Support of academic synthetic chemistry using separation technologies from the pharmaceutical industry

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Page	Contents
S1	Instrumentation
S2	Stationary phases

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Instrumentation

Reversed phase achiral UHPLC screening and optimization experiments were performed with a Waters Acquity UPLC H-Class (Waters Corp., Milford, MA) system equipped with a quaternary solvent delivery pump, a sampler manager – FIN autosampler, two auxiliary column managers allowing six installed columns, a photodiode array detector, and MassLynx[®] software (Waters Corp., Milford, MA) for instrument control and data processing.

Achiral SFC screening was carried out on an Agilent 1100 with an Aurora A5 Fusion SFC module. The Agilent stack was comprised a G1322 vacuum degasser, G1312A binary pump, G1367A high-performance autosampler, G1316A column compartment, G1315B diode array detector. An Agilent G1956B, single quadruple mass spectrometer was used to selectively identify the test compounds. The system was controlled by Agilent ChemStation B.03.01, SR1.

Chiral SFC screening and optimization experiments were carried out on Waters Acquity UPC² (Waters Corp., Milford, MA, USA) systems equipped with a fluid delivery module (a liquid CO₂ pump and a modifier pump), a sampler manager – FL autosampler, two auxiliary column managers allowing six installed columns, a photodiode array detector, a Corona Aerosol Detector, a SQ mass spectrometry detector and MassLynx[®] software.

Chiral SFC loading and purification studies were performed on a Berger Instruments Inc. system (Waters Corp., Milford, MA, USA) equipped with a Varian PrepStar solvent delivery module (a liquid CO2 pump and a modifier pump), a sampler manager, a diode array detector, and SFC ProNtO as software.

Achiral stationary phases

The 2.1 mm i.d. by 50 mm length, 1.8 μ m ZORBAX RRHD Eclipse Plus C18, SB-CN, SB-C8 and SBphenyl columns were purchased from Agilent (Palo Alto, CA, USA). The 2.1 x 50 mm, 1.9 μ m Hypersil GOLD (AQ and PFP) columns were obtained from Thermo Scientific (Rockford, IL, USA). A 4.6 mm × 150 mm, 3 μ m Luna HILIC column was purchased from Phenomenex (Torrance, CA, USA). The 4.6 mm × 250 mm, 5 μ m GreenSep BASIC, GreenSep Nitro, GreenSep DEAP, GreenSep Ethyl Pyridine and GreenSep Ethyl Pyridine II columns were purchased from ES Industries (West Berlin, NJ, USA). The 4.6 mm × 250 mm, 5 μ m Kromasil Silica, Diol, NH₂ and CN columns were obtained from AkzoNobel, Separation Products (Bohus, Sweden). The 4.6 mm × 250 mm, 5 μ m ZymorSPHER-HAP and ZymorSPHER-HADP columns were purchased from Zymor, Inc. (Wayne, NJ, USA).

Chiral stationary phases

Columns packed with Chiralpak (AD-3, AS-3, IA, IB, IC, IE and IF) and Chiralcel (OD-3, OJ-3 and OZ-3) were purchased from Chiral Technologies (West Chester, PA, USA), and Lux Amylose-2 and Lux Cellulose-4 columns were purchased from Phenomenex (Torrance, CA, USA). Dimensions and particle size for all columns were 150 x 4.6 mm I.D., 3µm. The 30 x 250 mm, 5µm columns packed with Chiralpak (AD-H and AS-H) and Chiralcel OJ-H were purchased from Chiral Technologies.