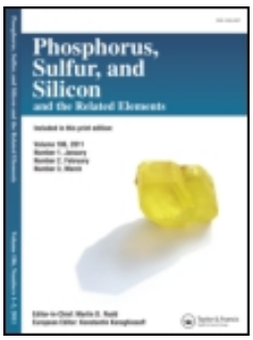

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Terpenyl tellurides—Synthesis and application in asymmetric epoxidation

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ABSTRACT

This work presents a synthesis of new group of unsymmetrical chiral tellurides obtained in the reaction of sodium methanetellurolate or sodium benzenetellurolate with appropriate terpenyl tosylates from p-menthane, carane, and pinane systems. Additionally, symmetrical diterpenyl tellurides were prepared according to our recently published method using sodium telluride and terpenyl tosylates. Methyl terpenyl, phenyl terpenyl, and diterpenyl tellurides were successfully used in tellurium ylide-mediated asymmetric epoxidation reaction. The best result of asymmetric epoxidation was obtained for dicaranyl telluride (d.r. cis:trans 11:89, e.r. trans 84:16).

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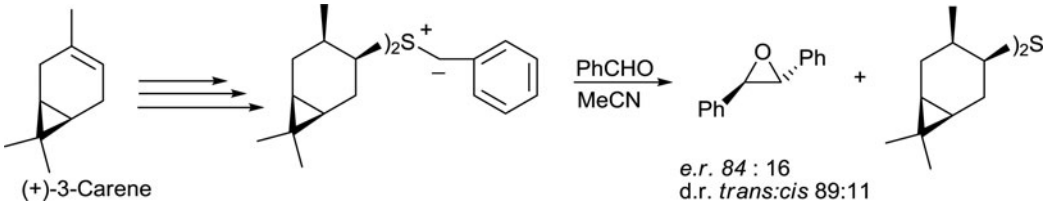
KEYWORDS
Tellurides; terpenes; asymmetric epoxidation

GRAPHICAL ABSTRACT



Introduction

Chalcogenides are highly valuable organic compounds which enable to create new carbon-carbon and carbon-heteroatom bonds. Analogical organosulfur, -selenium, and -tellurium reagents and catalysts differ in reactivity and selectivity in various reactions, thus it is essential to test and compare their applicability.[1] The catalytic efficiency of chalcogenides, mainly sulfur and selenium reagents, were widely studied in ylide-mediated epoxidation, cyclopropanation, or aziridination reactions.[2] Organotellurium compounds were less explored but the currently developed methodologies and obtained results are promising.[3–5] Till now, there are only few examples of tellurium ylide-mediated epoxidation. The reaction of propargyl tellurium salts **1** with benzaldehyde and base tends to favor the *cis*-epoxide, forming the product in high yield and good selectivity.[6] Catalytic amount of tellurium salt **2**, in the presence of Cs_2CO_3 and benzaldehyde, gave the epoxide also in high yield but lower diastereoselectivity (Scheme 1).[7]

According to our best knowledge, there is only one example of asymmetric epoxidation reaction catalyzed by a chiral telluride. Metzner and coworkers presented that compound **3**, applied in 20 mol% gave very good diastereoselectivity and enantioselectivity, but the yield was low (Scheme 2).[3]

In previous studies, we have developed an efficient methodologies for the synthesize of terpenyl sulfides, selenides, and diselenides, and have tested their reactivity in asymmetric synthesis,[8–17] e.g., optically active methyl terpenyl, phenyl terpenyl, and diterpenyl sulfides from carane, *p*-menthane, pinane, and bornane systems were obtained. Organosulfur reagents were tested in sulfur ylide-mediated asymmetric epoxidation reacton.[8] Quite recently, we have tested asymmetric epoxidation reaction using terpenyl selenides.[17] Formation of chiral selenides was generally based on the reaction of appropriate tosylate or chloride with sodium selenide, sodium diselenide, than methyl iodide or sodium benzeneselenolate. Methyl terpenyl, phenyl terpenyl and diterpenyl derivatives from carane, pinane, and *p*-menthane groups were obtained and tested with benzyl bromine, benzaldehyde, and NaOH to give epoxy stilbene. Selected the best results are shown in Figure 1. Chiral selenides and sulfides were also tested in asymmetric cyclopropanation with high enantioselectivities.[18]

In this paper, we present the preparation of unsymmetrical and symmetrical terpenyl tellurides and their use in asymmetric tellurium ylide-mediated epoxidation reaction.

Results and discussion

Recently, we have developed a new methodology for the synthesis of symmetrical mono- and ditellurides from *p*-menthane,

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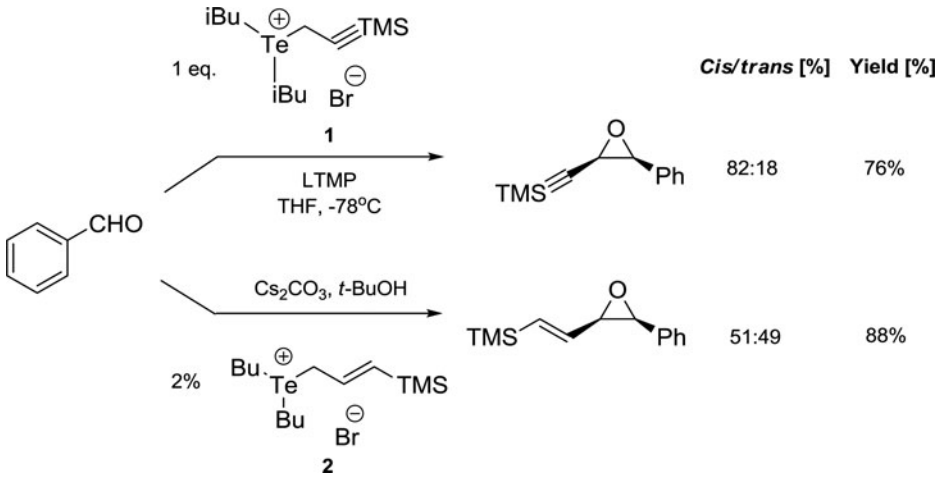
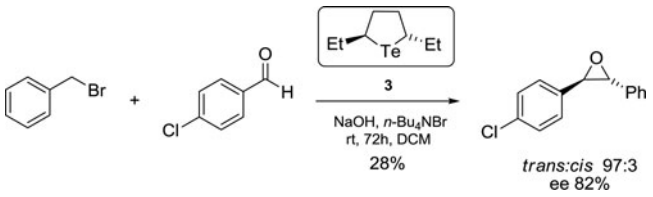
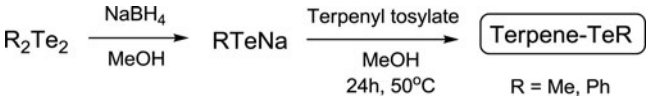
Dedicated to Prof. B. S.(Raj) Thyagarajan.



Scheme 1. Tellurium ylide-mediated epoxidation.



Scheme 2. Asymmetric tellurium ylide-mediated epoxidation reaction.



Scheme 3. Synthesis of unsymmetrical terpenyl tellurides.

carane, and pinane systems by the reaction of sodium telluride or ditelluride with corresponding terpenyl tosylate or chloride, e.g., dineomenthyl, dicaranyl, and dimyrtanyl tellurides **4–6** were obtained (Figure 2).[19]

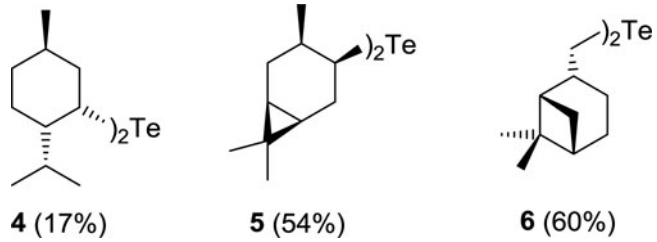


Figure 2. Chiral terpenyl tellurides.

Now we present the synthesis of new unsymmetrical chiral tellurides. Methyl terpenyl and phenyl terpenyl tellurides have been prepared by the reaction of appropriate tosylate with sodium methanetellurolate or sodium benzenetellurolate, generated *in situ* from the reaction of dimethyl ditelluride or diphenyl ditelluride with sodium borohydride (Scheme 3).

Using for the reaction menthyl, myrtanyl, and isocaranyl tosylates we have obtained methyl tellurides **7**, **9** and phenyl tellurides **8** and **10** from *p*-menthane and pinane series (Figure 3).

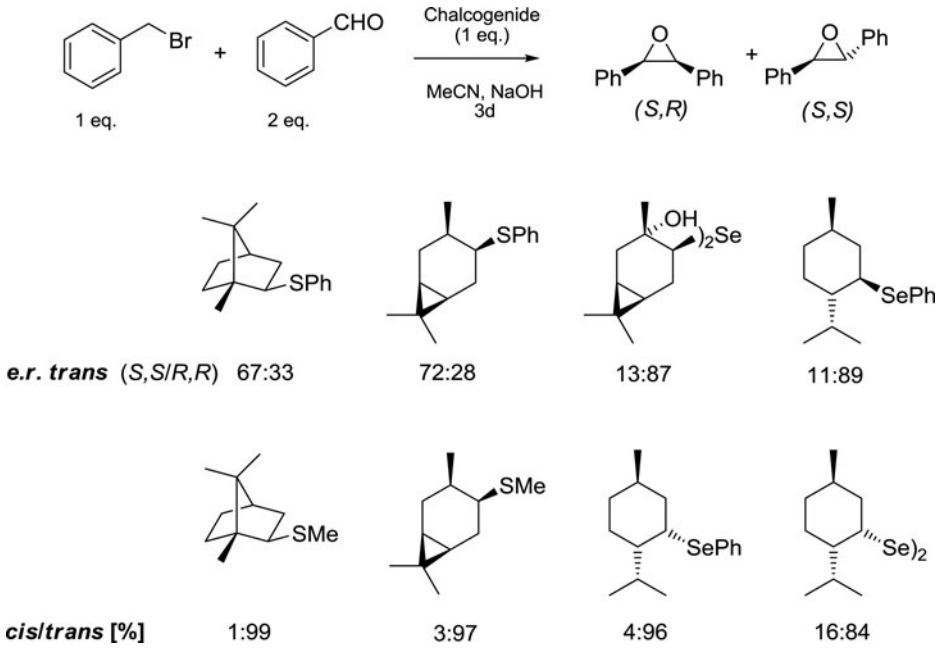


Figure 1. The best enantioselectivities and diastereoselectivities of sulfur and selenium ylide-mediated epoxidation reaction.

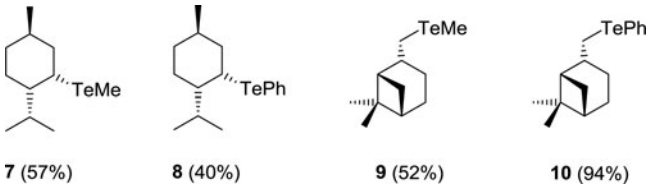


Figure 3. Obtained methyl and phenyl terpenyl tellurides.

Analogous tellurides were also obtained from isocarane tosylate. In this case, the epimerization was observed. For the methyl tellurides **11** and **12**, the ratio was 40:60 with a predominance of *trans* epimer, in case of phenyl analogues **13** and **14** the *cis* structure was in excess (Figure 4).

Symmetrical and unsymmetrical tellurides **4–10** were tested as reagents in tellurium ylide-mediated epoxidation reaction. Benzyl bromine reacts with phenyl aldehyde in the presence of appropriate chiral telluride to form epoxy stilbene (Table 1). As a base, we have used NaOH. The reaction was carried out in anhydrous conditions under argon atmosphere.

The best distereomeric ratios were obtained for myrtanyl phenyl telluride **10**. Dicaranyl telluride **5** gave the best enantioselectivity. The best results are comparable to the ones achieved for terpenyl sulfides and selenides presented in Figure 1. When we compare the results of asymmetric epoxidation of dicaranyl ditelluride **5** with his sulfur[8] and selenium[17] analogous, we can observed increase of enantioselenctivity, preference to create the *trans* product and increase of yield (Table 2).

Conclusion

Efficient methodology for the synthesis of unsymmetrical tellurides was presented. Methyl, phenyl, and diterpenyl tellurides from *p*-menthane, carane, and pinane groups were obtained. All compounds were successfully applied in asymmetric tellurium-mediated epoxidation reaction. The best result was obtained for symmetrical bicyclic caranyl system, and in this case telluride comparing to sulfur and selenium analogues gave the best yield, diastereo- and stereoselectivities.

Experimental

^{1}H NMR spectra were obtained at 200, 400, or 700 MHz and chemical shifts were recorded relative to $SiMe_4$ (δ 0.00) or solvent resonance ($CDCl_3$ δ7.26). Multiplicities were given as: s (singlet), d (doublet), dd (double doublet), ddd (double double doublet), t (triplet), dt (double triplet), and m (multiplet). The number of protons (n) for a given resonance was indicated by *n*H. Coupling constants were reported as a *J* value in Hz. ^{13}C NMR spectra were acquired at 100.6 Hz and chemical shifts were recorded relative to solvent resonance ($CDCl_3$ δ77.25). NMR spectra were carried out using ACD/NMR Processor Academic Edition. Commercially available solvents ethanol, methanol, diethyl ether, petroleum ether and acetonitrile and chemicals (Aldrich) were used without further purification. Column chromatography was performed using Silica Gel 60 Merck (70–230 mesh).

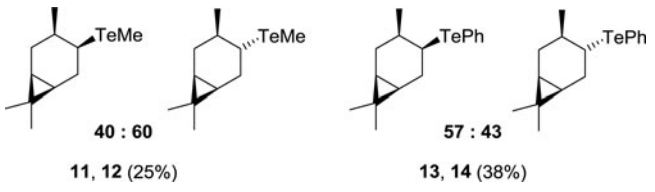


Figure 4. Synthesis of caranyl ditellurides.

General procedure for the synthesis of methyl terpenyl tellurides

To the solution of dimethyl ditelluride (1.43 g, 5.0 mmol) in methanol (50 mL) $NaBH_4$ (2.40 g, 64.2 mmol) was added. Tosylate (10.0 mmol) was added and the mixture was stirred for 24 h. Methanol was evaporated, the solution was poured on water (40 mL) and extracted with petroleum ether (3 × 30 mL). The combined organic layers were dried, evaporated, and the obtained crude product was purified by column chromatography (petroleum ether). The reaction and column chromatography was performed under argon atmosphere.

(1S,2S,5R)-(+)-Methyl neomenthyl telluride 7

Yield: 57%, $[\alpha]^{23}_D$ = + 93.0 (c 0.50, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.45–0.53 (m, 1H), 0.82–0.93 (m, 2H), 0.93 (d, *J* = 6.8 Hz, 3H, CH_3), 0.94 (d, *J* = 6.4 Hz, 3H, CH_3), 0.95 (d, *J* = 6.8 Hz, 3H, CH_3), 1.43–1.54 (m, 2H), 1.67–1.86 (m, 3H), 1.88 (s, 3H, CH_3), 2.13 (ddd, *J* = 2.4, 5.2, 14.0 Hz, 1H), 3.62 (dt, *J* = 2.8, 5.6 Hz, 1H) ppm; ^{13}C NMR (100.6 Hz, $CDCl_3$): δ = −21.6 (CH_3), 20.7 (CH_3), 20.9 (CH_3), 22.1 (CH_3), 28.8 (CH_2), 30.1 (CH), 33.7 (CH), 35.3 (CH), 35.7 (CH_2), 43.6 (CH_2), 50.0 (CH) ppm; IR (cm^{-1}, film): 3437, 2916, 1685, 1451, 1380, 1258, 1217, 1166, 1085. Elemental Anal. Calcd for $C_{11}H_{22}Te$ (281.89): C, 46.87; H, 7.87. Found: C, 46.89; H, 7.91.

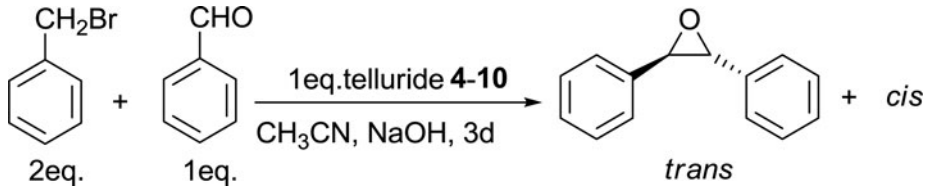
(1S,2R,5S)-(−)-Methyl myrtanyl telluride 9

Yield: 52%,$[\alpha]^{23}_D$ = −42.1 (c 0.55, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.89 (d, *J* = 10.0Hz, 1H), 1.03 (s, 3H, CH_3), 1.21 (s, 3H, CH_3), 1.45–1.56 (m, 1H), 1. 89 (s, 3H, CH_3), 1.82–2.15 (m, 5H), 2.27–2.40 (m, 2H), 2.75 (dd, *J* = 8.8, 11.6 Hz, 1H), 2.81 (dd, *J* = 7.6, 11.6 Hz, 1H) ppm; ^{13}C NMR (100.6 Hz, $CDCl_3$): δ = −25.3 (CH_3), 13.5 (CH_2), 23.3 (CH_3), 24.6 (CH_2), 26.3 (CH_2), 28.1 (CH_3), 33.6 (CH_2), 38.7 (C), 41.3 (CH), 43.1 (CH), 47.8 (CH) ppm; IR (cm^{-1}, film): 2920, 2866, 2345, 1461, 1415, 1379, 1358, 1218, 1176, 1166, 838. Elemental Anal. Calcd for $C_{11}H_{20}Te$ (279.88): C, 47.21; H, 7.20. Found: C, 47.28; H, 7.16.

(1S,3R,4S,6R)-(+)-4-Caranyl methyl telluride 11, (1S,3R,4R,6R)-(+)-4-izocaranyl methyl telluride 12

Yield: 25%, ^{1}H NMR (700 MHz, $CDCl_3$): δ = 0.91 (d, *J* = 7.0 Hz, 3H; CH_3), 0.94 (s, 3H, CH_3), **0.94 (s, 3H, CH_3)**, 0.95 (s, 3H, CH_3), **0.96 (s, 3H, CH_3), 0.99 (d, *J* = 6.3 Hz, 3H; CH_3)**, 1.33–1.68 (m, 3H + **3H**), 1.82 (s, 3H, CH_3), **1.83 (s, 3H, CH_3), 1.95–2.18 (m, 4H)**, 2.25–2.38 (m, 4H), **2.52–2.60 (m, 1H)** 3.40–3.45 (m, 1H) ppm; ^{13}C NMR (100.6 Hz, $CDCl_3$): δ = **−22.6 (CH_3)**, −21.5 (CH_3), **15.6 (CH_3)**, 16.1 (CH_3), **17.5 (C)**, 17.7 (C), **20.9 (CH), 20.9 (CH)**, 21.7 (CH), 22.6 (CH), 23.5 (CH_3), **24.1 (CH_3)**, 26.3 (CH_2), 27.9 (CH_2), **28.7 (CH), 28.9 (CH_2),**

Table 1. Results of synthesis of epoxy stilbene.



Entry	Telluride	Yield [%]	Ratio *Cis*/*trans* [%][a]	Ratio *trans* (*S*/*S*/*R*/*R*)[b]
<1	**4**	9	50/50	58:42
2	**5**	47	11/89	**84:16**
3	**6**	22	59/41	47:53
4	**7**	40	44/56	68:32
5	**8**	13	39/61	68:32
6	**9**	22	59/41	47:53
7	**10**	10	**9/91**	52:48

[a]Determined on the basis of ^{1}H NMR spectroscopy.
[b]Determined on the basis of HPLC analysis using a Chiralcel Daicel ODH column.

28.9 (CH_3), 29.6 (CH), 30.1 (CH_3), 31.3, **31.8 (CH_2), 35.7 (CH)** (CH) ppm. Mix of two epimers, (1S,3R,4S,6R)-(+)-4-Caranyl methyl telluride : (1*S*,3*R*,4*R*,6*R*)-(+)-4-Izocaranyl methyl telluride (40:**60**).

General procedure for the synthesis of phenyl terpenyl tellurides

To the solution of diphenyl ditelluride (1.02 g, 2.5 mmol) in methanol (50 mL) $NaBH_4$ (0.38 g, 10.0 mmol) was added. Tosylate (5.4 mmol) in methanol (20 mL) was added and the mixture was stirred for 24 h at 80°C. Methanol was evaporated, the solution was poured on water (100 mL), and extracted with petroleum ether (3 × 100 mL). The combined organic layers were dried, evaporated, and the obtained crude product was purified by column chromatography (petroleum ether). The reaction and column chromatography was performed under argon atmosphere.

(1S,2S,5R)-(+)-Neomenthyl phenyl telluride 8

Yield: 40%, $[\alpha]^{23}{}_D = +85.9$ (c 1.36, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.61–0.69 (m, 1H), 0.84 (d, J = 6.4 Hz, 3H, CH_3), 0.93 (d, J = 6.8 Hz, 3H, CH_3), 0.97 (d, J = 6.8 Hz, 3H, CH_3), 0.89–1.03 (m, 2H), 1.51–1.64 (m, 2H), 1.73–1.80 (m, 1H), 1.84–1.93 (m, 2H), 2.17 (ddd, J = 2.4, 4.8, 13.6 Hz, 1H), 3.50 (dt, J = 2.8, 5.6 Hz, 1H), 7.18–7.23 (m, 2H, 2x $CH_{arom.}$), 7.28–7.33 (m, 1H, $CH_{arom.}$), 7.83 (dd, J = 1.2, 8.0 Hz, 2H, 2x $CH_{arom.}$) ppm; ^{13}C NMR (100.6 Hz, $CDCl_3$): δ = 20.7 (CH_3), 20.8 (CH_3), 22.1 (CH_3), 29.1 (CH_2), 30.2 (CH), 33.8 (CH), 35.3 (CH_2), 41.5 (CH), 43.9 (CH_2), 50.3 (CH), 112.0 ($C_{arom.}$), 127.6 ($CH_{arom.}$), 129.0 (2x $CH_{arom.}$), 139.7 (2x $CH_{arom.}$) ppm; IR (cm^{-1}, film): 3400, 3065, 2945, 2868, 1686, 1574, 1473, 1442, 1384, 1275, 1260, 1222, 1167, 1062,1018, 853, 732, 692. Elemental Anal. Calcd for $C_{16}H_{24}Te$ (343.96): C, 55.87; H, 7.03. Found: C, 56.01; H, 7.00.

Table 2. Results of epoxidation with the use dicaranyl chalcogenides.

Entry	Dicaranyl chalcogenide	Yield [%]	Ratio *Cis*/*trans* [%][a]	Ratio *trans* (*S*/*S*/*R*/*R*)[b]
1	Sulfide	14	71:29	54:48
2	Selenide	10	52:48	59:41
3	Telluride **5**	47	11:89	84:16

[a]Determined on the basis of ^{1}H NMR spectroscopy.
[b]Determined on the basis of HPLC analysis using a Chiralcel Daicel ODH column.

(1S,2R,5S)-(−)-Myrtanyl phenyl telluride 10

Yield: 94%), $[\alpha]^{23}{}_D = -40.2$ (c 1.28, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.89 (d, J = 9.6 Hz, 1H), 1.02 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 1.48–1.60 (m, 1H), 1.82–2.01 (m, 3H), 2.03–2.17 (m, 2H), 2.33–2.46 (m, 2H), 3.08 (dd, J = 11.2, 30.0 Hz, 1H), 3.11 (dd, J = 11.6, 29.2 Hz, 1H), 7.19–7.24 (m, 2H, 2x $CH_{arom.}$), 7.26–7.32 (m, 1H, $CH_{arom.}$), 7.72–7.77 (m, 2H, 2x $CH_{arom.}$) ppm; ^{13}C NMR (100.6 Hz, $CDCl_3$): δ = 18.6 (CH_2), 23.2 (CH_3), 24.7 (CH_2), 26.3 (CH_2), 28.0 (CH_3), 33.6 (CH_2), 38.7 (C), 41.3 (CH), 43.0 (CH), 47.8 (CH), 112.1 ($C_{arom.}$), 127.4 ($CH_{arom.}$), 129.1 (2x $CH_{arom.}$), 138.3 (2x $CH_{arom.}$) ppm; IR (cm^{-1}, film): 3066, 2912, 1574, 1474, 1433, 1365, 1165, 1062, 1018, 908, 758, 729, 691. Elemental Anal. Calcd for $C_{16}H_{22}Te$ (341.95): C, 56.20; H, 6.48. Found: C, 56.29; H, 6.45.

(1S,3R,4S,6R)-(+)-4-Caranyl phenyl telluride 13, (1S,3R,4R,6R)-(+)-4-izocaranyl phenyl telluride 14

Yield: 38%, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.40–0.50 (m, 1H), 0.51–0.60 (m, 1H), 0.62–0.90 (m, 4H), 0.96 (d, J = 6.4 Hz, 3H; CH_3), 0.97 (s, 3H, CH_3), 0.98 (s, 3H, CH_3), 0.98 (s, 6H, 2xCH_3), 1.04 (d, J = 6.8 Hz, 3H; CH_3), 1.45–1.50 (m, 1H) 1.60–1.70 (m, 1H), 1.80–1.95 (m, 2H), 1.95–2.05 (m, 1H), 2.10–2.38 (m, 3H), 2.89–2.99 (m, 1H), 3.75–3.85 (m, 1H), 7.13–7.25 (m, 4H), 7.28–7.35 (m, 2H), 7.75–7.83 (m, 4H) ppm; ^{13}C NMR (100.6 Hz, $CDCl_3$): δ = 15.6 (CH_3), 16.0 (CH_3), 17.5 (C), 17.7 (C), 20.8 (CH), 21.2 (CH), 21.5 (CH), 22.9 (CH), 26.7 (CH_2), 27.9 (CH_2), 28.4 (CH), 28.9 (CH), 29.2 (CH_2), 31.25 (CH), 31.69 (CH_2), 34.69 (CH), 34.78 (CH), 35.947 (CH), 111.85 (C), 112.84 (C), 127.56 (CH), 127.80 (CH), 129.00 (2xCH), 129.04 (2xCH), 139.44 (2xCH), 140.37 (2xCH) ppm. Mix of two epimers, (1S,3R,4S,6R)-(+)-4-Caranyl phenyl telluride : (1*S*,3*R*,4*R*,6*R*)-(+)-4-Izocaranyl phenyl telluride (57:43).

General procedure for the epoxidation reaction

A mixture of telluride (2.18 mmol), benzyl bromide (0.749g, 4.38 mmol), sodium hydroxide (0.088g, 2.20 mmol), and benzaldehyde (0.231g, 2.18 mmol) in 10 mL of acetonitrile was stirred for 3 days. Acetonitrile was evaporated and water (80 mL) was added. The mixture was extracted with petroleum ether (3 × 75 mL) and the combined organic layers were dried, evaporated and the obtained crude product was purified by column chromatography (petroleum ether: ethyl acetate, 95:5).

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