

ICP-Optical Emission Spectroscopy

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The Analysis of Metals in Disposable, Non-Medical Face Masks in Accordance with ISO 18562-4:2017 Using the Avio 550 Max ICP-OES

Introduction

With COVID-19 becoming a global pandemic, the utilization of face masks to limit transmission of the viral illness has gained global traction. While a large variety of commercial and homemade face masks

are available, the most common type used is the 3-layer disposable, hygienic face mask consisting of an innermost layer (closest to the wearer's face), a middle layer, and an outer light blue layer, as shown in Figure 1.

As with any consumer product, it is important that these face coverings are free of chemicals or hazards for the people that wear them. Two categories of analytes that should be monitored are volatile organic compounds (VOCs) and metals. This work will discuss the determination of metals in disposable, non-medical face masks.

While no specific methods exist for the analysis of metals in non-medical face coverings, ISO method 18562-4:2017 Biocompatibility of Breathing Gas Pathways in Healthcare Applications – Part 4: Leachables in Condensate¹ may be relevant and applied, since it discusses the sample preparation, analytes and methodologies used to evaluate data quality.

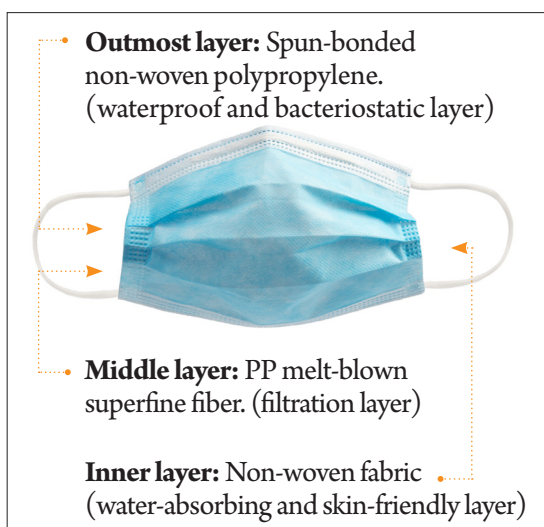


Figure 1. Components of a common, disposable, hygienic (non-medical) face mask.

ISO 18562-4:2017 states that the analytes and limits are defined by USP <232>,² and the methodology should be evaluated by USP <233>.³ Since face masks cover the nose and mouth, ISO 18562 states that the route of exposure is inhalation, with a maximum daily intake of condensate of 1 mL. Table 1 shows the analytes and maximum per daily exposure (PDE) for inhalation, as defined in USP <232>.

Table 1. Analytes and Maximum PDE for Inhalation, as Defined in USP <232>.

Elements	PDE (µg/day)
Au, Hg, Ir, Os, Pd, Pt, Rh, Ru, V	1
As	2
Cd, Co, Cr	3
Ni, Pb	5
Ag	7
Tl	8
Mo	10
Sb	20
Li	25
Cu	30
Sn	60
Se	130
Ba	300

To determine the analytical concentrations, USP <232> defines the relevant concentrations as the J value, according to the following equation:

$$J = \frac{\text{PDE}}{\text{Maximum Daily Dose} \times \text{Dilution Factor}}$$

PDE = maximum permissible daily exposure (µg/g)

Maximum Daily Dose = maximum amount of the medication consumed

Dilution Factor = dilution used in sample preparation = mass of sample/sample preparation volume

More details about USP <232> and <233> are available.⁴

ISO 18562-4:2017 also discusses sample preparation considerations, a summary of which is available.⁵ The important points are that analytes should be leached from the portion of the face mask closest in contact to the wearer at 37 °C (normal body temperature) for a time representative of the duration the face mask worn.

After sample preparation is complete, the quality of the methodology must be evaluated via the criteria specified in USP <233>, as summarized in Table 2.

This work describes the sample preparation and analysis of metals in disposable, non-medical face masks with the PerkinElmer Avio® 550 Max ICP-OES to meet the criteria defined in ISO 18562-4:2017. The disposable, non-medical face masks are not only the most used, but they also are the most challenging to analyze due to the light weight of the inner layer, which results in lower analyte concentrations from the J-value calculations.

Table 2. Analytical Criteria Defined in USP <233> for Quantitative Procedures.

Criteria	Description
Accuracy	Spike recoveries of 0.5 J, 1 J, and 1.5 J must be between 70-150%
Repeatability	The RSDs of measurements of six independent samples spiked at J must be less than 20%
Ruggedness	Six solutions must be analyzed on different days, with different instruments, or with different analysts. The RSDs over the 12 measurements must be less than 25%
System Suitability	The difference in the results of the high calibration standard (1.5 J) measured at the beginning and end of a batch must be < 20%

The Avio 550 Max is a fully simultaneous ICP and was chosen for this application due to its exceptional stability, its ability to conduct analysis in high resolution, which aids in interference removal, and its low argon consumption. The Avio 550 has a robust plasma at only 8 L/min plasma flow, which results in significant cost savings for the laboratory over the lifetime of the instrument.

Experimental

Samples and Sample Preparation

A variety of disposable, 3-ply, non-medical grade face masks were purchased locally and the inner-most inner layer removed. Depending on the brand, the inner layers ranged in mass from 0.50-0.58 g, with most being ≈ 0.55 g. To minimize the creation of additional surface area and to better simulate what a wearer would be exposed to, the inner layers were not cut into smaller pieces, but rather the mask was leached whole.

For sample preparation, each inner layer was added to a 50 mL autosampler tube and pushed to the bottom, followed by the addition of 10 mL of 10% HCl + 1% HNO₃ (v/v). Nitric acid was used to stabilize most elements, while the HCl was required to stabilize the platinum group elements; 10% HCl was required to stabilize gold (Au) in solution over the leaching time period. The tubes were capped and shaken before being heated in an SPB Sample Preparation Block (PerkinElmer Inc., Shelton, Connecticut, USA) at 37 °C for four hours to simulate a common time a person may wear a face mask continuously within a typical day. After four hours, the acid was decanted into 15 mL autosampler tubes for analysis.

The validation procedure for USP <233> requires several pre-sample preparation spikes to make sure that no analyte is lost during the sample preparation procedure. Sample spikes (0.5 J, 1 J, 1.5 J) were added to empty 50 mL autosampler tubes, followed by the inner layers. The inner layers were pushed to the bottom of the autosampler tubes to maximize contact with the spike solutions and allowed to sit for 30 minutes to absorb as much of the spike solutions as possible. The acid mixture was then added so that the final volume of liquid added to each tube was 10 mL. The sample preparation continued as for the unspiked mask inner layers.

Calibrations were made against external calibration curves at 0.5 J and 1.5 J (as specified in USP <233>), and yttrium (Y) was added to all blanks, calibration standards and samples as an internal standard. Table 3 shows the analytes and their concentrations at different J values used in this work.

Table 3. Calibration Concentrations at Different J Values.

Class	Element	0.5 J (mg/L)	1 J (mg/L)	1.5 J (mg/L)
1	As	0.055	0.110	0.165
	Cd	0.083	0.165	0.248
	Hg	0.028	0.055	0.165
	Pb	0.138	0.110	0.413
2	Ag	0.193	0.385	0.578
	Au	0.028	0.055	0.083
	Co	0.083	0.165	0.248
	Ir	0.028	0.055	0.083
	Ni	0.138	0.275	0.413
	Pd	0.028	0.055	0.083
	Pt	0.028	0.055	0.083
	Rh	0.028	0.055	0.083
	Ru	0.028	0.055	0.083
	Se	3.58	7.15	10.7
	Tl	0.220	0.440	0.660
	V	0.028	0.055	0.083
	3	Ba	8.25	16.5
Cr		0.083	0.165	0.248
Cu		0.825	1.65	2.48
Li		0.688	1.38	2.06
Mo		0.275	0.550	0.825
Sn		1.65	3.30	4.95

Instrumental Conditions

All analyses were performed on an Avio 550 Max ICP-OES using the conditions and parameters in Table 4, along with the analytes and wavelengths in Table 5. For maximum sensitivity, all analytes were measured with an axial plasma view, while high resolution was used to separate analytes from potential interferences. Despite the sensitivity reduction with high resolution, axial viewing allowed even the low concentration analytes to be measured with more consistent results. Multicomponent Spectral Fitting (MSF)⁶ was applied to Ir 208.882 to more easily resolve the peak from the baseline noise. These analyses were accomplished with a total argon consumption of 9 L/min, reducing argon usage and cost.

Table 4. Avio 550 Max ICP-OES Instrumental Parameters and Conditions.

Parameter	Description/Value
Nebulizer	SeaSpray
Spray Chamber	Baffled Glass Cyclonic
Sample Uptake Rate	0.9 mL/min
RF Power	1500 W
Injector	2.0 mm id alumina
Nebulizer Gas Flow	0.60 L/min
Auxiliary Gas Flow	0.2 L/min
Plasma Gas Flow	8 L/min
Torch Position	-3
Integration	Auto
Read Time Range	1-10 seconds
Replicates	3
Resolution	High
Plasma View	Axial

Table 5. Analytes and Wavelengths.

Element	Wavelength (nm)	Element	Wavelength (nm)
Ag	338.289	Pb	220.353
As	188.979	Pd	340.458
Ba	233.527	Pt	265.945
Cd	214.440	Rh	343.489
Co	238.892	Ru	240.272
Cr	267.716	Sb	217.582
Cu	327.373	Se	196.026
Hg	194.168	Sn	189.927
Ir	208.882	Tl	190.801
Li	670.784	V	309.310
Mo	202.031	Y (int std)	371.029
Ni	231.604		

Results and Discussion

Sample Analysis

To assess the presence of analytes in the mask inner layers, unspiked inner layers were measured after four hours. The concentrations for all elements in all samples were less than 0.3 J, a common actionable threshold which is more than three times lower than the PDE. These results indicate that the inner layers do not pose a danger to the wearer and that the acids used for the leaching do not extract excess analytes over time.

Meeting the USP <233> Criteria

To meet the criteria outlined in Table 2, the inner layers from one brand of masks were chosen to run through the full validation requirements from the four-hour leaches.

First, the system suitability was determined by measuring the 1.5 J standard at the beginning (after calibration) and end of a batch analysis. Figure 2 shows that the drift is 6% or less for all elements, well less than the acceptance criteria of 20%.

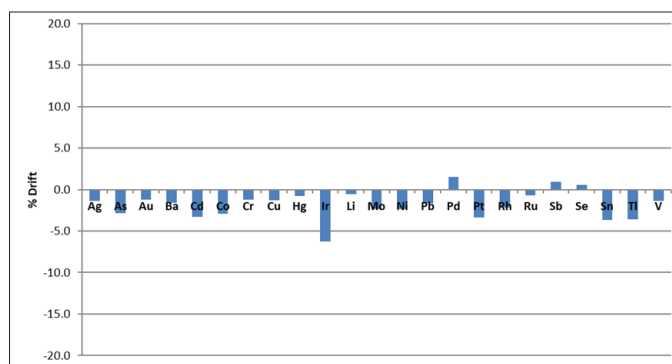


Figure 2. System suitability: drift over a batch analysis from a 1.5 J standard measured at the beginning and end of a batch analysis.

With the system suitability established, the accuracy of the methodology was evaluated. To meet the USP <233> criteria, pre-leach spikes of 0.5 J, 1 J, and 1.5 J were added as described earlier in the Sample Preparation section and carried through the complete sample preparation process. Figure 3 shows excellent recoveries for all analytes (with the exception of Au and Pd), easily meeting the USP <233> criteria, indicating the stability of the methodology. However, Au recovers at less than 70%, while Pd recovers at 70%.

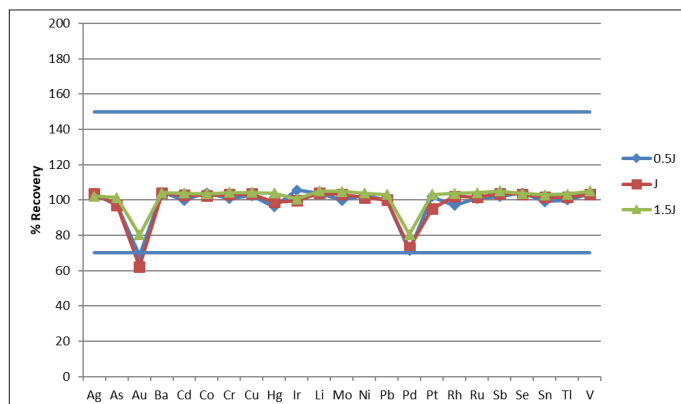


Figure 3. Accuracy study: 0.5 J, 1 J, and 1.5 J recoveries from a four-hour leach of mask inner layers. The blue lines represent the USP <233> limits (70-150%).

To investigate Au and Pd, another accuracy leach study was done, although this time, without the mask inner layers. The leach procedure was followed as described in Sample Preparation, except the mask inner layers were not added to the sample tubes – only the acid mixture was used. The recoveries from the blank accuracy study appear in Figure 4 and show that all elements recover within 10%, including Au and Pd. These results suggest that Au and Pd are sticking to the mask inner layers and are not extracted during the leach process. The implication is that wearers do not have to worry about inhaling Au and Pd if they are present in face mask inner layers.

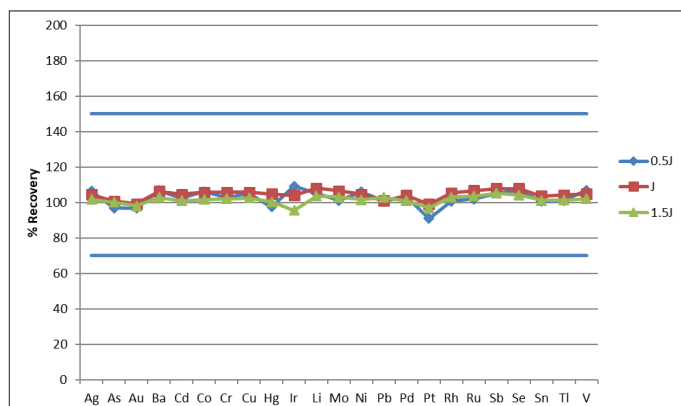


Figure 4. Accuracy study: 0.5 J, 1 J, and 1.5 J recoveries from a 4-hour leach of acid blanks.

With the accuracy established, the repeatability of the method was evaluated by spiking six mask inner layers at the 1 J values prior to leach. As shown in Figure 5, the relative standard deviations (RSDs) for the six samples are 4% or less, well below the 20% USP <233> limit.

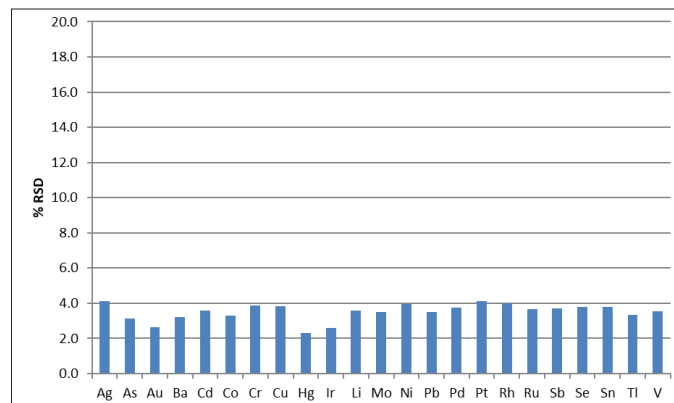


Figure 5. Repeatability study: %RSDs for six mask inner layers spiked at 1 J over four hours.

Finally, the ruggedness of the methodology was evaluated by analyzing the same six samples used for the repeatability study on a second day. The RSDs of the 12 measurements are 6% or less (Figure 6), well below the USP <233> criteria of < 25%.

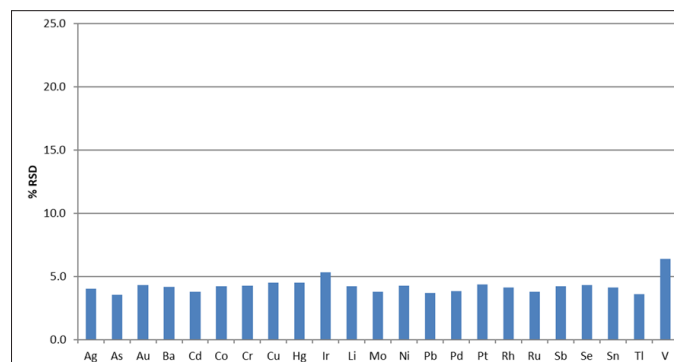


Figure 6. Ruggedness study: %RSDs for analysis of pre-leach 1 J spikes over two days (12 measurements).

Conclusions

This work demonstrates the ability of the Avio 550 Max ICP-OES to effectively measure metals in non-medical, disposable mask inner layers, following ISO 18562-4:2017, which references the analytes and validation from USP <232> and <233>. The four-hour leach simulates how a person would wear a mask during a typical day. The results indicate that the methodology easily meets the criteria for USP <233> for all analytes, with the exception of Au and Pd which had low spike recoveries, the result of their sticking to the mask inner layers. With low argon consumption, day-to-day stability, and the ability to be operated in high resolution mode, the Avio 550 Max is the ICP of choice for the evaluation of metals in mask inner layers.

References

1. ISO 18562-4:2017 Biocompatibility Evaluation of Breathing Gas Pathways in Healthcare Applications – Part 4: Leachables in Condensate, ISO 2017.
2. General Chapter <232> Elemental Impurities – Limits, 2nd Supplement of USP 35-NF 30, United States Pharmacopeia.
3. General Chapter <233> Elemental Impurities – Procedures, 2nd Supplement of USP 35-NF 30, United States Pharmacopeia.
4. “Implementation of USP New Chapters <232> and <233> of Elemental Impurities in Pharmaceutical Products”, PerkinElmer white paper, 2013.
5. “The Determination of Metals in Non-Medical Face Masks”, PerkinElmer white paper, 2020.
6. “Multicomponent Spectral Fitting”, PerkinElmer technical note, 2017.

Consumables Used

Component	Part Number
SeaSpray Nebulizer	N0811305
Sample Uptake Tubing, Black/Black (0.76 mm ID), PVC	N0777043 (Flared) 09908587 (Non-Flared)
Drain Tubing, Gray/Gray (1.14 mm ID), Santoprene (1.30 mm ID)	N0777444
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)
ICH Elemental Impurities Standards Kit – Inhalation PDEs	N9304375
Yttrium, 1000 mg/L	N9303810 (125 mL) N9300167 (500 mL)