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Short communication

Self-disproportionation of enantiomers of heterocyclic compounds having a tertiary trifluoromethyl alcohol center on chromatography with a non-chiral system

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ABSTRACT

Self-disproportionation of enantiomers of heterocycles having a tertiary trifluoromethyl alcohol center on an achiral silica-gel stationary phase is discussed. During the chromatographic separation of an enantiomerically enriched mixture of 1-(3,4-dimethoxyphenethyl)-3-hydroxy-3-(trifluoromethyl)-6,7-dihydro-1*H*-indole-2,4(3*H*,5*H*)-dione (**1**) by eluting with ether on a non-chiral regular silica-gel significant enantiomeric enrichment was observed. Separation of non-racemic samples of **1** with enantiomeric excess values of 10–54% was carefully investigated: enantiomerically pure **1** with 99.9% ee was obtained by the use of **1** with at least 40% ee. A remarkable enantiomeric enrichment in the faster eluting fractions was also observed for compound **1** with only 30% ee to transform into 80% ee. Other enantiomeric mixtures of heterocyclic molecules containing a trifluoromethyl alcohol moiety at their quaternary carbon center were also examined from an SDE view point.

1. Introduction

The unique chemical and physical properties as well as biological activities of fluorinated compounds have made these molecules attractive targets for organic and medicinal chemists [1]. Especially, heterocyclic compounds containing a trifluoromethyl group constitute a major contributing nucleus in agricultural and medicinal chemistry [2], and thus the development of a simple and flexible method to generate a trifluoromethylated heterocyclic system has received much attention [3]. We considered that incorporating a tertiary trifluoromethyl alcohol stereocenter into heterocycles could provide a novel drug candidate where the unique properties of tertiary trifluoromethyl alcohols may contribute to new biological activities. The key to constructing these molecules is indeed the control of absolute stereochemistry at their trifluoromethyl alcohol moieties. Two different basic approaches have been considered; the direct introduction of trifluoromethyl group into non-fluorinated molecules and the synthesis of trifluoromethylated compounds via a building block strategy in which the fluorinated substrates are used as starting materials [4]. Furthermore, each approach can be divided into two main categories: classical chiral auxiliary-controlled diastereoselective synthesis and the recently established enantioselective approach via chiral catalysts which is now very stereospecific [5]. However, obtaining enantiomerically pure molecules with >99% ee still remains methodologically unexploited. Whenever an optically pure enantiomer cannot be obtained in asymmetric synthesis, it is necessary to employ a resolution to isolate a pure form, for example by recrystallization [6]. However, this approach is applicable only to enantiomerically enriched crystalline samples, and liquid samples are generally difficult to obtain in an enantiomerically pure state. During the course of our research program in the design and synthesis of biologically attractive fluorinated compounds [7], we required an enantiomerically pure sample of 1-(3,4-dimethoxyphenethyl)-3-hydroxy-3-(trifluoromethyl)-6,7-dihydro-1*H*-indole-2,4(3*H*,5*H*)-dione (**1**) (Fig. 1).

We report herein on the chromatographic purification of the non-racemic mixtures of **1** by regular silica-gel column chromatography under achiral conditions based on the concept of "self-disproportionation of enantiomers" (SDE). Remarkable amplification of the SDE of **1** on achiral phase chromatography has been observed. Enantiomerically pure **1** with 99.9% ee was separated from the sample of **1** with only 40% ee without any help of chiral sources. Other enantiomeric mixtures of heterocyclic molecules containing a trifluoromethyl alcohol moiety at their quaternary carbon center were also examined from an SDE view point.

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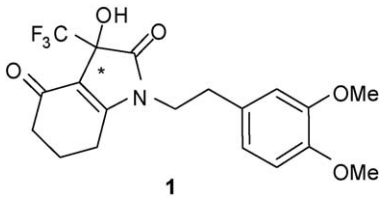


1

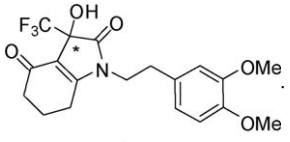
Fig. 1. Structure of 1-(3,4-dimethoxyphenethyl)-3-hydroxy-3-(trifluoromethyl)-6,7-dihydro-1*H*-indole-2,4(3*H*,5*H*)-dione (**1**).

2. Results and discussion

The term, self-disproportionation of enantiomers, or SDE, was introduced by Soloshonok in 2006 to describe a phenomenon, namely the transformation of an enantiomerically enriched system resulting in the formation of fractions of a different, as compared with the original, proportion of the enantiomers [8]. This phenomenon is fundamentally general for any enantiomeric mixtures and could be observed under any physical processes such as chromatography, sublimation and distillation [9]. Therefore the phenomenon itself might not be surprising; however, reports of SDE are really limited [10] because there is no general rule on the relationship between the phenomenon and compound structures; and consequently, it is impossible to predict whether substrates indicate this phenomenon. Soloshonok recently suggested that fluorinated compounds, especially involving a trifluoromethyl group at their stereogenic center, highly likely possess a significant potential of the SDE phenomenon of high magnitude. We hence attempted the purification of **1** with low to medium enantiomeric excesses to obtain an optically pure form on column chromatography with an achiral silica-gel (Table 1).

An experiment was begun to examine whether the SDE occurs for **1** based on conventional chromatographic conditions with regular silica-gel on an achiral stationary phase. Partially resolved

Table 1
Optimization of self-disproportionation of enantiomers of **1** on achiral silica-gel chromatography



1

Run	Starting ee of **1** (%)	Eluent	Columns (mm)	% ee min	% ee max	Δee
1[a]	52.3	$CHCl_3$	20 × 50	50.6	57.1	6.5
2[a]	53.9	H/A[d]	20 × 50	42.4	63.8	21.4
3[a]	52.0	Et_2O	20 × 50	34.1	66.8	32.7
4[b]	47.3	H/A[d]	20 × 50	33.0	60.1	27.1
5[b]	47.3	H/A[d]	10 × 50	39.0	61.4	22.4
6[b]	47.3	H/A[d]	30 × 50	34.8	61.0	26.2
7[b]	47.3	H/A[d]	20 × 20	27.9	61.2	33.3
8[b]	47.3	H/A[d]	20 × 80	25.0	80.2	55.2
9[b]	47.3	H/A[d]	20 × 110	33.6	76.0	42.4
10[b]	47.3	Et_2O	20 × 80	29.2	96.9	67.7
11[b]	52.1	Et_2O	20 × 80	33.0	99.9	66.9
12[c]	52.0	Et_2O	26 × 80	28.3	99.9	71.6
13[c]	40.0	Et_2O	26 × 80	25.1	99.9	74.8
14[c]	30.3	Et_2O	26 × 80	17.0	79.7	62.7
15[c]	9.9	Et_2O	26 × 80	4.2	44.6	40.4

[a] Regular silica-gel packed in glass column was used under atmospheric pressure.
[b] Flash silica-gel in glass column was used under atmospheric pressure.
[c] Flash silica-gel in polypropylene column was used under medium pressure (10 mL/min).
[d] H/A: hexane/EtOAc = 1:2.

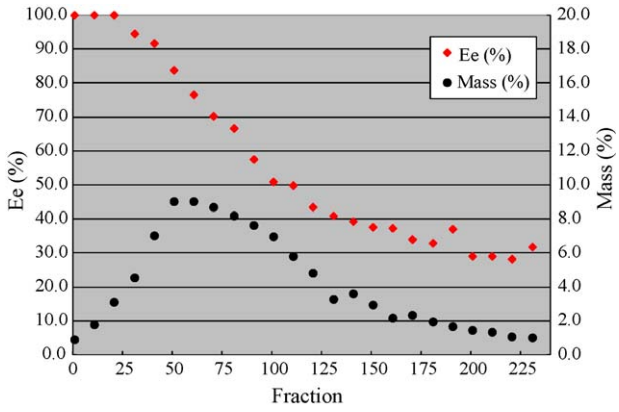


Fig. 2. Details of chromatography of **1** (52.0% ee) by medium-pressure liquid chromatography on achiral flash silica-gel.

(*ca.* 50% ee) compound **1** [11], which was prepared by HPLC enantiomer resolution of racemic **1** using Daicel CHIRALPAK AD-3, served as a loading substrate. Table 1 shows the data for the SDE of **1** on achiral silica-gel chromatography with different eluting conditions. We first examined the purification of **1** on a glass column filled with regular silica-gel (KANTO CHEMICAL CO., INC., Silica Gel 60N, spherical, neutral, 63–210 μm) as stationary phase at atmospheric pressure. The difference between the minimum and maximum ee for the chromatographic fractions is shown as an evaluation value of this phenomenon. The SDE effect was not observed when $CHCl_3$ was used as eluent (Δee, 6.5, run 1). The extent of SDE was pronounced when hexane/EtOAc (1/2) or ether was used as the eluent (runs 2 and 3). Next, purification was attempted under hexane/EtOAc conditions but using flash silica-gel (KANTO CHEMICAL CO., INC., Silica Gel 60N, spherical, neutral, 40–50 μm) as column packing material with different diameters/lengths of column (runs 4–9). The highest Δee for **1** was found to be 55.2% on a column of 20 mm diameter and 80 mm length, filled with flash silica-gel. The Δee value was more pronounced (67.7%) when ether was used as the eluent under the same conditions and separation of 99.9% ee of **1** was finally achieved from **1** with 52% ee as the loading sample. The Δee value was also slightly improved to 71.6% at medium-pressure chromatographic conditions with flash silica-gel filled in a polypropylene column (run 12). It is interesting to note that we observed a substantial SDE effect >99.9% ee (Δee value of 74.8%) with only 40% ee of **1** as starting substrate (run 13). According to the report by Soloshonok, the ee of the starting substrates tends to require at least 66.6% ee to achieve 99.9% ee of

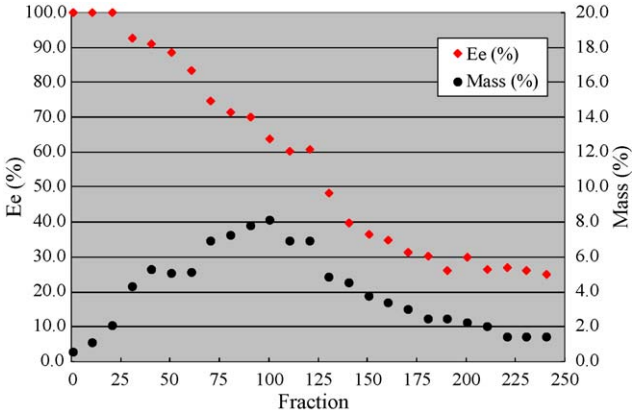


Fig. 3. Details of chromatography of **1** (40.0% ee) by medium-pressure liquid chromatography on achiral flash silica-gel.

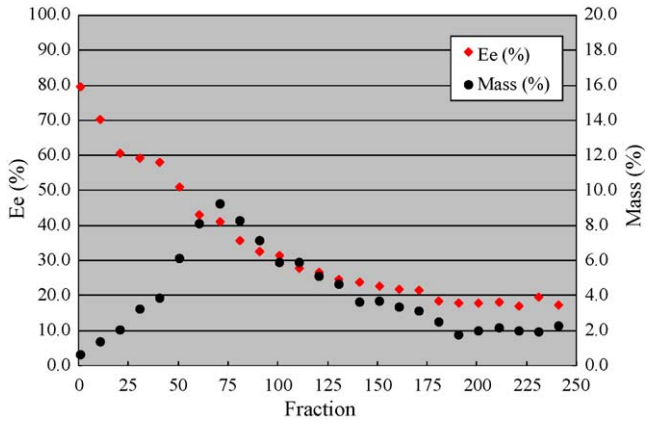


Fig. 4. Details of chromatography of **1** (30.3% ee) by medium-pressure liquid chromatography on achiral flash silica-gel.

separation. The Δee value decreased with a decrease in the enantiomeric purity of the loading samples (runs 14 and 15).

We next investigated the relationship between the ee value and mass of each fraction, which was estimated based on the peak area of **1** on HPLC analysis since the total recovery of **1** was quantitative at the end of chromatographic separation for each experiment. Figs. 2–5 show the details of chromatography of **1** with ee values of 52, 40, 30 and 10% with elution by ether using flash silica-gel packed in a polypropylene column under medium pressure (Figs. 2–5). The first fraction always has the highest ee value and the ee values will decrease as the number of fractions increases. Enantiomerically pure compound **1** can be separated when the staring ee is higher than 40%, 5.7% from **1** with 52% ee and 3.7% from **1** with 40% ee (Figs. 2 and 3). Although the masses are very low, this methodology could be useful if a very small amount of samples is sufficient for use, for example in biological screenings.

With these results in hand, we finally examined the generality of this phenomenon using other heterocyclic compounds containing a trifluoromethylated alcohol moiety at their quaternary carbon center. Enantiomeric mixtures of indole-like compounds **2**–**6** [11,12] were subjected to an "enantiomer self-disproportionation test" under similar optimized conditions. Results of the SDE test are shown in Table 2. Heterocyclic compound **2**, having the same skeleton as **1**, shows an ability of SDE; however, the enantiomer self-disproportionation effect was not observed for other types of heterocycles **3**–**6** independent of their enantiomeric purity (Table 2).

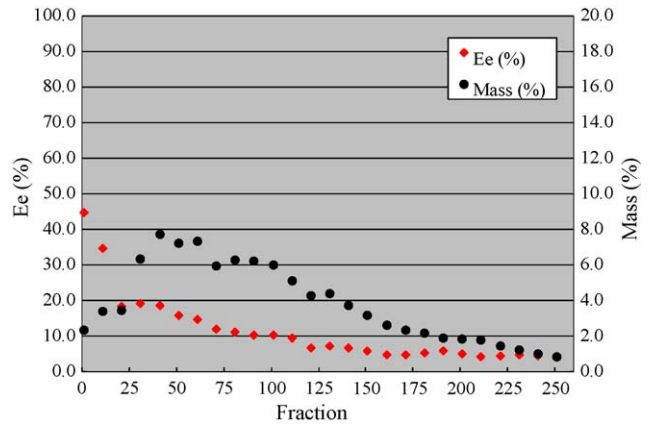
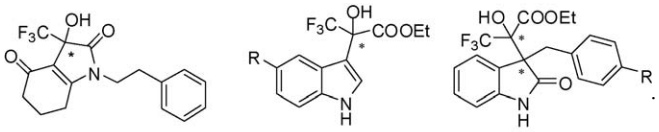


Fig. 5. Details of chromatography of **1** (9.9% ee) by medium-pressure liquid chromatography on achiral flash silica-gel.

Table 2
Enantiomer self-disproportionation test of compounds **2**–**6** on achiral silica-gel chromatography



2 **3**: R=H **4**: R=OMe **5**: R=OMe **6**: R=Br

Run	Substrate	Starting ee (%)	Eluent	% ee min	% ee max	Δee
1[a]	**2**	37.5	Et_2O	26.1	82.3	56.2
2[b]	**3**	52.7	H/E[c] (4/1)	52.2	52.4	0.2
3[b]	**4**	52.5	H/E[c] (4/1)	53.7	54.0	0.3
4[b]	**5**	96.5	H/E[c] (1/1)	96.0	97.0	1.0
5[b]	**6**	92.0	H/A[d] (9/1)	91.3	93.3	2.0

[a] Flash silica-gel was used under medium pressure.
[b] Flash silica-gel in glass column was used under atmospheric pressure.
[c] H/E: hexane/Et_2O.
[d] H/A: hexane/EtOAc.

3. Conclusion

In conclusion, the self-disproportionation of heterocycles enantiomers having a tertiary trifluoromethyl alcohol stereogenic center on an achiral silica-gel stationary phase has been examined. There is apparent generality of the core structure of **1** as one may assume that compounds of this type will show the SDE of a similar magnitude under the similar conditions. Probably the feature of these compounds **1** and **2** is that there is no rotation in the 5-membered ring and the chiral recognition is very pronounced. Moreover, compounds **3**–**6**, representing a different set of substituents and free rotation of the stereogenic carbon with (CF_3, OH, COOEt), still show some effect, although they are very small. The SDE is a fundamentally general phenomenon and it is manifestation and magnitude depend on the conditions applied. Therefore, SDE of **3**–**6** could be magnified by the different solvent system. The mechanism for the phenomenon of enantiomer self-disproportionation of **1** is now under investigation by comparing with experiments of a non-fluorinated analogue of **1**.

4. Experimental

In a typical optimal condition of a slurry packing system, 24 g of silica-gel (KANTO CHEMICAL CO., INC., Silica Gel 60N, spherical, neutral, 40–50 μm) was packed in a 26 mm × 80 mm polypropylene column with ether as the eluent using medium pressure (10 mL/min) at room temperature. In general, a solution of 10.0 mg of **1** dissolved in 0.50 mL of CH_2Cl_2 was loaded on this packed column following which this column was pressurized at the above-mentioned pressure and 225–250 (each 3.8 mL) fractions were collected until no more **1** was detected by TLC analysis. Each fraction was then subjected to high-performance liquid chromatography (HPLC) analysis to determine enantiomeric excess (ee). The racemic samples **1** and **2** were prepared according to the reported procedures and their enantiomeric mixtures were obtained by the purification using chiral HPLC enantiomer resolution. The samples **3**–**6** and their enantiomeric mixtures were prepared according to the reported procedures.

1: Colorless crystal; ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.81 (d, J = 8.2 Hz, 1H), 6.66 (d, J = 8.2, 1.8 Hz, 1H), 6.61 (d, J = 1.8 Hz, 1H), 4.65 (brs, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 3.78–3.91 (m, 1H), 3.61 (ddd, J = 14.0, 7.6, 5.3 Hz, 1H), 2.86–3.00 (m, 1H), 2.73–2.86 (m, 1H), 2.41 (dd, J = 17.2, 4.8 Hz, 1H), 2.12–2.28 (m, 1H), 1.81–1.95 (m,

4H); ^{19}F NMR (188 MHz, $CDCl_3$) δ −77.8 (s, 3F); ^{13}C NMR (50.4 MHz, $CDCl_3$) δ 191.5, 171.5, 169.0, 149.3, 148.2, 130.3, 123.0 (q, J = 286.7 Hz), 121.2, 112.2, 111.8, 111.5, 75.5 (q, J = 32.8 Hz), 56.4, 56.2, 43.8, 36.5, 34.8, 22.7, 21.3; IR (neat) 3422, 2951, 1754, 1645, 1605, 1518, 1422, 1351, 1264, 1240, 1187, 1159, 1081, 1027, 995, 810, 764, 714 cm^{-1}; MS (m/z, ESI) 422 ($M+Na^+$); HPLC (CHIRALPAK AD-3, hexane/i-PrOH = 90/10, 2.0 mL/min, 254 nm) tR (major) = 17.5 min and tR (minor) = 19.5 min); $[\alpha]_D^{25.2}$ −34.8 (c 0.20, $CHCl_3$, 57% ee); m.p. = 56.5–58.4 °C.

2: Colorless crystal; ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.21–7.36 (m, 3H), 7.09–7.13 (m, 2H), 4.50 (brs, 1H), 3.86 (ddd, J = 14.0, 9.8, 7.2 Hz, 1H), 3.62 (ddd, J = 14.0, 7.5, 5.7 Hz, 1H), 2.94–3.04 (m, 1H), 2.79–2.90 (m, 1H), 2.32–2.45 (m, 1H), 2.10–2.25 (m, 1H), 1.70–1.92 (m, 4H); ^{19}F NMR (188 MHz, $CDCl_3$) δ −77.9 (s, 3F); ^{13}C NMR (50.4 MHz, $CDCl_3$) δ 191.6, 171.5, 168.8, 137.7, 129.3, 129.1, 127.4, 123.0 (q, J = 286.1 Hz), 111.6, 75.5 (q, J = 32.8 Hz), 43.7, 36.4, 35.2, 22.6, 21.3; IR (neat) 3364, 2953, 1755, 1644, 1604, 1497, 1456, 1423, 1361, 1265, 1187, 1073, 1046, 1031, 992, 947, 898, 842, 743, 703 cm^{-1}; MS (m/z, ESI) 362 ($M+Na^+$); HPLC (CHIRALPAK AD-3 hexane/i-PrOH = 90/10, 0.5 mL/min, 254 nm) tR (minor) = 48 min, tR (major) = 50 min; $[\alpha]_D^{24.8}$ −32.2 (c 0.21, $CHCl_3$, 48% ee); m.p. = 128.0–128.5 °C.

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