MRI REPORT

ASSESSMENT OF AIRBORNE EXPOSURE AND DERMAL CONTACT TO ACRYLAMIDE DURING CHEMICAL GROUTING OPERATIONS

QUALITY ASSURANCE PROJECT PLAN
for the
Office of Toxic Substances

EPA Prime Contract No. 68-02-3938
Work Assignment No. 47
MRI Project No. 8501-A(47)

For

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

Attn: Mr. Tom Murray

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MIDWEST RESEARCH INSTITUTE 425 VOLKER BOULEVARD, KANSAS CITY, MISSOURI 64110 • 816 753-7600

SECTION 1.0

ASSESSMENT OF AIRBORNE EXPOSURE AND DERMAL CONTACT TO ACRYLAMIDE DURING CHEMICAL GROUTING OPERATIONS

Quality Assurance Project Plan

EPA Contract No. 68-02-3938
Work Assignment No. 47

Approval for:

MIDWEST RESEARCH INSTITUTE

Paul C. Constant
Program Manager

4/14/86
Date

Carol L. Green
Quality Assurance Officer

4/16/86
Date

Approval for:

ENVIRONMENTAL PROTECTION AGENCY

Joseph J. Breen
Project Officer

Date

Joseph S. Carra
EPA 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, J. McHugh, D. Hooton

Environmental Protection Agency:
J. Breen, J. Carra, E. Reilly-Weidow, T. Murray

National Institute of Occupational Safety and Health:
B. Hills

SECTION 3.0

PROJECT DESCRIPTION

The Environmental Protection Agency/Office of Toxic Substances (EPA/OTS) under the Existing Chemicals Program has initiated a plan to conduct field studies to assess airborne exposure and dermal contact to acrylamide during sewer grouting operations. The results obtained from these studies will be used to prepare a quantitative risk assessment.

The overall objectives of the proposed field studies are:

1. Quantitative measurement of occupational exposure to airborne acrylamide particulate and vapor in the breathing zone of chemical grouting operators during sewer line and manhole sealing operations.

2. Quantitative measurement of dermal contact to acrylamide during these same operations using direct and indirect methods.

3.1 Scope of Work

The scope of work will consist of the following subtasks:

A. Review applicable methodologies for assessing dermal contact to acrylamide.

B. Review applicable air sampling methods for measuring occupational airborne exposures to acrylamide.

C. Evaluate and finalize an analytical method for determination of acrylamide in air and dermal samples.

D. Develop a QA/QC project plan.

E. Perform the necessary laboratory analysis of the samples collected in the field.

SECTION 4.0

PROJECT ORGANIZATION AND MANAGEMENT

The project 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, will represent management and serve as program manager. He will be assisted in this effort by Mr. John Hosenfeld. Together they will:

- Assure that all necessary resources are available.
- Assure that the Quality Assurance Manager (QAM)/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 QAM/QAC regarding the project.
- Assure that any problems, deviations, etc., reported by the QAM/QAC receive immediate corrective action.
- Review all technical work and reports for overall technical accuracy.

4.2 Quality Assurance Manager (QAM)/Quality Assurance Coordinator (QAC)

Ms. Carol Green, Quality Assurance Manager, will represent QA management. She will be assisted by Mr. Jack Balsinger who will serve as QAC. Together they will:

- Assure that all QA policies and procedures are available and understood by project staff by conducting training courses.
- Assure MRI management that the facilities, equipment, personnel, methods, records, and controls are consistent with project objectives/requirements by conducting or directing inspections and/or audits. These inspection/audit results are reported to project and MRI management. Corrective action is requested in these reports.
- Help prepare the project QA plan.

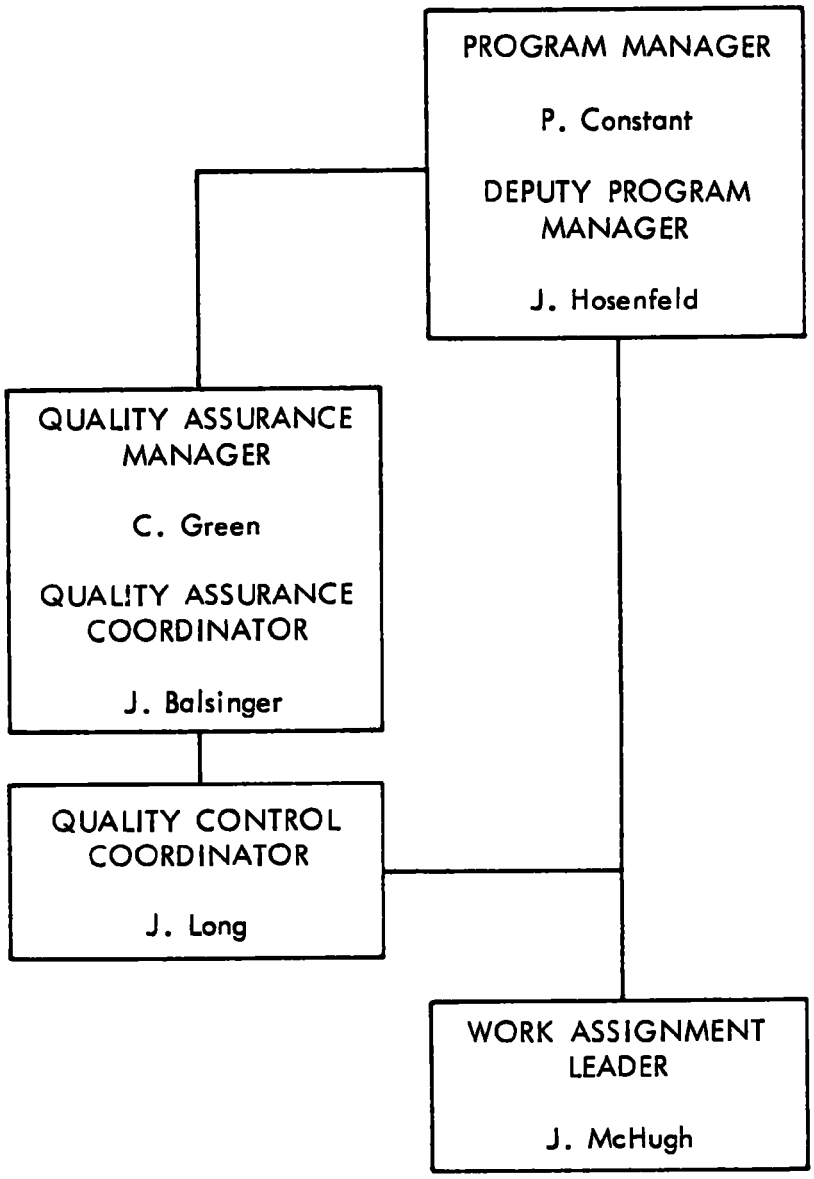

Figure 4-1. Project organizational chart.

- Reinspect or audit to assure that appropriate corrective actions were implemented. Report unresolved corrective actions to MRI's Associate Director of K.C. Operations and the Senior Vice President for resolution.
- Review and audit data reports and supporting evidence prior to submission to EPA.
- Prepare and direct the preparation of QA reports to be submitted to EPA.

4.3 QC Coordinator (QCC)

Ms. Julie Long will serve as QCC. She will:

- Conduct systems audit(s) and report findings to the QAM/QAC.
- Prepare performance audit samples.
- Review notebooks, chromatograms, printouts, and other hard copy information during systems audits.
- Report audit findings to project leader and program management after QAM/QAC approval.

4.4 Work Assignment Leader

Mr. James McHugh will be the work assignment leader. He will:

- Help prepare 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 and sampling 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 and documented.

- Be responsible for analytical data traceability.
- Take corrective action on any problems and communicate them in writing to the QAC/QAM, the QCC, and the program and department managements.
- Prepare and submit monthly and triannual reports.
- Prepare and submit other reports as requested by the work assignment manager in conjunction with project staff.

SECTION 5

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 recently been assigned to this position but has 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. James McHugh will serve as the Work Assignment Leader. He is a certified industrial hygenist and has served as Project Leader on field studies conducted for the Department of Defense and the National Institute of Occupational Safety and Health. His credentials were submitted in the proposal for this contract.

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. He has been with the QA Unit since June 1985. His credentials were previously submitted in the proposal for this contract.

Ms. Julie Long will serve as Quality Control Coordinator. She is skilled in spectrophotometric analysis, and generating and analyzing vapors and aerosols. Ms. Long has received QCC training from Mr. Jack Balsinger, the Quality Assurance Coordinator, and has functioned as a Quality Control Coordinator on EPA/OTS tasks 6 and 37.

SECTION 6.0

FACILITIES, EQUIPMENT, CONSUMABLES, AND SERVICES

6.1 Facilities

Sample preparation will be performed in MRI Lab 315W designated, in part, for the project. The laboratory will be equipped with fume hoods, glove boxes, and an analytical balance.

Sample analyses will be performed on a Varian 5000 Liquid Chromatograph located in MRI Lab 120N.

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:

- Varian 5000 Liquid chromotographic system with autosampler and chart recorder.
- 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 AE-163 analytical balance, capable of weighing to the nearest 0.1 mg.
- Battery operated air sampling pumps.

6.2.1 The LC system will be calibrated prior to sample analysis over a concentration range of ~ 0.1 to 100 µg/mL by injecting a series of four acrylamide (electrophoresis grade) standard solutions prepared from one stock standard plus a check standard solution prepared from an independently weighed standard. The linear regression equation parameters will be calculated from the standard data and plotted for visual evaluation of linearity. The correlation coefficient for the standard data should be greater than 0.995 to define a linear operating range for the analytical system. One of the midpoint standards will be injected after every fifth sample to monitor the precision of the system over the entire analysis; the responses for this standard will be plotted on a control chart and should exhibit less than a 10% relative standard deviation.

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

6.2.1.2 The Mettler AE-163 analytical balance is checked twice monthly to confirm performance according to manufacturer's specifications. The weight will be traceable to or checked against National Bureau of Standards weights.

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.

6.3 Consumables

All water used for the analysis will be deionized water filtered through a Milli-Q® system; pH adjustments will be made using a reagent grade sulfuric acid:water solution (1:10, v/v).

Blank collection media (dermal pads, surface wipes, air monitors, and hand rinses) will be spiked both in the field during sample collection and also in the laboratory to check for chemical recovery and for any loss of chemical during transit back to the laboratory. All sample collecting media spiking experiments, both in the laboratory and those spiked in the field during sample collection, will use collecting media identical to those used in actual field sample collection.

Table 6-1. Maintenance

Equipment	Service	Frequency
Varian 5000 Liquid Chromatograph	General	As needed
Hewlett-Packard 9826	Limited requirements	As needed
Balance	Cleaning and adjustment for calibration	1 year

SECTION 7.0

DATA GENERATION

7.1 Sample Data Collection

7.1.1 Field Sampling Plan

See Appendix A for the field sampling protocol

7.1.2 Air Sampling Data

Personal and area samples will be collected on a 0.8 u mixed cellulose ester filter and silica gel sampling train using calibrated, battery operated sampling pumps at a nominal flow rate of 1.0 L/min. One area sample will be collected inside the Mobil Reveal and Seal Unit where the mixing tanks are located. Personal samples will be collected in the breathing zone of workers during the chemical grout mixing operation and during the grout injection operation whenever the manhole is entered.

Air sampling data will be recorded on the Air Sampling Data Sheets (Figure 7-1). The information collected for each personal or area air sample collected will include: employee and work site data, sampling equipment data, sampling parameters, exposure data, calibration data and general observations.

7.1.3 Dermal Contact Assessment Data

Dermal contact assessment sampling will be performed using the dermal pad and hand rinse methods as described by Durham and Wolfe (1962). Observations made during a preliminary site visit to a chemical grouting operation indicated that significant dermal contact occurs on the face, neck, and forearms of workers protected with impervious clothing. The worker's torso, upper arms, and legs are protected by the impervious suit. Assuming that full protective clothing is used by the worker, dermal pads will be placed at six body locations to assess dermal contact to the face, neck and forearms. If protective clothing is not utilized dermal pads will be placed at ten body locations to assess dermal contact to the entire body. Hand rinses will be conducted using the bag techniques as first described by Durham and Wolfe (1962). Dermal contact assessments will be conducted during equipment assembly operations, grout mixing operations, grout injection operations, and equipment disassembly operations.

Company Name	Contract Number: 68-02-3938	Date (Mo/Da/Yr)
Site ID	Work Assigment No. 47	Substance Monitored: ACRYLAMIDE

EMPLOYEE AND WORK AREA DATA

Employee Name	Work Location Description
Job Title/Work Duties	
	Weather Conditions

SAMPLING EQUIPMENT

Instrument	Model No.	Serial No.

[] Personal [] Area [] Bulk	Sample Collection Media: 0.8u MIXED CELLULOSE ESTER FILTER AND SILICA GEL TUBE	Lot No.

FIELD SAMPLING INFORMATION

Field Sample ID Number						
Start Time						
Stop Time						
Sample Duration (mins.)						
Pump Flow Rate ( )						
Sample Air Volume ( )						
Laboratory Analysis	Laboratory Results	Air Concentration	Laboratory Results	Air Concentration	Laboratory Results	Air Concentration
Analyte / Units-->						
1. ACRYLAMIDE						
2.						
3.						

Signature:	Date:
Calculations Checked by:	Date:

Figure 7-1. Air sampling data sheet.

CALIBRATION DATA			
Calibration Method			
Volume		Resistance	
Pre Date	Post Date	Calculations	
1.	1.		
2.	2.		
3.	3.		
Signature	Signature	Average Time	Flowrate

Additional Comments, Observations, Diagrams, Data References, etc.

Figure 7-1 (continued)

Dermal sampling data will be recorded on Dermal Assessment Data Sheets (Figure 7-2). The information collected for each worker sampled will include: employee and work site data, weather conditions, protective equipment utilized, sampling parameters, exposure data and general observations.

7.1.4 Wipe Sampling Data

Wipe samples will be conducted using glass fiber filters moistened with distilled water. Wipe samples will be collected at a number of equipment surfaces and protective equipment surfaces which come in frequent contact with the skin.

Wipe sampling data will be recorded on Wipe Sampling Data Sheets (Figure 7-3). The information collected for each wipe sample will include: work site data, sampling parameters, and detailed description of the wipe sample location.

7.1.5 General Observation

The field observation sheets (Appendix B) will be completed for each site visited. The information collected will include: Grouting contractor information, a description of the site, workforce description, employee health information, safety procedures, protective equipment used, and general work practices.

7.2 Sample Traceability

Sample traceability protocol (SOP SC-1) will be followed for sample tracking for this project. Traceability records will start with sample collection. These records will accompany the samples when shipped to MRI. Examples of the field and laboratory traceability forms are shown in Figures 7-4 and 7-5.

7.3 Laboratory Analysis Procedures

See Appendix C for analytical protocol. A flow chart of the steps involved in the analytical method is shown in Figure 7-6.

7.4 Internal Quality Control Checks

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 method blanks, spikes, field blanks, and field spiked blanks.

Company Name	Contract Number 68-02-3939	Date (Mo/Da/Yr)
Site ID	Work Assignment No. 47	Substance Monitored ACRYLAMIDE

EMPLOYEE AND WORK AREA DATA

Employee Name	Job Title/ Work Duties:

PERSONAL PROTECTIVE EQUIPMENT

Eye Protection	NOTE: Specify type, material, brand, etc.
Boots/Shoes	Head Protection
Respirator	Gloves
Protective Suit/Coveralls	

DERMAL SAMPLING DATA

Dermal Pad Media: WHATMAN CHROMATOGRAPHY PAPER No. 17 (4 X 4 inch Pads) | Lot No.

Body Region	Start Time	Stop Time	Duration	Sample Numbers	
Back					
Chest					
Forearms				Left	Right
Shoulders				Left	Right
Thighs				Left	Right
Shins				Left	Right
Hand Rinses Solvent: DISTILLED WATER				Left	Right
				Left	Right
				Left	Right

Signature	Date	Checked by:

Figure 7-2. Dermal assessment data sheet.

Company Name	Contract Number 68-02-3938	Date (Mo/Da/Yr)
Site ID	Work Assignment No. 47	Substance Monitored: ACRYLAMIDE
Wipe Sampling Media and Method WHATMAN 37mm GLASS FIBER FILTER MOISTENED WITH DISTILLED WATER		Lot No.

Wipe Sample No.	Time	Location Description
		TOP OF THE CONTROL PANEL IN THE MOBILE REVEAL AND SEAL UNIT
		OUTSIDE OF THE ACRYLAMIDE MIXING TANK
		OUTSIDE OF THE CATALYST MIXING TANK
		HANDLE OF THE INJECTION GUN (Manhole sealing operation only) OR SIDE OF PACKER (main line or lateral line sealing operation only)
		HYDRAULIC HOSING ATTACHED TO INJECTION GUN OR PACKER
		SIDE OF SAFETY CONE OF THE ROAD
		INSIDE OF THE RESPIRATOR
		OUTSIDE OF THE RESPIRATOR
		BACK OF IMPERVIOUS WORK GLOVE

Signature	Date

Figure 7-3. Wipe sampling data sheet.

(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				

Figure 7-4. Sample traceability form.

FILLING OUT CHAIN OF CUSTODY/TRACEABILITY LOG

1 Check chain of custody, or

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 commments Column 14 When the inventory is completed, enter the date 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

Figure 7-4 (continued)

(See Back of Page for Explanation of Numbers)

LABORATORY CHAIN OF CUSTODY OR TRACEABILITY RECORD

① ☐ Chain of Custody Record
② ☐ Traceability Log
③ Project Number _____
④ Date of Laboratory Check-In _____
⑤ Location _____
⑥ Type of Samples _____
⑦ Container Type _____
⑧ Custody Office Storage Location _____

⑨ Laboratory Sample No.	⑩ Field Sample No.	⑪ Sample Description	⑫ Amount of Sample Removed (give date)	⑬ Comments

Laboratory/Area	⑭ Relinquished by: (signature)	⑮ Date/Time	⑯ Received by: (signature)	⑰ Date/Time
Custody Office				
Sample Preparation				
Metals				
GC/MS				
Other (Identify)				

⑱ Notebook Reference Pages _____ ⑲ Notebook No. _____
⑳ Analyst Comments _____

Figure 7-5. Laboratory traceability record.

FILLING OUT CHAIN OF CUSTODY/TRACEABILITY LOG

1. Check chain of custody or
2. Traceability log.
3. Enter project and task number.
4. Enter date samples were received by custody office.
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 container type, i.e., Tenax trap, XAD trap, petri dish, narrow mouth quart bottle, 2-dram vial, 2 oz Rx bottle, etc.
8. Enter storage location samples will be taken from and returned to for custody office possession.
9. Enter laboratory sample number.
10. Enter field sample number.
11. Enter any additional sample description, i.e., hazards in handling the sample, appearance, Tenax trap number, etc.
12. Enter any amount of sample expended and date.
13. Enter any comments pertinent to the tracking of the entire sample, i.e., sample composited to form a new sample, sample transfer to a new custody form, sample split to give two or more items to keep in custody, sample lost, etc. Enter references to appropriate laboratory notebook (page and book number). If comments pertain to all samples listed, enter as "see time 20," and include these in the appropriate space provied at the bottom of the form.

14-17. 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 columns 9 and 11 through 13 and the transfer recorded using 14 through 17. 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.

18 and 19. Enter location of sample records listed in the project sample custodian's notebook.

20. Enter any comments pertaining to (a) an individual sample noted above (use footnote) or to (b) all the samples listed above. (See also item 13.)

Figure 7-5 (continued)

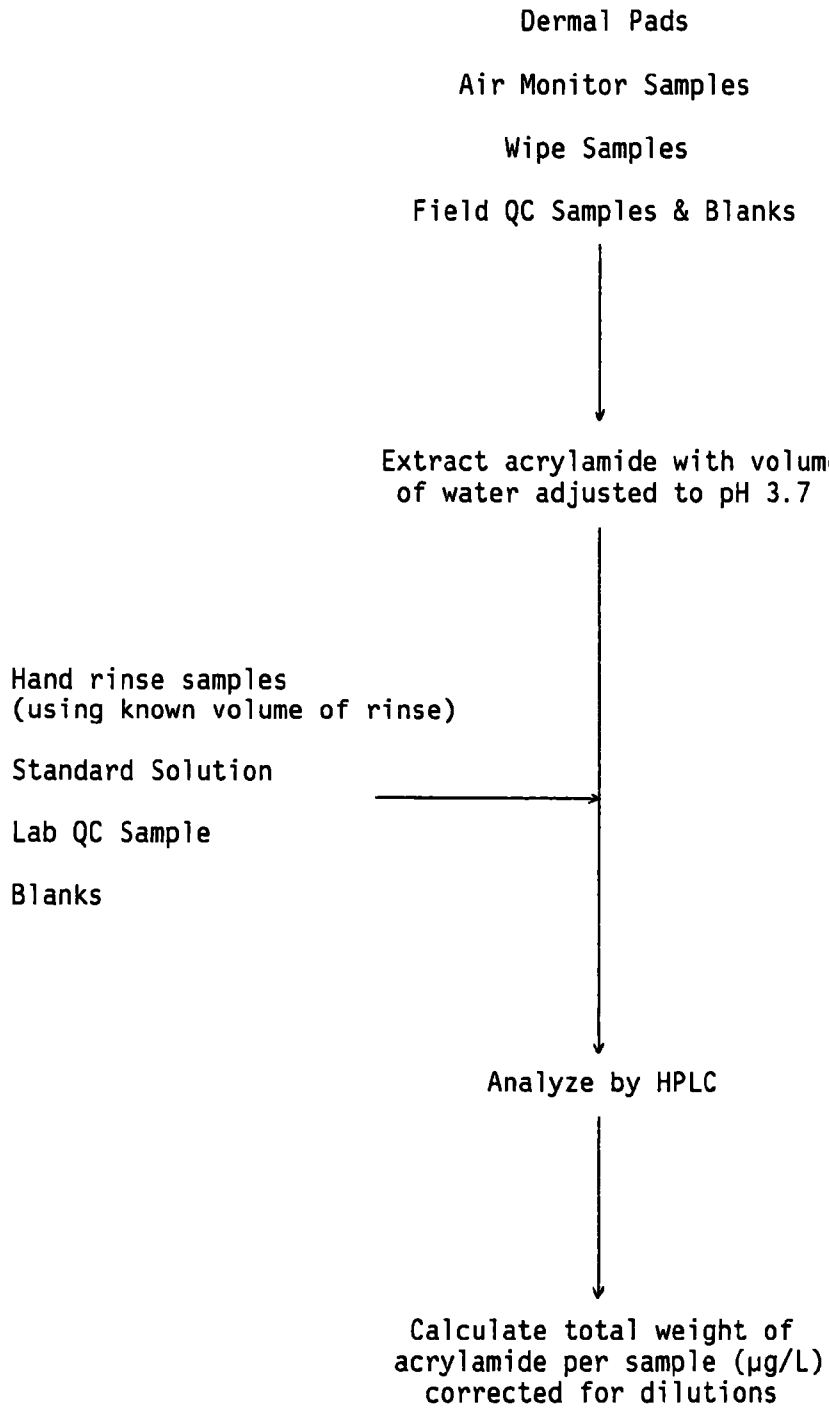

Figure 7-6. Flow chart for the determination of acrylamide in field samples.

7.4.1 General

New lots of reagents are checked prior to use, or current lots of reagents are checked when method blank problems are experienced.

7.4.2 The control checks that will be utilized are the following:

Method blanks: At least one per sample group tested.

Field filter blanks: At least two per sample group tested for each type of collection medium.

Spiked blank filters: Replicate samples will be prepared to check on extraction recovery.

7.4.3 Calibration

The LC system will be calibrated prior to sample analysis over a concentration range of ~ 0.1 to 100 µg/mL by injecting a series of four acrylamide (electrophoresis grade) standard solutions prepared from one stock standard plus a check standard solution prepared from an independently weighed standard. The linear regression equation parameters will be calculated from the standard data and plotted for visual evaluation for linearity. The correlation coefficient for the standard data should be greater than 0.995 to define a linear operating range for the analytical system. One of the midpoint standards will be injected after every fifth sample to monitor the precision of the system over the entire analysis; the responses for this standard will be plotted on a control chart and should exhibit less than a 10% relative standard deviation.

If response values for the midpoint standard vary from its mean response by more than 10% relative standard deviation, the source of loss in precision will be corrected and the calibration curve repeated before subsequent samples are analyzed.

7.4.4 Definitions

7.4.4.1 Method blanks: Procedural blanks are carried through the entire procedure to check for contamination.

7.4.4.2 Field blanks: The field filter blank is taken to the field and handled in the normal fashion except it is not exposed to the contaminant.

7.4.4.3 Spiked blanks: Collecting media will be spiked with known amounts of acrylamide.

7.5 Systems and Performance Audits

7.5.1 Systems audits: Systems audits by the QCC/QAC 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.
- Assisting/conducting data audit prior to report submittal.
- Preparing and submitting a report with recommended corrective actions to the QAC/QAM, and after approval, to the work assignment leader and program manager.
- Conduct additional audits as directed by the QAC/QAM.
- Assisting/preparing QA report for the EPA's work assignment manager.

7.5.2 Performance Audits

The performance audit sample is designed to check the operation of the equipment. Several blind performance samples will be independently prepared by the QCC and submitted for analysis before and during the analysis of the regular samples. Performance audit samples will also be analyzed if (1) the QCC believes the analysis procedures has changed, (2) analytical problems are suspected, or (3) the MRI work assignment leader of the EPA work assignment manager requests samples.

7.5.3 QAC/QAM Audits

Additional audits will be conducted or directed by the QAC/QAM as follows:

- Schedule and conduct additional audits as needed, e.g., staff credentials, quality control data and practices, documentation practices, data audit, and QA compliance.
- Review and approve the report and supporting evidence for accuracy and QA compliance prior to report submittal to OTS.

SECTION 8.0

DATA PROCESSING

8.1 Collection

Data collection will utilize both manual and computerized acquisition systems. All activities shall be legibly recorded using permanent ink in the project notebook or on worksheets. Each person who records data shall sign and date each sheet. Strip charts, magnetic tapes, etc., shall be labeled with a format identifier, project number, date, the ID(s) of the instrument, and the name of the person responsible for the data. Custody of the original data media will be the responsibility of assigned project staff until archived.

8.2 Data Reduction

Standard data reduction procedures with built-in checks will be used. For example, if an integrator or computer is used to calculate concentrations, the standards used to generate the curve must be back-calculated using the curve to ensure satisfactory curve fitting over the anticipated range. In addition, all sample manipulations (e.g., weighing, dilution, concentration, etc.) must be clearly documented.

8.3 Data Validation

The work assignment leader will be responsible for assuring data validity which will include:

- Validating all equations and computer programs and documenting the validating and evidence.
- Validating and checking electronic data transfer.
- Proofreading all data transfers by the analyst or a second project staff member.
- Screening data for consistency by a second project staff member.
- Checking calculations.
- Performing outlier checks.
- Reporting of all associated blank, standard, and QC data along with results for analyses of each batch of samples.

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

8.4 Transfers

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. These data will be checked for transfer errors.

8.5 Storage

Raw data will be documented in laboratory notebooks, on printed paper, as strip chart recordings, or on magnetic tape or disk. Permanent storage of work assignment data in the formal project file and hard copy from magnetic media will be archived (SOP-QA7). The storage of magnetic media will be reported.

SECTION 9.0

DATA QUALITY ASSESSMENT

The objective of precision for this method will be to obtain total weight of acrylamide for replicate spiked filter samples which have relative differences ± 20% of each other. The objective for accuracy will be to obtain total weights of acrylamide on replicate spiked filter samples which have relative errors ± 30% of the actual acrylamide present on the air filter. Average recovery efficiencies for acrylamide using spiked filters should fall within the range of 70 to 130% to yield meaningful data.

9.1 Analytical Precision

Precision is determined by performing replicate analysis. For data sets with a small number of points ($2 \leqq n \leqq 8$), the estimate of precision will be expressed as range percent (R%):

$$R\% = \frac{X_1 - X_2}{\bar{X}} \times 100$$

where X_1 = highest value determined

X_2 = lowest value determined

$\bar{X}$ = mean value of the set

and

$$\bar{X} = \sum_{i=1}^{n} \frac{X_i}{n}$$

where X_i = ith determination

n = number of determinations

The estimated detection limit (EDL) for the analytical system will be defined as the corresponding concentration equal to three times the noise level from the detector.

For large data sets ($n > 8$), the estimate of precision will be expressed as percent relative standard deviation (% RSD):

$$R.S.D. = \sqrt{\frac{\sum_{i-1}^{n} (Xi - \bar{X})^2}{n - 1}}$$

where n = number of replicate determinations.

$$\bar{X} = \text{mean} = \frac{\sum_{i=1}^{n} Xi}{n}$$

The precision of the analytical system will be monitored through replicate injections of a midpoint standard throughout the analysis and plotted on a control chart.

9.2 Accuracy

The accuracy of the analytical method can only be established for known spiked samples. Accuracy may be indicated by comparing the total weight of acrylamide on spiked sample collection media determined using the analytical method to the theoretical or actual amount of chemical that was spiked onto the blanks.

Accuracy will be measured by calculating the relative error (RE):

$$RE\ (\%) = \frac{F - A}{A} \times 100$$

where F = found weight of chemical
A = actual weight of chemical

9.3 Recovery

Recovery will be indicated from the results of the spiked blank analyses. Spiked acrylamide recovery will be determined by a direct comparison of the spiking solution to the solution obtained from extracting the spiked blank.

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

where c_{fil} = concentration of extract
c_{STD} = concentration of spiking solution

9.4 Traceability of Instrumentation

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

9.5 Traceability of Samples

All samples will have a unique identification number along with information on field site, monitoring location, exposure time and conditions, collection device, etc. The samples will be labeled with adhesive bar-code labels to identify the samples and trace them through the sampling and analytical procedures.

9.6 Traceability of Data

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

9.7 Completeness

Due to the variety of data points available per field test site, completeness of the data will be crucial in order to obtain meaningful data.

SECTION 10

CORRECTIVE ACTION

The work assignment leader has primary responsibility for taking corrective action; if he is unavailable, the program manager, and/or the QAC/QAM shall be contacted for instructions. Some of the types of problems and corrective actions to be taken are listed below. Unresolved problems are reported by the QAM to the Associate Director of K.C. Operations and to the Senior Vice President for resolution.

10.1 Performance/Systems Audits

If problems are detected during an audit:

- The auditor shall notify the person responsible, the work assignment leader, and the QAC/QAM of the problem(s) and any action(s) he has taken.
- The work assignment leader and the person responsible shall correct the problem, then notify the QAC/QAM.
- The auditor shall then prepare, and after QAC/QAM approval, send a problem/action taken memo to the program manager and the work assignment leader.

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 notebook; the work assignment leader then prepares and sends a problem/action taken memo to the QAC/QAM, and the program manager.
- If a large quantity of data is affected, the problem/action taken is documented in the notebook; the work assignment leader then prepares and sends a problem/action-taken memo to the QAC/QAM, project manager, and 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/QAM and program manager; if the problems are significant, the action is determined collectively.
- The action taken is documented in the notebook.
- The problem and action taken is reported to the EPA work assignment manager.

SECTION 11

DOCUMENTATION AND REPORTING

11.1 Documentation

- All documentation shall be in permanent ink.
- 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 an original page. It will be initialed, dated, and explained.
- All deviations from standard operating procedures (SOPs), procedures, and protocols 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.

11.2 Document Control

- Raw sampling data will be documented and stored in laboratory notebooks, on sequentially numbered sampling forms, on printer paper, on magnetic tape, and as strip chart recordings.
- A logbook of the data media created during each test period will be established to document the existence and flow of data through the data processing cycle.
- All project-related documents will be assigned a unique numerical designation in a document control system maintained by assigned project staff.

11.3 QA Reports to Management

The QAC/QAM, in cooperation with the work assignment leader, shall identify critical phases 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 QA plan.
- Assessment of data accuracy, precision, and completeness.

The results of inspections and audits will be reported quarterly by the QAM to MRI management; summaries will be reported to the EPA work assignment manager.

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

FIELD SAMPLING PROTOCOL

1.0 AIRBORNE EXPOSURE ASSESSMENT

1.1 Air Sampling Protocol

Airborne acrylamide aerosols and vapor are collected on a sampling train consisting of a mixed cellulose ester filter and silica gel tube at a flow rate of 1 L/min. The collected material is desorbed from the sampling media with deionized water followed by HPLC analysis.

The sampling media will consist of a Millipore 37 mm MAWP filter cassette assembly available from Millipore Corporation of Bedford, Massachusetts. On the outlet side of the filter cassette assembly a SKC No. 226-10 silica gel tube available from SKC, Inc. of Eighty Four, Pennsylvania shall be attached with a short section of tygon tubing of minimum length. The sampling train shall be attached to a calibrated, Ni-Cad battery operated personal sample pump. The sampling pump is calibrated before and after the sampling period using a volumetric buret and stop watch with the sampling train in line.

The MAWP filter cassettes and silica gel tubes are to be labeled prior to the site visit with a unique number using adhesive barcode labels. A set of six identical barcode labels will be printed for each sample. One of the adhesive labels in the set will be affixed to each filter cassette and silica gel tube. The samples are then placed in individual zip-lock plastic bags. Another label from the same set will be affixed directly to the outside of the plastic bag. The remainder of the labels in the set will be placed inside the plastic bag. The sample numbers will be logged with a laptop computer and light pen using barcode software.

At the survey site, a corresponding barcode label from the filter cassettes and silica gel tubes will be affixed to the Air Sampling Data Sheet which contains pertinent information concerning site location, employee and work area information, sampling equipment and methods, calibration data, and sampling and analytical data.

Breathing zone air sampling shall be conducted during the grouting chemical mixing operation and during sealing operations when the worker enters the manhole. Area sampling shall be conducted inside the Reveal and Seal Mobile Unit near the mixing tanks. If possible, an area sample will also be collected inside the maintenance and storage facility at the grouting contractor's place of business. The recommendation flow rate is 1.0 L/min. The recommended sample volume is 480 L. The pump rotometer should be checked frequently during the sampling period to maintain the calibrated flow rate. Pertinent information concerning the air sample such as ambient temperature, atmospheric pressure, sample duration, flow rate, site location, subject information shall be referenced to the sample number and recorded on an Air Sampling Data Sheet.

At the end of the sampling period, the sample train is disassembled and the filter cassette and silica gel tube recapped. Place the sampling media in a water tight container and store in a chest containing ice or blue ice packs for transport to the laboratory. Do not use dry ice. The samples will be sent to the laboratory in sealed ice chests using a next-day delivery service. A Chain-of-Custody or Traceability record will be filled out in the field and accompany all shipments to the laboratory.

1.2 Quality Control Field Samples

On each day of sampling two sets of field blanks and two sets of spiked samples shall be prepared. Field blanks are required in order to account for any possible contamination which may occur while collecting, transporting, or handling field samples prior to extraction in the laboratory. Spiked or fortified samples are required to account for stability and volatility of the grouting chemical.

Field blanks of the collection media should be handled in the same manner as the exposed sample except that no air is drawn through them. The end caps of the filter cassette blanks are to be removed in the field and immediately resealed and labelled. The ends of the glass tube on the silica gel blank shall be broken in the field and immediately recapped and labelled. One set of field blanks shall be prepared at the beginning of the sampling period and one set at the end of the sampling period. The blanks shall be stored and shipped with the exposed air samples.

Two filter cassettes and two silica gel tubes are to be spiked with a known quantity of acrylamide diluted to the approximate concentration used in the grouting material (100 g of acrylamide per liter of water). The amount of acrylamide added to the collecting media should be approximately the levels that are expected to impinge on the sampling media during the field studies. One spiked sample set shall be prepared at the beginning of the sampling period and the other at the end of the sampling period. Both samples should be handled, stored and shipped with the exposed samples.

2.0 DERMAL CONTACT ASSESSMENT

2.1 Background

The methodologies for assessment of dermal contact to toxicants have been developed principally for pesticides exposure monitoring. The methods described by Durham and Wolfe (1962) and Davis (1980) remain the established protocols utilized by pesticide manufacturers and formulators to conduct exposure monitoring of pesticide applicators.

Durham et al. (1962) reviewed the primary methods for measurement of dermal exposure. One method involves placing absorbent pads at various points on the worker's body. The worker then performs his usual job functions. The pads are removed and transported to a laboratory, where the toxicant is extracted and analyzed. The amount of pesticide that comes into contact with the skin is calculated using an anatomical model, which is extrapolated to the entire surface area of the body part represented by the pad. The necessary assumption that the pad area is representative of the entire body part being measured is a shortcoming of this technique.

A variation of the absorbent pads method involves the use of knit cotton garments that cover the study area during exposure. One such method was described by Davis (1980) to assess exposure to the hands using cotton gloves as the collecting media. Durham et al. felt that the use of absorbent

gloves might result in overestimations of exposure to the hands. The gloves tend to absorb much more liquids than could be expected to adhere to flesh. This method however, is quick and easy to implement in the field.

The other primary method described by Durham et al. involves swabbing or rinsing the skin with a solvent that will remove the toxicant. The rinse solutions are collected and analyzed. The swabbing technique uses surgical gauze moistened with solvent. The gauze pads are handled with forceps to rub the body surface with light pressure. The procedure is very tedious and time-consuming and will not remove residues that are absorbed into the skin during the exposure period.

Use of the swab method for estimating contamination of the hands was found to be unsatisfactory. It is not easy to swab correctly between fingers and around the fingernails. Durham and Wolfe described a bag rinse technique suitable for the hands. The procedure involves the use of polyethylene bags containing a suitable solvent for washing the pesticide from the hands. The hand is inserted into the bag containing a given quantity of solvent. While the bag is held tightly around the wrist to prevent leakage, the hand is shaken vigorously. The rinse is collected and transported to a laboratory for analysis. The procedure is faster than the swabbing technique; however, it also will not remove residues that are absorbed into the skin during the exposure period. Durham and Wolfe (1962) reported percentage of extractable parathion pesticide using the hand rinse method ranging from 77 to 94% for the first rinse and 89 to 98% for the second rinse. The hand rinse recoveries for acrylamide are expected to be higher because acrylamide is more soluble in water.

Taking into account the advantages and disadvantages of the various monitoring devices listed above, the simple absorbent pad method as described by Durham and Wolfe (1962) is recommended for all body parts except the hands. Durham et al. validated the effectiveness of alpha-cellulose pads by comparing the residues found on the pads to those found on adjacent areas of skin swabbed with ethanol and found acceptable agreement.

Most dermal studies of pesticide have essentially adopted the number and location of dermal pads used by Durham and Wolfe (1962). They recommended that each worker be monitored with a set of ten pads. The location of the pads are: the front of the legs just below the knees, the front of the thighs, the back of the forearms, on top of the shoulders, the back of the neck at the edge of the collar, and on the upper chest near the jugular notch. If protective clothing is worn, the worker is monitored by pads attached to the upper back and chest to assess exposures to the face and neck. In addition, pads attached to the forearm can be utilized to assess dermal contact to the arms if not protected by the impervious clothing.

Dermal exposure studies of pesticide workers demonstrate the important contribution of hand exposure to overall dermal contact. Hand exposure as a percentage to total dermal exposure range from 37 to 98%. The bag rinse method appears to be the method of choice. Durham and Wolfe (1962) found that they could recover approximately twice as much residue from the worker's hands by using the bag rinse method rather than the swab method.

2.2 Dermal Contact Assessment (except of hands) Using the Dermal Pad Method

2.2.1 Sampling Protocol

Pads to be used for estimating dermal contact are to be constructed from preparative chromatography paper (17 Chr) available in sheets from Whatman, Inc. of Clifton, New Jersey. The chromatography paper is cut into 4-in. (10.2-cm) squares which are then stapled to the center of a 5 in. square protective backing of glassine paper available from Schleicher and Schuell, Inc. Both materials are also readily available through laboratory supply companies. When handling the absorbent pads, skin contact should be avoided by wearing disposable surgical gloves.

The pads are to be labeled prior to the site visit using barcode labels which will identify the samples and track them through the sampling and analytical procedures. A set of six identical barcode labels will be printed for each sample. Each set of labels will have a unique number. One of the adhesive labels in the set shall be affixed to the back of the glassine protective backing. Each pad shall then be placed into individual zip-lock plastic bags. The other five adhesive labels from the same set shall be affixed to the outside of the plastic bag. The sample numbers will be logged with a laptop computer and light pen using barcode software.

At the survey site, a corresponding barcode label from the dermal pad will be affixed to a Dermal Assessment Data Sheet which contain pertinent information concerning the site location, sampling data, subject information, etc. Following the sampling period, each dermal pad shall be returned to its original container.

Assuming that full impervious protective clothing is used by the subject, six pads will be attached to the worker. The locations of the pads are:

1. On the right forearm midway between the wrist and elbow, on the side of the arm opposite the palm - the pad shall be positioned face up with the glassine backing taped flush against the skin. Attach the pad with surgical tape applied only to the glassine backing.

2. On the left forearm midway between the wrist and elbow, on the side of the arm opposite the palm - the pad shall be positioned face up with the glassine backing flush against the skin using surgical tape as described above.

3. On the subject's upper back immediately below the collar. The pad shall be positioned face up on the outside of the protective clothing using safety pins.

4. On the upper chest near the Jugular notch. The pad shall be positioned face up on the outside of the protective clothing using safety pins.

5. On the top of the right shoulder - the pad shall be positioned face up with the glassine backing against the outside of the workers' protective clothing using safety pins.

6. On top of the left shoulder - the pad shall be positioned face up with the glassine backing against the outside of the worker's protective clothing using safety pins.

If impervious protective clothing is not worn by the worker, 10 dermal pads will be attached to the worker. The locations of the pads are:

1. On the right forearm, midway between the wrist and elbow, on the side of the arm opposite the palm - the pad shall be positioned face up with the glassine backing flush against the outside of worker's street clothes using safety pins. If the worker is wearing a short sleeve shirt, attach the pad directly against the skin using surgical tape.

2. On the left forearm midway between the wrist and elbow, on the side of the arm opposite the palm - the pad shall be positioned face up with the glassine backing flush against the outside of the worker's street clothes using safety pins. If the worker is wearing a short sleeve shirt, attach the pad directly against the skin using surgical tape.

3. On the subject's upper back immediately below the collar. The pad shall be positioned face up on the outside of the worker's street clothes using safety pins.

4. On the upper chest near the jugular notch. The pad shall be positioned face up on the outside of the worker's street clothes using safety pins.

5. On the top of the right shoulder - the pad shall be positioned face up with the glassine backing against the outside of the worker's street clothes using safety pins.

6. On top of the left shoulder - the pad shall be positioned face up with the glassine backing against the outside of the worker's street clothes using safety pins.

7. On the right front thigh - the pad shall be positioned face up with the glassine backing against the outside of the worker's street clothes using safety pins.

8. On the left front thigh - the pad shall be positioned face up with the glassine backing against the outside of the worker's street clothes using safety pins.

9. On the right front shin just below the knee - the pad shall be positioned face up with the glassine backing against the outside of the worker's clothes using safety pins. If the worker is wearing shorts, the pad will be attached directly against the skin using surgical tape.

10. On the left front shin just below the knee - the pad shall be positioned face up with the glassine backing against the outside of the worker's clothes using safety pins. If the worker is wearing shorts, the pad will be attached directly against the skin using surgical tape.

Dermal pad sampling is to be performed during the grout equipment assembly, grout mixing operations, grout injection operations, and equipment disassembly operations. Sampling duration shall not exceed 4 h. If the grouting operations exceed 4 h duration, the pads shall be changed to fresh pads.

At the end of the sampling period the pads shall be carefully removed from the worker using surgical gloves. Care should be taken not to touch the absorbent pad. Slide the pad and glassine backing into its original prelabeled plastic bag and seal. Place the plastic bags from a single test subject in a sealed wide mouth jar. Seal the jar inside a plastic bag and store the samples in a chest containing ice or blue ice packs for transport to the laboratory. Do not use dry ice. Send the samples to the lab at the end of each day of sampling using a next-day delivery service. A Chain-of-Custody or Traceability Record will be filled out and accompany all samples sent to the laboratory. A barcode label from each sample in the shipment will be affixed to this form and sent with the sample to MRI.

2.2.2 Quality Control Field Samples

On each day of sampling two field blanks and two spiked samples shall be prepared. Field blanks of the dermal pads should be handled in the same manner as the exposed pads except they are not attached to the worker. An absorbent pad is to be removed from the plastic bag container and immediately returned to the bag and resealed. One field blank shall be prepared at the beginning of the sampling period and one at the end of the sampling period. The field blanks shall be stored and shipped with the exposed samples.

Two pads are to be spiked with a known quantity of acrylamide diluted to the approximate concentration used in the grouting material (100 g of acrylamide per liter of water). The amount of acrylamide added to the pad should be approximately the levels that are expected to impinge on the pads during the field studies. One spiked sample shall be prepared at the beginning of the sampling period and exposed to the same weather conditions as the exposed samples for the duration of the sampling period. The other spiked sample shall be prepared at the end of the sampling period. Both samples should be handled, stored and shipped in the same manner as the exposed samples.

2.2.3 Dermal Contact Calculations

Dermal contact will be calculated using the method described by Durham and Wolfe (1962). An anatomical model is used to calculate dermal contact from the amount of acrylamide found on the dermal pads. The anatomical model shown in Table A-1 represents surface areas of an adult male and is derived from the data of Berkow (1931), Diem and Lentner (1970), and Durham and Wolfe (1962). The suggested pairing of exposure pad locations and body

Table A-1. Dermal Exposure Pad Locations Used for Calculation of Dermal Contact and Surface Areas of those Regions

Body region	Exposure pads used to represent body regions	Surface area of regions (cm^2)[a]
Face	Shoulder pads	650
Back of neck	Back pad	110
Front of neck	Chest pad	150
Hands	Total residue in hand rinse	820[b]
Back	Back pad	3,550
Chest and stomach	Chest pad	3,550
Upper arms	Shoulder and forearm pads	1,320
Forearms	Forearm pads	1,210
Thighs	Thigh pads	2,250
Lower legs	Shin pads	2,380

[a]Calculated from data of Berkow (1931), Diem and Lentner (1970), and Durham and Wolfe (1962).
[b]Surface area of hands is only necessary if partial swabbing or pads attached to the hands are used for collection of residues.

regions is also shown in Table A-1. The amount of acrylamide per unit area of the pad is divided by the exposure time and then multiplied by the surface area of the unprotected body region represented by the pad. If more than one pad represents a body region, the amount of acrylamide per unit area per exposure time for each pad is averaged and then multiplied by the surface area of the body region represented by the pads. The dermal contact to the hand is simply the total residue as determined by the bag rinse, divided by the exposure time. The total dermal contact to the body is the sum of the calculated dermal contact to the individual body region.

2.3 Dermal Contact Assessment Using the Hand Rinse Method

2.3.1 Sampling Protocol

Hand rinses shall be performed using the bag technique as first described by Durham and Wolfe (1962). The bag shall be a 5-1/2 in. by 15 in. polyethylene Whirlpak (No. B1027) supplied by NASCO of Fort Atkinson, Wisconsin. The hand rinses shall be transferred into 402 mason jars which shall be cleaned and labeled prior to the site visit using a barcode system.

A set of six identical barcode labels will be printed for each sample. Each set of labels will have a unique number. Prior to the site visit, all six labels shall be affixed to the outside of the mason jar. The sample numbers will be logged with a laptop computer and light pen using barcode software. At the survey site, one of the corresponding barcode labels from the mason jar will be transferred to a Dermal Assessment Data Sheet which contains pertinent information concerning the site location, sampling data, subject information, etc.

Prior to sampling the polyethylene bag shall be rinsed twice with 100 mL of distilled water. Discard the rinse water. Add 50 mL of distilled water to the bag. Insert the subject's hand into the bag. While the bag tightly held below the wrist bone, the hand is shaken vigorously in the distilled water for 50 shakes. Allow the water to drain from the hand for 10 s before removing the hand. Transfer the wash water to a clean, 4 oz wide-mouth mason jar. The mason jars are to be cleaned prior to the site visit with Alconox followed by two rinses with deionized water. Rinse the bag with 25 mL of distilled water and add the rinse to the wash water in the mason jar. Seal the mason jar with a Teflon-lined cap. Seal each sample in a plastic bag and place them in a chest containing ice or blue ice packs for transport to the laboratory. Do not use dry ice. Ship the samples to the laboratory at the end of each sampling day using a next-day delivery service.

Hand rinses should be conducted at the start of the workshift, immediately after the equipment assembly operation and immediately after the equipment disassembly operation. For manhole sealing operations a hand rinse should be performed immediately after the injection gun operator exits the manhole. A hand rinse should be performed after the workers remove their protective clothing at break periods and at the end of the day.

2.3.2 Quality Control Field Samples

On each day of sampling two field blanks and two spiked samples shall be prepared. The field blanks are prepared using the same method and materials as the samples. Pre-rinse the fresh polyethylene bag as described above but instead of discarding the water transfer the rinse water into a clean prelabeled wide-mouth mason jar. Next add 50 mL of distilled water to the bag, shake vigorously 50 times and transfer the distilled water to a second prelabeled mason jar. Post-rinse the bag as described in the sampling procedure. Seal the mason jar with a Teflon-lined cap and place the field blank in the ice chest with the other samples. One field blank should be prepared at the beginning of the sampling period and the other at the end.

Two spiked field samples shall be prepared using the same method as the blank, except that a known amount of acrylamide is added to the distilled water just prior to shaking the bag. The concentration of acrylamide in the fortified samples should be approximately the concentration expected in hand washes. One spiked field sample should be prepared at the beginning of the sampling period and the other at the end.

3.0 EQUIPMENT WIPE SAMPLING PROTOCOL

Wipe samples shall be collected on representative surfaces of equipment regularly handled by the chemical grouting operators to evaluate surface contamination. The collecting media will be 37-mm glass fiber filters moistened with distilled water. Clean disposable surgical gloves (individual packaged powderless type) will be worn whenever the filters are handled. Care should be taken not to contaminate the exterior of the glove when they are donned.

The wipe samples are to be shipped back to the lab in glass vials. Prior to the site visit, the vials shall be cleaned with Alconox and rinsed twice with deionized water. The sample vials are to be labeled using barcode adhesive labels. A set of six identical barcode labels will be printed for each sample. Each set of labels will have a unique number. One of the adhesive labels in the set shall be affixed to the sample vial and then placed into individual zip-lock plastic bags. Another adhesive label from the same set shall be affixed to the outside of the plastic bag. The other labels in the set shall be placed inside the plastic bag. The sample numbers will be logged with a laptop computer and light pen using barcode software.

At the sampling site, remove the filter from the packaging while wearing clean disposable gloves. Moisten the filter with distilled water. Gently wipe approximately 100 cm^2 of the surface to be sampled. Use a measured 100 cm^2 template as a guide to judge the size of the area to be wiped. Without allowing the filter to contact any other surfaces, fold the filter with the exposed side in, then fold it again. Return the filter to the labeled glass vial and seal with a teflon lined cap. Affix one of the barcode labels on a Wipe Sampling Data Sheet and record the pertinent information concerning the site location, complete description of the sampling location, etc., that can be traced to the unique sample number. Seal the vials in its

original plastic bag and store them in a chest containing ice or blue ice packs for transport to the laboratory. Do not use dry ice. Ship the samples to the laboratory at the end of each sampling day using a next-day delivery service.

Wipe sampling shall be conducted on the following equipment; top of the control panel desk in the Mobile Reveal and Seal Unit, outside of each mixing tank after mixing operations, the back of work gloves, inside and outside of respirators, the handle of the injection gun (manhole sealing operations only), side of the packer (main and lateral line operations only), the hydraulic hose connected to the injection gun or packer, and the side of a safety cone in the road. Wipe sampling need not be limited to this list. Wipe samples should be collected on any other frequently handled equipment that appear to be contaminated with grouting material.

3.1 Quality Control Field Samples

On each day of sampling two field blanks and two spiked wipe samples shall be prepared. Field blanks of the collection media should be handled in the same manner as the exposed filter media. Moisten a wipe filter with distilled water and immediately place the filter into a labeled vial container. One field blank shall be prepared at the beginning of the sampling period and one at the end of the sampling period. The blanks are to be stored and shipped with the exposed samples.

Two glass fiber filters are to be spiked with a known quantity of acrylamide diluted to the approximate concentration used in the grouting material (100 g of acrylamide per liter of water). The amount of acrylamide added to the filters should be approximately the levels that are expected to be collected on the wipe samples during the field studies. One spiked sample shall be prepared at the beginning of the sampling period and the other at the end of the sampling period. The spiked samples should be handled, stored and shipped in the same manner as the exposed samples.

4.0 REFERENCES

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APPENDIX B

FIELD OBSERVATION SHEETS

GENERAL OBSERVATIONS - Sewer Chemical Grouting Operation

Site ID: _____ Date: _____ Time: _____
Signature: _____ Title: _____

I. GROUTING CONTRACTOR INFORMATION

Name: _____
Address: _____
_____

II. DESCRIPTION OF SITE

Street: _____
_____
County: _____ City: _____
State: _____

III. WORKFORCE

A. Number of employees on site: _____

B. Workforce Description

	Job Title:	Duties:
1.	_____	_____
2.	_____	_____
3.	_____	_____
4.	_____	_____
5.	_____	_____

Site ID:_____Date: _____Time: _____
Signature:_____Title: _____

IV. EMPLOYEE INFORMATION

NOTE: Fill out one information sheet for each employee on site

Employee Name: _____Job Title: _____

	Yes	No	Comments:
A. Has this employee received training? If yes, specify.	[]	[]	_____
B. Is this employee licensed? If yes, specify.	[]	[]	_____
C. Did the grouting material come in contact with this employee's skin? If yes, specify.	[]	[]	_____

D. Has this employee experienced any of the following symptoms since he/she has worked with grouting chemicals?

	Yes	No	
1. Shortness of Breath?	[]	[]	_____
2. Muscular weakness of the hands, arms legs or feet?	[]	[]	_____
3. Numbness or tingling of the hands or feet?	[]	[]	_____
4. Excessive sweating of the hands or feet?	[]	[]	_____
5. Red or peeling skin of the hands or feet?	[]	[]	_____
6. Excessive fatigue or lethargy?	[]	[]	_____

Site ID:_____Date: _____Time: _____
Signature:_____Title: _____

V. PRE-OPERATION SAFETY PROCEDURES:

A. Does this company have a written safety and health program?	Yes []	No []
B. Do the employees know the company's safety and health policy?	Yes []	No []
C. Are employees aware of the hazards of confined space entry?	Yes []	No []
D. Was an entry permit issued from the sewer district?	Yes []	No []
E. Was confined space atmospheric testing performed prior to entry into the sewer?	Yes []	No []
F. Was confined space atmospheric testing performed during the operation?	Yes []	No []
G. Was the airspace in the sewer ventilated prior to entry?	Yes []	No []
H. Was the airspace in the sewer ventilated during the qrouting operation?	Yes []	No []
I. Were procedures for stand-by, communication and rescue followed?	Yes []	No []
J. Is a first aid kit available?	Yes []	No []
K. Are any employees on site trained in first aid and/or CPR?	Yes []	No []
L. Are spillage cleanup kits available?	Yes []	No []
M. Is safety equipment kept in good operating condition?	Yes []	No []
N. Are adequate skin and eye washing facilities available on site?	Yes []	No []
O. Are respirators fit tested? Do beard, sideburn, or temple of glasses, etc. interfere with respirator fit?	Yes []	No []

Site ID:_____ Date: _____ Time: _____
Signature:_____ Title: _____

VI. PERSONAL PROTECTIVE EQUIPMENT (PPE)

NOTE: Fill out one information sheet for each employee on site

Job Title:_____

	Yes	No	Comments:
A. Impervious gloves? Specify type.	[]	[]	_____
B. Glove liners? Specify type.	[]	[]	_____
C. Impervious Coveralls? Specify type.	[]	[]	_____
D. Safety harness?	[]	[]	_____
E. Respiratory Protection? Specify type.	[]	[]	_____
F. Head protection? Specify type.	[]	[]	_____
G. Eye protection? Specify type.	[]	[]	_____
H. Impervious boots? Specify type.	[]	[]	_____
I. Change of Clothes?	[]	[]	_____

Comments on general condition of PPE:_____

Site ID:_____Date: _____Time: _____
Signature:_____Title: _____

VII. CHEMICAL GROUTING PROCESS:

Type of grouting operation performed? [] Main line sealing
[] Lateral line sealing
[] Manhole sealing

Step 1. Equipment preparation and assembly - WORKER SAMPLER _____

A. Is there visible grouting residue on the equipment? Yes [] No []

B. Are gloves and protective clothing worn by the operators during the assembly process? Yes [] No []

C. Was direct skin contact to grouting materials observed during this operation? Yes [] No []

General Observations: _____

D. Approximate duration of assembly operation. _____minutes

Step 2. Installing the equipment assembly in the manhole WORKER - SAMPLER _____

A. Are gloves and protective clothing worn by the operators during the installation process? Yes [] No []

B. Was direct skin contact to grouting materials observed during this operation? Yes [] No []

General Observations: _____

C. Approximate duration of installation operation. _____minutes

Site ID:_____Date: _____Time: _____
Signature:_____Title: _____

Step 3. Chemical grout mixing operation

A. Product name of grouting material(s):

_____
_____
_____

B. Name and address of manufacturer(s):

_____
_____
_____
_____

C. The monomer used was [] acrylamide [] acrylate ONSITE

	Yes	No
D. Was spillage observed during the mixing process?	[]	[]
E. Was spillage observed during the gel test?	[]	[]
F. Was spillage immediately cleaned up? (specify method in general comments section)	[]	[]
G. Was visible airborne dust observed during the mixing operation? (acrylamide only)	[]	[]
H. Was direct skin contact with chemical grouting materials observed during the mixing operation?	[]	[]
I. Was the mixing area ventilated? (specify in general comments section)	[]	[]
J. Was respiratory protection used during the mixing process?	[]	[]
K. Were bags, cups, grout test samples disposed of in enclosed containers?	[]	[]

L. Approximate duration of each batch mixing operation:

_____

M. Quantities of chemical grout materials used:

_____

General Comments:_____
_____
_____
_____
_____

Site ID:_____Date: _____Time: _____
Signature:_____Title: _____

Step 4. Chemical grout injection operation ON SITE

A. Was the injection process performed remotely using a packer and video camera?	Yes []	No []
B. Was the chemical grout injected manually using an injection gun?	Yes []	No []
C. Was respiratory protection used during manual injection operations?	Yes []	No []
D. Were there any apparent leaks in the injection equipment?	Yes []	No []
E. Was direct skin contact to grouting chemicals observed during the pumping operation?	Yes []	No []
F. Was the injection gun equipped with an anti-splash back guard? (manual operation only)	Yes []	No []

General Observations: _____

G. Approximate duration of pumping operation. _____minutes

Site ID:_____Date: _____Time: _____
Signature:_____Title: _____

Step 5. Equipment disassembly and cleanup

A. Was spillage observed during the equipment disassembly operation?	Yes []	No []
B. Was direct skin contact with grouting materials observed during the disassembly operation?	Yes []	No []
C. Was direct skin contact with contaminated protective equipment observed when it was removed?	Yes []	No []
D. Is grouting equipment routinely washed at the end of the day?	Yes []	No []
E. Are respirators and protective clothing routinely cleaned after each use?	Yes []	No []
F. Are respirator and protective clothing properly disposed of or otherwise stored after use?	Yes []	No []
G. Are disposal containers equipped with a tight fitting lid?	Yes []	No []
H. Are disposal and storage containers properly labelled as to their contents?	Yes []	No []

General observations and comments: _____

I. Approximate duration of disassembly operation. _____minutes

J. Approximate duration of cleanup operation._____minutes

Site ID:_____Date: _____Time: _____
Signature:_____Title: _____

VIII. SCHEMATICS

A. Worksite:

B. Service van:

APPENDIX C

ANALYTICAL PROTOCOL

1.0 Analytical Method for Acrylamide

1.1 This method has been developed for the determination of trace quantities of acrylamide in field test samples (dermal pads, surface wipes, air monitors, and hand rinses) for assessment of worker exposure to acrylamide during chemical grouting operations.

1.2 This method yields the total weight of acrylamide present per field sample. Information about the exposure levels of acrylamide must be related to the type of sample and collection parameters.

2.0 SUMMARY OF METHOD

This method describes the procedures to determine the total quantity of acrylamide present in field test samples. A general diagram of the method is shown in Figure C-1.

The analysis procedure will consist of extracting acrylamide from field test samples with a minimum known volume of acidic (pH 3.7) water. An aliquot of the sample extract will be filtered and analyzed by HPLC. The chromatograms of the sample extracts will then be obtained and digitized by means of an A/D converter box interfaced with the HPLC system. One or more field blank samples will be extracted and analyzed concomitantly for comparative purposes. All of the sample and blank chromatographs will be stored on floppy disks for future data manipulation.

3.0 INTERFERENCES

3.1 Due to the nature of the analytical technique used, this method is susceptible to low UV interferences. Glassware should therefore be thoroughly rinsed with the solvent before use.

3.2 All glassware used in the sampling and analytical procedures will be thoroughly cleaned with Alconox and rinsed twice with deionized water.

4.0 SAFETY

All manipulations made with acrylamide samples should be performed in a fume hood or glove box. Gloves and other appropriate safety apparel should be worn at all times. Solid and liquid waste should be disposed of in the proper manner.

5.0 APPARATUS AND MATERIALS

5.1 Solution Preparation

5.1.1 1,000 mL graduated cylinder

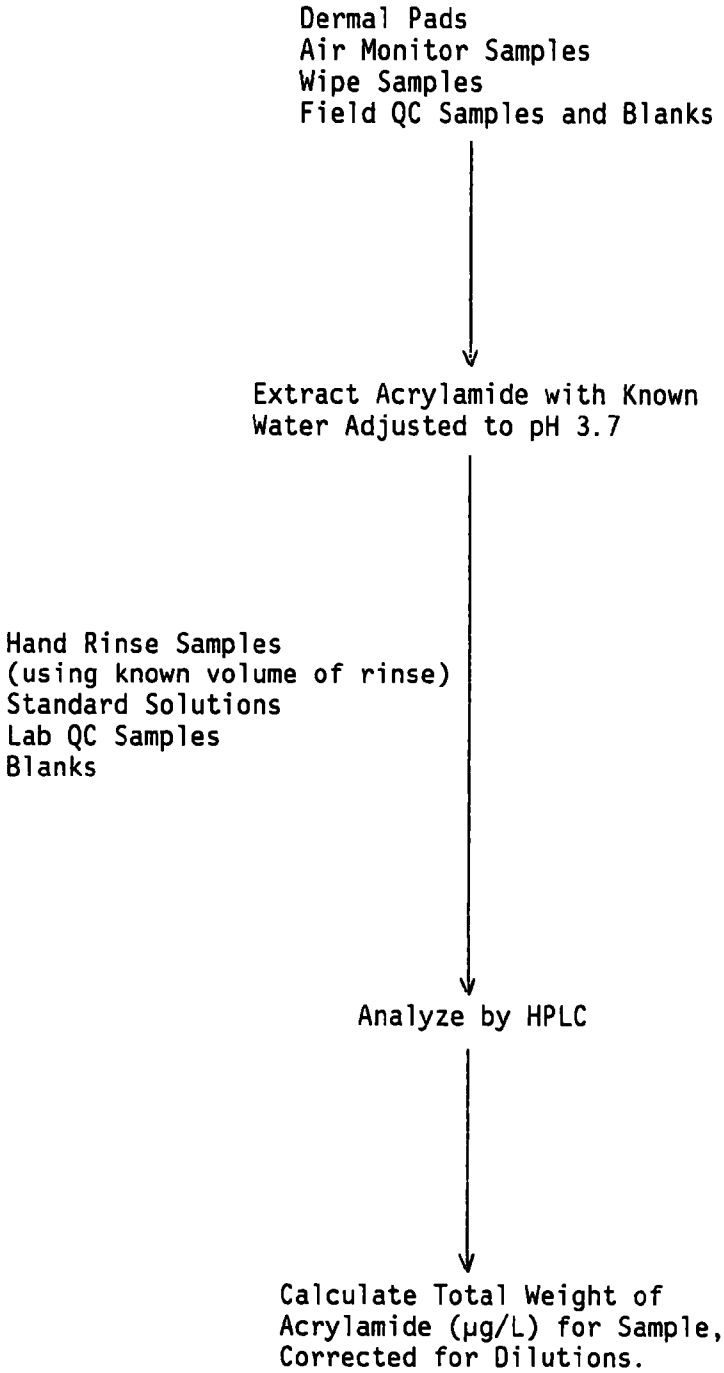

Figure C-1. Flow chart for the determination of acrylamide in field samples.

5.1.2 1 gal. glass bottle

5.1.3 Glass volumetric pipets (TD) - 2, 4, 5, 6, 8, and 10 mL

5.1.4 Volumetric flasks - 50, 100, and 250 mL (low actinic or foil-wrapped)

5.1.5 Disposable pipettes

5.1.6 Beakers - 100 mL

5.1.7 Filters - (0.2) µm Millex FG membranes (Millipore)

5.1.8 Glass jars (opaque) - 4 oz with Teflon®-lined lid liners

5.1.9 10-mL disposable syringe (Luer tip)

5.2 Balance - Analytical capable of accurately weighing to 0.00001 g.

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

5.4 Ultrasonic bath

5.5 HPLC data storage system

5.5.1 HPLC system

Instrument: Varian Model 5000 liquid chromatograph with autosampler Varian UV-50 variable wavelength detector, Heath Model 255B chart recorder

Column: Altex Ultrasphere (TM) ODS dp = 5u, 4.6 mm x 25 cm

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

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

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

6.0 REAGENTS

6.1 Acrylamide, electrophoresis grade

6.2 Deionized water, Milli-Q water system

6.3 Sulfuric acid, reagent grade

7.0 METHOD VALIDATION

The analytical method will be validated by evaluating the method for accuracy, linearity, and precision. Recovery will be determined by spiking duplicate blank sample collecting media with a known quantity of acrylamide. Validation will be confirmed if the average recovery efficiency for acrylamide falls in the range 70 to 130%.

8.0 SAMPLE STORAGE

8.1 Unless instructed otherwise, field samples will be stored in the dark at subambient temperature in their original packing containers until the analysis is completed.

8.2 Individual samples will be retained at the discretion of EPA. Subambient temperature storage is advised if samples are to be retained for long periods of time.

9.0 SAMPLE EXTRACTION

9.1 Carefully transfer the filter or train adsorbent sample to a 4-oz opaque glass jar. Pipette 20 mL of water (pH 3.7) into the cassette holder, cap tightly, and shake the cassette vigorously for 30 s.

9.2 Transfer the cassette rinse solution to the 4-oz jar using a disposable glass pipette.

9.3 Place the jar in a shaker for 10 min. The shaker must oscillate at least once per second.

9.4 The extract solution must be analyzed the same day of preparation.

10.0 STANDARD SOLUTION PREPARATION

10.1 Prepare a stock standard solution by accurately weighing (to the nearest 0.1 mg) approximately 500 mg of electrophoresis grade acrylamide and transferring the chemical to a 100-mL volumetric flask. Dissolve the chemical to a 100-mL volume by adding the acidic water-solution (concentration = 5,000 µg/mL).

10.2 Dilute the stock standard solution prepared in 10.1, 5 mL to 250 mL to make a 100 µg/mL standard solution.

10.3 Dilute the 100 µg/mL standard solution, prepared in 10.2, 5 mL to 50 mL to make a 10 µg/mL standard solution.

10.4 Dilute the 10 µg/mL standard solution, prepared in 10.3, 5 mL to 50 mL to make a 1 µg/mL standard solution.

10.5 Dilute the 1 µg/mL standard solution, prepared in 10.4, 5 mL to 50 mL to make a 0.1 µg/mL standard solution.

10.6 Standard solutions should be analyzed the same day of preparation.

10.7 Prepare check standard solutions by repeating steps 10.1 through 10.3.

11.0 PREPARATION OF SPIKED FILTER BLANKS

11.1 Place a blank filter into a 4-oz glass jar.

11.2 Using a 50-µL syringe, carefully load 30 µL of the acrylamide stock standard solution from Step 10.1 onto the filter.

11.3 Analyze this spiked filter blank with the samples the same day of preparation according to the procedures outlined in Steps 9.1-9.4.

11.4 Repeat Steps 11.1-11.5 for the duplicate spiked filter blank.

12.0 ANALYSIS OF ACRYLAMIDE SOLUTIONS

12.1 HPLC Operating System

Instrument: Varian Model 5000 Liquid Chromatograph with Autosampler
Varian UV-50 variable wavelength detector, Heath Model 255B chart recorder
Column: Altex Ultrasphere (TM) ODS du = 5 µ, 4.6 mm x 25 cm
Eluting Solvent: Water adjusted to pH 3.7 with sulfuric acid: H_2O (1:10 v/v)
Flow: 1.0 mL/min
Detection: UV at 200 nm
Chart: 0.1 in/min
Injection volume: 100 µL
Retention time of acrylamide: ~ 4 min

12.2 Analog/Digital Computer Interface Box Operating Parameters

Maximum input voltage: 10 V
Run time: 8 min
Sampling time: 1 point/s

12.3 Analysis of Acrylamide Solutions

12.3.1 Withdraw a portion of each standard, blank, or sample into a 10-mL disposable syringe, attach a 0.2-µm Millex GF filter onto the end, and filter into a autosampler vial.

12.3.2 Establish the calibration curve by injecting the series of standards prepared in Steps 10.2-10.6 in duplicate using the HPLC/data system described in 10.1-10.2. Adjust the absorbance range attenuation and injection volume to make the acrylamide peak of the highest concentrated standard ~ 90% full scale on the chart recorder. Analyze the samples using duplicate injection with a mid-point standard injected after every five samples to monitor the performance of the HPLC system. Start the A/D box at the beginning of the run.

12.3.3 If the concentration of the sample exceeds the linear range of the calibration curve, prepare an appropriate dilution of that sample and reanalyze it.

12.3.4 Plot the data file in the "re-detect" mode of the integration software.

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

13.0 CALCULATIONS

13.1 Using the integration software from Nelson analytical, prepare a peak summary table of the integrated areas of the analysis.

13.2 Evaluate the calibration curve by calculating the correlation coefficient and linear regression equation for the standard data. The correlation coefficient should be greater than 0.995. Calculate the relative standard deviation (RSD) for the responses of the mid-point standard solution that was injected throughout the analysis. The RSD should not exceed ±20%.

13.3 Calculate the total weight of acrylamide in each sample using peak areas and the linear regression equation computed from the standard data, corrected for sample dilution.

13.4 Calculate the recovery values for the spiked standards using the following formula:

$$\text{Recovery (\%)} = \frac{\text{Found weight of acrylamide}}{\text{Actual weight of acrylamide spiked}} \times 100$$

13.5 Evaluate the precision of the analytical system by preparing a control chart of the responses from the mid-point standard solution that was injected throughout the analysis of the samples.

14.0 REFERENCES

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