (*S,S*)-*anti*-**34** (5 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.64; ν_{\max} (film) cm⁻¹ 1774 (OC=O) and 1701 (NC=O); $[\alpha]_D^{20} = +128.9$ (c 3.5, CHCl₃) (Found MH⁺ 262.1434; C₁₅H₂₀NO₃⁺ requires 262.1443); the oxazolidin-2-one (*R,S*)-*syn*-**34** (96 mg, 57%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.43; ν_{\max} (CHCl₃) cm⁻¹ 1774 (OC=O) and 1703 (NC=O); $[\alpha]_D^{20} = -19.8$ (c 3.3, CHCl₃) (Found MH⁺ 262.1432; C₁₅H₂₀NO₃⁺ requires 262.1443); the oxazolidin-2-one (*R,S*)-*anti*-**46** (4 mg, 2%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; ν_{\max} (CHCl₃) cm⁻¹ 1794 (OC=O), 1747 (CC=O) and 1705 (NC=O); $[\alpha]_D^{20} = -130.5$ (c 2.1, CHCl₃); and the oxazolidin-2-one (*S,S*)-*syn*-**46** (90 mg, 47%) as a white powder; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.30; mp 97–99 °C; $[\alpha]_D^{20} = +24.8$ (c 5.3, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1793 (OC=O), 1747 (CC=O) and 1705 (NC=O); (Found MH⁺, 292.1195; C₁₅H₁₈NO₃⁺ requires 292.1185). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] – (*S,S*)-*anti*-**34** (R_F 0.64); (*R,S*)-*syn*-**34** (R_F 0.43); (*R,S*)-*anti*-**46** (R_F 0.42) and (*S,S*)-*syn*-**46** (R_F 0.30).

4.55. Parallel kinetic resolution of pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** (0.478 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**35** (ratio 96:4:*syn*:-*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**47** (ratio 95:5:*syn*:-*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (40–60 °C)/diethyl ether (7:3) to give oxazolidin-2-one (*S,S*)-*anti*-**35** (4 mg, 2%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 65–67 °C; $[\alpha]_D^{20} = +128.9$ (c 3.5, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O) (Found MH⁺, 276.1612; C₁₆H₂₂NO₃ requires 276.1600); and the oxazolidin-2-one (*R,S*)-*syn*-**35** (0.108 g, 60%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.53; $[\alpha]_D^{20} = -24.6$ (c 5.0, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1697 (NC=O); (Found MH⁺, 276.1587; C₁₆H₂₂NO₃ requires 276.1600); and the oxazolidin-2-one (*R,S*)-*anti*-**35** (6 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.48; $[\alpha]_D^{20} = -131.1$ (c 3.3, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1747 (CC=O) and 1705 (NC=O) (Found M⁺, 305.1258; C₁₆H₁₉NO₅ requires 305.1258); and the oxazolidin-2-one (*S,S*)-*syn*-**47** (0.124 g, 62%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.38; $[\alpha]_D^{20} = +30.0$ (c 8.2, CHCl₃) [for (*R,R*)-*syn*-**47**]; $[\alpha]_D^{20} = -24.8$ (c 5.3, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1747 (CC=O) and 1701 (OC=O) (Found M⁺, 305.1256; C₁₆H₁₉NO₅ requires 305.1258). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] – (*S,S*)-*anti*-**35** (R_F 0.63); (*R,S*)-*syn*-**35** (R_F 0.53); (*R,S*)-*anti*-**47** (R_F 0.48) and (*S,S*)-*syn*-**47** (R_F 0.38).

4.56. Parallel kinetic resolution of pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.499 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**36** (ratio 68:32:*syn*:-*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**48** (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 an inseparable mixture of oxazolidin-2-ones (*S,S*)-*anti*-**36** and (*R,S*)-*syn*-**36** (0.114 g, 61%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82; {for ratio (*R,S*)-*syn*-**36**:(*S,S*)-*anti*-**36**: 68:32 – $[\alpha]_D^{25} = +35.1$ (c 1.9, CHCl₃)}; characterisation data for (*S,S*)-*anti*-**36**; ν_{\max} (CHCl₃) cm⁻¹ 1770 (OC=O) and 1700 (NC=O) (Found MH⁺, 290.1751; C₁₇H₂₄NO₃⁺ requires MH⁺ 290.1751); characterisation data for oxazolidin-2-one (*R,S*)-*syn*-**36**; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.82; ν_{\max} (CHCl₃) cm⁻¹ 1772 (OC=O) and 1700 (NC=O) (Found MH⁺, 290.1751; C₁₇H₂₄NO₃⁺ requires MH⁺ 290.1751); the oxazolidin-2-one (*R,S*)-*anti*-**48** (14 mg, 7%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; $[\alpha]_D^{25} = +19.6$ (c 0.2, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1751 (CC=O) and 1700 (NC=O) (Found MNH₄⁺, 337.1761; C₁₇H₂₅N₂O₅⁺ requires MNH₄⁺ 337.1758); and the oxazolidin-2-one (*S,S*)-*syn*-**48** (0.118 g, 57%) as a colourless oil; R_F [light

petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; $[\alpha]_D^{25} = -8.4$ (c 0.9, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1755 (CC=O) and 1700 (NC=O) (Found MNH₄⁺, 337.1756; C₁₇H₂₅N₂O₅⁺ requires MNH₄⁺ 337.1758). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] - (S,S)-*anti*-**36** (R_F 0.82); (R,S)-*syn*-**36** (R_F 0.82); (R,S)-*anti*-**48** (R_F 0.55) and (S,S)-*syn*-**48** (R_F 0.42).

4.57. Parallel kinetic resolution of pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** (0.479 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**37** (ratio 95:5:*syn*:-*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**49** (ratio 95:5:*syn*:-*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (40–60 °C)/diethyl ether (7:3) to give the oxazolidin-2-one (*S,S*)-*anti*-**37** (5 mg, 3%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.63; mp 67–69 °C; $[\alpha]_D^{23} = +115.5$ (c 0.7, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1779 (OC=O) and 1702 (NC=O) (Found MNH₄⁺, 293.1857; C₁₆H₂₅N₂O₃⁺ requires MNH₄⁺, 293.1860); the oxazolidin-2-one (*R,S*)-*syn*-**37** (0.104 g, 58%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.50; mp 46–48 °C; $[\alpha]_D^{23} = -26.7$ (c 1.8, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1779 (OC=O) and 1700 (NC=O) (Found MNH₄⁺, 293.1858; C₁₆H₂₅N₂O₃⁺ requires MNH₄⁺, 293.1860); the oxazolidin-2-one (*R,S*)-*anti*-**49** (8 mg, 4%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.40; $[\alpha]_D^{23} = -125.6$ (c 2.5, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O), 1745 (CC=O) and 1702 (NC=O) (Found MNH₄⁺, 323.1596; C₁₆H₂₃N₂O₅⁺ requires MNH₄⁺, 323.1601); and the oxazolidin-2-one (*S,S*)-*syn*-**49** (0.128 g, 64%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.20; $[\alpha]_D^{23} = +34.6$ (c 0.6, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O), 1746 (CC=O) and 1700 (NC=O) (Found MNH₄⁺, 323.1607; C₁₆H₂₃N₂O₅⁺ requires MNH₄⁺, 323.1601). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)]—(*S,S*)-*anti*-**37** (R_F 0.63); (*R,S*)-*syn*-**37** (R_F 0.50); (*R,S*)-*anti*-**49** (R_F 0.40) and (*S,S*)-*syn*-**49** (R_F 0.20).

4.58. Parallel kinetic resolution of pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.539 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**38** (ratio 95:5:*syn*:-*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**50** (ratio 95:5:*syn*:-*anti*-). The crude residue was purified by flash chromatography on silica gel eluting with light petroleum ether (40–60 °C)/diethyl ether (7:3) to give oxazolidin-2-one (*S,S*)-*anti*-**38** (7 mg, 3%) as a colourless oil; R_F [light petroleum ether (40–60 °C)/diethyl ether (1:1)] 0.77; ν_{\max} (CHCl₃) cm⁻¹ 1776 (OC=O) and 1692 (NC=O); $[\alpha]_D^{25} = +117.3$ (c 1.3, CHCl₃) (Found MH⁺, 318.2062; C₁₉H₂₈NO₃ requires 318.2064); the oxazolidin-2-one (*R,S*)-*syn*-**38** (0.128 g, 62%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; ν_{\max}

(CHCl₃) cm⁻¹ 1778 (OC=O) and 1699 (NC=O); $[\alpha]_D^{25} = -33.0$ (c 1.2, CHCl₃) (Found M, 317.1979; C₂₉H₂₇NO₃ requires 317.1985); the oxazolidin-2-one (*R,S*)-*anti*-**50** (7 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.53; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1751 (CC=O) and 1701 (NC=O); $[\alpha]_D^{25} = -125.4$ (c 1.2, CHCl₃) (Found MNH₄⁺, 365.2069; C₁₉H₂₉N₂O₅ requires 365.2171); and the oxazolidin-2-one (*S,S*)-*syn*-**50** (0.135 g, 60%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.35; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1747 (CC=O) and 1699 (NC=O); $[\alpha]_D^{25} = +29.8$ (c 0.95, CHCl₃) (Found M+NH₄⁺, 365.2073; C₁₉H₂₉N₂O₅ requires 365.2071). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)]—(*S,S*)-*anti*-**38** (R_F 0.77); (*R,S*)-*syn*-**38** (R_F 0.55); (*R,S*)-*anti*-**50** (R_F 0.53) and (*S,S*)-*syn*-**50** (R_F 0.35).

4.59. Parallel kinetic resolution of pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.508 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**39** (ratio 90:10:*syn*:-*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**51** (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 oxazolidin-2-one (*S,S*)-*anti*-**39** (12 mg, 6%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.56; $[\alpha]_D^{23} = +101.5$ (c 5.8, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1702 (NC=O) (Found MNH₄(³⁵Cl)⁺ 313.1310; C₁₅H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺ 313.1313); the oxazolidin-2-one (*R,S*)-*syn*-**39** (0.107 g, 56%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.32; $[\alpha]_D^{23} = -32.4$ (c 1.9, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O); mp 63–65 °C (Found MNH₄(³⁵Cl)⁺ 313.1311; C₁₅H₂₂ClN₂O₃ requires MNH₄(³⁵Cl)⁺ 313.1313); and oxazolidin-2-one (*R,S*)-*anti*-**51** (6 mg, 3%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.31; $[\alpha]_D^{23} = -130.5$ (c 1.2, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1748 (CC=O) and 1700 (NC=O) (Found MNH₄(³⁵Cl)⁺ 343.1059; C₁₅H₂₀ClN₂O₅ requires MNH₄(³⁵Cl)⁺ 343.1055); and the oxazolidin-2-one (*S,S*)-*syn*-**51** (0.131 g, 62%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.20; $[\alpha]_D^{23} = +40.0$ (c 1.8, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1745 (CC=O) and 1700 (NC=O) (Found MNH₄(³⁵Cl)⁺ 343.1057; C₁₅H₂₀ClN₂O₅ requires MNH₄(³⁵Cl)⁺ 343.1055). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] - (*S,S*)-*anti*-**39** (R_F 0.56); (*R,S*)-*syn*-**39** (R_F 0.32); (*R,S*)-*anti*-**51** (R_F 0.31) and (*S,S*)-*syn*-**51** (R_F 0.20).

4.60. Parallel kinetic resolution of pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** with 4-isopropyl-oxazolidin-2-one (*S*)-**13** and ethyloxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-isopropyl-oxazolidin-2-one (*S*)-**13** (84 mg, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** (0.574 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**40** (ratio 93:7:*syn*:-*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**52** (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 the (*S,S*)-*anti*-**40** (8 mg, 4%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.51; $[\alpha]_D^{23} = +194.3$ (c 1.6, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1702 (NC=O) (Found MH⁺, 342.1707; C₂₀H₂₄NO₄⁺ requires MH⁺, 342.1700); (*R,S*)-*syn*-**40** (0.126 g, 57%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.34; $[\alpha]_D^{23} = -59.6$ (c 3.3, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1779 (OC=O) and 1700 (NC=O) (Found MH⁺, 342.1707; C₂₀H₂₄NO₄⁺ requires MH⁺, 342.1700); (*R,S*)-*anti*-**52** (5 mg, 2%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.28; $[\alpha]_D^{23} = -140.3$ (c 0.9, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O), 1744 (CC=O) and 1699 (NC=O) (Found MH⁺, 372.1445; C₂₀H₂₂NO₆⁺ requires MH⁺, 372.1442); and (*S,S*)-*syn*-**52** (0.112 g, 46%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.18; $[\alpha]_D^{23} = +55.7$ (c 3.0, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O), 1745 (CC=O) and 1700 (NC=O) (Found MNH₄⁺, 389.1703; C₂₀H₂₅N₂O₆⁺ requires MNH₄⁺, 389.1707). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)]—(*S,S*)-*anti*-**40** (R_F 0.51); (*R,S*)-*syn*-**40** (R_F 0.34); (*R,S*)-*anti*-**52** (R_F 0.28) and (*S,S*)-*syn*-**52** (R_F 0.18).

4.61. Parallel kinetic resolutions of active esters (*rac*)-**6**, (*rac*)-**7**, (*rac*)-**15**, (*rac*)-**16**, (*rac*)-**17**, (*rac*)-**18** and (*rac*)-**19** using a quasi-enantiomeric combination of oxazolidin-2-ones (*S*)-**13** and (*S*)-**14**

See Ref. 15.

4.62. Parallel kinetic resolution of pentafluorophenyl 2-phenylpropanoate (*rac*)-**15** with 4-phenyl-oxazolidin-2-one (*S*)-**8** and 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**8** (0.106 g, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-phenylpropanoate (*rac*)-**15** (0.458 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**41** (ratio 95:5:*syn*-:*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**46** (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 oxazolidin-2-one (*S,S*)-*anti*-**41** (6 mg, 3%) as a white crystalline solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.58; mp 158–160 °C; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O); $[\alpha]_D^{20} = +163.7$ (c 1.8, CHCl₃); {(*R,R*)-*anti*-**41**}; $[\alpha]_D^{20} = -165.2$ (c 2.0, CHCl₃) (Found MH⁺, 296.1282; C₁₈H₁₈NO₃⁺ requires 296.1287); the oxazolidin-2-one (*R,S*)-*syn*-**41** (0.128 g, 67%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; mp 140–142 °C; ν_{\max} (CHCl₃) cm⁻¹ 1778 (OC=O) and 1701 (NC=O); $[\alpha]_D^{20} = -92.8$ (c 2.6, CHCl₃); {(*S,R*)-*syn*-**41**}; $[\alpha]_D^{20} = +88.5$ (c 4.0, CHCl₃) (Found MH⁺, 296.1286; C₁₅H₁₈NO₃⁺ requires 296.1287); the oxazolidin-2-one (*R,S*)-*anti*-**46** (4 mg, 2%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; ν_{\max} (CHCl₃) cm⁻¹ 1794 (OC=O), 1747 (CC=O) and 1705 (NC=O); $[\alpha]_D^{20} = -130.5$ (c 2.1, CHCl₃) {(*S,R*)-*anti*-**46**}; $[\alpha]_D^{20} = -135.8$ (c 4.5, CHCl₃); $[\alpha]_D^{20} = -135.8$ (c 4.5, CHCl₃) (Found MH⁺, 292.1195; C₁₅H₁₈NO₅⁺ requires 292.1185); and the oxazolidin-2-one (*S,S*)-*syn*-**46** (0.131 g, 69%) as a white powder; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.30; mp 97–99 °C; ν_{\max} (CHCl₃) cm⁻¹ 1793 (OC=O), 1747 (CC=O) and 1705 (NC=O); $[\alpha]_D^{20} = +24.8$ (c 5.3, CHCl₃) (Found MH⁺, 292.1195; C₁₅H₁₈NO₅⁺ requires 292.1185). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)]—(*S,S*)-*anti*-**41** (R_F 0.58); (*R,S*)-*syn*-**41** (R_F 0.42); (*R,S*)-*anti*-**46** (R_F 0.42) and (*S,S*)-*syn*-**46** (R_F 0.30).

4.63. Parallel kinetic resolution of pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** with 4-phenyl-oxazolidin-2-one (*S*)-**8** and ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**8** (0.106 g, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-phenylbutanoate (*rac*)-**16** (0.478 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**42** (ratio 95:5:*syn*-:*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**47** (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 oxazolidin-2-one (*S,S*)-*anti*-**42** (6 mg, 3%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.55; mp 136–140 °C; $[\alpha]_D^{20} = +150.4$ (c 4.9, CHCl₃) {for (*R,R*)-*anti*-**42**}; $[\alpha]_D^{20} = -160.0$ (c 0.74, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O), 1703 (NC=O) and 1600 (Ph) (Found MH⁺, 310.1430; C₁₉H₂₀NO₃ requires 310.1443); and the oxazolidin-2-one (*R,S*)-*syn*-**42** (0.125 g, 62%) as a white solid; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.50; mp 82–84 °C; $[\alpha]_D^{20} = -95.6$ (c 3.0, CHCl₃) {(*S,R*)-*syn*-**42**}; $[\alpha]_D^{20} = +77.4$ (c 4.0, CHCl₃); ν_{\max} (film) cm⁻¹ 1780 (OC=O) and 1703 (NC=O) (Found MH⁺, 310.1437; C₁₉H₂₀NO₃ requires 310.1443); the oxazolidin-2-one (*R,S*)-*anti*-**47** (8 mg, 4%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.48; $[\alpha]_D^{20} = -131.1$ (c 3.3, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1747 (CC=O) and 1705 (NC=O) (Found M⁺, 305.1258; C₁₆H₁₉NO₅ requires 305.1258); and the oxazolidin-2-one (*S,S*)-*syn*-**47** (0.13 g, 65%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.38; $[\alpha]_D^{20} = +30.0$ (c 8.2, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1747 (CC=O) and 1701 (NC=O) (Found M⁺, 305.1256; C₁₆H₁₉NO₅ requires 305.1258). R_F differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)]—(*S,S*)-*anti*-**42** (R_F 0.55); (*R,S*)-*syn*-**42** (R_F 0.50); (*R,S*)-*anti*-**47** (R_F 0.48) and (*S,S*)-*syn*-**47** (R_F 0.38).

4.64. Parallel kinetic resolution of pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** with 4-phenyl-oxazolidin-2-one (*S*)-**8** and ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-phenyl-oxazolidin-2-one (*S*)-**8** (0.106 g, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-phenyl-3-methylbutanoate (*rac*)-**17** (0.499 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**43** (ratio 86:14:*syn*-:*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**48** (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 an inseparable mixture of oxazolidin-2-ones (*S,S*)-*anti*-**43** and (*R,S*)-*syn*-**43** (0.134 g, 64%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.64. Characterisation data for (*S,S*)-*anti*-**43**; colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.64; $[\alpha]_D^{20} = +9.4$ (c 0.3, CHCl₃) {(*S,R*)-*anti*-**43**}; $[\alpha]_D^{20} = -9.4$ (c 0.4, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O) (Found MNH₄⁺, 341.1860; C₂₀H₂₅N₂O₃⁺ requires MNH₄⁺ 341.1860); the oxazolidin-2-one (*R,S*)-*syn*-**43**; colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.61; $[\alpha]_D^{20} = -79.4$ (c 0.1, CHCl₃) {(*S,R*)-*syn*-**43**}; $[\alpha]_D^{20} = +78.4$ (c 0.5, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1700 (NC=O) (Found MNH₄⁺, 341.1860; C₂₀H₂₅N₂O₃⁺ requires MNH₄⁺ 341.1860); the oxazolidin-2-one (*R,S*)-*anti*-**48** (10 mg, 5%) as a colourless oil; R_F [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.645; $[\alpha]_D^{25} = +19.6$ (c

0.2, CHCl₃) CHECK; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1751 (CC=O) and 1700 (NC=O) (Found MNH₄⁺, 337.1761; C₁₇H₂₅N₂O₅⁺ requires MNH₄⁺ 337.1758); and the oxazolidin-2-one (S,S)-*syn*-**48** (0.118 g, 57%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; $[\alpha]_D^{25} = -8.4$ (c 0.9, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1755 (CC=O) and 1700 (OC=O) (Found MNH₄⁺, 337.1756; C₁₇H₂₅N₂O₅⁺ requires MNH₄⁺ 337.1758). R_f differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] - (S,S)-*anti*-**43** (R_f 0.64); (R,S)-*syn*-**43** (R_f 0.64); (R,S)-*anti*-**48** (R_f 0.55) and (S,S)-*syn*-**48** (R_f 0.42).

4.65. Parallel kinetic resolution of pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** with 4-phenyl-oxazolidin-2-one (S)-**8** and ethyl oxazolidin-2-one 4-carboxylate (S)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-phenyl-oxazolidin-2-one (S)-**8** (0.106 g, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (S)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(4-methylphenyl)propanoate (*rac*)-**18** (0.479 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (R,S)-*syn*- and (S,S)-*anti*-**44** (ratio 98:2:*syn*:*anti*-) and oxazolidin-2-ones (S,S)-*syn*- and (R,S)-*anti*-**49** (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 oxazolidin-2-one (S,S)-*anti*-**44** (2 mg, 1%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.47; mp 124–126 °C; $[\alpha]_D^{23} = +173.5$ (c 2.0, CHCl₃) {(R,R)-*anti*-**44**; $[\alpha]_D^{20} = -179.1$ (c 3.0, CHCl₃)}; ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O) and 1700 (NC=O) (Found MNH₄⁺, 327.1710; C₁₉H₂₃N₂O₃⁺ requires MNH₄⁺, 327.1700); the oxazolidin-2-one (R,S)-*syn*-**44** (0.118 g, 59%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.29; mp 120–122 °C; $[\alpha]_D^{23} = -116.8$ (c 0.8, CHCl₃) {(S,R)-*syn*-**44**; $[\alpha]_D^{20} = +121.6$ (c 0.6, CHCl₃)}; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1705 (NC=O) (Found MNH₄⁺, 327.1700; C₁₉H₂₃N₂O₃⁺ requires MNH₄⁺, 327.1700); the oxazolidin-2-one (R,S)-*anti*-**49** (6 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.40; $[\alpha]_D^{23} = -125.6$ (c 2.5, CHCl₃); 1779 (OC=O), 1750 (CC=O) and 1700 (NC=O) (Found MNH₄⁺, 323.1596; C₁₆H₂₃N₂O₅⁺ requires MNH₄⁺, 323.1601); and the oxazolidin-2-one (S,S)-*syn*-**49** (0.115 g, 58%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.20; $[\alpha]_D^{23} = +34.6$ (c 0.6, CHCl₃); 1780 (OC=O), 1748 (CC=O) and 1700 (NC=O) (Found MNH₄⁺, 323.1607; C₁₆H₂₃N₂O₅⁺ requires MNH₄⁺, 323.1601). R_f differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] - (S,S)-*anti*-**44** (R_f 0.47); (R,S)-*syn*-**44** (R_f 0.29); (R,S)-*anti*-**49** (R_f 0.40) and (S,S)-*syn*-**49** (R_f 0.20).

4.66. Parallel kinetic resolution of pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** with 4-phenyl-oxazolidin-2-one (S)-**8** and ethyl oxazolidin-2-one 4-carboxylate (S)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-phenyl-oxazolidin-2-one (S)-**8** (0.106 g, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (S)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(4-isobutylphenyl)propanoate (*rac*)-**7** (0.539 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (R,S)-*syn*- and (S,S)-*anti*-**10** (ratio 95:5:*syn*:*anti*-) and oxazolidin-2-ones (S,S)-*syn*- and (R,S)-*anti*-**10** (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 oxazolidin-2-one (S,S)-*anti*-**10** (9 mg, 4%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.62; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1701 (NC=O); mp 155–158 °C; $[\alpha]_D^{25} = +152.7$ (c 2.0, CHCl₃) {(R,R)-*anti*-**10**;

$[\alpha]_D^{25} = -145.7$ (c 3.0, CHCl₃) (Found MH⁺, 352.1913; C₂₂H₂₆NO₃ requires 352.1907); the oxazolidin-2-one (R,S)-*syn*-**10** (0.149 g, 65%) as a white crystalline solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.41; ν_{\max} (CHCl₃) cm⁻¹ 1779 (OC=O) and 1705 (NC=O); mp 86–88 °C; $[\alpha]_D^{25} = -120.3$ (c 2.8, CHCl₃); {(S,R)-*syn*-**10**; $[\alpha]_D^{25} = +118.7$ (c 6.0, CHCl₃) (Found MH⁺, 352.1909; C₂₂H₂₆NO₃ requires 352.1907); the oxazolidin-2-one (R,S)-*anti*-**50** (7 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.53; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1751 (CC=O) and 1701 (NC=O); $[\alpha]_D^{25} = -125.4$ (c 1.2, CHCl₃) (Found MNH₄⁺, 365.2069; C₁₉H₂₉N₂O₅ requires 365.2071); and the oxazolidin-2-one (S,S)-*syn*-**50** (0.135 mg, 60%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.35; ν_{\max} (CHCl₃) cm⁻¹ 1791 (OC=O), 1747 (CC=O) and 1699 (NC=O); $[\alpha]_D^{25} = +29.8$ (c 0.95, CHCl₃) (Found M+NH₄⁺, 365.2073; C₁₉H₂₉N₂O₅ requires 365.2071). R_f differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] - (S,S)-*anti*-**10** (R_f 0.62); (R,S)-*syn*-**10** (R_f 0.41); (R,S)-*anti*-**50** (R_f 0.53) and (S,S)-*syn*-**50** (R_f 0.35).

4.67. Parallel kinetic resolution of pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** with 4-phenyl-oxazolidin-2-one (S)-**8** and ethyl oxazolidin-2-one 4-carboxylate (S)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-phenyl-oxazolidin-2-one (S)-**8** (0.106 g, 0.65 mmol), 4-ethyl oxazolidin-2-one 4-carboxylate (S)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(4-chlorophenyl)propanoate (*rac*)-**19** (0.508 g, 1.45 mmol) gave a mixture of two diastereoisomeric oxazolidin-2-ones (R,S)-*syn*- and (S,S)-*anti*-**19** (ratio 95:5:*syn*:*anti*-) and oxazolidin-2-ones (S,S)-*syn*- and (R,S)-*anti*-**51** (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 oxazolidin-2-one (S,S)-*anti*-**45** (6 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.42; $[\alpha]_D^{23} = +161.4$ (c 1.0, CHCl₃) {(R,R)-*anti*-**45**; $[\alpha]_D^{23} = -156.3$ (c 1.3, CHCl₃)}; ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1700 (NC=O) (Found MNH₄⁺(³⁵Cl) 347.1154; C₁₈H₂₀ClN₂O₃ requires MNH₄⁺(³⁵Cl) 347.1157); the oxazolidin-2-one (R,S)-*syn*-**45** (0.124 g, 58%) as a white solid; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.27; mp 142–144 °C; $[\alpha]_D^{23} = -142.4$ (c 1.5, CHCl₃) {(S,R)-*syn*-**45**; $[\alpha]_D^{23} = +144.4$ (c 1.6, CHCl₃)}; ν_{\max} (CHCl₃) cm⁻¹ 1782 (OC=O) and 1700 (NC=O) (Found MNH₄⁺(³⁵Cl) 347.1154; C₁₈H₂₀ClN₂O₃ requires MNH₄⁺(³⁵Cl) 347.1157); the oxazolidin-2-one (R,S)-*anti*-**51** (6 mg, 3%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.31; $[\alpha]_D^{23} = -130.5$ (c 1.2, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1748 (CC=O) and 1700 (NC=O) (Found MNH₄⁺(³⁵Cl) 343.1059; C₁₅H₂₀ClN₂O₅ requires MNH₄⁺(³⁵Cl) 343.1055); and the oxazolidin-2-one (S,S)-*syn*-**51** (0.116 g, 55%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.20; $[\alpha]_D^{23} = +40.0$ (c 1.8, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1790 (OC=O), 1745 (CC=O) and 1700 (NC=O) (Found MNH₄⁺(³⁵Cl) 343.1057; C₁₅H₂₀ClN₂O₅ requires MNH₄⁺(³⁵Cl) 343.1055). R_f differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] - (S,S)-*anti*-**45** (R_f 0.42); (R,S)-*syn*-**45** (R_f 0.27); (R,S)-*anti*-**51** (R_f 0.31) and (S,S)-*syn*-**51** (R_f 0.20).

4.68. Parallel kinetic resolution of pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)propanoate (*rac*)-**6** with 4-phenyl-oxazolidin-2-one (S)-**8** and 4-ethyl oxazolidin-2-one 4-carboxylate (S)-**14**

In the same way as the oxazolidin-2-one (*rac*)-**20**, *n*-butyl lithium (0.58 mL, 2.5 M in hexane, 1.45 mmol), 4-phenyl-oxazolidin-2-one (S)-**8** (0.106 g, 0.65 mmol), 4-ethyl oxazolidin-2-one

4-carboxylate (*S*)-**14** (0.103 g, 0.65 mmol) and pentafluorophenyl 2-(6-methoxynaphthalene-2-yl)-propanoate (*rac*)-**6** (0.574 g, 1.45 mmol), gave a mixture of two diastereoisomeric oxazolidin-2-ones (*R,S*)-*syn*- and (*S,S*)-*anti*-**9** (ratio 95:5:*syn*-:*anti*-) and oxazolidin-2-ones (*S,S*)-*syn*- and (*R,S*)-*anti*-**52** (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 (*S,S*)-*anti*-**9** (10 mg, 4%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.45; {(*R,R*)-*anti*-**9**; $[\alpha]_D^{23} = -164.2$ (c 1.3, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1703 (NC=O) (Found MH⁺, 376.1545; C₂₃H₂₂NO₄⁺ requires MH⁺, 376.1543); (*R,S*)-*syn*-**9** (0.171 g, 70%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.33; {(*S,R*)-*syn*-**9**; $[\alpha]_D^{23} = +166.2$ (c 1.5, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1780 (OC=O) and 1702 (NC=O) (Found MH⁺, 376.1553; C₂₃H₂₂NO₄⁺ requires MH⁺, 376.1543); (*R,S*)-*anti*-**52** (3 mg, 1%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.28; $[\alpha]_D^{23} = -140.3$ (c 0.9, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1782 (OC=O), 1749 (CC=O) and 1700 (NC=O) (Found MH⁺, 372.1445; C₂₀H₂₂NO₆⁺ requires MH⁺, 372.1442); and (*S,S*)-*syn*-**52** (0.118 g, 49%) as a colourless oil; R_f [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] 0.18; $[\alpha]_D^{23} = +55.7$ (c 3.0, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1781 (OC=O), 1754 (CC=O) and 1701 (NC=O) (Found MNH₄⁺, 389.1703; C₂₀H₂₅N₂O₆⁺ requires MNH₄⁺, 389.1707). R_f differences [light petroleum ether (bp 40–60 °C)/diethyl ether (1:1)] - (*S,S*)-*anti*-**9** (R_f 0.45); (*R,S*)-*syn*-**9** (R_f 0.33); (*R,S*)-*anti*-**52** (R_f 0.28) and (*S,S*)-*syn*-**52** (R_f 0.18).

4.69. Hydrolysis of oxazolidin-2-ones

4.69.1. 2-Phenylpropanoic acid (+)-(S)-53

Lithium hydroxide monohydrate (71 mg, 1.69 mmol) was slowly added to a stirred solution of oxazolidin-2-one (*S,R*)-*syn*-**41** (0.25 g, 0.84 mmol) and hydrogen peroxide (0.47 mL, 3.53 M in H₂O, 1.69 mmol) in THF/water (1:1; 5 mL). The reaction mixture was stirred at room temperature for 12 h. The reaction was quenched with water (10 mL) and extracted with dichloromethane (3 × 10 mL). The combined organic layers were dried (over MgSO₄) and evaporated under reduced pressure to give the recovered 4-phenyloxazolidin-2-one (*R*)-**8** (127 mg, 93%) as a white solid; R_f [ethyl acetate/ethanol (9:1)] 0.71; mp 130–133 °C; $[\alpha]_D^{20} = -54.0$ (c 1.0, CHCl₃); ν_{\max} (CHCl₃)/cm⁻¹ 3262 (NH) and 1736 (C=O); δ_H (400 MHz; CDCl₃) 7.41–7.31 (5H, m, 5 × CH; Ph), 5.69 (1H, s, NH), 4.93 (1H, dd, *J* 8.6 and 6.9, PhCHN), 4.72 (1H, t, *J* 8.6, CH_AH_BO) and 4.17 (1H, dd, *J* 8.6 and 6.9, CH_AH_BO); δ_C (100 MHz; CDCl₃) 159.4 (C=O), 139.3 (*i*-C; Ph), 129.2,² 128.9¹ and 126.0² (5 × CH; Ph), 72.5 (CH₂O) and 56.3 (PhCHN) (Found MNH₄⁺, 181.0970; C₉H₁₃N₂O₂ requires 181.0972). The aqueous phase was acidified using HCl (3 M HCl) until the pH = 3, and extracted with diethyl ether (3 × 10 mL). The combined organic phases were dried (over MgSO₄) and evaporated under reduced pressure to give 2-phenylpropanoic acid (*S*)-**53** (113 mg, 90%) as a colourless oil; R_f [light petroleum spirit (bp 40–60 °C)/diethyl ether (1:9)] 0.5; $[\alpha]_D^{23} = +69.5$ (c 8.2, CHCl₃); ν_{\max} (CHCl₃)/cm⁻¹ 1706 (C=O); δ_H (400 MHz; CDCl₃) 7.45–6.98 (5H, m, 5 × CH; Ph), 3.75 (1H, q, *J* 7.2, PhCHCH₃) and 1.50 (3H, d, *J* 7.2, PhCHCH₃); δ_C (100 MHz; CDCl₃) 180.4 (C=O), 139.7 (*i*-C; Ph), 128.7,² 127.6² and 127.4¹ (5 × CH; Ph), 45.3 (PhCHCH₃) and 18.1 (PhCHCH₃) (Found MNH₄⁺, 151.0753; C₉H₁₁NO₂ requires 151.0759).

4.69.2. 2-Phenylpropanoic acid (-)-(R)-53

In the same way as the 2-phenylpropanoic acid (*S*)-**53**, oxazolidin-2-one (*R,S*)-*syn*-**34** (0.2 g, 0.76 mmol), lithium hydroxide monohydrate (64 mg, 1.53 mmol) and hydrogen peroxide (0.43 mL, 3.53 M in H₂O, 1.53 mmol) in THF/water (1:1; 3 mL) gave, the recovered 4-isopropyl-oxazolidin-2-one (*S*)-**13** (76 mg,

78%) as a white solid; R_f [ethyl acetate/ethanol (9:1)] 0.82; mp 71–73 °C; $[\alpha]_D^{20} = +13.0$ (c 2.6, CHCl₃); ν_{\max} (CHCl₃)/cm⁻¹ 3455 (NH) and 1750 (C=O); δ_H (400 MHz; CDCl₃) 7.26 (1H, broad s, NH), 4.34 (1H, t, *J* 8.7, CH_AH_BO), 4.00 (1H, dd, *J* 8.7 and 6.4, CH_AH_BO) and 3.53 (1H, tdd, *J* 8.7, 6.7 and 6.4, CHN); 1.67–1.57 (1H, br octet, *J* ~6.7, CH(CH₃)₂), 0.86 (3H, d, *J* 6.7, CH₃^ACHCH₃^B) and 0.80 (3H, d, *J* 6.7, CH₃^ACHCH₃^B); δ_C (100 MHz, CDCl₃) 160.6 (C=O), 68.4 (CH₂O), 58.2 (CHN), 32.6 (CH(CH₃)₂), 17.7 (CH₃^ACHCH₃^B) and 17.4 (CH₃^ACHCH₃^B) (Found MNH₄⁺, 147.1129; C₉H₁₅N₂O₂ requires MNH₄⁺, 147.1128); and 2-phenylpropanoic acid (*R*)-**53** (97 mg, 85%) as a colourless oil; R_f [light petroleum spirit (bp 40–60 °C)/diethyl ether (1:9)] 0.5; $[\alpha]_D^{23} = -68.5$ (c 2.4, CHCl₃); ν_{\max} (CHCl₃) cm⁻¹ 1710 (C=O) (Found MNH₄⁺, 151.0755; C₉H₁₁NO₂ requires 151.0759).

4.69.3. 2-Phenylpropanoic acid (+)-(S)-53

In the same way as the 2-phenylpropanoic acid (*S*)-**53**, oxazolidin-2-one (*S,S*)-*syn*-**46** (0.12 g, 0.41 mmol), lithium hydroxide monohydrate (34 mg, 0.82 mmol) and hydrogen peroxide (0.23 mL, 3.53 M in H₂O, 0.82 mmol) in THF/water (1:1; 3 mL) gave, the (*S*)-oxazolidin-2-one-4-carboxylic acid (24 mg, 38%) as a white solid; (*S*)-2-oxo-oxazolidine-4-carboxylic acid (24 mg, 38%) as a white solid; R_f (ethyl acetate) 0.10; $[\alpha]_D^{20} = -13.0$ (c 1.2, H₂O); mp 109–112 °C; ν_{\max} (film) 3493–3340 cm⁻¹ (NH and OH), 1741 cm⁻¹ (C=O) and 1652 cm⁻¹ (C=O); δ_H (400 MHz, DMSO-*d*₆) 8.13 (1H, br s, NH), 4.65–4.46 (1H, t, *J* 8.7, CHN), 4.33 (1H, dd, *J* 8.7 and 4.2, CH_AH_BO), 4.27 (1H, dd, *J* 8.7 and 4.2, CH_AH_BO) and 3.35 (1H, br s, OH); δ_C (100.7 MHz, acetone-*d*₆) 171.8 (OC=O), 158.6 (NC=O), 66.7 (CH₂O) and 53.4 (CHN) (Found MNH₄⁺, 149.0557. C₄H₉N₂O₄ requires 149.0557); and 2-phenylpropanoic acid (*R*)-**53** (53 mg, 87%) as a colourless oil; R_f [light petroleum spirit (bp 40–60 °C) / diethyl ether (1:9)] 0.5; $[\alpha]_D^{23} = +69.2$ (c 2.6, CHCl₃) (Found MNH₄⁺, 151.0757; C₉H₁₁NO₂ requires 151.0759).

4.69.4. 2-Phenylbutanoic acid (+)-(S)-54

In the same way as the 2-phenylpropanoic acid (*S*)-**53**, oxazolidin-2-one (*S,R*)-*syn*-**42** (0.2 g, 0.64 mmol), lithium hydroxide monohydrate (53 mg, 1.28 mmol) and hydrogen peroxide (0.36 mL, 3.53 M in H₂O, 1.28 mmol) in THF/water (1:1; 3 mL) gave, the recovered 4-phenyl oxazolidin-2-one (*R*)-**8** (95 mg, 82%) as a white solid; and 2-phenylbutanoic acid (*S*)-**54** (95 mg, 91%) as a colourless oil; $[\alpha]_D^{20} = +65.5$ (c 4.0, CHCl₃); ν_{\max} (CHCl₃)/cm⁻¹ 3295 (OH) and 1719 (C=O); δ_H (400 MHz; CDCl₃) 7.30–7.20 (5H, m, 5 × CH; Ph), 3.43 (1H, t, *J* 7.7; PhCHCO), 2.16–2.03 (1H, ddq, *J* 7.5, 7.5 and 7.4, CH_AH_BCH₃), 1.85–1.79 (1H, ddq, *J* 7.5, 7.5 and 7.4, CH_AH_BCH₃) and 0.90 (3H, t, *J* 7.4, CH₂CH₃); δ_C (100 MHz; CDCl₃) 181.0 (C=O), 138.6 (*i*-C; Ph), 128.8,² 128.2² and 127.6¹ (5 × CH; Ph), 53.6 (PhCHCO), 26.5 (CH₂CH₃) and 12.2 (CH₂CH₃) (Found M⁺, 164.0832; C₁₀H₁₂O₂ requires 164.0832).

4.69.5. 2-Phenyl-3-methylbutanoic acid (+)-(S)-55

In the same way as the 2-phenylpropanoic acid (*S*)-**53**, oxazolidin-2-one (*S,R*)-*syn*-**43** (74% de) (0.15 g, 0.46 mmol), lithium hydroxide monohydrate (39 mg, 0.92 mmol) and hydrogen peroxide (0.26 mL, 3.53 M in H₂O, 0.92 mmol) in THF/water (1:1; 3 mL) gave, the recovered 4-phenyl-oxazolidin-2-one (*R*)-**8** (67 mg, 90%) as a white solid; and 2-phenyl-3-methylbutanoic acid (*S*)-**55** (68 mg, 83%) as a colourless oil; $[\alpha]_D^{20} = +48.4$ (c 2.8, CHCl₃) (74% ee); ν_{\max} (CHCl₃)/cm⁻¹ 1705 (C=O); δ_H (400 MHz; CDCl₃) 7.35–7.23 (5H, m, 5 × CH; Ph), 3.14 (1H, d, *J* 10.6, PhCH), 2.39–2.28 (1H, m, CH₃CHCH₃), 1.08 (3H, d, *J* 6.6, CH₃^ACHCH₃^B) and 0.71 (3H, d, *J* 6.8, CH₃^ACHCH₃^B); δ_C (100 MHz; CDCl₃) 179.8 (C=O), 137.7 (*i*-C; Ph), 128.6,² 128.5² and 127.4¹ (5 × CH; Ph), 59.9 (PhCH), 31.5 (CH₃CHCH₃), 21.4 and 20.1 (2 × CH₃; CH₃^ACHCH₃^B) (Found M⁺, 178.0987; C₁₁H₁₄O₂ requires 178.0988).

4.69.6. 2-(4-Methylphenyl)propanoic acid (+)-(S)-56

In the same way as the 2-phenylpropanoic acid (S)-**53**, oxazolidin-2-one (S,R)-*syn*-**44** (0.2 g, 0.64 mmol), lithium hydroxide monohydrate (53 mg, 1.28 mmol) and hydrogen peroxide (36 mL, 3.53 M in H₂O, 1.28 mmol) in THF/water (1:1; 4 mL) gave, the recovered 4-phenyl-oxazolidin-2-one (R)-**8** (93 mg, 90%) as a white solid; and 2-(4-methylphenyl)propanoic acid (S)-**56** (93 mg, 89%) as a white solid; mp 59–60 °C; $[\alpha]_D^{20} = +64.8$ (c 4.0, CHCl₃); ν_{\max} (CHCl₃)/cm⁻¹ 1710 (C=O); δ_H (400 MHz; CDCl₃) 7.14 (2H, d, J 7.9, 2 × CH; Ar), 7.08 (2H, d, J 7.9, 2 × CH; Ar), 3.69 (1H, q, J 7.1, ArCHCH₃), 2.31 (3H, s, Me; Ar) and 1.47 (3H, d, J 7.1, ArCHCH₃); δ_C (100 MHz; CDCl₃) 180.1 (C=O), 137.1 and 136.9 (2 × *i*-C; Ar), 129.4² and 127.6² (4 × CH; Ar), 44.9 (ArCHCH₃), 21.1 (CH₃; Ar) and 18.2 (ArCHCH₃) (Found MNH₄⁺, 182.1175. C₁₀H₁₆NO₂ requires MNH₄⁺, 182.1176).

4.69.7. 2-(4-Isobutylphenyl)propanoic acid (+)-(S)-57

In the same way as the 2-phenylpropanoic acid (S)-**53**, oxazolidin-2-one (S,R)-*syn*-**10** (0.2 g, 0.57 mmol), lithium hydroxide monohydrate (48 mg, 1.14 mmol) and hydrogen peroxide (42 mg, 3.53 M in H₂O, 1.48 mmol) in THF/water (1:1; 3 mL) gave, the recovered 4-phenyl-oxazolidin-2-one (R)-**8** (81 mg, 87%) as a white solid; and 2-(4-isobutylphenyl)propanoic acid (S)-**57** (108 mg, 92%) as a colourless oil; $[\alpha]_D^{20} = +58.2$ (c 3.6, CHCl₃); δ_H (400 MHz, CDCl₃) 7.21 (2H, dt, J 7.9 and 2.1, 2 × CH; Ar); 7.11 (2H, dt, J 7.9 and 2.1, 2 × CH; Ar), 3.70 (1H, q, J 7.2, ArCHCH₃), 2.41 (2H, d, J 7.2, CH₂Ar), 1.90–1.75 (1H, br septet, J ~6.8, CH(CH₃)₂), 1.47 (3H, d, J 7.2, ArCHCH₃) and 0.87 (6H, d, J 6.5, 2 × CH₃; (CH₃)₂CH); δ_C (100 MHz, CDCl₃) 180.3 (C=O), 140.9 (*i*-C; Ar), 137.0 (*i*-C; Ar), 129.4² and 127.3² (2 × CH; Ar), 45.1 ((CH₃)₂CH), 44.8 (ArCHCH₃), 30.2 (ArCH₂), 22.4² ((CH₃)₂CH) and 18.1 (ArCHCH₃) (Found M⁺, 206.1270; C₁₃H₁₈O₂ requires 206.1268).

4.69.8. 2-(4-Chlorophenyl)propanoic acid (+)-(S)-58

In the same way as the 2-phenylpropanoic acid (S)-**53**, oxazolidin-2-one (S,R)-*syn*-**45** (0.2 g, 0.61 mmol), lithium hydroxide monohydrate (51 mg, 1.21 mmol) and hydrogen peroxide (0.34 mL, 3.53 M in H₂O, 1.21 mmol) in THF/water (1:1; 4 mL) gave, the recovered 4-phenyl-oxazolidin-2-one (R)-**8** (85 mg, 86%) as a white solid; and 2-(4-chlorophenyl)propanoic acid (S)-**58** (96 mg, 85%) as a white solid mp 49–53 °C; $[\alpha]_D^{20} = +48.5$ (c 4.0, CHCl₃); ν_{\max} (CHCl₃)/cm⁻¹ 1710 (C=O); δ_H (400 MHz; CDCl₃) 7.30 (2H, dt, J 8.8 and 2.2, 2 × CH; Ar), 7.25 (2H, dt, J 8.8 and 2.2, 2 × CH; Ar), 3.72 (1H, q, J 7.2, ArCHCH₃) and 1.50 (3H, d, J 7.2, ArCHCH₃); δ_C (100 MHz; CDCl₃) 178.9 (C=O), 138.2 and 133.3 (2 × *i*-C; Ar), 129.0² and 128.8² (4 × CH; Ar), 44.5 (ArCHCH₃) and 18.1 (ArCHCH₃) (Found MNH₄⁺, 202.0636. C₉H₁₃ClO₂ requires MNH₄⁺, 202.0629).

4.69.9. 2-(6-Methoxynaphthalene-2-yl)propanoic acid (+)-(S)-59

In the same way as the 2-phenylpropanoic acid (S)-**53**, oxazolidin-2-one (S,R)-*syn*-**9** (0.2 g, 0.53 mmol), lithium hydroxide monohydrate (45 mg, 1.06 mmol) and hydrogen peroxide (0.30 mL, 3.53 M in H₂O, 1.06 mmol) in THF/water (1:1; 5 mL) gave, the recovered 4-phenyl-oxazolidin-2-one (R)-**8** (77 mg, 89%) as a white solid; and 2-(6-methoxynaphthalene-2-yl)propanoic acid (S)-**59** (109 mg, 90%) as a white solid; R_f [diethyl ether] 0.56; mp 151–153 °C; $[\alpha]_D^{20} = +93.1$ (c 4.2, CHCl₃); ν_{\max} (CHCl₃)/cm⁻¹ 1710 (C=O); δ_H (400 MHz; CDCl₃) 7.70 (2H, dd, J 8.4 and 2.8, 2 × CH; Ph), 7.68 (1H, s, 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, CH₃O), 3.88 (1H, q, J

7.0, ArCHCH₃) and 1.59 (3H, d, J 7.0, ArCHCH₃); δ_C (100 MHz; CDCl₃) 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 (CH₃O), 45.2 (ArCHCH₃) and 18.1 (ArCHCH₃); *m/z* 230 (60%, M⁺) and 185 (100, ArCHCH₃) (Found M⁺, 230.0906; C₁₄H₁₄O₃ requires 230.0904).

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