Direct Quantification of Etching Bath Components Using Extended-Range Raman Spectroscopy

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ABSTRACT

Across industries ranging from semiconductor and advanced packaging to battery, photovoltaic, chemical, and metal-finishing manufacturing, wet-process baths are central to surface preparation, etching, and cleaning. These systems often involve corrosive mixtures, such as NH_4OH/H_2O_2, HF, or H_3PO_4, whose composition directly governs etch rate, selectivity, and product quality. Accurate, real-time concentration control is therefore critical to maintain yield and extend bath lifetime. To address the variability associated with indirect near-infrared (NIR) measurements, this study evaluates Raman spectroscopy as a direct and more robust analytical alternative for wet-etch and cleaning chemistry. Detailed spectral evaluation and model development were performed to establish calibration accuracy, spectral selectivity, and matrix independence across temperature and viscosity variations. Raman achieved $R^2 > 0.99$ and Standard Error of Prediction (SEP) values as low as 0.03–0.09 wt%, matching or surpassing NIR performance. Validation using multi-level check samples and repeated measurements confirmed excellent repeatability and stability.

INTRODUCTION

Industry Importance:

Across semiconductor, advanced packaging, battery, photovoltaic, chemical, and metal-finishing industries, wet-process baths are fundamental to surface preparation, etching, cleaning, and chemical conditioning. These baths support a wide range of production steps, from oxide removal in wafer fabrication to metal texturing in large-scale industrial manufacturing, and their chemistry must be carefully maintained to ensure consistent material performance and product quality.

Typical operations that rely on tightly controlled etching and treatment baths include HF-based SiO_2 removal, metal etching of aluminum, copper, or steel, and pickling processes used to strip oxide layers and prepare metallic surfaces for downstream manufacturing. Beyond etching, these chemistries are also central to pre-plating activation, cleaning and degreasing steps, and electrochemical workflows such as electroplating, electrolytic refining, and other surface-conditioning sequences where bath uniformity directly affects coating adhesion, surface energy, and final component reliability. In coating, anodizing, and corrosion-protection lines, consistent bath composition is equally important, small deviations can impact adhesion, conversion-coating uniformity, or the long-term corrosion resistance demanded by high-performance applications.

Maintaining these chemistries requires analytical techniques that can keep pace with modern manufacturing throughput while offering high reliability, minimal maintenance, and real-time insight. Near-infrared (NIR) spectroscopy has become a widely adopted solution due to its speed, non-destructive nature, and ability to simultaneously measure multiple chemical and physical parameters. Its operational simplicity makes it well-suited for continuous bath surveillance, enabling more frequent measurement than laboratory-based wet-chemical methods and reducing the burden on production staff.

However, NIR is fundamentally an indirect analytical technique: it measures broad overtone and combination bands that correlate with concentration rather than responding to the fundamental vibrational modes of individual species. As a result, NIR models must interpret spectral changes that can arise from multiple sources, not only analyte concentration, but also temperature shifts, viscosity changes, additives, aging effects, or bath byproducts. These influences do not undermine NIR's value but do require thoughtful calibration design, appropriate preprocessing, and ongoing model lifecycle management to keep predictions robust across varying operating conditions. In multicomponent or highly reactive baths, this calibration dependency can introduce additional effort when striving for the most precise, composition-specific feedback. Because of these inherent characteristics, some wet-etch and cleaning chemistries benefit from complementary analytical tools that provide more direct molecular information, especially when spectral selectivity or component-specific stability is critical. Raman offers such advantages by measuring fundamental vibrational modes

with sharp, well-resolved peaks and minimal water interference, Raman can directly target individual bath constituents and reduce reliance on indirect correlation models. This study evaluates Raman spectroscopy as a direct, robust alternative for process bath analysis, while acknowledging the continued strengths and relevance of NIR in industrial monitoring.

Analytical Context:

The initial portion of the study evaluated the feasibility of using near-infrared (NIR) spectroscopy for monitoring the etch chemistries investigated, since this technology is a widely established industry standard. As expected for liquid-phase etching baths with low-concentration analytes and rapidly exchanging matrices, several practical and spectral considerations shaped NIR performance in this application. Accurate measurement required a sampling interface with a larger optical path length to enable proper sample exchange and avoid signal suppression at low analyte concentrations. While the increased gap improved flushing dynamics, it also reduced absorbance intensity, reinforcing that highly aqueous, low-level analytes push NIR measurements toward the lower end of their effective sensitivity range.

Figure 1 shows overlaid NIR spectra of gravimetrically prepared etching-bath samples collected between 1000 and 2200 nm. The shaded region (1000–1300 nm) highlights the part of the spectrum where concentration-dependent variation is observable. Within this zone, spectral differences arise primarily from indirect matrix effects such as temperature, viscosity, and pH changes. Above ~1400 nm, the spectra converge due to strong water-dominated overtone absorption, resulting in low analyte specificity and illustrating why only the $R\text{-}NH_2$ third overtone remains accessible without saturation.

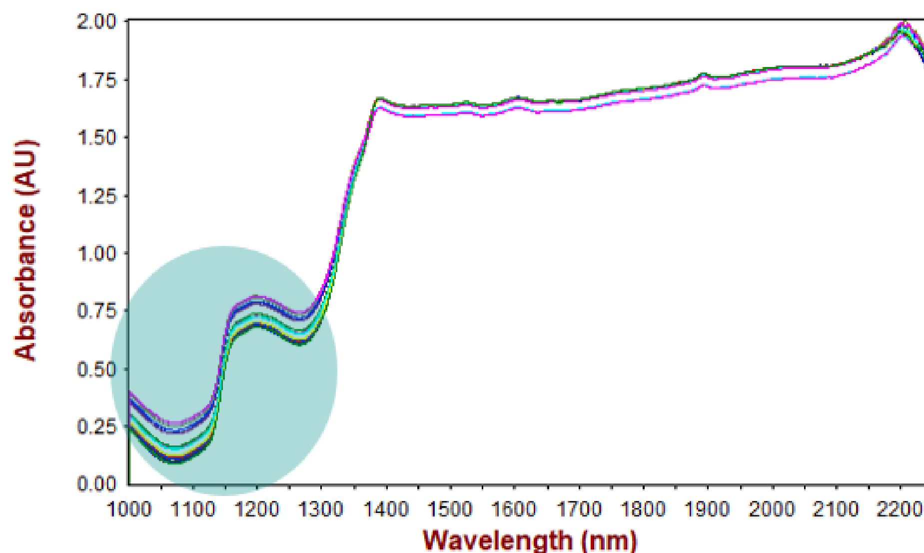


Figure 1*: Overlaid NIR spectra (1000–2200 nm) showing matrix-dependent baseline shifts. The circled region highlights the low-wavelength range where indirect concentration-related variation appears due to temperature, viscosity, and pH influences rather than distinct analyte-specific absorbance.*

Within the NIR spectral region, the R-NH_2 third overtone is the only feature that does not saturate under typical bath conditions, allowing ammonium-containing species to show measurable, analyte-correlated responses. In contrast, all other bath constituents—including H_2O_2 and H_3PO_4—fall within strongly absorbing regions where broad, overlapping water and matrix signals dominate. Consequently, their quantification depends entirely on indirect, correlation-based chemometric models rather than distinct analyte-specific absorption bands. This indirect-measurement behavior is well understood in NIR method development, but in highly reactive or dynamically changing chemistries it places added emphasis on model robustness and matrix stability.

For phosphoric acid in particular, spectral evaluation revealed consistent shifts in the NIR baseline and absorbance shape driven by temperature, viscosity, and pH changes within the bath. Because water-rich overtone behavior dominates this spectral region, these matrix-dependent variations can resemble true concentration changes, making indirect prediction less reliable unless the calibration set captures all relevant physical variation. In controlled environments—such as well-regulated flow cells, fixed temperatures, or processes with narrow operating windows, these effects can be successfully managed. However, in chemistries where temperature or viscosity drift naturally as the bath ages, or where replenishment chemistry alters pH and density, the indirect nature of the NIR response increases the effort needed to keep stable prediction performance.

EXPERIMENTAL

MAJOR SYSTEM COMPONENTS

- Metrohm 2060 Raman Process Analyzer, 785 nm excitation
- Metrohm 2060 NIR Process Analyzer

REAGENTS

- Ammonium hydroxide (NH_4OH)
- Hydrogen peroxide (H_2O_2)
- Phosphoric acid (H_3PO_4)

EXPERIMENTAL PROCEDURE

Sample Preparation

All samples were gravimetrically prepared, providing primary-standard-quality reference values. The standard deviation of this method is +/- 0.1wt%. No secondary analytical methods were used for calibration or validation. Concentration sets were designed to span industrially relevant ranges:

- H_2O_2: 0.0–3.0 wt%
- NH_4OH: 0.34–1.93 wt%
- H_3PO_4: 74.71–85.00 wt%

Raman Acquisition Parameters

- 5 mm focal point
- Measured at room temperature
- NH_4OH: 30 seconds
- H_2O_2: 11 seconds
- H_3PO_4: 1.5 seconds

NIR Data Collection Parameters:

- 5 mm gap size = 10 mm total pathlength
- 100 averages
- 10 sec integration time
- Measured at room temperature

Calibration & Validation

Models were developed using Partial Least Squares (PLS) regression. Preprocessing steps included:

- N-point smoothing (60 cm^{-1})
- Standard Normal Variate (SNV)
- First derivative filtering (10 cm^{-1}) where applicable.

Performance was evaluated with:

- Internal cross-validation
- Three independent check-sample levels per analyte
- Ten replicate measurements per level to assess precision, repeatability, and stability.

This approach allowed an objective assessment of both short-term measurement precision and the stability of the Raman-based prediction models.

RESULTS AND DISCUSSION

Quantification of hydrogen peroxide (H_2O_2) in ammonium hydroxide–hydrogen peroxide mixtures (APM):

Accurate quantification of hydrogen peroxide (H_2O_2) in ammonium hydroxide–hydrogen peroxide mixtures (APM) is essential for maintaining predictable etch behavior, particle removal efficiency, and bath lifetime in semiconductor and advanced cleaning processes. APM, commonly known as SC1, is a widely used alkaline cleaning solution composed of NH_4OH, H_2O_2, and water. Its performance depends directly on the balance between oxidative cleaning strength (driven by H_2O_2) and alkalinity (provided by NH_4OH). Because H_2O_2 decomposes over time and is sensitive to temperature, catalysis, and contamination, real-time monitoring of its concentration is critical to ensure consistent process outcomes, avoid under- or over-etching, and support reliable bath control strategies.

A total of 20 gravimetrically prepared samples (***Figure 2***), spanning 0.0–3.0 wt%, were used for calibration and internal cross-validation.

#	Theoretical Conc. (%wt.)	Actual H2O (g)	Actual APM (g)	Total (g)	Actual Conc. (%wt.)	
1	0	5	0	5	**0**	
2	0.5	4.1634	0.8273	4.9907	**0.50**	
3	0.6	4.0017	0.9979	4.9996	**0.60**	
4	0.7	3.8300	1.1683	4.9983	**0.70**	
5	0.8	3.6659	1.3335	4.9994	**0.80**	
6	0.9	3.4944	1.5253	5.0197	**0.91**	
7	1	3.3357	1.6659	5.0016	**1.00**	
8	1.1	3.1648	1.8559	5.0207	**1.11**	
9	1.2	3.0278	2.0399	5.0677	**1.21**	
10	1.3	2.8421	2.1798	5.0219	**1.30**	
11	1.4	2.6688	2.3378	5.0066	**1.40**	
12	1.5	2.5096	2.6026	5.1122	**1.53**	
13	1.8	2.0037	3.0211	5.0248	**1.80**	
14	1.9	1.8472	3.1654	5.0126	**1.89**	
15	2	1.6976	3.3713	5.0689	**2.00**	
16	2.2	1.3235	3.6917	5.0152	**2.21**	
17	2.4	1.0126	3.9955	5.0081	**2.39**	
18	2.6	0.6880	4.3538	5.0418	**2.59**	
19	2.8	0.3469	4.6914	5.0383	**2.79**	
20	3	0	5	5	**3.00**	
27	0.75	3.753	1.2541	5.0071	**0.75**	**Low**
28	1.65	2.2515	2.7666	5.0181	**1.65**	**Medium**
29	2.75	0.4149	4.5799	4.9948	**2.75**	**High**

Figure 2: *Gravimetrically prepared calibration and validation samples for the quantification of H_2O_2 in AMP covering low, medium, and high concentration levels. The table lists the target concentrations and corresponding actual masses used to produce precise 5 g samples for model development and validation.*

The corresponding Raman spectra (***Figure 3***) show a monotonic increase in peak height from 0 wt% to 3 wt%, confirming a strong analyte-specific response suitable for robust quantification. Validation of the model was further supported by three independent concentration-level check samples, each measured 10 times to assess standard deviation, average predicted concentration, and repeatability. These results confirmed the model's stability, reproducibility, and suitability for precise monitoring of H_2O_2 in APM across the investigated concentration range.

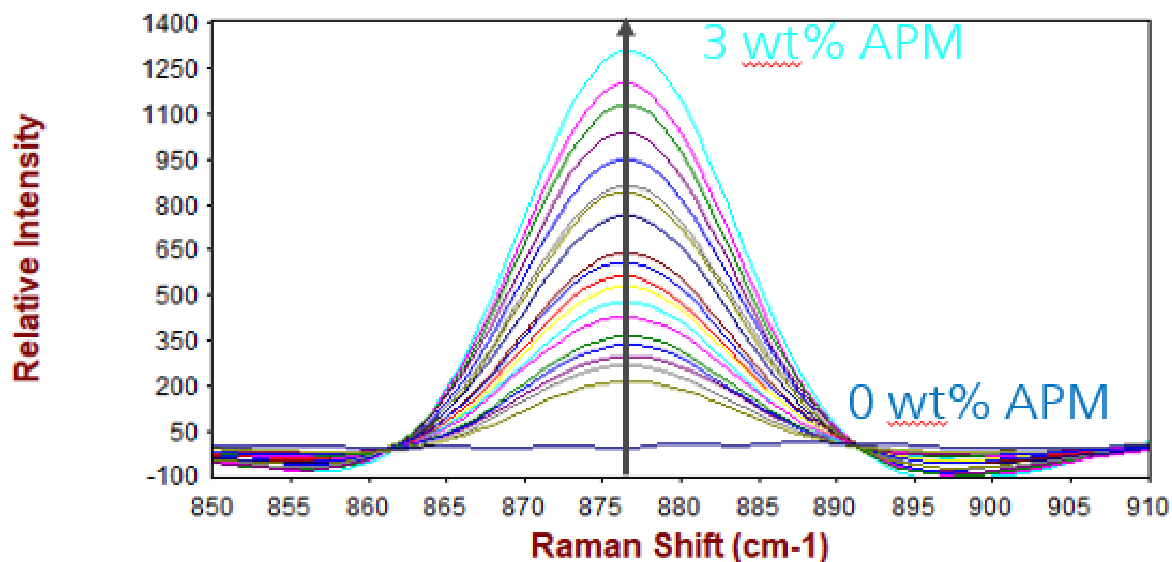
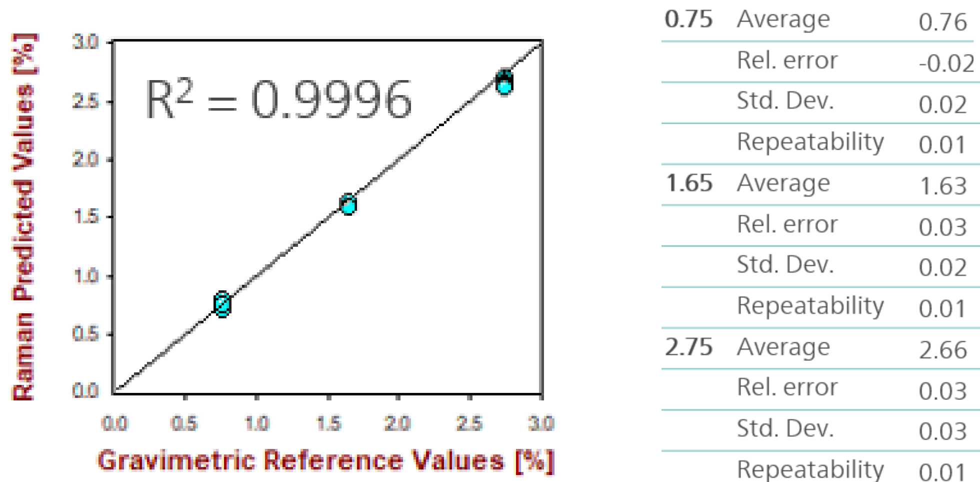


Figure 3: *Raman spectra of APM samples (0–3 wt%) showing the strong, concentration-dependent increase in the peroxide band near 875 cm⁻¹.*

The quantitative Raman model for H_2O_2 in APM was developed using a Partial Least Squares (PLS) regression approach optimized for the sharp peroxide band centered near 875 cm^{-1}. Modeling was performed over the 800–1000 cm^{-1} region, where spectral congestion is minimal and peroxide signal intensity shows a strong linear response to concentration. Spectral pretreatment consisted of N-point smoothing (60 cm^{-1} window), Standard Normal Variate (SNV) normalization, and a first derivative (10 cm^{-1}) to enhance peak definition and suppress baseline drift. Only one PLS factor was needed, reflecting the high signal specificity and clear concentration-dependent intensity response of the peroxide Raman band. Model performance metrics showed excellent predictive strength, with a Standard Error of Calibration (SEC) of ±0.04%, and a Standard Error of Cross-Validation (SECV) of ±0.04%.

Method performance was verified using three independent check-sample levels 0.75%, 1.65%, and 2.75% H_2O_2 in APM, each measured 10 times to assess prediction stability, precision, and overall model robustness. The resulting predicted concentrations, calculated gravimetric values, and residuals are summarized in ***Figure 4***.

Across all three levels, the Raman model showed excellent agreement with the gravimetric reference values. The average predicted concentrations for the check samples were 0.763%, 1.624%, and 2.662%, respectively, with corresponding standard deviations of 0.019%, 0.017%, and 0.027%. These values translate to repeatabilities of 0.040–0.055% and relative errors of 0.75%: ±0.02%, 1.65%: ±0.01%, and 2.75%: ±0.03%



0.75	Average	0.76
	Rel. error	-0.02
	Std. Dev.	0.02
	Repeatability	0.01
1.65	Average	1.63
	Rel. error	0.03
	Std. Dev.	0.02
	Repeatability	0.01
2.75	Average	2.66
	Rel. error	0.03
	Std. Dev.	0.03
	Repeatability	0.01

Figure 4: *Validation results for three check-sample levels (0.75%, 1.65%, 2.75%), each measured 10 times. Summary statistics reflect low standard deviation and excellent repeatability. The validation plot shows strong correlation (R^2 = 0.9996) between Raman-predicted and true concentrations.*

The validation plot further confirms the predictive strength of the method, showing a near-perfect correlation between Raman-predicted and gravimetric reference values (R^2 = 0.9996).

This strong linearity, combined with the low residuals and tight clustering of replicate measurements, shows that the Raman model provides reliable, precise, and highly repeatable quantification of H_2O_2 in APM across the full investigated concentration range.

Quantification of ammonium hydroxide (NH_4OH) in ammonium hydroxide–hydrogen peroxide mixtures (APM):

Accurate quantification of ammonium hydroxide (NH_4OH) in ammonium hydroxide–hydrogen peroxide mixtures (APM/SC1) is essential for keeping the cleaning and surface-conditioning performance needed in semiconductor and advanced manufacturing processes. NH_4OH governs the alkalinity, particle-lifting capability, and metal-ion complexation strength of the bath, directly influencing surface roughness, organic residue removal, and compatibility with sensitive materials and multilayer stacks. Even small deviations in NH_4OH concentration can shift the balance between effective cleaning and undesirable surface modification, impact bath lifetime, or alter the etching selectivity between materials. Because NH_4OH can volatilize, react, or dilute through bath aging and replenishment cycles, reliable quantification is critical for ensuring stable process behavior and keeping the high-purity conditions demanded in wet-chemistry workflows.

A total of 13 gravimetrically prepared samples (***Figure 5***), spanning 0.34–1.93 wt%, were used for calibration and internal cross-validation.

	NH4OH	
Actual Conc. (%wt)	**#**	**Actual Conc. (%wt)**
0.50	1	0.34
0.69	2	0.46
0.91	3	0.61
1.10	4	0.73
1.30	5	0.87
1.50	6	1.00
1.90	7	1.27
2.20	8	1.47
2.59	9	1.73
3.01	10	2.00
0.61	11	0.41
1.29	12	0.86
2.90	13	1.93

Figure 5*: Gravimetrically prepared calibration and validation samples for the quantification of NH_4OH in AMP covering low, medium, and high concentration levels. The table lists the target concentrations and corresponding actual concentrations.*

The Raman spectral overlay (***Figure 6***) shows a clear, concentration-dependent increase in peak intensity within the NH_4OH analytical window, confirming a strong linear response suitable for quantification.

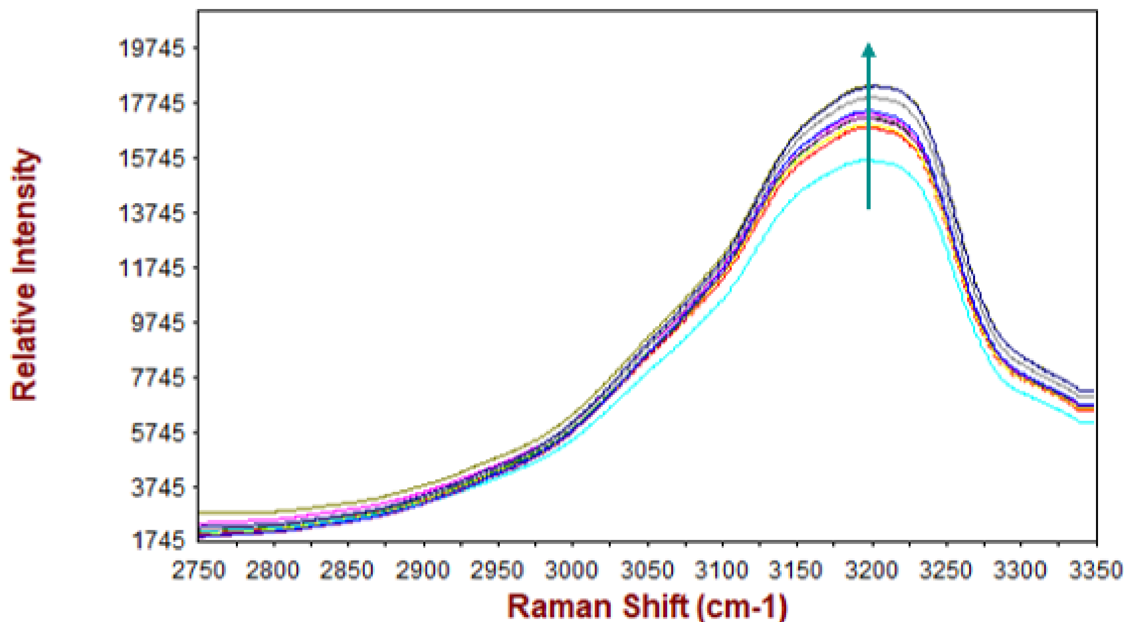


Figure 6*: Raman spectral overlay for NH_4OH in APM collected using a 30-second integration time. Detector saturation occurs outside the NH_4OH analytical region (2150–3320 cm^{-1}) and does not affect the spectral features used for quantification.*

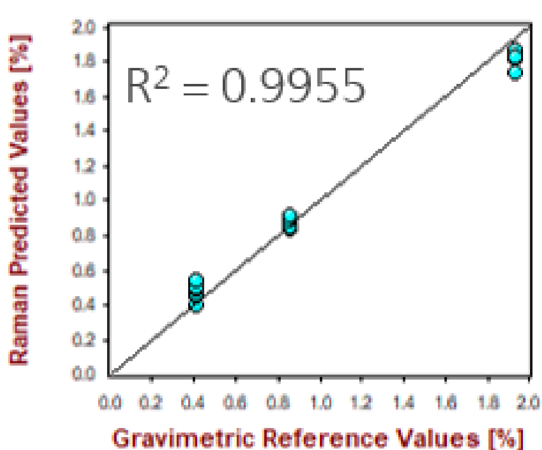
The NH_4OH calibration set was modeled using a Partial Least Squares (PLS) approach applied to the 2150–3320 cm^{-1} region, where NH_4OH shows a well-defined overtone structure. Spectral pretreatment consisted of N-point smoothing (60 cm^{-1}) and Standard Normal Variate (SNV) normalization, yielding a robust model requiring four PLS factors. The resulting figures of merit—SEC = ±0.05%, SECV = ±0.05%, and SEP = ±0.08%, show strong calibration performance across the full concentration range.

Validation of the model was further supported by three independent concentration-level check samples, each measured 10 times to assess standard deviation, average predicted concentration, and repeatability. These results confirmed the model's stability, reproducibility, and suitability for precise monitoring of NH_4OH in APM across the investigated concentration range.

Method performance for NH_4OH quantification in APM was evaluated using three independent check-sample levels 0.41%, 0.86%, and 1.93%, each measured 10 times to assess accuracy, precision, and repeatability. The full set of predicted values, gravimetric reference values, and residuals is shown in ***Figure 7***. confirming the

robustness of the method across the tested range. The validation plot further shows the strong linear relationship between Raman-predicted and gravimetric concentrations, with an R^2 value of 0.9955, showing excellent predictive accuracy and confirming the suitability of the Raman model for precise NH_4OH quantification in APM.

Across all validation levels, the Raman model showed excellent agreement with the gravimetric targets, yielding average predicted concentrations of 0.49%, 0.89%, and 1.83%, respectively. The corresponding standard deviations remained low (0.06%, 0.02%, and 0.03%), resulting in strong repeatabilities between 0.08% and 0.04%. The calculated relative errors, −0.19%, +0.02%, and −0.10%, produced an average relative error of only 0.06%,



0.41	Average	0.49
	Rel. error	-0.19
	Std. Dev.	0.04
	Repeatability	0.01
0.86	Average	0.87
	Rel. error	-0.02
	Std. Dev.	0.03
	Repeatability	0.01
1.93	Average	1.83
	Rel. error	0.05
	Std. Dev.	0.04
	Repeatability	0.01

Figure 7: *Validation results for NH_4OH using three check-sample levels (0.41%, 0.86%, 1.93%), each measured 10 times. Predicted versus gravimetric reference values show excellent agreement with low residuals, yielding an average relative error of 0.06%. The validation plot shows strong linearity ($R^2 = 0.9955$), confirming high accuracy and repeatability of the Raman quantification method.*

Quantification of phosphoric acid (H_3PO_4) in etching bathes:

Accurate quantification of phosphoric acid (H_3PO_4) is essential for controlling etching behavior, bath lifetime, and process stability in a wide range of semiconductor and advanced manufacturing workflows. H_3PO_4-based etchants are commonly used for oxide removal, surface texturing, and post-deposition cleaning steps, where the acid concentration directly influences etch rate, uniformity, and selectivity toward different material layers. Even small concentration deviations can lead to under- or over-etching, shift temperature-dependent viscosity profiles, or compromise adhesion and downstream process quality. Because H_3PO_4 etching solutions show strong temperature, viscosity, and pH dependencies, routine and reliable monitoring of acid concentration is necessary to ensure predictable bath performance and avoid unintentional process drift as the bath ages or is replenished.

A total of 25 gravimetrically prepared samples (***Figure 8***), spanning 74.71–85.00 wt%, were used for calibration and internal cross-validation.

#	Theoretical Conc. (%wt.)	Actual H2O (g)	Actual H3PO4 (g)	Total (g)	Actual Conc. (%wt.)	
1	0	5	0	5	**0**	
2	75	0.6105	4.4321	5.0426	**74.71**	
3	75.1	0.5867	4.4235	5.0102	**75.05**	
4	75.2	0.5811	4.4476	5.0287	**75.18**	
5	75.3	0.5713	4.4324	5.0037	**75.30**	
6	75.4	0.5742	4.4527	5.0269	**75.29**	
7	75.5	0.5679	4.4617	5.0296	**75.40**	
8	75.6	0.5423	4.4683	5.0106	**75.80**	
9	75.7	0.5459	4.4802	5.0261	**75.77**	
10	75.8	0.5474	4.482	5.0294	**75.75**	
11	75.9	0.5368	4.4917	5.0285	**75.93**	
12	76	0.5277	4.5114	5.0391	**76.10**	
13	76.5	0.4945	4.7524	5.2469	**76.99**	
14	77	0.4782	4.5382	5.0164	**76.90**	
15	77.5	0.4505	4.57	5.0205	**77.37**	
16	78	0.4282	4.6107	5.0389	**77.78**	
17	78.5	0.3857	4.6268	5.0125	**78.46**	
18	79	0.3726	4.6825	5.0551	**78.73**	
19	79.5	0.3393	4.7024	5.0417	**79.28**	
20	80	0.2999	4.7058	5.0057	**79.91**	
21	80.5	0.2744	4.7483	5.0227	**80.36**	
22	81	0.234	4.7858	5.0198	**81.04**	
23	82	0.1733	4.8837	5.057	**82.09**	
24	83	0.116	4.9324	5.0484	**83.05**	
25	84	0.0581	4.9544	5.0125	**84.01**	
26	85	0	5	5	**85.00**	
27	75.25	0.5711	4.4257	4.9968	**75.29**	**Low**
28	81.5	0.2122	4.8052	5.0174	**81.41**	**Medium**
29	84.5	0.0288	4.9919	5.0207	**84.51**	**High**

Figure 8: *Gravimetrically prepared calibration and validation samples for the quantification of H_3PO_4 covering low, medium, and high concentration levels. The table lists the target concentrations and corresponding actual concentrations.*

The H_3PO_4 calibration set, prepared gravimetrically across the full concentration range of interest, was modeled using a Raman-based approach focused on the strong phosphate vibrational band located above **900 cm⁻¹**, where H_3PO_4 exhibits a dominant and highly selective response (***Figure 9***). Spectra were preprocessed using N-point smoothing and Standard Normal Variate (SNV) normalization to enhance peak consistency and reduce baseline variability before applying Partial Least Squares (PLS) regression.

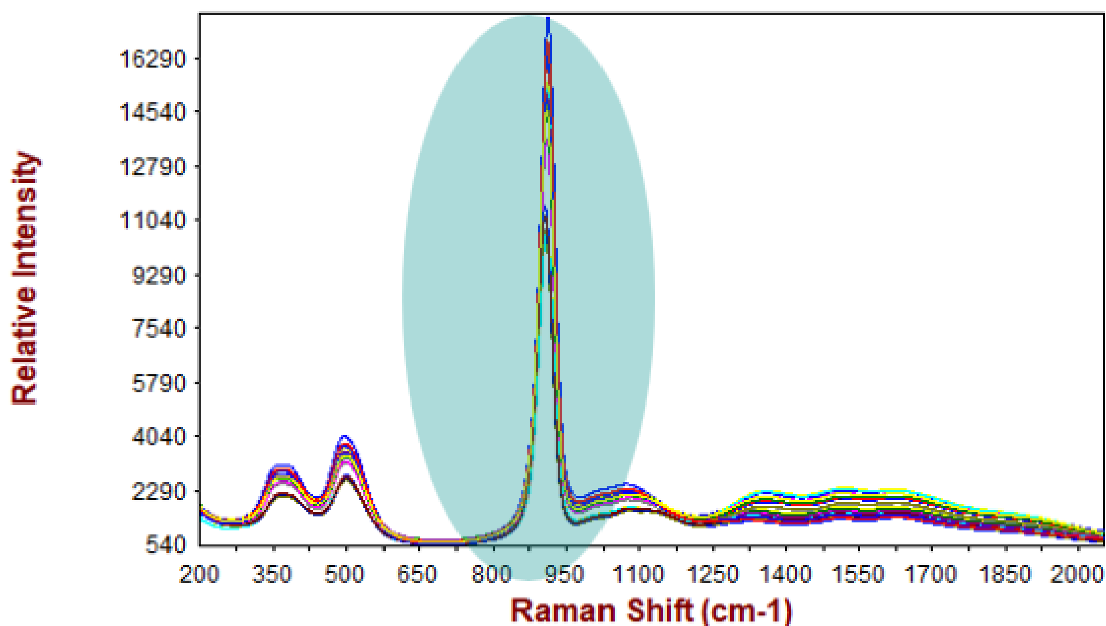
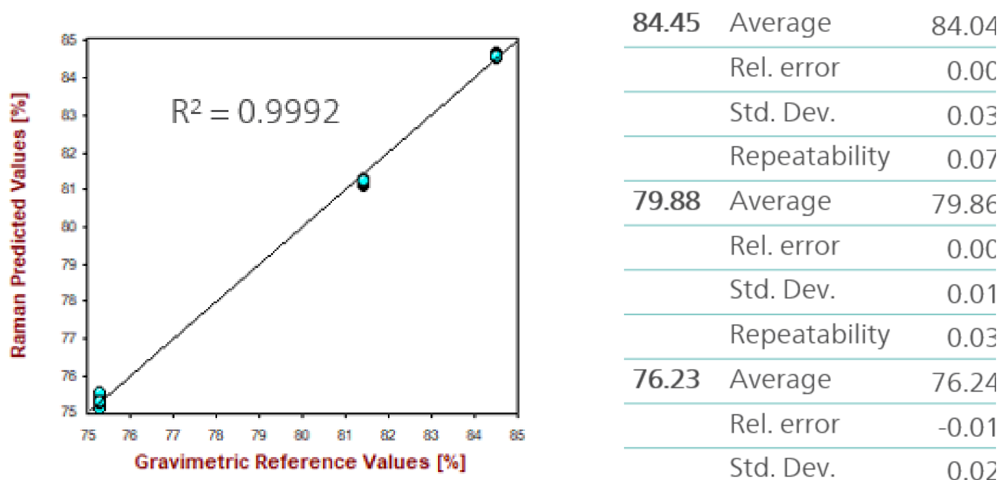


Figure 9: *Raman spectral overlay of H_3PO_4 showing the strong phosphate vibrational band above 900 cm $^{-1}$. The dominant, concentration-dependent peak provides a highly selective analytical target for direct quantification in etching bath solutions.*

The PLS model was developed over the 800–1000 cm^{-1} range using N-point smoothing (60 cm^{-1}), Standard Normal Variate (SNV) normalization, and a first derivative (10 cm^{-1}) to enhance peak resolution. Three PLS factors were needed to capture the spectral variability across the 75–85 wt% calibration range, resulting in figures of merit of SEC = ±0.16%, SECV = ±0.19%, and SEP = ±0.21%. Together, the calibration dataset and spectral trends confirm that Raman spectroscopy delivers a robust, selective, and sensitive approach for quantifying H_3PO_4 in etching bath formulations.

The Raman quantification method for H_3PO_4 was validated using three check-sample levels 75.29%, 81.41%, and 84.51%, each measured 10 times to assess prediction accuracy and measurement repeatability. The full set of predicted values, gravimetric reference values, and residuals is summarized in ***Figure 10***. Across all levels, the model demonstrated strong agreement with the gravimetric targets, with average predicted concentrations of 75.29%, 80.36%, and 83.39%, respectively. Corresponding standard

deviations remained low (0.13%, 0.02%, and 0.08%), resulting in excellent repeatability, as shown in the summary table. The resulting relative errors, ±0.01% for all three levels, produced an overall average relative error of 0.01%, confirming the stability and precision of the method. The validation plot further illustrates the strong correlation between Raman-predicted and gravimetric concentrations, with an R^2 value of 0.9992, demonstrating the robustness and reliability of the Raman model for high-concentration H_3PO_4 quantification.



84.45	Average	84.04
	Rel. error	0.00
	Std. Dev.	0.03
	Repeatability	0.07
79.88	Average	79.86
	Rel. error	0.00
	Std. Dev.	0.01
	Repeatability	0.03
76.23	Average	76.24
	Rel. error	-0.01
	Std. Dev.	0.02

Figure 10: *Validation results for H_3PO_4 using three check-sample levels (75.29%, 81.41%, 84.51%), each measured 10 times. Predicted values, gravimetric references, and residuals demonstrate excellent precision, low standard deviations, and an overall relative error of ±0.01%. The validation plot (R^2 = 0.9992) confirms strong linearity and high accuracy of the Raman quantification method.*

CONCLUSION

This work demonstrates that Raman spectroscopy is a powerful, direct, and highly selective analytical method for real-time monitoring of wet process etching and cleaning baths. Unlike NIR spectroscopy, whose indirect measurements are strongly influenced by temperature, viscosity, pH, and bulk matrix effects, Raman spectroscopy provides molecule-specific responses with minimal water interference and strong resistance to matrix-driven variability.

Across all analytes studied (H_2O_2, NH_4OH, and H_3PO_4), Raman-based calibration models exhibited:

- Outstanding linearity
- Low calibration, validation, and prediction errors
- Excellent repeatability and stability across multi-level validation

These results confirm the intrinsic robustness and precision of Raman spectroscopy for quantitative monitoring of critical bath components, supporting tighter process control, improved bath consistency, and enhanced manufacturing quality in semiconductor, metal finishing, and other advanced processing environments.

It is important to note that the results presented here were generated under controlled laboratory conditions and have not yet been deployed in an active production wet bath. In real-world wet process environments, bath chemistries typically contain additional additives, stabilizers, and reaction byproducts beyond the primary analytes studied. As with any multivariate spectroscopic technique, accurate Raman model performance under these conditions depends on representative calibration samples that capture the full range of expected matrix variability.
The molecule-specific nature of Raman spectroscopy provides a strong foundation for handling increased bath complexity; however, successful field deployment will require model development using samples that include relevant additives, process-induced byproducts, and concentration ranges representative of actual operating conditions.

Overall, the findings of this study establish Raman spectroscopy as a technically sound and promising analytical platform for next-generation wet process monitoring, with clear advantages in selectivity and resilience to matrix effects, and a well-defined path toward full-scale industrial implementation.