



Process Compatibility Parameters for Wet Bench Plastic Materials

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Process Compatibility Parameters for Wet Bench Plastic Materials

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Abstract: This report presents a test protocol of accepted analytical procedures to determine the process compatibility parameters for commonly used wet bench plastic materials (PP, FRPP, and PVC) and proposed plastic materials (CPVC, ECTFE, and PVDF) that may be used for wet bench construction. Three industry standard test methods were used to determine the outgassing, leaching, and extraction parameters, defined in combination as the process compatibility parameters. To supplement them, the presence of critical elements at the surface of the materials and the condition of the surface were examined before and after exposure to water and chemicals. Changes in the mass of plastic materials as a result of outgassing, leaching, and extraction were also used to supplement the parameters. The report includes details of the test procedures, data analysis, surface topography of the plastic materials, before and after exposure to water and chemicals, and discussion of the results.

Keywords: Construction Materials, Materials Testing, Contamination Control, Outgassing, Wet Stations, Bulk Chemicals, Ultrapure Water

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Definitions and Abbreviations

amu	Atomic mass unit
analyte	Organic compound or a mixture of organic compounds introduced into an instrument for qualitative and quantitative analysis
APM	Ammonium hydroxide-hydrogen peroxide mixture
CHF	Critical heat flux at or below which there is no ignition (kW/m^2)
CI	Rate of corrosion per unit mass concentration of the plastic material vapors ($\text{\AA}/\text{min}/(\text{g/m}^3)$)
common	Plastic materials currently used in the construction of wet benches (i.e., PP, FRPP, and PVC)
critical	Ions and elements identified in 1997 NTSR Roadmap and SEMI Draft Document 2840 and organic compounds with 10 to 30 carbon atoms
CPVC	Chlorinated poly(vinylchloride)
DI water	Deionized water (purity of the water is measured by its resistivity, with the standard being $18 \text{ M}\Omega$)
ECTFE	Poly(ethylenechlorotrifluoroethylene)
EDX	Energy Dispersive X-ray
eluent	Mobile phase used in chromatography. For ion chromatography eluents used are electrolyte solutions
EP	Extraction parameter (total mass of the critical elements in μg extracted per unit total exposed surface area of the plastic material in cm^2)
FPI	Fire Propagation Index (fire propagation propensity beyond the ignition zone, $(\text{m/s}^{1/2})/(\text{kW/m}^{2/3})$)
FR	Fire retarded
GC	Gas chromatograph
HEPA	High efficiency particulate attenuator
HPM	Hydrogen chloride-hydrogen peroxide mixture
ICP	Inductively Coupled Plasma-Mass Spectrometry
leachate	Ion or element in the solution obtained after soaking a material in ultrapure water, ozonated water, or a chemical
LP	Leaching Parameter (total mass of critical ions and elements, and TOC in μg leached per unit total exposed surface area of the plastic material in cm^2)
MS	Mass spectrometer
NTSR	National Technology Roadmap for Semiconductors
OP	Outgassing Parameter (total mass of critical organic compounds outgassed in μg per unit total exposed surface area of the plastic material in cm^2)
PP	Polypropylene
PVC	Poly(vinylchloride)

PVDF	Poly(vinylidene fluoride)
RL	Reporting limit
SC1	Standard Clean 1
SC2	Standard Clean 2
SEM	Scanning electron microscope
SPM	Sulfuric acid-hydrogen peroxide mixture
TD	Thermal desorber
TOC	Total oxidizable carbon
TRP	Thermal Response Parameter- ignition propensity of a material (kW-s ^{1/2} /m ²)
UPW	Ultrapure water (silica <1 ppb, total organic compounds (TOC) < 20 ppb, anions and ammonium ion < 0.05 ppb, prepared by reverse osmosis de-ionization)

1 EXECUTIVE SUMMARY

The purpose of this study was to

- Identify and select commonly used industry standard test methods for outgassing, leaching, and extraction
- Define parameters to be indications of process compatibility
- Evaluate the parameters against commonly used wet bench plastic materials as references
- Compare the parameters for proposed wet bench plastic materials

Three industry standard test methods were used to determine the process compatibility parameters: 1) outgassing for critical (10–30 carbon atom containing) organic compounds, 2) leaching for critical¹ ions and elements and total oxidizable carbon (TOC), and 3) extraction for critical elements. The process compatibility parameters were identified as the outgassing parameter (OP), the leaching parameter (LP), and the extraction parameter (EP). The values of these three parameters were determined for three commonly used wet bench plastic materials (used as reference materials) and three proposed wet bench plastic materials (used as test materials) to evaluate the test protocol. The fire behavior of the proposed plastic materials is characterized by no fire propagation beyond the ignition zone.

Results for the surface topography and mass change of plastic materials from outgassing, leaching, and extraction were used to augment the results obtained from the OP, LP, and EP values.

The test protocol developed in the study and the values of the process compatibility parameters presented in the report are based on plastic materials in their fully processed form, not on feedstock plastic materials. The actual OP, LP, and EP values obtained in the test protocol may be affected by the fabrication history by which the plastic sheets were produced in addition to any residuals from the polymerization processes or the manufacture of the feedstock resins. Thus, users must be cognizant of the level of processing a plastic sample has undergone before comparing the data. Furthermore, this study focused on plastic material used in non-wetted wet bench areas (i.e., structural components); it must be understood that the acceptable levels of contaminants for these wet bench construction materials may be much different from the levels for the wet bench plastic materials used for wetted parts.

1.1 Methodology

In the outgassing test method, the sample pieces were heated at 100°C for 30 minutes using a dynamic headspace method to accelerate the outgassing of the organic compounds. The outgassed compounds were analyzed by a gas chromatographic-mass spectrometric (GC-MS) technique. Masses of all the outgassed organic compounds in the range of 10–30 carbon atoms, identified as critical, were added. The sum of the masses of the critical organic compounds was divided by the total exposed surface area of the sample pieces and the result was used as the value of the outgassing parameter (OP) with units of $\mu\text{g}/\text{cm}^2$.

¹ Critical ions and elements are identified in the 1997 National Technology Roadmap for Semiconductors (NTRS) [4] and in the SEMI draft document 2840 [5].

In the leaching test method, the sample coupons were soaked for 30 days in UPW at 85°C in an oven without any shaking and for 24 hours in ozonated UPW at ambient temperature, shaking the solutions every four hours. An ion-chromatographic (IC) system, inductively coupled plasma-mass spectrometric (ICP-MS) system, and TOC analyzer were used to identify and measure the masses of ions, elements, and TOC, respectively. Masses of all the critical ions and elements and TOC leached by UPW and ozonated UPW from the sample coupon were added and divided by the total exposed surface area of the sample coupon. The result was taken as the value of the leaching parameter (LP) with UPW and with ozonated UPW, respectively.

The extraction test method was similar to the leaching test method, except five chemicals typically used in wafer processing in cleanrooms were used in place of UPW and ozonated UPW. The chemicals were shaken every 24 hours for the entire 30-day extraction period. The chemicals used were 1) APM (SC1), 2) HPM (SC2), 3) SPM (Piranha), 4) 70% w/w nitric acid (HNO₃), and 5) 49% w/w hydrofluoric acid (HF). The initial temperature of each chemical was the temperature of the freshly mixed components (usually higher than ambient, e.g., 110–130°C for SPM), which cooled to the ambient temperature during the extraction period. ICP-MS was used to identify and measure the masses of elements. Masses of all the critical elements extracted from the plastic material by each chemical were added and divided by the total exposed surface area of the sample coupon. The result was taken as the value of the extraction parameter (EP) for each chemical in $\mu\text{g}/\text{cm}^2$.

1.2 Results

The OP values for the proposed wet bench plastic materials (CPVC, ECTFE, and PVDF), used as the test materials, were comparable to the values for two of the common wet bench plastic materials (PP and PVC) and at least five times lower than the value for FRPP, which is reference material.

The LP values for the proposed test materials were generally lower than the values for the two common wet bench plastic materials (PP and FRPP) used as the reference materials. The LP value for CPVC was higher than PP with UPW, and PVDF was higher than PVC in ozonated UPW.

The EP values for the proposed test materials were nearly an order of magnitude lower than the values for the two common wet bench plastic materials (FRPP and PVC), used as the reference materials. CPVC however, had the highest EP value with HF, because large amounts of titanium, aluminum, and sodium were extracted from CPVC by HF. The amount of titanium extracted was at least seven times greater than any other element.

The leaching and extraction processes had little effect on the surfaces of the proposed plastic materials, whereas the surfaces of PP and FRPP, the common wet bench plastic materials, were affected (as observed in the microphotographs from the high magnification scanning electron microscope [SEM]).

The changes in the mass from outgassing, leaching and extraction were significantly less than 1% for all the plastic materials, except in the CPVC-HF interaction, where it was 1.6%. The higher mass change for the CPVC-HF interaction is possibly due to the removal of titanium (used as TiO₂ for whitening CPVC), aluminum, and sodium.

The values for the process compatibility parameters for the six plastic materials examined in the study are summarized in Table 1.

Table 1 Values for the Process Compatibility Parameters for Plastic Materials^a

Process	Agent	Parameter ($\mu\text{g}/\text{cm}^2$)	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Out-gassing	Helium	OP	<0.10	0.54	<0.10	<0.10	<0.10	<0.10
Leaching	UPW	LP	9.2	25	0.42	23	7.6	2.5
	Ozonated UPW ^b	LP	37	47	3.0	1.8	0.96	1.2
Extraction	APM (SC1)	EP	0.0061	0.055	0.77	0.57	0.0092	0.016
	HPM (SC2)	EP	0.019	0.064	0.80	0.18	0.0032	0.026
	SPM (Piranha)	EP	0.036	4.6	1.1	0.49	0.011	0.031
	70% HNO ₃	EP	0.0094	0.090	0.88	0.51	0.0079	0.025
	49% HF	EP	4.9	3.0	400	900	0.28	0.32

a: The sheet materials manufactured from the same plastic material can be different as far as contaminants are concerned since most contaminants arise from the additives, not from the resin.

b: Ozone concentration: $150 \pm 5 \text{ g}/\text{m}^3$ (ppm) in the gas stream and about $40 \text{ g}/\text{m}^3$ (ppm) in ozonated UPW.

2 INTRODUCTION

Recently, the fire protection community has responded to potential losses from fires in wet benches by seeking for new plastic materials (with no fire propagation beyond the ignition zone and low smoke and corrosive product release), reductions in the amount of combustible materials, and/or the inclusion of fire suppression systems. The material suppliers are now proposing several plastic materials² for fabricating semiconductor equipment, especially for the non-wetted wet bench shell materials. These materials, however, must be compatible with cleanroom processes, must not contribute any additional contaminants than the common wet bench plastic materials, must have appropriate physical properties (such as weldability), and must be cost effective.

Currently, wet benches are fabricated from polypropylene (PP), fire retarded polypropylene (FRPP), and poly(vinylchloride) (PVC) (all referred to as common wet bench plastic materials in this report). At this time, there is no systematic testing protocol for determining the process compatibility for the plastic materials. This depends on the extent of outgassing, leaching, and extraction of critical organic compounds, ions and elements, and TOC. It also depends on the surface damage and changes in the mass resulting from interactions with UPW and chemicals typically used for wafer processing in cleanrooms.

Types of critical ions and elements and TOC contamination for wafer processing in cleanrooms have been identified in the 1997 *National Technology Roadmap for Semiconductors* (NTRS) in the Front-End Processes section [4] and in the SEMI draft document 2840 [5]. These critical

² As of November 1998, the following plastic materials are available and described as low-combustibility plastic materials for the construction of semiconductor equipment, especially the wet benches [1,2,3]- fire retarded polypropylene (FRPP-FMPP3750); fire retarded polypropylene (FRPP-CP7-D); glass fiber reinforced phenolic; fire retarded poly(vinylchloride) (FRPVC-FMT300); chlorinated poly(vinylchloride) (CPVC-FMRC4910 Corzan white 120); poly(ethylenechlorotrifluoroethylene) (ECTFE-Halar 901); poly(vinylidene fluoride)(PVDF-Kynar2850); poly(vinylidene fluoride)(PVDF Kynar740); poly(vinylidene fluoride) (PVDF-Hylar MP-20); poly(vinylidene-fluoride) (PVDF-Kytec MP-20); fire retarded poly(tetrafluoroethylene)-fluoropolymer composite.

contaminants (ions and elements) are listed in Table 2. Organic compounds with 10–30 carbon atoms in the outgassed compounds from plastic materials, under normal operating conditions, are considered critical for wafer processing [6].

The presence of critical ions and elements such as Al, Cu, Au, Fe, K, Ag, and Na are known to be especially detrimental to semiconductor manufacturing. They can enter the Si lattice substitutionally, migrate in the Si lattice, and affect minority carrier lifetime and threshold voltage. Consequently, the concentrations of these individual species may have to be considered in addition to the OP, LP, and EP, which deal with the sum total of all the critical ions and elements. For example, the sum totals for a potential wet bench material may be acceptable, but individual concentrations of one or more of the species may still make the material unacceptable.

International SEMATECH's Environment, Safety, and Health Division thus undertook this study. The purpose of the study was two-fold:

- To define an analytical test protocol for evaluating the process compatibility parameters for the wet bench fabrication materials
- To develop a method of quantifying the process compatibility parameters for the wet bench fabrication materials with process chemicals including UPW and ozonated UPW. It should be noted that the wetted parts such as the immersion tanks, valves, filters, etc., are not necessarily made from the same construction materials as the shell of the wet bench.

Three industry standard test methods were used to determine the process compatibility parameters:

- The outgassing test determined amounts of critical organic compounds outgassed from the plastic materials.
- The leaching test determined amounts of critical ions and elements and TOC leached from the plastic materials by UPW and ozonated UPW.
- The extraction test determined amounts of critical elements extracted from the plastic materials by five different chemicals typically used in wafer processing in cleanrooms.

These three industry standard test methods are described in the Section 2 with details in Appendix A. The plastic materials used to develop the test protocol are listed in Table 3.

Table 2 Critical Ions and Elements for Cleanroom Processes^a

Contaminants	Contaminants	Contaminants	Contaminants
Ions			
Ammonium (NH ₄ ⁺)	Chloride (Cl ⁻)	Nitrate (NO ₃ ⁻)	Phosphate (PO ₄ ³⁻)
Bromide (Br ⁻)	Fluoride (F ⁻)	Nitrite (NO ₂ ⁻)	Sulfate (SO ₄ ²⁻)
Elements			
Aluminum (Al)	Copper (Cu)	Manganese (Mn)	Strontium (Sr)
Arsenic (As)	Chromium (Cr)	Molybdenum (Mo)	Thorium (Th)
Boron (B)	Iron (Fe)	Nickel (Ni)	Titanium (Ti)
Calcium (Ca)	Lead (Pb)	Potassium (K)	Tungsten (W)
Cadmium (Cd)	Lithium (Li)	Silver (Ag)	Vanadium (V)
Cobalt (Co)	Magnesium (Mg)	Sodium (Na)	Zinc (Zn)

a: information taken from Refs. 4 and 5

Table 3 Plastic Materials Examined in the Study

Plastic Material	Thickness (mm)	Color and Surface Condition ^a
Reference Materials (common wet bench plastic materials)		
Polypropylene (PP)	6	<u>Color</u> : white with a slight blue tint; <u>Surface</u> : smooth with small bumps
Fire retarded polypropylene (FRPP)	6	<u>Color</u> : white; <u>Surface</u> : slightly rough; some pits, no crystals on the surface
Poly(vinylchloride) (PVC -TSP 739)	6	<u>Color</u> : light bluish-white; <u>Surface</u> : rough, bumpy with very few pits
Test Materials (low-combustibility-proposed wet bench plastic materials)		
Chlorinated poly(vinylchloride) (CPVC-4910Corzan)	6	<u>Color</u> : light yellow-cream color. <u>Surface</u> : slightly rough, parallel line present, which may be extrusion marks. Sub-micron pits and particles are present on the surface
Poly(ethylenechlorotrifluoroethylene) (ECTFE -Halar 901)- copolymer of ethylene and chlorotrifluoroethylene	6	<u>Color</u> : dull white-yellowish translucent. <u>Surface</u> : smooth; occasional scratches on the surface
Poly(vinylidene fluoride)- copolymer of vinylidene -fluoride and hexafluoro-polypropylene (PVDF-HFP Kynar 2850)	3	<u>Color</u> : translucent. <u>Surface</u> : smooth; occasional scratches on the surface

a: color by visual observations and surface conditions by SEM.

3 TEST METHODS AND ANALYTICAL PROCEDURES

In this study, industry accepted analytical procedures and standard tests were used. All the experiments were performed at the Balazs Analytical laboratory. Standard analytical procedures were used by the Balazs Analytical Laboratory for cleanliness, operation of the instruments, calibrations, acquisition of the data, and data reporting. The reporting limit is defined as the minimum concentration of a substance that can be identified, measured, and reported with confidence that the analyte concentration is greater than zero. Where applicable, it is determined from the analysis of a sample in a given matrix containing analyte. The test conditions were selected by the project team on the advice of the International SEMATECH Working Group team members.

3.1 Samples

The samples of the plastic materials were shipped to Balazs Laboratory³ as 50 mm x 20 mm x 6 mm thick coupons (3 mm thick for PVDF). A total of 80 coupons were shipped in containers that prevented contamination. The coupons for the leaching and extraction tests were sent in clean polypropylene bags. The samples for the outgassing tests were sent wrapped in aluminum

³ It was recommended that coupons be cut in a manner representative of how they would be cut when making wet benches. It was recommended that the edge exclusions off at least 3 mm from the edge of the sheet stock should be used and the coupons should be cut using representative cutting tools that minimize contamination of the sample. The suppliers provided material identification, traceable lot numbers, and material safety data sheet (MSDS) with the samples. Preferred sample dimensions were 50 ± 5 mm x 20 ± 5 mm x 6 ± 1 mm thick.

foil (foil baked out at 250°C for one hour and cooled to room temperature) and placed in clean polypropylene bags. For the outgassing tests, the coupons were cut into sample pieces (about 20 mm x 3 mm x 3 mm thick).

A single small piece of each plastic material was used in the outgassing test. In the leaching test, two coupons in 150 mL of UPW and four coupons in 200 mL of ozonated UPW were used to determine ions. To determine TOC and elements, four coupons in 300 mL of UPW and four coupons in 200 mL of ozonated UPW were used. In the extraction test, two coupons in 150 mL of each chemical were used to determine the elements.

3.2 Outgassing Test Method

The outgassing test method was developed to quantify the OP—the total mass of the critical organic compounds outgassed in μg per unit total exposed surface area of the plastic material in cm^2 . Organic compounds with carbon atoms ranging from 10–30 atoms are considered critical for cleanroom processes [6]. These medium-to-high boiling organic compounds tend to adhere to surfaces more strongly than lower boiling compounds (with carbon atoms ≤ 10), making surfaces hydrophobic and thus difficult to clean [6].

The outgassing test method simulated accelerated conditions by heating the sample at 100°C for 30 minutes using dynamic headspace conditions. To remove the surface contaminants from shipping and handling, the samples were rinsed with UPW for 10 minutes, wiped with presoaked isopropyl alcohol wipers, rinsed again with UPW, and air-dried in a HEPA hood with a carbon prefilter for 24 hours. The cleaned coupons were cut into approximately 20 mm x 3 mm x 3 mm thick sample pieces with tools previously cleaned with an organic solvent and baked with a heat gun (about 250°C for 30 sec.). The sample pieces were weighed and their dimensions were measured. The sample pieces were placed in 90 mm long, 6 mm OD and 5 mm ID sample tubes made of stainless with a retaining screen near one end (Figure 1). The sample pieces were typically about 0.2 g in mass.

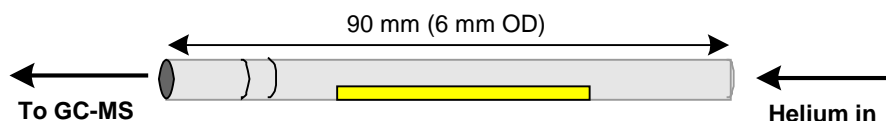
Each sample tube was heated in a clean automated thermal desorber (ATD) at 100°C for 30 minutes while continuously purging with an inert gas (helium) at 50 mL/min (and ambient temperature) (Figure 1). The outgassed compounds were carried by helium to the column of a gas chromatograph (GC), where each compound was separated (Figure 2 and Figure 3). The outgassed organic compounds were separated into three groups:

- Low boilers ($\text{C}_6\text{-C}_{10}$), defined as those compounds with retention times in the GC column between n-hexane (C_6H_{14}) and n-decane ($\text{C}_{10}\text{H}_{22}$),
- Medium boilers ($\geq \text{C}_{10}\text{-C}_{20}$), defined as those compounds with retention times in the GC column between n-decane ($\text{C}_{10}\text{H}_{22}$) and n-eicosane ($\text{C}_{20}\text{H}_{42}$),

High boilers ($>\text{C}_{20}$), defined as those compounds with retention times in the GC column beyond n-eicosane ($\text{C}_{20}\text{H}_{42}$) but up to at least n-triacontane ($\text{C}_{30}\text{H}_{62}$).

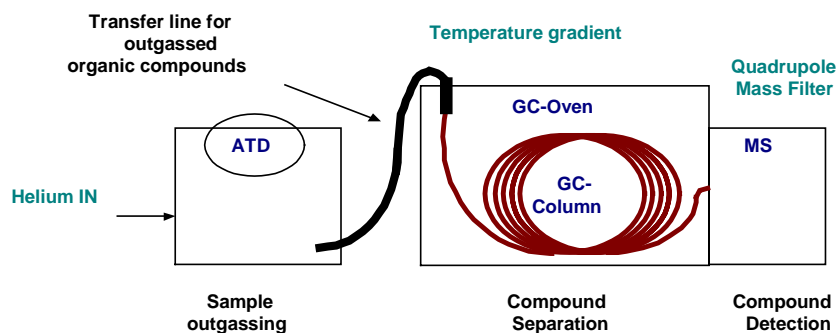
As each compound exited from the GC column, it was carried by helium into a quadrupole mass spectrometer (MS), where it was ionized by electron impact, compound identified, and its mass estimated (Figure 2 and Figure 4). Details of the procedure are contained in Appendix A.

The total mass of the medium-to-high boiling organic compounds (with carbon atoms between 10 and 30), as measured by GC-MS, per unit exposed surface area, was used as the OP value for the plastic material.



Note: A sample piece (20 mm x 3 mm x 3 mm thick) is shown inside the sample tube.

Figure 1 Schematic of Sample Tube for Automated Thermal Desorber (ATD) Used in the Outgassing Test



Note: Gas chromatograph = GC, mass spectrometer = MS (quadrupole).

Figure 2 Schematic of Arrangement of Instruments in the Outgassing Test

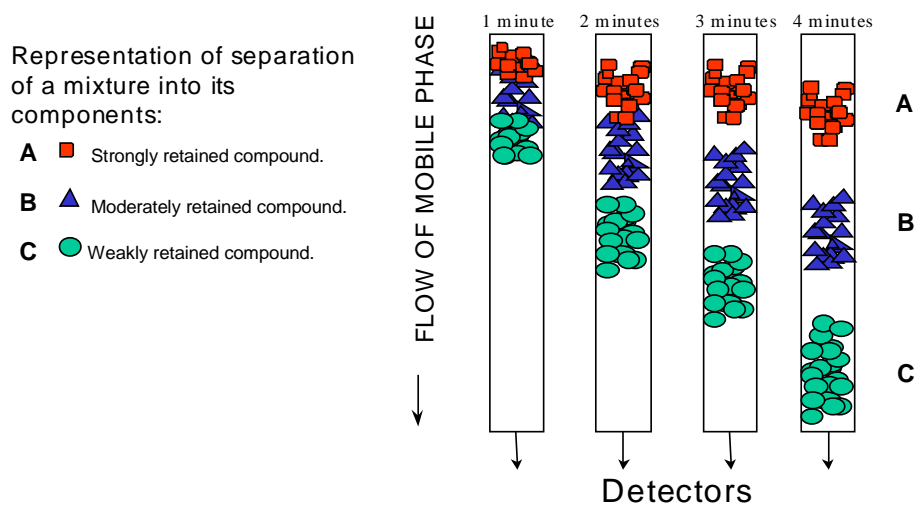


Figure 3 Separation of Individual Compounds in the Gas Chromatograph (GC) Column

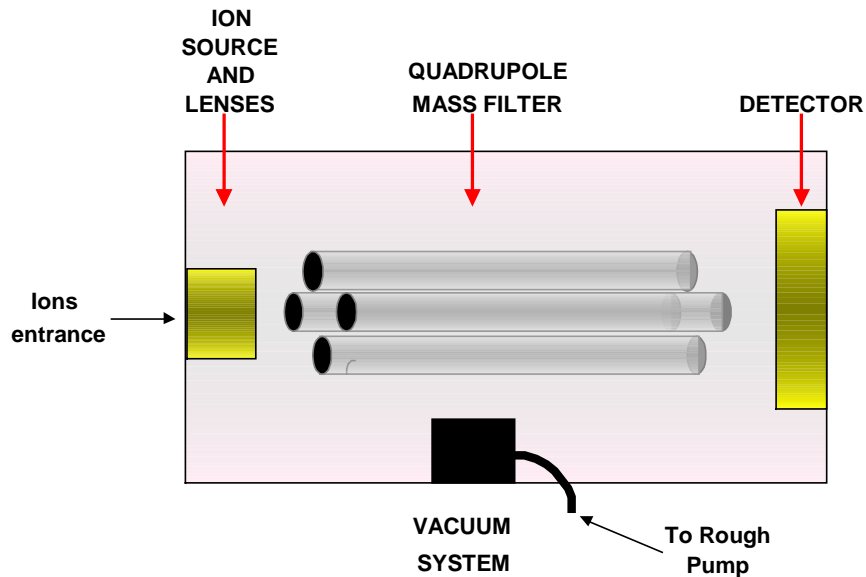


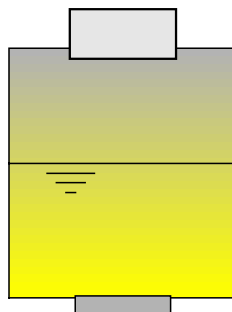
Figure 4 Schematic of Quadrupole Mass Spectrometer

3.3 Leaching Test Method

The leaching test method was developed to quantify the LP—the total mass of the critical ions and elements and TOC leached from the plastic material in μg per unit total exposed surface area in cm^2 . Critical ions and elements for cleanroom processes are listed in Table 2.

In the leaching test, plastic material coupons approximately 50 mm long and 20 mm wide were used. To remove the surface contaminants from shipping and handling, the coupons were rinsed with UPW for 10 minutes, wiped with presoaked isopropyl alcohol wipers, rinsed again with UPW, and dried under N_2 . The coupons were weighed and their dimensions were measured. Surface topography by SEM, surface composition by energy dispersive X-ray (EDX), and visual observations such as color and appearance were recorded for each sample coupon.

Each individual cleaned coupon was placed in a polypropylene bottle for the UPW soak to analyze for ions and in a fluoropolymer bottle to analyze for elements and TOC (Figure 5). For the ozonated UPW soak, a fluoropolymer bottle was used. The PP bottle was 250 ml in volume, 60 mm in diameter, and 100 mm high to the neck. The fluoropolymer bottle was 500 ml in volume, 70 mm in diameter, and 135 mm high to the neck.



Note: Polypropylene bottle was used for UPW soak and fluoropolymer bottle was used for ozonated UPW soak.

Figure 5 Schematic of Soaking Bottle with a Coupon for the Leaching Test

In each bottle, 75 mL of UPW per coupon was used for the UPW soak and 50 mL of UPW per coupon was used for the ozonated UPW soak. For each leach study, two bottles with UPW or ozonated UPW (no coupons) were also analyzed to record the blank data. To determine ions leached by UPW soak and ozonated UPW, two coupons (150 mL) and four coupons (200 mL) were used, respectively. To determine elements leached by UPW soak, four coupons in 300 mL were used and for ozonated UPW, four coupons in 200 mL were used.

In the UPW leaching tests, the coupons were soaked in closed bottles for 30 ± 2 days, in an oven, maintaining the UPW temperature at $85 \pm 2^\circ\text{C}$ for the soaking period without any shaking. In the ozonated UPW leaching tests, the coupons were soaked in fluoropolymer bottles for 24 hours with ozonated UPW at ambient temperature. The ozonated UPW solutions were shaken manually every four hours; 150 g/m^3 (ppm) of ozone gas stream was continuously purged through the UPW solution in the bottle to maintain ozone saturation of UPW with an estimated concentration of 40 g/m^3 (ppm) in UPW. The ozonated UPW soaking tests were performed under an exhaust hood, using fluoropolymer tubes to introduce the ozone. Ultrahigh purity oxygen was used as the feed gas for ozone generation to minimize NO_x formation. At the end of the soaking period, to avoid contamination⁴, the outside of each bottle was thoroughly cleaned with UPW under a HEPA hood before opening.

At the conclusion of the leaching test, each coupon was weighed and its surface condition and topography were examined visually and by SEM/EDX (for details, see Appendix A), respectively. The solutions from the leaching tests were analyzed for the following:

- Types and amounts of ions leached by UPW and ozonated UPW by an IC system (Figure 6, for details see Appendix A)
- Types and amounts of elements leached by UPW and ozonated UPW by ICP-MS (Figure 7, for details see Appendix A)
- Mass of TOC leached by UPW and ozonated UPW from the plastic materials by a TOC analyzer (for details see Appendix A).

The IC system (Figure 6) consists of reservoirs for eluents (electrolyte solutions, such as a solution of the mixture of sodium carbonate and sodium bicarbonate), a pump, a concentrator, a guard column, a sample loop, a sample injector, a separator column, a suppressor column, and a conductivity detector. To enable analyses in the low parts-per-billion (ppb) range, a chemically inert single piston pump is used to pump the sample through the concentrator column before injecting it into the separator column. The suppressor column is used to chemically reduce the high background conductivity of the electrolytes in the eluent and to convert the ions of interest into a more conductive form. The separator column is filled with particles of stationary phase over which mobile phase is pumped. When a sample is injected to the flowing mobile phase, it is carried into the column and the process of separation begins using the partition coefficients of the ions (an ion with a larger partition coefficient has a longer retention time than one with a

⁴ For ozonated UPW soaks, overall fluoride and nitrate ions data do not reflect amounts of these ions leaching from the plastic materials. Some fluoride ions come from the interaction of ozone with fluoropolymer bottles and tubing. The formation of ozone from oxygen containing trace nitrogen is known to produce N_2O_5 , which reacts with water to form HNO_3 . Thus, much of the nitrate detected may be from the ozone gas itself. To reduce the amount of nitrates, the ozone gas stream was passed through a UPW scrubber prior to use. All eight leaching tests were performed in parallel over the same 24-hour period.

lower partition coefficient). Each ion detected is identified using retention time data (confirmed against the daily calibration standard) (for details, see Appendix A).

In the ICP-MS system (Figure 7), the liquid sample injected is converted to fine aerosol droplets that are injected into argon ICP. Under extremely high temperature conditions of ICP (which can reach as high as $\sim 9000\text{K}$), the molecules are broken down into atoms and most elements are ionized with $>90\%$ efficiency. The ions are then injected into the ICP-MS interface where most of the gas is removed. The positive ions are extracted into a mass spectrometer and are identified based on their characteristic mass-to-charge ratio; their amounts are then quantified by a calibration standard (for details see Appendix A). The quadrupole mass filter used can analyze singly charged ions from 1–300 amu.

3.4 Extraction Test Method

The extraction test method was developed to quantify EP—the total mass of the critical elements in μg extracted per unit total exposed surface area of the plastic material in cm^2 . Elements considered critical for the cleanroom processes are listed in Table 2.

In the extraction test, plastic material coupons approximately 50 mm long, 20 mm wide, and 6 mm thick were used; the PVDF coupons, however, were 3 mm thick. To remove the surface contaminants from shipping and handling, the coupons were rinsed with UPW for 10 minutes, wiped with presoaked isopropyl alcohol wipers, rinsed again with UPW, and dried under nitrogen. The coupons were weighed, and their dimensions were measured. Visual observations such as color and appearance and surface topography by SEMI/EDX were recorded for each sample coupon.

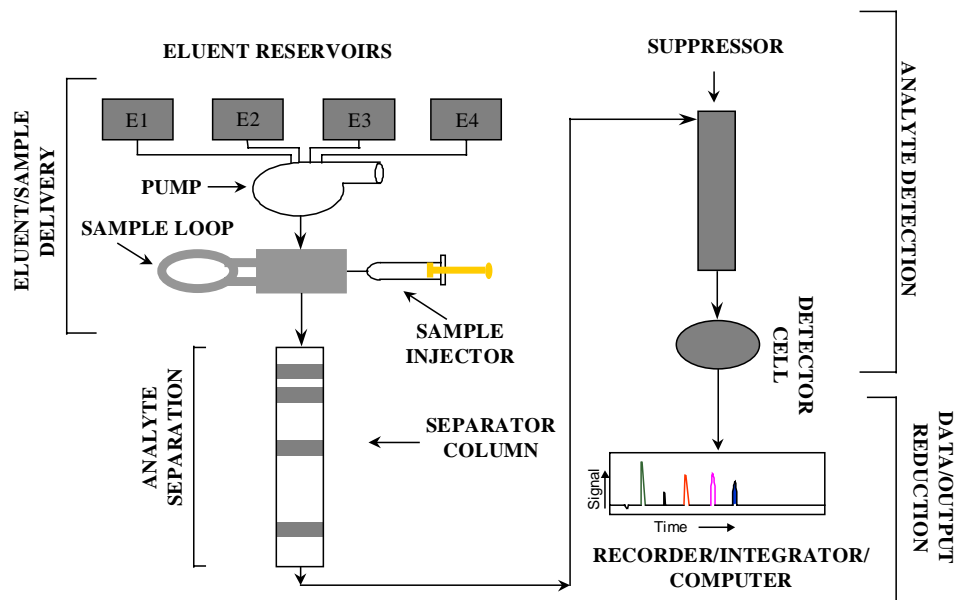


Figure 6 Schematic of Ion Chromatographic (IC) System Used to Determine Types and Amounts of Ions Leached by UPW and Ozonated UPW

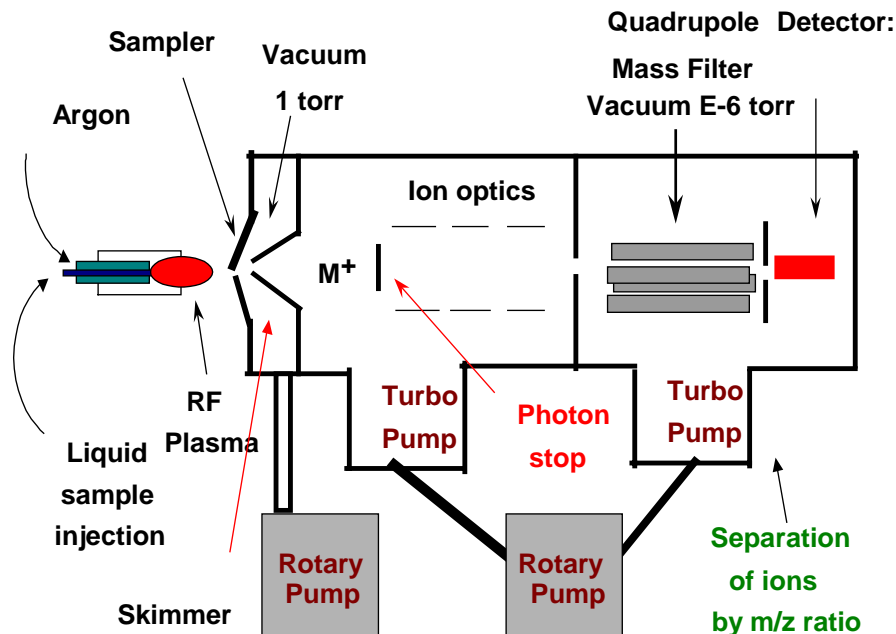


Figure 7 Schematic of Inductively Coupled Plasma Mass Spectrometric (ICP-MS) System

The sum of the masses of the critical ions and elements and TOC leached per unit exposed surface area of the plastic material was used as the LP value with UPW and with ozonated UPW, respectively.

The cleaned coupons were placed in fluoropolymer extraction bottles containing 75 gm of the solution of chemicals per coupon (Figure 5). For the tests, two coupons of each sample were placed in separate bottles containing 150 gm of the following chemicals:

- APM (also, known as SC1 = 1:1:5 (30% w/w NH_4OH : 31% w/w H_2O_2 : H_2O volume ratio)
- HPM (also known as SC2 = 1:1:6 (HCl 37% w/w: 31% w/w H_2O_2 : H_2O volume ratio)
- SPM (also known as Piranha = 5:1 v/v (96% w/w H_2SO_4 : 31% w/w H_2O_2)
- 70% (w/w) HNO_3
- 49% (w/w) HF

Note: Where w/w represents weight of the component/weight of the chemical.

The initial temperature of the chemical was the temperature of the freshly mixed chemicals, which cooled to the ambient temperature during the 30 ± 2 days extraction period. For example, the initial temperature of 96% by weight of H_2SO_4 and 31% by weight of H_2O_2 ranged from about 110–130°C, which cooled to the ambient temperature during the extraction period. The extraction was continued in the closed bottles, shaking the solutions every 24 hours. At the end of the extraction period, to avoid contamination, the outside of each bottle was thoroughly cleaned with UPW before opening. The weight of each coupon was measured and its surface condition and topography were examined visually and by the SEM/EDX technique.

To measure types and amounts of extracted elements, a known volume of each chemical from the extraction tests was evaporated at a temperature below the boiling point of the chemical to

concentrate the chemical. The concentrate was dissolved in dilute nitric acid and injected into an ICP-MS system (Figure 7 and Appendix A).

The sum of the masses of the critical elements extracted per unit exposed surface area of the plastic material was used as the EP value with the respective chemical.

4 OUTGASSING OF ORGANIC COMPOUNDS FROM PLASTIC MATERIALS

Outgassing is defined as the release of organic compounds as a result of heating the plastic materials at 10°C for 30 minutes. The amounts of outgassed organic compounds, measured by GC-MS, are normalized by the total exposed surface area of the sample coupon. All the outgassing test results⁵ in this report are presented in $\mu\text{g}/\text{cm}^2$.

4.1 Outgassing Data

Outgassing data is listed in Table 4 and Table 5 and shown in Figure 8 through Figure 14.

Table 4 Sample Weight, Dimensions and Total Mass Outgassed

Plastic Material	Weight x 10 ⁻⁶ (μg)		Dimensions (cm)			% Mass Change ^a	Total Mass Outgassed ($\mu\text{g}/\text{cm}^2$)
	Initial	Final	Length	Width	Thickness		
PP	0.18152	0.18150	1.91	0.369	0.352	-0.01	6
FRPP	0.19850	0.19837	2.11	0.397	0.288	-0.07	42
PVC	0.20894	0.20894	2.07	0.296	0.260	0.00	<0.1
CPVC	0.21133	0.21080	2.04	0.333	0.271	-0.25	201
ECTFE	0.18507	0.18490	1.78	0.245	0.267	-0.09	87
PVDF	0.20554	0.20490	1.62	0.379	0.243	-0.31	291

a: negative numbers indicate mass loss

In Table 5, the estimated reporting limit for the accelerated outgassing method is 0.1 $\mu\text{g}/\text{cm}^2$ for a sample surface area of about 2.0 cm^2 for any single organic compound outgassed. The reporting limit is equal to the estimated minimum amount of typical organic compound that can be routinely quantified.

⁵ ng = 10⁻⁹ g; μg = 10⁻⁶ g. To convert ng to μg , multiply ng by 10⁻³

Table 5 Organic Compounds Outgassed from the Plastic Materials^a

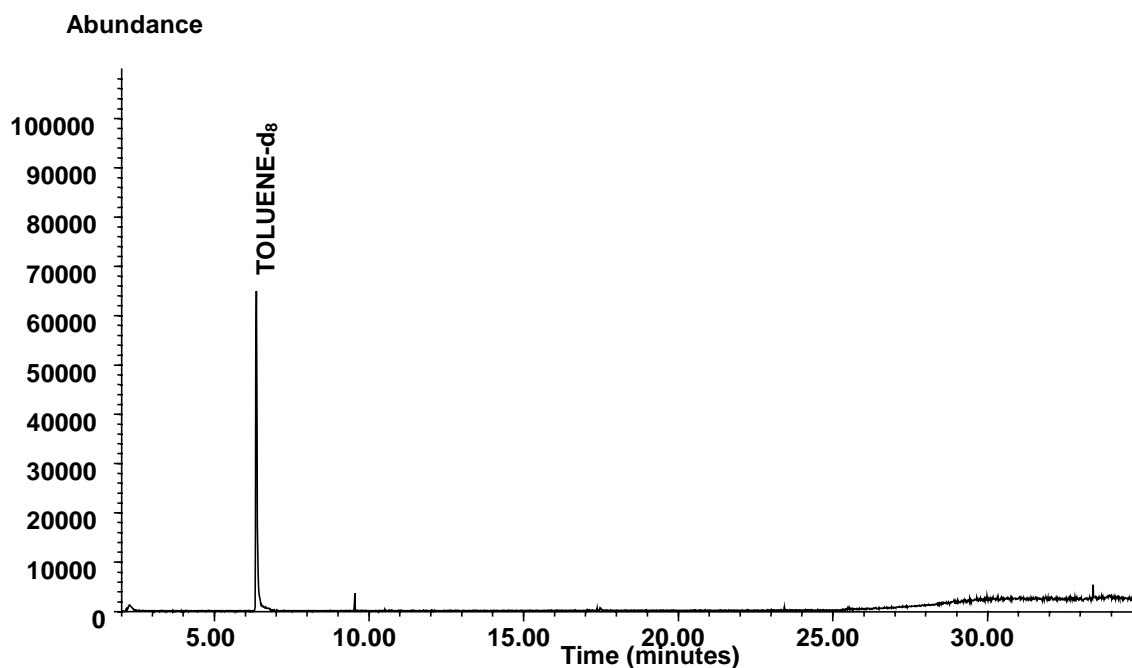
Compounds Outgassed	Mass of Outgassed Organic Compounds ($\mu\text{g}/\text{cm}^2$) ^b						
	Blank	PVC	PP	FRPP	ECTFE	CPVC	PVDF
Isopropyl alcohol ^c	*	*	*	0.12	*	*	*
Fluorohydrocarbons	*	*	*	*	*	*	< 0.1
n-Butanol	*	*	*	< 0.1	*	*	*
1-Bromobutane	*	*	*	< 0.1	*	*	*
Toluene	*	*	*	<0.1	*	*	*
C ₆ -C ₁₀ Hydrocarbons	*	*	< 0.1	< 0.1	*	*	*
(Di-t-Butyl)phenol	*	*	*	< 0.1	*	*	*
C ₁₁ -C ₂₀ Hydrocarbons	*	*	< 0.1	0.51	*	*	*
Low Boilers (C ₆ -C ₁₀) ($\mu\text{g}/\text{cm}^2$)	*	< 0.1	< 0.1	0.17	*	*	< 0.1
Medium Boilers ($\geq\text{C}_{10}$ -C ₂₀) ($\mu\text{g}/\text{cm}^2$)	*	< 0.1	< 0.1	0.54	< 0.1	< 0.1	*
High Boilers (>C ₂₀) ($\mu\text{g}/\text{cm}^2$)	*	*	*	*	*	*	*

a: Heat at 100 °C for 30 minutes.

b: Reporting limit: 0.1 $\mu\text{g}/\text{cm}^2$.

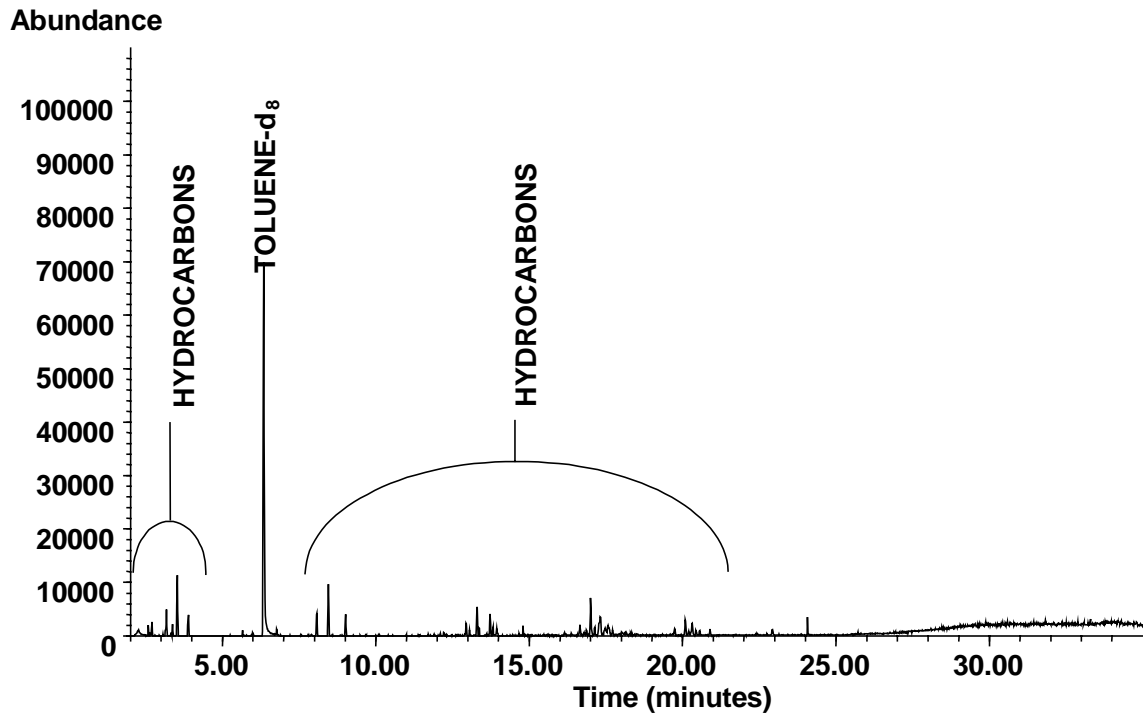
c: used for sample cleaning.

*: below the reporting limit.



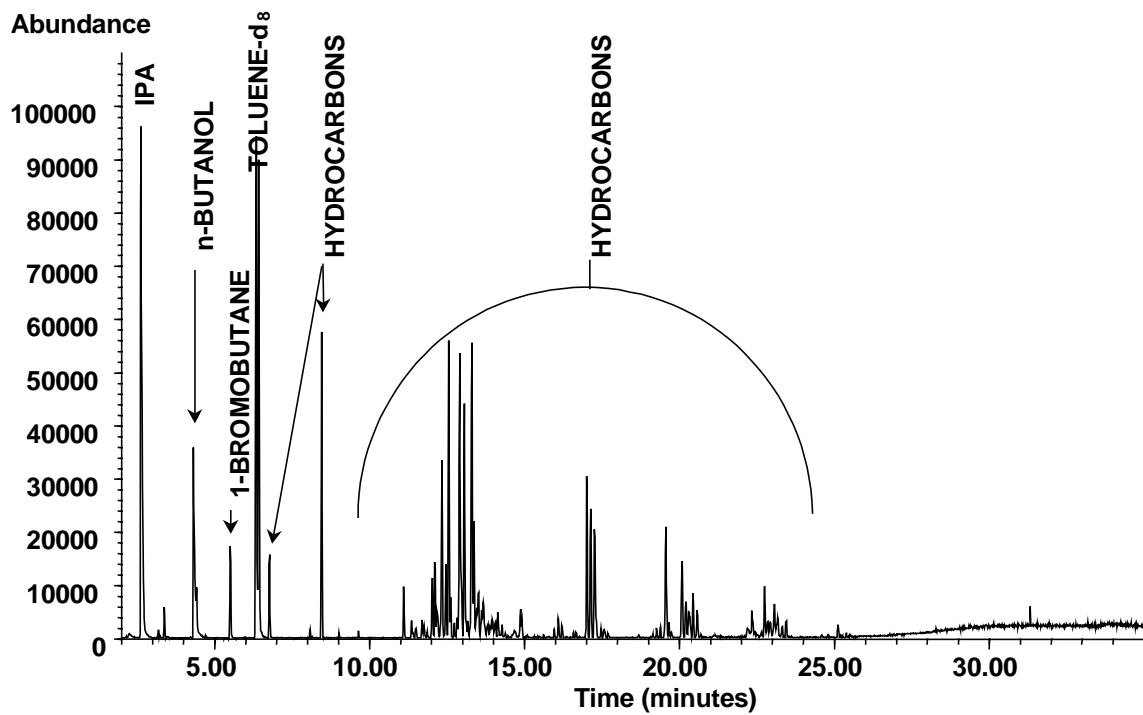
Note: Toluene-d₈ is the internal standard.

Figure 8 Organic Compounds Outgassed from the Blank Sample Tube Heated at 100°C for 30 Minutes



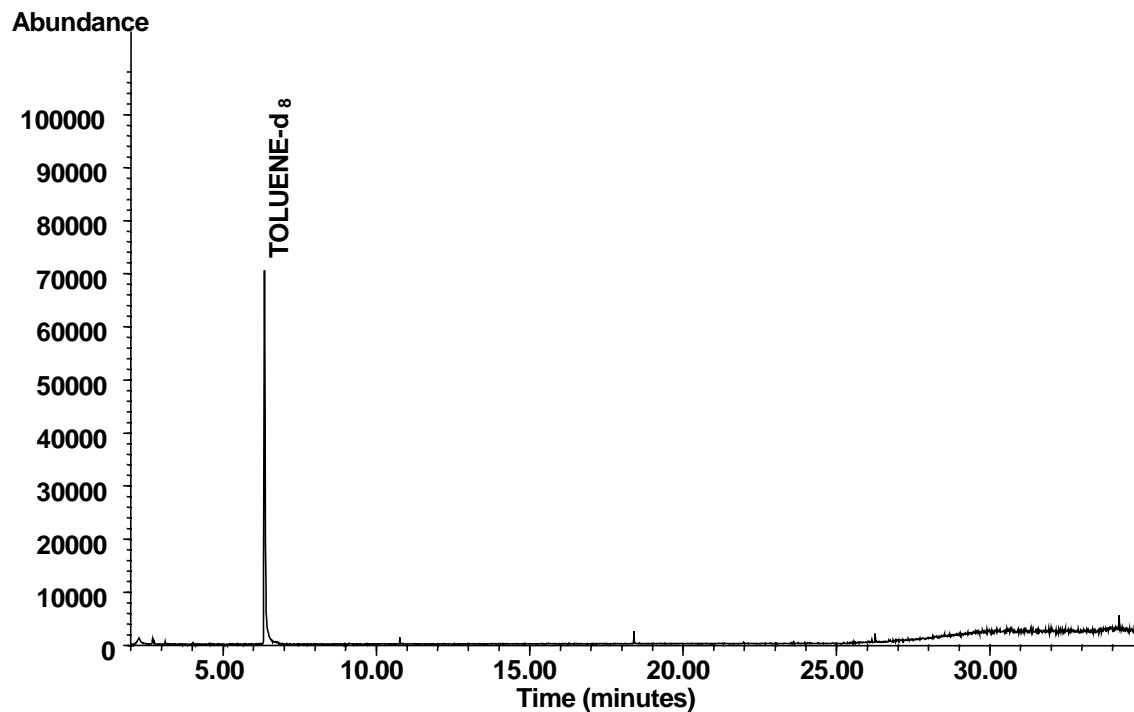
Note: Toluene-d₈ is the internal standard.

Figure 9 Organic Compounds Outgassed from PP Heated at 100°C for 30 Minutes



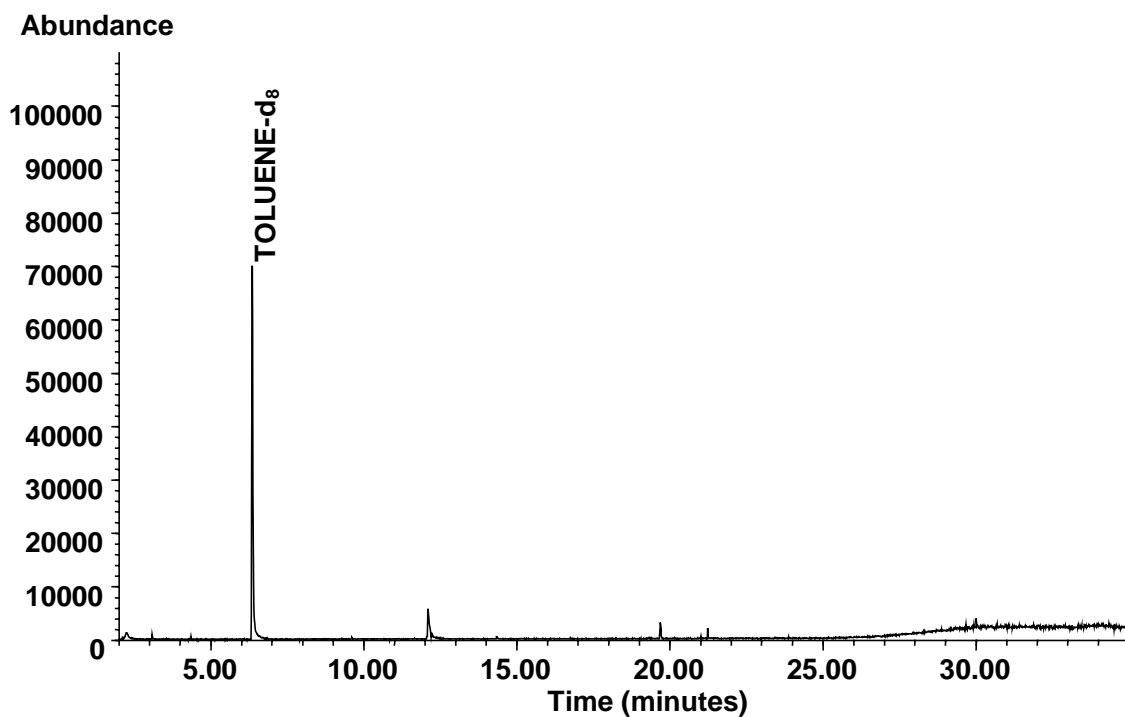
Note: Toluene-d₈ is the internal standard. IPA: Isopropyl alcohol is used for wiping the sample.

Figure 10 Organic Compounds Outgassed from FRPP Heated at 100°C for 30 Minutes



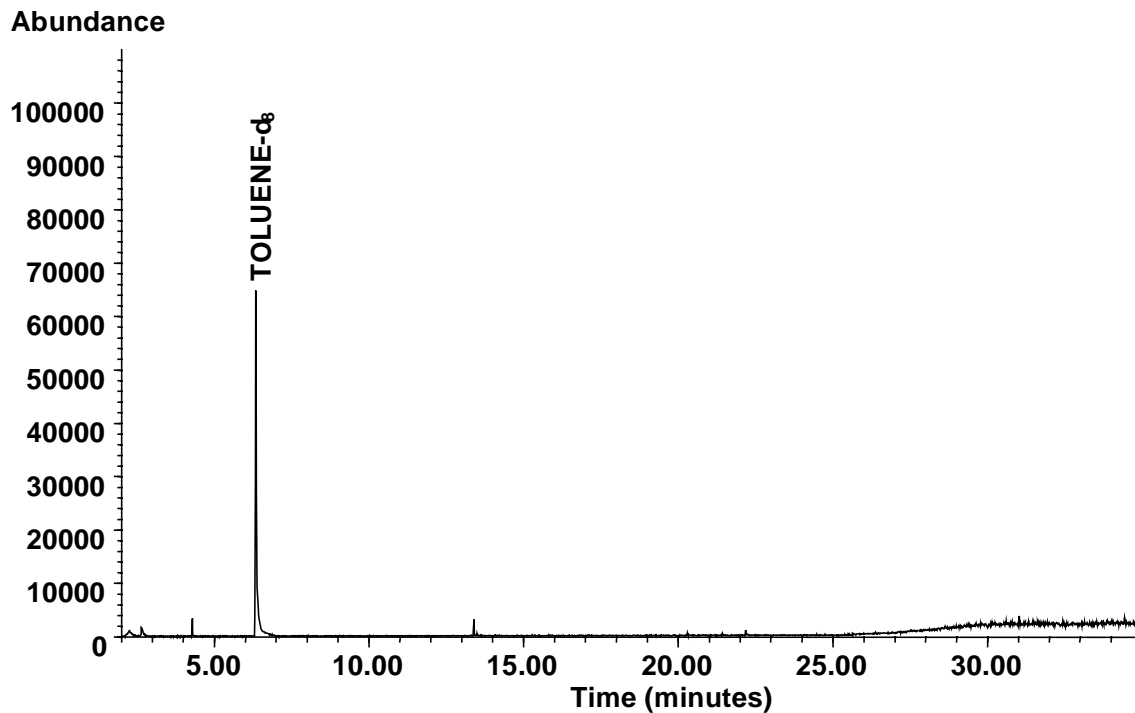
Note: Toluene-d₈ is the internal standard.

Figure 11 Organic Compounds Outgassed from PVC Heated at 100°C for 30 Minutes



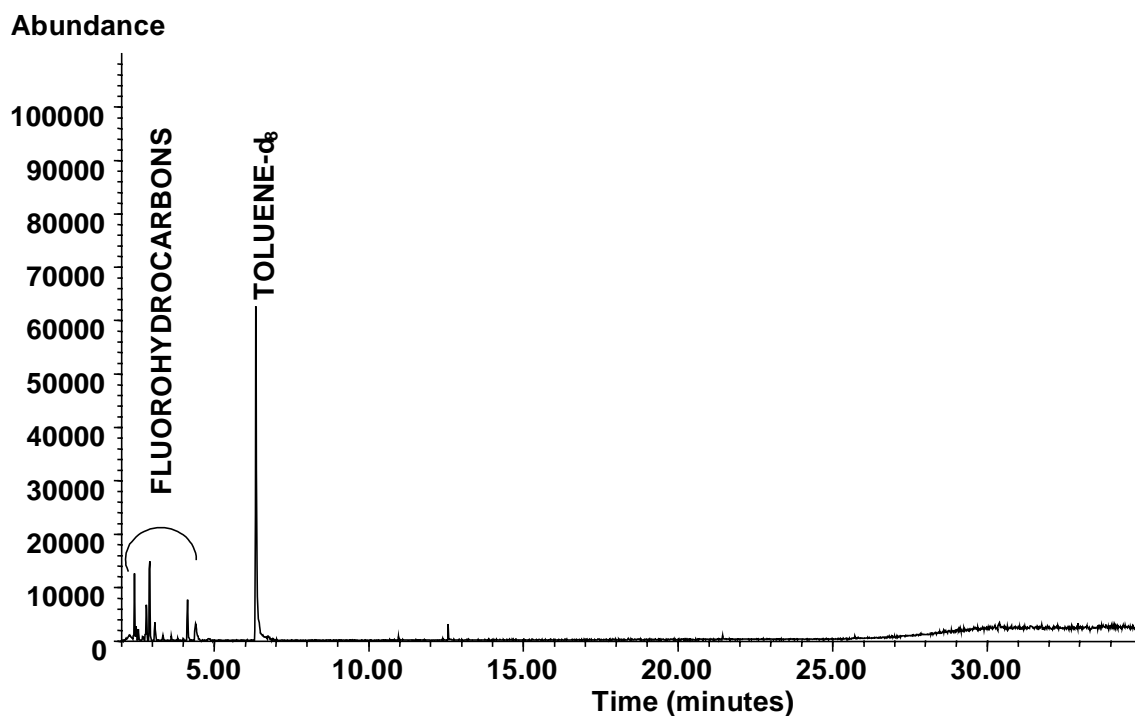
Note: Toluene-d₈ is the internal standard.

Figure 12 Organic Compounds Outgassed from CPVC Heated at 100°C for 30 Minutes



Note: Toluene-d₈ is the internal standard.

Figure 13 Organic Compounds Outgassed from ECTFE Heated at 100°C for 30 Minutes



Note: Toluene-d₈ is the internal standard.

Figure 14 Organic Compounds Outgassed from PVDF Heated at 100°C for 30 Minutes

In Figure 8 through Figure 14, the area under each curve roughly correlates with the amount of each organic compound present. PVC, CPVC, and ECTFE show negligible amounts of outgassed organic compounds. PVDF, which is a copolymer of vinylidene fluoride (VDF) and hexafluoropropylene (HFP) shows outgassing of small amounts of low molecular weight fluorohydrocarbons, possibly the HFP monomer (boiling point < C₆ hydrocarbons). PP shows outgassing of small amounts of hydrocarbons, whereas FRPP shows outgassing of higher amounts of hydrocarbons than PP, as well as butanol, bromobutane, toluene, and very small amounts of di-(t-butyl) phenol. The outgassing of bromobutane is likely associated with the brominated fire retardant in FRPP. Toluene and butanol may be used as solvents. Di-(t-butyl) phenol is likely to be used as an antioxidant. The low and medium-boiling hydrocarbons in PP and FRPP may be monomers trapped during the polymerization process or components of the FRPP lubricant system compounds.

The accelerated outgassing test has limitations. Some polar organic compounds with lower boiling points may not be trapped effectively and hydrocarbons with boiling points significantly lower than that of hexane (69°C) or other low-boiling compounds may not be recovered well. Thermally unstable compounds may decompose to other compounds, and some reactive compounds can interact to give new compounds or low recoveries. Amines may react with acidic components to give poor recovery.

4.2 Outgassing Parameter (OP)

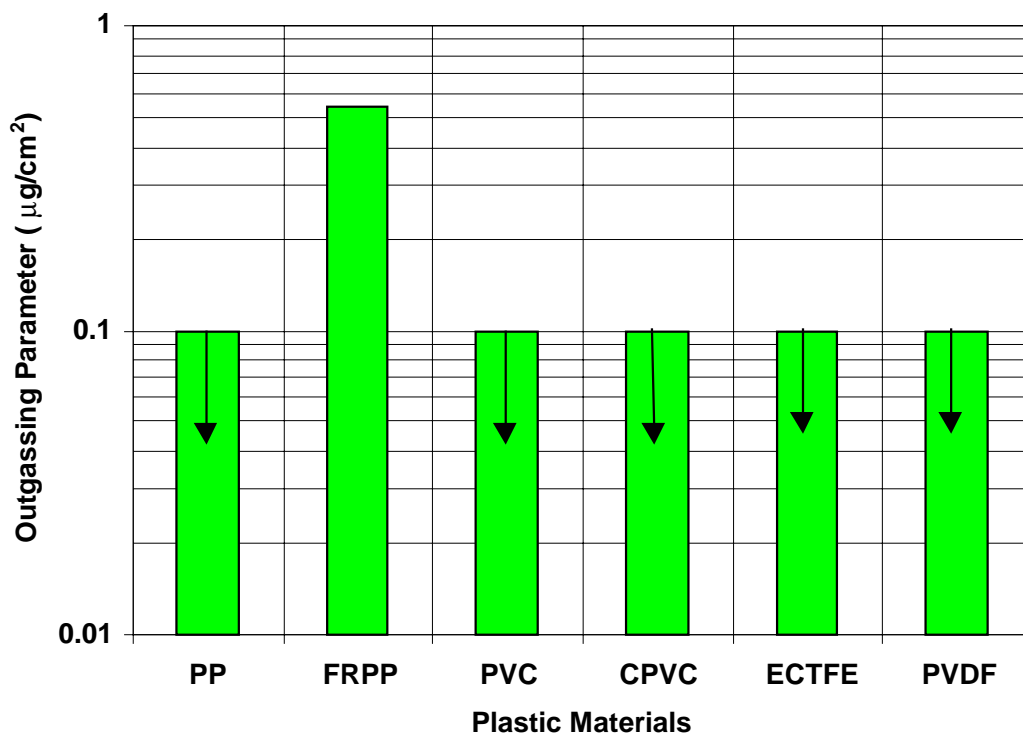
The OP is defined as the mass of the critical organic compounds (C₁₀ to C₃₀) outgassed in µg per unit total exposed surface area of the plastic material in cm². The OP values listed in Table 6 are calculated from the data from Table 5 and are plotted in Figure 15.

Table 6 Values of the Outgassing Parameter for the Plastic Materials^a

Material	Name	Outgassing Parameter (µg/cm ²)
Reference Materials	Polypropylene (<i>PP</i>)	<0.1
	Fire retarded Polypropylene (<i>FRPP</i>)	0.54
	Poly(vinylchloride) (<i>PVC</i>)	<0.1
Test Materials	Chlorinated poly(vinylchloride) (<i>CPVC</i>)	<0.1
	Poly(ethylenechlorotrifluoroethylene) (<i>ECTFE</i>)	<0.1
	Poly(vinylidene fluoride) (<i>PVDF</i>)	<0.1

a: Reporting limit: 0.1 µg/cm²

The OP values for all the plastic materials are low, although those for the proposed materials are lower than the value for FRPP. For some plastic materials, total mass outgassed is high (Table 4 for FRPP, PVDF, CPVC, and ECTFE), whereas for others it is low (PP and PVC). The differences between the OP values and total mass outgassed (measured directly by weighing the sample before and after the test) could be due to outgassing of low boiling point monomers and solvents trapped in the polymer matrix that could not be trapped by this method. Figure 14 is a typical example of the outgassing of low molecular weight fluorohydrocarbons from PVDF, probably trapped in the polymer matrix during the processing of the polymer, or perhaps hexafluoropropylene (HFP) monomer escaping from the copolymer matrix. In general, the low-boiling compounds outgassed are not considered critical in cleanroom processes unless they contain phosphorus or are reactive (amines)[6].



Note: Arrows indicate values less than $0.1 \mu\text{g}/\text{cm}^2$ (D.L.).

Figure 15 Values of the Outgassing Parameter for the Plastic Materials

5 LEACHING OF IONS, ELEMENTS, AND TOTAL OXIDIZABLE CARBON FROM PLASTIC MATERIALS BY UPW AND OZONATED UPW

Leaching is defined as the removal of ions, elements, and TOC from the plastic materials by UPW and ozonated UPW. Amounts of ions, elements, and TOC, measured by IC, ICP, and TOC techniques are normalized by the total exposed surface area of the sample coupon. All the leaching test results⁶ in this report are presented in $\mu\text{g}/\text{cm}^2$.

5.1 Leaching Data for Ions

Details of the ion leaching data for the test plastic materials by UPW and ozonated UPW are listed in Table 21 and Table 22 in Appendix B and are summarized in Table 7. Amounts of the critical⁷ ions leached from the plastic materials by UPW and ozonated UPW are plotted in Figure 16 (critical ions are listed Table 2).

It should be noted that ozonated UPW data include the contribution from fluoride ions generated from the interaction of ozone with fluoropolymer bottles and tubing (primary source) and the nitrate ions primarily formed from trace levels of N_2 in the oxygen feed gas used to generate the ozone gas stream. In addition, the effect of ozone on any free bromide or chloride ions in

⁶ $\text{ng} = 10^{-9} \text{ g}$; $\mu\text{g} = 10^{-6} \text{ g}$. To convert ng to μg , multiply ng by 10^{-3}

⁷ The term 'critical' in this report is defined by NTSR [4] and SEMI [5].

solutions is unknown if they oxidize to bromine or chlorine gas, which could purge out and remain undetected. Further oxidation could form bromine or chlorine oxides, which could also remain undetected.

The total amounts of the critical ions leached by UPW is highest for FRPP ($0.89 \mu\text{g}/\text{cm}^2$) and lowest for PP ($0.029 \mu\text{g}/\text{cm}^2$). For ozonated UPW leach, the total amount of the critical ions leached is high for FRPP ($5.2 \mu\text{g}/\text{cm}^2$) and lowest for PVDF ($0.89 \mu\text{g}/\text{cm}^2$).

Table 7 Amounts of the Ions Leached from the Plastic Materials by UPW and Ozonated UPW

Material	Name	Amounts of Ions ($\mu\text{g}/\text{cm}^2$) $\times 10^3$			
		UPW		Ozonated UPW	
		All Ions	Critical Ions	All Ions	Critical Ions
Reference Materials (Common)	Polypropylene (PP)	33	29	2,700	2,700
	Fire retarded Polypropylene (FRPP)	3,600	890	5,300	5,200
	Poly(vinylchloride) (PVC)	260	260	2,900	2,800
Low-Combustibility Test Materials	Chlorinated poly(vinylchloride) (CPVC)	830	730	1,800	1,600
	Poly(ethylenechlorotrifluoroethylene) (ECTFE)	208	176	940	940
	Poly(vinylidene fluoride) (PVDF)	760	760	890	890

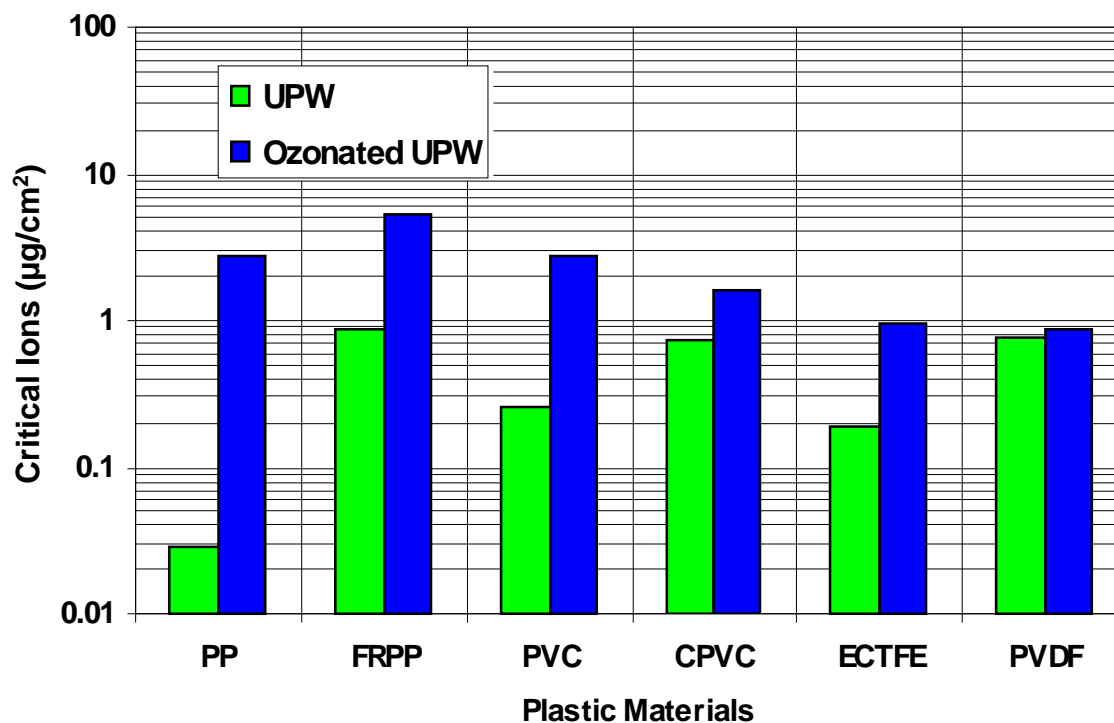


Figure 16 Total Amounts of the Critical Ions Leached from the Plastic Materials by UPW at 85°C in 30 Days and Ozonated UPW at Ambient Temperature in 24 Hours

5.2 Leaching Data for Elements

Details of the element leaching data for the plastic materials for UPW and ozonated UPW are listed in Table 23 and Table 24 in Appendix B and are summarized in Table 8. The amounts of the critical elements leached from the plastic materials by UPW and ozonated UPW are plotted in Figure 17.

From the UPW-soak studies, the highest amounts of the critical elements are leached from FRPP ($0.18 \mu\text{g}/\text{cm}^2$); the lowest levels, from ECTFE and PVDF ($0.0066 \mu\text{g}/\text{cm}^2$).

For the ozonated UPW leach, the total amount of critical elements leached is highest for CPVC ($0.16 \mu\text{g}/\text{cm}^2$) and FRPP ($0.15 \mu\text{g}/\text{cm}^2$) and lowest for ECTFE ($0.00020 \mu\text{g}/\text{cm}^2$).

Table 8 Amounts of Elements Leached from Plastic Materials by UPW and Ozonated UPW

Material	Name	Amounts of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$			
		UPW		Ozonated UPW	
		All Elements	Critical Elements	All Elements	Critical Elements
Reference Materials	Polypropylene (PP)	100	100	98	86
	Fire retarded Polypropylene (FRPP)	1,000	180	8,500	150
	Poly(vinylchloride) (PVC)	1,200	55	94	92
Test Materials	Chlorinated poly(vinylchloride) (CPVC)	510	140	180	160
	Poly(ethylenechlorotri-fluoroethylene) (ECTFE)	7.0	6.6	0.20	0.20
	Poly(vinylidene fluoride) (PVDF)	7.0	6.6	5.5	5.5

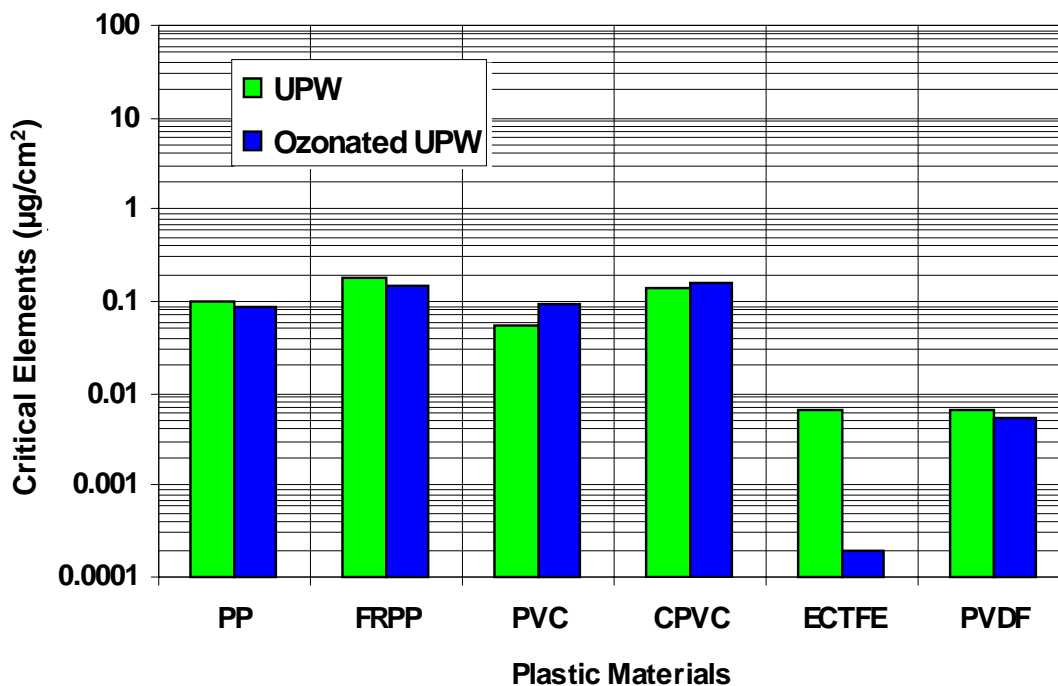


Figure 17 Total Amounts of Critical Elements Leached from Plastic Materials by UPW at 85°C in 30 Days and Ozonated UPW at Ambient Temperature in 24 Hours

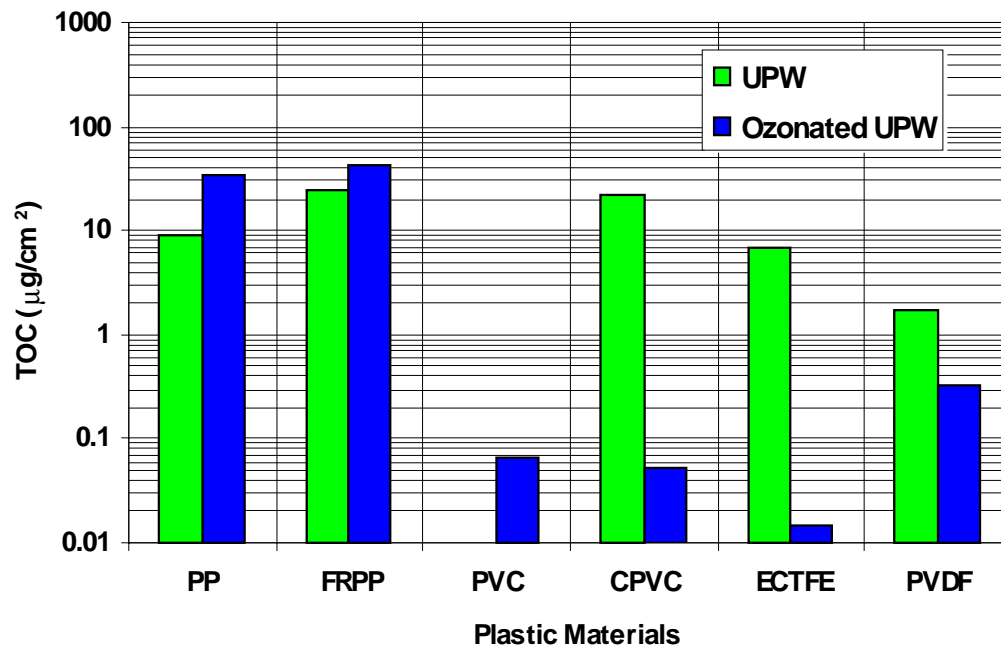
5.3 Leaching Data for TOC

TOC leaching data for the plastic materials for UPW and ozonated UPW are listed in Table 9 and plotted in Figure 18. The total amounts of TOC leached by UPW and ozonated UPW is highest for FRPP (24 and 42 µg/cm², respectively) and lowest for PVC (0.10 µg/cm²) and ECTFE (0.015 µg/cm²), respectively.

Table 9 Total Amounts of TOC Leached From Test Plastic Materials by UPW and Ozonated UPW^a

Amount of TOC (µg/cm ²) x 10 ³								
RL ^b	Blank I	Blank II	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Ultrapure Water at 85°C and Soaking Period of 30 Days								
5	50	100	9200	24000	(14)*	22000	7000	1800
Amount of TOC minus blank			9,100	24,000	(0)*	22,000	6,900	1,700
Ozonated Ultrapure Water at Room Temperature and Soaking Period of 24 Hours								
5	90	100	34000	42000	160*	147	110	420
Amount of TOC minus blank			34,000	42,000	65*	52	15	330

Notes: a: Data are reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data. b: reporting limit. Number of coupons: 4 in 300 mL UPW and about 200-mL ozonated UPW. Numbers in parentheses appear to be anomalies. In the future, these types of anomalies need to be resolved by repeating the test. *: Apparently anomalous data as evidenced by the loss of organic material from the surface displayed by the SEMs, Table 11 and Table 12, Figure 22b and Figure 22c.



Note: The amount of TOC leached from PVC by UPW was close to zero and appears to be an anomaly.

Figure 18 Total Amounts of Oxidizable Carbon (TOC) Leached from Plastic Materials by UPW at 85°C in 30 Days and Ozonated UPW at Ambient Temperature in 24 Hours

5.4 Leaching Parameter

The Leaching Parameter (LP) is defined as the mass of the critical ions and elements, and TOC leached by UPW and ozonated UPW in µg in the leaching test per unit total exposed surface area of the plastic material in cm². LP values are listed in Table 10 and plotted in Figure 19.

Table 10 Values of the Leaching Parameter for the Plastic Materials^a

Material	Sample	Leaching Parameter ^a (µg/cm ²)	
		UPW ^b	Ozonated UPW ^c
Reference Material	Polypropylene (PP)	9.2	37
	Fire retarded Polypropylene (FRPP)	25	47
	Poly(vinylchloride) (PVC)	0.42	3.0
Test Material	Chlorinated poly(vinylchloride) (CPVC)	23	1.8
	Poly(ethylenechlorotrifluoroethylene) (ECTFE)	7.1	0.96
	Poly(vinylidene fluoride) (PVDF)	2.5	1.2

a: Leaching Parameter (LP) = (total mass of critical ions + total mass of critical elements + mass of TOC) leached/ total exposed surface area of the plastic material from Tables 7 to 9; **b:** UPW at 85 ± 2°C. Leaching period: 30 ± 2 days; **c:** Ozonated UPW at ambient temperature. Leaching period: 24 hours.

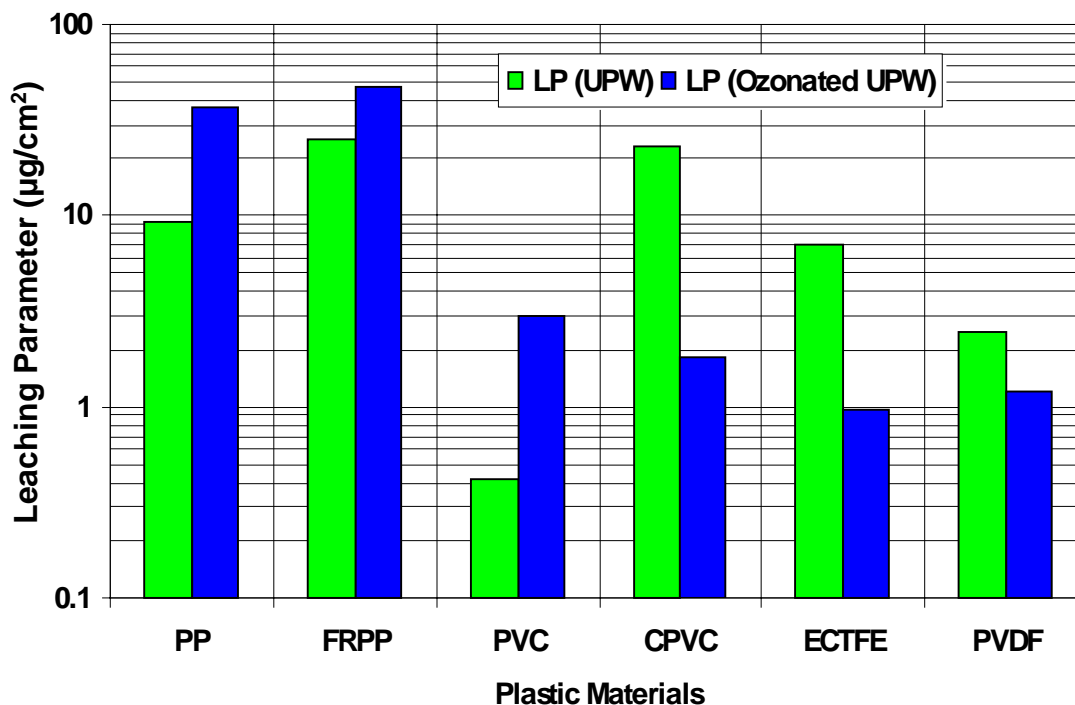


Figure 19 Values of the Leaching Parameter for the Plastic Materials Soaked in UPW at 85°C for 30 Days and in Ozonated UPW at Ambient Temperature for 24 Hours

5.5 Surface Conditions and Topography of the Plastic Materials Before and After the Leaching Tests

The surface conditions observed visually are listed in Table 11 and surface topographic examination results by SEM/EDX are listed in Table 12 and shown in Figure 20 through Figure 25. These data are used to augment the LP. Visual observations indicate that there is no change in the color of the surface of PP, ECTFE, and PVDF as a result of interactions with UPW and ozonated UPW.

The surface topographic examination (Figure 20 through Figure 25 and Table 12) indicates that the surface damage from interactions with UPW and ozonated UPW is not as severe for the proposed plastic materials as it is for the common materials. No additional critical elements are leached to the surface as a result of the interactions of the plastic materials with UPW and ozonated UPW.

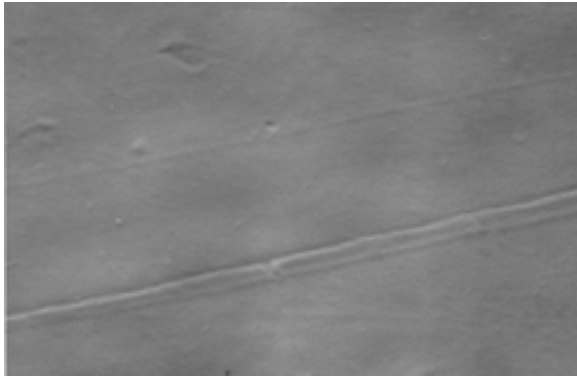
Table 11 Surface Condition of Plastic Materials Before and After the Leaching Tests Observed Visually

Material	Before Interaction	After Interaction	
		UPW (85°C, 30 days)	Ozonated-UPW soak (ambient temperature, 24 hours)
PP	White with slight blue tint	No visible change	No visible change
FRPP	White	Light cream-to-yellow color	Surface appeared to be rougher and not as shiny as before the soak
PVC	Light bluish-white	Pinkish-to-peach color	Color changed to off-white- yellowish
CPVC	Light yellow-cream color	Very light yellow color	Surface appeared to be slightly rougher
ECTFE	Dull white-yellowish translucent	No visible change	No visible change
PVDF	Translucent	No visible change	No visible change

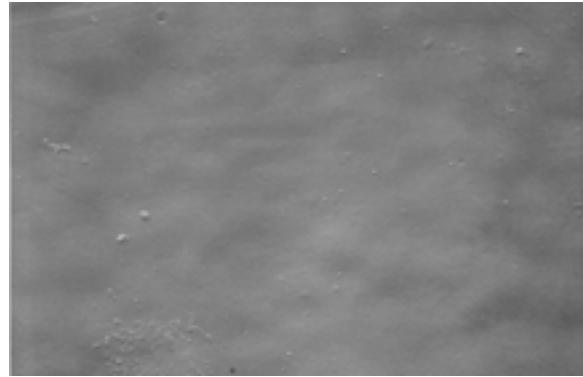
Table 12 Surface Topographic Examination Results for Plastic Materials Before and After the Leaching Tests

Material	EDX Results		SEM Results	
	Before	After	Before	After
UPW Leaching (85°C, 30 days)				
PP	C ^a , O, Ti	Same as before	Smooth surface with small bumps (Figure 20a)	Minimal change, parallel lines visible. Lines could be from extrusion or molding (Figure 20b)
FRPP	C ^a , O, Al, Br, Sb, Fe*	Same as before, No Sb and Fe*	Slightly rough surface; some pits, no crystals on the surface (Figure 21a)	Extensive leaching out of the material. There may be surface deposition of leached compounds or exposed crystalline fibers (Figure 21b)
PVC	C, O, Cl ^a , Ca, Ti, Fe*	Same as before, Al, No Ca	Rough bumpy surface with very few pits (Figure 22a)	Leaching out of material and pits on the surface with particles in the pits (Figure 22b)
CPVC	C, O, Cl ^a , Al, Si, Ti, Fe*	Same as before, No Si	Slightly rough surface, parallel line present, may be extrusion marks. Sub-micron pits and particles are present (Figure 23a)	Little change (Figure 23b)
ECTFE	C, F ^a , Cl, Fe*	No discernible change	Smooth surface; occasional scratches (Figure 24a)	No discernible change (Figure 24b)
PVDF	C, F ^a	No discernible change	Smooth surface (Figure 25a)	No discernible change (Figure 25b)
Ozonated UPW Leaching (Ambient Temperature, 24 Hours)				
PP	C ^a , O, Ti	Same as before + Fe*	Smooth surface with small bumps (Figure 20a)	Extensive cracking of the surface (Figure 20c)
FRPP	C ^a , O, Al, Br, Sb, Fe*	Same as before, No Fe*	Slightly rough surface; some pits, no crystals on the surface (Figure 21a)	Extensive cracking of the surface (Figure 21c)
PVC	C, O, Cl ^a , Ca, Ti, Fe*	Same as before. No Ca and Fe*	Rough, bumpy surface with very few pits (Figure 22a)	Similar to UPW-85° C soak (Figure 22c)
CPVC	C, O, Cl ^a , Al, Si, Ti, Fe*	Same as before. No Al, Si and Fe*	Slightly rough surface, parallel line may be extrusion marks. Sub-micron pits and particles (Figure 23a)	Minimal change; possibly fewer particles (Figure 23c)
ECTFE	C, F ^a , Cl, Fe*	No discernible change, No Fe*	Smooth surface; occasional scratches (Figure 24a)	No discernible change (Figure 24c)
PVDF	C, F ^a	No discernible change, no Fe*	Smooth surface (Figure 25a)	No discernible change (Figure 25c)

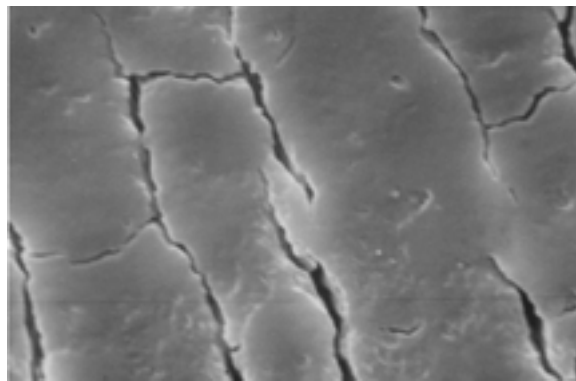
a: major; Fe*: may be an impurity incorporated during cutting of the plastic materials.



a) Original PP surface
(magnification: 3.87×10^3)

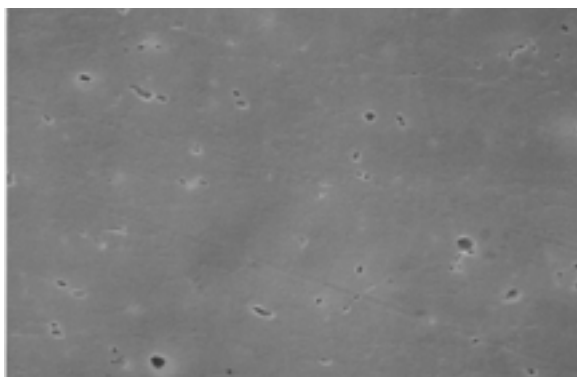


b) PP surface after interacting with UPW
(magnification: 3.94×10^3)



c) PP surface after interacting with ozonated UPW (magnification: 4.90×10^3)

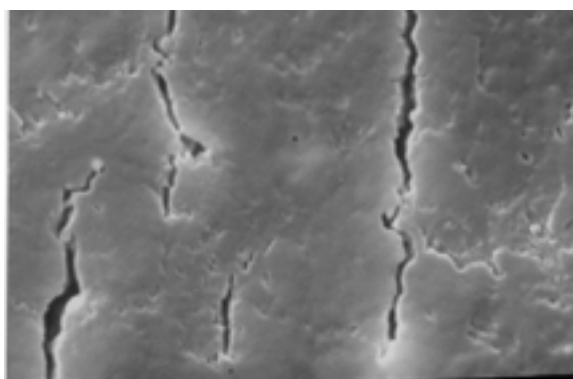
Figure 20 Polypropylene Surface Topography by SEM Before and After Interacting with UPW for 30 Days and Ozonated UPW for 24 Hours



a) Original FRPP surface
(magnification: 3.68×10^3)

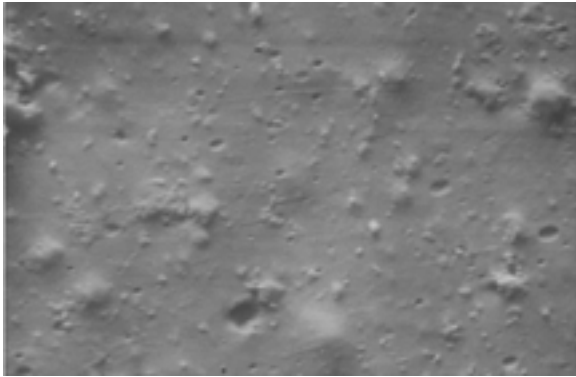


b) FRPP surface after interacting with UPW
(magnification: 7.77×10^3)

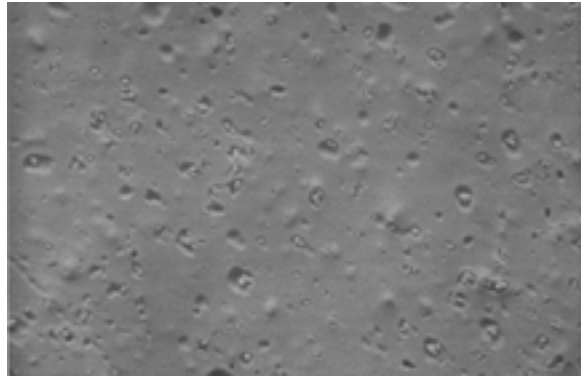


c) FRPP surface after interacting with Ozonated UPW (magnification: 6.36×10^3)

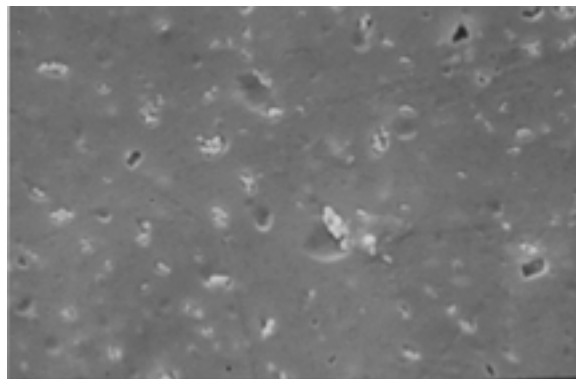
Figure 21 Fire Retarded Polypropylene Surface Topography by SEM Before and After Interacting with UPW for 30 Days and Ozonated UPW for 24 Hours



a) Original PVC surface (magnification: 3.87×10^3)

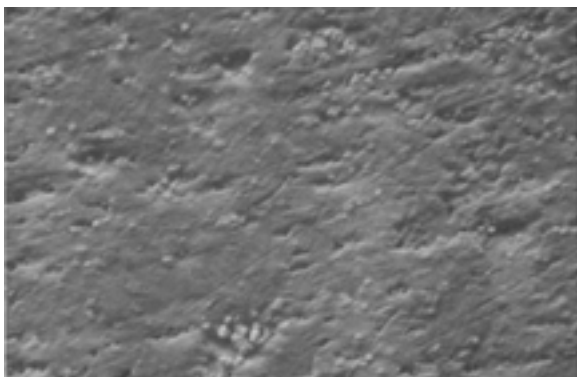


b) PVC surface after interacting with UPW (magnification: 3.80×10^3)

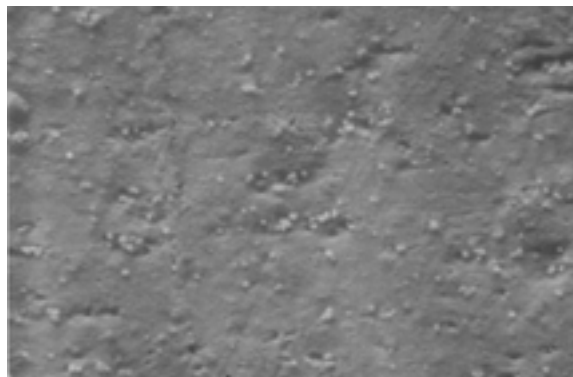


c) PVC surface after interacting with ozonated UPW (magnification: 5.52×10^3)

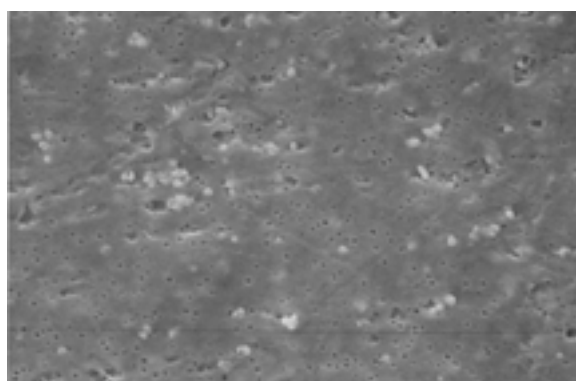
Figure 22 Poly (vinylchloride) Surface Topography by SEM Before and After Interacting with UPW for 30 Days and Ozonated UPW for 24 Hours



a) Original CPVC surface
(magnification: 3.94×10^3)

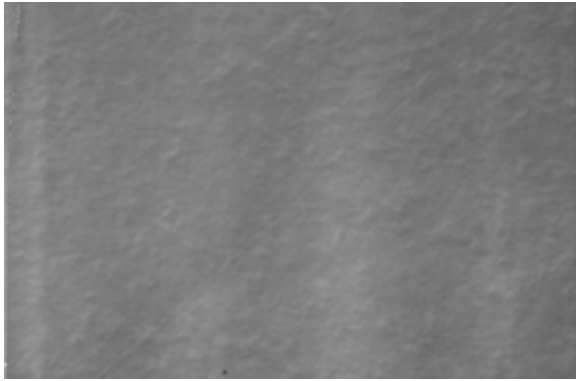


b) CPVC surface after interacting with UPW
(magnification: 3.94×10^3)

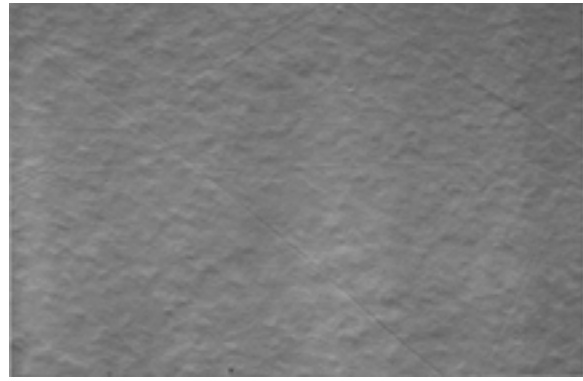


c) CPVC surface after interacting with ozonated UPW (magnification: 5.84×10^3)

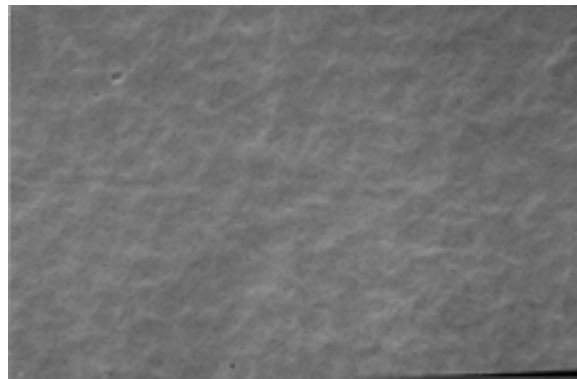
Figure 23 Chlorinated Poly(vinylchloride) Surface Topography by SEM Before and After Interacting with UPW for 30 Days and Ozonated UPW for 24 Hours



a) Original ECTFE surface
(magnification: 3.80×10^3)

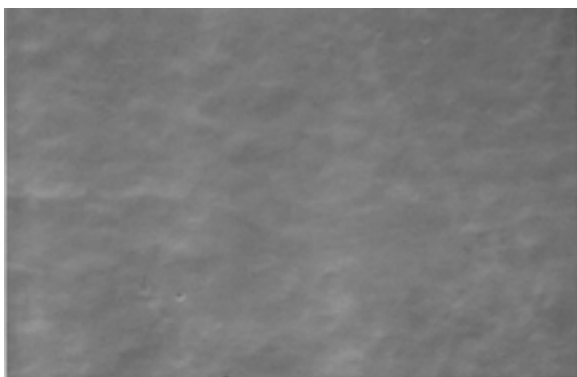


b) ECTFE surface after interacting with UPW
(magnification: 3.05×10^3)

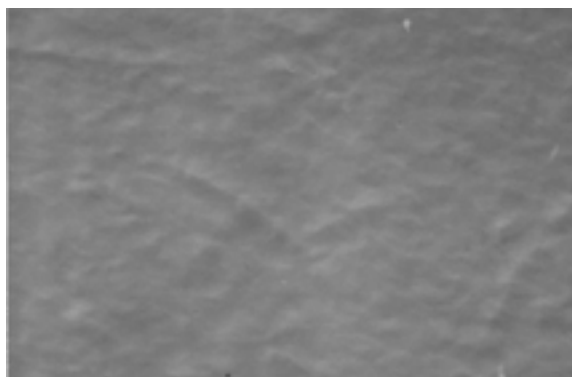


c) ECTFE surface after interacting with ozonated UPW (magnification: 6.25×10^3)

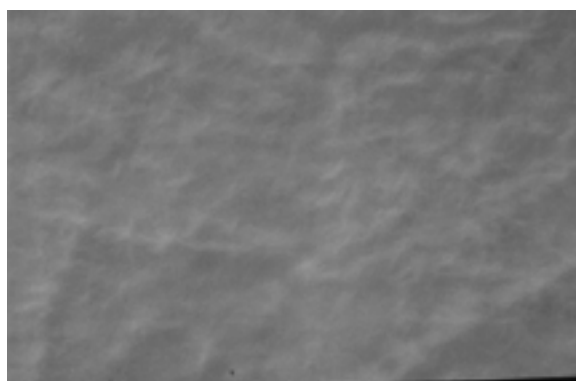
Figure 24 Poly(ethylenechlorotrifluoroethylene) Surface Topography by SEM Before and After Interacting with UPW for 30 Days and Ozonated UPW for 24 Hours



a) Original PVDF surface
(magnification: 3.61×10^3)



b) PVDF surface after interacting with UPW
(magnification: 3.31×10^3)



c) PVDF surface after interacting with ozonated UPW (magnification: 4.81×10^3)

Figure 25 Poly(vinylidene fluoride) Surface Topography by SEM Before and After Interacting with UPW for 30 Days and Ozonated UPW for 24 Hours

5.6 Change in Mass of the Plastic Materials in the Leaching Tests

Change in mass of the plastic materials in the leaching tests measured by direct weighing, which is listed in Table 13, is used to augment the LP.

Table 13 Change in Mass of the Plastic Materials in the Leaching Tests^a

Mass	PP	FRPP	PVC	CPVC	ECTFE	PVDF
UPW at 85°C and Soaking Period of 30 Days (Tests for Leachable Ions (2 coupons in 150 mL UPW))						
Initial (μg) $\times 10^{-6}$	21.420	28.140	30.360	35.000	39.140	24.610
Final (μg) $\times 10^{-6}$	21.400	28.110	30.320	35.200	38.960	24.590
Surface Area (cm^2)	110	130	110	110	120	110
Change ($\mu\text{g}/\text{cm}^2$) ^a	-182	-231	-364	1818	-1500	-182
% change of mass	-0.093	-0.11	-0.13	0.57	-0.46	-0.081
Ozonated UPW at Room Temperature and Soaking Period of 24 hours (4 coupons in about 200 mL UPW)						
Initial (μg) $\times 10^{-6}$	20.539	26.303	30.228	34.838	37.190	24.367
Final (μg) $\times 10^{-6}$	20.532	26.281	30.313	34.294	37.195	24.369
Surface Area (cm^2)	107	123	108	108	119	101
Change ($\mu\text{g}/\text{cm}^2$) ^a	-65	-179	787	-5032	42	20
% change of mass	-0.034	-0.084	0.29	-1.6	0.013	0.0080

a: positive number indicate mass gain and negative number indicate mass loss

PP and FRPP show mass loss with both UPW and ozonated UPW leaching. PVC, ECTFE, and PVDF show mass loss with UPW and mass gain with ozonated UPW. CPVC on the other shows mass gain with UPW and mass loss with ozonated UPW.

Mass loss or gain may indicate various types of interactions between UPW and/or ozone and the plastic materials. Mass loss of a plastic material could be a hydrolysis process, breaking of bonds, or dissolving of additives [7]. The physical properties of the plastic materials may change (e.g., tensile strength) [8]. Mass gain could be due to water penetrating the material matrix through spaces in the chains of the polymer or hydration of the additives. The plastic material may soften, swell, and gain weight (anything over 2% is cause for concern [8]). In the leaching test, none of the plastic materials, except CPVC, showed mass gain or loss close to 2%.

6 EXTRACTION OF ELEMENTS FROM PLASTIC MATERIALS BY CHEMICALS

Extraction is defined as the removal of elements from the plastic materials by chemicals typically used in the wafer processing in cleanrooms. The amounts of elements measured by ICP are normalized by the total exposed surface area of the sample coupon. All the extraction test results⁸ in this report are presented in $\mu\text{g}/\text{cm}^2$.

⁸ ng = 10^{-9} g; μg = 10^{-6} g. To convert ng to μg , multiply ng by 10^{-3}

6.1 Extraction Data

Extraction data are summarized in Table 14 with details listed in Table 25 through Table 29 in Appendix B.

Table 14 Amounts of Elements Extracted from the Plastic Materials by Chemical

Chemicals	Elements	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$					
		PP	FRPP	PVC	CPVC	ECTFE	PVDF
APM (SC1)	Total	8.0	130	780	590	11	17
	Critical ^a	6.1	55	770	570	9.2	16
HPM (SC2)	Total	20	1,100	820	194	4.0	29
	Critical ^a	19	64	800	180	3.2	26
SPM (Piranha)	Total	36	5,000	1,100	560	11	31
	Critical ^a	36	4,600	1,100	490	11	31
70% w/w HNO ₃	Total	9.4	1,000	890	660	9.7	26
	Critical ^a	9.4	90	880	510	7.9	25
49% w/w HF	Total	4,900	68,000	400,000	900,000	280	320
	Critical ^a	4,900	3,000	400,000	900,000	280	320

a: The term “critical” used in this report is defined by the NTRS [4] and SEMI [5] (see Table 2).

6.2 Extraction Parameter

The extraction parameter (EP) is defined as the mass of the critical elements extracted by chemicals typically used in the wafer processing in cleanrooms, in μg per unit total exposed surface area of the plastic material in cm^2 . The amounts of the critical elements listed in Table 14 are taken as the EP values of the plastic materials for each chemical. These EP values are listed in Table 15 and are plotted in Figure 26 through Figure 30.

Table 15 Values of the Extraction Parameter for the Plastic Materials

Chemicals	Extraction Parameter ($\mu\text{g}/\text{cm}^2$)					
	PP	FRPP	PVC	CPVC	ECTFE	PVDF
APM (SC1)	0.0061	0.055	0.77	0.57	0.0092	0.016
HPM (SC2)	0.019	0.064	0.80	0.18	0.0032	0.026
SPM (Piranha)	0.036	4.6	1.1	0.49	0.011	0.031
70% w/w HNO ₃	0.0094	0.090	0.88	0.51	0.0079	0.025
49% w/w HF	4.9	3.0	400	900	0.28	0.32

Note: Solutions initially at the mixing temperature of the chemicals, decaying to ambient temperature during the extraction period of 30 ± 2 days; EP = total mass of the critical elements extracted/total exposed surface area of the plastic material.

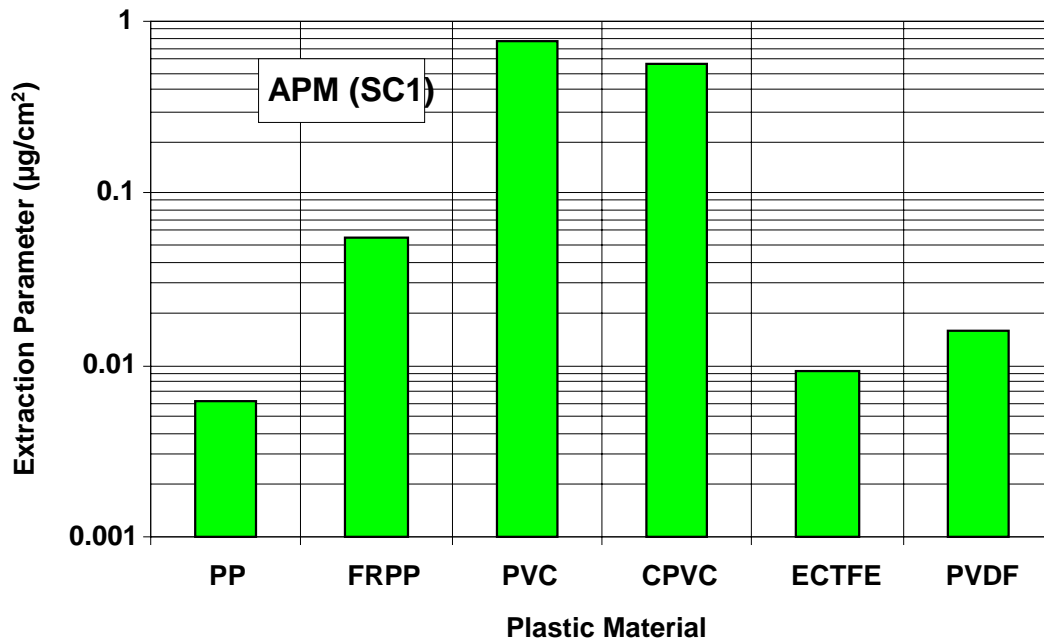


Figure 26 Values of the Extraction Parameter for the Plastic Materials with APM (SC1) Initially at the Mixing Temperature of the Chemicals and Decreasing to Ambient Temperature During the 30-Day Extraction Period

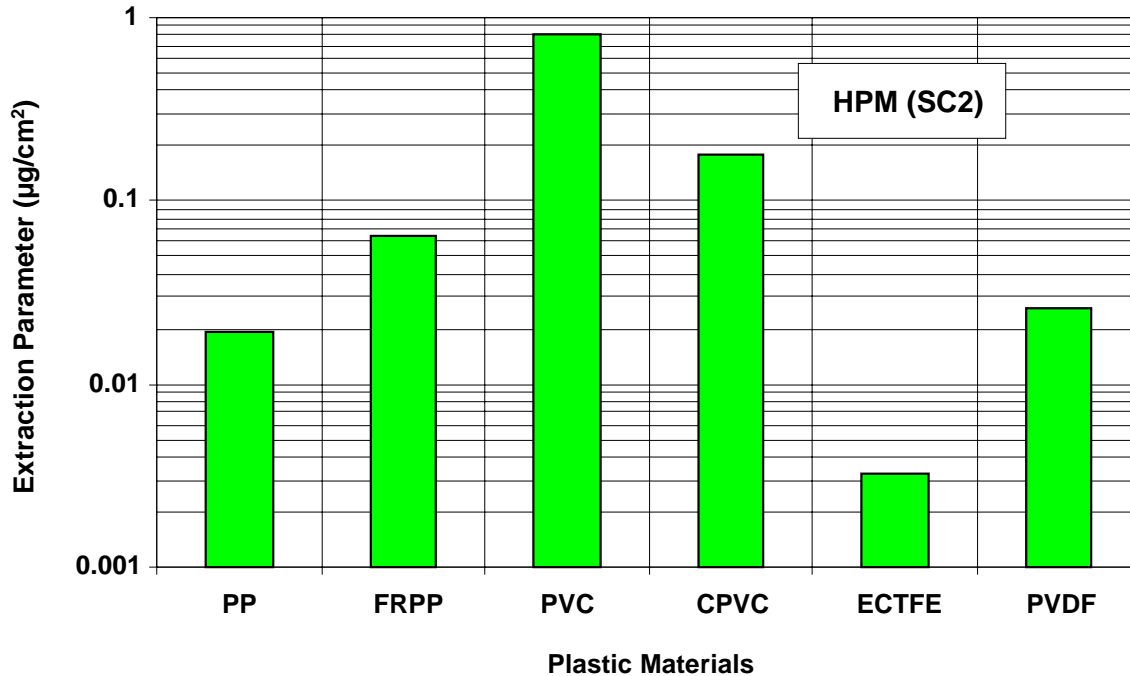


Figure 27 Values of the Extraction Parameter for the Plastic Materials with HPM (SC2) Initially at the Mixing Temperature of the Chemicals and Decreasing to Ambient Temperature During the 30-Day Extraction Period

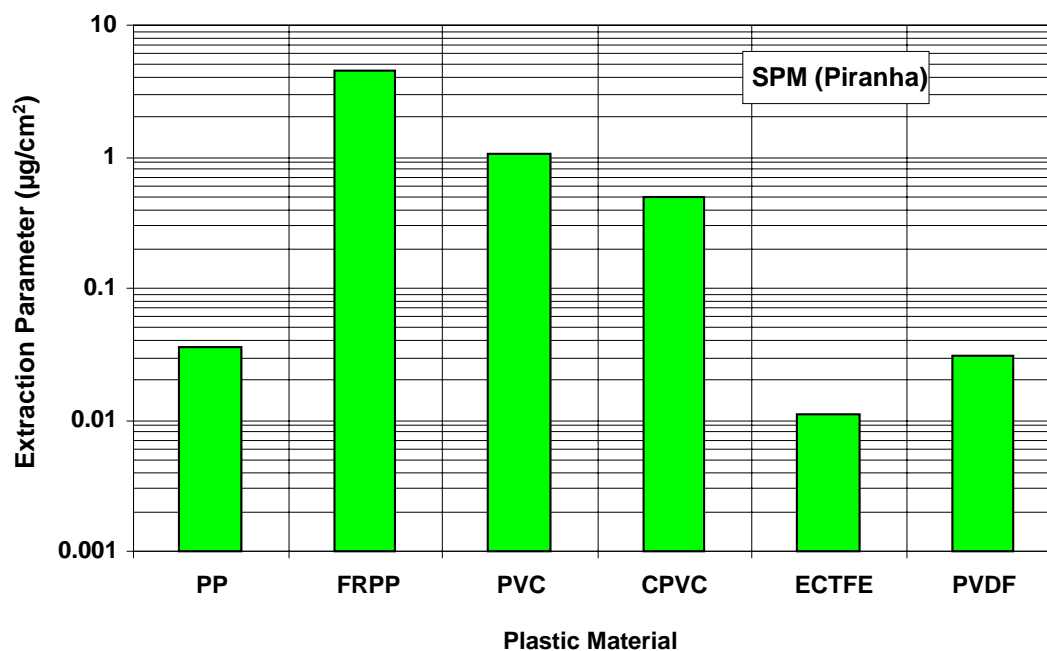


Figure 28 Values of the Extraction Parameter for the Plastic Materials with SPM (Piranha) Initially at the Mixing Temperature of the Chemicals and Decreasing to Ambient Temperature During the 30-Day Extraction Period

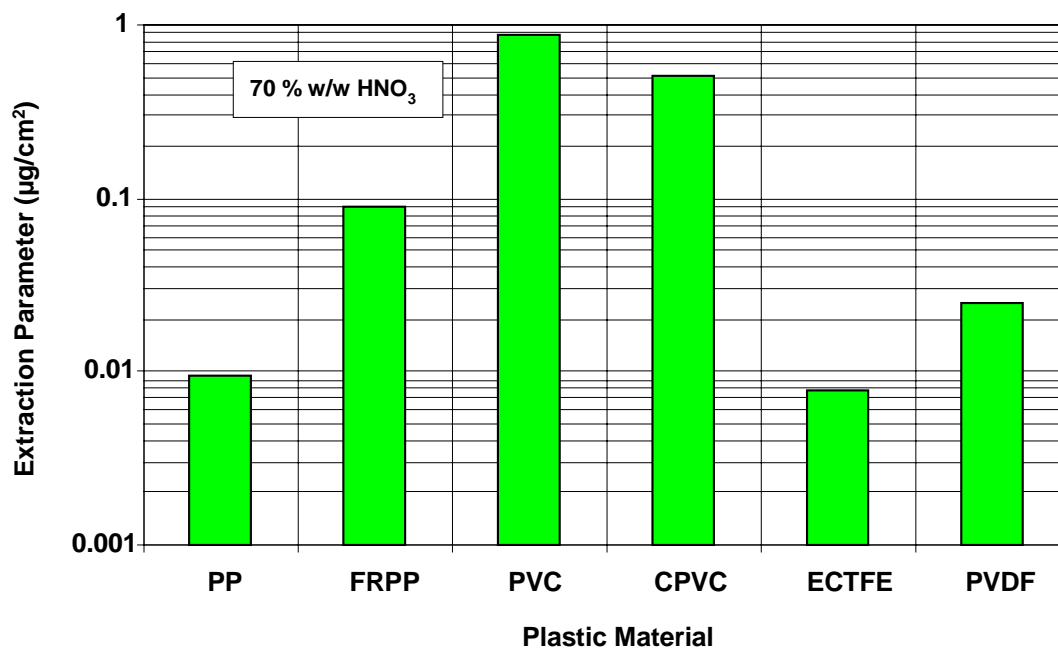


Figure 29 Values of the Extraction Parameter for the Plastic Materials with 70% HNO_3 Initially at the Mixing Temperature of the Chemicals and Decreasing to Ambient Temperature During the 30-Day Extraction Period

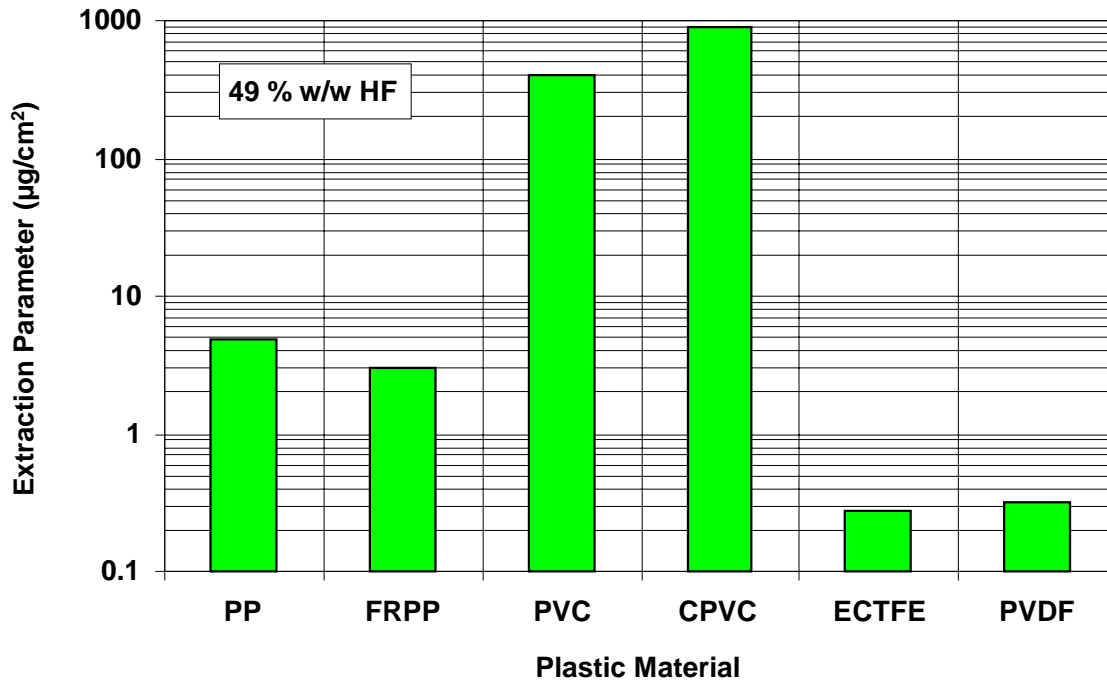


Figure 30 Values of the Extraction Parameter for the Plastic Materials with 49% HF Initially at the Mixing Temperature of the Chemicals and Decreasing to Ambient Temperature During the 30-Day Extraction Period

The EP values with APM (SC1), HPM (SC2), SPM (Piranha), and HNO₃ acid for the proposed materials are lower than the values for FRPP and PVC. The EP values with HF show some differences for the proposed plastics. The EP values with HF for ECTFE and PVDF are lower than the values for the common wet bench plastic materials (PP, FRPP, and PVC). The EP value of CPVC with HF, however, is higher than the values for PP, FRPP, and PVC. The high EP value for CPVC appears to be due to extraction of large amounts of titanium, aluminum, and sodium by HF.

6.3 Surface Conditions and Topographic Examination for Plastic Materials Before and After the Extraction Tests

The surface conditions observed visually are listed in Table 16 and topography examination results by SEM/EDX are listed in Table 17 and are shown in Figure 31 through Figure 36. The visual observations (Table 16) indicate no change in the color of the surface in the interactions of plastic materials with APM (SC1), HPM (SC2), and SPM (Piranha). With HNO₃, the color of the surface of all the plastic materials changes to light gray or yellow. With HF, no change in color occurs for ECTFE and PVDF, while the surfaces of other plastic materials change color.

The surface topographic examination (Figure 31 through Figure 36 and Table 17) indicates that the damage to the surface of the proposed materials in the extraction tests is not as severe as it is with common wet bench materials. The EDX results (Table 17) indicate no critical elements are extracted to the surface in the extraction tests.

Table 16 Surface Condition of the Plastic Materials Before and After the Extraction Tests Observed Visually

Material	Before Extraction	After Extraction				
		APM (SC1)	HPM (SC2)	SPM (Piranha)	70% HNO ₃	49% HF
PP	White with slight blue tint	No visible change	No visible change	No visible change	Turns light gray	Lost bluish tint
FRPP	White	No visible change	No visible change	No visible change	Turns light yellow	Turns light yellow
PVC	Light bluish-white	No visible change	No visible change	No visible change	Turns yellowish white	Turns light gray
CPVC	Light yellow-cream color	No visible change	No visible change	No visible change	Turns light yellow	Turns light brown
ECTFE	Dull white-yellowish translucent	No visible change	No visible change	No visible change	Turns light yellow	No visible change
PVDF	Translucent	No visible change	No visible change	No visible change	Turns light yellow	No visible change

Table 17 EDX and Surface Topographic Examination Results for Six Plastic Test Materials in the Extraction Tests

Chemical	EDX Results		SEM Results	
	Before	After	Before	After
PP				
APM (SC1)	C ^a , O, Ti	Same as before	Smooth surface with small bumps (Figure 31a)	Smooth surface (Figure 31b)
HPM (SC2)	C ^a , O, Ti	Same as before	Smooth surface with small bumps (Figure 31a)	Lines are visible (Figure 31c)
SPM (Piranha)	C ^a , O, Ti	Same as before	Smooth surface with small bumps (Figure 31a)	Sub-micron pits and lines are visible on the surface (Figure 31d)
70% HNO ₃	C ^a , O, Ti	Same as before	Smooth surface with small bumps (Figure 31a)	Minimal change. Parallel lines and some pits are visible (Figure 31e)
49% HF	C ^a , O, Ti	Same as + F (from HF)	Smooth surface with small bumps (Figure 31a)	Parallel lines still visible (Figure 31f)
FRPP				
APM (SC1)	C ^a , O, Al, Br, Sb, Fe*	Same as before, no Fe*	Slightly rough surface; some pits no crystals on surface (Figure 32a)	Many elongated structures, but fewer than UPW-85°C soaks (Figure 32b and Figure 21b)
HPM (SC2)	C ^a , O, Al, Br, Sb, Fe*	Same as before, no Fe*	Slightly rough surface; some pits, no crystals on surface (Figure 33a)	Similar to APM-soak (Figure 32c same as Figure 32b)
SPM (Piranha)	C ^a , O, Al, Br, Sb, Fe*	Same as before, S (from H ₂ SO ₄), Ti, no Fe*	Slightly rough surface; some pits, no crystals on surface (Figure 32a)	Extensive surface damage, cracks and pits (Figure 32d)
70% HNO ₃	C ^a , O, Al, Br, Sb, Fe*	Same as before, no Fe*	Slightly rough surface; some pits, no crystals on surface (Figure 32a)	Similar to APM-soak (Figure 32e and Figure 32b)
49% HF	C ^a , O, Al, Br, Sb, Fe*	Same as before (Al/Br major) + F (from HF), no Fe*	Slightly rough surface; some pits, no crystals on surface (Figure 32a)	Extensive elongated structures with many pits (Figure 32f)

Chemical	EDX Results		SEM Results	
	Before	After	Before	After
PVC				
APM (SC1)	C, O, Cl ^a , Ca, Ti, Fe*	Same as before, no Ca	Rough bumpy surface with very few pits (Figure 33a)	Similar to UPW-85°C soak (Figure 33b). Leaching out of material and pits on the surface with particles in the pits (Figure 22b)
HPM (SC2)	C, O, Cl ^a , Ca, Ti, Fe*	Same as before, no Ca and Fe*	Rough bumpy surface with very few pits (Figure 33a)	Similar to UPW-85°C soak (Figure 33c). Leaching out of material and pits on the surface with particles in the pits (Figure 22b)
SPM (Piranha)	C, O, Cl ^a , Ca, Ti, Fe*	Same as before, no Ca and Fe*	Rough bumpy surface with very few pits (Figure 33a)	Similar to UPW-85°C soak (Figure 33d). Leaching out of material and pits on the surface with particles in the pits (Figure 22b)

Table 17 EDX and Surface Topographic Examination Results for Six Plastic Test Materials in the Extraction Tests (continued)

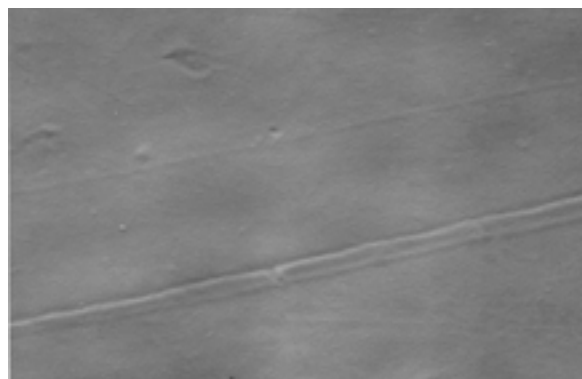
Chemical	EDX Results		SEM Results	
	Before	After	Before	After
70% HNO ₃	C, O, Cl ^a , Ca, Ti, Fe*	Same as before, no Ca and Fe*	Rough bumpy surface with very few pits (Figure 33a).	Similar to UPW-85°C soak (Figure 33e). Leaching out of material and pits on the surface with particles in the pits (Figure 22b).
49% HF	C, O, Cl ^a , Ca, Ti, Fe*	Same as before + F (from HF); no Ca and Fe*	Rough bumpy surface with very few pits (Figure 33a).	Pits on the surface, but no particles (Figure 33f)
CPVC				
APM (SC1)	C, O, Cl ^a , Al, Si, Ti, Fe*	Same as before, no Al, and Si	Slightly rough surface, parallel line present, which may be extrusion marks. Sub-micron pits and particles are present. (Figure 34a)	No discernible change (Figure 34b).
HPM (SC2)	C, O, Cl ^a , Al, Si, Ti, Fe*	Same as before, no Al, Si, and Fe*	Slightly rough surface, parallel line present, which may be extrusion marks. Sub-micron pits and particles are present (Figure 34a).	Surface smoother, fewer particles (Figure 34c).
SPM (Piranha)	C, O, Cl ^a , Al, Si, Ti, Fe*	Same as before, no Al and Fe*	Slightly rough surface, parallel line present, which may be extrusion marks. Sub-micron pits and particles are present (Figure 34a).	Some surface pitting, some surface deposition; lines are still visible (Figure 34d).
70% HNO ₃	C, O, Cl ^a , Al, Si, Ti, Fe*	Same as before, no Al, Si, and Fe*	Slightly rough surface, parallel line present, which may be extrusion marks. Sub-micron pits and particles are present (Figure 34a).	Some surface pitting, some surface deposition (Figure 34e).

Chemical	EDX Results		SEM Results	
	Before	After	Before	After
49% HF	C, O, Cl ^a , Al, Si, Ti, Fe*	Same as before + F (from HF); no Al, Si, and Fe*	Slightly rough surface, parallel line present, which may be extrusion marks. Sub-micron pits and particles are present (Figure 34a).	Some change of surface appearance (Figure 34f).
ECTFE				
APM (SC1)	C, F ^a , Cl, Fe*	No discernible change.	Smooth surface; occasional scratches (Figure 35a).	No discernible change (Figure 35b).
HPM (SC2)	C, F ^a , Cl, Fe*	No discernible change; No Fe*	Smooth surface; occasional scratches (Figure 35a)	No discernible change (Figure 35c)
SPM (Piranha)	C, F ^a , Cl, Fe*	No discernible change	Smooth surface; occasional scratches (Figure 35a)	No discernible change (Figure 35d)
70% HNO ₃	C, F ^a , Cl, Fe*	No discernible change	Smooth surface; occasional scratches (Figure 35a)	No discernible change (Figure 35e)
49% HF	C, F ^a , Cl, Fe*	No discernible change	Smooth surface; occasional scratches (Figure 35a)	No discernible change (Figure 35f)

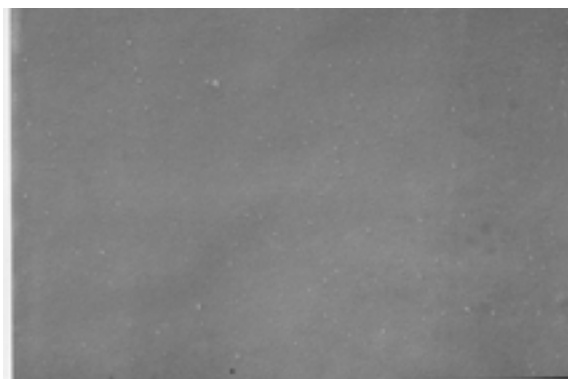
Table 17 EDX and Surface Topographic Examination Results for Six Plastic Test Materials in the Extraction Tests (continued)

Chemical	EDX Results		SEM Results	
	Before	After	Before	After
PVDF				
APM (SC1)	C, F ^a	No discernible change	Smooth surface (Figure 36a)	No discernible change (Figure 36b)
HPM (SC2)	C, F ^a	No discernible change; no Fe*	Smooth surface (Figure 36a)	No discernible change (Figure 36c)
SPM (Piranha)	C, F ^a	No discernible change	Smooth surface (Figure 36a)	No discernible change (Figure 36d)
70% HNO ₃	C, F ^a	No discernible change	Smooth surface (Figure 36a)	No discernible change (Figure 36e)
49% HF	C, F ^a	No discernible change	Smooth surface (Figure 36a)	No discernible change (Figure 36f)

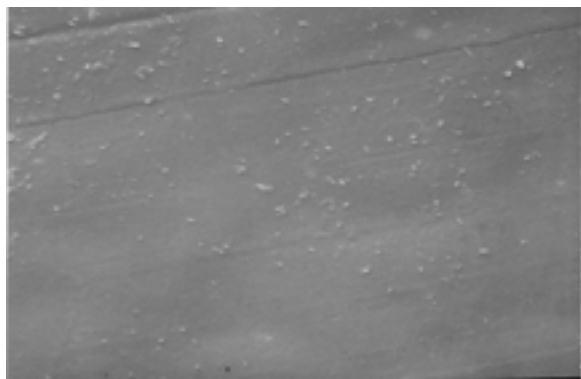
a: major element; Fe*: may be an impurity incorporated during cutting of the plastic materials.



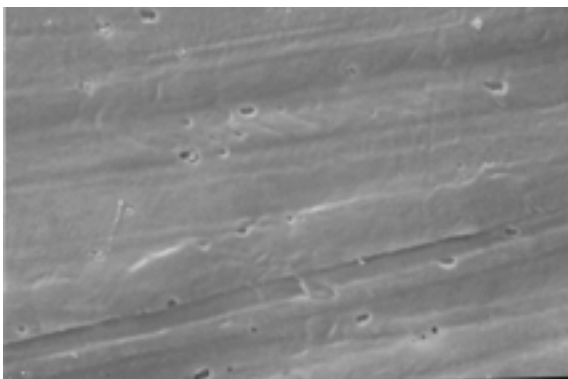
a) before interacting with the chemicals for 30 days
(magnification 3.87×10^3)



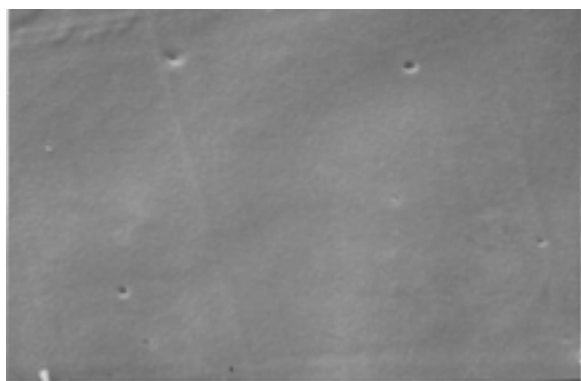
b) after interacting with APM (SC1) for 30 days
(magnification 4.20×10^3)



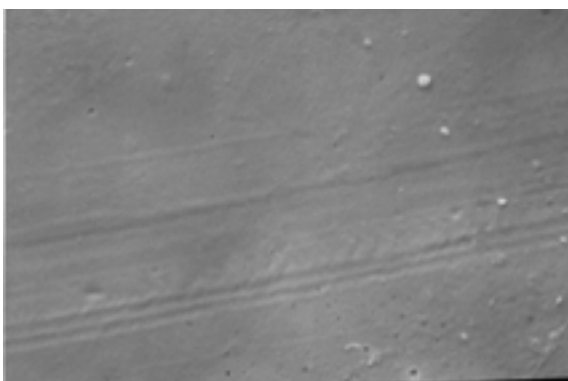
c) after interacting with HPM (SC2) for 30 days
(magnification 5.09×10^3)



d) after interacting with SPM (Piranha) for 30 days
(magnification 4.67×10^3)

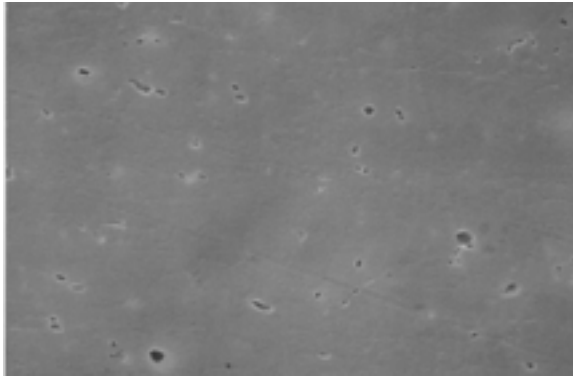


e) after interacting with HNO₃ for 30 days
(magnification 6.69×10^3)

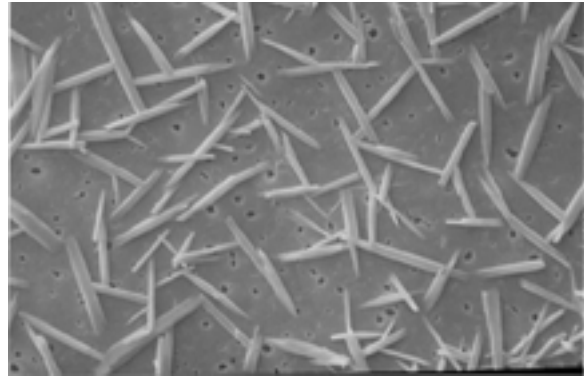


f) after interacting with HF for 30 days
(magnification 5.35×10^3)

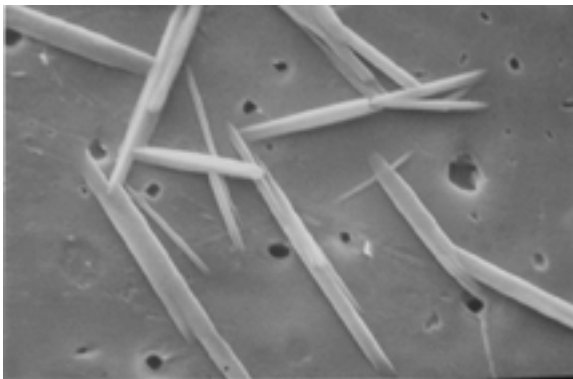
Figure 31 Polypropylene Surface Topography by SEM After Interacting with Solutions of Chemicals for 30 Days



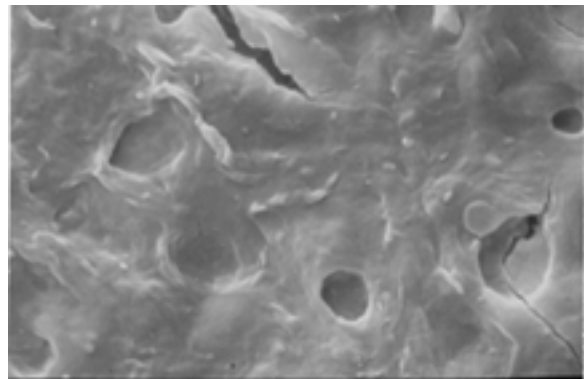
a) before interacting with the chemicals for 30 days
(magnification 3.68×10^3)



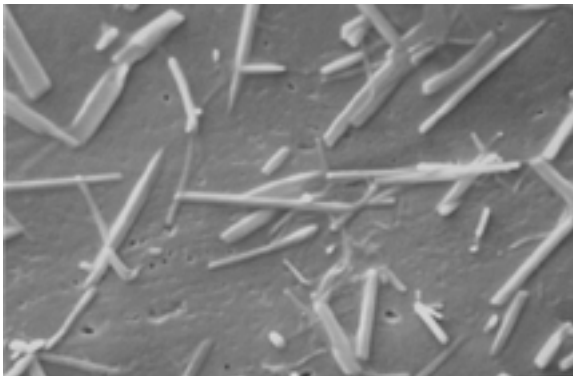
b) after interacting with APM (SC1) for 30 days
(magnification 2.39×10^3)



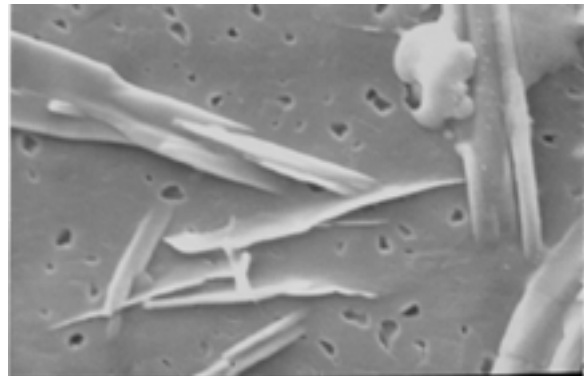
c) after interacting with HPM (SC2) for 30 days
(magnification 4.56×10^3)



d) after interacting with SPM (Piranha) for 30 days
(magnification 4.90×10^3)

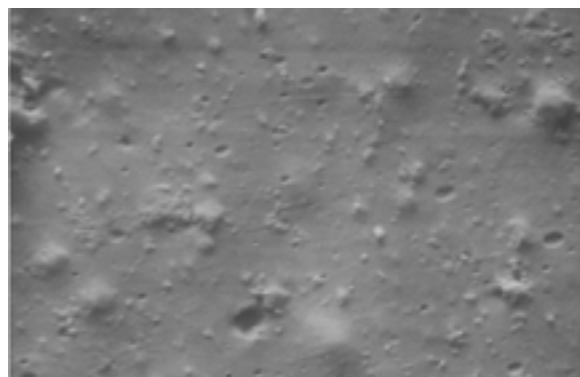


e) after interacting with HNO_3 for 30 days
(magnification 6.10×10^3)

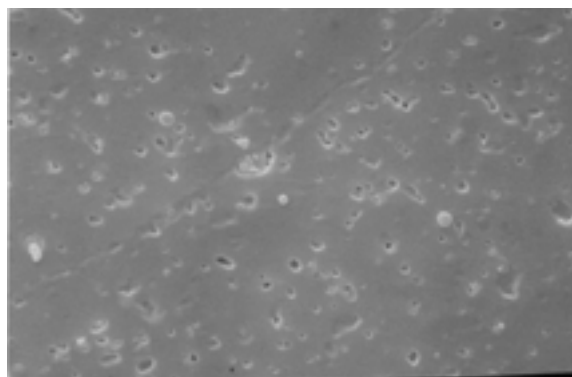


f) after interacting with HF for 30 days
(magnification 5.52×10^3)

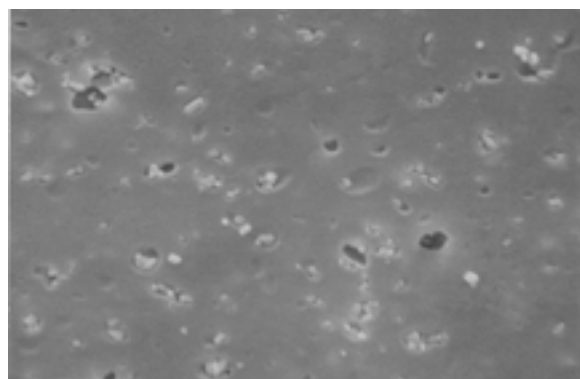
Figure 32 Fire Retardant Polypropylene Surface Topography by SEM After Interacting with Solutions of Chemicals for 30 Days



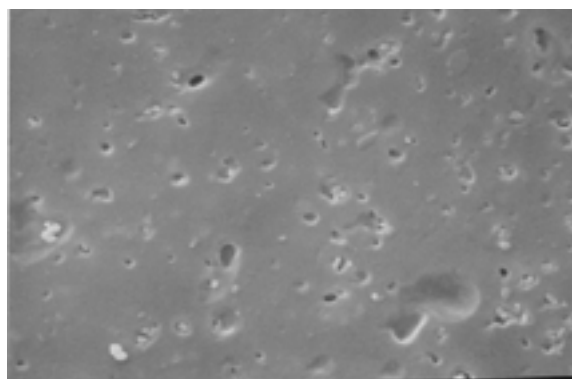
a) before interacting with the chemicals for 30 days
(magnification 3.87×10^3)



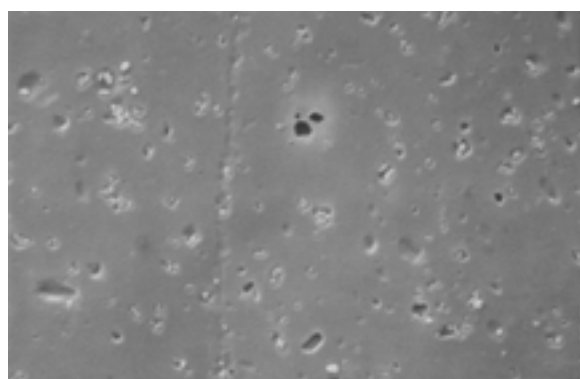
b) after interacting with APM (SC1) for 30 days
(magnification 4.63×10^3)



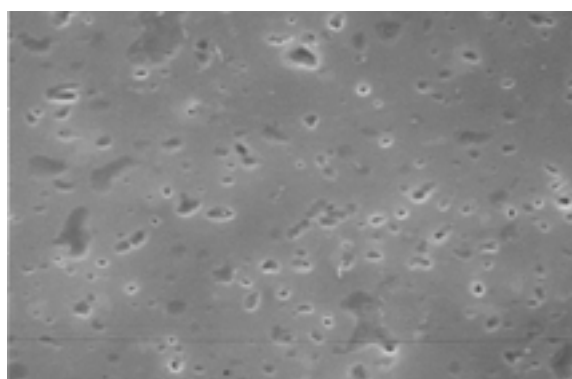
c) after interacting with HPM (SC2) for 30 days
(magnification 6.87×10^3)



d) after interacting with SPM (Piranha) for 30 days
(magnification 6.32×10^3)

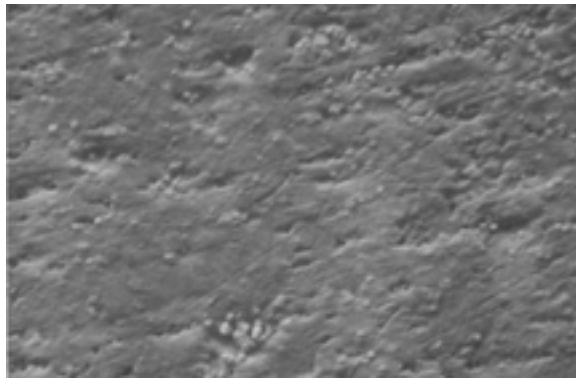


e) after interacting with HNO₃ for 30 days
(magnification 5.52×10^3)

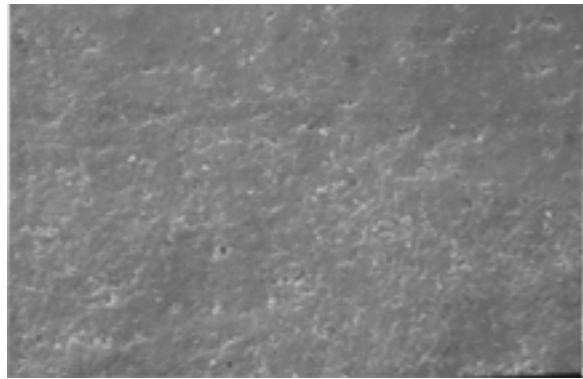


f) after interacting with HF for 30 days
(magnification 4.82×10^3)

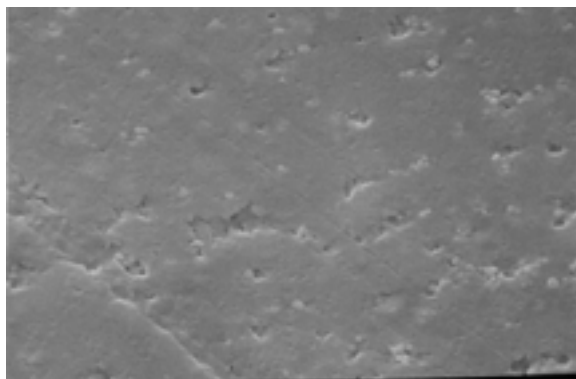
Figure 33 Surface Topography of Poly(vinylchloride) by SEM After Interacting with Solutions of Chemicals for 30 Days



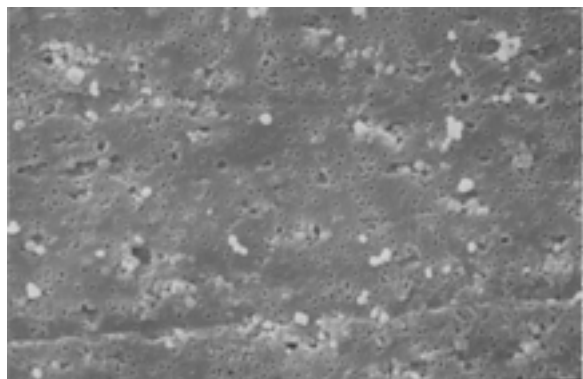
a) before interacting with the chemicals for 30 days
(magnification 3.94×10^3)



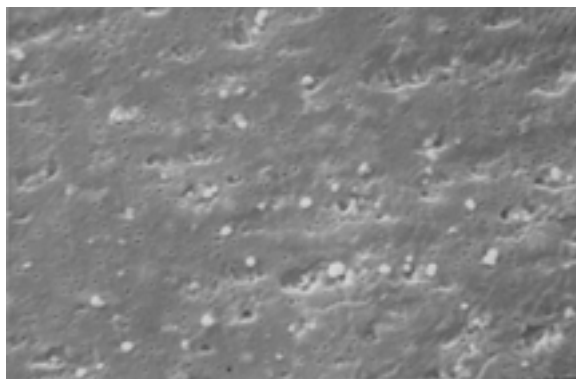
b) after interacting with APM (SC1) for 30 days
(magnification 4.28×10^3)



c) after interacting with HPM (SC2) for 30 days
(magnification 5.84×10^3)



d) after interacting with SPM (Piranha) for 30 days
(magnification 5.55×10^3)



e) after interacting with HNO₃ for 30 days
(magnification 5.55×10^3)



f) after interacting with HF for 30 days
(magnification 5.84×10^3)

Figure 34 Chlorinated Poly(vinylchloride) Surface Topography by SEM After Interacting with Solutions of Chemicals for 30 Days

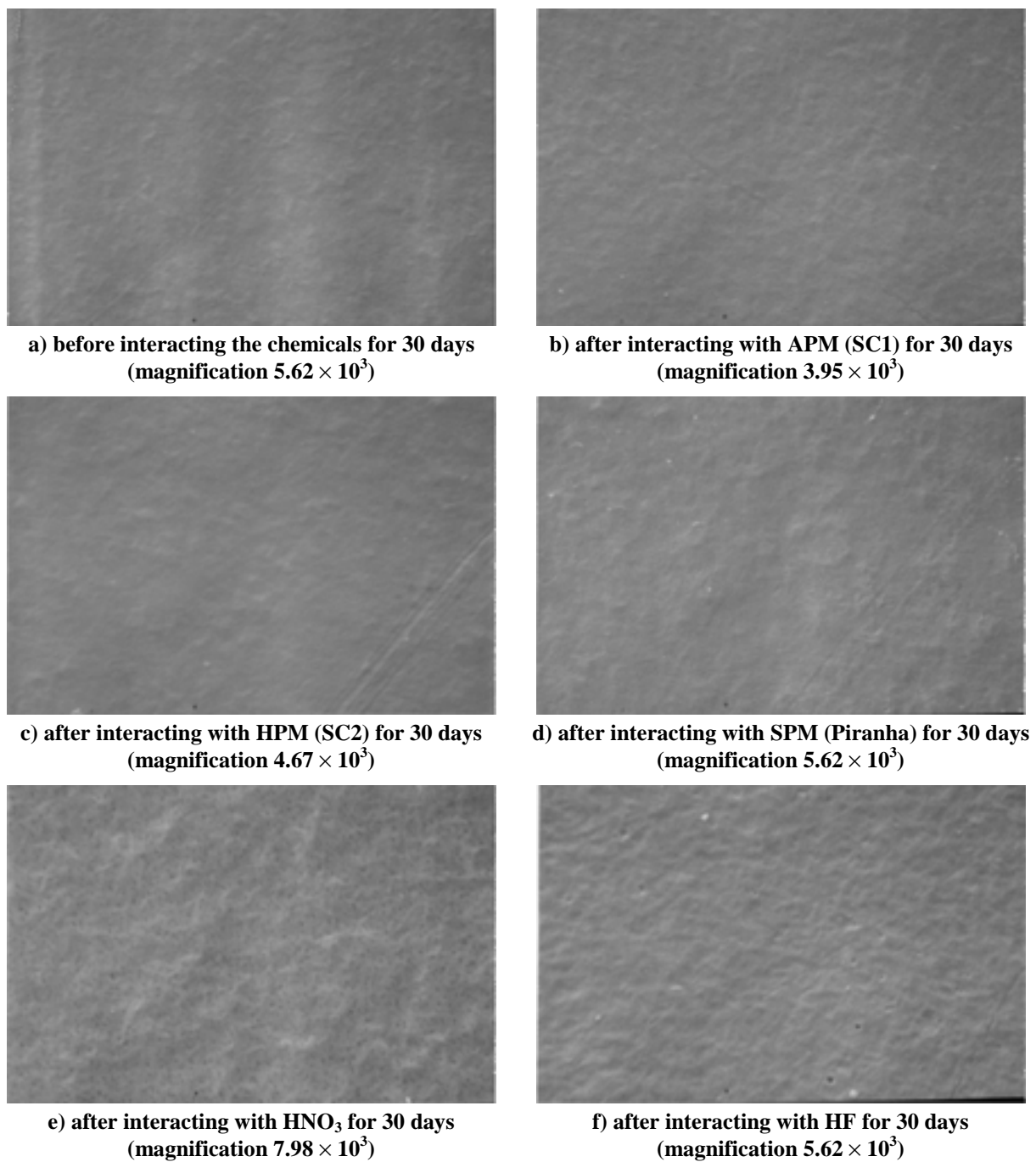
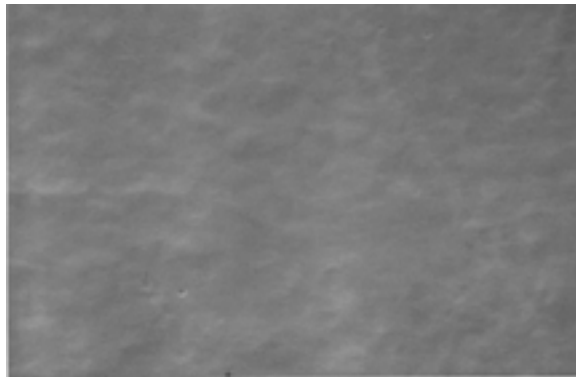
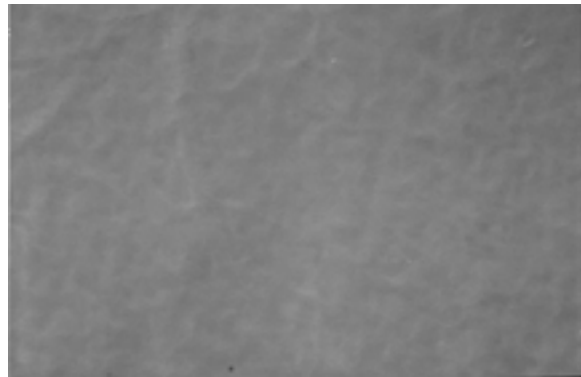


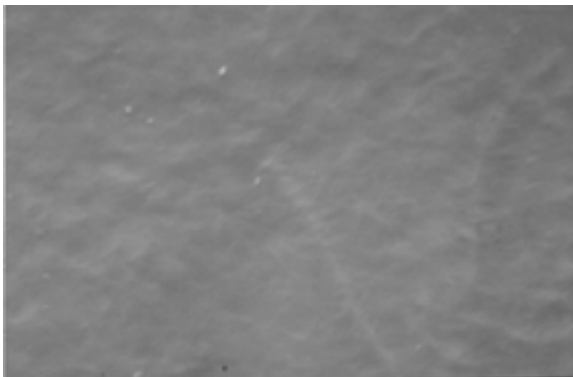
Figure 35 Poly(ethylenechlorotrifluoroethylene) Surface Topography by SEM After Interacting with Solutions of Chemicals for 30 Days



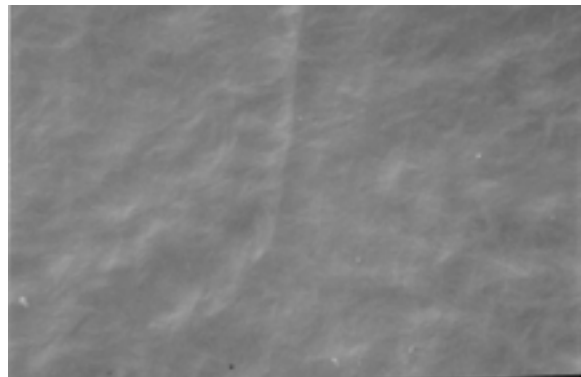
a) before interacting with the chemicals for 30 days
(magnification 3.61×10^3)



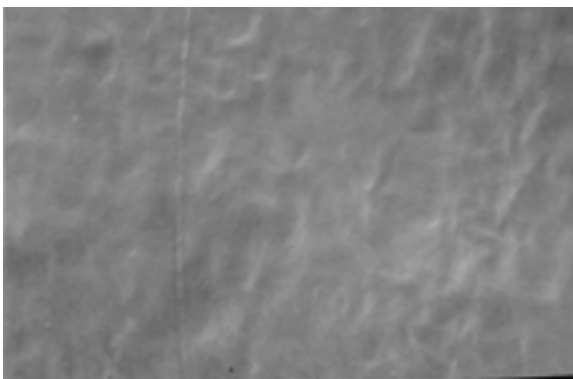
b) after interacting with APM (SC1) for 30 days
(magnification 4.05×10^3)



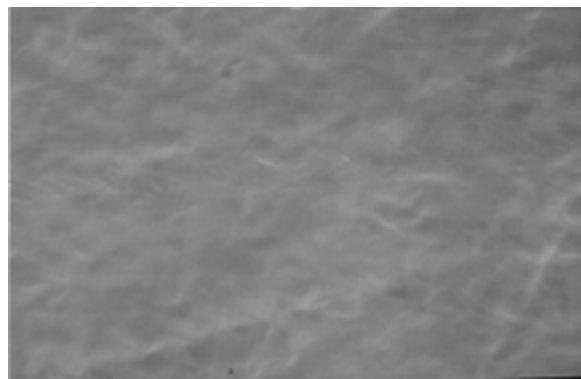
c) after interacting with HPM (SC2) for 30 days
(magnification 4.05×10^3)



d) after interacting with SPM (Piranha) for 30 days
(magnification 6.30×10^3)



e) after interacting with HNO₃ for 30 days
(magnification 7.22×10^3)



f) after interacting with HF for 30 days
(magnification 6.30×10^3)

Figure 36 Poly(vinylidene fluoride) Surface Topography by SEM After Interacting with Solutions of Chemicals for 30 Days

6.4 Mass Change of the Plastic Materials in the Extraction Tests

Changes in mass of the plastic materials in the extraction tests, measured by direct weighing, are listed in Table 18. The mass changes are used to augment the EP data. For all the plastic materials interacting with the chemicals, there is loss of mass, except the interactions of PP and ECTFE with APM (SC1) and the interactions of PP and FRPP with SPM (Piranha).

Table 18 Change in Mass of the Plastic Materials in the Extraction Tests

Mass	PP	FRPP	PVC	CPVC	ECTFE	PVDF
APM (SC1) [1:1:5 (NH₄OH: H₂O₂: H₂O volume ratio)] (Two Coupons)						
Initial (μg) x 10 ⁻⁶	10.148	13.863	15.203	16.813	19.354	12.257
Final (μg) x 10 ⁻⁶	10.151	13.855	15.188	16.801	19.361	12.248
Surface Area (cm ²)	54	62	54	53	59	51
Change (μg/cm ²) ^a	55	-130	-278	-225	118	-178
% mass change	0.030	-0.058	-0.099	-0.071	0.036	-0.073
HPM (SC2) [1:1:6 (HCl : H₂O₂ : H₂O volume ratio)] (Two Coupons)						
Initial (μg) x 10 ⁻⁶	10.211	13.375	15.260	16.831	19.762	12.309
Final (μg) x 10 ⁻⁶	10.203	13.356	15.256	16.680	19.664	12.308
Surface Area (cm ²)	107	123	108	108	119	101
Surface Area (cm ²)	54	62	54	51	57	51
Change (μg/cm ²) ^a	-147	-309	-74	-2961	-1718	-20
% mass change	-0.078	-0.142	-0.026	-0.897	-0.486	-0.008
SPM (Piranha) [5:1 v/v (96% w/w H₂SO₄: 31% w/w H₂O₂)] (Two Coupons)						
Initial (μg) x 10 ⁻⁶	10.197	13.833	15.130	17.328	20.189	12.292
Final (μg) x 10 ⁻⁶	10.201	13.843	15.129	17.298	20.165	12.279
Surface Area (cm ²)	54	60	53	53	59	51
Change (μg/cm ²) ^a	74	166	-19	-570	-404	-257
% mass change	0.039	0.072	-0.007	-0.173	-0.119	-0.106
70% (w/w) HNO₃(Two Coupons)						
Initial (μg) x 10 ⁻⁶	10.232	13.061	15.018	17.489	20.000	12.037
Final (μg) x 10 ⁻⁶	10.202	13.034	15.007	17.459	19.981	12.008
Surface Area (cm ²)	54	60	54	54	56	51
Change (μg/cm ²) ^a	-553	-449	-204	-553	-339	-574
% mass change	-0.293	-0.207	-0.073	-0.172	-0.095	-0.241
49% (w/w) HF(Two Coupons)						
Initial (μg) x 10 ⁻⁶	10.342	14.229	15.332	18.179	23.135	12.207
Final (μg) x 10 ⁻⁶	10.300	14.160	15.257	17.891	23.126	12.165
Surface Area (cm ²)	54	63	54	53	62	51
Change (μg/cm ²) ^a	-774	-1095	-1389	-5405	-146	-831
% mass change	-0.41	-0.49	-0.49	-1.6	-0.039	-0.34

a: positive number indicate mass gain and negative number indicate mass loss

Mass loss or gain in the extraction tests could be due to attack of the chemicals on the plastic materials, breaking the bonds in the backbone resulting in their degradation, or extraction of additives [7]. Mass gain could be due to chemicals penetrating the material matrix through spaces in the chains of the polymer or reactions between the chemicals and the plastic materials or additives. The plastic material could soften, swell, and gain weight (anything over 2% is cause for concern [8]).

The data in Table 18 indicates that for most of the plastic materials, the mass loss or gain is less than 2%, except the interaction of CPVC with HF. It appears that HF extracts unusually high amounts of aluminum, sodium, and titanium from CPVC.

7 DISCUSSION

In this study, a test protocol has been defined consisting of accepted analytical procedures to determine the process compatibility parameters for commonly used and proposed wet bench plastic materials. In this study, three industry standard test methods have been used to determine the outgassing, leaching, and extraction parameters (OP, LP, and EP), defined in combination as the process compatibility parameters.

The actual OP, LP, and EP values obtained in the test protocol may be affected by the fabrication history of the plastic sheets as well as residuals from the polymerization processes or the manufacture of the feedstock resins. Thus, users must be cognizant of the level of processing a given plastic material has undergone before comparing the data with other prior data.

Furthermore, this study focused on plastic materials used in non-wetted wet bench areas; it must be understood that the acceptable levels of contaminants for these wet bench construction materials may be much different than the levels for materials used for wetted parts.

The test data presented in the report for each plastic material are based on a single test for each process. Data repeatability was not established, as the objective was to develop a protocol rather than to provide test data. Also, test data from different parts of a non-uniform plastic material could vary if additives are not well mixed.

7.1 Process Compatibility and Fire Propagation Propensity for the Plastic Materials

Table 19, Table 20, and Figure 37 summarize the data for the process compatibility parameters from this study. Table 30 in Appendix D lists the values of the fire propagating and smoke and corrosive product release propensity (FPI and other properties). Physical properties of the plastic materials are also listed in Appendix D. Table 19 and Figure 37 show that at 100°C accelerated heating of the sample pieces, only a small amount of organic materials (0.01 to 0.31%) is outgassed. Only weakly bonded additives and solvents are outgassed, and chemical bonds between various atoms in the polymer matrix remain intact. The low values of the mass change also reflect the low OP values. The data show that fire retardant additives in PP are weakly bonded.

Data in Table 20 and Figure 37 show that the values of the process compatibility parameters for plastic materials with weakly bonded additives (i.e., FRPP) are highest in almost all cases, except with APM (SC1) and HF. These results also suggest that low values of the process compatibility parameters for leaching and extraction processes (LP and EP) and FPI appear to be inherent properties of the plastic materials having strong chemical bonds with negligible amounts of weakly bonded additives, such as CPVC, ECTFE and PVDF (except for CPVC-HF interaction).

Weakly bonded compounds of titanium, aluminum, and sodium in CPVC are responsible for deviation with HF interaction.

Table 19 Change of Mass (%) for the Plastic Materials after Testing

Agent	Mass Change (w/w) ^a					
	PP	FRPP	PVC	CPVC	ECTFE	PVDF
100°C Heating for 30 minutes (Outgassing Test)						
Helium (Heating)	-0.010	-0.070	(0.00)	-0.25	-0.090	-0.31
Interactions with UPW and Ozonated UPW Leaching Test						
UPW	-0.093	-0.11	-0.13	0.57	-0.46	-0.081
Ozonated UPW	-0.034	-0.084	0.29	-1.6	0.013	0.0080
Interactions with Chemicals, Extraction Test						
APM (SC1)	0.030	-0.058	-0.099	-0.071	0.036	-0.073
HPM (SC2)	-0.078	-0.14	-0.026	-0.90	-0.49	-0.0080
SPM (Piranha)	0.039	0.072	-0.007	-0.17	-0.12	-0.11
HNO ₃	-0.29	-0.21	-0.073	-0.17	-0.095	-0.24
HF	-0.41	-0.49	-0.49	-1.6	-0.039	-0.34

a: positive numbers indicate mass gain and negative numbers indicate mass loss. Number in parenthesis appears to be anomalous.

Table 20 Total Amounts of Ions, Elements, and TOC Leached/ Extracted

Species	Agent	Amount Leached/Extracted (µg/g Initial Weight)					
		PP	FRPP	PVC	CPVC	ECTFE	PVDF
Ions	UPW	0.52	14	1.2	2.8	6.5	3.7
	Ozonated UPW	30	39	21	15	13	16
Elements	UPW	0.51	4.0	3.9	1.6	0.019	0.031
	Ozonated UPW	0.51	40	0.37	0.59	0.017	0.057
	APM (SC1)	0.053	0.57	2.8	1.9	0.041	0.077
	HPM (SC2)	0.12	5.2	2.9	0.60	0.018	0.13
	SPM (Piranha)	0.24	22	3.8	1.7	0.055	0.15
	HNO ₃	0.053	4.7	3.2	2.1	0.027	0.11
	HF	46	310	2600	5000	0.80	1.4
TOC	UPW	48	110	0.36	69	21	7.5
	Ozonated UPW	180	200	0.24	0.16	0.046	1.5

Note: Bold numbers represent the highest value in each category.

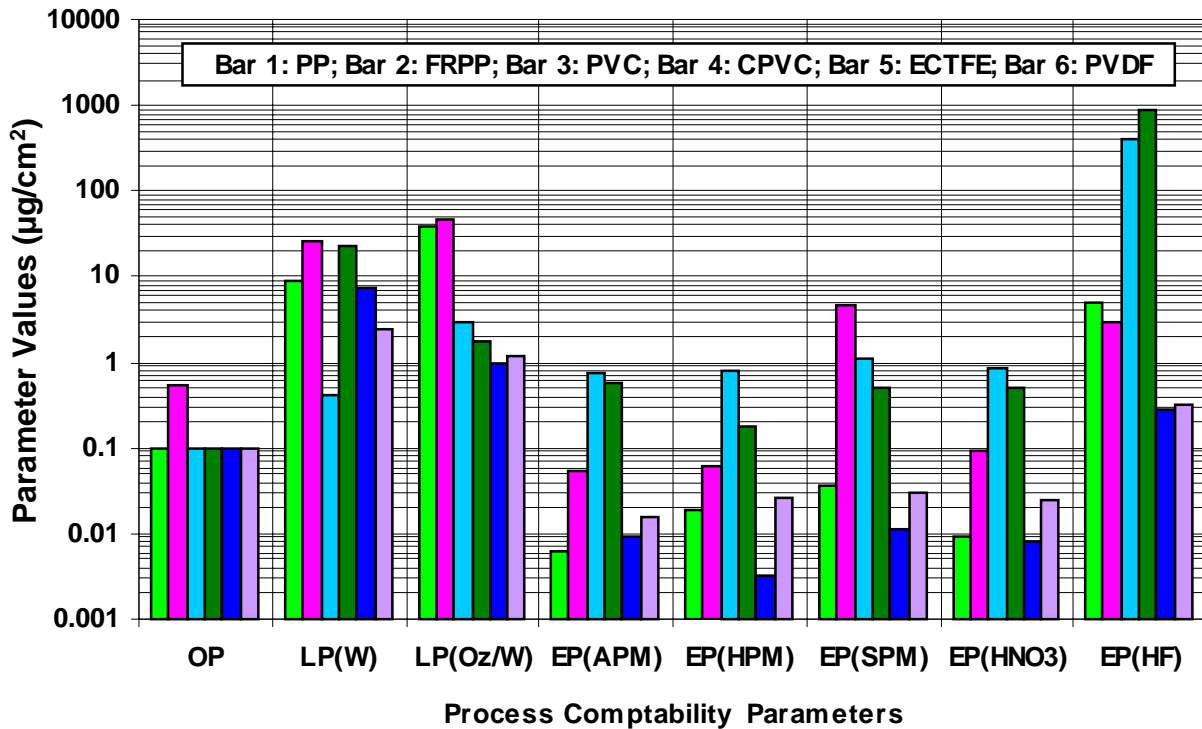


Figure 37 Values of the Process Compatibility Parameters for the Plastic Materials in Use or Proposed to be Used for the Fabrication of Wet Benches in the Wafer Processing in Cleanrooms

Data in Figure 37 shows that the most severe interaction is between the plastic materials and HF, followed by ozonated UPW, UPW and SPM. The chemical interactions between the plastic materials and APM, HPM, and HNO₃ are relatively weak and are about a similar order of magnitude.

7.2 Recommendation

This report presents a technique and test protocol to determine the values of the process compatibility parameters. Inputs from the International SEMATECH member companies suggest that

- The test protocol be used as a part of the guidelines for the purchase of future plastic materials for the fabrication of wet benches.
- The test protocol be presented to SEMI standards group for possible incorporation as a part of a SEMI standards addressing fire propagation behavior and process-compatibility for the plastic materials for fabricating wet benches.
- The test protocol be evaluated and extended to plastic materials used for wetted parts with changes in specifications limits.

8 WORK CITED

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APPENDIX A

Test Methods for Outgassing, Leaching, and Extraction

A.1 Outgassing Test

In the test method, the tube containing the sample tube was heated in a Perkin-Elmer ATD-400 (automated thermal desorber) at 100°C for 30 minutes under dynamic headspace conditions (Figure 1). The flow of inert gas (helium) through the tube was 50 ml/min. The outgassed organic compounds were carried by helium into a trap containing an adsorbent, where they were cryo-focussed onto the adsorbent. After 30 minutes, the trap was heated to 350°C to desorb the trapped compounds. The desorbed compounds were carried by helium into a non-polar poly (dimethylsiloxane) phase capillary column of a HP 6890 GC (Figure 2). Each compound passed down the GC column at a characteristic rate and was separated by programmed heating of the column: initial temperature held at 35°C for 3.5 minutes, ramped at a rate of 10°C/minute to 280°C and held at this temperature for 10 minutes.

As each compound exited the GC column, it was carried by helium into a HP 5972 quadrupole mass spectrometer (MS), where it was ionized by electron impact (70 eV) (Figure 2 and Figure 4). The MS collected a full mass spectrum (10–700 amu) approximately once every second. The compounds detected by the MS were first identified by searching a Wiley library of 275,000 mass spectra. In cases of no match, the mass spectrum was interpreted manually to give the best estimate of the most probable compounds. The mass of each compound was estimated by integrating the area under its peak and using the total ion count response factor of n-decane standard of known mass in the same mass spectrometer.

$$\text{Concentration of analyte} = \frac{(\text{area of analyte peak}) \times (\text{weight of decane in standard})}{(\text{area of decane peak in standard}) \times (\text{surface area of the sample})}$$

Where, analyte represents either an individual organic compound or range of organic compounds. Surface area of the sample (cm²) = 2 x [(width x length) + (width x thickness) + (length x thickness)], weight of decane in µg.

For GC/MS calibration, sensitivity of TD-GC-MS system, and boiling range information of the compounds that could be detected, a standard n-alkane mixture was used. In the mixture, 500 ng each of a series of n-alkanes from n-hexane (C₆H₁₄, boiling point: 69°C) to n-octacosane (C₂₈H₅₈, boiling point: 432°C, retention time of about 34 minutes in the GC column) was used. The sample tube with the standard n-alkane mixture was heated in the ATD-400 system at 350°C. The higher temperature was necessary to completely desorb all the hydrocarbons. The outgassed chemical compounds were separated into three groups:

- Low boilers (C₆–C₁₀), defined as those compounds with retention times in the GC column between n-hexane (C₆H₁₄) and n-decane (C₁₀H₂₂),
- Medium boilers (≥ C₁₀–C₂₀), defined as those compounds with retention times in the GC column between n-decane (C₁₀H₂₂) and n-eicosane (C₂₀H₄₂),
- High boilers (>C₂₀), defined as those compounds with retention times in the GC column beyond n-eicosane (C₂₀H₄₂) and up to at least n-triacontane (C₃₀H₆₂).

For GC-MS analysis, a toluene-d₈ standard of known concentration was used as an internal standard. A known volume of the standard was added to the ATD-400 system at the time the sample was placed into the system.

A.2 Leaching Test

A.2.1 Ions Leached from the Plastic Materials by UPW and Ozonated UPW

In the leaching test, types and amounts of ions leached from the plastic materials by UPW and ozonated UPW were measured by the ion-chromatograph (IC) system (Dionex Dual Channel Model DX-500) (Figure 6)

The IC system (Figure 6) consists of reservoirs for eluents (electrolyte solutions, such as a solution of the mixture of sodium carbonate and sodium bicarbonate), a pump, a concentrator, a guard column, a sample loop, a sample injector, a separator column, a suppressor column, and a conductivity detector. To enable analyses in the low parts-per-billion (ppb) range, a chemically inert single piston pump was used to pump the sample through the concentrator column before injection into the analytical column. The suppressor was used to chemically reduce the high background conductivity of the electrolytes in the eluent and to convert the ions of interest into a more conductive form. The analytical columns were filled with particles of stationary phase over which mobile phase was pumped. When a sample was injected to the flowing mobile phase, it was carried into the column and the process of separation began using the partition coefficients of the ions (An ion with a larger partition coefficient has a longer retention time than one with a lower partition coefficient). Each ion detected was identified using retention time data (confirmed against the daily calibration standard).

An initial calibration curve was generated with four points. The curve was verified using a second standard source. Daily calibration of the instrument consisted of running a blank followed by a calibration check. Closing blanks and standards were run at the end of the day. Peak areas were compared to check the performance of the instrument.

A.2.2 Elements Leached from the Plastic Materials by UPW and Ozonated UPW

For the measurements of types and amounts of elements leached from the plastic materials by UPW and ozonated UPW, ICP-MS (Figure 7) used the following instruments:

- Perkin Elmer model 6000 equipped with auto samplers
- Hewlett Packard 4500 equipped with auto sampler
- Varian Ultramass 700 with auto sampler
- Sample introduction systems

The sample introduction system converted the leachate solution into fine aerosol droplets that were injected into Ar ICP. Under extremely high temperature conditions of ICP (which can reach as high as ~ 9000K, the molecules are broken down into atoms and most elements are ionized with >90% efficiency). The ions were then injected into the ICP-MS interface where most of the gas was removed. The positive ions were extracted into a mass spectrometer and were identified based on their characteristic mass to charge ratio and their amounts were quantified by a calibration standard. The quadrupole mass filter used had the capacity to analyze singly charged ions from 1–300 amu.

A.2.3 Total Oxidizable Carbon (TOC) Leached from the Plastic Materials by UPW and Ozonated UPW

The amount of TOC in the leachate solution was quantified by the oxidation of the organic compounds to carbon dioxide by ammonium persulfate in the presence of UV radiation. A

sensitive and selective membrane-based conductometric detector was used to measure the amount of carbon dioxide generated in the oxidation reaction (this method is described in ASTM D5904-96). The following apparatus/instruments were used to quantify the mass of total oxidizable carbon in the leachate:

- Sievers 800 TOC Analyzer (detection limit is 0.5 parts-per-billion by weight)
- Sievers Autosampler
- 40-ml amber glass vials with aluminum foil wraps

For the quantification of the amount of TOC, after the leaching test, the leachate was transferred into the 40-ml amber glass vial. The vial was covered with aluminum foil to prevent airborne contamination. A system blank was analyzed before calibration of the instrument. The acid and oxidizer flow rates were preset. The instrument was calibrated and then the analysis of each leachate was performed in duplicate.

A.2.4 Surface Topography of Plastic Materials Before and After Interacting with UPW and Ozonated UPW

The surface topography of the plastic material was examined by the scanning electron microscope (SEM)/energy dispersive (EDX) X-ray technique, using a Cambridge Stereoscan Model 120 SEM with Kevex EDX equipped with Delta Four SuperQuantum detector. (The EDX microanalysis determines those elements present in approximately the top 1 μm of the surface). For SEM, the coupons were coated with gold for more suitable conduction properties. For EDX, uncoated material provided compositional information. In case of charging problems, the EDX samples were also coated with gold. High-resolution micrographs of the samples were used to provide important information about the changes at the surface due to chemical exposures.

A.3 Extraction Test

In the extraction test, the following instruments (ICP, Figure 7) were used to measure types and amounts of elements extracted from the plastic materials by chemicals, similar to the technique used in the leaching test:

- Perkin Elmer model 6000 equipped with auto samplers
- Hewlett Packard 4500 equipped with auto sampler
- Varian Ultramass 700 with auto sampler
- Sample introduction systems

The sample introduction system converted the extracted solution into fine aerosol droplets that were injected into Ar ICP. (Under extremely high temperature conditions of ICP, which can reach as high as $\sim 9000\text{K}$, the molecules are broken down into atoms and most elements are ionized with $>90\%$ efficiency). The ions were injected into the ICP-MS interface where most of the gas was removed. The positive ions were extracted into a mass spectrometer and were identified based on their characteristic mass to charge ratio and their amounts were quantified by a calibration standard. The quadrupole mass filter used had the capacity to analyze singly charged ions from 1–300 amu.

The surface topography of the plastic material coupons was examined by the SEM/EDX X-ray technique similar to that used for the leaching test.

APPENDIX B Leaching Test Data

B.1 Ions

The UPW and ozonated UPW leaching test data for ions are listed in Table 21 and Table 22 respectively. Standard reporting practice has been used (i.e., one significant figure for reporting limit and two significant figures for measured data). The reporting limit for ozonated UPW in Table 22 is higher because of the higher leachate concentrations.

Table 21 Ions Leached from Plastic Materials by UPW^a

Ion	Amounts of Ions ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank I	Blank II	PP	FR PP	PVC	CPVC	ECTFE	PVDF
Fluoride	5	*	*	8.0	30	6.8	19	**	740
Chloride	1	8.0	8.6	4.0	370	99	480	120	6.8
Nitrite	1	4.7	4.6	1.5	*	*	*	*	1.9
Bromide	2	*	*	*	2600	*	*	*	4.0
Nitrate	2	6.9	6.6	6.0	*	*	*	24	12
Phosphate	2	*	*	*	*	6.9	*	34	*
Sulfate	2	1.4	1.3	6.4	*	160	240	14	8.4
Lithium	1	*	*	*	*	*	*	*	*
Sodium	1	0.32	0.32	0.80	40	10	37	6.3	3.6
Ammonium	2	46	46	62	520	25	25	39	34
Potassium	2	*	*	*	*	*	7.9	3.3	3.9
Magnesium	2	*	*	*	36	*	2.1	*	*
Calcium	1	*	*	12	74	24	94	18	13
Total ions				100	3700	330	900	259	830
Total ions minus the blank				33	3600	260	830	203	760
Critical ions minus the blank				29	890	260	730	176	760

a: Data are reported following standard practice; i.e., one significant figure for the reporting limit and two significant figures for measured data; water temperature: 85°C; soaking period: 30 ± 2 days; soaking bottle: PP; Number of coupons: 2 in 150 mL UPW; **b:** reporting limit. *: below the reporting limit. **: anomalous data

Table 22 Ions Leached from Plastic Materials by Ozonated UPW^a

Ion	Amounts of Ions ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank I	Blank II	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Fluoride	200	2700	2800	3000	3500	4200	3000	2800	2700
Chloride	90	190 ^c	*	*	*	*	160 ^c	*	*
Nitrite	90	*	*	*	*	*	*	*	*
Bromide	200	*	*	*	900	*	*	*	*
Nitrate	200	330	300	2000	2300	1700	1600	1200	1200
Phosphate	200	*	*	*	270	*	*	*	*
Sulfate	200	*	*	760	1300	*	*	*	*
Lithium	0.5	*	*	*	*	*	*	*	*
Sodium	0.5	4.3	1.3	7.5	13	2.7	21	1.2	2.9
Ammonium	1	13	4.9	13	5.4	19	10	10	6.9
Potassium	1	5.6	1.5	3.9	4.6	3.0	9.0	2.7	3.4
Magnesium	1	1.8	*	1.7	20	*	2.8	*	2.8
Calcium	0.5	8.8	2.1	14	55	5.2	81	2.8	6.6
Total ions				5800	8400	5900	4900	4000	3900
Total ions minus blank				2700	5300	2900	1800	940	890
Critical ions minus blank				2700	5200	2800	1600	940	890

a: Data reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; ozonated water temperature: ambient; soaking period: 24 hours; soaking bottle: fluoropolymer; Number of coupons: 4 in about 200 mL ozonated UPW; **b:** reporting limit. The reporting limit is higher because of the higher leachate concentrations; **c:** one blank showed apparent contamination above the reporting limit. Hence the CPVC value may also be questionable. *: below the reporting limit.

B.2 Elements

The UPW and ozonated UPW leaching test data for elements are listed in Table 23 and Table 24, respectively. Standard reporting practice has been used (i.e., one significant figure for reporting limit and two significant figures for measured data).

Table 23 Elements Leached from Plastic Materials by Ultrapure Water^a

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank I	Blank II	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Aluminum (Al)	0.1	*	*	3.8	72	14	19	0.95	1.7
Antimony (Sb)	0.06	*	*	0.11	830	0.85	0.08	*	0.38
Arsenic (As)	0.6	*	*	*	1.6	*	*	*	*
Barium (Ba)	0.03	*	*	0.030	0.37	8.7	6.3	*	*
Beryllium (Be)	0.1	*	*	*	*	*	*	*	*
Bismuth (Bi)	0.1	*	*	*	*	*	*	*	*
Boron (B)	1	*	*	3.8	*	*	*	3.5	*
Cadmium (Cd)	0.08	*	*	*	0.070	*	*	*	*
Calcium (Ca)	8	*	*	14	42	22	79	*	*
Cerium (Ce)	0.03	*	*	*	*	*	*	*	*
Cesium (Cs)	0.06	*	*	*	*	*	*	*	*
Chromium (Cr)	0.08	*	*	*	0.14	*	0.25	*	*
Cobalt (Co)	0.06	*	*	*	0.050	*	*	*	*
Copper (Cu)	0.1	*	*	0.19	0.18	*	*	*	*
Dysprosium (Dy)	0.1	*	*	*	*	*	*	*	*
Erbium (Er)	0.06	*	*	*	*	*	*	*	*
Europium (Eu)	0.06	*	*	*	*	*	*	*	*
Gadolinium (Gd)	0.1	*	*	*	*	*	*	*	*
Gallium (Ga)	0.1	*	*	*	*	*	*	*	*
Germanium (Ge)	0.1	*	*	*	*	*	*	*	*
Gold (Au)	0.1	*	*	*	*	*	*	*	*
Hafnium (Hf)	0.08	*	*	*	*	*	*	*	*
Holmium (Ho)	0.03	*	*	*	*	*	*	*	*
Indium (In)	0.3	*	*	*	*	*	*	*	*
Iridium (Ir)	0.2	*	*	*	*	*	*	*	*
Iron (Fe)	0.3	*	*	0.50	1.2	*	0.30	0.30	*
Lanthanum (La)	0.06	*	*	*	*	*	*	*	*
Lead (Pb)	0.1	*	*	*	*	1100	*	*	*
Lithium (Li)	0.08	*	*	*	*	*	*	*	*
Lutetium (Lu)	0.03	*	*	*	*	*	*	*	*
Magnesium (Mg)	0.06	*	*	66	28	0.52	1.8	0.20	1.6
Manganese (Mn)	0.08	*	*	*	0.12	*	*	*	*
Mercury (Hg)	0.1	*	*	*	*	*	*	*	*
Molybdenum (Mo)	0.1	*	*	*	*	*	0.22	*	*
Neodymium (Nd)	0.06	*	*	*	*	*	*	*	*
Nickel (Ni)	0.1	*	*	*	0.23	*	*	*	*
Niobium (Nb)	0.06	*	*	*	*	*	*	*	*

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank I	Blank II	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Osmium (Os)	0.06	*	*	*	*	*	*	*	*
Palladium (Pd)	0.2	*	*	*	*	*	*	*	*
Platinum (Pt)	0.2	*	*	*	*	*	*	*	*
Potassium (K)	10	*	*	*	*	*	*	*	*
Praseodymium (Pr)	0.03	*	*	*	*	*	*	*	*
Rhenium (Re)	0.2	*	*	*	*	*	*	*	*
Rhodium (Rh)	0.06	*	*	*	*	*	*	*	*
Rubidium (Rb)	0.03	*	*	*	*	*	*	*	*
Ruthenium (Ru)	0.1	*	*	*	*	*	*	*	*
Samarium (Sm)	0.1	*	*	*	*	*	*	*	*
Scandium (Sc)	0.6	*	*	*	*	*	*	*	*
Selenium (Se)	20	*	*	*	*	*	*	*	*
Silicon (Si)	100	*	*	*	*	*	140	*	*
Silver (Ag)	0.08	*	*	*	*	*	*	*	*
Sodium (Na)	0.2	*	*	1.7	2.0	6.0	35	0.47	2.4
Strontium (Sr)	0.03	*	*	*	0.050	0.080	0.050	*	*
Tantalum (Ta)	0.06	*	*	*	*	*	*	*	*
Tellurium (Te)	0.1	*	*	*	*	*	*	*	*
Terbium (Tb)	0.06	*	*	*	*	*	*	*	*
Thallium (Tl)	0.6	*	*	*	*	*	*	*	*
Thorium (Th)	0.06	*	*	*	*	*	*	*	*
Thulium (Tm)	0.03	*	*	*	*	*	*	*	*
Tin (Sn)	0.06	*	*	0.71	12	*	230	*	*
Titanium (Ti)	0.1	*	*	*	*	*	*	*	*
Tungsten (W)	0.06	*	*	*	*	*	*	*	*
Uranium (U)	0.06	*	*	*	*	*	*	*	*
Vanadium (V)	0.08	*	*	*	*	*	*	*	*
Ytterbium (Yb)	0.08	*	*	*	*	*	*	*	*
Yttrium (Y)	0.06	*	*	*	*	*	*	*	*
Zinc (Zn)	0.2	*	*	4.4	32	12	0.71	0.87	0.93
Zirconium (Zr)	0.1	*	*	*	*	*	*	*	*
Total elements				100	1,000	1,200	510	7.0	7.0
Total elements minus blank				100	1,000	1,200	510	7.0	7.0
Critical elements minus blank				100	180	55	140	6.6	6.6

a: Data are reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; water temperature: 85°C soaking period: 30 ± 2 days; soaking bottle: PP; Number of coupons: 4 in 300 mL ozonated UPW; **b:** reporting limit; *: below the reporting limit.

Table 24 Elements Leached from Plastic Materials by Ozonated Ultrapure Water^a

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank I	Blank II	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Aluminum (Al)	0.09	5.3	1.7	29	36	24	52	0.73	1.9
Antimony (Sb)	0.04	0.050	0.090	0.19	8300	0.46	0.10	*	0.24
Arsenic (As)	0.4	*	*	*	6.8	*	*	*	*
Barium (Ba)	0.02	0.050	*	*	0.050	2.7	0.020	*	*
Beryllium (Be)	0.07	*	*	*	*	*	*	*	*
Bismuth (Bi)	0.07	0.11	*	*	3.9	*	*	*	*
Boron (B)	0.9	*	*	*	*	*	*	*	*
Cadmium (Cd)	0.06	*	*	*	2.3	*	0.28	*	*
Calcium (Ca)	6	*	*	14	60	15	74	*	*
Cerium (Ce)	0.02	*	*	*	0.030	*	*	*	*
Cesium (Cs)	0.04	*	*	*	*	*	*	*	*
Chromium (Cr)	0.06	0.11	0.33	0.21	0.29	0.17	0.28	0.14	0.39
Cobalt (Co)	0.04	*	*	*	0.030	*	*	*	*
Copper (Cu)	0.09	0.11	*	0.69	0.10	*	0.22	*	*
Dysprosium (Dy)	0.07	*	*	*	*	*	*	*	*
Erbium (Er)	0.04	*	*	*	*	*	*	*	*
Europium (Eu)	0.04	*	*	*	*	*	*	*	*
Gadolinium (Gd)	0.07	*	*	*	*	*	*	*	*
Gallium (Ga)	0.07	*	*	*	*	*	*	*	*
Germanium (Ge)	0.09	*	*	*	*	*	*	*	*
Gold (Au)	0.9	*	*	*	*	*	*	*	*
Hafnium (Hf)	0.06	*	*	*	*	*	*	*	*
Holmium (Ho)	0.02	*	*	*	*	*	*	*	*
Indium (In)	0.04	*	*	*	0.36	*	*	*	*
Iridium (Ir)	0.1	*	*	*	*	*	*	*	*
Iron (Fe)	0.2	5.5	2.4	6.0	6.0	1.5	3.2	1.4	2.8
Lanthanum (La)	0.02	*	*	*	*	*	*	*	*
Lead (Pb)	0.09	*	*	*	0.58	36	*	*	*
Lithium (Li)	0.06	*	*	*	*	*	*	*	*
Lutetium (Lu)	0.02	*	*	*	*	*	*	*	*
Magnesium (Mg)	0.04	2.0	0.44	2.1	24	0.68	1.7	0.37	1.8
Manganese (Mn)	0.06	*	*	0.080	0.11	*	*	*	*
Mercury (Hg)	0.9	*	*	*	*	*	*	*	*
Molybdenum (Mo)	0.09	*	*	0.71	0.36	*	*	*	*
Neodymium (Nd)	0.04	*	*	*	*	*	*	*	*
Nickel (Ni)	0.09	0.090	*	0.15	0.32	0.17	0.22	*	0.26
Niobium (Nb)	0.04	*	*	*	*	*	*	*	*

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank I	Blank II	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Osmium (Os)	0.04	*	*	*	*	*	*	*	*
Palladium (Pd)	0.1	*	*	*	*	*	*	*	*
Platinum (Pt)	6	*	*	*	*	*	*	*	*
Potassium (K)	9	*	*	*	*	*	10	*	*
Praseodymium (Pr)	0.02	*	*	*	*	*	*	*	*
Rhenium (Re)	0.1	*	*	*	*	*	*	*	*
Rhodium (Rh)	0.04	*	*	*	*	*	*	*	*
Rubidium (Rb)	0.02	*	*	*	*	*	*	*	*
Ruthenium (Ru)	0.09	*	*	*	*	*	*	*	*
Samarium (Sm)	0.07	*	*	*	*	*	*	*	*
Scandium (Sc)	0.4	*	*	*	*	*	*	*	*
Selenium (Se)	10	*	*	*	*	*	*	*	*
Silicon (Si)	90	*	*	*	*	*	*	*	*
Silver (Ag)	0.06	*	*	*	*	*	*	*	*
Sodium (Na)	0.1	1.8	0.93	14	5.8	5.6	28	1.4	0.97
Strontium (Sr)	0.02	*	*	0.020	0.030	0.030	0.040	*	*
Tantalum (Ta)	0.04	*	*	*	*	*	*	*	*
Tellurium (Te)	0.07	*	*	*	*	*	*	*	*
Terbium (Tb)	0.04	*	*	*	*	*	*	*	*
Thallium (Tl)	0.09	*	*	*	*	*	*	*	*
Thorium (Th)	0.04	*	*	*	*	*	*	*	*
Thulium (Tm)	0.02	*	*	*	*	*	*	*	*
Tin (Sn)	0.04	0.15	*	0.48	89	0.31	14	0.25	0.040
Titanium (Ti)	0.09	0.31	*	8.3	1.6	7.9	3.2	*	0.10
Tungsten (W)	0.04	0.11	0.53	5.0	2.9	0.62	0.58	0.44	4.7
Uranium (U)	0.04	*	*	*	*	*	*	*	*
Vanadium (V)	0.06	*	*	*	*	*	*	*	*
Ytterbium (Yb)	0.06	*	*	*	*	*	*	*	*
Yttrium (Y)	0.04	*	*	*	*	*	*	*	*
Zinc (Zn)	0.1	1.0	0.24	17	9.1	8.2	1.7	0.66	0.53
Zirconium (Zr)	0.09	*	0.36	0.15	0.15	0.17	*	*	*
Total elements				98	8,600	100	190	5.0	14
Total elements minus blank				86	8,500	94	180	0.20	5.5
Critical elements minus blank				86	150	92	160	0.20	5.5

a: Data are reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; ozonated water temperature: ambient; soaking period: 24 hours; soaking bottle: fluoropolymer; Number of coupons: 4 in about 200 mL ozonated UPW; **b:** reporting limit. *: below the reporting limit.

APPENDIX C Extraction Test Data

C.1 Elements

Data for the types and amounts of elements extracted from the plastic materials by chemicals are listed in Table 25 to Table 29. Standard data reporting practice has been used (i.e., one significant figure for reporting limit and two significant figures for measured data).

Table 25 Elements Extracted from the Plastic Materials by APM (SC1)^a

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Aluminum (Al)	0.3	*	*	3.1	4.7	59	63	1.8	7.5
Antimony (Sb)	0.3	*	*	*	66	*	*	*	*
Arsenic (As)	1.0	*	*	*	*	*	*	*	*
Barium (Ba)	0.3	*	*	*	*	6.2	*	*	0.30
Beryllium (Be)	0.3	*	*	*	*	*	*	*	*
Boron (B)	6	*	*	*	*	*	*	*	*
Cadmium (Cd)	0.3	*	*	*	0.25	*	0.85	*	*
Calcium (Ca)	8	*	*	*	39	20	20	*	*
Chromium (Cr)	0.3	*	*	*	*	*	*	*	*
Cobalt (Co)	0.1	*	*	*	*	*	*	*	*
Copper (Cu)	0.3	*	*	*	0.74	*	0.85	0.51	0.30
Gallium (Ga)	0.1	*	*	*	*	*	*	*	*
Germanium (Ge)	0.3	*	*	1.9	0.74	*	*	0.76	0.30
Gold (Au)	1.0	*	*	*	*	*	*	*	*
Iron (Fe)	6	*	*	*	*	*	*	*	*
Lead (Pb)	0.3	*	*	*	*	450	*	*	0.30
Lithium (Li)	0.1	*	*	*	*	*	*	*	*
Magnesium (Mg)	0.3	*	0.56	1.1	2.0	1.1	1.1	1.0	3.0
Manganese (Mn)	0.3	*	*	*	*	*	*	*	*
Molybdenum (Mo)	0.1	*	*	*	*	*	*	*	*
Nickel (Ni)	0.3	*	*	*	*	*	*	*	1.8
Potassium (K)	8	*	*	*	*	*	*	*	*
Silicon (Si)	1	*	*	*	*	*	*	*	*
Silver (Ag)	0.3	*	*	*	*	*	*	*	*
Sodium (Na)	0.3	*	1.4	1.9	2.2	4.8	5.7	1.8	2.1
Strontium (Sr)	0.1	*	*	*	*	*	*	*	*
Tin (Sn)	3.0	*	*	*	4.7	*	15.0	1.3	*
Titanium (Ti)	1.0	*	*	*	*	220	480	*	*

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Vanadium (V)	3.0	*	*	*	*	*	*	*	*
Zinc (Zn)	1.0	*	*	1.9	8.3	19	*	6.1	3.0
Total Elements				10	130	780	590	13	19
Total elements minus blank				8.0	130	780	590	11	17
Critical elements minus blank				6.1	55	770	570	9.2	16

a: APM (SC1) = 1:1:5 (NH₄OH: H₂O₂: H₂O volume ratio); data are reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; APM (SC1) was initially at the mixing temperature of the chemicals; extraction period: 30 ± 2 days; extraction bottle: fluoropolymer; Number of coupons: 2; **b:** reporting limit. *: below the reporting limit.

Table 26 Elements Extracted from the Plastic Materials by HPM (SC2)^a

Element	Amount of elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Aluminum (Al)	0.3	*	0.56	5.9	9.7	53	83	1.9	5.1
Antimony (Sb)	0.3	*	*	*	1046	7.3	4.1	*	2.4
Arsenic (As)	1	*	*	*	1.5	*	*	*	*
Barium (Ba)	0.3	*	*	*	0.73	7.0	*	*	*
Beryllium (Be)	0.3	*	*	*	*	*	*	*	*
Boron (B)	6	*	*	*	*	*	*	*	*
Cadmium (Cd)	0.3	*	*	*	*	*	*	*	*
Calcium (Ca)	8	*	*	11	21	13	59	*	*
Chromium (Cr)	0.3	*	*	*	*	*	*	*	*
Cobalt (Co)	0.1	*	*	*	*	*	*	*	*
Copper (Cu)	0.3	*	*	*	1.0	*	0.59	0.81	*
Gallium (Ga)	0.1	*	*	*	*	*	*	*	*
Germanium (Ge)	0.3	*	*	*	*	*	*	*	*
Gold (Au)	1	*	*	*	*	*	*	*	*
Iron (Fe)	6	*	*	*	19	*	8.0	*	15
Lead (Pb)	0.3	*	*	*	*	676	0.60	*	*
Lithium (Li)	0.1	*	*	*	*	*	*	*	*
Magnesium (Mg)	0.3	*	0.56	2.2	8.0	1.4	4.4	1.6	4.5
Manganese (Mn)	0.3	*	*	*	*	*	*	*	*
Molybdenum (Mo)	0.1	*	*	*	*	*	0.59	*	*
Nickel (Ni)	0.3	*	*	*	*	*	*	*	2.1
Potassium (K)	8	*	*	*	*	*	*	*	*
Silicon (Si)	1	*	*	*	*	*	*	*	*
Silver (Ag)	0.3	*	*	*	*	*	*	*	*
Sodium (Na)	0.3	0.28	1.4	2.5	1.5	3.7	17	1.3	2.1

Element	Amount of elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Strontium (Sr)	0.1	*	*	*	*	*	*	*	*
Tin (Sn)	3	*	*	*	13	*	6.5	0.54	*
Titanium (Ti)	1	*	*	*	*	42	12	*	*
Vanadium (V)	3	*	*	*	*	*	*	*	*
Zinc (Zn)	1	*	*	*	4.4	17	*	*	*
Total elements				22	1,100	820	200	6.0	31
Total elements minus blank				20	1,100	820	194	4.0	29
Critical elements minus blank				19	64	800	180	3.2	26

a: HPM (SC2) = 1:1:6 (HCl : H₂O₂ : H₂O volume ratio); data are reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; HPM (SC2) was initially at the mixing temperature of the chemicals; extraction period: 30 ± 2 days; extraction bottle: fluoropolymer; Number of coupons: 2; **b:** reporting limit. *: below the reporting limit.

Table 27 Elements Extracted from the Plastic Materials by SPM (Piranha)^a

Element	Amount of elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Aluminum (Al)	0.3	1.7	1.7	7.5	60	60	170	4.4	9.8
Antimony (Sb)	0.3	*	*	*	4275	7.7	3.2	3.3	3.1
Arsenic (As)	1	*	*	*	1.5	*	*	*	*
Barium (Ba)	0.3	0.17	0.19	0.20	2.0	9.1	0.26	0.18	0.21
Beryllium (Be)	0.3	*	*	*	*	*	*	*	*
Boron (B)	6	*	*	*	*	*	*	*	*
Cadmium (Cd)	0.3	*	*	*	*	*	*	*	*
Calcium (Ca)	8	*	*	*	78	16	117	*	*
Chromium (Cr)	0.3	*	*	*	*	*	*	*	*
Cobalt (Co)	0.1	*	*	*	*	*	*	*	*
Copper (Cu)	0.3	*	*	*	*	*	*	*	*
Gallium (Ga)	0.1	*	*	*	*	*	*	*	*
Germanium (Ge)	0.3	*	*	*	*	0.85	0.59	0.77	4.9
Gold (Au)	1	*	*	*	*	*	*	*	*
Iron (Fe)	6	*	*	*	43	*	6.7	*	10.7
Lead (Pb)	0.3	*	*	*	*	794	0.88	0.51	0.61
Lithium (Li)	0.1	*	*	*	*	*	*	*	*
Magnesium (Mg)	0.3	0.56	0.83	4.1	33	1.4	5.6	1.8	4.3
Manganese (Mn)	0.3	*	*	*	*	*	*	*	*
Molybdenum (Mo)	0.1	*	*	*	*	*	0.59	*	*
Nickel (Ni)	0.3	*	*	*	*	*	*	*	*

Element	Amount of elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Potassium (K)	8	*	*	*	*	*	11	*	*
Silicon (Si)	1	*	*	*	*	*	*	*	*
Silver (Ag)	0.3	*	*	*	*	*	*	*	*
Sodium (Na)	0.3	2.5	2.5	2.3	3.8	6.2	50	1.3	2.1
Strontium (Sr)	0.1	*	*	*	*	*	*	*	*
Tin (Sn)	3	*	*	*	402	*	70	*	*
Titanium (Ti)	1	*	4.7	32	63	170	132	3.8	*
Vanadium (V)	3	*	*	*	*	*	*	*	*
Zinc (Zn)	1	*	*	*	13	20	*	2.6	*
Total elements				46	5,000	1,100	570	19	36
Total elements minus blank				36	5,000	1,100	560	11	31
Critical elements minus blank				36	4,600	1,100	490	11	31

a: SPM (Piranha) = 5:1 v/v (96% w/w H₂SO₄: 31% w/w H₂O₂); data are reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; SPM (Piranha) was initially at the mixing temperature of the chemicals; extraction period: 30 \pm 2 days; extraction bottle: fluoropolymer; Number of coupons: 2; b: reporting limit. *: below the reporting limit.

Table 28 Elements Extracted from the Plastic Materials by Nitric Acid^a

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTF E	PVDF
Aluminum (Al)	0.3	*	*	5.3	12	59	150	1.9	4.8
Antimony (Sb)	0.3	*	0.56	0.55	870	3.7	1.7	1.1	1.5
Arsenic (As)	1	*	*	*	*	*	*	*	*
Barium (Ba)	0.3	*	*	*	0.50	7.3	*	*	*
Beryllium (Be)	0.3	*	*	*	*	*	*	*	*
Boron (B)	6	*	*	*	*	*	*	*	*
Cadmium (Cd)	0.3	*	*	*	*	*	*	*	*
Calcium (Ca)	8	*	*	*	35	15	160	*	*
Chromium (Cr)	0.3	*	*	*	*	*	*	*	*
Cobalt (Co)	0.1	*	*	*	*	*	*	*	*
Copper (Cu)	0.3	*	*	*	1.2	*	0.55	0.81	0.59
Gallium (Ga)	0.1	*	*	*	*	*	*	*	*
Germanium (Ge)	0.3	*	*	*	*	*	*	*	*
Gold (Au)	1	*	*	*	*	*	*	*	*
Iron (Fe)	6	*	*	*	25	*	9.1	*	13
Lead (Pb)	0.3	*	*	*	0.50	760	*	*	*
Lithium (Li)	0.1	*	*	*	*	*	*	*	*
Magnesium (Mg)	0.3	*	*	1.7	8.7	1.1	8.8	1.9	3.9

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE E	PVDF
Manganese (Mn)	0.3	*	*	*	*	*	*	*	*
Molybdenum (Mo)	0.1	*	*	*	*	*	0.55	*	*
Nickel (Ni)	0.3	*	*	*	*	*	*	*	*
Potassium (K)	8	*	*	*	*	*	14	*	*
Silicon (Si)	1	*	*	*	*	*	*	*	*
Silver (Ag)	0.3	*	*	*	*	*	*	*	*
Sodium (Na)	0.3	*	*	2.5	1.0	4.8	150	*	2.4
Strontium (Sr)	0.1	*	*	*	*	*	*	*	*
Tin (Sn)	3	*	*	*	60	0.20	150	0.81	*
Titanium (Ti)	1	*	*	*	*	34	16	*	*
Vanadium (V)	3	*	*	*	*	*	*	*	*
Zinc (Zn)	1	*	*	*	7.2	*	*	3.2	*
Total elements				10	1,000	890	660	10	26
Total elements minus blank				9.4	1,000	890	660	9.7	26
Critical elements minus blank				9.4	90	880	510	7.9	25

a: 70% (w/w) HNO₃; data are reported following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; HNO₃ was at ambient temperature; extraction period: 30 ± 2 days; extraction bottle: fluoropolymer; Number of coupons: 2; b: reporting limit. *: below the reporting limit.

Table 29 Elements Extracted from the Plastic Materials by Hydrofluoric Acid^a

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Aluminum (Al)	0.3	*	1.4	72	93	300	12000	11	14
Antimony (Sb)	0.3	*	*	*	65000	860	57	16	17
Arsenic (As)	1	*	*	*	*	*	*	*	*
Barium (Ba)	0.3	*	*	*	2.6	25	1.1	*	*
Beryllium (Be)	0.3	*	*	*	*	*	*	*	*
Boron (B)	6	*	*	*	*	*	*	*	*
Cadmium (Cd)	0.3	*	*	*	0.24	0.56	*	*	*
Calcium (Ca)	8	*	*	10	33	27	145	*	*
Chromium (Cr)	0.3	*	*	0.28	0.24	1.1	13	*	*
Cobalt (Co)	0.1	*	*	*	0.22	*	0.28	*	*
Copper (Cu)	0.3	*	*	3.1	3.6	170	530	0.73	0.60
Gallium (Ga)	0.1	*	*	*	*	*	2.6	*	*
Germanium (Ge)	0.3	*	*	*	*	*	0.28	0.49	*
Gold (Au)	1	*	*	*	*	*	*	*	*
Iron (Fe)	6	*	*	26	93	*	16	6	15

Element	Amount of Elements ($\mu\text{g}/\text{cm}^2$) $\times 10^3$								
	RL ^b	Blank Before	Blank After	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Lead (Pb)	0.3	*	*	*	6.5	8100	4.0	1.2	1.5
Lithium (Li)	0.1	*	*	*	*	*	*	*	*
Magnesium (Mg)	0.3	*	0.60	11	17	45	120	7.6	4.2
Manganese (Mn)	0.3	*	*	*	0.24	0.84	3.4	*	*
Molybdenum (Mo)	0.1	*	*	*	*	36	65	*	*
Nickel (Ni)	0.3	*	*	*	0.48	*	18	*	0.89
Potassium (K)	8	*	*	*	*	72	220	*	*
Silver (Ag)	1	*	*	*	*	*	*	*	*
Sodium (Na)	0.3	*	*	25	6.2	470	3100	1.2	3.9
Strontium (Sr)	0.3	*	*	*	*	0.84	2.3	*	*
Tin (Sn)	0.1	*	*	1.4	360	56	1800	0.73	0.30
Titanium (Ti)	3	*	*	4700	3100	390000	890000	250	280
Vanadium (V)	1	*	*	*	*	1.9	4.0	*	*
Zinc (Zn)	3	*	*	36	21	1700	10	2.9	*
Zirconium (Zr)	1	*	*	1.4	0.72	3.1	34	*	*
Total elements				4,900	68,000	400,000	900,000	300	320
Total elements minus blank				4,900	68,000	400,000	900,000	300	320
Critical elements minus blank				4,900	3,000	400,000	900,000	280	320

a: 49% (w/w) HF; data following standard practice, i.e., one significant figure for reporting limit and two significant figures for measured data.; HF was at ambient temperature; extraction period: 30 ± 2 days; extraction containers: fluoropolymer; Number of coupons: 2; **b:** reporting limit. *: below the reporting limit.

APPENDIX D
Propensity for Fire Propagation, Smoke, and Corrosive Product Release and Physical Properties of Plastic Materials Examined in the Study⁹

Table 30 lists the combustibility properties of the plastic materials examined in this study.

Table 30 Combustibility Properties of Plastic Materials Examined in the Study

Property	PP	FRPP	PVC	CPVC	ECTFE	PVDF
Ignition Properties						
Critical Heat Flux, CHF ^a (kW/m ²)	10	20	20	≥ 60	≥ 60	30
Thermal Response Parameter, TRP ^b (kW-s ^{1/2} /m ²)	347	200	603	f	f	465
Combustion Properties						
Chemical heat of combustion (kJ/g)	30.4	25.3	4.9	g	g	4.0
Yield of smoke (g/g)	0.09	0.14	0.07	0.05	0.07	0.06
Average corrosion rate (Å/min)	0.04	0.61	0.14	0.48	0.06	0.06
Material mass loss ^c (g)	40.9	44.8	43.6	48.1	56.7	34.7
Average Corrosion Index ^d (Å/min)/g/m ³)	0.08	1.4	0.47	1.6	0.14	0.12
Fire Propagation Property						
Fire Propagation Index, FPI ^e (m/s ^{1/2})/(kW/m) ^{2/3}	>>6	>> 6	4	< 6	<6	6
Low-combustible plastic	No	No	Yes	Yes	Yes	Yes

a: The value of the critical heat flux (CHF) represents the minimum heat flux at or below which there is no ignition [9]. CHF values are related to the decomposition and ignition temperatures.

b: The value of the thermal response parameter (TRP) represents the ignition delay for a plastic material exposed to heat [9].

c: For fixed surface area (0.0077 m²) and heat flux exposure (50 kW/m²) in normal air.

d: Average Corrosion Index (ACI) is the rate of corrosion per unit mass concentration of the plastic material vapors.

e: The fire propagation index (FPI) value represents the extent and rate of fire propagation beyond the ignition zone [9]. Plastic materials with FPI values ≤ 6 (m/s^{1/2})/(kW/m)^{2/3} are not expected to have fire propagation beyond the ignition zone and are thus defined as low-combustible plastic materials [9].

f: TRP > 700 kW-s^{1/2}/m²; **g:** combustion not sustained.

⁹ Data for fire propagation and release of smoke and corrosive products were measured in the flammability apparatus. The suppliers provided the physical property data.

D.1 Polypropylene (PP)**Supplier:** Compression Polymers Group, 801 Corey St, Moosic, PA**Chemical Formula:** C₃H₆

Properties	Units	Measured Value	Testing Method
Physical Properties			
Density	g/cm ³	0.905	ASTM D792
Melting Temperature	°C	164	Aristech
Processing Characteristics			
Melt flow	g/10 min	0.6	ASTM D 1238
Flammability			
Fire Propagation Index (FPI)	(m/s ^{1/2} /(kW/s) ^{2/3})	>>6	FMRC 4910
Mechanical Properties			
Tensile Strength	psi	4800	ASTM D638
Elongation Yield	%	14	ASTM D 638
Flexural Modulus	psi	195,000	ASTM D790
Izod Impact Strength Notched	ft-lb/in	1.2 (73°F)	ASTM D256
Rockwell Hardness	R Scale	92	ASTM D 785

D.2 Fire Retarded Polypropylene (FRPP)**Supplier:** Westlake Plastics, P.O. Box 127, Lenni, PA 19052**Chemical Formula:** C₃H₆

Properties	Units	Measured Value	Testing Method
Physical Properties			
Density	g/cm ³	0.963 (73°C)	ASTM D792
Melting Temperature	°C	164	Aristech
Water Absorption	% (change in weight)	0.0119	ASTM D570
	% (soluble matter lost)	0.1511	ASTM D570
Processing Characteristics			
Melt flow	g/10 min	0.6	ASTM D 1238
Thermal Properties			
Deflection Temperature	°F	145 (264 psi) 221 (66 psi)	ASTM D648
Flammability			
Limiting Oxygen Index	%	28.3	ASTM D2868
Burning Rate		V-0	UL Bulletin 94
Fire Propagation Index (FPI)	(m/s ^{1/2} /(kW/s) ^{2/3})	>>6	FMRC 4910
Mechanical Properties			
Tensile Strength Yield	psi	4520 (73°F)	ASTM D638
Tensile Elongation Yield	%	13 (73°F)	ASTM D638
Tensile Elongation Break	%	60 (73°F)	ASTM D 638
Flexural Modulus	10 ³ psi	220 (73°F)	ASTM D790
Izod Impact Strength notched	ft/lbs/in	0.93 (73°F)	ASTM D256
Izod Impact Strength Unnotched	ft/lbs/in	9.4 (73°F)	ASTM D256
Compressive Strength	In/in/°C	3.8 x 10 ⁻⁵ (0–30°C)	ASTM D695
Hardness (Shore D)		80 (73°F)	ASTM D 2240

D.3 Poly(vinylchloride) (PVC-TSP 739)

Supplier:	Takiron Co Ltd., Osaka Kokusai Building, Osaka, Japan
Material:	Blended plastic
Composition:	PVC resin: > 90; stabilizers, etc: < 10
Chemical Formula:	CH ₂ CHCl
Official Gazette:	No. 6-66 (main composite)
CAS Number:	9002-86-2
External Appearance:	Flat plate white in color

Properties	Units	Measured Value	Testing Method
Physical Properties			
Specific Gravity		1.45 (20°C)	NR
Processing Characteristics			
Softening Point	°C	73	NR
Thermal Properties			
Deflection Temperature	°C	75	JIS K6745
Flammability			
Flash Point	°C	391	NR
Ignition Point	°C	454	NR
Limiting Oxygen Index	%	53	ASTM D2868
Fire Propagation Index (FPI)	(m/s ^{1/2} /(kW/s) ^{2/3})	4	FMRC 4910
Mechanical Properties			
Tensile Strength	N/mm ²	55	JIS K6745
Elongation	%	100	JIS K6745
Tensile Modulus	N/mm ²	2500	JIS K6745
Flexural Strength	N/mm ²	94	ASTM D790
Modulus (Flexural)	N/mm ²	3100	ASTM D790
Izod Impact Strength	kJ/m ²	5.9	JIS K6745
Compressive Strength	N/mm ²	78	ASTM D 695
Shear Strength	N/mm ²	60	ASTM D732

D.4 Chlorinated Poly(vinylchloride) (CPVC Corzan)

Supplier: BFGoodrich, Cleveland, Ohio, USA

Chemical Formula: $C_2H_2Cl_2$

Properties	Units	Measured Value	Testing Method
Physical Properties			
Density	g/cm^3	1.55	ASTM D792
Water Absorption	%	0.03	ASTM D570
Rockwell Hardness	R-Scale	119	ASTM D785
Cell Class		23447-B	ASTM D1784
Thermal Properties			
Coefficient of Linear Thermal Expansion	In/in/°F	3.4×10^{-5}	ASTM D696
Thermal Conductivity	BTU/in/hr/ft ² /°F	0.95	ASTM C177
Heat Distortion Temperature	°C	103 (264 psi)	ASTM D648
Flammability			
Limiting Oxygen Index	%	60	ASTM D2868
Flame Spread		15	ASTM E-84
Smoke Developed		70–125	ASTM E-84
Burning Rate		V-0	UL Bulletin 94
Fire Propagation Index (FPI)	$(m/s^{1/2}/(kW/s)^{2/3})$	<<6	FMRC 4910
Mechanical Properties			
Tensile Strength	psi	8,000	ASTM D638
Tensile Modulus	psi	15,100	ASTM D790
Flexural Modulus	psi	415,000	ASTM D790
Compressive Strength	psi	10,100	ASTM D695
Compressive Modulus	psi	196,000	ASTM D695
Izod Impact Strength (notched)	ft-lb/in o.n.	1.5	ASTM D256

D.5 Poly(ethylenechlorotrifluoroethylene) (ECTFE Halar 901)

Supplier: Ausimont, 10 Leonards Lane, Thorofare, NJ 08086
Material: Copolymer of ethylene and chlorotrifluoro-ethylene (CTFE), Kel-F
Chemical Formula: C₂H₂Cl_{0.5}F_{1.5}

Properties	Units	Measured Value	Testing Method
Physical Properties			
Density	g/cm ³	1.68	ASTM D792
Water Absorption	%	<0.1	ASTM D 570
Processing Characteristics			
Thermal Properties			
Maximum Use Temperature	°F	300	
Thermal Expansion	In/in/°F	5.6 x 10 ⁻⁵	ASTM D696
Heat Distortion Temperature	°F	194 (66 psi)	ASTM D648
Flammability			
Limiting Oxygen Index	%	52	ASTM D863
Burning Rate		V-0	UL Bulletin 94
Fire Propagation Index (FPI)	(m/s ^{1/2} /(kW/s) ^{2/3})	<<6	FMRC 4910
Mechanical Properties			
Tensile Strength Yield	psi	4300	ASTM D638
Tensile Strength Break	psi	7800	ASTM D 638
Elongation Break	%	250	ASTM D638
Flexural Modulus	psi	245,000	ASTM D790
Izod Impact Strength Notched	ft-lb/in	2.3 (104°F)	ASTM D256
Hardness-Rockwell R	Shore D	90/71	ASTM D785
Abrasion Resistance	1000 revs	0.006	Taber D1044
Notch Sensitivity		Not sensitive	—

D.6 Poly(vinylidene fluoride) (PVDF, Kynar 2850)**Supplier:** Westlake Plastics, P.O. Box 127, Lenni, PA 19052**Material:** Copolymer of vinylidene fluoride (VDF) and hexafluoropropylene (HFP)**Chemical Formula:** C₂H₂F₂

Properties	Units	Measured Value	Testing Method
Physical Properties			
Density	g/cm ³	1.76–1.78	ASTM D792
Melting Temperature	°F	311–320	ASTM D3418
Water Absorption	%	0.04 (77°F, 24 hrs)	ASTM D 785
Processing Characteristics			
Melt Viscosity	poise	23000–27000 (450°F, 100 sec ⁻¹)	ASTM D3835
Melt flow	g/10 min	3.5–7.5 (450°F, 27.562 lb)	ASTM D 1238
Mold Shrinkage	%	2–3	ASTM D955
Thermal Properties			
Coefficient of Linear Thermal Expansion	°F ⁻¹	7.8 x 10 ⁻⁵	ASTM D696
Deflection Temperature	°F	237 (66 psi)	ASTM D648
Flammability			
Limiting Oxygen Index	%	42	ASTM D2868
Thermal Decomposition	°F	707 (1 wt. Loss)	TGA
Burning Rate		V–0	UL Bulletin 94
Fire Propagation Index (FPI)	(m/s ^{1/2} /(kW/s) ^{2/3})	6	FMRC 4910
Mechanical Properties			
Tensile Strength	psi	4500–5500	ASTM D638
Tensile Break Strength	psi	4000–6000	ASTM D 638
Break Elongation	%	300–400	ASTM D638
Tensile Modulus	psi	110000–140000	ASTM D638
Flexural Modulus	psi	160000–180000	ASTM D790
Izod Impact Strength Notched Unnotched	ft-lb/in	6–10 No break	ASTM D256
Hardness	Shore D	70	ASTM D2240

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