Continuous flow enantioselective arylation of aldehydes with ArZnEt using triarylborationoxins as the ultimate source of aryl groups

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Conditions for the analysis of diarylmethanols by GC and HPLC

General information:

The GC analyses were performed in an Agilent 6890N GC apparatus, with an Agilent HP-5 column and a FID detector. In every case, 1 μL of an approximately 15 μM solution of the product was injected. The injector was heated at 250 °C, and the mobile phase was He at 12 psi constant pressure, with a 25:1 split ratio. The detector was heated at 300 °C, with a 35 mL/min flow of H₂ and 350 mL/min of air.

The HPLC analyses were done in Agilent 1100 and 1200 series HPLC apparatus.

(S)-phenyl(p-tolyl)methanol:

\[
\text{GC: 70 to 115 °C (15 °C/min), then to 280 °C (25 °C/min), hold 0.4 min. } R_t = 3.93 \text{ min (aldehyde), 5.54 min (tridecane), 8.29 min (alcohol).}
\]

HPLC: Chiralpak AD-H column, hexane–IPA 98:2, 1 mL/min, 210 nm. \( R_t = 28.0 \text{ min (R), 30.4 min (S).} \)

(R)-(4-methoxyphenyl)(phenyl)methanol:

\[
\text{GC: 70 to 115 °C (15 °C/min), then to 280 °C (25 °C/min), hold 0.4 min. } R_t = 2.96 \text{ min (aldehyde), 5.54 min (tridecane), 9.05 min (alcohol).}
\]

HPLC: Chiralpak OJ column, hexane–IPA 95:5, 1 mL/min, 230 nm. \( R_t = 60.7 \text{ min (R), 69.4 min (S).} \)

(S)-(2-fluorophenyl)(phenyl)methanol:

\[
\text{GC: 70 to 115 °C (15 °C/min), then to 280 °C (25 °C/min), hold 0.4 min. } R_t = 2.84 \text{ min (aldehyde), 5.54 min (tridecane), 7.67 min (alcohol).}
\]

HPLC: Chiralpak AD-H column, hexane–IPA 98:2, 1 mL/min, 230 nm. \( R_t = 26.7 \text{ min (S), 28.5 min (R).} \)
**(S)-(4-chlorophenyl)(phenyl)methanol:**

GC: 70 to 115 °C (15 °C/min), then to 280 °C (25 °C/min), hold 0.4 min. \(R_t = 4.30\) min (aldehyde), 5.54 min (tridecane), 8.81 min (alcohol).

HPLC: Chiralpak AD-H column, hexane–IPA 95:5, 1 mL/min, 230 nm. \(R_t = 14.0\) min (\(R\)), 15.4 min (\(S\)).

**(S)-naphthalen-2-yl(phenyl)methanol:**

GC: 70 to 115 °C (15 °C/min), then to 310 °C (25 °C/min), hold 4.2 min. \(R_t = 5.54\) min (tridecane), 7.04 min (aldehyde), 10.50 min (alcohol).

HPLC: Chiralpak OD column, hexane–IPA 90:10, 1 mL/min, 230 nm. \(R_t = 13.1\) min (\(S\)), 15.6 min (\(R\)).

**(R,E)-2-methyl-1,3-diphenylprop-2-en-1-ol:**

HPLC: Chiralpak AD-H column, hexane–IPA 98:2, 0.5 mL/min, 254 nm. \(R_t = 42.1\) min (\(R\)), 43.9 min (\(S\)).