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MEASUREMENTS OF TRITIUM RELEASE FROM GAS CHROMATOGRAPH DETECTOR FOILS

R. G. Niemeyer

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MEASUREMENTS OF TRITIUM RELEASE FROM GAS CHROMATOGRAPH DETECTOR FOILS

R. G. Niemeyer

Isotopes Division

DECEMBER 1971

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MEASUREMENTS OF TRITIUM RELEASE FROM GAS CHROMATOGRAPH DETECTOR FOILS

R. G. Niemeyer

ABSTRACT

A study of tritium losses from foils used in gas chromatograph detectors showed that the losses increased slowly with temperature to a break point above which the losses increased at a much higher rate. The approximate break points for the 250-, 500-, and 1000-mCi/in.^{2} foils were 238, 198, and 200°C, respectively.

Tritium losses were relatively high (38 to 170 μCi/hr) during the first hour of operation of a new foil. After 2 to 10 hr of operation, the loss rates decreased to values which were 26 to 64% of the initial 1-hr loss rates.

Larger losses (21 to 353 μCi/hr) occurred during ultrasonic cleaning of new foils in 5% alcoholic KOH, heptane, hexane, and toluene; it is felt that less vigorous cleaning procedures should be sought to reduce these losses.

Variations in losses as a function of foil heating rate, carrier-gas flow rate, and gas composition were relatively insignificant.

Ninety-eight to 100% of the tritium losses were in the form of tritium oxide.

INTRODUCTION

Tritium foils used in certain types of gas chromatograph detectors were tested for tritium loss under various conditions of operation. These foils are made by evaporating titanium metal onto a stainless steel backing and then reacting tritium with the titanium. They are commonly made in activities of 0.25, 0.5, and 1.0 Ci of tritium per in.^{2} of foil surface for gas chromatograph use. The foils are used in three types of gas chromatograph detector units: argon ionization detector, electron capture detector, and cross-section detector. Although radium, ^{63}Ni, and ^{90}Sr are sometimes used as the radiation source in these detectors, a recent survey of manufacturers indicated that about 80% of the units sold using radiation type detectors employed tritium foils.[1] The usual range of activity in a tritium unit is from 100 to 300 mCi. The devices are generally licensed with the stipulation that they not be operated at temperatures in excess of 225°C. However,

[1]G. A. Pettigrew and J. A. Halperin, *The Use of Radioactive Materials in Gas Chromatography*, BRH/DMRE 70-5, U. S. Dept. of Health, Education, and Welfare, October 1970.

a gas chromatograph is usually designed to operate with any one of several different types of detector units and, therefore, the column oven is usually able to achieve temperatures up to about 500°C. Therefore, one of the operating variables of interest is the tritium release rate as a function of operating temperature. Other operating variables are listed in Table 1.

Table 1. Gas Chromatograph Tritium Foil Detector Operating Variables

Variable	Conditions
Temperature	Ambient to 500°C
Carrier gas	H_2, Ar, CH_4, N_2, and He
Foil specific activity	0.25, 0.5, and 1.0 Ci/in.^{2}
Gas flow rate	0.1, 2.0, and 10.0 ml/sec
Foil heating rate	10°C/min and 60°C/min
Run time	1, 2, and 3 hr
Foil cleaning method	Detergents, alcohol, ultrasonics

Foils having nominal tritium concentrations of 250, 500, and 1000 mCi/in.^{2} were obtained from the manufacturer for testing. The 250-mCi/in.^{2} foils were manufactured differently from the other foils in that the tritium was absorbed from a gas mixture containing ∿75% deuterium and ∿25% tritium; whereas for the other two types of foils, the tritium was absorbed from a pure tritium atmosphere. The areal density of the titanium deposits stated by the manufacturer was approximately 1, 0.5, and 1 mg/cm^2 for the 250-, 500-, and 1000-mCi/in.^{2} foils, respectively. The dimensions of the foils were 0.5 x 0.5 x ∿0.002 in.

Tests were made to determine the effect of temperature, heating rate, gas flow rate, type of gas, foil cleaning procedure, and operating time on the tritium losses. The amounts of elemental tritium and tritium oxide were determined separately in each case, since the biological effect of the oxide is much greater than that of elemental tritium. In all of the tests performed, it was found that 98-100% of the released tritium was in the form of tritium oxide.

TEST EQUIPMENT

The equipment used for the tests (Fig. 1) consisted of a containment vessel positioned in a tube furnace, a metered dry gas supply, and a tritium trapping system. The temperature of the foil and the gas stream was controlled by a calibrated temperature controller and thermocouple. The gas flow was controlled using rotometers calibrated for each type of gas used

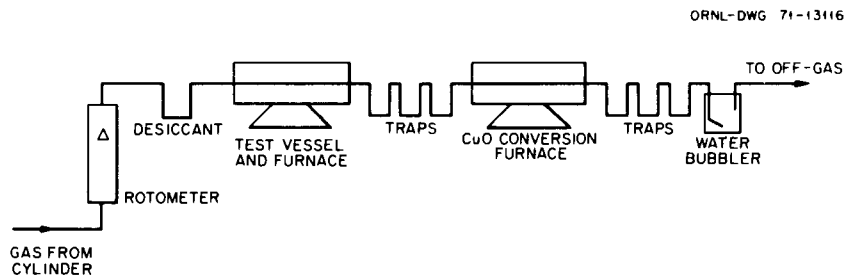

Fig. 1. Apparatus for Testing Tritium Foils.

in the test series. The trapping system consisted of a series of U-traps which were packed with glass beads and immersed in dry ice-trichloroethylene baths. The gas flowed through a tube of desiccant to remove any water vapor, then flowed past the test foil and through three cold traps to remove tritium oxide. The gas then passed through a bed of hot (700°C) copper oxide to convert the elemental tritium to tritium oxide, which was then removed from the gas stream in a second series of two cold traps. The exit gas was then passed through a backup water scrubber before going to the hot off-gas system. Analysis of the samples was by beta liquid scintillation counting. The activity found in the water scrubber was <0.001 μCi in every case.

STANDARD TEST PROCEDURE

In order for the results of the various tests to be comparable, the following standard test conditions were used:

1. A new foil was used for each test.

2. Foil operating temperature (also gas temperature) was 150°C.

3. Time used to heat the foil (and gas) from 20°C to the operating temperature was ∿4 min (average rate ∿32.5°C/min).

4. Sample time was 1 hr. Sampling started when foil reached operating temperature and continued for 1 hr.

5. Gas composition was argon.

6. Gas flow rate was 120 ml/min.

In order to test the effect of a particular variable, the standard test procedure was varied with respect to that variable, while all the other conditions were kept constant. These deviations are stated in the sections where the test results are given. Before inserting a foil into the equipment for testing, the air was removed from the system by purging with the carrier gas. During storage prior to testing, the foils were kept in a low-humidity environment by means of a desiccant. For each of the three

types of foils tested, selection of the test samples for each test series was based on the manufacturer's activity measurements so that activity differences between samples would be minimized.

Tritium collected in the traps was measured by liquid scintillation counting. In order to avoid dilution errors, the scintillation fluid was added directly to each trap following each run. Prior to counting each series of samples, the scintillation counter was standardized against an NBS standard and was found to be within ±1% of the standard. The limit of detection is 0.001 μCi/ml. In each of the tests, at least two and sometimes as many as ten separate determinations were made for each type of foil, and the reported results are an average of these determinations. Since there is substantial variation in the amount of activity on the foils as shown in the section on Total Activity Measurement, the overall results are estimated to be within ±25% accuracy.

EXPERIMENTAL RESULTS

Reproducibility Tests

Before determining the tritium losses from the foils under the various test conditions, a series of tests was performed to evaluate the reproducibility of the experimental results. These tests were designed to evaluate variations in the measured tritium losses due to inherent characteristics of the foils, procedures, and equipment used. The test conditions used for the reproducibility tests were the standard test conditions previously described, involving heating the foil from 20 to 150°C in 4 min in an argon flow of 120 ml/min and then obtaining a 1-hr sample at these conditions.

The results of these tests are given in Table 2. For each type of foil the tritium losses were within ±22% of the average loss.

The density thickness of the titanium layer is also given in Table 2, because the tritium loss appeared to be related to this variable. The 500- and 1000-mCi/in.^{2} foils were manufactured in the same manner except that the latter foils had a titanium layer which was twice as thick as that of the 500-mCi/in.^{2} foils. However, the losses of the 1000-mCi/in.^{2} foils were less than one-third as much as for the 500-mCi/in.^{2} foils. This apparent effect of decreasing tritium loss, occurring with increasing titanium layer thickness and increasing tritium concentration per unit area of foil, has also been observed when similar foils having a copper backing were tested for tritium loss. This same effect was observed in all of the tests made in this study.

Table 2. Tritium Losses in Reproducibility Tests

	Foil Activity (mCi/in.^{2})		
	250	500	1000
Number of foils tested	10	5	7
Density thickness of titanium layer (mg/cm^2)	1	0.5	1
Arithmetic mean loss (μCi)	4.9	51.1	14.9
Range of tritium losses (μCi)	3.95-5.97	44.1-55.2	12.6-18.0
Variance (μCi)	0.43	19.9	4.64
Standard deviation (μCi)	0.65	4.46	2.16

Total Activity Measurements

Radiation measurements were made to determine the relative range of activity in eight new foils taken from each of the three activity levels. The measurements were made at 0.7 cm with a cutie pie and at 3.6 cm with a G-M tube in a shielded counting chamber. Both detectors had 1.5-mg/cm^2 windows. Since these instruments were not calibrated for tritium, the measured dose rates are not accurate. However, the relative response of the instruments to each foil is a reasonable comparative indication of differences in the radiation output of the foils and, therefore, will show major differences in the contained activity on the foils. The data given in Table 3 show considerable variation between the foils in each group and also indicate that the saturation ion current measurement, used by the manufacturer to obtain the surface activity level of a foil, is not a sensitive measurement of the total tritium content of the foils.

Tests were then conducted to determine the total tritium content in the foils by heating the foils at 600°C for 2.5 hr in a flow of air. The amount of tritium released from the nominal 250-, 500-, and 1000-mCi/in.^{2} foils was determined for each of four foils of each activity level. The results (Table 4) indicate that considerably more tritium is present in the foils than is indicated by the manufacturer's stated nominal activity.

Table 3. Radiation Measurements of Tritium Foils

Foil No.	Saturation Ion Current[a] (Amp x 10^{-8})	Cutie Pie (mR/hr)	G-M Tube (counts/min)
	250-mCi/in.^{2} Foils		
1	1.2	68	34,200
2	1.2	54	27,200
3	1.2	54	28,600
4	1.2	90	44,600
5	1.2	95	46,900
6	1.2	52	26,900
7	1.2	93	45,400
8	1.2	87	44,800
	500-mCi/in.^{2} Foils		
1	3.7	110	53,300
2	3.7	110	54,700
3	3.5	93	47,000
4	3.5	100	48,800
5	3.5	98	47,800
6	3.5	105	51,400
7	3.4	98	48,300
8	3.0	84	41,200
	1000-mCi/in.^{2} Foils		
1	5.1	310	122,600
2	5.0	220	96,500
3	5.0	280	114,100
4	5.0	220	99,300
5	4.9	260	109,200
6	4.8	300	120,800
7	4.7	220	95,200
8	4.7	313	123,300

[a]Data reported by manufacturer of foils.

Table 4. Comparison of Manufacturer's Nominal Activity With Experimental Determination of Activity in Foils

Manufacturer's Nominal Activity		Average of Four Determinations	Determination for each Foil
mCi/in.^{2}	mCi/foil	(mCi)	(mCi)
250	62.5	124	108, 130, 147, 112
500	125	161	148, 235, 157, 106
1000	250	380	360, 550, 314, 498

Operating Temperature Tests

Tritium losses were determined with temperature as the only variable, because it was known from previous testing of similar foils having higher activity concentrations (1-15 Ci/in.^{2}) that temperature is a significant factor in the tritium loss rate and that the loss rate exhibits a marked increase at temperatures in the range of about 175-230°C. Since this marked increase in loss rate occurs within the useful temperature range of the gas chromatograph, it was desirable to determine more precisely the temperature at which it occurs. The number of foils tested at each temperature ranged from 2 to 10.

In measuring the losses, the standard test procedure was used except that tests were made at each of several temperatures ranging from 125 to 275°C in order to cover the range of interest. The results of the test are shown in Fig. 2. The losses increased slowly with temperature to a break point above which the losses increased at a much higher rate. The break points for the 250-, 500-, and 1000-mCi/in.^{2} foils were ∿238, 198, and 200°C, respectively. These points are the temperatures at which the observed data would no longer fall on a straight line of constant slope. However, as seen in Fig. 2, the sharp change in release rate occurs at a temperature of ∿260°C for all three types of foils.

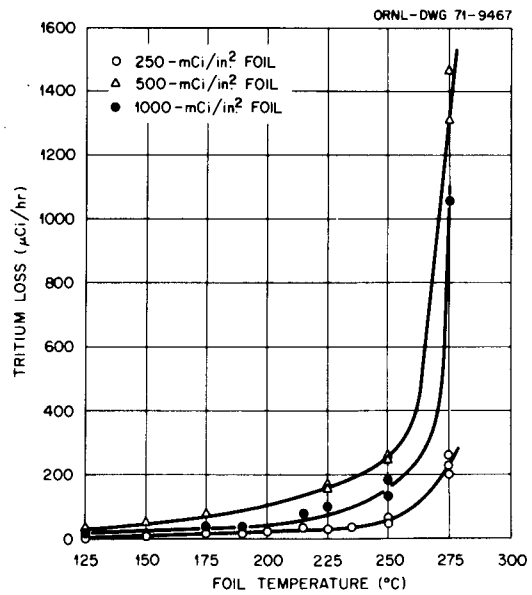

Fig. 2. Tritium Loss as a Function of Temperature in Argon.

These data also show that the 500-mCi/in.^{2} foils have a higher release rate than both the 250- and 1000-mCi/in.^{2} foils. This higher rate was observed consistently throughout all the different tests in this program and is attributed to the lower titanium areal density of the 500-mCi/in.^{2} foils as compared to the other two types.

Operating Time Tests

Tritium losses as a function of operating time at 225°C (maximum temperature allowed by USAEC regulations) were obtained in an argon flow for each type of foil. The losses were determined during each of the first two 1-hr periods and during each of the following eight 2-hr periods for each foil. The results are shown in Fig. 3. The losses shown at 1 and 2 hr are the total losses occurring during the first and second hour of operation, respectively. The loss shown at 4 hr is the total loss occurring during the third and fourth hour, since it covered a 2-hr

sample period. Similarly, the remaining samples up to 18 hr cover tritium losses for 2-hr periods. One foil of each type was tested.

As can be seen from Fig. 3, the initial losses from new foils were relatively high during the first few hours of operation. The losses decreased rapidly with time and reached a nearly constant value. Since 225°C is the maximum allowable temperature at which these foils can be used, the losses given in Fig. 3 represent the highest losses expected in normal operation under the test conditions. These foils should still be useful since the count rates as described in the section on Radiation Measurements were the same after the tests as they were before the tests.

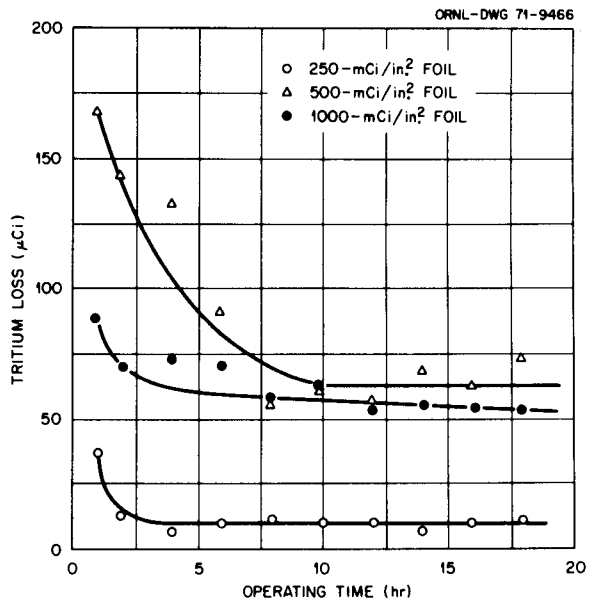

Fig. 3. Tritium as a Function of Time in Argon Flow at 225°C.

Heating Rate Tests

This test was performed to determine the effect of heating rate on the tritium losses from the foil. The standard test procedure (average heating rate, 32.5°C/min) was used for one set of foils, and a second set of foils was tested using the same conditions except that a much lower foil heating rate (average heating rate, 5.2°C/min) was used. The results of these tests (Table 5) show that foil heating rate had no significant effect on tritium losses obtained during the standard tests, within the range of heating rates tested. Two foils of each activity level were tested.

Table 5. Tritium Loss As a Function of Heating Rate

Foil Activity (mCi/in.^{2})	Time to Reach Operating Temperature (min)[a]	Tritium Loss (μCi/hr)
250	4	5.1
	25	5.2
500	4	50.1
	25	54.6
1000	4	17.5
	25	18.0

[a]Average heating rate of 32.5°C/min was used for 4-min test and 5.2°C/min was used for 25-min tests.

Tests were also performed to determine the tritium losses from two new 1000-mCi/in.^{2} foils during the period in which the foil is heated in an argon flow from room temperature to 150°C. The foils were new and were selected so that their radiation count rates at 3.6 cm from the detector were nearly the same (within 2%). The first foil was tested by determining the loss during heating the foil to 150°C in 4 min. The foil was then allowed to cool in an argon flow to room temperature, and the test was repeated. The cooled foil was heated to 150°C over a 24-min period, and the loss was determined over that time period. The tests of the second foil were the same as those of the first foil except that the heating rates were 24, 24, and 4 min for the respective tests as shown in Table 6.

The data (Table 6) show that the total loss is low (0.3 to 2.6 μCi) during heat-up in both of the heating rate tests of the foils. The results indicate that there may be some advantage on both average and overall bases to the longer heat-up cycle; however, the losses are minor in both cases when compared to the losses during operation.

Table 6. Tritium Loss from 1000-mCi/in.^{2} Foils as a Function of Heating Rate

Test No.	Time[a] to Reach 150°C (min)	Tritium Loss (μCi)	Average Tritium Loss Rate (μCi/min)
		First Foil	
1	4	2.60	0.650
2	4	1.22	0.305
3	24	1.47	0.061
		Second Foil	
1	24	3.45	0.144
2	24	0.81	0.034
3	4	0.32	0.080

[a]Average heating rate of 32.5°C/min was used for 4-min tests and 5.4°C/min was used for 24-min tests.

Gas Composition Effects Tests

Four of the common carrier gases used in gas chromatography were tested to determine if tritium loss varied due to inherent properties of the gas being used. The standard test procedure was used for these tests except for the type of carrier gas. The averaged results are shown in Table 7, which gives the loss rate for each type of foil when tested in each of the four gases. The results indicate that there are no major differences in the loss rate when any of the four carrier gases are used. There was

Table 7. Tritium Losses As a Function of Gas Compositon

Foil Activity (mCi/in.^{2})	Gas Composition	Tritium Loss (μCi/hr)
250	Argon	4.9
	Nitrogen	3.3
	Helium	4.1
	Ar-10% CH_4	4.1
500	Argon	51.1
	Nitrogen	50.0
	Helium	39.9
	Ar-10% CH_4	62.6
1000	Argon	14.9
	Nitrogen	16.1
	Helium	15.8
	Argon-10% CH_4	17.9

also no significant difference among the gases in the proportion of elemental tritium (∿2%) to tritiated water (∿98%) that was collected in the sample traps. Two to ten foils of each type were tested.

Flow Rate Tests

These tests were designed to detect any effect on tritium loss due to carrier-gas flow rate. Tritium losses were measured using the standard conditions except that one of the sets of foils was tested using a gas flow rate of 60 ml/min instead of the standard flow of 120 ml/min. The results (Table 8) indicate that gas flow rate was not a significant factor in the tritium loss rates. Three to ten foils of each type were tested.

Table 8. Tritium Loss As a Function of Argon Flow Rate

Foil Activity (mCi/in.^{2})	Argon Gas Flow Rate (ml/min)	Tritium Loss (μCi/hr)
250	60	3.3
	120	4.9
500	60	51.4
	120	51.1
1000	60	19.2
	120	14.9

Cleaning Procedure Tests

A series of tests was made to determine the effect of cleaning procedure on tritium losses. One foil of each activity (250, 500, and 1000 mCi/in.^{2}) was tested by ultrasonically cleaning for 1 hr. The cleaning solvents were 5% alcoholic KOH, heptane, hexane, and toluene. In these tests a new foil was placed in a vial containing 10 ml of solvent. The vial was sealed and immersed in water in a 125-W ultrasonic cleaner. The active surface of the foil was facing up to prevent abrasion of the titanium tritide surface. After the foil was cleaned, the solvent was filtered to remove any titanium tritide which may have been dislodged from the foil. The foil and filter were then rinsed with clean solvent to remove tritium held by the liquid. The combined rinse and filtrate was analyzed by liquid scintillation counting. The amount of activity in the filter was found by heating the filter to 600°C for 2.5 hr and trapping the tritium in a recovery train. The dry foil and the dry filter were examined under a microscope to observe the amount and size of the particulates on the filter and to determine whether particles had been lost from the surface of the foil. The tritium loss from the foil was then determined under the standard operating test conditions (150°C, 1 hr, argon). The entire procedure was repeated three times to obtain the effect of repeated cleaning cycles on the same foil. Radiation measurements made before and after the tests were nearly the same, indicating that the usefulness of the foils was not affected by the tests.

The results of the tests are given in Table 9. Several factors could have affected the tritium losses which occurred in these tests. The corrosion rate of solid titanium in 5% KOH at room temperature is reported to be 0.04 mil/year. The rate would be expected to be higher in the source material because of the porosity of the vapor deposition process used in manufacturing the foils. Even small amounts of corrosion could conceivably alter the surface characteristics of the titanium in such a way as to affect the tritium losses.

The ultrasonic cleaning created considerable shear force at the liquid-metal surface and may have removed any loosely held tritium from the surface. Since these forces are not uniform throughout the cleaner tank, some differences are to be expected due to position of the foil in the tank.

Operating time and temperature both affect the losses as previously shown in Figs. 2 and 3. The repeated cleaning of the foils would be expected to reduce the losses during operation immediately following cleaning by removing tritium at the foil surface faster than it was being replaced with tritium from the deeper layers of the foil.

After the tests were completed, it was found that the filtrate contained some solids which had passed through the 0.45-μm filters. The seven samples which had not been disposed of were centrifuged and assayed again. The resulting values for tritium in the filtrate indicated that 44 to 97% of the tritium in the filtrate was in the form of fine solids (average of 7 samples 53%). The test results for total tritium loss (filtrate plus

Table 9. Tritium Losses Due to Foil Cleaning Procedures

Cleaning No.	Losses During Cleaning (μCi)															
	In 5% Alcoholic KOH				In Hexane				In Heptane				In Toluene			
	Filtrate	Filter	Total	Standard Test	Filtrate	Filter	Total	Standard Test	Filtrate	Filter	Total	Standard Test	Filtrate	Filter	Total	Standard Test
							250-mCi/in.^{2} Foils									
1	27.8	48.4	76.6	4.0	0.18	80.7	80.9	4.1	0.78	41.7	42.5	8.3	0.62	42.7	43.3	2.6
2	8.9	21.8	30.7	2.3	0.05	77.3	77.4	1.8	0.14	76.9	77.0	1.4	0.54	64.5	65.0	1.3
3	4.6	15.3	19.9	2.1	0.03	8.7	8.7	1.7	0.02	22.1	22.1	1.0	1.62	4.5	6.1	1.4
							500-mCi/in.^{2} Foils									
1	75.2	74.6	149.8	50.7	0.16	87.2	87.4	22.3	0.31	55.4	55.7	15.3	2.11	102.5	104.6	27.3
2	19.6	13.5	33.1	25.2	0.05	19.9	20.0	7.2	3.72	107.7	111.4	7.4	1.09	108.0	109.1	4.4
3	6.9	15.2	22.1	18.5	0.11	23.9	24.0	0.4	0.14	61.1	61.2	6.2	0.29	93.5	93.8	6.0
							1000-mCi/in.^{2} Foils									
1	65.3	120.3	185.6	8.3	0.34	21.7	22.0	10.8	0.07	127.	127.1	10.6	0.30	353.5	353.8	9.4
2	1.5	46.4	47.9	3.3	2.44	199.3	201.7	3.9	0.09	76.8	76.9	3.7	0.43	135.8	136.2	2.5
3	0.8	12.3	13.1	3.6	0.13	32.9	33.0	5.6	0.37	93.9	94.3	2.0	0.11	145.1	145.2	5.2

filter) should not be significantly affected by these fine particles, but it is of interest to know that they exist when disposing of the waste cleaning solutions. As can be seen in Table 9, most of the losses were found on the filters, indicating that the ultrasonic cleaning was removing solids from the foil surface in significant quantities. The magnitude of these losses suggests that less vigorous cleaning procedures should be sought in order to reduce the losses during cleaning.

Several of the 0.45-μm filters were examined at a magnification of 400X to measure particle sizes of the solids filtered from the cleaning solutions. Each of the filters examined had one or two large particles having widths of about 50-75 μm. Most of the visible particles were in the range of 0.5-5 μm. The limit of measurement was ∿0.5 μm. Examination of the cleaned foils revealed the areas where the large particles had been dislodged. No instances were found in which separation or peeling of the titanium from the stainless steel backing occurred.

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