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LTV Steel Company  
Pittsburgh, Pennsylvania

NIOSH Investigators:  
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## I. SUMMARY

In October 1989, the National Institute for Occupational Safety and Health (NIOSH) received a request to investigate the occurrence of work-related dermatitis at the LTV Steel Company's coke oven plant in Pittsburgh, Pennsylvania. NIOSH investigators visited the facility on January 18-19, 1990, to assist in identifying a potential causative agent for the dermatological conditions experienced by employees who clean the coke oven gas inlets.

LTV Steel and the union representing LTV employees, the United Steelworkers of America (USWA), District 20, had been jointly investigating these skin problems. In 1989, 11 out of a total of 26 employees who were identified (by LTV and the USWA) as experiencing symptoms were referred to the University of Pittsburgh, Department of Dermatology for further evaluation. These employees were patch tested using suspected causative agents, which included coke oven gas condensates distilled into three fractions, monoethanolamine, and ferrameen. It was concluded that 9 of these 11 employees had an occupational allergic contact dermatitis; however, the specific occupational allergen was not determined.

The NIOSH investigation consisted of reviewing pertinent records maintained at the plant and the results from the patch testing performed by the University of Pittsburgh. Private interviews with 10 employees who had a history of dermatitis were conducted. The investigation also consisted of observing work practices, with particular attention to the process of cleaning the gas inlets and the use of personal protective equipment. Four bulk samples of the coke oven gas condensate were collected to be separated into the same fractions used in the patch testing and then analyzed to identify their constituents.

The 9 employees who were diagnosed as having occupational allergic contact dermatitis tested positive to at least one of the coke oven gas condensate fractions. These results indicated that the coke oven condensate was the most probable causative agent. Seven of these employees had the job classification of heater, while the other 2 included a helper and a patcher. The fractionation of the 4 bulk condensate samples did not proceed as expected and could not be used to isolate the causative agent. However, many compounds were identified as being present in the bulk condensate samples.

The dermatitis experienced by some of the LTV employees was probably caused by contact with the condensates from the coke oven gas. Fractionation attempts and subsequent laboratory analysis could not identify a particular causative agent. Recommendations are provided to assist in preventing contact with the condensates and the occurrence of further symptoms and new cases.

**KEYWORDS:** SIC 3312 (Steel Works, Blast Furnaces [Including Coke Ovens], and Rolling Mills), coke ovens, gas condensate, dermatitis, personal protective equipment.

## II. INTRODUCTION

In October 1989, the National Institute for Occupational Safety and Health (NIOSH) received a request to investigate the occurrence of work-related dermatitis at the LTV Steel Company's coke oven plant in Pittsburgh, Pennsylvania. The request was submitted by the United Steelworkers of America (USWA), District 20.

NIOSH investigators visited the facility on January 18-19, 1990. During this visit, an opening conference was attended by a representative from USWA Local 1843, management representatives from LTV Steel, and the company medical director. Following the opening conference, a walk-through survey of the coke oven batteries, both the Wilputte and Koppers ovens, and the by-products area was conducted. Informal discussions were held with 3 employees during the gas inlet cleaning process and a total of 4 bulk samples of the coke oven gas condensate were collected from both the Wilputte and Koppers batteries. Private interviews were held with employees having a history of dermatitis and pertinent records, including OSHA 200 logs, personnel, and medical, were reviewed.

Initial findings were presented to management and union representatives at the conclusion of the site visit. A subsequent status report was distributed on March 7, 1990.

## III. BACKGROUND

The LTV Steel coke oven facility consists of 5 batteries, with a total of 315 by-product ovens. Four of the 5 batteries consist of 59 Wilputte side-fire ovens per battery. The remaining battery consists of 79 Koppers-Becker underjet fired ovens. The coke oven gas from these batteries is routed to the by-products area, which consists of different processes to remove and recover the volatile products liberated during coking. The coke oven gas is then routed back to the ovens to be used as heating fuel.

Almost three years ago (1988), a skin problem of potential occupational origin was identified among the heaters, helpers, and patchers. This potential health hazard was brought to the union's attention when employees transferring into the above job categories began reporting skin problems. Apparently, many of the heaters, helpers, and patchers had skin problems for several years that were not previously recognized as potentially related to a common occupational exposure. Union and management have been working together to determine the nature of these skin problems. A list of 26 employees with skin problems was developed by union and management and provided to NIOSH investigators. These 26 employees had been evaluated by union and company physicians. In 1989, 11 of these employees were referred to the University of Pittsburgh, Department of Dermatology for further evaluation.

The suspected causative agent was a condensate from coke oven underfiring gas which collected on gas nozzle seats in the gas heating pipes of the Koppers and Wilputte coke oven batteries. Employees are exposed during the cleaning, patching, and repairing of the gas pipes. Samples of the condensate, its distilled fractions, and other possible constituents (monoethanolamine and ferrameen) were provided by LTV Steel to the University of Pittsburgh for patch testing.

Nine of the 11 employees had positive patch tests with the coke oven gas condensate obtained from the workplace and its distilled fractions. All 9 tested positive to the residue obtained by fractional distillation up to 200°C. Eight tested positive to the distilled fraction from 200°C to 250°C, and none tested positive to the distilled fraction

cut above 250°C. Seven employees tested positive to rich monoethanolamine, and none to ferrameen. It was concluded that these 9 employees had an occupational allergic contact dermatitis; however, the specific occupational allergen was not identified.

The cleaning process for the underjets of the Koppers-Becker is usually performed on a daily basis. These 79 coke ovens are heated by the combustion chambers contained in the 80 walls which separate the ovens. The pipes that feed gas to the underjets for each combustion chamber are alternately cleaned, so that the underjets for each oven are not cleaned on a daily basis. Three employees typically perform the cleaning operation. This task involves removing the caps from the extension pipes for each underjet, removing and cleaning the nozzles, and then cleaning the underjet pipe.

The potential exposures to the condensate initially occur when the caps are removed. The caps, located on the ends of the extension pipes which extend perpendicularly from the ceiling to a height approximately 7 feet above the floor, are usually filled with the condensate. Residual pressures within the pipes sometimes produce backfires which can spray the workers with small amounts of the condensate. These backfires are similar to those occurring in automobiles. The sidejets for the Wilputte ovens are also cleaned in a similar manner. However, the potential for exposure is not as great because the workers are not working directly beneath the pipes. The personal protective equipment worn by these employees included hard hats, safety glasses, coveralls, gloves, and Tyvek® sleeve extenders.

#### IV. METHODS

##### ENVIRONMENTAL

The industrial hygiene component of the investigation consisted of observing work practices, with particular attention to the process of cleaning the gas jets and use of personal protective equipment. Informal discussions were held with three employees during the cleaning process, and a total of 4 bulk samples of the coke gas condensate were collected from both the Koppers and Wilputte batteries. These samples were submitted to the NIOSH laboratory for separation via fractional distillation into the same fractions as used in the University of Pittsburgh's patch tests. A fractional distillation apparatus was set up, and approximately 50 milliliters (ml) of each sample were used for the fractionation procedure. Activated charcoal sorbent tubes were attached to the receiving flask exhaust to collect any vapors which may have escaped from the system. These sorbent tubes were then desorbed in either carbon disulfide or methylene chloride. The resulting distillation fractions and the eluates from the sorbent tubes were then analyzed by gas chromatography equipped with a mass selective detector (GC-MSD) to identify the major components.

##### MEDICAL

The medical investigation consisted of reviewing the OSHA 200 logs and records present at the plant, including both medical and personnel records. Private interviews were conducted with 10 employees who had a history of dermatitis. They were heaters, helpers, or patchers who work on the gas pipes under the Koppers and along side of the Wilputte coke oven batteries. Medical records of these employees, including dermatological evaluations conducted by the University of Pittsburgh, Department of Dermatology, by a dermatologist retained by the company, and by a consulting physician for the union, were reviewed. The Assistant Director of the Safety and Health

Department at the United Steelworkers of America International Union was contacted to discuss the occurrence of dermatitis at coke oven facilities.

## V. EVALUATION CRITERIA

Excluding disorders associated with repeated trauma, skin disorders are the most frequently reported occupational disease. Allergic contact dermatitis accounts for 25 to 30 percent of all occupational skin disorders. Allergic contact dermatitis is a cell-mediated hypersensitivity reaction beginning when a contact allergen penetrates the skin. The allergen is transformed to a complete antigen, whose presence stimulates T lymphocytes to divide. Clones of cells with specific sensitivity to the antigen are produced. On repeated contact, these cells evoke an inflammatory response characteristic of contact dermatitis.<sup>(1,2)</sup>

Exposure to a contact allergen produces sensitization in a small percentage of exposed individuals. Factors that may influence sensitization include nature of the allergen, exposure characteristics (e.g., dose, site, route, and frequency of exposure), host characteristics (e.g., preexisting irritant dermatitis, age, genetic predisposition, sex), and environmental conditions (e.g., temperature, humidity).<sup>(1,2)</sup>

The patch test is an integral part of the evaluation of patients with suspected allergic dermatitis. Properly conducted patch testing can often identify the specific substances causing the dermatitis. Approximately 2800 chemicals have been identified as sensitizers. From these, standard batteries of 20 to 30 different test materials have been developed. Nonstandard allergens can also be used in patch tests when specific chemical substances present in the workplace are suspected as the cause of contact dermatitis. Identification of the causative agent or source is crucial to preventing reoccurrence of dermatologic symptoms.<sup>(1,2)</sup>

## VI. RESULTS AND DISCUSSION

### ENVIRONMENTAL

The fractionation of the 4 bulk condensate samples did not proceed as expected. The temperature ranges for these fractions never reached the temperatures obtained for the fractions used in the patch testing. The temperature that was monitored at the top of the boiling flask never exceeded 160°C, and the temperature monitored at the top of the fractionating column rarely exceeded 100°C. Residue also coated the condenser, and white smoke was produced as the boiling flask temperature reached its upper limit. The smoke was collected using the charcoal tube at the receiving flask exhaust, and the residues were collected by rinsing with methanol. Since the fractionation did not proceed as expected, the potential differences between the fractions used during the patch test could not be determined.

The results for the analysis of the 4 bulk condensate samples identified many compounds. All of these bulks were collected from areas in the Koppers-Becker battery (#1-3) and the Wilputte battery adjacent to the Koppers-Becker (#4). Bulks 1, 3, and 4 were very similar, while Bulk 2 contained primarily water and could not be analyzed. Water was also a major component in the other samples, but these could still be analyzed. The other major components identified in these samples included hydrogen sulfide, acetonitrile, thiazole, pyridine, pyrrole, 1,2-ethanedithiol, 2-methylpyridine, and 2-thiazolidinethione. The charcoal tubes used during the fractionation of the bulks and

desorbed with carbon disulfide also had similar compounds. The chromatograms varied only in peak intensity; therefore, only one of these samples was analyzed by GC-MSD. The major peaks were identified as benzene and toluene. Other peaks identified were a  $C_6H_8$ , such as cyclohexadiene, thiophene, thiazole, pyridine, pyrrole, xylene isomers, and several aromatic hydrocarbons. Two of the charcoal tubes were desorbed with methylene chloride. Identified compounds in these samples included thiazole, pyridine, 2-methylpyridine, benzene, thiophene, toluene, xylene isomers, and several sulfur-containing compounds. Other components were also identified in all of these samples. The individual chromatographs and the other compounds identified can be found in a copy of the laboratory report which is provided as Appendix I. The residues remaining in the boiling pot during the fractionation attempts could not be analyzed by gas chromatography due to their high boiling points.

## MEDICAL

The 9 employees who were diagnosed as having occupational allergic contact dermatitis tested positive to at least one of the coke oven gas condensate fractions. Seven of these employees had the job classification of heater, while the other 2 included a helper and a patcher. Of the 2 employees who did not have positive reactions to any of the fractions of coke oven gas condensate, one was a laborer and the other a crane helper. Two employees required a job transfer, although one was transferred back upon his request. Reportedly, management and union have agreed to offer patch testing to 10 additional employees from the list of 26.

According to the interviewed employees, various types of personal protective equipment (PPE) have been used in the past, but none were totally effective at eliminating skin contact with the condensate. Presently, some of the exposed employees use disposable jackets with long sleeves or disposable long sleeves. Rubber, cloth, leather, or lid gloves (for hot jobs) are also used. Zinc oxide is used as a barrier cream. Several employees reported that handwashing facilities were easily accessible. Most stated that they wash their arms, hands, and/or face as frequently as needed to remove any contamination. Boraxo® liquid and powder soaps with paper towels are supplied in the restrooms. A physician from the University of Pittsburgh has recommended that middle of the forearm-length Allerderm® vinyl gloves be used in combination with cotton gloves as an inner layer.

The Assistant Director of the Safety and Health Department at the United Steelworkers of America International Union, the dermatologist from the University of Pittsburgh, and a consulting physician for the union were contacted to discuss dermatitis problems at coke ovens (including LTV). These individuals mentioned that there have been reports of workers at 2 other coke oven plants experiencing dermatitis problems. Individuals working on the gas jets under the coke oven batteries at one of the plants were reportedly experiencing an irritant-type of dermatitis. At the other coke oven plant, workers reportedly developed a dermatitis affecting the unexposed skin areas.

## VII. CONCLUSIONS

The dermatitis experienced by some of the LTV employees was probably caused by contact with the condensates from the coke oven gas. Fractionation attempts and subsequent laboratory analysis could not identify a particular causative agent. Recommendations are provided to assist in preventing contact with the condensates and the occurrence of further symptoms.

## VIII. RECOMMENDATIONS

1. Unfortunately, completely impervious chemical protective clothing does not exist. Therefore, the type of personal protective equipment that should be worn to prevent direct skin contact with the coke oven condensate should be based on the chemical resistance of the material to the agent being used. Many studies have demonstrated that some chemicals can permeate all commercially available chemical protective equipment.<sup>(3)</sup> Butyl rubber or Viton™ may be appropriate materials for gloves, while Saranex™ or polyethylene/ethylene vinyl alcohol laminated suits may be appropriate for whole body protection. Any protective suit worn should also have a hood which covers the head and the back of the neck.

The gloves that are selected should cover at least one-third of the forearm and fit snugly. Rubber and plastic gloves should be lined with cloth or another perspiration-absorbing material. Gloves should be replaced if they become torn or if the interior becomes contaminated. Also, the junction between the glove and the sleeve of the protective suit should be taped to prevent contamination via this route. It should be noted that, in some instances, gloves can sometimes cause skin problems, such as itching, sweating, and rashes. Possible causes for these reactions include allergenic substances in the glove material, the powder (if any) used on the interior of the glove, and occlusion effects.

Workers cleaning the gas jets should also wear hard hats equipped with full face shields to prevent condensate from contacting the face.

During the summer, heat stress may be a problem due to the hot environment created by the ovens and the personal protective suits. If heat stress is a concern, a variation in the protective equipment may be warranted. Since the greatest potential for exposure originates from above the worker's heads, the use of a hooded poncho, extending below the shoulders, may still provide adequate protection when used in combination with extension sleeves, gloves, hard hat, and face shield. The poncho and extension sleeves should be made from an appropriate material, such as those previously discussed. This type of equipment will reduce the risk of heat stress and may still adequately prevent skin contact. However, this variation should be studied to determine if it provides proper protection.

2. Exposure to the condensate may also occur during the donning and doffing of the protective equipment. Workers should be trained in the proper method of donning and doffing of their protective equipment. This training should also include the proper storage, maintenance, and replacement of all the protective equipment.
3. Any contamination of the skin should be removed promptly with mild soap and water. For difficult stains on the backs of the hands and forearms, a waterless hand cleaner should be used instead of abrasive soaps. The residual film from waterless hand cleaners should be washed off with mild soap and water. Abrasive soaps should only be used for removing difficult stains on the palms. Both waterless cleaners and abrasive soaps should be used sparingly and only when necessary. Use of a skin moisturizer after washing helps to replace skin oils that are removed by frequent washing.

In addition, it is particularly important that workers remove all contamination from their skin before going outdoors. Certain substances present in coal tar and pitch may cause a phototoxic dermatitis to develop following sun exposure. Reportedly, among the employees evaluated at the University of Pittsburgh, photopatch testing was negative.

4. Employees should shower at the end of the day in order to remove all traces of the condensate from the skin.
5. Any skin injury or disorder should be immediately reported to the plant medical department.
6. To monitor trends in the occurrence of dermatologic conditions, a log of skin complaints should be maintained by the plant medical department. Cases of

possible occupational dermatitis should be recorded by department and job category so that any problem areas can be identified as early as possible. This information, minus personal identifiers, can be made available to management and union representatives. Follow-up of high risk areas should include an industrial hygiene evaluation and a medical evaluation of similarly affected workers to determine work-relatedness. Based upon these evaluations appropriate control measures (e.g., engineering controls, product substitution, personal protective equipment, personal hygiene procedures) should be implemented.

7. Because of the importance of early diagnosis and treatment of occupational dermatoses, all skin problems more severe than dry skin should be referred to a dermatologist who is familiar with work-related skin diseases. The offering of patch testing should be based on a dermatologist's recommendations, and not on union and management decisions. A second medical opinion may sometimes be advisable because occupational dermatoses can be difficult to diagnose and treat.
8. Workers should be educated about the effects of the chemicals they work with and the types of work practices that will minimize their exposures to them.

#### IX. REFERENCES

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APPENDIX I



## APPENDIX I

## DEPARTMENT OF HEALTH &amp; HUMAN SERVICES

## Memorandum

Date July 11, 1990  
From Chemist, MDS, MRSB  
Subject Sequence 6989A,B,C; HHE 90-010: Analysis of Distillation Fractions of Coke Oven Gas Condensates.  
To Dawn Tharr, HETAB Lab Coordinator  
Attn: Greg Kinnes  
Through: Director, DPSE *GD* 7/12/90  
Chief, MRSB, DPSE *RL*

Introduction

Four bulk liquids of coke oven gas condensates were collected at LTV Steel. The bulks were submitted for fractionation via fractional distillation and the resulting fractions were to be analyzed qualitatively by GC-MS or other available techniques. Similar bulks have been fractionated by the company and three fractions were collected for each bulk. The first fraction was composed of components boiling at 200° C or less. The second contained components which boil between 200° and 250° C. The third fraction was the remaining residue which boiled above 250° C. Skin patch testing was done at the company using these various fractions and positive reactions were noted for all workers for the first fraction. Some workers also reacted positively to the second fraction. Photodermatitis due to PNA exposure was suspected as a cause for the positive skin patch reactions. Therefore, determination of PNA content was also requested for each fraction collected.

Method

A fractional distillation apparatus was set up and approximately 50 mL of each bulk was used for the fractionation procedure. The bulk was placed in a round bottom flask and heated via a heating mantle. The flask and the fractionating column were completely wrapped with glass wool for more uniform heating. The condenser was water cooled and led directly to a round bottom receiving flask. An SKC Lot 120 charcoal tube was attached to the receiving flask exhaust to collect any vapors which may have escaped from the system.

The fractionation did not proceed as expected. The temperature which was monitored at the top of the boiling flask never exceeded 160° C and the temperature monitored at the top of the fractionating column rarely exceeded 100° C. Residue also coated the condenser, although one fraction was collected in the receiving flask for each bulk. As the boiling flask temperature reached its upper temperature, white smoke poured through the system and was not condensed. Collection of

the smoke was accomplished somewhat using the charcoal tube at the receiving flask exhaust; however, the tube had to be changed for both Bulk 1 and 3 when the original tube became saturated. For each bulk, except Bulk 2, the condenser was rinsed with methanol and the rinse collected for analysis. There was no residue in the condenser for Bulk 2.

Each fraction collected was screened directly by GC-FID using a 30 meter DB-1 fused silica capillary column (split mode; 20:1 split), except for Bulk 3. The fraction collected from Bulk 3 solidified before analysis and had to be dissolved in methanol before analysis. It was screened along with the methanol rinses of the condenser. The front section of one charcoal tube sample associated with each bulk was desorbed for 30 minutes with 1 mL of CS<sub>2</sub>. The back sections and the remaining 2 charcoal tube samples from Bulks 1 and 3 were desorbed for 30 minutes with 1 ml of methylene chloride. The methanol rinses and the charcoal tube samples were screened as above except in the splitless mode.

#### Results and Discussion

The fraction from Bulks 1 and 4 were also analyzed by GC-MDS (splitless mode) for identification of detected peaks. The fraction from Bulk 2 was primarily water and extinguished the GC-FID flame during screening; therefore, it was not analyzed by GC-MSD. The peaks for each fraction were similar and water was identified as the major component. Other peaks also identified in both fractions were hydrogen sulfide, acetonitrile, thiazole, pyridine, pyrrole, 1,2-ethanedithiol, 2-methylpyridine, and 2-thiazolidinethione. Additional components identified in the fraction from Bulk 1 were 3-methylpyridine, a MW 89 C<sub>3</sub>H<sub>7</sub>NS such as thiazolidine, 2-methylthiazolidine, and ethanethioamide. Phenol was also detected in the fraction from Bulk 4.

The chromatograms of the fraction from Bulk 3 (which was dissolved in methanol) and the methanol rinse of the condenser from Bulk 3 were similar. Since the condenser rinse was more concentrated it was analyzed by GC-MSD (splitless mode) along with the condenser rinses from Bulks 1 and 4. The results of the mass spectral analyses are as follows:

Bulk 1: The major peaks identified were hydrogen sulfide, a C<sub>3</sub>H<sub>5</sub>NS such as ethyl thiocyanate, naphthalene, and a MW 136 such as 1,3-dithiolane-2-thione. Other peaks identified were thiazole, a MW 75 C<sub>2</sub>H<sub>5</sub>NO<sub>2</sub>, pyridine, pyrrole, 2-methylthiazole, 2- and 3-methylpyridine, 2-methylthiazoline, a C<sub>9</sub>H<sub>10</sub> such as a methylstyrene, indene or methylphenylacetylene, 1,4-dithione, 1,2,4-trithiolane, a methyl naphthalene, a MW 152 C<sub>4</sub>H<sub>8</sub>S<sub>3</sub>, a MW 152 such as biphenylene or acenaphthalene, a MW 168 such as dibenzofuran or naphthofuran, a MW 166 such as fluorene or phenalene, and a MW 178 such as anthracene or phenanthrene.

Bulk 3: The major peaks identified were the same as found in Bulk 1 as well as 2-methylthiazoline. Minor peaks were identified as thiazole, pyridine, pyrrole, 2-methylthiazole, 2- and 3-methylpyridine, phenol, indene or methylphenylacetylene, 1,4-dithiane, 1,2,4-trithiolane, a methylnaphthalene, and a MW 152  $C_4H_8S_3$ .

Bulk 4: The major peaks identified were hydrogen sulfide, indene or methylphenylacetylene, naphthalene, and a MW 136 such as 1,3-dithiolane-2-thione. Minor peaks identified were a MW 75  $C_2H_5NO_2$ , 2-methylthiazoline, styrene, a methylindene, 1,2,4-trithiolane, methylnaphthalenes, a MW 152  $C_4H_8S_3$ , a MW 154 such as acenaphthene or biphenyl, a MW 152 such as biphenylene or acenaphthalene, and a MW 168 such as dibenzofuran or naphthofuran.

The chromatograms of the charcoal tube samples desorbed with  $CS_2$  were all similar varying only in peak intensity; therefore, only one of the samples was analyzed by GC-MSD. Since the charcoal tube associated with Bulk 1 was most concentrated it was selected for GC-MSD analysis. The major peaks were identified as benzene and toluene. Other peaks identified were a  $C_6H_8$  such as cyclohexadiene, thiophene, thiazole, pyridine, pyrrole, the xylene isomers, MW 120 aromatic hydrocarbons such as trimethylbenzenes, benzofuran, a methylstyrene, indene or methylphenylacetylene, naphthalene, a  $C_9H_{10}OS$  such as S-ethyl thiobenzoate, and a MW 136 such as 1,3-dithiolane-2-thione.

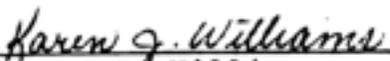
Both of the remaining charcoal tube samples desorbed with methylene chloride were analyzed by GC-MSD. The results of the analyses are as follows:

Bulk 1 CT-2: The major peaks identified were thiazole, pyridine, 2-methylpyridine, a xylene isomer or ethylbenzene, indene or methylphenylacetylene, and a MW 136 such as 1,3-dithiolane-2-thione. Other peaks identified were ethyl cyanide, n-propyl cyanide, benzene, thiophene, diethyl sulfide, pyrrole, methylthiophene isomers, 2-methylthiazole, 3-methylpyridine, xylene isomers, an ethyl thiophene isomer, styrene, MW 120 aromatic hydrocarbons such as trimethylbenzenes, indan or a methylstyrene, a methylindene, naphthalene, and MW 154, MW 152, and MW 184 sulfur containing compounds.

Bulk 3 CT-2: The major peaks identified were benzene, thiophene, thiazole, toluene, the xylene isomers, and a MW 154 sulfur containing compound. Other peaks identified were methyl ethyl ketone, isobutanol, diethyl sulfide, n-heptane, pyridine, pyrrole, 2-methylthiazole, n-octane, a MW 108  $C_3H_8S_2$  such as methyl ethyl disulfide, diethyl disulfide, dimethyl trisulfide, a MW 120 aromatic hydrocarbon such as a trimethylbenzene, and MW 140, MW 156, MW 170 and MW 188 sulfur containing compounds.

Copies of the reconstructed total ion chromatograms of the mass spectral analyses of the above samples are attached with all identified peaks labelled. No additional peaks not detected on the front section were detected on the backup section of the charcoal tube samples.

The third fraction generated from each of the bulk samples, consisting of residue remaining in the boiling pot, could not be analyzed by gas chromatography. The components had boiling points which were too high to analyze via this technique. Analysis of these fractions is now on hold.

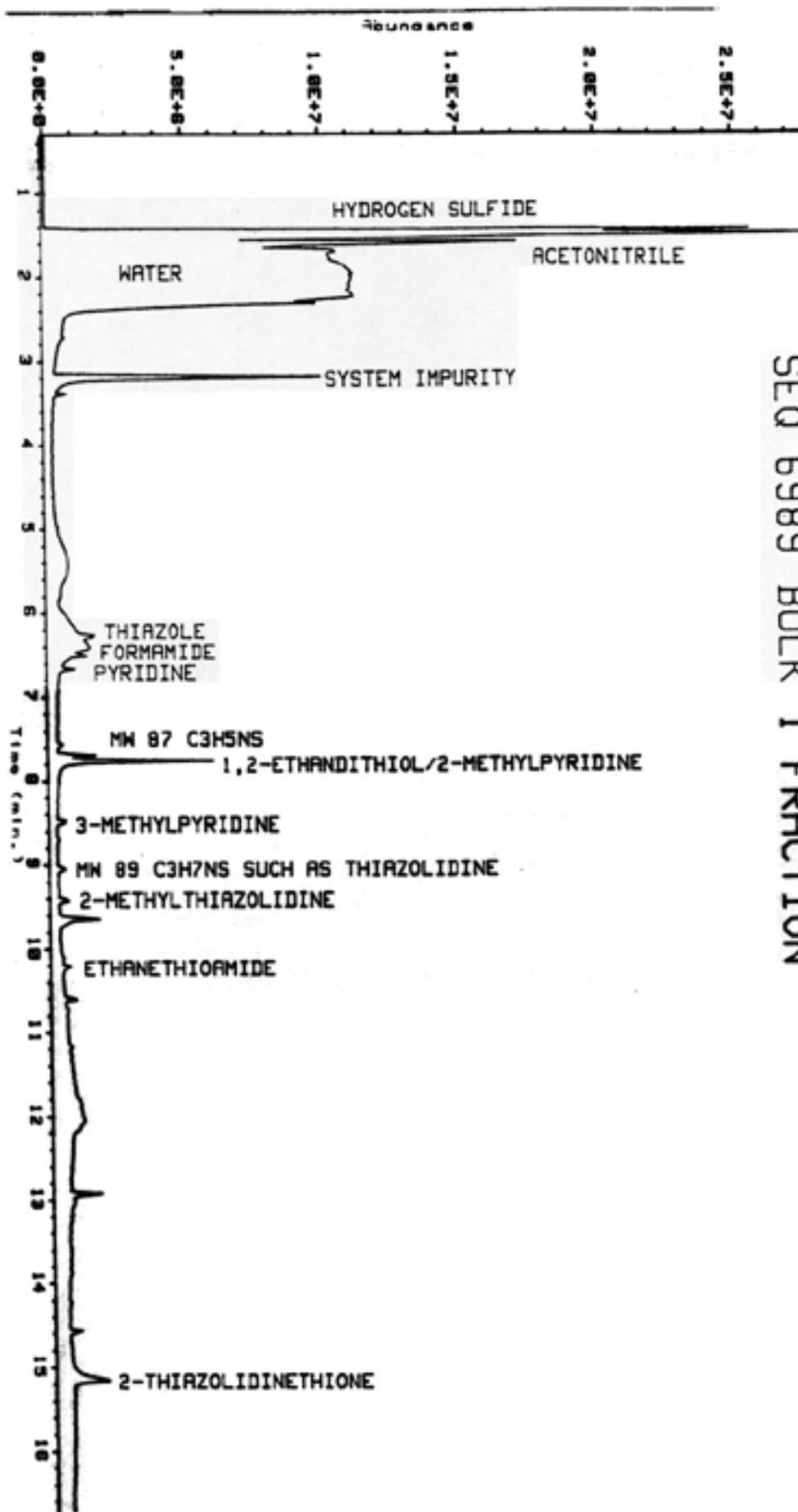
  
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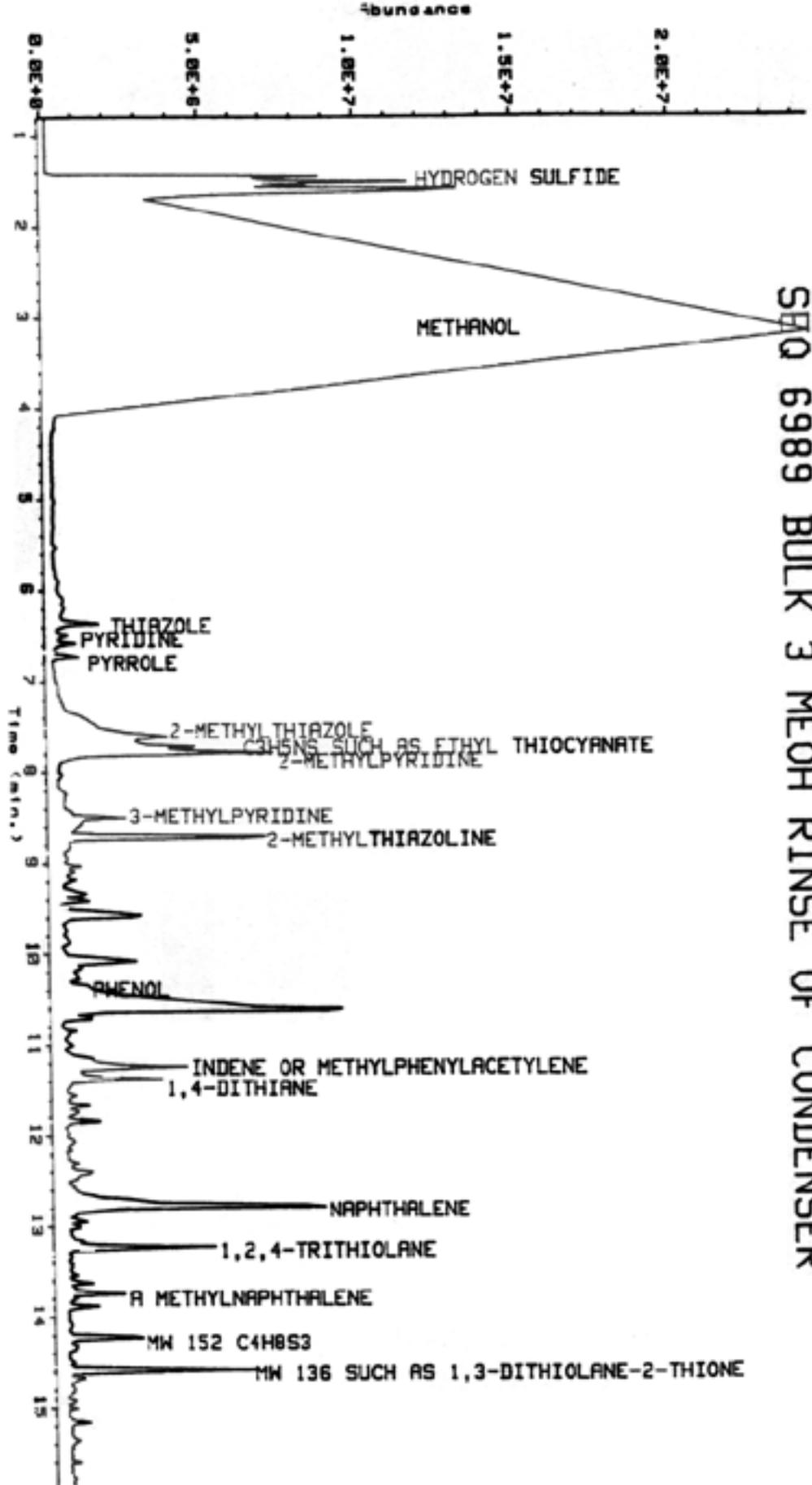
Attachment

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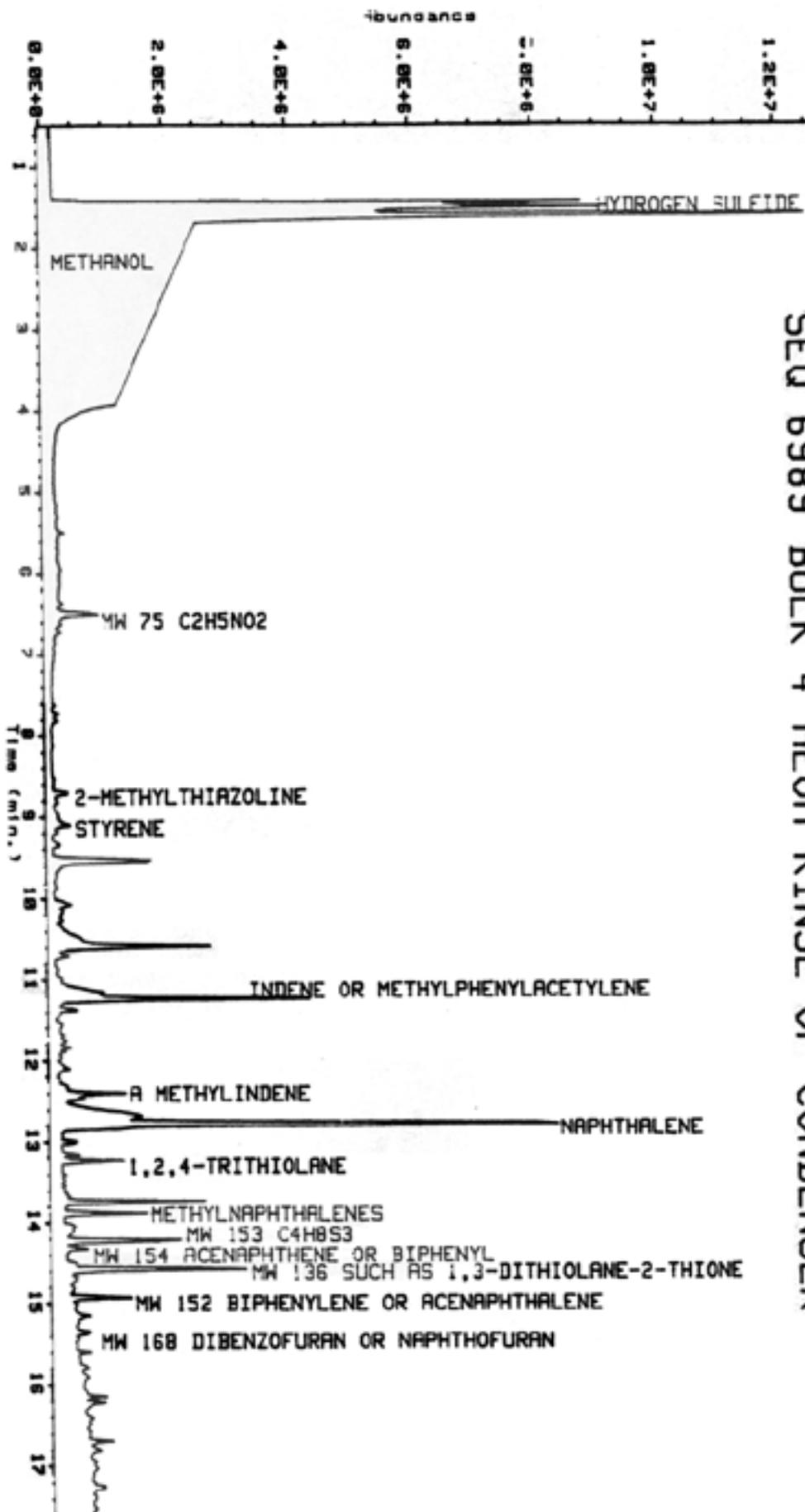
SEQ 6989 BULK 1 FRACTION

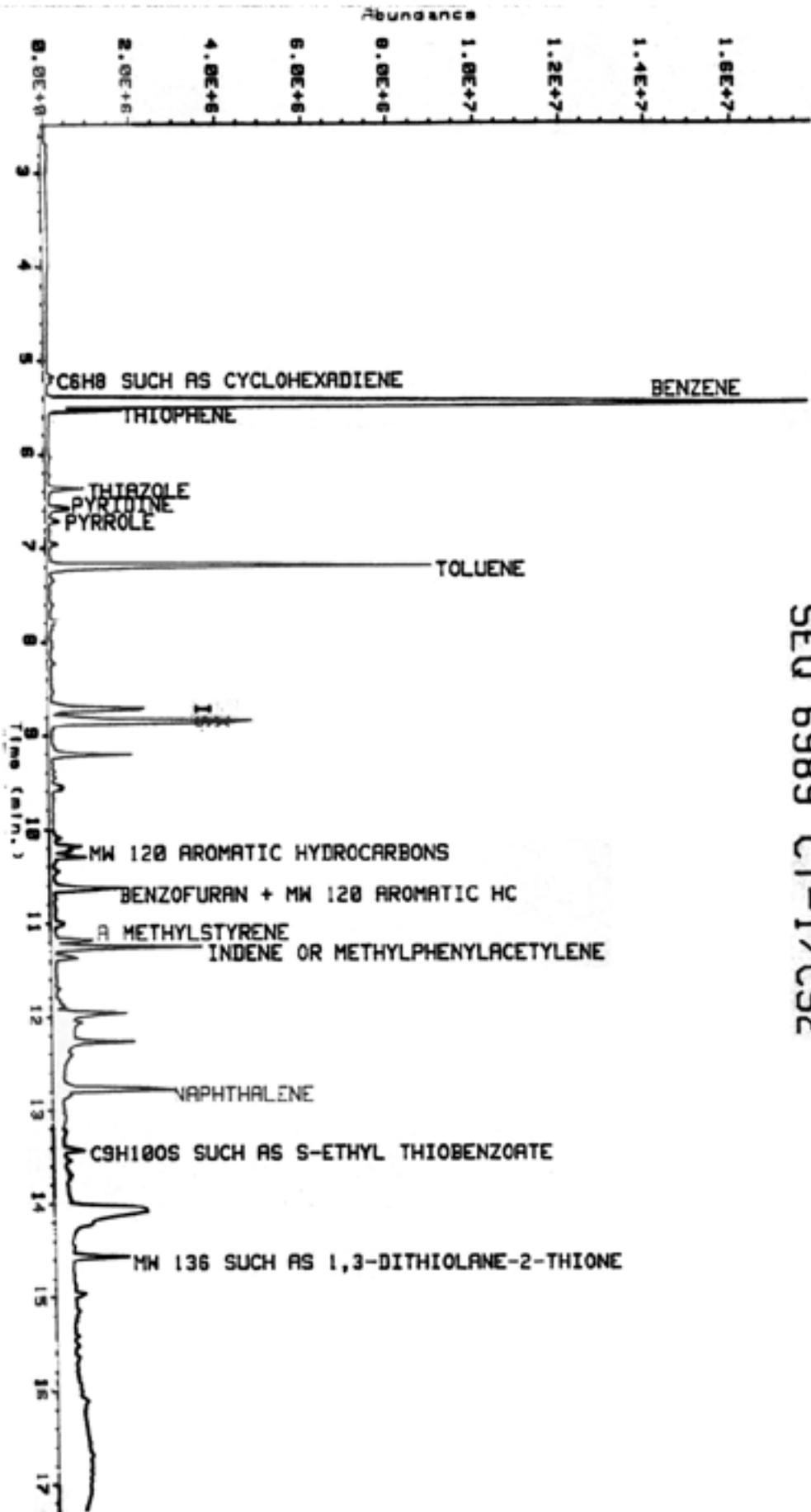


SAQ 6989 BULK 3 MEOH RINSE OF CONDENSER

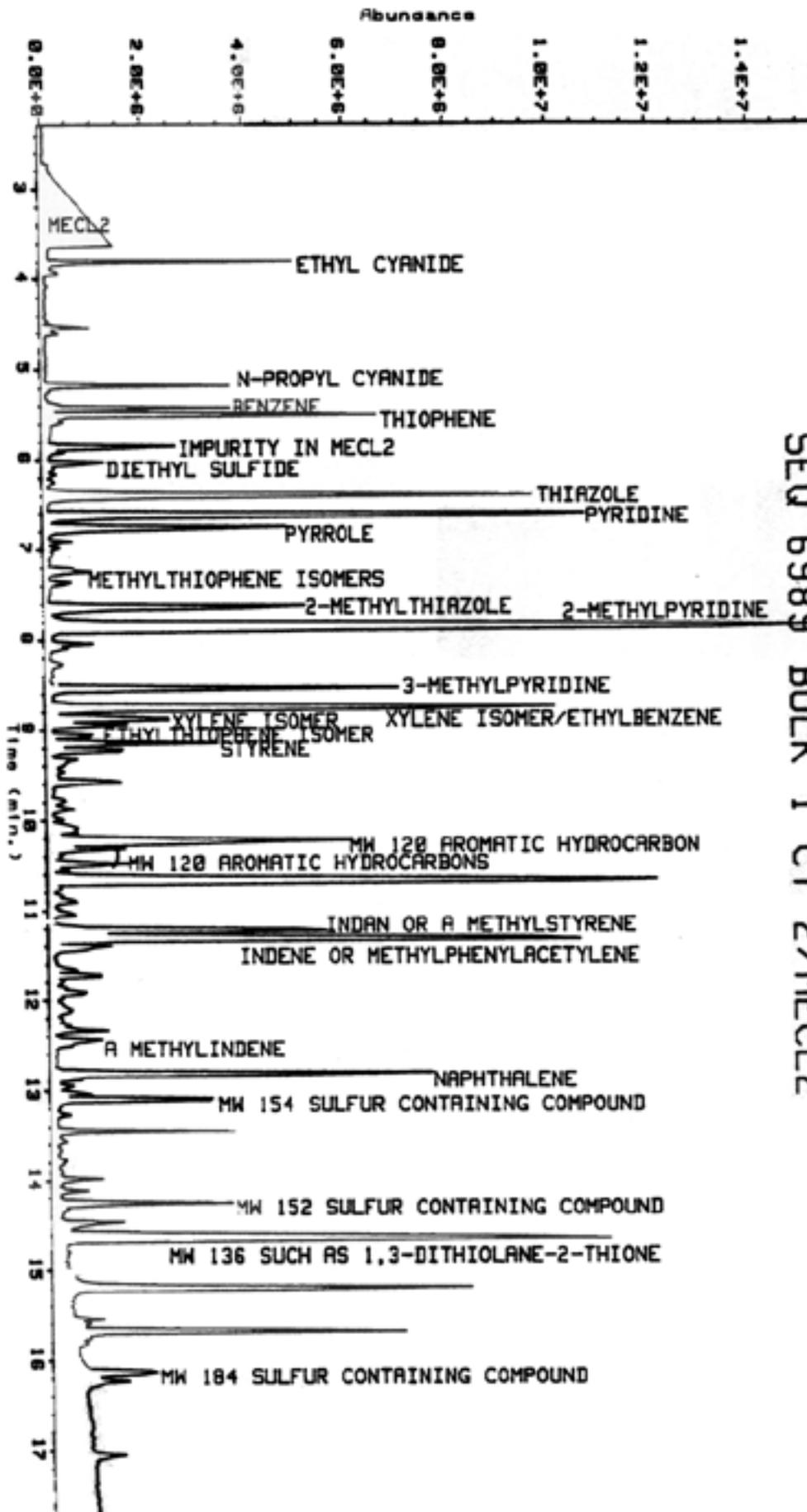


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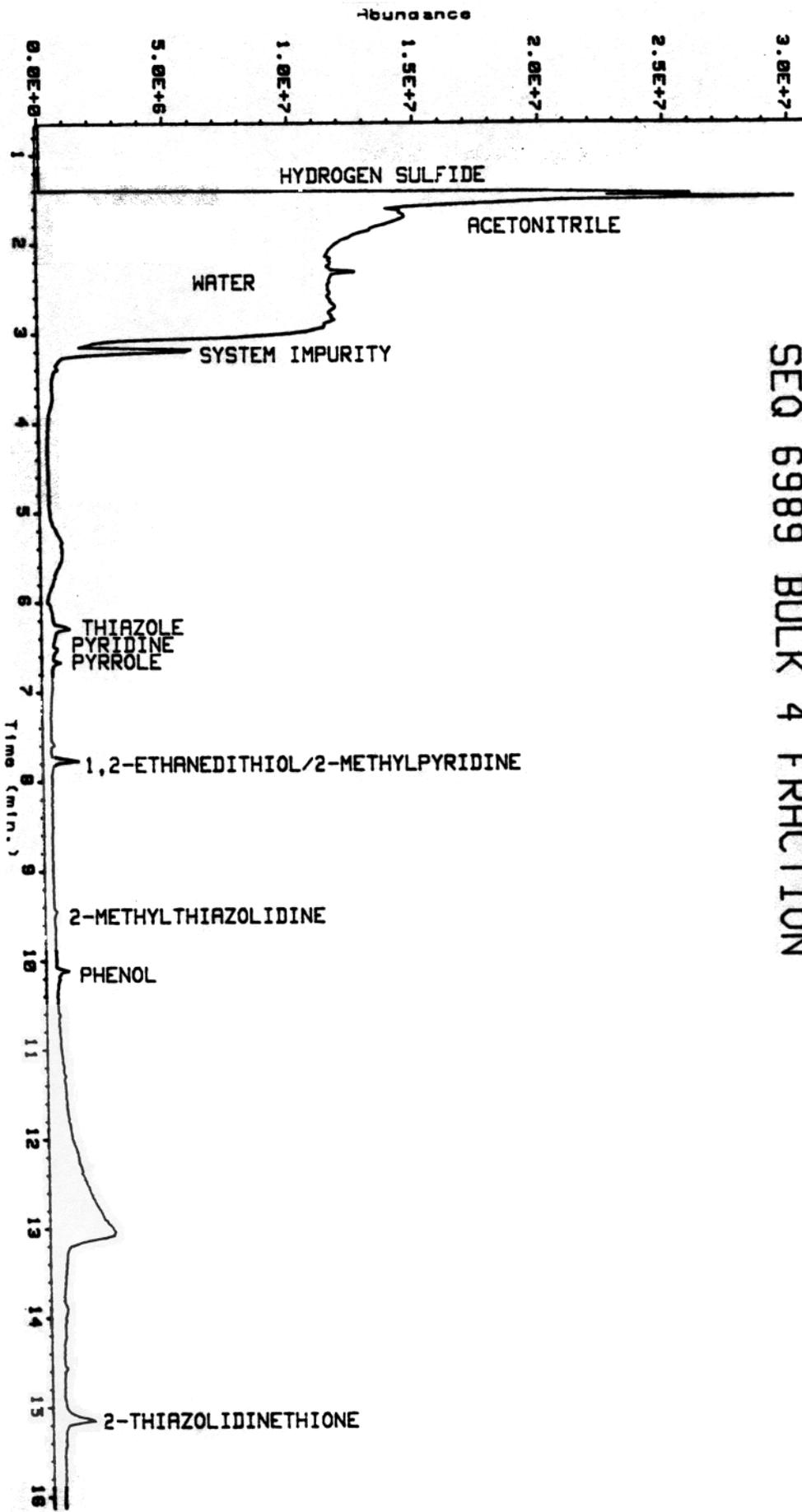


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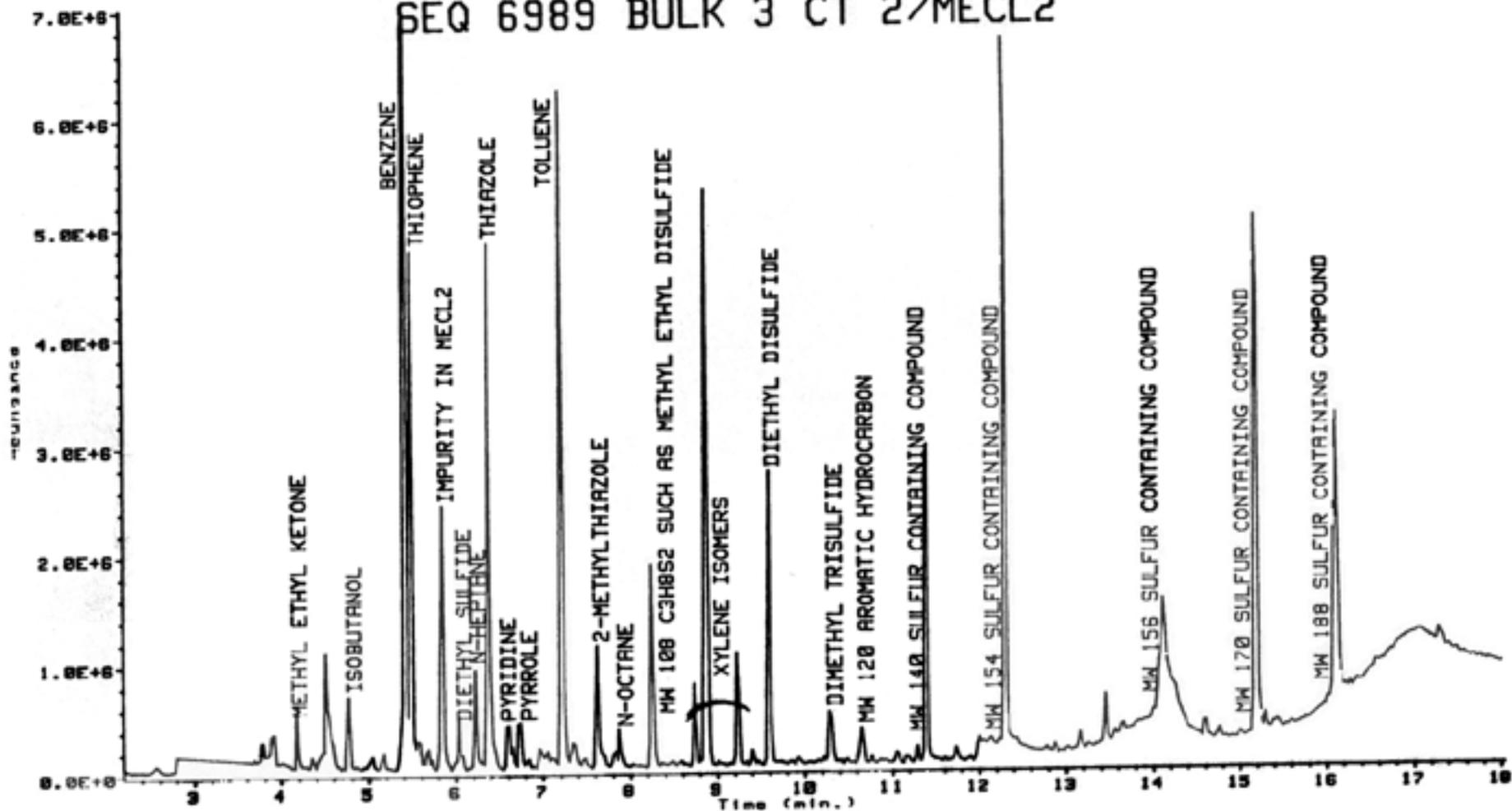


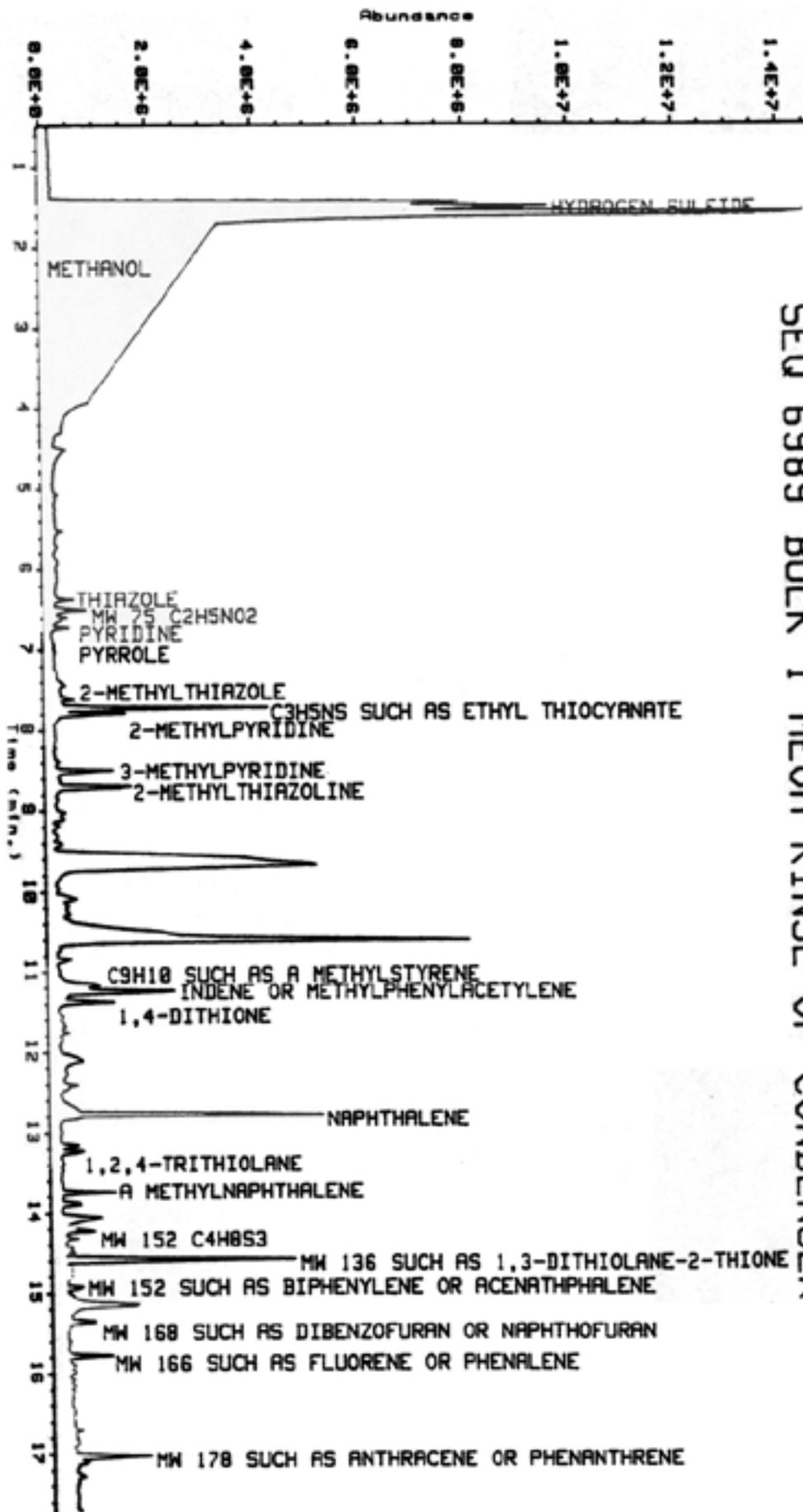
SEQ 6989 BULK 1 CT 2/MECL2

SEQ 6989 BULK 4 FRACTION



SEQ 6989 BULK 3 CT 2/MECL2





SEQ 6989 BULK 1 MEQH RINSE OF CONDENSER