

# 2

## Assessing Occupational Exposure in Employees

### 2.1 Introduction

Measurement of diacetyl and 2,3-pentanedione exposure is helpful in preventing flavorings-related lung disease, even with complex flavorings formulations. Exposures to diacetyl and 2,3-pentanedione can be monitored using personal and area (environmental) air samples because the predominant route of exposure is inhalation. Results from air sampling can be compared with established criteria such as the NIOSH RELs. Measuring employees' exposures to diacetyl or 2,3-pentanedione may help identify processes, locations, or tasks with exposures of concern; guide corrective actions such as engineering controls; identify improved work practices; and select appropriate respiratory protection.

This chapter discusses (1) available sampling and analytical techniques for monitoring diacetyl and 2,3-pentanedione vapor in the workplace; (2) techniques for measuring diacetyl and 2,3-pentanedione in airborne dust and bulk materials; (3) real-time techniques for measuring relevant airborne analytes and other flavoring compounds; and (4) results of some occupational exposure assessments by NIOSH and others of facilities that use diacetyl and 2,3-pentanedione.

Many work environments have mixed exposures, with multiple chemical agents present. Although the primary focus of this criteria document is diacetyl and 2,3-pentanedione, other compounds can also be of concern. Depending upon the processes employed in a workplace, sampling should be conducted

for agents of concern to maintain safe work environments. Common sampling and analytical methods to determine concentrations of diacetyl and 2,3-pentanedione are presented in Appendices A–E.

### 2.2 Time-integrated Air Sampling and Analytical Methods for Diacetyl and 2,3-Pentanedione Vapor

Personal breathing zone sampling is the preferred approach for estimating employee exposure. For personal sampling, an employee wears the air sampling equipment, and the inlet to the collection medium is positioned within the employee's breathing zone. Area sampling is performed for several purposes such as to evaluate exposure characteristics associated with an area or process, and to determine the efficiency of control systems. While the same sampling equipment may be used in some cases for both personal and area sampling, area sampling is stationary, in contrast to personal sampling, which allows for mobility by accompanying the employee throughout the sampling period.

#### 2.2.1 OSHA Methods 1012 and 1013

In response to the need for longer sampling time periods with a lower limit of detection or reliable quantitation limit, the Occupational Safety and Health Administration (OSHA) validated two sampling and analytical methods, OSHA Method 1012 and OSHA Method 1013, for diacetyl and acetoin in 2008 [OSHA

2008a, b]. OSHA Method 1013 is for monitoring low ppm levels, while OSHA Method 1012 is for monitoring ppb levels [OSHA 2008b]. These methods can be used for the simultaneous determination of diacetyl and acetoin. As of the publication of this document, these are the recommended methods for diacetyl.

OSHA Methods 1012 and 1013 use two 600 milligram (mg) sorbent tubes containing specially cleaned and dried silica gel (SKC Inc., Eighty Four, PA, Catalog no. 226-183) in series and air is sampled at a flow rate of 50 milliliters per minute (mL/min) for up to 180 minutes for determination of TWA concentrations, and a flow rate of 200 mL/min for 15 minutes for short-term concentration measurements. An opaque sampling tube protective cover should be used in conjunction with the sampler to prevent the glass sampling tube from breaking and to protect the sample from light, which can decompose diacetyl and acetoin. After sampling, the tubes should be separated, capped, and protected from light with aluminum foil or other opaque material. There is no requirement that samples be kept cold during shipping or storage.

OSHA Method 1013 has a reliable quantitation limit of 12 ppb (0.041 mg/m<sup>3</sup>) diacetyl for a 9-liter sample, and samples are analyzed by gas chromatography using a flame ionization detector (GC-FID). OSHA Method 1012 has a nearly 10 times lower RQL of 1.3 ppb (4.57 micrograms per meter cubed [µg/m<sup>3</sup>]) diacetyl for a 9-liter sample, which is achieved by derivatizing diacetyl with 2 milligram per milliliter (mg/mL) O-(2,3,4,5,6-pentafluorobenzyl) hydroxylamine hydrochloride in the extraction solution and analyzing by gas chromatography using an electron capture detector (GC-ECD). An advantage of OSHA Method 1013 is that sample preparation can be performed in one hour, whereas the derivatization step of OSHA Method 1012 requires 36 hours. After samples have been extracted and analyzed

using OSHA Method 1013, if needed (e.g., if sample concentration is not detectable), they can be derivatized and analyzed using OSHA Method 1012 to benefit from its lower detection capability.

### 2.2.2 OSHA Method 1016

OSHA Method 1016 [OSHA 2010] can be used to measure 2,3-pentanedione concentrations. OSHA Method 1016 uses the same sampling media, sample collection procedure and analytical procedure as OSHA method 1013. However, OSHA Method 1016 allows for the simultaneous analysis of 2,3-pentanedione, diacetyl, and acetoin by using a different analytical column to optimize the analytical separation of these compounds. In addition, OSHA Method 1016 requires samples to be shipped cold. If diacetyl and/or acetoin are not anticipated to be present, OSHA Method 1016 can be used to sample for an additional 20 minutes, or 200 minutes, at 50 mL/min to determine TWA concentrations of 2,3-pentanedione [OSHA 2010]. For a 10-liter sample, the RQL of 2,3-pentanedione is 9.3 ppb (38 µg/m<sup>3</sup>).

### 2.2.3 OSHA Method PV2118

Superseded by OSHA Methods 1012 and 1013, OSHA Method PV2118 [OSHA 2003] was developed as an air sampling method for diacetyl that uses two 150/75 mg silica gel sorbent tubes in series (SKC Cat. No. 226-10) at a recommended flow rate of 50 mL/min for one hour. In response to the limited capacity of this sampler in humid environments, a modified version of OSHA Method PV2118 was used by some practitioners in the field. The modified method uses larger 400/200 mg sorbent tubes packed with specially cleaned silica gel (SKC Cat. No. 226-10-03) allowing for greater

sample capacity without breakthrough of diacetyl. Sample analysis remained unchanged.

#### 2.2.4 NIOSH Method 2557

While no longer recommended for use, NIOSH developed NIOSH Method 2557 [NIOSH 1994] for measuring diacetyl vapor in air. It called for the collection of samples onto a 150/75 mg carbon molecular sieve sorbent tube (Cat. No. 226-121, SKC Inc., Eighty Four, PA) at a flow rate between 10 and 200 mL/min for a sample volume between 1 and 10 liters. The method specifies that samples be stored cold and analyzed within 7 days of sampling.

Until 2007, NIOSH Method 2557 was the predominant air sampling and analytical method for diacetyl used in the field, but it is no longer recommended for use [Ashley et al. 2008]. In 2007, field and chamber investigations indicated that NIOSH Method 2557 was adversely affected by humidity, resulting in an underestimation of true diacetyl concentrations. To aid in the evaluation of sampling and analytical methods for diacetyl, a field comparison study between new and existing sampling collection methods was conducted [Ashley et al. 2008]. Side-by-side field samples were collected in flavor manufacturing facilities and analyzed according to NIOSH Method 2557, OSHA Method PV2118, and a modified version of OSHA Method PV2118. The results of this field work confirmed the tendency of NIOSH Method 2557 to underestimate the true concentration of diacetyl as humidity increases. However, no mathematical correlation was found in this data set which would produce an adjustment factor to allow for correction of results.

As a result, NIOSH researchers collaborated with scientists at the OSHA Salt Lake Technical Center laboratory to study the effects of humidity on measured diacetyl air concentrations using NIOSH Method 2557. This laboratory has

chamber facilities for the generation of known diacetyl air concentrations with the ability to control both temperature and relative humidity (RH). Controlled test atmospheres of diacetyl were generated and sampled through an array of sampling tubes at calibrated flow rates. Test atmospheres were controlled for diacetyl concentration, temperature, and relative humidity. Results indicated that diacetyl recoveries for NIOSH Method 2557 were affected by absolute humidity (AH), storage time of sample tube prior to extraction, and diacetyl air concentration. The study resulted in the development of a mathematical procedure to adjust diacetyl concentrations previously measured using NIOSH Method 2557. The procedure is presented in Appendix F and is also published elsewhere [Cox-Ganser et al. 2011].

#### 2.2.5 Other Air Sampling Method(s) in Development

Because of current interest in occupational exposure to flavoring compounds, new methods continue to be developed for their measurement. At this time, however, none of these methods are validated.

A method is being developed by NIOSH to measure alpha-dicarbonyl compounds (such as diacetyl and 2,3-pentanedione) in air via derivatization with 1,2-phenylenediamine. This compound is known to react with alpha-dicarbonyl compounds to form stable quinoxaline derivatives [Rodrigues et al. 1999]. In this method, air is sampled through a sorbent tube containing silica gel coated with 1,2-phenylenediamine at 0.1% by weight. Samples are extracted in the lab and extraction solutions analyzed by gas chromatography-nitrogen/phosphorus detection (GC-NPD). A potential advantage of this method is greater sampling volume and sampling time without the breakthrough that would be experienced if sampling for an extended time with uncoated silica gel tubes. Experiments to date indicate

no breakthrough of diacetyl, 2,3-pentanedione, or 2,3-hexanedione from the sampling tubes after passing 144 liters of air at 80% RH. This enables sampling for 8 hours without changing out sampling tubes. Another advantage is the high sensitivity of NPD detection, which will enable measurement of alpha-dicarbonyl compounds below the proposed REL for diacetyl of 5 ppb.

A new method for collecting air samples using evacuated canisters has been evaluated for several VOCs [LeBouf et al. 2012]. The 450-milliliter canisters, which can be equipped with either instantaneous grab sampling attachments or restricted-flow controllers (for task-based or full-shift sampling), are suitable for collection of area and personal samples. The air samples are analyzed for VOCs using a preconcentrator/gas chromatography-mass spectrometry (GC-MS) system. At present, this canister method is in the process of being validated with three additional compounds, diacetyl, 2,3-pentanedione and 2,3-hexanedione, and is being reviewed for incorporation into the *NIOSH Manual of Analytical Methods*.

A method for priority flavoring compounds is being investigated that utilizes a novel sampler—the helium diffusion sampler (HDS) [Entech Instruments Incorporated 2011]. The HDS collects a whole air sample for either short-term or full-shift sampling. The advantages of HDS are that no air sampling pump is required, there is no concern about breakthrough of the sample components, and there is minimal sample handling in the laboratory. A portion of the collected air sample is analyzed by a preconcentrator/GC-MS in the selected ion monitoring mode. Although HDS will not support limits of detection achieved by TD-GC-MS because of the relatively small air volume sampled (~20 mL), it may have adequate sensitivity to measure diacetyl at the proposed REL.

### 2.2.6 NIOSH Method 2549 – Qualitative Determination of Volatile Organic Compounds

To sample for diacetyl, 2,3-pentanedione, as well as a wide range of other flavoring VOCs, thermal desorption sorbent tubes can provide a high degree of sensitivity. This is because desorption of compounds from thermal desorption tubes does not involve dilution into an extraction solvent. Instead, compounds are thermally desorbed from the sampling tubes in a thermal desorption system. This technique is primarily used for qualitative screening purposes because of the ability of thermal desorption tubes to capture a diverse range of VOCs, but specific compounds can be quantified if corresponding standards are analyzed along with the samples. The thermal desorption tube is usually a stainless steel tube configured and filled with a single sorbent bed or multiple beds of various sorbents including carbonaceous materials, carbon molecular sieves, and/or porous polymers. The sorbents can be heated to high temperatures without breakdown or the generation of artifacts, so thermal desorption tubes can be cleaned and reused multiple times. The tubes are analyzed with a thermal desorber-GC-MS (TD-GC-MS) [NIOSH 1994].

## 2.3 Sampling for Diacetyl and 2,3-Pentanedione in Airborne Dust and in Bulk Materials

Although diacetyl and 2,3-pentanedione are normally found in liquid form, they can also be encapsulated in or coated on a powder substrate. Air sampling for dust that may be generated during handling of powdered flavorings can be achieved by active sampling methods. During the sample collection, however, some of the diacetyl and 2,3-pentanedione may volatilize,

i.e., release from the dust particles and enter the vapor phase due to contact with moisture. In addition, environments in which dust is generated may also contain vapors of the flavoring compounds. Sorbent tubes used for the collection of vapor-phase diacetyl or 2,3-pentanedione cannot be used to adequately sample for dust at the low flow rates required by the tubes [OSHA 2008a]. As a result, modifications to the sampling methods are necessary to assess exposure to both vapor and dust.

### 2.3.1 Size-Selective Air Sampling for Dust

Measurement of airborne dust particles according to their size (e.g. inhalable, thoracic, and respirable) can help to understand where they may deposit in the respiratory tract. Several types of sampling devices are available (e.g., inhalable dust samplers, impactors, cyclones, and sampling cassettes) to provide measurements of different size fractions of airborne dust. In most cases, dust is collected onto a filter, and the filter can be analyzed via gravimetric means to provide the mass of the dust. Filters should be hydrophobic in nature (e.g., polyvinyl chloride) in order to minimize collection of moisture. After being measured gravimetrically, filters can be analyzed for diacetyl and other compounds by the procedure described in section 2.3.2. Validated methods such as NIOSH Method 0500 for total dust and NIOSH Method 0600 for respirable dust [NIOSH 1994] are available for the collection and gravimetric analysis of airborne dust.

### 2.3.2 Sampling for Diacetyl and 2,3-Pentanedione in Airborne Dust

A sampling and analytical method is being developed by NIOSH for the quantitative measurement of diacetyl, 2,3-pentanedione, and potentially other flavoring compounds in dust. A sampling cassette with a filter is

used to collect airborne dust. The filter is then extracted in water and the aqueous solution is heated to promote the transfer of volatile components to the headspace above the solution. The headspace is sampled using a solid-phase microextraction (SPME) fiber. Headspace SPME involves an equilibrium process in which the volatile analytes establish equilibria between the sample solution, the headspace above the solution, and the polymer-coated fused silica fiber. The mechanism by which the analytes are extracted from the headspace is based on absorption of the analytes onto the fiber. The fiber is inserted directly into a GC-MS. The analytes are extracted from the fiber in the hot injection port and concentrated onto an analytical column. Because the entire sample collected on the fiber is introduced into the GC-MS instrument, as opposed to an aliquot of the sample for methods in which a solvent extract is used, lower detection limits can be achieved. This same procedure can be used to measure diacetyl, 2,3-pentanedione, and potentially other flavoring compounds in samples of bulk powders.

### 2.3.3 Bulk Liquids and Solids

#### 2.3.3.1 Sample collection

Although the review of safety data sheets or other available product documentation may be helpful to identify flavor compounds and potential exposures, they are not always comprehensive or specific. Collection and analysis of bulk flavoring materials can be useful to identify and quantify chemical ingredients and guide exposure assessment strategies. Prior to collecting bulk samples, it is important to consider the physical state of the materials to be sampled (liquids, pastes, or powders), the need to sample opened or unopened containers, the sampling locations, the number of samples to collect, and the amount of sample to collect (often determined by requirements of the laboratory analysis). Bulk samples should

be representative; in other words, they should be derived from a variety of sampling locations and obtained from multiple batches to capture any variability in the bulk materials used.

When sampling, it is important to collect and transport the sample in a manner that does not contaminate or cross-contaminate the bulk materials. Only clean or unused sample containers that are compatible with the bulk materials sampled should be used. In general, glass containers are ideal because they will not react with most chemicals, but polyethylene or polypropylene containers may also be appropriate. A typical container is a 20-mL glass scintillation vial with a polytetrafluoroethylene (PTFE)-lined screw cap. Each container should be clearly labeled with information about the bulk sample including material sampled, company and product number, site of sampling, date of sampling, sample tracking number and any hazards or precautions to be taken when handling the bulk sample.

After sampling, consideration should be given to preserve the integrity of the bulk samples during storage and shipping. For example, care should be taken to keep samples cold and protected from light if necessary. In addition, bulk materials should not be shipped together with air samples. Established Department of Transportation (DOT) and International Air and Transport Association (IATA) shipping regulations of hazardous materials and dangerous goods should be followed if hazardous materials are to be shipped. Materials that are considered hazardous for the purpose of transportation under the DOT regulations are listed in the hazardous materials table in Title 49 of the Code of Federal Regulations (CFR), Section 172.101 [49 CFR 172.101]; materials that are considered dangerous goods for the purpose of shipping by air under IATA regulations are listed in the list of dangerous goods in IATA dangerous goods regulations, section 4.2 [IATA 2012]. The DOT and IATA regulations guide

the classification/identification and packaging of hazardous materials and the marking and labeling of shipping containers containing hazardous materials. If the materials to be shipped are known to be hazardous but the specific names of the materials are not found on either the DOT hazardous materials table or the IATA list of dangerous goods, then the materials must be classified into a hazard class according to section 3 of the IATA dangerous goods regulations handbook, and a proper shipping name must be assigned according to section 4 of the IATA dangerous goods regulations handbook. A person must be trained in DOT and IATA regulations and certified in order to mark a shipment as hazardous. If it is unknown whether the materials to be shipped are hazardous or not, then a person who is trained in DOT and IATA regulations should be consulted.

### ***2.3.3.2 Measurement of diacetyl or 2,3-pentanedione content of bulk powders***

The analytical procedure being developed for airborne dust samples described in section 2.3.2 will also be used for analysis of bulk powder samples.

## **2.4 Real-time Techniques for Diacetyl and Other Flavoring Compounds**

Several analytical methods provide real-time or near real-time measurements of volatile compounds in air such as diacetyl and 2,3-pentanedione. These methods have the unique advantage of providing continuous exposure information over very short averaging periods that can be viewed as it is being generated during sampling or later if the instrument has data-logging capabilities. The abundance of measurement information provides valuable insight into variations in concentrations

throughout the sampling period as well as the short-term concentration peaks that can possibly be associated with their sources. While real-time monitoring instruments generally lack sufficient sensitivity and specificity for monitoring REL levels of diacetyl and 2,3-pentanedione, they can be useful for screening, identifying appropriate work practices, and to find leaks and “hotspots.” This information can be very useful in the development of exposure controls.

### 2.4.1 Photoionization Detectors

Photoionization detectors (PIDs) can be used to monitor VOC air concentrations in industrial work environments, including flavoring manufacturing facilities, and have become favored instruments for on-site monitoring because of ease of operation, reliability, versatility, cost, and response to a wide variety of substances. PID instruments measure the relative concentration of VOCs by passing the molecules of those compounds past an ultraviolet lamp that emits radiation over a narrow wavelength range in the ultraviolet region of the electromagnetic spectrum. Photons of ultraviolet radiation will form a molecular ion by removing an electron from orbit around that molecule, allowing for electronic detection of that ion, hence the name.

The energy of the radiation emitted by the lamp is inversely proportional to its wavelength, and common PID lamps produce energy in the range from approximately 8 to 12 electron volts (eV). The amount of work required to form a molecular ion by removing an electron from orbit, a property known as ionization potential, varies by compound but for many hydrocarbons is in the range from 7 to 11 eV. Because nitrogen, oxygen, and many of the minor components of air (i.e., water vapor, carbon monoxide, carbon dioxide, argon) have ionization potentials significantly higher than 12 eV, they are not ionized by the photons emitted from a PID. This property allows for

the continuous monitoring of air to obtain an estimate of total hydrocarbon concentration.

PIDs respond to a broad range of VOCs and do not provide concentrations specific to any particular compound. They are often calibrated for isobutylene and can commonly detect total VOC concentrations from 1 to 2,000 ppm. Modern PIDs can be programmed to measure the concentration of VOCs at fixed time intervals and store these data for subsequent download to a computer.

### 2.4.2 Infrared Analyzers

The absorption of infrared (IR) radiation, while more commonly used as a qualitative tool, can also be used to quantify many substances by determination of response relative to known concentrations of that substance. Absorption of electromagnetic radiation in the IR region of the spectrum will produce transitions among vibrational and rotational states of the molecules absorbing that radiation. This absorption can only occur at wavelengths exactly matching the vibrational frequency of a chemical bond, and by selecting the proper analytical wavelength it is possible to obtain reasonable specificity in the compound being quantified.

Diacetyl can be detected and measured by using an IR gas analyzer such as the Thermo Electron MIRAN® “SapphIRE” (Thermo Fisher Scientific Inc., Waltham, MA), which is a portable direct-reading instrument that has the advantage of displaying real-time concentrations. The SapphIRE is a single beam IR spectrophotometer with a pathlength of 0.5 or 12.5 meters. It has a sample cell volume of 2.23 liters and a built-in pump that runs at approximately 14 liters per minute. Single sample analyses are updated every 0.5 seconds. The detector is available with preloaded factory calibrations for over 100 gases, but because diacetyl is not in this standard library it should be set up for this application by the factory. The concentration

range that can be measured is dependent on the compound in question. The high and low settings for the pathlength extend this range considerably.

The predecessor model, the Foxboro/Wilks MIRAN 1A, has adjustable wavelength and pathlength controls and can be calibrated for gases or vapors using the closed loop system available. Many MIRAN 1A models are still in use in the field. The best wavelength for measuring diacetyl is about 9 micrometers. Neither water nor carbon dioxide should interfere significantly at that wavelength. The minimum detectable concentration should be less than 0.5 ppm at the highest pathlength.

Fourier transform infrared gas analyzer (FTIR) spectroscopy can be used to analyze a sample of gaseous molecules for both chemical composition and for the concentration of individual chemical constituents. In this analysis, chemical functional groups absorb IR radiation at specific, unique frequencies producing a characteristic spectrum of absorbed versus transmitted radiation. From this spectrum, identification and quantitation of the gas is possible. FTIR analysis can produce real-time quantitation of flavoring compounds in air providing chemical specific full-shift, partial-shift, and peak concentration measures although interferences can pose analytical difficulties in quantifying specific flavoring compounds in complex environments with multiple organic chemicals present.

### **2.4.3 Photoacoustic Spectroscopy (Infrared Absorbance) Techniques**

Because the absorption of infrared radiation produces transitions among vibrational states of molecules, the application of rapid pulses of IR photons at the proper wavelength can be used to produce pressure variations in the air surrounding the molecules absorbing that radiation. Those pressure variations can be detected

as sound waves, the amplitude of which is proportional to the concentration of the analyte of interest. Using IR radiation and measuring this resultant amplitude to quantify an analyte is the technique of photoacoustic spectroscopy.

Diacetyl has been measured using the Innova photoacoustic infrared gas analyzers, which are direct-reading instruments that have the advantage of displaying real-time concentrations. Both personal and area concentrations were measured during tasks involving exposure to diacetyl in liquid and powder form and then 8-hour TWA exposures were calculated. The powder exposures only measured vapor released and did not include diacetyl adsorbed on the powder [Martyny et al. 2008].

Current available models of the photoacoustic analyzer are the 1314 and 1412, available from California Analytical Instruments, Inc., Orange, CA. The measurement system is based on photoacoustic infrared detection and provides the capability of measuring virtually any gas that absorbs in the infrared spectrum. Gas selectivity is achieved through the use of optical filters that provide both a means of detecting the gas of interest and compensating for interfering gases and water. Specifications on the unit indicate a dynamic range of 4 orders of magnitude and a repeatability of 1% of the measured value. The analyzer displays updated concentrations approximately every 30 seconds. The analyzer can be calibrated using diacetyl standards and can analyze diacetyl concentrations from the parts per billion range to hundreds or thousands of parts per million.

## **2.5 Industrial Hygiene Surveys and Exposure Assessments**

Several investigations have been completed by NIOSH and others within the flavoring and food production industries. Exposure

conditions vary widely, depending upon site-specific parameters and the processes employed. Many diacetyl samples have been collected to evaluate occupational exposures in the workplace and are described below. When pertinent data on absolute humidity and time to sample extraction were available, measurements obtained using NIOSH Method 2557 were subsequently corrected for the method's tendency to underestimate [Cox-Ganser et al. 2011]. An overview of diacetyl samples collected during multiple investigations is presented in Table 2-1.

### **2.5.1 NIOSH Microwave Popcorn Production Exposure Assessments**

NIOSH conducted health hazard evaluations at six microwave popcorn plants from 2000 to 2003 [Kanwal et al. 2006]. In these facilities diacetyl-containing butter flavorings (liquids, pastes, or powders) were mixed with heated soybean oil in large heated mixing tanks. Salt and coloring were added to the flavoring mixture which was transferred to packaging lines and combined with kernel popcorn in microwaveable bags. Diacetyl concentrations were measured with NIOSH Method 2557 in multiple production locations using personal and area samples.

In the plants, 29 area and 17 personal samples were collected in mixing areas, and 67 area and 65 personal samples were collected in packaging areas. Humidity-corrected mean diacetyl air concentrations ranged from 0.63 to 57.2 ppm for area samples and from 0.035 to 1.33 ppm for personal samples in the mixing areas. In the packaging areas, mean concentrations ranged from 0.019 to 3.0 ppm for area samples and from 0.023 to 1.16 ppm for personal samples. In general, diacetyl concentrations were higher

in the mixing rooms when the diacetyl-containing butter flavorings were heated.

In 2010, a microwave popcorn company asked NIOSH to evaluate chemical constituents in eight liquid butter flavorings because their supplier did not identify chemical substitutes they were using in place of diacetyl [Boylstein 2012]. Quantitative GC-MS analysis showed acetoin in five samples, 2,3-pentanedione in four, and 2,3-hexanedione in one, all at concentrations of 0.5% or less by weight, except for one acetoin sample at 2%. The more sensitive semiquantitative headspace analysis with thermal detection tubes found diacetyl and acetoin in all samples, 2,3-pentanedione in five, 2,3-hexanedione in one, and 2,3-heptanedione in one.

### **2.5.2 Other Microwave Popcorn Production Exposure Assessments**

White et al. [2010] conducted a comprehensive, repeated exposure monitoring campaign at four microwave popcorn plants. A total of 639 full shift diacetyl samples were collected during the day and night shifts in multiple production areas including all employees who worked in the slurry (mixing) room. In that study 49% of 639 samples were below their limit of detection with the maximum measurement of 11.72 ppm after correction for humidity [White et al. 2010]. Overall, exposures were higher for mixers compared to non-mixers and were consistent with diacetyl concentrations observed during previous NIOSH investigations. Diacetyl exposures declined substantially for mixers after the installation of engineering controls.

### **2.5.3 NIOSH Flavoring Manufacturing Exposure Assessments**

In 1985, NIOSH conducted a health hazard evaluation at a plant in Indiana that produced flavorings for the baking industry [NIOSH 1986]. Case histories showed severe fixed

Table 2-1. Multiple investigations of diacetyl in flavoring and food production industries

Study	Method*	Location	Diacetyl concentration in ppm (sample type)		
			Arithmetic mean	Geometric mean	Range
<b>Microwave popcorn plants</b>					
Facility G (first survey) [NIOSH 2006]	NIOSH Method 2557 (corrected)	Mixing room Packaging area QC lab Maintenance Other areas	57.2 (full-shift TWA) 2.8 (full-shift TWA) 0.8 (full-shift TWA) 0.9 (full-shift TWA) < 0.15 (full-shift TWA)	— — — — —	— — — — —
	NIOSH Method 2557 (corrected)	Packaging areas (area samples) Packaging areas (personal samples) Mixing rooms/areas (area samples) Mixing rooms/areas (personal samples)	0.019–3.0 0.023–1.16 0.63–57.2 0.035–1.33	— — — —	— — — —
Facilities G,J,K,L,N,O [Kanwal et al. 2006] [White 2011]	NIOSH Method 2557 (corrected)	Mixers Non-mixers	0.119–2.704 (full shift) 0.042–0.123 (full shift)	0.044–0.587 (full shift) < 0.001–0.019 (full shift)	0.004–11.72 (full shift) 0.004–1.984 (full shift)
	NIOSH Method 2557 (corrected)				
<b>Flavoring production plants</b>					
Facility B [NIOSH 2007a]	NIOSH Method 2557 (corrected)	Powdered flavoring production area	2.73 (full-shift TWA) 25.9 (partial shift)	— —	204 (real-time peak) —
	OSHA Method PV2118	Liquid flavoring production area Powdered flavoring production area Task-based (pouring diacetyl)	0.46 (full-shift TWA) 0.34 (full-shift TWA) —	— — —	— — 11 (10-minute peak)
Facility H [NIOSH 2008a]	OSHA Method PV2118	Liquid production room (area samples) Powder production room (area samples)	0.26 (full-shift TWA) 0.07 (full-shift TWA)	— —	— —
	NIOSH Method 2557	Liquid production (personal samples) Powder production (personal samples)	0.10 0.05	— —	— —

See footnotes at end of table.

(Continued)

Table 2-1 (Continued). Multiple investigations of diacetyl in flavoring and food production industries

Study	Method*	Location	Diacetyl concentration in ppm (sample type)		
			Arithmetic mean	Geometric mean	Range
Facility D [NIOSH 2009c]	OSHA Method PV2118	Starter distillate room	—	1.06 (full-shift TWA)	1.06 (full-shift TWA)
		Starter distillate room (personal samples)	—	1.78 (full-shift TWA)	1.78 (full-shift TWA)
		Spray dry room	—	1.07 (full-shift TWA)	1.07 (full-shift TWA)
		Spray dry room (personal samples)	—	0.756 (full-shift TWA)	0.756 (full-shift TWA)
		Flavors room	—	0.171 (full-shift TWA)	0.171 (full-shift TWA)
		Flavors room (personal samples)	—	0.329 (full-shift TWA)	0.329 (full-shift TWA)
		Spray dry room, task-based (moving diacetyl between containers)	—	—	—
Facility I [NIOSH 2011]	NIOSH Method 2557	Spray drying	—	0.169 (full-shift TWA)	—
		Spray drying (personal samples)	—	0.123 (full-shift TWA)	—
		Other production areas	—	0.375 (full-shift TWA)	—
		Other production areas (personal samples)	—	0.762 (full-shift TWA)	—
		Spray drying	—	0.167 (full-shift TWA)	—
		Spray drying (personal samples)	—	0.182 (full-shift TWA)	—
		Coffee and tea area	—	0.076 (full-shift TWA)	—
Facility Q [NIOSH 2013a]	Canister	Liquid compounding area (personal samples)	—	1.900 (full-shift TWA)	—
		Multiple locations (area samples)	—	—	No diacetyl detected
		—	—	—	(< 0.0029)(instant to 3 hrs)
[Martyny et al. 2008]	NIOSH Method 2557	All areas (personal samples)	2.48 (1–3 hours)	—	0.01–60 (1–3 hours)
<b>Diacetyl production</b>					
[van Rooy 2007]	—	Task specific	—	—	0.6–83 (real-time peaks)

See footnotes at end of table.

(Continued)



obstructive lung disease among employees in a mixing room. Data from previous air monitoring indicated a high dust concentration in the personal breathing zone of an employee during a mixing operation. Diacetyl was on a list of ingredients commonly used at this facility but airborne measurements of diacetyl or other flavoring compounds were not made. Although the investigators were unable to identify specific etiology at that time, they concluded that employees' disease was most likely caused by some agent in the mixing room at the plant.

NIOSH personnel conducted evaluations at three California flavoring manufacturing facilities where they measured exposures to diacetyl and other related compounds [NIOSH 2007a, 2008a, b, c]. The objectives of these surveys included identifying common work tasks, plant processes, and procedures, as well as characterizing potential occupational exposures within the flavoring industry. Most of the data collected were from the liquid and powder production areas, with some information also coming from spray drying, preproduction, quality assurance, administration, and research and development locations.

At one plant [NIOSH 2007a], the mean TWA diacetyl exposure, after NIOSH Method 2557 humidity-based correction, from full-shift air sampling in the powdered flavoring production area was 2.73 ppm. Measurements made with partial-shift air sampling during the production of butter and vanilla powdered flavorings showed a diacetyl exposure of 25.9 ppm. Employees' real-time diacetyl exposures measured with an FTIR monitor during the packaging of these powders were as high as 204 ppm. At another plant [NIOSH 2008b], mean TWA diacetyl air concentrations from full-shift air sampling using modified OSHA Method PV2118 in November 2006 (area and personal samples combined) were 0.46 ppm in liquid flavoring production and 0.34 ppm in powdered flavoring production. A task-based

personal air sample measured a diacetyl air concentration of 11 ppm when an employee poured diacetyl from a 55-gallon drum into multiple 5-gallon containers over a 10-minute period. Using modified OSHA Method PV2118 for area air sampling at the other plant [NIOSH 2008a], the mean full-shift concentration of diacetyl in the liquid production room was 0.26 ppm, while in the powder production room it was 0.07 ppm. For personal samples that were collected with NIOSH Method 2557 and not corrected for humidity and time to extraction, the mean concentrations in liquid production and powder production rooms were 0.10 ppm and 0.05 ppm. This work also indicated high variability in concentrations of volatile organic compounds (as measured with a PID) and dust (as measured with personal dust monitors) with time.

A health hazard evaluation was conducted at a facility in Wisconsin [NIOSH 2009c] that manufactured flavorings, modified dairy products, and bacterial additives. One of the flavoring products made at this plant was liquid starter distillate, a product of distillation of fermented milk stock, which contains about 4.5% diacetyl. Starter distillate and liquid diacetyl were used to make a variety of powdered (via spray drying processes) and liquid flavorings. NIOSH staff obtained 21 personal and 29 area air samples using modified OSHA Method PV2118 for diacetyl throughout the facility. They found the highest full-shift TWA concentrations in the starter distillate room (geometric mean of 1.78 ppm for personal and 1.06 ppm for area samples), followed by the spray dry room (0.756 and 1.07 ppm) and the flavors room (0.329 and 0.171 ppm). In the spray dry room, FTIR real-time measurements indicated peak diacetyl concentrations up to 90 ppm in the employee's breathing zone while dumping diacetyl from buckets to mixing tanks and while pumping diacetyl from a barrel into buckets. A peak exposure of about 18 ppm was measured in

the breathing zone of an employee in the same room while cleaning a barrel with a water hose.

Company air sampling data were obtained during a health hazard evaluation at an Indiana flavorings plant that used many ingredients, including diacetyl and starter distillate, in the batch production of a variety of liquid and powdered flavorings [NIOSH 2011]. Using NIOSH Method 2557 prior to the HHE request to measure diacetyl, they collected 22 samples. The geometric mean full-shift TWA diacetyl concentration in spray drying operations was 0.123 ppm for personal samples and 0.169 ppm for area samples, while in the other production areas, mean concentrations up to 0.762 ppm and 0.375 ppm were measured for personal and area samples, respectively. Because of the problems with NIOSH Method 2557, these results were likely underestimations of the true concentrations. No data on humidity or time from collection to analysis was available, so no correction could be estimated. Subsequent measurements (45 personal and 71 area samples) by the company, after some control intervention, were collected using validated OSHA sampling Methods PV2118 and 1012 for diacetyl. In the spray drying operations, the geometric mean for full-shift diacetyl personal samples was 0.182 ppm, and for area samples it was 0.167 ppm. The highest mean concentration in the other production areas was 1.900 ppm for personal samples (liquid compounding area) and 0.076 ppm for area samples (coffee and tea area).

Another health hazard evaluation was performed at a flavorings plant in Kentucky that produced flavors, colors, and food and beverage ingredients used in the manufacture of consumer products [NIOSH 2013a]. Diacetyl was not found in use during the NIOSH air sampling survey. Using evacuated canisters, diacetyl and 2,3-hexanedione were not detected in any of the instantaneous or 3-hour area air samples taken in several parts of the plant. 2,3-Pentanedione

was detected in two area air samples taken in the liquid samples room. The detection limits ranged from 1.4 to 2.9 ppb for diacetyl, 1.5 to 3.2 ppb for 2,3-pentanedione, and 1.7 to 3.6 ppb for 2,3-hexanedione. Of the two air samples that detected 2,3-pentanedione in the room, one was an instantaneous sample taken near a trash can for disposal of used pipettes while making a flavoring recipe and resulted in a level of 47 ppb. The other sample that detected 26 ppb 2,3-pentanedione was collected for 187 minutes in the center of the room. During the sampling period, several employees were preparing recipes, which included fruit and cheese flavors.

#### 2.5.4 Other Flavoring Manufacturing Exposure Assessments

In a study evaluating diacetyl exposures in 16 flavor manufacturing facilities, Martyny et al. [Martyny et al. 2008] measured levels of that compound from the limit of detection (0.01 to 0.18 ppm depending on sample duration) to as high as 60 ppm. Using a protocol designed to obtain measurements during worst-case exposures by collecting samples only during processes in which diacetyl was being used, 181 personal and area samples were collected generally for 1 to 3 hours. Samples for diacetyl were collected and analyzed using NIOSH Method 2557 [NIOSH 1994] which was subsequently found to underestimate actual diacetyl concentrations. Without sampling environment absolute humidity information to make corrections, the results of this study likely underestimate true values.

Results indicated personal exposures during the selected work processes ranged from <0.01 to 60 ppm, with a mean of 2.48 ppm. Eight-hour TWA concentrations were calculated with the assumption that there was no exposure to diacetyl during the unsampled 5 to 7 hours of a work shift. However real-time monitoring of airborne diacetyl vapor concentrations, made using a photoacoustic IR analyzer, indicated a

background of approximately 2 ppm diacetyl according to Figure 1 of that paper.

Data indicated that concentrations varied by process, with powder compounding having the highest mean and median diacetyl exposures. Martyny also concluded, “Compared with the microwave popcorn industry, there is wide variability in frequency and duration of use of diacetyl among flavor companies.”

### 2.5.5 NIOSH Flavored Food Production Exposure Assessments

NIOSH researchers conducted health hazard evaluations at food production facilities including a bakery mix production plant [NIOSH 2009a], a popcorn popping plant [NIOSH 2007b], three office building cafeterias [NIOSH 2009b], a cream cheese manufacturing plant [NIOSH 2013b], a snack food production plant [NIOSH 2013c], and a coffee production plant [Bailey et al. 2015; Duling et al. 2016].

At the bakery mix production facility, employees combined liquid and powdered flavorings with flour, sugar, salt and other solid ingredients to produce baking mixes. For about a year up to July 2008, the plant used a buttermilk flavoring that contained 15% to 20% diacetyl and then began using a reformulated buttermilk flavoring that contained less than 1% diacetyl. The reformulated flavoring also contained the diacetyl substitute 2,3-pentanedione. Diacetyl was detected in qualitative screening air samples using NIOSH Method 2549 during industrial hygiene air sampling by NIOSH investigators in late September 2008, but the concentrations were too low to be detected in any of the 9 personal or 10 area samples collected with the modified OSHA Method PV2118. Diacetyl was again not detectable in a second industrial hygiene survey in May 2009 when NIOSH investigators collected 13 personal and 11 area air samples using OSHA Method 1013; however, one personal sample showed an air

concentration of 2,3-pentanedione of 91 ppb (parts per billion parts air), and a corresponding area sample showed an air concentration of 78 ppb. Nearly half of the samples detected 2,3-pentanedione in the air. Area air sampling using a method under development, in-tube derivatization with 1,2-phenylenediamine (section 2.2.5 above), did not detect diacetyl in any of the 11 samples, but it measured 2,3-pentanedione in 7 samples, at concentrations ranging from 48 to 95 ppb. The sample that showed an air concentration of 95 ppb was obtained in the same area where a sample obtained with OSHA Method 1013 showed an air concentration of 78 ppb.

At the popcorn popping plant, neither the two personal nor the twelve area air samples found diacetyl concentrations above the minimum detectable concentration of 0.01 ppm using NIOSH Method 2557 during popcorn popping operations with butter-flavored oil. Diacetyl was detected in all three thermal desorption tube samples from the room with semiquantitative analyses (NIOSH Method 2549) but with very low abundances. A one-minute real-time concentration of 0.14 ppm diacetyl was measured with an FTIR monitor directly above the heated popping oil.

At the three cafeterias, two of seven cooking oil products being used contained diacetyl. Neither diacetyl nor acetoin was found at or above the minimum detectable concentration (0.02 ppm) using the modified OSHA Method PV2118 to collect 20 personal and area air samples during grilling operations.

At the cream cheese plant in 2011, several flavorings, including dairy, cheese, strawberry, blueberry, and smoke were found with headspace sampling to contain diacetyl with or without 2,3-pentanedione. Air sampling with OSHA Method 1012 during cooking, filling, and packaging of cream cheese made with some of those flavorings, measured area diacetyl concentrations (n=15 near full-shift) from

0.3 to 13.8 ppb. The four highest concentrations were greater than 11 ppb: three of these were collected in a cooking area and the other in a filling area. Fourteen near full-shift personal diacetyl exposures ranged from 0.4 to 8.3 ppb, while six short-term samples collected mostly while ingredients were added to cook kettles ranged from 4.4 to 15.1 ppb. Fourteen area and six personal concentrations of 2,3-pentanedione measured with OSHA Method 1016 were all less than limits of detection (15.8 to 48.8 ppb), as were two of the three also sampled with the more sensitive draft NIOSH method using 1,2-phenylenediamine-treated silica gel tubes (0.5 ppb limit of detection) – the detectable concentration was 0.9 ppb while using smoke flavoring. Of six area samples collected alongside cleaning operations with evacuated canisters for 2,3-pentanedione (1.2 to 2.9 ppb limits of detection) and 2,3-hexanedione (1.5 to 3.6 ppb limits of detection), one measured 2,3-pentanedione at 6.2 ppb and 2,3-hexanedione at 9.0 ppb during a nearly 3-hour cleaning procedure of cooking equipment containing strawberry cream cheese remnants while no cream cheese was being made in the room.

The snack food production plant applied powdered seasonings onto potato, corn, and tortilla chips after they were fried. Headspace analyses of bulk samples of seasonings found trace amounts of diacetyl, but no other alpha-diketone compounds, in four of the seven samples: barbeque, honey barbeque, cheddar sour cream, and chili cheese. Diacetyl, 2,3-pentanedione, and 2,3-hexanedione were not detected in the five 15- to 180-minute personal breathing zone evacuated canister air samples from processing line operators during nacho cheese tortilla chip production. The detection limits ranged from 2.8 to 6.0 ppb for diacetyl, 3.4 to 7.2 ppb for 2,3-pentanedione, and 3.2 to 6.8 ppb for 2,3-hexanedione. Although diacetyl was detected in three area samples collected instantaneously near the seasoning hopper, it was not

quantifiable. Because it was found between the detectable level of 1.3 ppb and the quantifiable level of 4.3 ppb, the reported concentrations of 1.4 to 1.7 ppb are considered estimates. The area samples did not detect 2,3-pentanedione or 2,3-hexanedione (detection limits of 1.5 and 1.6 ppb, respectively).

The coffee production plant produced flavored and unflavored whole bean and ground coffee. Full-shift area air samples collected for diacetyl with OSHA Method 1012 and for 2,3-pentanedione with OSHA Method 1016 had highest mean concentrations by location in the grinding/packaging room (103 ppb diacetyl, 63 ppb 2,3-pentanedione), flavoring room (90 ppb diacetyl, 151 ppb 2,3-pentanedione), and the production offices (62 ppb diacetyl, 32 ppb 2,3-pentanedione), which were located within the larger grinding/packaging room. These were followed by mean concentrations in the roasting room (20 ppb diacetyl, 6 ppb 2,3-pentanedione), green bean and finished goods warehouses (11 ppb diacetyl, <3 ppb 2,3-pentanedione), quality control room (8 ppb diacetyl, <3 ppb 2,3-pentanedione), maintenance shop (7 ppb diacetyl, <3 ppb 2,3-pentanedione), and the nonproduction offices (4 ppb diacetyl, <3 ppb 2,3-pentanedione). The flavoring room was under negative pressure with respect to the adjacent grinding/packaging room where unflavored roasted coffee was processed.

Personal sample mean concentrations by location in the coffee plant were highest for employees working in the grinding/packaging room (93 ppb diacetyl, 53 ppb 2,3-pentanedione), flavoring room (80 ppb diacetyl, 122 ppb 2,3-pentanedione), production offices (81 ppb diacetyl, 22 ppb 2,3-pentanedione), all over (59 ppb diacetyl, 39 ppb 2,3-pentanedione), and housekeeping (54 ppb diacetyl, 18 ppb 2,3-pentanedione). These were followed by those in the roasting room (26 ppb diacetyl, 7 ppb 2,3-pentanedione), quality control room (24 ppb diacetyl, 11 ppb 2,3-pentanedione), warehouse

(8 ppb diacetyl, <3 ppb 2,3-pentanedione), and nonproduction offices (7 ppb diacetyl, <3 ppb 2,3-pentanedione).

The mean area concentrations on the grinding/packaging and flavoring room mezzanines, where roasted whole and ground bean storage hoppers were located, were higher than those measured on the main production levels of the rooms. A 15-minute short-term air sample collected at the open hatch of a grinding/packaging room mezzanine hopper holding unflavored ground coffee above an active packaging line measured concentrations of 14,300 ppb diacetyl and 13,800 ppb 2,3-pentanedione. The location of the sample was representative of the proximity of employees' faces as they frequently and momentarily monitored coffee levels in the hoppers throughout their shift.

NIOSH also conducted a small industrywide study at some flavored food production facilities where diacetyl and other food flavorings were added to various food products. Seventy-four personal and 105 area samples were collected for diacetyl using OSHA Method 1013. With one exception where local exhaust ventilation was documented in some locations, no engineering controls were noted in any facility. Of the 179 total samples, 12 had detectable levels of diacetyl (LOD 0.5 – 1.0 ug/sample). The eight area samples ranged from 0.03 to 3.1 ppm, with three samples above 1 ppm (1.1, 2.1 and 3.1 ppm). The four personal samples ranged from 0.06 to 0.6 ppm [Curwin et al. 2015].

### **2.5.6 OSHA Site Visits Related to Diacetyl and Flavorings that Contain Diacetyl**

Between January 2008 and January 2010, an OSHA contractor measured diacetyl exposure

to employees in a series of 12 industrial hygiene surveys at various facilities that use (11 facilities) or manufacture (1 facility) formulated flavorings, including flavorings that contain diacetyl [Eastern Research Group 2008a, b, c, d, 2009a, b, c, d, e, 2010a, b, c]. In the first two surveys, conducted in January 2008, diacetyl was measured using OSHA Method PV2118. In the subsequent 10 surveys, OSHA Methods 1012 and 1013 were used. At all facilities, visual observation was made of engineering controls in place at the various operations evaluated.

The measured range of diacetyl concentrations are presented in Table 2-2 below, along with the type of facility and synopsis of controls. Eastern Research Group returned to OSHA G Facility in 2010 to remeasure airborne diacetyl concentrations following the installation of engineering controls and work-practice changes at that facility. In this follow-up study measurements were also made for 2,3-pentanedione in samples that contained diacetyl. 2,3-Pentanedione was not detected.

### **2.5.7 Other Exposure Assessments**

Pierce et al. characterized diacetyl exposures that could potentially occur in a simulated small coffee shop during the preparation and consumption of unflavored coffee. Mean estimated 8-hour TWA exposure concentrations ranged from 7 ppb to 13 ppb [Pierce et al. 2015].

Gaffney et al. evaluated exposures in a facility that roasts and grinds coffee beans. Results indicated that airborne concentrations of diacetyl and 2,3-pentanedione are similar to concentrations in food flavoring facilities [Gaffney et al. 2015].

**Table 2-2. Investigations of facilities using or producing diacetyl**

Facility	Product	Controls in place	Diacetyl measurement range
OSHA A [ERG 2008a]	Coffee	Dilution ventilation	ND–54 ppb (TWA) 8 ppb (short-term sample)
OSHA B [ERG 2008b]	Commercial bakery	Dilution ventilation, LEV, process containment	ND–2703 ppb (TWA) ND–1012 ppb (area)
OSHA C [ERG 2008c]	Seasoned snack product	Dilution ventilation; heat extraction for adjacent process	ND
OSHA D [ERG 2008d]	Baked snack food	Secondary heat ventilation for heating and powder dumping	ND–164 ppb (TWA) ND–139 ppb (short-term sample) ND–111 ppb (area)
OSHA E [ERG 2009a]	Sauce production	Engineering controls for other purposes, heat removal, etc.	ND–5.3 ppb (TWA) ND–10.5 ppb (short-term sample) ND–2.4 ppb (area)
OSHA F [ERG 2009b]	Low-calorie cracker	Canopy hoods in heated Production process #1, dilution ventilation	ND–195.7 ppb (TWA) ND–701.1 ppb (short-term sample) ND–13.1 ppb (area)
OSHA G [ERG 2009c]	Buttered popcorn production	Before: Heat extraction hoods, dilution ventilation  After: Dilution ventilation; slot hood at tumbler; modified tank cover; work practice change	24.8–71.2 ppb (TWA) 466.8–2298.7 ppb (short-term sample) 9.1–8660.2 ppb (area)  < 2.7–< 9.8 ppb (TWA) < 10.4–98.8 ppb (short-term sample) 2.7–5.4 ppb (area)
OSHA H [ERG 2009d]	Sour cream production	Exhaust ventilation for dust	ND–32.4 ppb (TWA) ND–138.6 ppb (short-term sample) ND–4.6 ppb (area)
OSHA I [ERG 2009e]	Ice cream	Controls for other purposes, immediate rinsing, cool temperature dilution ventilation	ND to 1.6 ppb (TWA) ND (short-term sample) ND (area)
OSHA J [ERG 2010a]	Cottage cheese	Dilution ventilation	ND–55.3 ppb (TWA) 32.3–317 ppb (short-term sample) 1.3–32.4 ppb (area)
OSHA K [ERG 2010b]	Food flavor production	Dilution ventilation; hose from tank to floor drain	ND–2,990 ppb (TWA) ND–12,373.1 ppb (short-term sample) 29.1–381.5 (area)
OSHA L [ERG 2010c]	Retail bakery	Dilution ventilation; oven room heat extraction	ND–50.4 ppb (TWA) ND–118.5 ppb (short-term sample) ND–30.9 ppb (area)

ND: not detected. The verification code for this document is 422299

TWA: a sample concentration determined over a full work shift

Short-term sample: a concentration measured for less than a full work shift

# References

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- 49 CFR 172.101. Purpose and use of hazardous materials table. Code of Federal Regulations. Washington, DC: U.S. Government Printing Office, Office of the Federal Register.
- Ashley K, McKernan LT, Burroughs E, Deddens J, Pendergrass S, Streicher RP [2008]. Analytical performance criteria. Field evaluation of diacetyl sampling and analytical methods. *J Occup Environ Hyg* 5(11):D111–116.
- Bailey RL, Cox-Ganser JM, Duling MG, LeBouf RF, Martin SB, Bledsoe TA, Green BJ, Kreiss K [2015]. Respiratory morbidity in a coffee processing workplace with sentinel obliterative bronchiolitis cases. *Am J Ind Med* 58(12):1235–1245.
- Boylstein R [2012]. Identification of diacetyl substitutes at a microwave popcorn production plant. *J Occup Environ Hyg* 9(2):D33–34.
- Cox-Ganser J, Ganser G, Saito R, Hobbs G, Boylstein R, Hendricks W, Simmons M, Eide M, Kullman G, Piacitelli C [2011]. Correcting diacetyl concentrations from air samples collected with NIOSH Method 2557. *J Occup Environ Hyg* 8(2):59–70.
- Curwin BD, Deddens JA, McKernan LT [2015]. Flavoring exposure in food manufacturing. *J Expo Sci Environ Epidemiol* 25(3):324–333.
- Duling MG, LeBouf RF, Cox-Ganser JM, Kreiss K, Martin SB, Bailey RL [2016]. Environmental characterization of a coffee processing workplace with obliterative bronchiolitis in former workers. *J Occup Environ Hyg* 13(10):770–781.
- Eastern Research Group, Inc. [2008a]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility A—a coffee roaster. OSHA Docket No. 2008–0046–0068. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2008b]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility B—a commercial bakery. OSHA Docket No. 2008–0046–0069. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2008c]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility C—seasoned snack product manufacturing. OSHA Docket No. 2008–0046–0070. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2008d]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility D—baked snack food production. OSHA Docket No. 2008–0046–0078. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2009a]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility E—sauce production. OSHA Docket No. 2008–0046–0079. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2009b]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility F—low calorie cracker production. OSHA Docket No. 2008–0046–0080. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2009c]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility G—buttered popcorn production (pre-popped). OSHA Docket No. 2008–0046–0081. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2009d]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility H—sour cream production. OSHA Docket No. 2008–0046–0082. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2009e]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility I—an ice cream manufacturing plant. OSHA Docket No. 2008–0046–0083. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2010a]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility J—dairy—cottage cheese production. OSHA Docket No. 2008–0046–0084. By Eastern Research Group, Inc. Lexington, MA.

- Eastern Research Group, Inc. [2010b]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility K—flavor production. OSHA Docket No. 2008–0046–0086. By Eastern Research Group, Inc. Lexington, MA.
- Eastern Research Group, Inc. [2010c]. Site visits related to diacetyl and flavorings that contain diacetyl: food production facility L—a retail bakery. OSHA Docket No. 2008–0046–0085. By Eastern Research Group, Inc. Lexington, MA.
- Entech Instruments Incorporated [2011]. HDS™ personal monitors. <http://www.entechnst.com/pdf/2011%20Updates/Seperate%20Sections/P78-81%20HDS%20Personal%20Monitors.pdf>. Date accessed: May 2011.
- Gaffney SH, Abelmann A, Pierce JS, Glynn ME, Henshaw JL, McCarthy LA, Lotter JT, Liang M, Finley BL [2015]. Naturally occurring diacetyl and 2,3-pentanedione concentrations associated with roasting and grinding unflavored coffee beans in a commercial setting. *Toxicol Rep* 2: 1171–1181.
- IATA [2012]. IATA Dangerous Goods Regulations. Montreal, Quebec, Canada: International Air Transport Association.
- Kanwal R, Kullman G, Piacitelli C, Boylstein R, Sahakian N, Martin S, Fedan K, Kreiss K [2006]. Evaluation of flavorings-related lung disease risk at six microwave popcorn plants. *J Occup Environ Med* 48(2):149–157.
- LeBouf RF, Stefaniak AB, Virji MA [2012]. Validation of evacuated canisters for sampling volatile organic compounds in healthcare settings. *J Environ Monit* 14(3):977–983.
- Martyny JW, Van Dyke MV, Arbuckle S, Towle M, Rose CS [2008]. Diacetyl exposures in the flavor manufacturing industry. *J Occup Environ Hyg* 5(11):679–688.
- NIOSH [1986]. Hazard evaluation and technical assistance report: International Bakers Services, Inc, South Bend, IN. By McConnell R, Hartle R. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 1985–0171–1710.
- NIOSH [1994]. NIOSH manual of analytical methods (NMAM). 4th ed. Schlecht PC, O'Connor PF, eds. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication 94–113 (August, 1994); 1st Supplement Publication 96–135; 2nd Supplement Publication 98–119; 3rd Supplement 2003–154.
- NIOSH [2006]. Hazard evaluation and technical assistance report: Gilster-Mary Lee Corporation, Jasper, MO. By Kanwal R, Kullman G, Fedan K, Kreiss K. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2000–0401–2991.
- NIOSH [2007a]. Hazard evaluation and technical assistance report: Carmi Flavor and Fragrance Company, Inc., Commerce, CA. By Kanwal R, Kullman G. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2006–0303–3043.
- NIOSH [2007b]. Hazard evaluation and technical assistance report: Yatsko's Popcorn, Sand Coulee, MT. By Kullman G, Sahakian N. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2006–0195–3044.
- NIOSH [2008a]. Evaluating occupational exposures and work practices at Agilex Flavors, Inc. formerly Key Essentials, Inc., Rancho Santa Margarita, CA. A technical assistance report to the California/Occupational Safety and Health Administration. By McKernan L, Dunn K. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2006–0361–2.
- NIOSH [2008b]. Hazard evaluation and technical assistance report: Gold Coast Ingredients, Inc., Commerce, CA. By Bailey R, McKernan L, Dunn K, Sahakian N, Kreiss K. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2007–0033–3074.
- NIOSH [2008c]. In-depth survey report: evaluation of engineering controls for the mixing of flavoring chemicals: Gold Coast Ingredients, Inc., Commerce, CA. By Dunn K, Echt A, Garcia A. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for

- Occupational Safety and Health, NIOSH EPHB Report No. 322-11a.
- NIOSH [2009a]. Hazard evaluation and technical assistance report. Report on an investigation of buttermilk flavoring exposures and respiratory health at a bakery mix production facility, General Mills, Los Angeles, CA. By Day G, Cummings K, Kullman G. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2008-0230-3096.
- NIOSH [2009b]. Hazard evaluation and technical assistance report. Respiratory symptoms in workers at three commercial kitchens, Aramark, New York, NY. By Gaughan D, Boylstein R, Iossifova Y, Piacitelli C, Bailey R, Day G. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2008-0125-0126-0127-3093.
- NIOSH [2009c]. Hazard evaluation and technical assistance report: Chr. Hansen, Inc., New Berlin, WI. By Sahakian N, Kullman G, Dunn K, Kanwal R. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2007-0327-3083.
- NIOSH [2011]. Hazard evaluation and technical assistance report: Lung function (spirometry) testing in employees at a flavorings manufacturing plant—Indiana. By Kreiss K, Piacitelli C, Cox-Ganser J. Morgantown, WV: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2008-0155-3131.
- NIOSH [2013a]. Hazard evaluation and technical assistance report: An evaluation of respiratory health at a flavoring manufacturing facility—Kentucky. By Cummings K, Boylstein R, Stanton M, Piacitelli C. Morgantown, WV: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2012-0012-3192.
- NIOSH [2013b]. Hazard evaluation and technical assistance report: Evaluation of respiratory concerns at a cream cheese manufacturing facility—New York. By Bailey R, Piacitelli C. Morgantown, WV: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2011-0102-3194.
- NIOSH [2013c]. Hazard evaluation and technical assistance report: Evaluation of respiratory concerns at a snack food production facility—New York. By Cummings K, Piacitelli C, Stanton M, Bailey R. Morgantown, WV: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2011-0037-3172.
- OSHA [2003]. OSHA Sampling and Analytical Methods: Diacetyl PV2118. <http://www.osha.gov/dts/sltc/methods/partial/t-pv2118/t-pv2118.html>. Date accessed: June 2009.
- OSHA [2008a]. OSHA Sampling and Analytical Methods: Acetoin/Diacetyl 1012. <http://www.osha.gov/dts/sltc/methods/validated/1012/1012.html>. Date accessed: June 2009.
- OSHA [2008b]. OSHA Sampling and Analytical Methods: Acetoin/Diacetyl 1013. <http://www.osha.gov/dts/sltc/methods/validated/1013/1013.html>. Date accessed: June 2009.
- OSHA [2010]. OSHA Sampling and Analytical Methods: 2,3-Pentanedione 1016. <http://www.osha.gov/dts/sltc/methods/validated/1016/1016.html>. Date accessed: July 2013.
- Pierce JS, Abelmann A, Lotter JT, Comerford C, Keeton K, Finley BL [2015]. Characterization of naturally occurring airborne diacetyl concentrations associated with the preparation and consumption of unflavored coffee. *Toxicol Rep* 2:1200-1208.
- Rodrigues JA, Barros AA, Rodrigues PG [1999]. Differential pulse polarographic determination of alpha-dicarbonyl compounds in foodstuffs after derivatization with o-phenylenediamine. *J Agric Food Chem* 47(8):3219-3222.
- van Rooy FG, Rooyackers JM, Prokop M, Houba R, Smit LA, Heederik DJ [2007]. Bronchiolitis obliterans syndrome in chemical workers producing diacetyl for food flavorings. *Am J Respir Crit Care Med* 176(5):498-504.
- White KL [2011]. Correction to microwave popcorn plant diacetyl exposure data. *J Occup Environ Hyg* 8(4) (Comment Letter):D25-26.
- White KL, Heikkila K, Williams R, Levin L, Lockey JE, Rice C [2010]. Diacetyl exposures at four microwave popcorn plants. *J Occup Environ Hyg* 7(4):185-193.

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