1992 DIFFUSION MONITOR TECHNICAL REPORT

January 14, 1993

EXECUTIVE SUMMARY

Accomplishments of the Diffusion Monitor program in 1992 include validation of the replacement carbon for the Organic Vapor Monitors, resolving formaldehyde manufacturing problems, MPDA and introduction of improved packaging for the formaldehyde monitor to reduce the blank, improving the quality control for the 3510 analysis, MPDA and introduction of the 3530 OVM (3520 with laboratory analysis) and validation of the OVM for a number of compounds.

Kuraray GA Acid Washed carbon was chosen as the replacement for the Witco 964 carbon which is no longer available. Less than a one year supply of the Witco carbon remains. A four to five year supply of the new Kuraray carbon was purchased. The carbon was ground and sieved locally (Particle Technology, Inc., Burnsville, MN). Two full-scale batches were milled successfully (December,1991 and December, 1992) A third batch is scheduled for January, 1993. Introduction of the new carbon is tentatively planned for September, 1993.

The Organic Vapor Monitor was revalidated for methylene chloride to provide additional documentation as the new OSHA standard is introduced. Many experiments were run to validate the use of our monitors in response to customer inquiries. This work will continue as an integral part of our Q90s effort.

The high and erratic formaldehyde blanks which have plagued us for many years were determined to come from outgassing of the enamel can lining at elevated temperatures. An MPDA was prepared to provide a temporary cure by first sealing the monitor in a nylon bag and also enclosing a scavenger strip in the can. Significant progress has been made in our search for a foil bag replacement for the can. We anticipate making this change in 1993. Improvements to the formaldehyde quality assurance procedure have led to fewer failing lots of wafers and better limits of detection.

An MPDA was prepared to introduce the 3530 Organic Vapor Monitor (3520 with Analysis) in June. Sales through December were $25,000.

CARBON QUALIFICATION

The Witco 964 petroleum-based carbon used in the Organic Vapor Monitors is no longer commercially available. Monitor Technical Reports for 1989-1990 and 1991 describe the work that resulted in the choice of th Kuraray GA Acid Washed coconut carbon as the replacement carbon. We ordered 1500 kg (3300 lbs.) from Kuraray which was ground and sieved by Particle Technology Incorporated of Burnsville, MN. This resulted in 1350 lbs. of monitor carbon (a 41% yield and approximately a 5 year supply). A full-scale batch of wafers was milled successfully in December, 1991 and again in December, 1992. Extensive testing of the December, 1991 lot (No. 236) was accomplished and results were described in detail in Make-Order Follow-Up Report H3500-52. Recovery and capacity data are shown in the accompanying tables. Testing of the December, 1992 lot (No. 259) is in progress. A third lot is scheduled for January, 1993. After completion of these trial lots and field testing by technical service, an MPDA will be prepared. We anticipate introduction of the new carbon in September, 1993.

The usual quality control tests for OVMs and ETO were run. The ETO wafers passed the recovery tests. The manufacturing QC MEK recovery was 0.919 +/- 0.012 (n=200) compared to a Witco control lot of 0.933 +/- 0.012 (n=35). Our lab evaluation showed a recovery after 1 day of 0.92 dry and 0.86 wet and after 2 weeks a recovery of 0.92 dry and 0.66 wet. Witco carbon after 2 weeks showed 0.91 dry and 0.52 wet. The manufacturing QC CCl_4 capacity was 5.36 +/- 0.21 mg/mil of wafer thickness (n=26) compared to 7.05 and 7.52 for two Witco control lots. We found 5.6 mg/mil for GAAW vs. 7.4 for Witco in our own lab tests. The density was 1.88 +/- 0.42 for GAAW and the caliper 14.3 +/- 0.94 mils vs. typical Witco of 1.61 and 15.5. The Kuraray GAAW carbon passed the wafer contamination test. No extraneous peaks were observed.

A change in the specifications will be necessary for the CCl_4 test and density.

Our lab test for determining capacity is to spike a monitor with an excess of solvent (usually 25 uL to 30 uL), allow it to stand capped overnight, allow it to stand open overnight, desorb with CS_2 and then determine the amount retained. When the capacity was determined in this manner by spiking with 40 mg of CCl_4, we found 41 mg on the GAAW and 32 mg on the Witco wafers. While the Witco wafers appear to pick up more CCl_4 initially in the manufacturing QC test, the GAAW wafers appear to retain more when exposed to air. Similarly, when the capacity for methylene chloride was determined, Witco retained 3.6 mg while GAAW retained 7.0 mg.

A six hour exposure to 50 ppm methylene chloride (2X the proposed OSHA standard) at 80 % RH showed that both Witco and GAAW in double decker monitors collected the same amount (2.1 mg). The Witco monitors showed more on the second section than the GAAW, and were just barely within our requirements for less than 50 % on the second section. The GAAW would allow for a little more leeway in meeting this requirement.

A 5 1/2 hour exposure to 100 ppm methylene chloride under dry conditions (less than 30 % RH) again showed that both Witco and GAAW monitors collected the same amount (4 mg) with approximately 10 % of the methylene chloride found on the second section of the Witco monitors compared to 2 % on the GAAW monitors.

A 7 1/2 hr exposure to 141 ppm of toluene at 50% RH showed that the Kuraray carbon collected within 2% of the Witco (Witco = 6.508 +/- 0.247 vs. Kuraray = 6.373 +/- 0.216 mg). The precision for Witco was 3.8% and for Kuraray was 3.4%.

Recoveries and capacities have been determined for 63 compounds as shown in Tables 1-4. The Kuraray GAAW recovery at the 100 ug level was within 10 % of the Witco recovery for 49 of the compounds, lower for 11 and higher for 3. At the 30 ug spike level some of the cellosolves and epichlorohydrin had significantly lower recoveries with CS_2 for the GAAW carbon. We may have to expand the number of compounds where we recommend alternate solvents for desorption. It will be even more necessary for customers to redetermine their own recoveries than for our previous Witco lot changes, but good industrial hygiene practice requires this anyway.

In summary, the Kuraray GAAW carbon can be used to prepare OVM monitors with the same sampling rates, similar but not identical recoveries, and somewhat improved capacities as the Witco carbon which is no longer available. Recoveries of very low concentrations of some oxygenated compounds are not as good as on the Witco petroleum carbon.

TABLE 1
KURARAY GAAW RECOVERIES - JAN-FEB, 1992
VS.
WITCO RECOVERIES - PREVIOUS WORK
(Spikes in Micrograms - CS_2 Elution)

CMPD.	WITCO SPIKE	WITCO REC	GAAW SPIKE	GAAW REC	GAAW SPIKE	GAAW REC	GAAW SPIKE	GAAW REC
Acetone		0.70A	32	0.77	95	0.84		
Acetonitrile		0.82T	31	0.54	94	0.63	786	0.71
Benzene		1.01A	35	0.96	105	0.95		
n-Butyl acetate		0.84A	35	1.00	106	0.96	882	1.07
n-Butyl alcohol	2430	0.80T	32	0.40	97	0.47		
Butyl cellosolve		0.73T 0.35A	33	0.13	108	0.45		
Butyl cellosolve acetate		0.98T	38	0.69	113	0.90		
Carbon tetrachloride		0.80A	64	0.87	191	1.00		
Cellosolve	280	0.33T	37	0.09	112	0.47		
Cellosolve acetate		1.03P 0.99A	39	0.75	117	0.89	975	0.99
Chloroform		0.89A	60	0.70	179	0.94	1492	1.06
Decane		1.04T	29	1.01	88	1.05		
Dimethyl formamide		0.32T	34	0.02	113	0.14		
Dodecane		1.04T	30	1.10	90	1.09		
Epichlorohydrin	350	0.89T	43	0.38	142	0.45		
Ethyl acetate		0.85A	36	0.82	108	0.98	902	0.91
Ethyl alcohol	2360	0.44T	31	0.23	94	0.31		
Heptane		1.00A	27	0.43	82	0.79	684	1.02
Hexane		1.02A	26	0.92	79	0.94		
Isopropyl alcohol	3930	0.66T 0.45A	31	0.48	94	0.49		
Methyl cellosolve		0.44T	37	0.00	111	0.05		
Methyl cellosolve acetate		1.00P	40	0.63	121	0.81		
Methylene chloride		0.91T	53	0.81	159	0.98		
Nonane			29	1.01	86	1.04		
Octane		1.02T	28	1.04	84	1.05		
Propylene glycol monomethyl ether		0.68T	39	0.00	116	0.23		
Styrene		0.94T 0.88A	36	0.77	109	0.82		
Tetrahydrofuran		1.09T 0.90A	35	0.72	106	0.86		
Trichloroethylene		0.97A	59	0.90	176	0.99		
Toluene		1.00T 1.06A	35	0.99	104	1.00		
1,1,1-Trichloroethane		1.04A	54	0.93	161	1.03		
m-Xylene		1.04T 1.02A	35	1.02	104	0.97		

T = DATA FROM MONITOR TECHNICAL REPORTS 3-91 & 2-92

A = MONITOR ANALYSIS LAB DATA

P = PINK SHEET

CONFIDENTIAL MATERIAL SUBJECT
TO PROTECTIVE ORDER

TABLE 2
KURARAY GAAW vs WITCO RECOVERIES
January-July, 1992
Spikes in Micrograms
CH_2CL_2 Elution

CMPD	SPIKE	KUR REC	WITCO REC	SPIKE	KUR REC	WITCO REC
Amyl alcohol	29	0.81	0.80	97	0.61	0.85
Butyl Cellosolve	33	0.61	0.70	108	0.89	0.91
Cellosolve	33	0.61	0.70	112	0.78	0.85
Diacetone alcohol	34	0.81	0.82	112	0.80	0.96
Dimethylformamide	34	0.07	0.12	113	0.65	0.74
Epichlorohydrin	43	0.66	0.93	142	0.72	0.95
Furfural	41	0.52	0.59	139	0.62	0.71
Furfuryl alcohol	41	0.72	0.75	138	0.71	0.76
Methyl Cellosolve	35	0.68	0.78	118	0.88	0.83
Methyl formate	35	0.32	0.78	117	0.76	0.93
PGME	33	0.75	0.85	111	0.86	0.92
Phenyl ether	39	0.30	0.43	131	0.41	0.52
Propyl nitrate	38	1.03	1.04	127	1.03	1.05

TABLE 3
KURARAY GAAW vs WITCO RECOVERIES
January-July, 1992
Spikes in Micrograms
CS_2 Elution

CMPD	SPIKE	KUR REC	WITCO REC	SPIKE	KUR REC	WITCO REC
Acrylonitrile	29	0.71	0.74	97	0.71	0.79
Allyl alcohol	31	0.31	0.36	102	0.30	0.33
n-Amyl acetate	34	0.80	0.82	114	0.83	0.73
Benzyl chloride	40	0.95	1.00	132	0.96	1.01
Bromochloro-methane	72	0.90	1.01	239	0.99	0.97
Bromoform	104	1.02	1.01	347	1.01	1.04
s-Butyl acetate	31	0.98	0.98	105	0.98	1.00
t-Butyl acetate	31	1.07	1.06	103	1.03	1.05
Butyl Cellosolve	33	0.13	0.34	108	0.45	0.45
Chlorobenzene	40	0.93	0.94	133	0.96	1.01
Cumene	31	0.96	0.98	104	1.01	1.04
Cyclohexanone	32	0.98	1.02	105	1.05	1.04
Cyclohexene	29	1.00	1.01	97	0.99	1.01
1,2-Dichlorobenzene	47	0.80	0.84	157	0.87	0.90
Dimethylformamide	34	0.02	0.05	113	0.14	0.25
Epichlorohydrin	43	0.38	0.82	142	0.45	0.79
Ethyl acrylate	33	0.95	0.96	111	0.93	0.96
Ethyl bromide	53	0.94	0.98	175	0.94	0.98
Ethyl ether	25	1.02	1.10	85	0.96	1.02
Isophorone	33	0.68	0.73	111	0.75	0.75
Mesitylene	31	1.01	1.00	104	1.05	1.08
Mesityl oxide	31	0.80	0.80	103	0.89	0.85
Methylal	31	0.91	0.94	103	0.97	0.92
Methyl t-butyl ether	27	1.21	1.24	91	1.11	1.19
Methylene chloride	2120	0.96	0.97	212	0.89	0.95
Methyl formate	35	0.29	0.90	117	0.29	0.79
Methyl methacrylate	34	1.01	0.99	112	0.98	1.05
Perchloroethylene	58	0.86	0.96	195	1.00	1.07
PGMEA	35	0.90	0.95	116	0.96	0.88
Phenyl ether	39	0.92	0.86	131	0.90	0.93
n-Propyl acetate	32	0.98	0.94	107	1.00	1.01
1,1,2,2-Tetrachloroethane	57	0.69	0.70	190	0.92	0.91

TABLE 4
KURARAY GAAW VS. WITCO CAPACITIES
January- July, 1992
Spikes in Milligrams
CS_2 Elution

Compound	Spike	Witco	Kuraray
Acetone	28	4.0	7.7
Acetonitrile	28	0.1	1.0
Acrylonitrile	28	1.9	5.1
Allyl alcohol	26	4.1	6.1
n-Amyl acetate	28	30	29
Amyl alcohol *	28	27	26
Benzene	26	16	22
Benzyl chloride	28	26	26
Bromochloromethane	30	7.9	18
Bromoform	29	30	31
n-Butyl acetate	26	26	26
s-Butyl acetate	26	26	27
t-Butyl acetate	26	25	25
Butyl cellosolve *	27	27	27
Butyl cellosolve acetate	28	30	30
Carbon tetrachloride	40	32	41
Cellosolve *	28	27	27
Cellosolve acetate	29	31	31
Chlorobenzene	28	27	28
Chloroform	30	12	21
Cyclohexanone	26	21	22
Cyclohexene	28	17	21
Cumene	26	28	27
Decane	26	27	27
Diacetone alcohol *	28	29	28
1,2-Dichlorobenzene	26	25	25
Dimethyl formamide *	30	27	28
Dodecane	26	27	27
Epichlorohydrin *	30	39	46
Ethyl acetate	27	15	20
Ethyl acrylate	28	24	27
Ethyl bromide	29	2.4	6.8
Ethyl ether	28	7.7	12
Furfural *	29	28	27
Furfuryl alcohol *	28	30	29
Heptane	27	28	28
Hexane	26	20	24
Isophorone	28	26	26
Isopropyl alcohol	27	4.8	9.2
Mesitylene	26	27	27
Mesityl oxide	26	24	26
Methylal	26	5.0	10
Methyl t-butyl ether	27	13	19
Methylene chloride	33	3.6	7.0
Methyl ethyl ketone	28	12	18
Methyl cellosolve *	29	24	26

TABLE 4 Cont'd

Compound	Spike	Witco	Kuraray
Methyl cellosolve acetate	25	25	26
Methyl formate	29	0.2	0.5
Methyl methacrylate	28	25	28
Nonane	25	26	27
Octane	25	26	25
Perchloroethylene	33	35	34
PGME *	28	26	27
Phenyl ether	27	26	27
PGMEA	29	30	30
n-Propyl acetate	27	24	25
Styrene	27	26	27
1,1,2,2-Tetrachloroethane	32	33	31
Tetrahydrofuran	27	8.4	15
Trichloroethylene	29	28	29
Toluene	26	25	26
1,1,1-Trichloroethane	27	20	22
m-Xylene	26	27	27

*Desorbed with methylene chloride.

__ determined for a number
nd charts.

is) was introduced in June.

to correct numerous errors
·ate new data.

) SAMPLING RATES

.UENT	SAMPLING RATE (cc/min)
·I2CL2	
H2CL2	
S2	
S2	
S2	
S2	
S2	27.9 +/- 1.3
S2	
:S2	
:S2	
:H2CL2	
:S2	31.5 +/- 1.1
	36.7 +/- 2.5 Witco
	36.6 +/- 0.9 Kur.

mg	SAMP RATE (cc/min)
	40.6 +/- 1.6
	37.3 +/- 1.2
	46.8 +/- 2.4

ınd Abscents with acetonitrile.

VALIDATION

validate the sampling rate for
duct d as follows:

/ = +/- 4.45%)

RH
in (CV = +/-6.8%)
ı rate = -3.2%

n (CV = +/-2.5%) Kuraray GAAW
ıracy = 8.4%

; are summarized in the following

ABLE 6
.ORIDE VALIDATION

IME	EXP.SAMP.RATE	NO.MON'S
	38.9 +/- 3.8	6
	39.3 +/- 1.7	12
	36.9 +/- 1.3	8
	37.1 +/- 2.2	6

rate was 38.1 +/- 2.2 cc/min compared
e calculated from the Hirschfelder
ratory validated sampling rate listed
uides is 37.9 +/- 0.3 cc/min. The
%. The indicated bias in these
'A Accuracy of 12.1 %. The average
' +/-0.056. The average recovery for
l8.

heir polypropylene resin supplier
3. A sample of film from the new
ne any possible effects on sampling
posure studies were conducted. No
rate was observed (see data below).

LE 7

)	Celgard D-209
078 mg	1.810 +/- 0.053 mg
60 ug	10.21 +/- 0.84 ug
68 ug	10.59 +/- 0.49 ug

3M 009063

A sample of 3M isoporous film from John Pournoor made by laser drilling of a uniform pattern of holes in a PET film in hopes that a high density of uniform holes could allow us to achieve a higher sampling rate than is possible with the Celgard. The sample resulted in 1.193 +/-0.077 mg of toluene being collected which was 34% less than the Celgard. Unless a much higher density of holes is possible this material does not appear to present any advantages to the Celgard.

The desire is to find a film which can be mathematically characterized, altered and studied and which when placed adjacent to the charcoal wafer would allow an increased sampling rate monitor to be developed without adverse boundary layer effects.

ETHYLENE OXIDE

In response to a customer inquiry, we compared the recovery after our recommended 1/2 hour elution time with 1, 2, 3 and 4 hours of elution and found no improvement in recovery with longer elution times.

FORMALDEHYDE

Formaldehyde monitors spiked with 8 ug were analyzed and the recovery determined to be 0.94.

Blank analyses performed on Lot 1303 (prepared with care by M. Chouanard in 11-91 and stored at room temperature) found 1.57 ug on 11-15-91 and 1.75 ug on 3-92.

Wafers prepared from Gelman A/E glass fiber had lower blanks than the standard S&S paper when aged at room and elevated (130 F) temperatures but had much lower recoveries in exposure tests. Analysis of the surface pH at CRL showed that the glass fiber pH was >10 vs. near neutral for the S&S paper. Treatment of the glass fiber by soaking in 1N HCl before treatment with bisulfite improved the recovery but then showed much higher blanks on elevated temperature ageing. This led to a number of experiments which conclusively proved that the high and erratic blanks that we have experienced in the last several years is due to outgassing of the interior coating on the can. This coating has been changed once and possibly twice without our knowledge since the can was originally qualified.

Canned monitors aged at 130 F for 5 days had blanks of 8-9 ug while monitors in glass jars or nylon bags remained under 2 ug. Canned acid treated glass fiber monitors also show elevated blanks when aged at 130 F but remain under 2 ug when packaged in glass jars or nylon bags. Canned monitors exposed to 130 F for 24 days increased to 24 ug while monitors packaged in nylon bags before canning remained about 2 ug. These experiments have led us to conclude that the high and erratic blanks which we have historically experienced are coming from outgassing of the cans at elevated temperatures which may occur in storage or shipping. An MPDA was written and implemented in May to provide a temporary fix by placing the monitor in a nylon bag and then sealing the bag in a can. A one square inch bisulfite containing paper wafer is being placed in the can as a scavenger until such time as a replacement for the can is found. Once the can is replaced, we can again pursue a lower blank by changing the wafer material.

Formaldehyde monitors were exposed to 1 ppm formaldehyde in air which had been dried over silica gel desiccant. Monitors without the wet pad showed only 30% of the expected amount. This confirmed the need for the wet second section.

Formaldehyde monitors were validated at 0.2 ppm and at 0.7 ppm. The quality control exposure test (OTM-2094) has been revised to use the 0.7 ppm level to bring the exposure in line with the new PEL. The accuracy and precision can be achieved only when the calibration curve standards are prepared at a comparable level. The QC test method OTM-2095 has been revised to achieve this lower detection limit.

In response to a further reduction in the OSHA standard from 1.0 ppm TWA to 0.75 ppm TWA, we have demonstrated the validity of the 3M monitor over a wide range of concentrations with an average bias of 4.7% and coefficient of variation of 5.3%. The OSHA accuracy equals 15.3% for 102 monitors exposed in batches of 6 to concentrations of 0.24, 0.40, 0.60, 0.61, 1.00, 1.13, 1.90, and 1.94 ppm.

MERCURY

Molding of the polystyrene substrates was moved to Plastech and Koral Labs reported much difficulty in coating them. The mold was repolished to remove blemishes and proper handling and packaging of the substrates by Plastech has improved the quality of the substrates although periodic difficulties are still being encountered.

ANALYSIS LABORATORY

A second GC was obtained and installed last year for OVM analysis to handle overflow and provide backup for the HP5880. Calibration of the new instrument was completed and it is now being used routinely due to erratic response of the HP5880.

The computer program was updated to perform the calculations for 3520/3530 OVMs with backup. The report fits on the existing report form.

The OVM analytical method was rewritten to include analysis of 3520/3530 monitors, analysis of standards with every set of samples, and to reflect the present capillary column parameters. The method is being reproduced for use by technical service to answer customer requests.

Several compounds were added to the analysis list and the Monitor Record Form was revised to include the new compounds as well as the 3530 Monitor Analysis.

The most recent lot of CS2 which we received from Aldrich has a very high benzene content (49 ug/mL) which prevents us from getting acceptable benzene analyses. I have evaluated another grade of CS2 which has a <1 ug/mL spec but which is 4 times the cost and have recommended use of the Aldrich 34,227-0 CS2 when benzene free CS2 is needed.

Tests were run in the pilot plant to determine the limit of quantitation for formaldehyde in an effort to reduce the minimum level which we report. We found that we are able to see 0.5 ug/3 mL sample and we are now reporting at this level rather than the 3.5 ug level used in the past. This will allow customers that submit blank monitors to receive an actual number on their report which they can subtract from the samples to achieve a lower detection limit. In the past almost all monitor blanks would be reported as "less than 3.5 ug". Our new packaging in nylon bags in cans should prevent blanks from reaching this level.

Several sets of quality control samples were submitted to the analysis lab. The most recent set was also sent to 3M Colombia and 3M Venezuela at the request of Paul Olson. No reports have as yet been received from Colombia or Venezuela. I discussed the PAT program statistics with NIOSH. The true value and standard deviation are determined from the mean of the results of 70 labs which have historically had consistently excellent results. An outlier is defined as a result which is greater than +/-3s from the mean. Typical relative standard deviations for organic solvents in the PAT Program are +/- 4-6%. Three standard deviations would then give you a range of 85 to 115 % of the amount expected. I determined the average per cent recovery for the recent set of three spiked samples that I sent to the analysis lab as shown below.

TABLE 8

COMPOUND	AVE %. EXPECTED	PP REC COEFF	R&D REC COEFF
March:			
Isopropanol	104	0.51	0.61-0.66
Toluene	92	1.06	1.04
p-Xylene	97	1.02	1.04
December:			
Toluene	100	1.06	1.04
1,1,1-TCE	108	1.04	1.04
Isopropanol	115	0.51	0.61-0.66

NEW MONITORS

CARBON MONOXIDE

Quantum CO Tests showed that significant exposure of the chip resulted from CO entering the monitor other than through the silica gel entrance slot. Sealing the slot and taping the outside of the monitor body reduced the sampling rate but did not eliminate the CO exposure.

After a meeting with Mark Goldstein and Michelle Oum, Quantum Group, it was decided that no further evaluation would be done by us until they have overcome several sensor chip manufacturing problems and we have developed marketing information.

THERMAL DESORPTION - NEW SORBENTS

I have demonstrated that the UOP Abscents silica molecular sieve material can be used to create a double decker monitor for alcohols. I have successfully desorbed methanol and ethanol from the wafer with acetonitrile solvent. I have determined the sampling rates and recoveries shown below (Table 9). I have also shown that it is possible to thermally desorb methanol from these wafers although much work remains to be done to make thermal desorption a quantitative method. A thermal desorber was obtained on loan from the CRL Industrial Hygiene lab for these initial tests. Pursuing this approach will require investing in an instrument of our own. I have also shown that it is possible to desorb some organics from the charcoal wafer although the efficiency will need to be improved.

I feel that many other compounds will be shown to work successfully with this silica molecular sieve material or similar materials and I think that it would significantly enhance our product line to have a monitor with these characteristics. A significant effort should be allotted for work in this area during the coming year.

TABLE 9

Compound	Recovery	Exp.Sampling Rate	Calc.Sampling Rate
MeOH	0.83	53.7 +/- 2.4 (+/-4.5%)	55.6 cc/min
EtOH	0.85	46.8 +/- 2.4 (+/-5.1%)	44.7 cc/min

In contrast, the EtOH recovery from Witco carbon with CS2 is 0.41 and MeOH doesn't work on carbon at all.

MISCELLANEOUS

EVALUATION OF DOW XPR-1531-D-01116-H3 FIBERS

The Dow fibers were tested for MEK recovery, Carbon Tetrachloride capacity, and Carbon Tetrachloride activity. Results (described below) were compared to carbon-Teflon wafers used in our Organic Vapor Monitors. I do not see any particular advantage to using the Dow fibers in a monitor application at this time, however further testing with dynamic organic vapor challenges may show an application to respirators.

Larry Brey suggested that a mixture of the fibers and chopped BMF be pressed into a disc which could be challenged using our breakthrough test procedures.

Test Data

MEK Recovery:

Approximately 160 mg of fibers was spiked with 97 ug of MEK in CS2. After sitting overnight the fibers were eluted with CS2 and the extract analyzed by GC. The ratio of the amount to the amount spiked is the recovery and was found to be 0.71 +/- 0.03 for 3 samples compared to a typical value for our carbon-Teflon wafers of 0.93.

Carbon Tetrachloride Capacity:

Approximately 160 mg of fibers was spiked with 32 mg of CCl4, allowed to sit overnight, the vial opened and again allowed to stand overnight. The fibers were then with CS2 and the extract analyzed by GC. The amount is considered to be the capacity and was found to be 28.3 +/- 0.9 mg for 3 samples compared to 32 mg for our carbon-Teflon wafers.

Carbon Tetrachloride Activity:

Approximately 300 mg of fibers was placed in an aluminum pan in a desiccator containing a saturated CCl4 atmosphere. The weight gain was determined periodically (30, 60, 135, & 195 minutes). The maximum weight gain for a carbon-Teflon wafer was achieved in 30 minutes (minimum time measured). Previous experiments have shown that the maximum weight is gained in less than 10 minutes. The maximum weight for the Dow fibers was achieved at 135 minutes although 89% was gained in the first 30 minutes. The weight gain divided by the amount of carbon or fiber times 100 is the CTA. The CTA for the carbon in the carbon-Teflon wafer was 74 while that for the fibers was 121.

MANUFACTURING MOVE TO PLASTECH

I participated in the evaluation of a proposal from Plastech, Corp., Rush City, MN to manufacture the monitors except for mercury. The proposal includes making the wafers, assembly and packaging the monitors and performing the quality control tests.

MOLD REDESIGN

I worked with Paul Flannigan to resolve a number of problems with molding parts by redesigning the molds for the elution cap, main body and primary cup. The design changes are complete and fabrication is in process with an anticipated introduction in April, 1993.

PACKAGING

The lining of the can causes high formaldehyde blanks and the cans are becoming more difficult t get. The size used f r formaldehyde is no longer available. A number of foil laminates and polymer films were screened for applicability (Table 10). The screening tests (Tables 11 and 12) included: OVM blank; OVMs exposed to saturated vapors of benzene, methylene chloride, MEK mixture, and carbon tetrachloride; 555 ppm ETO; saturated formaldehyde vapor; formaldehyde blank. Our first choice, a Printpack nylon/foil extrusion laminate, has an unacceptable lead time of 58 weeks. Our next choice, Graphic Packaging Vitek, is now undergoing extensive aging tests.

TABLE 10
ALTERNATIVE PACKAGING MATERIALS

PRINTPACK FOIL - Extrusion Laminated
60 ga. biax Nylon/primer/7# PE/0.0003 Foil/7# PE/1 mil 5% EVA

PRINTPACK METALLIZED POLYESTER - Machine debug for the 5000 respirator
48 ga. met. PET/2 mil PE

TORAYFAN METALLIZED POLYPROPYLENE

KAPAK SILVER FOIL - CADPAK N
60 ga. Nylon/15# PE/0.00033 Foil/0.0022 LDPE

KAPAK WHITE FOIL - With adhesive strip

LPS A - VF-52 PET/PE/Foil/PE to ACLAR/PE/Foil/PE

LPS B - VF-52 PET/PE/Foil/PE to VF-62 Nylon/Saran/PE

TEDLAR - Tursso Group - Polyvinylfluoride

VITEK - Graphic Packaging
92 ga. PET/7.2# Surlyn/0.001 Foil/14.4# Surlyn

SURLYN - 3M Medical Products
1 mil biaxial PP/14.3# LDPE/0.00035 Foil/26# Surlyn #1652

WHITE - Graphic Packaging
48 ga. PET/10# White LDPE/0.00035 Foil/21.6# EAA

CASCADE - Graphic Packaging
48 ga. PET/Ink/10# White LDPE/0.00035 Foil/7.5 EAA/2-1/2 LLDPE

LAMCOR - Cast Nylon/PE/Foil/PE

OMEGA - Graphic Packaging
48 ga. PET/14# LDPE/0.00032 Foil/adhesive/2 mil LLDPE

CAN

NYLON BAG

ELUTION CAP

CLOSURE CAP - No ports

PRIMARY CUP

TABLE 11
ALTERNATIVE PACKAGING TESTS
JUNE-JULY, 1992

PKG	OVM BLANK	OVM BENZENE	OVM CH2CL2	OVM MIX	MEK	OVM CCL4	FORMALDE HYDE
CAN	+	+/-,+/-	+	+			-,+/-,+
NYLON	+	+,+/-	-	-			-,-
ELUTION CAP	+	+/-,-					-
CLOSURE CAP	+	-	-	-			+/-
PRINTPACK FOIL	+	+	+	+		+/-	+
PRINTPACK METALLIZED POLYESTER	+/-	-/+,-				-	+,+
TORAYFAN METALLIZED POLY-PROPYLENE	+	-	-			-	-
KAPAK SILVER FOIL	+/-	-					
KAPAK WHITE FOIL	-	+/-					+,+
LPS A	+/-		+/-	+/-		-	+
LPS B	+		-	-			-
TEDLAR	+/-		+/-	+/-		+/-	-

+ = No permeation or background observed.
+/- = Small amount or variable results observed.
- = Large amount observed.

TABLE 12
ALTERNATIVE PACKAGING TESTS
NOV-DEC, 1992

PKG	OVM BLANK	OVM BENZENE	OVM CH2CL2	ETO	FORM VAPOR	FORM BLANK*
CAN	+	+/-		+	+	1.31
NYLON			-			
ELUTION CAP					-	
PRIMARY CUP		-			-	
PRINTPACK	+	+	+,+	+/-	+	1.50
VITEK	+	+	-,+/-	+/-	+	1.92
SURLYN	+	-	+,+	+/-	+	1.49
WHITE	+	+	+	+	+/-	1.94
CASCADE	-	-			+	1.65
LAMCOR	+	-	+/-,+/-	+	+	1.29
OMEGA	-	-			+/-	

+ = No permeation or background observed.
+/- = Small amount or variable results observed.
- = Large amount observed.
* = Blank after 15 days at 100 F.

Formaldehyde monitors, after 5 days at 100 F, gave >90% recovery for all tested packages - Cascade, Vitek, White, Surlyn, Printpack, Can, Can (Room Temp), and Lamcor.

Formaldehyde monitors, after 5 days at 100 F, showed no difference in blanks between Vitek bags and a nylon bag inside the Vitek bags.