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EXECUTIVE SUMMARY

For several years, National Oceanic and Atmospheric Administration (NOAA) employees occupying Floors 3 through 5 of the Silver Spring Metro Center Building One (SSMC-1) in Silver Spring, MD have complained about ailments which they have associated with poor indoor air quality. NOAA and the General Services Administration (GSA) commissioned at least six indoor air quality surveys to seek the causes of these complaints. In one of the later surveys [5], it was concluded that phenol emissions from an epoxy floor-leveling material used in leveling the floor slabs were causing the indoor air quality complaints from Floors 3 through 5. To obtain an independent analysis and assessment, NOAA and GSA asked the National Institute of Standards and Technology (NIST) to ascertain whether phenol (or any other volatile organic compound) was being emitted from the epoxy floor-leveling material and, if so, to recommend remedial actions for mitigating or eliminating the emissions.

Prior to investigating the epoxy floor-leveling material, a review was made of the construction and occupancy history of SSMC-1, the installation of the floor-leveling material, occupant complaints about the poor indoor air quality, and the indoor air quality surveys.

Based on this review, the following observations were made:

- 1) the indoor air quality complaints were mainly confined to Floors 3 through 5;
- 2) the epoxy floor-leveling material was installed on Floors 2 through 5;
- 3) occupant complaints associated with the indoor air quality on Floors 3 through 5 were received by NOAA's management almost immediately after these floors

were occupied in January 1990; there were no corresponding complaints from occupants of Floors 6 through 9;

- 4) although a large number of indoor air quality parameters were monitored, phenol was the only volatile organic compound having a concentration in the air which was considered to be high; and
- 5) phenol was present in, and was emitted by the floor-leveling material.

These observations strongly supported the suggestion that the floor-leveling material might be a primary source of the phenol in the air on Floors 2 through 5, and, therefore, it was decided that further investigation of this material was warranted.

NIST designed and executed a random sampling plan to obtain samples of the epoxy floor-leveling material on Floors 2 through 5. The chemical composition and physical properties of the material were determined using a variety of analytical procedures including thermogravimetric analysis, gas chromatography/mass spectrometry (GC/MS), liquid chromatography/mass spectrometry (LC/MS), and ultraviolet spectrophotometry (UV-spectrophotometry). It was concluded from these analyses that

1. The floor-leveling material on Floors 2 through 5 contained phenol; the measured concentrations of phenol ranged from 0.25 to 0.52 % of the material's mass. The measured phenol concentrations were likely to have been a little lower than the true concentrations, since some phenol would have been lost during sample

preparation.

2. Phenol was emitted from the floor-leveling material and was a major contributor to the phenol concentration in the air.
3. The concrete floor slab beneath the epoxy floor-leveling material was not contaminated by phenol.
4. Phenol was the volatile organic compound present in the greatest quantity in the floor-leveling material and it was the one emitted at the highest rate in the Gas Chromatography/Mass Spectroscopy studies, and, thus, it was the one most likely to adversely affect the indoor air quality on Floors 2 through 5.

Several possible strategies for mitigating or eliminating phenol emissions from the floor-leveling material were considered. They included baking out the phenol from the material, using activated carbon filters to scavenge phenol from the air, sealing or encapsulating the floor-leveling material, and removing the material. It was concluded that the most certain, and probably the most practical, strategy would be to remove the floor-leveling material from the building and replace it with a portland cement-based leveling material which contained no volatile organic compounds and would act as a scavenger of phenol left on the floor slab.

1. INTRODUCTION

Ever since Floors 3 through 5 of the Silver Spring Metro Center Number One (SSMC-1) building were occupied in January 1990, the occupants of these floors have complained about the air quality and a variety of ailments that they attributed to this poor air quality. The National Oceanic and Atmospheric Administration (NOAA) and the General Services Administration (GSA) commissioned at least six indoor air quality surveys between February 1990 and June 1993 to identify the source of the indoor air quality problem. In a recent survey [5], phenol emissions from the floor-leveling material on Floors 2 through 5 were implicated as a cause of the indoor air quality complaints. In August of 1993, the GSA and NOAA requested that the National Institute of Standards and Technology (NIST) investigate whether phenol, or any other volatile organic compound, was being emitted by the epoxy floor-leveling material and, if so, to recommend remedial strategies for eliminating or mitigating the emissions.

2. BACKGROUND

Prior to discussing the present studies of the epoxy floor-leveling material, the building's construction and occupancy history, the installation of the floor-leveling material, occupants' complaints, and the results of indoor air quality surveys will be reviewed. The objective is to organize the evidence chronologically (see Figure 1) and to determine if it suggests that phenol in the leveling material could indeed be a likely source of emissions affecting the indoor air quality on Floors 2 through 5.

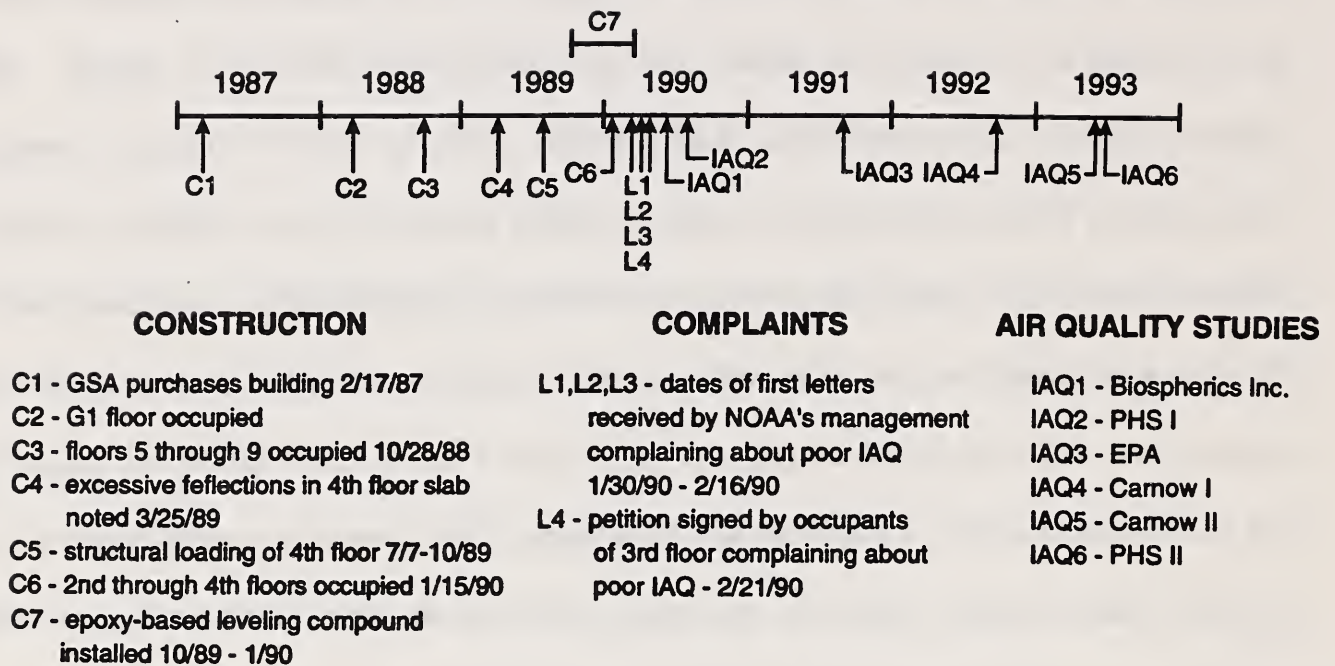


Figure 1. Time line for construction, occupancy, occupant complaints, and indoor air quality events.

2.1 Construction and Occupancy of the Building

The SSMC-1 Building is located in Silver Spring, MD. It is one of a group of four buildings housing employees of the National Oceanic and Atmospheric Administration (NOAA). SSMC-1 is a nine-story, reinforced concrete frame building with three levels of underground parking (designated G1 through G3). Floors 3 through 9 of SSMC-1 have basically the same

floor plan. Floor 2 has a smaller area because the first floor atrium extends through it. The few indoor air quality complaints from occupants of Floor 2 may be attributed to the additional flow of outdoor air resulting from the opening and closing of the doors in the lobby [5].

Base-Building construction of SSMC-1 began in 1984, and was completed in 1986. The building, which was purchased from the owner by GSA on February 17, 1987 [14], was occupied in several stages. On March 25, 1988, parts of G1 (the floor below the lobby) and the lobby were occupied and on October 28, 1988, a portion of the fifth floor and all of Floors 6 through 9 were occupied. The remainder of the building (Floors 2 through 4 and the balance of Floor 5) was scheduled to be occupied by June 1989. Occupancy of the building, however, was not completed until January 1990 because of the events described below.

On March 3, 1989, while furniture was being installed on Floor 4, significant deflections were observed in the floor slab, and a decision was made to temporarily cease furnishing this floor until the structural implications of the deflections could be ascertained [14]. This temporary delay was changed into a full halt in occupancy when excessive deflections were also noted in the floor slabs on all the floors.

Because of these deflections, GSA initiated a full-scale survey of the building to ascertain its as-built condition, and published the findings in General Services Administration Report GSA PCN Number ZMD 66130 [14]. Two outcomes of this study were that the concrete floor slabs on Floors 2 through 9 were reinforced with steel brackets in areas which were considered to be

structurally deficient, and Floor 4 was loaded to determine the structural response of a "typical floor slab". The structural loading of Floor 4 took place in July, 1989. Deflection diagrams were produced for Floors 3 through 9 [14] mapping the extent by which the slabs were out of level at each location (the deflection diagrams were made available for the present study, but no diagram was found for Floor 2). The GSA survey team also found that the builder had applied large amounts of an inorganic floor-leveling material on all nine floors of SSMC-1. This inorganic floor leveling material was removed from Floors 2 through 4 and most of Floor 5 during the structural retrofit of the building. It has not been ascertained why the material was only removed from these floors, but it may have been because these floors were the only unoccupied floors at the time the building was retrofitted.

An example of a deflection diagram from the GSA report [14] is shown in Figure 2. In this figure, the contour lines indicate the boundaries of each of the sunken areas in the floor and, thus, the probable boundaries of the "puddles" of inorganic floor-leveling material. For this reason, the deflection diagrams have often been called "puddle diagrams". The numbers within each puddle indicate the depth by which the floor is out-of-level in units of feet; thus, 0.2 is 0.2 feet so that the floor is 61 mm (2.4 inches) out-of-level at this location.

2.2 Installation of the Epoxy Floor-Leveling Material

As mentioned above, the inorganic floor-leveling material was removed from Floors 2, 3, and 4 and a portion of Floor 5 which was not occupied at the time the building was surveyed.

This inorganic floor-leveling material was replaced by a floor-leveling material containing an epoxy binder (hereinafter called the leveling material).

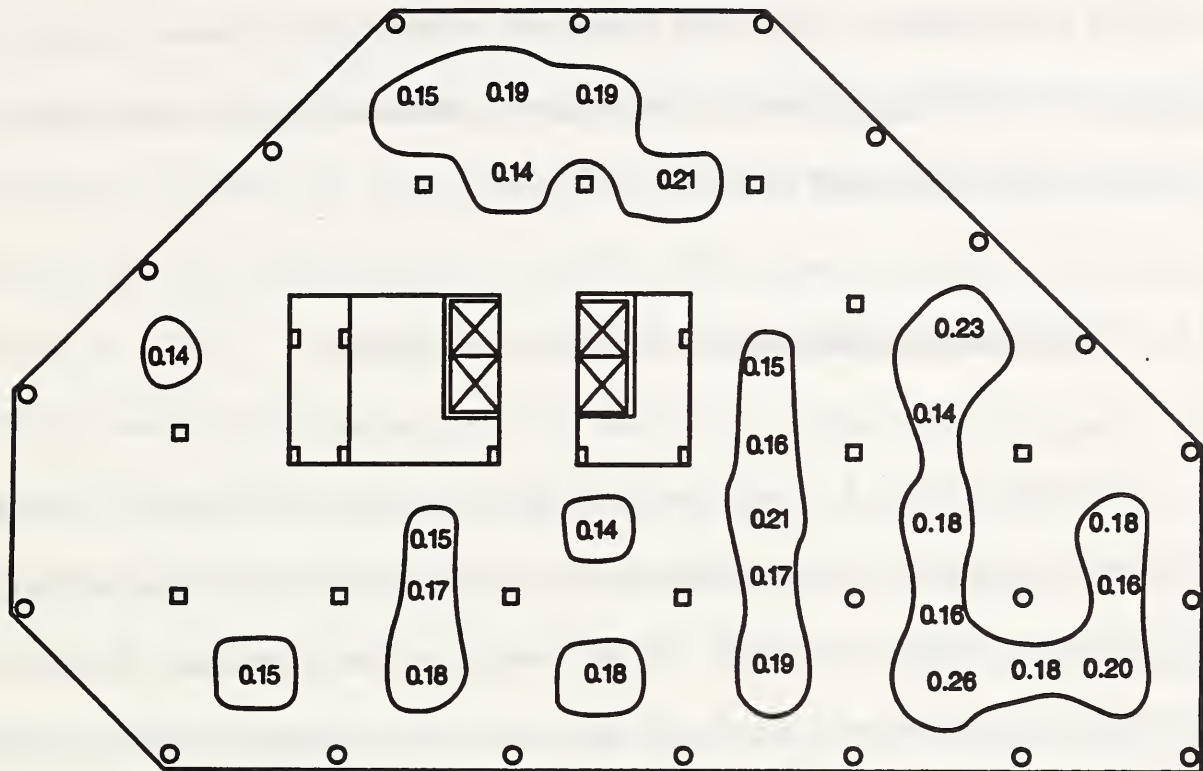


Figure 2. Deflection diagram for Floor 4 of SSMC-1. Numbers indicate out-of-levelness dimensions in feet.

From records and interviews with NOAA's employees, the majority of the leveling material was installed between October 1989 and January 1990 with the most likely dates being between late October, 1989 and early December, 1989. The October date was deduced from a letter dated September 25, 1989 in which a representative of the leveling material producer

gave the installer instructions on the installation of the material. The December date was deduced from knowledge that Floors 3 through 5 of SSMC-1 were occupied at the end of January 1990, and, prior to occupying a floor, the room partitions, carpet, and furniture would have had to be installed, and the heating, ventilation, and air conditioning (HVAC) system would have had to be balanced. These tasks would have required, at a minimum, several weeks of effort once the leveling material had cured and, thus, the latest that the leveling material could have been installed was early December, 1989.

2.3 Occupants' Complaints about the Indoor Air Quality

Soon after Floors 2 through 5 were occupied in January, 1990, NOAA's management began to receive letters complaining about ailments, mostly irritation of the eyes, nose and throat and nausea and fatigue, associated with the indoor air quality condition on these floors. Complaints continued to be received until these floors were vacated in August, 1993 [18].

The earliest letter was dated January 30, 1990; in it the writer complained about chest, eye, and throat irritations. In a letter dated February 16, 1990, six out of seven occupants on Floor 5 complained about headaches and nausea and inferred that these ailments were related to the installation of the leveling material on Floor 5. (Apparently, at the time this letter was written, Floor 5 was almost entirely unoccupied). In a letter dated February 17, 1990, an employee, who had recently moved from Floor 7 to Floor 3, complained about numerous ailments which had occurred since moving to Floor 3. On February 21, 1990, most (perhaps

all) of the occupants on Floor 3 signed a petition demanding an improvement in the indoor air quality on this floor.

2.4 Indoor Air Quality Surveys

On February 28, 1990, soon after receiving the initial complaints, NOAA's management commissioned an indoor air quality survey of SSMC-1. The survey was performed by Biospherics Inc. [1]. Since this survey, at least five other indoor air quality surveys have been performed in SSMC-1. In chronological order, the six surveys were conducted by 1) Biospherics Incorporated [1] (hereinafter called **Biospherics**); 2) the Federal Occupation Health Agency [13] (hereinafter called the Public Health Service I survey or **PHS I**); 3) the Environmental Protection Agency [11] (hereinafter called **EPA**); 4) Carnow, Conibear & Associates [5] (hereinafter called **Carnow I**); 5) Carnow, Conibear & Associates [6] (hereinafter called **Carnow II**); and 6) the Public Health Service [18] (hereinafter called **PHS II**). Site visit dates, report dates, and indoor air quality parameters measured in each survey are presented in Table 1.

The objective of these surveys was to identify the factor(s) which might be causing the indoor air quality complaints on Floors 2 through 5. Since these factors were not readily identifiable, the early surveys (**Biospherics** [1], **PHS I** [13], and **EPA** [11]) monitored comfort parameters (air movement, relative humidity, ambient temperature, and the concentrations of carbon dioxide (CO₂) and carbon monoxide (CO)), and a few other air quality parameters. The

results from these surveys served to establish baseline data, but did not identify any parameter that appeared likely to be a major cause of the complaints on Floors 2 through 5. Included among the air quality parameters were particulates, bioaerosols, aldehydes, volatile hydrocarbons, and the comfort parameters. The parameters monitored in each survey are indicated by an "X" in Table 1.

Table 1: Parameters Investigated in the Air Quality Surveys

Report	Biospherics	PHS I	EPA	Carnow I	Carnow II	PHS II
Report Date	3/21/90	undated	3/19/92	3/17/93	6/15/93	6/15/93
Site Visit Dates	2/28/90	7/12/91 7/19/91 8/ 2/91 8/29/91	8/27/91 8/28/91 8/29/91	11/18/92 12/ 8/92 1/13/93 1/26/93 1/29/93 2/ 5/93	5/10/93 6/ 4/93	5/28/93
Comfort Parameters	X	X	X	X		
particulates	X	X				
aldehyde		X	X	X		
NO _x			X	X		
bioaerosols				X		
ozone						X
hydrocarbons	X	X	X	X		
xylene		X	X	X		
ethyl-benzene				X		
phenol				X	X	

Overall, the first three surveys (Biospherics [1], PHS I [13], and EPA [11]) did not identify any compound having an abnormally high concentration. Instead, they emphasized the need for changes in the comfort parameters and, in response, improvements were made in the HVAC system. The surveys provided indirect evidence that the source of the indoor air quality complaints could be a volatile organic compound. For example, the Biospherics [1] survey reported "very strong chemical-like odors" on Floors 2 through 5; the PHS I [13] survey mentioned "formaldehyde-like odors"; and the EPA [11] survey noted "a new plastic or vinyl odor vapor" on Floors 2, 3, 4, and 7 of SSMC-1.

Since the improvements in the HVAC system did not lead to a cessation of the indoor air quality complaints, another indoor air quality survey was commissioned which was conducted by Carnow & Associates (Carnow I [5]). This survey included analyses for phenol. The Carnow I [5] survey, like the follow-up Carnow II [6] survey, detected high concentrations of phenol in the air; it concluded that "the distinct chemical solvent type odor noticeable on the 3rd and 4th floors is associated with phenol, which is a component of the floor-leveler used on these floors". The mean values of the phenol concentrations reported in the Carnow I and II reports are presented in Table 2.

In addition to the air quality survey, the Carnow I [5] report presented results of a headspace analysis of the flooring material (carpet, adhesive, and leveling material) in which a container was placed over the flooring materials for two days after which the air within the container was sampled for volatile organic compounds. From this analysis, it was concluded

that "the floor leveler compound used on the lower floors (two through five) emits volatile organic compounds, primarily **phenol, xylene, and ethyl benzene.**" Thus, the Carnow I [5] and II [6] surveys provided strong evidence of a connection between phenol in the air of Floors 2 through 5 and phenol emissions from the leveling material. Further, since the phenol concentrations in the air were highest on Floors 3 through 5 which had the most indoor air quality complaints, there was strong evidence that phenol emissions were associated with the complaints.

Table 2: Average phenol concentrations in the air on Floors 2 through 5.

Floor	Epoxy Floor-leveling Material	Carnow I survey (ppm)	Carnow II survey (ppm)
2	Present		
3	Present	0.01*	0.02
2	Present	0.01	0.01
5	Present	0.01	
6	Absent		
7	Absent	0.003	0.003
8	Absent	<0.005	
9	Absent		

* A phenol concentration of 0.27 ppm was recorded on this floor in an area in which the rug adhesive was being removed from the leveling material at the time of the survey. Since this reading was atypically high, it was not averaged in with the other readings on Floor 3.

3. EPOXY FLOOR-LEVELING MATERIAL

According to the manufacturer; the epoxy floor-leveling material is made by blending four to ten parts of sand with one part of an epoxy binder. The epoxy binder is a two-part system which is made by mixing four parts of an epoxy resin (PART A) with one part hardener (PART B).

PART A contains an epoxy resin, xylene (1 to 5% by volume), and triphenylphosphite. Xylene is added as a non-reactive diluent [10] to reduce the viscosity of the resin during pouring and mixing. Triphenylphosphite accelerates the cure of the binder and reduces the set time (gel time) of the binder [10, p 13-19].

PART B contains phenol (1 to 5% by volume) and a number of amine crosslinking agents including polyoxypropylene diamine (40 to 50% by volume), proprietary amine adducts, diethylenetriamine (1 to 5% by volume), triethylenetriamine (1 to 5% by volume), and triethanolamine (1 to 3% by volume). The amine crosslinking agents convert the normally linear epoxy resin into a three-dimensional, crosslinked network. Phenol is used in controlling the size of the crosslinked network which in turn controls the flexibility of the leveling material. It performs this function by terminating the polymerization reaction [3,10].

Phenol molecules taking part in the polymerization reactions become chemically bound to the epoxy resin and cannot easily be emitted into the atmosphere. However, chemical

reactions are seldom 100 % efficient and, hence, some portion of the phenol molecules do not take part in the chemical reaction. Unreacted phenol molecules are not chemically bound and are more likely to diffuse out of the leveling material. According to the manufacturer, the maximum amount of phenol present in a properly mixed leveling material in which ten parts of sand are mixed with one part of binder is 0.048 % by volume. The amount of free phenol in a properly mixed and cured leveling material (that is, phenol which has not taken part in the polymerization reaction), therefore, should be much less than 0.048 % by volume¹.

4. SAMPLING PLAN

A sampling plan was designed to detect large differences in the phenol concentrations between floors and to detect the presence of other contaminants. The design of the sampling plan was constrained by several practical considerations. These constraints were imposed by the occupancy of the building at the time that samples of the leveling material were obtained and the need to minimize the number of cuts in the overlying carpet. To minimize the number of cuts in the carpet, it was assumed that the deflection diagrams provided accurate representations of the locations of the leveling material on Floors 3 through 5; (as mentioned previously, a deflection diagram was not available for Floor 2.) For safety reasons, cores were not taken

¹Information in the manufacturer's literature and correspondence was in terms of volume, whereas the analytical results reported in section 5.0 are terms of mass. NIST was not able to obtain a sample of the epoxy binder from the manufacturer, so it was not possible to make the conversion between volume and mass with any accuracy. The density of cured epoxy resins is usually in the range from 1.1 to 1.4 g/cm³; while the density of sand is usually in the range from 2.65 to 2.67 g/cm³.

from highly "trafficked" areas like aisles and elevator lobbies.

Given the constraints, a plan was devised in which all areas containing the leveling material had an equal opportunity of being sampled. The protocol was as follows:

1. Program the floor plan for each floor into a computer and, for each floor, randomly select 100 locations and sequentially record and map each of these locations on the floor plan. Starting at the first number in the sequence, perform the actions described in Steps Two through Five.
2. When the prescribed location has been found to within 450 mm, decide whether it falls outside a puddle of the leveling material as mapped in the deflection diagram or within a heavily trafficked area. If it falls outside a puddle or within a heavily trafficked area, proceed to Step Four. Otherwise, cut the carpet and go to Step Three.
3. Obtain a core of the leveling material, using a 28 mm inside diameter coring bit. (This bit was selected because it readily cuts through the leveling material, but immediately halts once it reaches the concrete floor slab). The depth of the leveling material is recorded, and the specimen is placed in an air-tight jar. The jar was immediately labelled with the floor number, the location of the core, date, time, and the name of the technician.
4. Advance to the next location in the sequence, and repeat Steps Two and Three until 12 cores have been obtained from each floor or until all 100 locations have

been visited. (Note: 14 cores were obtained on Floor 2.)

5. Repeat Steps One through Four on the next floor.

Care was taken to ensure that the samples would not be contaminated or tampered with. When the cores had been brought into the laboratory, the top surfaces were cleaned of any residual carpet adhesive and the densities of the cores measured. The average density for the cores was 2.3 g/cm^3 .

Each core was crushed in a mortar and pestle until the maximum particle size was less than 3 mm in the longest dimension. The particles were then mixed and a one-gram sample -- a small portion of the total mass of the specimens -- was removed for analysis by GC/MS (gas chromatography/mass spectrometry). The remainder of the material was ground into a powder (100 % passing through a $300 \mu\text{m}$ mesh) in a ball mill; the powder was immediately put into an air-tight jar. The smell indicated that phenol continued to be released during the crushing and grinding of the leveling material; thus, the phenol concentrations reported in Section 5.2 may be a little lower than those that existed in the intact cores.

For each floor, the twelve powdered specimens were randomly assigned to one of two groups and, within each group of six specimens, a composite specimen was prepared by mixing an amount of material from each specimen which was proportional to its depth. (This was done to ensure that the contribution from each core was approximately proportional to the amount of material it represented). Thus, two composite specimens were made for each floor.

5. RESULTS FROM STUDIES OF LEVELING MATERIAL

5.1 Thickness, Area, Volume, and Mass of the Leveling Material

The total areas covered by the leveling material on Floors 3 through 5 were estimated from the areas of the puddles drawn on the deflection diagrams. For Floor 2, a deflection diagram was not available, so the total area covered by the leveling material had to be estimated from the sampling data. Specifically, out of the first 20 sampling locations, 14 cores were obtained, and, thus, approximately 70% of the area of Floor 2 was covered by the leveling material.

The thickness of the leveling material at any point sampled on Floors 2 through 5 was taken to be the depth of the core. As a check, the average thicknesses of the cores from a floor were compared with the average of the out-of-levelness readings recorded on the deflection diagrams for the floor. From Table 3, it was clear that the average thickness of the cores was always less than the out-of-level depth recorded on the deflection diagrams. This implies that, even after the leveling material was installed, the floors were not level.

Knowing the density (approximately 2.3 g/cm^3), the average thickness, and the area covered by the leveling material, it was possible to estimate the total volume and mass of the leveling material on each floor. These estimates are presented in Table 4. The total volume of the leveling material for all floors was approximately 37 m^3 and the total mass was approximately 85000 kg.

Table 3: Comparison of the average of the out-of-level measurements obtained from the deflection diagrams with the average thickness of the leveling material cores obtained from Floors 2 through 5.

Statistic	Floor 2		Floor 3		Floor 4		Floor 5	
	deflection depth (mm)	core tk (mm)	deflection depth (mm)	core tk (mm)	deflection depth (mm)	core tk (mm)	deflection depth (mm)	core tk (mm)
average	n.a.*	14	54	14	53	25	59	24
standard deviation	n.a.	10	10	11	9	13	14	15

A deflection diagram was not available for Floor 2.

Table 4: Thickness, area, volume, and mass of the leveling material on Floors 2 through 5.

Floor	thickness (mm)	area (m ²)	volume (m ³)	mass (kg)
2	16	750	11	25000
3	16	400	6	14000
4	25	400	10	23000
5	24	400	10	23000

5.2 Chemical Composition and Physical Properties of the Leveling Material

The chemical compositions and physical properties of the leveling material were determined using a variety of analytical procedures including: thermogravimetric analysis, gas chromatography/mass spectrometry (GC/MS), liquid chromatography/mass spectrometry (LC/MS), and ultraviolet spectrophotometry (UV-spectrophotometry) to identify and quantify

volatile organic compounds in the material. Efforts were also made to determine whether the volatile organic compounds detected were physically or chemically bound within the leveling material.

5.2.1 Thermogravimetric Analysis

Thermogravimetric analysis [19] was used in determining the mass fractions of sand, volatile organic compounds, and epoxy binder in the cured leveling material. The procedure entailed placing 45 mg of the powdered composite material onto a thermobalance and continuously monitoring the mass loss of the material, while heating it at a rate of 20° C per minute from room temperature to 600° C in a nitrogen atmosphere, and from 600° C to 800° C in air.

TG curves for one of the powdered composite samples from Floor 4 are shown in Figure 3. In this figure, TG curves for three replicates from each composite specimen were generated and averaged. In the discussion which follows and in Appendix A, reference is made only to the average curve.

In the presence of air and at temperatures of 800° C and above, all of the organic materials in the powdered leveling material are oxidized and volatilized as carbon dioxide and water, leaving only non-volatile inorganic materials, like sand. Thus, for this particular composite specimen, approximately 93.5% of the leveling material was non-volatile inorganic

materials. (Refer to Table 5 and Appendix A for data on the other samples taken from Floors 2 through 5). At temperatures between 200° C and 800° C, the epoxy binder undergoes thermal degradation causing the epoxy resin to break down into small molecular units by pyrolysis or oxidation which are volatilized along with the high boiling organic compounds, like amines. From the TG curve, the epoxy binder comprised approximately 5.7 % of the total mass of the leveling material (Figure 3). The remaining 0.8 % of the volatile material (100 % - 93.5 % - 5.7 % = 0.8 %) was volatilized between room temperature and 200° C. This would include volatile organic compounds like phenol which might contribute to the indoor air quality complaints in SSMC-1. Of this 0.8 %, between 0.1 and 0.2 % was water².

5.2.2 GC/MS and LC/MS Analyses

The volatile organic compounds emitted between room temperature and 200° C were identified using both GC/MS and LC/MS techniques. From the analyses, it was concluded that the predominant volatile organic compound liberated from the leveling material between room temperature and 200° C was phenol.

²A confirmatory experiment was conducted to verify this analysis. A sample of the leveling compound was heated to 200 °C for 46 h to drive off all the low boiling volatile organic materials and water. The oven-dried specimens were then placed in an open container in the laboratory for 200 h and exposed to 24 °C and 45% relative humidity (conditions which approximate those in SSMC-1). The mass of this specimen increased by approximately 0.12 %, as determined by TG, and this increase was attributed to water absorption.

Table 5: Average mass loss (percent) and Standard Deviation (number in parenthesis) of percent mass loss in three temperature ranges of the TG curves for material taken from Floors 2 through 5.

Floor	Mass Loss (%) (room temp to 200° C)	Mass Loss (%) (200 to 800° C)	Remaining Mass (%) 800° C
2	0.6 (0.1)	5.5 (0.8)	94.0 (0.9)
3	0.9 (0.09)	5.8 (0.6)	93.3 (0.6)
4	0.7 (0.01)	5.1 (0.8)	94.2 (0.8)
5	0.7 (0.01)	5.26 (0.65)	94.1 (0.5)

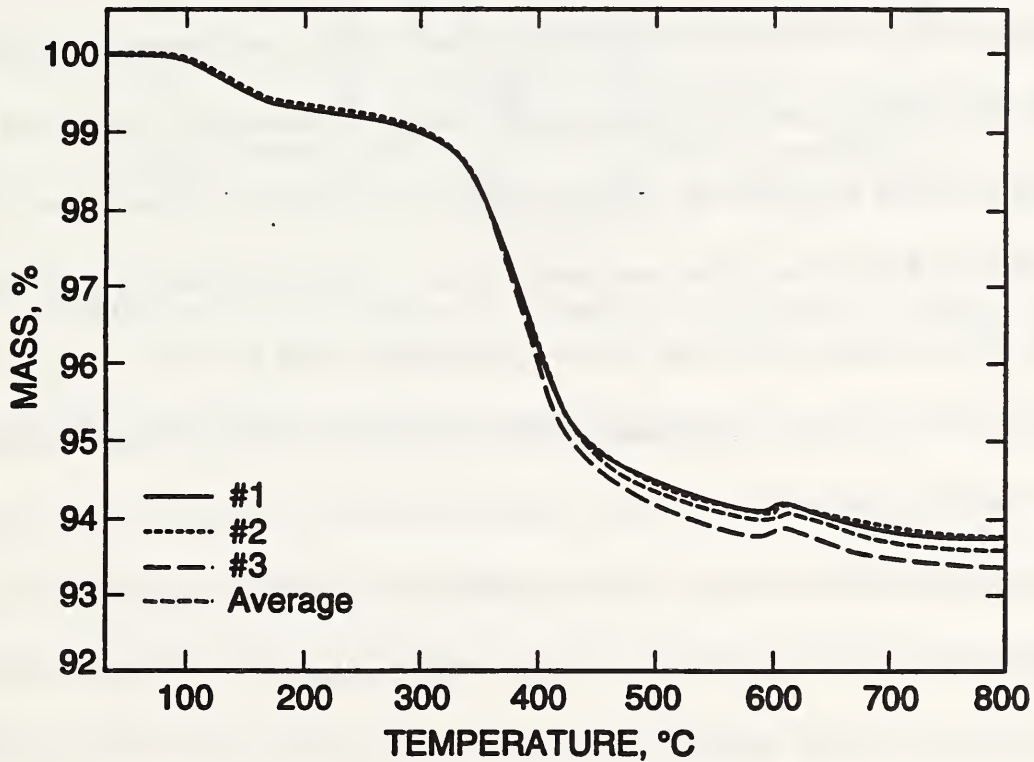


Figure 3. Thermogravimetric curve for one of the fourth floor composite samples heated from room temperature to 800 °C.

In the GC/MS experiment (see Appendix B), a one-gram sample of granulated floor-leveling material was placed in a stream of nitrogen which could be sampled by GC/MS. The volatile organic emissions from the leveling material were monitored at room temperature and 100° C. A GC/MS spectrum taken at 100° C is presented in Figure 4. The GC/MS spectrum at room temperature is similar to the one shown in Figure 4, except that the areas under the peaks are less. From this analysis, it was concluded that **phenol** is emitted from the leveling material along with smaller quantities of **xylene, ethyl benzene, and higher molecular weight hydrocarbons, such as terpenes**. These volatile organic compounds are the same as those reported by Carnow I [5].

The GC/MS was not equipped to detect amines, so a complementary LC/MS experiment was performed which could detect the presence of both amines and phenol (see Appendix C). The sample injected into the LC/MS was obtained from a condensation experiment in which approximately 40 g of the powdered leveling material was heated in a retort in two stages. In the first stage, the bulb of the retort was heated to 111° C for 96 h, while the condensate was collected at the air-cooled end of the retort. Immediately after the 111° C condensate was removed, the bulb of the retort was heated to 200° C for 96 h, while the condensate was again collected at the air cooled end of the retort. Samples from both the first and second stages were analyzed by LC/MS. The dominant volatile organic in the condensate was phenol. In addition, approximately 5 % of the mass of the condensate was triethanolamine. Traces of xylene and diethylenetriamine were also detected.

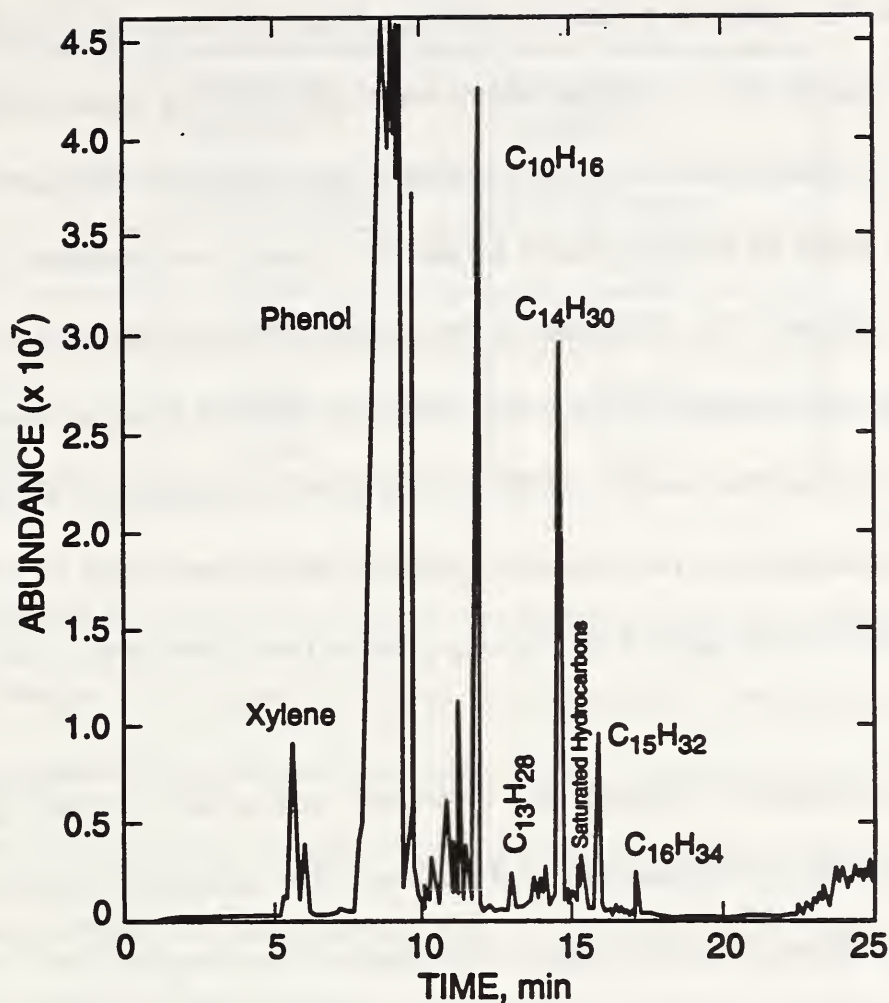


Figure 4. GC/MS spectrum of the epoxy floor-leveling material taken at 100 °C.

5.2.3 Quantification of Phenol in the Leveling Material

The previous two analysis procedures were designed to determine qualitatively the volatile organic emissions from the leveling material. Estimates of the phenol concentration in the leveling material were obtained through Soxhlet extractions.

In the first experiment (see Appendix D), between four and nine grams of the powdered leveling material was placed in a Soxhlet apparatus [16], and extracted for 24 h with water heated to approximately 90° C. The concentration of phenol in the extract was quantified by comparing the UV-absorption of the extract against a calibration curve obtained from the UV-absorptions of a series of aqueous phenol solutions of known concentration. In the second experiment (see Appendix C), 5.7 grams of the powdered leveling material was placed in a Soxhlet apparatus, and extracted for 24 h with methylene chloride heated to approximately 25° C. The results from the Soxhlet extractions with water are presented in Table 6. Note that the concentrations of phenol in the composite specimens taken from Floors 2 through 5 ranged from 0.25 to 0.52 % of the mass of the leveling material (sand plus epoxy binder).

A one-way analysis of variance was performed, at the 0.05 level of significance, to determine if the phenol concentration differed from one floor to another; it was concluded that at least two of the floors differed in phenol concentration from the other two. In addition, a Duncan's multiple range test was performed at the 0.05 level. From this, it was concluded that the phenol concentration on Floor 4 was higher than on Floors 2 and 5. The phenol concentration on Floor 3 was significantly higher than on Floor 2, but not significantly different from the phenol concentrations on Floors 4 and 5.

Table 6. Phenol concentrations in the two composite samples from Floors 2 through 5 as determined by Soxhlet extraction with water.

Sample	Composite Sample #1 (% total mass)	Composite Sample #2 (% total mass)
Floor 2	0.25	0.32
Floor 3	0.52	0.43
Floor 3	0.51	0.51
Floor 5	0.39	0.36

An important question was whether the phenol was able to escape from the leveling material or whether it was strongly bound in the epoxy matrix material and not able to escape. The low temperatures of the Soxhlet extractions strongly suggest that most or all of the phenol was "loosely-held" and, given enough time, the loosely-held phenol molecules would diffuse out of the leveling material and into the atmosphere. This suggestion was reinforced by the results from another experiment (see Appendix C) in which the emissions from the granulated leveling material were measured by GC/MS immediately after the material was removed from its container and again measured after purging the same specimen with nitrogen for one hour. Over this one hour period, the concentration of phenol in the gas stream decreased by a factor of five indicating that most of the phenol readily diffused out of the leveling material. It was concluded from these experiments that most the phenol molecules are "loosely held" within the matrix of the leveling material.

5.3 Test for Permeation of Phenol into the Concrete Floor Slabs

Concern was expressed by NOAA and GSA that, since the leveling material was in intimate contact with the concrete floor slabs, the concrete might be contaminated with phenol, and be a source of phenol emissions if the leveling material was removed. To test this possibility, cores of the concrete beneath the leveling material were taken from Floor 4. The leveling material was separated from the concrete (in several cases the leveling material was not well bonded to the structural concrete) and a faint odor of phenol was detected on the top surface of the core, but nowhere else. Starting from the top of the core, three 4 mm thick disks were cut from the concrete cores using a diamond saw. The wafers were crushed into coarse particles for analysis by GC/MS. The GC/MS results showed no detectable amounts of phenol in any of the cores (see Appendix B). This was not unexpected since portland cement concrete contains calcium hydroxide, a strong base, which combines with phenol, a weak acid, to form a salt, calcium phenolate [12].

6. POSSIBLE REMEDIAL STRATEGIES

From the analyses, it was concluded that significant concentrations of phenol were present in the leveling material on Floors 2 through 5, and that phenol was emitted, and continues to be emitted, into the atmosphere from the leveling material. Four remedial strategies were investigated for mitigating or eliminating these emissions including 1) building bake-out, 2) using activated carbon filters to absorb phenol from the air, 3) sealing or encapsulating the

leveling material, and 4) removing the epoxy material and replacing it with an inorganic floor-leveling material.

6.1 Bake Out of the Leveling Material

The first strategy considered for reducing phenol emissions from the leveling material was to bake out Floors 2 through 5 [4]. A floor would be baked out by substantially increasing the air temperature on the floor for an extended time while the floor was unoccupied and either furnished or unfurnished. A variation of this strategy would be to bake out the leveling material locally by first removing the furnishing and carpets and then directly heating the leveling material to a high temperature (100 to 200° C) using a thermal radiation source. These strategies were abandoned as being impractical once the TG and GC/MS results were available, because the results indicated that an impractically high temperature (greater than 200° C) would have to be applied for a very long time to remove the phenol.

6.2 Activated Carbon Filters to Absorb Phenol from the Air

The second strategy considered was the use of absorbents to remove phenol from the air. Activated carbon filters are known to be effective absorbers of phenol in the air [7,9] and such filters have been installed in the HVAC system of SSMC-1 in recent renovations. Experience with the carbon filters has shown that they are capable of greatly improving the air quality on Floors 2 through 5. On Floors 3 and 5, the filters reduced the concentrations of phenol in the

air to below phenol's odor threshold (approximately 0.05 ppm); while on Floor 4, the concentration of phenol remained above this threshold. Thus, activated carbon filters of suitable size, either alone or coupled with another remedial strategy (like encapsulation or sealing of the leveling material), might mitigate the concentration of phenol in the air, although these filters would have to be periodically changed over the life of the building.

6.3 Sealing or Encapsulation of the Leveling Material

It is well-known that some organic coatings and plastic sheets can act as barriers to the movement of vapors of volatile organic compounds (see, for example, Crissman and Schen [2], Kanare [8], Salame [15], and Thorp [17]). It might be possible, therefore, to seal or encapsulate the leveling material by painting it, by covering it with a plastic sheet like polyethylene, or by a combination of both. If necessary, the effectiveness of this strategy could be enhanced by coupling it with activated carbon filters.

This strategy, however, was judged **unlikely** to be cost-effective for several reasons. First, sealing or encapsulating the leveling material would cause phenol to be retained within the leveling material. Since the service lives of the sealing materials and plastic sheets would likely be much shorter than the life of the building, the air on Floors 2 through 5 would have to be periodically monitored for phenol over the life of the building to determine if and when resealing had become necessary. Moreover, since the life of the building is likely to be fifty years or more, there is a possibility that there would be a loss of institutional memory of the phenol

emission problem. For example, it is possible that NOAA or other future occupants of the building would rediscover an indoor air quality problem involving phenol.

6.4 Removal and Replacement of the Leveling Material

The most certain, and probably the most cost-effective, long-term solution considered is to physically remove the leveling material and replace it with a light-weight portland cement-based product. This remedial strategy would eliminate the major source of phenol emissions. Replacement of the leveling material with a portland cement-based floor-leveling material would have the added advantage that any residual phenol left on the surface of the concrete slab coming in contact with the portland cement-based material would be converted into a salt, calcium phenolate. This should effectively prevent any significant quantity of residual phenol from being volatilized into the atmosphere (see Section 5.3).

7. SUMMARY AND CONCLUSIONS

GSA and NOAA requested that the National Institute of Standards and Technology (NIST) investigate whether phenol, or any other volatile organic compound, was being emitted by the epoxy floor-leveling material and, if so, to recommend remedial strategies for eliminating or mitigating the emissions.

NIST designed and executed a random sampling plan for obtaining samples of the epoxy

floor-leveling material on Floors 2 through 5 of SSMC-1. The chemical composition and physical properties of this material were studied using a variety of analytical procedures including thermogravimetric analysis, gas chromatography/mass spectrometry (GC/MS), liquid chromatography/mass spectrometry (LC/MS), and ultraviolet spectrophotometry (UV-spectrophotometry). It was concluded from these analyses that

1. The epoxy floor-leveling material on Floors 2 through 5 contained significant concentrations of phenol and that phenol was emitted from the floor-leveling material at room temperature; also, phenol was the major organic emission from the material at room temperature.
2. The concentrations of phenol in the samples of epoxy floor-leveling material ranged from 0.25 to 0.52 % of the total mass of the leveling material.
3. The concrete floor slabs beneath the epoxy floor-leveling material are not contaminated by phenol.
4. The most certain, and probably the most cost-effective, long-term remedial strategy would be to physically remove the epoxy floor-leveling material and replace it with a light-weight portland cement-based product which contained no volatile organic compounds and would act as a scavenger for any traces of phenol left on the floor slab.

8. ACKNOWLEDGEMENTS

The authors were fortunate to be able to be able to access the talents of many individuals from a number of federal agencies in resolving this problem. In particular, the authors would like to acknowledge Mr. Douglas Benton and Mr. Gary Wells (GSA) for their continuing help and support throughout this project; Mrs. Jeannie Garvin (NOAA), who provided invaluable assistance on a multitude of matters concerning SSMC-1; Dr. Geoffrey Frohnsdorff (NIST), who originally set up the research team and provided guidance throughout; Dr. Barry J. Bauer (NIST), who provided insight into the formulation and chemistry of epoxy resins; Dr. Nicholas Carino (NIST), who provided insight into structural problems; Mr. Richard G. Christensen (NIST), who performed the LC/MS analysis; Dr. James A. Clifton (NIST), who provided assistance on issues related to the replacement of the epoxy leveling material with a inorganic material; Dr. Andy Persily (NIST) and Mr. Steve Emmerich (NIST), who helped interpret the indoor air quality survey results; and Mr. Frank Davis (NIST) and Mr. Jim Seiler (NIST), who spend several evenings and weekends collecting samples of the leveling compound.

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APPENDIX A: THERMOGRAVIMETRIC ANALYSIS

Thermogravimetric measurements were made on eight composite samples (two from each floor) which were prepared from the core samples obtained from Floors 2 through 5 (see Figure A1). The procedure for making these measurements entailed placing 45 mg of a powdered composite sample onto a thermobalance and continuously monitoring its mass loss, while heating it at a rate of 20° C per minute from room temperature to 600° C in a nitrogen atmosphere, and from 600° C to 800° C in air. In determining the composition of the leveling material and mass fraction of material which might contribute to volatile organic emissions, three regions of the TG curves were noted as follows: 1) room temperature to 200° C, 2) 200° C to 800° C, and 3) above 800° C.


In the presence of air and at temperatures of 800° C and above, all organic materials in the powdered leveling material are oxidized and volatilized off as carbon dioxide and water, leaving only non-volatile inorganic materials, like sand. The average percent of the mass of leveling material which consisted of inorganic material for Floors 2 through 5 was 93.9%. A one-way analysis of variance was performed at the 0.05 level of significance to determine if the mass fractions differed from one floor to another; it was concluded from this analysis that the mass fraction of inorganic materials were not significantly different. A box-and-whisker plot for the non-volatile materials from Floors 2 through 5 is shown in Figure A2. Each box encloses the range of data. The line in the center of the box indicates the average of the two values. (See Table A1 for the tabulated values).

For temperatures between 200° C and 800° C, the epoxy binder undergoes thermal degradation in which the epoxy resin breaks down into small molecular units by pyrolysis or oxidation; the small molecules are volatilized along with high-boiling organic compounds, like amines. The average percent of the total mass of material which consisted of less-volatile organic materials was 5.4 %. As before, a one-way analysis of variance was performed at a 0.05 level of significance to determine if the mass fractions of the less-volatile organic compounds from the different floors were significantly different; it was concluded that the mass fraction of "stable" organic compounds were not significantly different as shown in Figure A3.

The remaining 0.7 % of the material ($100 \% - 93.9 \% - 5.4 \% = 0.7 \%$) was volatilized between room temperature and 200° C. This material would include any volatile organic compounds which might contribute to the indoor air quality problem in SSMC-1. Of this 0.7 %, between 0.1 and 0.2 % was water and the remaining portion might be volatile organic compounds. A one-way analysis of variance was performed at the 0.05 level of significance to determine if the mass fraction differed from one floor to another; it was concluded from this analysis that the mass fraction of volatile organic compounds on Floor 2 was significantly less than on the other floors and that the mass fraction on Floor 3 was significantly greater than the concentration on Floors 4 and 5. A box-and-whisker plot of the concentrations of the volatile materials for Floors 2 through 5 is shown in Figure A4.

Table A1: Average mass loss (percent) and standard deviation (number in parenthesis) in three temperature regions of the thermogravimetric curves for Floors 2 through 5.

Floor	Mass Loss (%) (room temp to 200° C)	Mass Loss (%) (200 to 800° C)	Remaining Mass (%) 800° C
2	0.6 (0.1)	5.5 (0.8)	94.0 (0.9)
3	0.9 (0.09)	5.8 (0.6)	93.3 (0.6)
4	0.7 (0.01)	5.1 (0.8)	94.2 (0.8)
5	0.7 (0.01)	5.2 (0.5)	94.1 (0.5)


 Tinh Nguyen
 11/30/93


 Eric Byrd

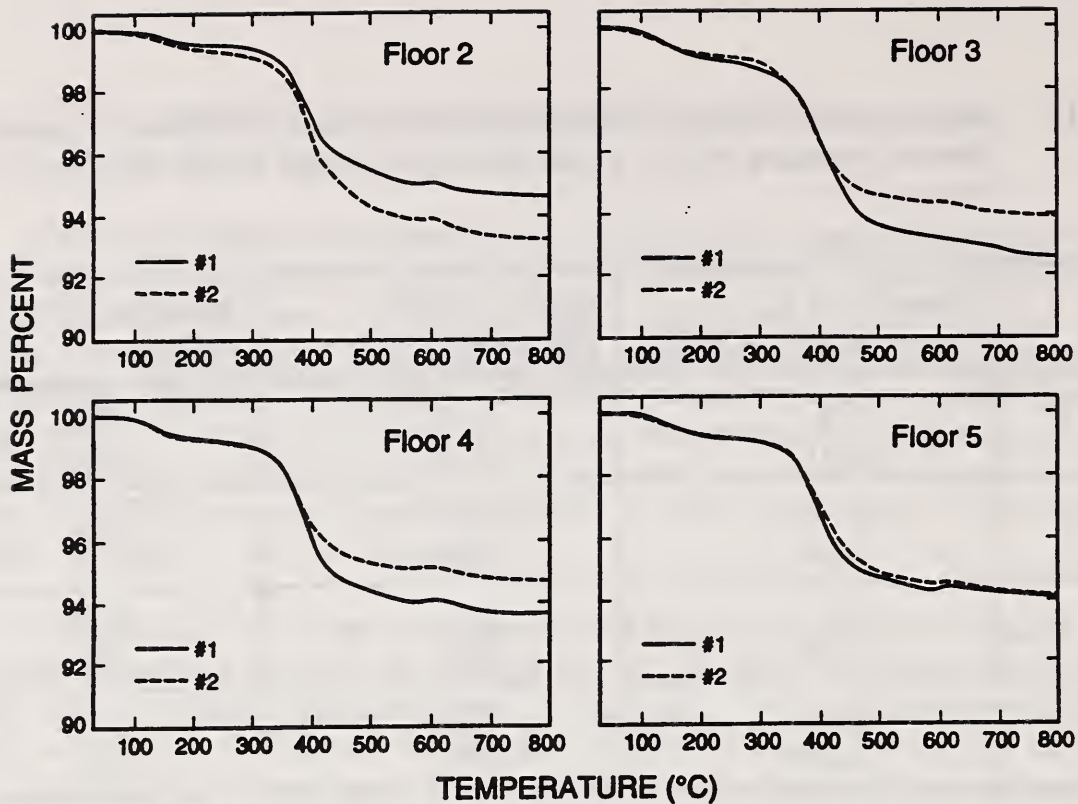


Figure A1. Thermogravimetric curves heated to 800 °C for composite samples made from core samples taken from Floors 2 through 5.

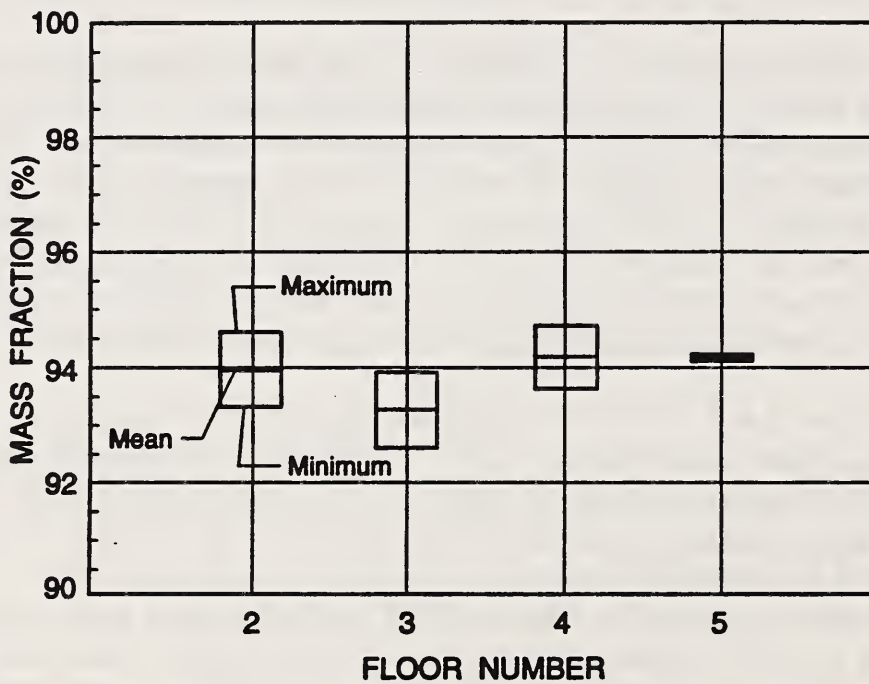


Figure A2. Box-and-whisker diagram for mass fraction of low volatile organic material volatilizing between 200 and 800 °C from the composite samples made from core samples taken from floors 2 through 5.

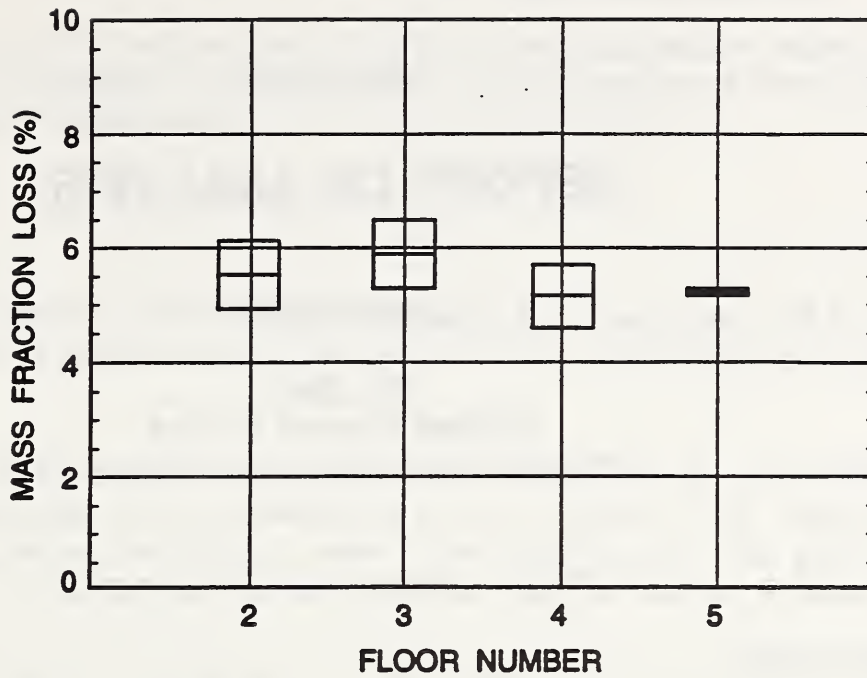


Figure A3. Box-and-whisker diagram for mass fraction of low volatile organic material volatilizing between 200 and 800 °C from composite sample made from cores taken from Floors 2 through 5.

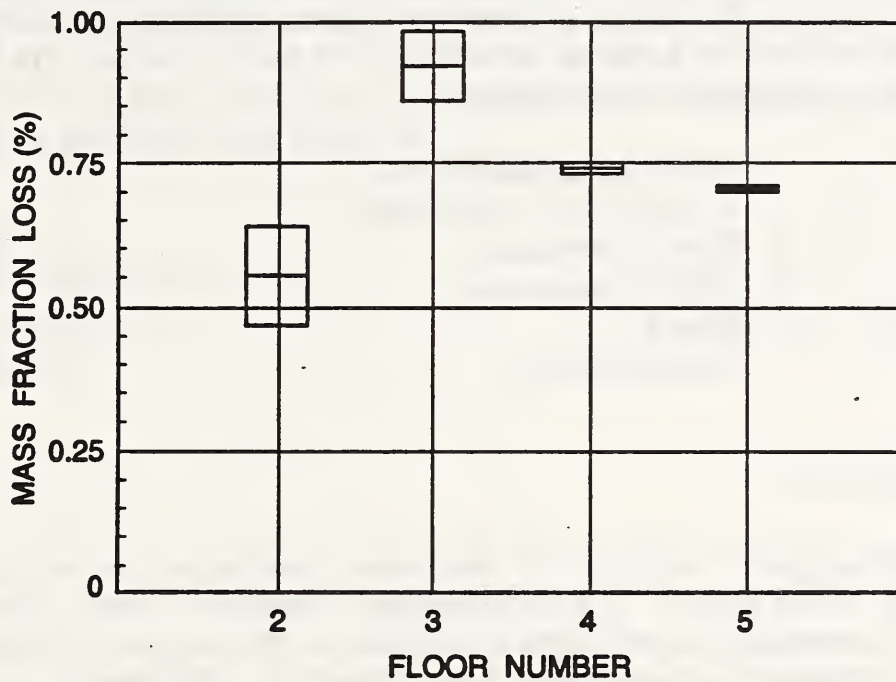


Figure A4. Box-and-whisker diagram for the mass fraction of volatile organic materials volatilizing between room temperature and 200 °C for composite samples made from core samples taken from Floors 2 through 5.

835-93-114

REPORT OF ANALYSIS

Submitted to:

Eric Byrd
Building Materials Division
Building and Fire Research Laboratory

Organic Compounds from Concrete

INTRODUCTION

Two samples were submitted; liquids which had been volatilized from powdered epoxy concrete at 111 °C and 200 °C (they will be identified in this report as S-111 and S-200). S-111 was a pale liquid with an odor of phenol. S-200 also had an odor of phenol, but was darker in color, and appeared to contain a droplet of water in addition to the organic layer (only the organic phase was sampled). It is desired to determine if these samples contain compounds which would indicate that an excess of hardener was added to the epoxy concrete. The MSDS of the epoxy concrete lists components of the hardener as:

Polyoxypropylenediamine
A proprietary "amine adduct"
Diethylenetriamine
Triethylenetetramine
Phenol
Triethanolamine

EXPERIMENTAL

Liquid chromatography (LC) with UV absorbance detection at 254 nm was used for the first investigation of the sample. The chromatographic conditions were: PRP-1 column, gradient from 0.1 M ammonium acetate buffer to methanol in 30 min, flow 1 mL/min. Aliquots of the sample and reference compounds were diluted 50-fold (i.e., 2% solution) in methanol. Injection volume was 10 µL retention times and peak heights in the resulting chromatograms were compared for compound identification and quantification.

Similar LC separation was also used with thermospray mass spectrometric detection (LC/MS) to look for compounds lacking suitable chromophores for UV absorbance detection. Single ion

monitoring of positive ions was used, since the amine compounds are expected to give (M+H)⁺ ions. Diethylenetriamine, triethylenetetramine, and triethanolamine should yield ions at m/z = 104, 147, and 150 respectively.

RESULTS


The LC measurements showed about 50% phenol in S-111, and about 30% in S-200. There may be a small amount of xylene present, but quantification is complicated by the presence of o,m,p-isomers.

The LC/MS measurements show about 5% triethanolamine in each sample. There is a chromatographic peak in the chromatogram of S-111 with m/z = 104, which would correspond to diethylenetriamine, but it is quite small. One would expect a very good ion yield for this compound in thermospray LC/MS, so there is probably not much of it present.

It is believed that the identification of the compounds for which quantitative numbers are given is reliable. They are characterized by chromatographic retention time, as well as by the distinctive odor in the case of phenol and by m/z in the case of triethanolamine.

The accuracy of the results is limited by the nature of the sample and the analyses used. It is the judgment of the analyst that the phenol numbers are probably not in error by more than $\pm 50\%$, and the triethanolamine numbers are probably within a factor of two of the true value.

The original data on which this report is based are in RGC Notebook Vol. 44, pp22ff, and on magnetic media identified in the Notebook.



Richard G. Christensen
Research Chemist



Willie E. May, Ph.D.
Chief

**APPENDIX C: GAS CHROMATOGRAPHY/MASS SPECTROSCOPY
(GC/MS) ANALYSIS**

835-93-122

REPORT OF ANALYSIS

Analysis of NOAA Building Floor Samples

Submitted to:

**Dr. John Martin
Division 862**

Job Number: 3044

Employees located in a building which NOAA occupies in Washington D.C. have been complaining about physical ailments associated with workplace exposure. The NOAA and GSA began investigating these complaints by commissioning several studies of the buildings air quality. In August of this year, NOAA and GSA asked NIST to help in determining the cause of the air quality problems in this building. Prior studies had indicated that a floor leveling material may be producing excessive phenol concentrations.

The Organic Analytical Research Division was asked by Division 862 to help in determining the potential sources of air contamination in the building. Since prior studies had indicated that the floor leveling material was suspect, NIST focused on this material. The objective handed to the Gas Metrology group was to determine what organic compounds would outgass from the material, and to provide some quantitative data on the major compounds.

Preparation and Analysis of Floor Samples for Outgassing Compounds

The samples came in as chunks of material. These pieces were crushed into a finer material and 1 gram were put into a 1/4" stainless steel tube with glass wool plugs at each end. The tube was then placed in a stream of pure dry nitrogen with a flow of 15 mL/minute. The samples flow was cryogenically trapped for 15 seconds and then injected onto the column.

The first set of samples sent for analysis from the NOAA building were of the bulk leveling material and from the floor surface where the leveling material had been applied. The GC/MS data showed that there was significant amounts of phenol (mass 94) in each sample as well as xylenes, ethylbenzene and heavy hydrocarbons such as terpenes.

Samples were then received from each of floors 2 through 5. The samples were analyzed several different times and on different days. Once again phenol was the predominate compound and xylenes, ethylbenzene and heavy hydrocarbons were also present. The phenol was estimated to be 2 orders of magnitude greater in concentration than the other compounds present in the sample. The results further showed that no real difference could be seen from floor to floor as to whether one was more concentrated in phenol than the other.

A sample from the 4th floor was prepared and then placed in an oven with a nitrogen purge through the trap. The sample was baked at 100°C for 4 hours. The trap was removed from the oven and placed in the nitrogen purged sample line and was injected onto the column. The GC/MS results from this analyses showed that the phenol had been virtually baked out of the sample.

Another sample trap containing material from the 5th floor was prepared and placed in the sampling line with a 15 mL/min nitrogen flow at ambient temperature. The sample was cryogenically trapped and analyzed. The GC/MS results showed a large amount of phenol present with an integration of the peak giving a count response of 4,944,000. After purging the sample trap for 1 hour with nitrogen at room temperature, the sample stream was analyzed again. The area integration for the phenol was 871,000, or a decrease in concentration by over a factor of 5. Another sample from the 3rd floor was also prepared and treated in the same manner but with different sampling times. An analysis after a 30 minute purge gave a response of 3,697,000; at the 90 minute mark the response was 1,215,000; and at the 180 minute mark the response was 990,000.

A concrete core sample taken from directly under an area where the leveling compound had been applied was also analyzed. Sample traps were prepared as before. The GC/MS results showed that there was no detectable amounts of phenol present. Likewise, samples injected from a concrete core sample taken from an area not affected by leveling compound showed no detectable levels of phenol.

Quantitative Analysis of Floor Samples for Phenol

In order to obtain quantitative information as to the amount of free phenol in the leveling compound, a portion of sample 4-1 was soxhlett extracted and analyzed by GCMS. The procedure used is as follows:

A finely crushed sample of floor 4 was weighed (5.7142 g) into a glass thimble with a coarse glass frit. The thimble was then placed into a soxhlett extractor and extracted with 150 mL of methylene chloride for 24 hours. The methylene chloride was spiked with 11.63 mg of d_6 -phenol prior to the start of the extraction. After extracting for 24 hours the methylene chloride was removed and analyzed by GC/MS. The ratio of the peak intensity of phenol (m/e 94) to the d_5 -phenol (m/e 99) was measured. This ratio was compared to a gravimetric solution of phenol and d_6 -phenol which was run under identical conditions.

The results of this experiment indicated that there was 0.43 ± 0.02 percent phenol in the floor sample.

In order to confirm these results an analysis of a water extracted sample was analyzed. This sample was prepared and extracted by Division 862, who than delivered to us 103 mL of the extractant. The water sample was acidified and extracted with 30 mL of methylene chloride spiked with 2.326 mg of d_6 -phenol. The methylene chloride extract was than analyzed by GC/MS as stated above. The results showed that there was 0.39 ± 0.05 percent phenol in the floor sample. Since this analysis involved more steps, and an estimate was used of the proportion of water extract that we received to the whole, we can only use this result to confirm the original result. Since both results agree well we can state that the floor sample contains 0.43 ± 0.02 percent phenol.

Conclusions

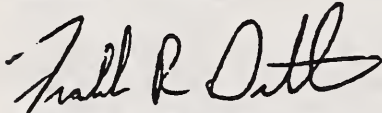
From our analyses we can conclude that all the floor samples contained phenol, and that this phenol did outgas from the floor material over time. We can further conclude that the phenol concentration exceeded all other detectable organic compounds by at least an order of magnitude. We can not conclude whether one floor was more concentrated in phenol over another floor. All the floors tested outgassed phenol under our experimental conditions.

From the extraction experiment we can conclude that the amount of phenol in the floor material is around 0.4%, and from our outgassing experiments, is distributed throughout the cores. The heating and purging experiments indicate that excessive conditions would be required to remove the phenol from the floor material in the building. However, the tests run on the concrete cores indicate that free phenol did not contaminate the concrete flooring.

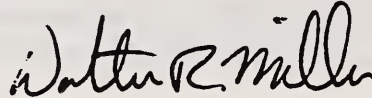
Ref.: Tox. Org. Anal. 10 GR, p. 51-95.



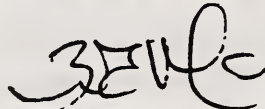
George C. Rhoderick
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Franklin R. Guenther, Ph.D.
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Willie E. May, Ph.D.
Chief

APPENDIX D: ULTRAVIOLET (UV) ABSORPTION

Phenol was extracted from a known mass of epoxy floor-leveling material over a 22 hour period via a Soxhlet extraction [17] using water heated to approximately 90° C as the solvent. The phenol concentration of the extracted solution was quantified by measuring its ultraviolet absorbance (UV-absorbance) at 270 nm and by determining where this absorption value falls on a calibration curve generated from a series of aqueous phenol solutions having known concentrations. A detailed description of this procedure follows.

Two milliliters of the extracted solution having pH 3 was placed in a 10-mm path length liquid cell of a UV spectrophotometer. The spectra were recorded in the 230-350 nm wavelength range using a scanning rate of 5 nm per minute. A typical UV-Vis spectrum of an extracted solution of the floor-leveling material sample is shown in Figure D1. For purposes of comparison, the UV spectra of water and of an aqueous phenol solution containing 100 mg of phenol in 1 liter of water are also shown. The spectrum of water alone is flat in the ultraviolet region between 235 and 295 nm wavelengths, indicating that water does not absorb in this range. The spectrum of aqueous phenol solution shows a characteristic phenol peak at 270 nm and two shoulders near 264 and 276 nm. Note that the spectrum of the epoxy floor-leveling material also contains the characteristic features of phenol.

Translation of the UV absorbance into the concentration of phenol was accomplished by generating a UV-absorbance calibration curve using aqueous phenol solutions of known concentration. The spectra of four different phenol concentrations in water are shown in Figure D2, which shows that the intensity of phenol (peak height or area under the curves) varies with its concentration. Using maximum height of the 270 nm peak to represent phenol intensity, an UV concentration-absorbance calibration curve for phenol was established (Figure D3). As seen in Figure D3, within the concentration range studied, there is a linear relationship between UV-Vis absorbance and phenol concentration in water having a squared correlation coefficient of 0.999.

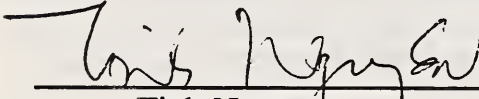
Figure D3 was used for determining the amounts of phenol extracted from the floor-leveling material on Floors 2, 3, 4 and 5. The amount was interpolated from the calibration curve using the absorbance value obtained from the UV-Vis spectrum for each extract. The percentages of phenol in the floor-leveling material for Floors 2 through 4 and the standard deviation between the samples (four of them) within each floor are given in Table 1. The phenol concentrations reported for each floor are the average of two measurements. The percent of phenol was calculated based on the total mass of phenol measured in each extract with respect to the total mass of the ground floor-leveling material (that is, sand plus binder) used in each extraction.

Table D1. Phenol concentrations in the floor-leveling material from Floors 2,3,4 ,and 5

Sample	Composite sample #1 (% total mass)	Composite Sample #2 (% total mass)
Floor 2	0.25	0.32
Floor 3	0.52	0.43
Floor 4	0.51	0.51
Floor 5	0.39	0.36

Table D1 shows the phenol concentrations of the composite samples for Floors 2 through 5. The floor-leveling materials on Floors 3 and 4 contain the highest levels of phenol, Floor 2 the lowest, and Floor 5 had an intermediate value.

The accuracy of the results depends on the identification and the extractibility of the phenol by hot water. The identification and determination of phenol in the extracted solutions is considered to be reliable. The extractibility of phenol by hot water should be high because of the small molecular size of water, which facilitates its diffusion into the sample, and the very high solubility of phenol in hot water. If hot water was not able to remove all of the phenol out of the floor-leveling material, then the results given in Table D1 would be lower than the true amounts.


 Tinh Nguyen
 11/30/93


 Eric Byrd

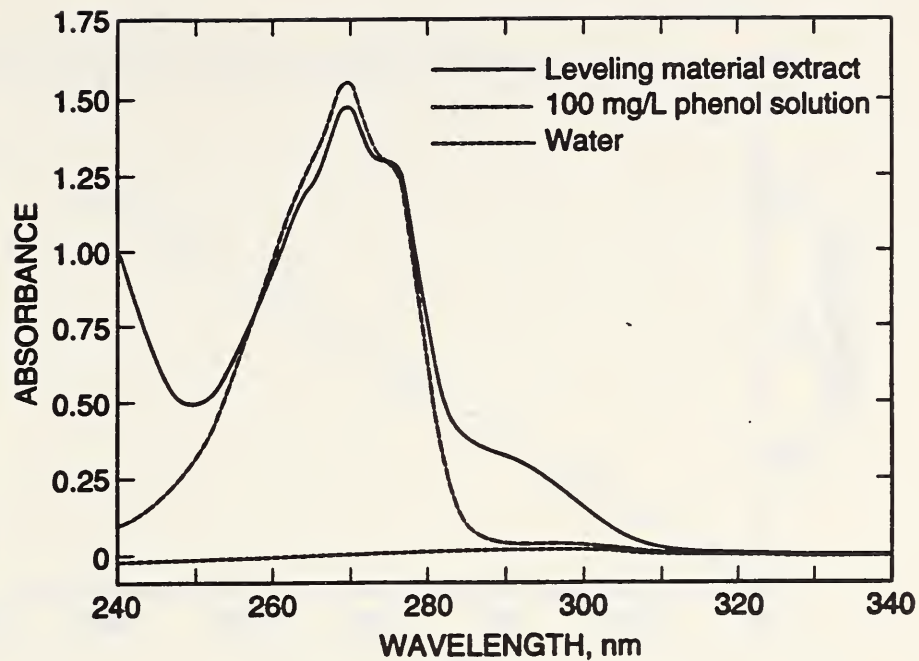


Figure D1. UV-spectra of water, a 100 mg/L aqueous phenol solution, and the Soxhlet water extract from the floor leveling material.

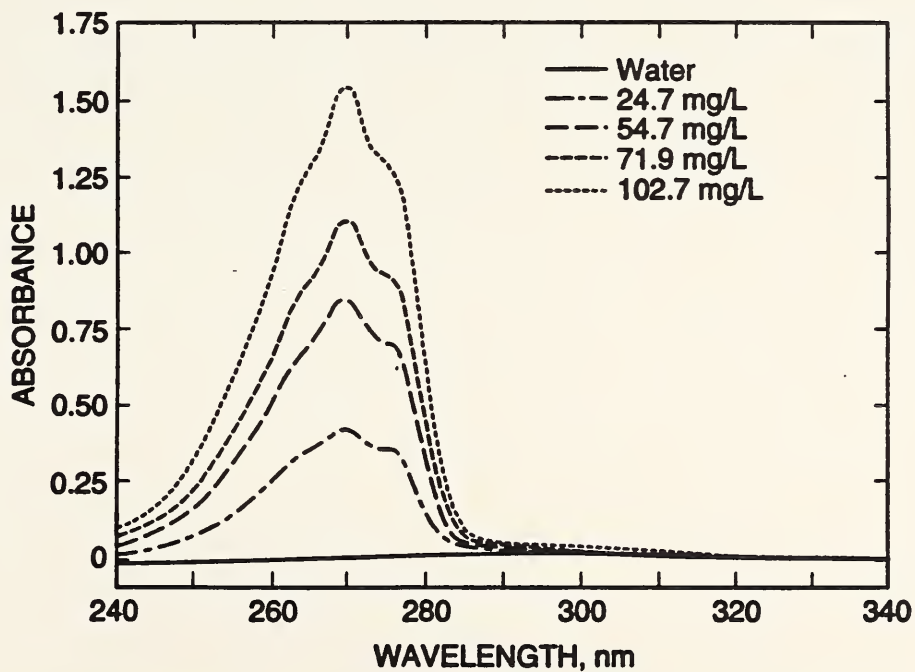


Figure D2. UV-absorbance spectra of phenol at four different concentrations in water.

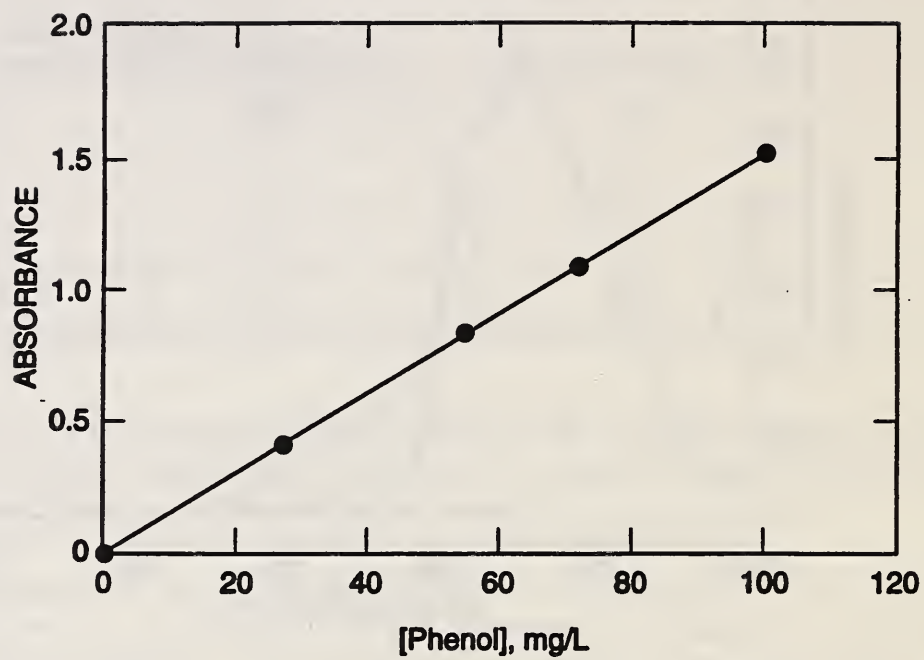


Figure D3. Concentration/UV-absorbance calibration curve for aqueous phenol solutions.

