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## Determination of Sodium and Potassium with Flame Atomic Absorption Spectrometry

### Scope and Application

Atomic Absorption can be used to determine the concentration of sodium and potassium in alcoholic beverages including wines, malt beverages, and distilled spirits. This method may also be used to determine the sodium concentration in nonbeverage products such as salted wines and soy sauces.

Sodium and potassium are important components of wines and are routinely determined to check the integrity and quality of a wine. A standard table wine may contain 5-50 ppm sodium and 400-1200 ppm potassium. A standard sake may contain 10-29 ppm sodium and 42-185 ppm potassium. A standard malt beverage may contain 14-36 ppm sodium and 43-126 ppm potassium.

### Regulatory Tolerances

If the concentrations of sodium and potassium are lower than the ranges mentioned above, the wine may fall into the category of a sub-standard or other than standard wine. Under U.S.C. 5010 (27 CFR, Part 19.21) a sub-standard wine becomes eligible for a tax credit when it is used in a distilled spirit product.

Salted wines, 27 CFR Part 24.215 (b), contain a minimum of 1.5% sodium chloride or 5900 ppm sodium.

### Levels and Limitations

1. Wines showing sodium and potassium above 400 mg/L and 800 mg/L, respectively, need to be diluted and retested. Dilution may be accomplished manually or using the auto-diluter system. Consult one of the co-principal analysts at the lab location for further instruction.
2. Salted wines and soy sauces should be appropriately diluted within the linear range for sodium.

Analyte	Quantitation Limit (ppm)	Linear Range (ppm)	Interferences
Sodium (beverage)	0.5	0.5-100	
Sodium (nonbeverage)	35	35-300	
Potassium	20	20-400	

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## Supplemental Documents

**SSD:WG:202** Flow Diagram for QC Failures.

### NLC

1. **NLC:WG:392** Perkin Elmer AA Maintenance Schedule
2. **NLC:WG:595** Interpreting Data from Perkin Elmer AA
3. **NLC:WG:510-1** Instructions for Operation of the Perkin Elmer Pinnacle 900F Flame AA using the Syngistix software

### BAL

1. **BAL:WG:596** Sodium and Potassium Standard and Sample Preparation

### CL

1. **CL:WG:016** Working Guideline for AA - Walnut Creek, CA Lab

### NPL

1. **NPL:WG:596** Sodium Standard and Sample Preparation for NPL

## Equipment

Equivalent instrumentation, glassware, and supplies may be used.

### Instrumentation:

**NLC uses** Perkin-Elmer Atomic Absorption Spectrometer (model: **Pinnacle 900F**), or equivalent, equipped with sodium and potassium hollow cathode lamps, Elemental Scientific M-PFF-AA prepFAST Flame Auto-sampler and Auto-diluter (Model: SC4 DX), or equivalent

**CL uses** Agilent (formerly Varian) Atomic Absorption Spectrometer (model: **AA240FS**), or equivalent, equipped with Na/K multi-element hollow cathode lamp, SP3 Sample Preparation System and SIPS Sample Introduction System, or equivalent

Computer, monitor, and a printer.

### Glassware and Supplies:

**NLC uses** mechanical pipettes, **Polymethylpentene (PMP)** volumetric flasks, **Polypropylene (PP)** centrifuge tubes for samples and standards, and other assorted laboratory plasticware\*.

**CL uses** class A volumetric pipettes, electronic and mechanical pipettes, volumetric flasks, test tubes for samples and standards, and other assorted laboratory glassware/plasticware\*.

\*NOTE: Examples of plasticware used include: **High Density Polyethylene (HDPE)** and **Low Density Polyethylene (LDPE)**, and **polyethylene terephthalate (PET)**.

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## Reagents, Sample Preparation and Handling

**Reagents and Standards:** (Specific vendors and product numbers are listed for convenience. Equivalent products may be used).

Nitric Acid (FW 63) — Certified ACS Grade or better (Fisher Scientific Product # A200)

NLC uses 1000 ppm Stock Sodium Standard Solution: Certified Concentration, AA Grade or better

Primary Source (CRM): SPEX CertiPrep Product # PLNA2-2T (ISO Guide 34 certified)

Secondary Source (RM): Inorganic Ventures Product # AANA1

CL uses 10000 ppm Stock Sodium Standard Solution: Certified Concentration, AA Grade or better

Primary Source (CRM): SPEX CertiPrep Product # PLNA2-3Y (ISO Guide 34 certified)

Secondary Source (RM): Agilent (formerly Ultra Scientific) Product # ICP-111 (ISO Guide 34 certified)

NLC uses 10000 ppm Primary Stock Potassium Standard Solution and 1000 ppm Secondary Stock Potassium Standard: Certified Concentration, AA Grade or better

Primary Source (CRM): SPEX CertiPrep Product # PLK2-3Y (ISO Guide 34 certified)

Secondary Source (RM): Inorganic Ventures Product # AAK1

CL uses 10000 ppm Stock Potassium Standard Solution: Certified Concentration, AA Grade or better

Primary Source (CRM): SPEX CertiPrep Product # PLK2-3Y (ISO Guide 34 certified)

Secondary Source (RM): Agilent (formerly Ultra Scientific) Product # ICP-119 (ISO Guide 34 certified)

NLC uses 1000 ppm Copper Stock Standard Solution (for burner head optimization, see NLC:WG:392)

Primary Source (CRM): Inorganic Ventures Product # AACU1

(BAL and CL) LCS: Commercially purchased bottles of varietal wine (typically white wine), with all bottles from the same lot or vintage.

(NPL) LCS: Mixture of cooking wine samples (may or may not be spiked with excess salt) with salt concentration determined by consensus values.

Deionized (DI) water—18 mega ohm or better

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## Reagent and Standards Preparation:

**Note: Each standard must have 3% nitric acid by volume to keep the metals in solution.**

1. The **Blank** is 3% Nitric Acid prepared with DI water.
2. **For Sodium and Potassium standards preparation refer to appropriate guidelines for more information.**

## Sample Preparation:

Samples can be diluted with 3% Nitric Acid to reduce the solid matrix. Refer to working guidelines for more information. The solid content of the matrix entering the burner should not exceed 0.5% (w/v). Carbonated samples are degassed before analysis.

## Procedure

1. Follow the appropriate working guidelines for set up, cleanup and analysis.
2. Make sure that there is enough volume of sample in each sample tube so that the tip of the sampler probe can reach into the sample. A stable baseline indicates the stability of the flame and the sensitivity of the instrument.

## Quality Control

1. Run the prepared **calibration** standards and the blank, which will represent the zero calibration point. Determine the linearity of the curve (correlation coefficient). Analysis should only proceed if the correlation coefficient is greater than 0.99.
2. Run LCS **in duplicate** for accuracy and precision. Record values on control charts. If values are outside of specified range, follow **the same troubleshooting on the next step and follow** the protocol in SSD:WG:202 Flow Diagram for QC Failures **as well**.
3. **Run the second or secondary source check standard (prepared from a different source than the calibration standards) before samples, then again after at least every 10 subsequent injections, and at the end of every sequence. Concentration should be within  $\pm 15\%$  of the expected value. If any of the second source checks fall outside this range, if possible, stop the run and inspect the peristaltic pump tubing for flat spots and also check if possibly clogged. Replace if necessary. Check that the lamp energy has an**

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acceptable value. For further troubleshooting refer to NLC:WG:510-1 in the troubleshooting section (NLC only). If drift is suspected, re-calibrate and run a second source check. If it still fails after troubleshooting and/or re-calibrating, follow the protocol in SSD:WG:202 Flow Diagram for QC Failures.

4. Any sample above the linear range of the standard curve needs to be diluted and retested. Insert blanks to check for carryover after any sample suspected of having high sodium or potassium.
5. Each sample is analyzed in triplicate by the instrument and a %RSD value is calculated. If the %RSD is  $\geq 10\%$ , the sample must be re-prepared and re-analyzed.

### Major Sources of Uncertainty

1. Standard Preparation
2. Dilution
3. Instrumental Factors such as the flame and lamps

### Calculation

Use the operating system software (a linear calibration algorithm) to calculate the linearity, slope, and concentrations of sodium and potassium.

**Note:** Use instrument software or LIMS to account for dilution or multiply the result by the correct dilution factor.

### Reporting Results

For beverage samples, report sodium and potassium as ppm to the nearest whole number, i.e. xx ppm.

For nonbeverage samples, report salt content to one decimal place, i.e. x.X g/100 mL.

### Safety Notes

Normal laboratory safety protocol should be followed.

Consult the MSDS for any chemicals that are unfamiliar. All chemicals should be considered hazardous - avoid direct physical contact.

### References

1. Association of Official Analytical Chemists (AOAC) Method 963.13 (17<sup>th</sup> Edition).

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2. Hobart H. Willard et al, *Instrumental Methods of Analysis*, Wadsworth, Inc., Belmont, California, 1988.
3. Lloyd A. Currie, *Detection in Analytical Chemistry*, American Chemical Society, Washington, DC 1988.
4. Wetz, B., *Atomic Absorption Spectroscopy*, Verlag Chemie, New York, 1976.

## Required Training, Certification and Re-certification

1. Initial in-house training by certified chemist in standard preparation, AAS operation.
2. Analyst has demonstrated competency after successfully obtaining values of 7 replicates of the LCS within LCS control limits. Refer to SSD:QPD:305 for more details.
3. Chemists will be recertified at least every 5 years using proficiency tests results or re-demonstration of competence. Refer to SSD:QPD:305 for more details.

## Revision History

Rev. 4 – General editorial changes and clarification identified in document review – 10/31/2008

Rev. 5 – changed linear range units to ppm from mg/L in order to be consistent with LOD, LOQ and reporting units – 3/22/2012

Rev. 6 – Changes to quantitation limit and linear range for sodium and potassium -- 5/9/2013

Rev. 7 – Addition of nonbeverage soy sauce and salted wines and autodilutor

Rev. 8 – update to associated documents

Rev. 9 – Extensive edits.