ENVIRONMENTAL HEALTH SURVEY REPORT

January 23, 1978

PHENOL AREA/ MONITORING AT THE CHLORINATED SOLVENTS PLANT

INTRODUCTION

The collection and determination of phenol in air using silica gel adsorption tubes has been accomplished with much success (R.G. Melcher and J.S. Warren, I.H. Analytical Method File, Method #559 of ML AL 77-50520).

Short term (10 minutes to 1 hour) as well as long term (4 to 8 hours) sampling is easily achieved with these tubes, even in highly humid air. Also, by using a mixed solvent composed of 10% methanol in ether by volume, high desorption efficiencies are obtained with a minimum of tailing problems in the analysis.

This report includes both a recovery efficiency study and the results of air samples collected at various locations within the Solvents Plant. A linearity study of the analytical method was also conducted and found to be within acceptable limits.

EXPERIMENTAL

Standard Preparation:

Stock solutions containing 38 and 19 milligrams of phenol per 100 ml. of solvent were prepared by injecting 35.5 and 17.8 microliters of the liquified compound into 100 ml. volumetric flasks and diluting to volume with a mixed solvent composed of 10% methanol in ether by volume. Working standards of 0.5, 1.5, 3.8 and 9.5 milligrams per 100 ml. solvent were prepared by pipetting appropriate amounts of the 38 milligram stock solution into 100 ml. volumetric flasks and diluting to volume with the mixed solvent. These standards were analyzed to check the linearity of the detector (See Graph 1).

Analytical Parameters:

A Hewlett-Packard,Model 5730 A, gas chromatograph equipped with a flame ionization detector was used for the analyses. Chromatographic conditions are as follow:

Analytical Parameters: (continued)

Column: 10' X 1/8" ss 10% FFAP on 100/120 Mesh Gas chrom W-(AW)

Temperatures: Oven - 200 C Isothermal
Injector - 200 C
Detector - 200 C

Carrier Gas: Nitrogen at 35 ml/minute flow

Range: X 10

Sample Size: 2 microliters

Phenol Elution Time: 4.8 minutes

Recovery Efficiencies:

The phase equilibrium recovery method was used to study recovery efficiencies. It consists of injecting a known amount of a compound into a suitable solvent, adding a known amount of adsorbent, and allowing the mixture to come to equilibrium. The recovery efficiency is determined by dividing the amounts found by the amounts added. These tests were performed on two section tubes containing 260/560 mg. of silica gel (Tek Labs Inc., catalog no. 226-15).

Recovery of phenol was approximately 100% for all concentration levels tested (See Table I). The high results observed in several of the samples may be due to partial solvent loss during desorption.

MONITORING PROCEDURES

Collection of Samples:

SP-1 personal air sampler pumps were used for collecting the samples. The silica gel tube was attached to the pump inlet via a 2 ft. piece of amber latex tubing (3/16" X 1/8"). All pumps were calibrated to operate at 150 ml/minute flow with a soap bubble meter. One, four, and eight hour samples were collected over a period of one week.

Sample Preparation:

Samples were desorbed by dumping the silica gel into 5 ml. of chilled mixed solvent and then shaking periodically for one hour. Analysis was performed by F.I.D. The peak height of the resulting trace was measured and the corresponding weight in milligrams read from a standard curve.

Sample Preparation: (continued)

A volume correction was made since the calibration curve was based on mg/100 ml. of mixed solvent and samples were desorbed with only 5 ml. of mixed solvent.

Corrected mg = C X (mg sample - mg blank)

where:

mg sample = milligrams phenol found in sample
mg blank = milligrams phenol found in blank
C = volume correction factor = 0.05

Calculations:

Concentrations of phenol in the air sample are expressed as follows:

(a) milligrams per cubic meter air

$$\text{mg/cu m air} = \frac{\text{corrected mg X 100 (liter/cu m)}}{\text{air volume sampled (liter)}}$$

(b) Parts per million by volume

$$\text{ppm(v)} = \text{mg/cu m} \times \frac{24.45}{MW} \times \frac{760}{P} \times \frac{T + 273}{298}$$

where:

P = pressure (mm Hg) of air sampled
T = temperature (degrees C) of air sampled
24.45 = molar volume (liter/mole) at 25°C and 760 mm Hg
MW = molecular weight (g/mole) of phenol = 94.11
760 = standard pressure (mm Hg)
298 = standard temperatures (degrees Kelvin)

RESULTS AND DISCUSSION

The ppm phenol and the corresponding mg/cu m value are shown in Table II. Eight hour samples were only collected in the vinylidene process area since no phenol was detected in the majority of the one and four hour samples obtained in other areas of the plant. All samples indicated that the airborne concentrations of phenol at the Chlorinated Solvents Plant are well within the 5 ppm or 19 mg/cu m air exposure limit set by OSHA.

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GRAPH 1 : LINEARITY PLOT

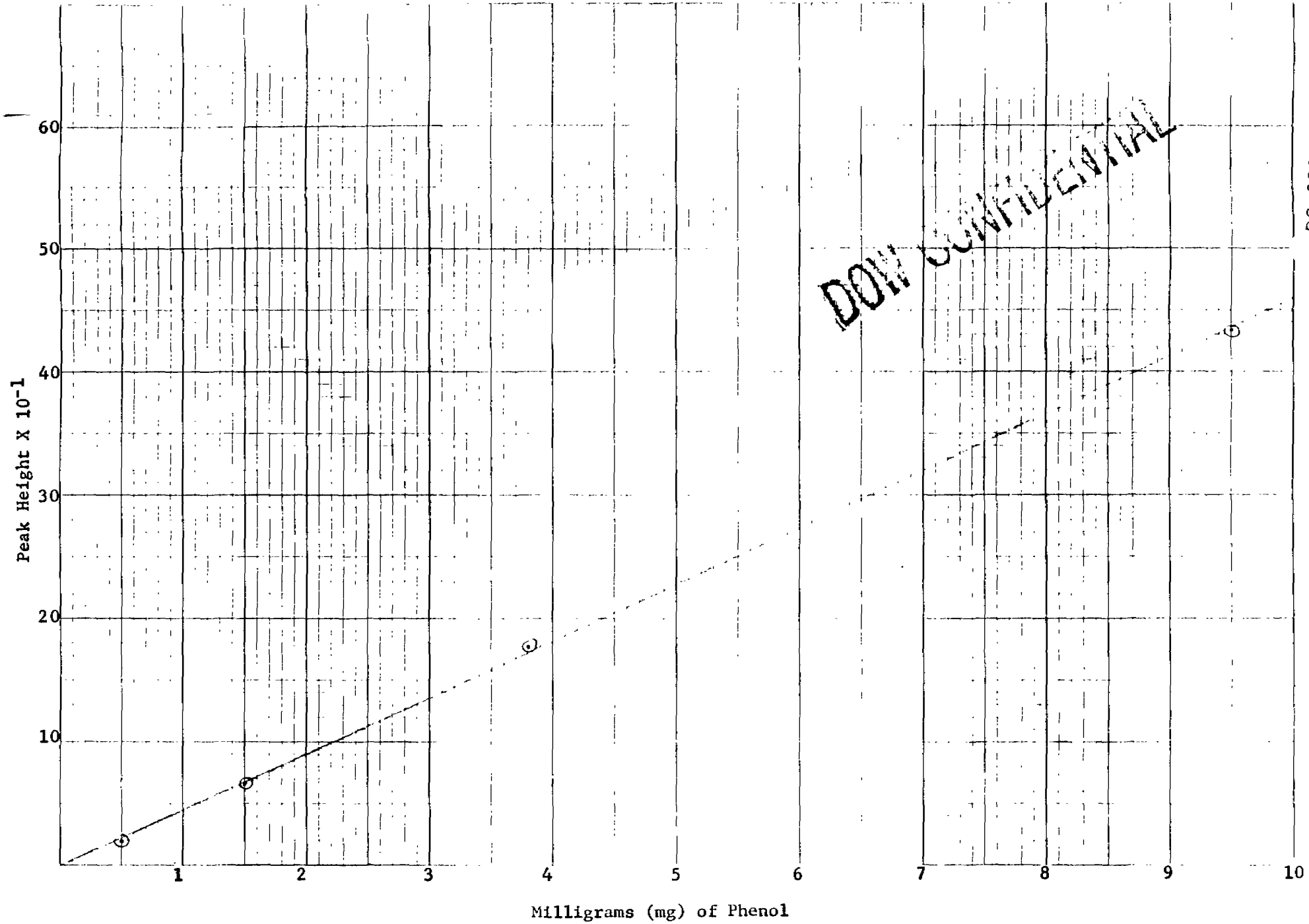

TABLE I

PHENOL PHASE EQUILIBRIUM RECOVERY DATA FOR SILICA GEL TUBES DESORBED WITH DIETHYL ETHER

CONCENTRATION:	2 X TLV	TLV*	1/2 TLV	1/5 TLV
Sample # 1	100	92	102	99
Sample # 2	100	94	101	100
Sample # 3	101	98	100	97
Average	100	95	101	99

* TLV = 19 milligrams

TABLE II

DOW CONFIDENTIAL

PHENOL CONCENTRATION LEVELS AT THE CHLORINATED SOLVENTS PLANT - 1978

SAMPLE #	SAMPLING DATE	SAMPLING TIME (HRS)	MG/CU M	PPM PHENOL	SAMPLE LOCATION:
100	1/13/78	1.0	N/D	N/D	Control room ✓
101	"	"	0.26	0.06	New lab ✓
102	"	"	0.44	0.11	Downwind C-94 Btms pump
103	"	"	N/D	N/D	Old lab
104	"	"	N/D	N/D	Downwind of T-97 & 98 pumps
105	"	"	N/D	N/D	Downwind of R-90
106	1/16/78	4.0	N/D	N/D	Control room ✓
107	"	"	N/D	N/D	Old lab
108	"	"	N/D	N/D	New lab
109	"	"	1.08	0.27	Downwind of C-94 Btms pump
110	"	"	N/D	N/D	Downwind of base of R-90
111	"	"	0.10	0.03	Downwind of T-97 & 98 pumps
112	1/19/78	8.0	0.39	0.09	Downwind of T-97 & 98 pumps
113	"	"	0.12	0.03	Downwind of T-94 pump
114	"	"	0.04	0.01	Downwind of C-94 Btms pump
115	"	"	0.02	0.01	Downwind of base of R-90
116	"	"	0.02	0.01	Downwind of tarpot pumps
117	"	"	trace (0.01)	0.01	Downwind of T-95 pumps

NOTE: Wind out of the North at 10-15 mph on 1/13/78.
Wind out of the South at 10 mph on 1/16/78.
Wind out of Northwest at 10 mph on 1/19/78.

N/D = None detected