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HIGHLIGHTS

- The largest contributors to odor activity values (OAV) were heptanoic acid (23.5%), hexanoic acid (17.6%), indole (14.7%), and dimethyl disulfide (DMDS) (9.50%).
- Measurements of volatile organic compounds (VOC) were greater from plots where manure was surface-applied than from plots with incorporated manure.
- Emissions of DMDS and dimethyl trisulfide (DMTS) (0.318 and 0.074 μg m^{-2} min^{-1}, respectively) were greater for plots with manure from beef cattle fed a diet containing 30% wet distillers grains with solubles (WDGS).
- Rainfall immediately after manure application influenced the types and amounts of VOC that were emitted.

ABSTRACT. *Land application of beef cattle manure may result in the emission of volatile organic compounds (VOC). This study was conducted to evaluate the effects of diet, land application method, soil water condition, and time since manure application on VOC emissions. Manure was collected from feedlot pens where cattle were fed diets containing 0%, 10%, or 30% wet distillers grains with solubles (WDGS). The effects of manure application method (surface-applied or incorporated) and soil water condition (saturated or wet) on VOC emissions were measured over a 48 h period. Heptanoic, hexanoic, isobutyric, and isovaleric acids contributed 23.5%, 17.6%, 9.26%, and 3.39% (0.034, 0.258, 0.030, and 0.014 μg m^{-2} min^{-1}), respectively, to total odor activity values (OAV). The aromatics indole and skatole contributed 14.7% and 8.84%, (0.005 and 0.0004 μg m^{-2} min^{-1}), respectively, to total OAV. Dimethyl disulfide (DMDS) contributed 9.50% (0.013 μg m^{-2} min^{-1}) and dimethyl trisulfide (DMTS) contributed 5.68% (0.030 μg m^{-2} min^{-1}) to total OAV. Emissions of the sulfur compounds (DMDS and DMTS) were substantially greater for the 30% WDGS diet. With the exception of heptanoic acid, flux measurements were greater from the plots where manure was surface-applied than from the plots where manure was incorporated. Emissions of each VOC were greater on the first day following manure application when a saturated soil water condition was present. VOC flux values were found to rapidly decrease following manure application. Effective best management practices for reducing VOC emissions are to incorporate manure soon after application and to delay land application when there is a high probability of rainfall.*

Keywords. *Air contaminants, Air quality, Environmental management, Land application, Manure management, Odor control, Odor emission, Odor evaluation, Volatile organic compounds, Volatile fatty acids.*

Airborne pollutants may be a concern to individuals living near animal feeding operations (AFOs) (Wright et al., 2005; Donham et al., 2007; Heederik et al., 2007; Katja et al., 2007; Thorne, 2007). Much of the previous odor research on cattle AFOs has focused on odor characteristics and emission rates from feedlots (Auvermann et al., 2007; Kyoung et al., 2007; Todd et al., 2008; Trabue et al., 2008). Utilization of ethanol by-products, including distillers grains, as a feed additive for beef cattle has influenced the characteristics of feedlot emissions (Gralapp et al., 2002; Varel et al., 2008, 2010; Spiehs and Varel, 2009).

Feeding wet distillers grains with solubles (WDGS) to cattle can increase both the intake and excretion of nitrogen, phosphorus, and sulfur. Feeding WDGS contributes to the

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production of odorous volatile organic compounds (VOC) such as long-chain and branched-chain volatile fatty acids (VFA) (Spiehs and Varel, 2009). VOC flux and odor activity values (OAV) were found to be similar from feces and urine obtained from cattle fed steam-flaked corn diets containing varying amounts of WDGS (Hales et al., 2012; Spiehs et al., 2012, 2018). The majority of the VOC emitted from cattle feeding operations is from urine deposition (Hales et al., 2015).

The frequency and duration of precipitation events on beef cattle feedlots cannot be altered. However, the rate at which the pen surface dries following a precipitation event can be modified. Feedlot management options that influence soil water condition and in turn odor emissions include (1) the amount of manure that is allowed to collect on the pen surface, (2) how well the drainage systems of the pens are maintained, and (3) the degree to which soil is mixed with manure on the pen surface (Woodbury et al., 2018).

Several areas associated with beef cattle AFOs may be a source of airborne pollutants, including feed storage areas, manure storage facilities, pen surfaces, and runoff holding ponds (Koelsch et al., 2004). Airborne pollutants from AFOs can include ammonia, bacteria, dust, endotoxins, hydrogen sulfide, particulate matter, and VOC (Mitloehner and Calvo, 2008). Malodorous VOC, including VFA, aromatics, and sulfides, are emitted during the microbial degradation of manure (Mackie et al., 1998; Miller and Varel, 2001; Miller and Berry, 2005; Rappert and Muller, 2005; Trabue et al., 2011).

The use of manure as a fertilizer and soil amendment can provide many soil quality benefits and contribute to improved agricultural sustainability (Tester, 1990; Fauci and Dick, 1994; Edmeades, 2003; Ferguson et al., 2005). However, airborne pollutants may also emanate from land on which cattle manure has been applied (McGinn et al., 2003). While VOC emissions from land-applied cattle manure originate from more diffuse sources than are typical for feedlot pen surfaces and manure storage facilities, land application areas may be closer to domestic housing areas than animal production facilities, creating a greater opportunity for odor concerns.

While there has been some research to evaluate the effects of land application of liquid dairy and swine manure on odor emissions (Hanna et al., 2000; Lau et al., 2003; Parker et al., 2013a; Liu et al., 2018), there has been limited research with beef cattle manure. Therefore, little information is available concerning best management practices for reducing VOC emissions following land application of solid manure. The objective of this study was to evaluate the effects of animal diet, land application method, soil water condition, and time since manure application on VOC emissions from areas on which beef cattle manure had been applied.

Materials and Methods

Study Site

Field experiments were conducted during the summer of 2012 at the University of Nebraska Rogers Memorial Farm, located 18 km east of Lincoln, Nebraska. The site had been cropped using a grain sorghum (*Sorghum bicolor* (L.) Moench), soybean (*Glycine max* (L.) Merr.), and winter wheat (*Triticum aestivum* L. cv. Pastiche) rotation, under a no-till management system, and was planted to winter wheat during the 2010-2011 cropping season. The area was left undisturbed following the wheat harvest in July 2011. Herbicide was applied as needed to control weed growth. Wheat residue was removed by hand raking prior to plot establishment.

Table 1. Mean air temperature and humidity at the study site during the two sampling periods.

Date	Mean Temperature (°C)	Mean Humidity (%)
18 June 2012	30	59
19 June 2012	29	60
20 June 2012	22	79
9 July 2012	27	55
10 July 2012	23	58
11 July 2013	22	62

The soil at the study site was classified as an Aksarben silt loam (fine, smectitic, mesic Typic Argiudoll). The soil contained 25% sand, 49% silt, and 26% clay and had a pH of 7.0 and organic matter content of 4.2%. Mean air temperatures and average humidity during the study are shown in table 1.

Beef Cattle Manure

Beef cattle manure was collected from feedlot pens located at the U.S. Meat Animal Research Center near Clay Center, Nebraska. Calves born during the spring of 2011 were placed in the pens in October 2011. The cattle were fed a dry rolled corn-based diet that contained 0%, 10%, or 30% WDGS. The animals were removed from the pens, and the surface was allowed to dry for several days. The manure was then collected from behind the feed bunk apron of the pens using a front-end loader. Under normal feedlot operating conditions, manure is usually stockpiled outdoors. However, in this study, the manure was stored indoors in 125 L plastic containers and allowed to air-dry until its weight no longer changed over time (approximately two weeks). A wood chipper was used to grind the manure until it passed through a 4 mm sieve, and then the ground manure was thoroughly mixed.

The manure was land-applied approximately 31 weeks (plots 501 to 506) or 34 weeks (plots 601 to 606) later on 18 June or 9 July 2012 (fig. 1). A composite consisting of ten grab samples was obtained prior to each land application date. The single composite sample obtained on each date was used to determine the physical and chemical characteristics of the manure including total nitrogen content (table 2) (Servi-Tech Laboratories, Hastings, Neb.).

Experimental Design

Twelve plots (0.75 m × 2.0 m) were established using a randomized block design (fig. 1). The experimental treatments included manure application method (surface-applied or incorporated immediately after manure application), diet (0%, 10%, or 30% WDGS), soil water condition (saturated or wet), and time since manure application: 0 (24), 1 (25), 2 (26), 6 (30), or 23 (47) h (sampling times for the wet soil water condition on day 2 are shown in parentheses). The

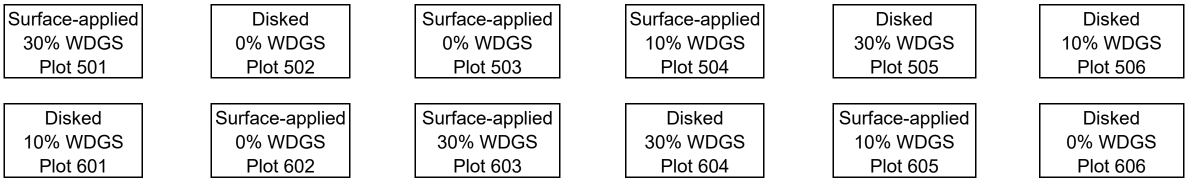


Figure 1. Schematic of plot layout, application method, and manure source. WDGS is wet distillers grains with solubles used in the diet. Plots 501 to 506 were contained in block 1, while plots 601 to 606 were contained in block 2.

Table 2. Laboratory analyses of manure from beef cattle fed diets containing wet distillers grains with solubles (WDGS).

	WDGS Content		
	0%	10%	30%
Total nitrogen (%)	1.36	1.75	1.94
Organic nitrogen (%)	1.30	1.66	1.74
Ammonium nitrogen (%)	0.028	0.045	0.190
Nitrate nitrogen (%)	0.030	0.021	0.007
Phosphorus (%)	0.359	0.415	0.857
Water (%)	26.9	25.1	11.7
Solids (%)	73.1	74.9	88.4
Organic matter (%)	31.3	46.5	42.3
Ash (%)	41.9	28.5	46.1
C:N ratio	13.4	15.4	12.7

experimental plots were contained within 20 cm wide sheet metal frames driven approximately 10 cm into the soil. The sheet metal frames prevented runoff from outside the plot from entering the test area.

For quantification of background emissions without manure addition, another set of six plots was established immediately adjacent to the twelve plots identified previously. Three of the six plots had water applied until runoff was observed at the bottom of the plots, and three were left in a dry condition. A hand-operated irrigation wand was used to apply water to the plot surfaces. Wind tunnels were installed, and background emissions were collected using the experimental procedures described below. Background values for each of the measured compounds are listed in table 3.

Flux measurements were taken on 18 to 20 June 2012 for plots 501 to 506 (1 to 2 days after the first manure application) and on 9 to 11 July 2012 (1 to 2 days after the second manure application) on plots 601 to 606 (fig. 1). There were six sets of sampling equipment, and flux samples were collected simultaneously from the six plots within each experimental block. Adjustments for differences in solar radiation, air temperature, turbulence, and other climatic conditions among sampling days were not made, but the ambient temperature was recorded (table 1).

Solid beef cattle manure was applied to meet the one-year N requirement for corn (151 kg N ha^{-1} $year^{-1}$ for an expected grain yield of 9.4 Mg ha^{-1}). Based on laboratory analysis of the manure, application rates for each plot were calculated assuming that the first-year N availability from the manure was 40% (Eghball et al., 2002). Manure was uniformly applied by hand across the plot surfaces using 19 L buckets for the surface application treatments. To ensure uniform distribution following tillage on the plots where manure was incorporated, the manure application covered an area slightly larger than the final plot dimensions. A 5 m tandem disc was used to incorporate the applied manure to a depth of approximately 8 cm. A single tillage pass occurred up and down the slope in the direction of overland flow within approximately 30 min after manure application.

Flux measurements were taken during two test periods under saturated (day 1) and wet (day 2) soil water conditions.

Table 3. Summary of measured compounds, method detection limits (MDL), calibration statistics, and background soil emission rates under dry and wet soil water conditions.

Compound	Molecular Weight	Retention Time (min)	Min. (ng)	Max. (ng)	MDL (ng)	MDL[a] (μg m^{-2} min^{-1})	RSD[b]	r^2	Background Emission (μg m^{-2} min^{-1}) Dry[c]	Wet[d]	Relative Contribution to OAV[e] (%)
Volatile fatty acids											
Acetic acid	60.0	12.4	30.2	4114	32.9	0.16	0.38	0.99	0.33	0.67	0.86
Butyric acid	88.1	15.1	2.9	3723	8.6	0.042	0.87	0.99	0.03	0.04	0.56
Heptanoic acid	130.2	19.5	0.66	502	1.6	0.0077	0.25	0.98	0.02	0.02	23.5
Hexanoic acid	116.2	18.1	5.3	8244	2.2	0.011	0.50	0.98	0.08	0.08	17.6
Isobutyric acid	88.1	14.2	2.4	2239	15.1	0.073	0.82	0.99	0.01	0.01	9.26
Isovaleric acid	102.1	15.7	0.57	1810	5.9	0.028	0.80	0.99	0.00	0.01	3.39
Propanoic acid	74.1	13.8	4.2	6721	15.9	0.077	0.89	0.99	0.04	0.13	0.87
Valeric acid	102.1	16.7	1.9	807	2.5	0.012	0.62	0.99	0.02	0.03	1.13
Aromatics											
4-Ethylphenol	122.2	22.2	0.07	7.66	6.0	0.029	0.20	0.97	0.00	0.00	1.21
4-Methylphenol	108.1	21.1	0.37	871	4.0	0.019	0.11	0.97	0.00	0.01	1.22
Indole	117.1	25.2	0.07	259	3.5	0.017	0.09	0.99	0.00	0.00	14.7
Phenol	94.1	20.2	5.0	2027	6.0	0.029	0.14	0.99	0.04	0.07	1.66
Skatole	131.2	25.6	0.015	9.84	4.8	0.023	0.12	0.98	0.00	0.00	8.84
Sulfides											
Dimethyl disulfide	94.2	5.2	2.4	1281	1.0	0.005	0.04	0.99	0.01	0.01	9.50
Dimethyl trisulfide	126.2	11.0	0.40	587	2.1	0.01	0.14	0.99	0.00	0.00	5.68

[a] Method detection limit for flux based on a 60 min sample with a wind tunnel flow rate of 1 L min^{-1}.
[b] Relative standard deviation (SD/mean) from seven replications at minimum mass analyzed.
[c] Background emission rate from soil receiving no water or beef manure (mean of three replicates).
[d] Background emission rate from soil receiving water but no beef manure (mean of three replicates).
[e] Relative contributions to total OAV are means obtained across all experimental variables.

An irrigation system was constructed that allowed water to be sprayed at a low rate onto the plot surface during day 1. The application intensity was adjusted so that the plot surface remained saturated, but runoff did not occur. VOC measurements were taken at 0, 1, 2, 6, and 23 h on day 1 (saturated soil water condition). The flux measurements obtained during day 1 can be used to determine if VOC emissions are impacted by extended periods of rainfall when saturated soil water conditions are present. The irrigation system was removed at the end of day 1. The effects of drying on VOC emissions were measured during the next 24 h (day 2, wet soil water condition) at 24, 25, 26, 30, and 47 h after manure application.

Wind Tunnel Flux Measurements

Flux measurements were made using six wind tunnels, one per plot within a given treatment block (fig. 2). Details on the operation of the small wind tunnels are provided by Parker et al. (2013b). Each wind tunnel had a 51 mm height, 305 mm length, and 152 mm width, with a footprint of 0.046 m^2 and internal volume of 2.36 L. The sweep air entered the wind tunnel through 17 holes (6 mm diameter) in three rows at heights of 17 mm (6 holes), 30 mm (5 holes), and 43 mm (6 holes) above the base. Air exited the wind tunnel through three 10 mm diameter holes equally spaced at a height of 27 mm above the surface at the opposite end of the tunnel. Sweep air (1 L min^{-1}) was supplied via Teflon tubing from a compressed air cylinder (Linweld, Lincoln, Neb.).

The wind tunnel was placed on the plot surface and pressed into the soil to establish a seal around the base of the tunnel. After an equilibration period of 21 min that allowed three volumes of sweep air to pass through the wind tunnel, VOC samples were collected from the air exiting the wind tunnel. Air samples were obtained in stainless steel sorbent tubes (89 mm × 6.4 mm outside diameter, Markes International, Bridgend, U.K.) filled with Tenax TA sorbent (Sigma-Aldrich, St. Louis, Mo.). Prior to use, the sorbent tubes were conditioned for 30 min at 230°C. Air was pulled through the sorbent tubes at a flow rate of 75 mL min^{-1} for 60 min using a vacuum pump (Pocket Pump 210 Series, SKC, Covington, Ga.).

Flux density (J) was calculated on a mass per unit area per unit time basis (μg m^{-2} min^{-1}) using equation 1:

$$J = \frac{Q C_{air}}{A} \tag{1}$$

where Q is the sweep airflow rate (m^3 min^{-1}), C_{air} is the VOC concentration of the exiting air (μg m^{-3}), and A is the footprint of the wind tunnel (m^2).

Air samples for VOC measurements were taken at 0, 1, 2, 6, and 23 h after manure application for the saturated soil water condition (day 1) and at 24, 25, 26, 30, and 47 h after manure application for the wet soil water condition (day 2). The irrigation system was turned off to prevent the vacuum pumps from becoming saturated during the period required to obtain VOC measurements on day 1. After the VOC samples were collected for a given sampling period on day 1, all of the sampling equipment was removed from the plots and irrigation was resumed until the next sampling interval. The wind tunnel was placed on different sections of the plot during each sampling interval on days 1 and 2. The same VOC sampling equipment, test intervals, and air sampling protocols were used on days 1 and 2.

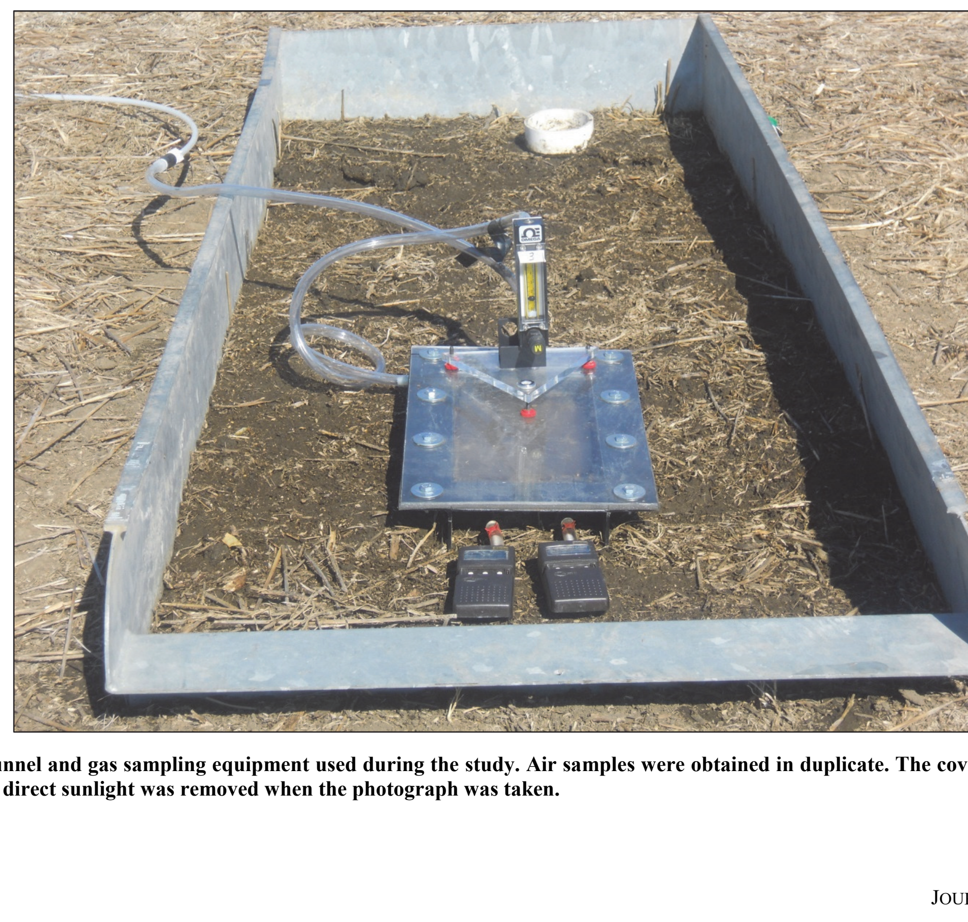


Figure 2. Wind tunnel and gas sampling equipment used during the study. Air samples were obtained in duplicate. The cover used to shade the wind tunnel from direct sunlight was removed when the photograph was taken.

Gas Chromatography/Mass Spectrometry

Sorbent tube samples were collected in duplicate on each plot. Two blocks were included in the experimental design (fig. 1); therefore, four measurements were obtained for a given soil water condition and sampling interval. The four measurements were treated as repeated measures in the statistical analyses.

A thermal desorption gas chromatography/mass spectrometry (TD GC/MS) system consisting of a Unity 2 thermal desorber with Ultra 2 autosampler (Markes International, Bridgend, U.K.) was used to analyze the sorbent tube samples. Samples were quantified with an Agilent GC/MS system (7890A/5975C, Agilent Technologies, Santa Clara, Cal.). The system used an Innowax (30 m × 0.25 mm inner diameter) polar capillary column (polyethylene glycol, 0.25 mm film thickness) that was operated at a constant airflow rate of 1.4 mL min^{-1}.

Samples were purged for 1 min at 40 mL min^{-1} to remove water and solvent. The tube was then desorbed for 10 min at 280°C with an ultra-high purity helium (carrier gas) flow of 50 mL min^{-1} and trapped on a cold trap maintained at -10°C. The cold trap was heated to 320°C for 1 min with a helium flow of 20 mL min^{-1}, and 1.4 mL min^{-1} was transferred to the GC/MS system. The column was held at 40°C for 3 min in the GC oven, and then the temperature was increased to 230°C at a rate of 8°C min^{-1} and held at 230°C for 5 min.

Samples were analyzed for three basic categories of compounds: VFA, aromatics, and volatile sulfur compounds (VSC). The compounds were selected based on the constituents that were most prevalent in our previous sorbent tube studies on emissions from beef cattle manure (Woodbury et al., 2014). The eight VFA were ethanoic (acetic), butanoic (butyric), heptanoic, hexanoic, 2-methylpropanoic (isobutyric), 3-methylbutanoic (isovaleric), propionic, and pentanoic (valeric) acids. For the remainder of this article, the more common names for the fatty acids (shown in parentheses) will be used. The five aromatic compounds were 4-ethylphenol, 4-methylphenol, indole, phenol, and skatole. The two sulfur-containing VOC were dimethyl disulfide (DMDS) and dimethyl trisulfide (DMTS).

Calibration standard solutions were prepared by diluting known masses of pure chemicals with methanol. The standards were prepared and analyzed within 48 h to establish standard curves. The prepared standards were periodically checked with stored standards to identify any changes in sensitivity of the analysis. All chemicals and solvents were FCC grade (Sigma Aldrich, St. Louis, Mo.). Standards were prepared using serial dilutions and then injected onto clean tubes while purified air was pulled through the tubes with a vacuum pump operated at 75 mL min^{-1}. The standard solutions were stored for periodic instrument calibrations.

Our experience has shown little evidence that VFA are ionized and become nonvolatile. We have seen nearly identical GC responses from freshly made standards when compared to standards that have been stored, which indicates minimal ionization or response issues. Within the linear range, standard curves were fit using linear regression with zero *y*-intercept. Coefficients of determination (r^2) for the standard curves ranged from 97.8% to 99.7% for the eight VFA, from 97.8% to 99.3% for the five aromatics, and were 99.8% for both DMDS and DMTS.

Andersen et al. (2012) showed that thermal desorption procedures similar to those used in this study can dimerize methanethiol (MT) to other VSC such as DMDS and DMTS. Because MT is sometimes reported in manure in anaerobic environments, it is difficult to definitively quantify whether VSC emissions are MT, DMDS, DMTS, or a combination of these constituents. For this reason, we estimated odor impacts as discussed in the following section using two methods: first using measured DMDS and DMTS concentrations, and second assuming that an equal mass of methanethiol was converted to DMDS and/or DMTS.

Odor Activity Values and Analyses

Concentrations of individual compounds were converted to their respective odor activity values (OAV). An assessment of the relative impact of each individual odor compound is provided by OAV analyses. Details on the conversion are provided by Parker et al. (2013a); a brief description is provided below.

The OAV is a ratio of the measured concentration of a single compound normalized to the single-compound odor threshold (SCOT) for that compound (Patton and Josephson, 1957; Friedrich and Acree, 1998; Trabue et al., 2006; Parker et al., 2010, 2013b). Therefore, the larger the OAV for an individual compound, the more likely that compound will contribute to the overall odor of a complex odor mixture.

The SCOT values for each compound were obtained from published odor thresholds (table 4). The relative contribution of each compound was calculated by subtracting the background emission for each compound and dividing by the sum of the OAV for all measured compounds. This approach does not account for possible synergistic or other complex interactions of the compounds (DiSpirito et al., 1994; Powers, 2001; Zahn et al., 2001).

Statistical Analyses

The effects of diet, land application method, soil water condition, and time since manure application on VOC measurements were determined using ANOVA (SAS, 2011). The MIXED procedure of SAS was used in the analyses, and the subsample error was adjusted out of the error term. The restricted maximum likelihood approach was used to provide estimates, and the profile method was used to concentrate the residual variance out of the optimization problem.

For a given plot, duplicate readings collected for a particular test interval and sample collection time were treated as repeated measures. Significant treatment differences were further evaluated using post-hoc protected multiple comparison tests (Fisher's LSD). A probability level of ≤0.05 was considered significant.

Results and Discussion

Contributions to total OAV are averages obtained across all diets, land application methods, soil water conditions, and time since manure application. VFA with notable contributions to total OAV included heptanoic, hexanoic, isobutyric,

Table 4. Summary of published single-compound odor thresholds (SCOT) for the 15 compounds measured in this study. Different statistical measures of central tendency are provided. All raw data on thresholds are from van Gemert (2003) unless otherwise noted.

Compound		N[a]	Minimum (μg m^{-3})	Maximum (μg m^{-3})	Arithmetic Mean (μg m^{-3})	Standard Deviation (μg m^{-3})	Geometric Mean (μg m^{-3})	Harmonic Mean (μg m^{-3})	Median (μg m^{-3})	Geometric Mean[b]
Volatile fatty acids	Acetic acid	8	25	7500	2480	2754	578	85	2050	467
	Butyric acid	11	0.4	105	25	34	6.9	1.4	13	23
	Heptanoic acid	3	22	300	118	157	60	38	3	-
	Hexanoic acid	5	12	510	182	226	69	31	40	83.1
	Isobutyric acid	2	0.8	285	145	198	38	10	145	41
	Isovaleric acid	5	0.22	14	5	5.5	2.3	0.81	4.1	4.7
	Propionic acid	7	3	890	303	344	106	18	80	101
	Valeric acid	6	0.8	75	24	30	8.8	3	9	11.7
Aromatics[c]	4-Ethylphenol	1	6.3	6.3	6.3	-	6.3	6.3	6.3	-
	4-Methylphenol	4	0.05	24	9.2	11.5	1.3	0.16	6.3	2.6
	Indole	2	0.6	7.1	3.8	4.6	2.1	1.1	3.8	1.9
	Phenol	9	39	4000	734	1290	206	88	200	127
	Skatole	4	0.35	0.78	0.51	0.19	0.48	0.46	0.45	1.6
Sulfides	DMDS	5	1.6	64	25	28	12	5.3	8.5	-
	DMTS	3	0.08	14	7.2	7	2	0.24	7.5	-

[a] N is the number of independent odor threshold observations used in the calculations.
[b] SCOT values from the compilation by Parker et al. (2010) are provided for comparison.
[c] 4-Ethylphenol threshold is from Trabue et al. (2008).

and isovaleric acids, with contributions of 23.5%, 17.6%, 9.26%, and 3.39% (0.034, 0.258, 0.030, and 0.014 μg m^{-2} min^{-1}), respectively (tables 3 and 5). Trabue et al. (2011) also reported that VFA were the most abundant of the major odorants from beef cattle feedlots.

The two aromatics that contributed substantially to total OAV were indole and skatole, with values of 14.7% and 8.84% (0.005 and 0.0004 μg m^{-2} min^{-1}), respectively (tables 3 and 5). The sulfides (DMDS and DMTS) contributed 9.50% and 5.68% (0.013 and 0.030 μg m^{-2} min^{-1}) to total OAV. Acetic acid, butyric acid, propanoic acid, valeric acid, 4-ethylphenol, 4-methylphenol, and phenol each had contributions that were less than 2% of the total measured OAV and are not included in further discussion. Measured values for isobutyric acid, isovaleric acid, indole, and skatole were in some cases less than the minimum detection limit.

With the exception of heptanoic acid, emissions of each of the measured VOC were greater from the plots where manure was surface-applied (table 5). The ratio of VOC emissions from the surface-applied to incorporated plots ranged from 1.00 for heptanoic acid to 11.0 for DMTS. Woodbury et al. (2014) also noted larger emissions from sites where beef cattle manure was surface-applied rather than incorporated.

Emission of DMDS for the 30% WDGS diet was 0.318 μg m^{-2} min^{-1}, compared to emissions of 0.032 and 0.053 μg m^{-2} min^{-1}, respectively, for the 0% and 10% WDGS diets (table 5). Similarly, emission of DMTS was 0.074 μg m^{-2} min^{-1} for the 30% WDGS diet, compared to emissions of 0.007 and 0.008 μg m^{-2} min^{-1}, respectively, for the 0% and 10% WDGS diets. Larger emissions of the sulfur compounds for the 30% WDGS diet were also noted by Woodbury et al. (2014).

Flux measurements for each VOC were greater under saturated soil water conditions than under wet conditions (table 5). The ratio of VOC emissions from saturated to wet soil water conditions ranged from 1.27 for heptanoic and hexanoic acids to 8.00 for indole. VOC flux values decreased

Table 5. Odorous volatile organic compound emissions as affected by application method, diet, soil water condition, and time since manure application. Values listed for a variable are averaged across all other variables. Means in the same column followed by different letters are significantly different at the 0.05 probability level bases on LSD test.

	Volatile Fatty Acids (μg m^{-2} min^{-1})				Aromatics (μg m^{-2} min^{-1})		Sulfides (μg m^{-2} min^{-1})	
	Heptanoic	Hexanoic	Isobutyric	Isovaleric	Indole	Skatole	DMDS	DMTS
Application method								
Surface-applied	0.034	0.268	0.036	0.018	0.007	0.0004	0.241	0.055
Incorporated	0.034	0.248	0.023	0.009	0.002	0.0003	0.028	0.005
Diet								
0% WDGS	0.033	0.264	0.028	0.009	0.002	0.0004	0.032	0.007
10% WDGS	0.040	0.281	0.026	0.011	0.006	0.0003	0.053	0.008
30% WDGS	0.029	0.230	0.035	0.021	0.006	0.0004	0.318	0.074
Soil water condition								
Saturated	0.038	0.289	0.038	0.016	0.008	0.0005 a	0.219	0.050
Wet	0.030	0.227	0.021	0.011	0.001	0.0002 b	0.050	0.010
Time since application[a]								
0 h (24)	0.044	0.344	0.053	0.014	0.007	0.0005	0.143	0.034
1 h (25)	0.038	0.312	0.025	0.015	0.007	0.0004	0.234	0.047
2 h (26)	0.021	0.165	0.022	0.012	0.005	0.0003	0.138	0.041
6 h (30)	0.039	0.240	0.021	0.014	0.002	0.0003	0.123	0.021
23 h (47)	0.028	0.228	0.027	0.012	0.001	0.0002	0.035	0.006

[a] Sampling of VOC emissions under saturated soil water conditions began immediately after manure application, while sampling under wet soil water conditions began 24 h later (at the times shown in parentheses).

substantially by the end of the test period. The isobutyric acid flux of 0.053 μg m^{-2} min^{-1} collected initially was greater than the measurements obtained during the later sampling intervals, which varied from 0.021 to 0.027 μg m^{-2} min^{-1}.

VOLATILE FATTY ACIDS

Mean emissions of heptanoic, hexanoic, isobutyric, and isovaleric acids were 0.034, 0.258, 0.030, and 0.014 μg m^{-2} min^{-1}, respectively. A significant interaction among diet, application method, soil water condition, and time since manure application was found for heptanoic acid (table 6). An heptanoic acid flux value of 0.103 μg m^{-2} min^{-1} occurred on the plots where manure from the 10% WDGS diet was surface-applied (fig. 3). Heptanoic acid emissions usually peaked at the 0 or 1 h sampling periods, and variations in flux values were usually minimal among the other sampling intervals. The results obtained for heptanoic acid were similar to those found for hexanoic, isobutyric, and isovaleric acids. Woodbury et al. (2014) noted that the largest hexanoic acid emissions occurred on plots where manure from the 10% WDGS diet was applied.

AROMATICS

The two aromatics that contributed substantially to total OAV were indole and skatole. The flux values for indole and skatole were 0.007 and 0.0004 μg m^{-2} min^{-1}, respectively, when manure was surface-applied compared to 0.002 and 0.0003 μg m^{-2} min^{-1}, respectively, for incorporated manure. The skatole emission of 0.0005 μg m^{-2} min^{-1} obtained under saturated soil water conditions was significantly greater than the 0.0002 μg m^{-2} min^{-1} obtained under wet conditions. The flux values of 0.007 and 0.0005 μg m^{-2} min^{-1} for indole and skatole, respectively, at the initiation of the tests were substantially greater than the corresponding values of 0.001 and 0.0002 μg m^{-2} min^{-1} obtained at the end of the sampling period.

SULFIDES

The emission trends for DMDS and DMTS were similar. A DMDS emission of 0.241 μg m^{-2} min^{-1} was obtained on the plots where manure was surface-applied compared to 0.028 μg m^{-2} min^{-1} on the plots where manure was incorporated. DMTS flux values of 0.055 and 0.005 μg m^{-2} min^{-1} were measured on the plots where manure was surface-applied and incorporated, respectively. Higher sulfide emission rates on the plots where manure was surface-applied occurred because of increased sulfide production on the soil surface.

The application of manure from a diet containing 30% WDGS resulted in a DMDS flux of 0.318 μg m^{-2} min^{-1} compared to the values of 0.032 and 0.053 μg m^{-2} min^{-1}, respectively, for the 0% and 10% WDGS diets. The DMDS flux of 0.219 μg m^{-2} min^{-1} obtained under saturated soil water conditions was greater than the 0.050 μg m^{-2} min^{-1} obtained under wet conditions.

The removal of the starch component of corn during the distillation process concentrates many of the conserved elements in the byproduct. Sulfur compounds may also be added during processing, which increases the sulfur content of the diet. WDGS are highly digested and have a higher sulfur concentration, which may result in rapid conversion to sulfide compounds under anaerobic conditions (Spiehs and Varel, 2009; Varel et al., 2010).

An increase in sulfide flux rates with time may also have been influenced by a reduction in the oxidation status of saturated soils, which favors the formation of sulfur compounds. Sulfides can be produced under anaerobic conditions for wet soil water conditions. Woodbury et al. (2014, 2016) reported the largest DMDS emissions under wet soil water conditions.

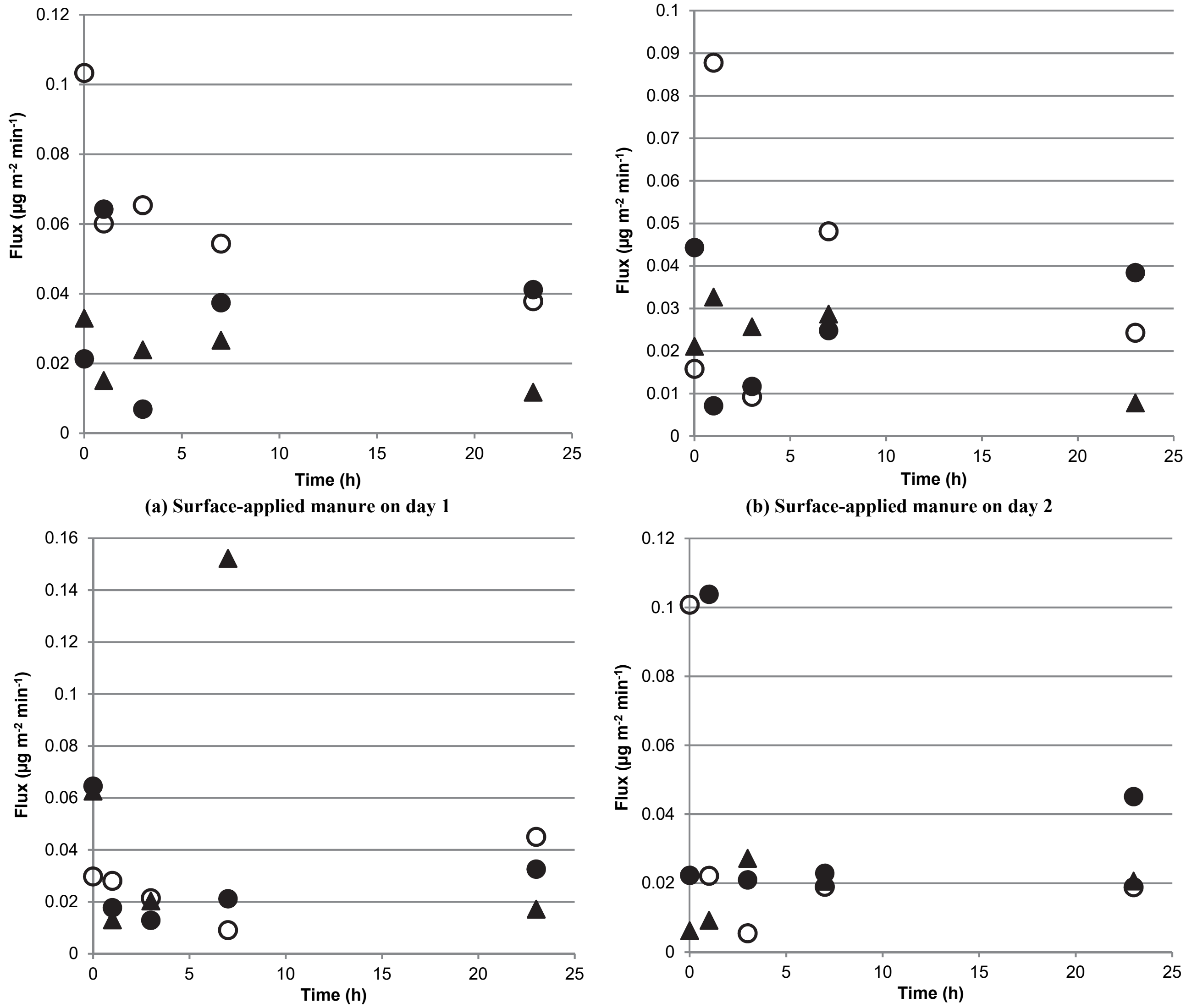
COMPARISONS WITH PREVIOUS STUDIES

Woodbury et al. (2014) measured VOC emissions after land application of manure from diets containing 0%, 10%, or 30% WDGS. Their study was conducted on a site where manure was either surface-applied or incorporated to meet the one-year N requirement for corn. In contrast to the present study, a single application of water was applied 24 h after manure application (at the beginning of day 2). The principal odorous VOC emissions that occurred during day 2 (wet soil water condition, table 5 of Woodbury et al., 2014) were VFA including butyric acid (0.127 μg m^2 min^{-1}), hexanoic acid (1.52 μg m^2 min^{-1}), isobutyric acid (0.050 μg m^2 min^{-1}), isovaleric acid (0.052 μg m^2 min^{-1}), propanoic acid

Table 6. Analysis of variance results (Pr > F) as affected by application method, diet, soil water condition, and time since application.[a]

Factors and Interactions	Volatile Fatty Acids				Aromatic		Sulfides	
	Heptanoic	Hexanoic	Isobutyric	Isovaleric	Indole	Skatole	DMDS	DMTS
Application method	0.95	0.80	0.24	0.32	0.21	0.52	0.13	0.21
Diet	0.62	0.85	0.75	0.56	0.72	0.62	0.18	0.29
Soil water condition	0.36	0.30	0.10	0.10	0.07	0.03*	0.07	0.16
Time	0.27	0.25	0.06	0.90	0.12	0.13	0.47	0.46
Application method × Diet	0.37	0.26	0.62	0.50	0.55	0.68	0.18	0.28
Application method × Soil water condition	0.73	0.86	0.31	0.74	0.20	0.62	0.10	0.20
Diet × Soil water condition	0.66	0.95	0.39	0.72	0.71	0.48	0.13	0.25
Application method × Diet × Soil water condition	0.27	0.25	0.65	0.80	0.55	0.60	0.13	0.24
Application method × Time	0.90	0.65	0.42	0.81	0.43	0.53	0.51	0.52
Diet × Time	0.33	0.50	0.79	0.80	0.35	0.93	0.71	0.67
Application method × Diet × Time	0.58	0.65	0.72	0.93	0.49	0.65	0.68	0.61
Soil water condition × Time	0.63	0.46	0.22	0.11	0.11	0.31	0.56	0.53
Application method × Soil water condition × Time	0.67	0.67	0.54	0.24	0.38	0.45	0.58	0.59
Diet × Soil water condition × Time	0.78	0.90	0.47	0.11	0.44	0.91	0.77	0.68
Application method × Diet × Soil water condition × Time	0.05*	0.09	0.74	0.12	0.48	0.31	0.73	0.63

[a] Asterisks (*) indicate significance at the 0.05 probability level.



(a) Surface-applied manure on day 1

(b) Surface-applied manure on day 2

(c) Incorporated manure on day 1

(d) Incorporated manure on day 2

Figure 3. Heptanoic acid flux as affected by diet and time for surface-applied manure under (a) saturated soil water conditions on day 1 and (b) wet soil water conditions on day 2 and for incorporated manure under (c) saturated soil water conditions on day 1 and (d) wet soil water conditions on day 2 (● = 0% WDGS, ○ = 10% WDGS, and ▲ = 30% WDGS).

(0.206 μg m^2 min^{-1}), and valeric acid (0.177 μg m^2 min^{-1}); aromatics including 4-methylphenol (0.056 μg m^2 min^{-1}) and indole (0.017 μg m^2 min^{-1}); and sulfides including DMDS (0.870 μg m^2 min^{-1}) and DMTS (0.187 μg m^2 min^{-1}).

In the present study, manure obtained from cattle diets containing 0%, 10%, or 30% WDGS was also applied to meet the one-year N requirement for corn. However, water was applied continuously for 24 h immediately following manure application on day 1. Principal odorous VOC emissions (saturated condition, table 5 of this study) were VFA including heptanoic acid (0.038 μg m^2 min^{-1}), hexanoic acid (0.289 μg m^2 min^{-1}), isobutyric acid (0.038 μg m^2 min^{-1}), and isovaleric acid (0.016 μg m^2 min^{-1}); aromatics including indole (0.008 μg m^2 min^{-1}) and skatole (0.0005 μg m^2 min^{-1}); and sulfides including DMDS (0.219 μg m^2 min^{-1}) and DMTS (0.050 μg m^2 min^{-1}).

A 24 h period of rainfall following manure application (day 1, table 5 of this study) produced substantially different types and amounts of VOC emissions than a single rainfall event occurring 24 h after land application (day 2, table 5 of Woodbury et al., 2014). For the principal VOC that were the same in both studies, the emissions reported by Woodbury et al. (2014) were greater. Greater concentrations of VOC constituents may have been present at the time of application in the manure collected by Woodbury et al. (2014). Emissions of VOC constituents in both studies were greater with surface application than with incorporated manure, except for heptanoic acid. With the exception of sulfides, which can be produced under anaerobic conditions, emissions of VOC were greater during the first 24 h after manure application.

Woodbury et al. (2015) conducted a laboratory study to evaluate the effects of pen location, soil water content, and temperature on VOC emissions from surface materials obtained from feedlot pens where cattle were fed a diet containing 30% WDGS. Surface materials were collected from the bunk, mound, and drainage areas of the pens, mixed with water to represent dry, wet, or saturated conditions, and then

incubated at temperatures of 5°C, 15°C, 25°C, and 35°C. Mean odorous VOC emissions measured by Woodbury et al. (2015) and those reported in the present study (shown in parentheses) were: VFA including heptanoic acid 0.042 (0.034) μg m^{-2} min^{-1} and hexanoic acid 0.334 (0.258) μg m^{-2} min^{-1}; aromatics including indole 0.003 (0.005) μg m^{-2} min^{-1} and skatole 0.0006 (0.0004) μg m^{-2} min^{-1}; and sulfides including DMDS 2.29 (0.135) μg m^{-2} min^{-1} and DMTS 0.170 (0.030) μg m^{-2} min^{-1}. Except for indole, the mean emissions measured by Woodbury et al. (2015) were greater than values obtained in this study. The larger VOC emissions reported by Woodbury et al. (2015) are attributed to greater amounts of VOC in the manure, which was derived from a single diet containing 30% WDGS.

Woodbury et al. (2016) also measured VOC emissions after land application of manure from cattle fed a diet of 30% WDGS. Manure was either surface-applied or incorporated at rates required to meet the 0.5, 1, or 2 year N requirements for corn. Approximately 24 h after manure application, a single application of water was added to the experimental sites until runoff occurred. Mean odorous VOC emissions measured by Woodbury et al. (2016) and those reported in the present study (shown in parentheses) were: VFA including hexanoic acid 0.369 (0.258) μg m^{-2} min^{-1} and isovaleric acid 0.028 (0.014) μg m^{-2} min^{-1}; aromatics including indole 0.003 (0.005) μg m^{-2} min^{-1}; and sulfides including DMDS 0.412 (0.135) μg m^{-2} min^{-1} and DMTS 0.067 (0.030) μg m^{-2} min^{-1}. If the VOC concentrations before manure application, climate factors, and other variables are assumed to be similar, the larger VOC emissions measured by Woodbury et al. (2016) are thought to be influenced by the greater manure application rates.

Conclusions

VOC emitted following land application of beef cattle manure may be a potential concern. VFA with notable contributions to total OAV included heptanoic, hexanoic, isobutyric, and isovaleric acids, with values of 23.5%, 17.6%, 9.26%, and 3.39% (0.034, 0.258, 0.030, and 0.014 μg m^{-2} min^{-1}), respectively. The two aromatics that contributed substantially to total OAV were indole and skatole, with values of 14.7% and 8.84% (0.005, and 0.0004 μg m^{-2} min^{-1}), respectively. The sulfides (DMDS and DMTS) contributed 9.50% and 5.68% (0.013 and 0.030 μg m^{-2} min^{-1}), respectively, to total OAV.

Incorporation of manure following land application reduced the flux values of each of the important VOC odor contributors, except heptanoic acid. The DMDS and DMTS emissions of 0.328 and 0.074 μg m^{-2} min^{-1}, respectively, for the 30% WDGS diet were substantially greater than the emissions measured for the 0% and 10% WDGS diets. Flux values were greater on the first day following land application (saturated soil water condition) than on the second day (wet soil water condition). For example, the skatole emission of 0.0005 μg m^{-2} min^{-1} measured for under saturated soil water conditions was significantly greater than the emission of 0.0002 μg m^{-2} min^{-1} measured under wet conditions.

From this study and the results reported by Woodbury et al. (2014), VOC emissions may be influenced by diet, application method, soil water content, and time since manure application. An effective best management practice for reducing VOC emissions is to incorporate beef cattle manure soon after application. If sulfide compounds are expected to be substantial contributors to OAV, then diets containing 30% WDGS or greater should be avoided.

The emission rates for the largest contributors to OAV occurred under saturated soil water conditions. The frequency and duration of precipitation events on land application sites cannot be controlled. However, delaying land application when there is a high probability of rainfall would reduce the potential of large VOC emissions. In this study, VOC emissions decreased substantially by the end of the test period. Therefore, off-site transport of VOC can be reduced if manure application occurs on days when wind speeds soon after manure application are minimal. Potential odor concerns may be diminished by employing a combination of best manure management and land application practices.

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NOMENCLATURE

AFO = animal feeding operation
DMDS = dimethyl disulfide
DMTS = dimethyl trisulfide
OAV = odor activity value
SCOT = single-compound odor threshold
VFA = volatile fatty acid
VOC = volatile organic compounds
VSC = volatile sulfur compounds
WDGS = wet distillers grains with solubles