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To cite this article: Susan R. Woskie , S. Katharine Hammond , Cynthia J. Hines , Marilyn F. Hallock , Elaina Kenyon & Marc B. Schenker (2000) Personal Fluoride and Solvent Exposures, and Their Determinants, in Semiconductor Manufacturing, Applied Occupational and Environmental Hygiene, 15:4, 354-361, DOI: [10.1080/104732200301476](https://doi.org/10.1080/104732200301476)

To link to this article: <https://doi.org/10.1080/104732200301476>



Published online: 30 Nov 2010.



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Personal Fluoride and Solvent Exposures, and Their Determinants, in Semiconductor Manufacturing

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Personal air sampling for fluorides and solvents was done at 35 semiconductor fabrication facilities in the United States. Fluoride compounds were used in etching and cleaning operations, and solvents were used in photoresist and developing operations. All personal solvent and fluoride levels were less than 2 percent of current Occupational Safety and Health Administration (OSHA) standards. Statistical models of the exposure determinants for the target agents found production level, as indicated by number of semiconductor wafer cassettes loaded/unloaded from the target machines or baths, was predictive of fluoride, xylene and 1-methoxy-2-propyl acetate exposures. The percent of fresh air ventilation and the percent of xylene in the photoresist were also significant determinants in the statistical model predicting personal xylene exposure levels.

Keywords Semiconductor Manufacturing, Chemical Exposures, Glycol Ethers, Fluoride, Solvents, Exposure Assessment, Exposure Determinants

The manufacture of semiconductor devices involves a series of chemical and physical processes performed on wafers of silicon. A given semiconductor manufacturing plant may fabricate a number of different types of devices. Although each type of device requires a different sequence of processing steps, virtually all devices are made using various iterations of six unit processes: oxidation, photolithographic patterning, etching, junction formation (i.e., doping), layer deposition, and metallization.

Each processing step is associated with different potential physical, chemical, and ergonomic exposure hazards.^(1–3)

Chemical exposures in the semiconductor industry are typically reported as less than 10 percent of Threshold Limit Values (TLVs[®]) published by the American Conference of Governmental Industrial Hygienists (ACGIH[®]).⁽⁴⁾ Nevertheless, in the late 1980s, chemical and physical exposures were identified as potential risk factors for an elevated risk of spontaneous abortions in semiconductor workers.⁽⁵⁾

A large, multidisciplinary investigation of reproductive and other health outcomes among semiconductor industry workers was conducted as a follow-up to those initial findings.⁽⁶⁾ This study was part of the exposure assessment component of those epidemiological investigations. As part of the exposure assessment component, chemical agents with the potential to cause adverse reproductive effects were selected.^(7,8) Of these targeted agents, personal air sampling was limited to fluoride, which is found in the acids used for etching and cleaning processes, and to seven organic solvents used for cleaning or in the photoresists and developers of the photolithography process. This article reports the results of personal sampling done at 35 semiconductor wafer fabrication facilities in the United States. Also discussed are the results of statistical modeling to predict a worker's exposure level based on process or facility data collected in conjunction with the personal air samples.

METHODS

Sample Collection

Personal air samples for solvents were collected with two activated charcoal sorbent tubes (100/50 mg) run side by side

at a flow rate of 200 ml/min for at least five hours. Personal air samples for fluoride were collected with a 37 mm closed-face cassette at a flow rate of 3 l/min using a cellulose support pad treated with a solution of sodium carbonate and glycerol (NIOSH Analytical Method 7902).⁽⁹⁾

For each personal sample, information regarding the facility, the chemicals, and the process used was collected by interview and observation (Table I). In addition, an Emission Control Score that accounted for engineering controls or work practices that might modify potential exposures was developed. During sampling, all equipment or machines operated by the worker were evaluated for each emission control indicator (Table I).⁽¹⁰⁾ Each emission control indicator was evaluated using a dichotomous scale: yes or no (present or absent). A summary emission control score for each sampled worker was estimated as the average of the emission control scores (percentage of emission controls in place) for all of the machines used by that worker.

Take, for example, a process with three machines and two emission control indicators evaluated. If two of the machines had controls in place for one factor and none in place for the other, the emission control score for each of those machines would be 50 percent. If the third machine the operator used had no controls in place, the emission control score for that machine would be 0 percent. Therefore, the summary emission control score for that worker is the weighted average of the scores for the three machines: $50\% + 50\% + 0\%/3 = 33\%$. Thus, the summary emission control score is highest for workers whose machines have the most controls (maximum value: 100%) and lowest for workers whose machines have the least controls (minimum value: 0%).

Sample Analysis

Solvent analysis was done by desorbing one charcoal tube for 30 minutes with 1 ml of 5 percent dichloromethane in methanol

TABLE I
Potential exposure determinants

Chemical	Potential exposure determinants
Photoresist and developer solvents: 2-methoxyethanol (ME), 2-ethoxyethyl acetate (EEA) 1-methoxy-2-propyl acetate (MPA) n-butyl acetate (nBA) xylene	Facility factors General ventilation rate Percent fresh air ventilation Process factors Process used (negative or positive photoresist application or negative photoresist developer application) Percent of solvent in photoresist or developer Number of wafer cassettes loaded/unloaded from machines Number of times resist/developer containers were emptied or filled using open pouring Emission control score based on: Automatic (vs. manual) wafer-boat loading Enclosure on spinner bowl Local exhaust on spinner bowl, bakeplate, chemical and waste reservoir Presence of local exhaust gauges No open pouring of chemicals
Cleaning acids: Fluorides	Facility factors General ventilation rate Percent fresh air ventilation Process factors Chemical used (concentrated hydrofluoric acid baths, baths with other HF dilutions, buffered oxide etch, ammonium fluoride, fluoboric acid) Number of wafer cassettes loaded/unloaded from acid baths Number of times acid baths emptied or refilled Emission control score based on: Top shield extending to front of bath Local exhaust ventilation-slot ring design Presence of local exhaust gauges No open pouring into baths

(v/v) for analysis for acetone, n-butyl acetate (nBA), 2-ethoxyethyl acetate (EEA) (also called ethylene glycol monomethyl ether acetate or cellosolve acetate), 1-methoxy-2-propyl acetate (MPA) (also called propylene glycol monomethyl ether acetate), and 2-methoxyethanol (ME) (also called ethylene glycol monomethyl ether or methyl cellosolve); the second charcoal tube was desorbed for 30 minutes with 1 ml of 1 percent sec-butanol in carbon disulfide (v/v) for analysis of isopropanol (IPA), and the xylene. All analyses were performed on a Hewlett-Packard model 5890 gas chromatograph with a model 5970 mass selective detector.⁽¹¹⁾ The injector and detector temperatures were 200°C. A 30 m DB-wax fused silica capillary column was used for all analysis.

For all solvents except acetone, the initial temperature of 50°C was held for five minutes, then increased at 7°C/min to 65°C, and then increased at 16°C/min to a final temperature of 150°C. The temperature program for acetone analysis started at 50°C, which was held for two minutes, then increased at 45°C/min to 200°C. The mass selective detector was used in selected ion mode to enhance sensitivity. The mass/charge peak at 43 atomic mass units (amu) was monitored for acetone, IPA, n-BA, 2-EEA, and MPA, and the peaks at 45 amu were used for 2-ME and xylene. The GC/MS limit of detection for most solvents was 0.05 µg/ml; this corresponds to approximately 0.1 µg/m³ for five-hour samples.⁽¹¹⁾ The limit of detection for acetone was 0.2 µg/ml; however, all samples were above this level.

A slightly modified NIOSH Analytical Method 7902⁽⁹⁾ was used for fluoride analysis. This involved desorbing the filters with an ionic buffer solution by sonication for 15 minutes, then analyzing with a fluoride selective electrode using an Orion ion meter. The limit of detection for the analysis was approximately 1 µg of fluoride per sample; this corresponds to approximately 0.1 µg/m³ for five hour samples.

Data Analysis

Examination of the logged and unlogged data by histograms, probability plots, and measures of skewness and kurtosis all indicated that the data fit the lognormal distribution better than the normal distribution. Therefore, all analyses were done using the natural logs of the air concentration to estimate the geometric mean and geometric standard deviation. For statistical analysis, one-half the limit of detection mass was used for those samples below the limit of detection of the analytical method.⁽¹²⁾

In semiconductor wafer fabrication facilities, a large fraction of the air is recirculated through a common plenum and very high-efficiency filters to remove particles. Therefore, general background or indirect exposure may result from air recirculation in the clean room. Direct exposure occurs when a worker operates a machine using a particular target chemical. However, the target solvents are generally not restricted to a single process; they may be found in several processes which may or may not be used by a particular machine operator. For instance, in this study, EEA and MPA were only used in positive photoresist processes, yet not all positive photoresists contain these solvents. On the other hand, nBA is found in some positive photoresists and some

negative developers. Xylene is a very common solvent, used in both negative and positive photoresists, as well as negative developers and other photolithography process chemicals.⁽¹⁰⁾

Therefore, to examine the potential determinants of any particular chemical exposure, subsets of the data were constructed. These subsets consisted of relatively homogeneous exposure groupings of workers whose jobs included direct, active use of the target agents on the day they were sampled. Homogeneous subsets of EEA (n = 16), MPA (n = 11), and nBA (n = 13) samples came from workers who used the target solvent only in a positive photoresist process on the sampling day. The subset of xylene exposures (n = 27) was limited to samples collected from workers who used xylene in either negative or positive photoresist, without concurrent use of xylene in another process.

The subset of samples from workers who used ME on the sample day only had four observations, so no further analysis was done on these data. Acetone and isopropanol were not used in these analyses because they are used as cleaning agents, not as process chemicals, so no auxiliary data on potential exposure determinants were collected for them. The fluoride data used for this analysis were restricted to samples collected from workers who used fluoride containing chemicals on the sampling day and whose exposures were over the limit of detection for the analysis method (n = 30).

A Pearson correlation matrix of all the potential exposure determinants was used to prioritize the regression modeling and evaluate potential collinearity. For the small subsets of data (n = 11–16), the non-parametric Spearman correlation coefficients were also calculated to evaluate the influence of extreme values on these small data sets.

Regression models were developed stepwise by addition of the most significant factors until the parameters lost significance and/or the model did not show an improved fit. The form of the model used in these analyses was:

$$\begin{aligned} \text{Ln}[\text{air conc}] = & B_0 + B_1 (\text{Exposure Determinant}_1) \\ & + B_2 (\text{Exposure Determinant}_2) \end{aligned}$$

where Ln[air conc] = log of the air concentration, B₀ = intercept, B₁ and B₂ are the parameter estimates for the increase (or decrease) in air concentration predicted to occur with a unit increase in the exposure determinant level. Residual analysis, statistical examination for outliers, and high leverage data values and evaluation of collinearity were conducted on the final models. In the case of the subset of fluoride data, where about half of the data were excluded from modeling because they were below the limit of detection, logistic regression with the whole data set was used to validate the subset findings. Samples were designated as less than or greater than the limit of detection. Then the probability (odds ratio) of being greater than the limit of detection as a function of a unit increase of a specific exposure determinant was calculated. If the exposure determinant remained a significant predictor of exposure level, as indicated by the dichotomous variable less than or greater than the limit of detection, then this supported the linear regression findings on the subset data.

RESULTS

Personal air samples for fluoride were collected on 62 workers. These samples ranged in exposure level from <0.0001 to 0.03 mg/m^3 (Table II). Although all but three of these samples were taken on workers whose job involved direct use of fluoride-containing acids, 31 (50%) of the samples were below the limit of detection (LOD) for the analytical method. The subset of fluoride data used in the examination of exposure determinants included those samples where the operator had direct contact with fluoride and the exposure level was over the limit of detection. These samples ($n = 30$) had a geometric mean (GM) exposure level of 0.0029 mg/m^3 and a geometric standard deviation (GSD) of 3.0.

Personal air samples for solvents showed that, except for the cleaning solvents isopropanol and acetone, there were a substantial number of personal exposures below the limit of detection among the semiconductor fabrication facility workers (Table II). When results were restricted to those where the worker sampled was directly using the target agent (Table III), only the fluoride and ME samples still contained values below the limit of detection (49% for fluoride). ME samples are not reported in Table III because three of the four samples were less than the limit of detection. The subset of xylene data used in the examination of exposure determinants included those samples where the operator used xylene-containing negative or positive photoresist, but had no concurrent use of xylene in another photolithography process. These 27 xylene samples had a GM exposure level of 131 ug/m^3 and a GSD of 4.1.

All levels measured were less than 2 percent of the lowest exposure limit recommended by OSHA or ACGIH, except EEA where the maximum measurement was 15 percent of the ACGIH-recommended exposure limit (Table II). Note that these exposure limits are based on an eight-hour average and most of the samples in this study were less than eight hours. However, in most cases conditions did not change across the workday so

TABLE III

Personal exposures of workers with direct use of target chemicals

Agent	Concentration (mg/m^3)		
	N ^A	GM ^A	GSD ^A
Fluoride	59	0.001	3.3
2-ethoxyethyl acetate (EEA)	16	0.124	4.4
n-butyl acetate	13	0.078	2.2
1-methoxy-2-propyl acetate (MPA)	13	0.047	2.3
Xylene	34	0.153	4.6

^AN = number; GM = geometric mean; GSD = geometric standard deviation.

that these results are representative of the daily average exposure level.

Exposure Determinants

Analysis was done on the subset of fluoride samples where the worker used fluoride-containing chemicals on the day of sampling and the exposures were over the limit of detection ($n = 30$). In preliminary examinations of exposure level, the highest and only statistically significant Pearson correlation coefficient was with the production rate, as indicated by the number of semiconductor wafer cassettes loaded/unloaded from the acid baths by the worker on the sample day (Table IV). Stepwise regression produced a single factor model with only production rate (# cassettes loaded/unloaded) as a significant predictor of exposure level ($R^2 = 0.18$, $F = 6.08$, $p = 0.02$) (Figure 1).

This result was, in part, because of collinearity between production rate and the emission control score ($r = 0.42$, $p = 0.02$),

TABLE II

Personal airborne exposures to solvents and fluoride in semiconductor fabrication facilities

Agent	Concentration (mg/m^3)				Occupational exposure limits (mg/m^3)	
	N ^A	Min ^A	Max ^A	%< LOD ^A	OSHA	ACGIH
Fluoride	62	<0.0001	0.03	50	2.5	2.5
2-ethoxyethyl acetate (EEA)	48	<0.001	4.00	40	540	27
n-butyl acetate (nBA)	48	<0.001	0.99	33	710	710
1-methoxy-2-propyl acetate (MPA)	48	<0.001	0.25	48	—	—
2-methoxyethanol (ME)	48	<0.001	0.01	96	80	16
Xylene	57	<0.001	1.99	14	435	435
Acetone	48	0.008	17.30	0	2400	1200
Isopropanol	57	0.025	6.98	0	980	980

^AN = number of samples; Min = minimum concentration; Max = maximum concentration; %<LOD = percentage of samples less than the limit of detection for the method.

TABLE IV

Correlation of facility and process factors with personal air exposures for workers actively using target chemicals

Potential exposure determinant	Pearson correlation coefficients (P value)				
	Log fluoride conc ^A (n = 30)	Log MPA conc (n = 11)	Log EEA conc (n = 16)	Log NBA conc (n = 13)	Log xylene conc ^B (n = 27)
Production rate (# wafer cassettes loaded/unloaded)	0.42 (0.02)	0.64 (0.03)	-0.01 (0.96)	0.05 (0.88)	0.28 (0.16)
Emission control score (%)	0.25 (0.18)	-0.15 (0.66)	0.46 (0.07)	0.02 (0.94)	-0.01 (0.96)
# Containers/baths filled/emptied	0.24 (0.20)	0.11 (0.74)	0.55 (0.03)	0.14 (0.66)	-0.13 (0.50)
Fresh air ventilation (%)	-0.22 (0.24)	0.54 (0.09)	0.04 (0.87)	0.01 (0.96)	-0.33 (0.10)
Ventilation rate (cfm)	0.07 (0.72)	-0.13 (0.70)	-0.33 (0.23)	0.30 (0.32)	0.45 (0.02)
% Target solvent in resist	—	-0.32 (0.33)	-0.35 (0.18)	0.11 (0.70)	0.80 (<0.01)

^ALimited to personal exposures over the limit of detection.^BLimited to active use of a single product with xylene (positive or negative photoresist).

as well as between production rate and the number of baths filled/emptied ($r = 0.58$, $p < 0.01$). Examination of the final model for outliers and points producing undue leverage suggested that the highest exposure value did have a very strong influence on the model.

To further examine the role of production rate in predicting exposure level, all 59 samples from workers using fluorides on the day of sampling were used in a logistic regression model. This larger data set includes 30 samples below the limit of detection. Thus, the model used estimated the probability of a sample

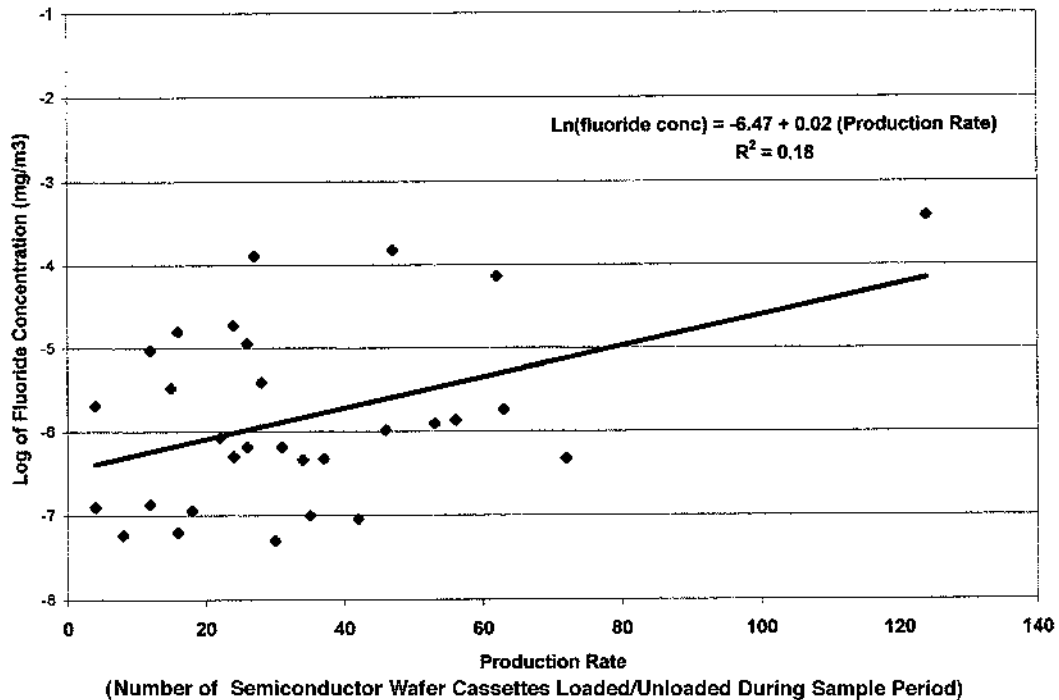


FIGURE 1

The effect of production rate on personal fluoride concentration.

being over the limit of detection as a function of the production rate. The resulting model was significant (chi square 12.57, df 1, $p < 0.01$) and the resulting odds ratio for production rate was 1.07 ($p < 0.01$, 95% CI 1.02–1.11). This model verifies the previous linear model by showing that for each unit increase in the production rate, the probability of a sample result being over the limit of detection increases.

For the subset of MPA samples where the worker had active and direct use of the target solvent on the sample day ($n = 11$), the only variable with a statistically significant Pearson correlation coefficient was production rate, as indicated by the number of cassettes loaded/unloaded from the positive photoresist spinners (Table IV). The non-parametric Spearman correlation coefficients were similar. Stepwise regression produced a single factor model with only production rate (# cassettes loaded/unloaded) as a significant predictor of exposure level ($R^2 = 0.42$ $F = 6.41$ $p = 0.03$) (Figure 2). Residual analysis and influence statistics did not detect any significant problems with this model.

For the subset of exposures where the worker involved had direct, active contact with EEA on the sampling day ($n = 16$), a significant Pearson correlation coefficient was indicated for the number of containers of positive photoresist filled/emptied on the sample day (Table IV). The non-parametric Spearman correlation coefficients were similar. Unfortunately, only three of the sixteen samples had any containers filled or emptied, so this is an unreliable variable for regression analysis. A t-test showed significantly higher levels among those who filled containers

with chemicals containing EEA compared to those who did not, although both groups loaded wafer cassettes into positive photoresist machines using this chemical ($t = -3.45$ $p < 0.01$). No other variables produced statistically significant models predicting personal EEA exposure levels.

For the subset of nBA concentrations where the worker actively used the target solvent ($n = 13$), there were no significant Pearson or Spearman correlation coefficients.

The xylene subset ($n = 27$) focused on those workers who actively used xylene-containing negative or positive photoresists, without concurrent use of any other xylene-containing photolithography chemical. A statistically significant Pearson correlation coefficient was found for the percent of xylene in the photoresist chemical used and with the ventilation rate in the fab (Table IV). However, because ventilation rate evidenced collinearity with the percent xylene in the resist ($r = 0.35$ $p = 0.07$), when a model with both variables was examined, ventilation rate was not a statistically significant parameter. Percent fresh air and ventilation rate were significantly negatively correlated ($r = -0.50$, $p = 0.007$). When percent fresh air ventilation was used in exposure models with percent xylene in the photolithography chemical, both were statistically significant and the model r^2 significantly improved. The final regression model contained the percent of xylene in the photoresist, the percent of fresh air ventilation, and the production rate (number of wafer cassettes loaded/unloaded) as significant predictors of personal xylene exposure level (Table V). Residual analysis and influence statistics did not detect any significant problems with the final model.

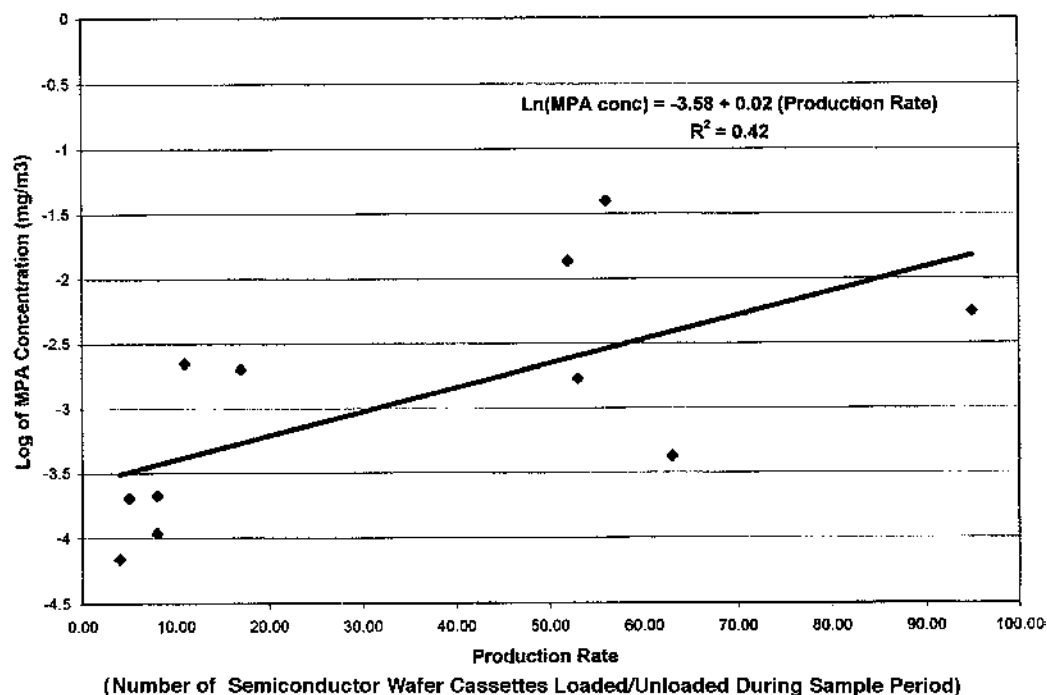


FIGURE 2

The effect of production rate on 1-methoxy-2-propyl acetate (MPA) concentration.

DISCUSSION

All solvent and fluoride concentrations measured were less than 2 percent of the lowest exposure limits recommended by OSHA or ACGIH, except EEA where the maximum measurement was 15 percent of the ACGIH-recommended exposure limit. This was true even when the analysis was restricted to those workers who worked directly with the target agent on the day of sampling. Scarpace et al.⁽⁴⁾ reported that full-shift time-weighted average (TWA) exposures were typically less than 10 percent of the recommended standards, although there was an occasional higher exposure level reported. Corn and Cohen⁽¹³⁾ also reported that the over 18,000 short-term (four-minute) area samples taken in a semiconductor fabrication facility by a real-time mass spectrometer were orders of magnitudes lower than existing standards.

Nevertheless, the risk of spontaneous abortions is elevated among female fab workers exposed to solvents in photoresist operations and to fluorides used in etching.^(14,15) Higher exposures are likely to occur during spills or maintenance tasks,⁽⁷⁾ and although these may contribute to worker risk, they were not reflected in the daily TWA exposure measurements reported here.

Exposure Determinants

The use of statistical models to examine personal exposure determinants in the semiconductor industry was hampered by the number of chemical mixtures in use across the industry and the variety of tasks performed by a single worker. Thus, the subsets of workers with active direct use of any specific target chemical without concurrent exposure to the same chemical in another process were quite small. Nevertheless, production rate, as indicated by the number of wafer cassettes loaded/unloaded into a machine or bath by a worker during the sampling day was a significant determinant of personal exposure levels for workers using solutions which contain MPA, xylene, and fluoride.

In two previous studies, measures of production rate were found to be predictive of solvent exposure concentrations in offset/rotogravure printing operations. In one case ($n = 21$, $r^2 = 0.57$),⁽¹⁶⁾ production rate was indicated by the number of plate

changes. In the other case ($n = 3-10$; $r^2 = 0.03-0.98$)⁽¹⁷⁾ production rate was indicated by the volume of solvent consumed per hour.

The xylene model of exposure determinants also indicated that increases in the percent of fresh air ventilation in the fab significantly decreased xylene exposure levels. Because the general ventilation rate was negatively correlated with percent fresh air, this result could also reflect the faster evaporation of the high vapor pressure xylene in high ventilation rate environments. The final xylene model for exposure determinants also contained a parameter for the percent xylene in the photoresist. This variable may have been significant in this model, in part because xylene containing resists had a much wider range of composition than any of the other target solvents (2-90%).

The emission control score, based on observation of machine-based engineering controls and work practices necessitated by the machine design, was not significant for any of the modeled exposures. This may be because the control scores did not, in general, have as wide a range as the other variables. This is a function of the constrained scale (0-100) as well the relatively high levels of control found on most machines across most of the semiconductor fabrication facilities (low variability).⁽¹⁰⁾

CONCLUSION

Personal air samples taken at 35 U.S. semiconductor fabrication facilities for the solvents found in photoresists and developers, as well as for the fluorides found in acid etching baths, were all quite low in comparison with current exposure limits. Although limited because of small sample sizes, statistical modeling of exposure determinants found production rate, as indicated by the number of semiconductor wafer cassettes loaded/unloaded from the machines or baths, to be a significant predictor of personal solvent and fluoride exposures. For xylene exposures, the fresh air ventilation rate and the percent of xylene in the photoresist also contributed significantly to a model predicting personal xylene exposure levels.

The use of statistical modeling of exposure determinants allows investigators to assess whether changes in production-rates, material composition, local ventilation, or machine-specific

TABLE V
Regression model for xylene exposures in photolithography

Variable	Coefficient	Standard error	P
Intercept	-3.03	0.305	<0.01
% Xylene in resist	0.03	0.004	<0.01
% Fresh air ventilation	-0.01	0.005	<0.01
Production rate (# wafer cassettes loaded/ unloaded during sample period)	0.01	0.003	0.04

Dependent variable: Ln [xylene concentration mg/m³]
N:27 R²:0.77 Model F-Ratio: 25.84 P: 0.0001

controls might lower chemical exposures during the performance of a specific task. The ability to examine the importance of specific environmental and process factors in determining the potential exposures to an agent can provide valuable information for prioritizing control strategies in the workplace.

ACKNOWLEDGMENTS

Support for this work was provided by the Semiconductor Industry Association. The authors gratefully acknowledge the cooperation of the company industrial hygienists, the field work of Ellen Anson and Fred Ramsey, and the laboratory work of Allison Draper, Louis J. Rouleau, and Coyla Woskie.

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