

Synthesis and structure of stereoisomers of 3,4-benzo-5,10-diphenyl-1,3-diaza-7-oxa-6-phosphabicyclo[4.3.1]decane-2,6-dione

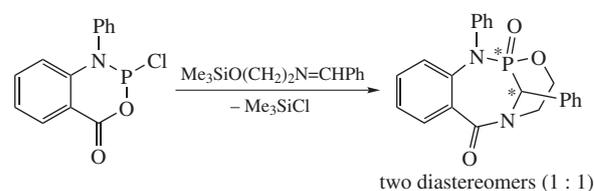
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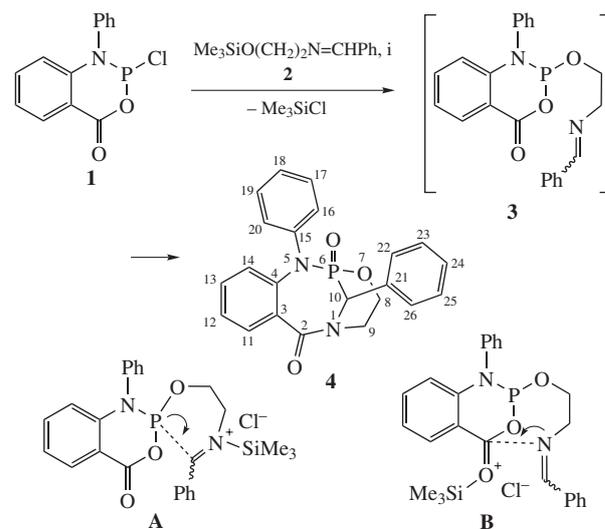
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A mild approach to the synthesis of the title cage amino-phosphonate has been developed based on the intramolecular cyclization of 2-(2-benzylideneaminoethoxy)-1-phenylbenzo[*e*]-1,3,2-aza-oxaphosphorin-4-one obtained from 1-phenyl-2-chlorobenzo[*e*]-1,3,2-aza-oxaphosphorin-4-one and (2-benzylideneaminoethoxy)trimethylsilane. The structure of isolated diastereomers of the product was determined by NMR spectroscopy and single crystal X-ray diffraction analysis.



α -Aminophosphonic acids have useful properties such as biological activity¹ and capability to form complexes with several metals.² The Kabachnik–Fields and Pudovik reactions³ provide versatile and popular approach to the synthesis of these compounds. Previously, we suggested to obtain 1-aminophosphonic acid esters by ring expansion reaction of 2-substituted benzo[*e*]-1,2,3-dioxaphosphorin-4-ones, containing the P–O–C(O) high-energy moiety, with imines. This approach resulted in only one of the two possible diastereomers of 3,4-dihydrobenzo[*f*]-1,4,2-oxazaphosphepine-2,5-diones with high selectivity.⁴ The intramolecular version of this reaction carried out with 2-(2-aryl-methylideneaminophenoxy)benzo[*e*]-1,3,2-dioxaphosphorin-4-ones, containing a C=N exocyclic bond, allowed us to obtain cage α -aminophosphonates with high stereoselectivity.⁵

In this study, we successfully implemented a new modification of this intramolecular approach to the synthesis of cage amino-phosphonates using the reaction of 2-chloro-1-phenylbenzo[*e*]-1,3,2-aza-oxaphosphorin-4-one **1** with (2-benzylideneaminoethoxy)trimethylsilane **2** (Scheme 1).[†] The formation of P^{III} derivative, 2-(2-benzylideneaminoethoxy)-1-phenylbenzo[*e*]-1,3,2-aza-oxaphosphorin-4-one **3**, was detected by ³¹P NMR (δ 120.3 ppm). Compound **3** was unstable under the reaction conditions and was immediately converted into the bicyclic cage amino-phosphonate **4** as 1:1 diastereomeric mixture.[‡] Lack in stability for compound **3** as compared to 2-(2-benzylidene-



Scheme 1 Reagents and conditions: i, CH₂Cl₂, 25 °C, 4 h.

[‡] 3,4-Benzo-5,10-diphenyl-1,5-diaza-7-oxa-6-phosphabicyclo[4.3.1]-decane-2,6-dione **4**. A solution of 2-chloro-1-phenylbenzo[*e*]-1,3,2-aza-oxaphosphorin-4-one **1** (1.53 g, 5.5 mmol) in dichloromethane (10 ml) was added dropwise to the solution of the trimethylsilane **2** in dichloromethane (20 ml) at 20 °C under stirring for 5 min. The reaction mixture that immediately acquired a bright crimson colour was left overnight. Then volatile components were removed *in vacuo* (12 Torr). The residue was treated with diethyl ether and dried *in vacuo* to give a light red powder of product **4** as 1:1 mixture of diastereomers *d*₁ and *d*₂. Yield 1.82 g (85%). MS (EI), *m/z* (%): 390 [M]⁺⁺ (83.9), 362 [M–C₂H₄]⁺ (19.4), 313 [M–C₆H₅]⁺ (66.8), 299 [M–P(O)OC₂H₄]⁺ (18.6), 285 [C₂₀H₁₇N₂]⁺ (2.9), 195 [C₈H₈N₂O₂P]⁺ (100.0), 167 [C₇H₈N₂OP]⁺ (58.9), 91 [C₆H₅CH₂]⁺ (23.5), 77 [C₆H₅]⁺ (28.4). Found (%): C, 67.28; H, 5.21; N, 7.09; P, 7.88. Calc. for C₂₂H₁₉N₂O₃P (%): C, 67.69; H, 4.87; N, 7.18; P, 7.95. Air-stable individual diastereomers **4**(*d*₁) [0.85 g, mp 263–264 °C (decomp.), δ _P: 15.3 ppm] and **4**(*d*₂) [0.74 g, mp 259–260 °C (decomp.), δ _P: 18.5 ppm] were sequentially isolated by chromatography on silica gel with chloroform and then chloroform–acetonitrile (5:1).

[†] (2-Benzylideneaminoethoxy)trimethylsilane **2**. A solution of chloro-trimethylsilane (8.1 g, 74.7 mmol) in diethyl ether (20 ml) was added dropwise to a mixture of 2-(benzylideneamino)ethanol (11.1 g, 74.5 mmol) and triethylamine (7.6 g, 75.3 mmol) in dry diethyl ether (100 ml) at 20 °C under an argon atmosphere, and the mixture was left overnight. The formed precipitate was filtered off, the volatiles from the filtrate were removed *in vacuo* and the residue was distilled. Yield 12.4 g (75%), light-yellow liquid, bp 171–173 °C (15 Torr), n_D^{20} = 1.5097. ¹H NMR (600 MHz, CDCl₃) δ : 0.11 (s, 9H, Me₃Si), 3.75 (t, 2H, NCH₂, ³J 5.8 Hz), 3.91 (t, 2H, OCH₂, ³J 5.8 Hz), 7.41 (m, 3H, *m*-H, *p*-H), 7.74 (m, 2H, *o*-H), 8.28 (s, 1H, =CH). Compound **2** was previously obtained as a crude material by different procedure.⁸

aminophenoxy)benzo[*e*]-1,3,2-dioxaphosphorin-4-one with a similar structure⁵ may be explained by the effect of chlorotrimethylsilane formed in the reaction, namely the activation of both the imine moiety (see Scheme 1, structure **A**) and the carbonyl group of **3** (see Scheme 1, structure **B**). Similar activation in polar acetonitrile by an equimolar amount or excess of chlorotrimethylsilane was demonstrated earlier for diethyl phosphite⁶ or triethyl phosphite⁷ addition to carbonyl compounds and imines.

The structure of the isolated diastereomers of product **4** was determined by NMR spectroscopy.⁸ The bicyclodecane moiety was deduced from the spin–spin coupling constants in the ¹³C and ¹³C-¹H NMR spectra for the carbon atoms located at a distance of 1–4 bonds from the phosphorus atom. There is a noticeable difference for the chemical shifts of the H-8AB and H-9AB protons, as well as for the C³, C⁴, C⁹, C¹⁰, C¹⁴, C²¹ and

⁸ Compound **4**(*d*₁). ¹H NMR (400 MHz, CDCl₃) δ: 3.24 (ddd, 1H, NC⁹H_X, ²J_{HH} 14.8, 12.9, 3.7 Hz), 4.02 (ddd, 1H, POC⁸H_B, ³J_{POCH} 19.2 Hz, ²J_{HH} 11.3, 3.7 Hz), 4.29–4.31 (m, 2H, POC⁸H_A, NC⁹H_A), 5.48 (d, 1H, PCH, ²J_{PCH} 12.1 Hz), 7.16 (m, 1H, H-12, ³J_{HH} 7.9, 7.3 Hz), 7.30 and 7.39–7.44 (two m, 11H, H-11, H-13, H-14, H-16 to H-20, H-22, H-26, H-24), 7.91 (m, 2H, H-23, H-25, ³J_{HH} 8.0, 7.6 Hz). ¹³C NMR (100.6 MHz, CDCl₃, hereinafter multiplicity for signal in ¹³C-¹H NMR is given after a slash) δ: 172.30 (m/s, C², ³J_{HC¹¹CC} 3.8 Hz, ³J_{HC¹⁴CC} 3.8–4.0 Hz), 132.71 (br. dd/s, C³, ³J_{HC¹²CC} 8.4 Hz, ³J_{HC¹⁴CC} 5.5 Hz), 141.25 (m/d, C⁴, ³J_{HC¹³CC} 11.5 Hz, ³J_{HC¹¹CC} 8.3 Hz, ²J_{PNC} 3.7 Hz, ²J_{HC¹⁴C} 2.0 Hz), 71.62 (dddd/d, C⁸, ¹J_{H_AC} 150.3 Hz, ¹J_{H_BC} 152.0 Hz, ²J_{POC} 11.1 Hz, ²J_{H_AC⁸C} 4.8 Hz, ²J_{H_BC⁸C} 2.1 Hz), 40.52 (ddd/d, C⁹, ¹J_{H_AC} 142.1 Hz, ¹J_{H_BC} 144.1 Hz, ³J_{POCC} 3.5 Hz, ²J_{HCC} 3.5 Hz), 55.74 (ddd/d, C¹⁰, ¹J_{HC} 136.4 Hz, ¹J_{PC} 126.0 Hz, ¹J_{H_AC⁹NC} 4.8 Hz, ³J_{HC^{22,26}CC} 4.8 Hz), 128.95 (br. dd/d, C¹¹, ⁴J_{PNC³C³C} 3.3 Hz), 128.04 (br. dd/d, C¹², ¹J_{HC} 164.8 Hz, ³J_{HC¹⁴CC} 8.2 Hz, ⁵J_{PNC³C³C¹¹C} 1.3 Hz), 132.83 (br. dd/s, C¹³, ¹J_{HC} 162.2 Hz, ³J_{HC¹³CC} 8.5 Hz), 127.54 (br. dd/d, C¹⁴, ¹J_{HC} 164.2 Hz, ³J_{HC¹²CC} 7.6 Hz, ³J_{PNC} 1.6 Hz), 141.99 (ttd/d, C¹⁵, ³J_{HC^{17,19}CC} 9.4 Hz, ²J_{PNC} 1.8 Hz, ²J_{HC^{16,20}C} 1.5 Hz), 125.07 (dm/d, C^{16,20}, ¹J_{HC} 161.8 Hz, ³J_{HC¹⁸CC} 7.8 Hz, ³J_{HC^{20,16}CC} 7.3–7.5 Hz, ³J_{PNC¹⁵C} 3.0 Hz), 129.15 (dd/s, C^{17,19}, ¹J_{HC} 160.6 Hz, ³J_{HC^{19,17}CC} 7.7 Hz), 125.68 (dt/s, C¹⁸, ¹J_{HC} 160.4, ³J_{HC^{16,20}CC} 7.7 Hz), 130.90 (tdd/d, C²¹, ³J_{HC^{23,25}CC} 7.8 Hz, ²J_{HC¹⁰C} 7.8 Hz, ²J_{PC¹⁰C} 4.2 Hz), 128.95 (dm/d, C^{22,26}, ¹J_{HC} 161.9 Hz, ³J_{PC¹⁰C²¹C} 7.9 Hz, ³J_{HC²⁴CC} 7.6 Hz, ³J_{HC^{26,22}CC} 7.2 Hz), 129.67 (dd/s, C^{23,25}, ¹J_{HC} 162.0 Hz, ³J_{HC^{25,23}CC} 8.2 Hz), 128.78 (dt/s, C²⁴, ¹J_{HC} 161.1 Hz, ³J_{HC^{22,26}CC} 7.3 Hz). ³¹P/³¹P-¹H NMR (162.0 MHz, CDCl₃) δ: 15.4 (dd/s, ³J_{POCH} 19.4 Hz, ²J_{PCH} 12.3 Hz).

Compound **4**(*d*₂). ¹H NMR (400 MHz, CDCl₃) δ: 3.58 (ddd, 1H, NC⁹H_B, ²J_{HH} 14.2 Hz, ³J_{HH} 12.3, 3.7 Hz), 4.22 (ddd, 1H, POC⁸H_B, ³J_{POCH} 20.3 Hz, ²J_{HH} 11.6 Hz, ³J_{HH} 3.7 Hz), 4.34 (dddd, 1H, POC⁸H_A, ²J_{HH} 11.6 Hz, ³J_{HH} 12.3, 2.9 Hz, ³J_{POCH} 1.9 Hz), 4.89 (dd, 1H, NC⁹H_B, ²J_{HH} 14.2 Hz, ³J_{HH} 2.9 Hz), 5.38 (d, 1H, PCH, ²J_{PCH} 21.0 Hz), 6.81 (br. dd, 1H, H-12, ³J_{HH} 8.1, 7.4 Hz), 6.95 (dd, 1H, H-11, ³J_{HH} 7.7 Hz, ⁴J_{HH} 1.6 Hz), 6.98 (br. d, 1H, H-14, ³J_{HH} 8.1 Hz), 7.03–7.05 (m, 3H, H-18, H-16, H-20, ³J_{HH} 7.4 Hz), 7.14 (br. dd, 1H, H-13, ³J_{HH} 7.9, 7.4 Hz), 7.18 (br. t, 1H, H-24, ³J_{HH} 7.1 Hz), 7.32–7.37 (m, 6H, H-17, H-19, H-22, H-26, H-23, H-25). ¹³C/¹³C-¹H NMR (100.6 MHz, CDCl₃) δ: 172.80 (m/s, C², ³J_{HC¹⁰NC} 6.8 Hz, ³J_{HC⁹NC} 6.6 Hz, ³J_{HC¹¹CC} 3.8 Hz), 135.45 (br. dd/s, C³, ³J_{HC¹²CC} 8.6 Hz, ³J_{HC¹⁴CC} 5.6 Hz), 138.70 (m/d, C⁴, ³J_{HC¹³CC} 11.5 Hz, ³J_{HC¹¹CC} 8.3 Hz, ²J_{PNC} 5.1 Hz, ²J_{HC¹⁴C} 2.0 Hz), 71.63 (dddd/d, C⁸, ¹J_{H_AC} 153.3 Hz, ¹J_{H_BC} 149.6 Hz, ²J_{POC} 11.8 Hz, ²J_{H_AC⁹C} 4.6 Hz, ²J_{H_BC⁹C} 2.5 Hz), 50.72 (br. ddm/br. s, C⁹, ¹J_{H_AC} 142.0 Hz, ¹J_{H_BC} 143.5 Hz, ²J_{H_AC⁸C} 2.0 Hz, ²J_{H_BC⁸C} 2.9 Hz), 62.49 (ddd/d, C¹⁰, ¹J_{HC} 134.0 Hz, ¹J_{PC} 125.4 Hz, ¹J_{H_AC⁹NC} 5.9 Hz, ³J_{HC^{22,26}CC} 5.9–6.0 Hz), 128.38 (br. dd/d, C¹¹, ⁴J_{PNC³C³C} 3.4 Hz), 128.40 (br. dd/d, C¹², ¹J_{HC} 164.5 Hz, ³J_{HC¹⁴CC} 7.8 Hz, ⁵J_{PNC³C³C¹¹C} 1.2 Hz), 132.03 (br. dd/s, C¹³, ¹J_{HC} 162.2 Hz, ³J_{HC¹¹CC} 8.5 Hz), 127.26 (br. dd/d, C¹⁴, ¹J_{HC} 163.2 Hz, ³J_{HC¹²CC} 7.9 Hz, ³J_{PNC⁴C} 1.5 Hz), 142.06 (ttd/d, C¹⁵, ³J_{HC^{17,19}CC} 9.6 Hz, ²J_{PNC} 1.6 Hz, ²J_{HC^{16,20}C} 1.5 Hz), 124.49 (dm/d, C^{16,20}, ¹J_{HC} 161.7 Hz, ³J_{HC¹⁸CC} 7.7 Hz, ³J_{HC^{20,16}CC} 7.3–7.5 Hz, ³J_{PNC¹⁵C} 3.2 Hz), 128.44 (dd/s, C^{17,19}, ¹J_{HC} 160.8 Hz, ³J_{HC^{19,17}CC} 7.6 Hz), 125.57 (dt/s, C¹⁸, ¹J_{HC} 164.8 Hz, ³J_{HC^{16,20}CC} 7.5 Hz), 133.79 (tdd/d, C²¹, ²J_{HC¹⁰C} 8.3 Hz, ³J_{HC^{23,25}CC} 7.5 Hz, ²J_{PC¹⁰C} 2.0 Hz), 126.27 (dm/d, C^{22,26}, ¹J_{HC} 159.0 Hz, ³J_{PC¹⁰C²¹C} 7.0 Hz, ³J_{HC²⁴CC} 7.0 Hz, ³J_{HC^{26,22}CC} 6.6–7.2 Hz), 129.77 (dd/s, C^{23,25}, ¹J_{HC} 161.8 Hz, ³J_{HC^{25,23}CC} 7.9 Hz), 127.47 (dt/s, C²⁴, ¹J_{HC} 161.5 Hz, ³J_{HC^{22,26}CC} 7.8 Hz). ³¹P/³¹P-¹H NMR (162.0 MHz, CDCl₃) δ: 18.6 (dd/s, ³J_{POCH} 21.1 Hz, ²J_{PCH} 20.2 Hz).

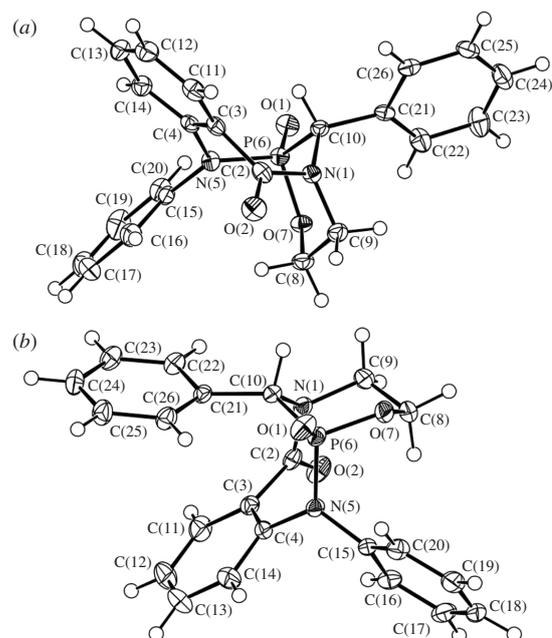


Figure 1 Molecular structures of (a) compound **4**(*d*₁) in the crystal, only the (*R*_p,*R*_{C(10)})-enantiomer of the racemic mixture is shown; (b) compound **4**(*d*₂) in the crystal, only the (*S*_p,*R*_{C(10)})-enantiomer of the racemic mixture is shown. Non-hydrogen atoms are presented as thermal ellipsoids with 50% probability.

C^{22,26} carbons in diastereomers *d*₁ and *d*₂, resulting from the different arrangement of the phenyl substituent at C¹⁰ with respect to the plane of the phenylene and oxazaphosphorinane moieties. In fact, for *d*₁ the difference in the chemical shifts between the H-9A and H-9B protons reaches 1.05 ppm, while it is 1.33 ppm for the *d*₂ counterpart. The C⁴, C^{22,26} and C²⁴ carbons are considerably shielded in *d*₂ in comparison with *d*₁, whereas the C³, C⁹, C¹⁰ and C²¹ carbons are noticeably deshielded in *d*₂. These differences can be interpreted based on the assumption that the C¹⁰ carbon atom in diastereomer *d*₂ has a configuration where the six-membered oxazaphosphorinane ring bears an equatorial phenyl moiety that overhangs the phenylene substituent, whereas diastereomer *d*₁ has a configuration with an axial phenyl in this six-membered ring. The NMR-based assignment of the diastereomers structure was further confirmed by single crystal X-ray diffraction data for both isolated compounds (Figure 1).[¶]

The seven-membered heterocycles of the bicyclodecane cage in both diastereomers have a boat conformation [the N(1)–C(2)–N(5)–P(6) moiety is planar within 0.053(1) Å for *d*₁ and 0.047(2) Å

[¶] X-ray diffraction data for the single crystals of **4**(*d*₁) and **4**(*d*₂) were collected in an ω/φ -scan mode on a Bruker Kappa Apex II CCD diffractometer equipped with an Oxford Cryostream LT device using graphite-monochromated MoK α (0.71073 Å) radiation at 150(2) K. Data were corrected for absorption based on the Laue symmetry using equivalent reflections as well as for systematic errors. The structures were solved by the direct method using SHELXT-2018/2⁹ and refined by the full-matrix least-squares on *F*² using SHELXL-2018/3.¹⁰ Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were inserted at the calculated positions and refined as riding atoms.

Crystal data for **4**(*d*₁). C₂₂H₁₉N₂O₃P, *M* = 390.36, monoclinic, *P*2₁/*c* (no. 14), *a* = 8.5028(6), *b* = 24.973(2) and *c* = 9.0183(7) Å, β = 102.140(4)°, *V* = 1872.1(3) Å³, *Z* = 4, *Z'* = 1, *d*_{calc} = 1.385 g cm⁻³, μ = 0.173 mm⁻¹, *F*(000) = 816, *T*_{max/min} = 0.7116/0.6386; 11798 reflections were collected (3.103° ≤ θ ≤ 25.247°), 3379 of which were unique, *R*_{int} = 0.0646, *R* _{σ} = 0.0714; completeness to θ of 25.242° was 99.8%. The refinement of 253 parameters with no restraints converged to *R*₁ = 0.0443, *wR*₂ = 0.0921 for 2367 reflections with *I* > 2 σ (*I*) and *R*₁ = 0.0783, *wR*₂ = 0.1048 for all data with *S* = 1.004 and residual electron density, peak/hole 0.265/–0.339 e Å⁻³.

for d_2 ; the C(3), C(4) and C(10) atoms deviate to one side from this plane by 1.044(2), 0.942(1) and 0.808(1) Å for d_1 and by 1.014(3), 0.906(3) and 0.850(3) Å for d_2 , respectively]. The six-membered heterocycles have a chair conformation; the N(1)–P(6)–O(7)–C(9) moiety is planar within 0.047(2) Å for d_1 and 0.045(2) Å for d_2 ; the corresponding deviations of the C(8) and C(10) atoms from this plane are 0.621(3) and –0.743(2) Å for d_1 as well as 0.619(2) and –0.814(1) Å for d_2 . The difference in configuration of the diastereomeric molecules is in the position of the phenyl substituent at C(10) atom towards the seven-membered heterocycle, *viz.*, an equatorial position for d_1 and an axial position for d_2 . The position of this substituent is opposite towards the six-membered heterocycle: it is axial for d_1 and equatorial for d_2 . The carbonyl group [C(2)=O(2)] in both diastereomers is out of plane of the phenylene moiety: the dihedral angles between the N(1)–C(2)–O(2)–C(3) and C(2)–C(3)–C(4)–N(5) planar moieties are 55.0(2)° for d_1 and 57.5(1)° for d_2 .

Thus, we have suggested an efficient approach to the synthesis of cage derivatives of aminophosphonic acids that involves the intramolecular reaction of the C=N exocyclic bond with the highly reactive P–O–C(O) moiety in 1,3,2-azaioxaphosphorin-4-ones. This approach can be extended to related cyclic systems where a phosphorus atom is bound to an exocyclic C=N bond through different possible spacers.

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Crystal data for 4(d₂). The structure was refined as a two-component twin. The twin law was found using the TwinRotMat routine of PLATON-200618¹¹ and the final model was refined against a combined set of diffraction indices. The minor domain with fractional contribution of 0.271(2) was rotated from the main one by reciprocal lattice two-fold twinning axis (1 0 0) and direct axis [5 0 1], the twin law was 1.000 0.000 0.335, 0.000 –1.000 0.000, 0.000 0.000 –1.000. C₂₂H₁₉N₂O₃P, $M = 390.36$, monoclinic, $P2_1/c$ (no. 14), $a = 9.2098(15)$, $b = 19.385(3)$ and $c = 10.5483(17)$ Å, $\beta = 101.048(8)^\circ$, $V = 1848.3(5)$ Å³, $Z = 4$, $Z' = 1$, $d_{\text{calc}} = 1.403$ g cm^{–3}, $\mu = 0.176$ mm^{–1}, $F(000) = 816$, $T_{\text{max/min}} = 0.5455/0.5115$; 54215 reflections were collected ($2.101^\circ \leq \theta \leq 28.620^\circ$), 4704 of which were unique, $R_\sigma = 0.0325$; completeness to θ of 25.242° was 99.7%. The refinement of 254 parameters with no restraints converged to $R_1 = 0.0392$, $wR_2 = 0.0996$ for 3958 reflections with $I > 2\sigma(I)$ and $R_1 = 0.0505$, $wR_2 = 0.1063$ for all data with $S = 1.040$ and residual electron density, peak/hole 0.302/–0.375 eÅ^{–3}.

CCDC 1863935 and 1863936 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via <http://www.ccdc.cam.ac.uk>.

Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.1016/j.mencom.2019.03.010.

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