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Increased sensitivity of OSHA method analysis of diacetyl and 2,3-pentanedione in air

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Abstract

Gas chromatography/mass spectrometry (GC/MS) operated in selected ion monitoring mode was used to enhance the sensitivity of OSHA Methods 1013/1016 for measuring diacetyl and 2,3-pentanedione in air samples. The original methods use flame ionization detection which cannot achieve the required sensitivity to quantify samples at or below the NIOSH recommended exposure limits (REL: 5 ppb for diacetyl and 9.3 ppb for 2,3-pentanedione) when sampling for both diacetyl and 2,3-pentanedione. OSHA Method 1012 was developed to measure diacetyl at lower levels but requires an electron capture detector, and a sample preparation time of 36 hours. Using GC/MS allows detection of these two alpha-diketones at lower levels than OSHA Method 1012 for diacetyl and OSHA Method 1016 for 2,3-pentanedione. Acetoin and 2,3-hexanedione may also be measured using this technique. Method quantification limits were 1.1 ppb for diacetyl (22% of the REL), 1.1 ppb for 2,3-pentanedione (12% of the REL), 1.1 ppb for 2,3-hexanedione, and 2.1 ppb for acetoin. Average extraction efficiencies above the limit of quantitation were 100% for diacetyl, 92% for 2,3-pentanedione, 89% for 2,3-hexanedione, and 87% for acetoin. Mass spectrometry with OSHA Methods 1013/1016 could be used by analytical laboratories to provide more sensitive and accurate measures of exposure to diacetyl and 2,3-pentanedione.

Keywords

2,3-pentanedione; diacetyl; increased sensitivity; mass spectrometry; OSHA Methods 1013/1016

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Introduction

Exposure to diacetyl and 2,3-pentanedione have been associated with development of obliterative bronchiolitis in animal studies.^[1] Sentinel cases of this illness were first reported in microwave popcorn facilities by researchers at the National Institute for Occupational Safety and Health (NIOSH).^[2] Most recently, investigations have been conducted at coffee processing facilities to assess alpha-diketone emissions from roasted coffee and flavoring coffee.^[3] NIOSH proposed a Recommended Exposure Limit (REL) of 5 ppb as a time-weighted average (TWA), and a 15-min short-term exposure limit (STEL) of 25 ppb, for occupational exposure to diacetyl. NIOSH also proposed a TWA of 9.3 ppb, and a STEL of 31 ppb for 2,3-pentanedione exposure. In 2012 the American Conference of Governmental Industrial Hygienists (ACGIH) recommended a threshold limit value-time-weighted average (TLV-TWA) of 10 ppb, and a threshold limit value-short-term exposure limit (TLV-STEL) of 20 ppb, for exposure to diacetyl.^[4]

The initial NIOSH sampling and analytical methods for assessing occupational exposure to flavoring compounds were Methods 2557 for diacetyl and 2558 for acetoin.^[5,6] These methods specify the use of dual bed (150/75 mg) Anasorb[®] Carbon Molecular Sieve sorbent tubes to collect up to 10 L of air. Limits of quantitation (LOQ) are 56.8 ppb for diacetyl and 83.3 ppb for acetoin. Gas chromatography with flame ionization detection (GC/FID) is used for both methods, but the two methods specify different extraction solvents requiring separate analysis for each compound. NIOSH Method 2557 was later found to underestimate diacetyl exposure depending on the diacetyl concentration, sampling site humidity, and sample storage time.^[7]

Method PV2118 was the first method issued by the Occupational Safety and Health Administration (OSHA) for diacetyl.^[8] With this method two dual bed (150/75 mg) silica gel sorbent tubes, placed in series, are used to collect a 60-min 3 L sample. Analysis is performed by GC/FID with a LOQ of 280 ppb. Due to increased interest in occupational exposure to flavorings, OSHA validated three additional methods with lower quantitation limits and longer sampling times: OSHA Methods 1012 and 1013 were validated for sampling diacetyl and acetoin, and OSHA Method 1016 for 2,3-pentanedione.^[9–11] With these methods two single-bed (600 mg) silica gel sorbent tubes, placed in series, are used to collect 180-min 9 L TWA samples or 15-min 3 L STEL samples. Samples prepared using OSHA Methods 1013 and 1016 are extracted in 95:5 ethanol:water for one hour and then analyzed by GC/FID. Diacetyl and acetoin samples prepared using OSHA Method 1012 are extracted with 95:5 ethanol:water, derivatized with *O*-pentafluorobenzyl hydroxylamine hydrochloride, and analyzed by GC using an electron capture detector (ECD). The limits of quantitation for diacetyl and acetoin by Method 1012 are significantly lower than by Method 1013, but the sample derivatization step requires 36 hours and multiple derivatives of diacetyl (isomers) are formed.

Laboratories will likely face difficulty in quantifying diacetyl and 2,3-pentanedione at the proposed NIOSH RELs, using Methods 1013/1016 because these methods have LOQs above the RELs. To increase the sensitivity of OSHA Methods 1013/1016 without the need to derivatize the sample, NIOSH and OSHA independently assessed the use of an alternate

detector, mass spectrometry operated in selected ion monitoring (SIM) mode. This article describes the positive effects of using mass spectrometry on sensitivity of the method. The SIM modification to OSHA Methods 1013/1016 could be adopted by laboratories to quantify alpha-diketones at concentrations lower than the NIOSH RELs.

Methods

Sampling

No modifications were made to the sampling portion of the OSHA Methods (1013/1016). Target analytes for the original methods were diacetyl, acetoin, and 2,3-pentanedione. The LOQ for 2,3-hexanedione was not included in the original validation of OSHA Methods 1013/1016, but was investigated and included in the current study. Sampling is conducted at 50 mL/min for 3 hr or 200 mL/min for 15 min. Analytes are collected on two silica gel tubes (7×110-mm, 600 mg, Cat. No. 226-183, SKC, Inc., Eighty Four, PA) in series. Samplers should be protected from light by sample holders or aluminum foil. Storage performance is the same (17 days refrigerated storage with cold shipment to the laboratory) as shown in the OSHA Method 1016.

Analysis

At NIOSH, samples were extracted in 2 mL of 95:5 ethanol:water (ALC213-1PTP, Undenatured, ACS/USP/NF Grade, MG Scientific, Pleasant Prairie, WI) with 3-pentanone as the internal standard (ISTD) (0.007 µL/mL) as outlined in the OSHA Methods 1013/1016. At OSHA, samples were also extracted in the same way as outlined in methods 1013/1016. Samples were extracted on a rotator. Do not sonicate or extract with any method that may heat up the extracts.

Modification to OSHA Methods 1013/1016 included the use of a mass spectrometer (MS) instead of a flame ionization detector attached to a gas chromatograph (GC). At OSHA, samples were analyzed with an Agilent 7890A/5975C GC/MS system (Santa Clara, CA). At NIOSH, samples were analyzed with an Agilent 7890B/5977A GC/MS system (Santa Clara, CA). The NIOSH-modified and OSHA-modified method parameters are displayed in Table 1 alongside OSHA Method 1016 parameters. MS was operated in SIM mode for increased sensitivity. Quantification ions were m/z 86 for diacetyl, m/z 100 for 2,3-pentanedione, m/z 88 for acetoin, m/z 71 for 2,3-hexanedione, and m/z 86 for 3-pentanone (ISTD). Qualification ions were m/z 43 for diacetyl, m/z 57 for 2,3-pentanedione, m/z 45 for acetoin, m/z 114 for 2,3-hexanedione, and m/z 57 for 3-pentanone (ISTD).

Detector performance

Limit of quantitation—LOQs were assessed by OSHA and NIOSH independently using a mass selective detector. At OSHA, 10 samplers were spiked with equally descending increments of analyte from 0.0191–0.191 µg diacetyl/sample and from 0.0186–0.186 µg 2,3-pentanedione/sample. The highest sample loading is the amount spiked on a sampler that would produce a peak approximately 10 times the response of a sample blank (i.e., estimated LOQ) at the chromatographic retention time of the analyte. These spiked samples and a sample blank were analyzed and the data obtained were used to calculate the reliable

quantitation limit (RQL) (NIOSH calls this the LOQ) as per OSHA document “Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis.”^[12] The RQL is considered by OSHA to be the lower limit for precise quantitative measurements as long as 75–125% of the analyte is recovered. At NIOSH, seven samples were spiked with descending increments of analyte from 0.01–0.2 µg/sample to cover the range from less than the expected limit of detection (LOD) of 0.02 µg/sample to no greater than 10 times the expected LOD. The expected LOD is the mass of analyte which gives a mean signal three times the standard deviation of the mean blank signal. The LOQ is considered by NIOSH to be the lowest mass that can be reported with acceptable precision and is the larger of the calculated LOQ or the mass above which recovery is 75%.^[13]

Extraction efficiency—The extraction efficiency was evaluated by spiking four separate samples at seven concentrations: 0.06, 0.1, 0.25, 0.5, 1, 1.5, and 2 times the NIOSH REL for diacetyl (5 ppb target concentration). Samples were spiked with 10 µL of a working standard solution serially diluted from a stock 1 mg/mL solution containing all four analytes. The NIOSH LOQs for diacetyl, 2,3-pentanedione, and 2,3-hexanedione were between the 0.1× and 0.25× target concentration. Percentage recoveries were calculated based on triplicate neat standards at each level (sample response/average neat response*100%). Sample response was first corrected for average ISTD response of the neat standards by the ratio of sample ISTD to average neat ISTD response (sample response*average ISTD response of standards/sampler ISTD response). To simulate humidity effects, four wet samples were also prepared at the target concentration by first spiking the media with 140 µL of 18 MΩ water to mimic 9 L of 22.2°C at 80% RH (15.7 mg/L) air. The wet samplers were allowed to sit for three hours before spiking with analytes. Average extraction efficiencies and coefficients of variation (CVs) were calculated for samples above the NIOSH LOQ. Wet samples were not included in the average.

Precision of the analytical method—The precision of the analytical method was measured as the mass equivalent to the standard error of estimate (SEE).^[12] SEE was determined from the linear regression of instrument response to the standard concentrations covering 0.1–2 times the TWA target concentration for diacetyl (5 ppb for a 9 L air sample). SEE is synonymous with the standard error of the regression used by NIOSH.^[13] A calibration curve was constructed for each analyte based on three separate standard preparations at each concentration and ISTD-corrected response.

Results

Detector performance

Limit of quantitation—Limits of quantitation of the modified vs. original methods are shown in Table 2. Calculated LOQs are lower than the lowest concentration used in the assessment (0.01 µg/sample for diacetyl, 2,3-pentanedione, and 2,3-hexanedione; and 0.02 µg/sample for acetoin). All NIOSH-modified LOQs were based on the lowest concentration used. The NIOSH-modified method has the lowest quantitation limits (1.05 ppb for a 9 L air sample) for diacetyl and 2,3-pentanedione. Acetoin LOQ is between OSHA Method 1012

and OSHA Method 1013. 2,3-Hexanedione has a quantitation limit of 1.05 ppb. All linear regressions used to calculate analyte LOQs had correlation coefficients > 0.999 (Figure 1).

Extraction efficiency—Individual extraction efficiencies were generally within 10% of the average extraction efficiency for each concentration except at the 0.06× target concentration which was below the LOQ for the analyte (Table 3). Average extraction efficiencies above the LOQ were $100 \pm 3.4\%$ (mean \pm CV) for diacetyl, $92 \pm 3.5\%$ for 2,3-pentanedione, $89 \pm 2.5\%$ for 2,3-hexanedione, and $87 \pm 4.7\%$ for acetoin. Average extraction efficiencies for wet samplers were within 10% of the dry samplers except for acetoin (16% difference).

Precision of the analytical method—Calibration curves for each analyte show good adjusted $R^2 > 0.99$ (Figure 2). The standard error of estimates was 0.0034 μg for diacetyl, 0.0063 μg for 2,3-pentanedione, 0.0057 μg for 2,3-hexanedione, and 0.0099 μg for acetoin.

Discussion

NIOSH- and OSHA-modifications using GC/MS in SIM mode exhibited lower quantitation limits than previously reported in OSHA Methods 1013/1016 using a flame ionization detector. NIOSH-modified LOQs were 6.2–6.6 times lower than those reported by OSHA due to differences in mass spectrometer models and instrument method parameters. Agilent reported a drop-in signal-to-noise from the 5975C (used at OSHA) to 5977A (used at NIOSH) (600:1 to 1500:1 calculated with injection of 1 μL of 1 $\text{pg}/\mu\text{L}$ of octafluoronaphthalene analyzed in standard scan mode using ion 272). The LOQs were calculated using the least abundant ions, m/z 86 for diacetyl and m/z 100 for 2,3-pentanedione, which are more selective for these compounds since they are the molecular ions.

For both analytes, the ion at m/z 43 was used as the qualifier ion. The use of m/z 43 is somewhat problematic due to the high background from the sample extraction solution (i.e., ethanol m/z 43 ion). At concentrations around the detection limit, the qualifier ion ratio may not be used for confirmation of diacetyl in field samples. The analyst should take care to match retention times and compare peak shape of the sample to that of the closest standard when confirming identity. Further work could include investigating the use of GC/MS grade methanol for sample extraction. Methanol, with a molar mass of 32 g/mol , would not produce an interfering ion at m/z 43.

Average extraction efficiencies were greater than 90% for diacetyl and 2,3-pentanedione but slightly less than 90% for 2,3-hexanedione and acetoin. Humidity effects were assessed by comparing spiked wet samples at the target concentration to dry samples. Extraction efficiencies for wet samples were within 10% of the dry samples, except for acetoin, meaning the samples may be used in humid environments at these low concentrations. The target concentration was designed for diacetyl and 2,3-pentanedione as these analytes are known to cause respiratory disease. Acetoin may perform better under humid conditions if the target concentration was increased. Some extraction efficiencies seen here are more variable than those seen in OSHA Method 1013/1016 due to the low concentrations assessed

in this study. OSHA 1012 is more sensitive (48 vs. 67 ng/sample LOQ compared to the NIOSH modified method) and may be more appropriate when assessing lower concentrations of acetoin.

The precisions of the analytical methods are significantly improved using mass spectrometry in SIM mode than those calculated in the OSHA Methods. The SEE calculated here was 5.8 times lower for diacetyl (0.0034 µg vs. 0.019 µg) and 5.3 times lower than acetoin (0.0099 µg vs. 0.052 µg) compared to OSHA Method 1012. The SEE was 78 times lower for 2,3-pentanedione compared to OSHA Method 1016 (0.0063 µg vs. 0.49 µg).

Conclusions

A need existed for increased sensitivity for diacetyl and 2,3-pentanedione using OSHA Methods 1013/1016. NIOSH and OSHA independently assessed the use of an alternate detector to achieve this goal. Method modifications included using a mass spectrometer operated in SIM mode in place of a flame ionization detector. Limits of quantitation using a mass spectrometer are lower than the more sensitive OSHA Method 1012 for diacetyl and OSHA Method 1016 for 2,3-pentanedione, and the method does not require derivatizing the samples. Acetoin and 2,3-hexanedione may also be measured using this modified method. Mass spectrometry with OSHA Methods 1013/1016 could be used by analytical laboratories to provide more sensitive and accurate measures of exposure to diacetyl and 2,3-pentanedione.

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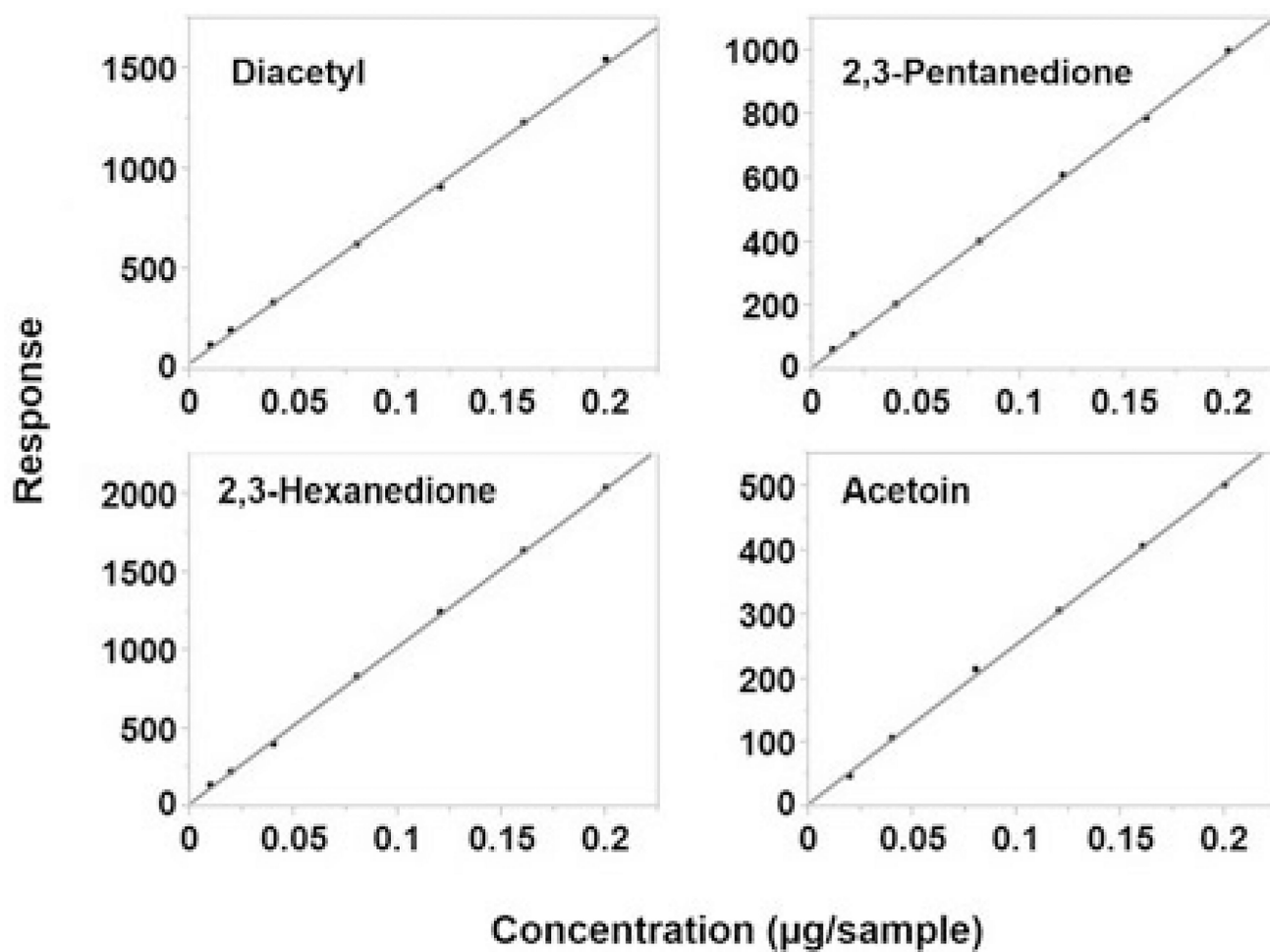


Figure 1.

LOQ linear regression plots for diacetyl ($y = 7444x + 39.9$), 2,3-pentanedione ($y = 4916x + 12.0$), 2,3-hexanedione ($y = 10052x + 27.7$), and acetoin ($y = 2503x + 6.56$).

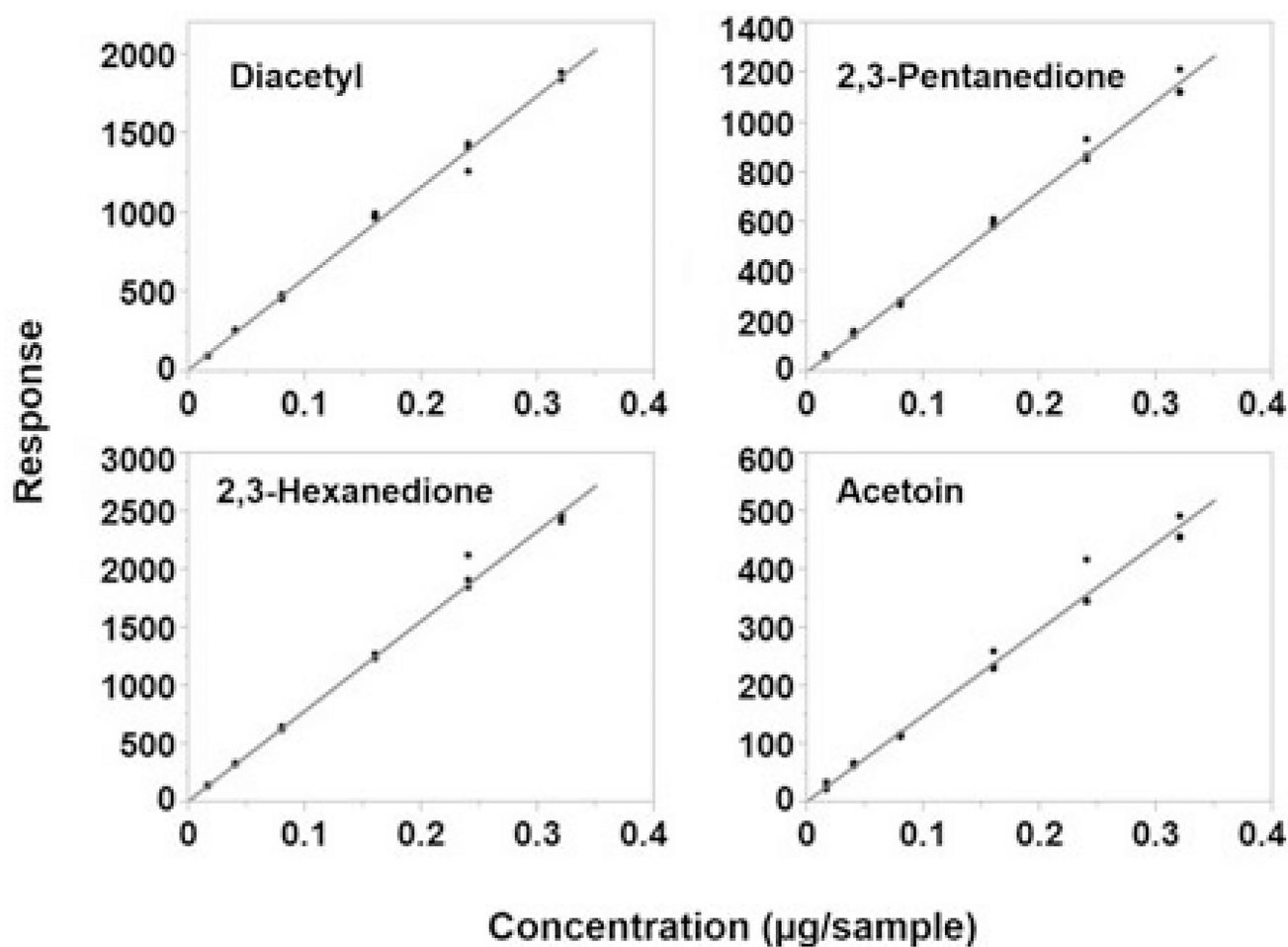


Figure 2. Calibration curves for diacetyl ($y = 5758x + 23.0$), 2,3-pentanedione ($y = 3619x + 4.9$), 2,3-hexanedione ($y = 7737x + 23.7$), and acetoin ($y = 1477z + 4.1$).

Table 1

Comparison of method conditions.

	NIOSH Modified	OSHA Modified	OSHA 1016
<u>GC column conditions</u>			
column:	Restek RTX1 60-m × 0.32-mm i.d. (1 µm df)	Agilent DB-624 UI 60-m × 0.32-mm i.d. (1.8 µm df)	Agilent DB-1 60-m × 0.32-mm i.d. (5 µm df)
flow:	1.2 mL/min (helium)	2.6 mL/min (helium)	1.8 mL/min (hydrogen)
column mode:	constant flow	constant flow	constant pressure (9.4 psi)
initial average velocity:	28.37 cm/s	41.8 cm/s	
<u>GC oven conditions</u>			
oven temperature:	60°C (hold 4 min), ramp to 110°C at 10°C/min (hold 1 min), ramp to 200°C at 40°C/min (hold 1 min)	60°C (hold 2 min), ramp to 120°C at 10°C/min (hold 0 min), ramp to 200°C at 25°C/min (hold 2.8 min)	60°C (hold 4 min), ramp to 150°C at 10°C/min (hold 5 min), ramp to 200°C at 20°C/min (hold 1 min)
run time:	13.25 min	14 min	21.5 min
<u>GC autosampler conditions:</u>			
injection volume:	1 µL	1 µL	1 µL
solvent wash:	95% Ethanol: water	95% Ethanol: water	
<u>GC inlet conditions</u>			
liner:	Restek Cat No. 21033 or equivalent low pressure drop liner w/ deactivated wool	Restek Cat No. 23309.1 or equivalent low pressure drop precision liner w/ wool	Agilent 5183-4647 or equivalent Liner w/ wool, split only, tapered, deactivated
temperature:	240°C	250°C	240°C
split ratio (flow):	10:1	10:1	2:1
septum purge:	3 mL/min (helium)	3 mL/min (helium)	
total flow:	16.2mL/min	31.6mL/min	
<u>Retention times</u>			
diacetyl:	5.67 min	5.61 min	10.0 min
2,3-pentanedione:	7.70 min	7.57 min	13.2 min
ISTD:	7.83 min	7.63 min	13.5 min
acetoin:	7.94 min		13.8 min
2,3-hexanedione:	9.80 min		
<u>Detector parameters</u>			
detector:	MS	MS	FID
mode:	EI	EI	
acquisition mode:	SIM	SIM	
solvent delay:	5.0 min	5.4 min	
EMV mode:	gain factor	gain factor	
gain factor:	1	15	
MS source:	300°C	250°C	
MS quad:	200°C	200°C	
MS transfer:	250°C	250°C	

	NIOSH Modified	OSHA Modified	OSHA 1016
dwel (ms)	100	100	
SIM parameters:	<u>mass (m/z)</u>	<u>mass (m/z)</u>	
	43	43.1	
	45		
	57	57.1	
	71		
	86	86.1	
	88		
	100	100	
	114		

Table 2

Limits of quantitation.

Analyte	Method	LOQ ^a (ng/sample)	9 L TWA sample (ppb)	3 L STEL sample (ppb)
Diacetyl	OSHA 1013	370	11.7	35
	OSHA 1012	41	1.3	3.9
	OSHA modified	97	3.1	9.2
	NIOSH modified	33	1.1	3.2
2,3-pentanedione	OSHA 1016	380	10.3	30.9
	OSHA modified	101	2.7	8.2
	NIOSH modified	33	1.1	3.2
2,3-hexanedione	NIOSH modified	33	1.1	3.2
Acetoin	OSHA 1013	350	11	30.9
	OSHA 1012	48	1.5	4.5
	NIOSH modified	67	2.1	6.3

^aLimit of Quantitation

Table 3

Extraction efficiency.

Compound	Type	×Target Concentration	Concentration (µg/sample)	Sample # Extraction Efficiency				
				1	2	3	4	Avg CV
diacetyl	dry	0.06	0.01	92	84	86	102	91 9.0
		0.1	0.016	95	106	96	104	100 5.7
		0.25	0.04	101	103	104	107	103 2.3
		0.5	0.08	97	102	100	100	100 2.0
		1	0.16	96	96	95	97	96 0.8
2,3-pentanedione	wet	1.5	0.24	103	100	103	102	102 1.5
		2	0.32	97	98	95	97	97 1.2
		1	0.16	92	96	95	94	94 1.7
		0.06	0.01	0	35	45	60	35 72
		0.1	0.016	78	92	86	97	88 9.1
	dry	0.25	0.04	88	86	86	88	87 1.2
		0.5	0.08	89	94	90	92	91 2.5
		1	0.16	91	92	94	92	92 1.5
		1.5	0.24	95	91	93	92	93 2.0
		2	0.32	95	94	95	98	95 1.9
2,3-hexanedione	wet	1	0.16	93	96	101	100	98 3.4
		0.06	0.01	33	35	47	56	43 25
	dry	0.1	0.016	89	92	96	91	92 3.3
		0.25	0.04	88	89	89	89	89 0.5
		0.5	0.08	91	90	93	88	90 2.2
acetoin	wet	1	0.16	90	90	88	88	89 1.2
		1.5	0.24	87	85	90	87	87 2.3
		2	0.32	92	93	90	94	92 2.1
		1	0.16	95	96	96	96	96 0.6
	dry	0.06	0.01	—	—	—	—	—
		0.1	0.016	67	85	78	91	80 13
		0.25	0.04	85	95	88	87	89 4.9

Compound	Type	×Target Concentration	Concentration (µg/sample)	Sample # Extraction Efficiency				Avg	CV
				1	2	3	4		
		0.5	0.08	93	92	90	88	91	2.1
		1	0.16	77	84	88	92	85	7.5
		1.5	0.24	86	84	82	84	84	1.7
		2	0.32	89	87	85	89	88	2.3
	wet	1	0.16	98	99	103	105	101	3.3