FINAL REPORT

CONTROL TECHNOLOGIES FOR SOLVENT EMISSIONS FROM COTTONSEED OIL EXTRACTION PLANTS

ARB Contract No. A0-074-31
PES Project No. 523

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State of California Air Resources Board

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August, 1982

ERRATA

Page 4-5 (Figure 4-3). Delete line above caption.

Page 4-9 (Table 4-1). In fourth line of table, in Column B, the "Difference"is <u>-9</u> instead of -11.

Page 4-12, line 16. The formula should be given as $t/(5\sqrt{N+.5}-6)$. Also, in line 17, the value should be given as <u>2.13</u> instead of 0.213.

Page 4-20 does not exist.

ABSTRACT

Five cottonseed oil manufacturing plants in California were surveyed, using an organic vapor analyzer to detect high concentrations of solvent vapor in the immediate vicinity of solvent-containing equipment, to measure concentrations in gases from accessible vents, and to measure gradients of hydrocarbon vapors (assumed to be hexane) in the ambient air on the grounds and at the peripheries of the plants.

From these measurements and from auxiliary information supplied by the manufacturers, the following conclusions are reached:

1. Hexane losses from the 5 plants totalled about 800,000 gallons for the 1980-81 operating season, ranging from 0.75 to 1.4 gallons per ton of seed processed at various plants.

2. A large fraction of the hexane lost enters the atmosphere as vapor; probably less than half the vapor is lost through scrubber vents, with most of the remainder issuing from leaks in process equipment. The most frequently detected leaks were in solvent extraction equipment, miscella refining and processing equipment, and stearine centrifuges.

3. Vapor losses are substantially greater in summer than in winter.

4. Hexane concentrations in the ambient air around these plants are demonstrably greater downwind thatn upwind, although the difference is marginal at the plant peripheries.

ACKNOWLEDGMENTS

Successful completion of the work described in this report was possible only because of the cooperation and collaboration of many people. In particular, PES owes gratitude to the National Cottonseed Products Association, Inc., and to member firms of the Association, who arranged and assisted in the industrial facility surveys:

Anderson Clayton Oil Seed Processing Division, Chowchilla, CA
J.G. Boswell Cotton Oil, Corcoran, CA
Producers Cotton Oil, Fresno, CA
Ranchers Cotton Oil, Fresno, CA

We express our appreciation, also, to the personnel of the California Air Resources Board and of the Air Pollution Control Districts of Fresno County, Kern County, Kings County, Madera County and Tulare County, for their assistance in providing appropriate records for our review.

PES personnel who participated in this study included Leslie Norton, who was initially the project manager, Todd Brody, Thomas McCabe and Gary Quinn. Special thanks are also due to Dr. Katherine Wilson, who served as an expert consultant and provided valuable help in interpreting results and in preparing this report.

Although numerous documents and publications were reviewed in the performance of this study, special acknowledgment is owed to the work of Frances L. Cameron, whose engineering report, "A Study of Solvent Losses at the Ranchers Cotton Oil Plant in Fresno, California" supplied much of the information needed in relating PES observations to the technological status of the industry.

This report was submitted in fulfillment of ARB Contract Number AO-074-31 by Pacific Environmental Services, Inc. (PES) under the sponsorship of the California Air Resources Board. Work was completed as of August, 1982.

DISCLAIMER

The statements and conclusions in this report are those of the contractor and not necessarily those of the California Air Resources Board. The mention of commercial products, their source or their use in connection with material reported herein is not be construed as either an actual or implied endorsement of such products.

EXECUTIVE SUMMARY

Cottonseed oil is manufactured at five plants in California by extracting cleaned and de-hulled cottonseed with hexane. This study was done to document losses of hexane vapor to the atmosphere in cottonseed oil extraction operations and to assess the need for additional field testing that could establish a basis for measures to control hexane emissions.

EPA Control Technology Guidelines for vegetable oil extraction recommend that a mineral oil scrubber be used to control vapor emissions from the main extractor vent and that measures be taken to minimize the amount of hexane remaining in the meal after the oil has been extracted. These measures have already been implemented at all of the cottonseed oil plants in California.

Previous work, reported in the literature, has indicated that losses of solvent vapor from the main extractor vent of one of the California plants accounted for a relatively small fraction of the average rate of loss, and that losses in products and other waste streams were even less. This implies that most of the hexane which disappears from inventory is lost to the atmosphere through fugitive emissions, assuming that the conditions observed were typical of operations in the industry.

The Air Resources Board has a need to know whether the results described for one plant are reasonably representative for all five plants. To clarify the need for control measures, the Board also requires a more direct assessment of the importance of leaks from process equipment as a cause of fugitive emissions in the manufacture of cottonseed oil. Finally, the Board wants a determination of the possible effects of these emissions on hydrocarbon levels in the ambient air in the vicinity of the existing cottonseed oil extraction plants.

Five cottonseed oil extraction plants were inspected as part of this project. Detailed information has been submitted to the Air Resources Board in a separate, confidential report. This report provides a generalized description of typical extraction plants, an account of survey procedures, a discussion of survey findings, and a more specific compilation of conclusions and recommendations.

A typical cottonseed oil extraction plant operates 24 hours a day, 7 days a week, and up to 330 days a year. A typical plant processes at least 100,000 tons of crushed cottonseed in a year and produces at least 20,000 tons of oil which is either refined on site or shipped to off-site refineries. The extraction medium is commercial hexane, which is continuously recovered and recycled. Typical plant operations are:

- Seed preparation and conditioning
- Prepressing and other feedcake operations
- Solvent extraction
- Miscella processing
- Miscella refining
- Meal processing
- Solvent recovery

The cottonseed oil extraction process is shown schematically in Figure 2-1. Materials fed into the process are raw cottonseed, hexane, and steam. The products are prepress oil, extracted oil, lint, and meal; wastes consist of solid wastes (hulls, trash, etc.), waste water and vent gases. Some plants also use caustic soda and produce stearine and soap.

Small amounts of hexane may be present in all output streams except the solid wastes and prepress oil. Fugitive losses of hexane as vapor may occur from the extractor and from equipment used in miscella processing or refining, meal processing, and solvent recovery.

Field surveys were carried out at each of the five plants between November 5, 1981 and January 20, 1982. These surveys were preceded by meetings and correspondence with personnel of the Air Resources Board, the National Cottonseed Products Association, the air pollution control agencies of the counties involved, and the managements of the five manufacturing plants.

Written protocols for these visits were transmitted to plant officials prior to the visits. PES personnel met with plant managers and technical personnel for orientation and gathering of specific plant information. Plant managers provided site plans and plant diagrams for use in identifying sampling points.

Using an organic vapor analyzer (OVA), the survey team obtained three sets of concentration readings:

1. Ambient air on the grounds of the facility
2. Ambient air along the perimeter of the grounds, and
3. Air at locations near possible sources of leakage from equipment in the plant.

On the grounds, sampling points were chosen by a randomizing procedure in each of thirty squares of a grid overlaid on the site plan. At the perimeter, points were designated at approximately equal intervals for a total of 8 to 15 points. Equipment leaks were probed as suggested by inspection, keeping the probe at a uniform distance of one inch from identified potential leakage points. On the average, more than 100 such points were tested at each plant.

Annual hexane usage for each plant ranged from 73,000 gallons to 245,000 gallons, according to information furnished by the plant managers. These amounts represent quantities that had to be added to the system during the year to replace hexane lost to the atmosphere or sewer or remaining in the oil or meal. Relative to cottonseed throughput, these amounts correspond to loss ratios of 0.75 to 1.4 gallons per ton of cottonseed crushed.

Comparison of hexane concentrations in ambient air upwind and downwind of the oil extraction plants revealed, as shown in Table 4-1, a statistically demonstrable increase on the downwind side of all but one of the plants surveyed. Concentrations ranged from 0 to 7.5 parts per million by volume (as hexane), with only a few observations exceeding 3 ppm. There were no obvious, well defined gradients of hexane concentrations in the ambient air; the isolated high levels were observed near the boilers at three of the plants and near a newly painted building at one plant.

Calculations were done for a hypothetical source of hexane emissions at a steady rate of 20 gallons per hour (which is about the same as the average rate of hexane loss from the plants surveyed in this study) to estimate plausible downwind concentrations for comparison with the ambient air readings. These calculations, described in Section 4.2.3, indicated that the observed ambient air concentrations, as well as the observed differences between downwind and upwind concentrations, were consistent with reasonable assumptions regarding emissions, wind velocities and atmospheric dispersion characteristics at the facilities surveyed.

Results of the surveys of leakage from the equipment in hexane service are summarized in Table 4-2. Hexane concentrations were measured in the immediate vicinity of vents, flanges, valves, seals, and other equipment components beginning with the solvent extraction unit and continuing along the process line. High concentrations of hexane were observed most frequently around solvent extraction equipment -- access doors, Rotocel plates, viewing windows and viewlight flanges. (Typically an extraction unit contains multiple access doors, viewing windows, etc., and hexane concentrations range from 10 ppm to above the range of the instrument (10,000 ppm) at a single extraction unit.) High hexane concentrations were also measured near the miscella processing and miscella refining units, but no one item of equipment was consistently associated with high concentrations. Other high concentrations were observed near stearine centrifuges, water sumps and floor drains. Meal processing equipment yielded high hexane concentrations at three of the plants, but not at the other two. These observations suggested that the observed high levels might be reduced by improving general maintenance practices, without necessitating any major changes in the unit operations.

Gases vented from the solvent recovery operations, whose hexane content is ostensibly controlled by the mineral oil scrubber, showed moderate hexane concentrations at two of the plants, but at the third plant the concentration in the vented gas was above the instrument range and it was estimated that the flow observed could be 13 grams per minute or more. If such a flow were steady, it would amount to at least 6 gallons per day, and possibly much more since the upper limit of the concentration could be many times the instrument limit, which was 10,000 ppm.

Hexane levels at the water sumps were relatively low at 4 of the 5 plants tested, suggesting that the amount of hexane lost in the waste water is substantially less than the amount vented through the main stack.

Some calculations were made to explore the plausibility of the hypothesis that leaks from the equipment could account for a major fraction of hexane losses in cottonseed oil extraction. Although the upper end of the range of the OVA instrument was 10,000 ppm hexane, there were a total of 33 observations of levels exceeding this range, out of 582 readings recorded at the 5 plants. Hexane concentrations in such cases might be anything up to about 200,000 ppm, being limited mainly by the vapor pressure of the solvent. The calculations showed (Section 4.3.2) that at an average concentration of 100,000 ppm and a reasonable escape velocity of 100 meters per minute, a total leakage area of about 50 square inches (0.033 square meters) would account for a hexane loss rate of 25 gallons per hour. This total leakage area corresponds to 60 linear feet of poorly gasketed seals with an average gap of about 1/16 inch.

Thus, the observations made in this study confirm that cottonseed oil extraction plants generally have readily detectable fugitive emissions of hexane, especially in connection with the extractor equipment. These fugitive emissions may potentially account for a large fraction of the solvent lost in the manufacturing operations, but neither this study nor published previous work provides precise estimates of the magnitude of losses due to leakage from various units of process equipment.

Besides the evidence of variable equipment leaks which might be reduced by improved maintenance, there is evidence that overall emission rates of hexane may be substantially dependent on ambient temperature and that use of refrigerated water in cooling the mineral oil scrubbers might effect important savings of hexane. Since the surveys reported in this project were done in November and January, they do not necessarily represent year-round conditions in these cottonseed oil plants, which may be expected to yield higher levels of emissions during the months of July, August and September. In agreement with these expectations, one plant provided monthly data on hexane replacement for the 1980-81 season, which indicated that the loss ratio (gallons of hexane lost per ton of cottonseed crushed) was 1.83 gallons of hexane lost per ton during the period May 22 to November 21, but only 0.82 during the other half of the year. At another plant, operators achieved short-term hexane losses as low as 0.3 to 0.4 gallons per ton crushed after installing new heaters and coolers in the hexane recovery system and were hoping to reduce their annual loss rate from 1.03 to 0.75.

CONCLUSIONS

Cottonseed oil extraction plants typically process 300 to 600 tons of crushed cottonseed per day, operating continuously for up to 330 days in a year. Losses of hexane, which is used as a solvent for extracting the oil, are substantial, totaling in one year about 800,000 gallons for the five plants in California.

Surveys conducted at these plants showed that hexane vapor leaks from process equipment at numerous points in the manufacturing process. Further, concentrations of hexane in the ambient air downwind of these plants were found to be higher than concentrations upwind. Measured at the plant perimeters, the average difference was 0.22 ppm; within the plant grounds, average upwind-downwind differences as large as 0.74 ppm were demonstrated.

Hexane concentrations in the ambient air were occasionally as high as 7.5 ppm, although most of the observations showed less than 3 ppm, even inside the plant grounds. Calculations for a hypothetical plant indicate that the order of magnitude of these observed concentrations and upwind-downwind differences is compatible with reasonable assumptions as to wind flow and source strength on the occasions of the plant visits.

Hexane concentrations in the immediate vicinity of process equipment demonstrated the existence of vapor leaks, especially near solvent extraction equipment, miscella processing and miscella refining equipment, and centrifuges. In many instances, the concentration of hexane exceeded the range of the measuring instrument and may have been from 10,000 to 200,000 ppm. Fugitive emissions from such leaks may account for a large fraction of the solvent lost in manufacturing operations.

Hexane losses appear to be relatively higher in summer months (May to November) than in the other part of the year. This incidental information is consistent with the hypothesis that the hexane used in the process remains warmer during summer and leaks out of the equipment more rapidly because it has a higher vapor pressure. However, hot weather may also reduce the efficiency of the mineral oil scrubbers which are used in solvent recovery, resulting in increased losses from the main vent, unless adequate cooling or refrigeration is maintained.

In summary, the findings reported here lead to the following conclusions:

1. Hexane losses in cottonseed oil manufacturing at five California plants range from 73,000 gallons to 245,000 gallons per year, totaling about 800,000 gallons for the 1980-81 operating season.
2. In relation to production, hexane losses ranged from 0.75 to 1.4 gallons per ton of cottonseed crushed.
3. A large fraction of the hexane lost is lost to the ambient atmosphere, with smaller amounts entrained in process products and waste water.
4. A substantial fraction of the vapor (but probably less than half) is lost through the vents of scrubbers used to control emissions.
5. The major part of the vapor lost to the ambient atmosphere constitutes fugitive emissions due to leaks in process equipment.
6. The most frequently detected leaks were in solvent extraction equipment, miscella refining and miscella processing equipment, and stearine centrifuges. In the main they were associated with doors, windows flanges, and other gasketed joints.
7. Vapor losses are substantially greater in summer months than in winter.
8. Hexane concentrations in the ambient air around cottonseed oil facilities are demonstrably greater in downwind locations than in upwind locations, although the magnitude of the difference is marginal at the peripheries of most of these plants.

RECOMMENDATIONS

1. Any further efforts to estimate emissions or emission factors for hexane from cottonseed oil extraction should be directed toward assessing the variability of these emissions as well as their magnitude. There should be a systematic program at a cooperating facility, with provision for monitoring and quantitating leaks from process equipment, emissions from process vents, and rates of evaporation from storage vessels, sumps and waste receptacles. These sources should be evaluated at regular intervals during day and night operations, and the plant-wide evaluation should be repeated at least three times during an annual operating cycle. One such plant-wide evaluation should be done during the hottest part of the operating year. Methods of assessing these emissions are reviewed in Appendix B.

2. Pending more accurate evaluation of the factors which affect hexane losses, estimates of emissions should be based on an overall emission factor of 6.6 pounds of hexane per ton of cottonseed crushed. (This is equivalent to 1.2 gallons of hexane per ton of cottonseed crushed, or 3.0 kg. per megagram.) Unless a particular facility has undertaken innovative measures to reduce emissions, this factor should be accurate to within 20 percent for annual emissions.

3. Estimates of daily emissions for summertime operations should be based on a factor of approximately 10 pounds of hexane per ton of cottonseed crushed, although this value is substantially less reliable than the overall emission factor cited in the previous recommendation. Implementation of tested control measures at any operating facility should, of course, be taken into account when such estimates are made.

4. In the expectation of reducing hexane losses, operators of cottonseed oil extraction facilities should institute programs of routine monitoring and maintenance beyond current practice, to detect and correct substantial leaks from equipment in hexane service.

5. Operators of cottonseed oil extraction facilities should consider installing systems to improve the efficiency of hexane recovery by using refrigerated water for cooling mineral oil used in the scrubbers, and possibly for cooling liquid hexane or condensing vapors at other points in the extraction process.

DISCLAIMER

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1.0 INTRODUCTION AND BACKGROUND

Cottonseed oil is manufactured at five plants in California by extracting cleaned and de-hulled cottonseed with hexane. A typical plant may process as much as 500 tons/day of cottonseed and operate for 330 days a year. The 1977 emission inventory compiled by the Air Resources Board attributed 1670 tons/year of reactive organic gases to the manufacturing of cottonseed oil. This quantity of organic material is not large when compared to the total inventory for the state, but it is sufficient to cause the Air Resources Board to consider whether controls might be applied to the process to reduce these emissions. The purpose of this study was to supplement the existing data base with field survey measurements at the five California plants and assess the need for additional field testing that would form the basis for control measures.

The Environmental Protection Agency (EPA) assessed the need for controlling vegetable oil plants and published findings in a Control Technology Guideline (CTG) document (Henz et al, 1978)*. This document was based on limited test data obtained at soybean extraction plants. These data were extrapolated to cottonseed extraction plants by taking into account the different percentages of oil, meal, hulls, and trash in cottonseed and soy beans. The authors recommend that a mineral oil scrubber be installed on the main extractor vent and that measures be taken to minimize the amount of hexane remaining in the meal after the extraction is completed. In California these controls were already in place before the EPA guideline document was published; therefore, the document is not particularly relevant to the California situation.

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*Henz, Donald, D. Louden, F. Hall, K. Brothers and F. Bunyard, "Control of Volatile Organic Emissions from Manufacture of Vegetable Oils," EPA Report No. 450/2-78-035, June 1978.

Recently, an investigation of solvent losses at the Ranchers Cotton Oil extraction plant in Fresno was undertaken by a graduate student at U.C. Davis as a thesis topic for a Master of Engineering degree (Cameron, 1981).* A material balance was computed for the extraction, meal drying and solvent recovery operations by measuring the flow rate and solvent content of each stream. The original intent was to estimate fugitive losses at each step in the process by taking the difference between the solvent entering and leaving the extractor, drier or other piece of equipment. The fugitive losses would represent a small difference between two large numbers. Unfortunately, the variability of the hexane-containing streams entering and leaving the extractor was so large that daily losses due to fugitive emissions could not be quantitated. Attempts were also made to compute a material balance for the overall plant operation, but the total solvent loss at the plant was considered proprietary information and could not be reported in the engineering report. Even though the material balances could not be computed as originally intended, Cameron's engineering report contains useful information on solvent losses from vents and waste water and the amounts remaining in the oil and meal products. The study identifies solvent losses of 188 ± 94 Kg/day while the plant was processing 600 tons/day of seed. This figure does not include fugitive losses from flanges, access doors, pump seals, etc.

The Air Resources Board has a need to know whether the test results obtained by Cameron for the Ranchers Fresno plant are reasonably representative of the other four plants. The Board needs to know more about fugitive losses at cottonseed oil extraction plants to help clarify the need for control measures. The study that is described in this report is an attempt to identify fugitive leaks and suggest

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*Cameron, Frances L. "A Study of Solvent Losses at the Ranchers Cotton Oil Extraction Plant in Fresno, California," submitted in partial satisfaction of the requirements for the degree of Master of Engineering in the Graduate Division of the university of California, Davis, 1981.

remedies. All five plants were surveyed to measure hexane concentrations at the plant perimeter, at various locations on the plant grounds and near individual items of equipment that might leak hexane from the process stream.

2.0 PROCESS DESCRIPTION

Five separate cottonseed oil extraction plants were inspected as part of this project. Detailed information on each plant has been submitted to the Air Resources Board in a separate, confidential data report. In this report, which is prepared for general distribution, the confidential information has been used to synthesize a "composite" extraction plant that is described in this chapter. All of the plants are sufficiently similar to permit this type of generalization. In Chapter 4 test data for individual plants are presented but the plants are not identified. For more detailed descriptions of oil extraction processes, references cited in the BIBLIOGRAPHY (Section 6.0) may be consulted.

2.1 OVERVIEW

Raw cottonseed is the starting material for oil production. The seed is cleaned and de-hulled and the meats are softened by steam cooking. The meats are pressed to remove some of the oil, and the residual feed cake is extracted with hexane* to remove the remainder of the oil. The hexane-oil mixture (miscella) is processed to separate the oil and recover the solvent, and the oil is stored and subsequently shipped. The process varies somewhat from one plant to the next and is discussed in more detail in the following sections of this chapter. The meal from the hexane extraction is treated to recover the residual hexane. The meal is stored and subsequently sold as an animal feed ingredient. The recovered hexane is reused many times.

Cottonseed oil plants are not enclosed in buildings in California except for onsite offices and laboratories. Roofed areas are provided for cottonseed storage and certain other operations, but

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*The major component is n-hexane but lesser amounts of methyl cyclopentane and methyl pentanes are also present.

almost the entire plant site is ventilated by the prevailing winds.

Typically, a cottonseed extraction plant operates 24 hours a day, 7 days a week for 330 days a year (fewer days for some plants). A typical plant will process 100 to 200 thousand tons of cottonseed per year. Some plants refine oil so that it can be sold directly to the consumer while other plants send the product to off-site refineries for final purification. All plants practice on-site recovery of hexane solvent.

2.2 SEED PREPARATION AND CONDITIONING

Raw cottonseed is delivered to the mill by truck. The trucks and their contents are weighed and the seed is then dumped into temporary storage facilities. Screw conveyors move the seeds into ventilated storage houses or piles. The storage piles are covered with large tarpaulins. The approximate mass composition of the raw cottonseed is as follows (Cameron, 1981):

Hulls - 36%
Meal - 34%
Oil - 19%
Lint - 11%

The seed is conveyed from the storage piles (or houses) to delintors where lint and short cotton fibers (motes) still clinging to the seed are removed. Lint is routed to beaters for further removal of motes and trash, and pressed into bales.

Cleaned seeds are moved by screw conveyors to a shaker and then to a huller that cracks them into meat and hull fractions. The meats are steam cooked to soften them so a good quality, workable flake can be formed in subsequent operations.

2.3 PREPRESSING AND OTHER FEEDCAKE OPERATIONS

The seeds are next conveyed to continuous screw presses (expellers) which remove a portion of the oil. This oil may be routed directly to storage or to "miscella refining" for processing along with extracted oil. (See 2.6 for more details). The meal exiting from the screw presses is cooled, granulated and then conveyed to the solvent extractor. Typically this meal contains about 12% oil.

2.4 SOLVENT EXTRACTION

The oil-laden meal is contacted with hexane at ambient temperature in an extractor that may utilize counter-current meal and solvent flow patterns or may pass meal through a series of solvent-meal compartments. Residence time of meal in the extractor ranges from 1/2 to 2 hours. Each of the extractors investigated in this study was slightly different from the others, but all extractors perform the function of providing efficient contact between meal and solvent in a limited space. To minimize solvent losses, extractors are usually operated at a slight negative pressure.

The oil hexane mixture discharged from the extractor is usually about 80% solvent by weight. This mixture is termed "miscella" and is routed from the extractor to a miscella processing unit or to a miscella refining unit, depending upon the plant.

2.5 MISCELLA PROCESSING

Miscella is passed from the extractor to filters and subsequently to single-stage or double-stage evaporators where the solvent is vaporized. Solvent vapors are passed to a solvent recovery unit that processes solvent vapors from several different unit operations at the cottonseed oil plant. Solvent recovery is discussed in Section 2.8. Solvent evaporators normally operate at slight negative pressures and most use cooling water that is not refrigerated; however, some plants are now utilizing refrigerated cooling water to reduce hexane losses. After most of the solvent is removed by the evaporators, the residual oil is transported to the oil stripper for removal of the remaining solvent. The residual solvent is removed by steam-stripping and the oil is routed to storage. The solvent-laden steam is routed to the Solvent Recovery Unit (Section 2.8).

2.6 MISCELLA REFINING

At plants that practice "miscella refining" rather than "miscella processing" the miscella from the extractor is pumped to a holding tank where it is combined with the prepressed oil. The mixture is treated with an alkali (caustic soda) in homogenizers to neutralize and remove free fatty acids. After heating and cooling, the mixture is centrifuged to separate the heavier soapstock from the oil and solvent. The soapstock removed is discharged into a desolventizer. The partially refined miscella may either be desolventized as described in Section 2.5 or be further processed for the removal of saturated fats and waxes.

The next miscella refining stage, called "Winterizing," consists of cooling the miscella to crystallize the stearine present in the oil and separating the stearine crystals out in a centrifuge. After desolventizing, the "winterized" oil can be marketed as salad oil.

2.7 MEAL PROCESSING

The solvent-laden meal cake from the extractor is conveyed to the desolventizer, where solvent is removed by one of the following methods:

- Indirect heating in steamjacketed heat exchangers;
- Applying steam directly to the meal (usually accomplished in either a tube drier or in a sequence of steam-heated trays); and
- Applying superheated solvent vapor to the meal to evaporate the residual solvent.

Solvent vapors from these processes are conveyed to the central solvent recovery unit.

In some plants, cake leaving the desolventizer is further dried and cooled before proceeding to storage. In others, it is processed in a deodorizer which removes, under vacuum, final traces of solvent and odoriferous materials. Meal exiting the deodorizer is cooled and then conveyed to storage as a final product.

2.8 SOLVENT RECOVERY

Solvent vapors* from miscella processing (or miscella refining) and from meal processing (and deodorizing) are conducted to the central solvent recovery unit. The vapors are passed through condensers that separate a mixture of water and hexane from the gas phase. The hexane phase is returned to the hexane storage tanks, and the water phase is heated to boiling to remove any remaining hexane. Vapors from this step are returned to the solvent recovery system.

The noncondensible vapors are routed from the condensers to a counter-current mineral oil scrubber. Gases from the scrubber are vented to the atmosphere. Hexane-laden mineral oil is heated, steam-stripped and cooled before being recycled to the top of the scrubbing

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*In one of the plants in this study, solvent vapors are condensed at the miscella-refining and meal-processing units and the condensate (rather than the vapor) is pumped to the central solvent recovery unit.

column. Hexane-laden vapors from the steam stripping are passed back into the solvent recovery system.

2.9 SUMMARY

The cottonseed oil extraction process is shown schematically in Figure 2-1. In brief, the materials fed into the process are raw cottonseed, hexane and steam. The products are prepress oil, extracted cottonseed oil, lint, and meal; wastes consist of solid wastes (hulls, trash, etc.), waste water and vent gases from the mineral oil scrubber. Some plants also use caustic soda and produce stearine and soap. Small amounts of hexane may be present in all output streams except the hulls, lint, trash and prepress oil, which are separated before the cottonseed is extracted with hexane. Fugitive hexane losses may occur at the extractor and at all points downstream; however, attempts are made to minimize hexane losses by maintaining a slight negative pressure in the extractor. A flow diagram is shown in Figure 2-2 for the Ranchers plant in Fresno, which practices miscella refining rather than miscella processing. Specific flow diagrams for other plants cannot be included because this information is considered confidential; however, the diagram for the Ranchers plant has been published in the open literature and can, therefore, be given as an example.

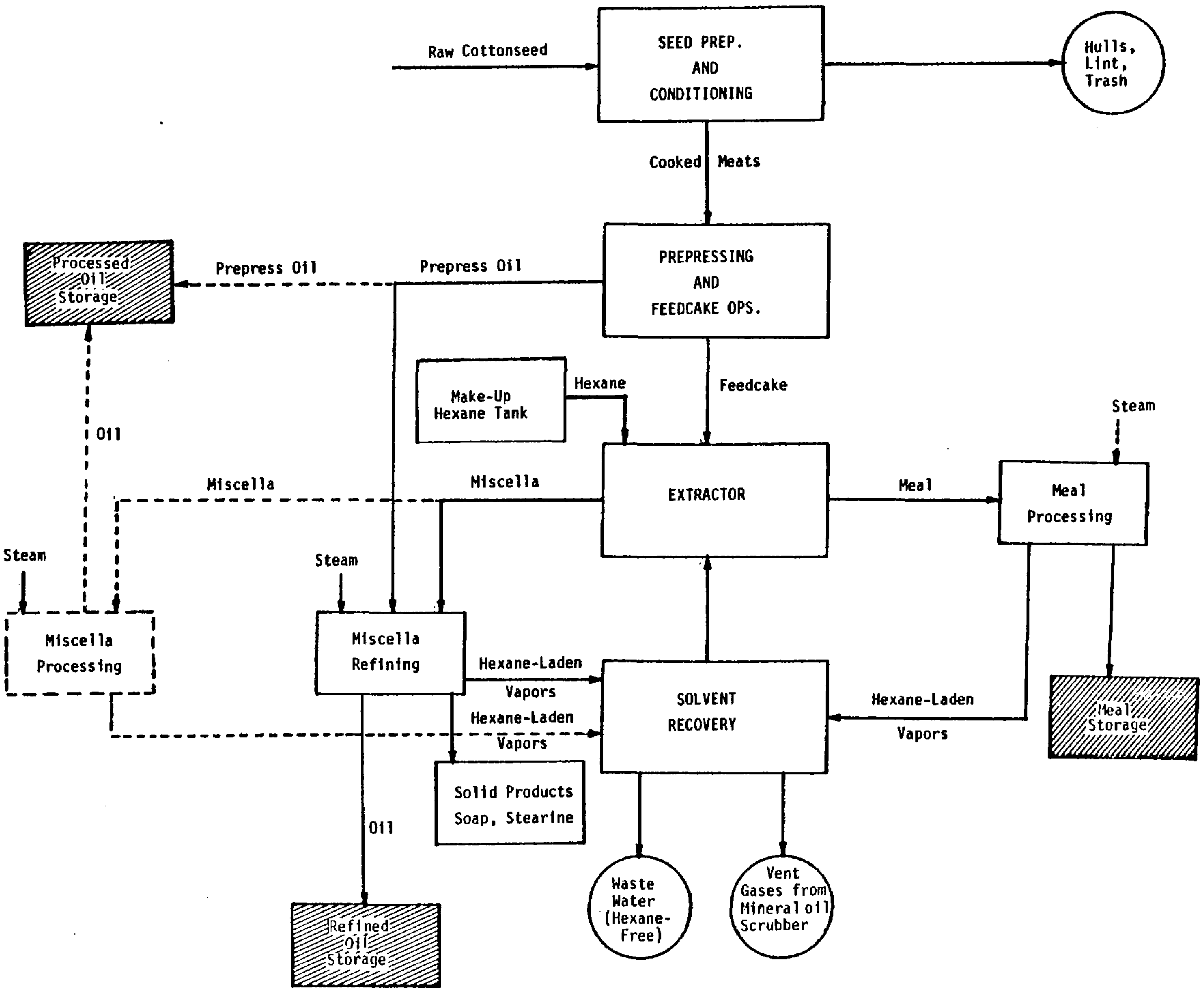

Figure 2-1. Cottonseed Oil Extraction Process (Schematic)

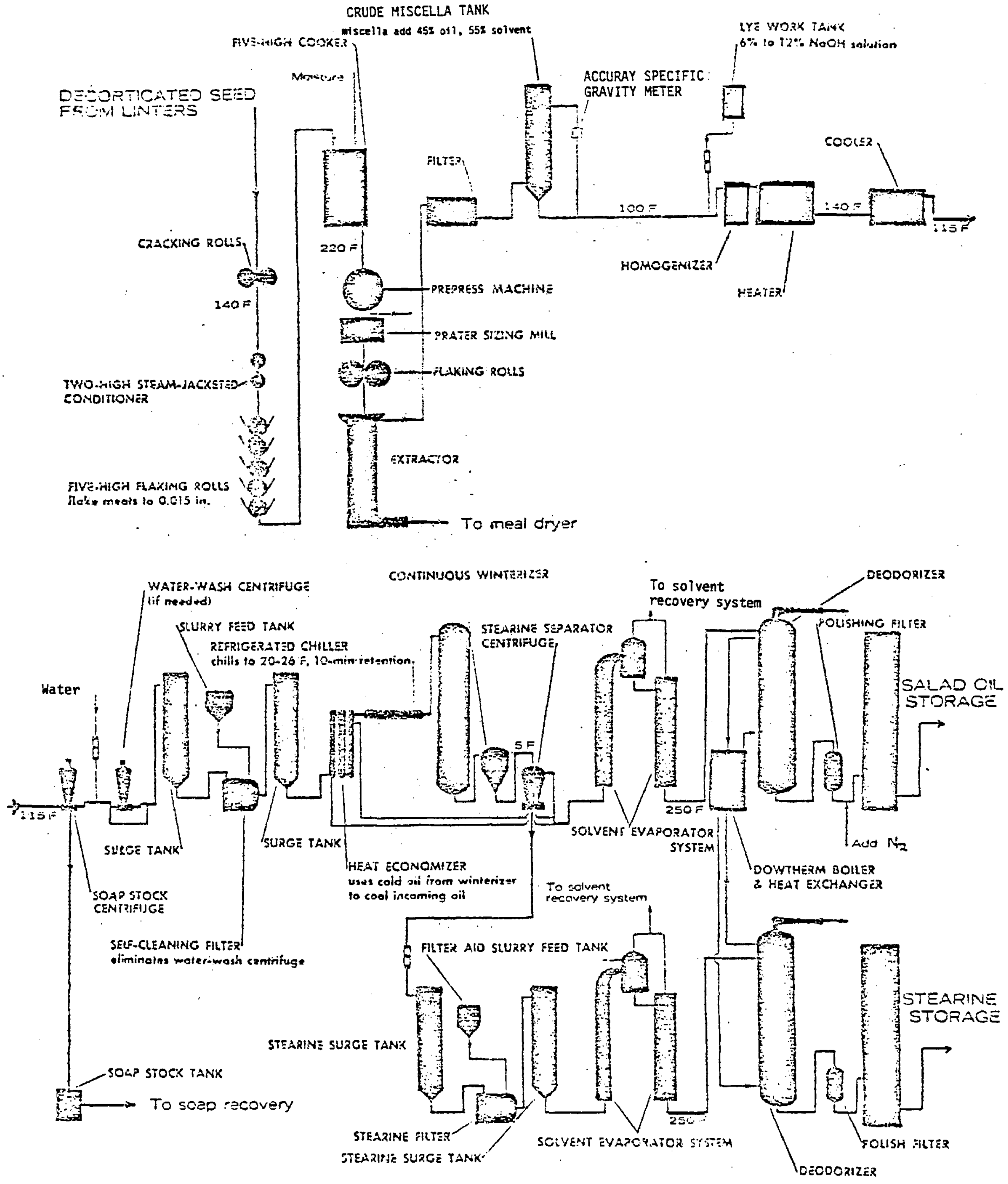

Figure 2-2. Flow Diagram of the Ranchers Solvent Plant (Source: G.C. Cavanagh, 1961)

3.0 FIELD SURVEY PROCEDURES

3.1 FACILITY VISITS AND PROTOCOLS

Field surveys designed to identify sources of fugitive emissions of hexane vapor were carried out at each of the five cottonseed oil manufacturing facilities, between November 5, 1981 and January 20, 1982. These surveys were intended to develop information for estimating the magnitude of fugitive emissions of hexane, using a portable organic vapor analyzer. For this purpose, three types of information were sought: organic vapor levels in the ambient air of the facility site; hexane levels in the immediate vicinity of operating equipment; and data on hexane consumption on a seasonal basis.

The facility visits were preceded by meetings and correspondence with personnel of the Air Resources Board, the National Cottonseed Products Association, the air pollution control agencies of the counties involved, and the managements of the five manufacturing plants.

Written protocols for these visits were developed through discussions in the meetings and subsequently transmitted to plant officials prior to the actual visits. The typical protocol was as follows:

> In preparation for each plant visit, PES will visit the pertinent local Air Pollution Control District (APCD) office and examine facility files. Typical types of information obtained from this practice include:
>
> - process description;
> - equipment specifications;
> - flow diagrams; and
> - previous process weight rates.
>
> This visit will take place on the morning of the first field day. In the early afternoon of the same day, PES will enter the facility. That afternoon will involve a complete review of the facility. Important elements to be made available will be:

- data concerning solvent usage rates;
- detailed schematics and process flow diagrams; and
- suggested key points in the process line for fugitive study focus.

It is anticipated that portions of the information obtained during this meeting will be considered proprietary.

Following a review of the process, PES will request a walking tour of the plant to observe the layout and examine potential sampling points. Based on review of all information, a sampling strategy will be developed.

The following morning, actual measurements will begin. A two member PES team will begin the sampling at the key locations in the developed strategy. The team will work all day and, depending on accessibility to selected points, anticipate being able to sample and document from 10 to 15 points per hour. The instrument to be used in the measurement aspect of the project is a portable Organic Vapor Analyzer (OVA) Model 108.

In summary, the PES visit will last 1-1/2 days. The first afternoon will involve gaining familiarity with plant operations and throughputs. Activities on the second, full day in the plant, will consist of data gathering and documentation.

This general protocol was followed in each facility visit, except that two of the plants were visited only on one day each.

3.1.1 PLANT DESCRIPTION

On each visit, PES personnel met with plant managers and technical personnel to inquire for the desired information, to be oriented to the site layout, to obtain process descriptions and to check the equipment specifications previously obtained at air pollution control district offices.

At this time the purpose and scope of the visit were also reviewed and a specific plant itinerary was agreed upon. Plant managers provided site plans and plant diagrams for use in identifying the specified sampling points.

3.1.2 SAMPLING PROTOCOLS

At each plant, the OVA was used to obtain three sets of readings. One set represented ambient air on the grounds of the facility; another, ambient air along the perimeter of the grounds; and the third, air at locations near possible sources of leakage from equipment in the solvent extraction plant (including all accessible units described in Section 2.3 to 2.8).

In order to specify a set of sampling points which would adequately represent the facility grounds each site plan was overlaid with a grid of 30 squares which approximately covered the grounds area, and a sampling point was chosen within each square, using a randomizing procedure to reduce bias due to subjective selection. If any point so chosen proved to be inaccessible, the nearest accessible point was substituted.

At the perimeter of the facility grounds, points were designated at approximately equal intervals, which resulted in a set of 8 to 15 perimeter points for each plant.

Finally, sampling points to represent the air in the extraction plant were designated, as a matter of practicality, on inspection of the various pieces of equipment identified in the plant description. The OVA probe was kept at a uniform distance of 1 inch (2.5 cm) from identified potential leakage points in this phase of the survey. On an average, more than 100 such points were tested at each plant.

3.2 VAPOR CONCENTRATION SURVEY

Solvent vapor concentrations were measured using a portable organic vapor analyzer (OVA), Model OVA-108, made by Century Systems Corporation. This instrument employs a hydrogen-flame ionization detector, which responds to carbon-containing gases and vapors in air taken in through a hand-held probe. It was used in conjunction with a strip-chart recorder which indicated detector response as a function of time, while the survey team noted the location of the probe as a function of time during the walk-through.

During the week preceding each facility visit, the OVA was calibrated so that readings could be interpreted directly in terms of hexane as parts per million by volume (ppmV, or simply ppm). To obtain equivalent concentrations in parts per million as carbon (ppmC), these readings would be multiplied by a factor of 6.0. Although the readings are thus expressed in terms of hexane, the concentrations observed may include contributions from other organic vapors or gases when these are present; thus, in principle, each reading reflects an upper limit for the amount of hexane present in the air sampled. In many of the ambient air readings, particularly in upwind locations, it is probable that the vapors detected originated in sources outside the plant and they may have contained no hexane at all.

Further details of the instrument, its principle of operation, and the calibration procedure will be found in Appendix A.

3.3 REVIEW OF FACILITY LAYOUT AND OPERATING DATA

In each of the five plants, managers and supervisors cooperated with the survey team by furnishing the requested information and site plans, and by responding to questions.

Data specifically sought included seasonal throughput in tons of cottonseed crushed, corresponding solvent usage (gallons), operating cycles (hours per day, days per week, weeks per year), plant capacity and current employment. In addition, samples of the solvent used for extracting the oil were requested; these were sent to ARB's laboratory in El Monte for analysis.

Because much of this information is considered confidential by the firms which assisted in the study, the detailed data are not presented in this report. Together with the flow sheets and plant descriptions, however, they have been used in developing the description of a typical facility for discussion herein (Section 2.0).

4.0 DATA ANALYSIS

The field surveys provided three kinds of data:

1. Hexane-use data (and hexane samples) provided by the cottonseed oil plant operators.
2. Ambient organic vapor* levels around the perimeter of each plant and in each of approximately 30 grid cells covering the plant grounds.
3. Hexane levels in the immediate vicinity of vents, flanges, valves, seals, etc. that appeared to be potential sources of hexane emissions.

The data are expected to provide a preliminary screening to characterize differences and similarities among plants and to help define the need for detailed follow-on studies. The program was not intended as an in-depth study of each individual plant.

4.1 HEXANE EMISSION RATES

The annual hexane usage by each of the plants in the 1980-81 season is summarized below:

Hexane Used	Loss per ton Crushed
73,000 gallons	0.75 gallons
130,000	1.3
132,000	1.03
220,000	1.2
245,000	1.4

These amounts represent the quantities that had to be added to re-

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*The OVA responds to all organic vapors; since it was calibrated with hexane, results are given in equivalent parts per million by volume as hexane. Especially in ambient air at low organic vapor levels, however, substantial fractions of the vapor sampled may be compounds other than hexane.

place the hexane lost to the atmosphere or sewer or remaining in the oil or meal. They are estimated by multiplying the annual cottonseed throughput by the hexane loss factor estimated by each manufacturer; these factors range from 0.75 to 1.4 gallons per ton.

It is instructive to estimate the total hexane throughput and compute the fraction of the throughput that is lost. Hexane throughput was not evaluated in the current study, but data are available from a recent master's thesis from UC Davis (Cameron, 1981). According to this study the solvent throughput was approximately equal to the throughput of cleaned meal. Assuming that the loss is about 1 gallon per ton of cottonseed crushed and that the cleaned meal is about one third of the cottonseed, the fraction of solvent throughput which is lost from the process may be estimated at about 1 percent. Cameron, however, was able to account for only about 0.13 gallons (per ton of seed crushed) by measuring solvent in vent, sewer and product streams. The solvent unaccounted for, nearly 90 percent of the total lost, is presumably lost as fugitive air emissions, through leaks from process equipment.

4.2 HEXANE IN AMBIENT AIR

4.2.1 UPWIND-DOWNWIND HEXANE OBSERVATIONS

The measured hexane* levels in ambient air are shown in Figures 4-1 through 4-5. The detailed plant layouts are considered confidential, but the relative locations of the extraction plant and the boiler are shown on each diagram for reference. The predominant wind direction is also shown.

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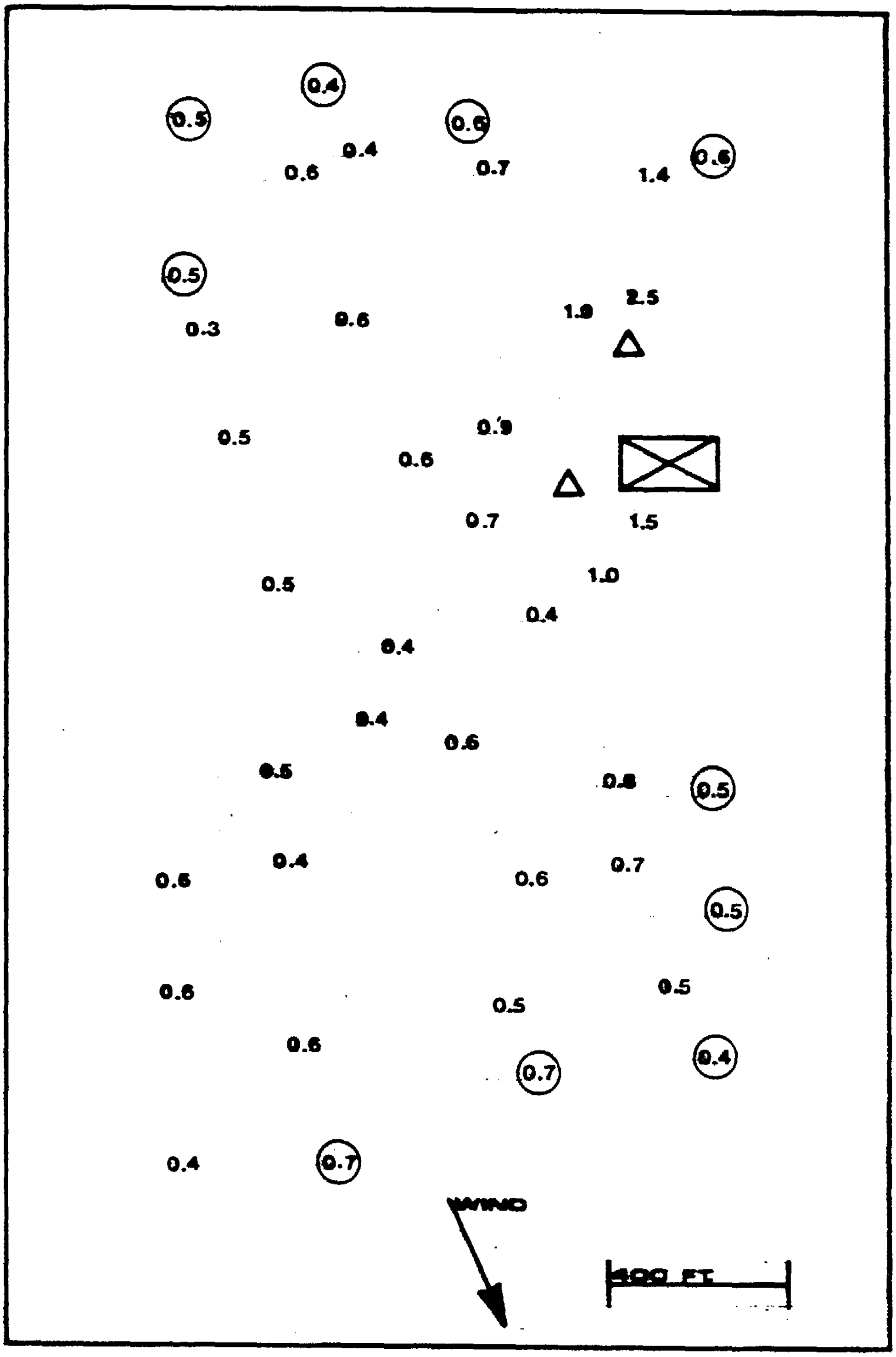
*Concentrations shown in figures and tables in this report have been corrected by subtracting 2.5 ppmV from the observed readings. This allows for any organics in the hydrogen gas supplied to the instrument and for organics (such as methane) which are always present in background air. The correction is selected to coincide with the lowest readings observed during the field studies.

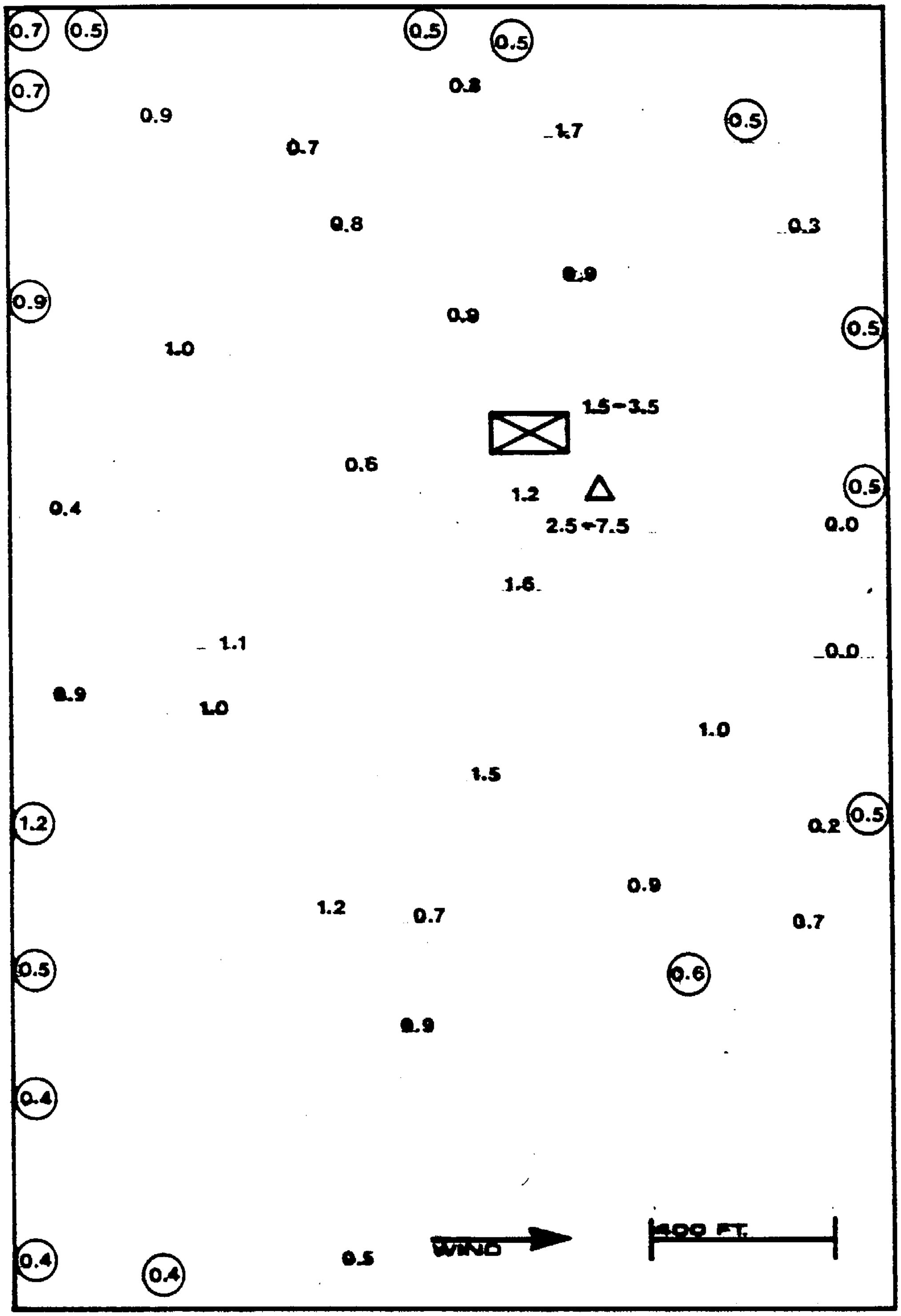


△ Boiler

☒ Extraction Plant

Circled values were measued approximately 1/2 hour later than the other values

Figure 4-1. PPM Hexane on Grounds of Plant A

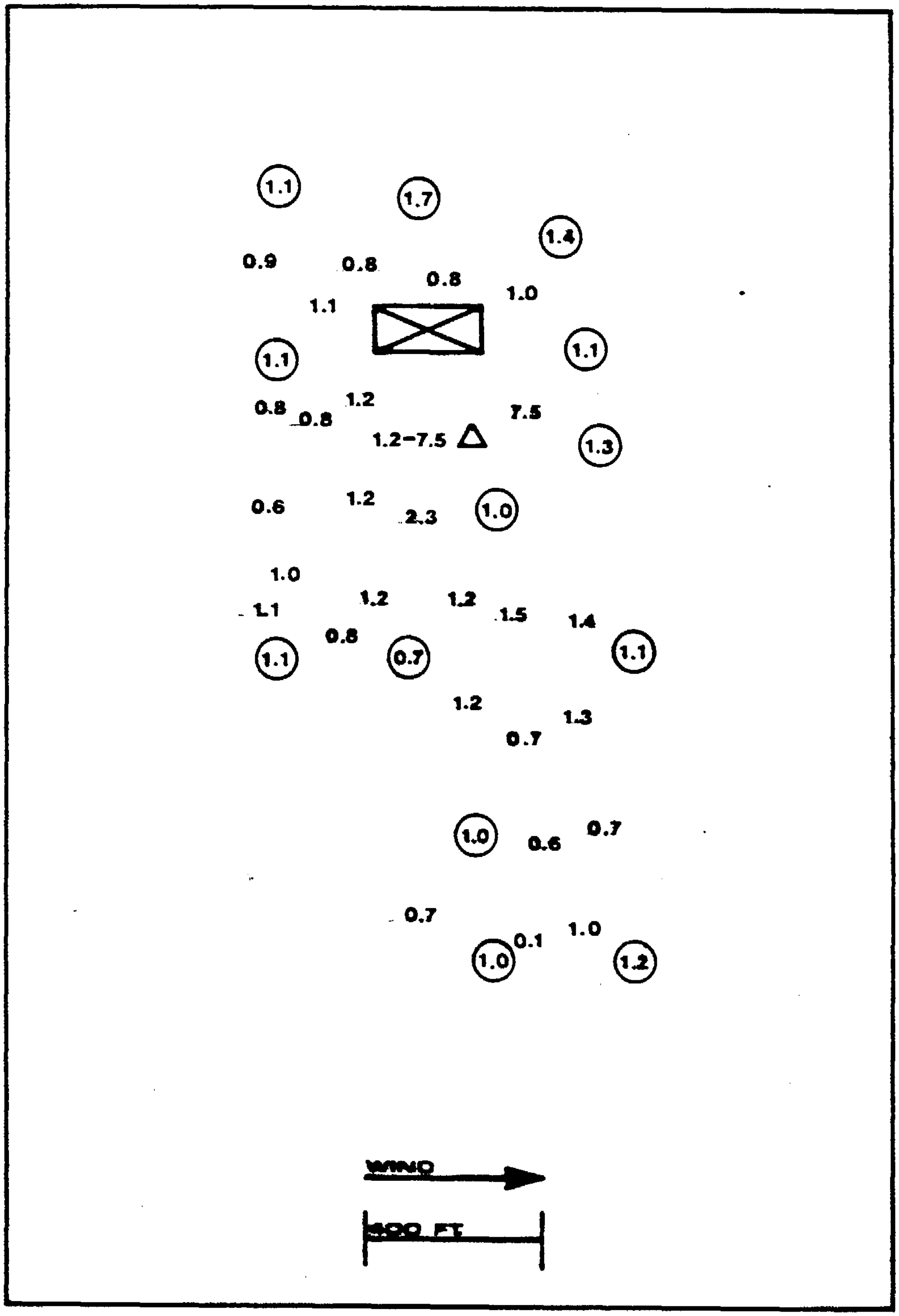


△ Boiler

☒ Extraction Plant

Circled values were measured approximately 1 hour later than the other values

Figure 4-2. PPM Hexane on Grounds of Plant B



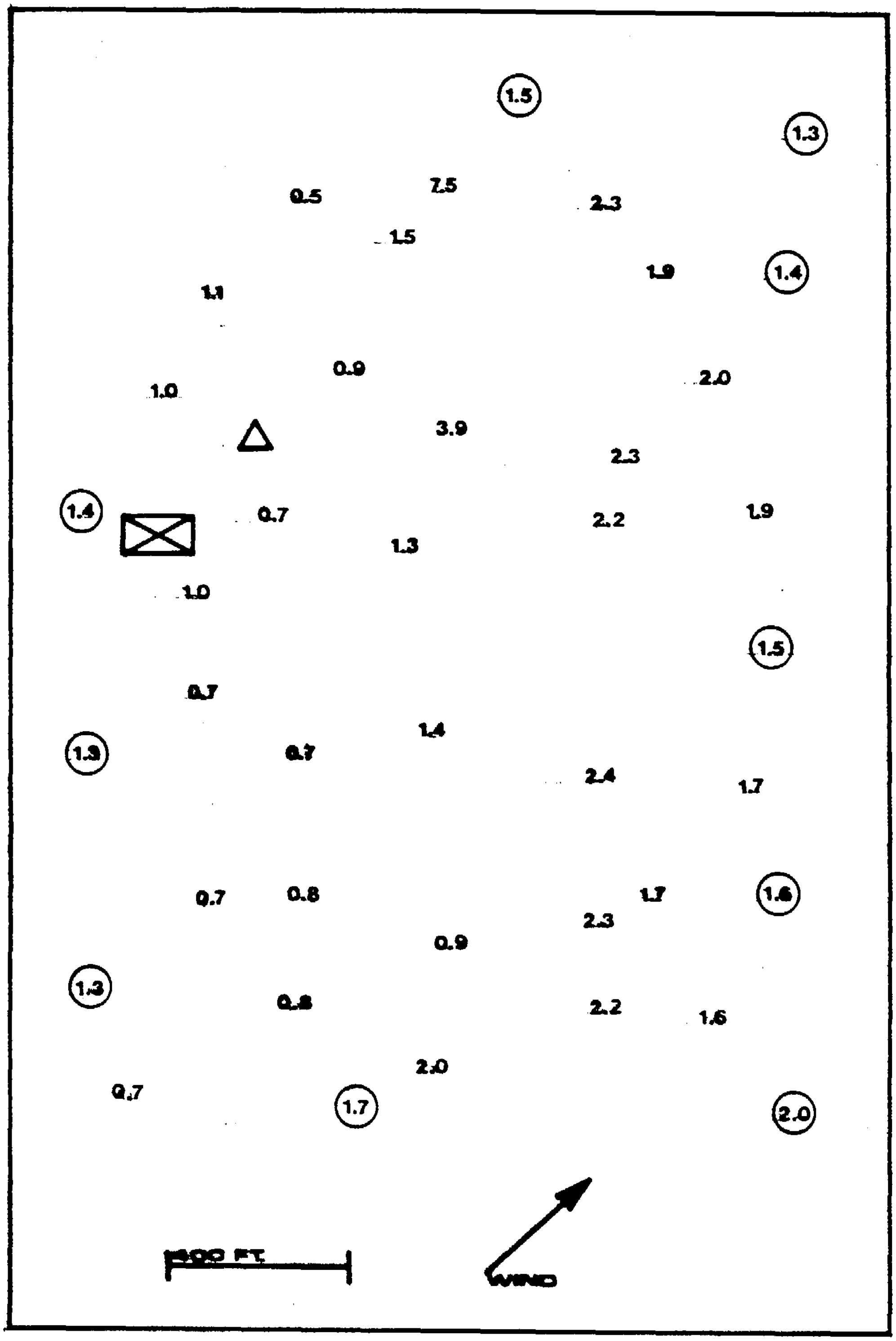
△ Boiler

☒ Extraction Plant

Circled values were measured approximately 1 hour later than the other values

*Next to recently painted building

Figure 4-3. PPM Hexane on Grounds of Plant C

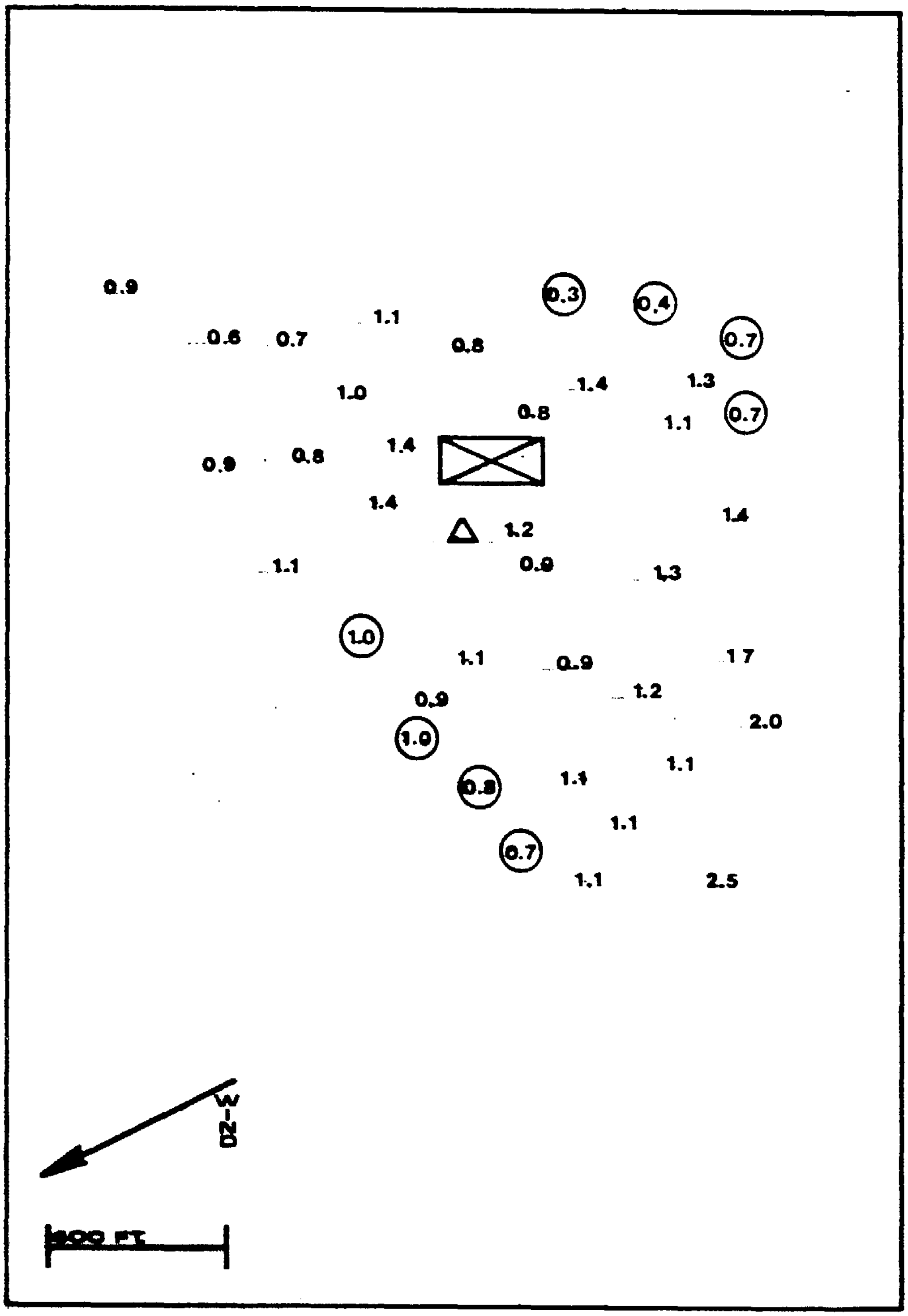


△ Boiler

☒ Extraction Plant

Circled values were measured approximately 1 hour later than the other values

Figure 4-4. PPM Hexane on Grounds of Plant D



△ Boiler

⊠ Extraction Plant

Circled values were measured approximately 4 hours later than the other values

Figure 4-5. PPM Hexane on Grounds of Plant E

Inspection of these figures shows immediately that there are no obvious, well defined hexane concentration gradients. Isolated high levels (i.e., greater than 3 ppm) were observed near the boilers at three of the plants and near a newly painted building at one of the plants. Hexane levels were normal in the grid cells adjacent to the cells where these high values were observed. These observations are consistent with the assumption that at ground level, the mass flows of hexane (or paint solvent) are very low and the ventilation was usually sufficient to dilute the hexane to low levels within a distance of 200 to 300 feet.

4.2.2 GRADIENT ANALYSIS

Hexane concentrations in the ambient air in the vicinity of vapor sources (such as the plants surveyed) vary strongly with time, due to the variability of wind speed and direction. As a result, when observed values are displayed on a chart (such as Figure 4-1 to 4-5), they may be suggest a rather random pattern, without readily detectable gradients. However, when a sufficient number of observations has been made, averaging can often serve to reduce the relative magnitude of the random component of variation and, thus, reveal information about the underlying spatial pattern.

To analyze the hexane observations depicted in Figures 4-1 to 4-5, three approaches were applied. The results, shown in Table 4-1, show clearly that organic vapor concentrations in the ambient air sampled during the plant surveys were, on the whole, higher at downwind locations than at upwind locations, as must be expected if large quantities of hexane are being emitted at these plants.

The first approach was a simple comparison of instrument readings at upwind and downwind locations near the plant perimeter. As shown in the first part of Table 4-1, the upwind-downwind difference at various plants ranged from -0.09 to +0.35 ppmV, where the plus

Table 4-1. ANALYSIS OF AMBIENT AIR SURVEY DATA*

Plant	A	B	C	D	E	Pooled
1. Perimeter						
Upwind	52:5	61:11	85:6	143:4	53:4	76:30
Downwind	56:5	52:5	116:8	155:6	88:4	98:28
Difference	+4	-11	+31	+12	+35	+22
2. Entire Site						
Upwind	68:18	71:28	108:18	108:13	120:19	92:96
Downwind	60:22	166:16	240:24	192:28	93:19	154:109
Difference	-8	+95	+132	+84	-27	+62
3. Quadrants						
Upwind	73:12	83:12	93:7	110:3	91:9	85:43
Crosswind	61:19	108:28	130:30	143:22	112:23	111:122
Downwind	58:9	158:4	250:5	207:16	110:6	159:40
Difference	-15	+75	+157	+97	+19	+74

*Format: a:b, where

a = average reading, parts per hundred million (pphm)

b = number of readings in average

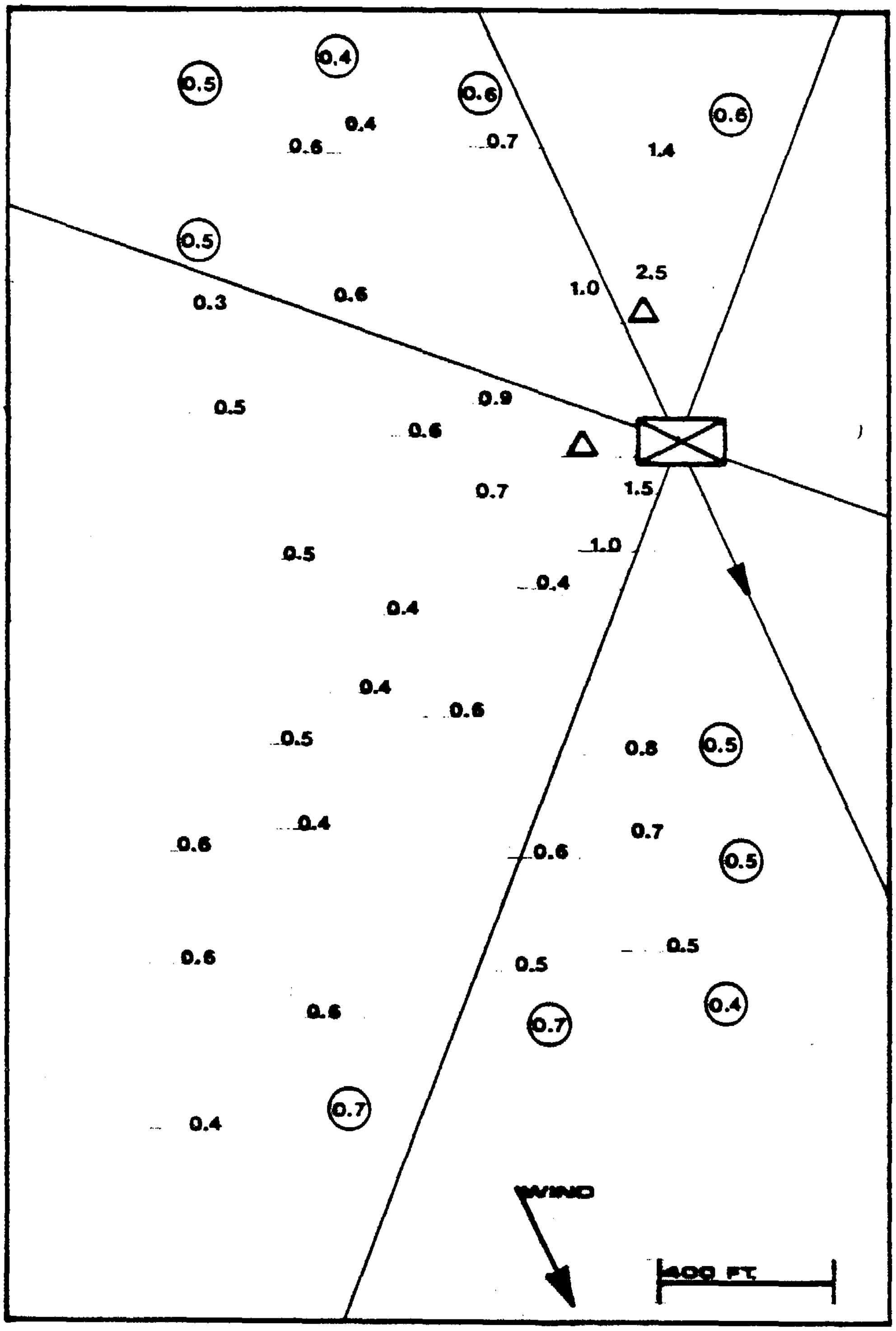
(Difference = average reading, downwind, minus average reading, upwind).

(Note:1 pphm = 0.01 ppmV)

sign indicates that the average downwind concentration was the higher. Although at one plant the difference was contrary to expectations, the average difference when all the data were pooled was positive and fairly substantial, viz., 0.22 ppmV.

Since it was obvious that the highest readings obtained in the survey were not found at plant perimeters, a second approach was taken. A line, perpendicular to the wind direction and passing through the location of the solvent plant on the site plan, was used to separate all the observed values at each plant into two groups, upwind and downwind. The differences between the averages for these groups are shown in part 2 of Table 4-1. They range from -0.27 to +1.32 ppmV. In this case, the difference found was negative for two of the plants; it is interesting to note that these two do not include the plant with a negative result from the perimeter data. Again, when all the data were pooled, the overall difference was positive (and much larger than that found from the perimeter approach), namely, 0.62 ppmV.

Examination of the plots indicated that the differences found with the second approach might be biased by random fluctuations at locations distant and nearly across the wind from the primary hexane source, the extractor. The third approach was devised to reduce this influence. Two lines were drawn through the extractor location and at angles of 45 degrees to the wind direction. As shown in Figure 4-6, these two lines divided the area into quadrants, such that one defined a 90-degree sector in the downwind direction, while the others defined similar sectors in the upwind direction and two opposite cross-wind directions. Results of averaging the observed values found in these sectors are shown in the third part of Table 4-1. The difference between downwind and upwind averages for various plants ranged from -0.15 to +1.57 ppmV. Again, when data from all plants were pooled, the overall result was positive and larger than found with the previous approaches, namely, 0.74 ppmV.



△ Boiler

⊠ Extraction Plant

Circled values were measured approximately 1/2 hour later than the other values

Figure 4-6. PPM Hexane on Grounds of Plant A
(Quadrant scheme for evaluating upwind-downwind difference)

These results, presented in part 3, Table 4-1, show that the survey technique is sensitive enough to confirm the expected effect of solvent emissions on ambient air concentrations. To show statistical significance, pooled differences shown in Table 4-1 were expressed as a fraction of the corresponding range of values of the tabulated differences. Three tests were made, one for each part of Table 4-1. The criterion value was 0.375, which corresponds to a 95% probability that the observed fraction is greater than the expected value of 0.0 (using a one-way null hypothesis). The observed values were,

for perimeter observations, 0.478 [= 22/(35+11)]
for entire site , 0.390 [= 62/(132+27)]
for wind-oriented quadrants, 0.430 [= 74/(157+15)]

Since the criterion value is exceeded in each case, it is concluded that the effect tested is demonstrated.

The criterion value, 0.375, is calculated according to the formula $t/(5\sqrt{N+.5})$, where N is the number of items in the set (in this case, 5) and $\underline{t}$ is the value 0.213, taken from standard t-tables for 4 degrees of freedom and an $\underline{\epsilon}$ of 0.10. The formula is derived from equations given by Lord (1947) quoted in Tate (1957).

A possible reason for discordant results at plant A is that the predominant wind direction at the time of the survey was such as to carry vapors emitted from the extractor beyond the plant perimeter with only a short path over the plant grounds, as can be seen in Figure 4-6; consequently, no readings were obtained in the downwind sector. Again, concentration values observed at this plant were substantially lower, on the whole, than those observed at the other plants, even though the plant has the largest reported annual cottonseed throughput of the five. It is probable that the sampling scheme, being confined to the plant grounds, simply missed the bulk of the actual hexane plume from this plant.

4.2.3 PLAUSIBILITY OF RESULTS

To establish the order of magnitude of hexane concentrations that it might be reasonable to expect in ambient air in the vicinity of a typical extraction plant, let us consider the example of a steady source of hexane vapor being fed into a wind tunnel of adjustable cross section. Suppose that hexane is evaporated at a rate of 20 gallons per hour, a source strength which is about the same as that of overall loss from a typical cottonseed oil extraction facility, using 100,000 gallons in 5,000 hours of operation.

At the given evaporation rate of 20 gallons per hour, in a wind tunnel having a cross section of 100 square meters (about 1,000 square feet), with air moving at a walking speed of 100 meters per minute, the average concentration of hexane in the tunnel would stabilize at about 25 ppmV. A stiff breeze of, say, 300 meters per minute (10 miles per hour) would reduce the steady-state concentration to about 8 ppmV. A smaller tunnel such as 10 square meters (100 square feet) would increase these concentrations by a proportional factor, to 250 ppmV for 100 meters per minute and 80 ppmV for 300 meters per minute, respectively.

In outdoor air in the vicinity of a source, as in a wind tunnel, concentrations should tend to be higher when wind velocities are lower, and vice versa. Under conditions of moderate dispersion, it may be reasonable to expect the vapors from a point source to affect an area of 100 square meters at a distance of 120 meters from the source. Thus, if the wind were really steady, a probe 120 meters from such a source and directly downwind of the source might be expected to register as much as 25 ppmV in a breeze of 100 meters per minute.

Nevertheless, the fact that no readings greater than 7.5 ppmV of hexane were observed in the surveys is readily consistent with the existence of hexane vapor sources of the postulated magnitude (20 gallons per minute). This is reasonable in view of the small probability of encountering outdoor sampling conditions meeting the specifications of the example: namely, a steady but gentle breeze, with the probe directly downwind of a point source, and the vapor from the source more or less confined to the postulated cross section (100 square meters). Again, the most important source at each extraction facility is very probably an elevated vent from the solvent recovery system; it is reasonable to suppose that dilution of vapor from these vents is enhanced by complex airflow patterns around the equipment sheds and that the vapors emitted at roof level may not readily mix uniformly to ground level, where the sampling is done.

In view of the uncertainties and uncontrolled conditions discussed above, it may be concluded that the survey measurements are reasonably consistent with the postulated loss of 10 to 20 gallons (25 to 50 kg) per hour, at the facilities visited.

Another question which may be similarly examined is whether the indicated downwind increase of hexane levels in ambient air at the plant perimeter is reasonably compatible with the presumed emission rate. Downwind plant perimeter readings actually ranged from 0.4 to 2.0 ppmV, with an average of 0.98 and an average increase (relative to upwind readings) of 0.22, as shown in Table 4-1.* Under conditions previously discussed, i.e., emissions of 20 gallons per hour and air moving at 100 meters per minute, an average concentration of 0.25 ppmV would be attained for an equivalent downwind area of 10,000 square meters. This could correspond to a vertical depth of 10 meters over a downwind site perimeter of 1,000 meters, or 20 meters over 500 meters, and so on.

This comparison seems appropriate and reasonable, in view of the fact that the areas surveyed, as shown in Figures 4-1 to 4-5, all had maximum dimensions between 500 and 1,000 meters. Vertical dispersion of effluent plumes is often much less than horizontal dispersion, since the horizontal components of wind direction are much more variable than the vertical component. We may, therefore, conclude

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*Note: 1 ppmV = 100 pphm.

that the average downwind concentration increase of 0.22 ppmV is consistent with reasonable assumptions about emissions, wind velocities and dispersion characteristics at the facilities surveyed.

4.3 HEXANE EMISSIONS FROM EQUIPMENT IN HEXANE SERVICE

Hexane concentrations were measured in the immediate vicinity of vents, flanges, valves, seals and other equipment components beginning with the solvent extraction unit and continuing along the process line. Between 100 and 200 readings were made at each plant. Every effort was made to survey all key components, but a few inaccessible items were encountered at each plant. The individual sampling locations and hexane concentrations are not presented because they were considered confidential; however, they are included in the supplementary data packages that has been submitted separately to the Air Resources Board. A summary of the results is given in Table 4-2. The equipment varies from one plant to the next, but an effort has been made to group the components into common categories that apply to most of the plants tested.

4.3.1 OBSERVED HEXANE CONCENTRATIONS NEAR EQUIPMENT

High hexane concentrations were observed most frequently around solvent extraction equipment -- access doors, Rotocel plates, viewing windows and viewlight flanges. However, low hexane concentrations were also measured near the same kind of equipment. Typically an extraction unit contains multiple access doors, viewing windows, etc., and hexane concentrations will range from 10 to >10,000 ppm hexane at a single extraction unit. Possibly the leakage may be associated with the condition of the sealing gaskets or with the care used in assembling and tightening the seal. The survey suggests that these emissions may be attributable to poor maintenance practices rather than to inadequate design of equipment.

Table 4-2. HEXANE CONCENTRATIONS (ppmV) NEAR EQUIPMENT IN HEXANE SERVICE

Plant:	A	B	C	D	E
Solvent Extraction					
Viewing windows	7-70	20-U	10-U	5-U	10-U
Viewlight flanges	10-30	50-U	U-U	5-500	n.a.
Rotocel plates	n.a.	50-U	n.a.	n.a.	10-U
Mechanical seals	4-U	10-100	U-U	4-400	3-1000
Equipment openings	100-U	100-200	10-U	U-U	30-100
Misc. flanges and seals	5-700	100-175	5-50	n.a.	5-4500
Access doors	20-U	4-300	7-200	5-250	n.a.
Miscella Processing	n.d.		n.d.	n.d.	
Valves		5-8			4
Pump seals		10-20			4-20
Flanges, access doors		10-U			4
Viewing windows		6-9			n.a.
Tank drain		10-100			4-8
Misc. packing glands, etc.		U			4-1000
Miscella Refining		n.d.			n.d.
Valves	n.a.		4-600	n.a.	
Pump seals	4-130		4-200	4	
Flanges	5-12		4-50	4-40	
Deodorizer, hot well	n.a.		n.a.	250	
Centrifuge access, discharge	U		n.a.	10-900	
Viewing windows, doors	5-U		4-150	50-300	
Floor washings, sumps	300-700		200	800	

Table 4-2 (Concluded) HEXANE CONCENTRATIONS (ppmV) NEAR EQUIPMENT IN HEXANE SERVICE

Plant:	A	B	C	D	E
Meal Processing					
Level controls	n.a.	n.a.	7-U	n.a.	n.a.
Access doors	4-20	7-U	10	U	30-800
Drier packing gland	n.a.	n.a.	n.a.	100-200	n.a.
Drier discharge	3-5	3-5	11	25	4
Misc. seals	5-50	n.a.	4-15	6-70	4
Pump seals	4-5	5-20	4-10	n.a.	n.a.
Feed conveyor	n.a.	4-1000	5-200	50-150	4
Solvent Recovery					
Water sump	18-25	10-20	70-500	15	4-8
Main vent	120-170	n.a.	U	300	n.a.
Hexane storage vent	n.a.	5-8	4	5-15	4
Blower, pump and valve seals	n.a.	7-7000	4-12	n.a.	4-1000
Access plates, seals	n.a.	5-20	n.a.	5-7	4-20

Notes: n.a. signifies equipment not accessible or not present

n.d. signifies operation not done at this plant

U indicates concentration above the range of the instrument, i.e., above 10,000 ppmV.

High hexane concentrations were also measured around the miscella processing and miscella refining units, but no one item of equipment was associated consistently with high concentrations. Since miscella refining has more unit operations than miscella processing, there are more potential sites for fugitive emissions. High hexane concentrations were measured near the stearine centrifuge at both plants where this operation is carried out and in the vicinity of water sumps and floor drains at all plants. Generally, high hexane levels result from small-scale, manual operations where residues are collected in open buckets or small sumps. The mass emissions of hexane are probably low since the solvent evaporates from a small surface area into relatively static air.

Meal processing is carried out at two of the plants without producing high hexane concentrations near any of the equipment. At the other three plants, high levels were measured near the inlet to the meal processing unit and slightly elevated levels near the meal discharge area. The latter is not surprising, since it is known that a small percentage of hexane remains in the meal after the solvent recovery step is completed. The observations suggest that the observed high levels might be reduced by improving general maintenance practices rather than making major changes in any of the unit operations.

Hexane emissions are associated with solvent recovery operations because the mineral-oil scrubber is less than 100 percent effective in removing hexane from the noncondensible gases (usually air or nitrogen) which accumulate in the system. In addition, small amounts of hexane remain dissolved in the waste water and are discharged to the sewer. These losses are inherent in the process, but their magnitude depends on equipment design and operational procedures. The main solvent recovery vents were accessible at plants A, C and D but inaccessible at plants B and E. Hexane concentrations in the vent gases ranged from 120 to 10,000 ppm. Since the flow rates through these vents were unknown, it was not possible to make an accurate estimate of hexane emissions.

However, at plant C, air flow from the scrubber vent was measured at approximately 15 cfm, with hexane concentration exceeding 10,000 ppm. This contrasts with values reported for Ranchers Cotton Oil in Fresno as 6,630 ± 240 liters per minute (234 ± 8.5 cfm), with hexane concentration of 3,600 ± 2,100 ppm (Cameron, 1981). If the air flow and concentration are assumed to be steady, the calculated scrubber emissions for plant C are at least 13 grams per minute (6 gallons per day), which may be compared with the corresponding estimate for Ranchers in Fresno, viz., 46 ± 28 gallons per day.

Hexane levels at water sumps were relatively low at 4 of the 5 plants tested and suggest that the amount of hexane lost in the waste water is substantially less than the amount vented through the main stack.

4.3.2 SUMMARY AND DISCUSSION OF POTENTIAL FUGITIVE EMISSIONS

The results displayed in Table 4-2 and discussed in Section 4.3.1 indicate that the principal sources of fugitive emissions may be leaks from equipment in hexane service. Although the upper end of the range of the instrument being used was 10,000 ppmV, almost six percent of the observations exceeded that level. (Table 4-3 shows the frequency of observed levels in various ranges in the plant surveys). There were, in fact, a total of 33 such observations at the 5 plants out of 582 readings recorded.

Concentrations exceeding the range of the instrument were not accurately quantifiable, but it is reasonable to suppose that vapor-laden gas escaping from the equipment may contain hexane near the saturation level, which, at ambient temperatures commonly encountered in these plant locations, may be as much as 200,000 ppmV, or 20 percent by volume. It is instructive to consider under what conditions such leaks might account for hexane losses of the magnitude reported by the manufacturers.

As an example, we may take the case of the Ranchers Cotton Oil plant in Fresno, cited by Cameron as processing 600 tons of crushed seed per day. Assuming a loss ratio of 1.0 gallon per ton in excess of the amounts accounted for by Cameron, the amount of hexane leaking would come to 600 gallons per day or 25 gallons per hour. This is roughly equivalent to 20 cubic meters of vapor per hour. If leaking gas from the equipment contained, on the average, 10 percent vapor (by volume), 200 cubic meters per hour would carry this much vapor. At a fairly low exit velocity of 100 meters per minute, this much flow would require a total opening of 0.033 square meters, or about 50 square inches.

Although 50 square inches seems a rather large opening for closed and gasketed equipment, it is nevertheless true that high concentrations of hexane were encountered at numerous sampling points in each plant. If, throughout a plant, there existed 60 linear feet of poorly gasketed seals with an average gap of about one sixteenth of an inch, it is conceivable that 25 gallons of hexane per hour could be lost through such leaks.

Alternatively, it is possible that the estimate of losses in product and waste streams, given by Cameron as about 75 gallons per day (about 3 gallons per hour), does not accurately represent these losses on a year-around basis. In this study, hexane concentrations in the gas from the main solvent recovery vent were 300 ppmV or less at two of the plants, but over 10,000 ppmV at another. Such a marked difference between conditions at various plants suggests that there may also be large variations with time at each plant. If this is true, it is possible that Cameron's samples were taken at a time when emissions from the scrubber vent happened to be unusually low, so that the results did not accurately reflect the annual average operation of the plant.

Thus, the observations made in this study confirm that cottonseed oil extraction plants generally have readily detectable fugitive emissions of hexane, especially in connection with the extractor equipment. These fugitive emissions may potentially account for a large fraction of the solvent lost in the manufacturing operations, but neither this study nor published previous work provides reliable estimates of the magnitude of losses due to leakage from process equipment.

Also indicative of substantial variation in conditions is the fact that readings observed near particular types of equipment were high in some cases, but in others, low. For example, referring again to Table 4-2, it is clear that concentrations exceeding the range of OVA were observed at viewing windows in most of the plants, but not at all plants and not at all windows in each plant. Such observations suggest that improved maintenance efforts might yield substantial benefits in controlling hexane leaks.

Besides the evidence of variable equipment leaks which might be reduced by improved maintenance, there is evidence that overall emission rates of hexane from these operations may be substantially greater in summer months than in winter, and that use of refrigerated water in cooling the mineral oil scrubbers might effect important savings of hexane. Monthly data on production and on hexane losses, provided by one firm, indicated that the loss rate between November 22 and May 22 was 0.82 gallons per ton crushed, while during the other half of the year the loss was 1.83 gallons per ton. Thus, despite a slightly smaller average operating rate in the summer, the hexane loss rate was more than twice as high as in the winter months. At another plant, operators observed hexane losses as low as 0.3 to 0.4 gallons per ton crushed after installing new heaters and coolers in the hexane recovery system and were hoping to reduce the annual loss rate to 0.75 from a 1980-81 season level of 1.03 gallons per ton.

Data for the Ranchers Cotton Oil plant in Fresno, cited by Cameron (1981), also indicate that the relative rate of solvent loss per day was substantially higher in the period, May to July 1980, than in the preceding six-month period.

The scrubber vent is undoubtedly the largest individual source of potential hexane losses in the typical plant. To put these losses in context we may note that a typical plant loses, on the average, perhaps 160 thousand gallons per year, or about 500 gallons per day. Thus the losses of more than 6 gallons per day crudely estimated for plant C, may appear negligible; but it should be remembered that "more than" 6 gallons per day might in fact be much more than 6 gallons per day. Again, the low concentrations observed at scrubber vents in plants A and D may not have been representative values for year-around operations at those plants. Uncertainties involved in the more quantitative study (Cameron, 1981) done at Ranchers Cotton Oil in Fresno were still quite large; for estimated losses from the scrubber vent, confidence limits were 18 to 74 gallons per day, apparently because of large variability in the air flow and in the concentrations of hexane in samples collected. Moreover, those estimates also referred to a single sampling period of a few hours; thus, even the best estimate for that period cannot automatically be assumed to be representative of a year of operations.

Operation of these plants in hot weather may be expected to cause increased hexane losses not only because of less efficient recovery of solvent in the mineral-oil scrubbers, but also because of increased temperatures of the solvent used in the processes. Thus, the vapor pressure of hexane at average ambient temperatures for Fresno varied from 115 mm Hg in December 1979 to 175 mm Hg in July 1980 (Cameron, 1981). Higher vapor pressures may be expected to increase the absolute pressure inside the process equipment, making the maintenance of a negative gauge pressure more difficult and increasing the concentration of vapor in any gas escaping from the system.

Since the studies reported herein were done in November and January, they do not represent summer-time conditions in these cottonseed oil plants, all of which may be expected to yield substantially higher levels of emissions during the months of July, August and September. The fragmentary information available suggests that emission in summer are at least 50 percent higher than year-around. The average hexane loss for all 5 plants on an annual basis is approximately 1.2 gallons per ton of cottonseed crushed, or 6.6 pounds per ton. A corresponding estimate for summer only would be, accordingly, 1.8 gallons (or 10 pounds) per ton.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

Cottonseed oil extraction plants operating in California typically process 300 to 600 tons of crushed cottonseed per day. They operate continuously for as much as 330 days in a year and may process up to 180,000 tons of seed in a year. Commercial hexane is used as a solvent in these operations, which result in substantial losses of hexane by evaporation to the atmosphere.

Although hexane is normally recycled in the standard process, fugitive losses are substantial. Managers at the five plants in California reported hexane losses ranging from 73,000 gallons to 245,000 gallons for the 1980-81 operating season. In relation to production, losses ranged from 0.75 to 1.4 gallons of hexane per ton of cottonseed crushed. The total of hexane lost in all these operations in that year was approximately 800,000 gallons or 2,200 tons.

In a published study it is reported that a relatively small fraction of the lost hexane, less than 0.15 gallons of hexane per ton of cottonseed crushed, could be accounted for in the principal process streams and waste streams when, at one of the large plants in California, these streams were systematically sampled and evaluated. These results imply that, at least under some conditions and probably in general, most of the hexane lost in these operations issues through uncontrolled leaks in the process equipment.

Observations made in the course of this study and the results of the analysis presented in Section 4.0 are consistent with the hypothesis that leaks from process equipment are responsible for a major fraction of the hexane lost in these operations. Hexane vapor concentrations were measured in ambient air on the grounds of each of the California cottonseed oil extraction facilities, to determine whether concentrations downwind from the plant would be detectably higher than those upwind. In general downwind concentrations were found to be higher than upwind concentrations (refer to Table 4.1).

When concentrations were measured at points on the perimeters of the facility grounds, the average difference was 0.22 parts per million (of hexane). When, additionally, concentrations were measured at many points within the perimeters, the average difference was found to be 0.47 ppm; and when a comparison was made between points more directly upwind and downwind from the extraction facilities, the average difference was 0.74 ppm.. (These average differences are all in the direction of higher concentrations downwind.)

Hexane concentrations observed in sampling the ambient air ranged from 0 to 7.5 ppm, with only a few observations exceeding 2 ppm. Calculations for a hypothetical example of a cottonseed oil extraction plant, discussed in Section 4.2.3, indicate that the order of magnitude of these observed concentrations is compatible with reasonable assumptions as to wind flow and source strength on the occasions of the plant visits.

Hexane concentrations measured in the immediate vicinity of various items of process equipment demonstrated the existence of vapor leaks in a number of places -- most frequently near solvent extraction equipment, especially access doors, Rotocel plates, viewing windows and viewlight flanges. Typically the extraction unit contains multiple doors, windows, flanges and gasketed joints, some of which appeared to be allowing vapor leakage. Other leaks were detected at miscella processing and miscella refining units and near stearine centrifuges, as well as high concentrations near sumps and floor drains. In an appreciable proportion of the observations, the concentration of hexane exceeded the range of the instrument and may have been from 10,000 to 200,000 ppm. These observations are summarized in Table 4-2.

Further calculations for the hypothetical cottonseed oil plant indicated that hexane losses of the order of magnitude of those experienced by the plants surveyed would be plausible if the average total leakage area in the system was as large as 0.033 square meters (about 50 square inches), equivalent to a length of about 60 feet with a gap of 1/16 inch.

Thus, the observations made in this study confirm that cottonseed oil extraction plants generally have readily detectable fugitive emissions of hexane, especially in connection with the extractor equipment. These fugitive emissions may account for a large fraction of the solvent lost in the manufacturing operations, but no reliable estimates of losses due to equipment leakage can be made at this time. It is probable that the configuration and magnitude of vapor leaks continually changes as the plant and its various units are started up, shut down, opened, closed, and generally subjected to use and maintenance procedures.

Evidence reported by the manufacturers suggests, also, that the rate of loss of hexane is greater in the summer than in the winter. In one case where data were furnished, the rate of replacement of hexane inventory was more than twice as great (relative to production) in the six-month period from May to November than in the other half of the year. Two factors are probably responsible for this hot-weather effect: reduced efficiency of the mineral-oil scrubber used to control emissions from the main vents in the solvent recovery units, and increased vapor pressure of the solvent throughout the system. These effects are both natural consequences of the high ambient air temperatures in the Central Valley of California in summer.

It was reported, by the management of one of the plants surveyed, that hexane losses were markedly reduced after new heaters and coolers were installed in the hexane recovery system, increasing the efficiency of separation. It has also been reported in the literature that hexane concentrations at the scrubber vent of one plant were different by 60 percent between November 1979 and June 1980. Such observations suggest that use of refrigerated water in cooling the mineral oil scrubbers might effect important savings of hexane.

In summary, the findings reported here lead to the following conclusions:

1. Hexane losses in cottonseed oil manufacturing at five California plants range from 73,000 gallons to 245,000 gallons per year, totaling about 800,000 gallons for the 1980-81 operating season.
2. In relation to production, hexane losses ranged from 0.75 to 1.4 gallons per ton of cottonseed crushed.
3. A large fraction of the hexane lost is lost to the ambient atmosphere, with smaller amounts entrained in process products and waste water.
4. A substantial fraction of the vapor (but probably less than half) is lost through the vents of scrubbers used to control emissions.
5. The major part of the vapor lost to the ambient atmosphere constitutes fugitive emissions due to leaks in process equipment.
6. The most frequently detected leaks were in solvent extraction equipment, miscella refining and miscella processing equipment, and stearine centrifuges. In the main they were associated with doors, windows flanges, and other gasketed joints.
7. Vapor losses are substantially greater in summer months than in winter.
8. Hexane concentrations in the ambient air around cottonseed oil facilities are demonstrably greater in downwind locations than in upwind locations, although the magnitude of the difference is marginal at the peripheries of most of these plants.

5.2 RECOMMENDATIONS

1. Any further efforts to estimate emissions or emission factors for hexane from cottonseed oil extraction should be directed toward assessing the variability of these emissions as well as their magnitude. There should be a systematic program at a cooperating facility, with provision for monitoring and quantitating leaks from process equipment, emissions from process vents, and rates of evaporation from storage vessels, sumps and waste receptacles. These sources should be evaluated at regular intervals during day and night operations, and the plant-wide evaluation should be repeated at least three times during an annual operating cycle. One such plant-wide evaluation should be done during the hottest part of the operating year. Methods of assessing these emissions are reviewed in Appendix B.

2. Pending more accurate evaluation of the factors which affect hexane losses, estimates of emissions should be based on an overall emission factor of 6.6 pounds of hexane per ton of cottonseed crushed. (This is equivalent to 1.2 gallons of hexane per ton of cottonseed crushed, or 3.0 kg. per megagram.) Unless a particular facility has undertaken innovative measures to reduce emissions, this factor should be accurate to within 20 percent for annual emissions.

3. Estimates of daily emissions for summertime operations should be based on a factor of approximately 10 pounds of hexane per ton of cottonseed crushed, although this value is substantially less reliable than the overall emission factor cited in the previous recommendation. Implementation of tested control measures at any operating facility should, of course, be taken into account when such estimates are made.

4. In the expectation of reducing hexane losses, operators of cottonseed oil extraction facilities should institute programs of routine monitoring and maintenance, to detect and correct substantial leaks from equipment in hexane service.

5. Operators of cottonseed oil extraction facilities should consider installing systems to improve the efficiency of hexane recovery by using refrigerated water for cooling mineral oil used in the scrubbers, and possibly for cooling liquid hexane or condensing vapors at other points in the extraction process.

6.0 BIBLIOGRAPHY

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APPENDIX A

ORGANIC VAPOR ANALYZER

1.0 INSTRUMENT DESCRIPTION

Solvent vapor concentrations were measured with a Century Systems Corporation Portable Organic Vapor Analyzer (OVA), Model 108 (see Figure 3-1). The OVA-108 is a hydrogen flame ionization detector (FID). The FID operates under the principle that a diffusion flame of pure hydrogen in air is essentially free of ions and is therefore nonconducting. However, when a small amount of sample gas containing hydrocarbons is premixed with the hydrogen, the concentration of ions increases greatly and the flame becomes conductive. When an electric potential of several hundred volts is established between the collector plates a current is detected that is proportional to the hydrocarbon concentration in the sample gas. The induced current is measured with a logarithmic electrometer preamplifier which has an output signal proportional to the log of the input or ionization current. A signal conditioning amplifier is used to amplify the signal from the preamplifier and to condition it for subsequent meter display.

Specific characteristics of the model OVA-108 are as follows:

Sensitivity - 1 ppmV (as methane; 0.2 ppmV as hexane)

Response Time - Less than 2 seconds

Readout - 250 degree logarithmic scaled meter in the 1 to 10,000 ppmV range

Fuel Supply - 75 cubic centimeter tank of hydrogen gas (less than 0.5 ppm methane) at a maximum pressure of 15.8 MPa (gauge)

Primary Electrical Power - Rechargeable and replaceable battery pack at 12 volts, direct current

Unbilical Cord - Cable between readout and sidepack with connectors for electrical cable and sample hose

Filtering - In-line disposable and permanent particle filters.

2.0 CALIBRATION

The Organic Vapor Analyzer (OVA) was calibrated to four different reference concentrations of hexane (balance ultra zero air) prior to each of the three weeks devoted to plant visits.

Calibration procedures conformed to the instructions described by the instrument manufacturer's manual ["Operating and Service Manual for Century Systems' Portable Organic Vapor Analyzer - Model OVA-108"]. Calibration data were recorded in a laboratory logbook. A summary of typical calibration data is as follows:

Hexane Concentration (ppm)		
Actual	Observed	Percent Error
53	55	3.8
318	320	0.63
753	750	0.40
6,000	6,000	neg.

Calibration Gas: The calibration gas, hexane in "ultra zero" air, was stored in metal cyclinders at the following concentrations - 53, 318, 753 and 6,000 ppm. These gases were certified by the manufacturer (Matheson) to contain hexane at recorded concentrations with a possible error of 2 percent or less.

Calibration Procedures: OVA calibration consists of three instrument adjustments - (1) electronic (or gain); (2) primary, and (3) gas selection. Electronic (or gain) calibration ensures that the "gain" of the system is properly aligned. This adjustment proceeds with the instrument turned on and all other controls and valves off or shut. A switch (designated CALIBRATE) is placed in the HIGH position. The meter should indicate a readout of 10,000 ppm. In the LOW position the OVA meter should indicate 10 ppm. If these readings cannot be obtained then adjustments internal to the instrument must be performed.

In all field use in this study, proper HIGH and LOW meter deflections were attained. Consequently, there was no need of internal instrument adjustment).

Primary calibration of the instrument utilizes the hexane mixtures. After the instrument is operating, a hexane sample is drawn into the instrument. A knob (designated CALIBRATE) is used to shift the readout meter indicator to correspond to the concentration of the calibration gas mixture. After a reference setting has been reached the hydrogen supply is cut off (eliminating background signal) and CALIBRATE is switched to the HIGH position. A GAS SELECT control knob is then used to align the meter indicator to full scale. The setting is then recorded. The purpose of the GAS SELECT is to establish resettable levels of internal calibration signals to correspond to the primary calibration.

The primary and gas selection adjustments were conducted for each of the four hexane concentrations. Reference hexane concentrations and corresponding GAS SELECT settings are as follows:

Observed Reference Concentration (ppm)	GAS SELECT Setting
55	516.5
320	490.5
750	486
6,000	190

APPENDIX B

SOURCE ASSESSMENT METHODS

APPENDIX B

SOURCE ASSESSMENT METHODS

Methods available for source testing to assess hexane emission rates are of two types. One method, an official EPA test method, is appropriate for determining rates of emissions from stacks and accessible vents. The other methods are directed toward the detection and quantification of fugitive emissions, especially those caused by leaks in equipment.

EPA Method 25, Determination of Total Gaseous Nonmethane Organic Emissions as Carbon,(1) describes EPA's officially approved method for sampling organic vapors in stack emissions. A sample is withdrawn from the stack at a constant rate through a chilled condensate trap by means of an evacuated sample tank. Organics are determined by combining the analytical results obtained from independent analysis of the condensate trap and sample tank fractions. Contents of the condensate trap are oxidized to carbon dioxide, of which a portion is reduced to methane and measured with a flame ionization detector (FID). The sample tank fraction is further fractionated by a gas chromatographic column, then the nonmethane organics are oxidized to carbon dioxide, reduced to methane, and measured by FID.

In test Method 25, several important components are not specified, but an Addendum (Addendum I) is provided which provides users with information regarding components which have been found satisfactory for use with the method. Diagrams and descriptions of these components are provided, but alternative components are said to be acceptable. The method is written in this manner to permit individual preference in choosing components and to encourage development and use of improved components. EPA has also circulated draft Methods 25a and 25b, in which the former provides for use of an FID system for measuring the organics, while the second depends on a non-dispersive infrared (NDIR) system.

For measuring fugitive emissions of organics (such as hexane), EPA has circulated a draft of a proposed Method 21, Determination of VOC Leaks. This method is essentially the same as the method used by Pacific Environmental Services in surveying cottonseed oil extraction facilities in California. It consists in the use of an organic vapor analyzer (OVA), with a defined probing procedure, to detect leaks, which are considered to require correction when they cause readings above the range of the instrument (10,000 ppm). For such leaks, it does not give a quantitative measure of the leak rate.

In a study at a cottonseed oil extraction plant,(2) Cameron attempted to use a tracer-gas method, with carbon monoxide as the tracer, to determine the rate of flow of vapors induced by the conveyor carrying cottonseed meal from the driers to storage. Although the application was unsuccessful, the principle of injection and tracking of a tracer gas is one that could likely be useful in more carefully designed studies of these fugitive emissions. However, even if a tracer gas were to be used, quantitation would still depend on determining both the concentration and the flow rate of leaking gas.

Cameron also reviewed the types of sampling systems reported to have been applied to measurement of fugitive vapor losses at refineries and petrochemical plants. The applications all involved construction of "tents" of plastic film, sealed around leak sources to form an enclosure from which the leaking vapor can be sampled. Cameron concluded that the most appropriate sampling system for the types of leaks encountered at the cottonseed oil facility would be one utilizing a dilution system, in which hexane-free air would be fed at a constant rate into the enclosure and the concentration of hexane would be measured in air leaving the enclosure. The principles and procedures exemplified by this method are straightforward, although on occasion substantial ingenuity may be required in order to fabricate an enclosure which will adequately contain the leak.

Other procedures reviewed by Cameron do not appear to be applicable to ordinary equipment leaks which would often be found in cottonseed oil extraction facilities, because they depend on the existence of a rather high pressure inside the equipment, whereas the equipment used in hexane service at these facilities is normally at or near atmospheric pressure. These methods involve (a) the inflation of a flexible bag and (b) the use of a suction system. Both methods are unreliable under the conditions encountered in the subject facilities.

To summarize, the most practical methods for source assessment for hexane emissions in the cottonseed oil industry are: EPA Method 25 for emissions from vents, such as the main vents from mineral oil scrubbers; and a flexible tent method for leaks and fugitive emissions, using a dilution system (as described by Cameron; see Figure B-1) and flame ionization detector to measure steady state concentrations. Alternatively, a tracer study using some gas foreign to the process, such as carbon monoxide, hydrogen, or sulfur hexafluoride, can be designed.

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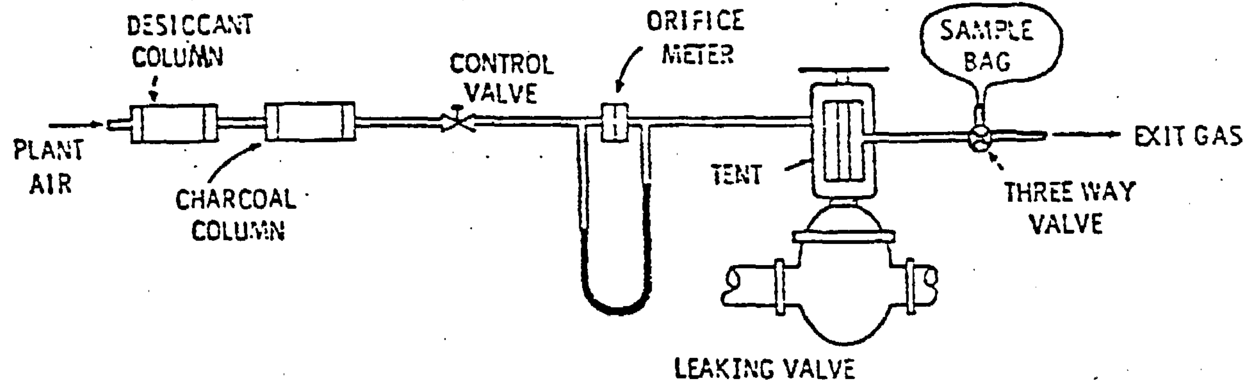

Figure B-1. Sampling Train for Measurement of Solvent Leak Rates, Using Plant Air for Dilution (Source: T.W. Hughes, 1979)