Asymmetric synthesis of cyclopropanes and dihydrofurans based on phosphine oxide chemistry†

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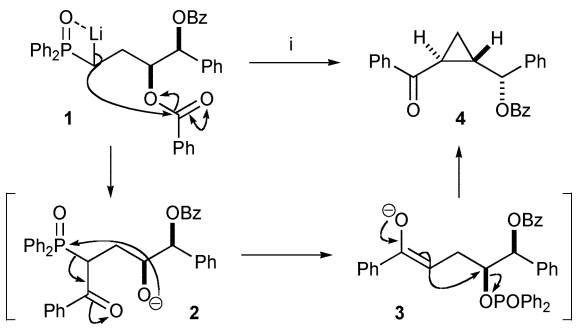
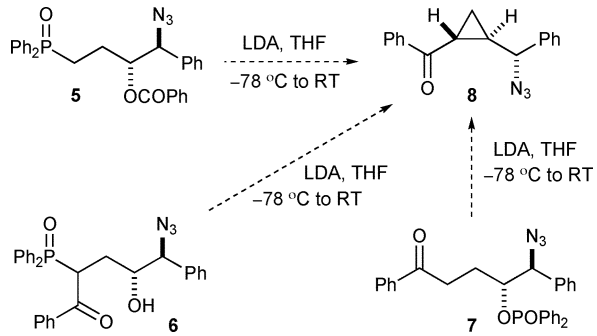
The asymmetric synthesis of γ-azido *trans*-cyclopropyl ketones is accomplished *via* a short, simple and efficient sequence. The cyclopropanation step is achieved by an intramolecular nucleophilic ring closure, with a diphenylphosphinate leaving group, to give *trans*-cyclopropane exclusively. β-Keto-diphenylphosphine oxides cyclise to form optically active dihydrofurans. All possible diastereoisomers of dihydrofurans can be prepared selectively starting from the same olefin.

Although the cyclopropane ring is a highly strained structure, it is found in a wide variety of naturally occurring compounds including terpenes, pheromones, fatty acid metabolites and unusual amino acids.[1,2] The rigidity of the three-membered ring makes it an appealing structural unit for the preparation of molecules with defined orientation of pendant functional groups. The stereocontrolled synthesis of cyclopropanes has therefore been the centre of much research effort.[3] The area of homogeneous metal-catalysed asymmetric addition of carbenes to olefins has been explored vigorously since the first examples were reported by Noyori and co-workers.[4,5] Methods involving palladium,[6] copper,[7–11] cobalt,[12–14] ruthenium,[14] and rhodium[11,15–17] have been reported. Asymmetric versions of the Simmons–Smith reaction[18–21] have also been used extensively in synthesis.[22] Michael-induced ring closure reactions to produce enantio-enriched cyclopropanes have also been studied using chloro-allyl amides,[23,24] sulfur ylides,[25–27] sulfoxonium and sulfonium ylides,[28–30] sulfoximines,[31] nitrogen ylides,[32,33] phosphorus ylides,[34,35] and phosphonates.[36] Cascade ring closing reactions involving phosphorus transfer, to generate both nucleophile and leaving group, have yielded cyclopropanes generally with high stereospecificity and selectivity. Phosphine oxides,[37–40] phosphonates,[10,41,42] and phosphonium salts[43] have been employed in this manner. We have previously reported that lithiated γ-benzoyloxy phosphine oxide **1** (Scheme 1) undergoes a three-step cascade reaction to produce *trans*-cyclopropane **4** in high yield without loss of stereochemical information.[38]



Scheme 1 *Reagents and conditions*: i) LDA, THF, −78 to 0 °C, 95% (>95% ee).



Scheme 2

Subjecting independently synthesised intermediates **2** and **3** to the same reaction conditions also produced cyclopropyl ketone **4**. Hence, three different precursors of the same cyclopropyl ketone product are possible. To determine if this approach to cyclopropyl ketones is a general synthetic concept we decided to explore the reaction in more detail. In particular, we were interested in introducing an amino group at the benzylic position to give us protected γ-amino *trans*-cyclopropane ketones **8** (Scheme 2). Moreover, we decided to invert the stereochemistry at the benzylic position to see what consequences this would have for the reaction outcome. Rather than a free amine we chose to use an azide substituent, which could be transformed into the desired amine equivalent at a later stage by a catalytic hydrogenation or a Staudinger reaction.[44]

We set out to synthesise three cyclopropane precursors **5**, **6** and **7** relying on chemistry developed by Chang and Sharpless.[45] They have demonstrated that styrenes **9** (Scheme 3) can be dihydroxylated and the product diols **10** converted to cyclic carbonates **11**. Regioselective benzylic ring opening with azide then produces *anti*-azido alcohols **12**.

The synthesis of cyclopropane precursor **5** (Scheme 4) was accomplished from the optically active diol[46] **13**, which was converted to cyclic carbonate **14** by treatment with 1,1′-carbonyldiimidazole in dichloromethane. Ring opening of the cyclic carbonate produced the desired regioisomer **15** exclusively and benzoylation of the alcohol produced cyclopropane precursor **5**.

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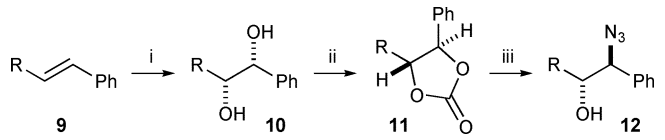
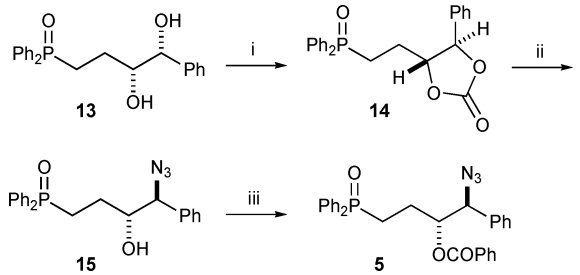


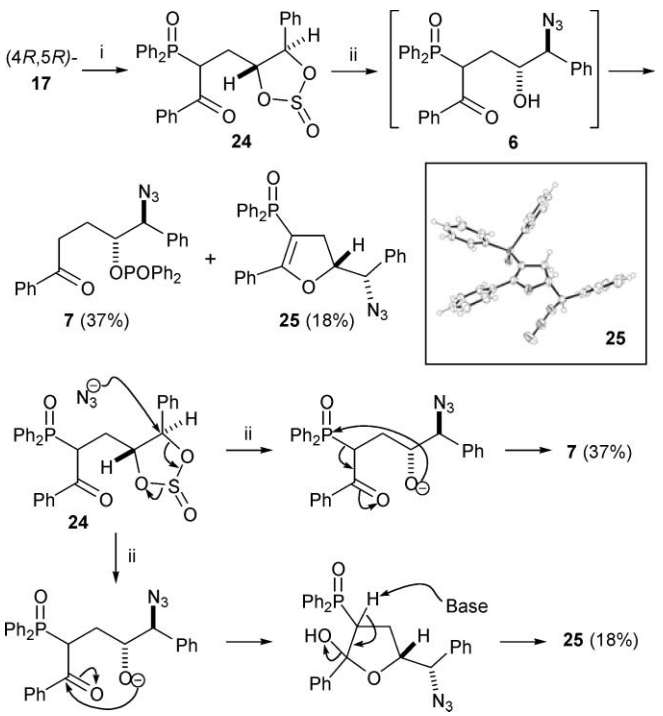
Scheme 3 i) Asymmetric dihydroxylation; *reagents and conditions*: ii) $(EtO)_2CO$, NaOH; iii) NaN_3, H_2O, DMF.



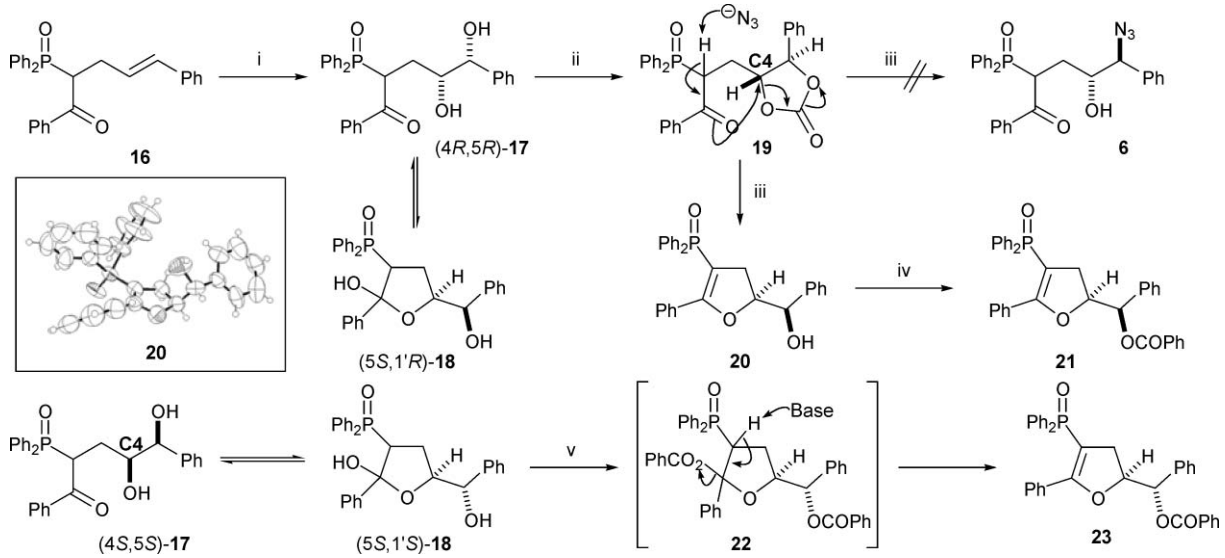
Scheme 4 *Reagents and conditions*: i) 1,1′-carbonyldiimidazole, CH_2Cl_2, 97%; ii) NaN_3, H_2O, DMF, 110 °C, 87%; iii) PhCOCl, DMAP, Et_3N, CH_2Cl_2, 73%.

The synthesis of cyclopropane precursor **6** was attempted by the same method starting from phosphine oxide[47] **16** (Scheme 5). Sharpless asymmetric dihydroxylation of olefin **16** produced a mixture of the open-chain diol (4*R*,5*R*)-**17** and the cyclic hemiketal (5*S*,1′*R*)-**18**, which was treated with 1,1′-carbonyldiimidazole in dichloromethane to give cyclic carbonate **19**. However, when ring opening was attempted none of the desired azide **6** was obtained. Instead, dihydrofuran **20** was formed as the only product in high yield. We suspected that the dihydrofuran was formed *via* intramolecular ring opening of the cyclic carbonate by the carbonyl oxygen with inversion of configuration at C4. An X-ray crystal structure of dihydrofuran **20** confirmed that this was indeed the case[48] (Scheme 5). Interestingly, when bis-benzoylation of diol[38] (4*S*,5*S*)-**17** was attempted dihydrofuran **23** was isolated. However, in this instance the dihydrofuran was formed with retention of configuration at C4 by dehydration of cyclic ketal (5*S*,1′*S*)-**18**, which was confirmed by comparison of NMR data of dihydrofurans **21** and **23**.

Ring opening of cyclic carbonates according to the procedure by Chang and Sharpless[45] requires forcing conditions (110 °C for 48 h) and we hoped that we could suppress dihydrofuran formation by using milder conditions. Hence, the cyclic carbonate was replaced with a more reactive cyclic sulfite. Treatment of diol (4*R*,5*R*)-**17** (Scheme 6) with thionyl chloride in dichloromethane produced the cyclic sulfite **24**. Treatment of the cyclic sulfite with



Scheme 6 *Reagents and conditions*: i) $SOCl_2$, pyridine, CH_2Cl_2, 47%; ii) NaN_3, DMF, 60 °C. Inset: X-ray crystal structure of azide **25** with ellipsoids at 50% probability.

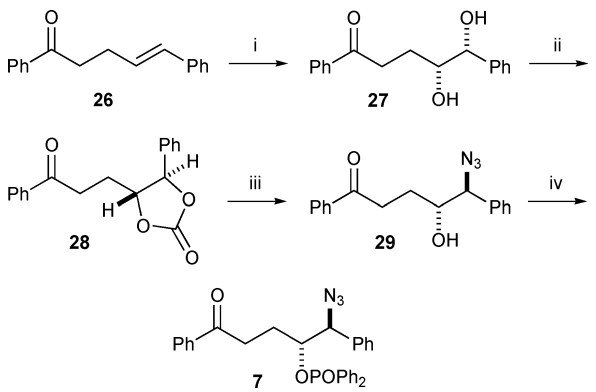


Scheme 5 *Reagents and conditions*: i) AD-mix-β, 74%; ii) 1,1′-carbonyldiimidazole, CH_2Cl_2, 98% (>95% ee); iii) NaN_3, H_2O, DMF, 110 °C, 89%; iv) PhCOCl, pyridine, 95%; v) PhCOCl, DMAP, Et_3N, CH_2Cl_2, 67%. Inset: X-ray crystal structure of alcohol **20** with ellipsoids at 50% probability.

sodium azide in DMF at 60 °C did indeed produce the desired *anti*-azido alcohol **6**. However, under the reaction conditions the reaction did not stop but proceeded to give a mixture of ketone **7** and azido dihydrofuran **25** as well as a small amount (6%) of the previously observed dihydrofuran **20**. Ketone **7** and dihydrofuran **25** can be formed after ring opening of the cyclic sulfite by either phosphinoyl transfer or dehydration of the cyclic ketal (Scheme 6). An X-ray crystal structure of dihydrofuran **25** confirmed the proposed stereochemistry.[48] Ketone **7** is one of the desired cyclopropane precursors and was spectroscopically identical to the same compound synthesised by a different method (*vide infra*).

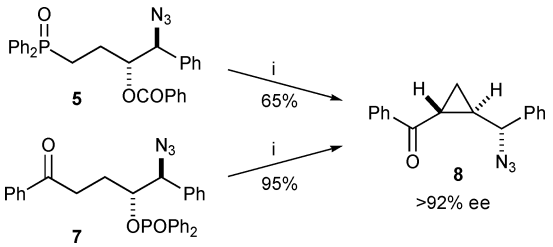
In the light of these experiments we concluded that it was unlikely that cyclopropane precursor **6** would provide an easy entry to cyclopropyl ketones and the approach was abandoned in favour of the remaining two cyclopropane precursors **5** and **7**. However, the discovery that dihydrofurans such as **20** and **23** (Scheme 5) can be obtained as single diastereoisomers is of some interest and will be discussed in more detail below.

Olefin[47] **26** was the starting point for the synthesis of the remaining cyclopropane precursor **7** (Scheme 7). Asymmetric dihydroxylation of olefin **26** gave diol **27** in excellent enantiomeric excess. Conversion of diol **27** to cyclic carbonate **28** using 1,1′-carbonyldiimidazole in dichloromethane followed by regioselective ring opening of the cyclic carbonate with sodium azide gave exclusively the desired regioisomer **29**. Phosphinoylation of alcohol **29** with diphenylphosphinoyl chloride in pyridine produced cyclopropane precursor **7**.



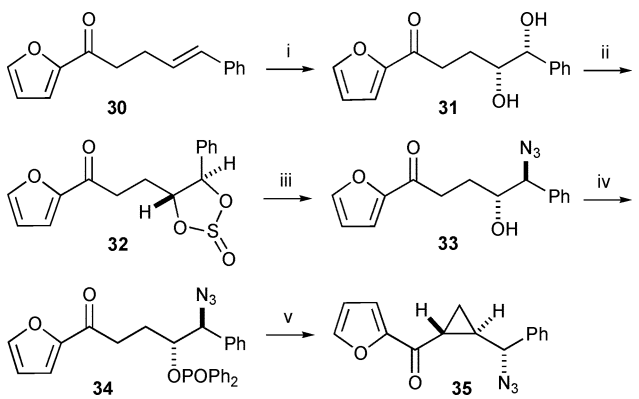
Scheme 7 *Reagents and conditions*: i) AD-mix-β, 94% (>95% ee); ii) 1,1′-carbonyldiimidazole, CH_2Cl_2, 92%; iii) NaN_3, DMF, H_2O, 110 °C, 92%; iv) Ph_2POCl, pyridine, 83%.

Cyclopropane precursors **5** and **7** were treated with LDA in THF at −78 °C and slowly allowed to warm to room temperature (Scheme 8). Phosphine oxide **5** underwent the three-step cascade reaction to give the desired cyclopropane **8** and ketone **7** produced the desired cyclopropane **8** by anionic ring closure to give the target cyclopropane. No *cis*-cyclopropane product was observed, presumably because the two substituents on the forming ring prefer to be *anti* in the transition state. In both cases the enantiomeric excess of cyclopropane **8** was determined to be >92% by NMR of Mosher's amide derivatives. The free amine necessary for this purpose was obtained by converting azide **8** to the amine by a Staudinger reaction (PPh_3, THF, H_2O).[44]



Scheme 8 *Reagents and conditions*: i) LDA, THF, −78 °C to room temperature.

From the results above it is evident that cyclopropane precursor **7** is ideally suited for our purpose. The synthetic sequence is short with an excellent overall yield (five steps, 62%, >92% ee) from easily prepared starting materials. Moreover, all experimental procedures are simple and inexpensive and the products are easily purified using conventional techniques. The method was applied to the synthesis of cyclopropane **35** starting from olefin[49] **30** (Scheme 9). Asymmetric dihydroxylation of olefin **30** gave diol **31** in excellent enantiomeric excess. Owing to the sensitive furan we anticipated that the Sharpless procedure for ring opening cyclic carbonates with sodium azide might prove too harsh. Hence, we decided to convert diol **31** to a cyclic sulfite, which could be ring opened using milder conditions. Reaction of diol **31** with thionyl chloride and pyridine in dichloromethane gave cyclic sulfite **32**. Ring opening of the cyclic sulfite using sodium azide in DMF at 60 °C produced the desired *anti*-azido alcohol **33** as a single regioisomer. Treatment of alcohol **33** with diphenylphosphinoyl chloride, triethylamine and DMAP in dichloromethane produced cyclopropane precursor **34**. Cyclopropanation of phosphinate **34** using LDA in THF gave the target cyclopropane **35** albeit in a low yield. However, no optimisation was undertaken and we expect that the final cyclopropanation step could be improved. The enantiomeric excess of cyclopropane **35** was determined to be >96% by NMR analysis of Mosher's amide derivatives made from the free amine obtained by a Staudinger reaction.[44]



Scheme 9 *Reagents and conditions*: i) AD-mix-β, 65% (>93% ee); ii) $SOCl_2$, pyridine, CH_2Cl_2, 99%; iii) NaN_3, DMF, 60 °C, 84%; iv) Ph_2POCl, Et_3N, DMAP, CH_2Cl_2, 88%; v) LDA, THF, −78 °C to room temperature, 30% (>96% ee).

As described above (Scheme 5) the treatment of cyclic carbonate **19** with sodium azide resulted in the formation of dihydrofuran **20** with inversion of stereochemistry at the C4 position. Moreover, we found that it was possible to prepare the diastereomeric

Table 1 Synthesis of dihydrofuran **20** from cyclic carbonate **19** (Scheme 5)

Entry	Base	Solvent	Time	Temp/°C	Conversion (%)	Isolated yield (%)
1	NaN_3	DMF	48 h	110	100[a]	89
2	NaH	DMF	19 h	110	100[a]	80
3[b]	—	DMF-d^7	15 d	70	62[c]	—
4[b]	—	CD_3CN	48 h	70	0[c]	—
5[b]	Et_3N	CD_3CN	19 d	70	66[c]	—
6[b]	DBU	CD_3CN	15 h	rt	100[c]	—
7	DBU	CH_2Cl_2	5 h	rt	100[a]	85

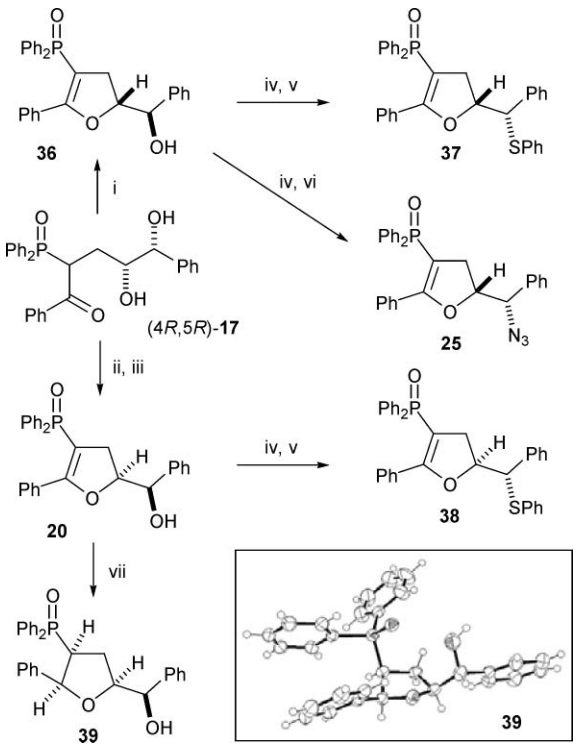
[a] Monitored by TLC. [b] Experiments performed in NMR tubes containing 5 mg of **19** in 0.5 cm^3 solvent, heated oil bath. [c] Monitored by ^1H NMR.

dihydrofuran **23** with retention of configuration at C4 starting from diol (4*S*,5*S*)-**17**. Because the stereochemistry of the diol is controlled in the asymmetric dihydroxylation this potentially gives a method for the selective synthesis of all four possible dihydrofuran diastereoisomers starting from the same olefin. In addition, the synthetic route makes it possible to vary the C4- and C1′-substituents considerably. Hence, a wide range of dihydrofurans should be readily accessible by this approach. The cyclisation of cyclic carbonate **19** to give dihydrofuran **20** was studied using a range of conditions (Table 1).

As it can be seen from Table 1 the cyclisation will occur even in neat DMF (entry 3). However, dimethylamine, a common impurity in DMF, may be acting as a base to accelerate the cyclisation. For comparison the same reaction conditions were examined with acetonitrile as the reaction medium (entry 4), which resulted in no detectable dihydrofuran formation by NMR. NaH (entry 2), triethylamine (entry 5) and DBU (entries 6 and 7) produced the desired dihydrofuran **20** in moderate to high yields. However conditions using DBU in dichloromethane (entry 7) proved to be superior due to ease of handling, short reaction time and simple purification to give dihydrofuran **20** in high yield. Optimised conditions for the synthesis of dihydrofuran diastereoisomer **36** and **20** starting from diol (4*R*,5*R*)-**17** (Scheme 10) were identified. Treatment of the diol with a small amount of thionyl chloride in methanol resulted in the rapid formation of dihydrofuran **36**. Alternatively, conversion to the cyclic carbonate followed by addition of DBU *in situ* gives dihydrofuran **20**.

The stereochemistry of dihydrofuran **36** was confirmed by conversion to the previously described azide **25** (Scheme 6) by mesylation of the C1′-alcohol and displacement of the mesylate with sodium azide. This proved to be a simple method for derivatisation at the C1′-position, which was demonstrated by the synthesis of sulfides **37** and **38** *via* displacement of the mesylates with benzene thiolate. The possibility of reducing the dihydrofurans to give tetrahydrofurans was also investigated. We anticipated that good facial selectivity could be achieved due to the substituent at the C2-position. Catalytic hydrogenation of dihydrofuran **20** with Pearlman's catalyst in a mixture of methanol and acetic acid at atmospheric pressure as expected gave exclusive reduction from the face opposite the C2-substituent. The stereochemistry of tetrahydrofuran **39** was confirmed by X-ray crystallography[48] (Scheme 10).

Similar methods for the synthesis of dihydrofurans have been reported for the cyclisation of the oxygens of stabilised enolates unto iodonium ions,[50–55] seleniranium ions,[56–58] Michael acceptors.[59–61] and epoxides.[62] However, unlike the previously reported strategies the present method has the advantage that it is ideally suited for making optically active dihydrofurans. We are currently seeking to extend this methodology to the synthesis of other substituted heterocycles.



Scheme 10 *Reagents and conditions*: i) $SOCl_2$, MeOH, 63%; ii) 1,1′-carbonyldiimidazole, CH_2Cl_2; iii) DBU, 63% (2 steps); iv) MsCl, Et_3N, CH_2Cl_2; v) PhSH, NaH, THF, **37**: 45% (2 steps), **38**: 40% (2 steps); vi) NaN_3, DMF, 60 °C, 62% (2 steps); vii) $Pd(OH)_2/C$, H_2, MeOH, AcOH, 58%. Inset: X-ray crystal structure of THF **39** with ellipsoids at 50% probability.

Acknowledgements

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48 Crystal data for **20**. $C_{29}H_{25}O_3P$, $M = 452.46$, hexagonal, space group $P6_1$, $a = 11.1634(1)$, $b = 11.1634(1)$, $c = 33.8352(5)$ Å, $\alpha = 90$, $\beta = 90$, $\gamma = 120°$, $U = 3651.68(7)$ Å^3, $Z = 6$, μ(Mo-Kα) = 0.141 mm^{-1}, 16946 reflections collected at 200(2) K using an Oxford Cryosystems Cryostream cooling apparatus, 3114 ($R_{int} = 0.046$); $R_1 = 0.058$, $wR_2 = 0.142$ [$I > 2\sigma(I)$], absolute structure parameter −0.02(17).
Crystal data for **25**. $C_{29}H_{24}N_3O_2P$, $M = 477.48$, triclinic, space group $P1$, $a = 8.4713(3)$, $b = 8.5479(3)$, $c = 17.4621(6)$ Å, $\alpha = 83.267(2)$, $\beta = 83.693(2)$, $\gamma = 74.518(2)°$, $U = 1206.09(7)$ Å^3, $Z = 2$, μ(Mo-Kα) = 0.146 mm^{-1}, 12870 reflections collected at 180(2) K using an Oxford Cryosystems Cryostream cooling apparatus, 8246 ($R_{int} = 0.068$); $R_1 = 0.070$, $wR_2 = 0.165$ [$I > 2\sigma(I)$], absolute structure parameter 0.01(13).
Crystal data for **39**. $C_{29}H_{27}O_3P$, $M = 454.48$, orthorhombic, space group $P2_12_12_1$, $a = 5.9419(1)$, $b = 14.3610(3)$, $c = 26.6529(7)$ Å, $U = 2274.34(9)$ Å^3, $Z = 4$, μ(Mo-Kα) = 0.151 mm^{-1}, 14297 reflections collected at 180(2) K using an Oxford Cryosystems Cryostream cooling apparatus, 2966 ($R_{int} = 0.095$); $R_1 = 0.046$, $wR_2 = 0.103$ [$I > 2\sigma(I)$], absolute structure parameter −0.02(13).
CCDC reference numbers 297304–297306. For crystallographic data in CIF or other electronic format see DOI: 10.1039/b606874j.
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