[11] In contrast to nitrogen atoms, which act as π donors and σ acceptors in amino substituents of persistent derivatives of **1** and **2**, the B–B units in **4**–**7** act as π acceptors and σ donors.
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[16] Distances in **10c** [pm]: B–B 176.1(4), BB–C 157.5(4), 158.0(4), ClB–C 155.2(3), 155.3(4), B–Cl 178.7(4), B···B (transannular) 238.0(6), 246.4(6).
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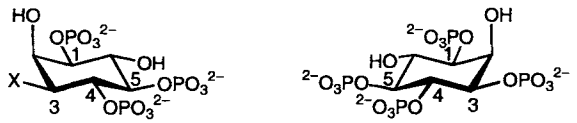
[18] GIAO-SCF/6-31G* gives 20.8 (2B), 22.2 (1B), and −19.6 (C) for **7u** and 94.1 (2B), 73.0 (1B), and 34.5 (C) for **10d**.
[19] X-ray structure analysis for **7a**·Li(THF)$_3$·Et$_2$O ($C_{44}H_{80}B_3LiO_4Si_2$): A yellow crystal (ca. 0.4 × 0.2 × 0.2 mm^3) was investigated on a four-circle diffractometer (CAD4, Enraf–Nonius) with $Cu_{K\alpha}$ radiation (λ = 154.178 pm) at −60 °C. Orthorhombic, space group $P2_12_12_1$, $Z = 4$, a = 1179.6(1), b = 1931.3(1), c = 2193.7(5) pm, $V = 4997.4(12) \times 10^{-30}$ m^3, ρ_{calcd} = 1.022 g cm^{-3}. A total of 4148 independent reflections were collected with $\omega/2\theta$ scans for $2\theta = 120°$ and subjected to LP and semi-empirical absorption corrections (Ψ scans, μ = 9.04 cm^{-1}, min./max. transmission = 0.83/0.87). The structure was solved by direct methods and refined with full matrix against the F^2 data. All heavy atoms were refined with anisotropic temperature factors. All hydrogen atoms were considered riding at idealized positions in groups with common isotropic temperature factors set to 1.2 and 1.5 times (methyl groups) the U_{eq} of the corresponding carbon atom. One THF molecule showed 1:1 disorder over two alternative orientations; high anisotropic distortion parameters indicate high flexibility or disorder in the cation and for several other groups, and reduce the accuracy of the structure determination: wR_2 = 0.2298 and R = 0.073 for 2331 observed reflections ($F_o > 4\sigma(F)$), max. residual electron density 0.28 e Å^{-3}. A Flack Parameter x = −0.07(8) proves that the correct enantiomer has been refined. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-100161. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: int. code +(1223)336-033; e-mail: deposit@chemcrys.cam.ac.uk).
[20] Compare for example the distances in Scheme 4 and the Wiberg bond indices: B1–B2 0.789, B2–B3 1.000, B1–C1 0.945, B2–C1 0.829, B2–H 0.946.

Rapid Synthesis of the Enantiomers of *myo*-Inositol-1,3,4,5-tetrakisphosphate by Direct Chiral Desymmetrization of *myo*-Inositol Orthoformate**

Andrew M. Riley, Mary F. Mahon, and Barry V. L. Potter*

D-*myo*-Inositol-1,4,5-trisphosphate (Ins(1,4,5)P_3, **1**) functions as an intracellular second messenger by eliciting the release of Ca^{2+} from non-mitochondrial stores in stimulated cells.[1] In mammalian tissues phosphorylation of Ins(1,4,5)P_3 at position 3 of the inositol ring by a highly specific cytosolic 3-kinase generates D-*myo*-inositol-1,3,4,5-tetrakisphosphate (D-Ins(1,3,4,5)P_4, **2a**), a molecule whose significance is much less



1 X = OH
2a X = OPO_3^{2-}
2b

well understood. Although binding sites for Ins(1,3,4,5)P_4 have been identified in a wide range of tissues,[2] the cellular function of Ins(1,3,4,5)P_4 remains unknown. Considerable interest has therefore followed the recent identification of an Ins(1,3,4,5)P_4-binding protein, which was purified from platelets, as a member of a family of GTPase activating proteins (GAP).[3] The high affinity of this protein (now designated GAP1^{IP4BP}) for Ins(1,3,4,5)P_4, and its extreme specificity for the 1,3,4,5 configuration of phosphate groups[4] suggest that it may be an Ins(1,3,4,5)P_4 receptor. Very recently it was reported that the interaction of Ins(1,3,4,5)P_4 with the pleckstrin homology (PH) domain of Bruton's tyrosine kinase (Btk) may be involved in B-cell activation and development.[5] Mutations in the Btk PH domain causing human X-linked agammaglobulinaemia (XLA) are associated with dramatically reduced Ins(1,3,4,5)P_4-binding activity.

The spreading interest in Ins(1,3,4,5)P_4 has created a need for an economical and rapid route to pure synthetic material.[6] Although several syntheses of D-Ins(1,3,4,5)P_4 have been published,[7] many are long or require the use of enzymes. Furthermore, the unnatural enantiomer L-Ins(1,3,4,5)P_4 (**2b**; alternative name D-Ins(1,3,5,6)P_4), for which there is only one reported synthesis,[7a] is increasingly being used as a biological tool in studies of D-Ins(1,3,4,5)P_4.[4a, 8] Here we describe a rapid and potentially useful large-scale route to both enantiomers of Ins(1,3,4,5)P_4 by desymmetrization of the well-known *myo*-inositol orthoformate **3**[9] (Scheme 1).

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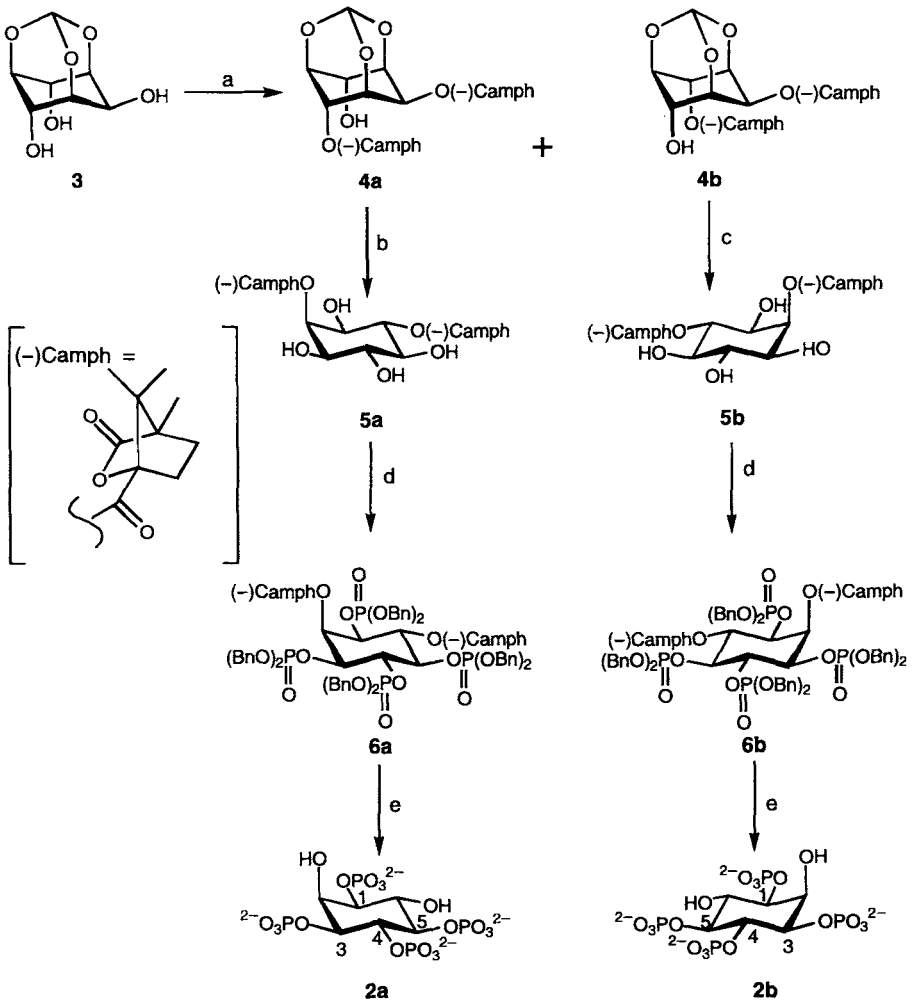

Scheme 1. Synthesis of **2a** and **2b** from *myo*-inositol orthoformate (**3**). a) (1*S*)-(−)-camphanic acid chloride (2.0 equiv), CH_2Cl_2, Et_3N, DMAP, 0 °C to room temperature (RT); b) 1 M HCl/MeOH 1/10, reflux, 6 h, 88%; c) CF_3COOH/H_2O 4/1, RT, 40 h, 85%; d) 1. $(BnO)_2PNiPr_2$, 1*H*-tetrazole, CH_2Cl_2; 2. *m*-CPBA, 78–86%; e) 1. H_2, 1 atm, Pd/C, MeOH/H_2O 19/1, RT; 2. concd aq NH_3, 60 °C, quantitative yield.

Table 1. Selected physical and spectroscopic data for **4a,b** and **2a,b**.

4a: $R_f = 0.32$ (CH_2Cl_2/ethyl acetate 3/1); $[\alpha]_D^{20} = -15.5$ ($c = 1$ in $CHCl_3$), $[\alpha]_D^{20} = -20.5$ ($c = 1$ in DMF). Crystallization from ethyl acetate/hexane, m.p. > 235 °C; elemental analysis calcd for $C_{27}H_{34}O_{12}$ (550.56): C 58.90, H 6.22; found: C 58.7, H 6.23; ^{1}H NMR (400 MHz, $CDCl_3$, TMS): $\delta = 1.00$, 1.03, 1.09, 1.10, 1.12, 1.14 (6s, 18H, camph-CH_3), 1.65–1.75 (m, 2H, camph-CH_2), 1.92–2.03 (m, 2H, camph-CH_2), 2.06–2.18 (m, 2H, camph-CH_2), 2.41–2.56 (m, 2H, camph-CH_2), 3.29 (d, $J = 6.8$ Hz, 1H, D_2O exchange, 4-OH), 4.36 (dddd, appears as dq, J = ca. 4, 2, 2, 2 Hz, 1H, C-3-H), 4.43 (dddd, appears as dq, J = ca. 4, 2, 2, 2 Hz, 1H, C-1-H), 4.55 (dddd, appears as tt, J = ca. 4, 4, 2, 2 Hz, 1H, C-5-H), 4.65–4.70 (br m, 1H, C-4-H), 5.30 (ddd, appears as partially resolved dt, J = ca. 2, 2, 1 Hz, 1H, C-2-H), 5.54 (d, $J = 0.98$ Hz, 1H, O_3CH), 5.63 (ddd, appears as dt, J = ca. 4, 4, 1.5 Hz, 1H, C-6-H); positive-ion FAB-MS: *m/z* (%) = 1101 (100) $[2M+H]^+$, 551 (80) $[M+H]^+$.

4b: $R_f = 0.23$ (CH_2Cl_2/ethyl acetate 3/1); $[\alpha]_D^{25} = +7.5$ ($c = 1$ in DMF). Crystallization from DMF/H_2O or 2-propanol, m.p. > 270 °C (decomp); elemental analysis calcd for $C_{27}H_{34}O_{12}$ (550.56): C 58.90, H 6.22; found: C 58.6, H 6.31; ^{1}H NMR (400 MHz, [D$_7$]DMF, TMS): $\delta = 0.95$, 0.96, 1.06, 1.08, 1.16, 1.17 (6s, 18H, camph-CH_3), 1.57–1.67 (m, 2H, camph-CH_2), 1.91–1.98 (m, 1H, camph-CH_2), 2.01–2.15 (m, 3H, camph-CH_2), 2.52–2.60 (m, 2H, camph-CH_2), 4.32–4.37 (m, 1H, C-1-H), 4.51–4.55 (m, 1H, C-5-H), 4.56–4.61 (m, 2H, C-3-H and C-6-H), 5.47–5.51 (m, 1H, C-2-H), 5.55–5.60 (m, 1H, C-4-H), 5.74 (d, $J = 0.91$ Hz, 1H, O_3CH), 6.21 (d, 1H, $J = 3.36$ Hz, D_2O exchange, C-6-OH); positive-ion FAB-MS: *m/z* (%) = 1101 (55) $[2M+H]^+$, 551 (100) $[M+H]^+$.

Optical rotations (cyclohexylammonium salts): **2a**: $[\alpha]_D^{22} = -2.5$ ($c = 2$ in H_2O), ref. [7a]; $[\alpha]_D^{25} = -2.5$ ($c = 1$ in H_2O); **2b**: $[\alpha]_D^{22} = 2.5$ ($c = 2$ in H_2O), ref. [7a]; $[\alpha]_D^{25} = 2.6$ ($c = 1$ in H_2O). All NMR data agree with the published values [7a, d].

Reaction of symmetrical **3** with 2.0 equivalents of (1*S*)-(−)-camphanic acid chloride gave two diastereoisomeric bis(camphanate) esters **4a** and **4b**, which were separated by flash chromatography and isolated as crystalline solids (Scheme 1, Table 1). A single-crystal X-ray crystallographic study of the less polar diastereoisomer[10] showed this compound to be 1D-2,6-di-*O*-[(−)-ω-camphanoyl]-*myo*-inositol orthoformate (**4a**, Figure 1). Diastereoisomer **4a** is therefore a synthetic precursor to D-Ins(1,3,4,5)P_4, whereas **4b** leads to L-Ins(1,3,4,5)P_4. It was possible to establish reaction conditions (see *Experimental Section*) that minimized production of the unwanted 2-monocamphanate and 2,4,6-tris(camphanate)s. Interestingly, under these conditions the reaction showed considerable diastereoselectivity in favor of **4a**, which allowed it to be isolated in up to 60% yield by careful chromatography. The more polar product **4b** was isolated in about 20% yield, and none of the possible side product 4,6-bis(camphanate) was detected.

Acid hydrolysis of the orthoformate protecting group from **4a** and **4b** gave tetraols **5a** and **5b**. Since these crystallized as hydrates, it was important that they were dried in vacuo at 60 °C before the next step. Phosphitylation with bis (benzyloxy) - *N,N* - diisopropylaminophosphine/1*H*-tetrazole followed by oxidation of the phosphites with *m*-CPBA gave the fully protected tetrakisphosphates **6a** and **6b**. As at the preced-

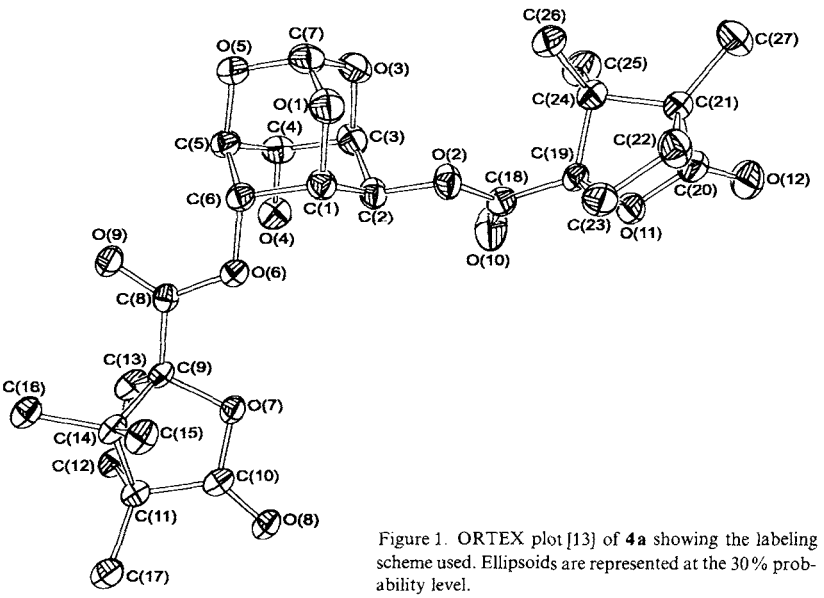

Figure 1. ORTEX plot [13] of **4a** showing the labeling scheme used. Ellipsoids are represented at the 30% probability level.

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ing stages, any diastereoisomer contamination of **6a** or **6b** would be apparent from an examination of the camphanate methyl resonances in the ^{1}H NMR spectra. At no stage was any such contamination seen. A two-step deprotection strategy involving removal of the benzyl protecting groups on phosphates by hydrogenolysis and then cleavage of camphanate esters in a concentrated solution of ammonia at 60 °C was effective with no migration of phosphate groups.[11] Tetrakis(phosphate)s **2a** and **2b** could then be isolated on a gram scale as the cyclohexylammonium[7a] or potassium[7d] salts in overall yields of 60–70% from **4a** and **4b**. The structures of **2a** and **2b** were confirmed by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy as well as high-resolution FAB-MS. We recently used bulk material prepared in this fashion to investigate the submolecular acid–base properties of Ins(1,3,4,5)P_4 by ^{31}P NMR titration experiments.[12] Alternatively, ion-exchange chromatography on Q-Sepharose Fast-Flow gave the pure tetrakisphosphates as their triethylammonium salts. A sample of **2a** was identical to biologically-derived D-Ins(1,3,4,5)P_4 with respect to its interaction with GAP1^{IP4BP}.

In conclusion, we describe rapid access to pure synthetic D- and L-Ins(1,3,4,5)P_4 from readily available starting materials by simultaneously using camphanate esters as desymmetrizing auxiliaries and protecting groups. This strategy should have wider applicability in the inositol phosphate field. Furthermore, the technique is capable of providing D-Ins(1,3,4,5)P_4 in quantities that will now be required for crystallographic and NMR studies of its interaction with the rapidly expanding range of Ins(1,3,4,5)P_4-binding proteins.

Experimental Section

4a, b: To a stirred suspension of **3** (2.00 g, 10.5 mmol) in dry CH_2Cl_2 (40 mL) at 0 °C were added Et_3N (3.3 mL, 23.7 mmol) and a catalytic amount of 4-dimethylaminopyridine (80 mg). A solution of (1*S*)-(−)-camphanic acid chloride (4.55 g, 21.0 mmol) in dry CH_2Cl_2 (10 mL) was added dropwise under an N_2 atmosphere at 0 °C. The cooling bath was removed after 30 min, and stirring continued for a further 30 min. After this time almost no solid remained, and TLC (CH_2Cl_2/ethyl acetate 3/1) showed two major products at $R_f = 0.32$ and 0.23. The solvents were removed in vacuo, and the residue was purified by flash chromatography (CH_2Cl_2/ethyl acetate 4/1) to give first **4a** (3.46 g, 6.28 mmol, 60% yield) and then **4b** (1.35 g, 2.45 mmol, 23% yield).

2a, b: A solution of **6a** or **6b** in MeOH/H_2O (19/1) was stirred vigorously with 10% Pd/C under an atmosphere of H_2 at room temperature overnight. The catalyst was removed by filtration, and the solvents in vacuo. The residue was dissolved in a concentrated solution of aq ammonia, and the solution stirred at 60 °C in a sealed Pyrex autoclavable bottle for 6 h. The solution was allowed to cool and then concentrated under reduced pressure. The residue was dissolved in deionized water, and the camphanamide removed by washing with CH_2Cl_2 (3 ×) followed by Et_2O. Purification by ion-exchange chromatography on Q-Sepharose Fast-Flow eluting with a gradient of triethylammonium bicarbonate buffer (pH 8, 0–1 M) gave the pure triethylammonium salts of **2a** or **2b**, which eluted at 730–850 mM buffer. For larger-scale production treatment with Dowex 50 H^+ resin gave solutions of the free acids of **2a** or **2b**, which were washed again with CH_2Cl_2 and Et_2O, and then converted into either the cyclohexylammonium [7a] or potassium [7d] salts in quantitative yield from **6a** or **6b**.

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[10] Crystal structure analysis of **4a**: Suitable crystals were grown by slow crystallization from 2-propanol (approximate crystal dimensions 0.3 × 0.3 × 0.3 mm): $C_{27}H_{34}O_{12}$, $M_r = 550.54$, monoclinic, $a = 6.755(1)$, $b = 31.139(2)$, $c = 7.035(1)$ Å, $\beta = 117.80(1)^\circ$, $V = 1309.0(3)$ Å^3, space group $P2_1$, $Z = 2$, $\rho_{calcd} = 1.397$ g cm^{-3}, $\mu(Mo_{K\alpha}) = 0.110$ mm^{-1}, $F(000) = 584$. Measurements were made at 293(2) K on a CAD4 automatic four-circle diffractometer in the range $2.61 < \theta < 21.93^\circ$. Data (1795 reflections) were corrected for Lorentzian and polarization but not for absorption effects. In the final least-squares cycles all atoms were allowed to vibrate anisotropically. Except for H1A, which was located in the penultimate difference Fourier and refined at a distance of 0.96 Å from O4, hydrogen atoms were included at calculated positions where relevant. The supramolecular structure contains a hydrogen-bonding feature. The structure was solved with SHELX86 and refined with SHELX93: based on 1326 with $F_o > 4\sigma(F_o)$ $R1 = 0.0318$ and $wR2 = 0.0755$; GOF = 1.029; max./min. residual densities 0.185/ − 0.175 e Å^{-3}. The asymmetric unit (Figure 1) and the labeling scheme were produced with ORTEX. Final fractional atomic coordinates, anisotropic temperature factors, and bond distances and angles are available as supplementary data. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-100275. Copies of the data can be obtained free of charge on application to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: int. code +(1223)336-033; e-mail: deposit@chemcrys.cam.ac.uk).

[11] An earlier strategy involving phosphitylation with bis(cyanoethyloxy)-*N*,*N*-diisopropylaminophosphine/1*H*-tetrazole and oxidation as before followed by a one-step deprotection with aqueous ammonia was not satisfactory. It gave rise to slight but detectable migration of phosphate groups at the deprotection stage.

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A New Cationic Domino Process to (±)-Uleine

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Dedicated to Professor Ekkehard Winterfeldt on the occasion of his 65th birthday

Domino processes play an important role in organic synthesis,[1] and cation-induced processes are of great significance in both organic synthesis and biosynthesis. Herein we report on a new cationic cascade reaction (Scheme 1), which we have applied to the stereoselective synthesis of uleine. The introduced process might also be very interesting for the construction of strychnos alkaloids.

Several syntheses have been described for the alkaloids of the uleine group such as uleine (**2**), which like the strychnos alkaloids has an azocino[4,3-*b*]indole skeleton.[2] However, most concepts that employ a carbocyclization in a late step (either by formation of the C-1/C-9a or C-4/C-4a bond) lead to 3-*epi*-com-

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