Enantioselective Syntheses and Sensory Properties of the 3-Mercapto-2-methylpentanols†

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3-Mercapto-2-methylpentanol, a new powerful flavor compound, exhibits two stereocenters giving rise to two pairs of diastereomers. To determine differences in the sensory properties, all four diastereomers and enantiomers were stereo- and enantioselectively synthesized. A highly diastereoselective aldol reaction using a chiral auxiliary was one of the key steps in the synthesis. Further derivatization yielded the enantiopure compounds. Odor thresholds in air and water were determined.

Keywords: *Enantioselective synthesis; chiral auxiliary; odor thresholds*

INTRODUCTION

3-Mercapto-2-methylpentan-1-ol has recently been discovered as a new powerful nature-identical flavor chemical (Widder et al., 2000). The odor threshold of this compound in air is extremely low. The general structure, containing two asymmetric centers, gives rise to four different diastereomers and enantiomers (Figure 1). In many cases properties of different diastereomeric and even enantiomeric forms of a molecule differ considerably (Guth, 1996; Pickenhagen et al., 1984 and references cited therein). Qualitative GC-O analysis indicated differences in the odor strengths of the two *syn*/*anti* diastereomers. To evaluate the sensory properties and to determine the odor thresholds of these four diastereomers, we undertook a stereoselective synthesis with total control of the two stereocenters at carbons two and three of the pentanol framework.

EXPERIMENTAL PROCEDURE

Synthesis. The two enantiomerically pure *syn*-2-methyl-1,3-pentanediols were both synthesized according to a literature method (Chong, 1994) by diastereoselective aldol reaction using Evan's chiral auxiliary.

(2S,3R)-1-Triisopropylsiloxy-2-methylpentan-3-ol. (2*R*,3*R*)-2-Methylpentane-1,3-diol (10.6 g, 90 mmol) and imidazole (12.23 g, 180 mmol) were dissolved in dry *N*,*N*-dimethylformamide under nitrogen and cooled to 10 °C. Triisopropylsilyl chloride (17.74 g, 92 mmol) was slowly added, and after the addition stirring was continued for 20 h. After cooling to 0 °C, the reaction mixture was acidified by addition of 2 N aqueous hydrochloric acid. The reaction mixture was extracted three times with diethyl ether, and the combined organic phases were washed once with saturated aqueous sodium bicarbonate solution, dried over sodium sulfate, and concentrated in vacuo to give 27 g of crude product, which was used as such in the next step.

^{1}H NMR ($CDCl_3$) δ 0.94 (d, 7 Hz, 3H, CH_3), 0.95 (t, 7.5 Hz, 3H, CH_3), 1.05 (m, 21H, TIPS), 1.48 (m, 2H, CH_2), 1.75 (m, 1H, CH), 3.18 (bs, 1H, OH), 3.74 (ddt, 2 Hz, 5 Hz, 8 Hz, 1H, CH−O), 3.80 (dd, 5 Hz, 10 Hz, 1H, CH_{2a}−O), 3.87 (dd, 4 Hz, 10 Hz, 1H, CH_{2b}−O).

^{13}C NMR ($CDCl_3$) δ 76.5 (3°, CH−OH), 69.1 (2°, CH_2−OSi), 38.5 (3°, CH), 27.0 (2°, CH_2), 18.0 (1°, isopropyl-CH_3), 11.8 (3°, isopropyl-CH), 10.7 (1°, CH_3), 10.2 (1°, CH_3).

IR (capillary film, cm^{-1}) 3439 (s), 2937 (s), 2863 (s), 1456 (s), 1379 (m), 1244 (w), 1105 (s), 1057 (s), 1010 (s), 880 (s).

*(2R,3R)-Methanesulfonic Acid 1-Ethyl-2-methyl-3-triisopropylsiloxypropyl Ester (**11**).* (2*S*,3*R*)-1-Triisopropylsiloxy-2-methylpentan-3-ol (20 g, 73 mmol) was dissolved in dry dichloromethane (120 mL) under nitrogen and cooled to 0 °C. Triethylamine (13.2 mL, 95 mmol) and then methanesulfonyl chloride (9.2 g, 80 mmol) were slowly added at 0−5 °C. After stirring for another 2 h at 0 °C, the reaction mixture was acidified by addition of 2 N aqueous hydrochloric acid. The reaction mixture was then extracted three times with dichloromethane, and the combined organic phases were washed once with saturated aqueous sodium bicarbonate solution and once with saturated aqueous sodium chloride solution, dried over sodium sulfate, and concentrated in vacuo.

^{1}H NMR ($CDCl_3$) δ 0.94 (d, 7 Hz, 3H, CH_3), 0.97 (t, 7.5 Hz, 3H, CH_3), 1.07 (m, 21H, TIPS), 1.84 (m, 2H, CH_2), 1.97 (m, 1H, CH), 3.03 (s, 3H, CH_3−SO_2), 3.64 (d, 7 Hz, 2H, CH_2−O), 4.84 (ddd, 3.5 Hz, 7 Hz, 7 Hz, 1H, CH−OMs).

^{13}C NMR ($CDCl_3$) δ 85.3 (3°, CH−OMs), 64.6 (2°, CH_2−OSi), 38.4 (3°, CH), 38.1 (1°, CH_3−SO_2), 25.4 (2°, CH_2), 18.0 (1°, isopropyl-CH_3), 12.0 (3°, isopropyl-CH), 10.7 (1°, CH_3), 9.8 (1°, CH_3).

IR (capillary film, cm^{-1}) 2938 (s), 2862 (s), 1712 (w), 1612 (w), 1457 (m), 1378 (m), 1353 (s), 1174 (s), 1099 (s), 915 (s).

*(2R,3S)-Thioacetic Acid S-[1-(1-Methyl-2-triisopropylsiloxyethyl)propyl] Ester (**12**).* (2*R*,3*S*)-Mesylate (**11**) (4 g, 11 mmol), potassium thioacetate (1.88 g, 16.5 mmol), and 18-crown-6 (100 mg) were refluxed in absolute acetonitrile (35 mL) under argon for ∼12 h. The reaction mixture was cooled to 0 °C, acidified by addition of 2 N aqueous hydrochloric acid, and then extracted three times with diethyl ether. The combined organic phases were washed once with saturated aqueous sodium bicarbonate solution and once with saturated aqueous sodium

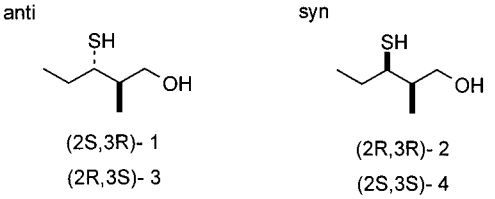

Figure 1. Diastereomers of 3-mercapto-2-methylpentanol.

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chloride solution, dried over sodium sulfate, and concentrated in vacuo. The crude product was purified by column chromatography (hexane/ether 2:1) to give 3.8 g (86% GC-purity) of the thiocetate.

^{1}H NMR ($CDCl_3$) δ 0.94 (t, 7 Hz, 3H, CH_3), 0.95 (d, 7 Hz, 3H, CH_3), 1.52 (m, 1H, CH_{2a}), 1.73 (m, 1H, CH_{2b}), 2.00 (m, 1H, CH), 2.32 (s, 3H, thioacetyl-CH_3), 3.54 (dd, 6.5 Hz, 9.5 Hz, 1H, CH−SAc), 3.61 (dd, 4.5 Hz, 9.5 Hz, 1H, CH_{2a}−O), 3.68 (dd, 6 Hz, 9.5 Hz, 1H, CH_{2b}−O).

^{13}C NMR (C_6D_6) δ 193.7 (4°, C=O), 66.5 (2°, CH_2−OSi), 48.9 (3°, CH−SAc), 40.4 (3°, CH), 30.3 (1°, thioacetyl-CH_3), 24.9 (2°, CH_2), 18.2 (1°, isopropyl-CH_3), 14.5 (1°, CH_3), 12.3 (3°, isopropyl-CH), 11.9 (1°, CH_3).

IR (capillary film, cm^{-1}) 2936 (s), 2862 (s), 1688 (s), 1456 (s), 1378 (m), 1363 (m), 1348 (m), 1105 (s), 1066 (s), 1010 (m), 994 (m), 949 (m), 880 (s).

*(2R,3S)-3-Mercapto-2-methylpentan-1-ol (**1**).* (2*R*,3*S*)-Thioacetate (**12**) (3.80 g from previous step, 86%) was dissolved in tetrahydrofuran under nitrogen and cooled to 0 °C before tetrabutylammonium fluoride trihydrate (3.41 g, 10.8 mmol) was added. After stirring for 2 h at room temperature, the reaction mixture was diluted with diethyl ether (100 mL), washed with saturated aqueous sodium bicarbonate solution and once with saturated aqueous sodium chloride solution, dried over sodium sulfate, and concentrated in vacuo. The crude product (3.67 g) was used in the next step.

Lithium aluminum hydride (0.54 g, 14.22 mmol) was suspended in dry diethyl ether (30 mL) under nitrogen and cooled to 0 °C. The crude (2*R*,3*S*)-acetylthiopentanol from the previous step (3.67 g) dissolved in dry diethyl ether (5 mL) was added dropwise. Stirring was continued at room temperature for 2 h before the reaction was quenched by careful addition of 1 N aqueous hydrochloric acid until acidic pH. The phases were separated, and the aqueous phase was extracted twice with diethyl ether. The combined organic phases were washed with saturated aqueous sodium bicarbonate solution and saturated aqueous sodium chloride solution, dried over sodium sulfate, and concentrated in vacuo. Upon column chromatography (hexane/ether 4:1) the final product (2*R*,3*S*)-3-mercapto-2-methylpentan-1-ol was obtained. Chiral GC analysis showed no traces of other enantiomers or diastereomers.

^{1}H NMR (C_6D_6) δ 0.85 (d, 7 Hz, 3H, CH_3), 0.92 (t, 7 Hz, 3H, CH_3), 1.12 (d, 8 Hz, 1H, CH_3), 1.25 (m, 1H, CH_{2a}), 1.52 (dqd, 4 Hz, 7 Hz, 15 Hz, 1H, CH_{2b}), 1.65 (bm, 2H, CH and OH), 2.59 (dddd, 4 Hz, 5.5 Hz, 8 Hz, 10 Hz, 1H, CH−S), 3.38 (dd, 6 Hz, 10.5 Hz, 1H, CH_{2a}−O), 3.43 (dd, 7 Hz, 10.5 Hz, 1H, CH_{2b}−O).

^{13}C NMR (C_6D_6) δ 65.3 (2°, CH_2−O), 45.7 (3°, CH−S), 42.1 (3°, CH), 28.4 (2°, CH_2), 14.4 (1°, CH_3), 12.2 (1°, CH_3).

MS (EI, 70 eV), *m*/*z* (%) 134 (M^+, 21), 116 (3), 100 (42), 83 (18), 75 (51), 74 (100), 71 (60), 55 (48), 47 (30), 45 (27), 41 (96), 31 (33).

IR (capillary film, cm^{-1}) 3342 (s), 2959 (s), 2926 (s), 2872 (s), 2560 (w), 1453 (m), 1375 (m), 1020 (s), 977 (m).

The (2*S*,3*R*)-3-mercapto-2-methylpentan-1-ol (**3**) was prepared accordingly, starting from D-phenylalanine (spectral data are identical to the above-mentioned).

(2R,3R)-3-Mercapto-2-methylpentan-1-ol (2). (2*R*,3*S*)-Mesylate (**11**) (10 g, 27.5 mmol), potassium chloride, and a catalytic amount of Aliquat 336 (0.61 g) were refluxed in water (10 mL) with efficient stirring while keeping the oil bath temperature at 105 °C. After ~8 h (TLC control), the reaction mixture was cooled, diluted with water, and extracted four times with hexane. The organic phase was washed once with saturated aqueous sodium chloride solution, dried over sodium sulfate, and concentrated in vacuo. The crude product (**13**) (8.1 g) was used in the next reaction.

The (2*R*,3*S*)-3-chloro-2-methyl-1-triisopropylsiloxypentane (**13**) from the previous step (8.1 g), potassium thioacetate (4.73 g, 41 mmol), and 18-crown-6 (2.5 g) were dissolved in dry acetonitrile (60 mL) and refluxed under argon for ~96 h. The reaction mixture was then cooled to room temperature, diluted with water, and extracted three times with diethyl ether. The combined organic phases were washed once with saturated aqueous sodium bicarbonate solution, dried over sodium sulfate, and concentrated in vacuo to give the crude (2*R*,3*R*)-thioacetate (**14**) (~52% GC purity), which was used in the next step.

The (2*R*,3*R*)-thioacetate (**14**) (7.7 g of 52% purity) from the previous step was dissolved in tetrahydrofuran (35 mL) under nitrogen and cooled to 0 °C. Tetrabutylammonium fluoride trihydrate (4.17 g, 13 mmol) was then added, and the reaction mixture was stirred for 2 h. The mixture was diluted with diethyl ether and washed twice with saturated aqueous sodium chloride solution, dried over sodium sulfate, and concentrated in vacuo. The crude product (7.5 g) was used in the next step.

Lithium aluminum hydride (1.94 g, 51 mmol) was suspended in dry diethyl ether (150 mL) and cooled to 0 °C. A solution of the crude (2*R*,3*R*)-pentanol (7.5 g) in dry diethyl ether (5 mL) was added dropwise. After completion of the addition, stirring was continued for 2 h at room temperature before the reaction was quenched by addition of 1 N aqueous hydrochloric acid until acidic pH. The phases were separated, and the aqueous phase was extracted twice with diethyl ether. The combined organic phases were washed with saturated aqueous sodium bicarbonate solution and saturated aqueous sodium chloride solution, dried over sodium sulfate, and concentrated in vacuo. Upon chromatography the final product (2*R*,3*R*)-3-mercapto-2-methylpentan-1-ol was obtained (0.62 g). Chiral GC analysis showed ~0.3% of the C2-epimeric diastereomer in addition to the correct product.

^{1}H NMR (C_6D_6) δ 0.70 (d, 7 Hz, 3H, CH_3), 0.89 (d, 8.5 Hz, 1H, SH), 0.90 (t, 7 Hz, 3H, CH_3), 1.35 (m, 2H, CH_2), 1.46 (bs, 1H, OH), 1.67 (m, 1H, CH), 2.84 (ddt, 4 Hz, 6 Hz, 9 Hz, 1H, CH−S), 3.28 (dd, 6 Hz, 10.5 Hz, 1H, CH_{2a}−O), 3.46 (dd, 8 Hz, 10.5 Hz, 1H, CH_{2b}−O).

^{13}C NMR (C_6D_6) δ 65.9 (2°, CH_2−O), 44.4 (3°, CH−S), 40.2 (3°, CH), 30.6 (2°, CH_2), 12.6 (1°, CH_3), 10.5 (1°, CH_3).

MS (EI, 70 eV), *m*/*z* (%) 134 (M^+, 21), 116 (3), 100 (42), 83 (22), 75 (52), 74 (100), 71 (60), 55 (50), 47 (31), 45 (31), 41 (97), 31 (34).

FTIR (gas phase, cm^{-1}) 3669 (m), 3583 (w), 2972 (s), 2939 (s), 2889 (s), 1465 (m), 1385 (m), 1035 (s).

The synthesis of the (2*S*,3*S*)-3-mercapto-2-methylpentan-1-ol (**4**) was performed accordingly from the mesylate derived from the reaction sequence starting from D-phenylalanine (spectral data were identical to the above-mentioned).

NMR Spectroscopy. ^{1}H NMR spectra of synthesized samples were recorded in C_6D_6 or $CDCl_3$ at 300 MHz on a Varian instrument. The ^{13}CNMR spectra of synthesized samples were recorded in C_6D_6 or $CDCl_3$ at 75 MHz. with $Si(CH_3)_4$ as internal standard.

Enantioselective MDGC Analysis. For the separation of the enantiomers, multidimensional gas chromatography was performed on a Siemens Sichromat 2-8 equipped with two independent column oven programs and a live-T-switching device for selective cutting from the first to the second column. Precolumn conditions were as follows: DB-Wax column (30 m × 0.25 mm, 0.25 μm; J&W Scientific, Fisons, Mainz, Germany); carrier gas, helium; gas flow rate, 1.07 mL/min; injector temperature, 200 °C, splitless injection; oven temperature, 60 °C raised at 4 °C/min to 200 °C. Main column conditions were as follows: MN Lipodex E column (25 m × 0.25 mm, 0.25 μm; Macherey and Nagel, Düren, Germany); carrier gas, helium; gas flow rate, 1.26 mL/min; oven temperature, 60 °C raised at 2 °C/min to 160 °C.

Odor Thresholds in Water. A defined amount of each compound, dissolved in 0.1 mL of ethanol, was added to water (1 L). After stirring for 10 min, this stock solution was diluted stepwise with water (1 + 1, v/v) and stirred for 5 min after each dilution step. Odor threshold values were determined by triangle tests. Every dilution step was presented with three glass beakers each containing 20 mL of liquid. One of these beakers contained the solution with the compound, the remaining two contained water. The samples were presented in order of decreasing concentrations. The odor threshold values evaluated by at least 15 judges were averaged.

Odor Thresholds in Air. Odor threshold values in air were determined by the olfactometric HRGC method described by Ullrich and Grosch (1987). The reference substance used for

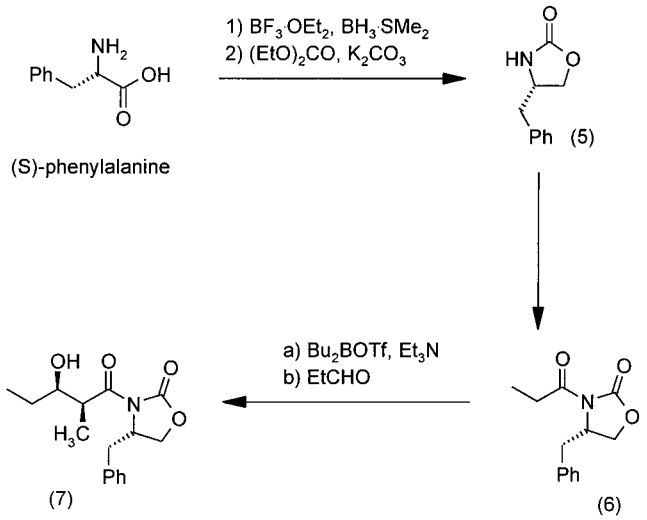

Figure 2. Synthesis of the chiral auxiliary and diastereoselective aldol reaction.

the calculation of the odor thresholds was (*E*)-2-decenal. Its odor threshold in air is 2.7 ng/L (Boelens and van Gemert, 1986).

RESULTS AND DISCUSSION

Retrosynthetic analysis suggested a *β*-hydroxy-α-methylpentanoic acid as a precursor for the mercaptopentanol. A very efficient route toward this class of compounds takes advantage of a stereoselective aldol reaction with a chiral auxiliary. One of the best known auxiliaries is a 1,3-oxazolidinone derived from phenylalanine first used by Evans (Evans et al., 1981; Ager et al., 1997). The stereoselective synthesis using this chiral auxiliary indeed afforded the four diastereomers.

The synthesis of (2*R*,3*S*)-3-mercapto-2-methylpentan-1-ol started with a boron trifluoride/borane-mediated reduction of L-phenylalanine to yield the phenylalaninol. This alcohol was reacted with diethyl carbonate to give the 1,3-oxazolidinone (**5**) (Evan's auxiliary), and through acylation with propionyl chloride one obtained the *N*-propionyl oxazolidinone (**6**). After the highly diastereoselective Evan's aldol reaction with propionaldehyde in the presence of dibutyl trifluoromethane sulfonate, the diastereomerically pure (*S*)-3-[(2*S*,3*R*)-3-hydroxy-2-methyl-1-oxopentyl]-4-phenylmethyl-1,3-oxazolidin-2-one (**7**) could be isolated in good yield after recrystallization (Figure 2). The enantiomerically pure 1,3-diol (**10**) was synthesized by selective cleavage of the side chain of the oxazolidinone and subsequent lithium aluminum hydride reduction of the *β*-hydroxy acid (**9**) (Chong, 1994). Protection of the more reactive primary hydroxyl group afforded the siloxy alcohol. A direct conversion of the secondary hydroxyl group into an acetylthio group by a Mitsunobu type reaction (Hughes, 1992) failed probably due to steric hindrance. Thus, a better leaving group had to be generated. Reaction of the hydroxyl group with mesyl chloride gave the mesylate (**11**), which was then subjected to an S_N2 type reaction with potassium thioacetate in the presence of a crown ether. The crude reaction mixture was taken on for the next steps. Straightforward deprotection of the primary hydroxyl group with tetrabutylammonium fluoride followed by lithium aluminum hydride reduction of the thioacetate afforded the final product (2*R*,3*S*)-3-mercapto-2-methylpentan-1-ol (**1**) after column chromatography (Figure 3). GC analysis on a chiral column

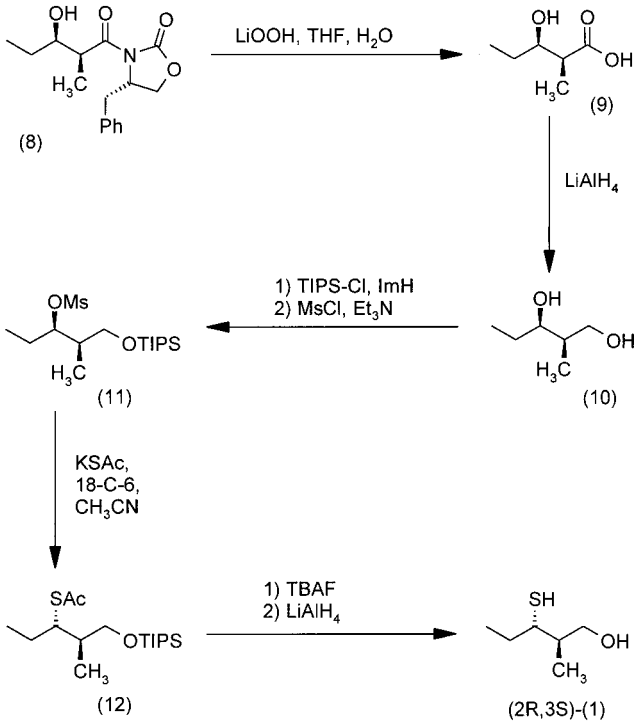

Figure 3. Synthesis of the *anti*-(2*R*,3*S*)-3-mercapto-2-methylpentanol.

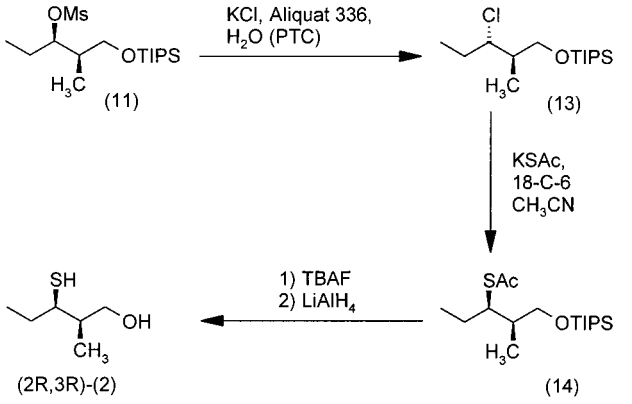

Figure 4. Formation of the *syn* product by double inversion of configuration.

showed no traces of either a second diastereomer or enantiomer.

The synthesis of the diastereomeric (2*R*,3*R*)-3-mercapto-2-methylpentan-1-ol (**2**) started from the same *syn*-mesylate (**11**). To obtain the final *syn* product, the stereocenter at carbon three had to be inverted twice by two consecutive S_N2 type reactions. First, the mesylate was converted to the secondary *anti*-configured alkyl chloride (**13**) by reaction with potassium chloride and Aliquat 336 under phase transfer conditions. The chloride was then directly replaced by a thioacetate group through reaction with potassium thioacetate in the presence of 18-crown-6. The resulting product (**14**) exhibited the correct *syn* configuration and subsequent deprotection with tetrabutylammonium fluoride and lithium aluminum hydride afforded the (2*R*,3*R*)-3-mercapto-2-methylpentan-1-ol (**2**) (Figure 4). After chromatographic purification, chiral GC analysis of this product revealed ~0.1−0.3% of the 2*R*,3*S* diastereomer. The impurity was caused by partial epimerization during the mesylate substitution reaction by a chloride

Table 1. Odor Thresholds of 3-Mercapto-2-methylpentanol Isomers

	odor threshold	
enantiomer	water (μg/L)	air (ng/L)
2*R*,3*S*	0.04	0.00007−0.0002
2*S*,3*R*	0.03	0.003−0.007
2*R*,3*S*	>12	nd[a]
2*S*,3*R*	>30	nd

[a] nd, not determined (see also text).

anion. The (2*S*,3*R*)-(**3**) and (*2S*,3*S*)-(**4**) diastereomers were prepared accordingly starting from D-phenylalanine.

Odor Characteristics of 3-Mercapto-2-methylpentanols. The odor thresholds of the broth-like, sweaty, and leek-like smelling enantiomers of 3-mercapto-2-methyl-pentanol were determined in water and in air. The results are compared in Table 1. Low odor thresholds in water could be found for the two *anti* enantiomers (0.04 and 0.03 ppb), whereas the thresholds of the *syn* enantiomers are higher by factors of 300 and 1000. These results clearly demonstrated that the thresholds strongly depend on the stereochemistry of the odorant.

The thresholds of the *syn* enantiomers could only be estimated as they contain small amounts of the corresponding *anti* isomer. The 2*R*,3*R* isomer contains ~0.3% of the 2*R*,3*S* enantiomer, and the 2*S*,3*S* was contaminated with 0.1% of the 2*S*,3*R* compound. On the basis of the amounts of these impurities and the thresholds determined for the *syn* isomers, calculations showed that the threshold values determined for the *syn* isomers may derive from the *anti* isomers and the real thresholds of the *syn* isomers should be higher than these values.

Due to the impurities in the *syn* isomers, odor threshold values in air were determined only for the *anti* isomers. The results in Table 1 indicated a very low odor threshold, especially for the 2*R*,3*S* enantiomer. This extremely low threshold places the 2*R*,3*S* enantiomer among the most potent flavor compounds.

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