

Diacetyl Emissions and Airborne Dust from Butter Flavorings Used in Microwave Popcorn Production

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In microwave popcorn workers, exposure to butter flavorings has been associated with fixed obstructive lung disease resembling bronchiolitis obliterans. Inhalation toxicology studies have shown severe respiratory effects in rats exposed to vapors from a paste butter flavoring, and to diacetyl, a diketone found in most butter flavorings. To gain a better understanding of worker exposures, we assessed diacetyl emissions and airborne dust levels from butter flavorings used by several microwave popcorn manufacturing companies. We heated bulk samples of 40 different butter flavorings (liquids, pastes, and powders) to approximately 50°C and used gas chromatography, with a mass selective detector, to measure the relative abundance of volatile organic compounds emitted. Air sampling was conducted for diacetyl and for total and respirable dust during the mixing of powder, liquid, or paste flavorings with heated soybean oil at a microwave popcorn plant. To further examine the potential for respiratory exposures to powders, we measured dust generated during different simulated methods of manual handling of several powder butter flavorings. Powder flavorings were found to give off much lower diacetyl emissions than pastes or liquids. The mean diacetyl emissions from liquids and pastes were 64 and 26 times larger, respectively, than the mean of diacetyl emissions from powders. The median diacetyl emissions from liquids and pastes were 364 and 72 times larger, respectively, than the median of diacetyl emissions from powders. Fourteen of 16 powders had diacetyl emissions that were lower than the diacetyl emissions from any liquid flavoring and from most paste flavorings. However, simulated handling of powder flavorings showed that a substantial amount of the airborne dust generated was of respirable size and could thus pose its own respiratory hazard. Companies that use butter flavorings should consider substituting flavorings with lower diacetyl emissions and the use of ventilation and enclosure engineering controls to minimize exposures. Until controls are fully implemented, companies should institute mandatory respiratory protection for all exposed workers.

Keywords bronchiolitis obliterans, butter flavoring, diacetyl, microwave popcorn

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INTRODUCTION

The National Institute for Occupational Safety and Health (NIOSH) has responded to multiple requests for health hazard evaluations to evaluate exposures and lung disease risk in microwave popcorn production workers. To date, NIOSH has performed evaluations at six microwave popcorn plants.⁽¹⁾ NIOSH received the first request in August 2000, when eight former workers at a microwave popcorn plant were reported to have the rare and disabling lung disease, bronchiolitis obliterans. This disease affects the small distal airways of the lung (bronchioles). Four of the eight worked as mixers of butter flavorings, soybean oil, salt, and colorings.^(2–4) NIOSH lung function tests in current workers at this plant revealed that the prevalence of abnormal lung function increased with increasing cumulative exposure to diacetyl, a diketone found commonly in butter flavorings and the predominant volatile organic compound (VOC) in the air of the plant. Toxicity studies conducted at NIOSH revealed damage to nasal passages and pulmonary airways in rats exposed to butter flavoring vapors, or to pure diacetyl, at concentrations similar to those found in the headspace of heated mixing and holding tanks at the index plant.^(5,6)

Microwave popcorn companies use a wide range of butter flavoring products to achieve variations in taste for the many types of popcorn they manufacture. Flavoring companies supply butter flavorings to the microwave popcorn producers as powders, liquids, or pastes. Workers measure flavorings in open containers and pour the ingredients into open tanks of heated soybean oil (approximate temperature 50°C). The oil and flavoring mixture, which also contains salt and coloring, is then transferred via pipes to packaging equipment where a measured amount is injected into bags containing popcorn kernels.

Diacetyl (2,3-butanedione) is the primary chemical used in food products to produce a buttery flavor and is a naturally occurring chemical found in butter, beer, and other food products.⁽⁷⁾ It is approved by the United States Food and Drug Administration for use in foods. Although diacetyl was known

as a potential eye and respiratory tract irritant prior to the report of lung disease in microwave popcorn workers, there were no previous reports in the published scientific literature that indicated that it might cause lung disease. To characterize exposures from different types of butter flavorings, NIOSH performed laboratory analyses of bulk flavoring samples and also performed air sampling during the use and handling of different types of flavorings at a microwave popcorn plant.

METHODS

Laboratory Analyses

We collected 40 bulk samples of butter flavorings (16 powders, 11 pastes, and 13 liquids) during a 3-year period from six different microwave popcorn production plants. The flavorings were made by 14 different flavor manufacturers. Laboratory analyses were conducted by NIOSH in the Division of Applied Research and Technology. The bulk flavorings were stored refrigerated prior to analysis and generally analyzed within 1 month of collection. Approximately 30 mg of each liquid, paste, or powder sample was placed in a glass tube and secured at both ends with glass wool. Each bulk sample was rapidly heated to 50°C and held for 10 min in a thermal desorption system (model 400 automatic thermal desorption system; PerkinElmer, Inc., Boston, Mass.). The thermal unit was interfaced directly to a gas chromatograph (GC, model 6890A gas chromatograph; Agilent Technologies, Inc., Palo Alto, Calif.), with a mass selective detector (model 5973 mass selective detector; Agilent) that was operated under normal electron impact conditions. A 30-m, DB-1 fused silica capillary column was used in the GC to separate components. Compounds were then identified by mass spectrometry by matching fragmentation patterns of the individual peaks to library spectra (Wiley 275 mass spectral library software). The chromatograms resulting from the analyses recorded the relative (not absolute) abundance of each chemical detected vs. a retention time. The area under the diacetyl peak was integrated electronically, and a ratio of peak area to the weight of material used, in micrograms, was calculated to allow for a comparison of diacetyl abundance from different bulk samples.

Air Sampling at a Microwave Popcorn Plant

In the room where butter flavorings were added to heated soybean oil as part of the microwave popcorn production process (i.e., the mixing room), we performed quantitative air sampling for VOCs, including diacetyl, and for total and respirable dust. Diacetyl samples were collected on carbon molecular sieve (CMS) tubes at a flow rate of 0.05 L/min and were analyzed quantitatively by gas chromatography according to NIOSH Method 2557⁽⁸⁾ with a limit of detection of approximately 0.001 ppm. We also assessed the potential for worker exposures to butter flavoring agents and diacetyl as solid particulates by dust sampling. Total dust samples were collected at a flow rate of 2.0 L/min on polyvinyl chloride (PVC) filters and respirable dust samples at 4.2 L/min on PVC filters behind a cyclone (GK 2.69

Respirable/Thoracic Cyclone; BGI Incorporated, Waltham, Mass.). The samples were analyzed gravimetrically according to NIOSH Methods 0500 and 0600, respectively.⁽⁸⁾ We obtained air samples from 8-hour work shifts over 3 days. On one of the days, only liquid and paste flavorings were used, whereas only powder flavorings were used during the other 2 days.

Within the same plant, in a separate room that was used to store and measure quantities of flavorings, we used a real-time optical particle counter (OPC, model 1.108; Grimm Technologies, Inc., Douglasville, Ga.) with simultaneous videotaping during the handling of four different powder butter flavorings to examine the potential for respiratory exposure. The Grimm OPC measures airborne particle concentrations in multiple size ranges regardless of chemical or physical form of the particles. These measurements were obtained while a NIOSH investigator scooped the powders from their containers into a weighing tub in three different handling manners: (1) *easy*: simulating an operator gently scooping powder; (2) *hurried*: simulating a worker who might be rushed while performing the task; and (3) *dropped*: where the powder was dropped from a height of 1 ft into the tub to simulate an extremely hastened transfer of powder.

RESULTS

Over 150 different VOCs were identified in the emissions from the bulk samples of butter flavorings. Diacetyl was one of the most abundant compounds found in all of the butter flavorings. Other compounds commonly detected were the ketones—acetoin and 2-nonanone—and butyric and acetic acids. Figures 1a to 1c show representative chromatograms obtained from heating a liquid, paste, and powder butter flavor (note that some compound peaks exceed the upper scale limits for these figures). The diacetyl emissions (abundances) from different flavorings, expressed as ratios of the area under the diacetyl peak to the weight of the bulk sample, are presented in Table I. The mean diacetyl abundances from liquids and pastes were 64 and 26 times larger, respectively, than the mean of diacetyl abundances from powders. The median diacetyl abundances from liquids and pastes were 364 and 72 times larger, respectively, than the median of diacetyl abundances from powders. The volatile diacetyl abundances for powders was statistically different from both liquids and pastes ($p < 0.001$). Fourteen of 16 powders had diacetyl abundances that were lower than the diacetyl abundances from any liquid flavoring, and also lower than the diacetyl abundances from most paste flavorings. A graphical comparison of diacetyl abundances from the three flavoring physical forms is presented in Figure 2.

Table II shows levels of total dust, respirable dust, and diacetyl during 3 consecutive days of air sampling in the mixing room of a microwave popcorn plant. During Day 1, only pastes and liquids were used, and the resulting 8-hour time-weighted average (TWA) area diacetyl air concentration was 0.57 ppm. During the next 2 days of sampling, only powder flavorings were used, and the resulting diacetyl air concentrations were

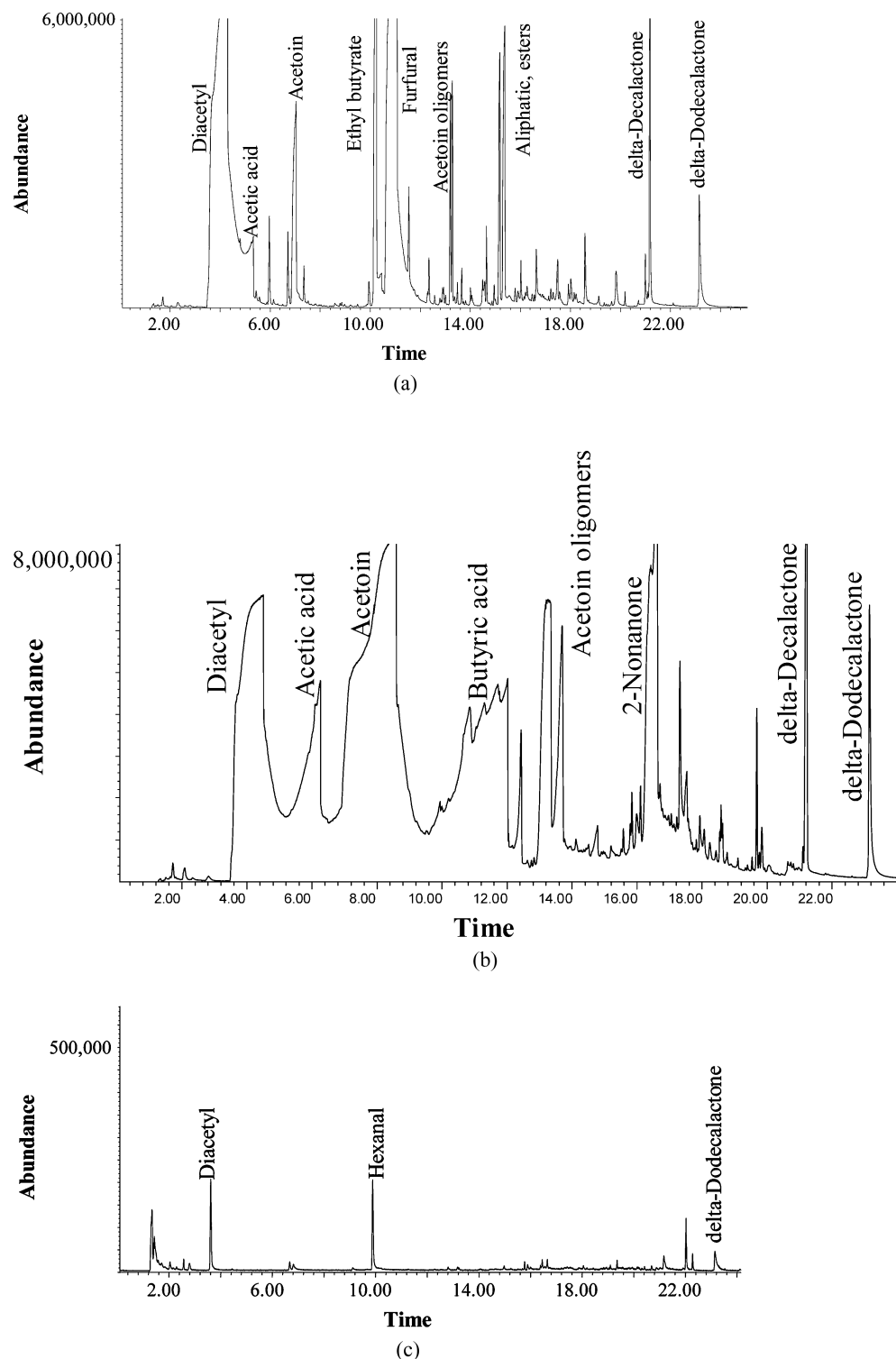


FIGURE 1. Thermal desorption-gas chromatography-mass spectrometry chromatograms of a bulk liquid, paste, and powder butter flavoring, respectively, when heated to 50°C. (a) Liquid, (b) Paste, (c) Powder

below the limit of detection (0.001 ppm). Total and respirable dust air concentrations were lowest when only pastes and liquids were used. Notably, respirable dust levels were 3 and 3.4 times higher on the 2 days when powder flavors were used.

The simulated handling of four powders used at this plant generated airborne dust that contained particles of respirable size (i.e., less than 10 μm in aerodynamic diameter) as shown in Figure 3. The highest air concentrations occurred when the

powders were dropped from a 1-foot-height into a weighing tub. One of the flavors, Powder 2 (oiled), had been misted with soybean oil by the flavoring manufacturer and generated much less dust, even when dropped from a height of 1 foot.

DISCUSSION

Butter flavorings can emit complex mixtures of VOCs during the production of microwave popcorn. Diacetyl is one of the predominant compounds found in most butter flavorings and has potential to cause respiratory damage.⁽⁶⁾ The lower diacetyl vapor emissions from powder flavorings (vs. liquid or paste flavorings) may offer an opportunity for substitution as a control method. However, the potential for respiratory health effects from powder flavoring particles containing diacetyl needs further study, especially those particles of respirable size that could be deposited in the bronchioles.

Powder butter flavorings are available in different forms, such as spray dried (encapsulated) or plated. Plated powders do not prevent evaporation of VOCs before and during use, but spray dried flavors are designed to limit the volatilization of the flavoring chemicals. Both spray-dried and plated powder types are generally water soluble and form a suspension when mixed with oil. The spray-dried variety keeps the flavoring chemicals trapped until steam from the popped corn causes release during heating in the microwave oven.

TABLE I. Summary of Diacetyl Abundance Found in Butter Flavors

	Liquid	Paste	Powder
	322,493	112,642	16,273
	248,936	82,396	4,322
	148,855	78,846	389
	103,698	65,385	342
	87,021	40,699	194
	57,546	10,604	152
	53,889	4,506	150
	50,493	2,597	148
	39,944	931	147
	31,234	117	140
	28,209	74	123
	2,508	—	38
	1,961	—	22
	—	—	20
	—	—	20
	—	—	10
Mean	90,522	36,254	1,406
Median	53,889	10,604	148

Note: Abundance is expressed as peak area divided by mass of sample in micrograms. Dashes indicate no sample collected.

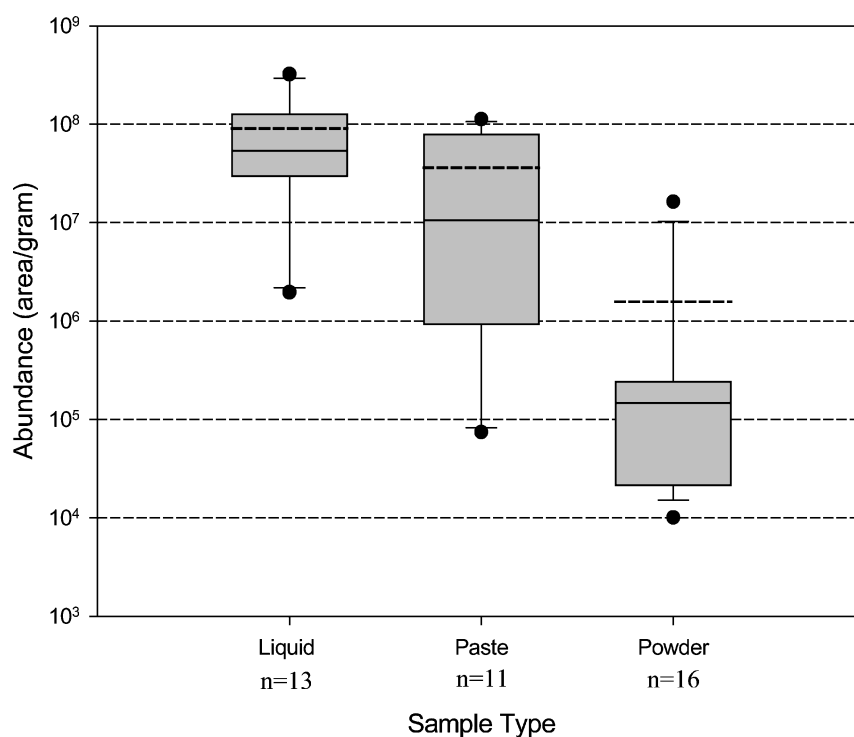


FIGURE 2. Box plots of the abundance (log scale) found in diacetyl abundances from the 40 butter flavors when heated to 50°C. The lower boundary indicates the twenty-fifth percentile, the solid line in the box marks the median, the dashed line marks the mean, and the upper boundary of the box indicates the seventy-fifth percentile. The error bars above and below mark the ninetieth and tenth percentiles. The dots mark the outliers.

TABLE II. Levels of Dust and Diacetyl in Mixing Room Air

Day	Total Dust (mg/m ³)	Respirable Dust (mg/m ³)	Diacetyl ^A (ppm)	Type of Flavoring
1	1.4	0.09	0.57	Liquid and Pastes
2	3.8	0.31	ND	Powder
3	2.2	0.27	ND	Powder

Notes: One full-shift, time-weighted area sample per analyte was collected each day over 3 consecutive days. ND = not detectable; diacetyl (in gas or vapor phase) measurements were below the limit of detection (0.001 ppm).

NIOSH did not have information on the formulation of the powders tested (i.e., spray dried vs. plated). However, the potential for diacetyl vapor exposures from powders is illustrated by the two powders with higher diacetyl vapor release as contrasted with the other powders in Table I. The fact that powder flavorings are water soluble presents a potential respiratory hazard if the powder is deposited

onto moist respiratory tissue. The delivery of water soluble diacetyl or other flavoring agents directly to the bronchioles in particles (by impaction or sedimentation) may generate exposure hazards comparable to those from gas and vapor exposures. However, the effects of these respirable particle exposures on the respiratory system are not known. Additional toxicology studies are necessary to help resolve this issue.

The results of our air sampling during simulated open handling of different powder flavors revealed the presence of particles of respirable size from all four powders tested, indicating the potential for inhalation of this airborne dust into the lungs. Powders designed to prevent the release of VOCs during use and that generate little airborne respirable dust (e.g., spray-dried, oil-misted powders) may pose less exposure risk to workers. Additionally, care should be taken when handling powders to minimize the generation of airborne dust.

Our findings from air sampling at a microwave popcorn plant complemented our laboratory findings regarding diacetyl emissions from paste, liquid, and powder butter flavorings. Volatile diacetyl air concentrations were orders of magnitude lower when powders were used as contrasted with paste or

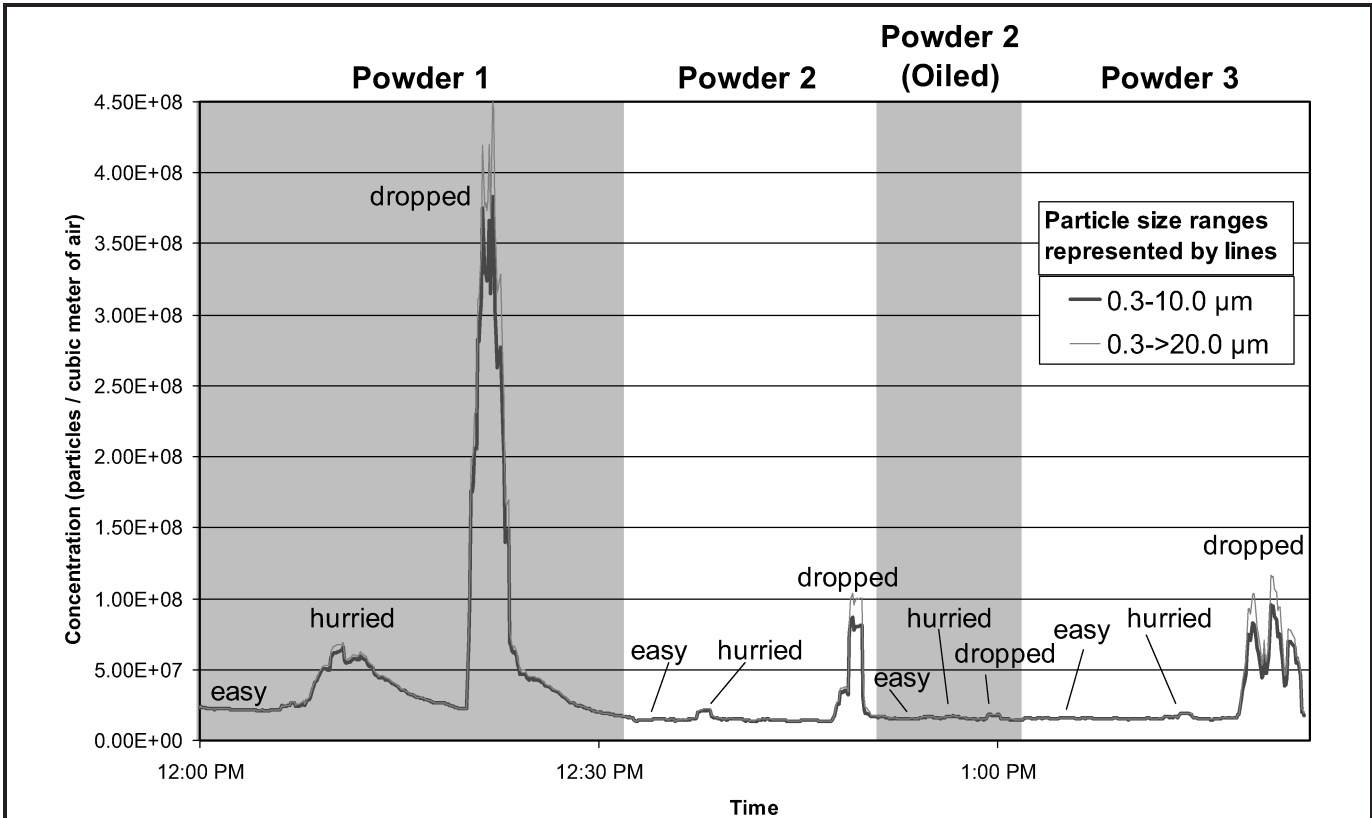


FIGURE 3. Real-time air sampling data during simulated hand scooping of four different powder butter flavorings. These measurements were obtained while a NIOSH investigator scooped the powders from their containers into a weighing tub in three different handling manners: *easy*—simulating an operator gently scooping powder; *hurried*—simulating a worker who might be rushed while performing the task; and *dropped*—where the powder was dropped from a height of 1 ft into the tub to simulate an extremely hastened transfer of powder.

liquid use during production. However, respirable dust levels were higher on days when powders were being used.

CONCLUSIONS

A workplace exposure standard or recommended exposure limit for diacetyl has not been established, and the same is true for many of the other chemicals found in butter flavorings.⁽⁹⁾ Until more is known regarding safe exposure levels for diacetyl and other chemicals in butter flavorings, the substitution of flavorings designed to reduce volatile emissions and respirable dusts (e.g., spray-dried, oil-misted powders) is recommended. Production and use of these flavorings should utilize closed processes whenever possible. Process isolation, along with sufficient local exhaust and general dilution ventilation, should be implemented for any open handling of butter flavoring ingredients. In addition, company management should institute mandatory respiratory protection (as part of a respiratory protection program that complies with the OSHA Respiratory Protection Standard, 29 CFR 1910.134) for workers exposed to airborne flavoring chemicals or dusts. Companies should provide workers with NIOSH-certified, half-facepiece, negative pressure respirators (or a more protective type) with organic vapor cartridges and particulate filters (oil-resistant or oil-proof filters if oil aerosols are present).

REFERENCES

1. Kanwal, R., G. Kullman, C. Piacitelli, R. Boylstein, N. Sahakian, S. Martin, K. Fedan, and K. Kreiss: Evaluation of flavorings-related lung disease risk at six microwave popcorn plants. *J. Occup. Environ. Med.* 48:149–157 (2006).
2. Kreiss, K., A. Goma, G. Kullman, K. Fedan, E. Simoes, and P. Enright: Clinical bronchiolitis in workers at a microwave-popcorn plant. *N. Engl. J. Med.* 347:330–338 (2002).
3. Akpınar-Elci, M., W.D. Travis, D.A. Lynch, and K. Kreiss: Bronchiolitis obliterans syndrome in popcorn plant workers. *Eur. Respir. J.* 24:298–302 (2004).
4. Kullman, K., R. Boylstein, W. Jones, C. Piacitelli, S. Pendergrass, and K. Kreiss: Characterization of respiratory exposures at a microwave popcorn plant with cases of bronchiolitis obliterans. *J. Occup. Environ. Hyg.* 2:169–178 (2005).
5. Hubbs, A.F., L.A. Battelli, W.T. Goldsmith, et al.: Necrosis of nasal and airway epithelium in rats inhaling vapors of artificial butter flavoring. *Toxicol. Appl. Pharmacol.* 185:128–135 (2002).
6. Hubbs, A.F., L.A. Battelli, R.R. Mercer, et al.: Inhalation toxicity of the flavoring agent, diacetyl (2,3-butanedione), in the upper respiratory tract of rats. *Toxicol. Sci.* 78(S1):438–439 (2004).
7. “National Toxicology Program. 431-03-8 Chemical.” [Online] Available at <http://ntp.niehs.nih.gov> (Accessed October 21, 2005).
8. National Institute for Occupational Safety and Health (NIOSH): Diacetyl, Method 2557. In *Manual of Analytical Methods (NMAM)*, 4th edition, Third Supplement, P.C. Schlecht and P.F. O'Connor (eds.). Cincinnati, Ohio: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Pub. No. 2003-154, 2003.
9. National Institute for Occupational Safety and Health (NIOSH): *ALERT: Preventing Lung Disease in Workers Who Use or Make Flavorings*. Cincinnati, Ohio: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Pub. No. 2004-110, 2003.