

Respirator Cartridge Breakthrough Testing of Substituted Silanes and Siloxanes

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ABSTRACT

Pairs of North half mask respirator cartridges were tested against commercially available substituted silanes. Tests were run at 32 liters/min, which represents a realistic average breathing rate, 25° C and 50 % relative humidity. Challenge concentrations varied from 0.1 to 1250 ppm depending on potential exposure conditions. The breakthrough of both the parent compound and any hydrolysis byproduct (usually ethanol, methanol, ammonia, acetic acid or hydrochloric acid) was measured. The silanes used as the challenge agents were tetramethoxysilane, methyltrimethoxysilane, vinyltrimethoxysilane, phenyltrimethoxysilane, triethoxysilane, tetraethoxysilane, methyltriethoxysilane, propyltrimethoxysilane, ethyltriethoxysilane, hexamethyldisiloxane, 50 % methyltriacetoxysilane / 50 % ethyltriacetoxysilane, polydimethylsiloxane, methyl tris(ketoximino) silane, trifluoropropyltrisiloxane, trimethylsilanol, hexamethyldisilazane, dimethyldichlorosilane, trimethylchlorosilane, vinyltrichlorosilane, silicon tetrachloride, and trichlorosilane. The cartridges lasted from 134 minutes to 480 minutes or more depending on the chemical. The alcohol hydrolysis byproduct breakthrough time was substantially less than organosilicon compounds and was detected as early as 2 minutes.

Keywords: respirator cartridge breakthrough, respirator cartridge testing, silanes, siloxanes, silazanes

INTRODUCTION

The service life of air purifying respirator cartridges has been a subject of great interest because of the need for accurate personal protective equipment recommendations and potential personnel exposure ramifications. Proper respirator selection and change schedules are a fundamental aspect of worker safety when working with hazardous chemicals. For the past three decades, a great body of information has been accumulated regarding the effectiveness of different types of respirator cartridges in capturing or removing various airborne chemical contaminants (Nelson and Correia, 1976; Nelson and Harder, 1974; Nelson et al., 1976; Moyer, 1983; and Smoot et al., 1977). But with the proliferation of new chemicals and respirator products, data accumulation continues to be an ongoing process. The need for additional experimental respirator breakthrough data is particularly true for the substituted silicon compounds. Although theoretical expressions exist for service life computation, their validity with silicon compounds, especially those that react with moist air, has not been demonstrated (Ackley, 1985; Yoon and Nelson, 1984A; Yoon and Nelson, 1984B; Wood, 1987; Wood, 1992; Wood, 1994; Wood, 2004; Wood, 2005).

The organosilicon compounds under examination include substituted alkoxysilanes, disiloxanes, disilazanes and chlorosilanes. This manuscript will describe tests conducted to measure the chemical breakthrough of 21 substituted silanes when contacting organic vapor, organic vapor/acid or acid gas air-purifying respirator cartridges. In addition, since the alkoxysilanes and chlorosilanes sometimes form alcohols or hydrochloric acid when hydrolyzed, the breakthrough of hydrolysis byproducts through the cartridges was measured as well.

Organosilanes

Organosilanes have been known since 1863 when Friedel and Crafts first synthesized tetraethylsilane from diethylzinc and silicon tetrachloride (Anderson et al., 1991). The first systematic studies of organosilanes were carried out by Frederick Kippling from 1898 to 1939⁽¹⁴⁾. It was not, however, until the 1940's that organosilicon chemistry proliferated following the success of the silicon polymers. The alkoxysilanes, disiloxanes and disilazanes are a class of compounds with the respective general molecular formulas $(\text{RO})_n\text{SiH}_{(4-n)}$ (where n equals 0 to 4), $(\text{R}_n\text{H}_{(3-n)}\text{Si})_2\text{O}$ and $(\text{R}_n\text{H}_{(3-n)}\text{Si})_2\text{NH}$ (where n equals 0 to 3). Some typical R substitutes are hydrogen, methyl, ethyl and phenyl groups. Depending on the type of compound and substituent group, some of these materials may be flammable, corrosive, toxic, irritating and/or water reactive releasing various hydrolysis by-products. For example, silazanes and chlorosilanes are corrosive and release ammonia or hydrochloric acid upon hydrolysis while the methoxysilanes tend to be toxic and release methanol on hydrolysis. In addition, compounds which contain hydrogen directly bonded to silicon (Si-H functionality), can decompose releasing hydrogen gas under certain conditions not relevant to these tests. For those materials that are water reactive, the preferred respirator must be suitable for both the organosilicon compound and its hydrolysis by-product.

MATERIALS AND METHODS

Materials

The Silicones Environmental Health and Safety Council (SEHSC) selected the challenge agents and respirator cartridges tested. The silanes were selected based on the extent of commercial use as well as their potential to represent a hazard to unprotected personnel. These compounds were tetramethoxysilane, methyltrimethoxysilane, vinyltrimethoxysilane, phenyltrimethoxysilane, triethoxysilane, tetraethoxysilane, methyltriethoxysilane, propyltrimethoxysilane, ethyltriethoxysilane, hexamethyldisiloxane, 50 % methyltriacetoxysilane / 50 % ethyltriacetoxysilane, polydimethylsiloxane, methyl tris(ketoximino) silane, trifluoropropyltrisiloxane, trimethylsilanol, hexamethyldisilazane, dimethyldichlorosilane, trimethylchlorosilane, vinyltrichlorosilane, silicon tetrachloride, and trichlorosilane. These chemicals were procured either from the Aldrich Chemical Company or United Chemical Technologies, Inc. The chemical purity was 95 % or greater and they were used as received. These compounds are listed in Tables I and II along with their CAS number.

The North organic vapor, organic vapor/acid gas or acid gas cartridges were chosen because of their common use within member companies of the SEHSC and availability. These cartridges are representative of typical air-purifying respirator cartridges readily available in the market place. Cartridge weights were measured before and after testing.

Table I. Cartridge Service Life Test Results for North OV N7500-1

Run No.	Test Gas	CAS No.	Conc. (PPM)	Initial Weight (Grams)	Final Weight (Grams)	Time to 1 PPM ROH (Min.)	Time to 20 PPM ROH (Min.)	Time to 200 PPM ROH (Min.)
11382	TETRAMETHOXYSilANE	681-84-5	10	78.2 76.9	79.8 78.4	12	61	-
11383	TETRAMETHOXYSilANE	681-84-5	10	77.7 78.6	79.8 80.3	11	57	-
11384	METHYLTRIMETHOXYSilANE	1185-55-3	50	78.5 78.9	81.7 82.0	15	39	-
11385	METHYLTRIMETHOXYSilANE	1185-55-3	50	76.4 76.9	79.5 80.4	15	40	-
11386	VINYLTRIMETHOXYSilANE	2768-02-7	100	77.9 77.1	83.3 82.4	8	25	180
11387	VINYLTRIMETHOXYSilANE	2768-02-7	100	78.0 78.6	83.0 83.9	8	27	188
11388	PHENYLTRIMETHOXYSilANE	2996-92-1	250	78.5 77.1	93.7 93.5	2	31	>480
11389	PHENYLTRIMETHOXYSilANE	2996-92-1	250	76.1 77.3	95.7 96.3	2	28	>480
11390	TRIETHOXYSilANE	998-30-1	100	77.9 78.0	85.9 86.3	78	162	360
11391	TRIETHOXYSilANE	998-30-1	100	76.3 77.2	84.8 85.6	73	153	328
11392	TETRAETHOXYSilANE	78-10-4	100	78.4 77.2	86.2 85.3	174	306	>480
11393	TETRAETHOXYSilANE	78-10-4	100	78.4 76.9	86.4 85.3	176	310	>480
11394	METHYLTRIETHOXYSilANE	2031-67-6	1000	78.1 76.7	95.6 93.8	10	93	137
11396	METHYLTRIETHOXYSilANE	2031-67-6	1000	76.6 77.0	93.8 94.4	10	89	130
11398	PROPYLTRIMETHOXYSilANE	1067-25-0	1250	78.1 76.2	96.8 94.5	3	14	48
11400	PROPYLTRIMETHOXYSilANE	1067-25-0	1250	76.7 78.5	95.2 97.4	3	14	46
13612	ETHYLTRIETHOXYSilANE	78-07-9	500	85.1	103.4	99	156	201

				86.7	105.3			
13613	ETHYLTRIETHOXYSILANE	78-07-9	500	87.4	105.0	86	148	191
				85.3	103.5			
13614	HEXAMETHYLDISILOXANE	107-46-0	50	80.9	83.9	>480	-	-
				83.6	86.4			
13615	HEXAMETHYLDISILOXANE	107-46-0	50	85.3	88.0	>480	-	-
				82.0	85.1			
13616	50 % METHYLTRIACETOXYSILANE/ 50 % ETHYLTRIACETOXYSILANE	4253-34-3 17689-77-9	100	80.7	86.6	>480	-	-
				82.5	88.7			
13617	50 % METHYLTRIACETOXYSILANE/ 50 % ETHYLTRIACETOXYSILANE	4253-34-3 17689-77-9	100	82.5	88.8	>480	-	-
				85.2	91.0			
13618	POLYDIMETHYLSILOXANE	631-62-9	≈ 90	80.5	84.7	>480	-	-
				83.8	88.4			
13619	POLYDIMETHYLSILOXANE	631-62-9	≈ 78	84.4	88.7	>480	-	-
				81.9	85.8			
13620	METHYL TRIS(KETOXIMINO) SILANE	22984-54-9	100	80.7	85.6	>480	-	-
				82.8	88.3			
13621	METHYL TRIS(KETOXIMINO) SILANE	22984-54-9	100	83.4	89.4	>480	-	-
				84.7	91.2			
13622	TRIFLUOROPROPYLTRISILOXANE	2374-14-3	0.10	79.6	80.8	>480	-	-
				82.4	83.9			
13623	TRIFLUOROPROPYLTRISILOXANE	2374-14-3	0.10	82.7	84.2	>480	-	-
				84.0	85.3			
13624	TRIMETHYLSILANOL	1066-40-6	500	79.8	93.1	138	174	>204
				82.4	96.5			
13626	TRIMETHYLSILANOL	1066-40-6	500	83.1	98.0	147	184	>225
				80.9	95.6			
13628	HEXAMETHYLDISILIZANE	999-97-3	250	79.9	92.0	246*	312*	>378*
				82.3	94.8			
13630	HEXAMETHYLDISILIZANE	999-97-3	250	83.4	94.8	211*	279*	>348*
				80.5	92.3			

* Approximately 100 PPM NH₃ breaks through immediately and continues throughout the test.

Test condition: 25° C, 32 LPM and 50 % relative humidity.

Table I. Cartridge Service Life Test Results for North OV N7500-1 (Continued)

Run No.	Test Gas	CAS NO.	Max ROH Conc. (PPM)	Time of Max ROH Conc. (Min.)	Time to 1 % Silane Conc. (Min.)	Time to 10 % Silane Conc. (Min.)
11382	TETRAMETHOXYSilANE	681-84-5	29	480	>480	>480
11383	TETRAMETHOXYSilANE	681-84-5	32	480	>480	>480
11384	METHYLTRIMETHOXYSilANE	1185-55-3	104	480	>480	>480
11385	METHYLTRIMETHOXYSilANE	1185-55-3	115	480	>480	>480
11386	VINYLTRIMETHOXYSilANE	2768-02-7	220	480	>480	>480
11387	VINYLTRIMETHOXYSilANE	2768-02-7	205	480	>480	>480
11388	PHENYLTRIMETHOXYSilANE	2996-92-1	176	480	>480	>480
11389	PHENYLTRIMETHOXYSilANE	2996-92-1	206	480	>480	>480
11390	TRIETHOXYSilANE	998-30-1	260	480	>480	>480
11391	TRIETHOXYSilANE	998-30-1	277	480	>480	>480
11392	TETRAETHOXYSilANE	78-10-4	89	480	>480	>480
11393	TETRAETHOXYSilANE	78-10-4	94	480	>480	>480
11394	METHYLTRIETHOXYSilANE	2031-67-6	530	165	138	165
11396	METHYLTRIETHOXYSilANE	2031-67-6	555	159	134	159
11398	PROPYLTRIMETHOXYSilANE	1067-25-0	830	164	138	164
11400	PROPYLTRIMETHOXYSilANE	1067-25-0	855	169	140	169
13612	ETHYLTRIETHOXYSilANE	78-07-9	500	267	186	267

13613	ETHYLTRIETHOXSILANE	78-07-9	525	258	181	258
13614	HEXAMETHYLDISILOXANE	107-46-0	<1	480	>480	>480
13615	HEXAMETHYLDISILOXANE	107-46-0	<1	480	>480	>480
13616	50 % METHYLTRIACETOXSILANE/ 50 % ETHYLTRIACETOXSILANE	4253-34-3 17689-77-9	<1	480	>480	>480
13617	50 % METHYLTRIACETOXSILANE/ 50 % ETHYLTRIACETOXSILANE	4253-34-3 17689-77-9	<1	480	>480	>480
13618	POLYDIMETHYLSILOXANE	631-62-9	<1	480	>480	>480
13619	POLYDIMETHYLSILOXANE	631-62-9	<1	480	>480	>480
13620	METHYL TRIS(KETOXIMINO) SILANE	22984-54-9	<1	480	>480	>480
13621	METHYL TRIS(KETOXIMINO) SILANE	22984-54-9	<1	480	>480	>480
13622	TRIFLUOROPROPYLTRISILOXANE	2374-14-3	<1	480	>480	>480
13623	TRIFLUOROPROPYLTRISILOXANE	2374-14-3	<1	480	>480	>480
13624	TRIMETHYLSILANOL	1066-40-6	160	204	168	204
13626	TRIMETHYLSILANOL	1066-40-6	170	225	197	225
13628	HEXAMETHYLDISILIZANE	999-97-3	250	378	300	378
13630	HEXAMETHYLDISILIZANE	999-97-3	220	348	269	348

Table II. Cartridge Service Life Test Results

Run		Test	CAS NO.	Conc. (PPM)	Initial	Final	Time to	Time to
No.	Cartridge	Gas			Weight (Grams)	Weight (Grams)	1 PPM HCL (Min.)	1 PPM Silane (Min.)
9074	AG, NORTH 7500-2	DIMETHYLDICHLOROSILANE	75-78-5	50	90.0 89.5	93.9 92.9	>480	>480
9075	AG, NORTH 7500-2	DIMETHYLDICHLOROSILANE	75-78-5	50	90.1 88.1	93.4 91.7	>480	>480
9076	OV/AG, NORTH 7500-3	DIMETHYLDICHLOROSILANE	75-78-5	50	87.4 89.7	92.2 94.6	>480	>480
9077	OV/AG, NORTH 7500-3	DIMETHYLDICHLOROSILANE	75-78-5	50	90.3 87.2	94.8 91.8	>480	>480
9078	AG, NORTH 7500-2	TRIMETHYLCHLOROSILANE	75-77-4	50	89.9 90.7	92.7 93.4	>480	>480
9079	AG, NORTH 7500-2	TRIMETHYLCHLOROSILANE	75-77-4	50	89.2 90.2	91.7 92.9	>480	>480
9080	OV/AG, NORTH 7500-3	TRIMETHYLCHLOROSILANE	75-77-4	50	87.0 88.0	89.6 90.7	>480	>480
9081	OV/AG, NORTH 7500-3	TRIMETHYLCHLOROSILANE	75-77-4	50	89.6 88.4	92.0 90.2	>480	>480
9082	AG, NORTH 7500-2	VINYLTRICHLOROSILANE	75-94-5	50	90.6 88.3	93.3 91.5	>480	>480
9083	AG, NORTH 7500-2	VINYLTRICHLOROSILANE	75-94-5	50	90.5 90.1	93.1 93.0	>480	>480
9084	OV/AG, NORTH 7500-3	VINYLTRICHLOROSILANE	75-94-5	50	86.9 89.8	90.2 92.6	>480	>480
9085	OV/AG, NORTH 7500-3	VINYLTRICHLOROSILANE	75-94-5	50	90.5 87.9	93.4 90.7	>480	>480
9086	AG, NORTH 7500-2	SILICON TETRACHLORIDE	10026-04-7	50	90.6 88.2	95.0 92.9	>480	>480
9088	OV/AG, NORTH 7500-3	SILICON TETRACHLORIDE	10026-04-7	50	88.2 90.1	93.5 94.8	>480	>480
9090	AG, NORTH 7500-2	TRICHLOROSILANE	10025-78-2	50	90.8 89.7	95.8 94.7	>480	>480
9092	OV/AG, NORTH 7500-3	TRICHLOROSILANE	10025-78-2	50	87.9 89.5	93.2 94.9	>480	>480

Note: Test condition: 25°C, 32 LPM and 50 % relative humidity.

Test Method

Pairs of new, unused North NIOSH approved organic vapor (N7500-1), organic vapor/acid gas (N7500-3) or acid gas (N7500-2) respirator cartridges were tested in the condition as received from the vendor. The tests were conducted at 32 liters per minute representative of a normal average breathing rate, 50 % relative humidity and 25° C. The target challenge concentration varied from 0.1 to 1250 ppm depending on the chemical under study. The selected target was based on the physical-chemical characteristics of the compounds and if there were any existing exposure limits or recommended exposure limits for either the parent compounds or their byproducts. Where an exposure limit existed the challenge atmosphere was targeted to be ten times the exposure limit, assuming that the most common respirator used in the workplace would be a half-mask with an assigned protection factor of ten.

In general, the test method consisted of an air flow-temperature-humidity control system, a contaminant source, the cartridge holder and appropriate gas analyzers. With duplicate tests run on each compound.

The alkoxy silanes were tested in the following manner. A Miller-Nelson Research HCS-401 flow-temperature-humidity control system produces an air stream flowing at 32 liters per minute, 25° C, and 50 % relative humidity. Injecting the solvent into the air stream using a motor driven syringe produces the vapor. Concentrations above 500 ppm required a special vaporizer (Miller-Nelson Research Model SV-2000). Here the liquid is evaporated and mixed with the humid air. The challenge concentration is calculated from the syringe injection rate and the air flow.

The mixture then passes through a Foxboro Miran 1A infrared analyzer where the challenge concentration is continuously monitored. Each silane vapor is scanned before testing to determine the best absorption wavelength. The vapor mixture then enters the cartridge holder and then passes through the cartridges. The downstream concentration is first measured with an infrared analyzer to determine if the silane is present (any alcohol reaction product does not normally interfere with this measurement). The gas stream is then measured with a Beckman Model 400 Hydrocarbon Analyzer (a continuous flame ionization detector (FID)). This gives the total alcohol and silane concentrations. However, it was determined that the alcohol reaction product (ethanol or methanol) appears far in advance of the parent alkoxy silane. To make sure the FID reading was the alcohol, an infra red scan was undertaken on the downstream products during the actual test.

The generation and detection of the chlorosilanes was somewhat different than the alkoxy silanes. An HCS-401 produces a primary air stream flowing at 22 liters/min, 25° C and 73 % relative humidity. Since many of the chlorosilanes react with humidified air, the vapor is produced by injecting the solvent into 10 liters/min of dry (< 3 % RH) air using the syringe pump. Here the liquid is evaporated and mixed with the dry air. The humidified air and the vapor laden air are then combined producing the final test gas mixture at 32 liters/min and 50 % relative humidity. The challenge concentration is calculated from the syringe pump injection rate. The challenge concentrations listed in Tables I and II are accurate to within ± 5 % or less of the calculated value.

The mixture then immediately enters the cartridge holder to minimize any reaction with water vapor and then passes through the cartridges. The downstream concentration of the chlorosilane is measured with the FID and the hydrochloric acid (HCl) is measured with an Interscan Series 1000 HCl analyzer. The detection limit of the HCl analyzer is approximately 0.1 ppm.

The concentrations from the analyzers were monitored with a strip chart recorder. The tests were run for 8 hours or until 10 % breakthrough of the parent alkoxy silane, whichever came first.

Calibration was conducted on each system component prior to testing. The airflow was checked against a Singer DTM-115 dry gas meter, which in turn had been compared, to an American Meter Controls five cubic foot spirometer. The humidity was checked using the magnesium nitrate/sodium nitrate saturated salt technique. The temperature probe was compared to an U. S. Bureau of Standards certified mercury in glass thermometer. The FID analyzer was calibrated by setting the syringe pump to generate to 200 ppm. Past experience on over 4,000 different hydrocarbon tests at Miller-Nelson Research has shown that the response of the FID analyzer is linear up to levels of 10,000 ppm. The detection of the limit of the FID is approximately 0.01 ppm. The infrared analyzer is calibrated using the

known concentration of the challenge vapor and/or a closed loop calibration pump at 5-6 points along the calibration curve. The detection limit of the infrared analyzer is approximately 0.5 ppm depending on the compound.

Challenge concentrations of the contaminant vapor were calculated from

$$C = 24.5 \times 10^6 qD/MQ$$

Where C is the concentration in ppm, q is the liquid injection rate in ml/min, D is the density in gm/ml, M is the molecular weight in grams/mole, and Q is the air flow rate.

RESULTS

The results are shown in Table I and II. Table I shows that the organic vapor cartridges proved effective against the parent organosilane compound. Only five alkoxy silanes (methyltriethoxysilane, propyltrimethoxysilane, ethyltriethoxysilane, trimethylsilanol and hexamethyldisiloxane) showed any parent molecule breakthrough in less than 8 hours. However, all these tests showed early alcohol breakthrough. Since the time weighted average (TWA) for methanol is 200 ppm, the cartridge capacity for the alcohols can be the limiting factor in service life determination.

The organic vapor cartridge was not suitable for hexamethyldisilazane. Although the parent compound was captured, the compound decomposed to form approximately 100 ppm of ammonia. This persisted throughout the entire test. This particular problem could probably be solved by using a multipurpose cartridge that was also certified for ammonia.

Table II shows the results from the chlorosilanes. Both the acid gas and organic vapor/acid gas cartridges proved effective for at least 8 hours against 50 ppm for the five chlorosilanes tested.

CONCLUSIONS

Organic vapor respirator cartridges provide adequate protection from substituted silicon compounds. However, challenge concentrations in the range of 500 ppm and above can produce a significant amount of alcohol as a decomposition product. These alcohols are poorly adsorbed and quickly pass through the cartridges to the wearer. In addition hexamethyldisilazane produces a significant amount of ammonia rendering organic vapor cartridges unsuitable for this vapor, one of the newer multi-contaminant "universal" cartridges which are rated for ammonia as well as organic vapors and acid gases might be a better choice for these compounds. Change schedules thus should be established using actual tests since it is difficult to predict the decomposition of silicon compounds from current adsorption theory. Acid gas and organic vapor/acid gas cartridges provided protection for the 5 chlorosilanes at the 50 ppm exposure level.

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