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Letter

One out of Four: Kinetic Resolution of Stereoisomeric Mixtures of Secondary Alcohols with a Quaternary Carbon Atom in the β-Position by Cu−H-Catalyzed Enantioselective Silylation

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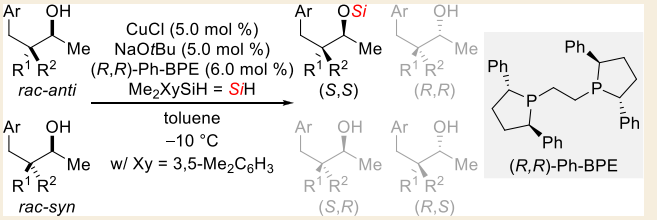
ABSTRACT: An enantio- and diastereoselective Cu−H-catalyzed silylation of acyclic secondary alcohols with a vicinal quaternary stereocenter is reported. The reaction kinetically selects one out of four stereoisomers, affording the fastest-reacting stereoisomer as the silyl ether in enantio- and diastereomerically enriched form. The obtained motif with a quaternary carbon atom in the β-position of the hydroxy group is otherwise not easy to access.

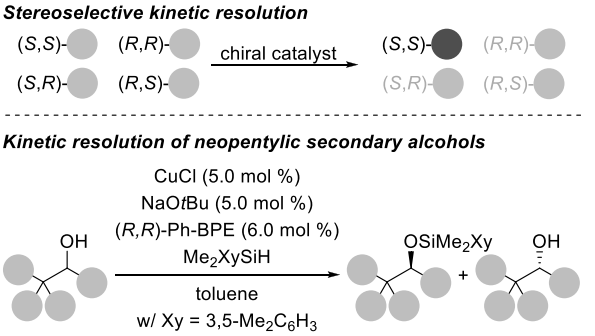


KEYWORDS: *asymmetric catalysis, copper, dehydrogenative coupling, kinetic resolution, quaternary centers, silicon*

Asymmetric synthesis can be achieved in numerous ways.[1] For example, stereoconvergent[2,3] as well as stereodivergent[4−6] methods are viable strategies. These have been employed for establishing a single stereocenter with great success, but there are fewer methods available for simultaneous, *independent* control over the formation of vicinal stereocenters.[7] A possible approach toward full control of absolute and relative configuration is stereodivergent dual catalysis.[8,9] Starting from the same set of prochiral starting materials, the use of the different combinations of two enantiomeric catalysts leads to the stereoselective formation of all four stereoisomers. Alternatives to that challenging synthesis of a single stereoisomer are dynamic kinetic asymmetric transformations where a mixture of stereoisomers as starting material converges to one product stereoisomer.[10] All of the aforementioned techniques become exceedingly complicated with one of the vicinal stereogenic carbon atoms being quaternary. Such molecules are interesting candidates for stereoselective kinetic resolution[11,12] in order to preferentially convert one stereoisomer out of a mixture of four (Scheme 1, top). The downside is low yields, but the approach can nevertheless be especially useful for motifs containing quaternary carbon atoms.

With our long-standing interest in silylation-based kinetic resolution of alcohols,[13−15] we set out to apply their enantioselective Cu−H-catalyzed Si−O coupling with achiral tertiary hydrosilanes[16−18] to the problem outlined above. We recently showed that sterically congested secondary alcohols with a nonstereogenic quaternary carbon atom in the β-position can be kinetically resolved with good selectivity factors.[19] We found this substance class particularly promising to probe their stereoselective kinetic resolution (Scheme 1, bottom).[20,21] The synthesis of similar acyclic alcohols with a vicinal quaternary stereocenter is mainly achieved by reagent- and catalyst-controlled carbonyl allylation to arrive at the corresponding homoallylic alcohols.[22−31] In this Letter, we present an enantio- and diastereoselective Cu−H-catalyzed silylation of stereoisomeric mixtures of those alcohols that enriches the fastest-reacting stereoisomer as the silyl ether.

Scheme 1. Kinetic Resolution of Neopentylic Secondary Alcohols Containing an Achiral Quaternary Center



Guided by our earlier study using 3,5-xylyl-substituted tertiary hydrosilane **2e**,[19] we chose the acyclic secondary alcohol *rac*-**1a** and subjected each diastereomer separately to the reaction conditions (Scheme 2, top). The diastereomers reacted with different selectivity factors $s = 26$ and $s = 18$. The

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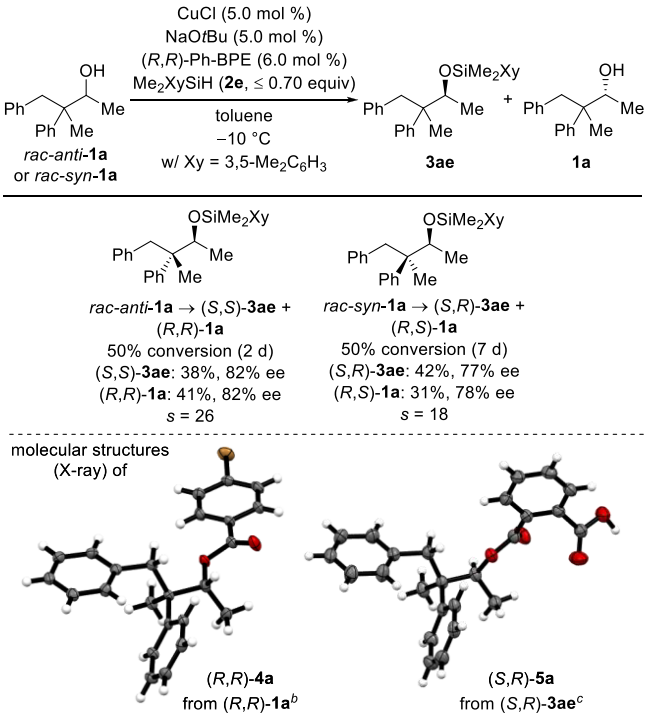
Scheme 2. Cu–H-Catalyzed Kinetic Resolution of Individual Racemic Diastereomers *anti*-1a and *syn*-1a[a]

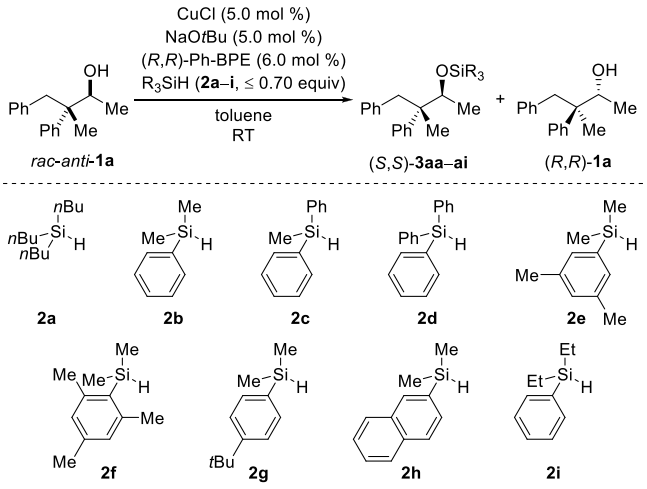


[a]Unless otherwise noted, reactions were performed on a 0.2 mmol scale and monitored by ^{1}H NMR spectroscopy or GLC analysis. Conversion was estimated by HPLC analysis and calculated according to conversion = $ee_{unreacted\ alcohol}/(ee_{silyl\ ether} + ee_{unreacted\ alcohol})$. Enantiomeric excesses were determined by HPLC analysis on chiral stationary phases (after cleavage of the silyl ether). With these data, selectivity factors were calculated according to $s = \ln[(1 - C)(1 - ee)]/\ln(1 - C)(1 + ee)]$, where $ee = ee_{unreacted\ alcohol}/100$ and C = conversion/100. [b]Obtained by derivatization of (*R*,*R*)-**1a** with 4-bromobenzoyl chloride. [c]Obtained from (*S*,*R*)-**3ae** by deprotection of the silyl ether and derivatization with phthaloyl chloride.

reaction of *anti*-**1a** was substantially faster than that of *syn*-**1a** (2 days versus 7 days), thereby qualifying the catalytic system for the stereoselective kinetic resolution. For alcohol (*R*,*R*)-**1a** from *anti*-**1a** and (*S*,*R*)-**3ae** from *syn*-**1a**, the relative and absolute configurations were assigned by X-ray diffraction after derivatization to the 4-bromobenzoate (*R*,*R*)-**4a** and phthalate derivative (*S*,*R*)-**5a**, respectively (Scheme 2, bottom). The asymmetric induction is in agreement with previous results.[16−19]

Several hydrosilanes **2** were tested using fast-reacting *anti*-**1a** as the model substrate (Table 1). There was no reaction with nBu_3SiH (**2a**; entry 1).[16] A set of $Me_{3-n}Ph_nSiH$ with n = 1 to 3 was probed (entries 2−4). Me_2PhSiH (**2b**) was sufficiently reactive and led to s = 12; significantly lower selectivity factors were obtained with sterically more hindered $MePh_2SiH$ (**2c**) and Ph_3SiH (**2d**). Similar to our previous study,[19] the 3,5-xylyl-substituted Me_2XySiH **2e** showed a good selectivity factor of 15 (entry 5). Conversely, mesityl-substituted **2f** was far less effective (entry 6). Moderate to good selectivity factors were seen with *tert*-butyl-substituted **2g** (s = 9.5; entry 7) and naphth-2-yl-derived **2h** (s = 14; entry 8). Ethyl instead of methyl groups at the silicon atom were detrimental (**2i**; entry 9).

Table 1. Hydrosilane Screening[a,b]

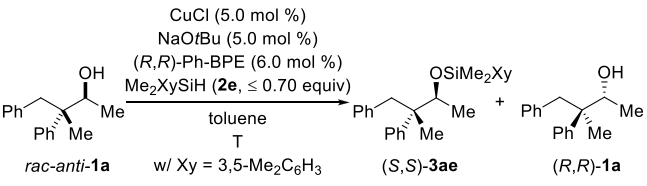


entry	hydrosilane	conv (%)	time (h)	ee of (*S*,*S*)-**3a** (%)	ee of (*R*,*R*)-**1a** (%)	*s*
1	**2a**	nr				
2	**2b**	55	18	66	82	12
3	**2c**	68	18	42	90	6.8
4	**2d**	50	18	17	17	1.6
5	**2e**	46	18	77	65	15
6	**2f**	52	42	32	33	2.6
7	**2g**	48	18	67	63	9.5
8	**2h**	50	18	73	73	14
9	**2i**	50	18	42	42	3.7

[a]See the caption of Scheme 2 for details. [b]See the Supporting Information for the complete optimization.

To identify an acceptable compromise between selectivity and reaction time, further optimization included variation of the reaction temperature (Table 2). At −20 °C, an excellent s value of 70 was achieved in the kinetic resolution of racemic *anti*-**1a** with hydrosilane **2e** (entry 1). However, the reaction time of 10 days is not practical. A stepwise increase of the reaction temperature to −15, −10, and 0 °C resulted in higher reaction rates while maintaining high levels of selectivity (entries 2−4). A selectivity factor of 25 and a reaction time of 46 h are still synthetically useful, and we proceeded with −10 °C as the reaction temperature (entry 3). For completion, the

Table 2. Temperature Screening[a,b]



entry	*T* (°C)	time[c]	conv[c] (%)	ee of (*S*,*S*)-**3ae**[c] (%)	ee of (*R*,*R*)-**1a**[c] (%)	*s*[c]
1	−20	10 d	49	92	89	70
2	−15	67 h	47	88	79	41
3	−10	46 h	51	80	85	25
4	0	29 h	46	82	71	21
5	rt	19 h	45	75	61	13

[a]See the caption of Scheme 2 for details. [b]See the Supporting Information for the complete optimization. [c]Average values of multiple runs.

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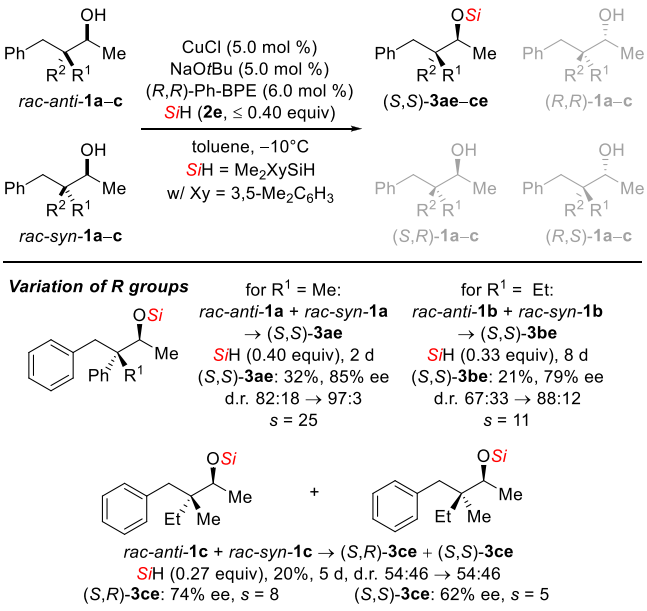
above trend continued when running the kinetic resolution at room temperature (entry 5).

We then applied the optimized conditions to the resolution of a mixture of four stereoisomers (Scheme 3). To avoid

Scheme 3. Substrate Scope I[a]

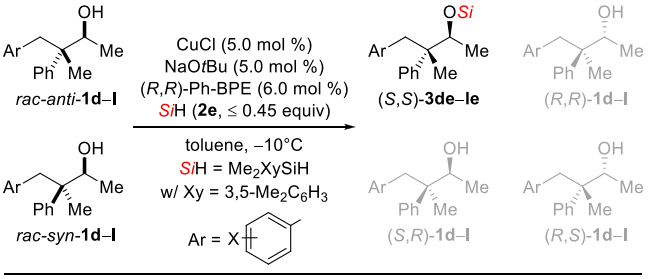


[a]Unless otherwise noted, reactions were performed on a 0.2 mmol scale and monitored by ^{1}H NMR spectroscopy. Conversion was estimated by HPLC analysis and calculated according to conversion = $ee_{unreacted\ alcohol}/(ee_{silyl\ ether} + ee_{unreacted\ alcohol})$. With these data, selectivity factors were calculated according to $s = \ln[(1 - C)(1 - ee)]/\ln(1 - C)(1 + ee)]$, where $ee = ee_{unreacted\ alcohol}/100$ and C = conversion/100. Diastereomeric ratios were determined by HPLC analysis and confirmed by ^{1}H NMR spectroscopy. Enantiomeric excesses were determined by HPLC analysis on chiral stationary phases (after cleavage of the silyl ether).

chromatographic separation of diastereomeric products, we adjusted the amount of the hydrosilane to the diastereomeric ratio of the starting material, that is, the faster-reacting diastereomer. Model substrate *rac*-**1a** was subjected to the Cu−H-catalyzed kinetic resolution as a mixture of the diastereomers *rac-anti*-**1a** and *rac-syn*-**1a** with dr = 82:18. We were pleased to find that silyl ether (*S*,*S*)-**3ae** (*anti*) formed from the major diastereomer *rac-anti*-**1a** with high diastereoselectivity (dr = 97:3); the *s* value was also high (*s* = 25). When the methyl was replaced with an ethyl group at the quaternary carbon atom as in *rac*-**1b**, the reaction proceeded with a decreased selectivity factor (*s* = 11 for (*S*,*S*)-**3be**); the diastereoselection was still satisfactory (dr = 88:12). A fully alkyl-substituted quaternary carbon atom as in **1c** (dr = 55:45) did not allow for the kinetic resolution of the diastereomers (dr = 54:46 for **3ce**). The corresponding pairs of enantiomers were, however, resolved with moderate selectivity factors of *s* = 8 for (*S*,*R*)-**3ce** and *s* = 5 for (*S*,*S*)-**3ce**.

Maintaining the established substitution pattern at the quaternary center, we investigated the electronic variation of the benzylic aryl group (Scheme 4). Functional groups are generally well tolerated for this transformation.[16−19] Derivatives with electron-withdrawing and -donating groups in the *para*- and *ortho*-positions, such as *rac*-**1d**−**i** and **k**−**l**, successfully underwent the diastereo- and enantioselective

Scheme 4. Substrate Scope II[a]



Variation of X

for X = 4-F:
rac-anti-**1d** + *rac-syn*-**1d** → (*S*,*S*)-**3de**
*Si*H (0.40 equiv), 7 d
(*S*,*S*)-**3de**: 11%, 88% ee
d.r. 83:17 → >99:1
s = 24

for X = 4-Cl:
rac-anti-**1e** + *rac-syn*-**1e** → (*S*,*S*)-**3ee**
*Si*H (0.40 equiv), 7 d
(*S*,*S*)-**3ee**: 30%, 87% ee
d.r. 84:16 → 97:3
s = 25

for X = 4-Br:
rac-anti-**1f** + *rac-syn*-**1f** → (*S*,*S*)-**3fe**
*Si*H (0.40 equiv), 9 d
(*S*,*S*)-**3fe**: 29%, 86% ee
d.r. 91:9 → >99:1
s = 21

for X = 4-I:
rac-anti-**1g** + *rac-syn*-**1g** → (*S*,*S*)-**3ge**
*Si*H (0.45 equiv), 5 d
(*S*,*S*)-**3ge**: 37%, 90% ee
d.r. 91:9 → 96:4
s = 36

for X = 2-Br:
rac-anti-**1h** + *rac-syn*-**1h** → (*S*,*S*)-**3he**
*Si*H (0.40 equiv), 5 d
(*S*,*S*)-**3he**: 31%, 84% ee
d.r. 78:22 → 96:4
s = 22

for X = 4-Me:
rac-anti-**1i** + *rac-syn*-**1i** → (*S*,*S*)-**3ie**
*Si*H (0.45 equiv), 7 d
(*S*,*S*)-**3ie**: 32%, 90% ee
d.r. 90:10 → 99:1
s = 35

for X, X = 3-Me, 5-Me:
rac-anti-**1j** + *rac-syn*-**1j** → (*S*,*S*)-**3je**
*Si*H (0.40 equiv), 7 d
(*S*,*S*)-**3je**: 36%, 43% ee
d.r. 80:20 → 99:1
s = 9

for X = 4-OMe:
rac-anti-**1k** + *rac-syn*-**1k** → (*S*,*S*)-**3ke**
*Si*H (0.55 equiv), 11 d
(*S*,*S*)-**1k**: 44%, 91% ee
d.r. 90:10 → >99:1
s = 24

for X, X = 3-OMe, 5-OMe:
rac-anti-**1l** + *rac-syn*-**1l** → (*S*,*S*)-**3le**
*Si*H (0.40 equiv), 14 d
(*S*,*S*)-**3le**: 24%, 86% ee
d.r. 79:21 → 97:3
s = 22

[a]See the caption of Scheme 3 for details.

silylation with high selectivity factors. For 3,5-disubstituted substrate *rac*-**1j**, the *s* value decreased while the diastereoselection remained at a high level.

In conclusion, we have developed a method for the enantio- and diastereoselective kinetic resolution of acyclic secondary alcohols with two vicinal stereocenters[32] by applying an adapted protocol of our Cu−H-catalyzed enantioselective silylation. This procedure allows for selective silylation of one stereoisomer out of a mixture of four. By this, chiral neopentylic alcohol motifs can be accessed without the need for prior separation of the diastereomers.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsorginorgau.1c00050.

General procedures, experimental details, characterization, and spectral data for all new compounds and crystal data and structural refinement for compounds (*R*,*R*)-**4a** and (*S*,*R*)-**5a** (PDF)

Accession Codes

CCDC 2103765 and 2120080 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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