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**Insights into the Key Compounds of Durian (*Durio zibethinus* L. 'Monthong') Pulp Odor
by Odorant Quantitation and Aroma Simulation Experiments**

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1 **ABSTRACT:** Sixteen compounds, previously identified as potent odorants by application of an
2 aroma extract dilution analysis and the gas chromatography-olfactometry analysis of static
3 headspace samples were quantitated in the pulp of durians, variety Monthong, and odor activity
4 values (OAVs) were calculated by dividing the concentrations obtained by the odor thresholds of
5 the compounds in water. In combination with data recently reported for hydrogen sulfide and
6 short-chain alkanethiols, OAVs > 1 were obtained for 19 compounds, among which ethyl (2*S*)-2-
7 methylbutanoate (fruity; OAV 1700000), ethanethiol (rotten onion; OAV 480000), and 1-
8 (ethylsulfanyl)ethanethiol (roasted onion; OAV 250000) were the most potent, followed by
9 methanethiol (rotten, cabbage; OAV 45000), ethane-1,1-dithiol (sulfury, durian; OAV 23000),
10 and ethyl 2-methylpropanoate (fruity; OAV 22000). Aroma simulation and omission
11 experiments revealed that the overall odor of durian pulp could be mimicked by only two
12 compounds, namely ethyl (2*S*)-2-methylbutanoate and 1-(ethylsulfanyl)ethanethiol, when
13 combined in their natural concentrations.

14 **KEYWORDS:** *durian, Durio zibethinus, stable isotope dilution assay, SIDA, omission test,*
15 *ethyl (2*S*)-2-methylbutanoate, 1-(ethylsulfanyl)ethanethiol, dithiohemiacetal.*

16 Introduction

17 Durian fruit is exceedingly popular in many East Asian countries. Its edible part, a
18 yellowish pulp, consists of the arils encasing the seeds. The pulp exhibits a sweet taste and a
19 pleasant custard-like consistency. Its odor, which combines some fruitiness with a dominating
20 sulfury note, however, is extremely strong and, from the point of view of an American or
21 European not used to durian consumption, very extraordinary and rather repellent. To approach
22 the compounds being responsible for the characteristic odor properties, we recently screened the
23 volatile fraction of durian pulp, variety Monthong, by aroma extract dilution analysis (AEDA)
24 and gas chromatography–olfactometry (GC-O) of headspace samples.¹ Results revealed high
25 flavor dilution (FD) factors for fruity smelling ethyl (2*S*)-2-methylbutanoate, honey-like smelling
26 ethyl cinnamate, and roasted onion-like smelling 1-(ethylsulfanyl)ethane-1-thiol, followed by 1-
27 (ethyldisulfanyl)-1-(ethylsulfanyl)ethane (sulfury, onion), 2(5)-ethyl-4-hydroxy-5(2)-
28 methylfuran-3(2*H*)-one (caramel), 3-hydroxy-4,5-dimethylfuran-2(5*H*)-one (soup seasoning),
29 ethyl 2-methylpropanoate (fruity), ethyl butanoate (fruity), 3-methylbut-2-ene-1-thiol (skunky),
30 ethane-1,1-dithiol (sulfury, durian), 1-(methylsulfanyl)ethane-1-thiol (roasted onion), 1-
31 (ethylsulfanyl)propane-1-thiol (roasted onion), and 4-hydroxy-2,5-dimethylfuran-3(2*H*)-one
32 (caramel). Hydrogen sulfide (rotten egg), acetaldehyde (fresh, fruity), methanethiol (rotten,
33 cabbage), ethanethiol (rotten, onion), and propane-1-thiol (rotten, durian) were additionally
34 identified as potent odorants by headspace GC-O analysis. The concentrations of hydrogen
35 sulfide, methanethiol, ethanethiol, and propane-1-thiol in Monthong durian pulp were recently
36 determined by newly developed stable isotope dilution assays employing sodium (³⁴S)sulfide,
37 sodium (²H₃)methanethiolate, (²H₅)ethanethiol, and (²H₇)propane-1-thiol as internal standards
38 and 3-buten-2-one as derivatization reagent.² Results showed an exceptional high odor activity

39 value (OAV; ratio of concentration to odor threshold) for ethanethiol (480000) and also a
40 comparably high OAV for methanethiol (45000), whereas OAVs for propane-1-thiol (6300) and
41 hydrogen sulfide (330) were lower. Data therefore suggested that in particular ethanethiol
42 contributes to the overall aroma of Monthong durian pulp. To verify this assumption and clarify
43 the odor impact of other compounds, the aim of the present study was to complete the
44 quantitation of all odorants detected with high FD factors during the screening by AEDA,
45 validate the results of identification and quantitation through sensory evaluation of an aroma
46 reconstitution model prepared accordingly, and finally perform omission tests.

47 **Materials and Methods**

48 **Durian Pulp.** Durians were purchased from a local internet shop during years 2012 and
49 2013. They were handpicked from trees of *Durio zibethinus* 'Monthong' in Thailand and sent to
50 Germany by air. The fruits were allowed to reach full ripeness indicated by cracks in the husk
51 and release of the typical odor, which was typically the case one or two days after arrival. The
52 husk was opened by hand and the pulp was carefully removed from the seeds with a spoon.

53 **Reference Odorants.** Isotopically unmodified odorants were obtained as detailed
54 recently.¹

55 **Stable Isotopically Substituted Durian Odorants.** (²H₆)-**13** was purchased from
56 aromaLAB, Planegg, Germany. (¹³C₂)-**11**³ and (¹³C₂)-**12**⁴ were synthesized as detailed in the
57 literature. The esters (²H₃)-**3**, (²H₃)-**4**, (²H₅)-**5**, (²H₃)-**8**, and (²H₃)-**14** were synthesized from the
58 corresponding acids and alcohols through Fischer esterification.⁵ In brief, sulfuric acid (50 μL)
59 was added to a mixture of carboxylic acid (20 mmol) and alcohol (2 mmol), the mixture was
60 heated to 80 °C for 30 min, diluted with water (50 mL), and extracted with diethyl ether (100
61 mL). The ethereal extract was washed with aqueous sodium carbonate (0.5 mol/L; 3 × 100 mL)

62 and water (100 mL), dried over anhydrous sodium sulfate, and the solvent was finally removed
63 in vacuo. ($^2\text{H}_{10}$)-**1**, ($^{13}\text{C}_2$)-**2**, ($^{13}\text{C}_2$)-**6**, ($^{13}\text{C}_2$)-**7**, ($^{13}\text{C}_2$)-**9**, ($^2\text{H}_5$)-**10**, and ($^{13}\text{C}_2$)-**15** were synthesized
64 as detailed below.

65 **Miscellaneous Chemicals.** ($^{13}\text{C}_2$)Acetaldehyde, bromo($^2\text{H}_5$)ethane, di-*tert*-butyl
66 dicarbonate, dithiothreitol, ethynylmagnesium bromide (0.5 M in THF), lithium
67 diisopropylamide mono(tetrahydrofuran) (1.5 M in cyclohexane), and sodium sulfide
68 nonahydrate were purchased from Sigma-Aldrich (Taufkirchen, Germany). Dichloromethane,
69 and diethyl ether were freshly distilled before use. Mercurated agarose gel was prepared from
70 Affi-Gel 10 (Bio-Rad, Munich, Germany).⁶

71 **Syntheses.** ($^2\text{H}_{10}$)Diethyl trisulfide ($^2\text{H}_{10}$)-**1**. The compound was synthesized following an
72 approach detailed for the synthesis of the isotopically unmodified compound.⁷ To ethanol (10
73 mL), powdered sulfur (0.077 g, 2.4 mmol) and sodium hydroxide (0.097 g, 2.4 mmol) were
74 added. The mixture was refluxed under continuous stirring for 1 h. After cooling,
75 bromo($^2\text{H}_5$)ethane (0.182 g, 1.6 mmol) in ethanol (5 mL) was added. The mixture was stirred for
76 3 h, poured into cold water (70 mL), and extracted with diethyl ether (2 × 100 mL). The
77 combined organic phases were washed with water (50 mL), dried over anhydrous sodium sulfate,
78 and concentrated using a Vigreux column (60 cm) at 40 °C. The concentrate was taken up in
79 dichloromethane (50 mL). GC-MS analysis indicated a mixture of ($^2\text{H}_{10}$)diethyl disulfide and
80 ($^2\text{H}_{10}$)diethyl trisulfide. The concentration of ($^2\text{H}_{10}$)diethyl trisulfide in the solution was
81 determined by GC-FID using methyl octanoate as internal standard and a response factor
82 previously determined from the analysis of a mixture of isotopically unmodified diethyl trisulfide
83 and methyl octanoate. MS-EI, m/z (intensity in %) 46 (12), 64 (21), 66 (70), 82 (7), 94 (10), 98
84 (38), 130 (5), 164 (100; M^+); MS-CI (methanol), m/z (intensity in %) 165 (100; $[\text{M}+\text{H}]^+$). The

85 detailed MS-CI spectrum is provided in the Supporting Information.

86 $(^{13}\text{C}_2)$ Ethane-1,1-dithiol ($(^{13}\text{C}_2)$ -2). The compound was synthesized from
87 $(^{13}\text{C}_2)$ acetaldehyde (0.35 g; 7.5 mmol) and sodium sulfide nonahydrate (3.6 g, 15 mmol) using
88 the approach recently detailed for the synthesis of the isotopically unmodified compound.¹ MS-
89 EI, m/z (intensity in %) 46 (27), 59 (10), 60 (18), 61 (36), 62 (32), 63 (100), 96 (26; M^+); MS-CI
90 (methanol), m/z (intensity in %) 63 (100). The detailed MS-CI spectrum is provided in the
91 Supporting Information.

92 *1-(Ethylsulfanyl)-1-(ethylsulfanyl)($^{13}\text{C}_2$)ethane* ($(^{13}\text{C}_2)$ -6). The compound was
93 synthesized from $(^{13}\text{C}_2)$ -9 (0.061 g; 0.5 mmol) and ethanethiol (0.062 g; 1 mmol) using the
94 approach recently detailed for the synthesis of the isotopically unmodified compound.¹ MS-EI,
95 m/z (intensity in %) 46 (8), 61 (28), 62 (11), 63 (51), 66 (4), 91 (100), 121 (3); MS-CI
96 (methanol), m/z (intensity in %) 123 (100). The detailed MS-CI spectrum is provided in the
97 Supporting Information.

98 *(1,2- $^{13}\text{C}_2$)But-3-yn-2-ol*. A solution of $(^{13}\text{C}_2)$ acetaldehyde (1.0 g, 21.7 mmol) in anhydrous
99 THF (15 mL) was added dropwise to a solution of ethynylmagnesium bromide (0.5 M in THF;
100 100 mL) at $-78\text{ }^\circ\text{C}$ in an argon atmosphere. Then, the mixture was stirred at room temperature
101 for 3 h. Saturated aqueous ammonium chloride solution (300 mL) was added and the mixture
102 was extracted with dichloromethane (150 mL). The organic phase was washed with brine (200
103 mL) and dried over anhydrous sodium sulfate. The solvent was removed in vacuo to afford 0.75
104 g of the target compound as a colorless oil in 80% purity (GC-FID) corresponding to 38% yield.

105 *tert-Butyl (1,2- $^{13}\text{C}_2$)but-3-yn-2-yl carbonate*. The compound was synthesized by adapting
106 the approach published for the synthesis of the isotopically unmodified compound.^{4, 8} Pyridine
107 (2.1 mL, 25.8 mmol) and 4-(dimethylamino)pyridine (0.105 g, 0.86 mmol) were added to a

108 solution of (1,2-¹³C₂)but-3-yn-2-ol (0.60 g, 8.6 mmol) in dichloromethane (20 mL). To the
109 mixture, di-*tert*-butyl dicarbonate (4.12 g, 18.9 mmol) in dichloromethane (10 mL) was added
110 dropwise. After stirring for 4 h, the mixture was poured into an aqueous citric acid solution (0.5
111 M; 50 mL) and the phases were separated. The aqueous phase was further extracted with
112 dichloromethane (50 mL). The combined organic phases were dried under anhydrous sodium
113 sulfate. The solvent was removed in vacuo to afford 1.63 g of the target compound as a colorless
114 oil.

115 *Di-tert-butyl (1,2-¹³C₂)hept-3-yne-2,5-diyl biscarbonate.* The compound was synthesized
116 by adapting the approach published for the synthesis of the (²H₃)-substituted isotopologue.⁴ The
117 *tert*-butyl (1,2-¹³C₂)but-3-yn-2-yl carbonate obtained above was dissolved in anhydrous THF (15
118 mL). Lithium diisopropylamide mono(tetrahydrofuran) (1.5 M in cyclohexane; 5.7 mL, 8.6
119 mmol) was added dropwise at -78 °C under an argon atmosphere. After stirring (30 min),
120 propanal (0.6 g, 10.3 mmol) in anhydrous THF (5 mL) was added and stirring was continued for
121 1 h. Di-*tert*-butyl dicarbonate (2.25 g, 10.3 mmol) dissolved in anhydrous THF (10 mL) was
122 added dropwise and stirring continued for further 1 h. The mixture was allowed to warm up to
123 room temperature, diluted with diethyl ether (30 mL), and washed with aqueous sodium
124 hydroxide (2 M; 100 mL), followed by water (100 mL) and brine (100 mL). The organic phase
125 was dried under anhydrous sodium sulfate and the solvent was removed in vacuo to afford 3.8 g
126 of the target compound as a colorless oil.

127 *Di-tert-butyl (1,2-¹³C₂)hept-3,4-dione-2,5-diyl biscarbonate.* The compound was
128 synthesized by adapting the approach published for the synthesis of the (²H₃)-substituted
129 isotopologue.⁴ Potassium permanganate (1.63 g, 10.3 mmol) was dissolved in a mixture of
130 acetone (50 mL), water (10 mL), and acetic acid (1.5 mL) at 0 °C. The di-*tert*-butyl (1,2-

131 $^{13}\text{C}_2$)hept-3-yne-2,5-diyl biscarbonate obtained above was added and the mixture was stirred for
132 3.5 h at 0 °C. Aqueous sodium hydrogen sulfite (5 %; 15 mL) was added and the mixture was
133 filtered through sea sand. The acetone was removed in vacuo and the residual aqueous phase was
134 extracted with dichloromethane (3×30 mL). The combined organic extracts were washed with
135 saturated aqueous sodium hydrogen carbonate (100 mL), dried over anhydrous sodium sulfate
136 and the solvent was removed in vacuo to afford 2.25 g of the target compound as a colorless oil.

137 *2(5)-Ethyl-4-hydroxy-5(2)-(^{13}C)methyl(2(5)- ^{13}C)furan-3(2H)-one* ($(^{13}\text{C}_2)$ -7). The
138 compound was synthesized by adapting the approach published for the synthesis of the ($^2\text{H}_3$)-
139 substituted isotopologue.⁴ The Di-*tert*-butyl (1,2- $^{13}\text{C}_2$)hept-3,4-dione-2,5-diyl biscarbonate
140 obtained above was refluxed in a mixture of water (20 mL) and oxalic acid (1.8 g) for 6 h under
141 an argon atmosphere. After cooling to room temperature, brine (20 mL) was added and the
142 mixture was extracted with dichloromethane (5×30 mL). The combined organic extracts were
143 washed with saturated aqueous sodium hydrogen carbonate (150 mL), and dried over anhydrous
144 sodium sulfate to afford a stock solution of the target compound. GC-MS (DB-5 column)
145 revealed a tautomeric mixture in the ratio of 29% (first eluted isomer) to 71% (second eluted
146 isomer). MS-EI (minor tautomer), *m/z* (intensity in %) 57 (100), 59 (42), 69 (6), 87 (14); MS-EI
147 (major tautomer), *m/z* (intensity in %) 41 (24), 43 (37), 45 (100), 46 (18), 53 (13), 55 (11), 57
148 (23), 71 (63), 99 (30), 116 (10), 129 (33), 144 (81; M^+); MS-CI (methanol; both tautomers), *m/z*
149 (intensity in %) 145 (100; $[\text{M}+\text{H}]^+$). The detailed MS-CI spectrum is provided in the Supporting
150 Information.

151 *1-(Ethylsulfanyl)($^{13}\text{C}_2$)ethane-1-thiol* ($(^{13}\text{C}_2)$ -9). The compound was synthesized from
152 ($^{13}\text{C}_2$)acetaldehyde (0.22 g; 5 mmol) and ethanethiol (0.31 g, 5 mmol) using the approach
153 recently detailed for the synthesis of the isotopically unmodified compound.¹ The synthesis

154 afforded 0.28 g of the target compound as colorless oil in 80% purity (GC-FID) corresponding to
155 36% yield. MS-EI, m/z (intensity in %) 46 (12), 47 (6), 60 (11), 61 (35), 62 (40), 63 (59), 91
156 (100), 124 (29; M^+); MS-CI (methanol), m/z (intensity in %) 91 (100), 125 (13; $[M+H]^+$). The
157 detailed MS-CI spectrum is provided in the Supporting Information.

158 *1-(Ethylsulfanyl)(2,2,3,3,3- 2H_5)propane-1-thiol* ($(^2H_5)$ -**10**). The compound was
159 synthesized from (2,2,3,3,3- 2H_5)propanal (0.32 g; 5 mmol), sodium sulfide nonahydrate (1.2 g, 5
160 mmol), and ethanethiol (0.31 g; 5 mmol) using the approach recently detailed for the synthesis of
161 the isotopically unmodified compound.¹ The synthesis afforded 0,21 g of the target compound as
162 colorless oil in 52% purity (GC-FID) corresponding to 15% yield. MS-EI, m/z (intensity in %)
163 46 (17), 45 (49), 47 (11), 49 (11), 62 (24), 77 (11), 79 (38), 80 (27), 108 (100), 141 (29; M^+);
164 MS-CI (methanol), m/z (intensity in %) 108 (100), 142 (11; $[M+H]^+$). The detailed MS-CI
165 spectrum is provided in the Supporting Information.

166 *1-(Methylsulfanyl)($^{13}C_2$)ethane-1-thiol* ($(^{13}C_2)$ -**15**). The compound was synthesized from
167 ($^{13}C_2$)acetaldehyde (0.22 g; 5 mmol), sodium sulfide nonahydrate (1.2 g, 5 mmol), and sodium
168 methanethiolate (0.35 g, 5 mmol) using the approach recently detailed for the synthesis of the
169 isotopically unmodified compound.¹ The synthesis afforded 0.21 g of the target compound as a
170 colorless oil in 95% purity (GC-FID) corresponding to 36% yield. MS-EI, m/z (intensity in %)
171 43 (24), 45 (25), 46 (43), 47 (40), 48 (17), 49 (12), 60 (18), 61 (46), 62 (34), 63 (53), 77 (100),
172 110 (26; M^+); MS-CI (methanol), m/z (intensity in %) 77 (100), 111 (15; $[M+H]^+$). The detailed
173 MS-CI spectrum is provided in the Supporting Information.

174 **Enzymatic Quantitation.** The concentration of acetaldehyde in Monthong durian pulp
175 was determined by employing a commercial enzymatic assay (Roche Yellow line, R-Biopharm,
176 Darmstadt, Germany). In the presence of aldehyde dehydrogenase, acetaldehyde was

177 quantitatively oxidized by NAD^+ to acetic acid and the NADH formed was photometrically
178 determined at 340 nm.

179 **Stable Isotope Dilution Assays (SIDAs).** Portions of durian pulp (10-170 g) were spiked
180 with stable isotopically substituted durian odorants (0.5-50 μg) in dichloromethane (100 mL) and
181 each mixture was homogenized using a stainless steel blender. The organic phase was separated,
182 dried over anhydrous sodium sulfate, and nonvolatile material was removed by solvent-assisted
183 flavor evaporation (SAFE)⁹ at 40 °C. For the quantitation of non-thiol compounds, the distillate
184 was concentrated to final volumes of 0.2-1 mL, first using a Vigreux column (60 cm) and
185 subsequently a Bemelmans microdistillation device,¹⁰ and aliquots (1-2 μL) of this concentrate
186 were directly subjected to two-dimensional heart-cut gas chromatography–mass spectrometry
187 (GC-GC-MS). For the quantitation of thiols, including the dithiohemiacetals, the distillate
188 obtained by SAFE was concentrated to 5 mL and applied onto mercurated agarose gel (1 g).
189 Using the approach detailed recently,¹¹ non-thiols were washed off, the thiol fraction was eluted
190 with an excess of dithiothreitol, and the bulk of dithiothreitol was removed by SAFE. The
191 distillate was concentrated (0.2 mL) and aliquots (1-2 μL) were subjected to GC-GC-MS.

192 Peak areas corresponding to analyte and internal standard were obtained from the extracted
193 ions chromatograms using the quantifier ions detailed in Table 1. The concentration of each
194 target compound in the durian pulp was then calculated from the area counts of the analyte peak,
195 the area counts of the standard peak, the amount of durian pulp used and the amount of standard
196 added, by employing a calibration line equation previously obtained from the analysis of
197 analyte/standard mixtures in known concentrations.²

198 **Gas Chromatography–Flame Ionization Detector (GC-FID).** For the exact quantitation
199 of stable isotopically substituted durian odorants in stock solutions used in the SIDA experiments

200 and for the determination of the purity of synthetic products, a system consisting of a Trace GC
201 Ultra gas chromatograph equipped with a cold-on-column injector and an FID (Thermo
202 Scientific, Dreieich, Germany) was employed. The GC was equipped either with a fused silica
203 column DB-FFAP, 30 m × 0.32 mm i.d., 0.25 μm film, or with a fused silica column DB-5, 30 m
204 × 0.32 mm i.d., 0.25 μm film (both Agilent, Waldbronn, Germany). The carrier gas was helium
205 at 70 kPa. Injection volume was 1 μL. The initial oven temperature was 40 °C (2 min), followed
206 by a gradient of 6 °C/min, which was run until an end temperature of 240 °C. The FID was
207 connected to a computer and chromatogram recording and peak area count calculation was
208 performed by ChromQuest 5.0 (Thermo).

209 **Gas Chromatography–Mass Spectrometry (GC-MS).** Mass spectra of synthetic products
210 in the electron ionization mode (MS-EI) were recorded at 70 eV using a HP 5890 gas
211 chromatograph (Hewlett-Packard, Heilbronn, Germany) connected to an MAT 95 mass
212 spectrometer (Finnigan, Bremen, Germany). Mass spectra in the chemical ionization mode (MS-
213 CI) were acquired with a 3800 gas chromatograph connected to a Saturn 2200 mass spectrometer
214 (Varian, Darmstadt, Germany) and methanol as reagent gas. The GC column employed was a
215 DB-FFAP, 30 m × 0.25 mm i.d., 0.25 μm film, or a DB-5, 30 m × 0.25 mm i.d., 0.25 μm film
216 (both Agilent). Oven programs corresponded to those detailed for GC-FID analyses.

217 **Two-Dimensional Heart-Cut Gas Chromatography–Mass Spectrometry (GC-GC-MS).**
218 A Trace GC Ultra (Thermo) was equipped with a Combi PAL autosampler (CTC Analytics,
219 Zwingen, Switzerland), a cold-on-column injector (Thermo) and a DB-FFAP capillary, 30 m ×
220 0.32 mm i.d., 0.25 μm film (Agilent). The column end was connected to a moving column
221 stream switching system (MCSS) (Thermo). The computer-operated MCSS conveyed the
222 column eluate through uncoated fused silica capillaries (0.32 mm i.d.) either to an FID (Thermo)

223 and a tailor-made sniffing port¹² serving as monitor detectors or via a transfer line to a second
224 column inside the oven of a CP 3800 GC (Varian). This GC was connected to a Saturn 2000
225 mass spectrometer (Varian) operated in CI mode with methanol as the reactant gas.

226 For the analysis of the SIDA samples, the column in the second oven was a DB-5, 30 m ×
227 0.25 mm i.d., 0.25 μm film (Agilent). Start temperatures were 40 °C and temperature gradients
228 were 4-6 °C/min in the first and second dimension. For the determination of the enantiomeric
229 distribution of 1-(ethylsulfanyl)ethanethiol, the column in the second oven was a BGB-175, 30 m
230 × 0.25 mm i.d., 0.25 μm film (BGB Analytik, Rheinfelden, Germany). Start temperatures were
231 40 °C and temperature gradients were 6 °C/min in the first and 2 °C/min in the second
232 dimension.

233 During analysis, a heart-cut of the eluate of the first column containing the respective target
234 compound(s) was transferred during 1-1.5 min via the MCSS and the transfer line to the second
235 oven. Transferred substances were refocused by a stream of cold (−196 °C) nitrogen gas directed
236 at the end of the transfer line inside the second oven. Then, cooling was turned off and second
237 oven and mass spectrometer were started. The retention times of the target compounds in the first
238 and second dimensions were previously determined using reference compounds.

239 **Odor Thresholds.** On the basis of the procedure for the determination of odor and taste
240 thresholds by a forced-choice ascending concentration series method of limits of American
241 Society for Testing and Materials (ASTM),¹³ orthonasal odor thresholds in water were
242 determined using the experimental setup detailed previously.¹⁴

243 **Aroma Model Solutions.** Aliquots (50-200 μL) of aqueous or ethanolic stock solutions of
244 reference odorants were combined and diluted with aqueous buffer (5 mmol citric acid; pH 6.2).
245 Sunflower oil was added to obtain a final oil concentration of 3 %, which is the approximate

246 natural lipid content in durian pulp. The concentrations of the stock solutions, the size of the
247 aliquots, and the dilution with the water/oil matrix were adjusted to yield final concentrations of
248 each odorant in the model solution identical to the concentrations previously determined in the
249 durian fruit pulp and to maintain final ethanol concentration below 1 g/L.

250 **Quantitative Descriptive Analysis (QDA).** Samples of freshly homogenized durian pulp
251 (10 g) and samples of an aroma simulation model (20 compounds, OAVs ≥ 1 ; 10 g) prepared as
252 described above were placed into cylindrical (40 mm \times 65 mm high) ground neck borosilicate
253 glasses with lids (VWR, Darmstadt, Germany) and orthonasally evaluated by trained panelists.
254 Descriptors were defined on the basis of the odor of a reference compound dissolved in water at
255 a concentration of 100 \times its respective orthonasal odor threshold value. Reference odorants used
256 in the sensory experiments were ethyl (2*S*)-2-methylbutanoate (fruity), 4-hydroxy-2,5-
257 dimethylfuran-3(2*H*)-one (caramel), 3-hydroxy-4,5-dimethylfuran-2(5*H*)-one (soup seasoning),
258 1-(ethylsulfanyl)ethanethiol (roasted onion), ethanethiol (rotten onion), hydrogen sulfide (rotten
259 egg), and acetaldehyde (fresh). Panelists rated the intensity of each descriptor in both samples on
260 a seven-point scale ranging from 0 to 3 with 0.5 increments and with 0 = not detectable, 1 =
261 weak, 2 = moderate, and 3 = strong. Ratings of 14 panelists (males and females, ages 20 to 46)
262 were averaged by calculating the arithmetic mean.

263 **Omission Tests.** Aroma model solutions were prepared as described above and presented to
264 the trained panelists in the borosilicate glasses detailed before. The models from which one or
265 more odorants had been omitted were orthonasally tested against two samples of a reference
266 model in a triangle test with forced choice. For each omission experiment, the *p*-value was
267 calculated from the total number of panelists and the number of correct answers.¹⁵

268 **Results and Discussion**

269 **Development of Stable Isotope Dilution Assays.** Sixteen substances recently detected
270 among the most odor-active compounds during the application of an AEDA and the GC-O
271 analysis of static headspace samples were quantitated in the pulp of durians, variety Monthong.
272 These 16 compounds included the entire set of 12 odorants for which FD factors ≥ 256 had been
273 determined.¹ Except for acetaldehyde, which was quantitated employing a commercial enzymatic
274 assay, quantitation was aspired by SIDA using ²H- or ¹³C-substituted analogues of the target
275 compounds as internal standards to compensate for losses during the work-up procedures.

276 The majority of isotopically substituted odorants needed was obtained by synthesis, either
277 following literature procedures or by adapting procedures published for the synthesis of the
278 respective isotopically unmodified compound. In detail, synthesis of (²H₁₀)diethyl trisulfide
279 ((²H₁₀)-**1**) was accomplished by reacting bromo(²H₅)ethane with trisulfide, which was generated
280 together with the disulfide in situ from elemental sulfur in alkaline solution (Figure 1).⁷
281 Isotopically substituted analogues of the 1,1-dithio compounds ethane-1,1-dithiol (**2**), 1-
282 (ethylsulfanyl)-1-(ethylsulfanyl)ethane (**6**), 1-(ethylsulfanyl)ethane-1-thiol (**9**), 1-
283 (ethylsulfanyl)propane-1-thiol (**10**), and 1-(methylsulfanyl)ethanethiol (**15**) were synthesized as
284 described for the isotopically unmodified compounds,¹ but using (¹³C₂)acetaldehyde instead of
285 acetaldehyde and (2,2,3,3,3-⁵H₅)propanal instead of propanal. Isotopically substituted esters were
286 synthesized from the corresponding acids with the deuterated alcohols (2,2,2-²H₃)ethanol,
287 (²H₅)ethanol, or (²H₃)methanol.⁵ For the synthesis of 2(5)-ethyl-4-hydroxy-5(2)-
288 (¹³C)methyl(2(5)-¹³C)furan-3(2H)-one ((¹³C₂)-**7**), a method published by Blank et al.⁴ for the
289 synthesis of the deuterated compound was adapted. Blank et al. reported the synthesis of 2(5)-
290 (2,2,2-²H₃)ethyl-4-hydroxy-5(2)-methylfuran-3(2H)-one starting from but-3-yn-2-ol. Protection
291 by a *tert*-butyloxycarbonyl (Boc) group yielded *tert*-butyl but-3-yn-2-yl carbonate. This

292 compound was reacted with lithium diisopropylamide, followed by (3,3,3-²H₃)propanal and the
293 resulting alkoxide was in situ derivatized with di-*tert*-butyl dicarbonate to yield di-*tert*-butyl
294 (7,7,7-²H₃)hept-3-yne-2,5-diyl biscarbonate. We used the same approach, but started from (1,2-
295 ¹³C₂)but-3-yn-2-ol, which was prepared from (¹³C₂)acetaldehyde and ethynylmagnesium
296 bromide, and employed isotopically unmodified propanal instead of the deuterated compound to
297 obtain di-*tert*-butyl (1,2-¹³C₂)hept-3-yne-2,5-diyl biscarbonate (Figure 2). As reported for the
298 deuterated analogue,⁴ oxidation of the triple bond to the diketone by aqueous potassium
299 permanganate followed by removal of the Boc groups and cyclisation under acidic conditions
300 finally led to the target compound.

301 In summary, for each of the 15 target compounds a stable isotopologue was finally available,
302 which then was used as internal standard in the quantitation assays (Figure 3).

303 **Concentrations and OAVs of Monthong Durian Odorants.** Results of the SIDA
304 experiments and the enzymatic quantitation of acetaldehyde revealed concentrations roughly
305 ranging between 1 µg/kg and 100 mg/kg (Table 2), thus covering five orders of magnitude. In
306 combination with the data recently reported for hydrogen sulfide (**19**) and short-chain alkane
307 thiols in Monthong durians,² high concentrations were particular determined for acetaldehyde
308 (**16**; 98400 µg/kg), ethanethiol (**17**; 45400 µg/kg), methanethiol (**18**; 26700 µg/kg), and ethyl
309 (2*S*)-2-methylbutanoate (**5**; 13800 µg/kg), whereas concentrations of 3-methylbut-2-ene-1-thiol
310 (**13**), ethyl cinnamate (**4**), and 1-(ethylsulfanyl)propane-1-thiol (**10**) were in the low µg/kg range.

311 To achieve an approximation of the odor potency of the individual compounds, an OAV was
312 calculated for each odorant as ratio of its natural concentration in the Monthong durians to its
313 orthonasal odor detection threshold in water (Table 3). Nineteen of the 20 compounds exceeded
314 their respective odor threshold value, many of them by far. Exceptional high OAVs were

315 obtained for fruity smelling ethyl (2*S*)-2-methylbutanoate (**5**; OAV 1700000), rotten onion-like
316 smelling ethanethiol (**17**; OAV 480000), and roasted onion-like smelling 1-
317 (ethylsulfanyl)ethanethiol (**9**; OAV 250000). OAVs of this size are beyond the range typically
318 found for aroma-active compounds in food,¹⁶ however they very well corresponded to the
319 extraordinary intensity of durian pulp odor.

320 The high OAV calculated for 1-(ethylsulfanyl)ethanethiol prompted us to investigate its
321 enantiomeric distribution in durian, as enantiomers may substantially differ in their olfactory
322 properties. Analysis of a fruit pulp extract by GC-GC-MS using a chiral column in the second
323 chromatographic dimension revealed the presence of a racemic mixture in durian. This suggested
324 a nonenzymatic formation from acetaldehyde, hydrogen sulfide and ethanethiol in the fruit pulp
325 and retrospectively justified OAV calculation based on the odor threshold of the racemate.

326 A key role of ethyl (2*S*)-2-methylbutanoate (**5**) for the fruity odor note of Monthong durian
327 pulp has already been suggested from its high FD factor obtained by AEDA.¹ Ethyl 2-
328 methylbutanoate has widely been reported as a major volatile in the pulp of different durian
329 varieties,¹⁷⁻²⁸ although in some varieties e.g. ethyl propanoate was more abundant.^{18, 24, 25} A
330 crucial impact of ethyl 2-methylbutanoate for durian odor has been claimed before such as by
331 Baldry et al.¹⁷, Weenen et al.,¹⁹ and Voon et al.²³ Ethanethiol (**17**) has also for a long time been
332 known as durian volatile,^{17, 18, 21, 22, 25} however, its high odor potency was yet not appropriately
333 recognized. 1-(Ethylsulfanyl)ethanethiol (**9**) has been reported from durian only by Wong and
334 Tie,²⁰ who assumed that the sum of sulfur compounds including 1-(ethylsulfanyl)ethanethiol
335 account for the oniony note in durian odor, but did not assess the impact of individual
336 compounds. Other authors suggested rather propane-1-thiol (**20**),¹⁷ 3,5-dimethyl-1,2,4-
337 trithiolane,¹⁹ or methyl propyl disulfide²² as crucial component for the oniony odor note in

338 durians. Näf and Velluz²⁰ reported a series of dithioacetals from durian, but did not find
339 dithiohemiacetals, which, according to our results,¹ are the more potent odorants. The workup
340 procedures used might account for that. Näf and Velluz applied simultaneous distillation-
341 extraction, whereas Wong and Tie²⁰ applied a rather mild volatile isolation technique, which was
342 similar to our approach and was based on a vacuum distillation at moderate temperatures of 28-
343 30 °C.

344 Apart from ethyl (2*S*)-2-methylbutanoate, ethanethiol, and 1-(ethylsulfanyl)ethanethiol, high
345 OAVs in the range of 330 to 45000 were also calculated for 13 further compounds (**18**, **2**, **8**, **14**,
346 **3**, **20**, **16**, **15**, **10**, **13**, **1**, **6**, and **19**) (Table 3). 2(5)-ethyl-4-hydroxy-5(2)-methylfuran-3(2*H*)-one
347 (**7**), 3-hydroxy-4,5-dimethylfuran-2(5*H*)-one (**11**), ethyl cinnamate (**4**), and 4-hydroxy-2,5-
348 dimethylfuran-3(2*H*)-one (**12**), on the other hand, exhibited comparatively low OAVs of 1 to 22,
349 although their FD factors obtained by AEDA were quite high (128 to 4096). The odor potency of
350 these substances might have been overestimated during AEDA, where compounds are assessed
351 completely vaporized. Their rather high boiling points and the good water solubility of the
352 furanones may substantially contribute to a low release from aqueous solution, which would
353 have resulted in rather low OAVs.

354 **Monthong Durian Odor Simulation.** To demonstrate that the 20 odor-active compounds
355 quantitated in this study included the key players in the overall olfactory profile of durian pulp,
356 an odor simulation model was prepared. The model was based on a buffered (pH 6.2) mixture of
357 97 % water and 3 % sunflower oil serving as an approximation of fresh durian pulp matrix and
358 included all 20 compounds in their natural concentrations as compiled in Table 2. The simulation
359 model exhibited a strong and characteristic durian odor and, when orthonasally compared to
360 fresh Monthong durian pulp in a QDA, showed a good agreement in the overall rating of major

361 odor descriptors. Both, the fruit material and the model, exhibited a dominating roasted onion-
362 like odor, followed by fruity, rotten onion-like, and rotten egg-like notes, whereas fresh, caramel-
363 like, and soup seasoning-like notes were rated less intense (Figure 4).

364 **Omission Tests.** The successful simulation of Monthong durian odor allowed for the
365 application of omission tests to study the contribution of individual odorants to the overall odor
366 profile. In an omission test, one or more compounds are omitted from the odor simulation model
367 and this incomplete model is then compared to the complete model in a triangle test. In case the
368 test results in a significant difference, a singly omitted compound has proven its odor impact.²⁹

369 In a first experiment (Table 4, test O1), the complete Monthong durian odor simulation
370 model (20 odorants, 1–20) was tested against a model from which all ten odorants with OAVs <
371 5000 (1, 4, 6, 7, 10–13, 15, 19) had been omitted. The resulting mixture of ten odorants (2, 3, 5,
372 8, 9, 14, 16–18, 20) could not significantly be distinguished from the complete model ($p \gg 5\%$),
373 indicating that the omitted odorants are of minor importance. Therefore, the model containing
374 only the 10 odorants with OAVs > 5000 was used as reference for the following tests. From this
375 model, the omission of single odorants was performed in ten separate experiments (Table 4, tests
376 O2–O11). None of the ten tests resulted in a significant difference between test and reference
377 sample and therefore were not helpful to rank the odor contribution of the individual odorants.
378 Obviously, omission of any single odorant from the model could be compensated by the other
379 nine compounds. A similar observation was recently reported from a mango aroma model.³⁰ In
380 the case of durian, the compensating effect is very plausible. First, because the ten odorants
381 basically exhibited only two types of odor qualities, fruity and sulfury-oniony, and second,
382 because the concentrations of some odorants were so far above their odor threshold values that
383 the corresponding olfactory receptors might already act beyond their saturation level. In this

384 case, e.g. the omission of ethyl (2*S*)-2-methylbutanoate (**5**), although exhibiting an OAV as high
385 as 1700000, might be fully compensated by ethyl 2-methylpropanoate (**8**; OAV 22000), methyl
386 (2*S*)-2-methylbutanoate (**14**; OAV 13000), and ethyl butanoate (**3**; OAV 11000), which in
387 combination would be able to saturate the odor receptor for fruitiness. In the same way, the
388 omission of oniony smelling ethanethiol (**17**; OAV 480000) could be compensated by 1-
389 (ethylsulfanyl)ethanethiol (**9**; OAV 250000) and vice versa. Following this idea further, it
390 seemed to be possible that the typical durian odor could possibly be evoked by the combination
391 of only two potent compounds, a fruity smelling one and an oniony smelling one. This
392 hypothesis was tested in omission experiment O12 (Table 4). A model only containing the
393 odorants ethyl (2*S*)-2-methylbutanoate (**5**) and 1-(ethylsulfanyl)ethanethiol (**9**) was tested against
394 the reference model of ten compounds. The result indicated no significant difference. Thus, the
395 typical durian pulp odor could be evoked by a mixture of only two odor-active compounds
396 present in their natural concentrations. In contrast, when ethyl (2*S*)-2-methylbutanoate (**5**) was
397 additionally omitted, thus 1-(ethylsulfanyl)ethanethiol (**9**) alone was tested against the reference
398 model of ten compounds, the result was a clear sensory difference (Table 4, test O13). Although
399 the oniony note is clearly dominating durian pulp odor, the fruity note is obviously of vital
400 importance, too. Similar binary mixtures such as a mixture of ethyl (2*S*)-2-methylbutanoate (**5**)
401 and ethanethiol (**17**) in the respective natural concentrations were also able to evoke a smell
402 clearly reminiscent of durian pulp (data not shown).

403 In summary, the molecular sensory science approach including screening of odor-active
404 compounds by AEDA and static headspace GC-O, structural assignment of the most odor-active
405 compounds, their exact quantitation, calculation of OAVs, and finally the execution of omission
406 tests revealed a surprisingly small number of only two key compounds, namely ethyl (2*S*)-2-

407 methylbutanoate and 1-(ethylsulfanyl)ethanethiol, being sufficient to evoke the characteristic
408 smell of durian pulp.

409 **Associated Content**

410 **Supporting Information**

411 MS-CI spectra of synthesized isotopically substituted odorants (PDF)

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416 **Notes**

417 The authors declare no competing financial interest.

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420 **Abbreviations Used**

421 AEDA, aroma extract dilution analysis; Boc, *tert*-butyloxycarbonyl; CI, chemical ionization; EI,
422 electron ionization; FD factor, flavor dilution factor; FFAP, free fatty acid phase; FID, flame
423 ionization detector; GC, gas chromatography; MS, mass spectrometry; GC-O, gas
424 chromatography–olfactometry; GC-GC-MS, two-dimensional heart-cut gas
425 chromatography–mass spectrometry; MCSS, moving column stream switching system; NAD,
426 nicotinamide adenine dinucleotide; OAV, odor activity value; QDA, quantitative descriptive
427 analysis; SAFE, solvent-assisted flavor evaporation; SIDA, stable isotope dilution assay.

428 **Nomenclature**

429 Dithiohemiacetal, 1-(alkylsulfanyl)alkane-1-thiol; diethyl trisulfide, diethyltrisulfane; ethyl

430 cinnamate, ethyl (2*E*)-3-phenylprop-2-enoate; 2(5)-ethyl-4-hydroxy-5(2)-methylfuran-3(2*H*)-
431 one, also called homofuraneol, tautomeric mixture of 2-ethyl-4-hydroxy-5-methylfuran-3(2*H*)-
432 one and 5-ethyl-4-hydroxy-2-methylfuran-3(2*H*)-one; 2(5)-ethyl-4-hydroxy-5(2)-
433 (¹³C)methyl(2(5)-¹³C)furan-3(2*H*)-one, tautomeric mixture of 2-ethyl-4-hydroxy-5-
434 (¹³C)methyl(2-¹³C)furan-3(2*H*)-one and 5-ethyl-4-hydroxy-2-(¹³C)methyl(5-¹³C)furan-3(2*H*)-
435 one; 3-hydroxy-4,5-dimethylfuran-2(5*H*)-one, also known as sotolon; 4-hydroxy-2,5-
436 dimethylfuran-3(2*H*)-one, also known as Furaneol[®]; 4-hydroxy-2(5)-methyl-5(2)-
437 (¹³C)methyl(5(2)-¹³C)furan-3(2*H*)-one, tautomeric mixture of 4-hydroxy-2-methyl-5-
438 (¹³C)methyl(5-¹³C)furan-3(2*H*)-one and 4-hydroxy-5-methyl-2-(¹³C)methyl(2-¹³C)furan-3(2*H*)-
439 one.

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529 **Figure Captions.**

530 **Figure 1.** Synthesis of ($^2\text{H}_{10}$)diethyl trisulfide based on the approach reported earlier for the
531 isotopically unmodified compound.⁷

532 **Figure 2.** Synthesis of 2(5)-ethyl-4-hydroxy-5(2)-(^{13}C)methyl(2(5)- ^{13}C)furan-3(2*H*)-one based
533 on the approach reported earlier for the deuterated compound.⁴ Dots indicate the positions of ^{13}C
534 atoms.

535 **Figure 3.** Structures of the stable isotopically substituted durian odorants used as internal
536 standards in the stable isotope dilution assays. Dots indicate the positions of ^{13}C atoms.

537 **Figure 4.** Aroma profile of the Monthong durian aroma model (20 compounds, $\text{OAV} \geq 1$) in
538 comparison with the aroma profile of fresh pulp obtained from ripe Monthong durians. Panelists
539 rated the intensity of each descriptor orthonasally on a scale from 0 to 3 with 0.5 increments and
540 with 0 = not detectable, 1 = weak, 2 = moderate, and 3 = strong.

Table 1. Isotopically Substituted Compounds and Quantitation Parameters Used in the Stable Isotope Dilution Assays

target analyte	isotopically substituted internal standard	quantifier ion (<i>m/z</i>)		calibration line ^a
		analyte	standard	
diethyl trisulfide (1)	(² H ₁₀)diethyl trisulfide	155	165	y = 0.98 x - 0.02
ethane-1,1-dithiol (2)	(¹³ C ₂)ethane-1,1-dithiol	61	63	y = 0.88 x + 0.07
ethyl butanoate (3)	(2,2,2- ² H ₃)ethyl butanoate	117	120	y = 1.00 x - 0.02
ethyl cinnamate (4)	(2,2,2- ² H ₃)ethyl cinnamate	177	180	y = 0.90 x + 0.01
ethyl (2 <i>S</i>)-2-methylbutanoate (5)	(² H ₅)ethyl 2-methylbutanoate	131	136	y = 0.94 x + 0.02
1-(ethylsulfanyl)-1-(ethylsulfanyl)ethane (6)	1-(ethylsulfanyl)-1-(ethylsulfanyl)(¹³ C ₂)ethane	121	123	y = 0.94 x + 0.11
2(5)-ethyl-4-hydroxy-5(2)-methylfuran-3(2 <i>H</i>)-one (7)	2(5)-ethyl-4-hydroxy-5(2)-(¹³ C)methyl(5(2)- ¹³ C)furan-3(2 <i>H</i>)-one	143	145	y = 0.94 x + 0.02
ethyl 2-methylpropanoate (8)	(2,2,2- ² H ₃)ethyl 2-methylpropanoate	117	120	y = 0.93 x + 0.03
1-(ethylsulfanyl)ethane-1-thiol (9)	1-(ethylsulfanyl)(¹³ C ₂)ethane-1-thiol	89	91	y = 1.10 x + 0.00
1-(ethylsulfanyl)propane-1-thiol (10)	1-(ethylsulfanyl)(2,2,3,3,3- ⁵ H ₅)propane-1-thiol	75	80	y = 0.99 x - 0.20
3-hydroxy-4,5-dimethylfuran-2(5 <i>H</i>)-one (11)	3-hydroxy-4-methyl-5-(¹³ C)methyl(5- ¹³ C)furan-2(5 <i>H</i>)-one	129	131	y = 0.98 x - 0.02
4-hydroxy-2,5-dimethylfuran-3(2 <i>H</i>)-one (12)	4-hydroxy-2(5)-methyl-5(2)-(¹³ C)methyl(5(2)- ¹³ C)furan-3(2 <i>H</i>)-one	129	131	y = 1.03 x - 0.01
3-methylbut-2-ene-1-thiol (13)	3-(² H ₃)methyl(4,4,4- ² H ₃)but-2-ene-1-thiol	69	75	y = 0.98 x - 0.20
methyl (2 <i>S</i>)-2-methylbutanoate (14)	(² H ₃)methyl 2-methylbutanoate	117	120	y = 1.02 x - 0.02
1-(methylsulfanyl)ethanethiol (15)	1-(methylsulfanyl)(¹³ C ₂)ethane-1-thiol	75	77	y = 0.97 x + 0.05

^ay = peak area standard / peak area analyte; x = concentration standard (μg/mL) / concentration analyte (μg/mL).

Table 2. Concentrations of Major Odor-Active Compounds in Monthong Durian Pulp

odorant	conc ^a ($\mu\text{g}/\text{kg}$)	CV ^b (%)
acetaldehyde (16)	98400	18
ethanethiol (17)	45400 ^c	20 ^c
methanethiol (18)	26700 ^c	13 ^c
ethyl (2 <i>S</i>)-2-methylbutanoate (5)	13800	12
ethyl butanoate (3)	8380	8
hydrogen sulfide (19)	3340 ^c	6 ^c
propane-1-thiol (20)	3070 ^c	22 ^c
ethane-1,1-dithiol (2)	2940	16
ethyl 2-methylpropanoate (8)	2000	8
methyl (2 <i>S</i>)-2-methylbutanoate (14)	602	6
1-(ethylsulfanyl)ethanethiol (9)	403	34
2(5)-ethyl-4-hydroxy-5(2)-methylfuran-3(2 <i>H</i>)-one (7)	376	32
diethyl trisulfide (1)	196	46
1-(methylsulfanyl)ethanethiol (15)	176	9
4-hydroxy-2,5-dimethylfuran-3(2 <i>H</i>)-one (12)	51	30
1-(ethyldisulfanyl)-1-(ethylsulfanyl)ethane (6)	16	34
3-hydroxy-4,5-dimethylfuran-2(5 <i>H</i>)-one (11)	13	25
1-(ethylsulfanyl)propane-1-thiol (10)	7.7	20
ethyl cinnamate (4)	4.5	20
3-methylbut-2-ene-1-thiol (13)	0.9	37

^aMean of triplicates. ^bCoefficient of variation; calculated by dividing the standard deviation by the mean value. ^cData taken from a previous publication.²

Table 3. Odor Thresholds and OAVs of Major Odor-Active Compounds in Monthong Durian Pulp

odorant	odor quality	odor threshold ^a ($\mu\text{g}/\text{kg}$)	OAV ^b
ethyl (2 <i>S</i>)-2-methylbutanoate (5)	fruity	0.0080	1700000
ethanethiol (17)	rotten onion	0.095 ^c	480000 ^c
1-(ethylsulfanyl)ethanethiol (9)	roasted onion	0.0016	250000
methanethiol (18)	rotten, cabbage	0.59 ^c	45000 ^c
ethane-1,1-dithiol (2)	sulfury, durian	0.13	23000
ethyl 2-methylpropanoate (8)	fruity	0.089	22000
methyl (2 <i>S</i>)-2-methylbutanoate (14)	fruity	0.048	13000
ethyl butanoate (3)	fruity	0.76	11000
propane-1-thiol (20)	rotten, durian	0.49 ^c	6200 ^c
acetaldehyde (16)	fresh, fruity	16	6100
1-(methylsulfanyl)ethanethiol (15)	roasted onion	0.038	4600
1-(ethylsulfanyl)propane-1-thiol (10)	roasted onion	0.0063	1200
3-methylbut-2-ene-1-thiol (13)	skunky	0.00076	1200
diethyl trisulfide (1)	fried shallot	0.20	980
1-(ethylsulfanyl)-1-(ethylsulfanyl)ethane (6)	sulfury, onion	0.018	890
hydrogen sulfide (19)	rotten egg	10 ^c	330 ^c
2(5)-ethyl-4-hydroxy-5(2)-methylfuran-3(2 <i>H</i>)-one (7)	caramel	17	22
3-hydroxy-4,5-dimethylfuran-2(5 <i>H</i>)-one (11)	soup seasoning	1.7	7
ethyl cinnamate (4)	honey	1.7	2
4-hydroxy-2,5-dimethylfuran-3(2 <i>H</i>)-one (12)	caramel	54	1

^aOrthonasal odor threshold in water. ^bOdor activity value, ratio of concentration (cf. Table 2) to odor threshold. ^cData taken from a previous publication.²

Table 4. Omission Tests Applied to the Monthong Durian Aroma Model

test	odorant(s) omitted ^a	reference model ^a	correct answers/ panelists ^a	<i>p</i> value (%)	level of significance ^b
O1	10 odorants, OAV < 5000	20 odorants, OAV ≥ 1	6/18	59	–
O2	ethyl (2 <i>S</i>)-2-methylbutanoate (5)	10 odorants, OAV > 5000	8/18	22	–
O3	ethanethiol (17)	10 odorants, OAV > 5000	9/18	11	–
O4	1-(ethylsulfanyl)ethanethiol (9)	10 odorants, OAV > 5000	9/18	11	–
O5	methanethiol (18)	10 odorants, OAV > 5000	7/18	39	–
O6	ethane-1,1-dithiol (2)	10 odorants, OAV > 5000	8/18	22	–
O7	ethyl 2-methylpropanoate (8)	10 odorants, OAV > 5000	6/18	59	–
O8	methyl (2 <i>S</i>)-2-methylbutanoate (14)	10 odorants, OAV > 5000	7/18	39	–
O9	ethyl butanoate (3)	10 odorants, OAV > 5000	5/18	77	–
O10	propane-1-thiol (20)	10 odorants, OAV > 5000	8/18	22	–
O11	acetaldehyde (16)	10 odorants, OAV > 5000	7/18	39	–
O12	all except 5 and 9	10 odorants, OAV > 5000	9/19	15	–
O13	all except 9	10 odorants, OAV > 5000	17/19	< 0.1	***

^aNumber of correct answers resulting from the triangle test and total number of panelist participating. ^b–, not significant ($p > 5\%$); *, significant ($5\% \geq p > 1\%$); **, highly significant ($1\% \geq p > 0.1\%$); ***, very highly significant ($p \leq 0.1\%$).

Figure 1.

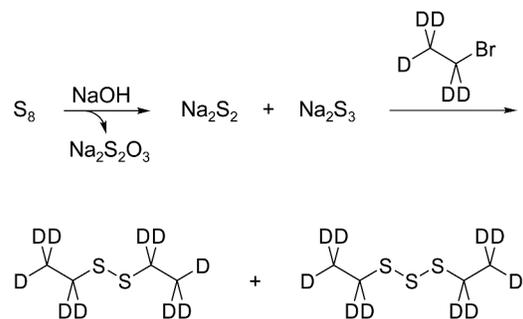


Figure 2.

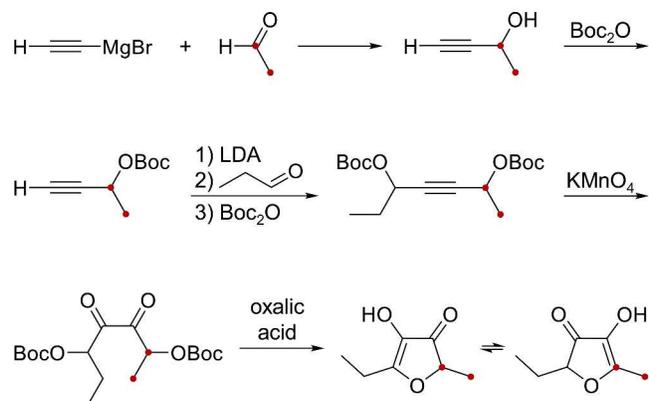


Figure 3.

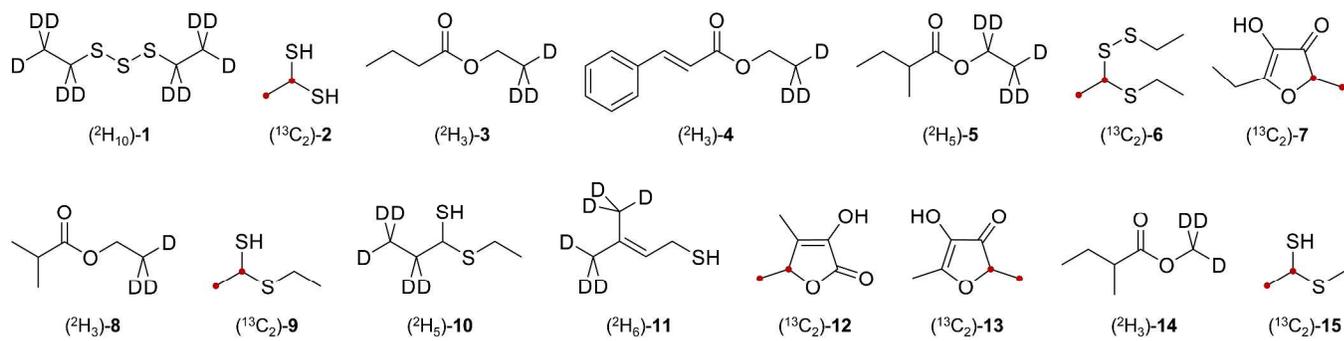
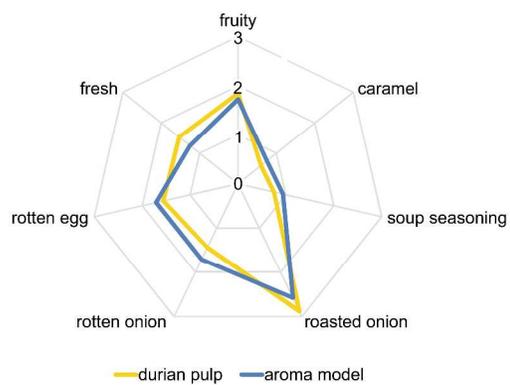


Figure 4.



TOC graphic.

