# ASSESSMENT OF PHTHALIC ANHYDRIDE AS A POTENTIAL AIR POLLUTION PROBLEM

VOLUME XIV

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U.S. ENVIRONMENTAL PROTECTION AGENCY
Research Triangle Park
North Carolina 27711

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# ASSESSMENT OF PHTHALIC ANHYDRIDE AS A POTENTIAL AIR POLLUTION PROBLEM

Volume XIV

by

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# ABSTRACT

This report is one of a series which assesses the potential air pollution impacts of 14 industrial chemicals outside the work environment. Topics covered in each assessment include physical and chemical properties, health and welfare effects, ambient concentrations and measurement methods, emission sources, and emission controls. The chemicals investigated in this report series are:

Volume	I	Acetylene
ACTOMIC	4.	VCCC) TGIIC

Volume II Methyl Alcohol

Volume III Ethylene Dichloride

Volume IV Benzene

Volume V Acetone

Volume VI Acrylonitrile

Volume VII Cyclohexanone

Volume VIII Formaldehyde

Volume IX Methyl Methacrylate

Volume X Ortho-Xylene

Volume XI Maleic Anhydride

Volume XII Dimethyl Terephthalate

Volume XIII Adipic Acid

Volume XIV Phthalic Anhydride.

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#### SECTION I

#### SUMMARY AND CONCLUSIONS

Phthalic anhydride is a white, crystalline solid with a characteristic choking odor. It is produced by the oxidation of either naphthalene or ortho-xylene, and it is a key industrial intermediate in the manufacture of resins, polyesters, dyes, and plasticizers.

The major acute toxic effect of exposure to phthalic anhydride dust or vapor is its irritating action on the skin, eyes, and mucous membranes. A concentration of  $25 \text{ mg/m}^3$  (4.2, ppm) will cause mucous membrane irritation. There is no record of serious injury due to acute exposure. Phthalic anhydride dust or vapor in contact with moist skin will form free phthalic acid. The U.S. occupational standard for exposure is  $12 \text{ mg/m}^3$  (2 ppm) for an 8-hour time weighted average.

Simple diffusion modeling estimates place the likely maximum 1-hour average ambient concentration at about 0.7 mg/m $^3$ , with a 24-hour average of about 0.3 mg/m $^3$ .

About 1 billion pounds of phthalic anhydride were produced at 16 locations in 1974, and production is expected to increase 6 percent per year through 1978. The primary end use is the manufacture of plasticizers, with polyester resin production second. The major emission sources in descending order are product storage, manufacturing, end product manufacturing, and product handling. Total emissions are estimated to have been about 5 million pounds in 1974.

Scrubbers and incinerators are currently being used by industry to control phthalic anhydride emissions with efficiencies of 86 and 99 percent, respectively. The product is stored in the molten state and blanketed with dry nitrogen to prevent fire and hydrolysis to phthalic acid. Currently used practices to control the continuous gaseous effluent are the use of knock-out pots for removal of solid phthalic anhydride and the venting of storage tanks to the main process vent gas incinerator.

Based on the results of the health effects research presented in this report, and the ambient concentration estimates, phthalic anhydride as an air pollutant apparently does not pose a threat to the health of the general population. In addition, phthalic anhydride does not appear to pose other environmental insults which would warrant further investigation or restriction of its use at the present time.

#### SECTION II

#### AIR POLLUTION ASSESSMENT REPORT

#### PHYSICAL AND CHEMICAL PROPERTIES

Phthalic anhydride is a white, crystalline solid with a characteristic choking odor. It is manufactured by the oxidation of either naphthalene or ortho-xylene. Phthalic anhydride is a key industrial intermediate in the manufacture of resins, polyesters, dyes, and plasticizers. Significant properties are given in Table 1.

#### HEALTH AND WELFARE EFFECTS

# Effects on Man

Acute Poisoning - The major acute toxic effect of exposure to phthalic anhydride dust or vapor results from its marked irritating properties on the skin, eyes, and mucous membranes of the nose and respiratory tract. A concentration of 25 mg/m³ (4.2 ppm) causes some mucous membrane irritation. A concentration of 30 mg/m³ (5.0 ppm) causes definite conjunctival irritation as well as increased mucous membrane irritation.¹ Symptoms of exposure to such concentrations include coughing, sneezing, a burning sensation in the throat and increased mucous secretion.²,³ Continued exposure or exposure to higher levels may result in general respiratory tract irritation, nasal ulceration and bleeding, loss of smell, and hoarseness.³ Pulmonary sensitization and allergic reaction is possible.² However, there is no record of serious injury due to short exposures to high concentrations.⁴

Table 1. SIGNIFICANT PROPERTIES OF PHTHALIC ANHYDRIDE

Synonyms	phthalic acid anhydride, phthalandione, 1, 2-benzene dicarboxylic acid anhydride	
Chemical formula	C C C C C C C C C C C C C C C C C C C	
Molecular weight	148.11	
Boiling point	284.5 <sup>0</sup> C (sublimes)	
Melting point	130.8°C	
Specific gravity	1.527 (4°C)	
Vapor pressure	1.4 mm Hg at 110 <sup>°</sup> C 42 mm Hg at 180 <sup>°</sup> C	
Solubility	Soluble in ethanol, benzene Slightly soluble in water, ethyl ethers, carbon disulfide	
Minimum explosive concentration	16.5 g/m <sup>3</sup>	
Explosive limits	1.7% at 140°C 10.5% at 193°C	
Ignition temperature	584 <sup>o</sup> c	
Flash point	151°C	
At 25°C and 760 mm Hg	1 ppm vapor = 6.0 mg/m <sup>3</sup> 1 mg/r <sup>3</sup> = 0.17 ppm	

Phthalic anhydride dust and vapor are particularly irritating to moist skin as a result of the formation of free phthalic acid. Contact produces redness and chapping, with urticaria possible. It is a skin sensitizing agent. The worst symptom of eye irritation is a persistent conjunctivitis, with no permanent injury resulting.

Chronic Poisoning - Workers chronically exposed to phthalic anhydride vapors or dust suffered from aggravated, acute exposure symptoms. Clinical findings included atrophy of the nasal mucous membrane (reversible), sometimes with blood-stained discharge, irritation of the larynx, and hoarseness and cough, and sometimes with blood-stained sputum. Bronchitis, irritation of the gastric mucous membrane with loss of appetite and weight, and asthma and allergic rhinitis (inflammation of the nose lining) due to sensitization have been seen. The allergy can develop such that lower doses of phthalic anhydride can no longer be tolerated. Direct dose-response data for chronic exposure are lacking in the literature. The U.S. occupational standard for exposure is a 12 mg/m<sup>3</sup> (2 ppm) 8-hour time weighted average, based on human sensory response.

# Effects on Animals

Acute Poisoning - Animal response to acute exposure has not been well documented in the literature. On the basis of available data, mucous membrane irritation, eye irritation and related symptoms would occur in animals at concentrations above 30 mg/m<sup>3</sup> (5.0 ppm). Sensory response and exposure symptoms would be the same as those seen in humans.

Values for the acute oral  $LD_{50}$  in the rat have been reported as high as 2,500 to 5,000 mg/kg, and as low as 800 to 1,600 mg/kg body weight.  $^{1,2}$  Death after ingestion was due to tubular necrosis (cell death) of the kidney. In cats the  $LD_{50}$  is 800 mg/kg, and for guinea pigs it is 100 mg/kg.  $^{7}$ 

Chronic Poisoning - Cats exposed to 3,700 mg/m<sup>3</sup> (617 ppm) for 6 hours a day for 7 days became drowsy, showed a loss of appetite, and vomited. Liver and kidney damage was seen. Rats and rabbits exposed to 10,000 mg/m<sup>3</sup> (1,667 ppm) for 4 hours per day for several days experienced 25 percent fatalities.<sup>3</sup>

Skin sensitization in guinea pigs has been illustrated by the intracutaneous injection of 20 mg·of phthalic anhydride dissolved in dioxane and clive oil twice a week for 2 weeks. Animals tested 2 weeks after the last injection were markedly sensitized.

# Effects on Vegetation

The effects of phthalic anhydride on plants have not been documented in the literature.

# Other Effects

The dust and vapors form explosive mixtures with air. The lower explosive limit is 1.7 percent and the upper explosive limit is 10.4 percent. The flash point by closed cup method is 151°C.

AMBIENT CONCENTRATIONS AND MEASUREMENTS

#### Ambient Concentration Estimates

The largest emission source of phthalic anhydride is a production facility with a capacity of 130 million pounds per year. This facility is located near Chicago, which has a population of over 3 million. The emission factor for phthalic anhydride is 0.0017 pound per pound produced.

Hence, the emission rate is

(0.0017 emission factor) (130 x 
$$10^6$$
 lb/yr) (453.6 g/lb)  
3.1536 x  $10^7$  sec/yr

= 3.2 g/sec of phthalic anhydride.

Some assumptions must be made regarding this chemical release to the atmosphere. First of all, the emissions do not all come from one source location, but rather from a number of locations within the plant where phthalic anhydride leaks to the atmosphere. Thus, the emissions can be characterized as coming from an area source which will be taken to be 100 meters on a side. Secondly, the emissions occur at different heights, and an average emission height of 10 meters is assumed.

Ground level concentrations can then be estimated at locations downwind of the facility. <sup>10</sup> To do this, a virtual point source of emission is assumed upwind of the facility at a distance where the initial horizontal dispersion coefficient equals the length of a side of the area divided by 4.3. In this case:

$$\sigma_{\rm vo} = 100 \, \rm m/4.3 = 23.3 \, m.$$

Assuming neutral stability conditions (Pasquill-Gifford Stability Class D) with overcast skies and light winds, the upwind distance of the virtual point source is approximately 310 meters. With consideration of the plant boundary, it is reasonable to assume that the nearest receptor location is thus 500 meters from the virtual point source. Finally, taking 2 m/sec as an average wind speed, the ground level concentration may be calculated from:

$$X = \frac{Q}{u\pi\sigma_y\sigma_z} e^{-1/2\left(\frac{H}{\sigma_z}\right)^2}$$

or 
$$\chi = \frac{3.2}{(2) \pi (36) (18.5)} e^{-1/2 \left(\frac{10}{18.5}\right)^2}$$

$$= 0.66 \text{ mg/m}^3$$

for a 10-minute average concentration. Over a period of an hour this becomes  $0.66~(0.72) = 0.48~\text{mg/m}^3$  (or 0.08~ppm vapor) 1-hour average concentration. Over a 24-hour period, the average concentration might roughly be expected to be about  $0.26~\text{mg/m}^3$  (0.04 ppm vapor).

# Phthalic Anhydride Measurement Techniques

Phthalic anhydride present as an airborne particulate should be collected on Whatman No. 1 filter paper or on a glass filter. For the collection of vapor or fumes, two gas washing bottles or bubblers in series containing appropriate absorbing solutions such as ethanol or 0.1 N HCl are used.

Three methods are available for the determination of the concentration of phthalic anhydride in air: colorimetric, ultraviolet absorption, and gas chromatography. However, only gas chromatography is suitable for ambient concentrations.

In the colorimetric method 11 phthalic anhydride is extracted from the filter paper using benzene. The sample is dried, treated with hydroquinone and sulfuric acid, left alone for 2 hours, and then diluted with water and benzene. An aliquot of the water layer is treated with potassium hydroxide and ascorbic acid. The absorbence of the aqueous layer

is then measured on a spectrophotometer at 560 mµ and compared to a standard curve. Concentrations in the range of 80 ppm have been determined by this method.

The ultraviolet absorption method  $^{11}$  involves the extraction of phthalic anhydride in 0.1 N HCl. The absorbency of the resulting solution is measured at 275 m $\mu$  and compared to a standard calibration curve. Concentrations detected by this method are similar to those of the colorimetric method.

The gas chromatograph method, which is used when small sample volumes are available, is the most accurate of the three methods. Concentrations as low as 1 ppb can be detected if used in conjunction with a flame ionization detector. 12

SOURCES OF PHTHALIC ANHYDRIDE EMISSIONS

# Phthalic Anhydride Production and Consumption

The production of phthalic anhydride in 1974 is estimated to have been 1,030 million pounds, and it is expected to increase at 6 percent per year through 1978. 13 Phthalic anhydride is primarily used in the production of plasticizers, accounting for 50 percent of total production. Alkyd resins and polyester resins account for a major portion of the remaining production. Twelve companies at 16 locations are manufacturing phthalic anhydride as listed in Appendix A. The consumption of phthalic anhydride for final products is shown in Table 2. This table also presents the expected growth rates for each sector of the market.

Table 2. PHTHALIC ANHYDRIDE CONSUMPTION - 1974

	Million pounds	% Annual growth
Plasticizers	515.0	6
Alkyd resins	195.7	0
Polyester resins	247.2	15
Export and miscellaneous	72.1	Variable
Total	1,030.0	6%

# Phthalic Anhydride Sources and Emission Estimates

Primary sources of emissions of phthalic anhydride result from phthalic anhydride production, bulk storage and handling, and end product manufacture. Total emissions from all categories are estimated to be 4.86 million pounds, representing 0.5 percent of total production. See Table 3.

Table 3. SOURCES AND EMISSION ESTIMATES
OF PHTHALIC ANHYDRIDE - 1974

Source	Million pounds
Phthalic anhydride manufacturing	1.56
Product handling	0.10
Product storage	1.64
End product manufacturing	1.56
Total	4.86

Phthalic anhydride is produced from two raw materials, naphthalene and ortho-xylene. Ortho-xylene accounts for 54 percent of production and naphthalene for the remaining 46 percent.

Emissions from the manufacturing of phthalic anhydride occur from process reactors and vents, product handling, and product storage. A recent study of phthalic anhydride production has estimated losses from reactors and vents. An emission rate of 0.0017 and 0.0013 ton of phthalic anhydride per ton of phthalic anhydride produced from the ortho-xylene and naphthalene processes, respectively, have been reported. Using these factors, and the corresponding production rate from each process, will result in 0.96 million and 0.60 million pounds, respectively, emitted from the ortho-xylene and naphthalene processes. These emission rates are based on extensive controls already in use by the industry.

Product handling losses, estimated from the same report, are based on 90 percent of the product sold in the molten state, and the remaining 10 percent sold as a solid flake. It is estimated that 0.0001 ton of product is lost per ton of phthalic anhydride produced. This results in 0.01 million pounds lost from the bagging operation and 0.09 million pounds lost from truck loading of molten product.

Phthalic anhydride is stored in fixed roof storage tanks in the molten state (149°C). The reported emission factor is 0.0016 ton emitted per ton of product stored. Therefore, losses from bulk storage are estimated to be 1.64 million pounds.

The last major source of phthalic anhydride emissions results from the production of end products. The literature does not contain any data on the emission rates from these processes. It is, therefore, estimated that the emission rates are the same as the emission rates for the production of phthalic anhydride. Emissions from end product manufacturing are estimated to be 1.56 million pounds.

#### PHTHALIC ANHYDRIDE EMISSION CONTROL METHODS

Two types of control devices — scrubbers and incinerators — are currently being used by the industry. In a recent study it was reported that these two control methods are used extensively in the manufacture of phthalic anhydride. The main source of emissions from the ortho-xylene process occurs from the main process vent gas stream. The main source of emissions from the naphthalene process occurs from the switch-condenser vent. Depending on the plant, one or two types of control devices may be used: scrubbers and/or incinerators. The water scrubber is reported to have an efficiency of 86 percent, and the incinerator has a reported 99 percent efficiency. Cost data for the various systems are presented in Tables 4, 5, and 6.

Table 4. WATER SCRUBBER AND INCINERATOR 4,14

	Water scrubber	Incinerator
Feed gas, 1b/hr	536,962	10,460
Gaseous effluent, 1b/hr	544,005	39,235
Gaseous effluent, SCFM	122,100	9,700
Total capital investment, \$	1,398,000	444,800.
Total operating cost, b \$	335,000	198,900
Net annual cost, \$		533,900 <sup>c</sup>
Emission control efficiency	86%	99% <sup>d</sup>

a Costs updated to first quarter 1975.

b Includes depreciation, interest charges, maintenance, labor and utilities.

Combined annual cost: water scrubber with incinerator.

d Combined efficiency: water scrubber with incincrator.

Table 5. DIRECT INCINERATION a, 14

Feed gas, 1b/hr	536,962
Gaseous effluent, 1b/hr	540,052
Gaseous effluent, SCFM	120,200
Total capital investment, \$	1,093,000
Total operating cost, b \$	502,000
Net annual cost, \$	502,000
Emission control efficiency	92%

<sup>&</sup>lt;sup>a</sup>Costs updated to first quarter 1975.

Table 6. INCINERATION AND WASTE HEAT BOILER<sup>a,14</sup>

Feed gas, 1b/hr	536,962
Gaseous effluent, 1b/hr	545,767
Gaseous effluent, SCFM	122,350
Total capital investment, \$	1,589,000
Total operating cost, b \$	1,101,000
Steam production (credit), c \$	591,000
Net annual cost, \$	510,000
Emission control efficiency	92%

<sup>&</sup>lt;sup>a</sup>Costs updated to first quarter 1975.

b Includes depreciation, interest charges, maintenance, labor and utilities.

<sup>&</sup>lt;sup>b</sup>Includes depreciation, interest charges, maintenance, labor and utilities.

 $<sup>^{\</sup>rm c}$ \$0.59/thousand pounds (450 psig, 750 $^{\rm o}$ F).

Most production plants store phthalic anhydride in the molten state and maintain it at 149°C. In several cases, the tanks are blanketed with dry nitrogen to prevent the entry of oxygen (fire) or water vapor (hydrolysis to phthalic acid). Consequently, there is a continuous gaseous effluent. Current control practices are the use of knock-out pots (settling and/or impactor chambers) for removal of solid phthalic anhydride and the venting of storage tanks to the main process vent gas incinerator. No cost data are available for these processes, but it is expected that capital and operating costs would be fairly low.

#### SECTION III

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APPENDIX A

PHTHALIC ANHYDRIDE MANUFACTURERS 14

		Capacity, million pounds/year
Allied Chemical	El Segundo, Calif.	40
Allied Chemical	Philadelphia, Pa.	100
Allied Chemical	Ironton, Ohio	33
BASF Wyandotte	Kearny, N. J.	130
Exxon Chemical Co.	Baton Rouge, La.	90
W. R. Grace	Fords, N. J.	75
Koppers Co.	Bridgeville, Pa.	95
Koppers Co.	Chicago, Ill.	130
Monsanto Co.	Bridgeport, N. J.	80
Monsanto Co.	Texas City, Tex.	130
Reichhold Chemicals	Morris, Ill.	100
Sherwin-Williams	Chicago, Ill.	20
Standard Oil of California	Richmond, Calif.	50
Stepan Chemical Co.	Millsdale, Ill.	50
Union Carbide Corp.	Institute, W. Va.	75
United States Steel Corp.	Pittsburgh, Pa.	125
Total		1,323

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#### 16. SUPPLEMENTARY NOTES

# 16. ABSTRACT

This report is one of a series which assesses the potential air pollution impacts of 14 industrial chemicals outside the work environment. Topics covered in each assessment include physical and chemical properties, health and welfare effects, ambient concentrations and measurement methods, emission sources, and emission controls. The chemicals investigated in this report series are:

Acetylene	Volume VIII	Formaldehyde
Methyl Alcohol	Volume IX	Methyl Methacrylate
Ethylene Dichloride	Volume X	Ortho-Xylene
Benzene	Volume XI	Maleic Anhydride
Acetone	Volume XII	Dimethyl Terephthalate
Acrylonitrile	Volume XIII	Adipic Acid
Cyclohexanone	Volume XIV	Phthalic Anhydride
	Methyl Alcohol Ethylene Dichloride Benzene Acetone Acrylonitrile	Methyl Alcohol Volume IX Ethylene Dichloride Volume X Benzene Volume XI Acetone Volume XII Acrylonitrile Volume XIII

17. KEY WORDS AND DOCUMENT ANALYSIS			
a. DESCRIPTORS		b.IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
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