

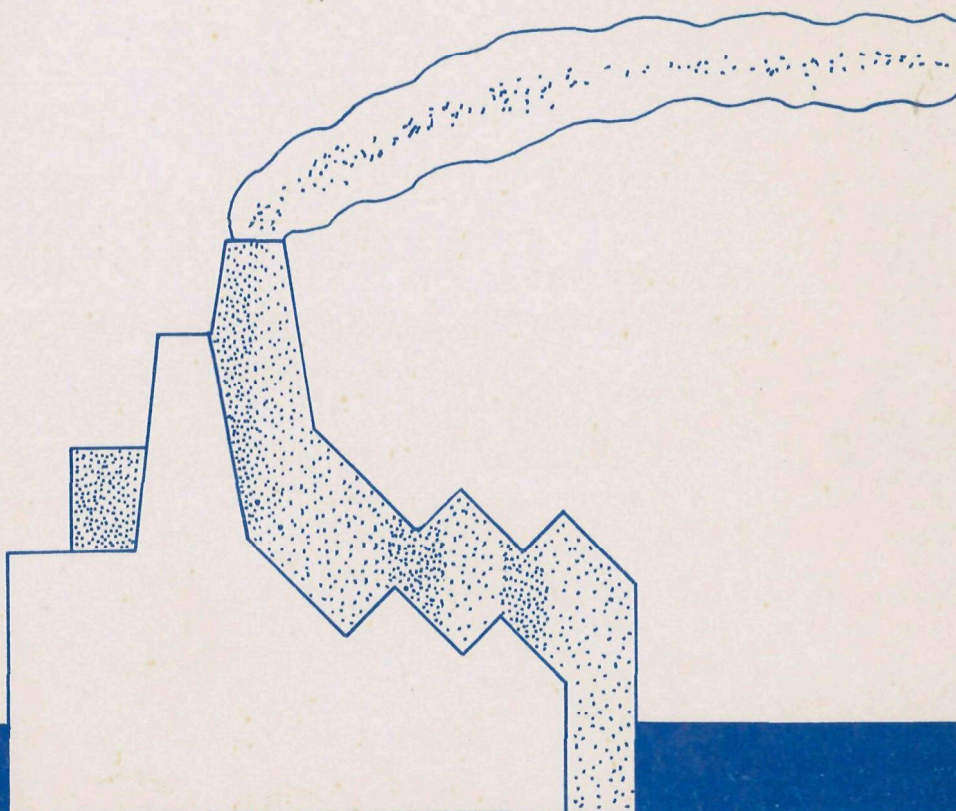
AIR POLLUTION CONTROL TECHNICAL REPORT

ODOR CONTROL INVESTIGATIONS

B. N. Murthy and B. C. Eusebio

DCS/RLB - 71-2

March 1, 1971



U. S. ENVIRONMENTAL PROTECTION AGENCY

Air Pollution Control Office

Division of Control Systems

ANNUAL REPORT

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Research Laboratory Branch
Division of Control Systems
Cincinnati, Ohio

ENVIRONMENTAL PROTECTION AGENCY

Air Pollution Control Office

SUMMARY

The odor control investigations in the Control Device Development Section include both in-house and contract-support research work for the development of suitable devices & processes for control of odorous emissions from stationary sources. DPCE started the investigations in 1968 with a literature survey on the state of the art of odor control technology, which resulted in a report including recommendations for research and development to be supported by DPCE.

The in-house work during 1970 consisted of laboratory studies aimed at development of suitable wet-scrubbing reagents for odorous compounds representing emissions from stationary sources. Two compounds in each of three groups of odorous emissions (mercaptans, amines and aldehydes) were treated separately with several of the possible aqueous scrubbing reagents under identical conditions, for comparison of the reagent capabilities for absorption of odors from air. The tests enabled the identification of specific reagents having the maximum scrubbing efficiency for each odorant species. Detailed kinetic studies to determine the optimum operating conditions and design data for scrubbers have been planned.

A contract project for studies on the adsorption of odorants on activated charcoal was started during the year at Kansas State University. The studies are aimed at determining equilibrium adsorption conditions and deriving mathematical models useful for the design of large scale adsorption control devices for odor control. Literature survey and apparatus construction were in progress at the end of 1970.

INTRODUCTION

DPCE started the odor control investigations in 1968, with a literature survey on the state of the art of odor control technology. The survey resulted in a report which included recommendations for research on odor control to be supported by DPCE (Appendix A).

Based on the recommendations, the in-house odor control project aimed at development of suitable wet scrubbing was started in late 1969. Odorous compounds representing those present in the emissions of the most objectionable stationary industrial sources were selected for the studies. A laboratory study was planned to determine in the in-house laboratories, the most promising set of reagents which could economically absorb and remove odors in wet scrubbers.

Besides wet scrubbers, the control devices which were identified as requiring detailed investigations for improvement and adaption to odor control were:

- adsorption units
- catalytic converters
- afterburners
- combinations of control devices

A contract for Fundamental studies on the adsorption of odorants on activated charcoal was signed with Kansas State University.

DISCUSSION

In-house screening tests.

An experimental bench-scale apparatus including gas purifiers, metering devices, temperature controls, absorption section and sampling ports, was constructed. A schematic design of the screening test system is shown in Fig. 1. The entire test system was set-up inside a large fume hood to prevent odor pollution episodes in the laboratory. An adsorption filter was installed in the fume discharge duct which removed odors from the fume hood.

The following odorants were selected for studies on the comparative efficiencies of their removal from air by adsorption in several reagents:

<u>Odorants</u>	<u>Common Industrial Sources</u>
Organic sulfur compounds- butyl and methyl mercaptans	Paper and pulp, agricultural chemicals, plasticizers, rubber products
Nitrogenous compounds - mono, di and tri methyl <i>cinnamyls</i>	Fish processing industries, rendering plants
Aldehydes Butyraldehyde Propionaldehyde Iso-valeraldehyde	Rendering plants, incinerators plastics

From a knowledge of the chemistry of the odorants, and from the literature survey, the following reagents were selected for experimental studies on odor adsorption:

- a) alkalis
 - sodium hydroxide
 - calcium hydroxide
 - sodium carbonate
- b) acids
 - hydrochloric
 - sulfuric
 - sulfamic
- c) oxidizing agents
 - potassium permanganate
 - sodium and calcium hypochlorites
- d) other reactants
 - bisulfites

The inlet concentration of each odorant in the air stream was held constant at 5000 ppm. The reagents were compared at concentrations ranging from 0.5 to 5 percent in water.

Analysis of the odorant before and after treatment with the reagent was by gas chromatography. Suitable combinations of column type, temperature, and detector were developed for the analysis of each odorant. The reagents were analyzed by standard wet chemical methods.

The complete results of the screening tests will be reported in a paper being prepared for publication. Some typical results are shown in Figs. 2, 3, and 4. The following main observations have been made from the screening test results:

- * Amines are efficiently removed by hydrochloric or sulfuric acid.
- * Sodium hydroxide is the best scrubbing reagent for mercaptans, while calcium hydroxide slurry is a promising scrubber from the economic aspect.
- * Aldehydes are scrubbed best by sodium and calcium bisulfites.
- * Potassium permanganate is a uniform scrubber for all the odorants, but is not the most efficient for any odorant. (Efficiency of odor removal was defined as percent of inlet odor absorbed by the same volume of reagent under identical conditions of contact time and reagent concentration.)
- * The efficiency of potassium permanganate depends on the pH of the solution, and is maximum in the pH range of 8 to 10.

The effect of operating conditions on the rate of odorant removal will be studied in detail with the best reagents for each type of odorant. Mathematical models useful for the design of wet scrubbers for odor control will be derived from the kinetic studies.

Program Plans

A proposed 5-year program plan for odor control projects to be conducted in-house and by contract was prepared.

The in-house projects identified will include:

- a) Kinetic studies for odor control
- b) Pilot scale wet-scrubber development studies
- c) Semi-pilot scale adsorption studies for odor control
- d) Catalysis and afterburner development
- e) Development of combined two-stage control devices and optimization studies
- f) Design and development work for odor control demonstration

Contract projects identified were:

- a) Odor adsorption studies using activated charcoal
- b) Selective sorption device development
- c) Odor control devices system study
- d) Follow-up R & D studies from odors control and related system studies
- e) Economic analysis and development of prototype odor control programs
- f) Demonstration studies

Procurement plan for the system study project to be supported on contract is being prepared and a strong recommendation to fund the study during FY72 will be made.

Existing Contract Activity

The in-house literature survey on the state of the art of odor control had revealed the need for developmental work on adsorption control devices as a high priority project.

A project was initiated with the contract signed with Kansas State University for conducting fundamental studies relating to equilibrium conditions and kinetics of adsorption of odorants in dilute concentrations appearing in the emissions of stationary sources. The detailed work plan is attached as Appendix B.

Literature survey, experimental apparatus construction, and calibration are in progress. The contract will be completed in September, 1972.

CONCLUSIONS

The state of the art survey on odor control has pointed out the urgent need for research and development work in the following areas:

- * Wet scrubber development
- * Adsorption
- * Afterburners and catalysis
- * System study on odor control devices
- * Optimization and engineering economic studies of combined two stage systems

In-house screening tests have shown that the following combinations of odorant-reagent require detailed investigations for wet-scrubber development:

Mercaptans - Sodium and calcium hydroxide
Aldehydes - Sodium and calcium bisulfites
Amines - Hydrochloric and sulfuric acids
All odorants - Potassium permanganate

RECOMMENDATIONS

1. Follow-up work to the screening tests should be conducted in-house.

Kinetic studies for odor control by gas-liquid contacting should be initiated to develop mathematical models correlating operating variables with rate of reaction. Mass-transfer effects in the experimental system should be kept at the least rate-limiting level to enable the determination of chemical reaction steps.

The follow-up studies on wet-scrubber development will use the results of the kinetic studies for developing optimum scrubber designs for efficient and economic odor control systems.

2. Semi-pilot and pilot scale studies on adsorption and regeneration should be conducted to determine the effect of fluid dynamic conditions on power requirements and material costs.

The mathematical models developed from fundamental laboratory studies should be used in the design of pilot scale fixed and fluidized bed absorbers.

3. Though activated charcoal is widely used as an adsorbent for odor control, it is a poor adsorbent for some odorants. Selective adsorbents, either through modification of activated charcoal or by investigation of other surface active materials, should be developed for efficient odor control in specific cases. Examples of odorants not effectively controlled by activated charcoal are:

Hydrogen sulfide
Carbonyl sulfide
Acetaldehyde
Amines

4. A system study on the present application of odor control devices and their limitations is needed to define the problems with existing control devices, and to identify research and development needs for effective odor control. The potential of existing control technology for odor control applications should be investigated.

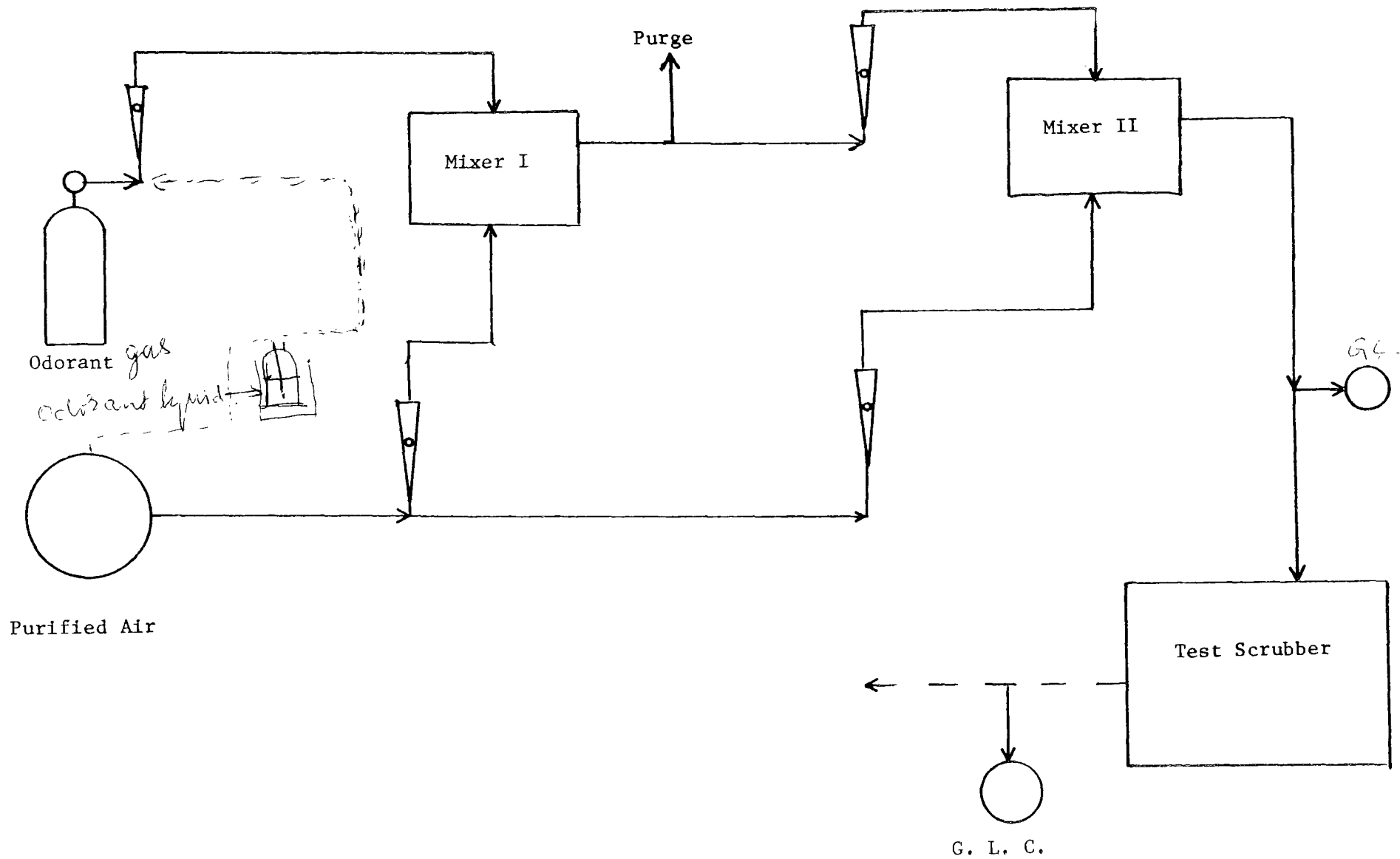


Fig 1

SCREENING TESTS - SCHEMATIC DESIGN OF TEST SCRUBBER - BENCH SCALE

mlt 3-10-70

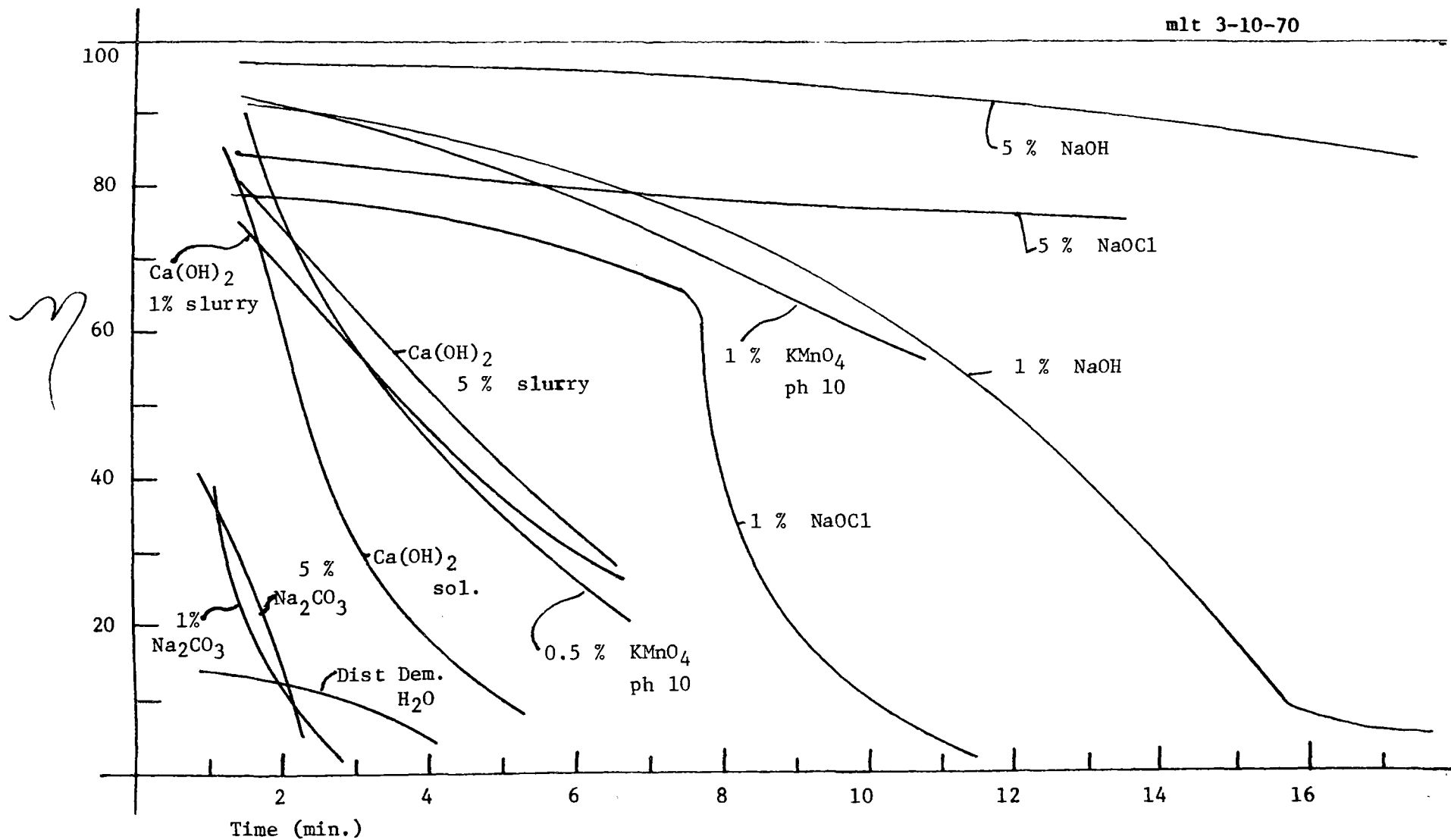


Fig. 2

BUTYL MERCAPTAN SCREENING TESTS

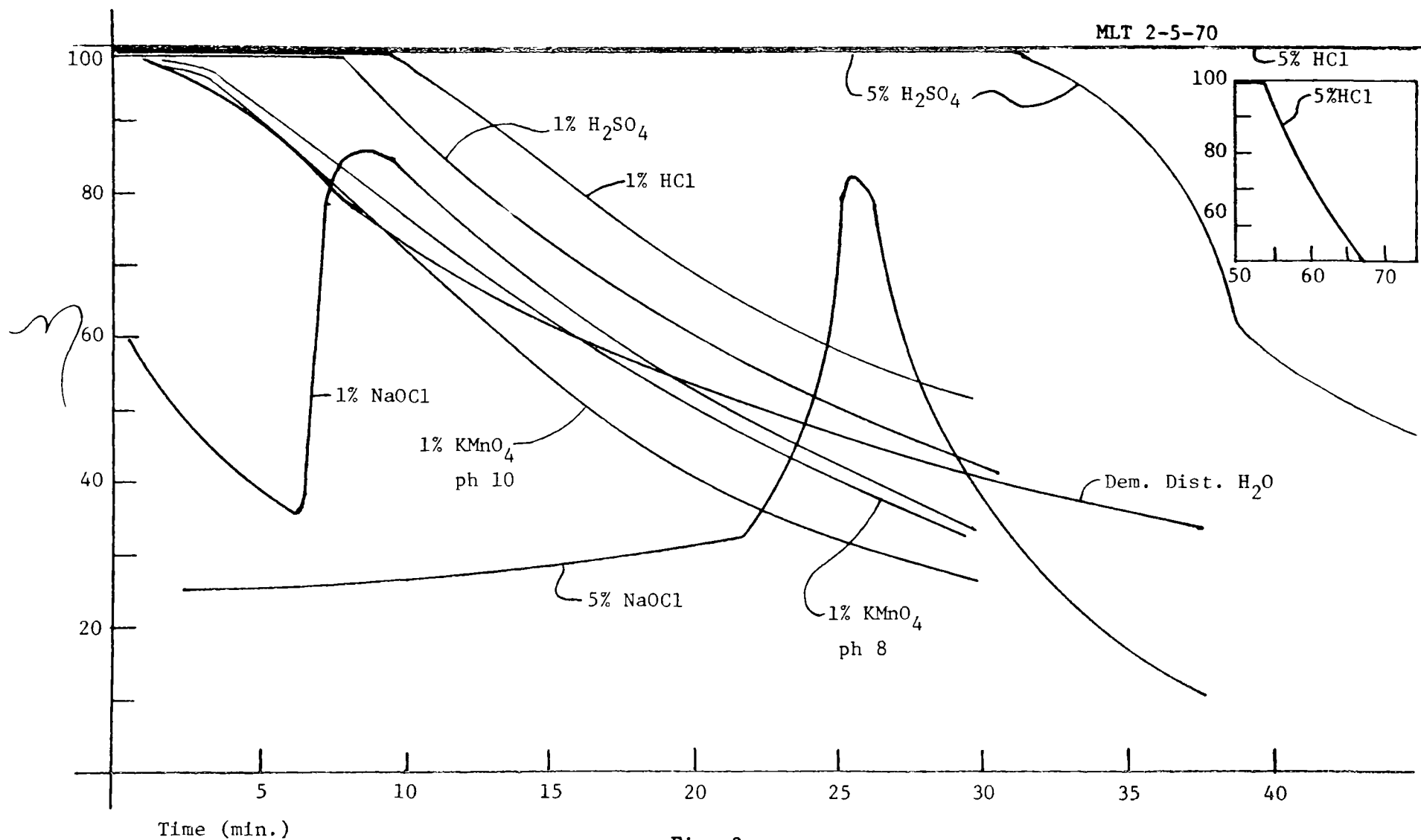
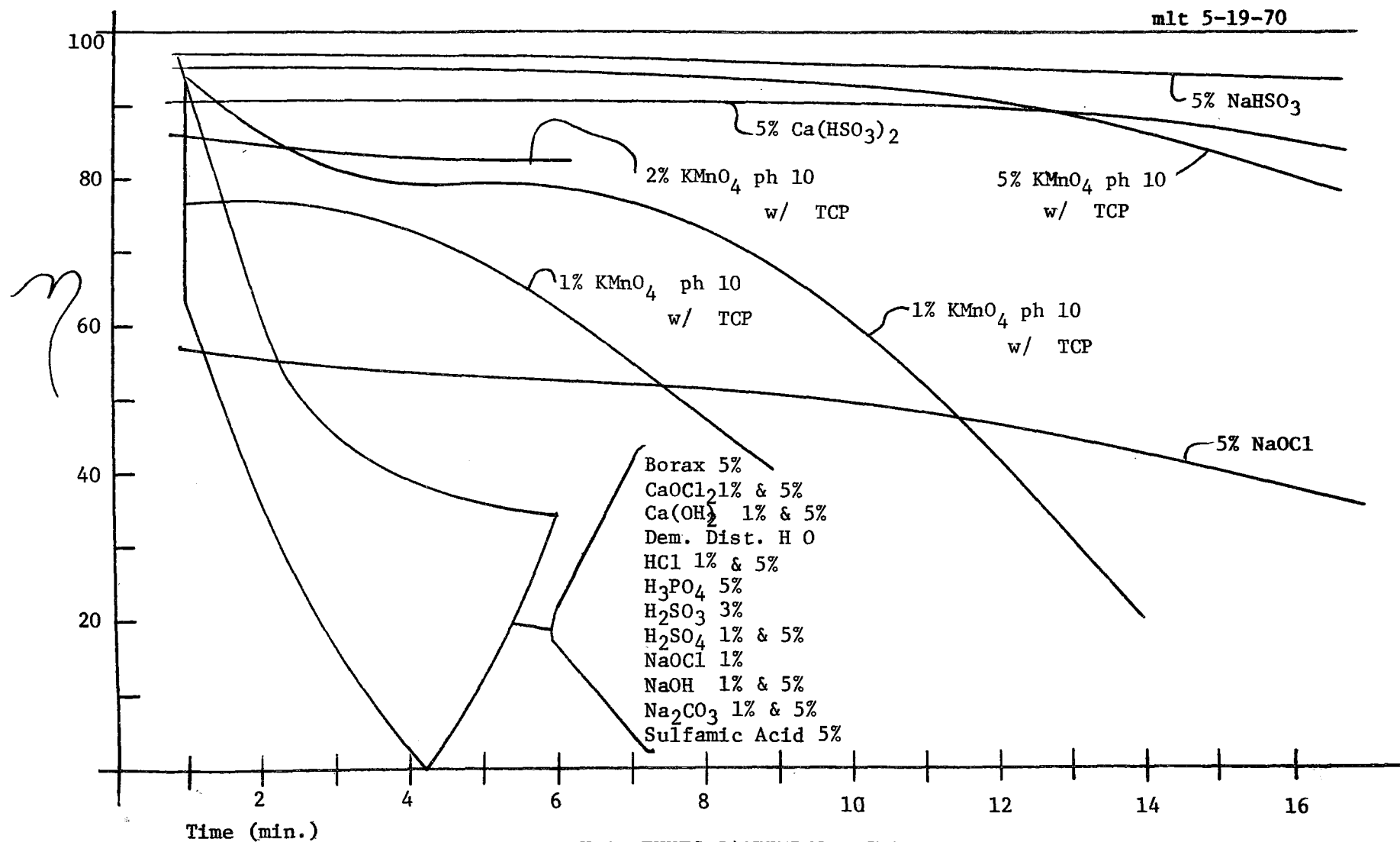


Fig. 3

DIMETHYLAMINE SCREENING TESTS

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BUTYRALDEHYDE SCREENING TESTS

BUTYRALDEHYDE SCREENING TESTS

APPENDIX A

ODOR CONTROL RESEARCH

A State-Of-The-Art Report and Research Recommendations

ODOR CONTROL RESEARCH

March, 1969

SUMMARY

This report is a review of the state of technology for odor control and evaluation. The emission of odorous compounds forms a significant portion of the air pollution problem. It has been established that objectionable odors produce nausea, disturb sleep, and reduce property value.

Odor surveys indicate that the most common odor emission sources are chemical and pulp manufacturers, petroleum and mineral refineries, and food stuff waste product and animal rendering industries.

The technology of odor evaluation and control has not advanced to keep pace with the growing problem. No suitable method or instrument is available for measuring odors objectively. Odor measurement techniques presently being used are mainly subjective. Consequently, there is a marked deficiency of quantitative information on odor emissions from industrial sources.

Techniques that can be used for odor control are ventilation, absorption chemical reaction, process changes, odor modification, combustion, and scrubbing. Of these, scrubbing offers the advantage of not being highly specific to particular odorous emissions.

Research needs for the development of scrubbers to control odors are discussed in this summary report. The report recommends that simple odorous systems amenable to accurate analysis be selected initially. Gas chromatographic equipment will be used for analysis of odorants. The effect of operating variables on the efficiency of odor control will be studied on a bench scale. Results from these studies will be used to form the input for pilot-scale scrubber studies.

INTRODUCTION

One of the major uncontrolled air pollution problems is the emission of odorous compounds into the atmosphere as a result of industrial activity. Although gross atmospheric pollution in the form of smoke and dust has been subjected to appraisal, regulation, and statutory restriction, attempts to control odors have been highly complicated as a result of the lack of uniform standards for measurement and classification of odors. The magnitude of the overall odor pollution problem, as indicated by the number of complaints about odors in ambient air surrounding industries, is increasing.

Odor is defined as the characteristic of a substance that stimulates the sense of smell, either pleasantly or unpleasantly.¹ The substance is usually airborne. When a solid or liquid gives out an odor, the stimulus is caused by the volatile molecules from the substance reaching the sensory nerves in the nose.

The relationship between the physical and chemical property of a substance and its odor quality has not been definitely established. Our understanding of the sense of smell is considerably less advanced than our understanding of other senses. Attempts to classify odors according to molecular structure have not been very successful. For instance, aldehydes, carbonyls, sulfur compounds, and hydroxy compounds cause certain typical smells, but take on entirely different characteristics at various concentrations in air. The odor of a mixture of odorous compounds cannot be predicted from a knowledge of their individual odors. The problem of classifying odors in terms of basic types is further complicated by psychological and physiological factors. An odor that is offensive to one person may be tolerable or even pleasant to another. But some smells fall definitely into the class of offensive odors. These include the putrefractive and fecal odors and some chemical odors that are generally considered objectionable.

Until recently, odors were considered something inevitably present around industrial areas and few attempts were made to control them. It has now been established that objectionable odors produce nausea, disturb sleep, and reduce property value.² Some odors, like emissions from acrylate industries, are toxic even at low concentrations.³ Although all industrial odors are not inherently harmful by nature, some

authorities believe that many odors may adversely affect public health by causing an ionic imbalance in the atmosphere. The presence of excessive positive ions in air irritates mucous membranes, interferes with ciliary activity, and may increase general susceptibility to viral infection.⁴

Since industrial odors are a definite part of the overall air pollution problem, people concerned with the design, supervision, or administration of chemical plants should be familiar with the latest techniques available for controlling odors. The purpose of this report is to review the state-of-the-art regarding odor evaluation and control methods and to make recommendations for a plan of research that will lead to the development of effective odor control techniques.

SOURCE OF ODOR

Before an effective odor control program can be launched, the sources and nature of odorous emissions must be identified. At present, there is a marked deficiency of published literature on the quantities, types, and concentrations of odorous emissions.

In 1955 an opinion survey was conducted by Pendray and Company in 67 major industrial cities.⁵ Members of the communities were questioned about the sources of odors. This survey resulted in an estimate of the qualitative nature of the problem, and indicated that the most common sources of odors were the chemical manufacturers (Appendix A).

More recently, in 1966 the U.S. Public Health Service conducted an odor survey in the St. Louis communities bordering Missouri and Illinois. This survey again was a qualitative assessment of the frequency and objectionability of odors as sensed in ambient air by volunteers geographically distributed over the area. The survey showed that 80 percent of the chemical odors and 98 percent of the animal odors (from rendering plants and stockyards) were considered to be objectionable (Table 1).

The magnitude of the odor problem and an estimate of the quantity of odorants to be removed at their source can be determined by the objectionability, odor threshold, and physiological effects of odors at ambient concentrations. An objectionability scale ranging from "like extremely" to "dislike extremely" has been proposed by Turk⁷ to determine subjective reactions to odors. Objectionability alone cannot be a sufficient criterion for control. For example, hydrogen sulfide at concentrations sufficient to produce harmful effects loses its characteristic offensive odor and produces a pleasant smell. The characteristic

smells and odor thresholds for some of the commonly occurring pollutants are given in Table 2.⁴ The odor threshold for an odorous substance in the atmosphere is the minimum concentration that can be detected by the human sense of smell.

In this review the common sources and compositions of odorous emissions have been compiled from the literature and are listed in Appendix B. To assess the relative contributions of the sources of air pollution, the major types and quantities of pollutants emitted have to be classified. The U.S. Public Health Service has published a compilation of air pollutant emission factors,⁸ along with quantitative estimates of a few odorous emissions. An emission factor is defined as the statistical average of the rate at which pollutants are emitted from the processing of a given quantity of raw material. We may conclude from the study of emission factors that the major chemical source contributing to the odor problem is the pulp and paper industry. The characteristic odor from a pulp mill results mainly from a group of organic sulfur compounds of which the most important are methyl mercaptan and its oxidation products, dimethyl sulfide and dimethyl disulfide.¹⁰

A survey of the odor complaints received by the Intercommunity Air Pollution Control Program of the city of Cincinnati indicated that most of the complaints were about odors from rendering plants, asphalt processing, and plastics industries. No quantitative information on these sources is available, however.

As part of its contract program, the Division of Economic Effects Research of the National Air Pollution Control Administration is conducting a study of the odor problem in the nation in terms of sources, description of odorants (character, acceptability, and other measures), population affected, and geographic distribution.¹¹ The study is aimed primarily at the promulgation of air quality criteria. Phase I of the study is expected to identify the industrial processes and other sources and describe major odorants. Phase II includes an estimation of the socio-economic impact of odors on the community. There is a great need for quantifying the type of data expected to be obtained in the study mentioned above, so that the results can be used to develop control techniques.

ODOR MEASUREMENT

The most effective odor control methods are those that prevent the release of odorous pollutants into the atmosphere. Accurate assessment of the odor problem and control equipment performance requires adequate

techniques for measuring odors over a wide range of concentrations. At this time, however, there are no fully satisfactory techniques for determining the character and intensity of an odor. This deficiency is undoubtedly due in part to the lack of a satisfactory theory or even a working hypothesis covering the physiology, psychology, physics, chemistry, and other aspects of the sense of smell.⁴

Measurement of Character and Intensity of Odors

Some attempts have been made in the past to characterize odors in terms of basic odor qualities. The number of suggested basic odor types varies from four upwards. Crocker and Henderson¹² have used (1) fragrant, (2) sour (acid), (3) burnt, and (4) goatly (caprylic). The object of this kind of classification is to simulate an odor by mixing the basic types. Henning¹³ has suggested six basic types: (1) spicy, (2) flowery, (3) fruity, (4) balsamic (resinous), (5) burnt (empyreumatic), and (6) foul (offensive). Zwaardemaker⁴ and others have suggested from six to eighteen basic odor types. For practical use in the development of control technology, none of these classifications has been generally acceptable.

Most industrial odors are complicated mixtures of several chemical components. Since instruments have not been perfected to analyze odors objectively, subjective evaluation by human observers is often used to estimate the character and intensity of industrial odors. There are mainly two methods by which the subjective (or sensory) evaluation is practiced: (1) dilution and (2) suprathreshold or matching standard.¹ In both methods the intensity of an odor is measured in terms of 'odor units'. An odor unit is defined as the amount of odor necessary to contaminate 1 cubic foot of clean odor-free air to the threshold level.^{13,14,15} The number of times a given volume of the sample gas has to be diluted with clean odorless air to bring it to the threshold level (detected by 50 percent of a panel of observers) is the value of the intensity in odor units. The product flow rate times the odor intensity gives the rate of odor emission.

Dilution Methods

Four major techniques are used to apply the "dilution" method of measuring odor intensity in terms of odor units:

1. Odorant air mixture with odor-free air in a container is brought to an opening and sniffed.

2. Part or all of the diluted mixture in a container is injected into the nose.
3. The diluted mixture is ducted into a hood or chamber enclosing the observer or only his face.
4. Odor-free and odorous air are inhaled in controlled proportions.

Of these methods, those in which the nose is immersed in the diluted mixture give more accurate results than the injection or inhalation method, because in the immersion method the dilution rates and the rate of odor delivery are exactly controlled by an unbiased operator. In the injection method the panel member himself controls the rate of release, the rate of injection, and the amount of inhalation. Also, it is impossible to measure dilution by air that is displaced by the sample injected into the nose.

Several devices for diluting gases for odor measurement have been described in literature.¹⁶⁻²² Among these, the ASTM syringe technique²² is widely used to measure the strength of odors at effluent sources for rough evaluations of the performance of control devices. The method consists of drawing the odorous gas into a graduated 100-ml syringe, sampling it into a 2-ml syringe, and diluting it with odor-free air in another 100-ml syringe. A special sampling and diluting device is constructed for the method from standard hypodermic needles, which fit the syringes. The method is simple and gives rapid approximate measurements of odor intensity.

Matching Standard Methods

For the matching standard method, a set of standards is calibrated to indicate objectionability and odor intensity on a scale. Odorants of known concentrations are prepared as standards. A nine-point scale of the type shown in Figure 1 was used by Duffee and co-workers¹ to represent both objectionability and intensity.

An advantage of this method is that a concentration above the threshold level can be directly matched against a standard mixture. The method is rapid, but not very accurate since the intensity of odor from a complex mixture is easily affected by extraneous factors like impurities (even in traces), humidity of ambient air, and temperature.

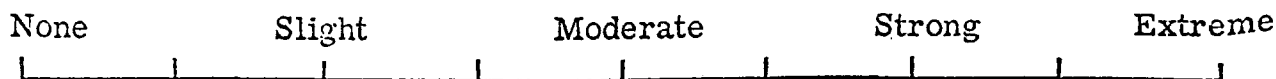


Figure 1

Analytical Methods

For developing or evaluating a control device, the best method of odor measurement is one that gives absolute concentrations of odorous compounds in emission streams. The analytical methods used for measurement of odorant concentrations are chemical or instrumental. In many practical situations the composition of the odorous effluent can be determined from a knowledge of the chemistry of the process or by analysis of emission samples. The odor intensities of pure compounds have largely been found to follow the Weber-Fechner equation:¹

$$P = K \log S$$

where P = odor intensity

K = a constant

and S = concentration of the odorant in air

For a mixture of compounds whose odor threshold data are available through previous determinations, gas chromatographic analysis has been successfully employed to estimate the odor intensity directly.²⁰⁻²⁶ Gas chromatography offers an accurate method for analyzing complex mixtures over a wide range of concentrations.

In some cases, a correlation between the concentration of a particular constituent and the odorant concentration or odor intensity can be established. An example of the application of this approach involves the use of devices to measure the concentration of carbon monoxide as an index of the intensity of domestic incinerator effluent odor.²³ This approach assumes a constant relationship between the components of the effluent stream.

ODOR CONTROL

The development of devices specifically suitable for odor control has not received sufficient attention in the past because of the diverse nature of the problem. The total quantity of gaseous emissions that must be

handled for the removal of odorants usually is large and results in high energy consumption and equipment costs. Thus, the choice of a particular odor control method depends on economic as well as technical considerations.

There are four major ways to control odors;

1. Ventilation and diffusion.
2. Chemical conversion - combustion, chemical reaction, or change of process.
3. Odorant capture - adsorption and absorption.
4. Masking or counteraction.

Ventilation and Diffusion

Ventilation is the most common method of removing odorous air from enclosed spaces. Industrial effluents are often diffused through stacks into the atmosphere so that the odorous effluent is substantially diluted and reduced to below the threshold level in the ambient air. This method cannot be successful if a large quantity of odorant is discharged or if the quality of the outdoor air is unsatisfactory.

Chemical Conversion

Combustion is the most commonly used method for odor control. This method of control can be attained by direct incineration or catalytic oxidation. Direct incineration is usually accomplished by supplementing the odorous effluent with a fuel such as natural gas to provide incineration temperatures of 1200 to 1500°F. Odorants can be incinerated at substantially reduced temperatures (600-900°F) if combustion catalyst is used. Oxy-cat Company³³ has developed catalysts to reduce the incineration temperature of odorants to less than 600°F. The main advantages claimed for the catalytic combustion method are low energy requirement and nearly complete elimination of odors. For both modes of incineration, however, when the concentration of combustible odorant in the effluent stream is low, the need to heat the large quantity of admixed air and water vapor to the combustion temperature of the odorant makes incineration economically unattractive.

A number of processes employed by industries fall in the category of chemical reaction. Examples include the kraft pulping industry, where sulfate black liquor is oxidized to prevent the emission of mercaptans and hydrogen sulfide into the atmosphere.^{31,32}

Scrubbing may be used to remove odorous pollutants from a gas stream by condensing or absorbing the pollutants in a liquid. Water is widely used for this purpose. If an odorant can be reacted with a liquid to form soluble compounds or precipitates, chemical reaction accompanying correlation or adsorption completely removes odors. Examples of aqueous scrubbing include the work of Borger,³⁶ who has described the development of a method for successfully eliminating dimethylamine odors by scrubbing with water and dilute acid in absorption towers. Many odorous pollutants can be reacted with aqueous permanganate solution to eliminate the odors.³³ This method is economically applicable under conditions where effluents with high moisture contents are to be treated. Rendering plant odors can largely be controlled by use of scrubbing devices.³⁵

Essentially all kinds of scrubbing equipment (spray towers, cyclone scrubbers, packed beds, venturi, etc.) can work at high efficiencies when the odorant is absorbed easily. If the reaction or solubility in the liquid is slow, packed towers or plate towers provide the residence time needed to achieve the required mass transfer. Designs using free liquid jet or spray are useful when simultaneous adsorption of odorous components and precipitation of solids are required. Packed towers and tanks are not suitable for this purpose, however, because the solids remain suspended. In general, pressure drop is high in plate towers and agitated tanks, moderate in packed towers, and low in spray towers and cyclone scrubbers.

The advantage of using scrubbers is that large quantities of odorous effluent can be handled continuously and economically.

Odorant Capture

Active carbon is widely used to adsorb odorous components from air. When the carbon is saturated with the odorous component, it is deactivated and must be reactivated. Carbon adsorption is used to control enclosed atmospheres and to eliminate solvents and vapors from effluents. Other substances like silica gel and activated alumina are used as adsorbents in some cases.³⁴

Masking

Odor masking can be employed to cover up non-toxic pollutants. In this method a pleasant smelling or counteracting reagent is injected into the odor-contaminated air. The masking agent acts in one or more of the following ways: (1) overwhelms the odor, (2) modifies the properties of the odorant to produce a less objectionable odor, or (3) numbs the sense of smell momentarily so that the odor does not seem offensive. Masking agents are usually aromatic compounds like benzyl acetate and phenyl-ethyl alcohol. The advantage of this method is its low capital investment. Masking generally cannot be recommended where odorants in large quantities are emitted.

RESEARCH AND DEVELOPMENT OF ODOR CONTROL

As noted earlier in this paper, odor control problem definition and quantification, odor measurement, and odor control are largely undeveloped technologies. Very little research and development to bridge the gaps in this air pollution control area are underway. Limited work is being done to identify and deal with the problems of odor control from motor vehicles; however, no analogous programs can be identified for the equally or more important problem of odor control from stationary sources.

Some odor control research relating to kraft mill effluents has been going on for more than 10 years under the sponsorship of the Public Health Service^{6,37} and the British Columbia Research Council.³⁸ To a large extent, research in this field has been oriented to a detailed study of the reactions that contribute to odor pollution so that processes may be changed to overcome this problem. The principal control methods developed are incineration, alkaline absorption (scrubbing), heat recovery, black liquor oxidation, and chlorine treatment. The problem of kraft mill odors has not been completely solved; further research on basic changes in both the pulping and recovery stages would aid in overcoming the odor problem.

Research and development is urgently needed to control chemical and rendering plant odors, since the methods now being employed are inadequate. The large number of complaints received in surveys on odor problems attest to this need.⁹

A well-supported research program extending over the next 5 to 10 years will be required to develop technology for control of odor pollution to a level comparable to those for control of particulate and many gaseous pollutants.

RECOMMENDATIONS FOR DPCE RESEARCH AND DEVELOPMENT ON ODOR CONTROL

A. Simple odorous gases and vapors that are known to be present as objectionable effluents from industries should be selected for control research. Examples of pure compounds that have been identified in complaints include

Acrylates	{	Plastic industries
Acrolein		
Acrylonitrile		
Methyl mercaptan	{	Pulp mills, rendering plants
Dimethyl sulfide		
Dimethyl disulfide		
1,5 diaminopentane (cadaverine) - Rendering plants		

The Manufacturing Chemists Association has very recently published their latest findings on odor threshold research on 53 odorous commercial compounds (Appendix C). This information should provide a basis for evaluating odorant removal efficiency.

B. Selected odorant compounds should be analyzed directly by instrumental methods. A gas chromatographic analyzer appears to be most suitable for accurately analyzing mixtures of odorant compounds over a wide range of concentrations (pure compound to < 0.5 ppm).

C. A screening study of scrubber reactants for controlling odorant compounds should be conducted:

1. The effectiveness of various scrubbing liquids or catalysts in removing odorants from gas streams should be determined in bench-scale screening tests. Gas bubblers and liquid-gas reaction vessels should be employed.
2. Rates of reaction or absorption under variable conditions of composition of scrubbing reagent, pH of the liquid, temperature, and inlet gas concentration should be suited.

3. Results of the screening test should be used in the selection of a scrubber system for further research.

D. A preliminary pilot- or bench-scale odor control process design should be developed. The economics of the process should be evaluated.

E. Different types of scrubbers should be evaluated for their efficiency in separation of odorants from effluent streams.

1. The design features of the scrubber unit should be developed from studies of the effect of operating conditions on efficiency of odor control.
2. Scrubbers as gas-liquid chemical reactors should be evaluated on a pilot scale.
3. Economically advantageous related phenomena like condensation should be incorporated in scrubber designs.

F. Fundamental principles and relationships that guide the improvement of scrubber systems as control devices should be investigated. For example, combined adsorption of odorants on solid particles and removal in scrubbers may lead to the development of systems that would remove particulate and gaseous pollutants in a single unit.

A proposed work schedule for the above program is attached as Appendix D.

Table 1
ST. LOUIS SURVEY: FREQUENCY
OBJECTIONABILITY OF ODOR TYPES⁶

Odor Type	<u>Averages</u>	
	% Frequency ^{a/}	% Objectionability ^{b/}
Chemical	17.8	80.9
Food	2.7	12.1
Combustion	30.7	65.3
General industrial	5.3	62.0
Animal	4.4	98.0
Combustion waste	22.8	50.6
Decomposition	4.3	83.2
Vegetation	2.5	8.7
Miscellaneous	3.7	51.7
No description	6.3	60.7

^{a/}% frequency = positive observations ÷ total positive observations, %.

^{b/}% objectionability = unpleasant observations ÷ positive observations, %.

Pollutant	Formula	Odor	Odor Threshold, ppm
Vanillin	$\text{CH}_3\text{O} \cdot \text{C}_6\text{H}_3(\text{OH})\text{CHO} \cdot$	Sweet, aromatic	$32 \cdot 10^{-9}$
Skatole	$\text{C}_6\text{H}_4 - \text{C}(\text{CH}_3) \cdot \text{CH} \cdot \text{NH}$	Fecal	75
Musk, synthetic	$\text{C} \cdot \text{CH}_3 \cdot \text{CH}_3 \cdot \text{CH}_3 \cdot \text{C}_6(\text{NO}_2)_3 \cdot \text{CH}_2 \cdot \text{CH}_2$	Musk	$42 \cdot 10^{-8}$
Ethyl Seleno mercaptan	$\text{C}_2\text{H}_5 \cdot \text{SeH}$	Foul, foetid	$18 \cdot 10^{-7}$
Ethyl mercaptan	$\text{C}_2\text{H}_5 \cdot \text{SH}$	Decayed cabbage	$16 \cdot 10^{-6}$
Allyl mercaptan	$\text{CH}_2 \cdot \text{CH} \cdot \text{CH}_2 \cdot \text{SH}$	Garlic	$5 \cdot 10^{-5}$
Ethyl selenite	$\text{C}_2\text{H}_5 \cdot \text{Se} \cdot \text{C}_2\text{H}_5$	Putrid, nauseating	$62 \cdot 10^{-6}$
Propyl mercaptan	$\text{C}_3\text{H}_7 \cdot \text{SH}$	Unpleasant	75
Allyl disulfide	$\text{CH}_2 \cdot \text{CH} \cdot \text{CH}_2 \cdot \text{S}_2 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{CH}_2$	Garlic	$1 \cdot 10^{-4}$
Hydrogen sulfide	H_2S	Rotten eggs	11
Ethyl sulfide	$\text{C}_2\text{H}_5 \cdot \text{S} \cdot \text{C}_2\text{H}_5$	Foul, garlic	$25 \cdot 10^{-5}$
Butyric acid	$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{COOH}$	Rancid, perspiration	28
Iodoform	CHI_3	Antiseptic	37
Valeric acid	$(\text{CH}_3)_2 \cdot \text{CH} \cdot \text{CH}_2 \cdot \text{COOH}$	Unclean body odor	62
Methyl mercaptan	$\text{CH}_3 \cdot \text{SH}$	Decayed cabbage	$11 \cdot 10^{-4}$
Apiole	$\text{CH}_3\text{O} \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{CH}_2\text{CH}_3\text{O} \cdot \text{O} \cdot \text{CH}_2 \cdot \text{O}$	Parsley	63
Chlorine	Cl_2	Pungent	$1 \cdot 10^{-2}$
Pyridine	$\text{C}_5\text{H}_5 \cdot \text{N} \cdot$	Empyreumatic	$12 \cdot 10^{-3}$

Table 2. Cont'd

Pollutant	Formula	Odor	Odor Threshold, ppm
Dimethyl sulfide	$\text{CH}_3 \cdot \text{S} \cdot \text{CH}_3$	Decayed cabbage	$2 \cdot 10^{-2}$
Diacetyl	$\text{CH}_3 \cdot \text{CO} \cdot \text{CO} \cdot \text{CH}_3$	Sweet butter	$25 \cdot 10^{-3}$
Ammonia	NH_3	Pungent	37
Ozone	O_3	Irritating	$1 \cdot 10^{-1}$
Hydrogen selenide	$\text{H} \cdot \text{SeH}$	Putrid	3
Phenol	$\text{C}_6\text{H}_5 \cdot \text{OH}$	Empyseumatic	3
Dimethylamine	$\text{CH}_3 \cdot \text{CH}_3 \cdot \text{HN}$	Fishy	6
Carbondisulfide	CS_2	Rotten	7.7
Acrolein	$\text{CH}_2 \cdot \text{CH} \cdot \text{CHO}$	Hot fats	15
Camphor	$\text{CH}_3 \cdot \text{CH}_3 \cdot \text{C} \cdot \text{C}_6\text{H}_6\text{O} \cdot \text{CH}_3$	Aromatic	16
Sulfur dioxide	SO_2	Pungent	30
Trimethylamine	$(\text{CH}_3)_3\text{N}$	Fishy-ammoniacal	4
Trichloroethylene	$\text{CH} \cdot \text{Cl} \cdot \text{C} \cdot \text{Cl}_2$	Aromatic	250

APPENDIX A

RESULTS OF AN OPINION SURVEY CONDUCTED IN 1955⁵

<u>Source</u>	<u>Frequency, %^{a/}</u>
Chemicals	62
Vehicles	52
Paint and Varnish	49
Food Processing	47
Domestic (homes, etc.)	45
Rendering Plants	43
Plastics	33
Oil Refineries	31
Coke Works	31
Rubber	27
Steel	25
Insulation	21
Fish	21
Gas Works	19
Pharmaceutical	19
Soaps and detergents	17
Breweries	15

$$a/\text{Frequency} = \frac{\text{no. of times the source was mentioned}}{\text{total no. of questionnaires}}$$

APPENDIX B

SOURCES OF ODORS AND THEIR COMPOSITION

<u>Source</u>	<u>Composition</u>
Chemical manufacturers:	Chlorine
Organic chemicals byproduct, electrolysis	
Dye-making, explosives, lacquers, refrigeration, textiles, chemicals	Ammonia, hydrogen cyanide
Soal making, fats, oils, glycerine, thermal decomposition, food processing	Aldehydes, dimethyl amine
Resins, adhesives, rubber, paints, varnish coatings	Phenolics, sulfur compounds, formaldehyde, solvents
Petroleum, petrochemicals	Hydrogen sulfide, organic sulfur compounds, hydrocarbons
Pulp and paper manufacture	Hydrogen sulfide, organic sulfur compounds
Fertilizers, fish wastes, spent acids	Amines, mercaptans, reduced sulfur compounds
Food products, canning	Decomposition products of nitro- genous compounds
Rendering, tanneries	Amines, reduced sulfur compounds, caproic acids, ammonia
Pharmaceuticals, breweries, fermentation	Amines, reduced sulfur compounds
Municipalities, dumps, lagoons, settling ponds	Decomposition products of nitro- geneous compounds
Textiles, paper	Urea, starch decomposition products

APPENDIX B Cont'd

<u>Source</u>	<u>Composition</u>
Coal gas manufacture	Sulfur compounds (hydrogen sulfide, carbon disulfide, thiophene, thiols, carbon oxysulfide)
Natural gas	Hydrogen
Diesel exhaust	Aldehydes
Coffee and chicory roasting	Aldehydes, hydrogen sulfide, mercaptans, phenols, organic acids, hydrocarbons
Domestic incinerators	Organic acids, aldehydes, hydrocarbons, nitrogen oxides, ammonia

APPENDIX C

Table 1. ODOR THRESHOLDS IN AIR
(ppm by volume)

<u>CHEMICAL</u>	<u>RESPONSE</u>	
	<u>50%</u>	<u>100%</u>
Acetaldehyde	0.21	0.21
Acetic acid	0.21	1.0
Acetone	46.8	100.0
Acrolein	0.1	0.21
Acrylonitrile	21.4	21.4
Allyl chloride	0.21	0.47
Amine, dimethyl	0.021	0.047
Amine, monomethyl	0.021	0.021
Amine, trimethyl	0.00021	0.00021
Ammonia	21.4	46.8
Aniline	1.0	1.0
Benzene	2.14	4.68
Benzyl chloride	0.01	0.047
Benzyl sulfide	0.0021	0.0021
Bromine	0.047	0.047
Butyric acid	0.00047	0.001
Carbon disulfide	0.1	0.21
Carbon tetrachloride (chlorination of CS ₂)	10.0	21.4
Carbon tetrachloride (chlorination of CH ₄)	46.8	100.0
Chloral	0.047	0.047
Chlorine	0.314	0.314
p-Cresol	0.00047	0.001
Dimethylacetamide	21.4	46.8
Dimethylformamide	21.4	100.0
Dimethyl sulfide	0.001	0.001
Diphenyl ether (perfume grade)	0.1	0.1
Diphenyl sulfide	0.0021	0.0047

Table 1. Cont'd

<u>CHEMICAL</u>	<u>RESPONSE</u>	
	<u>50%</u>	<u>100%</u>
Ethanol (synthetic)	4.68	10.0
Ethyl acrylate	0.0001	0.00047
Ethyl mercaptan	0.00047	0.001
Formaldehyde	1.0	1.0
Hydrochloric acid gas	10.0	10.0
Hydrogen sulfide (from Na ₂ S)	0.001	0.0047
Hydrogen sulfide gas	0.00021	0.00047
Methanol	100.0	100.0
Methyl chloride	[Above 10 ppm]	
Methylene chloride	214.0	214.0
Methyl ethyl ketone	4.68	10.0
Methyl isobutyl ketone	0.47	0.47
Methyl mercaptan	0.001	0.0021
Methyl methacrylate	0.21	0.21
Monochlorobenzene	0.21	0.21
Nitrobenzene	0.0047	0.0047
Perchloroethylene	4.68	4.68
Phenol	0.021	0.047
Phosgene	0.47	1.0
Phosphine	0.021	0.021
Pyridine	0.01	0.021
Styrene (inhibited)	0.047	0.1
Styrene (uninhibited)	0.047	0.047
Sulfur dichloride	0.001	0.001
Sulfur dioxide	0.47	0.47
Toluene (from coke)	2.14	4.68
Toluene (from petroleum)	2.14	2.14
Tolylene diisocyanate	0.21	2.14
Trichloroethylene	21.4	21.4
p-Xylene	0.47	0.47

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APPENDIX B

WORK PLAN

Odor Removal from Air by Adsorption
on Charcoal

Contract No. EHSD 71-4

Submitted by

Benjamin G. Kyle
Professor of Chemical
Engineering

N. Dean Eckhoff
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September 1970

WORK PLAN

In delineating the work plan for this contract research several distinct tasks can be identified. These tasks are described below and are scheduled on the accompanying "Milestone Chart."

1. Construction of Experimental Apparatus. A volumetric type of adsorption system will be constructed which utilizes radioactive tracers for measurement of the extremely low concentration of odorous compounds in the gas phase. The gas containing the tagged odorous compound will be circulated through a shallow bed of charcoal while the concentration of the tagged compound in the gas phase is continuously monitored with a radiation detector. A material balance calculation gives the adsorbent loading corresponding to any adsorbate partial pressure. Because the output of the radiation detector is continuous, both kinetic and equilibrium data may be obtained.

Initially a gas flow cell filled with powdered anthracene crystals will be used in conjunction with a Model 3375 Packard TRI-CARB Scintillation Spectrometer. A portion of the tagged (with ^{14}C , ^{35}S , or ^3H) gas being tested will flow through the cell. The anthracene crystals will serve to detect the beta particles resulting from the decay of the radioactive isotope disintegration. The signal from the anthracene crystal scintillator is analysed by a pulse height analyzer and the result is stored in one or more memory units. The Model 3375 TRI-CARB system consists, basically, of three single channel analyzers which have the capability of varying the lower level discriminator as well as the window

width for each channel. This variability allows one to discern between three different beta particles emitting radio-isotopes, provided their maximum energies are well separated. For example, it is quite easy to discern ^3H from ^{14}C , but not as easy to discern ^{14}C from ^{35}S . However, it should be possible to tag three compounds with ^3H , ^{14}C , and ^{35}S and to determine the relative concentration of each compound which flows through the cell.

If the flow cell scintillation detector yields unusable data, a thin window barrier Si semiconductor detector will be inserted in place of it. These detectors are not as efficient as the flow cell scintillation detector nor can different isotopes be discerned, but they are compact, rugged, inexpensive, and can be used as a beta particle detector.

Finally if neither of these systems work we will have to purchase the Johnson Laboratory TRITON flow ionization chamber as listed in the proposal. A copy of the specifications of this detector is attached.

2. Literature Search. A literature survey will be conducted covering the following topics:

Equilibrium and kinetic data for the adsorption of odors on charcoal.

General gas phase adsorption equilibrium -- theoretical treatment and engineering correlations.

General gas adsorption kinetics -- theoretical treatment and engineering correlations.

3. Testing and Evaluating the Experimental Apparatus. The apparatus will be tested by determining the adsorption equilibrium data for Ethyl Mercaptan (tagged with Carbon 14) on Pittsburgh BPL activated carbon.

These data can be compared with those of Grant, Manes, and Smith (1) who report the adsorption isotherm for this system in the range 10^{-1} to 10^{-5} atmospheres.

4 Experimental Study -- Adsorption of Specific Odorous Compounds. When the experimental apparatus and technique have been perfected, adsorption equilibrium and kinetic data will be obtained for certain selected compounds. The specific compounds chosen for study will be selected on the basis of their importance as air pollutants (e.g. mercaptans, disulfides, and aldehydes). To better understand the adsorption equilibrium behavior of odorous compounds it will be necessary to study several compounds within a homologous series. Adsorption equilibrium data for mercaptans on charcoal have been reported (1) for the moderate to low pressure range and a study of these systems should indicate whether adsorption equilibrium data at ultra low pressure can be obtained from data at moderate pressures.

5. Correlation of Adsorption Equilibrium Data. Concurrent with the experimental study work will be directed toward obtaining an understanding of the thermodynamics of adsorption at low concentrations, and developing correlations for the adsorption phase equilibrium data required for design of systems for adsorption of odorous compounds. The work of Grant, Manes, and Smith (1) implies that adsorption forces depend mainly on the nature of the functional groups comprising the adsorbate molecule and suggests that separate adsorption equilibrium correlation curves may be expected for each homologous series. Such a development would allow the prediction of adsorption equilibrium data for any member

of a homologous series once these data have been determined experimentally for one member of the series. This approach might be extended to adsorbates with several types of functional groups by means of a "group contribution" approach similar to that employed by Pierotti et al. (2) for correlating liquid phase solution behavior. It is also possible that the corresponding states principle (3), which has proved useful in dealing with gas phase interactions, would allow the correlation of adsorption equilibrium data if the significant molecular parameters can be identified.

6. Modeling of Experimental Kinetic Data. The experimental kinetic data will be analysed using both the differential and integral approaches. With the differential approach one determines the instantaneous rate of adsorption at various times from the quantity adsorbed versus time data and uses this to test rate expressions arising from various proposed mechanisms. The appropriate mechanism is established from the condition that for a single run parameters determined from the instantaneous rate of adsorption evaluated at different times should be identical. In addition to this, these parameters should be physically realistic. The integral approach is based on integrating these various rate expressions and comparing the resulting quantity adsorbed - time relationships with the experimentally determined relationship. If an integrated rate expression can be fitted to the experimental data and the evaluated parameters appear realistic, that mechanism is said to be valid. Both the differential and integral approaches will be considered in analyzing the kinetic data. The differential approach has the advantage of being capable of testing any postulated mechanism, while the integral approach is restricted to those simpler mechanisms whose rate expressions are integrable.

The object of the modeling study is to establish the rate mechanism and to determine the dependence of the parameters in the rate expression upon the system variables so that design of adsorption systems for odor removal can be accomplished.

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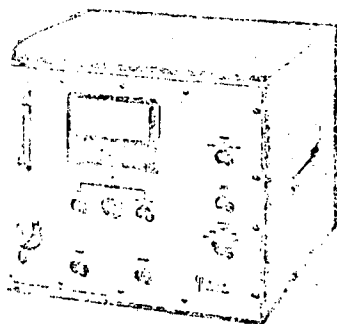
TRITON

Systems • Accessories

Low Level Detection • Measurement
for Radioactive Gases

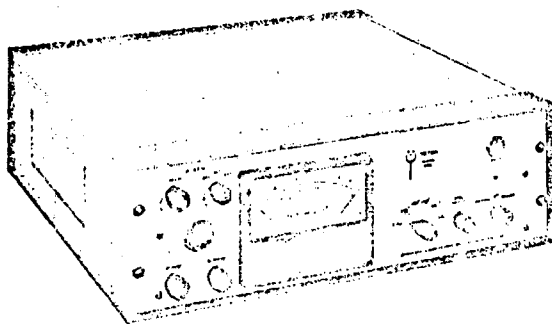
H³ • C¹⁴ • A⁴¹ • Rn²²² • S³⁵ • Kr⁸⁵ • Xe¹³³ • Xe¹³⁵ •

TRITON Model 955B



Sensitivity: H³: 10 μ Ci/M³ F.S. •
Accuracy $\pm 10\%$ •
Sensitivity, Gamma: 50 μ R/hour F.S. •
Input Filter: 0.5 micron • Electrostatic Precipitator •
Gamma Compensation: to 5 mR/hour •
Flow Chambers: 10 liter volume • Cleanable •
Positive Displacement Air Pump: 1 to 10 liters/min. •
Alarm: Variable Set Point • Visual • Aural •
Closed and Open Loop Operation • Gas Tight •
Recorder Output • Remote Accessory Connector •

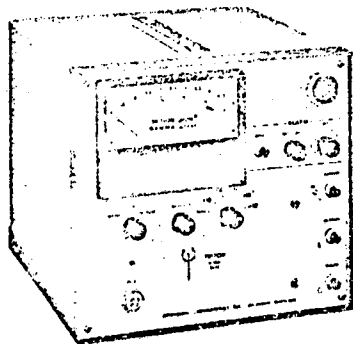
TRITON Model 755C



similar to 955B except:

Sensitivity, H³: 100 μ Ci/M³ F.S. •
Sensitivity, Gamma: 0.5 mR/hour F.S. •
Flow Chamber: 1.2 liter volume •
Flow Rate: 9 liters/min. •

TRITON Model 1055B



similar to 755C except:

Sensitivity, H³: 50 μ Ci/M³ F.S. •
Sensitivity, Gamma: 0.25 mR/hour F.S. •
Portable • Rechargeable Ni-Cad Batteries •
Flow Rate: 2 liters/min. •

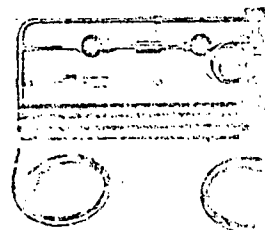
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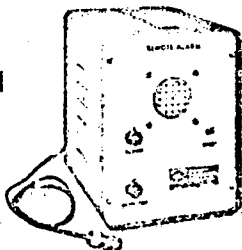
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Compact • Visual and Aural Alarm •
Powered from main instrument •
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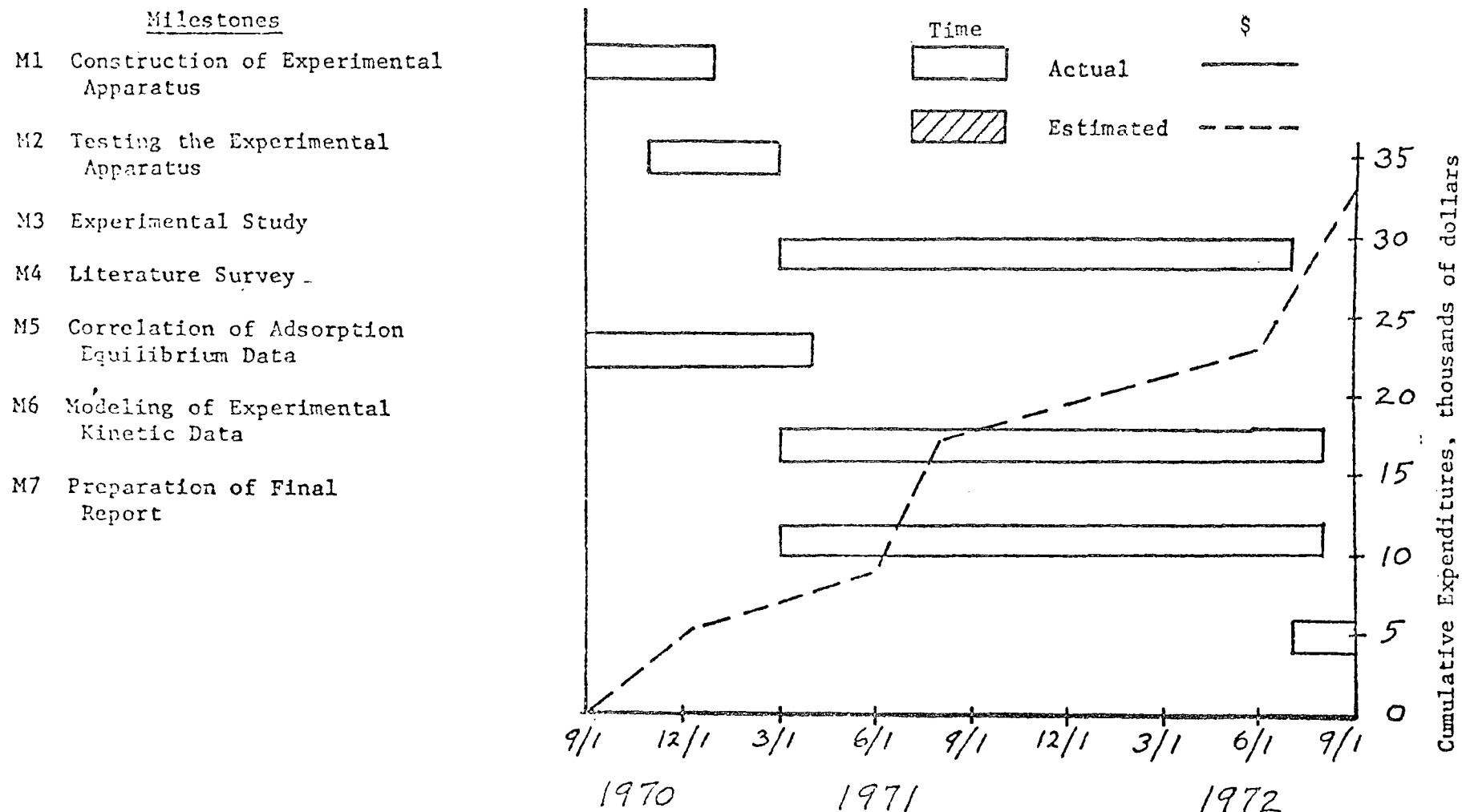
Contract: Odor Removal from Air by Adsorption
on Charcoal

Contract No.
EHSD 71 - 4

Contractor: Kansas State University
Manhattan, Kansas

Contract Dates
8/27/70 - 8/27/72

Project Officer
Dr. Belur N. Murthy



Completion Date
of Milestones

Actual
Estimated

1 2 4

3 5 7
6

MILESTONE CHART