

IX. APPENDIX I

SAMPLING METHOD FOR AIRBORNE BENZOYL PEROXIDE

The sampling method for airborne benzoyl peroxide is adapted from general particulate sampling methods [98,99, R O'Gee, written communication, August 1976].

General Requirements

Collect breathing zone samples representative of the individual employee's exposure. Collect enough samples to permit calculation of a TWA concentration for every operation or location in which there is exposure to benzoyl peroxide. At the time of sample collection, record the sampling location and conditions, equipment used, time and rate of sampling, individual performing the sampling, and any other pertinent information.

Air Sampling

(a) Collect breathing-zone samples as close as practicable to the employee's face, without interfering with the employee's freedom of movement. This may be accomplished by use of a glass-fiber filter mounted in a personal cassette attached to the employee's clothing. The samples should characterize the exposure from each job or specific operation dealing with the manufacture, formulation, or application of benzoyl peroxide. Sampling flow rates should be checked frequently. If filters become clogged so that airflow is too restricted, change the filters and

initiate the collection of new samples.

(b) Collect samples using a portable, explosion-proof sampling pump whose flow can be determined to an accuracy of $\pm 5\%$ at 1-2 liters/minute. Connect the pump to the filter unit, which consists of a preweighed glass-fiber filter (Type A, 37 mm in diameter) mounted in a polystyrene, 37-mm, two-piece cassette holder and supported by a backup pad. The filter should be weighed to the nearest 0.01 mg after being brought to a constant weight in a chamber that is kept at a constant humidity by a 42.5% aqueous sulfuric acid solution [100].

(c) Static charges should be dissipated on the balance and filter with a nuclear static eliminator during all weighing operations.

(d) Operate the pump at a known flow rate of 2 liters/minute for a maximum of 1 hour/sample. Record the total sampling time. A sample size of 90 liters is recommended. Before reweighing the filter after sampling, bring it to constant relative humidity (50%) in a chamber containing an aqueous sulfuric acid solution (42.5%).

(e) With each batch of 10 samples, submit one filter from the same lot used for sample collection, subjecting it to exactly the same handling as the samples except that no air is drawn through it. Label this as a blank.

Calibration of Sampling Trains

The accurate calibration of a sampling pump is essential for the correct interpretation of the volume indicated. The frequency of calibration is dependent on the use, care, and handling to which the pump is subjected. Pumps should be recalibrated if they have been misused or if

they have just been repaired or received from a manufacturer. If the pump receives hard use, more frequent calibration may be necessary. Regardless of use, maintenance and calibration should be performed on a regular schedule, and records of these operations should be kept.

Ordinarily, pumps should be calibrated in the laboratory both before and after they are used in the field. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely on where the calibration is to be performed. For laboratory testing, a 1- or 2-liter buret for a soapbubble calibration or wet-test meter is recommended, although other standard calibrating instruments, such as a spirometer, Marriott's bottle, or dry-gas meter, can be used.

Instructions for calibration with the soapbubble meter follow. If another calibration device is selected, equivalent procedures should be used. Since the flow rate is dependent on the pressure drop of the sampling device, the pump must be calibrated while being operated with a representative filter and backup pad in line.

(a) While the pump is running, check the voltage of the pump battery with a voltmeter to assure that it is adequate for calibration. Charge the battery if necessary.

(b) Turn on the pump and immerse the buret in the soap solution; draw bubbles up the inside until they are able to travel the entire length of the buret without bursting.

(c) Adjust the pump flow controller to provide the desired flow rate.

(d) Start a soapbubble up the buret and measure with a stopwatch the time the bubble takes to move from one calibration mark to another.

(e) Repeat the procedure in (d) at least three times, average the results, and calculate the flow rate by dividing the volume between the preselected marks by the time required for the soapbubble to traverse the distance. If, for the pump being calibrated, the volume of air sampled is the product obtained by multiplying the number of strokes times a stroke factor (given in units of volume/stroke), the stroke factor is the quotient obtained by dividing the volume between the two preselected marks by the number of strokes.

(f) Data for the calibration include volume measured, elapsed time or number of strokes, pressure drop, air temperature, atmospheric pressure, serial number of the pump, date, and the name of the person performing the calibration.

X. APPENDIX II

ANALYTICAL METHOD FOR BENZOYL PEROXIDE

An initial gravimetric analysis for total dust should be performed with a preweighed glass-fiber filter [98,99, R O'Gee, written communication, August 1976]. If the total airborne dust exceeds the environmental limit for benzoyl peroxide, a colorimetric analysis, adapted from the method described by Dolin [62], should be performed on the particles trapped in the filter.

Principle of the Method

(a) Air samples are drawn through glass-fiber filters; the filters are then analyzed by a general gravimetric method.

(b) If the total airborne dust exceeds the environmental limit for benzoyl peroxide, a chemical analysis should be performed, using the filter(s) that collected airborne dust in excess of the environmental limit.

(c) The preferred chemical method, a colorimetric analysis, is based on the development of color in an aqueous solution of starch and potassium iodide.

(d) The intensity of the blue color resulting from the released iodine is read in a spectrophotometer. The concentration of benzoyl peroxide in the sample is read from a standard curve prepared from standard solutions of benzoyl peroxide treated the same as the sample solutions.

The concentration of benzoyl peroxide in the sample can also be determined visually by comparing the color of the sample to standard solutions of known concentrations.

Range and Sensitivity

With a spectrophotometer, the lower limit of the working range is 1 $\mu\text{g}/\text{sample}$; when visual comparisons are made, the lower limit is 3 $\mu\text{g}/\text{sample}$. There is no upper limit because the sample solutions can always be sufficiently diluted to allow spectrophotometric readings within the limits set by standard curves or to match the absorption of standard solutions.

Interferences

Other peroxides will also react in the colorimetric analysis to release the iodine, and other oxidizing or reducing agents present in the sample may interfere.

Advantages of the Method

- (a) It provides a method suitable for determination of total peroxides in the air.
- (b) The sampling device is small and portable and involves no liquids.
- (c) The analysis is readily accomplished.
- (d) No elaborate equipment is required.

Disadvantage of the Method

The method is not specific for benzoyl peroxide.

Precision and Accuracy

(a) The precision and accuracy of the gravimetric sampling method is defined by the limits of sensitivity of the balance used to weigh the filter.

(b) It is important that the standard and sample solutions are prepared and used at the same time. An error of as high as 25% may occur if the standard solutions have been prepared as little as 1 day before the sample solution. No further data were given on the accuracy or precision of this method.

Apparatus

(a) Spectrophotometer.

(b) Cuvettes that allow the reading of solutions in the spectrophotometer.

(c) Balance for gravimetric analysis.

Reagents

(a) Standard aqueous solutions of known concentrations of benzoyl peroxide, 0.5-10 μg in increments of 0.5 μg are suggested.

(b) Aqueous potassium iodide, 0.75% (w/v).

(c) Aqueous starch solution, 0.5% (w/v).

Analysis of Samples

(a) Gravimetric analysis

(1) A glass-fiber filter is placed in a chamber over an aqueous sulfuric acid solution for 24 hours to bring the filter to a constant weight at 50% relative humidity.

(2) The initial weight of the glass-fiber filter is recorded to the nearest 0.01 mg. A nuclear static eliminator on the balance will remove static charges that might interfere with obtaining accurate, reproducible weights of the filter.

(3) A known volume of air is drawn through the preweighed glass-fiber filter to collect airborne dust, including airborne benzoyl peroxide.

(4) After sampling, the filter is replaced in the chamber for 24 hours and again brought to a constant weight at 50% humidity.

(5) The filter is reweighed on the balance used for the preweighing, and the weight is recorded to the nearest 0.01 mg. If the difference in the initial and final weights of the filter, divided by the known volume of air sampled, equals or is less than the environmental limit for benzoyl peroxide, nothing further need be done.

Filter(s) found to contain a dust concentration higher than the environmental limit should be analyzed by the following colorimetric procedure.

(b) Colorimetric analysis

(1) Put each glass-fiber filter that was used for the sampling of total dust in a clean, dry flask with 10 ml of double-distilled water, 1 ml of potassium iodide solution, and 1 ml of starch solution. Mix

the contents of the flask and allow it to stand for 12 hours.

(2) Treat portions of each of the standard aqueous benzoyl peroxide solutions in a similar manner.

(3) Determine the absorption of the sample solution and of the standards at the absorption band maximum in the spectrophotometer. Make dilutions of the sample solutions if necessary.

(4) Construct a standard curve of the percent transmittance versus μg benzoyl peroxide, using the data obtained from the standard solutions.

(5) Read the concentrations of the sample solutions from the standard curve, or visually compare the standard and sample solutions to obtain the closest color match and the corresponding concentration of benzoyl peroxide. This visual comparison can be performed instead of steps (c) and (d) when less sensitivity is needed.

(6) The concentration of benzoyl peroxide in air can be expressed as milligrams of benzoyl peroxide/cu m of air, which is numerically equal to micrograms of benzoyl peroxide/liter of air:

$$\text{mg benzoyl peroxide/cu m} = \mu\text{g benzoyl peroxide/V}$$

where:

$$\begin{aligned} \mu\text{g benzoyl peroxide} &= \text{micrograms of benzoyl peroxide} \\ &\quad \text{(from the calibration curve)} \\ V &= \text{volume of air sampled (in liters) at 25 C and} \\ &\quad 760 \text{ mmHg} \end{aligned}$$

XI. APPENDIX III
MATERIAL SAFETY DATA SHEET

The following items of information which are applicable to a specific product or material shall be provided in the appropriate block of the Material Safety Data Sheet (MSDS).

The product designation is inserted in the block in the upper left corner of the first page to facilitate filing and retrieval. Print in upper case letters as large as possible. It should be printed to read upright with the sheet turned sideways. The product designation is that name or code designation which appears on the label, or by which the product is sold or known by employees. The relative numerical hazard ratings and key statements are those determined by the rules in Chapter V, Part B, of the NIOSH publication, An Identification System for Occupationally Hazardous Materials. The company identification may be printed in the upper right corner if desired.

(a) Section I. Product Identification

The manufacturer's name, address, and regular and emergency telephone numbers (including area code) are inserted in the appropriate blocks of Section I. The company listed should be a source of detailed backup information on the hazards of the material(s) covered by the MSDS. The listing of suppliers or wholesale distributors is discouraged. The trade name should be the product designation or common name associated with the material. The synonyms are those commonly used for the product, especially formal chemical nomenclature. Every known chemical designation or

competitor's trade name need not be listed.

(b) Section II. Hazardous Ingredients

The "materials" listed in Section II shall be those substances which are part of the hazardous product covered by the MSDS and individually meet any of the criteria defining a hazardous material. Thus, one component of a multicomponent product might be listed because of its toxicity, another component because of its flammability, while a third component could be included both for its toxicity and its reactivity. Note that a MSDS for a single component product must have the name of the material repeated in this section to avoid giving the impression that there are no hazardous ingredients.

Chemical substances should be listed according to their complete name derived from a recognized system of nomenclature. Where possible, avoid using common names and general class names such as "aromatic amine," "safety solvent," or "aliphatic hydrocarbon" when the specific name is known.

The "%" may be the approximate percentage by weight or volume (indicate basis) which each hazardous ingredient of the mixture bears to the whole mixture. This may be indicated as a range or maximum amount, ie, "10-40% vol" or "10% max wt" to avoid disclosure of trade secrets.

Toxic hazard data shall be stated in terms of concentration, mode of exposure or test, and animal used, eg, "100 ppm LC50-rat," "25 mg/kg LD50-skin-rabbit," "75 ppm LC man," or "permissible exposure from 29 CFR 1910.1000," or, if not available, from other sources of publications such as the American Conference of Governmental Industrial Hygienists or the American National Standards Institute Inc. Flashpoint, shock sensitivity,

or similar descriptive data may be used to indicate flammability, reactivity, or similar hazardous properties of the material.

(c) Section III. Physical Data

The data in Section III should be for the total mixture and should include the boiling point and melting point in degrees Fahrenheit (Celsius in parentheses); vapor pressure, in conventional millimeters of mercury (mmHg); vapor density of gas or vapor (air = 1); solubility in water, in parts/hundred parts of water by weight; specific gravity (water = 1); percent volatiles (indicated if by weight or volume) at 70 degrees Fahrenheit (21.1 degrees Celsius); evaporation rate for liquids or sublimable solids, relative to butyl acetate; and appearance and odor. These data are useful for the control of toxic substances. Boiling point, vapor density, percent volatiles, vapor pressure, and evaporation are useful for designing proper ventilation equipment. This information is also useful for design and deployment of adequate fire and spill containment equipment. The appearance and odor may facilitate identification of substances stored in improperly marked containers or when spilled.

(d) Section IV. Fire and Explosion Data

Section IV should contain complete fire and explosion data for the product, including flashpoint and autoignition temperature in degrees Fahrenheit (Celsius in parentheses); flammable limits, in percent by volume in air; suitable extinguishing media or materials; special firefighting procedures; and unusual fire and explosion hazard information. If the product presents no fire hazard, insert "NO FIRE HAZARD" on the line labeled "Extinguishing Media."

(e) Section V. Health Hazard Information

The "Health Hazard Data" should be a combined estimate of the hazard of the total product. This can be expressed as a TWA concentration, as a permissible exposure, or by some other indication of an acceptable standard. Other data are acceptable, such as lowest LD50 if multiple components are involved.

Under "Routes of Exposure," comments in each category should reflect the potential hazard from absorption by the route in question. Comments should indicate the severity of the effect and the basis for the statement if possible. The basis might be animal studies, analogy with similar products, or human experiences. Comments such as "yes" or "possible" are not helpful. Typical comments might be:

Skin Contact--single short contact, no adverse effects likely; prolonged or repeated contact, possibly mild irritation.

Eye Contact--some pain and mild transient irritation; no corneal scarring.

"Emergency and First Aid Procedures" should be written in lay language and should primarily represent first-aid treatment that could be provided by paramedical personnel or individuals trained in first aid.

Information in the "Notes to Physician" section should include any special medical information which would be of assistance to an attending physician including required or recommended preplacement and periodic medical examinations, diagnostic procedures, and medical management of overexposed employees.

(f) Section VI. Reactivity Data

The comments in Section VI relate to safe storage and handling of hazardous, unstable substances. It is particularly important to highlight instability or incompatibility to common substances or circumstances, such as water, direct sunlight, steel or copper piping, acids, alkalies, etc. "Hazardous Decomposition Products" shall include those products released under fire conditions. It must also include dangerous products produced by aging, such as peroxides in the case of some ethers. Where applicable, shelf life should also be indicated.

(g) Section VII. Spill or Leak Procedures

Detailed procedures for cleanup and disposal should be listed with emphasis on precautions to be taken to protect employees assigned to cleanup detail. Specific neutralizing chemicals or procedures should be described in detail. Disposal methods should be explicit including proper labeling of containers holding residues and ultimate disposal methods such as "sanitary landfill," or "incineration." Warnings such as "comply with local, state, and federal antipollution ordinances" are proper but not sufficient. Specific procedures shall be identified.

(h) Section VIII. Special Protection Information

Section VIII requires specific information. Statements such as "Yes," "No," or "If necessary" are not informative. Ventilation requirements should be specific as to type and preferred methods. Respirators shall be specified as to type and NIOSH or US Bureau of Mines approval class, ie, "Supplied air," "Organic vapor canister," etc. Protective equipment must be specified as to type and materials of construction.

(i) Section IX. Special Precautions

"Precautionary Statements" shall consist of the label statements selected for use on the container or placard. Additional information on any aspect of safety or health not covered in other sections should be inserted in Section IX. The lower block can contain references to published guides or in-house procedures for handling and storage. Department of Transportation markings and classifications and other freight, handling, or storage requirements and environmental controls can be noted.

(j) Signature and Filing

Finally, the name and address of the responsible person who completed the MSDS and the date of completion are entered. This will facilitate correction of errors and identify a source of additional information.

The MSDS shall be filed in a location readily accessible to employees exposed to the hazardous substance. The MSDS can be used as a training aid and basis for discussion during safety meetings and training of new employees. It should assist management by directing attention to the need for specific control engineering, work practices, and protective measures to ensure safe handling and use of the material. It will aid the safety and health staff in planning a safe and healthful work environment and in suggesting appropriate emergency procedures and sources of help in the event of harmful exposure of employees.

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MATERIAL SAFETY DATA SHEET

I PRODUCT IDENTIFICATION		
MANUFACTURER'S NAME	REGULAR TELEPHONE NO. EMERGENCY TELEPHONE NO.	
ADDRESS		
TRADE NAME		
SYNONYMS		
II HAZARDOUS INGREDIENTS		
MATERIAL OR COMPONENT	%	HAZARD DATA
III PHYSICAL DATA		
BOILING POINT, 760 MM HG		MELTING POINT
SPECIFIC GRAVITY (H ₂ O=1)		VAPOR PRESSURE
VAPOR DENSITY (AIR=1)		SOLUBILITY IN H ₂ O, % BY WT
% VOLATILES BY VOL		EVAPORATION RATE (BUTYL ACETATE=1)
APPEARANCE AND ODOR		

IV FIRE AND EXPLOSION DATA				
FLASH POINT (TEST METHOD)		AUTOIGNITION TEMPERATURE		
FLAMMABLE LIMITS IN AIR, % BY VOL.		LOWER		UPPER
EXTINGUISHING MEDIA				
SPECIAL FIRE FIGHTING PROCEDURES				
UNUSUAL FIRE AND EXPLOSION HAZARD				
V HEALTH HAZARD INFORMATION				
HEALTH HAZARD DATA				
ROUTES OF EXPOSURE				
INHALATION				
SKIN CONTACT				
SKIN ABSORPTION				
EYE CONTACT				
INGESTION				
EFFECTS OF OVEREXPOSURE				
ACUTE OVEREXPOSURE				
CHRONIC OVEREXPOSURE				
EMERGENCY AND FIRST AID PROCEDURES				
EYES				
SKIN:				
INHALATION:				
INGESTION				
NOTES TO PHYSICIAN				

VI REACTIVITY DATA	
CONDITIONS CONTRIBUTING TO INSTABILITY	
INCOMPATIBILITY	
HAZARDOUS DECOMPOSITION PRODUCTS	
CONDITIONS CONTRIBUTING TO HAZARDOUS POLYMERIZATION	
VII SPILL OR LEAK PROCEDURES	
STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED	
NEUTRALIZING CHEMICALS	
WASTE DISPOSAL METHOD	
VIII SPECIAL PROTECTION INFORMATION	
VENTILATION REQUIREMENTS	
SPECIFIC PERSONAL PROTECTIVE EQUIPMENT	
RESPIRATORY (SPECIFY IN DETAIL)	
EYE	
GLOVES	
OTHER CLOTHING AND EQUIPMENT	

IX SPECIAL PRECAUTIONS

PRECAUTIONARY
STATEMENTS

OTHER HANDLING AND
STORAGE REQUIREMENTS

PREPARED BY _____

ADDRESS _____

DATE _____

XII. APPENDIX IV

TWO TENTATIVE HAZARD CLASSIFICATION SYSTEMS

The following hazard classification system is adapted from a tentative NFPA definition of peroxide classes (O Mageli, written communication, January 1977).

Class I contains organic peroxide formulations which burn like Class II materials (ie, like nitrocellulose and Class IA flammable liquids) and deflagrate. These materials do not detonate.

Class II contains moderately reactive organic peroxide formulations which burn rapidly like polystyrene and Class C flammable liquids.

Class III contains moderately reactive organic peroxide formulations which burn rapidly like cellular polyethylene and Class II combustible liquids.

Class IV organic peroxide formulations have a reactivity hazard and, like wood, paper, and Class II combustible liquids, will not sustain combustion.

Class V contains organic peroxide formulations that will not sustain combustion.

The following hazard classification system for organic peroxides was developed by the Factory Mutual Research Corporation and was published in March, 1972 [72]. A table with further information on commercial products

containing benzoyl peroxide has been compiled by Factory Mutual Research Corporation [70].

Hazard classifications are based on tests designed to evaluate the total energy release, the rate of energy release, and the ease of ignition and/or decomposition and storage containers when exposed to normal temperatures and when exposed to heat, fire, or mechanical shock.

These tests include: burning rate (solids or pastes); flash point (liquids); impact sensitivity (drop weight test); self-accelerating decomposition temperature (temperature at which self-heating to decomposition is initiated) and the evaluation of the violence of this decomposition; heat exposure to a sample in a vented pressure vessel to evaluate rate and violence of decomposition; and lead block deformation test (exposure to heavy shock) to evaluate violence of decomposition.

Peroxides frequently do not react consistently in the various tests. Therefore, a peroxide may be classified on the basis of the most hazardous rating attained in any one of the series of tests.

Classification of each peroxide is based on its normal shipping container. If a peroxide is shipped in a different container or transferred to a different container, the normal hazard classification may no longer apply. In general, a stronger container will increase the hazard.

Classification is also based on a specific product of a specific manufacturer. The same type product in the same type of container by various manufacturers will not necessarily be in the same class. Differences in manufacturing procedures may have an effect on the hazard of a peroxide which can be determined only by tests.

The classifications are as follows:

Class I. Class I peroxides present a high explosion hazard through easily initiated, rapid explosive decomposition. This group may include peroxides that are relatively safe under highly controlled temperatures or in a liquid solution where loss of temperature control or crystallization out of solution can result in severe explosive decomposition.

Class II. Class II peroxides present an intermediate explosion hazard. That is, an explosive decomposition is not as rapid, violent or complete as that produced by a Class I

material. As with Class I materials, this group may also contain peroxides that are relatively safe under controlled temperatures or when mixed with a diluent.

Class III. Class III peroxides present moderate explosion and severe fire hazards. They have characteristics of rapid burning, high heat liberation or vapor-air explosion hazards of the products of decomposition.

Class IV. Class IV peroxides have moderate fire hazard characteristics that can be easily contained by normal sprinkler systems and fire walls.

Class V. Class V peroxides present a low or negligible fire hazard. With these peroxides, combustible packing materials may present a greater hazard than the peroxide itself.

XIII. APPENDIX V

INACTIVATION OF BENZOYL PEROXIDE

The following method for inactivating benzoyl peroxide for subsequent disposal is recommended for pure benzoyl peroxide (96-99%) and wet benzoyl peroxide formulations; "BPO-78" means 78% benzoyl peroxide plus 22% water.

Pure benzoyl peroxide (98+%) and water-wet benzoyl peroxide formulations (70% or 78% wetted products) can be hydrolyzed with dilute sodium hydroxide to form sodium benzoate and a solution of hydrogen peroxide in caustic. The hydrogen peroxide decomposes in the caustic solution.

Procedure

Slowly add the BPO-98 (BPO-78 or BPO-70) in small portions to a rapidly stirred 10% sodium hydroxide solution, the amount of such solution being 10 times the weight of the actual benzoyl peroxide to be hydrolyzed. The sodium hydroxide solution must be no warmer than room temperature [25 C] at the time of addition. The reaction is only mildly exothermic, so cooling is not necessary. When all the benzoyl peroxide has been added, continue stirring until the solution is free of solids. The solution will be cloudy. When the temperature is maintained at about 25 C, the time for hydrolysis will be about three hours.

When the solution is free of solids, the benzoyl peroxide has been hydrolyzed and the solution can be disposed of, in accordance with any regulations which apply to disposal of a dilute sodium hydroxide solution containing benzoic acid salts (O Mageli, written communication, January 1977).

XIV. TABLES

TABLE XIV-1

PHYSICAL AND CHEMICAL PROPERTIES OF BENZOYL PEROXIDE

Molecular formula	(C ₆ H ₅ CO) ₂ O ₂
Formula weight	242.23
Autoignition temperature	80 C
Specific gravity (25 C)	1.3340
Melting point	104 C (with explosive decomposition above 105 C)
Boiling point	Explodes
Solubility	Soluble in ethanol, diethyl ether, acetone, benzene, and carbon disulfide; very slightly soluble in water and methanol

Adapted from references 1 and 2

TABLE XIV-2

OCCUPATIONS WITH POTENTIAL EXPOSURE TO BENZOYL PEROXIDE

Automobile body repair workers	Pharmaceutical products makers
Bakers	Pharmacists
Benzoyl peroxide makers	Physicians
Cheesemakers	Plastic products makers
Dentists	Polyester makers
Dental assistants	Printers
Flour-mill workers	Silicone rubber makers
Miners	Styrene makers
Nurses	Telephone repair workers

Adapted from references 10 (pp 277-284) and 20

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