# Enabling Inert, High-Temperature Spectroscopy with Specac-Harrick Reaction Chambers

## A SilcoTek® Case Study with:

Specac
analysis advancing life

## The Objective:

Researchers at Washington State University needed to study the photocatalytic interaction between dimethyl methylphosphonate (DMMP) and titania using diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS). The experiment required a reaction chamber that could:

- Maintain sample temperature precisely at 18°C during UV photoirradiation to eliminate thermal decomposition artifacts.
- Remain completely chemically inert toward organophosphorus compounds, which are known to react with bare 316L stainless steel.
- Allow in-situ spectral acquisition without introducing contamination that could interfere with the measurement.

## The Challenge:

DMMP and related organophosphorus compounds are notoriously reactive with metallic surfaces. Uncoated 316 stainless steel catalyzes decomposition of these compounds, generating spurious reaction products that contaminate the spectra and obscure true surface chemistry on the target mineral sample.

- Catalytic Interference: Bare stainless steel catalyzes organophosphorous compound decomposition, generating artifacts and false spectral bands that obscure true catalyst chemistry.
- Material Cost: The conventional solution of using Hastelloy C-22 requires costly exotic alloys and specialized fabrication, significantly increasing hardware expense.
- Temperature Control: UV photoirradiation introduces heat that can cause thermal decomposition of the simulant which is indistinguishable from genuine photocatalytic chemistry unless sample temperature is tightly controlled.

## The Solution:

Specac partnered with SilcoTek to apply Silcolloy® 2000 to all wetted internal surfaces of the Praying Mantis High Temperature Reaction Chamber. The complete solution included:

- Silcolloy® 2000-coated Specac-Harrick high temperature reaction chamber via chemical vapor deposition (CVD).
- Integrated cooling cartridge (chiller set to 18°C) to counteract UV lamp heat and maintain isothermal conditions throughout irradiation.
- Praying Mantis diffuse reflection accessory for efficient collection of DRIFTS signal from the powder sample.

## The Results and Benefits:

By applying Silcolloy® 2000 to a standard 316 stainless-steel Specac-Harrick high temperature reaction chamber, Washington State University researchers obtained clean, artifact-free DRIFTS spectra of DMMP photocatalysis on P25 titania. The coating eliminated the need to specify exotic Hastelloy C-22 hardware while delivering inertness that exceeded bare C-22 by over three orders of magnitude in independent ICP-MS testing. The result: scientifically valid data, reduced hardware cost, and extended reliable instrument lifetimes.

Why Silcolloy® Instead of Hastelloy C-22?

SilcoTek ICP-MS testing demonstrated that Silcolloy-coated C-22 outperforms bare C-22 by over 1,000x in metal ion leaching, reducing contamination from >1,000 ppb to approximately 1.2 ppb after 30 days in solvent (Figure 3). Silcolloy-coated 316L SS performs comparably to Silcolloy-coated C-22, meaning researchers achieve superior inertness at standard stainless steel pricing. Rebuilding chambers in exotic Hastelloy alloy is no longer necessary to achieve long-lasting, accurate analysis.

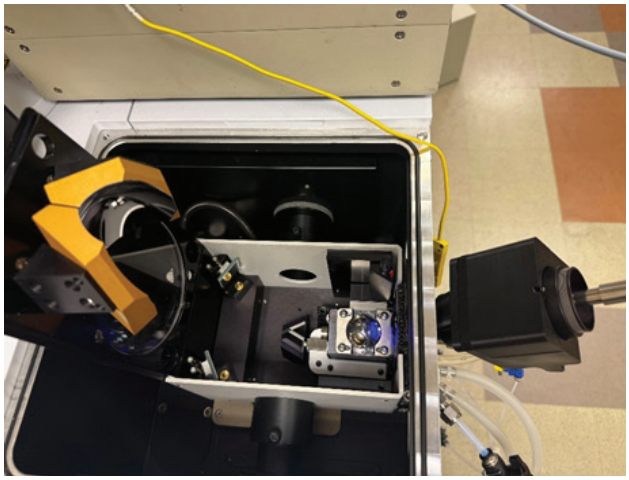

*Figure 1. Experimental setup. The Harrick Scientific reaction chamber can be seen in the center of the photo. It is placed inside the Praying Mantis accessory. Using a liquid light guide with a lens assembly (Newport), the top of the sample is illuminated by UV light, inducing photocatalytic reactions.*

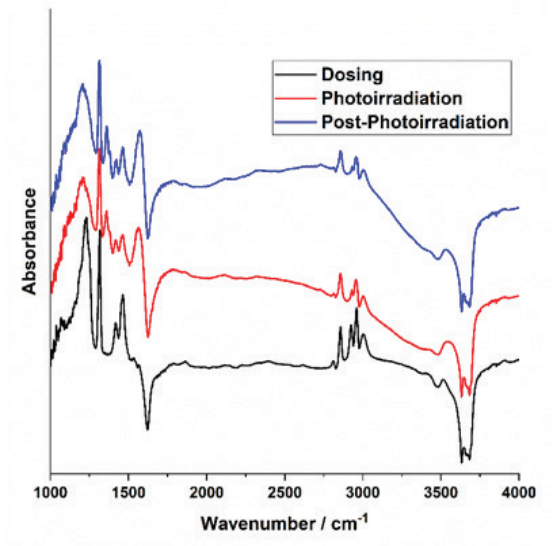


*Figure 2. DRIFTS spectra of P25 titania exposed to vapor-phase DMMP. During dosing, we observe the formation of the expected DMMP bands. The negative bands reflect the fact that DMMP is replacing surface hydroxyl groups. During photoirradiation, we observe the formation of formic acid bands near 1350 and 1575 cm-1, and of a surface-bound methoxy band near 2810 cm-1.*

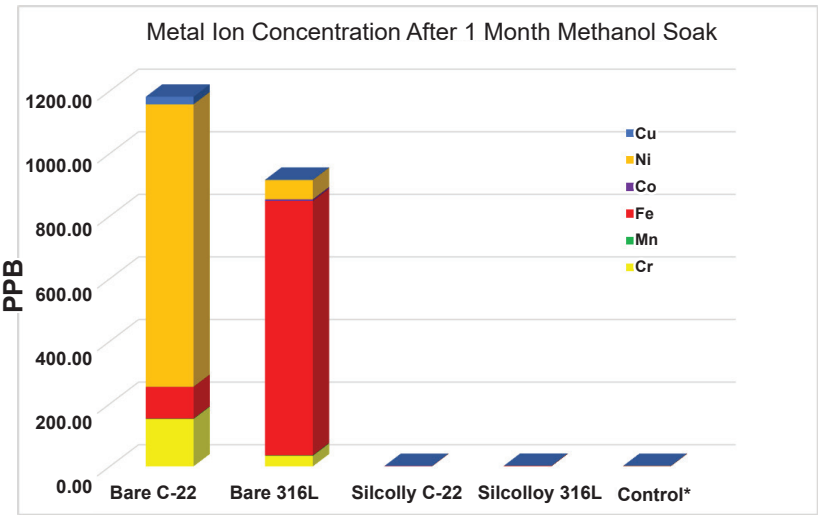


*Figure 3. This image shows the amount (PPB) of metal contamination found after soaking the samples in methanol for 30 days. The Silcolloy-coated C-22 outperformed the uncoated C-22 by 1000x. Method- Solvent Extraction ICP-MS. Technique- Parts were soaked in methanol for 30 days to allow for measurable metal extraction. Samples were then analyzed by mass spectroscopy to investigate metal ions present in solution.*

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