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HETA 89-374-2197
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EXXON BAYTOWN REFINERY
BAYTOWN, TEXAS

NIOSH INVESTIGATORS:
Gregory M. Kinnes, M.S.
Melody M. Kawamoto, M.D., M.S.

I. SUMMARY

On April 19-20, 1990, the National Institute for Occupational Safety and Health (NIOSH) conducted a Health Hazard Evaluation (HHE) at the Exxon Baytown Refinery in Baytown, Texas. The request, from the Gulf Coast Industrial Workers Union (GCIWU), reported that some process technicians experienced "heart attack-type symptoms" during or after unloading cetane improver (2-ethylhexyl nitrate, octyl nitrate) from tanker trucks.

The Exxon Baytown Refinery adds cetane improver to refined diesel fuel to improve its cetane rating (analogous to octane ratings for gasoline). The quantity of cetane improver used depends on the quality of the diesel fuel being processed. The cetane improver is received in tanker trucks which are unloaded into a storage tank before blending with the diesel fuel. Procedures and personal protective equipment used during the unloading of the cetane improver were evaluated during an actual operation. Although there is no current NIOSH analytical method for 2-ethylhexyl nitrate, an attempt was made to collect environmental samples for 2-ethylhexyl nitrate using a variation of Exxon Method 89MR392. Nine personal breathing-zone and general area air samples were collected during the cetane improver unloading operation using Tenax™ solid sorbent tubes. Confidential medical interviews were conducted with 11 process technicians who were randomly selected from 20 employees identified as having unloaded cetane improver tanker trucks.

The environmental samples were initially analyzed using Exxon Method 89MR392. However, since the limit of detection reported in this method could not be reproduced or achieved, the samples were analyzed using an alternate method. A total of nine samples were collected, but only three had detectable levels of 2-ethylhexyl nitrate. The airborne concentrations of 2-ethylhexyl nitrate on these three samples ranged from 0.05 to 0.48 parts per million (ppm). The difficulties encountered during the laboratory analysis and significant discrepancies in the results make the validity of the data questionable. NIOSH, the Occupational Safety and Health Administration (OSHA), and the American Conference of Governmental Industrial Hygienists (ACGIH) have not established any evaluation criteria for 2-ethylhexyl nitrate at this time.

Six of the 11 interviewed employees reported symptoms related to cetane improver exposure. Although symptomatic episodes were rare, they were often temporally related to direct skin exposure. Symptoms included headache, lightheadedness or dizziness, chest discomfort or palpitations, and nausea. Five employees reported no symptoms related to handling the cetane improver. Symptomatic employees reported that use of personal protective equipment and precautionary procedures instituted in September 1989, appeared to be effective; therefore, further NIOSH evaluation of exposures was not warranted.

Although the results of environmental sampling could not be interpreted, and the exposures to Exxon employees are only intermittent and of short duration, some employees reported symptoms consistent with excessive exposure to nitrated esters. In addition, the occurrence of symptomatic episodes has reportedly decreased since the implementation of personal protective equipment procedures. Because of the potential for excessive exposure, and because the health effects related to exposure to nitrated esters can be serious, Exxon should continue to take measures to reduce the potential for exposure to 2-ethylhexyl nitrate.

KEYWORDS: SIC 2911 (Petroleum Refining), cetane improver, 2-ethylhexyl nitrate, octyl nitrate, nitrated esters, heart attack symptoms, refinery.

II. INTRODUCTION

On September 25, 1989, the National Institute for Occupational Safety and Health (NIOSH) received a request from the Gulf Coast Industrial Workers Union (GCIWU) to conduct a Health Hazard Evaluation (HHE) at the Exxon Baytown Refinery in Baytown, Texas. The request reported that some process technicians experienced "heart attack-type symptoms" during or after unloading cetane improver (2-ethylhexyl nitrate, octyl nitrate) from tanker trucks.

NIOSH investigators visited the facility on April 19-20, 1990. The opening conference was attended by both Exxon and GCIWU representatives. The specific issues that prompted the HHE request, as well as Exxon's current and future efforts to control exposure to cetane improver, were discussed. After the conference, a walk-through inspection of the cetane improver unloading area was conducted. Interviews were held with potentially exposed employees and the refinery's medical director, and pertinent records were reviewed. Personal breathing-zone and general area air samples were collected for 2-ethylhexyl nitrate during a tanker unloading operation.

Initial findings were presented to management and union representatives at the conclusion of the site visit. A subsequent status report was also distributed on May 9, 1990.

III. BACKGROUND

The Exxon Baytown Refinery adds cetane improver to refined diesel fuel to improve its cetane rating (analogous to octane ratings for gasoline). The quantity of cetane improver used depends on the quality of the diesel fuel being processed. Therefore, orders are made on an as-needed basis. Cetane improver is delivered to the facility in a 6,000-gallon tanker truck approximately every five days. The cetane improver is unloaded into a 15,000-gallon storage tank using a pneumatic slurp pump and a 3-inch diameter hose. During unloading, the tanker truck is open to the outside atmosphere. The storage tank is always vented to the outside atmosphere. Typically, the unloading operation is performed by an Exxon process technician, with some assistance from the truck driver. Tasks with obvious potential for exposure included collection of product sample from the tanker, connection and disconnection of the hose to the tanker, and spills. The transfer operation lasts approximately two to three hours. The cetane improver is piped from the storage tank and blended with refined diesel fuel as needed.

In November 1988, an employee reported to Exxon that he had experienced medical problems related to unloading cetane improver on at least two occasions. In August 1989, another employee also reported experiencing lightheadedness, headache, and nausea during and after the unloading operation. On October 5, 1989, the plant physician and industrial hygienist issued a written summary of an evaluation conducted to address occupational health issues related to employee exposure to cetane improver. Five out of the 18 employees who unloaded cetane improver reported similar symptoms. Industrial hygiene monitoring on two separate days showed that short-term exposure to 2-ethylhexyl nitrate ranged from 20 to 100 parts per billion (ppb) during unloading tasks. One of the two process technicians monitored had a history of symptoms related to the unloading operation. He reported symptoms during the monitored operation, and the timing of his symptoms corresponded to a transient increase in his pulse rate. Exxon considered these findings to be subtle, but consistent with exposure to 2-ethylhexyl nitrate.[1]

As a result of the Exxon study, unloading procedures were modified and employees with pre-existing medical conditions, which could be exacerbated by exposure, were restricted from working with cetane improver. The use of personal protective equipment (PPE) to prevent vapor inhalation and skin contact was required. This equipment includes a full face respirator fitted with organic vapor cartridges, a neoprene apron, neoprene gloves, and rubber boots. Before the Exxon study, the only PPE used, in addition to that required on refinery premises, were polyvinyl chloride (PVC) gloves and safety glasses. A 2-hour training program was developed for process technicians with a potential for exposure; it reviewed the health hazards of 2-ethylhexyl nitrate and the required work practices. Sampling requirements for cetane improver shipments were re-evaluated and practices modified so that the technician unloading cetane improver no longer had to retrieve a bulk sample from the top hatch of the tanker truck. Exxon also began to review the engineering design of the existing unloading facility to determine what changes could be made to reduce exposure potential. A preliminary plan for a new cetane improver unloading station was already developed at the time of the NIOSH investigation.

IV. EVALUATION PROCEDURES

A. ENVIRONMENTAL

Nine personal breathing-zone (PBZ) and general area air samples were collected for 2-ethylhexyl nitrate during the cetane improver unloading operation. Two samples were collected from the unloading area before the actual operation was initiated. These pre-unloading area samples were used to determine background concentrations. The remaining samples were collected during the unloading process. Four (two at each location) of these were general area air samples collected at locations near the tanker discharge valve or the cetane transfer pump. Two PBZ samples were collected on the process technician. One of these two samples was collected throughout the entire duration of the unloading process. The other was collected only during manual operations, when potential for exposure was greatest, and not during the significant portion of time the worker spent observing the operation from an upwind location. In addition, a hand-carried, intermittent sample was collected by the NIOSH investigator to simulate the one collected from the process technician.

The samples for 2-ethylhexyl nitrate were collected using Tenax™ solid sorbent tubes attached via flexible tubing to personal sampling pumps calibrated at a flow rate of either 0.5 liter of air per minute (lpm) or 50 cubic centimeters of air per minute (cc/min). One background area sample, one PBZ sample worn by the technician, and one of the simulated PBZ samples were collected at the higher flowrate (0.5 lpm) to ensure that a detectable quantity of 2-ethylhexyl nitrate was accumulated. Since NIOSH does not have a specific analytical method for 2-ethylhexyl nitrate, a modification of Exxon Method 89MR392 by gas chromatography (GC) equipped with an electron capture detector (ECD) was initially used to determine 2-ethylhexyl nitrate concentrations in sub-microgram (:g) levels. However, after experiencing a variety of problems associated with the analysis of the samples by GC-ECD (most notably the inability of this procedure to achieve the sensitivities reported in the Exxon method), the samples were analyzed by GC equipped with a flame ionization detector (FID). The FID method had a comparable limit of detection (LOD) to that of the ECD method. Results from the FID method also had the advantage of being determined from more reproducible integration data (chromatographic peak shapes were more consistent) and retention times in the presence of lower background noise levels.

As specified in the Exxon method, the front and back sections of each field sample, and the front section of media blanks, were desorbed separately for 30 minutes in 2 milliliters (ml) of acetonitrile. One milliliter aliquots were then transferred to autosampler vials and analyzed initially by GC-ECD and then by GC-FID using a HP5890 GC containing a DB-1 (0.53 mm ID, 1.5 :m film thickness) fused-silica capillary column.

B. MEDICAL

The NIOSH medical officer conducted confidential interviews with 11 process technicians over three shifts. The interviewed employees were randomly selected from a list of twenty employees identified by the company as having unloaded the cetane improver tankers. The interviews focused on training programs, the use of PPE, mechanisms of exposures, and symptoms. Pertinent medical records and the OSHA 200 logs were reviewed, and an interview was conducted with the refinery's medical director.

V. EVALUATION CRITERIA

As a guide to the evaluation of the hazards posed by workplace exposures, NIOSH field staff employ environmental evaluation criteria for the assessment of a number of chemical and physical agents. These criteria are intended to suggest limits of exposure to which most workers may be exposed up to 10 hours per day, 40 hours per week for a working lifetime without experiencing adverse health effects. It is, however, important to note that not all workers will be protected from adverse health effects even though their exposures are maintained below these limits. A small percentage may experience adverse health effects because of individual susceptibility, a preexisting medical condition, and/or a hypersensitivity (allergy). In addition, some hazardous substances may act in combination with other workplace exposures, the general environment, or with medications or personal habits of the worker to produce health effects even if the occupational exposures are controlled at the limit set by the criterion. These combined effects are often not considered in the evaluation criteria. Also, some substances are absorbed by direct contact with the skin and mucous membranes, and thus potentially increase the overall exposure. Finally, evaluation criteria may change over the years as new information on the toxic effects of an agent become available.

The primary sources of environmental evaluation criteria for the workplace are the following: 1) NIOSH Recommended Exposure Limits (RELs)[(2)], 2) the American Conference of Governmental Industrial Hygienists' (ACGIH) Threshold Limit Values (TLVs)[(3)], and 3) the U.S. Department of Labor, Occupational Safety and Health Administration (OSHA) Permissible Exposure Limits (PELs)[(4)]. The OSHA PELs may be required to take into account the feasibility of controlling exposures in various industries where the agents are used; the NIOSH RELs, by contrast, are based primarily on concerns relating to the prevention of occupational disease. In evaluating the exposure concentrations and the recommendations for reducing these concentrations found in this report, it should be noted that neither NIOSH, ACGIH, nor OSHA currently have evaluation criteria for 2-ethylhexyl nitrate. However, most manufacturers or users of chemicals employ the same scientific reasoning, as discussed earlier, to establish their own internal criteria.

A time-weighted average (TWA) exposure refers to the average airborne concentration of a substance during a normal 8- to 10-hour workday. Some substances have recommended short-term exposure limits (STELs) or ceiling values which are intended to supplement the TWA where there are recognized toxic effects from high, short-term exposures.

A. 2-ETHYLHEXYL NITRATE

2-Ethylhexyl nitrate is a nitrated ester and is chemically related to nitroglycerin (NG) and ethylene glycol dinitrate (EGDN). Nitrated esters are well known for their explosive properties and are used in the manufacture of munitions or as rocket or small arms propellants. In medicine, NG is best known for its use in the treatment of angina (chest pain related to coronary artery disease or spasm). Inhalation and skin contact are the major routes of occupational exposure to nitrated esters. Acute health effects of exposure to NG and EGDN include headache, dizziness, nausea, increase in heart rate, decrease in systolic blood pressure, and change in diastolic blood pressure.[(5)] Exposed workers develop tolerance to the vasodilatory effects.[(5)] Some workers develop nitrate withdrawal symptoms, such as angina, after exposure has ended.[(6)] Occupational exposures to NG and EGDN have also been associated with sudden death and an increased risk of dying from cardiovascular or cerebrovascular disease.[(7)]

Chemical factory workers have been reported to develop throbbing headaches during the dilution of 2-ethylhexyl nitrate with simple aliphatic alcohols in the production of diesel oil additive. 2-Ethylhexyl nitrate in workroom air was measured in the range of 5 to 20 parts per million (ppm), but skin absorption could not be excluded. The clinical picture was noted to closely resemble the headaches experienced by dynamite workers, except that the chemical workers exposed to 2-ethylhexyl nitrate did not develop tolerance. This was attributed to the short exposure periods.[8]

NIOSH, OSHA, and the ACGIH have not established any evaluation criteria for 2-ethylhexyl nitrate at this time. However, the material safety data sheet provided to Exxon by Dupont lists an internal Dupont exposure limit of 5 ppm as an 8-hour TWA. Exxon's Occupational Exposure Limit Committee does not agree with Dupont's exposure limit as a TWA and is currently developing a STEL recommendation for 2-ethylhexyl nitrate. Exxon's best estimate of short term exposure levels which may cause symptoms in susceptible individuals is in the 50-100 ppb range, which is lower the Dupont TWA of 5 ppm.

VI. RESULTS AND DISCUSSION

A. ENVIRONMENTAL

The duration of the unloading operation sampled during this investigation was approximately two hours. The process technician who performed the operation wore the PPE currently required by Exxon. This equipment included a full face respirator fitted with organic vapor cartridges, a neoprene apron, neoprene gloves, and rubber boots. The technician was assisted by the tanker truck driver, who was also required to wear the same PPE. The technician and the truck driver attached the hose from the cetane improver transfer pump to a hose from the tanker truck. This hose was then attached to the discharge valve of the tanker truck, and a plastic barrel was placed under the tanker discharge valve to contain any spills during the transfer. Both individuals climbed atop the tanker to open (for venting) a hatch to allow free-flow of the cetane improver. Once the actual unloading was initiated, the workers spent a significant portion of time observing the operation from an upwind location. During this period, the workers removed their gloves and respirators. These individuals would make only periodic checks of the operation until the hoses were ready to be disconnected. The total portion of time performing the connection, periodic checks, and disconnection was approximately 17 minutes.

Quantitative analysis of the samples by GC-ECD (Exxon Method) yielded indeterminant results, including a LOD value of 5-10 micrograms per sample (:g/sample). The measured LOD was approximately 20 times less sensitive than the ECD sensitivity reported in the Exxon method. Because of the discrepency, the samples were then analyzed by GC-FID since no interfering hydrocarbons were detected. The LOD was comparable to that obtained by the GC-ECD analysis, and the latter method was found to have more reproducible and consistent peak areas. The results of the analysis by GC-FID for 2-ethylhexyl nitrate are reported in Table I. These results are reported as airborne concentrations during the sampling intervals and have not been corrected to reflect 8-hour TWAs.

A total of nine samples were collected, but only three had detectable levels of 2-ethylhexyl nitrate. The remaining six samples had levels below the LOD of 5 :g/sample. The airborne concentrations of 2-ethylhexyl nitrate on the three samples with detectable quantities ranged from 0.052 to 0.475 ppm. Two of the detectable samples were collected from the same area downwind of the unloading area before the actual unloading operation was initiated. One sample was collected at a flow rate of 0.5 lpm and one at 50 cc/min. Even though these samples were collected from the same location, the airborne concentrations of 2-ethylhexyl nitrate varied significantly. The low volume sample had a concentration of 0.475 ppm, while the concentration for the high volume sample was only 0.052 ppm. This variation may be due to decreased collection efficiency at the higher flow rate. The third detectable sample was collected from an area 3 feet from the tanker discharge valve (downwind) during the pumping operation. This sample had an airborne concentration of 0.229 ppm, even though the other sample collected from this area did not have a detectable concentration of 2-ethylhexyl nitrate. The background samples collected before unloading had greater concentrations than the samples collected during the actual operation. Two of the three detectable airborne concentrations were also higher than the concentrations reported by Exxon during their evaluations of the operation.

Due to the difficulties encountered during the laboratory analysis and the significant discrepancies in the results, the validity of the sampling results is questionable. There are several possible causes that could have lead to these difficulties and discrepancies. The Tenax™ sorbent tubes prescribed by Exxon Method 89MR392 are specially prepared, while commercially available Tenax™ sorbent tubes were used during this investigation. This could have affected the results do to the possibility of different collection efficiencies, interferences, and/or solvent-analyte interactions. The LOD reported in the Exxon method could not be reproduced by the NIOSH laboratory. Therefore, the method was modified during the attempt to achieve reproducible results for this analysis. This modified method for analysis used GC-FID instead of GC-ECD as used in the Exxon method. Due to sample stability constraints, insufficient time was allowed for preparational and conformational testing of the Exxon method or the modified NIOSH version. Another possible reason for the differences could have been the use of acetonitrile as a solvent to desorb the samples. Acetonitrile is generally not considered a suitable solvent for use in gas chromatography because it is not very volatile, has a broad peak, and can produce carryover effects. Since the samples had already been desorbed in acetonitrile as dictated by the Exxon method, experimentation with an alternate solvent could not be conducted. Also, only small amounts of 2-ethylhexyl nitrate were confirmed in the high volume background sample (0.052 ppm) using GC equipped with a mass selective detector.

B. MEDICAL

The confidential employee interviews revealed that potential sources of exposure to cetane improver included leaks in hoses, pumps, and valves; the connection and disconnection of the hose to and from tankers; the sampling of contents from top of the tanker; injection of cetane improver from the storage tank into the diesel lines; evaporation from the slab, the storage tank vent, the tanker vent, and ground spills; and improper handling of contaminated equipment (e.g., worn hoses, PPE). Employees reported exposure potential to be worse during hot and humid weather or when the technician stands downwind from the atmospheric drum or tanker.

Work practices for disconnecting hoses were reportedly variable, resulting in differences in exposure potential. Two employees reported closing the tanker valve before turning the pump off, allowing the pump to drain the hose before disconnecting it from the tanker and, thereby, minimizing spillage. Employees who did not use this procedure reported that they placed buckets under the tanker to contain spills.

Six of the 11 interviewed employees reported symptoms related to cetane improver exposure. Although symptomatic episodes were rare, they were often temporally related to direct skin exposure. Episodes of headaches were reported by five, lightheadedness or dizziness by three, chest discomfort (chest pain and heartburn) by two, and nausea by two. Three reported episodes of heart palpitations, but two could not directly relate symptoms to work exposures. The remaining five employees reported no symptoms related to handling cetane improver.

Symptomatic employees reported that use of PPE and precautionary procedures instituted in

September 1989, appeared to decrease the number of symptomatic episodes. Some of the asymptomatic employees reported that they do not use the respirators and aprons as required.

The symptoms reported by Exxon process technicians (i.e., headaches, dizziness, nausea, palpitations, chest pain) are consistent with exposure to nitrated esters (in this case, 2-ethylhexyl nitrate). Therefore, it is possible that some of the reported symptoms were related to exposure to 2-cetane improver, especially during nonroutine exposures (e.g., skin contact, spills). This possibility is also supported by the reported temporal relationship of symptoms to work with cetane improver and by the reported decrease in symptoms with the use of PPE and changes in work practices. However, general symptoms such as headaches, dizziness, and nausea commonly develop from a variety of causes. Chest discomfort and palpitations may be related to underlying heart conditions, and to other causes as well. The medical evaluations that would be necessary to determine the cause of chest symptoms in each symptomatic Exxon employee is beyond the scope of the NIOSH investigation.

VII. CONCLUSIONS

Although the results of environmental sampling could not be confidently interpreted, and the exposures to Exxon employees are only intermittent and of short duration (unlike the long-term, daily exposures to nitroglycerin and ethylene glycol dinitrate by workers in studies reported in the medical literature), some employees reported symptoms consistent with excessive exposure to nitrated esters. In addition, the occurrence of symptomatic episodes has reportedly decreased since the implementation of personal protective procedures.

VIII. RECOMMENDATIONS

1. All employees, including asymptomatic employees, performing the cetane improver unloading operation should wear the PPE required by Exxon, especially when performing any of the manual tasks (handling hoses, opening the tanker hatch, working with the valves or pumps, etc.).

2. Before beginning the cetane improver unloading operation, employees should ensure that the hoses, pumps, and valves are not worn or leaking. Any equipment that is not in satisfactory condition should be replaced or repaired before unloading the tanker. Personal protective equipment should be used at all times during the performance of these maintenance tasks.

3. When not performing required manual tasks, employees performing the cetane improver unloading operation should not remove their respirators and gloves unless they remain at a location which is a significant distance (approximately 50 yards) upwind from the unloading area.

4. After completion of the unloading operation, the tanker discharge valve should be closed before turning off the transfer pump. Employees reported that this allows the pump to drain the hose before it is disconnected from the tanker discharge valve, thereby, minimizing spillage. In the event of any spillage, precautions, such as the use of the plastic containers to contain spills, should be used. Any recovered cetane improver should be properly disposed at the waste treatment facility.

5. In the event that PPE becomes contaminated, it should be discarded in a covered bin located near the unloading station until it can be properly disposed.

6. Due to the difficulties encountered during the laboratory analysis, further validation studies should be conducted on Exxon Method 89MR392 to determine its reliability.

7. Because of the potential for exposure, and because the health effects related to 2-ethylhexyl nitrate can be serious, Exxon should continue to take measures (such as the construction of the specially designed unloading station) to reduce the potential for employee exposures. In addition, the potential for exposure during blending of cetane improver and diesel fuel should be evaluated and controlled, if necessary.

8. Reports of symptoms potentially related to exposure to nitrated esters (e.g., headaches, dizziness, nausea, chest discomfort, and palpitations) should be actively investigated.

IX. REFERENCES

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X. AUTHORSHIP AND ACKNOWLEDGEMENTS

Report Prepared by:	Gregory M. Kinnes, M.S. Industrial Hygienist Industrial Hygiene Section
	Melody M. Kawamoto, M.D., M.S. Medical Officer Medical Section
Originating Office:	Hazard Evaluations and Technical Assistance Branch Division of Surveillance, Hazard Evaluations and Field Studies
Analytical Support:	Stephanie M. Pendergrass Measurements Development Section Measurements Research and Support Branch Division of Physical Sciences and Engineering Cincinnati, Ohio 45226

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Copies of this report have been sent to:

1. Exxon Company, U.S.A.
2. Gulf Coast Industrial Workers Union
3. OSHA, Region VI
4. NIOSH, Atlanta

For the purpose of informing affected employees, copies of this report shall be posted by the employer in a prominent place accessible to the employees for a period of 30 calendar days.

Table I

Personal and Area Air Samples for 2-Ethylhexyl Nitrate

Exxon Baytown Refinery
Baytown, Texas
HETA 89-374

April 19, 1990

Type	Sample Description	Duration (minutes)	Volume (liters)	Concentration (ppm)
GA-1	Background, downwind Area (High Volume)	99	48	0.052
GA-1	Background, downwind Area (Low Volume)	99	5.0	0.475
PBZ	Process Technician (Entire Operation)	101	5.0	ND
PBZ	Process Technician (Manual Tasks Only)	17	8.5	ND
Sim*	Carried Sample	17	8.5	ND
GA-2	Tanker Discharge Valve	110	5.5	0.229
GA-2	Tanker Discharge Valve	110	5.5	ND
GA-3	Transfer Pump	108	5.4	ND
GA-3	Transfer Pump	108	5.4	ND

NIOSH, OSHA, nor ACGIH have established exposure criteria for 2-ethylhexyl nitrate; however, Exxon has used an internal 8-hr TWA criteria of 5 ppm established by the supplier (Dupont) and is proposing a short-term exposure limit which is lower than the 8-hr TWA.

GA-# - General Area Sample, same numbers denote paired samples.
PBZ - Personal Breathing-Zone Sample.
ND - Non-Dectected.
Sim* - Simulated sample that was carried near Process Technician during manual tasks.

NOTE - The results presented in this table are suspect due to difficulties
›oratory analysis.