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Subject: Consumer Panel Estimates of the Odor Threshold Concentration, Odor Recognition Concentration and Odor Objection Concentration for Crude 4-methylcyclohexanemethanol in Water

EXECUTIVE SUMMARY

On January 9, 2014, "Crude" 4-methylcyclohexanemethanol (MCHM) spilled into the Elk River in West Virginia, which contaminated the water supply treated by West Virginia American Water and resulted in licorice odor complaints by residents. A team of experts was hired to understand the odor characteristics of the spilled chemical. The team developed a methodology based on ASTM Method E679-04 (2011) to estimate the Odor Threshold Concentration (OTC), Odor Recognition Concentration (ORC) and Odor Objection Concentration (OOC) for Crude MCHM in water during a single panel session. An Expert Panel used the methodology and estimated these thresholds.¹ The same methodology was used in this study to estimate these thresholds using an untrained Consumer Panel.

Two qualifiers should be attached to the findings of this report:

- 1. Sixty consumer panelists with equal gender distribution were used in the study. The panelists were not a statistically representative sample of consumers from the area served by West Virginia American Water.
- 2. No chlorine was in the water samples assessed by the panel

Table ES-1 summarizes the estimated OTC, ORC and OOC concentrations that were determined by the Consumer Panel and compares them to the values determined in the Expert Panel study. The Consumer Panel study showed that panelists were able to detect this compound at a concentration in water (0.55 ppb) at least as low as the most sensitive analytical method available to date (0.5 ppb).

Results	Expert Panel Geometric Mean, ppb	Consumer Panel Geometric Mean, ppb
Number of Panelists	9	60
Odor Threshold Concentration (OTC)	less than 0.15	0.55
Odor Recognition Concentration (ORC)	2.2	7.4
Odor Objection Concentration (OOC) Based on Degree of Liking	4.0	7.7
Odor Objection Concentration (OOC) Based on Objection/Complaint	4.0	9.5

Table ES-1. Comparison of OTC, ORC and OOC Values for Expert and Consumer Panels

The estimated thresholds determined in the Consumer Panel study support consumer observations in Charleston, WV that people recognized and objected to the licorice odor caused by Crude MCHM in their drinking water even in the presence of high concentrations of chlorine and even though the analytical reports were showing non-detect at a minimum reporting level of 10 ppb.

INTRODUCTION

On January 9, 2014, approximately 10,000 gallons of "Crude" 4-methylcyclohexanemethanol (MCHM) spilled into the Elk River from the property of Freedom Industries a short distance above the drinking water intake of the West Virginia American Water (WVAW) water treatment plant. Shortly after the spill began, consumers located in the area served by WVAW (Charleston, WV and environs) began complaining of a licorice odor in their drinking water. On February 9, an expert team was hired to help the state of West Virginia understand the odor characteristics of the spilled chemical and the reactions of the customers served by WVAW.

It was urgent that the odor characteristics of the chemical be understood in a scientific context in a short period of time. Therefore, an Expert Panel was convened within 15 days, which estimated the OTC, ORC and OOC values for Crude MCHM. The Expert Panel results were used to devise the concentration range for the Consumer Panel study that was held two weeks later.

The objectives of the work described in this technical memorandum were to:

1. Apply the Expert Panel methodology to a Consumer Panel study that would estimate the Odor Threshold Concentration (OTC), Odor Recognition Concentration (ORC) and Odor Objection Concentration (OOC) for the licorice-smelling substance in water.

- 2. Use the sample spiking methodology developed with Eurofins Laboratory to prepare samples of the licorice-smelling substance in a reference water for presentation to a Consumer Panel.
- 3. Understand how the Expert and Consumer Panel results could be used to explain consumer observations in Charleston, WV where people smelled a licorice odor in their drinking water immediately after the spill and for many weeks following the spill even after repeated system flushing.
- 4. Make recommendations for additional work to supplement and confirm the Consumer Panel findings.

DEFINITIONS OF DETECTION (THRESHOLD), RECOGNITION AND OBJECTION CONCENTRATIONS

Understanding how consumers react to off-odors in their drinking water is a complex problem that presents a unique set of challenges. To understand this phenomenon, it is important to appreciate the distinction between detectable odors and the concepts of recognizable and objectionable odors. Peer-reviewed scientific literature has recognized the concepts of detection, recognition and objection in drinking water and other substances.^{2, 3, 4} Table 1 organizes the concentrations of odorants in drinking water into aesthetic response levels.

Odor Response	Description	Aesthetic Response Levels
Detection (Threshold)	Chemical concentration usually determined in a laboratory setting where approximately 50% of the panelists can just detect the odor of a chemical	Odor threshold concentration—OTC
Recognition	Concentration of a chemical where a fraction of panelists (defined in the method) can correctly recognize and describe the odor characteristics of the chemical	Odor recognition concentration—ORC
Objection/Complaint	Chemical concentration determined either in a laboratory or field setting that causes consumers to object to their water supply and to call and complain	Odor objection concentration—OOC

Table 1. Odor Response Levels for Concentrations of Chemicals in Water

The same principles in Table 1 apply to the sense of taste. For example, the taste thresholds for sodium chloride and calcium chloride are in the range of 200 to 300 mg/L.⁵ At or above the taste threshold, panelists can describe the "salty" taste resulting in

recognition. As the sodium chloride concentration is increased further, the salty taste becomes objectionable.

Concentrations of minerals (including sodium chloride) that are objectionable to consumers in actual drinking water distribution systems have been described by detailed surveys of households. Bruvold and Daniels found that total dissolved solids (TDS) concentrations above 450 mg/L resulted in a significant number of consumers to reject their water supply and to seek alternatives. This concentration is just below the Secondary Maximum Contaminant Level for TDS and is equivalent to the taste objection concentration for TDS.⁶

PANEL METHODOLOGY

Panel Recruitment

Panelists for this study had to meet the following criteria:

- Untrained consumers
- Between the ages of 18 and 65
- Balance of women and men (approximately 50:50)
- Pregnant women could not participate
- Non-smokers only
- Anyone with a history of severe asthma or sinus problems was excluded
- Anyone currently suffering from a cold, the flu or any upper-respiratory disease at the time of testing was excluded
- No eating or drinking anything but water for one hour prior to testing

The Atkins Research Group recruited the panelists for the Consumer Panel study. They randomly selected a group of people from their database of 85,000 respondents, targeting some of the selection criteria that were provided to them. They sent an email blast to the sample of potential panelists with several screening questions including smoking status and other factors. Based on the panelist responses to the email blast, the Atkins Research Group selected a short list of qualified respondents.

A week before the Consumer Panels were held, the Atkins Research Group sent an email to each respondent, invited the potential panelist to one of four specific sessions, and asked them to confirm their participation. As time for the panels drew closer, schedule conflicts arose and panelists dropped out. People on the short list were then contacted to fill in the needed places. For each panel of 15, a total of 18 panelists were invited to attend to cover no-shows and last minute attendance problems. Sixty consumer panelists participated in the study. Four Consumer Panel sessions were held at 5:30 and 7:30 pm on Monday, March 3 and Wednesday, March 5 at the Atkins Research Group facility at 4929 Wilshire Boulevard, Los Angeles, California.

The gender split for the Consumer Panel was 50:50, 30 females and 30 males. Figure 1 shows the distribution of ages of the panel. Most of the panelists (67%) were in the middle age range of 30 to 53.

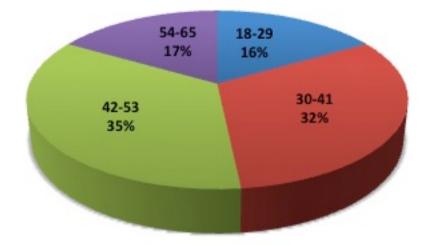


Figure 1. Age Distribution of Consumer Panel Threshold Study

The Consumer Panel was held in a market research room at the Atkins facility. There were no fugitive odors in the room that interfered with odor detection. On March 3, dividers separated the panelists to promote privacy—see Figure 2. Two moderators were in the room at all times and the panelists kept their worked covered with a sheet of paper assuring privacy and independent analysis. The head moderator decided that the dividers were not necessary for the March 5 panels.



Figure 2. Consumer Testing Facilities

Source of Crude MCHM

A 100 mL sample of the same Crude MCHM that spilled into the Elk River was collected by SGT Brian Spotloe and SGT Charles Cook of the West Virginia Army National Guard on February 12 and shipped to the Los Angeles area the next day. The sample came from Tank SV35927LM at the Poca Blending Facility. The contents of that tank were transferred from the leaking tank on the property of Freedom Industries sometime after the leak was discovered. A subsample of this sample was shipped to Eurofins Laboratory for spiking purposes.

Odor Assessment of Chemical to Spike into Water for Threshold Determinations

I assessed the odor characteristics of Crude MCHM and a pure standard of MCHM obtained from the chemical supply company TCI America. The Crude MCHM had a licorice odor that was penetrating, irritating and sharp. The pure MCHM had a definite licorice odor, but it was milder than the Crude. The MSDS form for Crude MCHM that accompanied the sample of the spilled tank contents showed that pure MCHM was the major component but other minor constituents were present. Figure 3 shows a chromatogram of Crude MCHM in methanol that was run on the Varian 450GC/220MS instrument in the UCLA laboratory showing MCHM and some of the tentatively identified minor constituents. We know from smelling a pure standard that dimethyl 1,4-cyclohexanedicarboxylate does have a licorice odor that is about the same characteristic and intensity as pure MCHM.

On March 13, 2014, several experts assessed the odor characteristic of a pure standard of cyclohexanemethanol (CHM), the first peak to elute on the chromatogram shown on Figure 3). The experts characterized the odor as penetrating, irritating, medicinal, green grass, sweet and pine. The CHM odor is definitely not as sweet as Crude or pure MCHM. Even though CHM is present at a much lower concentration than MCHM, it appears that CHM is contributing to the sharp characteristic of the Crude MCHM odor that has been experienced in panel studies and by the consumers of water in the Charleston area. More work is needed with difficult-to-obtain pure standards before the contributions of all of the minor components of Crude MCHM to the overall odor can be stated with confidence.

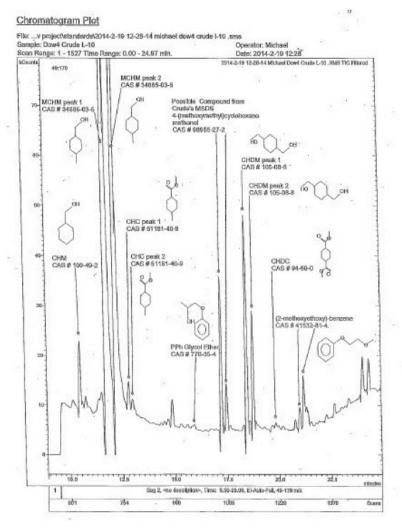


Figure 3. Chromatogram of Crude MCHM Showing Minor Components

To be certain that the Consumer Panel was presented with the same odor characteristics as experienced in the WVAW distribution system, Crude MCHM was spiked into the water that was presented to the consumer panelists. An odor assessment of propylene glycol phenyl ether (PPH), which was listed as one of the minor components of Crude MCHM, showed that PPH does not have a licorice odor.⁷

Selection of Matrix Water

We could not use treated water from the Elk River as the water for our tests because of the obvious problem that we did not know if the licorice problem was really absent from that source. Also, the goal was to conduct the threshold studies on water without chlorine. Chlorine interference with odor thresholds is well established and the impact of chlorine on the odor characteristics of MCHM in water is the subject of future research.

For this study, a spring water was selected for the panel matrix water.^{8, 9, 10} Arrowhead spring water was chosen because it is widely available in Southern California where the Consumer Panel studies would take place. The odor quality of Arrowhead spring water is consistent, and off-odors in that product have not been reported.

Table 2 shows the inorganic quality of Arrowhead spring water compared to a sample of water taken from the WVAW water treatment plant on March 11, 2014. While the total dissolved solids concentration of Arrowhead spring water is higher, neither water is highly mineralized. None of the minerals in the Arrowhead or WVAW treatment plant water would mask or interfere with consumers detecting, recognizing or objecting to levels of Crude MCHM in their tap water.

Table 2. Inorganic Water Quality of Arrowhead Spring Water and a Water Sample from
the WVAW Treatment Plant

Parameter	Units	WVA Treatment Plant Effluent, March 11, 2014	Arrowhead Spring Water
рН	Std. Units	7.3	7.9
Total Dissolved Solids	mg/l	73	228
Specific Conductance	umhos/cm	157	453
Calcium	mg/l	12	50
Magnesium	mg/l	6	20
Potassium	mg/l	1.3	3.2
Sodium	mg/l	8	18
Chloride	mg/l	9	7
Nitrate-Nitrogen	mg/l	0.52	0.85
Sulfate	mg/l	34	23
Total Alkalinity	mg/l as CaCO3	16	195

Thirty-nine gallons of Arrowhead spring water were purchased directly from Arrowhead in 3-gallon containers, delivered to the Atkins Research Group facility and used as the blank water in the Consumer Panel threshold tests.

Preparation of Spiked Samples and Determination of Crude MCHM Concentrations

Eurofins Laboratory in Lancaster, PA prepared the spiked samples of Crude MCHM. Eurofins is using an MCHM analytical method with a method detection level (MDL) of 0.5 ppb and a method reporting level (MRL) of 1.0 ppb—the lowest MCHM concentrations currently being determined by any laboratory in the U.S. Concentrations in the spiked samples were based on spiking 100% crude MCHM. The laboratory measured total peak area for the *trans* and *cis* isomers of MCHM and used this marker to determine the recovery of spiked concentrations in water.

The following is a summary of the Eurofins MCHM analytical method: A water sample is serially extracted with methylene chloride. The resulting extract is reduced in volume and an aliquot injected into a gas chromatograph equipped with a mass spectrometer detector (GC/MS). The GC/MS analytical system is tuned and calibrated following the principles outlined in SW-846, Method 8270D. This includes tuning the system to decafluorotriphenylphosphine (DFTPP) relative mass abundance criteria and calibration using a minimum of five calibration points from 1 ppb to 60 ppb. The analytical system is tuned and the calibration responses are checked every 12 hours.

As a routine part of the extraction procedure, a method blank, a laboratory control sample (LCS) and an MRL LCS are extracted along with every group of field samples that are analyzed. A method blank that is free of target compounds and an LCS and MRL LCS with acceptable recoveries of the target compounds is required for an extraction batch to be considered acceptable.

Arrowhead spring water is not available in Lancaster, PA. Sixty-four liters of Arrowhead spring water were purchased in Southern California and shipped to the Lancaster facility. The matrix water was spiked with Crude MCHM at eight levels with concentrations ranging from 0.027 to 60 ppb. Subsamples of the spiked water were analyzed immediately using the Eurofins analytical method. Eurofins analyzed the top six concentrations. The bottom two concentrations (0.027 and 0.082 ppb) were so far below the MDL and MRL that no effort was made to detect them. The two low concentrations were assured by the results of the higher concentrations and careful dilution procedures used by Eurofins laboratory staff.

Six liters each of the eight levels of spiked samples were shipped to the Atkins Research Group facility for delivery on March 3. Two of the bottles were broken in transit. Eurofins shipped replacements overnight from the samples being held for later analysis.

On Thursday, March 6 after the Consumer Panels were complete, Eurofins analyzed surviving subsamples of the spiked matrix water. One of the spiked results was lost during the extraction procedure.

Panelist Procedures

Prior to conducting the Consumer Panels, individual panelists were taken into a separate room and told that they would be sniffing water that may have odors that were similar to a reference odor that I would be presenting to them. They were also told that they might not recognize any odors in the water samples that were anything like the reference odor. They were asked to carefully sniff a diluted sample of Crude MCHM and explain in their own words how they would describe the odor. Their descriptions of the reference odor were recorded on a form. To avoid prejudicing the consumer panelists, no mention was made of the relation of the odor to the chemical spill in West Virginia. Appendix A contains a script used to elicit reference odor responses from each of the panelists.

Each Consumer Panel only required 15 panelists. Because17-18 people were recruited for each panel, the moderator eliminated from the final panel people who were clearly anosmic (i.e., they could not smell anything in the dilute MCHM sample), people who had trouble describing the odor using reasonable descriptive terms and those who by their actions and attitude were not interested in participating.

When the 15 consumer panelists were assembled, the panel moderator read a script, which described the methods and procedures that they would use, see Appendix B. While the panel was underway, the moderator walked around the panel testing area asking for any questions or clarifications and observing whether or not the panelists were following instructions.

Odor Threshold Methodologies

The well-known methodology referred to as ASTM E679-04 (2011) was used to estimate the OTC for Crude MCHM.¹¹ The same method was used in 1999 to determine the OTC for methyl *tert*-butyl ether (the gasoline additive MTBE) using a Consumer Panel¹² and to determine four thresholds of Crude MCHM by an Expert Panel.¹³

For the Consumer Panel study, three ounces of spiked and blank water were placed in nine-ounce odor-free plastic cups and covered with watch glasses, see Figure 4. Each panelist was presented with three cups at a time. One of the cups contained the spiked sample and the other two cups contained blank water. The panelists were asked to pick up the cup and watch glass, gently swirl the water in the cup, lift the watch glass and sniff the headspace above the water replacing the watch glass when they were finished. The panelists were instructed to choose the cup containing the odor that was different from the other two.



Figure 4. Sample Presentation to Panelists

Even if the panelists could not tell the difference between the three cups, they had to choose one of them as different. They could re-sniff the cups if they wished. The panelists received the lowest concentration of spiked water first. Subsequent groups of three cups contained one spiked sample with increasing concentrations of Crude MCHM to a maximum of 60 ppb. They recorded their observations by circling the code of the different cup on the score sheet. Appendix C shows the score sheet used in the Consumer Panels.

Random numbers were used to code all of the cups. The location of the different cup containing the spiked sample was roughly split between the left, middle and right cup. This presentation and scoring methodology is generally referred to as a forced-choice triangle, ascending (concentration) series. Temperatures of the spiked and blank water in the cups during both nights of testing ranged from 19 to 21 degrees Celsius.

Next, panelists were asked to record on the score sheet what they thought the water in the different cup smelled like. They were told that they could use any terminology that described the characteristic of the odor in the different cup, or they could use the terms that they provided for the reference odor if they recognized it. If the water smelled like nothing (had no odor), the panelists could write "nothing."

The ASTM E679 technique recognizes the determination of an ORC as part of its methodology. "...*recognition threshold*—the lowest concentration of a substance in a medium relating to the lowest physical intensity at which a stimulus is recognized as determined by the best-estimate criterion."¹⁴ (Italics in original)

There is no generally accepted methodology for determining a level of objection to the odor of an organic compound in water. In this research, two methods were used to answer the question: When do we know that panelists object to something in the water they are testing?

The first approach used the widely accepted methodology¹⁵ of presenting a stimulus to a panelist and asking how much the panelist liked or disliked the stimulus using a hedonic scale for the panelist to score his/her judgments. The nine-point hedonic scale used for this work was taken from *Standard Methods*.¹⁶ Using the nine-point hedonic scale to estimate the OOC was first reported by Suffet, Leavey and colleagues for determining odor and flavor objection concentrations in conjunction with a study of ethyl *tert*-butyl ether (ETBE) in drinking water.^{17, 18, 19}

The panelists were asked to rate how much they liked or disliked the odor of the water in the different cup using the degree of liking scale shown in Appendix D. They recorded their rating of the odor in the different cup on the score sheet in the "Degree of Liking" column.

The second approach is based on water utility experience determining when the concentration of a substance in water has reached the objection level in a distribution system. When a significant number of consumers object to an odor in their drinking water, some of them will pick up the telephone and call to complain. Not all who object will complain, but it will be clear to water utility management when the telephone calls start rolling in that they have a problem.

Experiences at the Metropolitan Water District of Southern California with earthy-musty odor problems suggest that there is a clear "tipping point" (concentration) when consumers begin to complain. Earthy-musty odors are generally caused by blue-green algae producing two compounds: geosmin and 2-methylisoborneol (MIB). Published OTCs for these compounds vary, but they are generally around 4 parts per trillion (ppt). When 10 ppt of either compound (or both adding up to 10 ppt) is being served to consumers, some of them will definitely call and complain. For both of these compounds, 10 ppt is the Odor Objection Concentration. A number of water utilities have set 10 ppt as their treatment goal to avoid complaints. Other utilities that desire a more stringent goal have set 5 ppt for both geosmin and MIB.

It was suggested to the panelists that they might find some of the odors in the different cups objectionable. If the odor was objectionable and the panelist would complain to their water utility or bottled water company, they were instructed to answer "Yes" in the "Object/Complain?" column.

After the eighth sample set was completed, the moderator collected the score sheets and checked them to make sure that the panelists had followed all of the instructions properly and that all of the descriptions and scores were filled in. The data from the panels were entered into an Excel spreadsheet and the best estimate thresholds for individuals and the panel as a whole were determined using the geometric mean calculation specified in ASTM E679.

RESULTS AND DISCUSSION

Analytical Results of Spiked Samples

Table 3 shows the concentrations of Crude MCHM recovered and the percent recoveries for samples analyzed before and after the Consumer Panels were conducted. The data show good recoveries for spiked Crude MCHM concentrations (based on the sum of the *cis* and *trans* isomer peak areas for pure MCHM) above the method MRL of 1 ppb ranging from 90 to 116 percent (within the acceptable range of 80 to 120%). As expected, the one recovery below the MRL is outside the generally acceptable range. None of these results nor the results from the Expert Panel spiking²⁰ indicates that the spiked concentrations of Crude MCHM degraded over the holding period. These data do not indicate if any of the minor compounds in the Crude MCHM mixture are changing over time, because their peak areas were not quantified.

Spiked Crude MCHM,	Crude MCHM R	ecovered, ppb	Percent Crude MCHM Recovered			
ppb	Pre-Panel Analysis	e-Panel Analysis Post-Panel Analysis P		Post-Panel Analysis		
0.027	(2)	(2)				
0.082	(2)	(2)				
0.25	ND	(3)				
0.74	0.54	(3)	72%			
2.2	1.9	2.3	83%	102%		
6.7	7.0	7.4	104%	111%		
20	22	(4)	112%			
60	66	65	110%	109%		

Table 3. Spiked Recoveries of Crude MCHM by Eurofins Laboratory

Notes:

(1) Pre-Panel analyses were conducted on 2/26 & 27/14; Post-Panel analyses on 3/6/14

(2) Not analyzed because concentration too low for MCHM analytical method

(3) Not analyzed due to broken bottle replacement

(4) Lost during extraction

ND = Not detected; All results rounded to two significant digits

Odor Threshold Concentration

Appendix E shows the results of the OTC determination for the 60 consumer panelists. The estimate of the individual odor thresholds is calculated as the geometric mean of the concentration where the last incorrect cup was chosen and the next higher concentration where the correct cup was chosen from there upward. An incorrect cup choice is recorded on Appendix E as a "0" and a correct choice as a "+". Thus, the estimate of the OTC for Panelist 02 is the geometric mean of 2.2 and 6.7 or 3.8 ppb.

For the 14 sensitive panelists who correctly chose the different cup at all eight concentrations, the estimate of their individual OTC is the geometric mean of the lowest concentration presented (0.027 ppb) and the concentration at the next theoretical lower

step, which in this case would be 3.0 times lower or 0.0091 ppb. Thus, the estimate of the OTC for Panelist 01 is the geometric mean of 0.027 and 0.0091 or 0.016 ppb.

The calculated estimate of the OTC for Crude MCHM determined by the Consumer Panel is the geometric mean of the 60 individual geometric means, or 0.55 ppb. The Consumer Panel study showed that consumers are able to detect Crude MCHM in water at concentrations at least as low as the most sensitive analytical method available to date for MCHM. Most of the individual OTC concentrations were within the range of concentrations presented, 0.027 to 60 ppb. However, 14 of the 60 panelist responses correctly chose the different cup for all eight concentrations.

Figure 5 shows the cumulative percentage plot for the 60 OTC responses. Using a log concentration scale, the plot shows good agreement with a straight line, which is similar to findings for the 57-panelist-OTC results for MTBE.²¹

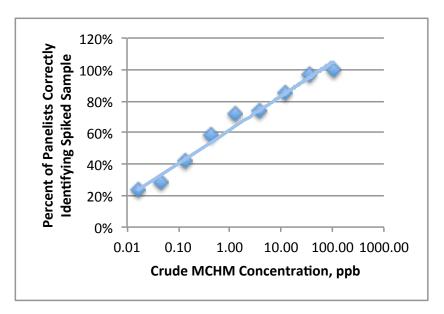


Figure 5. Cumulative Percentage Plot of Individual Odor Threshold Concentrations

The Expert Panel OTC for Crude MCHM was less than 0.15 ppb. It is not surprising that trained panelists are more sensitive to odors than untrained panelists. Nonetheless, the OTC of the Consumer Panel shows that the detection level is quite low when compared to other organic compounds.²²

Figure 6 shows the estimated OTC values for individual panelists plotted against panelist age. For this study, there did not appear to be any relationship between age and odor sensitivity over four orders of magnitude of the Crude MCHM concentration. Other studies have shown an age-OTC relationship.^{23, 24} However, Doty noted that the decrease in odor sensitivity was not severe below the age of 65.²⁵

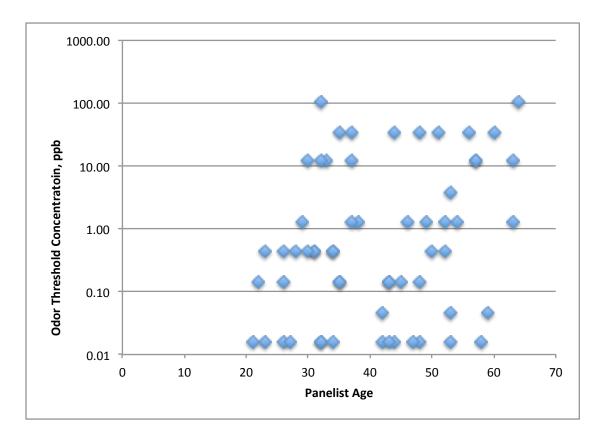
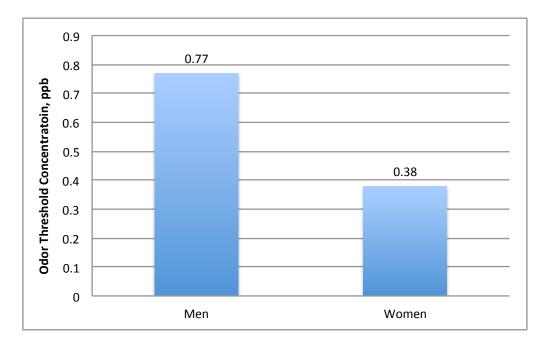
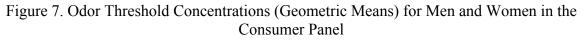


Figure 6. Plot of Consumer Panelist Age versus Estimated Individual OTC

Figure 7 indicates that OTC values for men and women on the consumer panel appeared to be different. A check of the gender OTC data sets showed that they were not normally distributed nor log normally distributed. Therefore, parametric statistics could not be used to check for differences. A nonparametric statistical test (Wilcoxon Rank Test) suggested that the two data sets were not statistically different. It appears that the variation in the data over four orders of magnitude make it difficult to determine differences as small as the one shown on Figure 7. Other studies have found inconsistent results comparing odor acuity comparisons between men and women.^{26, 27, 28, 29}





Odor Recognition Concentration

Appendix F shows the results of the ORC determination for the 60 consumer panelists. An ORC was only recorded for concentrations at or above the individual panelist's OTC. The best estimate of the individual panelist's ORC is the geometric mean of the two concentrations where there is a change from "other" descriptors to the reference odor descriptor and that change remains consistent to the highest concentration, which is noted with gray-shaded cells. For panelist 01 for example, the descriptor of "smelled fresh" at 0.74 ppb changed to "strawberry, fruity, familiar smell" at 2.2 ppb. The panelist's individual ORC is the geometric mean of those two concentrations, 1.3 ppb.

Two panelists were able to characterize the odor of the water in the different cup as their reference odor at the lowest concentration presented, 0.027 ppb. The estimate of their individual ORCs is the geometric mean of the lowest concentration presented (0.027 ppb) and the concentration at the theoretical next lower step, which in this case would be 3.0 times lower or 0.0091 ppb. Thus, the estimate of the ORC for Panelist 16 is the geometric mean of 0.027 and 0.0091 or 0.016 ppb.

For the panelists that did not describe their reference odor even at 60 ppb, the individual OOC was calculated as the geometric mean of 60 ppb and the theoretical next highest step, which in this case would be 3.0 times higher or 180 ppb. Therefore, the estimate of the OOC for panelist 02 is the geometric mean of 60 and 180 or 100 ppb.

Many of the panelists described their reference and descriptor odors using some variation of the term "sweet." Some judgment had to be applied to the many descriptors used by

the panelists to establish a continuum of odor descriptors up to the highest concentration of Crude MCHM presented. Appendix G lists the many sweet reference and descriptor odors used by the panelists.

The calculated estimate of the ORC for Crude MCHM determined by these panelists is the geometric mean of the 60 individual geometric means, or 7.4 ppb.

Odor Objection Concentration

As noted in the Panel Methodology section of this memorandum, two methods were used to estimate the OOC for Crude MCHM. Appendix H shows the results for the OOC determination based on the degree of liking scale. The OOC was only recorded for concentrations at or above the individual panelist's OTC. For this study, the best estimate of the panelist's individual OOC is the geometric mean of the two concentrations where there is a jump in the degree of liking score to 6 or above, which is noted by gray-shaded cells on Appendix H.

The previous studies that used the nine-point degree of liking scale chose the level 5 for objection and a level of 6 for rejection. It was not clear from those publications why two levels were chosen because a consumer who objects to an odor in water will most likely reject it. It was clear from their own data and the data from this study that the objection level in the nine-point degree of liking scale is 6. There was no need to determine an odor rejection concentration as was done in the other studies.³⁰

The same methods as described above for OTC and ORC were used to calculate the individual OOC levels when the panelist scored the lowest concentration of Crude MCHM as a 6 or the highest concentration as a number less than 6. Therefore, the estimate of the OOC for panelist 39 is the geometric mean of 0.027 and 0.0091 or 0.016 ppb. The estimate of the OOC for panelist 01 is the geometric mean of 60 and 180, or 100 ppb.

The calculated estimate of the OOC for Crude MCHM using the degree of liking scale is the geometric mean of the 60 individual geometric means, or 7.7 ppb.

Appendix I shows the results for the OOC determination based on objection/complaint. The OOC was only recorded for concentrations at or above the individual panelist's OTC. The best estimate of the panelist's individual OOC is the geometric mean of the two concentrations where there is a change to a consistent answer of "Yes" to the question: Would you object/complain about the odor in the different cup? The gray-shaded cells on Appendix I note the two concentrations used to calculate the individual geometric means.

The same methods as described above for OTC, ORC and OOC (Liking) were used to calculate the individual OOC levels when the panelist scored the lowest concentration of Crude MCHM as a "Yes" or the highest concentration as a "No."

The calculated estimate of the OOC for Crude MCHM using the objection/complaint criterion is the geometric mean of the 60 individual geometric means, or 9.5 ppb.

Limitations of the Methodology and Results

As with all research, there are limitations associated with this work that must be understood so that errors will not be made extrapolating the results to other applications.

- Sixty consumer panelists with equal gender distribution were used in the study. The panelists were not a statistically representative sample of consumers from the area served by West Virginia American Water.
- No chlorine was in the water samples assessed by the panel

A substantial number of the individual ORC and OOC concentrations were at the highest concentration presented to the panelists, 60 ppb. While it would have been preferable to have more individual ORC and OOC values in the middle of the concentration range presented, it appeared that the panelists were already having trouble describing the odor and deciding if they objected to the odor at 60 ppb. There was evidence that the panelists were becoming fatigued at the highest concentration presented. Raising the upper end of the odor concentration range presented to the panelists would have aggravated that problem.

Applicability of Consumer Panel Results to Understanding how Consumers Respond to Crude MCHM in Drinking Water

Table 4 summarizes the estimated OTC, ORC and OOC concentrations that were determined by the Consumer Panel. The Consumer Panel study showed that panelists were able to detect this compound at a concentration in water (0.55) at least as low as the most sensitive analytical method available today (0.5 ppb).

Odor Thresholds	Geometric Mean, ppb	Factor: Greater than OTC
Odor Threshold Concentration (OTC)	0.55	
Odor Recognition Concentration (ORC)	7.4	14
Odor Objection Concentration (OOC) Based on Degree of Liking	7.7	14
Odor Objection Concentration (OOC) Based on Objection/Complaint	9.5	17

Table 4. Summary of Consumer Panel Odor Threshold Estimates

The OTC is limited in its ability to predict how consumers assess odors in their tap water. The OTC is determined in a controlled environment with no masking odors like chlorine present in the water. The panelists were striving under laboratory conditions to detect odor differences between three cups at eight concentration levels. That situation is far different than taking a glass of water from a kitchen faucet.

ORC is a much better indicator than OTC for the point where consumers recognize an odor. The ORC level determined in this Consumer Panel study is higher than the OTC by a factor of 14. OOC levels are 14 and 17 times higher than the Consumer Panel's OTC. Peer-reviewed literature does not provide much guidance on how high or low factors like this should be.

Figure 8 shows the cumulative percentage plots for all of the thresholds determined in the Consumer Panel studies. As shown before on Figure 5, the OTC plot appears to be a straight line with the Crude MCHM concentrations presented on a log scale. The other three plots are not linear and are indicative of cumulative percentages plotted for higher threshold concentration levels.

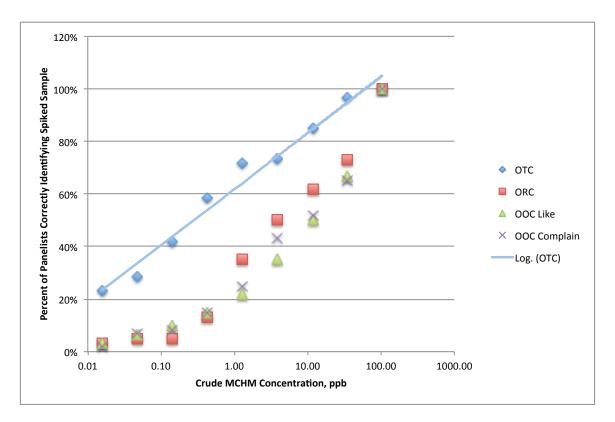


Figure 8. Cumulative Percentage Plots of Individual OTC, ORC and OOC Values

The ORC level of 7.4 and the OOC levels of 7.7 and 9.5 ppb are better values to use to gauge how consumers would respond to an odor event than the OTC. California used the taste objection concentration in 1999 when they set a 1 ppb secondary standard for Thiobencarb (a rice herbicide) that generated a bitter taste upon chlorination. Instead of using the Expert Panel findings they used the level of complaints from consumers objecting to the taste of the water.³¹

In the specific case of the Crude MCHM spill above the WVAW water intake, consumers would have been able to recognize and would have objected to concentrations of Crude MCHM in their tap water at low ppb concentrations (lower than those listed in Table 4) because they had become sensitized to it, they had the odor identified as licorice by the media and they had learned first hand how objectionable the licorice odor was when the first concentrations had been released into the water system at about 3,000 ppb.

Table 5 compares the OTC, ORC and OOC values for the Expert and Consumer Panels. While the Expert Panel determined lower values for all four thresholds, the actual thresholds that the consumers of WVAW tap water would have experienced during and after the spill were probably between the two sets of values. Once again, the consumers learned and became more sensitive to the detection, recognition and objection of concentrations of Crude MCHM because they had been subjected to it for weeks at concentration levels far above the concentrations presented on Table 5. It is clear from press reports that members of the public in Charleston and environs were able to recognize Crude MCHM in their tap water even with the presence of high concentrations of free chlorine, approximately 3.5 ppb (and below).

Results	Expert Panel Geometric Mean, ppb	Consumer Panel Geometric Mean, ppb
Number of Panelists	9	60
Odor Threshold Concentration (OTC)	less than 0.15	0.55
Odor Recognition Concentration (ORC)	2.2	7.4
Odor Objection Concentration (OOC) Based on Degree of Liking	4.0	7.7
Odor Objection Concentration (OOC) Based on Objection/Complaint	4.0	9.5

Table 5. Comparison of OTC, ORC and OOC Values for Expert and Consumer Panels

It is not appropriate to look at only a portion of the responses by individual panelists in this study and extrapolate their determinations to the public at large. We have no idea if the individual responses of these 60 consumer panelists represent responses by any segment of the Charleston population. However the collective responses (with qualifiers) can give us guidance to consumer responses.

The most important finding of this work can be stated succinctly. The estimated thresholds determined in the Consumer Panel study support consumer observations in Charleston, WV that people recognized and objected to the licorice odor caused by Crude MCHM in their drinking water even in the presence of high concentrations of chlorine and even though the analytical reports were showing non-detect at a minimum reporting level of 10 ppb.

The only appropriate use of the results of this work is to cite the geometric means of the data generated by the panelists, which resulted in estimates of the OTC, ORC and OOC concentrations. The fact that these composite numbers reflect the general experience of the consumers exposed to Crude MCHM contaminated tap water strengthens the appropriateness of this conclusion.

Not surprisingly, many people in Charleston did not use tap water even after the "Do Not Use" restriction was lifted. They also did not start using tap water after they were told that the concentration of MCHM was non-detect. They continued not using tap water because their sense of smell recognized it and objected to its presence. For many people, smelling an off-odor in tap water means that it is not safe for them to drink it.³²

SUMMARY AND CONCLUSIONS

Based on the assessments in this report, the following points can be concluded:

- 1. A methodology was used based on ASTM Method E679 to estimate the OTC, ORC and OOC concentrations for Crude MCHM in water during a single panel session. The methodology was tested using an Expert Panel and then applied to the Consumer Panel determinations.
- 2. Spiked concentrations of Crude MCHM were measured by a sensitive analytical method and found to be within acceptable percent recoveries.
- 3. The estimate of the Odor Threshold (Detection) Concentration for Crude MCHM in water determined by the Consumer Panel was 0.55 ppb. The Consumer Panel study showed that panelists were able to detect this compound at a concentration in water (0.55) at least as low as the most sensitive analytical method available to date (0.5 ppb).
- 4. The estimate of the Odor Recognition Concentration for Crude MCHM in water determined by the Consumer Panel was 7.4 ppb.
- 5. The estimates of the Odor Objection Concentrations for Crude MCHM in water determined by the Consumer Panel were 7.7 and 9.5 ppb when measured using the Degree of Liking and Objection/Complaint methods, respectively.
- 6. The estimated thresholds determined in the Consumer Panel study support consumer observations in Charleston, WV that people could recognize and object to the licorice odor caused by Crude MCHM in their drinking water even in the presence of high concentrations of chlorine and even though the analytical reports were showing non-detect at a minimum reporting level of 10 ppb.

RECOMMENDATIONS

As a result of the findings from this study, the following actions are recommended:

1. Investigate the impact of free chlorine residuals on the ability of consumers to detect, recognize and object to the licorice odor of Crude MCHM in drinking water.

2. Conduct oxidation studies of Crude MCHM with chlorine and potassium permanganate and determine if the odor characteristic or intensity of the licorice odor is changed after oxidation.

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Appendices

Appendix A

Script for Reference Odor Determination by Consumer Panelists

How are you? Thanks coming in. We're going to sniff water samples tonight. We will present water samples to you, have you sniff them and tell us in your own words what the water smells like. We are not looking for any particular answer. We are not digging for one way to describe the odors. There is certainly no right or wrong answer. We want to hear from you what the water smells like in your own words.

To begin with, I will present you with one sample and ask you to tell us in your own words what the sample smells like. (Moderator unscrews the cap of a small bottle containing a dilute solution of MCHM. Moderator sniffs the opening at the top of the bottle and then presents the bottle to the panelist.) Just lean forward and sniff.

So what does it smell like? (Panelist responds with odor descriptors. If the descriptors are not clear, the panelist is asked again how the sample smells. The panelist is given a second opportunity to sniff the contents of the bottle. All of the panelist's responses are written down on a form.)

(I then say the first name of the panelist and repeat the odor descriptors that he/she gave. For example:) Ok, Kevin. You said that the odor smells "sweet flowery." So, "Kevin's odor" is sweet flowery. That is your reference odor for this study. If you smell Kevin's odor in any of the samples we present to you, please call it sweet flowery. And if the odor doesn't then don't call it that. Please tell us in your own words what the samples smell like.

Thank you.

Appendix B

Procedure for Consumer Panel

Panelists check in. After they complete checkin, they are directed to another room. In that room, a person will hand them a cup of water and ask them to sniff it. After the panelist sniffs the example odor in the cup, the staff person will say, *"The odors in the water samples may contain substances that smell like what is in this bottle. Please describe in your own words what you think this smells like."* The panelist's response is marked on a score sheet by the staff person.

The panelist is then directed to the consumer panel testing room. Once all (15) of the panelists are seated, the panel session begins and the script is read.

Script for Consumer Panel

All 15 people are seated. In front of each person is:

- 1. Coding sheet
- 2. Pen (not a pencil)
- 3. Degree of Liking Scale
- 4. Cup of plain water (color of cup is different from the others)

Good evening. Thank you all for helping us out. Tonight we will be testing a compound that is sometimes found in drinking water. At very low concentrations some people find the aroma of the compound quite pleasant. At higher concentrations other people find it not so pleasant. Some people find nothing wrong with odor of the compound at all. We are trying to figure out how a large group of people in a controlled environment react to the odor of this compound.

We will present three cups of water to each of you 8 different times. For each cup, pick up the watch glass and cup just like this, swirl it gently, lift the watch glass and sniff the air above the water. Replace the watch glass. For each set of 3 cups, choose the cup that is different from the other two. Even if it is difficult for you to detect a difference in the three cups, you must select one cup that is different. If you want, you can re-sniff the cups. Circle the code on your score sheet representing the cup that is different. **Leader demonstrates.**

After you choose the cup that is different, write down the **Odor Description** representing the water in the different cup on the score sheet. Describe the smell of the water in your own words. If you smell the example odor that we presented to you in the other room, use that descriptor in the Odor Description blank. If the water in the cup smells like nothing, you can write "*nothing*."

Next, we want you to tell us how much you **like or dislike** the water with the odor in the cup that was different. Use the Degree of Liking Scale at your place.

Finally, some of the odors we are presenting to you may be objectionable. If the odor is **objectionable** and you would complain to your water utility or the bottled water company about it, please mark "Yes" in the Object/Complain column. Otherwise, mark "No."

Please remember that there are no "wrong" answers here. We are trying to understand how you perceive the water samples.

The plain water is available for you to use at any time during the panel session.

Let's begin with the first set of three cups. (Three cups are delivered to each panelist.)

Are there any questions?

Leader walks around the room answering questions and making sure that everyone is filling in the coding sheets as the session continues.

After finishing the 8th and last set of three cups, all the panelists stay in their seats. The leader collects the coding sheets and makes sure that they are all filled out. Then all the panelists can leave.

Appendix C

Sa	Imple Cup Cod	es	Office Use	Odor Description	Degree of Liking	Object/ Complain? Y or N
473	475	088				
298	332	649				
030	275	900				
874	503	301				
263	253	451				
547	152	636				
063	195	140				
827	841	607				

Score Sheet for Panelist Number _____

Date:_____

Start Time:_____

Instructions:

1. Circle the number of the sample cup that has a *different* odor from the other two cups.

2. Describe the odor in the cup that is different. Use your own words. If the odor is like the odor in the sample you smelled before the panel, use that descriptor.

3. Record how much you like or dislike the odor in the different cup using the Degree of Liking Scale provided.

4. Do you object to the odor in the different cup? Would you call your water utility or bottled water company to complain about the odor in the different cup?

Appendix D

Degree of Liking Scale

- 1. I would be very happy to accept this water as my everyday drinking water.
- 2. I would be happy to accept this water as my everyday drinking water.
- 3. I am sure that I could accept this water as my everyday drinking water.
- 4. I could accept this water as my everyday drinking water.
- 5. Maybe I could accept this water as my everyday drinking water.
- 6. I don't think that I could accept this water as my everyday drinking water.
- 7. I could not accept this water as my everyday drinking water.
- 8. I could never drink this water.
- 9. I can't stand this water in my mouth and I could never drink it.

Appendix E. Consumer Panel Results for Odor Threshold Concentration

			Cor	centrations	of Crude MCI	IM Presented	to Panelists,	ppb		Best Estimate Threshold, ppb
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Value
01	3/3/14	+	+	+	+	+	+	+	+	0.016
02	3/3/14	0	0	0	+	0	+	+	+	3.8
03	3/3/14	0	0	0	+	+	+	0	+	35
04	3/3/14	0	0	0	+	+	+	+	+	0.43
06	3/3/14	0	0	0	+	+	+	+	+	0.43
07	3/3/14	+	+	+	+	+	+	0	0	100
08	3/3/14	0	+	+	+	+	+	+	+	0.047
09	3/3/14	+	+	+	+	+	+	+	+	0.016
10	3/3/14	0	0	0	0	0	0	+	+	12
11	3/3/14	+	+	0	+	+	+	+	+	0.43
12	3/3/14	+	+	+	+	+	+	+	+	0.016
13	3/3/14	+	+	0	+	+	+	+	+	0.43
14	3/3/14	+	0	+	+	+	+	+	+	0.14
15	3/3/14	+	0	+	+	+	+	0	+	35
16	3/3/14	+	+	+	+	+	+	+	+	0.016
19	3/3/14	0	0	+	0	+	+	+	+	1.3
20	3/3/14	+	+	+	+	+	+	+	+	0.016
22	3/3/14	+	0	+	+	+	+	+	+	0.14
23	3/3/14	+	0	+	+	+	+	+	+	0.14
24	3/3/14	+	0	0	+	+	+	+	+	0.43
25	3/3/14	+	+	0	+	+	+	+	+	0.43
27	3/3/14	0	0	0	+	+	0	+	+	12
28	3/3/14	+	0	+	+	+	+	+	+	0.14
29	3/3/14	0	0	0	0	+	0	+	+	12
31	3/3/14	+	+	0	0	+	+	+	+	1.3
32	3/3/14	+	0	+	0	+	+	+	+	1.3
33	3/3/14	0	+	0	0	0	0	+	+	12
34	3/3/14	0	0	0	0	+	+	+	+	1.3
35	3/3/14	+	+	+	+	+	+	+	+	0.016
36	3/3/14	+	0	+	+	+	+	+	+	0.14
		•								
38	3/5/14	+	+	+	+	+	+	+	+	0.016
39	3/5/14	+	+	+	+	+	+	+	+	0.016
41	3/5/14	0	+	+	0	+	+	+	+	1.3
42	3/5/14	0	0	0	+	+	+	+	+	0.43
43	3/5/14	+	0	+	+	+	+	+	+	0.14
44	3/5/14	+	+	0	+	+	+	+	+	0.43
45	3/5/14	0	0	+	0	+	+	0	+	35
46	3/5/14	0	0	0	+	0	0	+	+	12
47	3/5/14	0	0	+	+	+	+	+	+	0.14
48	3/5/14	+	+	+	0	+	+	+	+	1.3
49	3/5/14	0	0	0	0	+	0	+	0	100
50	3/5/14	0	0	+	+	0	+	0	+	35
51	3/5/14	0	+	0	0	+	+	+	+	1.3
53	3/5/14	0	+	+	+	+	+	+	+	0.047
54	3/5/14	+	0	0	+	+	+	+	+	0.43
55	3/5/14	+	+	+	+	+	+	+	+	0.016
56	3/5/14	+	0	0	+	+	+	0	+	35
57	3/5/14	0	0	+	+	+	+	+	+	0.14
59	3/5/14	+	+	+	+	+	+	+	+	0.016
62	3/5/14	+	+	+	+	+	+	+	+	0.016
63	3/5/14	0	+	+	+	0	0	+	+	12

Appendix E. Consumer Panel Results for Odor Threshold Concentration

			Concentrations of Crude MCHM Presented to Panelists, ppb											
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Value				
64	3/5/14	+	+	+	+	0	+	0	+	35				
65	3/5/14	0	+	+	+	+	+	+	+	0.047				
66	3/5/14	+	+	0	0	+	+	+	+	1.3				
67	3/5/14	+	+	0	+	+	+	+	+	0.43				
68	3/5/14	+	+	+	+	+	+	+	+	0.016				
69	3/5/14	+	+	+	+	+	+	+	+	0.016				
70	3/5/14	+	0	0	0	0	0	+	+	12				
71	3/5/14	+	0	0	+	+	0	0	+	35				
72	3/5/14	+	+	+	+	+	+	+	+	0.016				

Note: "0" indicates that the panelist selected the wrong sample of the set of three; "+" indicates that the panelist selected the correct sample; the individual OTC is the geometric mean of the two concentrations where there is a change from "0" to consistent answers of "+" which is noted by gray-shaded cells.

Geometric Mean, ppb = 0.55

				Concentra	ations of Crude N	ICHM Presented 1	o Panelists, ppl	þ			Best Estimate Threshold, ppb
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Reference Odor	Value
1	3/3/14				Smelled fresh	strawberry, fruity, familiar smell	strawberry, fruity, familiar smell	strawberry, fruity, familiar smell (more like candy)	strawberry, fruity, familiar smell	strawberry, fruity	1.3
2	3/3/14								It's the one that smells different	perfumey, ocean breeze	100
3	3/3/14							Clear, clean, light, no odor	pepperminty, room freshner	floral, earthy, tan, minty	35
4	3/3/14					Nothing	Cut grass & vanilla	smells sweet like vanilla	cotton candy	sweet, vanilla, cut grass	3.8
6	3/3/14			chemicals	rubber, chemicals	Glue	Glue, chemicals	chemicals, glue, chlorine	chemicals, glue, rubber	licorice, chemical, not good	0.43
7	3/3/14								chemical smell	minty, basil	100
8	3/3/14				dewy	floral	sweet, fruity	sweet	sweet	fruity, flowery	1.3
9	3/3/14								plastic melted	Bubble bath, flowery	100
10	2/2/44						clean, fresh,	sweet, carbonated,	sweet syrupy, cola: reminds me of the one I smelled before		
10	3/3/14						pure	fruity	the panel	syrupy, coca cola rosewater, candy	12
11	3/3/14					antiseptic wipe,	plastic	jolly rancher artificial	jolly rancher oranges, mixed	(jolly rancher)	12
12	3/3/14 3/3/14			sharp, cutting	mold-like, grassy, pungent	cleaning agent chemical, metal, pungent	fruity gas-like, pungent, potent	grape, fruity chemical, dirty, potent	fruit, fruity nail polish remover, varnish	fruity, licorice medicine, pungent, rotten	<u>3.8</u> 0.43
14	3/3/14			clean, fresh, no smell	fruity	fruity	licorice	licorice, sweet	Jet fuel, medicine	medicine, fruity, perfumey	0.43
15	3/3/14	fairly sweet, tangy, moutain	citrus, dewy,		dull citrus, dull	mountain-dewy, monster energy	citrus, energy drink, almost lemony, jolly	a bit smoky	licorice citrus, lemon, lemonhead	licorice medicinal,	35
16	3/3/14	dew, citrus	fruity	citrus, lemony	candy	drink	rancher	citrus, Pinesol	candy	neutral, sweet	0.016
19	3/3/14				seltzer	strawberry	fresh produce section of grocery store	old cantaloupe	lime, strawberry	cherry cough syrup, sweet	1.3
20	3/3/14								hair spray	medicinal, kids cough syrup	100
22	3/3/14							chemical detergent	wild cherry (Ludens) cough drops	cherry cough drops (Ludens)	35

			1	Concentr	ations of Crude N	//CHM Presented	to Panelists, ppl	b			Best Estimate Threshold, ppb
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Reference Odor	Value
								Similar to aroma during	Interview aroma, flowers, bathroom air	Bathroom air	
23	3/3/14						Fresh fruity	interview	freshner	freshner, flowery	12
24	3/3/14						vinegar, cleaner smell	cherry coke	cherry cough medicine, Robitussin	spicy, herb, fruity	12
25	3/3/14						Nothing	fruity, sweet	fruity, sweet	fruity, sweet, natural	12
27	3/3/14							chemical smell	medicine	mediciney	35
28	3/3/14		Fairly basic smell	sweet	flowery	coca cola, sugar cane syrup	cola, sugar cane syrup, sugary, sweet	sugary sweet	sweet soda	floral, brown sugar	0.14
29	3/3/14								mint-like, protein drink	algae fish tank, medicine	100
31	3/3/14					musty, chemical	grape, fruity	citrusy	chemical and artificial	citrusy, floral	3.8
32	3/3/14				clean air	floral, potpourri	lemon dish soap (sweet fruity)	cherry, Kool Aid fruit punch	cherry, Kool Aid fruit punch	dark cherry (floral and fruity)	1.3
33	3/3/14								garden hose, dirt and rubber	pine nut sage cookie	100
34	3/3/14				smells off	cherry cough syrup	cherry cough syrup	cherry cough syrup	cherry cough syrup	cherry cough syrup	1.3
35	3/3/14				fresh smell	smelled like reference sample	like ref sample, sage/pine	medicine	household cleaner	fruity, sage	1.3
36	3/3/14								hair spray	vanilla, baking flavoring	100
38	3/5/14				gas from the oven	sweet like test smell, slight perfumey acetone	perfumey acetone	sweet nail polish remover	sweet like a jelly bean	sweet candy, jelly bean, vanilla, watermelon	1.3
	5, 5, 17				medicinal,	chemical,	chemical,	chemical,	chemical,	Flower Bomb (perfume brand floral, spicy,	1.5
39	3/5/14	medicinal	medicine	medicinal	chemical	medicinal	medicinal	medicinal	medicinal	fruity), almond	0.016
41	3/5/14					Rum	licorice	berry, sweet	cherry clean, fresh	spicy, sweet	3.8
42	3/5/14				chemical	sweet, juicy fruit	sweet and surgary	sweet, hard candy	sweet smell like before the panel- licorice	licorice	1.3
43	3/5/14								garbage, sewer water	Candle vanilla, lavender, waxy flower	100

			I		Best Estimate Threshold, ppb						
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Reference Odor	Value
44	3/5/14						chemical, jet fuel	perfumey, sweet, cloying	sweet, vanilla, sasparilla	tapioca, vanilla, sweet medicinal	12
45	3/5/14								7 Up	peppermint, pine (old tree)	100
46	3/5/14						Stephens flower paint thinner	perfumey flower	heavy plastic	flowery, terpentine, paint thinner	12
47	3/5/14			fruity	nutty	lemon (sweet fruity)	medicine like cherry Nyquil	almond	cherry, fruity	nutty, sweet, vanilla	0.43
48	3/5/14				hint of an actual aroma	citrus, sweetness	musky sweetness	sugary	perfumey, musty	floral, citrus	1.3
49	3/5/14								fruit	Sweet, fruity	100
50	3/5/14							sewage	sweet	medicinal, sweet candy	35
51	3/5/14					foul	minty chemical	watered down minty chemical	raspberry	minty chemical	3.8
53	3/5/14					dirty diaper, plastic	vinegar, plastic, fruity	ammonia, smog, vapor rub	sweet, chemical, ammonia	sweet cherry, menthol	3.8
54	3/5/14					cleaning solution	almond-like	almond-like	almond-like	almond extract	3.8
55	3/5/14							plastic, chemical	fruity, black licorice	fruity, black licorice	35
56	3/5/14								unpleasant	soapy, fruity, cough syrup	100
57	3/5/14								magic marker, gasoline	coconut, medicinal, nutty	100
59	3/5/14					nail polish	grape kids medicine	grape	mixed fruit Pedialyte	Nyquil medicine (licorice)	3.8
62	3/5/14							rubber glue	Blue Nyquil	Blue Nyquil (minty)	35
63	3/5/14								nothing	black licorice	100
64	3/5/14								water with no filtration	rose water	100
65	3/5/14				sewage	cleaning odor	cleaning agent	cleaning agent	cleaning agent	cleaning chemical	1.3

				Best Estimate Threshold, ppb							
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Reference Odor	Value
66	3/5/14				no odor	licorice	licorice	licorice	licorice	Playdough (candy)	1.3
67	3/5/14								nothing	cough syrup, mediciney, Robutussin	100
					pungent sulfur,	sweet, acidic,	organic,	sweet raspberry, flowery, lavender,		sweet, carmelized	
68	3/5/14				sour, egg-like	orange or lime	flowery	chemical	chemical	brown sugar	1.3
69	3/5/14					boiled eggs	perfume	bubblegum, vanilla	peanuts (nutty)	bubblegum, vanilla, flowery	3.8
70	3/5/14						orange	nail polish remover (fruity)	fruity, grape	Robutussin, grape, vanilla	12
71	3/5/14								melted plastic	black licorice	100
72	3/5/14				nothing	flower	alcohol	flower	flower/alcohol	jasmine, flowery, alcohol	1.3
					Ŭ Ŭ						

Notes: The ORC was only recorded for concentrations at or above the OTC; the individual ORC is the geometric mean of the two concentrations where there is a change from other descriptors to the reference odor descriptor which is noted by gray-shaded cells. Descriptors are not shown below individual ORC thresholds.

Geometric Mean, ppb = 7.4

Appendix G. Sweet Reference and Descriptor Odors for MCHM

Reference Odor	Descriptor
strawberry, fruity	strawberry, fruity, candy
sweet, vanilla, cut grass	cut grass, vanilla, cotton candy
fruity, flowery	floral, sweet, fruity
syrupy, coca cola	sweet, carbonated, fruity, syrupy
rosewater, candy, jolly rancher	jolly rancher
fruity, licorice	artificial grape, fruity, oranges, mixed fruit
medicine, fruity, perfumey	fruity, licorice, sweet, jet fuel, medicine
licorice	licorice
medicinal, neutral, sweet	citrus, dewy, fruity, lemony, dull candy, mountain dew, monster drink, citrus, Pinesol, lemonhead candy
	strawberry, fresh produce section of grocery store, old
cherry cough syrup, sweet	cantaloupe, lime, strawberry
cherry cough drops (Ludens)	wild cherry (Lundens) cough drops
spicy, herb, fruity fruity, sweet, natural	cherry coke, cherry cough medicine, Robutussin fruity, sweet
mediciney	like medicine
floral, brown sugar	sweet, flowery, coca cola, sugar cane syrup, sugary sweet, sweet soda
citrusy, floral	grape, fruity, citrusy, chemical and artificial
dark cherry (floral and fruity)	floral, potpourri, lemon dish soap, cherry, Kool Aid fruit punch
cherry cough syrup	cherry cough syrup
fruity, sage	like reference sample, sage/pine, medicine, household cleaner
	sweet like test smell, perfumey, acetone sweet nail polish
sweet candy, jelly bean, vanilla, watermelon	remover, sweet like a jelly bean
Floral Bomb perfume, floral, spicy, fruity, almond	medicine, chemical
spicy, sweet	licorice, berry, sweet, cherry
	sweet, juicy fruit, sweet, sugary, hard candy, fresh sweet smell
licorice	like before the panel, licorice
tapioca, vanilla, sweet medicinal	sweet, vanilla, perfumey, sweet, cloying, sasparilla
nutty, sweet, vanilla	nutty, lemon, medicine like cherry Nyquil, almond, cherry, fruity
floral, citrus	citrus sweetness, musky sweetiness, sugary, perfumey, musty
sweet, fruity	fruit
medicinal, sweet candy	sweet
	vinegar, plastic, fruity, ammonia, smog, vapor rub, sweet,
sweet cherry, menthol	chemical, ammonia
almond extract	almond-like
fruity, black licorice	fruity, black licorice
Nyquil medicine, licorice	grape kids medicine, grape, mixed fruit Pedialyte
Blue Nyquil, minty	Blue Nyquil
Playdough, candy	licorice
	sweet, acidic, orange, lime, organic, flowery, sweet raspberry,
sweet, carmelized brown sugar	flowery, lavendar, chemical
bubblegum, vanilla, flowery	bubblegum, peanuts (nutty)
Robutussin, grape, vanilla	nail polish remover (non-acetone, fruity), fruity, grape
jasmine, flowery, alcohol	flower, alcohol,

Appendix H. Consumer Panel Results for Odor Objection Concentration Based on Degree of Liking

	Concentrations of Crude MCHM Presented to Panelists, ppb								Best Estimate Threshold, ppb	
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Value
01	3/3/14	3	5	2	2	2	2	1	1	100
02	3/3/14	6	5	7	6	2	8	8	6	3.8
03	3/3/14	6	4	3	6	3	7	2	6	35
04	3/3/14	1	5	8	5	1	7	7	7	3.8
06	3/3/14	8	7	8	8	7	9	9	9	0.43
07	3/3/14	6	4	3	3	1	4	1	6	35
08	3/3/14	3	6	5	5	4	5	5	5	100
09	3/3/14	7	8	3	9	5	8	9	8	3.8
10	3/3/14	2	5	2	4	7	1	6	6	12
11	3/3/14	7	5	6	4	7	5	6	8	12
12	3/3/14	2	3	1	5	5	6	8	7	3.8
13	3/3/14	9	8	9	6	8	9	9	9	0.43
14	3/3/14	2	2	2	4	4	5	6	9	12
15	3/3/14	4	5	1	2	3	8	6	4	100
16	3/3/14	3	3	3	4	3	2	3	2	100
19	3/3/14	8	7	5	6	4	6	7	5	100
20	3/3/14	1	5	7	8	8	9	9	9	0.14
22	3/3/14	6	3	5	5	7	8	8	8	1.3
23	3/3/14	4	3	6	7	6	4	7	7	12
24	3/3/14	4	1	5	8	9	9	6	7	0.43
25	3/3/14	6	8	5	6	7	5	2	2	100
27	3/3/14	5	3	6	6	4	5	7	7	12
28	3/3/14	3	5	4	2	7	7	7	7	1.3
29	3/3/14	5	4	4	6	5	5	5	4	100
31	3/3/14	6	3	3	5	8	4	6	7	12
32	3/3/14	4	6	5	2	4	6	4	6	35
33	3/3/14	5	6	3	4	3	5	6	7	12
34	3/3/14	4	4	7	5	5	6	7	7	3.8
35	3/3/14	6	4	4	3	5	5	7	9	12
36	3/3/14	5	7	5	5	4	7	7	7	3.8
38	3/5/14	5	4	4	7	5	7	8	8	3.8
39	3/5/14	6	6	7	7	8	9	9	9	0.016
41	3/5/14	5	8	5	6	8	7	8	6	1.3
42	3/5/14	6	3	5	6	3	5	3	3	100
43	3/5/14	3	5	7	7	8	8	8	9	0.14
44	3/5/14	4	9	5	6	9	9	7	7	0.43
45	3/5/14	1	1	3	2	1	2	2	4	100
46	3/5/14	4	1	5	5	4	6	4	6	35
47	3/5/14	1	1	2	5	7	5	5	6	35
48	3/5/14	5	5	4	4	3	6	5	6	35
49	3/5/14	2	2	2	3	2	2	2	2	100
50	3/5/14	7	3	3	2	7	2	7	2	100
51	3/5/14	6	4	5	4	7	8	7	8	1.3
53	3/5/14	9	9	9	9	9	9	9	9	0.047
54	3/5/14	6	2	5	7	5	8	9	9	3.8
55	3/5/14	7	5	6	7	7	7	7	8	0.14
56	3/5/14	5	4	3	9	9	8	1	9	35
57	3/5/14	3	3	7	5	7	8	7	8	1.3
59	3/5/14	6	8	8	5	7	7	7	6	1.3
62	3/5/14	6	6	8	8	8	6	6	8	0.016
63	3/5/14	1	8	5	2	1	1	5	2	100

Appendix H. Consumer Panel Results for Odor Objection Concentration Based on Degree of Liking

		Concentrations of Crude MCHM Presented to Panelists, ppb									
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Value	
64	3/5/14	4	6	6	3	5	5	2	5	100	
65	3/5/14	9	8	9	9	8	9	8	9	0.047	
66	3/5/14	5	2	1	1	2	4	4	4	100	
67	3/5/14	5	5	4	4	5	4	4	4	100	
68	3/5/14	4	4	5	6	5	5	7	8	12	
69	3/5/14	3	2	3	2	7	5	5	5	100	
70	3/5/14	2	6	7	2	2	2	8	2	100	
71	3/5/14	1	1	1	9	1	8	1	9	35	
72	3/5/14	1	2	1	1	2	7	1	1	100	

Note: The OOC was only recorded for concentrations at or above the OTC; the individual OOC is the geometric mean of the two concentrations where there is a jump in the degree of disliking to a score of 6 or above which is noted by gray-shaded cells.

Geometric Mean, ppb = 7.7

Appendix I. Consumer Panel Results for Odor Objection Concentration Based on Objection/Complaint

	Concentrations of Crude MCHM Presented to Panelists, ppb									Best Estimate Threshold, ppb
Donalista	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Value
Panelists 01	3/3/14	N	0.082 Y	0.25 Y	0.74 Y	<u> </u>	0.7 Y	20 Y	Y	0.047
01	3/3/14	Y	N N	Y	Y Y	N	Y	Y Y	Y	3.8
02	3/3/14	Y	N	N	Y Y	N	Y	N	Y	3.8
04	3/3/14	N	N	Y	N N	N	Y	Y	Y	3.8
04	3/3/14	Y	Y	Y	Y	Y	Y	Y	Y	0.43
07	3/3/14	Ŷ	N	N.	N	N	N	N	Y	100
08	3/3/14	N	Y	N	N	N	N	N	N	100
09	3/3/14	Y	Y	N	Y	N	Y	Y	Y	3.8
10	3/3/14	Ν	N	N	N	Y	N	N	N	100
11	3/3/14	Y	N	N	N	Y	N	N	Y	35
12	3/3/14	N	N	N	N	N	Y	Y	Y	3.8
13	3/3/14	Y	Y	Y	N	Y	Y	Y	Y	1.3
14	3/3/14	N	N	N	N	N	Y	Y	Y	3.8
15	3/3/14	N	Y	N	N	N	Y	Y	N	100
16	3/3/14	N	N	N	N	N	N	N	N	100
19	3/3/14	N	N	N	N	N	N	N	N	100
20	3/3/14	Ν	Y	Y	Y	Y	Y	Y	Y	0.047
22	3/3/14	Ν	N	N	N	Y	Y	Y	Y	1.3
23	3/3/14	Ν	N	N	Y	N	N	N	N	100
24	3/3/14	Ν	N	N	Y	Y	Y	Y	Y	0.43
25	3/3/14	Y	Y	N	Y	Y	N	N	N	100
27	3/3/14	Ν	N	Y	Y	N	N	Y	Y	12
28	3/3/14	Ν	N	N	N	Y	Y	Y	Y	1.3
29	3/3/14	Ν	N	N	Y	N	N	N	N	100
31	3/3/14	Ν	N	N	N	Y	N	Y	Y	12
32	3/3/14	Ν	Y	N	N	N	Y	N	Y	35
33	3/3/14	Ν	N	N	N	N	N	N	Y	35
34	3/3/14	N	N	Y	N	N	Y	Y	Y	3.8
35	3/3/14	Ν	N	N	N	N	N	Y	Y	12
36	3/3/14	N	Y	N	N	N	Y	Y	Y	3.8
38	3/5/14	Ν	N	N	N	N	Y	Y	Y	3.8
39	3/5/14	Y	Y	Y	Y	Y	Y	Y	Y	0.016
41	3/5/14	N	Y	N	Y	Y	Y	Y	Y	1.3
42	3/5/14	Y	N	N	Y	N	N	N	N	100
43	3/5/14	N	N	Y	Y Y	Y Y	Y	Y Y	Y Y	0.14
44	3/5/14	N	Y	N			Y			0.43
45 46	3/5/14 3/5/14	N N	N N	N N	N N	N N	N N	N N	N Y	100 35
46	3/5/14 3/5/14	N N	N N	N N	N N	N Y	N Y	N N	Y Y	35
47	3/5/14 3/5/14	N N	N N	N N	N N	Y N	Y N	N	N Y	35 100
48 49	3/5/14 3/5/14	N	N N	N N	N N	N N	N	N N	N	100
	3/5/14	Y	N	N N	N N	Y	N	Y	N	100
51	3/5/14	Y	N	N	N	Y	Y	Y Y	Y	1.3
53	3/5/14	Y	Y	Y	Y	Y	Y	Y Y	Y	0.047
54	3/5/14	N	N	N	Y	N	Y	Y	Y	3.8
55	3/5/14	Y	N	N	Y	Y	Y	Y	Y	0.43
56	3/5/14	N	N	N	Y	Y	Y	N	Y	35
57	3/5/14	N	N	Y	N	Y	Y	Y	Y	1.3
59	3/5/14	N	Y	Y	N	Y	Y	N	N	1.0
62	3/5/14	Y	Y	Y	Y	Y	N	Y	Y	3.8
63	3/5/14	N	N	N	N	N	N	N	N	100

Appendix I. Consumer Panel Results for Odor Objection Concentration Based on Objection/Complaint

			Concentrations of Crude MCHM Presented to Panelists, ppb									
Panelists	Date Study Conducted	0.027	0.082	0.25	0.74	2.2	6.7	20	60	Value		
64	3/5/14	N	N	N	N	N	N	N	N	100		
65	3/5/14	Y	Y	Y	Y	Y	Y	Y	Y	12		
66	3/5/14	N	N	N	N	N	Y	Y	Y	3.8		
67	3/5/14	N	N	N	N	N	N	N	N	100		
68	3/5/14	N	N	N	Y	N	N	Y	Y	12		
69	3/5/14	N	N	N	N	Y	N	N	N	100		
70	3/5/14	N	N	Y	N	N	N	Y	N	100		
71	3/5/14	N	N	N	Y	N	N	N	Y	35		
72	3/5/14	N	N	N	N	N	Y	N	N	100		

Note: The OOC was only recorded for concentrations at or above the OTC; the individual OOC is the geometric mean of the two concentrations where there is a change to a consistent answer of Yes to the question: Would you object/complain about the odor in the different cup? Noted by gray-shaded cells.

Geometric Mean, ppb = 9.5