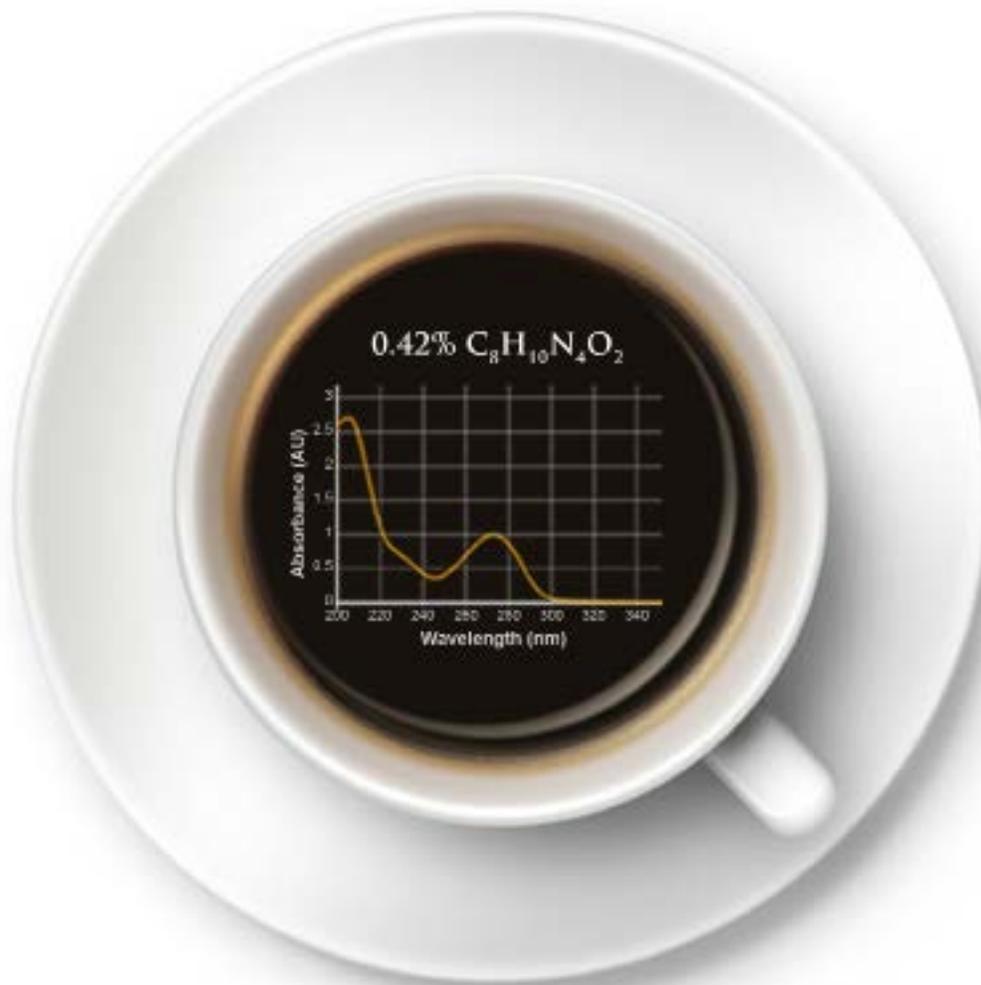


Measuring Caffeine Concentration

Applied Analytics Application Note No. AN-019



Application Summary

Analytes: **caffeine ($C_8H_{10}N_4O_2$)**

Detector: **OMA-300 Process Analyzer (UV-Vis spectrophotometer)**

Introduction

While it may mystify those of us that need strong coffee to get through the workday, some people actually drink decaf (decaffeinated coffee), whether for health reasons (high blood pressure, hypertension, sleep difficulties), pregnancy cautions, or personal preference.

In lieu of more firm legal standards, decaf is supposed to have its caffeine content reduced by no less than 97.5% of the source coffee (USDA guideline). Accurate validation of the caffeine levels in decaffeinated coffee bean batches would reduce

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sensitive consumers' health risks by preventing the wildly inconsistent caffeine levels among available "decaf" blends (as documented by the Journal of Analytical Toxicology). Moreover, producers can use real-time caffeine monitoring to conclude the decaffeination cycle as soon as the specified caffeine threshold is achieved, thus wasting no more caffeine solvent or production time than absolutely necessary.

An online analysis solution would provide continuous, live caffeine measurement in an automated fashion. The High-Performance Liquid Chromatography (HPLC) methods currently used in some caffeine applications is quite costly to own and maintain while delivering slow response.

Using OMA to Measure Caffeine Concentration in Decaffeination Processing

While certain selective solvents are extremely effective at removing caffeine from coffee beans, many of these chemicals have been classified as carcinogenic or toxic. Though less powerful, the supercritical CO₂ extraction method is a harmless decaffeination process common in large-scale operations.

Supercritical CO₂ fluid is a hybrid gaseous/liquid resource maintained by exceeding the critical temperature (31 °C) and pressure (73 atm) of CO₂; for the supercritical CO₂ to act as an effective solvent for caffeine molecules, it actually has to be kept at more extreme conditions (around 94 °C and 225 atm). These conditions are expensive to maintain, and the 10-hour process cycle is time-consuming. The caffeine extract is sold off to pharmaceutical and soft drink companies, but this only partially alleviates process cost.

With real-time caffeine monitoring at ±1 ppm accuracy, the OMA-300 Process Analyzer allows decaf coffee producers to automatically end an extraction cycle when the specified caffeine maximum is reached. Doing away with the preset 10-hour cycle, this method banishes both overprocessing (which wastes production resources) and underprocessing (which upsets customers).

While highly suitable for monitoring the 0-1,000 ppm caffeine range established by international standards as "legally" decaf, the OMA-300's UV-Vis spectrophotometer has a true detection limit of 1 ppm caffeine, thus theoretically capable of validating a cup of perfect decaf.

Importance of Using a UV-Vis Spectroscopic Method

The defining advantage of the ultraviolet-visible (UV-Vis) range in caffeine analysis is the transparency of the process background to the detector. Systems using infrared (IR) spectroscopy will suffer interference from water or CO₂ because these compounds absorb IR radiation; these systems may require drying the sample or expensive sample handling.

Since water and CO₂ have no absorbance in the UV-Vis range, they appear totally transparent to the OMA spectrophotometer. This is extremely important for typical caffeine applications such as:

- » caffeine in supercritical CO₂ stream
- » caffeine in water or liquids containing water

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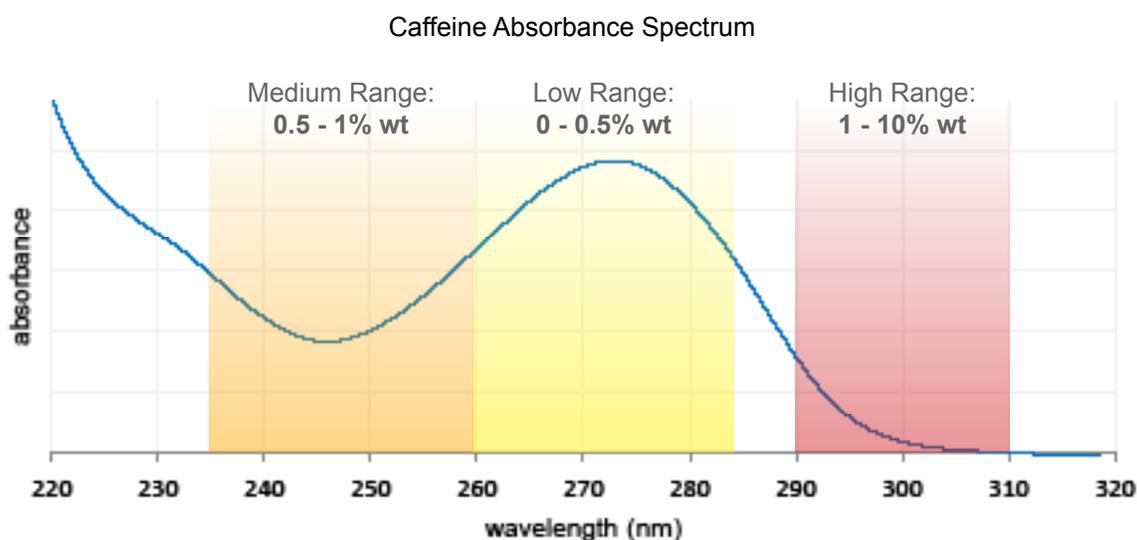
Dynamic Range in Caffeine Measurement

Any single photodiode measurement is vulnerable to noise, signal saturation, or unexpected interference. This susceptibility to error makes a lone photodiode data point an unreliable indicator of one chemical's absorbance.

As accepted in the lab community for decades, the best way to neutralize this type of error is to use collateral data in the form of 'confirmation wavelengths,' i.e. many data points at many wavelengths instead of a single wavelength.

The same principle holds true for sustaining accuracy across a very wide concentration range. An instrument that only measures one wavelength at a pre-determined absorbance peak will suffer from signal clipping: above a certain concentration, all of the signal at that wavelength will be absorbed and higher concentrations cannot be measured. The full-spectrum technology used by the OMA removes this limitation by measuring at many wavelengths simultaneously.

To illustrate this capability, the spectrum below shows the shape of the UV-Vis absorbance curve intrinsic to the caffeine molecule. Each range of caffeine concentration has a most suitable wavelength region of analysis based on the relative height of the absorbance curve within that region:



When processing the raw data from the spectrophotometer, the OMA's proprietary ECLIPSE software runs three analysis models in parallel—each assigned to a unique wavelength region—to measure caffeine concentration at different ranges. These parallel models ensure that the measurement always compensates for an error at a single photodiode due to over-absorbance or light saturation.

Consider the example of the Low Range: when caffeine concentration rises above 0.5% wt, the sample will absorb strongly enough that the diodes measuring at the 260-284 nm region will register zero light. At >0.5% wt concentration level, these diodes cease to be reliable due to signal clipping. Meanwhile, the absorbance curve is naturally lower in the 235-260 nm region (Medium Range), and the diodes reading these wavelengths are still receiving an optimal absorbance signal.

The OMA system always runs all three models simultaneously, reporting each as a separate output. Optional system logic can create a unified output which always reports from the appropriate model (Low, Medium, or High) based on the current caffeine concentration in the sample.

In this manner, the OMA provides seamless dynamic range with sustained accuracy through large fluctuations.

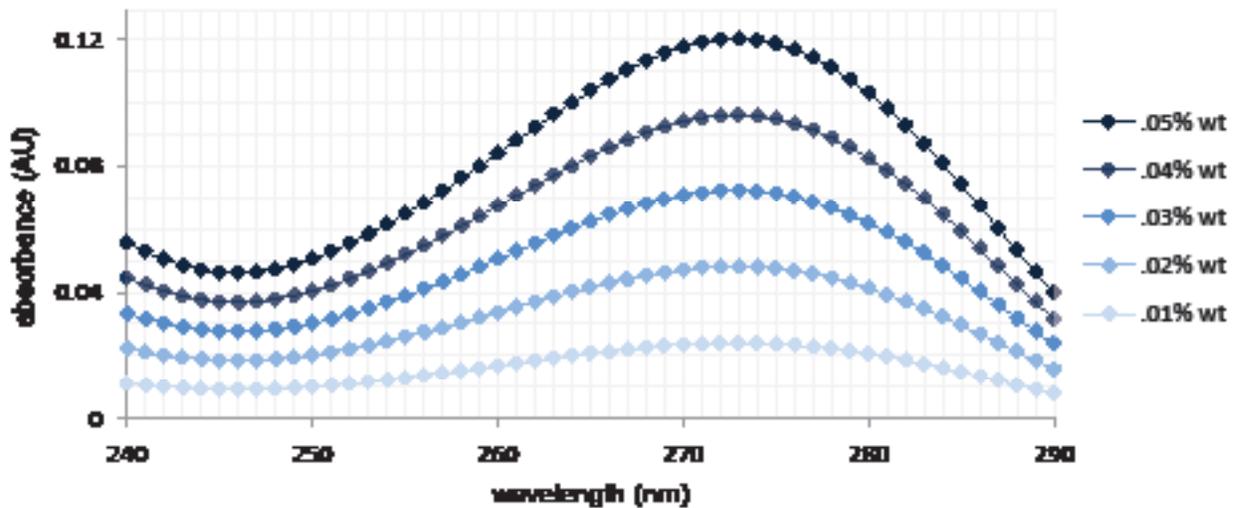
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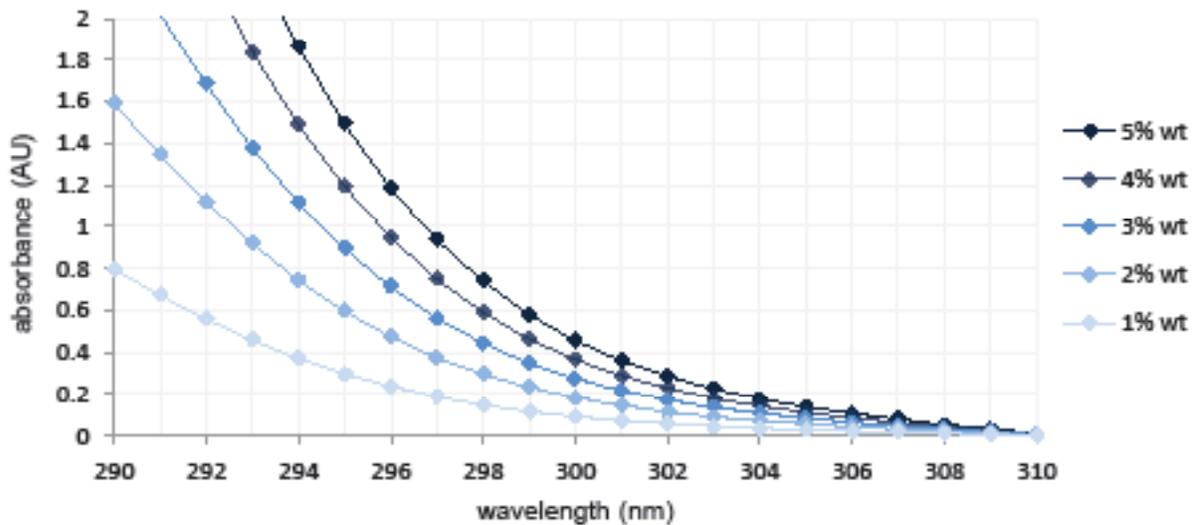
Absorbance-to-Concentration Correlation

In the figures below, each diamond in a curve represents a single photodiode and data point. The OMA measures absorbance at each integer wavelength from 200-800 nm and produces a caffeine absorbance curve. After being calibrated on a full spectrum of pure caffeine at known concentration, the OMA knows the absorbance-concentration correlation for each wavelength. This is the equivalent of using hundreds of single-wavelength photometers simultaneously, averaging the concentration calculated from each data point to eradicate the effect of noise at any single photodiode.

Caffeine Absorbance Curves at **Low Range** (0.01% wt to 0.05% wt)



Caffeine Absorbance Curves at **High Range** (1% wt to 5% wt)



Only a full-spectrum instrument can provide the rich raw data necessary to accurately measure caffeine concentration from very low to very high levels.

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The specifications below represent performance of the OMA-300 Process Analyzer in a typical caffeine application.

For technical details about the OMA-300 Process Analyzer, see the data sheet:

http://aai.solutions/documents/AA_DS001A_OMA300.pdf

All performance specifications are subject to the assumption that the sample conditioning system and unit installation are approved by Applied Analytics. For any other arrangement, please inquire directly with Sales.

Subject to modifications. Specified product characteristics and technical data do not serve as guarantee declarations.

Application Data	
Performance Specifications	
Accuracy	<i>Custom measurement ranges available; example ranges below.</i>
caffeine	0-100 ppm: ± 1 ppm 0-1,000 ppm: $\pm 1\%$ full scale 0-10%: $\pm 1\%$ full scale

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Revised 12 April 2017

Further Reading

Subject	Location
OMA Series Process Analyzers Brochure	http://aai.solutions/documents/OMA.pdf
OMA-300 Process Analyzer Data sheet	http://aai.solutions/documents/AA_DS001A_OMA300.pdf
Advantage of Collateral Data Technical Note	http://aai.solutions/documents/AA_TN-202_CollateralData.pdf

References

McCusker, Rachel R., Brian Fuehrlein, Bruce A. Goldberger, Mark S. Gold, and Edward J. Cone. "Caffeine Content of Decaffeinated Coffee." *Journal of Analytical Toxicology* 30.8 (2006): 611-13.



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