JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Quantitation of the Intense Aroma Compound 3-Mercapto-2-methylpentan-1-ol in Raw and Processed Onions (*Allium cepa*) of Different Origins and in Other *Allium* Varieties Using a Stable Isotope Dilution Assay

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A stable isotope dilution assay was developed for the quantitation of the potent onion odorant 3-mercapto-2-methylpentan-1-ol (**1**) using mass chromatography and synthesized [$^2H_2$]-3-mercapto-2-methylpentan-1-ol as the internal standard. Application of the newly developed method on onions from different origins revealed amounts between 8 and 32 μg/kg in raw onions, whereas 34−246 μg was found in sliced, stored (50 min), and then cooked onions. In extracts prepared by simultaneous steam distillation−extraction the highest concentrations of **1** were formed, amounting to >1200 μg/kg. The much higher content of 3-mercapto-2-methylpentan-1-ol in cooked onions suggested its formation from specific, yet unkown, precursors enzymatically formed during cutting of raw onions. **1** was for the first time identified and also quantified in other *Allium* species such as chives, scallions, and leek, whereas surprisingly garlic and bear's garlic did not contain the aroma compound.

KEYWORDS: 3-Mercapto-2-methylpentan-1-ol; stable isotope dilution assay; onions; leek; chives; garlic

INTRODUCTION

Because of their characteristic aroma, onions (*Allium cepa* L.) are among the most important culinary ingredients in many food recipes all over the world. Other vegetables, such as garlic, leek, bear's garlic, shallots, or chives belonging to the same plant family (Liliaceae; variety: Alliaceae), are also very often used as important "flavor improvers" in many foods.

It is well-known that volatile sulfur compounds, which are formed during cutting by a very fast enzymatic degradation of cysteine sulfoxides, followed by a cascade of chemical reactions, predominantly contribute to the aroma of raw onions (*1*−*4*). The chemistry of onion volatiles is, however, quite complex, in particular, because significant changes in the spectrum of volatiles occur during storage and/or processing after disruption of the cell walls (*3*, *4*).

Widder et al. (*5*) recently applied GC/Olfactometry to an extract of volatiles isolated from thermally processed flavors containing onion extracts and detected a very potent odorant eliciting an intense, meat broth-like, onion-like aroma quality. In an extract prepared by vacuum distillation of raw onions, this compound was identified for the first time as 3-mercapto-2-methylpentan-1-ol (**1**) on the basis of NMR experiments and synthesis. The odor threshold of a mixture of the four possible stereoisomers (**Figure 1**) was determined to be 0.15 μg/L in water (*5*). In a subsequent publication of the same group (*6*), the two *anti* enantiomers 2*R*,3*S* and 2*S*,3*R* were characterized as the most odor-active isomers, showing odor thresholds of 0.04 and 0.03 μg/L in water, respectively. Very recently, **1** has been suggested as an inhibitor of peroxynitrite formation, which is involved in numerous diseases (*7*).

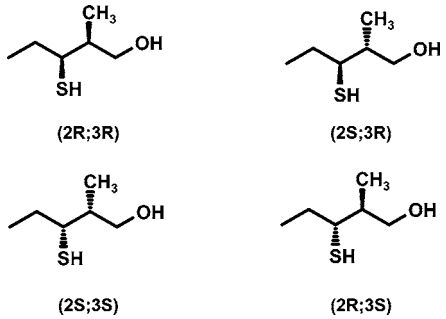

Figure 1. Structures of the four possible diastereoisomers of 3-mercapto-2-methylpentan-1-ol (**1**).

Up to now, no quantitative data are available on the amounts of **1** in onions depending on the processing state (raw, cooked, deep-fried, etc.). Furthermore, it is unknown whether **1** also occurs in other members of the *Allium* variety. This prompted us to look for its occurrence in various other *Allium* varieties and vegetables and to develop a reliable method for its quantitation, because 3-mercapto-2-methylpentan-1-ol should be quite unstable due to the sulfhydryl group, which might be oxidized into the corresponding disulfide as previously shown by us for other thiol aroma compounds (*8*). Because stable

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10.1021/jf049874l CCC: $27.50
Published on Web 04/27/2004

isotope dilution assays have been proven to be a potent tool for the quantitation of, in particular, unstable thiols in foods (*8*), the purpose of this study was (i) to develop a stable isotope dilution analysis for **1** and (ii) to determine its concentrations in fresh and processed onions as well as in other *Allium* varieties and vegetables.

EXPERIMENTAL PROCEDURES

Materials. Yellow onions (Germany), Spanish onions (Spain), white onions (Italy) (*Allium cepa*), shallots (France) (*Allium ascalonicum*), chives (Israel) (*Allium schoenoprasum*), scallions (Italy) (*Allium fistulosum*), leek (Austria) (*Allium porrum*), garlic (Argentina) (*Allium sativum*), and bear's garlic (Germany) (*Allium ursinum*) as well as broccoli (*Brassica oleracea*), winter radish (*Raphanus sativus* L. var. *niger*), celeriac (*Apium graveolens* var. *rapaceum*), fennel (*Foeniculum vulgare*), and asparagus (*Asparagus officinalis*) (all from Germany) were purchased at a local market in the period from July 2002 to July 2003. About two to six different batches from the same country were analyzed.

Chemicals. 2-Methyl-2-pentenal, piperidine, lithium aluminum hydride, and lithium aluminum deuteride were from Aldrich (Sigma-Aldrich Chemie, Taufkirchen, Germany); thioacetic acid and sodium chlorite were from Fluka (Sigma-Aldrich Chemie, Taufkirchen, Germany). All other chemicals were of analytical grade.

Syntheses. *3-Mercapto-2-methylpentan-1-ol*. The synthesis was carried out according to the procedure described by Widder et al. (*5*) with slight modifications:

Piperidine (0.07 g; 0.82 mmol) was mixed with 2-methyl-2-pentenal (6.74 g; 0.07 mol) in an argon atmosphere at 10 °C. Thioacetic acid (7.9 g; 0.10 mol) was dropwise added within 10 min, then the reaction mixture was stirred for 18 h at room temperature, and finally diluted with diethyl ether (50 mL). The solution was washed with hydrochloric acid (10 mL; 1 mol/L) followed by an aqueous saturated sodium hydrogencarbonate solution (total volume 60 mL) and finally brine. After drying of the organic phase over sodium sulfate, the solvent was evaporated and the crude thioester obtained was slowly added to a suspension of lithium aluminum hydride (2.5 g, 66 mmol) in 80 mL of dry diethyl ether at 0 °C in an atmosphere of argon. The mixture was refluxed for 150 min and, after cooling to 0 °C, treated with a saturated ammonium chloride solution (40 mL) followed by hydrochloric acid (20 mL; 2 mol/L). The organic phase was washed with an aqueous saturated sodium hydrogencarbonate solution (total volume = 50 mL), dried over sodium sulfate; after filtration, the solvent was distilled off at 40 °C, and the target compound was obtained as a clear liquid.

[$^2H_2$]-3-Mercapto-2-methylpentan-1-ol. In a first step, 3-(acetylthio)-2-methylpentanal was synthesized from 2-methyl-2-pentenal and thioacetic acid following the procedure described above for the unlabeled compound. The crude 3-(acetylthio)-2-methylpentanal obtained was then oxidized into the corresponding acid using sodium chlorite (*9*): To a solution of 3-(acetylthio)-2-methylpentanal (0.87 g; 5 mmol) and sulfamic acid (0.63 g; 6.5 mmol) in water/ethanol (350 mL; 25:10; v/v) was slowly added sodium chlorite (0.61 g; 6.7 mmol) dissolved in water/ethanol (20 mL; 1+1; v/v). After 2 h of stirring at room temperature, the solution was extracted with diethyl ether (total volume = 250 mL). After drying over Na_2SO_4, the solvent was evaporated, and aliquots of the crude product were purified using a water-cooled column (10−12 °C, 25 × 1 cm) packed with a slurry of silica gel (silica 60, 0.063−0.200 mm, VWR International, formerly Merck, Darmstadt, Germany) in diethyl ether. Isolation was performed using diethyl ether as the effluent (2 mL/min; 50 mL).

The 3-(acetylthio)-2-methylpentanoic acid obtained (0.44 g; 2.2 mmol) was dissolved in anhydrous diethyl ether (5 mL), and slowly added to a suspension of lithium aluminum deuteride (0.4 g; 9.5 mmol) in anhydrous diethyl ether (10 mL) at −5 °C under an argon atmosphere with stirring. Stirring was continued for another 150 min under reflux. After cooling, the mixture (0 °C) was treated with an aqueous, saturated ammonium chloride solution (15 mL), and then hydrochloric acid (10 mL; 2 mol/L) was added. The organic phase was separated, and the aqueous phase was extracted with diethyl ether (total volume 50 mL). The combined ethereal phases were washed with aqueous saturated sodium hydrogencarbonate solution (total volume = 30 mL) and dried over sodium sulfate. The concentration of the target compound was determined using methyl octanoate as the internal standard and using a response factor of 0.93 calculated from data obtained by GC-FID analysis of a mixture of unlabeled **1** and methyl octanoate.

Isolation and Quantitation Procedures. *Raw Onions.* After tap water (150 mL) and onions (100 g) were mixed, the onions were minced for 1 min using a homogenizer (Ultraturrax, Jahnke & Kunkel, IKA-Labortechnik, Staufen, Germany). The sample was then spiked with [$^2H_2$]-**1** [0.5−2 μg, dissolved in dichloromethane (0.5 mL), depending on the amounts expected] and stirred for 50 min in a closed vessel. After the addition of dichloromethane (200 mL), the sample was stirred for another 50 min. The mixture was centrifuged (4000 rpm, 5 °C; centrifuge GR 412, Jouan, Unterhaching, Germany) to break the emulsion formed, and, after the solvent had been decanted off, the extraction was repeated twice (total volume = 100 mL). The organic layers were combined, dried over Na_2SO_4, and, after the solvent had been distilled off to ∼100 mL, the volatile compounds were separated from the nonvolatile material using the solvent-assisted flavor evaporation (SAFE) technique (*10*). The distillate was dried over sodium sulfate and concentrated to ∼1 mL on a Vigreux column (60 × 1 cm) and to ∼300 μL using a microdistillation device (*11*). The same procedure was also applied to the other raw *Allium* varieties or vegetables, respectively.

Intactly Cooked Onions. One whole, peeled onion (∼150 g) was cooked for 20 min in a pressure cooker (Fissler, 2 L, Idar-Oberstein, Germany). After cooling, the sample was homogenized (Ultraturrax, Jahnke & Kunkel, IKA-Labortechnik, Staufen, Germany), and [$^2H_2$]-**1** (0.5 μg), dissolved in dichloromethane (0.5 mL), was added. The volatiles (including **1** and [$^2H_2$]-**1**) were then isolated as described above for the raw onions.

Deep-Fried Onions. Onions (100 g) were finely sliced and immediately deep-fried at 150 °C for 165 s in a chip pan (frifri, Freiburg, Germany) using 5 kg of palm butter. After deep-frying, the surplus palm butter was removed from the deep-fried material using a paper towel. The material was frozen in liquid nitrogen, and, after addition of sodium sulfate (100 g), the sample was homogenized in a blender (Moulinex, Radolfzell, Germany). After the addition of [$^2H_2$]-**1** (0.5−1.5 μg), dissolved in dichloromethane (0.5 mL), the material was extracted twice with diethyl ether (total volume = 300 mL) for a total time of 2 h. The volatiles were isolated as described above for the raw onions.

Minced, Cooked Onions. Tap water (150 mL) and onions (100 g) were mixed, and the onions were minced for 1 min using a homogenizer (Ultraturrax; Jahnke & Kunkel, IKA-Labortechnik) and left for 50 min in a closed vessel. Then, the material was cooked for 20 min in a pressure cooker (Fissler, 2 L). After cooling to room temperature, [$^2H_2$]-**1** (10−25 μg) was added. The sample was stirred for 15 min and after the addition of dichloromethane (200 mL) for another 50 min. Then, the mixture was centrifuged (4000 rpm, 5 °C; centrifuge GR 412, Jouan, Unterhaching, Germany) and, after the solvent had been decanted off, the extraction was repeated twice. The volatile compounds were isolated as described above for the raw onions.

Simultaneous Steam Distillation−Extraction (SDE). Onions (100 g) were homogenized for 1 min in tap water (150 mL) using a homogenizer (Ultraturrax, Janke & Kunkel, IKA-Labortechnik). After spiking with [$^2H_2$]-**1** (25−80 μg), the samples were immediately heated and continuously steam-distilled and extracted with diethyl ether (150 mL) for 2 h (*12*). After drying over sodium sulfate, the extract was concentrated to 1.5 mL using a Vigreux column (60 × 1 cm).

High-Resolution Gas Chromatography (HRGC)−Mass Spectrometry (MS). HRGC-MS analyses were performed by means of a Hewlett-Packard gas chromatograph, type 5890 series II (Hewlett-Packard, Waldbronn, Germany), coupled to a sector field mass spectrometer, type MAT 95 S (Finnigan MAT, Bremen, Germany), running either in the electron impact (EI) mode (70 eV ionization energy) or in the chemical ionization mode (CI) using isobutane as the reagent gas (115 eV ionization energy). The samples were injected by the cold on-column technique at 40 °C onto an FFAP fused silica capillary column [30 m × 0.25 mm i.d.; 0.25 μm film thickness (J&W Scientific, Folsom, CA)]. The oven temperature was held for 2 min

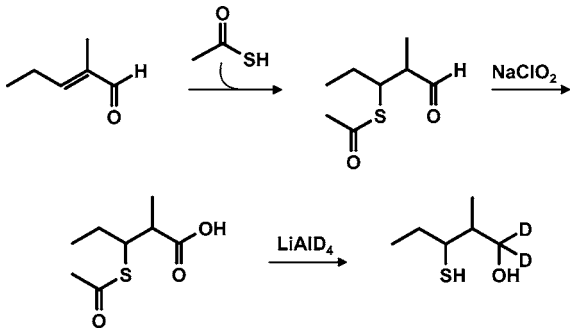

Figure 2. Reaction sequence used in the synthesis of [$^2H_2$]-3-mercapto-2-methylpentan-1-ol ([$^2H_2$]-**1**).

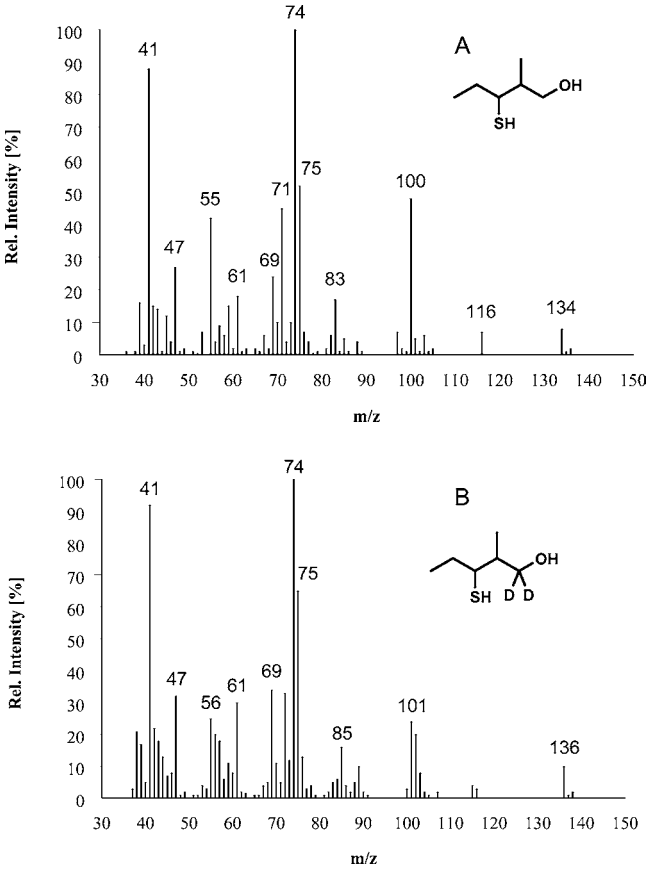

Figure 3. Mass spectra (MS/EI) of 3-mercapto-2-methylpentan-1-ol (**A**) and [$^2H_2$]-3-mercapto-2-methylpentan-1-ol (**B**).

isothermally, then raised by 40 °C/min to 60 °C, held for 1 min, and then raised by 6 °C/min to 230 °C.

Quantitation of 1 by Stable Isotope Dilution Analysis. This was performed by means of two-dimensional GC-MS as described recently (*13*). For this purpose, a Thermo Finnigan gas chromatograph, type Trace 2000 series (Thermo Finnigan, Egelsbach, Germany), equipped with a moving capillary stream switching (MCSS) system was coupled via a second gas chromatograph (CP 3800, Varian, Darmstadt, Germany) to an ion trap detector Saturn 2000 (Varian) running in the chemical ionization mode (70 eV ionization energy) with methanol as reagent gas. The traces *m*/*z* 117 (**1**) and *m*/*z* 119 ([$^2H_2$]-**1**) were recorded, and the concentrations were calculated from a response curve measured as described recently (*14*).

Odor Threshold Determination. A defined amount of 3-mercapto-2-methylpentan-1-ol, dissolved in 100 μL of ethanol, was added to boiled tap water (1 L). After stirring, this stock solution was diluted stepwise (1 + 1 by volume). Odor threshold values were determined using triangle tests as described recently (*15*).

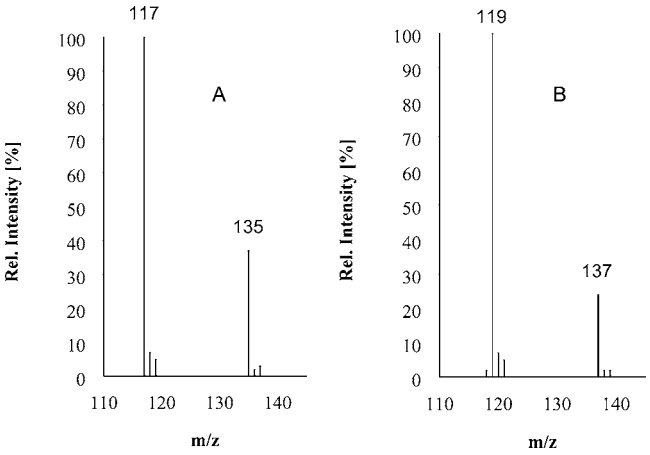

Figure 4. Mass spectra (MS/CI) of 3-mercapto-2-methylpentan-1-ol (**A**) and [$^2H_2$]-3-mercapto-2-methylpentan-1-ol (**B**).

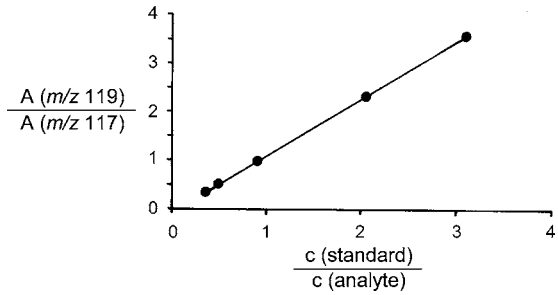

Figure 5. Standard curve used in the determination of the response factor for [$^2H_2$]-3-mercapto-2-methylpentan-1-ol. Area ratios (*A*) of *m*/*z* 119/*m*/*z* 117 versus the concentration ratios (*c*) of standard and analyte are given.

RESULTS AND DISCUSSION

Development of the Stable Isotope Dilution Assay. Isotopomers of aroma compounds containing a 100% stable isotope label are scarcely commercially available. Thus, the first step in the development of a stable isotope dilution assay is the synthesis of the respective isotopomer. For this purpose, a position in the molecule that is not eliminated during mass spectrometry has to be chosen for labeling.

As summarized in **Figure 2**, a three-step reaction sequence was used to prepare [$^2H_2$]-3-mercapto-2-methylpentan-1-ol ([$^2H_2$]-**1**) doubly labeled at carbon-1. The first step was carried out as a Michael addition of thioacetic acid at the double bond of 2-methyl-2-pentenal according to a procedure also used in the preparation of the unlabeled aroma compound. To introduce the labeling, the aldehyde was oxidized to yield 3-(acetylthio)-2-methylpentanoic acid, and the label was finally introduced by a reduction of the acid into the alcohol using $LiAlD_4$ (**Figure 2**). This procedure starts from the intermediate 3-(acetylthio)-2-methylpentanal, already used in the synthesis of the unlabeled thiol, thus saving time.

The mass spectrum of unlabeled **1** is shown in **Figure 3A**. In agreement with a recently published spectrum (*5*), a weak molecular ion (*m*/*z* 134) was monitored and also the ions formed by elimination of H_2O (*m*/*z* 116) and H_2S (*m*/*z* 100) were measured.

In the mass spectrum of the newly synthesized isotopomer [$^2H_2$]-3-mercapto-2-methylpentan-1-ol ([$^2H_2$]-**1**) (**Figure 3B**), a weak molecular ion at *m*/*z* 136 was observable, confirming the expected incorporation of two deuterium atoms into the target molecule. Analysis by MS/CI, using methanol as the reagent gas, gave a base peak at *m*/*z* 119 and a molecular mass at *m*/*z* 137 [$(M + 1)^+$ (**Figure 4B**)]. By comparison with the MS/CI

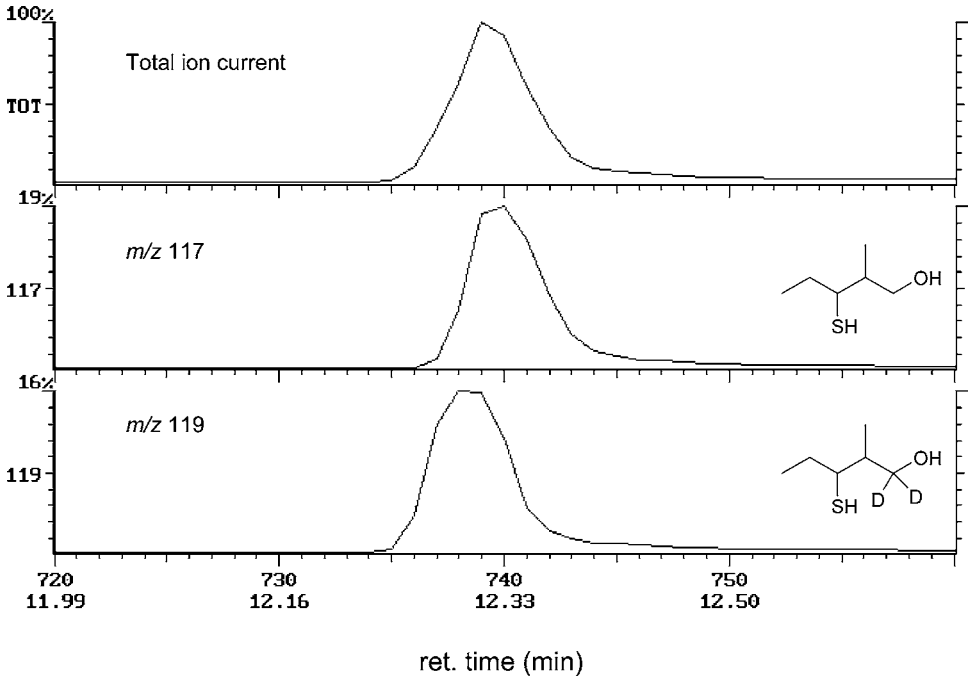

Figure 6. Mass chromatogram obtained during quantitation of 3-mercapto-2-methylpentan-1-ol in a yellow onion sample, *m*/*z* 119 ([$^2H_2$]-**1**) and *m*/*z* 117 (**1**).

Table 1. Concentrations and Odor-Activity Values (OAVs) of 3-Mercapto-2-methylpentan-1-ol in Raw Onions and Other *Allium* Varieties of Different Origins

expt	sample	concn[a] (μg/kg)	n[b]	OAV[c]
1	yellow onions (Germany)	9.8 (7.1–12.0)	3	6100
2	white onions (Italy)	32.3 (28.6–35.9)	2	20000
3	red onions (Spain)	18.7 (16.8–20.6)	2	12000
4	Spanish onions (Spain)	8.0 (5.4–10.8)	3	5000
5	shallots (France)	25.9 (20.6–32.7)	5	16000
6	chives (Israel)	125.4 (105.2–146.3)	3	78000
7	scallions (Italy)	21.1 (17.1–25.3)	3	13000
8	leek (Austria)	33.0 (27.9–42.0)	4	21000
9	garlic (Argentina)	<0.4[d]	3	
10	bear's garlic (Germany)	<0.4[d]	3	

[a] Mean values (concentration range). [b] Number of different batches analyzed. [c] OAVs were calculated by dividing the concentrations of 3-mercapto-2-methylpentan-1-ol by its recognition threshold. [d] Limit of detection.

of the unlabeled **1**, the incorporation of two deuterium atoms was proven. Besides the fragment *m*/*z* 117, the unlabeled compound also showed a small ion at *m*/*z* 119 (**Figure 4A**) resulting from the isotope mass of sulfur (roughly 4%).

Five mixtures containing known amounts of **1** and [$^2H_2$]-**1** (molar ratios of 5:1 to 1:5) were then analyzed by MS/CI for the ions *m*/*z* 117 (unlabeled **1**) and *m*/*z* 119 (labeled **1**) to generate a response curve. From the gradient of the resulting curve (**Figure 5**) a response factor of 0.91 was calculated.

Quantitation of 1 in Onions. In a first experiment, the amounts of **1** in four samples of raw, sliced, and stored onions (*A. cepa* L.) from different origins were determined. As an example, the mass chromatogram obtained for the quantitation of **1** in the yellow onion sample is shown in **Figure 6**. The concentrations of the thiol were quite low and varied between 8 μg/kg in Spanish onions (expt 4; **Table 1**) and 32 μg in white onions (expt 2; **Table 1**). The differences in the concentrations between two and six different onion batches of the same origin were quite pronounced; however, the white onions were always highest in the amounts of **1**.

Odor activity values (OAVs; ratio of concentration to odor threshold) are a helpful tool to approximate the contribution of single odorants to a given aroma (*15*). A calculation of the odor threshold of synthesized **1** in boiled tap water revealed a detection threshold of 0.7 ng/L and a recognition threshold of 1.6 ng/L. These values determined in our study were by a factor of nearly 100 lower than those reported recently (*5*) for the racemic mixture and were even lower by a factor of 20−40 compared to previous data reported for the most odor-active 2*S*,3*R* diastereoisomer (*6*). However, differences in odor thresholds determined in different studies are common in this order of magnitude, but need to be fine-tuned, if necessary.

A calculation of the OAV of **1** in the four different raw onions (expts 1−4; **Table 1**) suggested that this compound should undoubtedly contribute much to the aroma of raw onions, because its concentration was at least 5000-fold above its odor threshold.

Four other types of Alliaceae varieties (expts 5−8; **Table 1**) also contained relatively high amounts of the flavor compound with chives (expt 6; **Table 1**) being highest in the concentration of the odorant, whereas garlic and bear's garlic, although having an intense sulfury smell, did not contain the thiol.

It should be mentioned that these results are the first report of **1** in shallots, chives, scallions, and leek.

Also, further vegetables, such as broccoli, winter radish, celeriac, fennel, and asparagus, were analyzed for 3-mercapto-2-methylpentan-1-ol. However, none of these foods contained the thiol in amounts >0.4 μg/kg (limit of detection).

In the literature (*5*) it has been proposed that **1** is formed by a reaction sequence involving a cascade of enzymic and chemical reactions as shown in **Figure 7**, for example, an Aldol reaction of propanal, followed by the addition of H_2S to the double bond of the 2-methyl-2-pentenal formed, and finally a reduction into the alcohol (*5*).

To confirm that enzymatic reactions are necessary to generate **1**, Spanish onions were sliced and immediately deep-fried (expt 1; **Table 2**). In this experiment, no formation of **1** was observed, thereby confirming that enzymatic reactions are undoubtedly

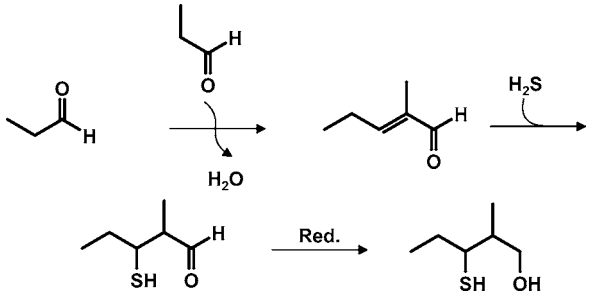

Figure 7. Hypothetical formation pathway of 3-mercapto-2-methylpentan-1-ol in onions modified according to ref *5*.

Table 2. Influence of a Thermal Treatment on the Formation of **1** from Spanish Onions

expt	sample	concn[a] (μg/kg)	n[b]
1	deep-fried	<0.2[c]	3
2	cooked (entire bulb)	1.5 (1.3–1.7)	4
3	cooked (minced)	34.6 (21.8–46.7)	3

[a,b] For footnotes *a* and *b* see **Table 1**. [c] Limit of detection.

Table 3. Concentrations of 3-Mercapto-2-methylpentan-1-ol in Onions and Other Cooked Vegetables

expt	sample[a]	concn[b] (μg/kg)	n[c]
1	yellow onions (Germany)	51.3 (45.6–56.6)	3
2	white onions (Italy)	246.6 (185.1–279.8)	6
3	red onions (Spain)	101.2 (87.5–113.3)	3
4	Spanish onions (Spain)	34.6 (22.8–47.7)	3
5	shallots (France)	177.0 (147.7–208.9)	6
6	chives (Italy)	347.6 (320.1–371.4)	3
7	scallions (Italy)	102.0 (94.9–110.0)	3
8	leek (Austria)	367.2 (331.1–401.2)	3

[a] Same batches as in **Table 1**. [b,c] See footnotes *a* and *b* in **Table 1**.

needed to generate precursors of **1**, such as propanal or 2-methylpentanal (**Figure 7**). In a following experiment, an entire onion was cooked in a pressure cooker. Also in this experiment (expt 2; **Table 2**), only very low amounts of **1** were generated, corroborating the assumption that a cell disruption is clearly necessary to generate the aroma compound, because, when the same batch of onions was minced, left at room temperature for 50 min, and then cooked, the thiol was formed (expt 3; **Table 2**).

Because our preliminary results had shown that the amounts of **1** could be much increased by a thermal processing, the influence of heat on the formation of the odorant was investigated in more detail. Cutting of onions followed by a 35 min rest at room temperature and, finally, a 20 min cooking process increased the concentrations of **1** in all onion samples as well as in shallots, scallions, chives, and leek (**Table 3**) as compared to the raw, minced, and stored onions (**Table 1**). In total, the amounts were higher by factors of 3−7 compared to the raw samples (cf. **Tables 1** and **3**). Cooking of leek was most effective in generating the thiol, because the amount analyzed in the cooked sample was 11-fold higher than in the uncooked material (cf. **Tables 1** and **3**).

Steam distillation is known to generate flavor compounds from precursors during cooking (*12*, *17*). Homogenization of several onion samples for only 1 min, followed by SDE, led to the highest amounts of **1** in four onion samples as well as in shallots (**Table 4**). In white onions, >1.2 mg/kg of the aroma compound was formed. Compared to the raw samples (**Table 1**), the amounts formed were by factors of 16−50 higher in the extracts obtained by SDE (**Table 4**). These results suggest that a high content of 3-mercapto-2-methylpentan-1-ol can be formed only after a yet unknown enzymatic reaction yielding a precursor which is finally degraded into the thiol by application of heat. Because steam distillates of sliced onions do contain the highest amount of this aroma-active, but also bioactive, compound, this "cooking" procedure can be recommended to obtain the highest yields of **1** from onions.

Table 4. Concentrations of 3-Mercapto-2-methylpentan-1-ol in Onions from Different Sources Isolated by Simultaneous Steam Distillation–Extraction

expt	sample[a]	concn[b] (μg/kg)	n[c]
1	yellow onions (Germany)	198.5 (185.7–211.3)	2
2	white onions (Italy)	1201.7 (1070.8–1332.6)	2
3	red onions (Spain)	298.2 (286.7–309.6)	2
4	Spanish onions (Spain)	421.1 (406.6–435.6)	2
5	shallots (France)	691.0 (648.0–735.0)	2

[a–c] For footnotes see **Table 3**.

ACKNOWLEDGMENT

We are grateful to S. Heinel for skillful technical assistance.

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Received for review January 23, 2004. Revised manuscript received March 17, 2004. Accepted March 17, 2004.

JF049874L