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Odor and Chemical Emissions from Dairy and Swine Facilities: Part 1—Project Overview and Collection Methods

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Abstract
Livestock facilities have received numerous criticisms due to their emissions of odorous air and chemicals. Hence, there is a significant need for odor emission factors and identification of principle odorous chemicals. Odor emission factors are used as inputs to odor setback models, while chemical emission factors may be compared with regulations to demonstrate possible health impacts.

Additional measurements were incorporated into the National Air Emissions Monitoring Study (NAEMS) to establish odor and chemical emission factors for confined animal feeding operations. This investigation was conducted by the University of Minnesota, Iowa State University, West Texas A&M University, and Purdue University. The project objectives were to: 1) determine odor emission rates using common protocols and standardized olfactometry methods, 2) develop a chemical library of the most significant odorants, and 3) correlate the chemical library with olfactometry results.

This paper describes the sampling and evaluation methods for the odor and chemical measurements at two freestall dairy farms, one sow (gestation and farrowing) site, and one finishing pig facility. Odor and chemical samples were collected in Tedlar™ bags and sorbent tubes, respectively at barn inlet and exhaust locations using the sophisticated NAEMS gas sampling systems. Quality assurance protocols including inter-laboratory comparison tests are also discussed. The inter-lab sessions were designed to identify variations between olfactometry labs. While differences between olfactometry labs were observed, the variations appeared random and the odor data are considered reliable.

Keywords
Animal feeding operation, odor, chemical, emission, methods

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ODOR AND CHEMICAL EMISSIONS FROM DAIRY AND SWINE FACILITIES: PART 1 – PROJECT OVERVIEW AND COLLECTION METHODS

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ABSTRACT

Livestock facilities have received numerous criticisms due to their emissions of odorous air and chemicals. Hence, there is a significant need for odor emission factors and identification of principle odorous chemicals. Odor emission factors are used as inputs to odor setback models, while chemical emission factors may be compared with regulations to demonstrate possible health impacts.

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This paper describes the sampling and evaluation methods for the odor and chemical measurements at two freestall dairy farms, one sow (gestation and farrowing) site, and one finishing pig facility. Odor and chemical samples were collected in Tedlar™ bags and sorbent tubes, respectively at barn inlet and exhaust locations using the sophisticated NAEMS gas sampling systems. Quality assurance protocols including inter-laboratory comparison tests are also discussed. The inter-lab sessions were designed to identify variations between olfactometry labs. While differences between olfactometry labs were observed, the variations appeared random and the odor data are considered reliable.

KEYWORDS. Animal feeding operation, odor, chemical, emission, methods

INTRODUCTION

Livestock facilities have long been the target of criticisms and complaints from people working and living near them, due to their emissions of odorous air and chemicals and the resulting potential health implications. A National Research Council report (2003) further stressed the important adverse impacts of odorous emissions (i.e. public annoyance, nuisance lawsuits) on the surrounding local community. From this, a significant need was realized for baseline odor emission rates from livestock facilities and identification of the principle chemicals in the annoying odorous air. These emission rates are used as inputs to odor setback models, which recommended distances between facilities and the surrounding neighbors based on odor risk.

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In 2007, the nationwide, 24-month National Air Emissions Monitoring Study (NAEMS) study was launched to provide accurate representation of livestock barn exhaust/air flow, gaseous chemical and particulate matter measurements, and identification of diurnal/seasonal trends for 14 confined animal feeding operations (CAFOs) in the egg layer, broiler, dairy, and swine production industries (Heber, et. al., 2008). The overall goal was to establish representative emission rates for livestock production and provide the U.S. Environmental Protection Agency (USEPA) with a scientific basis for the appreciation of existing air pollution regulations on livestock facilities.

While odor nuisance is not addressed by USEPA regulations, it is important at the state and local levels. An add-on study to NAEMS was therefore conducted to measure odor emission rates and identify key chemicals associated with CAFOs. This involved collecting a series of odor samples from ventilation inlet and outlet locations, similar to the study reported by Jacobson et al. (2002), and simultaneous chemical samples from the same locations. The goals of this study were to:

1. Determine odor emission rates at four NAEMS sites using common protocol and standardized olfactometry for use in air dispersion models and evaluation of controls.
2. Develop a comprehensive chemical library that delineates the most significant odorants.
3. Correlate the observed chemical analysis with olfactometry results.

This paper is part one of a five-paper series and presents the sampling methods and results of interlaboratory comparison tests for olfactometry facilities. Part 2 focuses on odor emissions as measured using triangular forced-choice olfactometry. Part 3 discusses the VOC concentrations/emissions as measured by the GC/MS-Olfactometry (GC/MS-O). Part 4 details the correlations between the sensory (olfactometry) and chemical measurements while part 5 presents correlations between GC/MS-O sensory data and chemical measurements.

FARM DESCRIPTIONS

Odor and associated trace chemicals were sampled from November 2007 to May 2009 at four of the 14 NAEMS sites (WI5B-dairy, IN5B-dairy, IN3B-finishing pigs, and IA4B-sow). The characteristics of these sites are summarized in Table 1:

Table 1. Barn and management characteristics of NAEMS sites tested for odor and trace chemicals.

		WI5B	IN5B	IN3B	IA4B
Barn type	Barn 1&2 Barn 3	Freestall	Freestall	Finishing[1]	Gestation Farrowing[2]
Barn capacity	Barn 1&2 Barn 3	275 / 375	1500-1700	1000[1]	1100 24[2]
Bedding / floor type	Barn 1&2 Barn 3	Pine shavings / Sand[3]	Digested manure	Slatted	Slatted Iron/plastic
Ventilation type	Barn 1&2 Barn 3	Crossflow	Tunnel	Tunnel	Tunnel Mechanical
Number of fans (pit fans)	Barn 1&2 Barn 3	59 & 66	76	4 / 1 (3)	20 (9) 2 (1)
Fan diameter (pit fan), cm	Barn 1&2 Barn 3	130	140	120 / 90 (60)	120 (60) 60 (25)
Barn dimensions, m	Barn 1&2 Barn 3	93 x 28 & 107 x 30	472 x 29	61 x 12	86 x 25 21.3 x 6.5
Manure removal system	Barn 1&2 Barn 3	Flush / scrape[3]	Scrape	Deep pit	Deep pit Pull plug
Manure removal frequency	Barn 1&2 Barn 3	8 h	8 h	180 d	180 d 24 d

[1] Barns 1 and 2 at IN3B correspond to two rooms in the same 4-room finishing barn
[2] Barn 3 at IA4B corresponds to one room in a 16-room farrowing barn
[3] WI5B manure management system changed from flush to scrape in Sept. 2008

LABORATORY IDENTIFICATION

Three olfactometry laboratories and a single chemical lab were utilized to provide the odor and chemical analyses. These facilities are identified as the following:

Table 2. Odor and chemical laboratory details.

Lab ID	Type	Sites Analyzed	University	Reference
O1	Olfactometry	WI5B / IN3B	Minnesota	Jacobson, et.al (2008)
O2	Olfactometry	IN5B / IN3B	Purdue	Lim, et.al (2004)
O3	Olfactometry	IA4B	Iowa State	ISU (2005)
C1	Chemical	All	Iowa State	Zhang, et. al (2010)

METHODS

Sampling

Odor and chemical samples were collected, at each site listed in Table 1, approximately every two weeks for 52 weeks during a 17 months span of time, beginning in November 2007. Four rounds of sampling occurred at each site that lasted 13 weeks per round, with six sampling events for each site per round. Additionally, one interlab comparison (IC) sampling event occurred once at each site during the 13th week of a round, for a total of 25 events per site. Odor samples were collected through a positive-pressure bleed valve on a gas sampling system (GSS) that included Teflon™ sampling lines and pump diaphragm and Teflon™-lined stainless steel control solenoids. For most sampling events, a flow-splitting Teflon™ manifold was also utilized. Chemical samples were pumped through a line connection in the side of the GSS. Sampling locations from each site were chosen to represent the background inlet (or ambient) air and the ventilation barn exhaust air at each site. Selection of all sampling locations for the GSS was controlled by a computerized data acquisition program (Ni et al, 2009).

The odor samples were collected and transported for analysis via 0.05-mm thick Tedlar™ bags (10L size) with polypropylene fittings. Flow rates into the bags were measured with a flow calibrator (Gilibrator-II, Sensidyne, LP, Clearwater, FL) before and after sample collection, and adjusted as needed. The chemical laboratory utilized sorbent tubes that were double passivated, 304-grade stainless steel tubes packed with 65 mg of Tenax TA. Each tube was sampled with a pocket pump (SKC Part No. 210-1002). The flow rate (70 ml/min) through the sorbent tubes was monitored during sample collection with a low-flow bubble meter connected at the tube outlet, in series with the pocket pumps.

Each sampling event consisted of eight odor samples collected among the representative inlet and exhaust sampling locations. There were two inlet samples and three barn exhaust samples per barn (total of six barn samples) at sites WI5B, IN5B, and IN3B. At IA4B, there were two inlet samples, two exhaust samples from a farrowing room, and four exhaust samples from the gestation barns (total of six barn samples). Each event also included chemical sampling with sorbent tubes, one per sampling location. The chemical samples were drawn simultaneously or in parallel with the odor samples at each site, and usually included breakthrough sorbent tubes. Sampling with three (four from IA site) sorbent tubes occurred every other time odor samples were taken. Hence, chemical results existed for 50% of the sampling events.

Initially, and for the first six sampling events at each site (i.e. the first round of collection), two sampling regimes (A and B) were implemented. These regimes corresponded to the bi-weekly routine of the sorbent tube collections. A third sampling regime (C) was employed after the first sampling round, and was maintained for the remaining three rounds (39 weeks) of sample collection. During regime C, each site attached a Teflon™ manifold to their GSS bypass valve connection for improved collection of the sorbent tubes in parallel with the odor sampling. Additionally, duplicative or triplicative odor bag samples from a given location were collected simultaneously (with replication) through a Teflon™ manifold. The collected samples, flow rates, collection style, and sample period are summarized in Table 3 for each of the sampling regimes.

Table 3. Characteristics of the three sampling regimes.

Regime	Sites	Sample media	Flow rate, ccm	Collection mode	Sampling period, min
A	WI5B & IN5B	Bags (inlet / barn)	450 / 660	Sequential	15 / 10
	IN3B				
	IA4B	Bags (all)	300	Sequential	15
B	WI5B & IN5B	Bags (inlet / barn)	225 / 330	Sequential	30 / 20
	IN3B	Sorbent tubes	70		60
	IA4B	Bags (all)	220	Sequential	30
		Sorbent tubes	70		60
C	WI5B & IN5B	Bags (inlet / barn)	220 / 330	Simultaneously	60
	IN3B	Sorbent tubes	70		60
		(every other week)			
	IA4B	Bags (all)	100	Simultaneously	60
		Sorbent tubes	70		60
		(every other week)			

Every 13 week round of sampling concluded with an IC. One IC was conducted for each of the four sampling sites, and was analyzed as a quality control measure of each olfactometry laboratory (see ‘Quality Control and Assurance Methods’). During each IC, the odor samples were collected into bags in triplicate, resulting in a total of six inlet and 18 exhaust samples. These samples were divided randomly into three sampling sets comprised of eight samples, with two from the inlet location and six from the barns (and with at least one sample per barn). Each sample set was then distributed to each olfactometry laboratory. The first IC occurred at the Wisconsin dairy (WI5B). Three sets of samples were collected in parallel using the 4-port Teflon™ manifold, which was also used later for all odor samples. However, the limited number of ports on the manifold restricted sampling and the nine samples were drawn as three sequential sets. This resulted in sampling times of 30 min for every three inlet samples and 20 min for every three barn exhaust samples. A larger 10-port Teflon™ manifold was developed by the Purdue University sampling team for simultaneous odor sampling during the final three IC events.

Olfactometry

Odor samples were evaluated within 30 h of sample collection with a commercial olfactometer (AC'SCENT® International Olfactometer, St. Croix Sensory, Inc., MN). This olfactometer was operated in accordance with United States (ASTM, 1997) and European (CEN, 2001) standards. The odor assessment procedure included dynamic, triangular, forced-choice olfactometry, with a panel of eight or more trained assessors. The starting dilution level was chosen below and incrementally increased to the panelist’s detection threshold (DT). The panel’s average (geometric mean) of the individual panelists’ DTs provided a measure of a sample’s odor concentration (odor units per cubic meter, OU/m^3) and represented the sample’s odor emission (CEN, 2001). Another measure, the European odor unit (OU_E), was reported, wherein the panel’s average concentration was normalized by individual panelists’ sensitivity to a standard mixture of n-butanol in air.

Panelists were screened to determine if their sensitivity to a standard odor (n-butanol) was within the “normal” range. To ensure that panelists maintained their “normal” sensitivity without excessive variability, their DTs for 40 ppb n-butanol were tracked over time. Each panelist’s running average (during the last 20 samples) was required to lie between 20 and 80 ppb, otherwise the panelist’s results were disqualified. A final quality assurance strategy for panelists’ sensitivity, defined by the CEN standard, required that no sample response be accepted into a data set if the log standard deviation of a panelist’s individual DT for the sample varied more than ± 2.3 (McGinley and McGinley, 2006).

Three other quality assurance procedures were used in this study. First, while traditional triangular-forced-choice olfactometry ceases evaluation once the panelist correctly recognizes the

odorous air stream, this study continued evaluations until three consecutive correct responses were given. This strategy was chosen so that subjective measurements of odor (intensity, character, hedonic tone) were made at a high enough odor concentration for the panelist to draw definitive assessments of the odor. A second analysis procedure standardized the hedonic tone scale to -4 to +4, with 0 being neutral, so that they all utilized the same scale. Lastly, the mixtures of n-butanol in water for evaluating odor intensity were common to all three labs, as defined by the CEN standard.

Chemical Analysis

The chemical lab analyzed for the following fifteen common chemical species: acetic acid, propanoic acid, isobutyric acid, butyric acid, isovaleric acid, valeric acid, hexanoic acid, phenol, p-cresol, 4-ethylphenol, 2-aminoaceto phenone, indole, skatole (3-methylindole), heptanoic acid, and guaiacol.

All sorbent tubes were conditioned by thermal desorption (260 °C for 5 h) with nitrogen at 100 mL/min and background chromatograms were investigated for cleanliness. For re-used tubes, sufficient cleanliness was found with a pre-conditioning of 260 °C for 30 min. Sorbent tubes were shipped in a cooler with ice packs and temperatures were recorded upon delivery. The tubes were analyzed with an ATD inlet (Microanalytics Model 3200, Round Rock, TX, USA) for the Agilent 6890 GC and a Microanalytics multidimensional GC–MS with Olfactometry. The general GC run parameters used were as follows: injector, 260 °C; FID, 280 °C, column, 40 °C initial, 3 min hold, 7 °C/min, 220 °C final, 10 min hold; carrier gas, GC-grade helium. Odor results were collected from a trained human panelist for the separated VOC's through the GC-MS-O sniff port. Due to the targeting of odorants in the samples, tube evaluations were conducted within two different concentration ranges. For each concentration range, a six-point calibration curve was developed using standard solution mixtures (Zhang et al., 2010).

QUALITY CONTROL AND ASSURANCE METHODS

Quality control measures were implemented by each lab to ensure data reliability and comparability of results among the laboratories. Analysis procedures and sampling protocols were harmonized among labs. For example, all olfactometry labs documented panelist sensitivity using 40 ppm n-butanol and collected samples with true replication. In addition, inter-lab comparison sampling events were conducted for direct comparison of results from the olfactometry labs.

Olfactometry Inter-laboratory Comparison (IC) Tests

The IC tests were conducted at the end of each 13-week sampling round. Each olfactometry laboratory evaluated a set of eight co-located samples from each site. In each IC event, the panel average dilution-to-threshold (DT) for the eight odor samples and n-butanol standard was compared among labs. Inter- and intra-lab comparisons were made using standard comparative methods (ASTM, 2009). Additional olfactometry results can be found in Akdeniz et. al (2010).

The panel's geometric average DT's for each sample are presented in Table 2. The sample number corresponds to the order that samples were taken at WI5B (Inlet, Barn 1, Barn 2), IN5B (Inlet, Barn 1, Barn 2), IN3B (Inlet, Barn 1, Barn 2), IA4B (Inlet, Barn 1, Barn 2, Barn 3) and the n-butanol standard. From these data, the reproducibility standard deviation, replication standard deviation, *h*-consistency, and *k*-consistency statistics were calculated for each sample. As described in ASTM E691-09 (ASTM, 2009), measure of data consistency from an interlaboratory study is achieved by examining the consistency of a test result between labs (*h*-value) and the consistency of within-lab precision between labs (*k*-value). Hence, the data was tested against the critical values for *h*- and *k*-parameters at the 0.5% significance level, as given in the E691-09 standard. For *h*-statistics, the critical value was determined from an unpaired t-test based on the number of labs in the study. Similarly, the critical values of *k*-statistics were calculated from an F-

ratio based on the number of labs in the study and the number of replicates per sample. Due to the difference in number of sample replications, the critical *k*-values were 1.72, 1.67, and 1.61 for inlet/IA4B barns (two replications), barns (WI5B, IN5B, and IN3B, three replications), and n-butanol samples (four replications), respectively. The *h*-parameters were ±1.15 due to three participating labs. Plots of the *h*-values per lab and per sample are given in Figure 1.

Table 2. Odor concentrations (OU/m^3) from the O1, O2, and O3 labs per site, per sample, and for n-butanol (n-b) from the IC events with replications identified.

		Sample Number												
		WI5B			IN5B			IN3B			IA4B			
Lab	Rep	1	2	3	4	5	6	7	8	9	10	11	12	13
O1	1	116	409	229	24	76	44	33	214	179	108	3323	3922	2546
	2	189	414	2023	40	82	51	39	195	179	109	5038	5105	1166
	3			462		82	44		253	195				
	n-b	799			719			656			786			
O2	1	556	70	303	58	128	128	111	625	525	282	6539	5463	555
	2	76	303	777	53	117	117	103	525	525	219	4146	6539	603
	3		189	191		117	149		525	483				
	n-b	267			646			675			657			
O3	1	1431	34	87	41	1271	61	76	283	347	61	4456	843	349
	2	41	1271	61	34	501	2577	126	283	274	98	5055	2634	391
	3		501	2577		87	310		244	314				
	n-b	310			1431			1125			309			

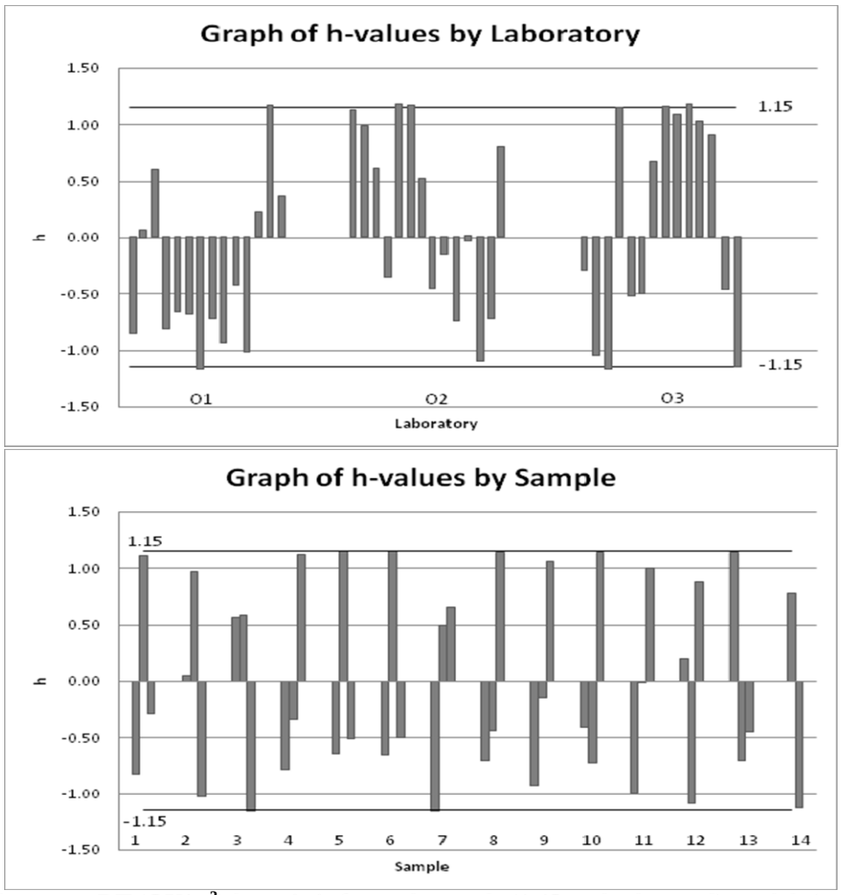

Figure 1: Panel average DT (OU/m^3) *h*-statistic for each sample (1-13 and n-butanol) per lab (top) and for each lab (O1-O3, left to right) per sample (bottom).

According to the interlaboratory comparison standard, laboratory *h*-values provide a measure of how each lab performs on a sample-to-sample basis as compared with combined laboratory data. Similarly, lab *k*-values measure the within-lab imprecision between replicate samples. The general pattern of the lab *h*-graph (Figure 1, left) indicates that O1 tended to have more negative samples and O3 more positive samples, as compared with the combined lab average. The plot also indicates that the number of negative lab samples is roughly equal to the number of positive lab samples for the entire data set. Collectively, these observations do not indicate any lab needing extra investigation for errors and that all labs experienced some degree of expected variability. Investigation of the lab *k*-values indicates that O1 had one sample, O2 had 5 samples, and O3 had no samples approaching or exceeding the threshold. Similarly, all labs had very few samples (one to five) approaching zero. Collectively, these observations show that none of the labs had a majority of samples (14 in total) near the zero or critical value. This indicates that each lab performed individually with reasonable variability and no evidence was provided that lab procedures were not comparable.

Investigations of the lab *h*- and *k*- values provided information about individual samples that may need further attention for discontinuity. Both *h* and *k* for odor concentration (OU/m^3) agreed that four samples were just at or above the critical value and in need of further review. These samples corresponded to samples taken from: IA4B-Barn 3/O1, WI5B-Barn 1/O2, IN5B-Barn 1/O2, and IN5B-Barn 2/O2. Review of field notes for the sample collections showed no marked difference in sample continuity for these particular samples. Hence, the samples were retained within the data set and considered reliable. A similar sample *h*- and *k*- value investigation was made using European odor units (OU_E/m^3) to determine the effects of panel sensitivity between the three labs. The analysis showed that the sample *h* and *k* agreed that one sample was in need of further investigation: IN5B-Barn 1/O2. As stated, the collection of this particular sample did not indicate obvious discontinuity from the other samples, and the data was retained.

CONCLUSIONS

From November 2007 to April 2009, 100 odor and chemical sampling events occurred at four National Air Emissions Monitoring Study sites – WI5B, IN5B, IN3B, and IA4B. Each sampling event involved a series of eight odor and three chemical samples, collected with a computer-controlled gas sampling system (GSS). The odor samples were collected into Tedlar™ bags, while sorbent tubes were used to collect odor-associated organic chemicals. One chemical and three olfactometry labs were involved in the analysis of these samples.

This study aimed to achieve continuous laboratory comparability and quality assurance by using a uniform set of sampling procedures for all four livestock facilities. This included taking odor and chemical samples with replication, taking comparable chemical samples bi-weekly, collecting three sets of odor samples every 13 weeks for inter-lab olfactometry comparisons, and ensuring odor samples were evaluated within 30 h of collection. In addition to the sample collection, the olfactometry lab-analysis procedures were also standardized. This was founded primarily on the basis of internationally accepted panel selection and monitoring principles, but also consisted of evaluations of odor intensity and hedonic tone at the third correct detect or recognition response.

Evaluations of results per-lab following standard comparative methods showed a reasonable amount of variability between and within the three labs. Per-sample statistical comparisons were also made from odor concentrations (both OU/m^3 and OU_E/m^3) to determine the amount of variability between labs due to panel sensitivity. Of 42 odor samples, only one was highlighted for investigation and the variability between and within labs due to panel sensitivity was considered inherent. Statistical evaluation for each lab versus sample location indicated a slight interaction between lab and sample location. It was determined that the sampling and analysis procedures were comparable between labs and all data were reliable.

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