8H, 2-adamantanol CH), 0.45 (s, 4H, 2-adamantanol CH), 0.24 (d, 8H, $J$ = 12.5 Hz, 2-adamantanol $CH_2$), 0.10 (d, 8H, $J$ = 12.5 Hz, 2-adamantanol $CH_2$), 0.06 (d, 8H, $J$ = 12.5 Hz, 2-adamantanol $CH_2$), −0.10 (s, 4H, 2-adamantanol CH), −0.43 (s, 8H, 2-adamantanol $CH_2$); $^{13}$C NMR (125 MHz, $D_2O$): $\delta$ = 170.1 (Cq), 153.4 (CH), 145.2 (Cq), 126.0 (CH), 74.0 (2-adamantanol CH), 47.8 ($CH_2$), 37.2 (2-adamantanol $CH_2$), 36.1 (2-adamantanol $CH_2$), 31.0 (2-adamantanol $CH_2$), 27.3 (2-adamantanol CH), 26.8 (2-adamantanol CH).

**1**·(**6**)$_4$: $^1$H NMR (500 MHz, $D_2O$): $\delta$ = 9.29 (d, $J$ = 6.6 Hz, 24H, Py H$\alpha$), 8.65 (d, $J$ = 6.6 Hz, 24H, Py H$\beta$), 4.09 (s, 12H, trimethoxybenzene CH), 2.98 (s, 24H, -$NCH_2CH_2$N-), 2.43 (br s, 36H, $CH_3O$); $^{13}$C NMR (125 MHz, $D_2O$): $\delta$ = 169.9 (Cq), 160.6 (trimethoxybenzene Cq), 153.3 (CH) 145.9 (Cq), 126.2 (CH), 92.0 (trimethoxybenzene CH), 54.5 ($CH_3O$) 47.8 (-$NCH_2CH_2$N-).

**1**·**7**: $^1$H NMR (500 MHz, $D_2O$): $\delta$ = 9.28 (br s, 24H, Py H$\alpha$), 8.72 (br s, 24H, Py H$\beta$), 4.8 (s, 3H, **7** Ar CH), 2.99 (s, 24H, -$NCH_2CH_2$N-), −0.12 (s, 27H, *t*Bu); $^{13}$C NMR (125 MHz, $D_2O$): $\delta$ = 170.1 (Cq), 153.3 (CH) 149.0 (**7** Ar Cq), 146.3 (Cq), 126.0 (CH), 47.8 (-$NCH_2CH_2$N-), 33.8 (*t*Bu Cq), 30.4 (*t*Bu $CH_3$).

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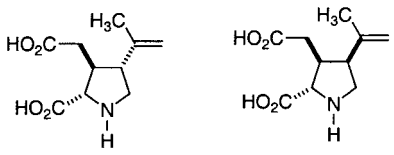
[18] The broadening of the signals is probably due to a slow exchange on the NMR time scale between the free and complexed host.
[19] The assignment of the signals was confirmed by H – H COSY, C – H COSY, NOESY, H – C HMBC.
[20] The 1:4 stoichiometry was estimated by the Job plot method.
[21] Force-field calculation clearly shows that tri-*tert*-butylbenzene is slightly larger than the portals of **1**.

# Nickel and Palladium Catalysis in the Stereoselective Synthesis of Functionalized Pyrrolidines: Enantioselective Formal Synthesis of (+)-$\alpha$-Allokainic Acid

Maxim V. Chevliakov and John Montgomery*

(+)-$\alpha$-Allokainic acid and (−)-$\alpha$-kainic acid are representative members of the kainoid family of neuroexcitatory amino acids whose biological activity has been attributed to their action as conformationally restricted analogues of glutamate (Scheme 1).[1] Extensive structure – activity studies have demonstrated that the C-4 isopropenyl substituent is the




(+)-$\alpha$-allokainic acid (−)-$\alpha$-kainic acid

Scheme 1. Kainoid natural products.

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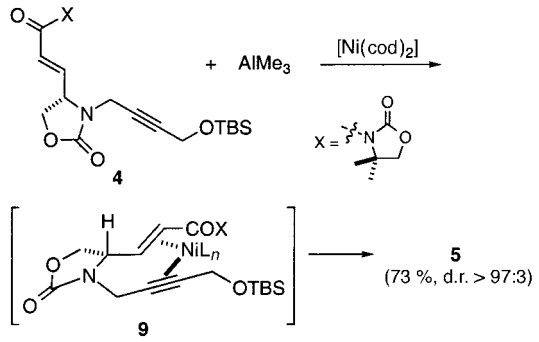
principal site at which structural variation is allowed without compromising neuroexcitatory activity.[2] Given the important role of these natural products in pharmacological investigations, we set out to develop a synthetic entry to this class of compounds that allows facile late-stage modification of the C-4 substituent and access to both the kainic acid and allokainic acid configurations.[3] Herein, we report our initial studies in this regard with a direct and highly stereoselective formal total synthesis of (+)-$\alpha$-allokainic acid. The strategy involves the nickel-catalyzed cyclization of a D-serine-derived alkynyl enone with trimethyl aluminum followed by a palladium-catalyzed allylic carbonate reductive transposition (Scheme 2).

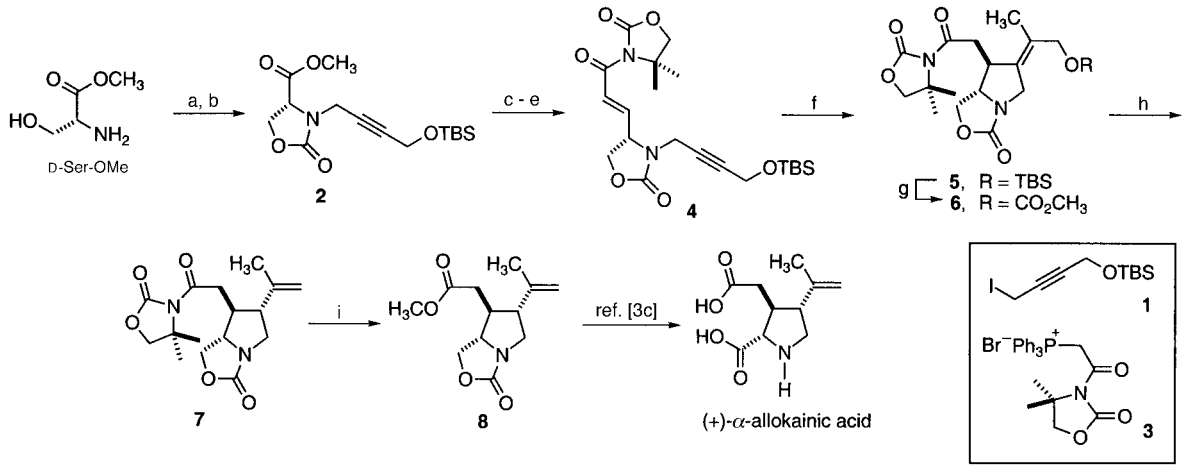
D-Serine methyl ester was efficiently converted to the central cyclization substrate **4** in straightforward fashion (Scheme 2). Condensation of D-serine methyl ester with triphosgene,[4] followed by N-propargylation with iodide **1** using KHMDS afforded **2** in 49% yield over two steps. Chemoselective ester reduction with $NaBH_4$,[5] oxidation under Swern conditions, and Wittig olefination with oxazolidinone **3**[6] gave substrate **4** in 74% overall yield from **2**.

Cyclization of **4** with MeLi/$ZnCl_2$ and 10 mol% $[Ni(cod)_2]$ by employing conditions previously reported[7–8] afforded high yields but moderate diastereoselectivities (ca. 3:1). Commercial trimethylaluminum or dimethylzinc, however, were found to be superior to MeLi/$ZnCl_2$ in achieving the desired *trans* stereochemical relationship of the C-2 and C-3 substituents. Cyclization of **4** with commercial trimethylaluminum and $[Ni(cod)_2]$ (10 mol%) in THF afforded a 73% yield of **5** with a diastereomeric ratio (d.r.) of >97:3 in favor of the desired *trans* isomer, and commercial dimethylzinc under identical conditions afforded a 67% yield of **5**, also with a >97:3 diastereomeric ratio. We believe that a parallel orientation of the two reactive $\pi$ components in chelated structure **9** is responsible for the *trans* relationship of the two substituents in product **5** (Scheme 3).




Scheme 3. Stereoselectivity of nickel-catalyzed alkylative cyclization.

A silyl to carbonate protecting group transposition was carried out in 73% yield to prepare substrate **6** for a palladium-catalyzed reduction with allylic transposition. Related systems were reported by Tsuji et al. to rearrange with palladium catalysis via intermediate $\pi$-allyl complexes to give the terminal alkene by hydride delivery to the more hindered allyl terminus.[9] The stereochemistry of oxidative addition ultimately sets the stereochemistry of the overall process, since decarboxylation and C–H bond reductive elimination both proceed with overall retention of configuration. Treatment of **6** with $[Pd_2(dba)_3]$/$PBu_3$ and $HCO_2H$/$Et_3N$ cleanly produced **7** in 74% yield with a 95:5 diastereomeric ratio in favor of the all-*trans* stereochemical relationship of the three substituents at the pyrrolidine ring, as seen in allokainic acid. This result is consistent with *exo*-selective oxidative addition, *syn* to the adjacent side chain, to ultimately deliver hydride to the $\beta$-face of **6** (Scheme 4). The formal total synthesis of (+)-$\alpha$-allokainic acid was then completed by converting the acyloxazolidinone unit to the corresponding methyl ester **8** upon treatment with $CH_3OMgBr$ (Scheme 2).[10] Compound **8** was previously converted to (+)-$\alpha$-allokainic acid by Hanessian et al.[3c]




Scheme 2. Formal synthesis of (+)-$\alpha$-allokainic acid. a) Triphosgene, THF, 65 °C, 4 h; b) KHMDS (1.1 equiv), THF, 0 °C, 30 min; then **1** (3 equiv) in THF; 40 °C, 24 h, 49% (2 steps); c) $NaBH_4$, EtOH, 0–25 °C, 3 h, 91%; d) $(COCl)_2$ (1.5 equiv), DMSO (3 equiv), $Et_3N$ (4 equiv), $CH_2Cl_2$, –78 °C; e) **3** (1.5 equiv), DMAP (3 equiv), $CH_2Cl_2$, –20 to 25 °C, 1.5 h, 81% (two steps); f) $Me_3Al$ (3 equiv), 10 mol% $[Ni(cod)_2]$, THF, 0 °C, 40 min., 73% (97:3 diastereoisomeric ratio); g) HF·pyridine, THF, 0–25 °C, 24 h, 86%; then methylchloroformate (3 equiv), pyridine, $CH_2Cl_2$, 0–25 °C, 3 h, 83%; h) 10 mol% $[Pd_2(dba)_3]$, 40 mol% $PBu_3$, $Et_3N$ (1.5 equiv), $HCO_2H$ (1.5 equiv), THF, 65 °C, 74%, (95:5 diastereomeric ratio); i) MeOMgBr (3 equiv), 25 °C, 3 h, 54%. cod = cycloocta-1,5-diene, dba = 1,5-diphenylpenta-1,4-dien-3-one, DMAP = 4-dimethylaminopyridine, HMDS = hexamethyldisilazanide, TBS = *tert*-butydimethylsilyl.

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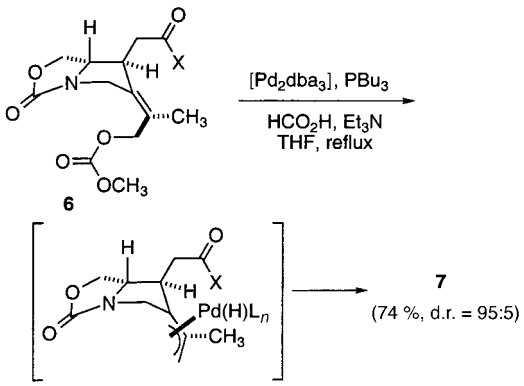


Scheme 4. Palladium-catalyzed reductive transposition.

The above strategy should provide simple variation of the C-4 substituent of allokainic acid simply by selection of a more complex organoaluminum or organozinc reagent. In addition, the introduction of chirality in sequential operations provides the opportunity for modification of the above strategy to allow preparation of the epimeric series of kainoids typified by kainic acid itself through a late-stage common intermediate.[11]

*Experimental Section*

**5**: Trimethylaluminum (2.0 M in hexanes, 1.44 mmol) was added dropwise to a solution of $[Ni(cod)_2]$ (13.2 mg, 0.05 mmol) in THF (1.5 mL) at 0 °C. After 5 min, the mixture was transferred to a solution of **4** (210 mg, 0.48 mmol) at 0 °C in THF (3 mL) by cannula, and stirring was continued for 40 min at 0 °C. The reaction was quenched with $NH_4Cl/NH_4OH$ pH 8 buffer, extracted with EtOAc (four times), dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (EtOAc/hexanes 1/1) afforded **5** (160 mg, 0.35 mmol, 73 %) in a 97:3 diastereomeric ratio: $[\alpha]_D^{25} = -90.9$ ($c = 0.5$ in $CHCl_3$); $^1$H NMR (500 MHz, $CDCl_3$): $\delta = 4.64$ (dd, $J = 4.5$, 3.5 Hz, 1H), 4.41–4.48 (m, 2H), 4.01–4.09 (m, 4H), 3.73 (dt, $J = 8.0$, 4.5 Hz, 1H), 3.59 (d, $J = 15$ Hz, 1H), 3.36 (d, $J = 16$ Hz, 1H), 2.91–3.02 (m, 2H), 1.68 (s, 3H), 1.58 (s, 3H), 1.55 (s, 3H), 0.89 (s, 9H), 0.06 (s, 3H), 0.05 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta = 173.0$, 161.1, 154.0, 132.8, 130.2, 75.4, 70.5, 65.1, 64.8, 60.5, 48.5, 44.2, 41.1, 25.9, 24.85, 24.81, 18.3, 16.3, −5.3, −5.4; IR (film): $\tilde{\nu} = 2928$, 1772, 1695, 1396, 1311; HRMS: *m/z*: calcd. for $C_{18}H_{27}N_2O_6Si$ 395.1639, found 395.1642 $[M - C(CH_3)_3^+]$.

**7**: Tributylphosphane (6.1 mg, 0.03 mmol) was added to a solution of $[Pd_2(dba)_3]$ (6.4 mg, 0.007 mmol) in THF (0.3 mL) at 0 °C. Formic acid (4.83 mg, 0.1 mmol) and triethylamine (10.6 mg, 0.1 mmol) were added neat, followed by carbonate **6** (25 mg, 0.06 mmol) in THF (0.3 mL), and the reaction mixture was refluxed (bath temperature 80–85 °C) for 3 h. The mixture was filtered through florisil, and the solvent was removed. Flash chromatography (EtOAc/hexanes 1/1) afforded **7** (15 mg, 0.05 mmol, 74 %) in a 95:5 diastereomeric ratio: $[\alpha]_D^{25} = -22.4$ ($c = 1.45$ in $CHCl_3$); $^1$H NMR (500 MHz, $CDCl_3$): $\delta = 4.87$ (br. s,1H), 4.85 (t, $J = 1.5$ Hz, 1H), 4.45–4.49 (m, 2H), 4.02 (s, 2H), 3.74 (m, 1H), 3.47 (dd, $J = 8.5$, 11.0 Hz, 1H), 3.38 (dd, $J = 9.5$, 11.5 Hz, 1H), 3.20 (dd, $J = 17.0$, 4.0 Hz, 1H), 2.75 (m, 2H), 2.25 (dq, J = 9.5, 4.0 Hz, 1H), 1.70 (s, 3H), 1.54 (s, 6H); $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta = 172.4$, 160.7, 154.0, 141.6, 114.7, 75.3, 68.0, 64.8, 60.6, 54.9, 48.6, 42.9, 38.2, 24.7, 24.6, 18.2; IR (film): $\tilde{\nu} = 1774$, 1696, 1381, 1308; HRMS (EI): *m/z*: calcd for $C_{16}H_{22}N_2O_5$ 322.1529, found 322.1535 $[M^+]$.

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## Acrylate-Assisted Arene–Chromium Bond Cleavage: Generation of a $[Cr(CO)_2]$ Fragment under Mild Conditions*

E. Peter Kündig,* Mikhail Kondratenko, and Patrick Romanens

Ligand exchange is a key reaction in stoichiometric and catalytic applications of transition metal compounds. It is instrumental in the coordination of a substrate and, following metal-mediated reactions, in the release of the product. Therefore the rate of these processes often determines the efficiency of the overall transformation. Acceleration of ligand exchange and the increase in selectivity that results

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