Supporting Information

Covalent anchoring of a cellulose per(phenyl carbamate) chiral selector onto silica gel through alkyne-azide *click* chemistry and its utilization in HPLC

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Abbreviations

α	Selectivity (chromatographic evaluation parameter)
ATR-FTIR	Attenuated total reflection Fourier-transformation infrared
AzPS	3-azidopropyl-functionalized silica
CDMPC	Cellulose *tris*(3,5-dimethylphenyl carbamate)
ClPS	3-chloropropyl-functionalized silica
CS	Chiral selector
CSP	Chiral stationary phase
DCC	Dicyclohexylcarbodiimid
DCM	Dichloromethane
DMAP	4-(Dimethylamino)pyridine
DS	Degree of substitution
EA	Elemental analysis
e.e.	Enantiomeric excess
HPLC	High-performance liquid chromatography
NMR	Nuclear magnetic resonance spectroscopy
R_s	Resolution (chromatographic evaluation parameter)
RT	Room temperature
(R); (R)-enantiomer	*(R)*-enantiomer; nomenclature according to Cahn-Ingold-Prelog for chiral molecules
(S); (S)-enantiomer	*(S)*-enantiomer; nomenclature according to Cahn-Ingold-Prelog for chiral molecules
TGA	Thermogravimetric analysis

1 Syntheses

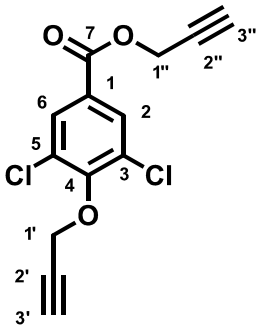
1.1 Synthesis of the reactive, anchor-containing group (3,5-dichlorophenyl-4-propargyloxyphenyl isocyanate, **4**)

The synthesis of 4-propargyloxy-3,5-dichlorophenyl isocyanate (**4**) was carried out according to published procedures (Wolrab et al. 2013; Hettegger et al. 2014) implementing minor modifications.

Synthesis of 3,5-dichloro-4-propargyloxybenzoic acid propargyl ester (**1**): A suspension of 14.6 g of 3,5-dichloro-4-hydroxybenzoic acid (70.4 mmol, 1.0 equiv.), 36.6 mL of propargyl bromide (80 % in toluene, 427 mmol, 6.1 equiv.) and 36.7 g of anhydrous powdered potassium carbonate (266 mmol, 3.8 equiv.) in 80 mL of acetone was heated to 60 °C and stirred at this temperature overnight. The brown suspension was cooled to RT and poured into 300 mL of water. After extraction with EtOAc (3 × 140 mL), the combined organic phases were washed with 100 mL of H_2O and 60 mL of saturated aq. NaCl solution, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The product was obtained as a colorless solid and dried under vacuum for 48 h (19.7 g, 69.8 mmol, 99 %).

^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 8.01 (s, 2H, H-2, H-6), 4.92 (d, 2H, J = 2.5 Hz, H-1'/H1''), 4.87 (d, 2H, J = 2.5 Hz, H-1'/H1''), 2.542 (t, 1H, J = 2.5 Hz, H-3'/H-3'')2.541 (t, 1H, J = 2.5 Hz, H-3'/H-3'').

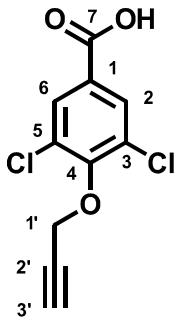
^{13}C NMR ($CDCl_3$, 100.61 MHz): δ 163.3 (C-7), 154.1 (C-4), 130.4 (C-2/C-6), 130.3 (C-3/C-5), 127.1 (C-1), 77.3 (C-2'/2''), 77.1 (C-2'/2''), 76.80 (C-3'/3''), 75.60 (C-3'/3''), 60.6 (C-1'), 53.1 (C-1'').



Synthesis of 3,5-dichloro-4-propargyloxybenzoic acid (**2**): Compound **1** (19.6 g, 69.5 mmol, 1.0 equiv.) was dissolved in 140 mL of EtOH and a solution of 5.18 g NaOH (129 mmol, 1.9 equiv.) in 40 mL of water was added. The reaction mixture was heated to a refluxing temperature for 1 h. Subsequently, the brown solution was diluted with 40 mL of EtOH and 40 mL of H_2O and cooled down to 0 °C using an ice bath. Upon acidification with concentrated HCl to pH~2, a colorless precipitate formed. The solid product was isolated by filtration, washed with 2 × 100 mL of cold H_2O, and lyophilized (15.9 g, 64.9 mmol, 93 %).

^{1}H NMR (CD_3OD, 400.13 MHz): δ 7.98 (s, 2H, H-2, H-6), 4.89 (d, 2H, J = 2.5 Hz, H-1'), 3.00 (t, 1H, J = 2.5 Hz, H-3').

^{13}C NMR (CD_3OD, 100.61 MHz): δ 166.7 (C-7), 154.9 (C-4), 131.3 (C-2/C-6), 131.2 (C-3/C-5), 130.1 (C-1), 78.4 (C-2'), 78.2 (C-3'), 61.6 (C-1').

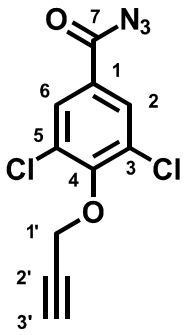


Synthesis of 3,5-dichloro-4-propargyloxybenzoyl azide (**3**): (a) Compound **2** (6.11 g, 24.9 mmol, 1.0 equiv.) was slowly added to 40.5 mL of oxalyl chloride (472 mmol, 19 equiv.) at 0 °C and four drops of dry DMF as a catalyst were carefully added. The suspension was stirred at 0 °C for 15 min and then heated up to 30 °C for 45 min whereupon the colorless solid dissolved and the color changed from colorless to yellowish with little brown drops. Subsequently, the unreacted oxalyl chloride was distilled off and 90 mL of petroleum ether was added to the reaction mixture together with activated carbon (approx. 200 mg) for purification. After refluxing for 3 min, the reaction mixture was filtered hot and evaporated to dryness under reduced pressure. Due to the limited stability of the acyl chloride, product **3a** was used without further purification or characterization and directly converted to the corresponding azide.

(b) A solution of 4.00 g of the acyl chloride **3a** (15.2 mmol, 1.0 equiv.) in 40 mL of cold acetone was cooled to 0 °C using an ice bath and 3.06 g of NaN_3 (47.1 mmol, 3.1 equiv.) dissolved in 16.7 mL of H_2O was added dropwise to the acyl chloride solution under vigorous stirring during 1 h at this temperature, whereupon a voluminous, colorless precipitate formed in the orange solution. The solid was filtered off and washed with cold H_2O (2 × 50 mL). For effective drying, the resulting solid was dissolved in 150 mL of EtOAc, washed with 20 mL of H_2O, 20 mL of saturated aqueous NaCl solution, and dried over anhydrous $MgSO_4$. After concentration *in vacuo* at room temperature, the product was obtained as a colorless solid (2.9 g, 11.0 mmol, 71%). The compound was stored at -20 °C until further use.

ATR-FTIR: υ [cm^{-1}] = 2165 ($-N_3$).

^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 7.98 (s, 2H, H-2, H-6), 4.88 (d, 2H, J = 2.4 Hz, H-1'), 2.55 (t, 1H, J = 2.4 Hz, H-3').

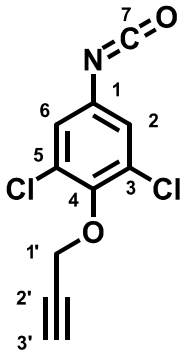


Synthesis of 3,5-dichloro-4-propargyloxyphenyl isocyanate (**4**): 4-Propargyloxy-3,5-dichlorophenyl azide (**3**, 0.416 g) was suspended in 20 mL toluene, dried by azeotropically distilling off 20 mL of the solvent using a Dean-Stark trap and further heated at refluxing temperature for 3 h. The solution was subsequently evaporated to dryness *in vacuo* and the product was obtained as a colorless solid in quantitative yield. Due to the high reactivity and water sensitivity of the isocyanate, the compound was freshly prepared before use to avoid prolonged storage and used without purification.

ATR-FTIR: υ [cm^{-1}] = 2258 (-N=C=O).

^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 7.07 (s, 2H, H-2, H-6), 4.77 (d, 2H, J = 2.4 Hz, H-1'), 2.54 (t, 1H, J = 2.4 Hz, H-3').

^{13}C NMR ($CDCl_3$, 100.61 MHz): δ 148.1 (C-4), 130.7 (C-1), 130.5 (C-3/C-5), 125.0 (C-2/C-6), 120.4 (C-7) 77.5 (C-2'), 76.5 (C-3'), 60.6 (C-1').



1.2 General procedure for the synthesis of the ibuprofen amides (M-P)

A Schlenk-flask was charged with DCC (1.5 equiv.), DMAP (0.5 equiv.) and the corresponding amine (1.2 equiv.). Dry DCM (c = 0.1 M) was added before ibuprofen (1.0 equiv.) was joined to the reaction mixture. After 3 h, DCM was distilled off and the residue was directly subjected to column chromatography (cyclohexane/ethyl acetate, 10/1) to obtain the corresponding amide after the removal of all volatiles.

2 Additional Analytical Data

2.1 NMR Spectra

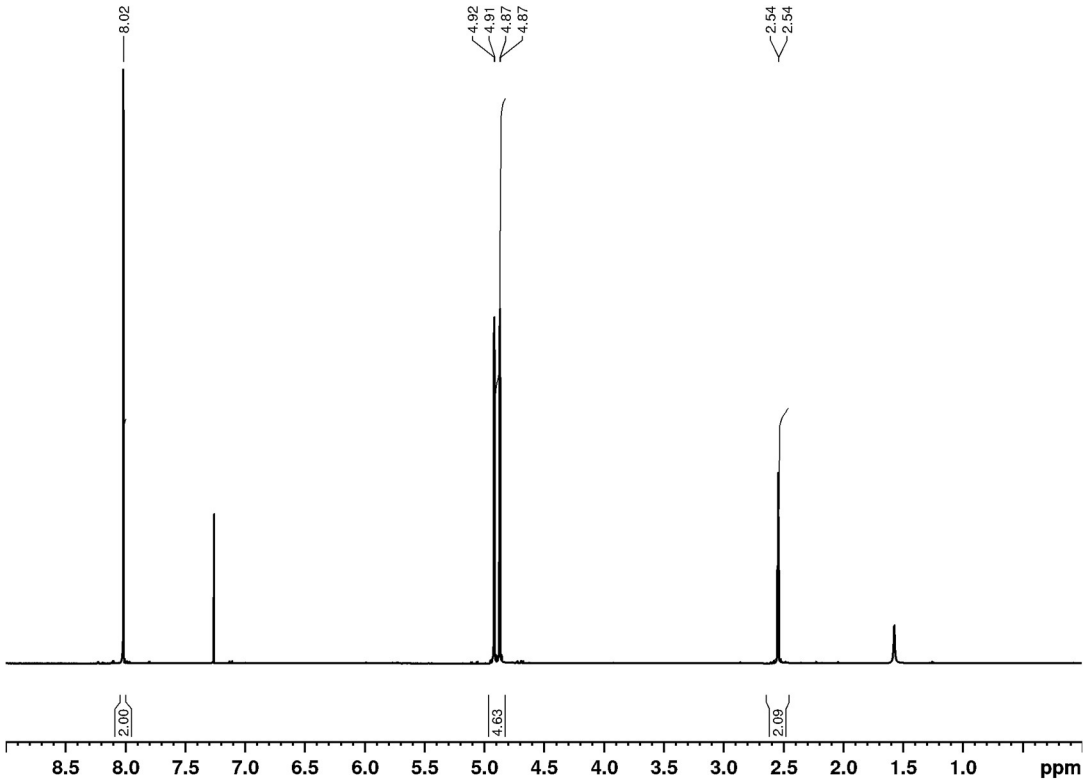


Figure S1: *^{1}H NMR spectrum of 3,5-dichloro-4-propargyloxy benzoic acid propargyl ester (**1**) in $CDCl_3$.*

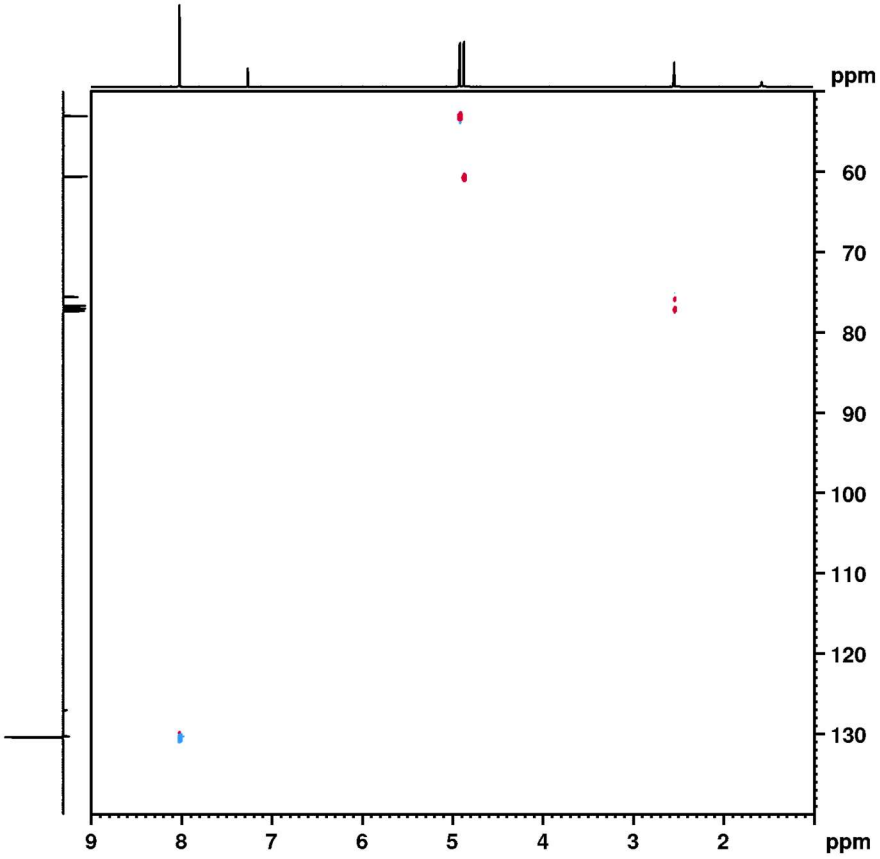


Figure S2: *Multiplicity-edited ^{1}H-^{13}C HSQC NMR spectrum of 3,5-dichloro-4-propargyloxy benzoic acid propargyl ester (**1**) in $CDCl_3$ (CH/CH_3 groups are shown in blue, CH_2 groups are shown in red).*

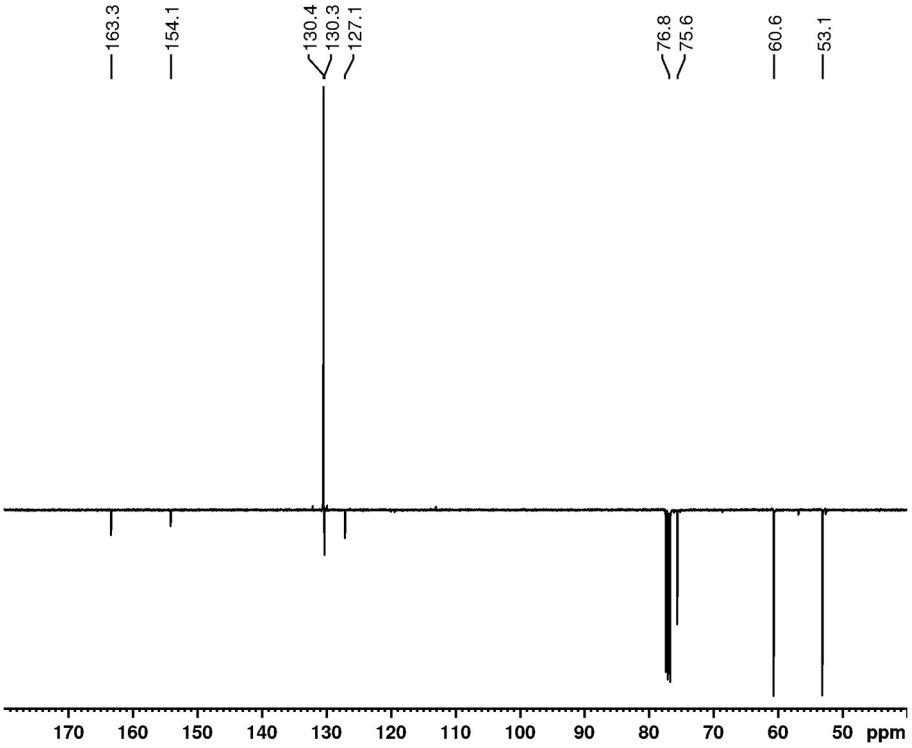


Figure S3: *^{13}C NMR spectrum of 3,5-dichloro-4-propargyloxy benzoic acid propargyl ester (**1**) in $CDCl_3$.*

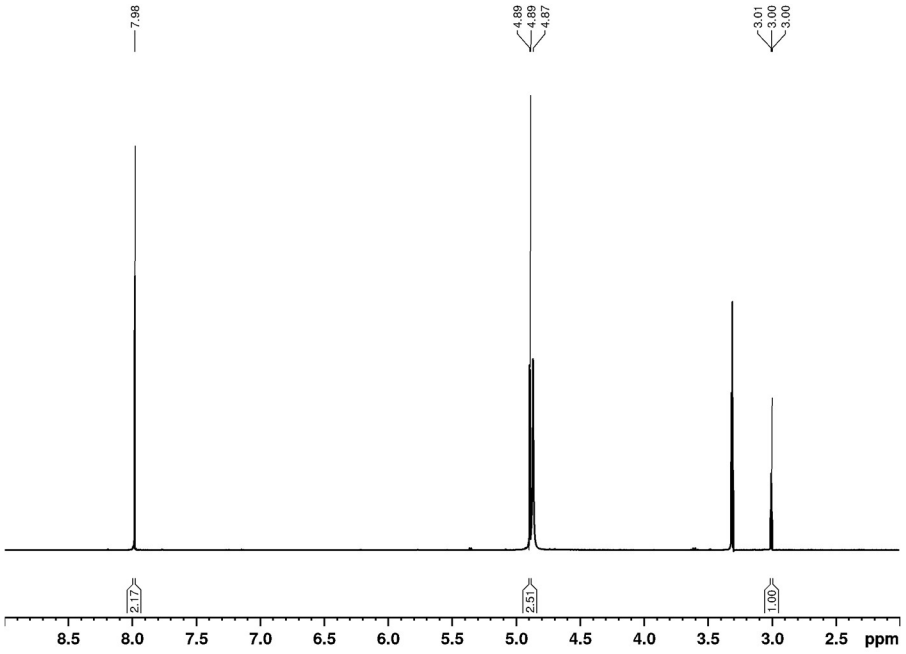


Figure S4: *^{1}H NMR spectrum of 3,5-dichloro-4-propargyloxy benzoic acid (**2**) in CD_3OD.*

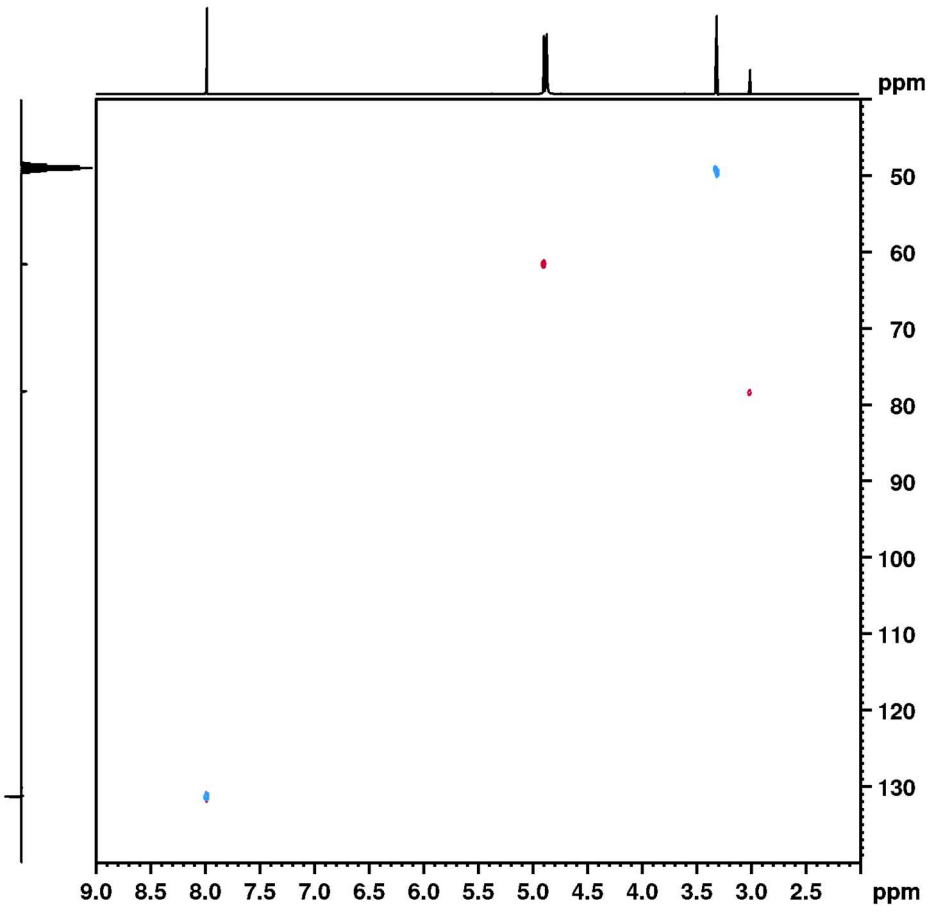


Figure S5: *Multiplicity-edited ^{1}H-^{13}C HSQC NMR spectrum of 3,5-dichloro-4-propargyloxy benzoic acid (**2**) in CD_3OD (CH/CH_3 groups are shown in blue, CH_2 groups are shown in red).*

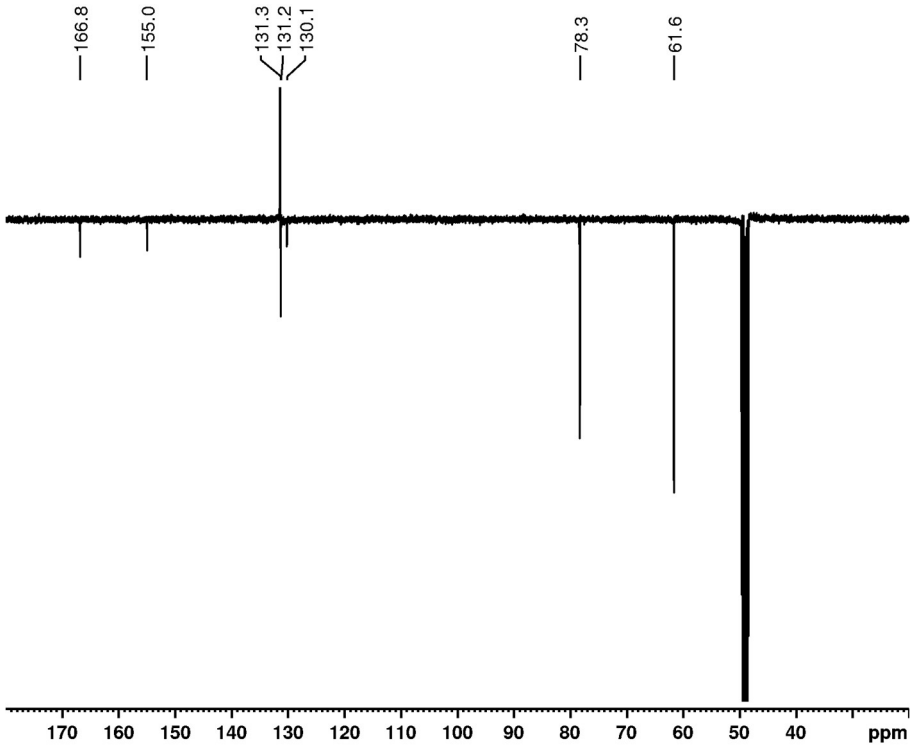


Figure S6: *^{13}C NMR spectrum of 3,5-dichloro-4-propargyloxy benzoic acid (**2**) in CD_3OD.*

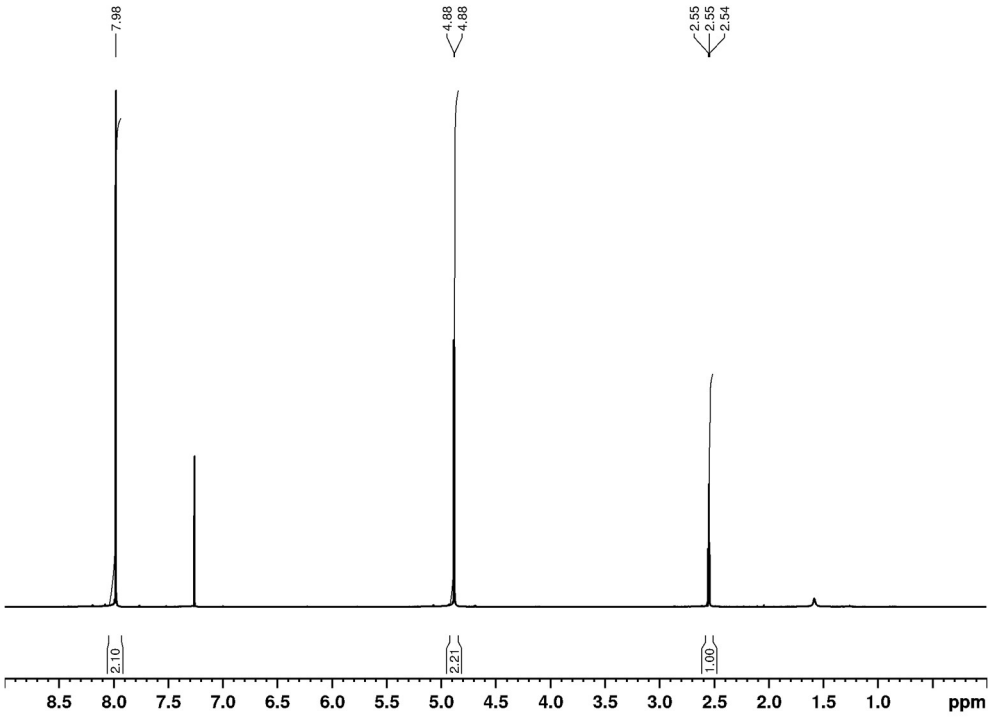


Figure S7: *^{1}H NMR spectrum of 3,5-dichloro-4-propargyloxy benzoyl azide (**3**) in $CDCl_3$.*

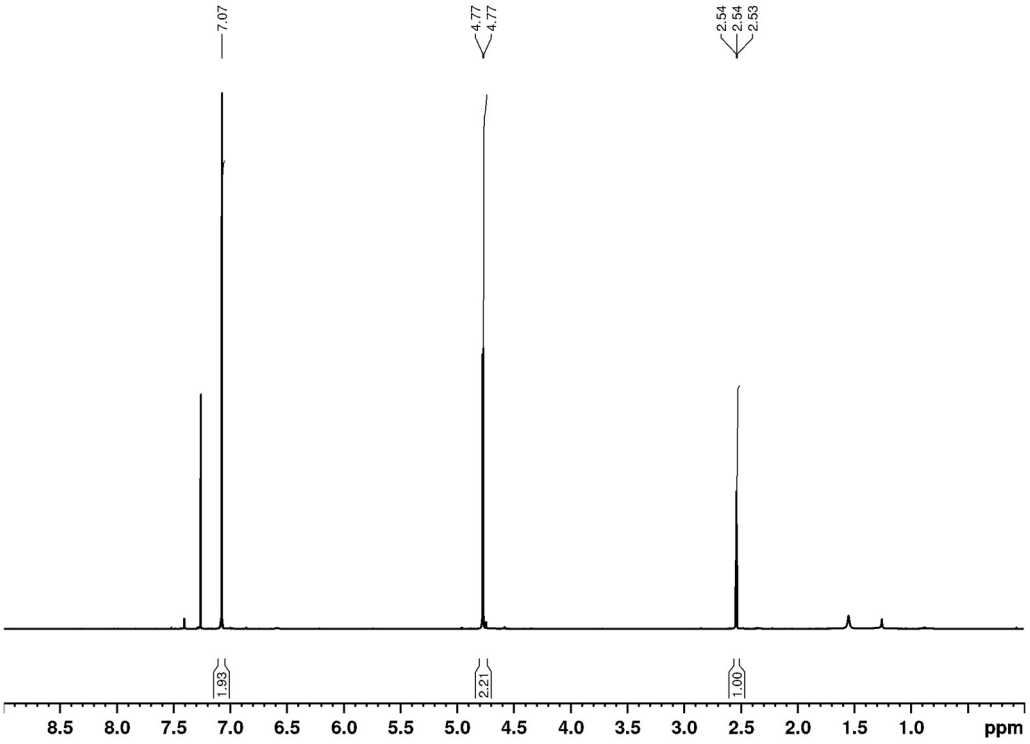


Figure S8: *^{3}H NMR spectrum of 4-propargyloxy-3,5-dichlorophenyl isocyanate (**4**) in $CDCl_3$.*

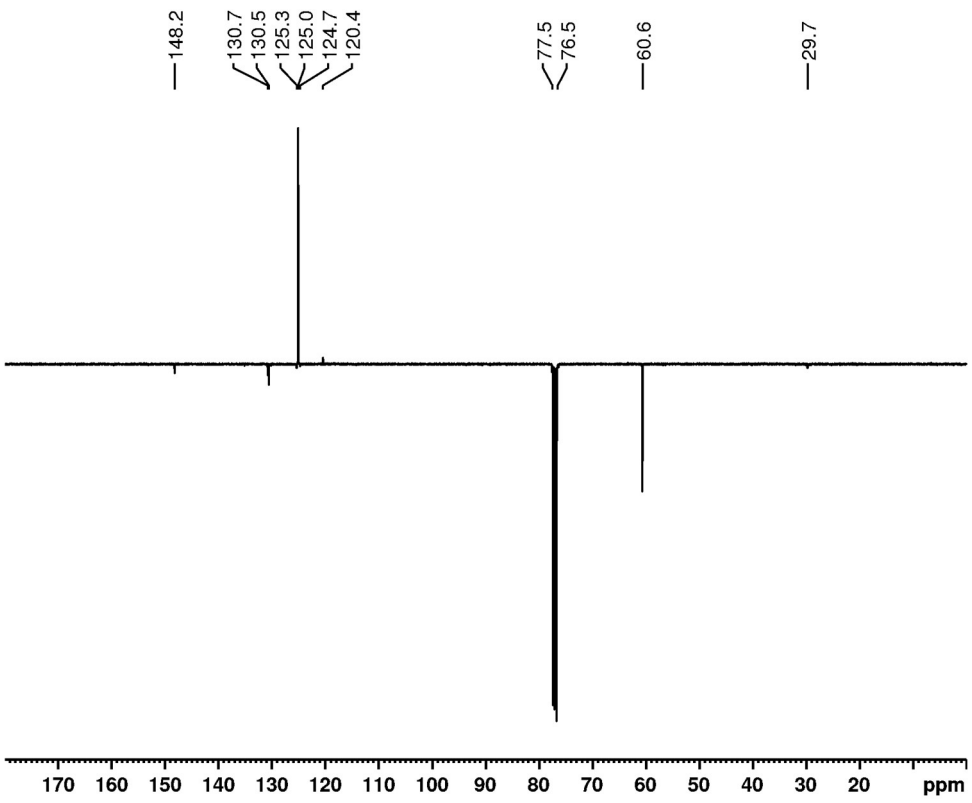


Figure S9: *^{13}C NMR spectrum of 4-propargyloxy-3,5-dichlorophenyl isocyanate (**4**) in $CDCl_3$.*

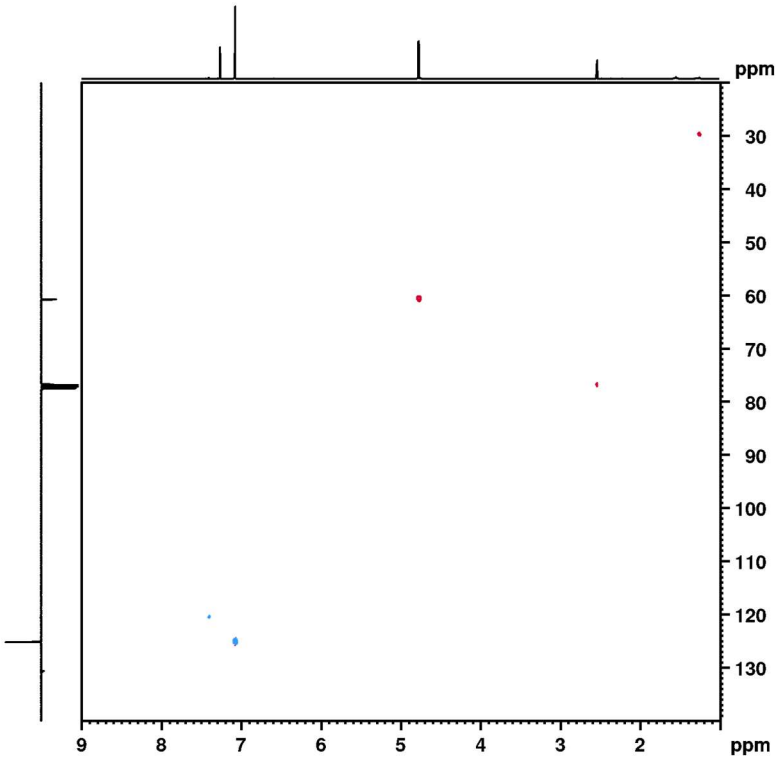


Figure S10: *Multiplicity-edited ^{1}H-^{13}C HSQC NMR spectrum of 4-propargyloxy-3,5-dichlorophenyl isocyanate (**4**) in $CDCl_3$ (CH/CH_3 groups are shown in blue, CH_2 groups are shown in red).*

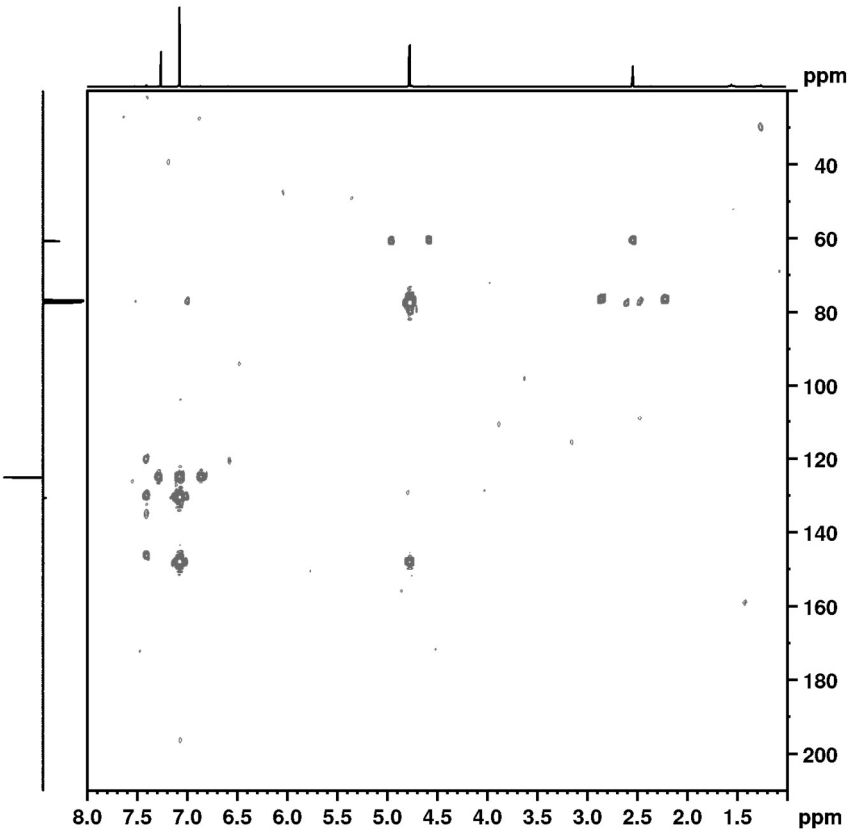


Figure S11: *^{1}H-^{13}C HMBC NMR spectrum of 4-propargyloxy-3,5-dichlorphenyl isocyanate (**4**) in $CDCl_3$.*

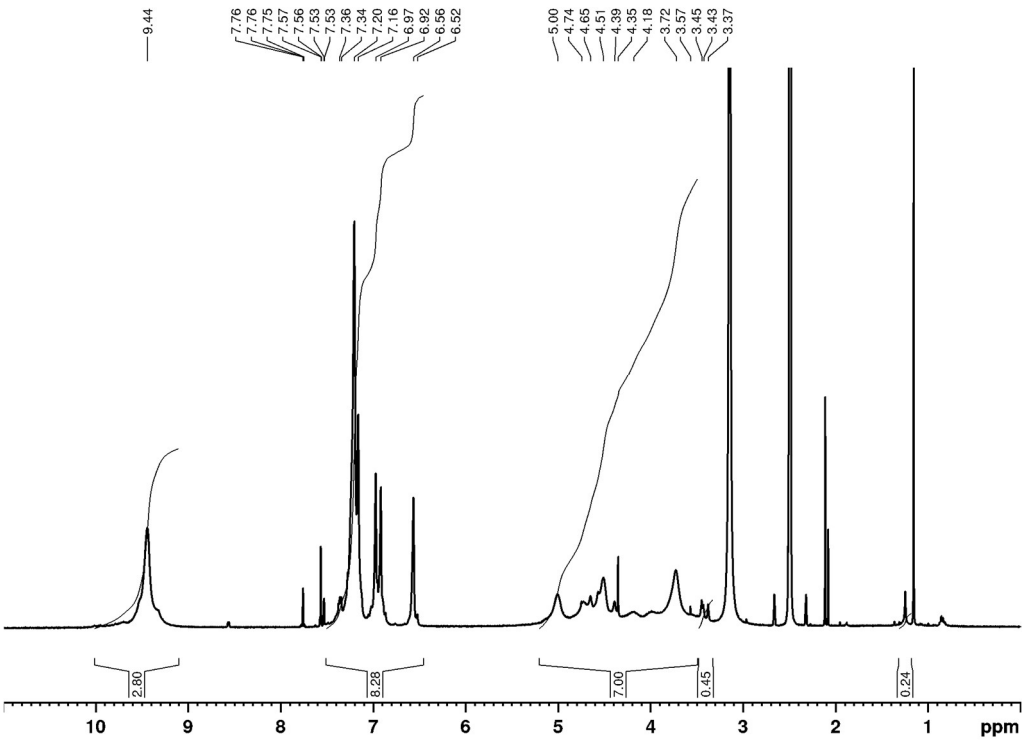


Figure S12: *^{1}H NMR spectrum of **CS1** in DMSO-d_6 (343 K).*

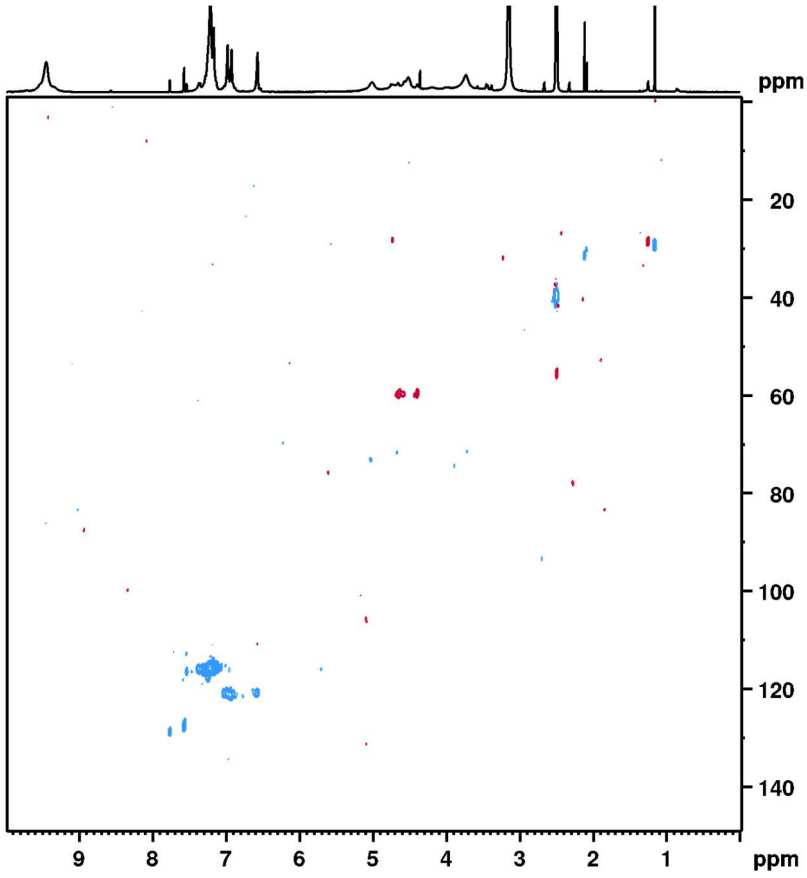


Figure S13: *Multiplicity-edited ^{1}H-^{13}C HSQC NMR spectrum of **CS1** in DMSO-d_6 (343 K) (CH/CH_3 groups are shown in blue, CH_2 groups are shown in red).*

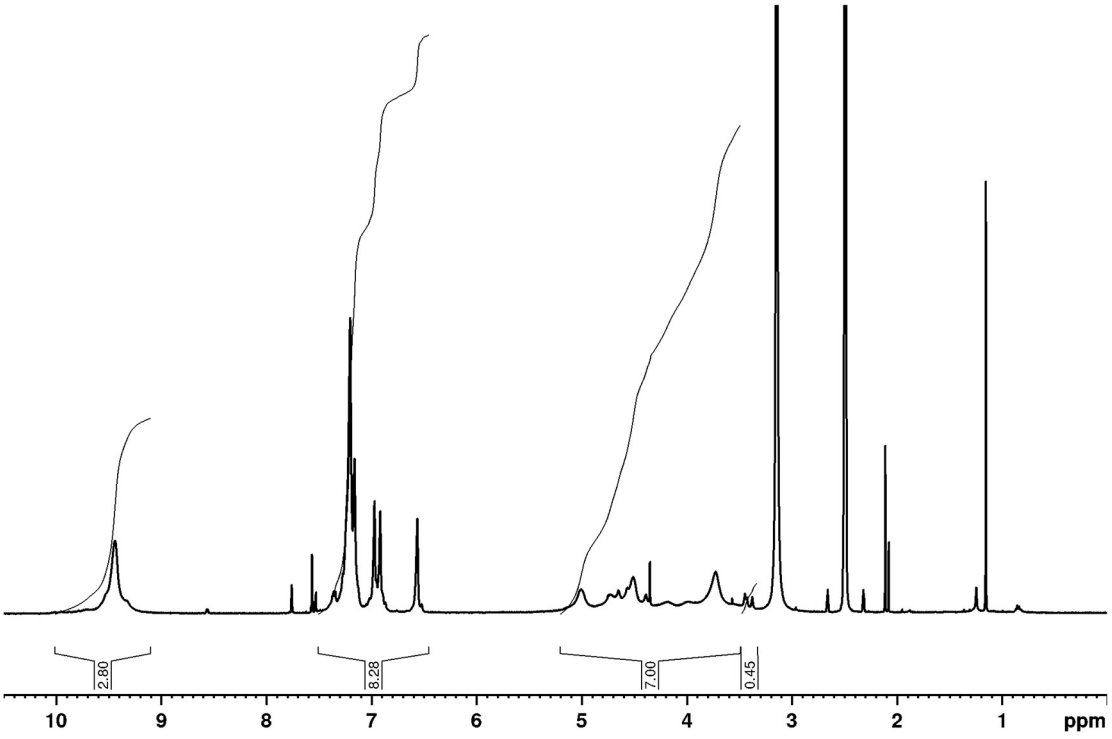


***Figure S14**: Quantitative ^{1}H NMR spectrum of **CS1** in DMSO-d_6 (T = 343 K; d1 = 10 s).*

2.2 FTIR Spectra

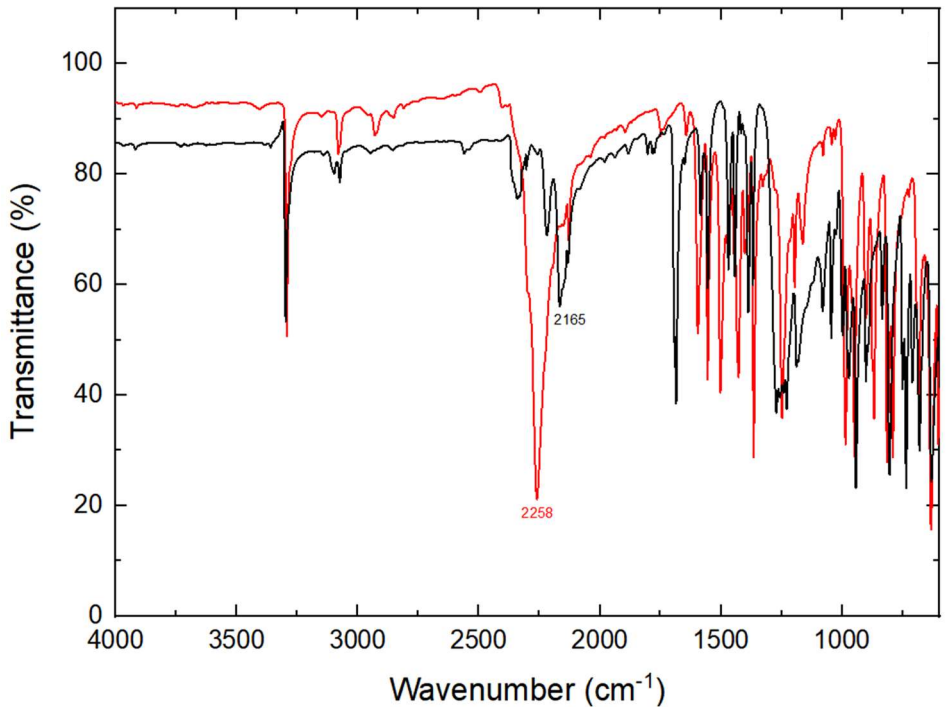


***Figure S15:** FTIR spectra of 3,5-dichloro-4-propargyloxybenzoyl azide (red): υ = 2165 (-N=N=N); 3,5-dichloro-4-propargyloxyphenyl isocyanate (black): υ = 2258 (-N=C=O) cm^{-1}.*

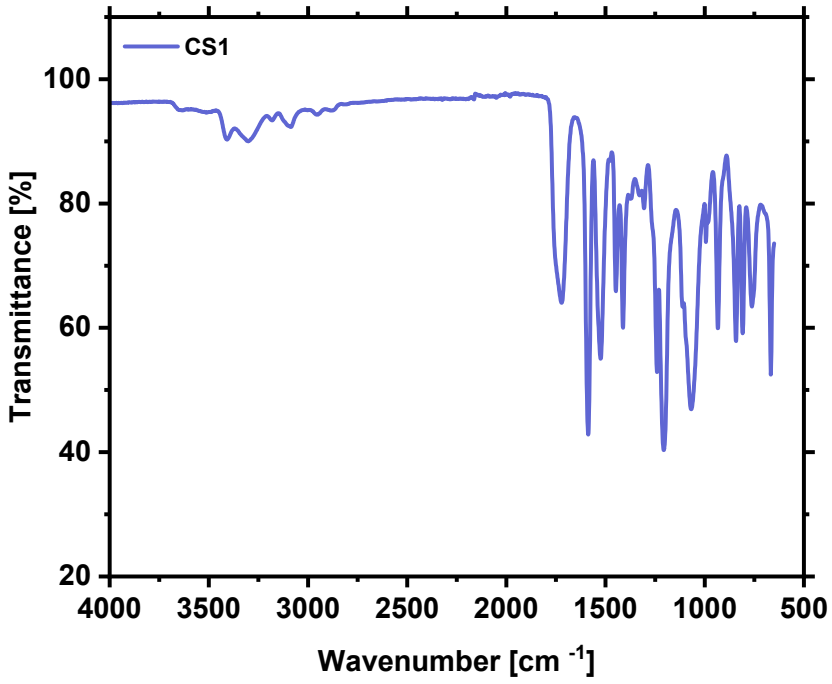


Figure S16: *FTIR spectrum of* ***CS1****; υ = 3408, 3179, 3105 (N-H), 2960, 2870 (C-H), 2137 (C≡C-H), 1718 (C=O), 1584, 1242, 1071, 934 (aromatic rings), 1205 cm^{-1} (C-O-C).*

2.3 Determination of cellulose carbamate DS_{anchor} by NMR

The degrees of substitution (DS) of the chiral selector **CS1** for both, the overall carbamate functionalization ($DS_{overall}$) and the anchor-containing carbamate (DS_{anchor}) were estimated by integration of the quantitative ^{1}H NMR spectrum. The selector was dissolved in DMSO-d_6, and the spectrum was obtained at 70 °C (343 K). Peak assignments were based on coupled ^{1}H-^{13}C HSQC experiments (SI, section 2.1.3).

For the estimation of the overall carbamate functionalization of cellulose (*i.e.* $DS_{overall}$), the integrals of the sugar backbone (δ_H = 3.5 - 5.5 ppm, $\mathbf{I_{SUGAR}}$) corresponding to the C*H*-O/CH_2-O groups were considered as an internal reference. Thus, their integral area was normalized to a total of 7.0 units corresponding to 7 protons *per* glucopyranose unit. The overall carbamate $DS_{overall}$ was determined by directly relating the integral of the carbamate NH-spectral region (broad signals at δ_H = 10.65 – 9.00 ppm, $\mathbf{I_{NH}}$) to the integrals of the sugar backbone. After reference to the sugar backbone region, this directly corresponds to the estimated overall $DS_{overall}$ (see **Table S1**).

However, it should be noted that within the sugar backbone spectral region, the *O*-methylene protons of the propargyloxy group resonate (δ_H = 4.35 ppm), causing a slight underestimation of the effective $DS_{overall}$.

The DS of the anchor-containing group 4-propargyloxy-3,5-dichlorophenyl carbamate was estimated by integration of the signals attributed to the alkyne protons (at δ_H = 3.37, 3.42 and 3.45 ppm; $I_{CH\text{-}alkyne}$). By referencing the latter to the C*H*-O/CH_2-O cellulose backbone region, a DS_{anchor} of 0.45 was calculated. This corresponds to a ratio of 16:84 of anchor- to non-anchor-containing carbamate units (A:NA).

Table S1: *DS calculation using integrals of the quantitative ^{1}H NMR spectra of* ***CS1****. Spectral region shifts are given in ppm and were referenced to the residual solvent signal (DMSO-d_6).*

	$I_{CH\text{-}alkyne}$ [3.35-3.50]	I_{SUGAR} [3.50-5.50]	$I_{H\text{-}Ar}$ [7.50-7.25 + 6.80-6.30]	I_{NH} [10.65-9.00] = $\underline{DS_{overall}}$	$\underline{DS_{anchor}}$ = $I_{CH\text{-}alkyne}$	$\underline{DS_{no\ anchor}}$
CS1	0.45	7.00	8.28	2.80	0.45	2.35

2.4 Elemental analysis

The chiral selector **CS1** was analyzed by elemental analysis in order to estimate the overall DS of the anchor groups. The theoretical elemental composition was calculated based on the NMR analysis of the substitute groups (16 % anchor, 84 % non-anchor), considering the elemental composition of the individual functional groups, more specifically the deprotonated cellulose-backbone, the anchor-containing carbamate group, and the non-anchor containing carbamate group (see **Table S2**). Consequently, the theoretical elemental composition was calculated according to **Equation S1** using carbon as an example. The theoretical molar mass and elemental composition were subsequently calculated by multiplying with each element's molar mass and calculating the theoretical weight percentage of the individual elements.

Equation S1: *Calculation of the theoretical number of C atoms per repeating unit.*

$$C_{theor} = C_{CB} + 3 \times C_{NAU} + 0.161 \times C_{AU-N}$$

$$C_{theor} = 7 + 3 \times 7 + 0.161 \times 3$$

$$C_{theor} = 27.48\ [C - atoms/repeating\ unit]$$

Table S2: *Elemental analysis data obtained for the chiral selector* ***CS1****. The theoretical elemental composition was calculated for the ratio of anchor/non-anchor groups determined by NMR (0.45/2.35, equals 16 % anchor and 84 % non-anchor; see above). Numbers in the table correspond to the number of atoms per repeating unit unless indicated otherwise.*

Functional group	C	H	N	O	Cl	Molar mass [g mol^{-1}]
Cellulose backbone (CB)	6	7	0	5	0	159.12
Anchor-carbamate unit (AU)	10	6	1	2	2	243.06
Non-anchor-carbamate unit (NAU)	7	4	1	1	2	189.02
Difference AU-NAU	3	2	0	1	0	
Theoretical composition for 16 % LU [# of atoms per repeating unit]	27.48	19.32	3.00	8.161	2.00	-
Theoretical composition [g mol^{-1}]	329.79	19.474	42.020	130.56	212.70	734.54
Theoretical composition [wt%]	44.897	2.6512	5.7206	17.775	28.957	-

The overall DS of the carbamate groups attached to the chiral selector was calculated using **Equation S2** with N_{found} being the N-content of the respective CS, $N_{theoretical}$ the theoretical N content for a maximum overall DS of 3 (see **Table S4**, bottom line) and DS_{max} the maximum DS of 3.

Equation S2: *Calculation of the overall DS of the carbamate groups.*

$$DS_{CS} = \frac{N_{found}[wt\%]}{N_{theoretica}\ [wt\%]} \times DS_{max}$$

Table S3: *Elemental analysis data obtained for the chiral selector* ***CS1****. The theoretical elemental composition was calculated for the ratio of anchor/non-anchor groups determined by NMR (0.452/2.35, equals 16 % anchor and 84 % non-anchor groups; see above).*

	C [wt%]	H [wt%]	N [wt%]	O [wt%]	Cl [wt%]	Overall DS
Calculated for DS = 3 (16 % LU)	44.90	2.65	5.72	17.77	28.96	-
CS1	**44.67 ± 0.04**	**2.88 ± 0.03**	**5.04 ± 0.03**	**22.01 ± 0.34**	**27.49***	**2.643**

* Calculated (100 % minus the sum of the C,H,N,O content)

The 3-azidoproyl-loading of **AzPS**, L_{AzP}, was determined by the N-content using **Equation S3**.

Equation S3: *Calculation of the 3-azidopropyl loading based on the nitrogen content determined by elemental analysis.*

$$L_{AzP}\ [\mu mol\ g^{-1}] = \frac{N_{content}[wt\%]}{M_{N3}[g\ mol^{-1}]} \times 10000$$

$$L_{AzP}\ [\mu mol\ g^{-1}] = \frac{1.214}{14.0067 \times 3[g\ mol^{-1}\}} \times 10000$$

$$L_{AzP} = 288.83\ \mu mol\ g^{-1}$$

Table S3: *Elemental analysis data obtained for the modified silica gel samples (**ClPS** and **AzPS**) and the chiral stationary phases (**CSP1**, **CSP2**, and **CSP3**).*

Sample	Selector; type of loading (nominal loading)	Loading [%]		C [%] *(mmol/g)*	H [%] *(mmol/g)*	N [%] *(mmol/g)*	Cl [%] *(mmol/g)*	N_3-groups *(µmol/g)*
		TGA	EA					
Cl-PS	–	-	**4.62**	**1.38**	**0.45**	**n.d.**	**0.96**	–
Az-PS	–	**3.65**	**4.69**	**1.46**	**0.41**	**1.21**	**0.05**	*288.8*
CSP1	**CS1** – clicked (9%)	**8.21**	**7.65**	**4.94** *(4.11)*	**0.54** *(5.44)*	**1.46** *(1.04)*	**2.21** *(0.63)*	–
CSP2	**CS1** – coated (9%)	**9.62**	**10.3**	**6.44** *(5.36)*	**0.75** *(7.42)*	**1.56** *(1.11)*	**2.37** *(0.66)*	–
CSP3	**CS1** – coated (20%)	**17.1**	**18.7**	**10.1** *(8.36)*	**0.97** *(9.62)*	**1.88** *(1.34)*	**4.56** *(1.28)*	–

2.5 Thermogravimetric analysis

TGA was performed on a TG 209 F1 Iris thermo-microbalance (Netzsch GmbH & Co. KG, Selb, Germany). About 10 to 15 mg of each sample was weighted and equilibrated at room temperature. The measurement was carried out under an oxidizing atmosphere (N_2:O_2 = 4:1, v/v) at a gas flow rate of 20 mL min^{-1}. The temperature was programmed at a gradient of 20 °C min^{-1}. A drying step at 105 °C (hold time: 10 min) was included to remove residual water before cooling back down to 30 °C and heating to 1000 °C (temperature program see **Figure S17**). All measurements were done in triplicate. Proteus software (version 8.0.3.0) was used for data processing TGA results.

To evaluate the results, the total mass loss between t = 20 min (weight after the drying step) and t = 65 min (weight after completion of the measurement) was determined for all samples. To exclude the mass loss due to the evaporation of residual water, the mass at t = 20 min was used as $m_{s,0}$ (original sample mass) to determine the total organic content (TOC). The TOC of

AzPS was calculated according to **Equation S4** with $m_{AzPS,0}$ as the original mass at t = 20 min and $m_{AzPS,1}$ as the final residual mass at t = 65 min. The selector loading L_{CS} was determined using the TOC of the respective CSPs according to **Equation S5** with $m_{s,0}$ as the original sample mass at t = 20 min and $m_{s,1}$ the final residual mass at t = 65 min using L_{AzPS} as a blank. All measurements were done in duplicate. The results are presented in **Table S4**.

Equation S4:

$$L_{AzPS}\ [wt\%] = \frac{m_{AzPS,0} - m_{AzPS,1}}{m_{AzPS,0}} \times 100\ \%$$

Equation S5:

$$L_{CS}\ [wt\%] = \frac{m_{s,0} - m_{s,1}}{m_{s,0}} \times 100\ \% - L_{AzPS}$$

Table S4: *TGA results of the CSs.*

Sample	wt% @ 20 min	wt% @ 65 min	Mass loss [wt%]	CS loading [wt%]	CS loading if B = 100 % (after drying)
AzPS	99.66	96.02	**3.65**	-	
CSP1	98.65	86.92	11.74	8.10	**8.21**
CSP2	98.96	85.80	13.16	9.52	**9.62**
CSP3	99.10	78.53	20.57	16.93	**17.08**

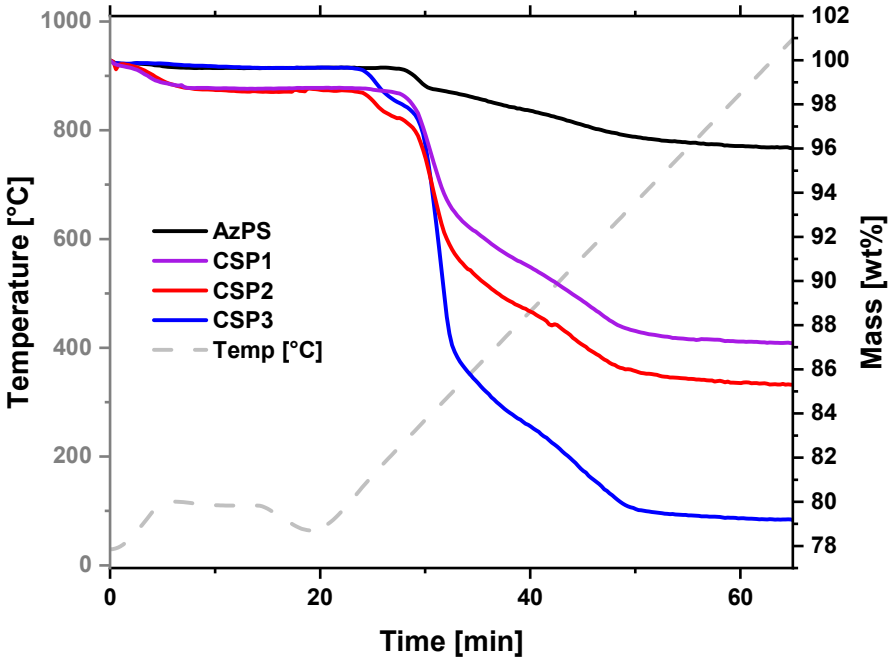


Figure S17: *TGA curves of* ***AzPS*** *(black),* ***CSP1*** *(purple),* ***CSP2*** *(red), and* ***CSP3*** *(blue) (raw data); the temperature program is depicted in grey (dashed line).*

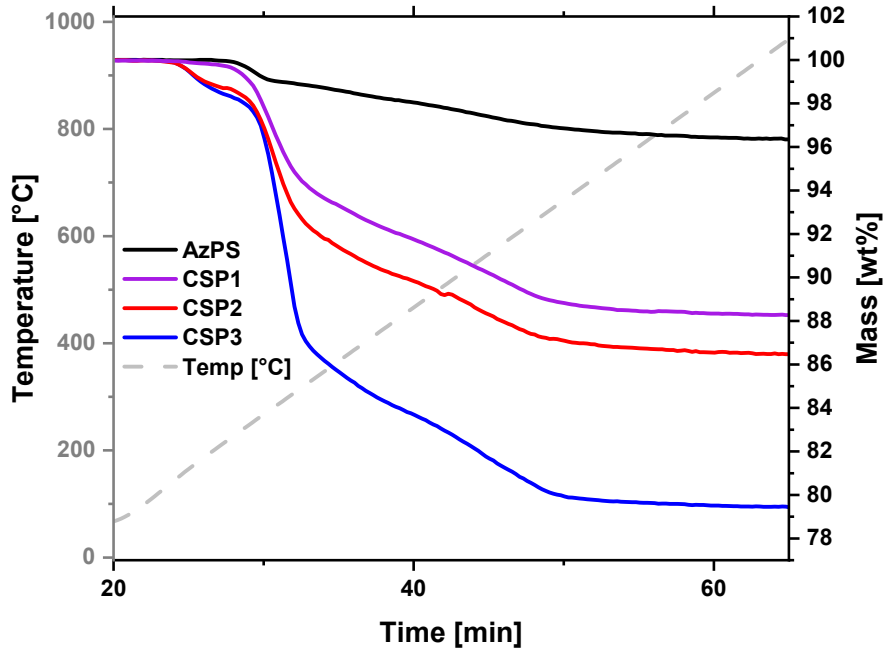


Figure S18: *TGA curves of* ***AzPS*** *(black),* ***CSP1*** *(purple),* ***CSP2*** *(red), and* ***CSP3*** *(blue) (normalized data); the temperature program is depicted in grey (dashed line).*

3 HPLC Testing

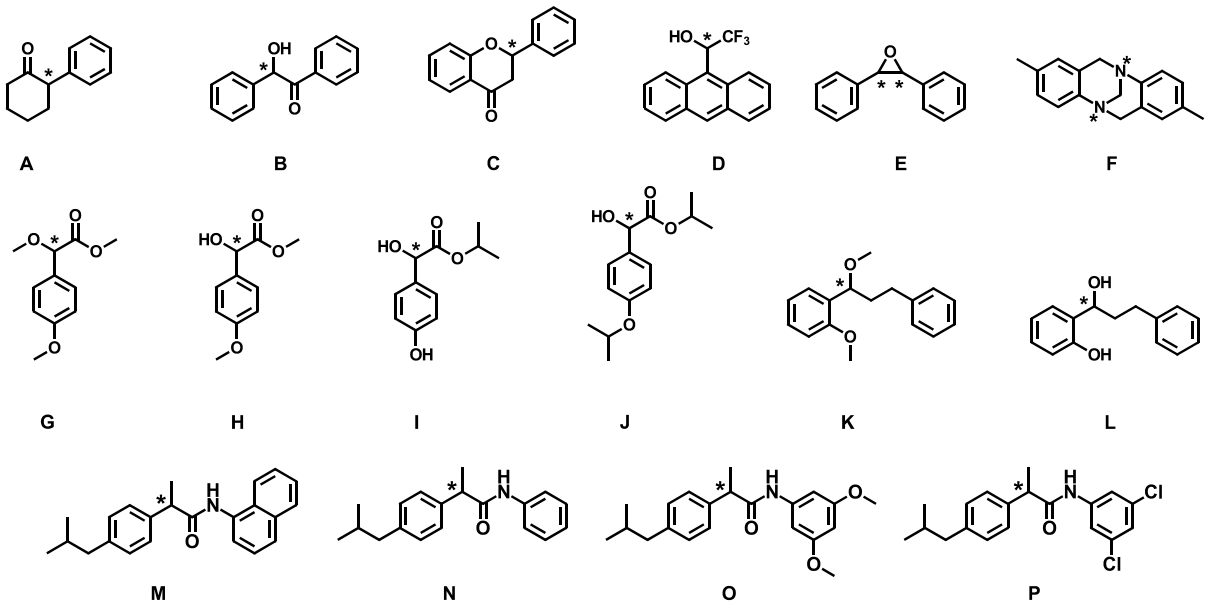


***Figure S19**: Chemical structures of the chiral analytes **A-P**.*

3.1 CSP1 (immobilized, 9 %)

3.1.1 Isocratic elution with *n*-Hex/*i*-PrOH 90/10 (v/v)

***Table S5**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH 90/10 (v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.910 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	2.054	2.268	0.0760	0.0840	1.26	1.49	1.19	**1.50**	4047	4039
B	2.591	2.725	0.1120	0.1140	1.85	1.99	1.08	**0.68**	2965	3165
C	1.701	1.755	0.0840	0.0710	0.87	0.93	1.07	**0.41**	2272	3385
D	1.153	1.194	0.1120	0.1410	0.27	0.31	1.17	**0.19**	587	397
E	1.122	1.217	0.0580	0.0620	0.23	0.34	1.45	**0.90**	2073	2135
F	1.961	2.030	0.0670	0.1000	1.15	1.23	1.07	**0.50**	4746	2283
G	2.163		0.0800		1.38				4050	
H	3.208		0.1570		2.53				2313	
I	3.494		0.1400		2.84				3451	
J	2.082	2.173	0.0920	0.0860	1.29	1.39	1.08	**0.59**	2837	3537
K	1.020		0.0640		0.12				1407	
L	1.483		0.0680		0.63				2635	
M	3.717	4.669	0.1380	0.1720	3.08	4.13	1.34	**3.24**	4019	4082
N	2.034	2.171	0.0870	0.0890	1.24	1.39	1.12	**0.89**	3028	3296
O	6.098	8.408	0.2510	0.3420	5.70	8.24	1.45	**3.95**	3270	3348
P	1.233	1.333	0.0890	0.0900	0.35	0.46	1.31	**0.63**	1063	1215

3.1.2 Isocratic elution with *n*-Hex/*i*-PrOH/EtOAc 94/1/5 (v/v/v)

***Table S6**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/EtOAc 94/1/5 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.909 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	2.257	2.564	0.0790	0.0870	1.48	1.82	1.23	**2.04**	4522	4812
B	3.350	3.669	0.1180	0.1330	2.69	3.04	1.13	**1.43**	4465	4216
C	1.844	1.918	0.0790	0.0690	1.03	1.11	1.08	**0.58**	3018	4281
D	1.436	1.585	0.1440	0.1690	0.58	0.74	1.28	**0.54**	551	487
E	1.156	1.265	0.0570	0.0580	0.27	0.39	1.44	**1.07**	2279	2635
F	3.229	3.340	0.0930	0.1820	2.55	2.67	1.05	**0.54**	6679	1866
G	2.704		0.0940		1.97				4584	
H	5.859		0.1920		5.45				5159	
I	12.607	13.165	0.5140	0.5620	12.87	13.48	1.05	**0.60**	3333	3040
J	3.040	3.105	0.1170	0.0740	2.34	2.42	1.03	**0.43**	3740	9754
K	1.047		0.0630		0.15				1530	
L	2.430		0.1010		1.67				3207	
M	6.603	8.411	0.2260	0.2880	6.26	8.25	1.32	**3.69**	4729	4725
N	2.918	3.257	0.1030	0.1140	2.21	2.58	1.17	**1.74**	4446	4522
O	10.032	15.022	0.3590	0.5510	10.04	15.53	1.55	**5.40**	4326	4118
P	1.401	1.523	0.0680	0.0710	0.54	0.68	1.25	**0.99**	2352	2549

3.1.3 Isocratic elution with *n*-Hex/*i*-PrOH/EtOAc 89/1/10 (v/v/v)

***Table S7**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/EtOAc 89/1/10 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.895 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	1.762	1.946	0.0680	0.0720	0.97	1.17	1.21	**1.47**	3720	4047
B	2.382	2.561	0.0960	0.1010	1.66	1.86	1.12	**1.03**	3411	3562
C	1.514		0.0950		0.69				1407	
D	1.212	1.266	0.0990	0.1150	0.35	0.41	1.17	**0.29**	830	671
E	1.079	1.140	0.0600	0.0590	0.21	0.27	1.33	**0.59**	1792	2068
F	2.615		0.1910		1.92				1038	
G	1.933		0.0750		1.16				3680	
H	3.526		0.1160		2.94				5119	
I	5.341	5.578	0.2170	0.2310	4.97	5.23	1.05	**0.61**	3356	3230
J	2.053		0.1050		1.29				2118	
K	0.990		0.0540		0.11				1862	
L	1.602		0.0790		0.79				2278	
M	3.661	4.631	0.1270	0.1590	3.09	4.17	1.35	**3.57**	4604	4700
N	1.840	2.034	0.0760	0.0810	1.06	1.27	1.21	**1.38**	3247	3493
O	4.270	6.479	0.1530	0.2310	3.77	6.24	1.65	**5.61**	4315	4358
P	1.135	1.188	0.0620	0.0600	0.27	0.33	1.22	**0.50**	1857	2172

3.1.4 Isocratic elution with *n*-Hex/*i*-PrOH/EtOAc 79/1/20 (v/v/v)

***Table S8**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/EtOAc 79/1/20 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.887 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	1.337	1.418	0.071	0.070	0.51	0.60	1.18	**0.66**	1965	2273
B	1.570	1.627	0.082	0.076	0.77	0.83	1.08	**0.42**	2031	2539
C	1.217		0.072		0.37				1583	
D	1.036		0.209		0.17				136	
E	1.01		0.073		0.14				1060	
F	1.898		0.130		1.14				1181	
G	1.387		0.063		0.56				2685	
H	2.003		0.077		1.26				3749	
I	2.266	2.325	0.081	0.085	1.55	1.62	1.04	**0.41**	4336	4145
J	1.400		0.068		0.58				2348	
K	0.944		0.054		0.06				1693	
L	1.171		0.061		0.32				2042	
M	1.983	2.405	0.078	0.090	1.24	1.71	1.39	**2.69**	3581	3956
N	1.262	1.350	0.079	0.078	0.42	0.52	1.23	**0.64**	1414	1660
O	1.912	2.653	0.083	0.105	1.16	1.99	1.72	**3.97**	2940	3537
P	0.995		0.106		0.12				488	

3.1.5 Isocratic elution with *n*-Hex/*i*-PrOH/THF 94/1/5 (v/v/v)

***Table S9**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/THF 94/1/5 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.899 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	2.133	2.386	0.0760	0.0830	1.37	1.65	1.21	**1.77**	4364	4578
B	3.170	3.902	0.1110	0.1540	2.53	3.34	1.32	**2.98**	4518	3557
C	1.816	1.888	0.0770	0.0690	1.02	1.10	1.08	**0.57**	3081	4148
D	1.389	1.488	0.1130	0.1190	0.55	0.66	1.20	**0.49**	837	866
E	1.132	1.247	0.0550	0.0570	0.26	0.39	1.49	**1.15**	2347	2652
F	2.452		0.1680		1.73				1180	
G	2.257		0.0860		1.51				3816	
H	4.636		0.2590		4.16				1775	
I	8.686		0.3320		8.66				3792	
J	2.571		0.0960		1.86				3973	
K	1.029		0.0560		0.14				1871	
L	1.996	2.069	0.0800	0.0770	1.22	1.30	1.07	**0.54**	3449	4000
M	5.966	7.788	0.2090	0.2750	5.64	7.66	1.36	**3.91**	4514	4443
N	2.620	3.044	0.0940	0.1080	1.91	2.39	1.25	**2.30**	4304	4401
O	7.864	13.345	0.2870	0.5100	7.75	13.84	1.79	**6.47**	4159	3793
P	1.342	1.471	0.0640	0.0670	0.49	0.64	1.29	**1.11**	2436	2670

3.1.6 Isocratic elution with *n*-Hex/*i*-PrOH/THF 89/1/10 (v/v/v)

***Table S10**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/THF 89/1/10 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.885 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	1.558	1.667	0.0660	0.0690	0.76	0.88	1.16	**0.92**	3087	3234
B	2.036	2.399	0.0810	0.0960	1.30	1.71	1.32	**2.23**	3500	3460
C	1.430		0.0810		0.62				1727	
D	1.169		0.1810		0.32				231	
E	1.045	1.096	0.0540	0.0540	0.18	0.24	1.32	**0.54**	2075	2282
F	1.749		0.0990		0.98				1729	
G	1.599		0.0660		0.81				3252	
H	2.652		0.1560		2.00				1601	
I	3.539		0.1350		3.00				3807	
J	1.683		0.0690		0.90				3296	
K	0.978		0.0580		0.11				1575	
L	1.387		0.0970		0.57				1133	
M	3.167	4.075	0.1120	0.1420	2.58	3.60	1.40	**3.74**	4430	4562
N	1.642	1.859	0.0690	0.0740	0.86	1.10	1.29	**1.68**	3137	3496
O	3.178	5.372	0.1180	0.1980	2.59	5.07	1.96	**6.50**	4018	4078
P	1.101	1.148	0.0610	0.0610	0.24	0.30	1.22	**0.44**	1805	1962

3.1.7 Isocratic elution with *n*-Hex/*i*-PrOH/THF 79/1/20 (v/v/v)

***Table S11**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/THF 79/1/20 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.885 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	1.207		0.0840		0.38				1144	
B	1.347	1.477	0.0660	0.0690	0.54	0.68	1.28	**1.08**	2308	2538
C	1.135		0.0680		0.29				1543	
D	1.011		0.1520		0.15				245	
E	0.984		0.0610		0.12				1442	
F	1.291		0.0770		0.47				1557	
G	1.197		0.0580		0.36				2360	
H	1.555		0.0830		0.77				1945	
I	1.626		0.0700		0.85				2989	
J	1.180		0.0580		0.35				2293	
K	0.934		0.0580		0.06				1437	
L	1.076		0.0590		0.23				1843	
M	1.631	1.948	0.0680	0.0770	0.86	1.22	1.42	**2.36**	3187	3546
N	1.145	1.222	0.0680	0.0690	0.31	0.39	1.29	**0.64**	1571	1738
O	1.471	2.069	0.0660	0.0830	0.68	1.36	2.01	**4.02**	2752	3443
P	0.970		0.0760		0.11				902	

3.1.8 Isocratic elution with *n*-Hex/*i*-PrOH/$CHCl_3$ 94/1/5 (v/v/v)

***Table S12**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/$CHCl_3$ 94/1/5 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.893 min) was determined using p-cymene. Sample **I** was insoluble in the mobile phase.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	3.058	3.567	0.1030	0.1230	2.42	2.99	1.24	**2.46**	4883	4659
B	5.843	6.264	0.1890	0.2290	5.54	6.01	1.09	**1.15**	5295	4145
C	2.336	2.456	0.0990	0.1000	1.62	1.75	1.08	**0.69**	3085	3342
D	2.847	3.670	0.2370	0.3100	2.19	3.11	1.42	**1.57**	799	776
E	1.463	1.644	0.0620	0.0620	0.64	0.84	1.32	**1.63**	3085	3895
F	3.991		0.2850		3.47				1086	
G	3.482		0.1210		2.90				4588	
H	10.380		0.5010		10.62				2378	
I	-	-	-	-						
J	5.214	5.435	0.2110	0.1910	4.84	5.09	1.05	**0.64**	3383	4486
K	1.040	1.100	0.0490	0.0920	0.16	0.23	1.41	**0.55**	2496	792
L	6.951	7.348	0.2630	0.2790	6.78	7.23	1.07	**0.84**	3870	3843
M	8.372	10.004	0.2970	0.3580	8.38	10.20	1.22	**2.69**	4402	4326
N	4.814	5.075	0.1850	0.2130	4.39	4.68	1.07	**0.75**	3751	3145
O	34.601	42.542	1.4200	1.7090	37.75	46.64	1.24	**2.71**	3289	3433
P	2.651	2.918	0.1010	0.1130	1.97	2.27	1.15	**1.40**	3817	3694

3.1.9 Isocratic elution with *n*-Hex/*i*-PrOH/$CHCl_3$ 89/1/10 (v/v/v)

***Table S13**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/$CHCl_3$ 89/1/10 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.890 min) was determined using p-cymene. Sample **I** was insoluble in the mobile phase.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	2.384	2.741	0.0840	0.1010	1.68	2.08	1.24	**2.13**	4462	4080
B	4.428	4.752	0.1500	0.1660	3.98	4.34	1.09	**1.17**	4828	4540
C	1.918	1.993	0.0820	0.0720	1.16	1.24	1.07	**0.57**	3031	4245
D	2.591	3.344	0.1880	0.2420	1.91	2.76	1.44	**1.83**	1052	1058
E	1.215	1.362	0.0580	0.0630	0.37	0.53	1.45	**1.35**	2431	2589
F	3.402		0.2740		2.82				854	
G	2.573		0.0950		1.89				4064	
H	7.519		0.3730		7.45				2251	
I	-	-	-	-						
J	4.077	4.196	0.1490	0.1280	3.58	3.71	1.04	**0.50**	4148	5953
K	1.058		0.1150		0.19				469	
L	6.039	6.463	0.2160	0.2310	5.79	6.26	1.08	**1.08**	4330	4337
M	6.716	7.912	0.2500	0.2970	6.55	7.89	1.21	**2.38**	3998	3932
N	3.864	4.052	0.1720	0.1590	3.34	3.55	1.06	**0.66**	2796	3598
O	21.514	26.453	0.8840	1.0380	23.17	28.72	1.24	**2.74**	3281	3598
P	2.212	2.413	0.0890	0.0970	1.49	1.71	1.15	**1.22**	3422	3428

3.1.10 Isocratic elution with *n*-Hex/*i*-PrOH/$CHCl_3$ 79/1/20 (v/v/v)

***Table S14**: HPLC data of **CSP1** for isocratic elution with n-Hex/i-PrOH/$CHCl_3$ 79/1/20 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.884 min) was determined using p-cymene. Sample **I** was insoluble in the mobile phase.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	1.741	1.937	0.0680	0.0730	0.97	1.19	1.23	**1.55**	3632	3901
B	2.925	3.100	0.1140	0.1190	2.31	2.51	1.09	**0.86**	3647	3760
C	1.496		0.0940		0.69				1403	
D	2.030	2.546	0.1590	0.1990	1.30	1.88	1.45	**1.52**	903	907
E	1.524	1.623	0.0610	0.0620	0.72	0.84	1.15	**0.92**	3458	3796
F	2.495		0.1620		1.82				1314	
G	1.752		0.0700		0.98				3470	
H	4.504		0.2550		4.10				1728	
I	-	-	-	-						
J	2.686		0.1370		2.04				2130	
K	0.985		0.0840		0.11				762	
L	4.153	4.479	0.1480	0.1580	3.70	4.07	1.10	**1.21**	4362	4452
M	3.768	4.333	0.1380	0.1600	3.26	3.90	1.20	**2.09**	4130	4063
N	2.401	2.530	0.1100	0.1080	1.72	1.86	1.09	**0.68**	2639	3040
O	9.527	11.937	0.3740	0.4580	9.78	12.50	1.28	**3.06**	3595	3763
P	1.628	1.752	0.0760	0.0780	0.84	0.98	1.17	**0.91**	2542	2795

3.1.11 Stability test of CSP1 with *n*-Hex/*i*-PrOH/$CHCl_3$ 79/1/20 (v/v/v)

***Table S15**: HPLC data of the stability test for **CSP1** with isocratic elution with n-Hex/i-PrOH/$CHCl_3$ 79/1/20 (v/v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.884 min) was determined using p-cymene. The column was constantly flushed with mobile phase for a total of 33 h, injecting analyte **O** every 4 h. Retention times t_{r1} and t_{r2} differ from the ones obtained for analyte **O** during analyte screening (see above, section 3.1.10, **Table S14**) due to the $CHCl_3$ used for the mobile phase (for chiral analyte screening measurements: 99.8 % stabilized with 1 % ethanol (Sigma-Aldrich, Vienna, Austria) vs. for stability test: HPLC grade, stabilized with amylene (TCI Europe N.V., Zwijndrecht, Belgium), leading to enhanced retention of the analyte.)*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
O - 0 h	12.045	15.084	0.4900	0.5970	12.63	16.06	1.27	2.96	3348	3537
O - 4 h	12.379	15.394	0.4890	0.5950	13.00	16.41	1.26	2.95	3550	3708
O - 8 h	12.034	15.049	0.4900	0.5960	12.61	16.02	1.27	2.94	3341	3532
O - 12 h	12.020	15.033	0.4900	0.5950	12.60	16.01	1.27	2.94	3334	3536
O - 16 h	11.994	14.999	0.4900	0.5950	12.57	15.97	1.27	2.93	3319	3520
O - 20 h	11.964	14.963	0.4900	0.5940	12.53	15.93	1.27	2.93	3303	3515
O - 24 h	11.941	14.940	0.4900	0.5950	12.51	15.90	1.27	2.92	3290	3493
O - 28 h	11.896	14.829	0.4890	0.5910	12.46	15.77	1.27	2.88	3279	3488
O - 32 h	11.872	14.824	0.4890	0.5920	12.43	15.77	1.27	2.89	3265	3474

3.2 CSP2 (coated, 9 %)

3.2.1 Isocratic elution with *n*-Hex/*i*-PrOH 90/10 (v/v)

***Table S16**: HPLC data of **CSP2** for isocratic elution with n-Hex/i-PrOH 9/1 (v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.910 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	1.963	2.192	0.1230	0.1400	1.16	1.41	1.22	**0.97**	1411	1358
B	2.478		0.1200		1.72				2362	
C	1.650	1.726	0.0670	0.0850	0.81	0.90	1.10	**0.58**	3360	2284
D	1.128		0.0930		0.24				815	
E	1.130		0.0950		0.24				784	
F	1.139		0.1070		0.25				628	
G	2.202		0.1830		1.42				802	
H	3.066		0.1980		2.37				1328	
I	2.862		0.1740		2.15				1499	
J	1.915	2.013	0.0930	0.1230	1.10	1.21	1.10	**0.53**	2349	1484
K	0.992		0.0710		0.09				1081	
L	1.407		0.0700		0.55				2238	
M	3.136	3.875	0.1870	0.2350	2.45	3.26	1.33	**1.87**	1558	1506
N	1.765	1.875	0.0970	0.1230	0.94	1.06	1.13	**0.58**	1834	1287
O	5.047	7.346	0.3780	0.5640	4.55	7.07	1.56	**2.43**	988	940
P	1.150	1.215	0.0760	0.0950	0.26	0.34	1.27	**0.44**	1268	906

3.3 CSP3 (coated, 20 %)

3.3.1 Isocratic elution with *n*-Hex/*i*-PrOH 90/10 (v/v)

***Table S17**: HPLC data of **CSP3** for isocratic elution with n-Hex/i-PrOH 9/1 (v/v) (retention time (t_{r1}, t_{r2}); full width at half maximum (w_1, w_2); retention factor (k_1, k_2); selectivity (α); resolution (R); theoretical plate number (N_1, N_2). The dead time (t_0 = 0.874 min) was determined using p-cymene.*

Analyte	t_{r1} [min]	t_{r2} [min]	w_1 [min]	w_2 [min]	k_1	k_2	α	R	N_1	N_2
A	2.926	3.405	0.1110	0.1410	2.35	2.90	1.23	**2.09**	3850	3231
B	3.811		0.1670		3.36				2885	
C	2.190	2.359	0.0950	0.1010	1.51	1.70	1.13	**0.98**	2944	3022
D	1.123		0.0970		0.28				743	
E	1.247	1.463	0.0560	0.0620	0.43	0.67	1.58	**1.99**	2747	3085
F	2.130	2.353	0.1050	0.1180	1.44	1.69	1.18	**1.12**	2280	2203
G	3.246	3.505	0.1270	0.1410	2.71	3.01	1.11	**1.10**	3619	3423
H	5.058	5.140	0.1700	0.1810	4.79	4.88	1.02	**0.27**	4904	4468
I	4.550		0.2380		4.21				2025	
J	2.880	3.117	0.1290	0.1430	2.30	2.57	1.12	**0.99**	2761	2632
K	1.057		0.0900		0.21				764	
L	1.745		0.0930		1.00				1950	
M	5.107	6.593	0.2530	0.3310	4.84	6.54	1.35	**2.66**	2257	2198
N	2.359	2.586	0.1120	0.1250	1.70	1.96	1.15	**1.08**	2458	2371
O	9.091	14.040	0.5290	0.8180	9.40	15.06	1.60	**3.56**	1636	1632
P	1.256	1.403	0.0780	0.0850	0.44	0.61	1.38	**1.01**	1436	1509

4 References

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