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# Analytical Methods Used in a Study of Coke Oven Effluent

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**In a coke oven study conducted by NIOSH, selected chemical analyses of airborne particulates, vapors, and metals in the emissions from five coke ovens were done. Eight sampling procedures and seven analytical techniques were used to analyze samples collected for the study. Six of the analytical methods used are discussed.**

## Introduction

THE REDUCTION OF IRON ORE to iron in the production of steel requires the use of a relatively pure form of carbon. The most popular source of carbon for steel making in the United States is coke, which is produced by baking coal in ovens at temperatures near 2800°F for 15 to 20 hours. During this baking process, volatile matter is emitted from the ovens. Most of the emissions are collected by an exhaust ventilation manifold, but a considerable amount of the volatiles leaks into the atmosphere during oven charging (filling) and pushing (emptying) and through faulty door and charging port seals. This leakage exposes coke battery workers to an atmosphere containing a variety of compounds including many polynuclear aromatic hydrocarbons (PNA's), some of which have been identified in animal studies as carcinogens.

Because of concern for the possible effects of the PNA emissions on the health of the coke oven workers, a statistical investigation of the mortality of coke oven employees was conducted by Lloyd.<sup>1</sup> The investigators found that coke oven workers have from 2.5 to 5 times greater incidence of lung cancer than the general population. Since as-

Mention of commercial concerns or products does not constitute endorsement by the National Institute for Occupational Safety and Health.

phalt workers, roofers, and other groups handling coal tars are exposed to similar PNA emissions, the number of workers affected is quite large.

In order to assess the extent of exposure to polynuclear aromatic hydrocarbons and to evaluate sampling and analytical methods for coke oven emissions, a study was jointly undertaken by the National Institute for Occupational Safety and Health (NIOSH) and the American Iron and Steel Institute (AISI). NIOSH designed and procured the sampling equipment used in the study and collected and analyzed the samples. The AISI supplied technical information from their experience with sampling of this nature and recruited five steel plants, representing the major coal mining areas of the United States, for participation in the program.

## Experimental

The sampling program was divided into two parts, which were designated Round I and Round II. Each round consisted of one week of sampling at each of the five plants. Round I was completed in June 1971 and Round II, in January 1972. Altogether, 1,440 samples were collected by eight different sampling methods and analyzed by seven analytical methods.

Two types of samples were used: area samplers, operated at a given station in the

plant; and personal samplers, worn by the coke oven employees.

The area samplers consisted of a package of seven different samplers. These were:

(1) A Dorr-Oliver 10-mm cyclone operated at 1.7 liters per minute (1pm), exhausting into a filter held by a 37-mm Millipore Field Monitor Cassette—chosen for a respirable sample with a widely used device.

(2) A British Cast Iron Research Association (BCIRA) aluminum alloy cyclone operating at 2.0 1pm, exhausting into a filter held by a 37-mm Millipore Field Monitor Cassette—chosen for a respirable sample to compare with the Dorr-Oliver cyclone.

(3) A 5-plate horizontal elutriator operating at 5.0 1pm, exhausting into a filter held by a 37-mm Millipore Field Monitor Cassette—chosen because it is a fairly well established method for sampling respirable particulates.

(4) A filter held in a 37-mm Millipore Field Monitor Cassette operated with the face cap removed at 5.0 1pm with inlet facing down—chosen to collect a gross sample at a flow rate near the rate for peak efficiency of the filter.

(5) Same as (4), but with a different filter type for comparison.

(6) A charcoal tube sampled at 0.5 1pm to sample for organic solvents.

(7) A general Metal Works high-volume air sampler with an 8" × 10" glass filter without an organic binder operated at 20 cubic feet per minute (cfm)—chosen to have a sample large enough to analyze by all the chemical methods used.

The first six of the above samplers were attached to a small carrier. The carrier samplers (1 through 6) and the high-volume unit (7) were operated in close proximity to collect simultaneous samples in approximately the same area of contamination.

The second type of sampler, the personal sampler, was used to collect samples in the breathing zone of the workers. A personal unit consisted of a Mine Safety Appliance, Model G, battery operated vacuum pump

with pulsation dampener operated at 1.7 1pm with a Dorr-Oliver 10-mm nylon cyclone which exhausted into a filter held by a 37-mm Millipore Field Monitor Cassette.

Samplers 1 through 4 and the personal samplers used a glass fiber filter without organic binders, followed by a silver membrane filter and backed with a cellulose pad. A single glass fiber filter without organic binders, with cellulose backup pad was used for sampler 5.

The analytical methods used were generally modifications of published methods. A brief description of each of the methods follows.

#### *Method 1. Benzene Soluble Extract<sup>2,3</sup>*

This method involves an extraction of the filter with hot benzene using a Soxhlet extractor and a determination of the weight of the benzene soluble material by the difference between the filter weights before and after extraction.

#### *Method 2. Gas Chromatography for Polynuclear Aromatic Hydrocarbons<sup>4,5</sup>*

This method requires the gas chromatographic separation of fluoranthene, pyrene, benz(c)acridene, chrysene, benz(a)anthracene, benz(a)anthrone, benzo(b)fluoranthene, benzo(j)fluoranthene, benzo(k)fluoranthene, benzo(e)pyrene, benzo(e)pyrene, benzo(a)pyrene, dibenz(a,h)anthracene, benzo(g,h,i)perylene, and anthanthrene using a 3% Dexsil 300 GC column. Identification and quantitation were done using either a UV-Vis spectrophotometer or a spectrofluorometer. The sample was transferred from the gas chromatograph (GC) to the spectrophotometer by means of a trapping assembly.

#### *Method 3. Thin Layer Chromatography for Benzo(a)pyrene<sup>6-8</sup>*

Benzo(a)pyrene (BaP) is separated from other aromatic hydrocarbons using thin-layer chromatography. The developing solvent is a pentane-ether mixture. The BaP is ex-

tracted from the plate with diethyl ether and ultimately dissolved in H<sub>2</sub>SO<sub>4</sub>. BaP content is measured fluorometrically at 470-nm excitation and 540-nm emission wavelengths.

*Method 4. Liquid Column Chromatography for Polynuclear Aromatic Compounds<sup>9,10,11</sup>*

Pyrene, fluoranthene, benz(a)anthracene, chrysene, benzo(e)pyrene, perylene, benzo(k)fluoranthene and anthanthrene are separated using an alumina-packed column and n-pentane-ether mixtures as elution solvents. The fractions collected are scanned by absorption spectroscopy and specific fractions are examined by fluorometry in order to quantitate the amounts present.

*Method 5. Emission Index<sup>12</sup>*

The sampling filters are extracted with cyclohexane, partitioned with a dimethylsulfoxide-phosphoric acid mixture, and then partitioned with iso-octane. The iso-octane extract is used to obtain the UV-Vis spectrum from 200-600 nm, the fluorometric

emission spectra from 300-500 nm with excitation at 350, 400, and 450 nm, and the fluorometric excitation spectra from 300-500 nm with emission at 350, 400, and 450 nm.

*Method 6. Trace Metals by Atomic Absorption<sup>13</sup>*

The sampling filters are wet-ashed with ultra-pure acids and the resulting ash is taken into solution and diluted to 25 ml. The solution is analyzed using the atomic absorption spectrophotometer for cobalt, chromium, copper, manganese, nickel, and zinc.

*Method 7. Gas Chromatography for Organic Solvents<sup>14,15</sup>*

Organic solvents in the atmosphere are collected on activated charcoal and then desorbed from the charcoal by elution with carbon disulfide. An aliquot of the CS<sub>2</sub>-solution is injected into a gas chromatograph and the recorded areas under the benzene,

TABLE I  
Sampling and Analytical Schema

Sampling Procedure	Method of Analysis*	Number of Samples
1. BCIRA cyclone	1	60
	5	60
2. Dorr Oliver cyclone	1	60
	5	60
3. Gross glass fiber filter	1	60
	5	60
4. Gross silver filter	1 & 2	20
	1 & 3	20
	1 & 4	20
	2 & 5	20
	3 & 5	20
	4 & 5	20
5. Horizontal Elutriator	1 & 2	20
	1 & 3	20
	1 & 4	20
	2 & 5	20
	3 & 5	20
	4 & 5	20
6. Charcoal tube	7	120
7. Hi Vol glass fiber filter	1,2,3,4,5, & 6	120
8. Personal monitor	1 & 3	300
	2	300
		1,440

\*Number refers to methods listed previously.

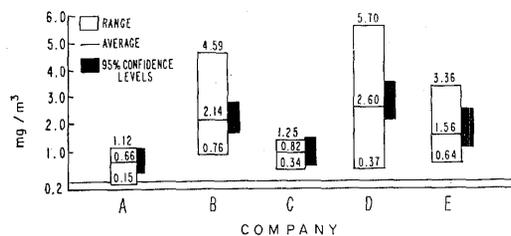


Figure 1. Summary of concentrations of benzene soluble fraction ( $\text{mg}/\text{m}^3$ ) collected by hi-vol samplers in Round I.

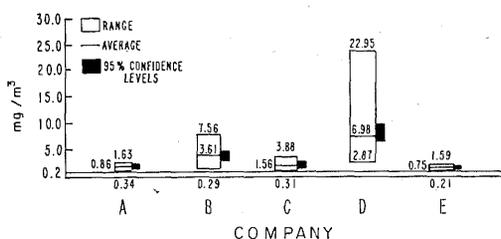


Figure 2. Summary of concentrations of benzene soluble fraction ( $\text{mg}/\text{m}^3$ ) collected by hi-vol samplers in Round II.

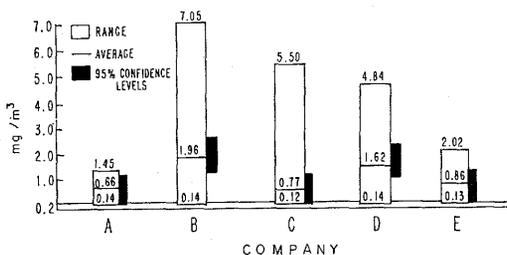


Figure 3. Summary of concentrations of benzene soluble fraction ( $\text{mg}/\text{m}^3$ ) collected by personal samplers in Round I.

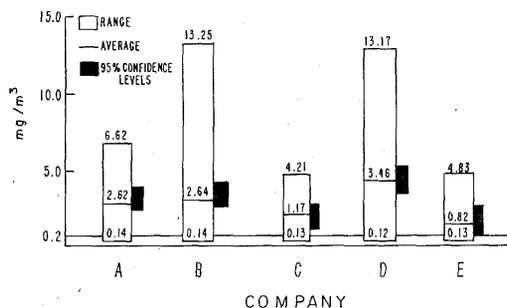


Figure 4. Summary of concentrations of benzene soluble fraction ( $\text{mg}/\text{m}^3$ ) collected by personal samplers in Round II.

toluene, xylene, and naphthalene peaks are integrated. An 8% SE-30 column was used to attain separation.

The division of the samples among the various sampling and analytical techniques is shown in Table I. This report will deal only with the evaluation of analytical methods.

## Results

After the analytical results had been received and the data had been reduced, the methods were compared and a statistical evaluation of the data begun.

The first comparative studies were done on the results from the benzene soluble analyses. Total benzene soluble concentrations were determined on both high-volume and personal samples. Twelve high-volume filters were sampled on the catwalks at each of the five batteries during each round of sampling. Figure 1 shows the range, average, and 95% confidence intervals for the average of the benzene soluble data collected from Round I samples. The 95% confidence intervals indicate that a 95% probability exists that the true average concentration for each company lies within the given intervals. The intervals are not symmetric about the average because the data are log-normally distributed. Figure 2 illustrates the results of the benzene soluble analysis of Round II samples. In both Round I and Round II, a large portion of the samples lie above the threshold limit value of  $0.2 \text{ mg}/\text{m}^3$  benzene soluble material. The average benzene soluble concentrations and the 95% confidence intervals of Round II samples were greater than those of Round I.

Figures 3 and 4 illustrate the range, average, and 95% intervals for the personal samples collected during Rounds I and II. As with the high-volume samples, most of the personal samples lie above  $0.2 \text{ mg}/\text{m}^3$  of benzene soluble material.

The average benzene soluble concentration of the personal samples collected at

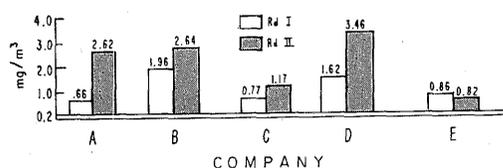


Figure 5. Comparison for Round I vs. Round II of concentrations of benzene soluble fraction collected by personal samplers.

each of the five plants during Rounds I and II is compared in Figure 5. The average benzene soluble concentration for the second round of samples is greater than that for the first round for each of the five companies, with the exception of Company E. This is also true for the samples collected by the high-volume filters. Several factors may have contributed to this difference: (a) samples collected in Round I were stored for several months in a refrigerator prior to analysis, while the Round II samples were analyzed a few weeks after collection, and (b) Round I was sampled in the spring, while Round II was sampled in the winter, hence operating conditions at the ovens may have changed between sampling rounds.

The Occupational Safety and Health Standard for exposure to coke oven emissions is based on the concentration of benzene soluble material in the air (0.2 mg/m<sup>3</sup> ben-

zene soluble material).<sup>16</sup> This is a non-specific standard which does not distinguish between carcinogenic and non-carcinogenic organics. In order to have a more complete characterization of coke oven emissions, we measured the concentration of several polynuclear aromatic hydrocarbons. Three methods were used to determine the PNA content of the coke oven atmosphere: the gas chromatographic-ultraviolet spectrophotometric analysis for 14 PNA's (Method 2), the thin-layer chromatographic method for BaP (Method 3), and the liquid chromatographic analysis for 9 PNA's (Method 4). The results of the benzene soluble analysis were correlated with the PNA content as determined by both the TLC method (BaP only) and the GC-UV method (total of 14 PNA's). The benzene soluble concentration was highly correlated with the BaP concentration as measured by TLC when two of the sampling methods, the high volume sampler and the horizontal elutriator were used. However, with all other sampling devices, the correlation coefficient was quite low. No significant correlation was found between the benzene soluble material and the sum of the concentrations of the 14 PNA's as determined by GC-UV. The correlation coefficients are given in Table II.

TABLE II  
Correlation Coefficients: Benzene Soluble versus: GC-UV and TLC Data

Methods compared	Sampler used	Number of samples		Correlation coefficient		Student's <i>t</i> -Test for <i>r</i> =0 Probability	
		Rd-I	Rd-II	Rd-I	Rd-II	Rd-I	Rd-II
B.S. vs. GC-UV	Hi-Vol	60	60	.581	.784	.0000	.0000
B.S. vs. GC-UV	Horizontal Elutriator	10	8	.726	.359*	.0175	.3832
B.S. vs. GC-UV	Gross	10	8	.383*	.791	.2746	.0193
B.S. vs. TLC	Hi-Vol	60	60	.713	.970	.0000	.0000
B.S. vs. TLC	Personal	144	148	.564	.635	.0000	.0000
B.S. vs. TLC	Horizontal Elutriator	10	9	.934	.884	.0001	.0016
B.S. vs. TLC	Gross	10	9	.818	.489*	.0038	.1815

\*No significant difference from zero.

The data gathered on benzo(a)pyrene concentration levels were also examined. The BaP levels were measured using three different analytical methods and several sampling techniques. All three of the analytical methods, GC-UV (Method 2), thin-layer chromatography (Method 3), and liquid chromatography (Method 4) were used to determine the BaP levels on each of the high-volume filters. Figure 6 is a comparison of the average BaP concentrations of 12 high-volume filters from each company as determined by each of the three analytical methods. These data are taken from Round I samples. The same comparisons were made on Round II data and are expressed in Figure 7. Examination of Figure 6 shows that the

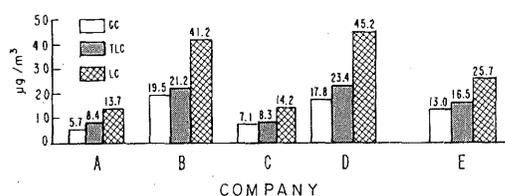


Figure 6. Comparison of concentrations of BaP found in Round I as determined by three analytical methods.

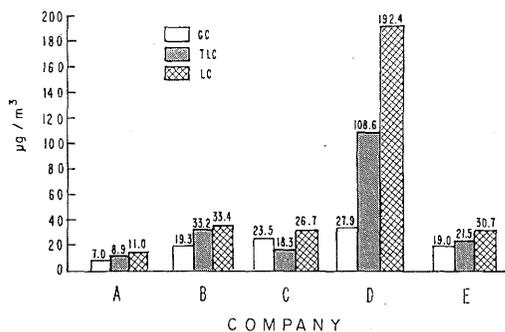


Figure 7. Comparison of concentrations of BaP found in Round II as determined by three analytical methods.

BaP concentrations detected by the GC-UV and TLC methods are fairly close, whereas the liquid chromatographic method yielded values twice as high as the other two methods. The analyses of Round II samples resulted in average BaP levels that were in fairly good agreement. Company D, Round II, is a notable exception.

Correlation coefficients for the methods were calculated and are given in Table III. A good correlation exists between the BaP levels found using the thin-layer chromatographic and liquid chromatographic methods, while the correlation is poor between the TLC and GC-UV methods. A high correlation coefficient indicates that there exists a strong linear relationship between the amounts of BaP found by the two methods. It does not indicate that the same amount of BaP was detected by both methods.

A gas chromatographic-ultraviolet spectrophotometric analysis for 14 PNA's (Method 2) was performed on four types of samples: high-volume filters, personal samples, horizontal elutriators, and gross samples. The average total amount of the 14 PNA's found in each of the sampling types is given in Table IV. Since the high-volume and gross samples monitor the same atmosphere and collect both respirable and non-respirable particulates, the difference in the total

TABLE IV  
Mean Concentration of 14 PNA's

Sampler used	Average total concentration of 14 PNA's ( $\mu\text{g}/\text{m}^3$ )	
	Rd-I	Rd-II
Hi-Vol	138	188
Personal	35.4	50.6
Horizontal Elutriator	106	119
Gross	91.2	116

TABLE III  
Comparison of Methods of Analysis for Benzo[a]pyrene

Methods compared	Number of samples		Correlation coefficient		Student's <i>t</i> -Test for $r=0$ Probability	
	Rd-I	Rd-II	Rd-I	Rd-II	Rd-I	Rd-II
TLC vs. GC-UV	60	60	.536	.558	.0000	.0000
GC-UV vs. LC	60	60	.582	.479	.0000	.0001
TLC vs. LC	60	60	.776	.973	.0000	.0000

amount of PNA's detected using these two sampling devices could reflect a basic difference in the efficiency of the sampling devices and/or the error of the analytical method.

There were definite trends in the detection of the PNA's. In the high-volume, horizontal elutriator, and gross samples, six of the 14 PNA's were most abundant: chrysene, fluoranthene, pyrene, benz(a)anthracene, benzo(k)fluoranthene, and benzo(a)pyrene. Three compounds were present in fairly low concentrations: anthanthrene, benz(c)acridine, and dibenz(a,h)anthracene. The personal samples contained only two major components, chrysene and benz(a)anthracene, and only minute or non-detectable quantities of the other fourteen. Benz(a)anthrone was not found in any of the high-volume or personal samples, but several compounds besides the fourteen were found. One of these additional peaks was identified as phenanthrene and two others were found to have molecular weights of 230 and 246.

A subset of the 14 PNA's was correlated with the entire set in order to develop a less time-consuming method of analysis. The subset consisted of those PNA's which were carcinogenic,<sup>17</sup> benz(a)anthracene, benzo(b)-fluoranthene, benzo(j)fluoranthene, and benzo(a)pyrene, plus the isomers of these compounds which were included in the original fourteen, benzo(e)pyrene and benzo(k)fluoranthene. The subset, consisting of six compounds, was called the carcinogenic subset.

Correlation coefficients for the total concentration of PNA's in the carcinogenic subset and the 14 PNA total were calculated.

These coefficients, given in Table V, were found to be generally greater than 0.9 for all four sampling methods in both Round I and Round II. The high correlation coefficients indicate that the six compounds in the subset will indicate exposure to the total 14 compounds.

The data collected on the high-volume filters is fairly consistent. The weight of the benzene soluble extract is generally 20-40% of the weight of the entire sample. The 14 PNA's comprised about 5% of the benzene soluble extract, while the six compounds in the carcinogenic subset were 2%, and the BaP was 1%, of the extract.

The trace metal distribution of the high-volume filters was investigated and found to be fairly uniform from one sample to another. Over half of the trace metal concentration could be attributed to zinc, while intermediate concentrations of copper and manganese, and only small amounts of chromium and nickel were detected. Correlation coefficients were calculated for the sum of the trace metal concentrations and the benzene soluble concentration, the sum of the 14 PNA's as determined by GC, the BaP concentration determined by TLC, and the sum of the 9 PNA's analyzed by liquid chromatography. These correlation coefficients, presented in Table VI, are low, indicating that there is a very weak linear relationship between the amount of trace metals present and the benzene soluble or PNA levels.

The charcoal tubes, which were sampled on the catwalks above the ovens, were analyzed for benzene, toluene, xylene, and nap-

TABLE V  
Comparison of the Total Concentration of 14 PNA's with the Total Concentration in the Carcinogenic Subset

Sampler used	Number of samples		Correlation coefficient		Student's <i>t</i> -Test for $r=0$ Probability	
	Rd-I	Rd-II	Rd-I	Rd-II	Rd-I	Rd-II
Hi-Vol	60	60	.956	.939	.0000	.0000
Personal	122	120	.931	.959	.0000	.0000
Horizontal Elutriator	20	18	.993	.869	.0000	.0000
Gross	20	18	.988	.944	.0000	.0000

TABLE VI  
Comparison of Trace Metal Concentration and Concentration of  
Organic Material

Trace metal conc. vs.	Correlation coefficient		Student's <i>t</i> -Test for $r=0$ Probability	
	Rd-I	Rd-II	Rd-I	Rd-II
B.S. conc.	.146*	.270	.2794	.0382
14 PNA's (by GC-UV) conc.	.112*	.284	.4070	.0292
BaP (by TLC) conc.	.021*	.208*	.8745	.1137
BaP (by LC) conc.	.134*	.192*	.3222	.1456

\*No significant difference from zero.

thalene. Although these compounds are cited in the literature as being present in fairly large amounts in coking operations, only microgram quantities were found on the tubes. We suspected that the charcoal tube was not a good collection device at the extreme temperatures and humidities found on a coke oven. However, charcoal tubes which were sampled at a later date to check the trapping ability of the charcoal contained appreciable quantities of all four organic solvents. Therefore, it is most likely that either benzene, toluene, xylene, and naphthalene were not present in any of the coking operations which were visited, or that some sampling error occurred and these compounds were not trapped.

A select group of the emission index spectra were studied to determine if any correlation existed between the spectra and the concentrations of one or more of the PNA's in the benzene soluble fraction.<sup>18</sup> No correlations were found.

### Discussion

The benzene soluble fraction of coal tar pitch volatiles, upon which the present standard is based, does not correlate well for all sampling techniques with any of the tested analytical methods for PNA's. The lack of correlation requires that epidemiological studies be conducted to determine if the polynuclear aromatic hydrocarbons will be a good index of the hazards of exposure to coal tar pitch volatiles. It is desirable to find a new analytical method for assessing coal tar pitch exposures because of the obvious drawbacks of the benzene

soluble method. As its name implies, the method is non-specific, analyzing for all compounds soluble in benzene. There are also problems involved in the procedure itself: the weights of benzene soluble material may be erroneous due to a disintegration of the filter and loss of particulates during handling and extraction or due to a change in the water content of the filter between the tare and final weight after extraction. We feel that a method which is specific for certain compounds or classes of compounds is needed. At present, no method can be singled out as the best method. More work must be done to determine exactly which components of coke oven emissions are hazardous and at what concentrations they affect the worker adversely. Once this is known, the Federal standard must be re-evaluated, and suitable analytical methods selected. Gas chromatographic and high-pressure liquid chromatographic methods hold promise, because of their specificity and ease of operation.

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### Employment Information

See pages p A-5, A-9, A-10, and A-20 for information on positions available and positions sought.