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Analysis of Emissions Collected from Four Types of Iron Casting Molds*

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The levels of polycyclic aromatic hydrocarbons (PAH) and related compounds, phenols and particulates were determined in emissions collected from iron casting molds composed of four different types of chemical binders: furan, urethane, green sand with sea coal and phenol-formaldehyde resins in shell molds. The shell sample, with 50% particulates, contained the most water-soluble material; green sand, 25% particulates; furan, 10% particulates; and urethane, less than 2% particulate material. The portion of the particulate fraction soluble in cyclohexane varies from 16 to 36% between mold types; emissions from urethane and furan molds contained the lowest quantities of cyclohexane-soluble components and of PAH and related compounds. Phenol, which was found in all four foundry samples, was present in the highest concentration in emissions from urethane molds. Shell mold emissions contained the highest levels of 2- and 4-nitrophenol.

Introduction

Iron foundry workers, in particular molders and casters, may have a two- to three-fold greater risk of developing lung cancer than persons in the general population.^(1,2) This figure is derived from epidemiologic studies of workers whose first foundry exposures occurred more than 20 years ago, at a time when green sand molds were the most widely used mold type in the industry. Many of the molds used today are constructed of synthetic chemical binders. These mold binders have been used for too short a time to have contributed significantly to the elevated lung cancer rates observed in the epidemiologic studies. Only a few investigations have examined the chemical and physical characteristics of emissions released from the newer molds during the casting and cooling processes,^(1,3,4) and little is known about the chronic health effects of exposure to them. The current study was part of a larger effort designed to compare health effects associated with emissions from molds composed of the newer synthetic chemical binders with those of the more traditional green sand molds.

Benzo(a)pyrene (BaP), a carcinogenic polycyclic aromatic hydrocarbon (PAH), as well as other PAHs, have been found in green sand foundry dusts and fumes;⁽⁵⁻⁷⁾ these compounds are generally present in airborne emissions as condensates on particle surfaces. Extracts of green sand foundry dusts containing PAHs as well as other classes of organic compounds are mutagenic in the Salmonella/Ames test.^(8,9) In this paper we report the results of a comparison of the levels of phenols and some PAHs and related compounds in emissions from molds composed of four different types of chemical binders; furan (furfuryl alcohol, urea, toluenesulfonic acid), urethane (phenolic-isocyanate), green sand with sea coal and phenol-formaldehyde resins in shell molds.

Material and Methods

Sampling

Samples were collected from pouring lines at which grey iron was cast. Sampling locations, which would minimize contamination from other processes, were selected. The mold ingredients typified those used at most foundries. Cores, when present, were composed of the same materials as the molds. Because the greater part of the collected samples were destined for use in long-term animal studies which require large quantities of material,^(10,11) emissions were collected from highly concentrated sources with a high-volume sampler. Personal exposure samples were not collected.

Emissions from each of the mold types were collected at different foundries. The green sand molds were approximately 7'L x 3'W x 3'H and the shell molds were approximately 2'L x 1'W x 6"H. Both of these mold types were poured on automatic conveyor lines and carried into narrow cooling tunnels soon after pouring. The emissions from the green sand and shell molds were collected from their respective cooling tunnels shortly downstream of the pouring area. The furan samples were collected from molds approximately

TABLE I
Solubility in Water and Cyclohexane^A

Mold	% Sample Insoluble in Water	% Particulate Soluble in Cyclohexane	% Total Sample Soluble in Cyclohexane
Furan	10-15	19	2-3
Urethane	1-2	16	0.2-0.3
Shell	50	35	18
Green Sand	20-25	36	7-9

^AEach determination was performed three times for the shell and green sand samples and twice for the urethane and furan samples.

*Mention of company name or product does not constitute endorsement by the National Institute for Occupational Safety and Health.

TABLE II
Carbon and Hydrogen Content^A of
Cyclohexane-Washed Particulates

Mold	Percent Carbon	Percent Hydrogen	Ratio of Moles C: Moles H
Furan	22.95	4.78	0.4:1
Urethane	30.99	4.38	0.6:1
Shell	85.16	1.89	3.7:1
Green Sand	44.93	4.4	0.8:1

^AThe carbon and hydrogen contents of the washed particles were determined by combustion analysis at Galbraith Laboratories (Knoxville, Tenn.).

4'L × 4'W × 4'H. The molds were poured on the open foundry floor and emissions were collected directly from vent holes in the top of the molds. The urethane molds were approximately 2'W × 3'L × 1'H. The molds were placed in a cooling hood shortly after the metal was poured. The emissions were captured by a hood located closely above the cooling molds.

Emissions were transported from the collection site to the sampler through a short section of ductwork. The wet scrubber used for sample collection was described previously.⁽¹²⁾ In brief, the scrubber collects respirable particles between 0.2 and 10 μm in diameter and water-soluble vapors that evolve from the molds. The air being sampled is first drawn through the cyclone pre-cleaner at the rate of 47.2 L/sec (100 ft³/min), which removes particles larger than 10 μm in diameter from the airstream. Air discharged from the cyclone passes into the venturi scrubber through a restricted throat in which the velocity is increased to about 3050 m/min (10 000 ft/min). The high velocity air impinges on a stream of distilled water which is dispersed into fine droplets in which particles larger than 0.2 μm in diameter are captured. The water/particle agglomerates are removed from the airstream by cyclonic action in the entrainment separator. Water is removed from the sump of the entrainment separator and recirculated to the venturi scrubber where it is reused for further sample collection. The fractional removal

efficiency for particulates increased with particle size from a low of about 30% of particles less than 0.3 μm in diameter to about 80% of particles with a diameter of 15 μm.

Characterization of Water-Insoluble Sample Constituents

The collected samples were a suspension of dispersed particulates in an aqueous medium. The ratio of dissolved solids to water-insoluble particulates was determined by measuring the quantity of a known volume of sample retained by a pre-weighed Millipore filter (0.22 μm pore size). The sample and its fractions were dried in tared vessels at 60°C in a circulating hot-air oven (Lipshaw Laboratory Oven, Model 218, Detroit, Mich.) and desiccated overnight before weighing.

To determine the quantity of absorbed organic materials and the carbon content of the particulate fraction, the particulates were collected on filters and washed with 500 mL water to remove adhering water-soluble compounds. A weighed, dry aliquot was Soxhlet-extracted with cyclohexane for 16 hours to remove associated organic materials. After this, the particles and cyclohexane-soluble material were dried at 60°C in a circulating hot-air oven, desiccated and weighed.

PAH Determinations

The concentrations of eight compounds (*i.e.*, acridine, naphthalene, carbazole, phenanthrene, benzo(a)anthracene, chrysene, BaP and dibenzo(a,h)anthracene) were determined separately in the aqueous and particulate phases of the mold emissions. These eight compounds were selected for analysis to characterize complex effluent mixtures by such features as number of arene rings, nitrogen content and heterocyclic character. The compounds selected are representative of the range of compounds that have been found in previous studies. Particulates were removed from the aqueous phase by filtration and washed with three 50 mL aliquots of distilled water to remove residual water-soluble materials. They were dried overnight in a desiccator at room temperature, weighed, and the base-neutral fraction was extracted by sonication in cyclohexane. Using a continuous extractor, the base-neutral fraction was extracted from the aqueous

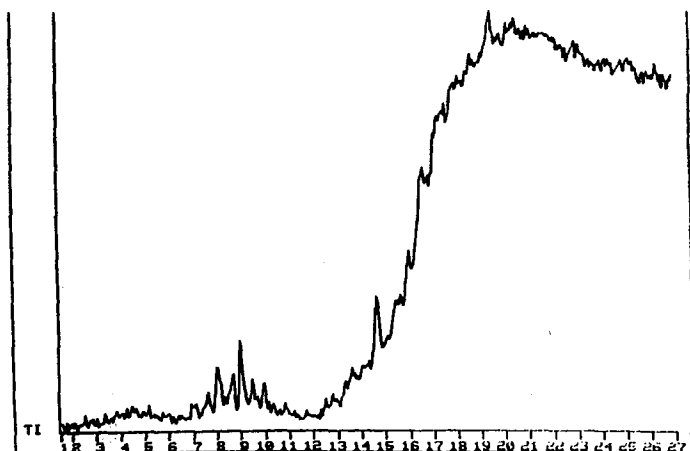


Figure 1a — Total ion current chromatogram by GC/MS of cyclohexane extract of water-insoluble particulate in furan sample for the determination of PAH-type compounds.

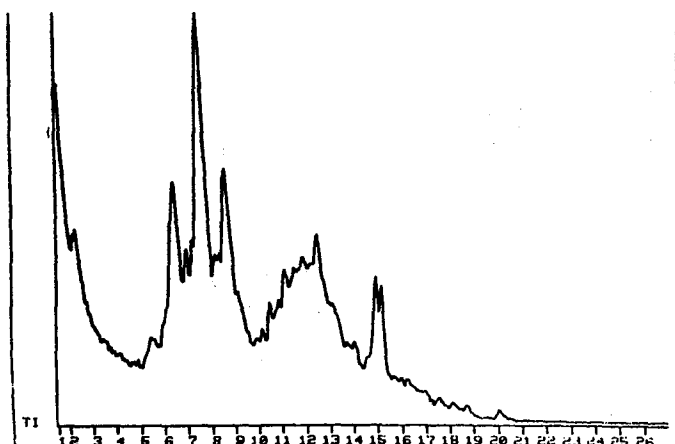


Figure 1b — Total ion current chromatogram by GC/MS of methylene chloride extract of aqueous phase in furan sample for the determination of PAH-type compounds

TABLE III
Concentrations of PAH and Related Compounds in
Cyclohexane Extracts of Water-Insoluble Particulate
($\mu\text{g/g}$ Particulate)

Compound	Furan	Urethane	Shell	Green Sand
Acridine	<0.6	<0.6	<0.6	<0.6
Naphthalene	<0.6	12	<0.6	<0.6
Carbazole	<0.6	<0.6	<0.6	<0.6
Phenanthrene	31	230	1500	7200
Benzo(a)anthracene	<0.6	5.4	350	1100
Chrysene				
Benzo(a)pyrene	<0.6	<0.6	270	230
Dibenzo(a,h)anthracene	<0.6	<0.6	<0.6	<0.6

phase with methylene chloride after adjustment to pH 11 with 50% sodium hydroxide. The residual aqueous phase was saved for extraction of phenols, described below.

The cyclohexane and methylene chloride extracts were concentrated by using Kuderna-Danish evaporators to a final volume of 2 mL. One microliter of each of the base-neutral concentrates was analyzed by gas chromatography/mass spectroscopy (GC/MS — Hewlett Packard 5985A) on a 6-ft glass column (2 mm ID) packed with 1% SP-2250 coated on 100/120-mesh Supelcoport. The column oven was temperature-programmed from 50 to 270°C at 15°C per minute. The mass spectrometer scanned from 45 to 450 atomic mass units (amu) in 3 seconds.

The remainder of the base-neutral extracts (cyclohexane/methylene chloride) was subjected to cleanup on silica-gel columns, concentrated and solvent exchanged to acetonitrile by evaporation of the original solvent under a nitrogen stream and replacement with acetonitrile. This procedure was repeated two times. It was then analyzed by high-pressure liquid chromatography coupled to a fluorescence detector (HPLC/UV) using a reverse-phase column, HC-ODS Sil-X, 250 mm long \times 2.6 mm ID (Perkin-Elmer Part No. 089-0716). Isocratic elution was employed for 5 minutes with 40% acetonitrile/60% water; then gradient elution to 100%

acetonitrile was employed over 35 minutes. The solvent flow rate was 0.5 mL/min. The HPLC injection volume was 25 μL .

Phenols

To extract the phenols, the aqueous phase from each sample that had been held in reserve after extraction of the base-neutral fraction was adjusted to pH 2 with 12 N HCl and was again extracted in a continuous extractor with methylene chloride to remove acidic compounds. The extracts were concentrated by using Kuderna-Danish evaporators to a final volume of 2 mL. One microliter was analyzed for phenols by GC/MS using a 6-ft column (2 mm ID) packed with 1% SP-1240 DA on 100/120-mesh Supelcoport. The column oven was temperature-programmed from 70 to 190°C at 8°C per minute. The mass spectrometer scanned from 45 to 350 amu in 3 seconds.

Results

Particulates

The collected samples contained components that were soluble in water as well as a particulate fraction that was water-insoluble. Table I shows the mean percent of the total dry weight of each sample that was present as water-insoluble particulates. The shell sample, with an average of 50% particulates, contained the most water-insoluble material, followed by green sand, composed of 25% particulates, and furan with 10% particulates. The urethane sample had less than 2% particulates, which was the lowest value observed among the four types of molds examined.

Most of the PAH and other organic compounds which are associated with the water-insoluble fraction can be extracted from the particulates with cyclohexane.⁽¹³⁾ The portion of the particulate fraction soluble in cyclohexane varied from 16 to 36% between mold types; the values from urethane and furan molds were lower than those from shell and green sand molds (Table I, percent particulate soluble in cyclohexane). The cyclohexane-soluble components from the urethane and furan samples were 16 and 19% of the total particulate fraction respectively, while those from shell and green sand were approximately 35%. This is of interest because most of

TABLE IV
Concentrations of PAH and Related Compounds in the Whole Sample ($\mu\text{g/g}$ Sample)^A

Compound	Furan		Urethane		Shell		Green Sand	
	Aqueous	Particulate	Aqueous	Particulate	Aqueous	Particulate	Aqueous	Particulate
Acridine	13	--	25	--	--	--	3.7	--
Naphthalene	--	--	--	0.2	--	--	--	--
Carbazole	--	--	--	--	--	--	--	--
Phenanthrene	--	3.4	--	4.6	230	720	8	1800
Benzo(a)anthracene	--	--	--	0.1	--	170	--	280
Chrysene								
Benzo(a)pyrene	--	--	--	--	--	130	--	58
Dibenzo(a,h)anthracene	--	--	--	--	--	--	--	--

^AThe dashed line (--) indicates not detectable.

TABLE V
Concentrations of Phenols in the Aqueous Phase
($\mu\text{g/g}$ Sample)

Compound	Furan	Urethane	Shell	Green Sand
Phenol	2000	50 000	1600	1000
Pentachlorophenol	3	< 2	< 2	< 2
4-Nitrophenol	48	420	1800	< 2
2-Nitrophenol	< 2	< 2	2300	< 2
2,4-Dimethylphenol	< 2	21	60	< 2

the airborne carcinogenic PAHs and some mutagenic compounds are known to be present in mold emissions as condensates on the surface of fine particles.^(4,8) Several studies have indicated that adsorption onto the surface of metal oxide particles increases the carcinogenic potency of PAHs.^(14,15) Considering the quantity of the cyclohexane-soluble material in terms of the fraction it represents of the whole sample (Table I, percent total sample soluble in cyclohexane), the cyclohexane-soluble fraction is actually about 18% of the total dry weight of the shell sample. This value is 7 to 9% for green sand, 2 to 3% for furan, and 0.2 to 0.3% for urethane.

To compare the concentrations of inorganic carbon, the water-insoluble fraction of each sample was washed with 500 mL distilled water to remove adhering water-soluble chemicals, and Soxhlet extracted with cyclohexane for 16 hours to remove associated organic materials. Table II shows the percent of carbon and hydrogen in the washed particulate fraction from each of the foundry samples. The shell sample contained considerably more carbon than the particulates from the other three samples. Carbon represented 85% of the total dry weight of the washed shell particles, 45% of the green sand, 31% of the urethane and only 23% of the furan particulates.

The ratios of the moles of carbon atoms to the moles of hydrogen atoms in the particulate fraction of the furan and urethane samples were 0.4:1 and 0.6:1, respectively, indicating that some of the carbon may have been present in resid-

ual organic compounds that were not extracted with cyclohexane. The ratio of 1:1 in the green sand sample indicates that much of the carbon was present in the elemental form. In the shell sample, the ratio of almost 4:1 shows that most of the carbon is present as inorganic carbon. These values may be pertinent to interpretations of the health effects of foundry emissions because the biological activity of toxic materials adsorbed to carbon particles may differ from those adsorbed to metal oxide particles.⁽¹⁶⁾

PAH Analysis

The concentrations of eight PAHs and related compounds in the mold emissions were determined in extracts of both the aqueous phase and the particulates. Figures 1a - 4a show reconstructed total ion current chromatograms of the cyclohexane extracts of the particulates in each of the samples. The largest peak in the green sand sample contains phenanthrene and anthracene, two closely related compounds which were not separated by the GC/MS system used. Some of the other PAHs in this sample are benzo(a)pyrene, fluoranthene, chrysene, benzo(a)anthracene, and their methylated and dimethylated derivatives. Chrysene and benzo(a)anthracene also cannot be differentiated by the GC/MS system used.

The PAH concentrations in the cyclohexane extracts of particulates from the four foundry samples are shown in Table III. The data are presented in terms of microgram PAH per gram particulate. To measure phenanthrene, benzo(a)anthracene and chrysene, which eluted in pairs with other PAHs in the GC/MS system, the samples were also analyzed by HPLC. Although this technique tends to be more subject to interferences for PAH than the GC/MS, HPLC can differentiate between certain combinations of compounds, such as benzo(a)anthracene and chrysene, which were not distinguished by GC/MS. Table III gives the combined concentrations, as determined by GC/MS, of benzo(a)anthracene and chrysene. Using HPLC, it was found that chrysene represented a considerably greater portion of the total benzo(a)anthracene/chrysene GC/MS peak than did benzo(a)anthracene in three of the four foundry

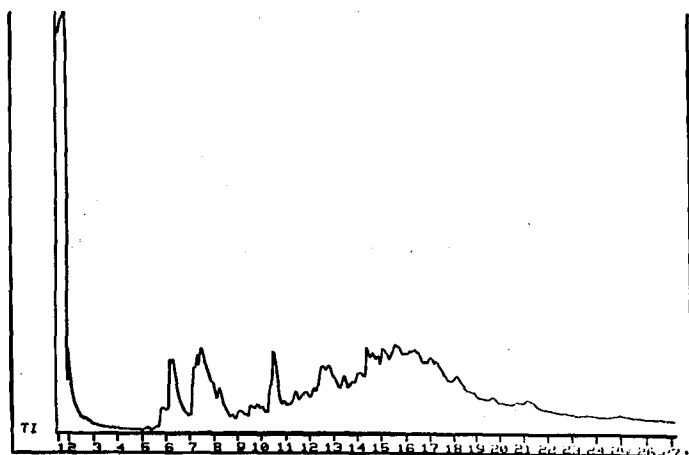


Figure 2a — Total ion current chromatogram by GC/MS of cyclohexane extract of water-insoluble particulate in urethane sample for the determination of PAH-type compounds.

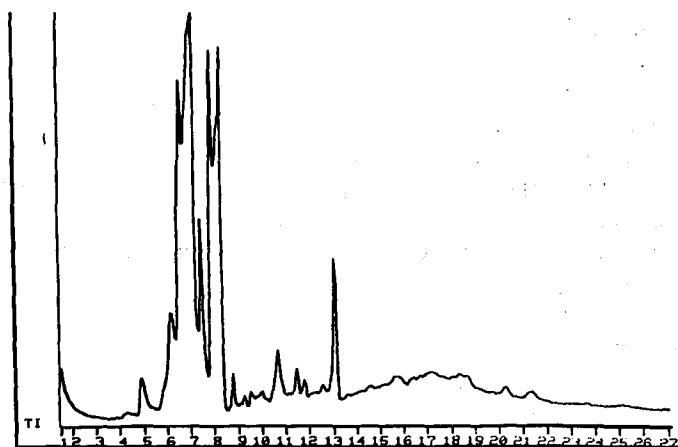


Figure 2b — Total ion current chromatogram by GC/MS of methylene chloride extract of aqueous phase in urethane sample for the determination of PAH-type compounds.

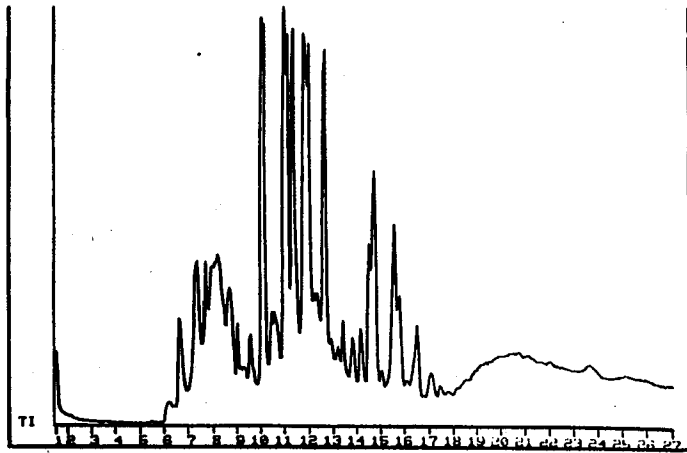


Figure 3a — Total ion current chromatogram by GC/MS of cyclohexane extract of water-insoluble particulate in shell sample for the determination of PAH-type compounds.

samples. Of these two compounds, only chrysene was detected in the furan sample; there was approximately four times as much chrysene as benzo(a)anthracene in the urethane and shell samples. The reverse situation occurred in the green sand sample, which had about five times as much benzo(a)anthracene as chrysene. Acridine, carbazole, and dibenzo(a,h)anthracene were not detected in any of the samples. Naphthalene was found in only the urethane sample; phenanthrene was present in all four samples. Measurable quantities of BaP were present only in particulates from the shell and green sand samples. The chromatograms of the methylene chloride extracts of the aqueous phase from each of the samples are shown in Figures 1b-4b. In contrast to the chromatographic profiles of the extracts from the particulates, the aqueous phase had little material which eluted in the PAH region of the chromatograph. The only PAH which eluted as a distinct peak in the chromatographs of the aqueous phase of any of the samples was phenanthrene in the green sand sample (Figure 1b).

The data for the base-neutral compounds are summarized in Table IV. The values are presented in terms of microgram per gram whole sample (water-soluble and insoluble materials combined) rendering the data for the PAH concentrations in the aqueous and particulate phases in all samples directly comparable. Of the compounds analyzed, only acridine in the furan, urethane and green sand samples, and phenanthrene in the shell and green sand samples were detected. These data for the most part reflect the water-solubility of the compounds studied. The combined concentrations of the base-neutral compounds in the aqueous and particulate phases were markedly lower in the urethane and furan samples than in the shell and green sand samples.

Phenols

The concentrations of five phenols were determined in each of the foundry samples (Table V). Because they are highly soluble in water, only the aqueous phase was examined. Of these compounds, phenol itself was the most prevalent and was found in all four foundry samples. 4-Nitrophenol, the second most prevalent phenol, was present in all but the

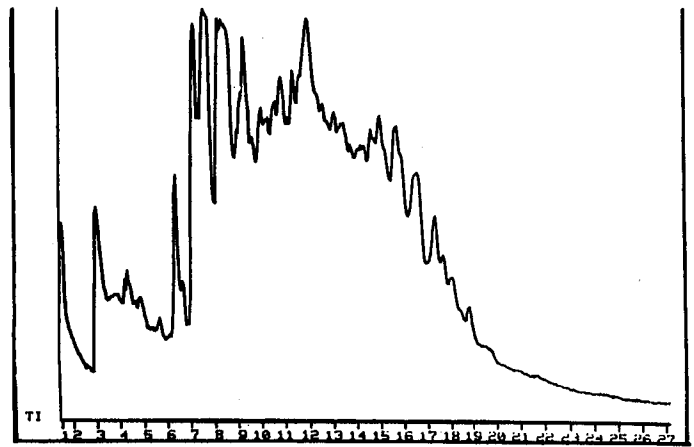


Figure 3b — Total ion current chromatogram by GC/MS of methylene chloride extract of aqueous phase in shell sample for the determination of PAH-type compounds.

green sand sample. 2-Nitrophenol was found in the shell sample only, and 2,4-dimethylphenol was found in the shell and urethane samples.

Discussion

The levels of PAH, phenols, and particulates in emissions collected from four different mold types were measured. These analyses were performed in support of a large-scale animal bioassay for which a maximum quantity of sample had to be collected in a minimum time period; hence, it was not possible to collect samples in a way which enabled the quantity of materials captured per unit time to be related to the emissions produced by any one mold. Also, to enable the efficient collection of materials, source samples were collected; therefore, the data cannot be directly transcribed into breathing zone exposures.

Samples collected from shell and green sand molds had markedly higher levels of particulates with about twice as much associated organic matter and greater total quantities of PAH than did samples collected from furan and urethane molds. Phenol was found in samples collected from all four mold types but was particularly high in that collected from urethane (phenolic-isocyanate) molds. In an earlier study, emissions from urethane molds were also found to have more phenol than those from 11 other mold types.⁽³⁾

The total quantities of particulates and BaP which evolve from twelve different types of molds were compared earlier by R. James and W. Scott at Southern Research Institute.⁽⁴⁾ They found that the total quantity of particulates and BaP in the mold effluents was lowest for urethane, followed by the furan and shell molds. The highest quantity of particulates was evolved from green sand molds. Our data concur with those findings with the exception that the portion of particulates and BaP per gram sample was higher for shell than for green sand molds. An important difference between the two studies is that the former was performed in a laboratory setting under which parameters such as sand-to-binder and sand-to-metal ratios could be carefully controlled. This was not feasible in the current study because mold effluents were

collected in commercial foundries during a normal day-to-day production schedule. In the current study, the sand-to-metal ratio in the green sand molds was higher than that in the other mold types. The larger bulk of sand surrounding the iron casting could have trapped much of the particulates and prevented their escape from the mold. Because BaP in mold effluents is associated with particulate material, this may account for the differences in the relative quantities of particulates and BaP found between green sand and shell molds in the two studies.

The relatively good agreement between the current data and those from the controlled laboratory studies described above^(3,4) indicates that the source samples collected with the wet scrubber for use in animal bioassays are fairly representative of the materials emitted from cooling molds. The high quantities of phenols that evolve from urethane molds are of concern because of their known toxicity as well as their possible activity as tumor promoters.⁽¹⁷⁾ Although the PAH levels (in particular, the known carcinogen BaP) are low in emissions from urethane and furan molds, further studies are needed to determine whether or not other classes of carcinogens are present in emissions from urethane and furan molds. Also, biological investigations are essential for exploring the potential health effects from exposure to these mold emissions.

Conclusion

In conclusion, there are vast differences in the ratio of particulate to water-soluble materials in emissions from different types of molds. A comparison of data from the current study and those conducted by Scott and James^(3,4) indicates that the differences in the ratio of particulate to water-soluble materials actually reflects a difference in the total amount of particulate released from the molds during cooling. Much of the particulate material in the shell mold emissions is represented by carbonaceous soot. Shell molds are very thin and have a much lower sand-to-metal ratio than do the other mold types studied. As a result of the close proximity of the sand binder to the molten metal, much of

the sand binder burns as the poured molds cool. In contrast, the other three types of molds have a high sand-to-metal ratio and very little open burning occurs while they cool. The difference in the degree of combustion that these mold binders undergo accounts for much of the difference in the quantities of elemental carbon present in the shell mold emissions and in those from the other types of molds studied. The binder content and the ultimate sand temperature also contribute to differences in the elemental carbon content of the different types of mold emissions.

Particulates and their associated PAH have long been suspected to be those components of the foundry atmosphere that are most likely to produce chronic health effects. The emissions from furan and urethane molds contain substantially less particulate and PAH than do those from the other mold types. This may lead to a significant decrease in the health hazards arising from exposures to those molds. On the other hand, an increase in water-soluble materials, such as phenols from urethane molds, may represent a health hazard unique to modern foundries. Detection of this material at the source of emissions does not necessarily imply that it will be present in the breathing zone, but the potential for this is always there.

Acknowledgement

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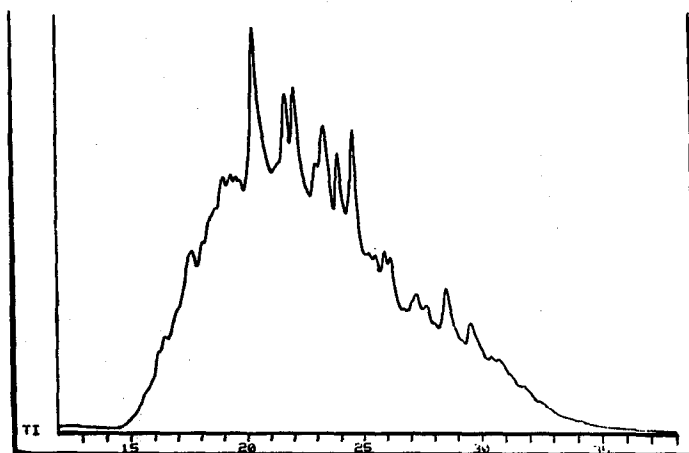


Figure 4a — Total ion current chromatogram by GC/MS of cyclohexane extract of water-insoluble particulate in green sand sample for the determination of PAH-type compounds.

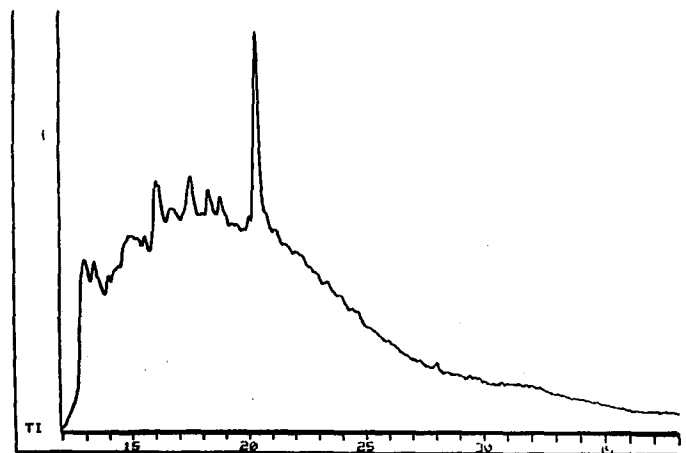


Figure 4b — Total ion current chromatogram by GC/MS of methylene chloride extract of aqueous phase in green sand sample for the determination of PAH-type compounds.

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