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Office of
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Washington, D.C. 20460

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April 1990

Toxic Substances

EPA

TEXTILE DYE WEIGHING MONITORING STUDY

Supplement

EPA 560/5-90-010
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TEXTILE DYE WEIGHING MONITORING STUDY

SUPPLEMENT

Exposure Evaluation Division
Economics and Technology Division
Office of Toxic Substances
U.S. Environmental Protection Agency
401 M Street, S.W.
Washington, D.C. 20460

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16. Abstract (Limit: 200 words)

Dye weighers in textile dyeing and printing plants are involved in the weighing and transfer of relatively small quantities of numerous powder dyes and other chemicals. This results in a potential exposure to a diverse range of chemicals which exhibit a broad spectrum of toxicological properties. In order to gain more detailed information about workplace exposure to poweder dyes, a study has been conducted to measure concentrations of dyes in the workplace air, and to characterize worker activities and industrial hygiene practices. This study was unique in that both government (U.S. Environmental Protection Agency) and industry (American Textile Manufacturers Institute and Ecological and Toxicological Association of Dyestuffs Manufacturing Industry) collaborated on an impartial basis and the dyehouses studies participated on a strictly voluntary basis.

The study included a survey of 24 randomly selected textile dyeing or printing sites which used power dyes. At each site, one worker was observed for an 8-hour shift; personal monitoring and area sampling data were taken. Certified industrial hygienists recorded worker activities, duration of potential expsoure, personal and engineering controls in use, and quantities and frequency of use of each dye that was handled during the monitoring period. Bulk samples of each dyes were also taken. The particulates collected on the air monitoring filters were analyzed for commercial dye content using a spectrophotometric method developed for the study.

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SUPPLEMENTAL A

QUALITY ASSURANCE PROJECT PLANS

QUALITY ASSURANCE PROJECT PLANS

THE WASHINGTON CONSULTING GROUP

August 10, 1986

QUALITY ASSURANCE PROJECT PLAN, Part I

for the

TEXTILE DYE DRUG ROOM STUDY

by

Bradley Schultz

Washington Consulting Group
1625 Eye Street, N.W.
Washington, D.C. 20006

EPA Contract No. 68-02-4229

Work Assignment 5

EPA Task Manager: Margaret G. Conomos
EPA Project Officer: Philip Robinson

Design and Development Branch
Office of Toxic Substances
Exposure Evaluation Division (TS-798)
U.S. Environmental Protection Agency
401 M Street, S.W.
Washington, D.C. 20460

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Work Assignment 5

Approval for:
WASHINGTON CONSULTING GROUP

Bryan K Porter 09/02/86
Date

Bryan K. Porter
Quality Assurance Officer

Approval for:
ENVIRONMENTAL PROTECTION AGENCY

Margaret G. Conomos 10/10/86
Date

Margaret G. Conomos
Task Manager

Date

Philip Robinson
Project Officer

Eileen Reilly-Wiedow 10/31/86
Date

Eileen Reilly-Wiedow
Quality Assurance Officer

QUALITY ASSURANCE PROJECT PLAN

There are two parts to this quality assurance plan. The first part is the statistical design and analysis section. Following this is Part II on the chemical analysis and field collection procedures. An overview of the study may be found in the data quality objectives for this study.

Part I Contents

QUALITY ASSURANCE PROJECT PLAN

Quality assurance (QA) is an integral part of the textile dye study. The following describes the components of the QA program.

1. FRAME CONSTRUCTION

The list of textile plants using powder dyes (the frame) was first constructed by using the names provided by Davison's Blue Book of textile manufacturers. This was first cross-checked with the Standard Industrial Classification listings to identify plants within classifications which include textile dyeing and printing plants. Then this list was cross-checked with a list of plants known to discharge dye-containing effluent. This list was then supplemented by EPA personnel familiar with the textile dyeing industry. Finally, as a last check, textile dye industry representatives affiliated with the American Textile Manufacturers Institute (ATMI) and the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD) revised the list based on their intimate knowledge of the industry. Although as with virtually any frame, it is known that this list is not perfect, numerous cross-checkings and reviews were made to ensure the highest quality possible at a reasonable cost.

The final examination involved drawing a (simple) random sample of 240 plants from the list of 1390 plants on the list. This allowed EPA and industry representatives to carefully examine a list of manageable size to insure that no systematic omissions were being made. Furthermore, this allowed for a detailed examination of the eligibility of each plant in this rapidly changing industry (i.e., an eligible plant is one that uses powder dyes to color textiles).

2. FIRST PHASE QUESTIONNAIRE

Although in-plant monitoring of dye exposure levels is the most important aspect of the study, the first phase questionnaire accomplishes a number of QA tasks, for the monitoring study:

1. allows a final check on the frame. If the 240 plants (randomly) selected were to have deviated in some systematic manner from the known make-up of the dye industry then the frame's accuracy would have come into question. The random sample of 240 provides a manageable list to carefully examine.

2. enhances knowledge of the dye industry for preparation of in-plant monitoring

3. allows the possibility of stratification of the sample for in-plant monitoring (the entire frame of 1390 is too large to accurately classify with reasonable cost).

For the first phase questionnaire itself, responses are being hand checked for consistency between answers by EPA Chemical Engineering Branch personnel familiar with the dye industry. Results of the questionnaire are being double-entered to keep data entry errors to a very low level.

3. RESPONSE RATE FOR SECOND PHASE

One of the most critical components of a sound study is a high response rate, since each refusal adds an uncontrolled bias to the results. Given the potential for such problems in a voluntary survey, we should take steps to increase the acceptance rate of in-plant monitoring before the first contact is made.

In conformance with government standards and good scientific practice, we should aim for a target of 75-80% participation. Due to the inherent difficulties in this particular study and the poor quality of the available data, a response rate of 60% of the 171 eligible plants is a reasonable lower limit on the participation rate, since the necessary caveats attached to such results should still not negate the worthwhile contribution of the study. But given potential refusals, we need a strategy in place to encourage participation in every way possible.

In preparation for actual methods to bolster the response rate, possible benefits and disadvantages of the in-plant monitoring from the plant management's perspective were considered.

Since there are only 30 plants for the on-site monitoring, the plan is to contact plants in an individualized manner. However, the sequence of contacts (extensive follow-up will be made with plants that initially refuse to participate) for most plants will be:

1. a telephone contact to a high level company official asking for permission to monitor dye concentrations at the plant. If permission is granted, a carefully crafted letter on ATMI letterhead will be sent explaining the study, confirming the telephone agreement, and describing the potential benefits (in an industry-wide sense, and with respect to free plant monitoring and a written report on the site visit).

Based on the type of refusal met, the following contacts will be made in an order appropriate for the particular plant contacted:

2. a letter from another appropriate trade group (e.g., Carpet and Rug Institute, or hosiery group, if appropriate)

3. a letter from Bill Dyson (Health & Hygiene) describing procedure

4. a personal contact to the Chief Executive Officer of the company, from a Chief Executive Officer of another firm favorable to the study

5. a letter from a plant that Bill Dyson has already monitored stating that the monitoring team "was practically invisible"

6. a letter from EPA assuring the reluctant plant in writing that enforcement action will not be taken based on the site visit

These letters will be carefully crafted to maximize the likelihood of acceptance of monitoring.

If a response rate of at least 60% is not achieved, the study will be reevaluated at that time. (But this worst case scenario is not anticipated.)

The refusing plants will be characterized to determine if there is any detectable pattern in those refusing plants versus the cooperative ones.

For cooperating plants, the on-site arrangements will be made by Bill Dyson.

4. SELECTION OF PLANTS

Of the 1390 plants in the U.S. with the potential for dyeing textiles 240 were chosen by simple random sampling and mailed questionnaires; the result was:

- 69 were determined to be ineligible (i.e., the plants were out of business, or for some other reason did not really use powder dyes to dye or print textiles, with mechanical equipment);
- 81 had responded by the cutoff date, February 18, 1986;
- 90 did not respond to the mailed-out questionnaire, but were determined to eligible by EPA (CEB), ATMI, or ETAD (determination coordinated by CEB's George Heath).

Several criteria were considered for stratification, but the one arrived at was to group the 171 eligible plants by whether or not the plant responded to the mailed-out questionnaire. Although whether a plant responded to the mailed-out questionnaire was not considered to be of direct inherent value as a dividing

characteristic between types of drug rooms, examination of the responses to the mailed-out questionnaire suggested that it serves as a proxy for carpet plants, since it was conjectured that a higher percentage of non-respondents were carpet plants than the percentage among respondents. Also the act of responding to the questionnaire is seen to separate into two groups based on the likelihood of allowing in-plant monitoring (and thus reducing somewhat the refusal bias by replacing in-plant refusals with another plant in the same strata).

A completely random sample of 14 respondent eligibles will be drawn from the list of 81 respondent eligibles and 16 from the list of 90 (for a total of 30 plants for in-plant monitoring) in order to represent plants from each strata with the number of plants proportional to the total number of plants in each strata. If any refuse to allow dye level monitoring, they will be replaced by a plant from the same list that the refusing plant was on.

5. SELECTION OF WORKER WITHIN PLANT

One weigher from each plant will be selected. First, the shift of the worker will be selected, based upon the facility's (or plant manager's) seven digit telephone number (obtained from the first phase questionnaire) as follows:

No of shifts operated	Middle 3 digits of telephone number	Shift to be observed
1	000-999	1st
2	000-499	1st
	500-999	2nd
3	000-333	1st
	334-666	2nd
	667-999	3rd

This will ensure a random selection of shifts for observation and will provide a mechanism for knowing the shift prior to the visit. Although not exact, it will allow for a nearly random selection of worker within plant.

The employee to be monitored will be selected within the chosen shift as follows:

1. collect last three digits of social security number (SSN) for all weighers from all dye areas
2. select worker with last three SSN digits closest to 500

6. PILOT PLANT MONITORING

Methods for in-plant monitoring, recording observations, and making the chemical analysis, will be pre-tested at a pilot plant (i.e., an actual plant with textile dyeing operations).

Of particular note are the testing of:

- the ability of the two member team to collect all of the information, particularly the number of weighings and the mass of dye measured
- the chemical analysis method

7. QA VISITS

Three QA visits will be made during the on-site visits, one at the beginning, one near the middle, and one near the end of the site visits. A report will be made on the quality of the data collected based on information collected as shown in Appendix B. (At the beginning of the study, corrections may be made to procedures.) This will also allow the task team to become intimately acquainted with the usefulness and limitations of the collected data. Two site visits will be conducted by the Design and Development Branch of OTS (one of these by their contractor, The Washington Consulting Group (WCG)) and one by the Field Studies Branch of OTS. Besides enhancing understanding, the site visits will check the procedure for selecting the shift and weigher chosen (and thus also the drug room chosen), the determination of the number of weighers, and all other data collected at the site -- but with particular attention paid to those items which are recorded in prespecified categories. Part II, sections 7 and 10 discuss chemical audits in detail.

8. VERIFYING IN-PLANT RECORDING FORMS

In-plant data collection forms will be compared by the two on-site industrial hygienists for accuracy of the information.

9. DATA PROCESSING AND ENTRY

ATMI mailed out the first phase questionnaire. After return to ATMI the coded, unidentified questionnaires were forwarded to EPA and then to WCG. All questionnaire responses were computerized by WCG and double-entered to reduce keypunch errors to a minimum. George Heath, of EPA's Chemical Engineering Branch, who is familiar with the dyeing industry and its operations, may provide a list of obviously incorrect questionnaire answers where there was a misunderstanding or other error on the part of the respondent (if corrections appropriate). The list of changes would be provided to the study partners (EPA and industry) along with a brief explanation, and this list and explanation would be provided with the summary table results. At the end of the study, all response forms will be returned to ATMI and destroyed.

Most of the second-phase data handling will be done in the field and at the laboratory (discussed in part II of QA plan). After the site visit and the verification of collected information, the data collection protocol will be split into two parts: the dye monitoring cartridges and accompanying information, and the observational results. The observational results will be forwarded to Margaret Conomos of the Design and Development Branch, and George Heath.

Margaret Conomos will then forward the entire data collection record to Bradley Schultz of WCG, to conduct the statistical analysis. WCG will then enter the following information, for each of the 30 plants, into a computer data base for statistical analysis, and presentation of summary tables:

1. 2-digit plant identification number
2. date (month, day, year)
3. shift (categorized as closest to 1st shift (7am-3pm), 2nd(3pm-11pm), or 3rd(11pm-7am))
4. total number of weighers working at plant (all work areas, all shifts)
5. number of printing machines running during observed shift (average of beginning and end of shift)
6. number of continuous/semi-continuous dyeing machines running during observed shift (average)
7. number of batch dyeing machines running during observed shift (average)
8. number of weighers working in observed drug room shift (note difference with item 5)
9. number of other workers working in observed drug room shift
10. age of worker at time of site visit (number of years)
11. life time work experience of monitored worker handling powder dyes (number of years)
12. experience at observed facility as powder dye handler (number of years)
13. whether or not weigher wore dust mask during shift (yes/no)
14. whether or not weigher ate in drug room during shift (yes/no)
15. whether or not weigher smoked in drug room during shift (yes/no)
16. total amount of all dyes weighed by worker observed during shift (nearest pound)
17. number of dyes weighed by worker observed during shift
18. number of hours weigher monitored was in drug room (nearest tenth of hour)
19. dye level measurement (mg/m^3), time-weighted 8-hour average from personal monitor
20. dye level measurement (mg/m^3), time weighted 8-hour average from area sampler
21. number of hours personal monitor running (nearest tenth of hour)
22. gravimetric weight of all dust on personal monitor cartridge
23. gravimetric weight of all dust on area sampler cartridge

All data will be double-entered to keep keypunch errors to a minimum. Bradley Schultz will examine summaries of the data to look for gross errors from what might be expected. Suspicious results will be reported to EPA's Margaret Conomos.

Any computer programs written by WCG (i.e., Douglas Marder) to fulfill the needs of the project will be checked for accuracy by Bradley Schultz. Reputable commercial computer packages will be assumed accurate; however, Doug Marder of WCG will briefly examine output as a final check. The choice of software used, appropriateness of the statistical analysis, and use of tables will be with the approval of both Bradley Schultz and David Cox.

10. DATA ANALYSIS

There will be two major components to the data analysis:

1. characterizing the distribution of dye levels in plants and among weighers
2. examining the correlation of dye level with other factors

Other items of interest are discussed in section 10.4 - 10.5.

10.1 DISTRIBUTION OF DYE LEVELS IN PLANTS

At each of the 30 plants, the 8-hour time-weighted exposure (mg/m^3) will be taken from the weigher's personal monitor. From these 30 data values, the distribution of plant dye levels will be characterized.

In particular, the average plant exposure level will be calculated, along with its 95% confidence interval. Also, the 85th, 90th, and 95th percentiles will be calculated and 95% nonparametric tolerance limits will be obtained for the 85th and 90th percentiles. Although a nonparametric (distribution-free) approach for 95% tolerance limits is not possible due to the number of plants in the study, the use of probability distribution-based approaches (such as using the normal distribution) will be explored for obtaining a 95% confidence interval for the 95th percentile.

10.2 ESTIMATE OF AVERAGE WORKER EXPOSURE

Estimates of average worker exposure in the population of textile dye weighers and the 85th percentile of worker exposure (the dye weighers are the only workers directly covered in this study).

In contrast with the analysis of section 10.1, this estimate takes into account the number of weighers working at each plant and is weighted according to the number working at the monitored

plant during a typical 24 hour period. For example statements could be made about the 85th percentile of worker exposure by weighting plant estimates by the number of weighers in each plant. This would result in a statement such as "It is estimated that 85% of weighers are exposed to levels lower than xxx mg/m^3 during an 8-hour shift." Note that this differs from the estimate of 85th percentile of plant levels (objective 1) which results in a statement such as: "It is estimated that the average exposure in 85% of textile dyeing and printing plants is less than zzz mg/m^3, time-weighted 8-hour average" (xxx and zzz determined from study).

This estimate will use the total dye exposure (mg/m^3) obtained from each worker's personal monitor.

10.3 CORRELATION OF DYE LEVEL WITH OTHER FACTORS

10.3A Primary focus

The exposure level (mg/m^3) will be examined for correlation with:

1. mass of weighing during shift by worker of interest
2. number of weighings during shift by worker of interest
3. combination of the two above factors.

If such a correlation exists, a functional relationship will be explored between the dye level and other factors.

10.3B Secondary measurements to examine association with dye level

The rate of exposure (mg/m^3/hr) will also be examined for correlation with several other variables of secondary importance. These are:

1. Production volume of textiles (pounds per year), from mailed-out questionnaire
2. Management of dye house (vertical, commission or both), from mailed-out questionnaire
3. Management of dye house (public or private), from mailed-out questionnaire
4. Color index class of dyes used, for any dye used during observed shift by monitored weigher (acid, basic/cationic, reactive, direct, disperse, other), from site visit log as classified by Chemical Engineering Branch
5. Total number of dyeing and printing machines serviced by monitored weigher (average of beginning and end of shift numbers), from site visit questionnaire
6. Fiber type dyed or printed (acrylic/modacrylic, rayon/cotton, nylon, polyester, other), from mailed-out questionnaire

It should be noted that a data set of only 30 observations is likely to result in some spurious large correlation when many correlations are calculated. Thus the correlations of this section (10.2.B) will be interpreted in that light.

10.4 AMOUNT OF DYE WEIGHED OUT

The average, and distribution of, amount of individual dye compund weighed out during a shift will be calculated and displayed (averages and histograms will be presented by dye class and aggregated).

10.5 SUMMARY TABLES

10.5.1. On-site questionnaire

Summary tables will be presented for several other variables collected from the on-site questionnaire with categorized responses. These will include averages or proportions as appropriate. The variables to be tabulated are:

1. Number of textile dyeing plants in each EPA geographical region
2. Number of plants by number of weighers, at all shifts during a typical 24 hour period
3. Number of plants by pounds of dye weighed during shift
4. Number of plants by number of dyes weighed during shift
5. Number of plants by number of dye weighings during shift
6. Number of workers by amount of time in drug room
7. Number of workers that used dust mask during site visit
8. Number of workers that used respirator during site visit
9. Number of workers that smoked in drug room area during site visit
10. Number of workers that ate in drug room area during site visit

10.5.2 Mailed-out questionnaire

As an appendix in the final report, the following variables will be tabulated from the first-phase, mailed-out questionnaire:

*11. Number of textile dyeing plants in each EPA geographical region
12. Number of plants by number of dyeing or printing operations within the company that owns the selected plant
@13. Number of plants by management of house (vertical, commission, or both)
@14. Number of plants by management of house (public, or private)
@15. Distribution of plants by product volume
16. Number of plants by product line (carpet, yarn, fabric, other)
#17. Number of plants by type of dyeing or printing equipment available (batch, semi-continuous/continuous, printing)
@18. Number of plants by fiber dyed or printed (acrylic/modacrylic, rayon/cotton, nylon, polyester, other)
@19. Number of plants by color index class of powder dye (acid, basic/cationic, reactive, direct, disperse, other)
20. Number of plants by number of dyes weighed per 24 hours (less than 10, 10 to 20, over 20)
&21. Number of plants by pounds of dye used per 24 hours (less than 50, 50 to 200, over 200)
&22. Number of plants by number of powder dye weighings per 24 hours (less than 50, 50 to 500, over 500)
23. Number of plants by number of dye weighing rooms (1 room, 2 or more rooms)
24. Number of plants by number of worker shifts per 24 hours (1, 2, 3)
25. Number of plants by number of operating days per week (1 to 4, 5, 6 or 7)
26. Number of plants by number of employees exposed to powder dyes (1, 2, 3, 4 or more)

Notes

* This is also tabulated from site visit data
\# Similar information is also collected in the on-site questionnaire and is used in the secondary correlation analysis (see Appendix A)
@ A portion of the data on this variable will be used in the secondary correlation analysis (see Appendix A)
& Similar information also collected in the on-site questionnaire and is used in the primary correlation analysis

10.6 AREA SAMPLER RESULTS

Although the area sampler measurements will be chemically analyzed for each plant, the results will be used solely for post-study exploratory work and possibly for quality assurance purposes (if a strong correlation is found between personal samplers and area sampler results). The area samplers may provide a useful quality assurance role at the data analysis phase. If the personal monitor result is suspect at one plant for some reason, the area sampler measurement provides a rough cross-check for such a suspect value. As in any study, it is hoped that no such incidents will take place.

APPENDIX: PROJECT PERSONNEL

Bradley Schultz is the WCG work assignment leader. He will be the statistician involved in the design and analysis of the study. David Cox is the overall Project Director for the WCG contract with EPA. Doug Marder and Keith Johnston will set up and oversee the creation and use of the data bases. Terri Stiteler will manage the data bases and coordinate the data entry. Credentials are on file with the Design and Development Branch of EPA.

APPENDIX B

TEXTILE DYE/DRUG ROOM EXPOSURE STUDY

QUALITY ASSURANCE AUDIT FOR A PLANT VISIT

Section I. Basic Audit Information

A. Auditor Information

1. Name(s)/Affiliation: _____

_____

_____

_____

2. Date of Audit: _____ ___, 19__

B. Textile Plant Information

Plant I.D._____

C. Industrial Hygienists:

1. Health and Hygiene, Inc. _____

2. PEI _____

Section II. Sampling Design

1. Was the selection of the shift and/or the weigher done on a random basis? Yes___ No___

Section III Air Monitoring Methods

1. Were all air sampling instruments calibrated accurately prior to field use? (Note means of verification). Yes___ No___

---

2. Were the personal samplers operating at a flow rate between 2 and 2.5 liters/minute? Yes___ No___

---

3. Did anything occur that might interfere with the airflow on the personal sampler (i.e tubing became twisted)? Yes___ No___

---

4. Were the area samplers operating at a flow rate between 5 and 8 liters/minute? Yes___ No___

---

5. Was the location of the area samplers appropriate? Yes___ No___

---

6. Were filter blanks taken into the field? Yes___ No___

---

7. Did splashing of liquids occur onto the filter? Yes___ No___

Section IV Drug Room Observations

1. Was each container of bulk dye taken labelled and appropriately identified on the corresponding form?
   Yes___ No___

---

2. Were appropriate methods used to collect bulk dye samples, i.e. non-obtrusive and non-dust-generating? Yes___ No___

---

3. Were all entries and exits into and out of the drug room by the weighes recorded? Yes___ No___

---

4. Were all weighings recorded with name of the dye noted and corresponding to the name of the bulk dye sampled?
   Yes___ No___

---

5. Was a validation of all dyes recorded conducted between the two industrial hygienists? Yes___ No___

Section V Qualitative Performance of the Field Visit

1. Was a positive rapport evident between management and the visiting industrial hygienists? Yes___ No___

2. Was management well-informed about the objectives of the study and fully co-operative? Yes___ No___

3. Was the weigher who was wearing the sampling device informed about the objectives of the study and fully cooperative? Yes___ No___

4. Were the weigher's work activities altered or interrupted by the visiting industrial hygienists? Yes___ No___

QUALITY ASSURANCE PROJECT PLANS

PEI ASSOCIATES

SECTION 1.0

EPA/ETAD/ATMI

DYE EXPOSURE STUDY

QUALITY ASSURANCE PROGRAM
FOR
FIELD SAMPLE AND DATA COLLECTION

SECTION 2.0

TABLE OF CONTENT

SECTION 3.0

PROJECT DESCRIPTION

The American Textile Manufacturers Institute (ATMI), the U.S. Operating Committee of the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD), and the U.S. Environmental Protection Agency (EPA) are jointly sponsoring a study to assess the potential exposure of dye weighers in textile drug rooms to airborne dye dust. Data from this study will be used to estimate worker exposure as new dyes are proposed for introduction into commerce and in addressing concerns on existing products.

The study is divided into three parts. The first is the selection of textile facilities to be visited. This will be done on a random basis from the total universe of textile dyeing and printing facilities in the U.S. by the Washington Research Group under contract to the EPA with assistance from ETAD and ATMI. The second is the collection of data and samples at the 30 facilities selected. This will be done by Health & Hygiene, Inc. as a contractor to ETAD and ATMI, and is the subject of this quality assurance plan. The third is the analyses of samples collected at the facilities to determine the dye content of the airborne dust in the drug rooms. This will be done by Midwest Research Institute as a contractor to the EPA and is the subject of a separate quality assurance document.

The field sample and data collection part of this study is comprised of the following activities:

3.1 Preliminary Arrangements

Agreement by a selected facility to participate in the study will be obtained by ATMI with assistance from ETAD and others. Once consent has been given, telephone contact will be made by Health & Hygiene to schedule a visit. Preliminary information necessary to prepare for the visit such as the number of shifts of drug room operation and the approximate number of dyes weighed per shift, will be obtained during this contact (Attachment 3-1). Other activities including obtaining necessary supplies, calibrating air sampling pumps, and preweighing sample filters and blanks will be done prior to the visit.

3.2 Field Visits

Visits to the selected facilities will be made by a two person team of industrial hygienists, one each from Health & Hygiene, Inc. and PEI Associates, Inc. The team will arrive at the facility at least four hours prior to the start of the shift selected for sampling/observation to obtain information about the facility, determine how to obtain dye weighing data most efficiently, and take a brief familiarization tour. During the selected shift, personal and area air samples will be taken; bulk samples of dyes weighed during the shift collected; temperature, barometric pressure, and relative humidity measurements made; the time the monitored employee spends in the drug room recorded; data on the number and quantity of dyes weighed obtained; and observations about conditions and controls in the drug room made. Upon completion of the sampling and observations a closing conference will be held with management.

3.3 Gravimetric Determinations

Total potential dust exposure of the employee during the monitoring period will be determined by reweighing the air sampling filters collected at each facility. After weighing, the filters and bulk dye samples will be sent to Midwest Research Institute (MRI) for analytical determination of the dye content on the filters. The gravimetric determination of total dust will provide an upper bound against which the nonspecific analysis by MRI can be compared.

ATTACHMENT 3-1

PRE-VISIT TELEPHONE CONTACT

Facility: _____

Address: _____

_____

_____

_____

Contact: _____

_____

Telephone No. _____

_____

Scheduled Visit Date: _____

Number Of Shifts Drug Room Operated: _____

Shift to be observed/sampled: _____

Comments: _____

_____

_____

_____

Approximate number dyes weighed/shift: _____

Approximate number dyes weighings/shift: _____

Local accommodations: _____

_____

_____

SECTION 4.0

FACILITIES, EQUIPMENT, CONSUMABLES, AND SERVICES

4.1 Facilities

Filter weighing and reweighing will be done at Burlington Industries' Industrial Hygiene Laboratory, Doyle Street, Greensboro, North Carolina. This laboratory has a constant temperature-humidity room in which the filters and blanks can be equilibrated prior to each weighing.

Sample pump calibrations will be performed in the industrial hygiene laboratory at Health & Hygiene, Inc.

4.2 Equipment

Equipment which will be used on this project includes:

- Mettler ME 30 microbalance, capable of weighing to the nearest microgram
- Gilian HFS 113 air sampling pumps with timers
- Gast Model 1531 vacuum pumps with critical orifices
- GCA/Precision Scientific wet test meter
- Buck Model M-5 mini-calibrator
- Bacharach Sling Psychrometer

4.2.1 Calibration

The Mettler ME 30 microbalance is checked prior to each set of weighings with an internal 100 mg weight. Zero checks are made periodically during the weighings to assure that drift is not occurring. If the balance cannot be calibrated or zeroed a Mettler service representative will be called.

Flow rates for the Gilian sampling pumps will be calibrated in the laboratory prior to field visits with an SKC 311-100 soap film calibrator. Prior to site visits while in the field the Buck mini-calibrator will be used. Further, the

rotameter setting will be noted during calibration and checked periodically during sampling. Should low flows be noted during sampling, a post-sampling flow rate will be determined with the mini-calibrator and an average value used.

Flow rates for the Gast vacuum pumps with critical orifices will be determined prior to field visits with the wet test meter.

4.2.2 Maintenance

Maintenance of equipment used in this project will be done according to the manufacturer's specifications. The Mettler microbalance has just been serviced and is on a yearly schedule. All other equipment is serviced as needed.

4.3 Consumables

Consumables supplies which will be used during this project include:

- Gelman Vinyl Metricel VM-1 filters with support pads
- Gelman 4339 3-piece cassettes
- UV Light Absorbing Plastic Bottles (for bulk samples)
- Plastic spoons
- Plastic bags

4.4 Services

Filter weighing will be done at Burlington Industries' Industrial Hygiene Laboratory. Services to be provided by this laboratory include:

- Equilibrate, preweigh, and place filters in numbered cassettes prior to field visits, including
- field blanks
- Equilibrate and reweigh filters and blanks after sampling

- Place samples in unused cassettes
- Record results in a permanent laboratory notebook
- Send samples, cassettes, field blanks, filter blanks and recorded results of weighings to MRI

SECTION 5.0

SAMPLE AND DATA GENERATION

5.1 Employee Selection

For each of the thirty (30) facilities visited, only one dye weigher will be selected for exposure monitoring. This will be done in a random fashion. the shift to be observed and sampled will be selected based on the middle three digits of the facility's (or facility contact's) seven digit telephone number as follows:

No. Shifts Operated	Middle 3 digits of telephone number	Shift to be observed/sampled
1	001-999	1st
2	001-500	1st
	501-999	2nd
3	001-333	1st
	334-666	2nd
	667-999	3rd

This will ensure a random selection of the shift for sampling and will provide a mechanism for knowing the shift prior to the visit. If contact with the facility indicates that the shift selected in the above fashion is unreasonable due to the dye weighing activities being conducted, then an alternative selection will be made based on professional judgment.

Where the facility has more than one dye weigher on the selected shift, the one selected for monitoring will be the dye weigher whose last three Social Security Number digits are closest to 500. Assistants and helpers will not be considered for inclusion. Selection of the employee will be done in the opening conference with management. The method of selection will be documented for both shift and weigher if different from normal procedure (page 2 of survey form.

5.2 Air Sample Collection

At each facility visited, a total of four air samples will be collected, two worn by the dye weigher selected for monitoring and two at stationary locations in the drug room. Air will be drawn through pre-weighed 37 mm polyvinyl chloride filters (Gelman VM-1) in three-piece cassettes. Personal samples will be taken using portable, battery operated pumps worn by the employee as he performs his work. Flow rates of approximately 2.0 L/min and a sampling duration of seven or more hours, including breaks, will be used. Stationary area samples will be obtained using electrical vacuum pumps operated at approximately 7.4 L/min for roughly the same duration as the personal samples. Sampling will be done in an "open-face" configuration. After collection, the samples will be recapped, returned to the laboratory, equilibrated, reweighed to the nearest microgram, and submitted to MRI for determination of dye content.

5.2.1. Sample Identification

As the filters are preweighed and placed into cassettes, a unique identifying number will be written on the bottom section of the cassette with an indelible marker. This will serve as the sample identifier on all documentation of the visit and subsequent analysis. When the filters are reweighed after collection and placed into new cassettes, the bottom section of the new cassettes will be identified by the same number as the sample plus a prime (') mark to distinguish it from the original cassette. Both will be submitted to MRI.

5.2.2 Duplicate Personal Samples

Both personal samples at a facility will be collected from the same dye weigher.

5.2.3. Air Flow Checks

The personal sampling pumps used have electronic feedback systems which maintain set flow rates even as dust on the filter increases resistance. These pumps also have rotameters. The setting of the rotameter will be noted during calibration and

observed two to three times during sampling to ensure that no change has occurred. If it appears that the air flow rate has changed, a post-sampling flow calibration check will be made using the mini-Buck calibrator. Should significant variation be found the average of the pre- and post-sampling calibrations will be used as the flow rate for dust level calculations.

Air flow rates for the two area samplers will be maintained with critical orifices. These are generally quite steady. A rotameter will be used to check air flow periodically during the sampling period. Should significant variation be found, a post-sampling calibration will be made and the average value used as the flow rate.

5.3 Data Collection

Information on the time the dye weigher being observed actually spends in the drug room, the number of dye weighings made, and the total quantity of each dye weighed during the sampling period will be obtained. Observations will be made of ventilation in the drug room, the use of personal protective equipment, work practices being used, and cleanliness of the area. In addition, limited demographic data will be obtained from the dye weigher being monitored/observed and temperature, barometric pressure, and relative humidity in the drug room will be recorded. The use of any particularly dusty dyes will be recorded.

5.3.1 Exposure Duration

Dye weighers are typically quite mobile. They move freely in and out of the drug room. Since it is assumed that the majority of exposure occurs while he is in the drug room, this parameter will be measured.

The boundary of the drug room will be established by agreement between the two site visitors. A rough sketch of the dye weighing area will be made indicating the boundary chosen. The actual time the dye weigher being observed spends in the drug room will be measured by recording the time, to

the nearest minute, that he enters and exits the area. The total potential exposure time will be the sum of the periods he spends in the drug room.

5.3.2 Weighings of Solid Materials

All weighings of solid materials made during the sampling period will be observed to determine three important parameters - the total number of individual weighings made, the total number of individual solids weighed, and the total mass or quantity of each solid weighed. Whenever possible, batch tickets used by the dye weigher will be obtained and the weighing data recorded from these. If batch tickets are not available, the data will be obtained by direct observation and, if necessary, questioning the dye weigher.

The method for obtaining these data will be discussed with management in the preliminary conference. Their suggestions as to the most efficient way to obtain the data in their facility will be considered and used where possible.

5.3.3 General Observations

During the sampling period, general observations will be recorded about the type of personal protective equipment used by the dye weigher, work practices used, the cleanliness of the drug room, and engineering controls such as ventilation used to reduce dust exposure. Whether or not the dye weigher smokes will also be recorded.

5.3.4 Dye Weigher Interview

During a break period, the dye weigher will be asked several questions to determine his age and how many years he has been handling dyes, both at the facility visited and elsewhere.

5.3.5 Temperature/Relative Humidity

Temperature, barometric pressure, and relative humidity will be measured intermittently during the sampling period.

5.4 Bulk Dye Samples

For each individual dye weighed during the sampling period, a bulk sample of approximately one ounce will be obtained. These samples will be taken from the original dye drums used by the weigher. Disposable plastic spoons will be used to avoid contamination.

The bulk dye sample bottles will be labeled sequentially using the labels provided by MRI. Corresponding labels (of the same number) will be placed on the data sheet where the full name of the dye, its lot number, if possible, and its supplier will be recorded along with the name used on the batch ticket or weighing record. Samples will also be taken of those solids wheighed by the weigher which may interfere with the dye analysis (e.g. colored materials or chemicals which may react with dyes).

5.5 Filter Weighings

Air sampling cassettes will be returned to Burlington Industries' Industrial Hygiene Laboratory. They will be equilibrated overnight in the constant temperature and humidity area prior to being reweighed to the nearest microgram. Ten field blanks - preweighed filters in cassettes through which no air has been drawn which have been handled and transported with the sample filters - will be equilibrated and weighed at the same time. The average weight change of these ten blanks will be used as the blank correction values in calculating dust exposure levels. Significant problems in filter weighing should be detected through the use of these blanks.

Note: The ten field blanks may apply to air samples from more than one facility if two or more are visited in the same week.

5.6 Shipping and Handling

After collection and reweighing, all materials will be shipped to MRI. This includes bulk samples, air sample filters, and data collection forms.

Bulk sample bottles will be placed in plastic bags in groups of four. These will be shipped to MRI separately from the air samples to avoid the potential for contamination. To minimize handling, bulk samples will be sent directly from Health & Hygiene.

Air samples will be hand carried to Burlington Industries' Industrial Hygiene Laboratory. After reweighing, they will be shipped directly to MRI. For each facility, these shipments will include the four air sample filters in new cassettes, four empty field cassettes, two field blanks, and ten unused filters from the same lot.

Data collection forms, minus the identifying cover sheet, will be hand delivered along with the air samples to Burlington's laboratory. After post-sampling weights are recorded, the forms will be sent along with the samples to MRI. The facility identification sheet will be mailed to ATMI from Health & Hygiene.

SECTION 6.0

DATA PROCESSING

6.1 Collection

All data will be collected manually. Recording will be done legibly in permanent ink on worksheets. Each person involved in recording data will sign and date the worksheet Corrections will be initialed.

6.2 Data Reduction

All sample manipulations will be clearly documented. Standard data reduction techniques will be used.

6.3 Data Validation

The data validation process will include:

- Air flow rate checks
- Timing checks with second watch
- Checking calculations
- Comparisons with original batch tickets
- Reviews for internal consistency by site visitors
- Use of field blanks

The site visitor from Health & Hygiene will be responsible for assuring data validity.

6.4 Transfer

Original recording sheets will be included with field visit documentation to allow checking of data transfers at a later date.

SECTION 7.0

DATA QUALITY ASSESSMENT

7.1 Filter Weighings

The microbalance used for filter weighings is capable of ±1 microgram precision and, since net weight change is being measured, accuracy. However, zero drift is slightly greater than this, approximately ±3 micrograms. Zero will be reset after every third weighing.

Greater variability is caused by moisture collection on the filters. A constant temperature and relative humidity room is used to equilibrate the filters before weighings to reduce this variability. Ten blank filters through which no air has been drawn are weighed and reweighed with the sample filters. The average weight change of these blanks is used as a blank correction in calculating dust levels. This correction is generally less than ±30 micrograms.

7.2 Traceability of Samples and Data

All air and bulk samples will have unique identification numbers. All data collected on the samples will be related to these numbers. Simplified traceability logs will be completed and signed when samples are transferred to Burlington's laboratory and to MRI.

7.3 Completeness

The Health & Hygiene site visitor will review all data collected prior to leaving the facility to assure completeness. This will include a verification that the record of dyes weighed/sampled is consistent.

SECTION 8.0

CORRECTIVE ACTION

The Health & Hygiene site visitor has primary responsibility for taking corrective actions as necessary. Examples of problems which might be encountered and possible corrective actions are as follows:

- Personal sampling pump stops - Record time from built-in timer, submit sample, make second sample primary
- Flow rate variation during sample - average pre- and post-sampling rates
- Large blank filter weight variation - check balance, examine filters for loss of material or contamination from backup pad.
- Failure to obtain bulk dye sample - Contact facility for assistance in obtaining
- Incomplete dye weighing data - Obtain original batch tickets from facility if possible
- Water spray or other inadvertent contamination of air sampling filter - use second sample if not contaminated, conduct sampling again

SECTION 9.0

DOCUMENTATION AND REPORTING

9.1 Documentation

Field sample and data collection will be documented in permanent ink on the forms provided by the EPA. The data related to sample collection will be compiled by the Health & Hygiene site visitor and Burlington lab personnel. Other data will be completed by the PEI, Inc. site visitor. Any corrections will be marked through and initialed. Raw data on air flow calibrations and filter weighings for which no space is provided on the EPA forms will be recorded on standard forms from Health & Hygiene and accompany the documentation for a facility visit.

9.2 Transmittal to MRI

MRI is acting as the central repository for all data related to this study. Data collected prior to and during facility visits will be submitted to MRI along with the samples for that facility by both Health & Hygiene and PEI. The identification sheet for each facility will be sent separately to ATMI by Health & Hygiene.

9.3 Reports

Trip reports will be prepared by the PEI, Inc. site visitor for each facility visited. Upon completion of the study, a composite report of dye dust exposure in textile drug rooms from an industrial hygiene point of view will be prepared jointly by PEI, Inc. and Health & Hygiene, Inc.

SECTION 10.0

PERSONNEL AND MANAGEMENT

10.1 Health & Hygiene Personnel

Dr. William L. Dyson, CIH, Vice President will have primary management responsibility for this project at Health & Hygiene. Other personnel who may participate in the study are Ronald Hill, CIH and David S. Davis, both industrial hygienists. The Burlington Industries Industrial Hygiene Laboratory person for the project is Sharon Lonon. She has more than seven years experience with filter weighing for cotton dust and other sampling at Burlington.

10.2 PEI, Inc. Personnel

Personnel from PEI who may participate on this project are Leslie J. Ungers, CIH and Robert W. Willson, CIH.

10.3 Project Coordination

Scheduling visits to the textile facilities will be done by Health & Hygiene. Conducting the preliminary management conference and coordinating sample and data collection at the facility are the responsibility of the Health & Hygiene site visitor.

QUALITY ASSURANCE PROJECT PLANS

MIDWEST RESEARCH INSTITUTE

DYES - ANALYTICAL METHODOLOGY DEVELOPMENT AND ANALYSIS OF FIELD SAMPLES

DRAFT QUALITY ASSURANCE PROJECT PLAN
for the
Office of Toxic Substances

EPA Prime Contract No. 68-02-4252
Work Assignment No. 56
MRI Project No. 8856-A(01)

For

U.S. Environmental Protection Agency
Office of Toxic Substances
Field Studies Branch, TS-798
Washington, D.C. 20460

Attn: Mr. Richard Kent

SECTION 1.0

DYES - ANALYTICAL METHODOLOGY DEVELOPMENT AND ANALYSIS OF FIELD SAMPLES

Draft Quality Assurance Project Plan

EPA Contract No. 68-02-4252
Work Assignment No. 56

Approval for:		Approval for:	
MIDWEST RESEARCH INSTITUTE		ENVIRONMENTAL PROTECTION AGENCY	
Paul Constant	29 April 87		
Paul Constant Program Manager	Date	Joseph J. Breen Project Officer	Date
Jack Balsinger	29 April '87		
Jack Balsinger Quality Assurance Coordinator	Date	Eileen Reilly-Wiedow Quality Assurance Officer	Date

SECTION 2.0

TABLE OF CONTENTS

List of Plan Holders:

Midwest Research Institute:
J. Spigarelli, J. Going, P. Constant, J. Hosenfeld, J. Balsinger, C. Green, D. Harbin, J. Long, R. Ayling, R. Rembecki

Environmental Protection Agency:
J. Breen, E. Reilly-Wiedow, R. Kent

SECTION 3.0

PROJECT DESCRIPTION

The Environmental Protection Agency (EPA) has initiated a joint study with the Ecological and Toxicological Association of Dyestuffs Manufacturing Industry (ETAD) and the American Textile Manufacturers Institute (ATMI) to assess the exposure of textile plant dye weighers to airborne dye particles which are present in so-called "drug rooms" in the textile facilities. As prime contractor for the Office of Toxic Substances, Midwest Research Institute (MRI) has been directed to develop the analytical methodology necessary to determine the total amount of dyes present on air sampling filters.

After extensive discussions between industrial dye chemists and analytical chemists at MRI, it was concluded that conventional quantitation of individual dyes on each air filter was not feasible, given the low quantities expected to be present. Various alternative methods based on measuring physical properties of dyes as a class of compounds were considered. A method based on spectrophotometry was deemed to be the most applicable to the analysis of dyes in general, especially at low levels.

The use of quantitation methods that are general (i.e., nonspecific) for a class of compounds will frequently result in final values which are more uncertain than those values obtained from a more specific method. The spectrophotometric approach to the determination of total dyes on an air filter will result in an estimation of the amount of dyes that are present. This is because the method assumes that all dyes that are weighed in the drug room are present on the air filter and appear in amounts proportional to the amount of each dye handled during the air monitoring period. Adding to the uncertainty of the value is the inability to determine (in most instances) the suitability of using these analytical assumptions in analyzing the actual samples. One distinct advantage of the spectrophotometric method, however, is that its accuracy improves with the number of dyes present on the filter, i.e., increasing sample complexity will give better dye estimates.

A means of assessing the approximate uncertainty of the dye estimate can be found by undertaking a statistical treatment of the absorbance characteristics of the individual dyes comprising the sample set. In this fashion, probable errors in the dye estimate can be generated for different dye mixture scenarios of the sample set. Absorbance spectrum profiles and/or drug room dye utilization data can then be employed to either favor or rule out certain dye mixture scenarios.

The scope of work is comprised of two tasks as described below.

3.1 Subtask A: Analytical Methodology Development

MRI will develop analytical methodology for estimating the amount of dyes collected on a filter during air sampling in dye drug rooms. The

experimental lab study will focus on estimation of unknown quantities of dyes by a visible absorbance method. Various aspects of this dye estimation method will be investigated. These include determining the detection limits of various groups of dyes, establishing the uncertainty of the estimate, devising an efficient filter extraction scheme, and determining dye recoveries from air filters for different groups of dyes.

3.2 Subtask B: Analysis of Dye Drug Room Field Samples

MRI will analyze air filter samples collected during surveys of a number of dye drug rooms. These analyses will be carried out by the methodology developed in Subtask A. Both area and personal air sampling will take place. The monitoring period will consist of one complete work shift at each drug room site. Two personal air samplers will be worn by one drug room worker per plant. Field air filter blanks and filter lot blanks will be collected for background correction and determination of dye recoveries for certain groups of dyes. Samples of the bulk dyes handled in the drug room will be taken at the end of the shift after pump shut-off, or during the shift if it is the judgment of the industrial hygienist that this will not affect the air samples.

Sample analysis will consist of extracting all dyes from the air filters, measuring and storing the visible absorbance data points from the extract solutions, and calculating the estimated quantity of dyes present on each filter based on a physical constant derived from the individual bulk dyes which were handled during the monitoring period. The uncertainty in the value of this constant will be proportional to the precision of the individual dye absorptivity values. The precision of the absorptivity values will be documented by performing them in duplicate for the trial plant analysis. An average airborne dye concentration will be calculated by dividing the total dye estimate by the volume of air sampled during the monitoring period.

SECTION 4.0

PROJECT ORGANIZATION AND MANAGEMENT

The work assignment organizational chart is shown in Figure 4-1. All MRI personnel may be reached by telephone at (816) 753-7600.

4.1 Program Management

Mr. Paul Constant, Program Manager, will represent management. He will be assisted in this effort by Mr. John Hosenfeld, Deputy Program Manager. Together they will:

- Assure that all necessary resources are available.
- Assure that the Quality Assurance Coordinator (QAC) is fully informed and involved in the project.
- Assure that all personnel are informed of project QA policy.
- Review all communication from the QAC or QAM regarding the project.
- Assure that any problems, deviations, etc., reported by the QAC or QAM receive immediate corrective action.
- Assure that the financial standing of the project is fully reported to the EPA project officer and work assignment manager.
- Review all technical work and reports for overall technical accuracy.

4.2 Quality Assurance Manager (QAM)

Ms. Carol Green, Quality Assurance Manager (QAM), will represent MRI.

- Review the project QA plan to assure that it is consistent with corporate and client policies and procedures.
- Assure MRI management that the facilities, equipment, personnel, procedures, and records are consistent with corporate and client QA objectives and requirements by conducting or directing independent inspections and/or audits.
- Monitor the work assignment QA activities.
- Report unresolved corrective actions to corporate management for resolution.

Work Assignment Organization Chart

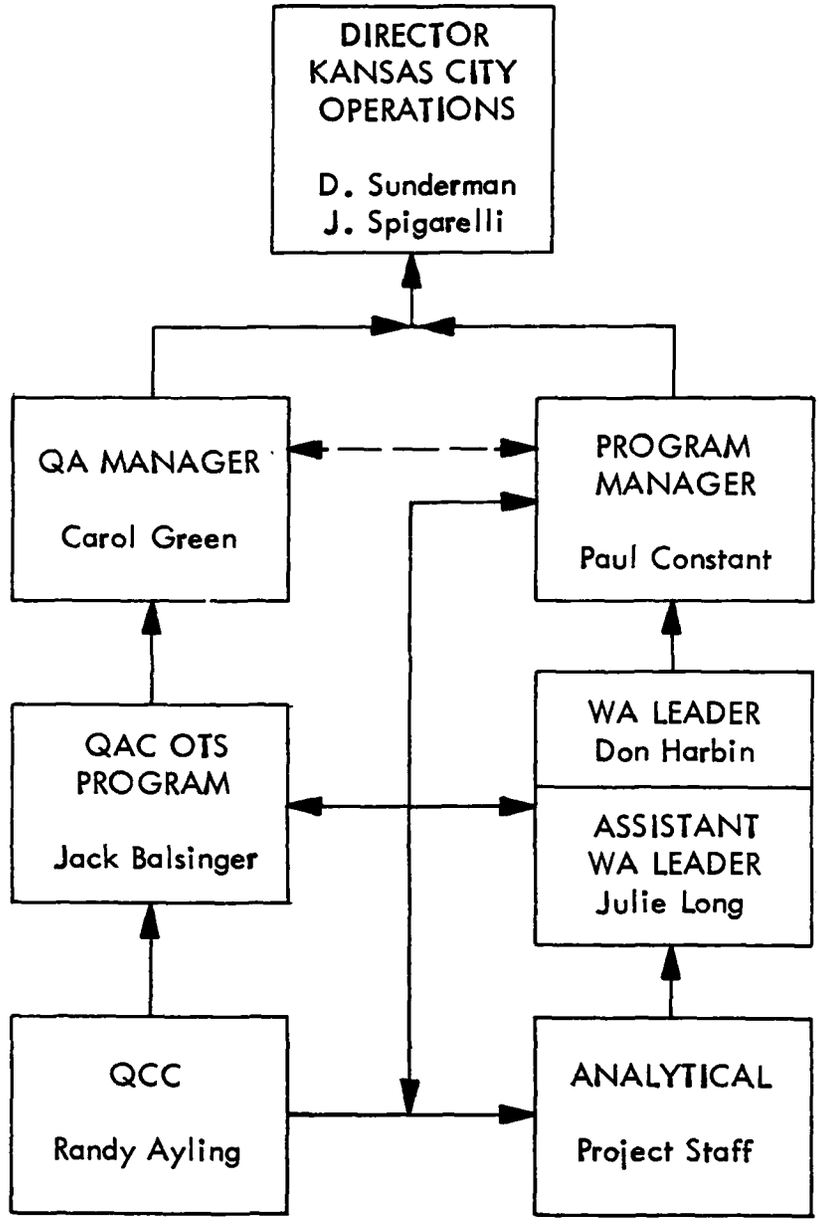

Figure 4.1

4.3 The Quality Assurance Coordinator (QAC)

Mr. Jack Balsinger will serve as the Quality Assurance Coordinator and will represent program management with respect to quality assurance. He will:

- Assure that all corporate and client QA policies and procedures are available and understood by the project staff by conducting inspections and audits.
- Help prepare the project QA plan.
- Approve the project QA plan.
- Assure that the facilities, equipment, personnel, methods, records, and controls are consistent with project objectives and requirements by conducting or directing inspections and/or audits. Inspection/audit results and corrective action requests will be reported to the program management, MRI management, and the QAM.
- Reinspect or audit to assure that appropriate corrective actions were implemented. Report unsolved actions to the program management and the QAM for resolution.
- Conduct additional audits as directed by the program manager and/or QAM.
- Review and audit data reports and supporting evidence prior to submission to EPA.
- Prepare QA reports to be submitted to EPA.

4.4 Quality Control Coordinator (QCC)

Mr. Randy Ayling will serve as the QCC. He will:

- Conduct systems audit(s), which include reviewing notebooks, chromatograms, printouts, and other hardcopy information and report the findings to the QAC.
- Prepare performance audit samples.
- Report audit findings to program manager after QAC review and approval.
- Conduct additional audits as directed by the QAC.

4.5 Work Assignment Leader/Assistant Work Assignment Leader

Mr. Don Harbin will be the work assignment leader. He will be assisted by Ms. Julie Long, assistant work assignment leader. Together they will:

- Help prepare and update the project QA plan.
- Be responsible for training staff where required.
- Be responsible for sample receipt and traceability.
- Enforce instrument calibration and maintenance procedures.
- Maintain document control of lab data, notebooks, records, and other hard copy information.
- Review and approve all data prior to submittal to EPA.
- Review/validate raw data (e.g., notebooks, forms, strip charts, etc.).
- Ensure that any deviations from protocol are approved, documented, and reported.
- Be responsible for analytical data traceability.
- Take corrective action on any problems and communicate them in writing to the QAC and the program and department managements.
- Prepare and submit monthly reports.
- Prepare and submit other reports as requested by the EPA work assignment manager in conjunction with project staff.

SECTION 5.0

PERSONNEL QUALIFICATION

Mr. Paul C. Constant and Mr. John Hosenfeld will serve as program manager and deputy manager, respectively. Mr. Hosenfeld will assist Mr. Constant. Mr. Constant has also served as program liaison officer and as deputy program manager on the previous contract. Their credentials were previously submitted in the proposal for this contract.

Mr. Don Harbin will serve as the Work Assignment Leader. He has significant experience in the high pressure liquid chromatographic analysis of dyes as well as trace quantitation methods for the determination of organic compounds. His credentials were previously submitted in the proposal for this contract.

Ms. Julie Long will serve as the Assistant Work Assignment Leader (effective September 1986). She previously served as Quality Control Coordinator for this program. She has contributed to a number of research programs requiring her instrumental skills for the analysis of toxic compounds.

Ms. Carol Green will be the Quality Assurance Manager. She has served in this capacity since May 1983. Her credentials were previously submitted in the proposal for this contract.

Mr. Jack Balsinger will be the Quality Assurance Coordinator. His credentials were previously submitted in the proposal for this contract.

Mr. Randy Ayling will serve as Quality Control Coordinator (effective September 1986). He is skilled in spectrophotometric analysis and in conducting performance audits and systems audits.

Dr. Jairus D. Flora, Jr., will perform the statistical analyses of the data from each plant site. His credentials were previously submitted in the proposal for this contract.

Mr. Roger Rembecki will serve as Senior Technician conducting the laboratory analyses for this project. He joined MRI in November 1986. He has been trained in the dye analysis protocols by Julie Long.

SECTION 6.0

FACILITIES, EQUIPMENT, CONSUMABLES, AND SERVICES

6.1 Facilities

Sample preparation will be performed in a laboratory designated, in part, for this project (MRI Lab 324-W). This laboratory is equipped with fume hoods and an analytical balance contained in a vented glove box. The windows are covered with low actinic film. The fluorescent lights have been replaced with red lighting.

Sample analyses will be performed on a Cary 219 spectrophotometer (located in MRI Lab 324-W) or other equivalent spectrophotometers.

Data file processing will be performed on a Hewlett-Packard 9826 microcomputer located in MRI Lab 119N.

6.2 Equipment

The equipment used on this task includes:

- Cary 219 spectrophotometer or equivalent; modified to allow an analog detector signal to be output to an integration device.
- Nelson Analytical A/D interface box and related chromatography software package (Model 4400).
- Hewlett-Packard Model 9826 microcomputer and peripherals used to run the software.
- Mettler H20 analytical balance or equivalent; capable of weighing to the nearest 0.01 mg.
- DuPont P4000 personal monitoring pumps or equivalents.
- Volumetric glassware, Low Actinic.

6.2.1 Calibration

6.2.1.1 The spectrophotometer is checked on a weekly basis by qualified MRI personnel. A holmium oxide film traceable to the National Bureau of Standards is used to ensure that wavelength readings meet the manufacturer's specifications. Oxford Spectrochek® QA buffer solutions are used to ensure that absorbance readings meet specifications. If the instrument performance falls outside of the acceptable range, it will be reported to the Instrument Services Group of MRI and corrective action will be taken.

6.2.1.2 The Nelson Analytical chromatography software and Hewlett-Packard hardware have built-in system checks to monitor their performance. Error messages wil be displayed if problems occur. A copy of the specific version of the software program used for processing the data points will be archived.

6.2.1.3 The analytical balance is checked before use with weights that are traceable to or checked against National Bureau of Standards weights to confirm performance according to manufacturer's specifications.

6.2.1.4 Personal air sampling pumps will be of the feedback flow-adjusting type. Each sampling pump will be calibrated for an airflow of 2 to 2.5 L/min prior to use and checked afterwards.

6.2.2 Maintenance

Maintenance of the analytical equipment used in this task will be done according to manufacturer's specifications and at their recommended frequency. This is summarized in Table 6.1

Table 6.1. Maintenance

Equipment	Service	Frequency
Spectrophotometer	General	As needed
Hewlett-Packard 9826	Limited requirements	As needed
Balance	Cleaning and adjustment for calibration	1 year
DuPont P4000 personal monitor pumps	Replace belts and inlet filters	As needed

6.3 Consumables

All dimethyl sulfoxide (DMSO) used will be A.C.S. reagent grade or better. All pH 7.0 and pH 3.0 buffer will be reagent grade. All filter spiking experiments will use filters identical to those used during the field sampling. Bulk sample containers will be amber pill vials.

6.4 Services

Health and Hygiene, Inc., will conduct the air sampling at the selected drug room sites and will perform the following services:

- Preweigh and postweigh air sampling filters, including field air filter blanks.
- Provide calibrated air sampling pumps for use at the site.
- Take bulk samples of all powder dyes handled in the drug room during the monitoring period.
- Take bulk samples of all non-dye compounds handled in the drug room during the monitoring period which might interfere with the analytical analysis method.
- Provide all survey information regarding site conditions and monitoring period activity by the drug room worker.
- Transfer field air filter samples to new cassettes (not required for field air filter blanks) after postweighing and ship to MRI along with original cassettes and support pads.
- Provide suitable documentation for calibration and maintenance of all air sampling and weighing equipment for inclusion in the work assignment archives.

SECTION 7.0

DATA GENERATION

7.1 Experimental Design

The sampling design will be prepared by EPA. A flow chart of the steps involved in the analytical method is shown in Figure 7-1.

7.2 Sample Collection (Health and Hygiene, Inc.)

7.2.1 Area Sampling

Area sampling will be performed using samplers operating under critical flow conditions of 5 to 8 L/min. One area sampler will be placed near the drug room weighing station and another will be located at a bulk dye storage area. The sampling period will be for the entire 8-h shift.

7.2.2 Personal Sampling

One drug room worker will be monitored at each plant. The worker will wear two personal air samplers. The 37-mm open-face sampling cassettes will be operated at air flow rates of 2 to 2.5 L/min. When sampling, the inlet of the cassettes will be pointed downward so that only airborne material will be collected during the 8-h shift.

7.2.3 Blanks

Filter lot blanks and field air filter blanks will be provided by Health and Hygiene. Filter lot blanks will be filters from the same lot as those used to collect field samples, but which will not be sent out to the drug room site. Field air filter blanks will be filters which are handled in the same manner as the sampling filters except that no air will be drawn through them.

7.2.4 Bulk Dye Samples

Small samples of each powder dye handled in the drug room during the monitoring period will be taken and labeled with unique bar-code stickers provided by MRI. An identical bar-code sticker will be placed on the bulk dye inventory sheet along with the full dye name, manufacturer, and lot number. Bulk samples will be taken with disposable spoons (one for each dye).

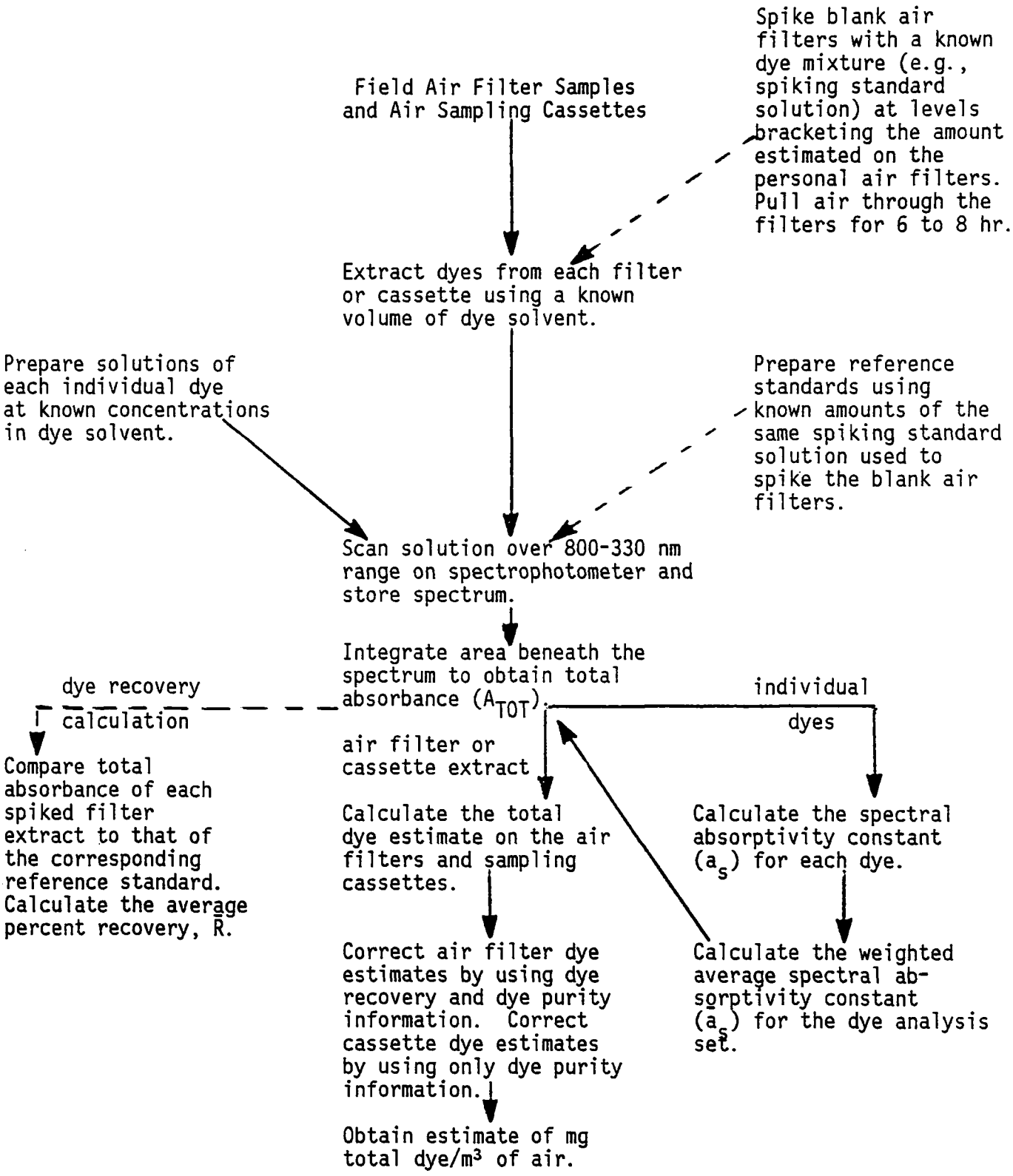

Figure 7-1. Flow chart for the estimation of total dyes on an air filter.

7.2.5 Collection Conditions

Temperature, relative humidity, and barometer measurements will be monitored and recorded during the test. A general description of the airflow characteristics in the drug room will be recorded. The sampling time interval will also be documented.

7.2.6 Shipping and Handling

After gravimetric analyses of the field air filters have been performed at Health and Hygiene, Inc., the air filters, original cassettes and support pads, bulk dye samples, field air filter blanks, filter lot blanks, and the field data forms will be shipped to MRI by overnight courier. Bulk dye samples will be shipped separately to minimize the chances of contamination.

7.3 Sample Traceability

Tracking of field air filter samples, field air filter blanks, and bulk dye samples will be achieved using the field data forms (Figure 7-2). During each phase of field testing (e.g., air sampling, gravimetric analysis) all samples will be assigned/identified using a unique sample identification number.

7.4 Laboratory Analysis Procedures

See Appendix B for the analytical protocol.

7.5 Internal Quality Control Checks for Sample Analyses

7.5.1 General

New and current lots of reagents are checked prior to use.

7.5.2 Calibration

Proper instrument performance will be confirmed and documented (see Section 6.2.1).

PERSONAL AREA AIR SAMPLING DATA SHEET

Plant ID No:	Sampling Performed by:	Date (Mo/Da/Yr)
Worker Monitored:	Job Title/Work Duties:	

SAMPLING EQUIPMENT AND CALIBRATION

Flowmeter Model Number:	Flowmeter Serial Number:
Flowmeter Calibration Date:	Calibration Traceable to:

Sampling Pump Model No:		
Sampling Pump Serial No:		
Pre-sampling Flowrate:		
Date:		
Time:		
Post-sampling Flowrate:		
Date:		
Time:		
Signature(s):		

FIELD SAMPLING DATA

Sample ID Number:		
Sampler Location:	left [] right []	left [] right []
Sample Start Time:		
Sample Stop Time:		
Sample Duration: (min)		
Pump Flow Rate: (L/min)		
Sample Air Volume: (m3)		

Signature:	Date:
Calculations Checked by:	Date:

TRACEABILITY RECORD

Sent by:	Date:	Time:	------->	Rec'd by:	Date:	Time:
Sent by:	Date:	Time:	------->	Rec'd by:	Date:	Time:

Figure 7-2

STATIONARY AREA AIR SAMPLING DATA SHEET

Plant ID No:	Sampling Performed by:	Date (Mo/Da/Yr)

SAMPLING EQUIPMENT AND CALIBRATION

Flowmeter Model Number:	Flowmeter Serial Number:
Flowmeter Calibration Date:	Calibration Traceable to:

Sampling Pump Model No:		
Sampling Pump Serial No:		
Pre-sampling Flowrate:		
Date:		
Time:		
Post-sampling Flowrate:		
Date:		
Time:		
Signature(s):		

FIELD SAMPLING DATA

Sample ID Number:		
Sampler Location:		
Sample Start Time:		
Sample Stop Time:		
Sample Duration: (min)		
Pump Flow Rate: (L/min)		
Sample Air Volume: (m3)		

Signature:	Date:
Calculations Checked by:	Date:

TRACEABILITY RECORD

Sent by:	Date:	Time:	------->	Rec'd by:	Date:	Time:
Sent by:	Date:	Time:	------->	Rec'd by:	Date:	Time:

Figure 7-2 (continued)

ANALYTICAL DATA

GRAVIMETRIC ANALYSIS (Health and Hygiene)						
	Personal Filters		Area Filters		Blank Filters	
Sample ID Number:						
Filter Preweight:						
Filter Postweight:						
Sample Weight:						
Blank Correction:						
Adjusted Weight:						
Signature(s):		Date:		Calculations Checked by:		

VISIBLE SPECTROPHOTOMETRIC ANALYSIS (MRI)						
Filter Extract. Date:						
Data File Number:						
Sample Prepared by:						
Total Absorbance:						
Corr. Total Absorbance:						
Absorptivity: (As)						
Dye Estimate: (ug)						
Avg. Recovery: (%)						
Corr. Dye Est: (ug)						
Est. Airborne Dyes: (ug/m3)						
Data Reference Number:						
Signature(s):		Date:		Calculations Checked by:		

Figure 7-2 (concluded)

7.5.3 Internal QC Samples

With each batch of samples, appropriate QC samples will be included so the quality of the sample data can be assessed. These QC samples include reagent blanks, field air filter blanks, and spiked filter controls for determining extraction efficiency.

7.5.3.1 Reagent blanks: At least one reagent blank will be analyzed each analysis day to check for solvent interferences. No filters will be used for this determination.

7.5.3.2 Field air filter blanks: The field air filter blanks are filters from the same lot as the air sampling filters. Field air filter blanks will be subjected to the same handling procedures as the field air filter samples except that no exposure to the drug room environment will be allowed. At least one field air filter blank will be collected and analyzed for each drug room site monitored.

7.5.3.3 Spiked filter controls: Filter lot blanks will be spiked with known amounts of a dye mixture comprising a subset of the total number of dyes handled in the drug room. This dye mixture (i.e., the spiking standard solution) will be composed of the individual dyes which account for at least 80% of the total quantity of dye handled by the drug room worker during the monitoring period. The relative amounts of the component dyes in the spiking standard solution will reflect their actual usage during the monitoring period. Spiked filter controls will be prepared in replicate to check the precision of the recovery experiments.

7.6 Systems and Performance Audits

7.6.1 Systems audits: Systems audits by the QAC or QCC shall include:

- Inspecting facilities and equipment for adequacy, appropriateness, and safety during use.
- Reviewing actual practices versus written procedures and protocols.
- Inspecting the records of maintenance and calibration.
- Inspecting QC practices.
- Preparing and submitting a report with recommended corrective actions to the QAC, and after approval, to the work assignment leader, program management, and the QAM.

7.6.2 Performance Audits

A performance audit sample (PAS) is designed to check the operation of the equipment as well as the analytical method and data reduction procedures. Performance audit samples will be prepared independently by the QAC or QCC using the bulk dyes and will be analyzed along with regular samples. The audit samples will be included periodically (beginning, middle, and end) during analysis of each drug room set (e.g., during field air filter analysis, during bulk dye analysis, and during the dye recovery experiments). The analyst will report to the QCC or QAC the total absorbance of each PAS. The QAC or QCC will calculate a "found" concentration for each PAS using the spectral absorptivity constant of the specific dye(s). If the found concentration does not agree within ± 30% of the actual bulk dye concentration, one or more of the actions listed below will be taken:

1. The QAC or QCC will supply another audit sample for analysis, using the same bulk dye(s) as before.

2. Calibration check of the Cary 219 spectrophotometer to verify that absorptivity and wavelength requirements fall within specifications.

3. Confirm the spectral absorptivity constant of the dye(s) used for the PAS by repeat analysis.

All performance audit sample results and any corrective actions taken will be reported to the work assignment leader, program management, and the QAM.

Performance audit samples will also be analyzed if (1) the QCC or QAC believes the analysis procedure has changed, (2) analytical problems are suspected, (3) the MRI work assignment leader or the EPA work assignment manager requests samples.

7.6.3 QAC Data Audits

Data audits will be conducted or directed by the QAC by reviewing, auditing, and approving all reports and supporting evidence for accuracy and QA compliance prior to report submittal to EPA.

SECTION 8.0

DATA PROCESSING

8.1 Collection

Data collection will utilize both manual and computerized acquisition systems. All raw data obtained manually shall be legibly recorded using permanent ink. Each person who records data shall sign/initial and date the data. Strip charts shall be labeled to allow traceability of all data. All data collected and manipulated using computerized aquisition systems shall be retained in hardcopy form for archiving. Custody of the original data media will be the responsibility of assigned project staff until archived. All data will be handled in a confidential manner.

8.2 Data Reduction

Standard data reduction procedures, as given in Appendix B, will be used. All data reduction will be performed manually or by using a database computer software program. In addition, all sample manipulations (e.g., weighing, dilution, etc.) will be clearly documented.

8.3 Data Validation

The data validation process will include:

- Checking field data forms to ensure accuracy and completeness of bulk dye sampling, amounts of dyes weighed, and the associated number of weighings.
- Validating all equations and computer programs and documenting the validation and evidence.
- Validating and checking electronic data transfer.
- Proofreading all data entries for transfer errors. Data transfer will be kept to a minimum to prevent errors. The analytical data will be transferred manually from a computerized output to data tables, then from the data tables to a computerized data reduction program to obtain final results. Data will be checked for transfer errors.
- Screening data for consistency by a second project staff member.
- Checking calculations, randomly.
- Performing outlier checks.
- Documentating of all associated blank, standard, and QC data along with results of analyses for each batch of samples.

- Examining QC data and QC checks.
- Maintaining records of reviews and validation.
- Examining data/information for completeness, representativeness, and comparability.
- Reviewing and approving of all records by the work assignment leader.
- Reporting protocol deviations and assumptions with the results.

The work assignment leader will be responsible for assuring data validity.

8.4 Storage

Raw data will be documented in laboratory notebooks, on data forms or printer paper, as strip chart recordings, or as hardcopy originals from magnetic tapes or disks. Permanent storage of work assignment records will be archived in a formal project file (SOP-QA7).

SECTION 9.0

DATA QUALITY ASSESSMENT

This analytical method gives an estimate of total dye content on an air filter and should not be considered as a quantitative determination of the amount of dyes present. The precision of the analytical method should meet expected standards for a spectrophotometric method which involves the extraction of microgram quantities of analytes from an air filter. The objectives of precision for this method will be to obtain dye recoveries for replicate spiked filter samples which have relative differences ± 25% of each other. The objective for accuracy will be to obtain total dye estimates on replicate spiked filter samples which have relative errors ± 50% of the actual dye present on the air filter. Average extraction efficiencies for the dyes using spiked filters should fall within the range of 60 to 140% recovery to yield meaningful data.

9.1 Precision

The precision of the analytical method will be determined by analyzing replicate spiked filter samples and calculating their respective percent recoveries. Percent relative difference, R.D., will be calculated as follows:

$$\text{R.D. } (\%) = \frac{R_1 - R_2}{\bar{R}} \times 100$$

where R_1 = the % recovery for one replicate

R_2 = the % recovery for the other replicate

$\bar{R}$ = the average % recovery calculated from R_1 and R_2

9.2 Accuracy

The accuracy of the analytical method can only be established for known dye spike samples. Accuracy may be indicated by comparing the total dye estimate for spiked filter controls to the actual amount spiked on the filters. Accuracy will be measured by calculating the relative error, R.E., of the total dye estimate:

$$\text{R.E. } (\%) = \frac{D_{EST} - D_{ACT}}{D_{ACT}} \times 100$$

where D_{EST} = the total dye estimate in µg
D_{ACT} = the actual total dye quantity spiked in µg

9.3 Uncertainty

The uncertainty of the total dye estimation will be primarily dependent on the specific group of dyes being analyzed. The dye estimation is based on the weighted average absorptivity of all of the dyes handled during the monitoring period. In general, the uncertainty of the dye estimate will be proportional to the standard deviation of the individual dye absorptivities. For this reason it is not possible to establish a specific uncertainty value for the total dye estimate.

Probable errors in the total dye estimate can be obtained by using a statistical computer program that selects dye mixtures from the given group of dyes. Specific dye handling information is input into the program to weight the dye selection process. As a result, the more heavily used dyes have a higher probability of being selected than the minor use dyes. In this manner the errors associated with various subsets of the entire group of dyes can be approximated. Additional data, such as the number of dye weighings or dustiness observations, can then be used to focus in on dye subsets which are more likely to occur on the air filter.

9.4 Recovery

The efficiency of the filter extraction procedure will be indicated from the recovery results of the spiked filter analyses. Dye recovery will be determined by a direct comparison of the spiking solution (e.g., reference standard) to the solution obtained from extracting the spiked filter.

$$R\ (\%) = \frac{A_{fil}}{A_{STD}} \times 100$$

where A_{fil} = total absorbance of spiked filter extract
A_{STD} = total absorbance of reference standard

9.5 Traceability of Instrumentation

All collection and measuring instrumentation will have a unique identification number. Maintenance, calibration, and use logs will be maintained.

9.6 Traceability of Samples

All samples will have a unique identification number along with information about the worker being monitored, the plant site, monitoring location, exposure time and conditions, collection device, etc.

9.7 Traceability of Data

Data will be documented and filed to allow complete reconstruction, from initial field records to data archiving.

9.8 Completeness

Due to the very small number of data points available per drug room site, completeness of the data will be crucial in order to obtain meaningful data.

SECTION 10.0

CORRECTIVE ACTION

The work assignment leader has primary responsibility for taking corrective action; if he is unavailable, the program manager shall be contacted for instructions. Unresolved problems are reported to the program manager and the QAM. The Associate Director of K.C. Operations and the Senior Vice President are notified of unresolved problems by the QAM.

Some of the types of problems and corrective actions to be taken are listed below.

8.1 Performance/Systems Audits

If problems are detected during an audit:

- The auditor shall notify the person responsible, the work assignment leader, and the QAC of the problem(s) and any action(s) that have been taken.
- The work assignment leader and the person responsible shall correct the problem, then notify the QAC.
- The auditor, with the approval of the QAC, shall prepare a corrective action request and send it to the program manager, work assignment leader, and the QAM.

10.2 Loss of Data

The work assignment leader shall investigate the problem, then perform one or more of the following actions:

- If the problem is limited in scope, the problem/action taken is documented in the project records; the work assignment leader then prepares and sends a problem/action taken memo to the QAC and the program manager.
- If a large quantity of data is affected, the problem/action taken is documented in the project records; the work assignment leader then prepares and sends a problem/action-taken memo to the QAC, the program manager, and to the EPA work assignment manager.

10.3 Significant QA Problems

In general, the work assignment leader shall identify technical problems.

- The work assignment leader prepares and sends a problem memo to the QAC and program manager; if the problems are significant, the action is determined collectively.
- The action taken is documented in the project records.
- The problem and action taken is reported to the EPA work assignment manager.

SECTION 11.0

DOCUMENTATION AND REPORTING

11.1 Documentation

- All documentation shall be in permanent ink or on computer printouts.
- Corrections will be performed as follows: Draw a single line through an incorrect entry so that the original entry remains legible. Add the correct entry; then explain, initial, and date the correction.
- New information may be added to original raw data. It will be initialed, dated, and explained.
- All deviations from standard operating procedures (SOPs), procedures, and protocols will be documented.
- All assumptions and interpretations will be documented.
- Strip charts, magnetic tapes, etc., will be labeled with a format identifier, the date, the ID(s) of the sampling equipment, and the name of the person responsible for the data recording equipment. Hardcopies of all magnetic data will be generated for archiving purposes.

11.2 Document Control

- Raw data will be documented in laboratory notebooks, on sampling forms, on analytical forms, on printer paper, as hardcopies from magnetic tape, and as strip chart recordings.
- A raw data packet for each drug room site monitored will be generated, along with data tracking forms to document the existence and flow of data through the data processing cycle.
- All project-related documents will be maintained by assigned project staff until archived.

11.3 QA Reports to Program Management

The QAC, in cooperation with the work assignment leader, shall identify critical areas of the project which will be subject to inspection. The inspection will include a review of:

- Staff credentials.
- Equipment maintenance and calibration records.
- Equipment performance.

- Documentation practices.
- Recordkeeping practices.
- Adherence to protocols, SOPs, and the QA project plan.
- Assessment of data accuracy, precision, and completeness.

The results of inspections and audits will be reported to the work assignment leader, the program manager, and QAM.

11.4 Report Design

Progress, draft final, final reports, and QA summary reports will be submitted in accordance with the provisions for reporting in the contract. Verbal status reports will be made biweekly to the work assignment leader.

APPENDIX A

STANDARD OPERATING PROCEDURE FOR CHECKING THE CALIBRATION OF THE CARY 219 SPECTROPHOTOMETER

STANDARD OPERATING PROCEDURE FOR CHECKING THE CALIBRATION OF THE CARY 219 SPECTROPHOTOMETER

Absorbance and wavelength verification of the Cary 219 spectrophotometer (or equivalent) is performed and documented weekly by MRI. The absorbance is monitored using Oxford Spectrochek® QA solutions (or equivalent). The wavelength is monitored using a NBS traceable holmium oxide film. If the instrument does not pass verification, the Instrument Services Group of MRI will be notified and corrective action will be taken. Verification documents will remain on file at MRI.

The following steps outline the operating procedure for conducting the spectrophotometric calibration check:

I. Wavelength Verification via Holmium Oxide Film

A. Turn main power switch to "on" position. Allow Vis-UV light source to warm up for at least 20 min.

B. Initially, an air versus air scan will be performed to zero the instrument. The conditions for setup are identical to those listed in Appendix B, Section 13.1.2 except for the following changes:

- Slit: 1.0 nm
- Scanning rate: 0.5 nm/s
- Chart display: 5

C. Close the covers to the cuvette compartments on the spectrophotometer.

D. Set the upper wavelength limit at 750 nm using the wavelength 1 dial and set the lower wavelength limit at 250 nm using the wavelength 2 dial.

E. Using the "scan" dial, turn dial (+) or (-) to set the wavelength at 750 nm.

F. Turn the timer mode knob fully clockwise, then back to "wavelength."

G. Turn the autobaseline knob fully clockwise to the "record" position and hold for a couple of seconds, making sure the red recording light comes on.

H. Adjust the balance knob to give an absorbance reading of 0.1000 (or as close as possible) on the digital display.

I. Insure measurement dial is set on serial.

J. Press "step" button and scan the wavelength range.

K. When the scan is finished, the instrument will return the wavelength to 750 nm.

L. Turn the pen off.

M. Turn the timer mode knob fully counter-clockwise to "off" position. Line up pen on a dark graduation.

N. Adjust the balance knob so the digital readout shows 0.000 (or as close as possible).

O. Turn timer mode knob fully clockwise, then back to "wavelength."

P. Turn measurement knob to overlay.

Q. Turn pen on and press "step" button to begin scan.

R. When scan is finished, turn the pen off. Watch to be sure the paper retracts properly.

S. Turn the timer mode knob to "off."

T. Remove the sample cover, place the holmium oxide film into the sample turret, and close the lid.

U. Check to be sure the pen has realigned itself to the same starting point as in step M.

V. Turn timer mode knob fully clockwise, then back to "wavelength."

W. Turn pen on and press "step" button to begin scan.

X. When the scan has finished, turn the pen and the timer mode knob to off positions.

Y. Remove the holmium oxide film.

II. Absorbance Verification via Oxford Spectrochek® QA Buffer Solutions

A. Initally, a water versus water scan will be performed to zero the instrument, using two 1-cm path width cuvettes filled with deionized water. Clean the outer surface of each cuvette with an appropriate tisue to remove any smudges.

B. Place one cuvette in the reference compartment and place the other cuvette in the sample compartment.

C. Dial in 690 nm for the upper wavelength limit and 300 nm for the lower wavelenght limit using the wavelength 1 and 2 dials, respectively. Using the "scan" dial, turn the dial to (-) to set the wavelength at 690 nm.

D. At this point, follow the steps outlined in F through S for the wavelength verification via the holmium oxide film. (Switch scan rate to 2.0 nm/s at this point.)

E. Remove the cuvette from the sample compartment. Fill with solution no. 2, rinsing the cuvette prior to filling with ~ 2 mL of the same solution. Wipe the cuvette surface and replace in sample compartment.

F. Reset the wavelength range to scan from 690 to 400 nm.

G. Fully turn the timer mode knob clockwise, then back to "wavelength."

H. Turn the pen on and press "step" button to start the scan.

I. When the scan is finished and the wavelength has returned to 690 nm, turn the pen and the timer mode knob off.

J. Open the sample compartment and remove the sample cuvette. Rinse the cuvette with solution no. 1 and fill. Replace into sample compartment and close lid.

K. Repeat steps G through I.

L. Remove the sample cuvette, rinse with solution no. 4 and fill. Place back into the sample compartment and close lid.

M. Reset the wavelength range to scan from 400 nm to 300 nm. Using the "scan" dial, turn the dial to (-) to set the wavelength at 400 nm.

N. Repeat steps G through I.

O. Remove the sample cuvette and repeat steps G through I using solution no. 3.

APPENDIX B

ANALYTICAL PROTOCOL

ANALYTICAL METHOD FOR THE ESTIMATION OF TOTAL DYES IN TRACE QUANTITIES ON AIR SAMPLING FILTERS

1.0 SCOPE AND APPLICATION

1.1 This method has been developed for the estimation of trace quantities of groups of dyes from the following dye classes: acid, basic, direct, disperse, and reactive. Dyes from other major classes have not been investigated.

1.2 This method yields an estimate of the total quantity of a group of dyes present on an air sampling filter. Information about the quantity of each individual dye on the air filter is not possible with this method.

1.3 This method is suitable for use by technicians possessing nominal experience with spectrophotometric equipment and procedures.

2.0 SUMMARY OF METHOD

This method describes the procedures for estimating the total quantity of a group of dyes present on an air sampling filter. A general diagram of the method is shown in Figure B-1.

The analysis procedure consists of extracting the dyes from each air filter using a minimum volume of buffered dye solvent. An aliquot of each filter extract is passed through a 0.45 μm Gelman Acrodisc (or equivalent) and transferred to the measuring cell in the spectrophotometer. The visible absorption spectrum of each extract is obtained and digitized by means of an A/D converter box interfaced with the spectrophotometer. Air sampling cassettes and one or more field air filter blanks are also extracted and scanned. All spectra are stored on floppy disks for future data manipulation.

Spectra of each individual dye handled in the drug room during the air monitoring period are obtained by analyzing known solutions of bulk dye samples collected at the plant site. These dye spectra are then used to form a data base for estimating the total quantity of dye on the air filters (and sampling cassettes) taken during that monitoring period. Spectra obtained on identical bulk dyes at different plants will not be employed so as to avoid possible lot-to-lot variations in absorption characteristics.

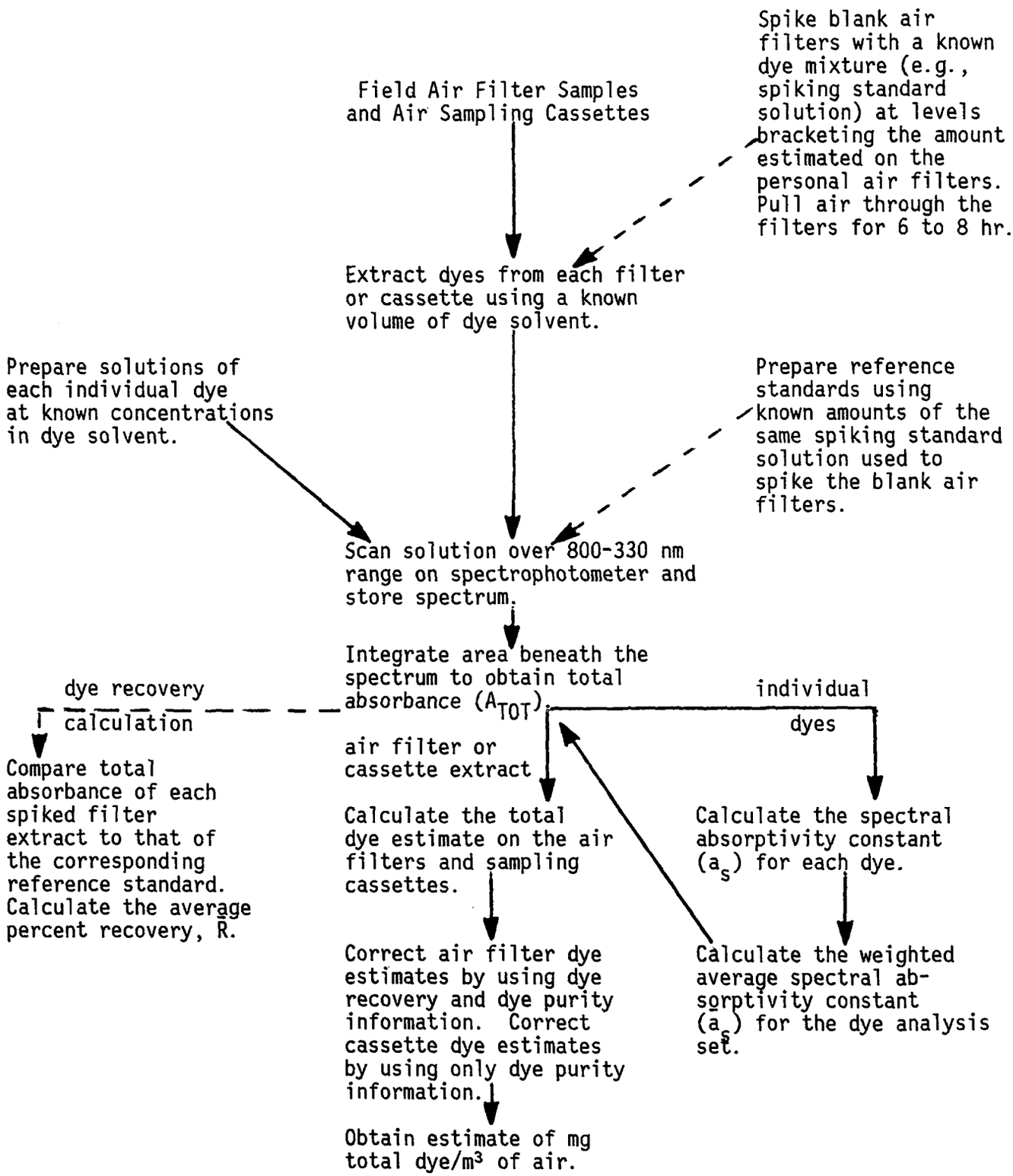

Figure B-1. Flow chart for the estimation of total dyes on an air filter.

The total sample absorbance in the visible wavelength region is used to estimate the total quantity of dye present on each air filter and its corresponding cassette. This entails electronically integrating the area beneath the absorption spectra of each filter or cassette extract and each individual dye. The area values are readily converted to total absorbance (A_{TOT}) by using an appropriate factor (i.e., area count value $\div 10^6$ area counts/ absorbance unit).

Spectral absorptivity (a_s) constants for each individual dye used during the monitoring period are calculated based on Beer's law. From the individual a_s values, and dye handling information, a weighted average spectral absorptivity constant ($\bar{a}_s$) for the group of dyes is calculated.

The total amount of dyes contained on each air filter, and its corresponding cassette, is then calculated using this weighted average. The dye estimates are corrected wherever appropriate for the amount of active ingredient in each dye (i.e., purity) and for the average recovery ($\bar{R}$) of dyes off the air filters.

3.0 INTERFERENCES

3.1 Because of possible reactivity with the dye solvent at the established pH (pH 7), some basic dyes may not be quantifiable using the standard analytical procedure. Use of pH 3 dye solvent is recommended if the bulk dyes handled at the plant site are predominantly of the basic class.

3.2 Due to the nature of the analytical technique used, this method is not particularly susceptible to other interferences except those from other dyes, i.e., cross-contamination. Therefore, after each use, glassware is immediately rinsed with methanol to remove all traces of dye and dye solvent. The glassware is then washed with soap/H_2O, rinsed with deionized water followed by acetone, and allowed to air dry prior to re-use.

3.3 Since fluorescent brighteners are not to be included in the total dye estimate, the lower wavelength limit of the absorbance scan may have to be shifted upward if brighteners were handled during the air sampling period. If this change in the wavelength scan range should be necessary, all the individual dye and filter/ cassette extract solutions in the dye analysis set must be scanned over this same wavelength range.

4.0 SAFETY

All manipulations made with dyestuff samples are performed in a fume hood or glove box. Gloves and other appropriate safety apparel are

worn at all times. Solid and liquid waste is disposed of in the proper manner.

5.0 APPARATUS AND MATERIALS

5.1 Solution Preparation

5.1.1 500 and 1,000 mL graduated cylinders
5.1.2 1 gal glass bottle with 10 mL Repipettor®
5.1.3 Volumetric pipets (TD) - 2, 3, 5, 6, 8, 10, and 20 mL
5.1.4 Volumetric flasks - 10, 25, 50, 100, 250, 500, and 1,000 mL (low actinic)
5.1.5 Disposable pipets
5.1.6 Beakers - 100 mL
5.1.7 Filters - 0.45 µm Gelman Acrodisc (or equivalent)
5.1.8 Glass jars (amber) - 4 oz with DMSO-resistant lid liners (optional)
5.1.9 10-mL disposable syringe (Luer tip)
5.1.10 Stainless steel forceps

5.2 Balance - Analytical; capable of accurately weighing to 0.01 mg

5.3 Shaker - Capable of shaking 4-oz jars at 1 oscillation/s. If a wrist-type shaker is employed, DMSO-resistant lid liners must be used on the glass jars.

5.4 Ultrasonic bath

5.5 Spectrophotometer/data storage system

5.5.1 Spectrophotometer - Dual beam instrument capable of scanning in the visible wavelength region (800-330 nm). The spectrophotometer must have a 1 or 10 V analog signal output, or allow such a signal to be obtained (such as by using the strip chart pen signal).

5.5.2 Cuvettes - Standard 1 cm pathlength.

5.5.3 Nelson Analytical Model 4400 Chromatography Data System, or equivalent.

5.5.4 Nelson Analytical A/D interface box, or equivalent.

5.5.5 Magnetic media for data storage - 5-1/4 in. floppy disks, or equivalent.

6.0 REAGENTS

6.1 Dimethyl sulfoxide (DMSO), ACS certified grade

6.2 Buffer, pH 7 and pH 3 - reagent grade (uncolored)

6.3 Dye solvent (pH 7) - prepared by adding 9 parts DMSO to 1 part pH 7.0 buffer

6.4 pH 3 dye solvent - prepared by adding 9 parts DMSO to 1 part pH 3.0 buffer

7.0 METHOD VALIDATION (Average Recovery Determination, $\bar{R}$)

The analytical method is validated using a subset of the dyes handled in the drug room during the monitoring period. This subset is composed of the individual dyes used most during the monitoring period (based on weight). The component dyes in the subset should represent at least 80% of the total quantity of dye handled and be in proportion to their usage.

Duplicate blank filters are spiked at two levels with a solution of the dye subset (i.e., spiking standard solution) so as to bracket the absorbance of the personal air filters. After drying, clean, humid air is pulled through the filters using similar conditions (time, flow rate) used in the field. Validation is confirmed if the average recovery for the spiked filters falls in the 60 to 140% range.

8.0 SAMPLE RECEIPT AND STORAGE

8.1 Sample Receipt

8.1.1 Samples received from the field consist of:

1. Personal and area air sampling filters, which were removed from their original cassettes and stored in fresh cassettes after gravimetric weight determination.

2. The original air sampling cassettes used during the monitoring period.

3. Field air filter blanks in cassettes, which were carried in the field along with the actual air filter samples.

4. Filter lot blanks (unexposed air filters for use in the recovery experiments).

5. Individual bulk dyes.

8.1.2 Upon receipt, each filter/cassette sample is examined and logged-in using Form A-0. Individual bulk dyes, identified by bar-code labels, are logged-in via use of an appropriate bar-code reader.

8.2 Sample Storage

8.2.1 Unless instructed otherwise, samples are stored in the dark in room 324W at room temperature in their original packing containers until the analysis is completed. After analysis, bulk dye samples are retained at the discretion of EPA.

8.2.2 Although field air filter samples should be analyzed as soon as possible after receipt at MRI, analysis will not be performed without (a) a performance audit sample (PAS) being analyzed concurrently, and (b) without knowledge of what type of dye solvent should be used to perform the analyses (i.e., pH 7 or pH 3). When air filters arrive at MRI, the EPA work assignment manager will be contacted. Air filter analysis will be delayed until (a) the individual bulk dyes (which are used to prepare the PAS) arrive at MRI and (b) basic dye information regarding the dyes handled during the monitoring period is received. The above measures will be observed unless instructions to the contrary are given by EPA.

9.0 EXTRACTION OF PERSONAL AND AREA FILTERS, CORRESPONDING AIR SAMPLING CASSETTES, AND FIELD AIR FILTER BLANKS

9.1 Using stainless steel forceps, carefully transfer the air filter and filter support from the cassette holder to a 4-oz amber glass jar. (Note: Each original air sampling cassette will contain only a filter support.)

9.2 Using the Repipettor® calibrated to 8.0 mL, pipet approximately 2 mL of the dye solvent into the cassette holder, dispensing the remaining ~ 6.0 mL of dye solvent into the jar.

9.3 Cap the cassette holder tightly and shake vigorously for 30 s. Transfer the cassette rinse solution to the 4-oz jar using a disposable glass pipette. Cap the jar securely.

FORM A-0. SAMPLE RECEIPT FORM

8856-A(01)

Plant ID: _____

Test Date: _____

Date Received: _____

Received by: _____

Were all samples received in good condition? Yes No (circle one)

Sample Storage: _____

Sample ID No.	Sample Description (Check One)			
	Original Air Filter Cassette	Personal Air Filter	Area Air Filter	Field Blank Air Filter

Were any blank filters received? Yes No (circle one)

Brand and Lot No.: _____

9.4 Place the jar in a shaker for 30 min. The shaker must oscillate at least once per second. After 30 min, visually examine the air filter and/or filter support. If the dye extraction appears incomplete, place the jar in an ultrasonic bath for a maximum of 10 min.

9.5 The extract solution must be analyzed within 45 min of initial contact with the dye solvent (i.e., Step 9.2). Dye analysis schedules (Form A-1) are prepared and followed for each set of filters/cassettes analyzed.

9.6 Only when deemed appropriate after consultation between MRI and EPA, should the pH 3 dye solvent (Step 6.4) be used to perform the extractions.

10.0 INDIVIDUAL DYE SOLUTION PREPARATION

Note: Dye solutions must be analyzed within 45 min of initial contact with the dye solvent (Step 10.2). Dye analysis schedules (Form A-1) are prepared and followed for each set of bulk dyes analyzed.

10.1 Accurately weigh (to the nearest 0.01 mg) approximately 40 mg of the bulk dye sample into a pre-tared 100-mL volumetric flask (low actinic). Record the weights on the Individual Dye Solution Preparation Form (Form A-2).

10.2 Add 50-70 mL of dye solvent to the flask and place in an ultrasonic bath for 10 min. After the flask has cooled to room temperature, dilute to volume with dye solvent and mix well.

10.3 Pipet 10.0 mL from the flask in Step 10.2 to a 250-mL low actinic volumetric flask. Dilute to volume with dye solvent and mix well.

10.4 Repeat Steps 10.1 through 10.3 for every dye in the dye analysis set.

10.5 If pH 3 dye solvent was used in Steps 9.2-9.5, use the pH 3 dye solvent (Step 6.4) to prepare the dye solutions.

11.0 PREPARATION OF SPIKED FILTER CONTROLS AND BLANKS (Forms A-3 and A-4)

11.1 Based on the average total dye estimate for the personal air filters (obtained from Step 16.5 and using Section I of Form A-3), calculate the total dye level which is 130% of that value. This calculated dye level is the target total dye concentration for the spiking standard solution.

FORM A-1. DYE ANALYSIS SCHEDULE

8856-A(01)

Date: _____ Plant(s): _____

Name(s): _____

LNB: page no. _____

Samples Analyzed:
Air Filters: _____
Bulk Dyes: _____
Spiked Filter Controls: _____

Extraction/dilution schedule					Analysis schedule					
Time	Plant I.D.	Sample I.D.	1° Diln.[a]		Time	Sample I.D.	Cycle no.	2° Diln.	A_{TOT}	Corrected[b] A_{TOT}
8:00					8:00					
:15					:15					
:30					:30					
:45					:45					
9:00					9:00					
:15					:15					
:30					:30					
:45					:45					
10:00					10:00					
:15					:15					
:30					:30					
:45					:45					
11:00					11:00					
:15					:15					
:30					:30					
:45					:45					
1:00					1:00					
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:30					:30					
:45					:45					
3:00					3:00					
:15					:15					
:30					:30					
:45					:45					
4:00					4:00					
:15					:15					
:30					:30					
:45					:45					
5:00					5:00					

[a] Sonicated all PASs and bulk dyes for 10 min after primary dilution (1° diln.). Agitated all filters on shaker for 30 min after primary dilution.

[b] Corrected A_{TOT} = A_{TOT} field air filter sample extract - A_{TOT} field air filter blank extract.

Form A-2
8856-A(01)

INDIVIDUAL DYE SOLUTION PREPARATION FORM[a]

Plant ID: _____ Prepared by: _____ Date: _____

Analytical Balance ID: _____ Model No.: _____ MRI No.: _____

Calibration Weights Used: _____ MRI No.: _____

Instrument Calibration: Post-Tare _____
Pre-Tare _____
_____ mg weight = _____ (accuracy = _____%)

Sample ID No.	Pre-Tare (g)	Post-Tare (g)	Dye Weight (g)

[a]See Dye Analysis Schedule for dilutions of the above weighed dyes.

11.2 Based on the dye usage data from the drug room site, determine the major use dye subset (i.e., those dyes which comprise at least 80% of the total dye quantity handled during the monitoring period). Using Section II of Form A-3, calculate the relative weight fraction for each of these dyes. Using this value and the target dye concentration value (calculated in Step 11.1), calculate target concentrations of each dye in the dye subset.

11.3 Using Form A-4 and the dye solvent, prepare the spiking standard solution containing all of these dyes in proportion to their weight fractions. The total dye concentration of the spiking standard solution should be as close to the target value (calculated in Step 11.1) as possible.

11.4 Place a blank air filter (i.e., filter blank from the same lot as was used at the drug room site) and pad into a three-piece 37-mm cassette and secure with the extension piece.

11.5 Using a 100-µL syringe, carefully dispense 100 µL of spiking standard solution from Step 11.3 onto the air filter. The volume delivered represents the 130% high level of total dyes calculated in Step 11.1. Deliver the spike in a scattered pattern over the entire filter. Set aside to dry.

11.6 Repeat Steps 11.4 and 11.5 for the duplicate high-level spiked filter control.

11.7 Deliver the same volume of spiking standard solution used in Step 11.5 into an empty 4 oz amber glass jar. Set aside to dry, leaving the jar uncapped. This is the reference standard at the high level.

11.8 Repeat Step 11.7 for the duplicate high-level reference standard.

11.9 Repeat Steps 11.4 through 11.6 for the spiked filter controls at the low dye level, using 50 µL of the spiking standard solution prepared in Step 11.3.

11.10 Repeat Steps 11.7 and 11.8 for the low-level reference standards using 50 µL of the spiking standard solution for each.

11.11 Repeat Steps 11.4 through 11.6 for the spiked filter blanks, using dye solvent in place of the spiking standard solution. Use the same volume used to spike the high-level spiked filter controls, e.g., 100 µL for each blank.

11.12 If the pH 3 dye solvent was used in Step 9.2, use the pH 3 dye solvent (Step 6.4) to perform Steps 11.3-11.11.

Form A-3

8856-A(01)

SPIKING STANDARD SOLUTION ESTIMATION FORM

Plant: _____
Date: _____ Name: _____

I. Total Dye Estimate on Personal Air Filters

Filter ID No.	Corrected A_{TOT}	Total Dye Estimate (μg)[a]	Average Total Dye Estimate (μg)

[a] where $\mu g = \frac{A_{TOT} \times \text{Dilution Volume}}{\bar{a}_s}$; $\bar{a}_s$ = _____

II. Target Values for Dye Subset Comprising ≧ 80-90% of Total Quantity Dye Handled:

Spiking Standard Solution's Target Total Dye Concentration =

$$\frac{130\% \times \quad \mu g \text{ (avg. total dye estimate)}}{100\ \mu L \text{ (high level spike volume)}} = \_\_\_\_\ \mu g/\mu L$$

Bulk Dye ID No.	% of Total Quantity of Dye Handled	Relative Weight Fraction = $\frac{\%\ \text{Total for Dye}}{\%\ \text{Total in Dye Subset}}$	Target Conc. (μg/μL) (Rel. Wt. Fraction x Target Tot. Dye Conc.)

% Total in Dye Subset = _____

Form A-4

8856-A(01)

SPIKING STANDARD SOLUTION PREPARATION FORM AND DATA ON SPIKED FILTERS

Plant: _____ Date: _____

Analyst: _____ LNB: page no.: _____

I. Spiking Standard Solution Preparation:

Analytical balance ID: _____
Calibration weight ID: _____
Calibration:
Post-Tare: _____
Pre-Tare: _____
_____ mg weight = _____ (accuracy = _____%)

II. Dye Weights (Use additional sheets if necessary)

Dilution Volume: _____

Post-Tare:	Post-Tare:	Post-Tare:
Pre-Tare: _____	Pre-Tare: _____	Pre-Tare: _____
Dye No. ___ wt.: _____	Dye No. ___ wt.: _____	Dye No. ___ wt.: _____
Actual Conc.: _____ µg/µL	Actual Conc.: _____ µg/µL	Actual Conc.: _____ µg/µL
Post-Tare:	Post-Tare:	Post-Tare:
Pre-Tare: _____	Pre-Tare: _____	Pre-Tare: _____
Dye No. ___ wt.: _____	Dye No. ___ wt.: _____	Dye No. ___ wt.: _____
Actual Conc.: _____ µg/µL	Actual Conc.: _____ µg/µL	Actual Conc.: _____ µg/µL

III. Spiked Filter Data

A. Filter ID: _____
No. of High-Level Spiked Filter Controls Prepared ___; Vol. Spiked ___ µL
No. of Low-Level Spiked Filter Controls Prepared ___; Vol. Spiked ___ µL
No. of Spiked Filter Blanks (DMSO only) Prepared ___; Vol. DMSO used ___ µL
No. of "Dry" High-Level Spiking Std. Controls Prepared___; Vol. Spiked ___ µL
No. of "Dry" Low-Level Spiking Std. Controls Prepared___; Vol. Spiked ___ µL

B. Date filters placed on sampling pumps: _____ by _____ (name)
Date filters and "dry" controls are to
be extracted/analyzed: _____ short-term
_____ long-term
_____ longest-term

No. of filters to be prepared/analyzed:

	Filters	"Dry" Controls
(short-term)	_____ high; _____ low; _____ blank	_____ high; _____ low
(long-term)	_____ high; _____ low; _____ blank	_____ high; _____ low
(longest-term)	_____ high; _____ low; _____ blank	_____ high; _____ low

11.13 Very low dye spiking levels (e.g., < 50 µg) result in weak absorbances which are difficult to quantitate with a high degree of confidence. As a result, recovery data based on low spike levels are more uncertain than those obtained for higher dye spiking levels. To address this potential problem, low personal air filter dye estimates will be bracketed by the high and low spiking levels, but spiking will also be done at an elevated level (e.g., 100 µg) as well.

12.0 EXPOSURE OF SPIKED FILTER CONTROLS, BLANKS, AND REFERENCE STANDARDS

12.1 Calibrate personal air pumps to draw air at a rate of 2 to 2.5 L/min.

12.2 Attach the dry spiked filter cassettes prepared in Section 11.0 to individual personal air pumps and draw air through the cassettes for 6 to 8 h. (Relative humidity in the immediate vicinity should be in the range of 30 to 70%. Fluorescent lights should be on during the exposure period.)

12.3 During Step 12.2, place the reference standards in the immediate vicinity of the aerating filters so they are exposed to the same lighting and humidity conditions.

13.0 ANALYSIS OF DYE SOLUTIONS

13.1 Spectrophotometer Operating Parameters

13.1.1 Turn main power switch to "on" position. Allow Visible-UV light source to warm up for at least 20 min.

13.1.2 Adjust the following parameters to their proper settings:

Source select: auto
Beam interchange: normal
Mode: autogain
Timer mode: off
Slit: 2.0 nm
Abs. zero suppression: 0
Range: 1.0 absorbance units full scale
Chart: off
% zero suppression: off
Period: 0.5 s
Pen: on
Autobaseline: on

Scanning rate: 1 nm/s
Chart display: 50
Scan: off
Function: off
Measurement: serial
Log A offset: 0.1
Concentration: turn knob completely to left
Analog signal output: 0-10 V[a]

13.1.3 Set the upper wavelength limit (e.g., 800 nm[b]) using the wavelength 1 dial. Set the lower wavelength limit (e.g., 330 nm[c]) using the wavelength 2 dial.

13.1.4 Using the "scan" dial, turn dial (+) or (-) to set the wavelength at the upper limit (e.g., 800 nm).

13.2 Analog/Digital Computer Interface Box Operating Parameters

Maximum Input Voltage: 10 V
Run Time: interface box run time must exceed actual analysis run time required to scan the wavelength range, e.g., at 1 nm/s from 800 nm to 330 nm, the analysis run time is 7.83 min. Therefore, an interface run time of > 8 min is required.
Sampling Time: 1 point/s

13.3 Absorbance Measurement of Filter Extract Solutions

13.3.1 Zero the spectrophotometer by placing the dye solvent (filtered using a 0.45 µm Gelman Acrodisc) in both the sample and reference cuvettes. Turn the autobaseline knob to "record" and check to see that red light comes on. (Autobaseline knob will automatically return to "on" position.) Adjust the balance knob to give an absorbance reading of ∿ 0.1000 on the digital display. Turn timer mode knob to "sample and wavelength," then back to "wavelength." Depress "step" button and scan the wavelength range (note: upon completion of the scan, the upper wavelength will automatically be reset). After the wavelength is reset, turn the timer mode knob to the "off" position. Adjust the balance knob to give an absorbance reading of ∿ 0.0500 on the digital display. Turn timer

a The maximum voltage must correspond to the maximum signal input of the A/D box.
b This setting can be anywhere in the range 750-800 nm.
c This setting can be anywhere in the range 300-350 nm. It is usually determined by the point where the grating in the spectrophotometer changes from the visible to the ultraviolet region.

mode knob to "sample and wavelength," then back to "wavelength." Depress "step" button, simultaneously starting the Nelson interface box, and scan the wavelength range to obtain a flat baseline from 800-330 nm. Empty the sample cuvette.

13.3.2 Analyze each filter extract solution, according to the Dye Analysis Schedule, by withdrawing the filter extract solution into a 10-mL disposable syringe. Attach a 0.45 µm Gelman Acrodisc filter onto the end of the syringe and filter the extract solution into the sample cuvette. Retain the remainder of the extract solution.

13.3.3 Scan the filter extract solution over the wavelength range, starting the A/D box at the beginning of the scan. Examine the scan during the analysis run. If the dye absorbance exceeds 1.0, abort the analysis run immediately[a] and make an appropriate dilution of an aliquot of the extract solution. Scan immediately. Document dilution and time deviations on the Dye Analysis Schedule.

13.4 Absorbance Measurement of Individual Dye Solutions

13.4.1 Zero the spectrophotometer according to the procedure outlined in Step 13.3.1.

13.4.2 Analyze each dye solution, according to the Dye Analysis Schedule, by withdrawing the dye solution from Step 10.3 into a 10-mL disposable syringe, attaching a 0.45 µm Gelman Acrodisc filter onto the end, and filtering into the sample cuvette.

13.4.3 Scan the dye solution over the same wavelength range used for the filter extract solutions, starting the A/D box at the beginning of the scan.

13.4.4 The maximum absorbance in the dye spectrum should be in the range of 0.1 to 1.0 absorbance units. If the dye absorbance does not fall within this range, abort the analysis run immediately and repeat Step 13.4.3 using an appropriate dilution of an aliquot from the flask in Step 10.2. Scan immediately. Document dilution and time deviations on the Dye Analysis Schedule.

---

a Turn the timer mode knob to "off" and stop the Nelson Box. Turn the scan knob to (+) side and increase scan rate until the wavelength is ~ 790 nm. Return scan rate to 1.0 nm/s. Turn scan knob to "off" position at 800 nm.

14.0 ANALYSIS OF SPIKED FILTER CONTROLS AND BLANKS

14.1 Extract all spiked filter controls and blanks as outlined in Steps 9.1-9.6.

14.2 Scan each spiked filter extract solution as outlined in Steps 13.3.1 through 13.3.3.

14.3 Dilute each reference standard with 8.0 mL of dye solvent, cap, and mix well.

14.4 Scan each reference standard solution in the same manner as the spiked filter extracts in Step 14.2.

15.0 Determination of Total Area Counts in a Dye Scan

15.1 For spectrum integration purposes, calculate, to the the nearest 0.01 min, the time required to scan the sample over the desired wavelength range. (Scanning from 800-380 nm at 1 nm/s takes 420 s = 7.00 min.)

15.2 Plot the data file in the "re-detect" mode of the Nelson integration software. Using the cursor, manually integrate the area above the baseline from 50 s (0.8333 min = 750 nm) to the calculated time (e.g., 7.00 min = 380 nm).

Note: The height of the baseline is determined by integration of the dye solvent over the calculated time period. In some cases, due to baseline drift, the baseline used for integrating data files may deviate slightly.

15.3 Obtain a hardcopy of the file integration for archiving purposes.

16.0 CALCULATIONS

16.1 Total Absorbance (A_{TOT}): The integration software from Nelson Analytical divides the maximum signal into 10^6 parts (counts). Since the full scale signal at any particular wavelength is equal to 1.0 absorbance units, there are 10^6 area counts per absorbance unit. Therefore, by dividing the total area counts (obtained in Section 15.0) by a factor of 10^6, a total absorbance value is obtained.

Note: A_{TOT} values for field air filter samples and air sampling cassettes are corrected for background absorbance by subtracting the A_{TOT} value for the corresponding field air filter blank(s). A_{TOT} values for individual bulk dye solutions are not subject to these background absorbances and therefore need no correction.

16.2 Spectral Absorptivity Constant (a_s) for Individual Dyes: Based on Beer's law, each individual dye's spectral absorptivity constant (a_s) is calculated using the formula:

$$a_s = \frac{A_{TOT}}{b \times c}$$

where b is the cuvette pathlength in cm (e.g., 1 cm), c is the dye concentration in µg/mL, and A_{TOT} is the total absorbance of the dye solution. The dye concentration will be expressed in terms of a commercial dye basis (i.e., assume 100% purity) and an active ingredient basic (i.e, using dye purity values supplied by ETAD).

16.3 Weighted Average Spectral Absorptivity Constant ($\bar{a}_s$) for the Dye Analysis Set: This value is obtained from the individual dye a_s constants in the dye analysis set, using the dye handling information to obtain the appropriate weight fraction for each component dye.

16.4 Dye Recovery Determination for Spiked Filter Controls: The recovery value for the group of major use dyes is calculated from the A_{TOT} values obtained from the spiked filter controls and the corresponding reference standard solutions. Percent recovery, R, is calculated using the formula:

$$R = \frac{A_{TOT} \text{ of Spiked Filter Control} - A_{TOT} \text{ of Spiked Filter Blank}}{A_{TOT} \text{ of Reference Standard}} \times 100$$

16.5 Total Dye Estimate on the Air Filter: This is calculated using the formula:

$$\text{Total Dye Estimate (µg)} = \frac{A_{TOT} \times V}{\bar{a}_s \times b}$$

where V is the volume of the filter extract solution in mL (A_{TOT}, a_s, and b are defined above).

16.6 Correction of Dye Estimate for Percent Active Ingredient of Dyes: The total dye estimate is also reported in terms of the active ingredient content from the individual dyes. This correction, made by using dye lot purity information provided by ETAD, is performed on all field air filters as well as their corresponding cassettes.

16.7 Correction of Dye Estimate for Extraction Efficiency: The total dye estimate is also corrected for the extraction efficiency of the analytical method. The overall average dye recovery value is used to make the correction. This correction is performed only on the field air filter samples.

16.8 Average Airborne Dye Concentration: This is calculated by dividing the total dye estimate (in mg) by the total volume of air sampled during the monitoring period (in m^3).

SUPPLEMENTAL B

DATA QUALITY OBJECTIVES

8-28-86

DATA QUALITY OBJECTIVES FOR THE TEXTILE DYE DRUG ROOM STUDY

I. STUDY OBJECTIVES

1. Estimate the distribution of the 30 breathing zone dye concentrations observed in the monitored plants (8 hour time-weighted concentration), specifically:

   a. the average breathing zone dye concentration at the plants
   b. the upper percentiles (85th percentile)
   c. confidence intervals for both estimates

2. Determine if dye concentration in breathing zone, adjusted for time in drug room is correlated with mass of dye weighed, number of weighings/shift, or other factors (see Appendix A), and if so, determine a functional relationship between concentration and factors.

3. Estimate the average, and distribution of, mass and number of weighings of individual solid dye compounds weighed out during a shift (averages and histograms need to be presented by dye class and aggregated)

4. Summarize selected drug room observations and general plant information in table form (see Appendix B for items to be summarized)

5. Obtain an extensive first-hand, qualitative view of drug room operations (the final result will be an individual industrial hygiene report for each of the 30 plants visited)

Objective 1 is the most important; items 2-5, all of roughly equal importance, are supplemental to objective 1 but will also enhance the knowledge of dye exposure. Besides the objectives of the study, other aspects of the data will be explored (see Appendix C).

II. BACKGROUND

About 1000 domestic textile dyeing or printing sites have been identified where there is potential for workers to be exposed to numerous powder dyes via inhalation or dermal routes. There are reasonable indications that some dyes or their metabolites

might be carcinogens or mutagens. Data which document potential exposure levels of workers associated with the weighing or mixing of powder dyes are limited, some of which were obtained from non-textile dyeing operations. The objective here is to conduct a well designed study of textile dye weighing rooms in order to improve the assessment of exposure (and associated risk) with the use of powder dyes in the American textile industry. This study is being sponsored jointly by the American Textile Manufacturers Institute (ATMI), the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD) and the Office of Toxic Substances (OTS) of EPA.

III. DATA COLLECTION APPROACH

A two phase approach was chosen for the study. The first phase was a mailed out questionnaire to 240 plants selected as a simple random sample from 1390 textile facilities thought to potentially use powder dyes. The first phase goals were: to make sure that the list of 1390 plants was not missing any major groups of dyeing plants (and thus provide a pool from which to draw that is not grossly unrepresentative); to provide valuable general information on drug room operations including data useful to industrial hygienists in preparing for in-plant monitoring; and possibly to provide assistance for stratified random sampling.

The second phase will be a stratified random sample with the two strata being: 1. Respondents to first phase questionnaire; 2. Non-respondents to first phase questionnaire. Plants will be selected with representation proportional to strata sizes. Actual in-plant airborne dye-level monitoring and drug room observation will take place in 30 plants at this phase. The two strata were chosen to separate first phase respondents from nonrespondents since there might be differences in the nature of exposure levels, and since it is desired that the final representation from the two groups reflect the actual population of dyeing plants.

Within each of the 30 plants selected, a two member team will record measurements and observations in the plant to satisfy the study objectives (see Appendices A and B for the approximately 40 items recorded and the Quality Assurance Project Plan for the methods). For one randomly selected dye weigher at each plant, a more extensive examination of practices and potential exposure will be conducted (only one weigher will be chosen to balance the needs of the six objectives between themselves in the presence of limited resources). This weigher will be observed and monitored during one randomly selected shift. The monitoring will take place during nearly an entire 8-hour period by personal monitoring equipment designed to collect solids from the air in the breathing zone of the selected worker. These will be laboratory analyzed to determine total dust mass and total dye mass. For the monitored weigher, observers will also record the mass of each powder dye

and chemical weighed, the total number of powder dyes weighed, the amount of time the weigher spends in the weighing area, and other qualitative and quantitative measurements. Also, the observers will record information on size of dyeing operation, cleanliness, ventilation, possible routes of exposure, and other qualitative information.

IV. DATA QUALITY OBJECTIVES

1. Sources of Variability

a. Error Associated with Chemical Analytical Procedures

The 95% confidence bounds for the measured dye level will differ from plant to plant (since essentially a unique material will be collected at each plant). These error bounds will also differ within any given plant depending on whether dyes measurement is on a commercial dye or an active ingredient basis. The potential range of variability is so great, in fact, that it is not feasible to predict the magnitude of the 95% confidence bounds for any dyes measurement until all of the component dyes have been analyzed.

After examination of the 95% confidence bounds for the results from each of the 24 plant sites in the study, it can be stated that these confidence bounds will generally be $\pm$ 70% of the reported value on a commercial dye basis and $\pm$ 80% on an active ingredient basis. The largest part of the uncertainty for any of the dye measurements is the use of a weighted average a_s constant to approximate the absorption characteristics of the dye mixture on the air filter. Other sources of error in the dye measurement, such as those due to systematic errors in the laboratory, method precision, or uncertainties in the dye recovery values, are relatively minor components of the total error estimates described above.

b. Sampling Error Associated with the Survey

The sampling error discussed here is the error introduced from selecting a sample of textile dyeing plants versus monitoring one shift for the entire population of textile dyeing plants.

The total sampling error introduced will depend upon the variability between plants. However, using the data collected by the National Institute for Occupational Safety and Health (NIOSH) and the CIBA-GEIGY dye firm, for three textile dyeing and one paper dyeing plant, the standard error expected for estimating the average exposure is approximately 15% of the estimated average (2 standard errors = $\pm$ 30% on this basis), due to the sampling error as described. For estimating the 85th percentile, a nonparametric 95% tolerance interval would utilize the second highest measured plant value as its upper bound (again, this includes error associated with the plant sampling procedure and not such things as chemical measurement error); however more precise intervals are likely to be obtained through the use of probability distributions to estimate the 85th percentile and its confidence interval.

c. Nonrandom Error Associated with the Survey

There are several potential sources of error (i.e., nonsampling errors) besides the four random-type errors, such as refusal to allow monitoring (this is a voluntary study), and possibly encountering artificially clean conditions at the time of the site visit. Only qualitative, subjective judgments can be made about these factors. However, to help preserve the usefulness of the study, an energetically pursued target of obtaining entry to at least 75% of selected plants has been set. But given the existing data, a lower bound of 60% monitoring acceptance has been set. Should the acceptance rate drop below 60% the current objectives will not be met and a reevaluation of the approach will be made.

2. Completeness

The survey collection is considered complete when 30 plants have been monitored, with a second phase response rate of at least 60%, and all 30 chemical analyses are determined to be possible.

3. Comparability

All chemical analyses will be done at a single laboratory, Midwest Research Institute. All field collection will be conducted by Health and Hygiene, Inc. under contract with participating industry trade associations, and by the firm PEI Associates, Inc. under contract with EPA. All observational data will be collected via answers to direct questions or open ended questions with suggested answers provided, wherever possible, to maintain comparability between responses at the 30 plants. Also, directing the scope of the answers on the data collection form insures that the reported observations actually answer the questions that EPA and the dye industry want answered.

No other such data collection has been done in the past, nor has the method for estimating total amount of dye been used before. The earlier studies by NIOSH and CIBA-GEIGY measured the amount of a limited number of specific dyes, and only three of the plants were textile mills.

4. Representativeness (assumptions and universe of interest)

a. The stratified simple random sampling plan will provide estimates on a national scale and with an (approximately) known amount of uncertainty due to plant sampling.

b. Plants were considered to be within the scope of the study if they dyed or printed textiles using powder dyes with mechanical equipment. Any amount of dyeing was considered within the scope of the study.

Drug rooms are considered within the scope of the study if any amount of weighing takes place there on a regular basis.

Weighers are the only type of worker represented in the study. Interest focuses on the weigher since he or she is the one handling the largest amount of powder dyes. No direct statement can be made about other workers except that they are assumed to be exposed to

substantially lower levels of dye.

c. Although the primary goal is to obtain estimates of average plant levels and the distribution of plant levels, it is possible to obtain estimates of average weigher exposure by randomly selecting a worker within the plant. For example, statements could be made about the 85th percentile of worker exposure by weighing plant estimates by the number of weighers in each plant. This would result in a statement such as "It is estimated that 85% of weighers are exposed to levels lower than xxx mg/m^3 during an 8-hour shift." Note that this differs from the estimate of 85th percentile of plant levels which results in a statement such as: "It is estimated that the average exposure in 85% of textile dyeing and printing plants is less than zzz mg/m^3, time-weighted 8-hour average" (xxx and zzz determined from study).

APPENDIX A SECONDARY MEASUREMENTS TO EXAMINE FOR ASSOCIATION WITH DYE LEVEL

The dye concentration adjusted for time in drug room, (total dye collected)(time personal monitor on/time in drug room), will also be examined for correlation with several other variables of secondary importance. These are:

1. Production volume of textiles (pounds per year), from mailed-out questionnaire
2. Management of dye house (vertical, commission or both), from mailed-out questionnaire
3. Management of dye house (public or private), from mailed-out questionnaire
4. Color index class of dyes used, for any dye used during observed shift by monitored weigher (acid, basic/cationic, reactive, direct, disperse, other), from site visit log as classified by Chemical Engineering Branch
5. Total number of dyeing and printing machines serviced by monitored weigher (average of beginning and end of shift numbers), from site visit questionnaire
6. Number of fiber types dyed or printed (acrylic/modacrylic, rayon/cotton, nylon, polyester, other) at site, taken from mailed-out questionnaire

It should be noted that a data set of only 30 observations is likely to result in some spurious large correlation when many correlations are calculated. Thus the correlations of this section will be interpreted in that light.

APPENDIX B SUMMARY TABLES

1. ON-SITE QUESTIONNAIRE

Summary tables will be presented for several other variables collected from the on-site questionnaire with categorized responses. The variables to be tabled are:

1. Number of plants by number of weighers, at all shifts during a typical 24 hour period
2. Number of plants by pounds of dye weighed during shift
3. Number of plants by number of dyes weighed during shift
4. Number of plants by number of dye weighings during shift
5. Number of workers by amount of time in drug room
6. Number of workers that used dust mask during site visit
7. Number of workers that used respirator during site visit
8. Number of workers that smoked in drug room area during site visit
9. Number of workers that ate in drug room area during site visit

2. MAILED-OUT QUESTIONNAIRE

As an appendix in the final report, the following variables will be tabulated from the first-phase, mailed-out questionnaire:

*11. Number of textile dyeing plants in each EPA geographical region
12. Number of plants by number of dyeing or printing operations within the company that owns the selected plant
@13. Number of plants by management of house (vertical, commission, or both)
@14. Number of plants by management of house (public, or private)
@15. Distribution of plants by product volume
16. Number of plants by product line (carpet, yarn, fabric, other)
#17. Number of plants by type of dyeing or printing equipment available (batch, semi-continuous/continuous, printing)
@18. Number of plants by fiber dyed or printed (acrylic/modacrylic, rayon/cotton, nylon, polyester, other)
@19. Number of plants by color index class of powder dye (acid, basic/cationic, reactive, direct, disperse, other)
20. Number of plants by number of dyes weighed per 24 hours (less than 10, 10 to 20, over 20)
&21. Number of plants by pounds of dye used per 24 hours (less than 50, 50 to 200, over 200)
&22. Number of plants by number of powder dye weighings per 24 hours (less than 50, 50 to 500, over 500)
23. Number of plants by number of dye weighing rooms (1 room, 2 or more rooms)
24. Number of plants by number of worker shifts per 24 hours (1, 2, 3)
25. Number of plants by number of operating days per week (1 to 4, 5, 6 or 7)
26. Number of plants by number of employees exposed to powder dyes (1, 2, 3, 4 or more)

Notes

* This is also tabulated from site visit data
\# Similar information is also collected in the on-site questionnaire and is used in the secondary correlation analysis (see Appendix A)
@ A portion of the data on this variable will be used in the secondary correlation analysis (see Appendix A)
& Similar information also collected in the on-site questionnaire and is used in the primary correlation analysis

APPENDIX C NOTES ON STUDY OBJECTIVE 1

Although the primary objective of the study is as stated in objective 1, the approach chosen warrants discussion.

1. The main objective and procedure

One worker (dye weigher) will be monitored at each of the 30 plants visited. This measured value will be used to represent a typical measurement from that plant. These 30 breathing zone dye concentrations will be used for an estimate of the distribution of dye concentrations typically found in plants across the U.S.

2. Another way of looking at exposure distribution which will be presented in a report appendix

Note that the above procedure focuses on levels typically found in textile dyeing plants. Another approach is to look at the distribution of weigher (worker) exposures. This implies that a plant with three (3) workers should receive three (3) times as much weight as a plant with one (1) worker, since the workers there represent three (3) times as much of the population. This approach has considerable appeal; however, practical problems are discussed in Section 4 of this Appendix.

3. Distinction with the main objective

Although Section 2 of this Appendix discusses the main difference between the chosen procedure focusing on typical levels found in plants and the alternative approach, this section elaborates upon the distinction.

In contrast with objective 1, the alternate approach takes into account the number of weighers working at each plant and is weighted according to the number working at the monitored plant during a typical 24-hour period. For example, statements could be made about the 85th percentile of worker breathing zone dye concentration by weighing plant estimates by the number of weighers in each plant. This would result in a statement such as "It is estimated that 85% of weighers are potentially exposed to concentrations lower than xxx mg/m^3 during an 8-hour shift." Note that this differs from the estimate of the 85th percentile of plant levels (objective 1) which results in a statement such as: It is estimated that the average concentration in 85 percent of textile dyeing and printing plants is less than zzz mg/m^3, time-weighted 8-hour average" (xxx and zzz determined from study).

4. Reason for chosen emphasis

Although it would be ideal to sample every weigher at every shift of the sampled plants, it is not feasible for several reasons:

- o cost: monitoring more than one weigher per visit would raise laboratory and site visit costs dramatically. At least one extra person would need to travel to the plant site to record the dyes weighed by an extra weigher, the relative amounts of each dye weighed, set up the personal air samplers, and collect specimens of each dye weighed by the additional weigher. The laboratory would then need to develop a new procedure to chemically analyze the mixture of dyes weighed by the second worker.

- o personnel maximum: monitoring workers by having more than two (2) observers per site is often not feasible due to limited workspace at the sites. Furthermore, cooperation in this voluntary study could be impaired by requesting to send a larger delegation.

- o balance with other objectives: sending a larger team to each site would allow for some information on the importance between worker variability and allow for a somewhat better estimate of the distribution of breathing zone dye concentrations associated with the population of dye weighers. However, much of the study concentrates on observations made on conditions and possible routes of exposure at the plant/drug room visited (i.e., more dye measurements would not necessarily improve these aspects of the study appreciably); the potential routes of exposure on a plant by plant (or drug room by drug room) basis are also of direct interest.

- o correlation between reported dye concentrations at the same plant: simply monitoring two workers at the same plant does not provide for two independent estimates of dye concentration due to the nature of the chemical analysis method. That is, the error in the chemical measurement is such that repeated measurements at the same site will tend to be repeatedly too high or too low if the same dyes tend to be used (and, less importantly, that the mechanism for exposure remains the same). This is due to the fact that the proportional amount of dyes in the mixture on the filter is not known, so that the error in using the average absorbance value tends to be in the same direction for repeated chemical analyses of similar mixtures (also, the percent recovery estimate will have a similar tendency to be repeatedly too high or too low for the same unknown mixture). However, this should cancel out when estimating the 30-plant average.

SUPPLEMENTAL C

LETTERS, TO ENCOURAGE PLANT PARTICIPATION

AMERICAN TEXTILE MANUFACTURERS INSTITUTE, INC.
1101 CONNECTICUT AVENUE, N.W., SUITE 300, WASHINGTON, D.C. 20036
TWX: 710-822-9489
TEL: 202 862-0500

November 18, 1985

Dear

The American Textile Manufacturers Institute (ATMI) and the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD) are performing a study to determine the extent to which textile workers may be exposed to dye dust when weighing and mixing powder dyes. This study is being done in cooperation with the U.S. Environmental Protection Agency (EPA).

Your plant is one of 200 textile dyeing or printing sites which has been randomly selected for surveying. The purpose of this preliminary survey is to gather information about production capabilities, fibers processed, product lines, dyeing/printing operations and prevailing practices of worker protection in order to obtain a broad picture on the use of dyestuffs for textiles and the potential for worker exposure. From these 200 companies, 30 will be selected as representing typical plants in the textile dyeing/printing industry. They will be requested to permit voluntary monitoring using personal monitors of the workers in drugrooms by a private consulting firm, Health and Hygiene Company of Greensboro, North Carolina, who will be accompanied by an EPA representative. The professional staff of Health and Hygiene have considerable textile experience and are highly suited and well qualified for this assignment.

Monitoring data will be used by industry and EPA as a basis for estimating potential levels of exposure of workers to powder dyes. No regulatory action will be generated against individual participants as a

result of this data gathering program. Data will be coded by ATMI and submitted only in coded form to EPA. After the survey forms have been reviewed for completeness, the code will be destroyed.

You are requested to voluntarily complete the enclosed survey forms which will provide information which is necessary to initiate the program. To aid you, we have included with the survey forms, instructions and an example of a completed survey. If any proprietary business information is provided which should be treated as confidential, so indicate by encircling the Y on the confidential business information (CBI) line at the top of the appropriate page(s). Your request will be respected. We would appreciate hearing from you by December 5, 1985. If you have any questions, please contact Maggie Dean, (202) 862-0580. We extend our thanks for your cooperation and assistance in this industry-government cooperative program.

Sincerely,

Carlos Moore

Carlos Moore
Executive Vice President

Enclosure
OIS/7T/1-2/CS

AMERICAN TEXTILE MANUFACTURERS INSTITUTE, INC.
1101 CONNECTICUT AVENUE, N.W., SUITE 300, WASHINGTON, D.C. 20036
TWX: 710-822-9489
TEL: 202.862-0500

December 20, 1985

Dear Mr. Garton:

This confirms our recent telephone conversation relative to the Textile Drug Room Monitoring Study. Your participation in this survey is of the utmost importance. The answers you provide will help us move to the second phase of a survey whose ultimate goal is to gather information that will be shared with the U.S. Environmental Protection Agency (EPA) to characterize the workplace exposure levels of the dyes in dye weighing and mixing rooms of textile dyehouse and printing operations.

The value for an exposure level which EPA currently uses in existing chemical assessments is based on the level detected in the leather industry, which is probably a higher number than might be observed in the textile industry. Ultimately, in this survey, the measured levels of dye in the air of 30 drug rooms of the sampled textile dyeing and printing plants will provide a more representative, and most likely a lower, value than the one the EPA is currently using. This survey has the support of ATMI and also the Dyestuff Manufacturers. The conclusions drawn from the current survey will affect all members of the textile and dye industry.

We look forward to your completed questionnaire. Please feel free to call Maggie Dean of ATMI at (202) 862-0850 if you have any questions.

Sincerely,

Carlos Moore
Executive Vice President

CRAFTED WITH PRIDE IN U.S.A.

AMERICAN TEXTILE MANUFACTURERS INSTITUTE, INC.
1101 CONNECTICUT AVENUE, N.W., SUITE 300, WASHINGTON, D.C. 20036
TWX: 710-822-9489
TEL: 202/862-0500

This letter is in regard to a contact you had recently with WESTAT relative to a joint industry/EPA sponsored study of textile drug rooms. We want you to know ATMI is actively supporting this effort and your participation in the survey is very important. The answers you provide will help us move to the second phase of the study, which is to assess exposure levels to dyes in weighing and mixing rooms of textile dyehouse and printing operations. The information is intended to help EPA in evaluating applications for manufacture of new dyes. You perhaps are aware that the Toxic Substances Control Act requires the agency to assess potential health risks before approving the manufacture of any new chemicals or chemical compounds.

EPA's current strategy for assessing dyes is based on exposure levels found in the leather industry thereby resulting in fewer approvals of new dyes. Ultimately, this study will establish levels representative of dye in the air of 30 textile drug rooms. We expect it to support our position that exposure levels in the textile industry are much lower than those assumed by EPA.

We urge you to join in this study and we look forward to receiving your completed questionaire. If you have any questions, please feel free to call Maggie Dean of ATMI at (202) 862-0580.

Sincerely,

O'Jay Niles
Director, Government
Reglations/Regulatory

AMERICAN TEXTILE MANUFACTURERS INSTITUTE, INC.
1101 CONNECTICUT AVENUE, N.W., SUITE 300, WASHINGTON, D.C. 20036
TWX: 710-822-9489
TEL: 202 862-0500

May 23, 1986

Thank you for participating in the first phase of the joint study with ATMI, the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD) and the U.S. Environmental Protection Agency (EPA) to determine worker exposure to dye dust. The second phase of this project involves the measurement of actual exposure levels in a representative group of U.S. textile plants. Your plant is one of thirty textile plants selected at random for the second part of this important study. The validity of the study depends upon achieving a high level of participation from the 30 plants. We encourage your participation for this reason.

The next phase will be conducted on-site by a field team of two certified industrial hygienists. Total time on-site over a 2-day period will be about 14-16 hours. Every effort will be made to minimize any inconvenience to your plant from the visit. In fact, the validity of the study can only be maintained by avoiding disruption of workplace activities. An individual site report will be prepared and you will receive a copy following completion of the study. The overall survey results will be presented in a 30-site composite report, individual plants will not be identified in this final report.

More details of the study are provided in the attached statement on objectives and protocol.

There will be some direct benefits to participants in this study; they include:

1. A confidential report which characterizes industrial hygiene practices at the plant and measures dye concentration levels in the drug room.

2. An opportunity for participating companies to compare their results with those of other participating plants.

3. The current EPA approach, which is based on worst case assumptions, is excluding from the U S. market many new dyes which are available to U S. competitors abroad. A direct objective of this study is to secure a more realistic assessment of potential risks.

4. A final report which characterizes the industry based on consolidation of results from the questionaire and monitoring study of all participants. This report will not identify specific sites.

Within the next few days Ms. Maggie Dean, ATMI's Director of Safety, Health and Environment, will phone you to discuss arrangements and possible dates for the two-day on-site visit by the field study team. Currently, we plan to begin site visits the week of June 16. Shortly after, Dr. William Dyson will phone you to schedule the on-site visit and answer any specific questions about the monitoring procedure. Your participation in this second and final phase of the study is important and I personally encourage you to take part. If you have any questions, please contact Maggie at (202) 862-0580.

Sincerely,

Carlos Moore
Executive Vice President

CM:jt

AMERICAN TEXTILE MANUFACTURERS INSTITUTE, INC.
1101 CONNECTICUT AVENUE, N.W., SUITE 300, WASHINGTON, D.C 20036
TWX: 710-822-9489
TEL 202/862-0500

January 15, 1987

Dear

A study is currently being sponsored jointly with ATMI, the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD) and the U.S. Environmental Protection Agency (EPA) to determine worker exposure to dye dust. Phase I surveyed 200 textile dyeing or printing sites to obtain a broad picture on the use of dyestuffs for textiles and the potential for worker exposure. The study will not be used to develop regulations. The objective is to provide a more realistic assessment of textile workers exposure to powdered dyes which we hope will help dye manufacturers pass premanufacturing notification requirements for new dyes. Strict confidentiality of plant identification will be kept by ATMI and not be available to EPA.

The next phase will be conducted on-site by a field team of two certified industrial hygienists and an EPA representative. Total time on-site over a 2-day period will be about 14-16 hours. Every effort will be made to minimize any inconvenience to your plant from the visit. In fact, the validity of the study can only be maintained by avoiding disruption of workplace activities. An individual site report will be prepared and you will receive a copy following completion of the study. The overall survey results will be presented in a 30-site composite report; individual plants will not be identified in this final report.

More details of the study are provided in the attached statement on objectives and protocol.

There will be some direct benefits to participants in this study; they include:

1. A confidential report which characterizes industrial hygiene practices at the plant and measures dye concentration levels in the drug room.

2. An opportunity for participating companies to compare their results with those of other participating plants.

3. The current EPA approach, which is based on worst case assumptions, is excluding from the U S. market many new dyes which are available to U S. competitors abroad. A direct objective of this study is to secure a more realistic assessment of potential risks.

4. A final report which characterizes the industry based on consolidation of results from the questionaire and monitoring study of all participants. This report will not identify specific sites.

Your participation in this second and final phase of the study is very important to the project's success and we personally encourage you to take part. We plan to conduct site visits during the next few months and will phone you next week to secure your agreement and answer any questions about the study and monitoring procedures.

Sincerely,

Maggie Dean
Director
Safety Health and Environment
ATMI

Eric Clarke
Executive Secretary
Ecological and Toxicological
Association of the Dyestuffs
Manufacturing Industry

TEXTILE DRUG ROOM MONITORING STUDY (TDRMS)

OBJECTIVES AND MECHANISM

About 1000 domestic textile dyeing or printing sites have been identified where there is potential for workers to be exposed to numerous powder dyes via inhalation or dermal routes. There are reasonable indications that some dyes (both new submissions and existing) or their metabolites might be carcinogens or mutagens. Data which document potential exposure levels of workers associated with the weighing or mixing of powder dyes are limited, some being derived from other than the textile industry. Our objective is to conduct a well-designed study of textile dye weighing rooms in order to improve the assessment of exposure (and associated risk) with the use of powder dyes in the American textile industry. This study is being sponsored jointly by the American Textile Manufacturers Institute (ATMI), the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD) and the Office of Toxic Substances (OTS) of EPA.

The mechanism of accomplishment will consist of monitoring one randomly selected dye weigher in each of 30 randomly selected sites which use solid dyestuffs in the dyeing or printing of textile fibers. The on-site field team will consist of two certified industrial hygienists. One will be an employee of Health and Hygiene, Inc. (H+H) of Greensboro, NC. The other will be an employee of PEI Associates, Inc. (PEI) of Cincinnati, OH.

On site observers will record the identity of each solid substance weighed by the person being monitored, the number of weighings of each, the total mass of each substance weighed and the duration of time that the worker is within the weighing area. They will characterize each work area in respect to number of dyeing/printing units in operation, materials flow, cleanliness and ventilation including engineering controls. They will also characterize each person being monitored relative to work habits, work history and protection equipment utilized including clothing and personal protective controls.

Over an 8 hour period, solids will be collected from the air in the breathing zone of each selected worker. At a later time, collection filters will be analyzed for total dust and for total dye. Observers will also collect analytical standardization controls consisting of samples of each dye and other materials which may interfere with the analysis.

The field team is expected to be at each site for a period no greater than 2 consecutive days. Projected activities and duration will be as follows:

Sequence	Activities	Duration
Pre-Monitoring	Gain site familiarity Characterize site	2-4 hrs
Monitoring	Monitor worker Record Dyes/chemicals weighed Characterized worker	8 hrs

Post-monitoring	Collect samples	2-4 hrs
	Review and completion of data/sample collection	
	Review with plant offical	

Observers will preserve the confidentiality of operations and formulations of the sites visited. As a courtesy and to ensure validity, they will conscientiously refrain from any interference in workers' performance of duties. Within one month of the monitoring date, observers will issue a joint report which characterizes each site and the monitored individual.

June 12, 1987

Dear :

The American Textile Manufacturers Institute, Inc., the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry and the Office of Toxic Substances of the U.S. Environmental Protection Agency, thank you for participating in the joint industry-government textile drug room monitoring study.

The monitoring phase of the survey of dustiness in textile drug rooms of 24 randomly selected volunteer plant sites was completed in May 1987. Reports are now being prepared characterizing, but not identifying, each site which participated. A copy of the report on your facility will be forwarded to you within the next two months. In addition, at a later date, you will recieve a copy of the final report which summarizes data gathered from all participating sites without identifying any individual participants.

We believe this study will provide new insights on worker exposure to dyes in textile drug rooms. Again, we greatly appreciate the time and assistance you provided. The study could not have been done without your cooperation and that of the other volunteer participants.

Very truly yours,

Maggie Dean
Director
Safety, Health and Environment
ATMI

MMD/sgt

Eric Clarke
Executive Secretary
Ecological and Toxicological
Association of the Dyestuffs
Manufacturing Industry

☆U.S. GOVERNMENT PRINTING OFFICE:1990 -725-029/

SUPPLEMENTAL D

FIRST PHASE QUESTIONNAIRE

PLEASE GIVE THE ACTUAL PHYSICAL LOCATION OF THE FACILITY RATHER THAN THE MAILING ADDRESS (IF DIFFERENT FROM LABEL).

Street Address: _____

City: _____

STREET OR P.O. BOX

State: _____ Zip Code: |__|__|__|__|__|

PLEASE RECORD THE NAME AND ADDRESS OF THE PARENT COMPANY (CORPORATE HEADQUARTERS) OF THIS FACILITY. IF THIS FACILITY HAS NO PARENT COMPANY, PLEASE WRITE 'NOT APPLICABLE' IN THE SPACE PROVIDED FOR PARENT COMPANY NAME.

Parent Company Name: _____

Address: _____

STREET

_____ |__|__|__|__|__|

CITY STATE

PLEASE RECORD THE NAME, TITLE AND PHONE NUMBER OF THE PERSON WHO MAY BE CONTACTED FOR FURTHER INFORMATION.

Name of Contact: _____

Title: _____

Phone Number: (___)_____

ID#: |__|__|__|__|__|

GENERAL INSTRUCTIONS

- All information requested concerns solid (powder or granular) dyes only. Exclude information for operations using only pigments or liquid dyes.

- Located in the upper right corner of each page of the questionnaire is a box in which you may indicate the presence of confidential business information (CBI) on that page. Please circle Y (yes) or N (no) to indicate the presence or absence of proprietary information on each page.

- Most questions will have specific instructions to assist you in their completion. If you have a situation not covered by the instructions, please don't hesitate to call the number in the box below for assistance.

- An identification number will be assigned by ATMI as a reference in order to protect the identity of each participant. This number will appear in the top right corner of each page.

PLEASE COMPLETE THIS QUESTIONNAIRE AS SOON AS POSSIBLE. AS SOON AS IT HAS BEEN COMPLETED, RETURN IT TO _____ IN THE ENCLOSED, POSTAGE-PAID ENVELOPE. IF YOU HAVE ANY QUESTIONS ABOUT HOW TO COMPLETE THE QUESTIONNAIRE, PLEASE CALL _____ AT ( ) _____.

PLEASE RECORD THE FOLLOWING INFORMATION ONLY IF DIFFERENT FROM LABEL ABOVE.

Name of Dyeing or Printing Facility: _____

Mailing Address: _____
STREET OR P.O. BOX

_____ |__|__|__|__|__|
CITY STATE ZIP

NOVEMBER 1985

CBI: Y N
ID#: |__|__|__|__|__|

1. What are the total number of sites within the entire corporation where dyeing or printing operations are performed? (PLEASE COUNT EACH SEPARATE DYE HOUSE OR PRINT SHOP.)

   # OF SITES: _____

2. Is this particular facility publicly or privately owned? (MARK AN 'X' IN THE APPROPRIATE BOX.)

   PUBLIC |___| PRIVATE |___|

3. Does this facility operate on a vertical (integrated) or commission basis? (MARK AN 'X' IN THE APPROPRIATE BOX.)

   VERTICAL |___| COMMISSION |___|

CBI: Y N
ID#: |__|__|__|__|__|

PRODUCT LINES DYED OR PRINTED

4. In the table below, please specify the following information:

COLUMN 1: IN THIS COLUMN PLEASE MARK AN 'X' IN THE BOX CORRESPONDING TO EACH PRODUCT EITHER DYED OR PRINTED AT THIS FACILITY. PRODUCT LINES WHICH DO NOT FIT ANY OF THE INDICATED CATEGORIES SHOULD BE ENTERED ON THE OTHER (SPECIFY) LINES.

COLUMN 2: IN THIS COLUMN, INDICATE ON THE APPROPRIATE LINE THE AMOUNT OF EACH PRODUCT WHICH IS SUBJECTED TO A DYING OPERATION USING POWDER DYES. THIS SHOULD BE POUNDS OF GOODS PROCESSED. IF IT IS INCONVENIENT FOR YOU TO SPECIFY THE NUMBER OF POUNDS PLEASE MARK AN 'X' IN THIS BOX [ ] AND RECORD YOUR ANSWER IN PERCENT OF TOTAL PRODUCTION. THE TOTAL FIGURE AT THE BOTTOM OF THE COLUMN SHOULD BE GIVEN IN POUNDS OF FIBER DYED (WITH POWDER DYES) PER YEAR.

COLUMN 3: IN THIS COLUMN, INDICATE ON THE APPROPRIATE LINE THE AMOUNT OF EACH PRODUCT WHICH IS PRINTED WITH POWDER DYES. THIS SHOULD BE AS POUNDS OF GOODS PROCESSED. IF IT IS INCONVENIENT FOR YOU TO SPECIFY THE NUMBER OF POUNDS PLEASE MARK AN 'X' IN THIS BOX [ ] AND RECORD YOUR ANSWER IN PERCENT OF TOTAL PRODUCTION. THE TOTAL FIGURE AT THE BOTTOM OF THE COLUMN SHOULD BE GIVEN IN POUNDS OF FIBER PRINTED (WITH POWDER DYES) PER YEAR. IF ANY GOODS ARE BOTH DYED AND PRINTED AT THIS SITE, LIST THE QUANTITIES IN BOTH COLUMN 2 (DYE) AND COLUMN 3 (PRINT) BRACKETED BY PARENTHESES.

PRODUCT LINE	COLUMN 1 PRODUCT DYED OR PRINTED	COLUMN 2 AMOUNT DYED PER YEAR IN POUNDS	COLUMN 3 AMOUNT PRINTED PER YEAR IN POUNDS
a. Staple	|__|	|_____|	|_____|
b. Yarn/thread	|__|	|_____|	|_____|
c. General apparel - woven	|__|	|_____|	|_____|
d. General apparel - knitted	|__|	|_____|	|_____|
e. Carpet/rug (include automotive)	|__|	|_____|	|_____|
f. Other home furnishings	|__|	|_____|	|_____|
g. Other transportation fabrics	|__|	|_____|	|_____|
h. Other pile fabrics	|__|	|_____|	|_____|
i. Outerwear/cloaking fabrics	|__|	|_____|	|_____|
j. Towels/terry cloth	|__|	|_____|	|_____|
k. Sheets/linens	|__|	|_____|	|_____|
l. Linings/woven continuous filament	|__|	|_____|	|_____|
m. Narrow fabrics	|__|	|_____|	|_____|
n. Light weight fabrics	|__|	|_____|	|_____|
o. Hosiery/intimate wear	|__|	|_____|	|_____|
p. Fabricated goods (as sweaters, socks, etc.)	|__|	|_____|	|_____|
q. Other woven fabrics (SPECIFY)	|__|	|_____|	|_____|
_____	|__|	|_____|	|_____|
_____	|__|	|_____|	|_____|
_____	|__|	|_____|	|_____|
r. Other knitted fabrics (SPECIFY)			
_____	|__|	|_____|	|_____|
_____	|__|	|_____|	|_____|
_____	|__|	|_____|	|_____|
s. Other substrates (SPECIFY)			
_____	|__|	|_____|	|_____|
_____	|__|	|_____|	|_____|
_____	|__|	|_____|	|_____|
Total pounds per year		|_____|	|_____|

CBI: Y N
ID#: |__|__|__|__|__|__|

5. Which of the following <u>fibers</u> are dyed or printed at this facility? (MARK AN 'X' IN THE BOX FOR EACH FIBER USED. IF FIBERS OTHER THAN THOSE LISTED ARE USED, PLEASE SPECIFY IN THE SPACE PROVIDED.)

Acrylic/Modacrylic. . . |__|	Acetate . . . . . |__|
Rayon . . . . . |__|	Wool. . . . . |__|
Cotton. . . . . |__|	Other (SPECIFY)
Nylon . . . . . |__|	_____. . |__|
Polyester . . . . . |__|	_____. . |__|

6. Which of the color index classes of powder (solid) dyes are used at this facility? (MARK AN 'X' IN THE BOX FOR EACH DYE CLASS USED. IF DYE CLASSES OTHER THAN THOSE LISTED ARE USED, PLEASE SPECIFY IN THE SPACE PROVIDED.)

Acid (include metallized). . . |__|	Chrome/Mordant. . . . . |__|
Basic/Cationic . . . . . |__|	Sulfur. . . . . |__|
Reactive . . . . . |__|	Vat . . . . . |__|
Direct . . . . . |__|	Other (SPECIFY)
Disperse . . . . . |__|	_____. . |__|
Naphthol/Azoic . . . . . |__|	_____. . |__|

CBI: Y N
ID#: |__|__|__|__|__|

7. How many units of the following types of dyeing or printing equipment are used at this facility on a regular basis? (RECORD THE NUMBER OF PRODUCTION SIZE UNITS OF EQUIPMENT USED AT THIS FACILITY IN THE BOX PROVIDED. IF EQUIPMENT OTHER THAN THAT LISTED IS USED, PLEASE SPECIFY IN THE SPACE PROVIDED.)

A. Batch

Beck	|_____|	Rotary	|_____|
Beam	|_____|	Stock/Top	|_____|
Jet	|_____|	Skein	|_____|
Jigg	|_____|	Other (SPECIFY)	
Package	|_____|	_____	|_____|
Paddle	|_____|	_____	|_____|

B. Continuous/Semi-Continuous

Pad	|_____|	Warp Dye	|_____|
Flood	|_____|	Other (SPECIFY)	
J-Box	|_____|	_____	|_____|
Spray	|_____|	_____	|_____|

C. Print

Flat Bed Screen:		Space Dye	|_____|
Hand	|_____|	Polychromatic	|_____|
Machine	|_____|	Other (SPECIFY)	
Rotary Screen	|_____|	_____	|_____|
Roller	|_____|	_____	|_____|

CBI: Y N

ID#: |__|__|__|__|__|

FREQUENCY AND VOLUME OF POWDER DYE HANDLING

8. In the table below, please specify the following information:

COLUMN 1: IN THIS COLUMN, PLEASE RECORD YOUR BEST ESTIMATE OF QUANTITIES SPECIFIED FOR A TYPICAL DAY/WEEK BASED ON PRODUCTION FIGURES OVER THE PAST YEAR.

COLUMN 2: IN THIS COLUMN, PLEASE RECORD YOUR BEST ESTIMATE OF BOTH THE LOW AND HIGH LEVELS OF QUANTITIES SPECIFIED BASED ON PRODUCTION OVER THE PAST 12 MONTHS. DO NOT INCLUDE EXTREMES WHICH ARE THE RESULT OF ABNORMAL CONDITIONS SUCH AS ZERO PRODUCTION DURING A CHRISTMAS WEEK CLOSING, OR EXCESSIVELY HIGH PRODUCTION RESULTING FROM A "CATCH-UP WEEK" FOLLOWING AN EXTENDED POWER OUTAGE.

	Column 1 Typical	Column 2 Low/High for Year
a. Number of individual powder dyes weighed per 24 hour day; (INCLUDE EACH DYE ONLY ONCE REGARDLESS OF THE NUMBER OF TIMES IT IS USED)		/
b. Number of pounds of powder dyes weighed per 24 hour day;		/
c. Number of weighings of powder dyes per 24 hour day; (COUNT EACH DYE WITHIN A FORMULA SEPARATELY. INCLUDE BOTH STARTING FORMULAS AND COLOR ADDS OR FEEDS);		/
d. Number of dye weighing rooms or areas at this facility;		/
e. Number of shifts operating per typical day/24 hours;		/
f. Number of days of operation per typical week;		/
g. Number of employees who weigh, mix, handle or are otherwise exposed to powder dyes on a typical day.		/

CBI: Y N
ID#: |__|__|__|__|__|

DRUG ROOM EXPOSURE CONTROLS WHEN WEIGHING POWDER DYE

9. In the table below, please specify the following information:

COLUMN 1: IN THIS COLUMN, PLEASE MARK AN 'X' IN THE BOX FOR EACH CONTROL IF IT IS AVAILABLE BUT ITS USE IS NOT REQUIRED.

COLUMN 2: IN THIS COLUMN, PLEASE MARK AN 'X' FOR EACH CONTROL IF ITS USE IS MANDATORY EITHER BY COMPANY DECREE OR REGULATION.

GENERAL: ANY CONTROL NOT MARKED IN EITHER COLUMN WILL BE ASSUMED TO NOT BE IN USE AT THIS FACILITY. IF CONTROLS OTHER THAN THOSE LISTED ARE USED, PLEASE SPECIFY IN THE SPACE PROVIDED.

	Column 1 Available Not Required	Column 2 Mandatory Use
a. General ventilation	|__|	|__|
b. Local exhaust or hood	|__|	|__|
c. Dust mask	|__|	|__|
d. Respirator	|__|	|__|
e. Long sleeve clothing	|__|	|__|
f. Impervious gloves	|__|	|__|
g. Goggles	|__|	|__|
Other (SPECIFY)		
h. _____	|__|	|__|
i. _____	|__|	|__|

SUPPLEMENTAL E

IN-PLANT QUESTIONNAIRE

Plant I.D. (2 digits)____/____
Recorder: _____

JOINT DYE/DRUG ROOMS MONITORING SURVEY
OF TEXTILE DYEING & PRINTING INDUSTRIES BY

ATMI - American Textile Manufacturers Institute
ETAD - Ecological & Toxicological Association of the Dyestuffs Manufacturing Industry
US EPA - United States Environmental Protection Agency

Name of Dyeing or Printing Facility: _____

Street Address: _____

City: _____, State: _____ Zip Code: / / / / / /

Name of Contact: _____

Title: _____ Phone Number: ( )_____

SELECTION OF WORK SHIFT

Identify the number of shifts where powder dyes are weighed. Circle number in Column A. Based on the above phone number, circle the appropriate digits in Column B. Circle the observed shift in Column C.

A	B	C
No. of shifts where powder dye weighed	Middle 3 digits of 7-digit telephone number	Shift to be observed
1	000-999	1st
2	000-499 500-999	1st 2nd
3	000-333 334-666 667-999	1st 2nd

Record Times of Shift: From ____:___ a.m./p.m. To ____:___ a.m./p.m.

SELECTION OF WEIGHER IN OBSERVED SHIFT TO BE ASSIGNED MONITOR

To select the weigher to be monitored from the chosen shift, collect the last 3 digits of social security numbers (SSN) for all weighers from all drug room(s) operating during observed shift. List:

__ __ __/__ __ __/__ __ __/__ __ __/__ __ __/__ __ __/__ __ __/__ __ __/__ __ __/__ __ __

Select the weigher with the last three SSN digits closest to 500.
Recorder the Weigher's Name: _____

SSN: _____

I.D. #_____

Date: ____/____/____
mo. date year

Plant I.D. (2 digits)____/____
Recorder: _____

SAMPLING AREA INFORMATION

SKETCH LAYOUT OF DRUG ROOM/AREA FOR WEIGHING POWER DYES.

Include: walls, windows, entrances, exits, tables, storage containers, air intake points, vents, fans. Mark stations (table/scales) where weighing occurs (*W). Note approximate dimensions of room/area. Indicate at margins location within facility and give locational name (e.g., "east wing"). Give North/South orientation. Define with arrow and dotted lines the flow of dyes from storage through mixing.

TEMPERATURE AND HUMIDITY

Record the temperature and humidity at the beginning, middle, and end of the shift, and at times when the room conditions change noticeably.

Temperature							
Humidity							
Time Recorded	:	:	:	:	:	:	:

Plant I.D. (2 digits)____/____
Recorder: _____

WEIGHER WORKER INTERVIEW

To be asked of weigher (Name) _____ selected on first page.

I'd like to ask some questions about you and your work history handling powder dyes.

1. When were you born? ____/____/____ (month / day / year)
2. How many years have you worked at this site? _____ years
3. How many years have you handled (weighed and/or mixed) powder dyes at this site? _____ years
4. In your lifetime work experience, indicate how long you have worked in any industry handling (weighing and/or mixing) powder dyes? _____ years
5. What information have you received in the safe handling of dyes, such as training, courses, and literature? _____

_____

OBSERVATION OF WEIGHER PRACTICES (Part I)

		If yes, describe
1. Did monitored weigher wear dust mask or respirator?	No / / Yes / /	_____
2. Did monitored weigher smoke in weigh area?	No / / Yes / /	_____
3. Did monitored weigher eat in weigh area?	No / / Yes / /	_____

QUESTIONS FOR WEIGHER'S SUPERVISOR

1. What is the total number of dyeing or printing machines being serviced today by the monitored weigher:

Equipment	Number at Start of Shift	Number at End of Shift
A. Printing		
B. Semi-continuous/Continuous		
C. Batch dyeing		

2. What is the total number of weighers at your plant on a typical day?
   a. first shift _____
   b. second shift _____
   c. third shift _____
   d. Total _____

Plant I.D. (2 digits)____/____
Recorder: _____

DETAILED WORKER/SAMPLING AREA OBSERVATIONS

Actual Time Periods Monitored: From ____:___ a.m./p.m. To ____:___ a.m./p.m.

Job Category: _____

Specific Duties: _____

_____

A. Clothing & Personal Protective Equipment Utilized: _____

_____

_____

B. Worker Practices: Note equipment used and dermal contact: _____

_____

Drum Relocation: _____

Weigh: _____

_____

_____

Mix: _____

_____

Strain: _____

_____

_____

Transport: _____

_____

C. General Cleanliness:

Walls: _____
Equipment: _____
Inventory: _____
Floors: _____
Spills: _____

D. Engineering Controls:

Ventilation: _____
Drainage: _____

_____

E. Building: _____

Ventilation: _____

_____

Plant I.D. (2 digits)____/____
Recorder: ____________________

TIME IN/OUT OF THE DRUG ROOM

Weigher I.D. #__________

ENTER	EXIT	TOTAL TIME	ENTER	EXIT	TOTAL TIME
HR. MIN.	HR. MIN.	HR. MIN.	HR. MIN.	HR. MIN.	HR. MIN.
				GRAND TOTAL FOR PAGE	

Page _____ of _____

Plant I.D. (2 digits)____/____
Recorder: _____

MASS OF EACH WEIGHING OF POWDERED DYE OR CHEMICAL

DYE OR CHEMICAL WEIGHED BATCH TICKET NAME	MASS OF WEIGHINGS (SPECIFIC UNITS) 1	2	3
	( ) ( )	( ) ( )	( ) ( )
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Complete this information if this is a continuation sheet page ___ of ___ pages.

Plant I.D. (2 digits)____/____
Recorder: ________________

	INVENTORY OF POWDERED DYES (D) AND CHEMICALS (C) WEIGHED				
Bulk Sample I.D.	Batch Ticket Name	FULL TRADE NAME	D/C	Lot No.	Supplier

Complete this information if this is a continuation sheet page ___ of ___ pages.

PERSONAL AIR SAMPLING DATA SHEET

Company:	Plant ID No.	Date (Mo/Da/Yr)
Worker Monitored:	Worker ID No.	SS No.
Job Title/Work Duties:		
Sampling Performed by:		

SAMPLING EQUIPMENT AND CALIBRATION

Flowmeter Model Number:	
Flowmeter Serial Number:	
Flowmeter Calibration Date:	
Calibration Traceable To:	

PERSONAL	PUMP #1			PUMP #2		
Model No.:						
Serial No:						
Flowrate:						
Time:						
Date:						

Signature(s):

FIELD SAMPLING DATA

Sample ID Number:	PUMP #1	PUMP #2
Sampler Location:		
Sample Start Time:		
Sample Stop Time:		
Sample Duration: (MIN)		
Pump Flow Rate: (L/MIN)		
Sample Air Volume: (m3)		

Signature: Date: Calc. Checked By:

STATIONARY AREA AIR SAMPLING DATA SHEET

Company:		Plant ID No:	Date (Mo/Da/Yr)
Sampling Performed by:			
SAMPLING EQUIPMENT AND CALIBRATION			
Flowmeter Model Number:			
Flowmeter Serial Number:			
Flowmeter Calibration Date:			
Calibration Traceable to:			
Sampling Pump Serial Number:			
Sampling Pump Model Number:			
Pre-sampling Flowrate:			
Date:			
Time:			
Post-sampling Flowrate:			
Date:			
Time:			
Signature(s):			
FIELD SAMPLING DATA			
Sample ID Number:			Field Blank
Sampler Location:			
Sample Start Time:			
Sample Stop Time:			
Sample Duration: (min)			
Pump Flow Rate: (L/min)			
Sample Air Volume: (m3)			
Signature:		Date:	
Calculations Checked by:		Date:	

ANALYTICAL DATA

GRAVIMETRIC ANALYSIS			
Sample ID Number:			Filter Blank
Filter Preweight:			
Filter Postweight:			
Sample Weight:			
Blank Correction:			
Adjusted Weight:			
Signature(s):		Date:	Calculations Checked by:

VISIBLE SPECTROPHOTOMETRIC ANALYSIS			
Filter Extraction Date:			
Data File Number:			
Sample Prepared by: (signature)			
Total Absorbance:			
Corrected Total Absorbance:			
Absorptivity: (As)			
Dye Estimate: (ug)			
Average Filter Spike Recovery: (%)			
Corrected Dye Estimate: (ug)			
Estimate, Airborne Dyes: (ug/m3)			
Data Reference Number:			
Signature(s):		Date:	Calculations Checked by:

(See Back of Page for Explanation of Numbers)

CHAIN OF CUSTODY OR TRACEABILITY RECORD

① ☐ Chain of Custody Record
② ☐ Traceability Log
③ Project Number _____
④ Date of Field Sampling _____
⑤ Location _____
⑥ Type of Sample _____
⑦ Container No. _____
⑧ Storage Requirements _____

⑨ Sample No.	⑩ Description	⑪ Other Ident.	⑫ Sampler	⑬ Inventory / Level Check					⑭ Comments
			⑮ Date						

⑯ Seal Intact													
⑰ Date													
⑱ Checked by (Initials)													

⑲ Relinquished by				
⑳ Date				
㉑ Time				
㉒ Received by				

FILLING OUT CHAIN OF CUSTODY/TRACEABILITY LOG

1. Check chain of custody form.

2. Traceability log.

3. Enter project and task number.

4. Enter dates the first and last samples were collected that are recorded on each log sheet.

5. Enter sampling location: plant name and/or city.

6. Enter type of sample, i.e., Tenax trap, condensate, bulk feed, etc. Record only one type of sample on a form.

7. Enter shipping container number in which samples are packed. Each shipping container must contain only one type of sample.

8. Enter storage requirements, i.e., wet ice, dry ice, in plastic bags, etc.

9. Enter entire sample number.

10. Enter any other sample description required.

11. Enter other sample identification, i.e., Tenax tube numbers.

12. Enter name or initials of person collecting sample.

13. Four columns are provided for inventory checkoff each time sample custody is transferred. As the samples are inventoried, place a checkmark in the appropriate box. If samples are liquid, the liquid level should be confirmed at the same time. Changes in the level should be noted and dated under the comments Column 14. When the inventory is completed, enter the data in 15 directly under the column checked off.

    Sixteen through 18 are provided for samples collected under Chain of Custody. Each shipping containers with samples must be sealed with evidence tape when not in the custodian's presence. The seal is not to be broken by any other person. Evidence tape must be placed over the joint between the container and container lid; the tape is signed and dated by the custodian. Each container with samples must be inspected at the beginning of each day. Check off that the seal is intact in 16, record the inspection date in 17, and initial in 18. After seal inspection, the seal may be broken to add additional samples or ice. The container may remain unsealed while in the presence of the custodian.

    Columns 19 through 22 must be used to record a change in sample custodian following either Chain of Custody or Traceability. Each time the custodian is changed, the samples must be inventoried using Column 13 and the transfer recorded using 19 through 22. Samples never change custody without being inventoried and signed off. The Chain of Custody Record or Traceability Log must travel with the samples until transferred to the laboratory custodian. After transfer, provide the laboratory custodian with copies of the forms and give the originals to the crew chief.