Electronic Supplementary Information (ESI)

Inhibitors of the Kinase IspE: Structure–Activity Relationships and Co-Crystal Structure Analysis


\[a\] Laboratorium für Organische Chemie, ETH Zürich, HCI, CH-8093 Zürich (Switzerland). Fax: +41 44 6321109; Tel. +41 44 6322992; E-mail: diederich@org.chem.ethz.ch

\[b\] Division of Biological Chemistry and Drug Discovery, College of Life Sciences, MSI/WTB Complex, University of Dundee, Dow Street, Dundee DD1 5EH (United Kingdom). Fax: +44 138 232 2558; Email: w.n.hunter@dundee.ac.uk

\[c\] Technische Universität München, Lehrstuhl für Organische Chemie und Biochemie, Center for Integrated Protein Research, Lichtenbergstraße 4, D-85748 Garching (Germany). Fax: +49 89 289 1336; E-mail: felix.rohdich@ch.tum.de
**Fig. 1ESI.** Active site of *E. coli* IspE from the ternary complex with CDP-ME and the non-hydrolysable ATP analogue 5’-adenyl-β,γ-amidotriphosphate (AppNp) (PDB code: 1OJ4). Colour code: protein skeleton: C: grey; inhibitor skeleton: C: green; O: red; N: blue; P: orange. The colour code is maintained throughout the ESI, if not otherwise stated.
**Fig. 2** MOLOC-generated molecular model of inhibitor 22 in the active site of *E. coli* IspE (PDB code: 1OJ4). Colour code: S: yellow. Distances are given in Å. The units for the indicated distances are maintained throughout the ESI.
**Fig. 3ESI.** Exemplary IC₃₀ curves for inhibition of *E. coli* IspE by inhibitors (+)-9 (a) and 22 (b). [CDP-ME] = 1 mM; [IspE] = 2.5 μg/mm³.

a)

![IC₃₀ curve](image1)

b)

![IC₃₀ curve](image2)
Fig. 4ESI. Exemplary kinetics for the inhibition of *E. coli* IspE by inhibitors (±)-9 (a) and 22 (b). Inhibitor concentrations were 0, 2, 4, 8, 16 and 32 μM (a) and 0, 8, 16, 31, 62 and 125 μM (b).

a)

![Graph](image1)

b)

![Graph](image2)
**Fig. 5ESI.** MOLOC-generated molecular model of (±)-3,\textsuperscript{12} showing hydrophobic contacts in the small, hydrophobic pocket of *E. coli* IspE (PDB code: 1OJ4).\textsuperscript{19}
**Fig 6ESI.** MOLOC-generated molecular model of inhibitors featuring $n$-alkyl chains bound within the active site of *E. coli* IspE (PDB code: 1OJ4). Colour code: C-skeleton of (±)-2$^{12}$: green, C-skeleton of (±)-3$^{12}$: cyan, C-skeleton of (±)-4$^{12}$: magenta, C-skeleton of (±)-6: light pink.
**Fig 7ESI.** MOLOC-generated molecular model of inhibitor (±)-16 in the active site of *E. coli* IspE (PDB code:1OJ4).\textsuperscript{19}
**Fig. 8** a) Omit difference density maps for 22 in the active site of *A. aeolicus* IspE. The purple chicken wire represents the $F_o-F_c$, acalc calculation, where $F_o$ and $F_c$ are the observed and calculated structure factors, respectively and acalc the model phases calculated from all atomic positions except for the ligand itself; contoured at the 1 $\sigma$ (left) and 2.5 $\sigma$ level (right). Colour code: protein skeleton: C: light pink; inhibitor skeleton: C: cyan. b) X-ray crystal structure of *A. aeolicus* IspE co-crystallised with 22 and diphosphate (PDB code: 2VF3). Shown is active site A. c) Superposition of 22, as observed in active site A of *A. aeolicus* IspE, onto 22, as observed in active site B of *A. aeolicus* IspE (PDB code: 2VF3). Colour code: C-skeleton of 22 as observed in active site A: light pink.
b)

![Diagram](image1)

c)

![Diagram](image2)
Fig. 9ESI. Superposition of the active sites from the X-ray crystal structures of *A. aeolicus* IspE with a cytidine-based ligand and 22 (PDB codes: 2V2V and 2VF3, respectively).\(^{13}\) a) Gly-rich loop; b) Cytidine-binding pocket. Colour code: protein skeleton of 2VF3: C: green.

a)
Scheme 1ESI. Synthesis of inhibitors (±)-5 – (±)-8 and (±)-11. (i) Et$_3$N, CH$_2$Cl$_2$, 0 °C → 25 °C, 15–30 min, 30, 33 35 (quantitative), (±)-36 (35%), 37 (31%), 40 (78%); (ii) (±)-29, 12 Et$_3$N, [PdCl$_2$(PPh$_3$)$_2$], CuI, DMF, 25 °C, 2.5–3 h, (±)-5 (72%), (±)-6 (41%), (±)-7 (72%), (±)-8 (92%), (±)-11 (66%).
Scheme 2ESI. Synthesis of inhibitors (±)-9 and (±)-10. (i) Mg, Et₂O, reflux, 30 min; (ii) SO₂Cl₂, CH₂Cl₂, 0 °C → 25 °C; (iii) propargyl amine, CH₂Cl₂, 0 °C → 25 °C, 15–30 min, 38 (31%), 39 (30%); (iv) (±)-29,12 Et₃N, [PdCl₂(PPh₃)₂], CuI, DMF, 25 °C, 2.5 h, (±)-9 (54%), (±)-10 (75%).
Scheme 3ESI. Synthesis of inhibitors 12 and 13. (i) Cs₂CO₃ or NaH, DMF, 50 °C, 16 h or 8 h, 41 (37%), 42 (54%); (ii) 26,¹² Et₃N, [PdCl₂(PPh₃)₂], CuI, DMF, 25 °C, 2.5 h, 12 (90%), 13 (74%).
**Scheme 4**. Synthesis of inhibitor (±)-14. (i) NaH, MeI, DMF, 25 °C, 1 h, 91%; (ii) Et₃N, [Pd(PPh₃)₄], CuI, DMF, 50 °C, 26 h, 31%.
Scheme S5ESI. Synthesis of inhibitors (±)-15 – (±)-17. (i) Et₃N, [PdCl₂(PPh₃)₂], CuI, DMF, 25 °C, 2 h, (±)-15 (95%), (±)-16 (43%), (±)-17 (53%).

31 R = 4-morpholinyl
32 R = 1-piperidinyl
33 R = 1-pyrrolidinyl

(±)-15 R = 4-morpholinyl
(±)-16 R = 1-piperidinyl
(±)-17 R = 1-pyrrolidinyl
Scheme 6ESI. Synthesis of inhibitors 19 – 21. (i) NaH, DMF, 25 °C, 21 h or 15 h, 44 (89%), 45 (67%); (ii) 26 or 34,12 Et3N, [Pd(PPh3)4] or [PdCl2(PPh3)2], CuI, DMF, 50 °C or 25 °C, 3.5 h or 20 h, 20 (75%), 21 (73%); (iii) Et3N, H2O, reflux, 1 h, 90%.
Table 1ESI. X-ray co-crystal structure: statistics for data collection and refinement.

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Numbers in parenthesis represent the highest resolution bin of width approx. 0.12 Å.

$^a$ Diffraction-component Precision Index.$^{43}$
Synthesis ESI

General procedures

General Procedure B for the preparation of a sulfonamide from a sulfonyl chloride

To a solution of propargyl amine (1.0 eq) in dry CH$_2$Cl$_2$, Et$_3$N (1.1 eq) and the sulfonyl chloride (1.0 eq) were slowly added at 0 °C. After completion of the addition, the mixture was left to stir at 25 °C for 15–30 min and concentrated in vacuo. The residue was purified by CC (SiO$_2$; EtOAc–cyclohexane 1:2).

General procedure C for the base-mediated alkylation of 23$^{24}$

A solution of 23$^{24}$ (1.0 eq) and NaH (1.1 eq) in dry DMF was left to stir at 25 °C for 1 h. The alkyl bromide (1.1 eq) in dry DMF was slowly added, and the mixture was left to stir at 25–50 °C for 8–21 h and concentrated in vacuo. NaH was used as suspension of NaH in mineral oil (55–65%).

General procedure D for the preparation of sulfonamides from bromides

To a suspension of Mg turnings (1.7 eq) in dry Et$_2$O, a solution of the alkyl bromide (1.0 eq) in dry Et$_2$O was slowly added in small portions under strong stirring. After the initial exothermic reaction had ceased, the mixture was further heated to reflux for 30 min. The suspension was cooled to 25 °C and slowly added to a solution of sulfuryl chloride (3.0 eq) in dry CH$_2$Cl$_2$ at 0 °C. The mixture was warmed up to 25 °C and concentrated in vacuo. The residue was extracted with n-hexane and concentrated in vacuo. The remaining oil was used without further purification and slowly added to a solution of propargyl amine (1.0 eq) and Et$_3$N (1.1 eq) in dry CH$_2$Cl$_2$ at 0 °C. After completion of the addition, the mixture was left to stir at 25 °C for 15–30 min and then concentrated in vacuo. The residue was purified by CC (SiO$_2$; EtOAc–cyclohexane 1:2) to afford the corresponding sulfonamide.
Preparation of the precursors

**N-Prop-2-yn-1-ylhexane-1-sulfonamide (35):**

General procedure B, starting from propargyl amine (0.13 cm$^3$, 2.0 mmol), Et$_3$N (0.30 cm$^3$, 2.2 mmol) and hexanesulfonyl chloride (0.30 cm$^3$, 2.0 mmol) in dry CH$_2$Cl$_2$ (15 cm$^3$). Purification by CC afforded 35 (387 mg, quantitative) as a yellow oil (Found C 52.9, H 8.4, N 6.9. Calcd for C$_9$H$_{17}$NO$_2$S: C 53.2, H 8.4, N 6.9%); $\nu_{\text{max (neat)}}$ cm$^{-1}$ 3274, 2930, 2860, 1431, 1325, 1251, 1143, 1078, 993, 920, 835, 663; $\delta_H$(300 MHz, CDCl$_3$) 0.90 (t, $J = 6.9$, 3 H), 1.28–1.48 (m, 6 H), 1.79–1.89 (m, 2 H), 2.35 (t, $J = 2.7$, 1 H), 3.11–3.16 (m, 2 H), 3.96 (dd, $J = 2.7$, 6.3, 2 H), 4.44 (br s, 1 H); $\delta_C$(75 MHz, CDCl$_3$) 13.9, 22.3, 23.5, 27.9, 31.2, 32.6, 53.5, 72.9, 109.8; EI-HR-MS: calcd for C$_9$H$_{17}$NO$_2$S$^+$ ([M-H]$^+$): 202.0897; found: 202.0894.

**(±)**-**N-Prop-2-yn-1-ylbutane-2-sulfonamide ((±)-36):**

General procedure B, starting from propargyl amine (0.13 cm$^3$, 2.0 mmol), Et$_3$N (0.30 cm$^3$, 2.2 mmol), and sec-butanesulfonyl chloride (0.13 cm$^3$, 2.0 mmol) in dry CH$_2$Cl$_2$ (15 cm$^3$). Purification by CC afforded (±)-36 (120 mg, 35%) as a yellow oil; $\nu_{\text{max (neat)}}$ cm$^{-1}$ 3275, 2974, 2935, 2889, 1458, 1315, 1140, 1078, 985, 918, 853, 719, 645; $\delta_H$(300 MHz, CDCl$_3$) 1.02 (t, $J = 7.5$, 3 H), 1.38 (d, $J = 6.9$, 3 H), 1.49–1.64 (m, 1 H), 2.00–2.13 (m, 1 H), 2.32 (t, $J = 2.6$, 1 H), 2.98-3.10 (m, 1 H), 3.93 (dd, $J = 2.6$, 6.2, 2 H), 4.81 (br s, 1 H); $\delta_C$(75 MHz, CDCl$_3$) 11.2, 13.3, 23.4, 32.7, 59.7, 72.6, 79.2; EI-HR-MS: calcd for C$_7$H$_{13}$NNaO$_2$S$^+$ ([M+Na]$^+$): 198.0565; found: 198.0560.

**N-Prop-2-yn-1-ylpropane-2-sulfonamide (37):**

General procedure B, starting from propargyl amine (0.13 cm$^3$, 2.0 mmol), Et$_3$N (0.30 cm$^3$, 2.2 mmol) and isopropanesulfonyl chloride (0.23 cm$^3$, 2.0 mmol) in dry CH$_2$Cl$_2$ (15 cm$^3$). Purification by CC afforded 37 (100 mg, 31%) as a red oil; $\nu_{\text{max (neat)}}$ cm$^{-1}$ 3274, 2956, 2930, 2863, 1458, 1324, 1248, 1143, 1076, 990, 925, 838,
668; $\delta$(300 MHz, CDCl$_3$) 1.40 (d, $J = 6.9$, 6 H), 2.33 (t, $J = 2.4$, 1 H), 3.29 (sept, $J = 6.9$, 1 H), 3.94 (dd, $J = 2.4$, 6.0, 2 H), 4.75 (br s, 1 H); $\delta$(75 MHz, CDCl$_3$) 16.5 (2 C), 32.8, 53.9, 72.7, 79.2; EI-HR-MS: calcd for C$_6$H$_{11}$NO$_2$S$^+$ ([M$^+$]): 161.0510; found: 161.0497.

N-Prop-2-yn-1-ylcyclobutanesulfonamide (38):

General procedure D, starting from Mg turnings (100 mg, 4.2 mmol) in dry Et$_2$O (4.0 cm$^3$) and cyclobutyl bromide (0.20 cm$^3$, 2.5 mmol) in dry Et$_2$O (4.0 cm$^3$); sulfuryl chloride (0.60 cm$^3$, 7.4 mmol) in dry CH$_2$Cl$_2$ (6.0 cm$^3$); propargyl amine (0.13 cm$^3$, 2.0 mmol) and Et$_3$N (0.30 cm$^3$, 2.2 mmol) in dry CH$_2$Cl$_2$ (15 cm$^3$).

Purification by CC afforded 38 (110 mg, 31%) as a brown oil; $\nu_{\text{max}}$(neat)/cm$^{-1}$ 3274, 2952, 2873, 1434, 1318, 1282, 1243, 1143, 1076, 1011, 913, 812, 737, 623; $\delta$(300 MHz, CDCl$_3$) 1.96–2.07 (m, 2 H), 2.29–2.41 (m, 2 H), 2.34 (t, $J = 2.7$, 1 H), 2.45–2.58 (m, 2 H), 3.91–3.95 (m, 3 H), 4.57 (br s, 1 H); $\delta$(75 MHz, CDCl$_3$) 16.8, 23.9, 32.7, 55.0, 72.6, 79.3; EI-HR-MS: calcd for C$_7$H$_{11}$NO$_2$S$^+$ ([M$^+$]): 173.0505; found: 173.0507.

N-Prop-2-yn-1-ylcyclopentanesulfonamide (39):

General procedure D, starting from Mg turnings (150 mg, 6.3 mmol) in dry Et$_2$O (4.0 cm$^3$) and cyclopentyl bromide (0.36 cm$^3$, 3.3 mmol) in dry Et$_2$O (4.0 cm$^3$); sulfuryl chloride (0.90 cm$^3$, 11.1 mmol) in dry CH$_2$Cl$_2$ (6.0 cm$^3$); propargyl amine (0.18 cm$^3$, 2.7 mmol) and Et$_3$N (0.45 cm$^3$, 3.3 mmol) in dry CH$_2$Cl$_2$ (15 cm$^3$).

Purification by CC afforded 39 (150 mg, 30%) as a brown oil that was taken directly to the next step without full characterisation; $\nu_{\text{max}}$(neat)/cm$^{-1}$ 3279, 2963, 1640, 1493, 1306, 1127, 1076, 972, 839, 783, 719; $\delta$(300 MHz, CDCl$_3$) 1.57–1.70 (m, 2 H), 1.76–1.89 (m, 2 H), 1.95–2.14 (m, 4 H), 2.34 (t, $J = 2.7$, 1 H), 3.61 (quint, $J = 7.5$,...
1 H), 3.94–3.98 (m, 2 H), 4.54 (br s, 1 H); EI-HR-MS: calcd for C₈H₁₂NO₂S⁺ ([M-H]⁺) 186.0584; found: 186.0585.

**N-Prop-2-yn-1-ylcyclohexanesulfonamide (40):**

General procedure B, starting from propargyl amine (0.13 cm³, 2.0 mmol), Et₃N (0.30 cm³, 2.2 mmol) and cyclohexanesulfonyl chloride (0.30 cm³, 2.0 mmol) in dry CH₂Cl₂ (15 cm³). Purification by CC afforded 40 (320 mg, 78%) as a yellow oil; νmax(neat)/cm⁻¹ 3273, 2938, 2860, 1454, 1317, 1269, 1144, 1078, 985, 893, 861, 838, 668; δH(300 MHz, CDCl₃) 1.17–1.37 (m, 3 H), 1.48–1.61 (m, 2 H), 1.68–1.74 (m, 1 H), 1.89–1.94 (m, 2 H), 2.20–2.25 (m, 2 H), 2.35 (t, J = 2.5, 1 H), 3.02 (tt, J = 3.5, 12.0, 1 H), 3.95 (dd, J = 2.4, 6.0, 2 H), 4.41 (br s, 1 H); δC(75 MHz, CDCl₃) 25.1, 25.2 (2 C), 26.3 (2 C), 32.7, 61.6, 72.6, 79.2; EI-HR-MS: calcd for C₉H₁₄NO₂S⁺ ([M+H]⁺): 200.0740; found: 200.0742.

**4-Amino-1-cyclopentyl-5-iodopyrimidin-2(1H)-one (41):**

General procedure C, starting from 23²d (240 mg, 1 mmol), Cs₂CO₃ (360 mg, 1.1 mmol) and cyclopentyl bromide (0.12 cm³, 1.1 mmol) in dry DMF (15 + 5.0 cm³). The mixture was left to stir at 50 °C for 16 h and concentrated in vacuo. Purification by CC (SiO₂; CH₂Cl₂–MeOH 96:4) afforded 41 (110 mg, 37%) as a white solid; mp > 210 °C (decomposition); νmax(neat)/cm⁻¹ 3441, 2953, 2867, 2661, 2567, 2235, 2040, 2025, 1984, 1967, 1946, 1897, 1822, 1767, 1610, 1514, 1481, 1400, 1309, 1283, 1243, 1189, 1099, 1067, 1026, 913, 850, 777, 725, 643; δH(300 MHz, (CDCl₃–CD₃OD 7:1) 1.43–1.72 (m, 6 H), 1.97–2.03 (m, 2 H), 4.79 (quint, J = 7.9, 1 H), 7.50 (s, 1 H); δC(75 MHz, CDCl₃–CD₂OD 7:1) 23.7 (2 C), 31.6 (2 C), 56.1, 58.1, 147.5, 155.9, 163.0; MALDI-HR-MS: calcd for C₉H₁₃IN₃O⁺ ([M+H]⁺): 306.0098; found: 306.0103.
4-Amino-1-(cyclobutylmethyl)-5-iodopyrimidin-2(1H)-one (42):

General procedure C, starting from: 23\textsuperscript{24} (240 mg, 1 mmol), NaH (44 mg, 1.1 mmol) and cyclobutylmethyl bromide (0.12 cm\textsuperscript{3}, 1.1 mmol) in dry DMF (15 + 5.0 cm\textsuperscript{3}). The mixture was left to stir at 50 °C for 8 h. Purification by CC (SiO\textsubscript{2}; CH\textsubscript{2}Cl\textsubscript{2}–MeOH 96:4) afforded 42 (164 mg, 54%) as a white solid (Found C 35.4, H 4.0, N 13.7. Calcd for C\textsubscript{9}H\textsubscript{12}IN\textsubscript{3}O: C 35.4, H 4.0, N 13.8%); mp 154–156 °C; \(\nu\text{max}(\text{neat})/\text{cm}^{-1}\) 2967, 2933, 2569, 2497, 2427, 1808, 1603, 1467, 1429, 1358, 1312, 1280, 1241, 1191, 1156, 1128, 1015, 951, 906, 867, 773, 736, 676, 632; \(\delta\text{H}(300 \text{ MHz, CD\textsubscript{3}OD})\) 1.75–2.06 (m, 6 H), 2.72 (quint, \(J = 7.6, 1 \text{ H}\)), 3.79 (d, \(J = 7.6, 2 \text{ H}\)), 8.02 (s, 1 H); \(\delta\text{C}(75 \text{ MHz, CD\textsubscript{3}OD})\) 1.75–2.06 (m, 6 H), 2.72 (quint, \(J = 7.6, 1 \text{ H}\)), 3.79 (d, \(J = 7.6, 2 \text{ H}\)), 8.02 (s, 1 H); MALDI-HR-MS: calcd for C\textsubscript{9}H\textsubscript{13}IN\textsubscript{3}O\textsuperscript{+} ([M+H]\textsuperscript{+}): 306.0098; found: 306.0098.

N-Methyl-N-prop-2-yn-1-ylcyclopropanesulfonamide (43):

A suspension of 26\textsuperscript{12} (87 mg, 0.55 mmol) and NaH (26 mg, 1.1 mmol) in dry DMF (7.0 cm\textsuperscript{3}) was left to stir at 25 °C for 1 h. Methyl iodide (68 mm\textsuperscript{3}, 1.1 mmol) was added, and the mixture was left to stir at 25 °C for 1 h. The resulting mixture was quenched with water (10 cm\textsuperscript{3}) and extracted with CH\textsubscript{2}Cl\textsubscript{2} (3 x 15 cm\textsuperscript{3}). The combined org. phases were dried over Na\textsubscript{2}SO\textsubscript{4}, filtered and concentrated in vacuo. Filtration through a plug of silica gel (EtOAc–cyclohexane 1:2) afforded 43 (87 mg, 91%) as a yellow oil (Found C 48.35, H 6.4, N 7.9. Calcd for C\textsubscript{7}H\textsubscript{11}NO\textsubscript{2}S: C 48.5, H 6.4, N 8.1%); \(\nu\text{max}(\text{neat})/\text{cm}^{-1}\) 3273, 2969, 1635, 1456, 1328, 1306, 1200, 1148, 1067, 1041, 995, 927, 907, 888, 827, 783, 760, 742, 691; \(\delta\text{H}(300 \text{ MHz, CDCl\textsubscript{3}})\) 0.96–1.03 (m, 2 H), 1.16–1.21 (m, 2 H), 2.36 (t, \(J = 2.5, 1 \text{ H}\)), 2.39–2.46 (m, 1 H), 2.96 (s, 3 H), 4.06 (d, \(J = 2.5, 2 \text{ H}\)); \(\delta\text{C}(75 \text{ MHz, CDCl\textsubscript{3}})\) 5.1 (2 C), 27.3, 34.7, 39.8, 74.1, 77.0; HR-ESI-MS: calcd for C\textsubscript{7}H\textsubscript{11}NNaO\textsubscript{2}S\textsuperscript{+} ([M+Na]\textsuperscript{+}): 196.0403; found: 196.0403.
Ethyl (4-Amino-5-iodo-2-oxopyrimidin-1(2H)-yl)acetate (44):  

General procedure C, starting from: 2324 (950 mg, 4.0 mmol), NaH (110 mg, 4.4 mmol) and ethyl bromoacetate (740 mg, 4.4 mmol) in dry DMF (60 + 20 cm3). The mixture was left to stir at 25 °C for 21 h. Purification by CC (SiO2; CH2Cl2–MeOH 95:5) afforded 44 (1.2 g, 89%) as a white solid (Found C 29.8, H 3.1, N 13.1. Calcd for C8H10IN3O3: C 29.7, H 3.2, N 13.0%); mp 207–209 °C; νmax(neat)/cm⁻¹ 3460, 2977, 2250, 1723, 1633, 1478, 1412, 1397, 1368, 1356, 1330, 1282, 1230, 1207, 1093, 1012, 958, 920, 876, 819, 776, 731, 710, 645, 625; δH(300 MHz, CDCl3–CD3OD 7:1) 1.30 (t, J = 7.2, 3 H), 4.25 (q, J = 7.2, 2 H), 4.51 (s, 2 H), 7.57 (s, 1 H); δC(75 MHz, CDCl3–CD3OD 7:1) 14.0, 49.6, 55.6, 60.9, 152.2, 154.5, 164.3, 168.4; MALDI-HR-MS: calcd for C8H11IN3O3⁺ ([M+H]+): 323.9840; found: 323.9846.

Methyl 4-[(4-Amino-5-iodo-2-oxopyrimidin-1(2H)-yl)methyl]benzoate (45):  

General procedure C, starting from: 2324 (470 mg, 2.0 mmol), NaH (53 mg, 2.2 mmol) and methyl 4-(bromomethyl)benzoate (500 mg, 2.2 mmol) in dry DMF (30 + 10 cm3). The mixture was left to stir at 25 °C for 15 h. Purification by CC (SiO2; CH2Cl2–MeOH 97:3) afforded 45 (520 mg, 67%) as a white solid (Found C 40.6, H 3.2, N 10.7. Calcd for C13H13IN3O3: C 40.5, H 3.1, N 10.9%); mp 228–230 °C; νmax(neat)/cm⁻¹ 3441, 3037, 2945, 1707, 1622, 1488, 1471, 1428, 1413, 1367, 1347, 1320, 1275, 1216, 1189, 1104, 1017, 965, 943, 923, 872, 793, 772, 757, 747, 706, 688, 645, 614; δH(300 MHz, CDCl3–CD3OD 7:1) 3.81 (s, 3 H), 4.91 (s, 2 H), 7.26 (d, J = 8.3, 2 H), 7.57 (s, 1 H), 7.91 (d, J = 8.3, 2 H); δC(75 MHz, CDCl3–CD3OD 7:1) 51.8, 52.0, 56.4, 127.4 (2 C), 129.5, 129.8 (2 C), 140.9, 150.8, 156.1, 164.0, 166.7; MALDI-HR-MS: calcd for C13H13IN3O3⁺ ([M+H]+): 385.9996; found: 385.9992.
Reference