QUICK DETERMINATION OF WATER PHASE SALT CONTENT OF SMOKED FISH

Microwave Oven Procedure

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Achieving proper levels of salt and moisture in the production of smoked fish is a critical step in preservation. Salt analysis by official methods can be expensive and time consuming and requires skilled technicians. However, a good estimate of salt and moisture can be obtained quickly using a much simpler (but perhaps less accurate) test which relies on "Chloride titrators" and a microwave oven. This technique is accurate enough for quality control purposes, requires relatively inexpensive equipment, and is easy to learn. However, to be sure smoked products meet regulatory requirements, samples should be submitted to qualified laboratories as necessary to verify accuracy of in-house testing.

The equipment and procedures listed below offer alternatives which generally trade accuracy for time, convenience, or expense. The most accurate procedure uses a drying oven for moisture analysis with silver nitrate titration for salt and will require 10 to 12 hours to complete.

EQUIPMENT & MATERIALS	**APPROXIMATE COST (2000)**
Chloride titrators	
Quantab high range (300-6,000 ppm Chloride/.05 to 1.0% as NaCl) Hach Chemical Co, bottle of 40. catalog # 27513-40	$31.95
Food blender with canning jar type threaded screw-on base	
Oster household models (discount houses)	$40.00 to $60.00
Mini blend jars, one cup/lid, pack of 2 (#4888-3)	$10.00
Processing unit (blades)/sealing ring (#4901-3)	$10.00
or Power Blend Blade only (#4961) same price	$10.00
Blender jar base (#4902-3)	$6.00
Blender seals, pack of 2 (#4900.3)	$2.00

Note: to conveniently handle multiple samples without stopping to clean and dry equipment, use two Mini blend jars, two Processing Units, and one blender base for each sample. Commercial (heavy duty) Oster® blenders and accessories can be obtained from

some scientific supply companies. Use Sunbeam® Service Center Locator (www.sunbeam.com) to find Oster® accessories (and sometimes blenders). Half pint wide-mouth glass canning jars or plastic oyster containers with standard screw threads to fit Oster blender blades are available at discount stores for about 12.00 per case of 12. However, glass jars can be dangerous. See safety precaution below.

Balance, Minimum 200 gram capacity, 0.1 gram readability

New Ohaus ® Compact Scales
Cole-Parmer catalog # U-11003-02 $100.00

Power adapter, 120 VAC, 50/60 Hz
Cole-Parmer catalog # U-11003-55 $21.00

Calibration mass (200 gram)
Cole-Parmer catalog #U-11953-27 $42.80

Note: balances of higher capacity can be more versatile and cost effective an example is an Acculab® portable top loading balance with 0.1 g readability and 2400 g capacity for $350 including power adapter. Weighing mass is extra.
Cole-Parmer catalog # U-11321-28

Graduate cylinder, 100 ml plastic

Cole-Parmer U-06137-56 $5.00

Distilled water (per gallon**)** $2.00

Note: clean tap water can be used if tests show it contains no salt

Filter Paper

Whatman #1, 11cm dia (per 100)
Cole-Parmer U-06648-13 $7.90

Note: clean salt free paper towels or napkins can be used if tests show they contain no salt

Microwave oven (Carousel type) with timed seconds control (discount store) $150

Note: any type microwave will work but without a timed control reading in seconds, a stopwatch should be used.

Glass fiber sample pads

C.E.M Corp, 10 cm round, part # 1002, per box of 200 $19.50

Paper plates, 6 inch dia.

from local grocery store (per 100) $2.00

Wash battle, 250 ml polyethylene

Cole-Parmer, Nalgene Economy, pack of 6 $15.50

EQUIPMENT SOURCES: The following companies are among many who supply good equipment at comparable prices. The use of names here is for example only and does not imply any endorsement.

Environmental Test Systems PO Box 4659 Elkhart, Ind 46514 219 262-2060 Quantab Chloride Titrators (Catalog no. 27449-40, .05 to 1.0% salt range) can now be obtained from: Hach Chemical Co., 800 227-4224, or <www.hach.com>	C.E.M. Corp PO Box 200 Matthews, NC 28106 C.E.M Corp is now selling glass fiber sample pads (part no. 1002, $50 min order) through: Data Support Co., PO Box 261338, Encino CA 91436, 800 344-6317	Fisher Scientific (1-800-766-7000) <www.fishersci.com>
Osterizer® Div. of Sunbeam Corp. 1-800-597-5978 (US) 1-800-667-8623 (Canada) Service Center Locator <www.sunbeam.com>	VWR Scientific 1-800-932-5000 <www.vwrsp.com>	Cole-Parmer 1 800-323-4340 <www.coleparmer.com>

PROCEDURE FOR WATER PHASE SALT ESTIMATES

Note: For saftey reasons read the entire procedure before beginning!

Conduct moisture analysis before salt determination. Knowing the moisture content of the sample prior to the salt analysis will improve the accuracy of determining dilution factors. Quantab© procedures for "Solid or Semi-Solid" samples does not consider sample moisture content and uses a one to ten dilution factor which will slightly overstate the salt content of smoked fish.

SAMPLE PREPARATION

1. Cut 150 to 200 gram sample (about 1 x 1 x 3 inches) from thickest section of largest piece of fish from batch. Remove skin, place in jar, attach blender blades, and pulse blend until well chopped and mixed.

> **CAUTION: using glass canning jars can be dangerous, do not operate blender while holding the jar. Severe injury can result if the jar breaks. Plastic Oster brand plastic Mini Blending jars are recommended.**

2. Place lid (canning jar lids and rings) on blended sample and retain in refrigerator until satisfactory completion of salt and moisture analysis.

MOISTURE ANALYSIS - (using microwave oven and glass fiber pads).

> Note: Total time needed to dry sample to constant weight will vary with sample size, texture, and moisture content. Practice will show the operator the best sequence of heating times to give the quickest results without scorching the sample. Several trial runs need to be made to calibrate procedure to requirements of sample and microwave oven. Vary oven heat level and dwell time to optimize drying rate. BE SURE TO DRY TO CONSTANT WEIGHT and DO NOT BURN OR SMOKE SAMPLE.

1. Dry two CEM glass fiber sample pads and two microwave safe paper plates to constant weight in microwave oven without scorching (less than one minute).

2. Zero the balance. Place one pre-dried glass pad on the balance (use forceps). Place one paper plate containing the second glass pad on top of the first pad. Weigh and record weight.

3. Weigh and record weight of about 10 grams ± 0.1 g ground sample on the glass fiber pad which is nested in the paper plate. Spread sample, and cover with second pad and a second paper plate (inverted). Mark the top plate with sample number and weights - it will not be weighed.

4. Remove sample, pads, and plates from balance then compress sample by pressing firmly on top plate (do not contaminate the sample with grease from fingers or other foreign matter).

5. Microwave for 30 seconds or less, record time, lift top plate and pads using forceps, and blot any moisture from the lower plate or microwave platform with paper towel. Weigh bottom plate, pads, and sample. Record weight.

6. Replace top plate and pads. Microwave for another 30 seconds or less and reweigh. Record time and weight of bottom plate, pads, and sample.

7. If moisture condensation is observed on the balance platform wipe it off with a dry paper towel. If moisture persists, set the paper plates and sample on dry paper towel in the microwave. In some cases it may be helpful to invert the two pads on the lower plate, replace upper plate. Microwave for additional 15 seconds or less. Record time and weight. Repeat step 7 if necessary until no further weight loss is observed. Do not "smoke" sample.

> **Caution: Do not leave the microwave unattended when heating. Scorching of samples or paper plates can cause fire.**

8. Record final weight, compute weight loss, and calculate percent moisture as weight loss divided by sample weight

SALT ANALYSIS (adapted from Quantab© procedure)

1. Place exactly 10.0 grams of sample directly into a clean dry blender jar. Zero balance first if the balance has the capacity.

2. Add 90 mls (or 90 grams) of boiling distilled water. Use boiling tap water only if a test with a Quantab© shows the tap water to be salt free.

3. Attach blender blades and can then pulse blend for 30 seconds, wait one minute (or longer if particles are large), and stir another 30 seconds to insure all salt is extracted from sample.

4. Place salt titrator in sample (enclosed in filter paper) as prescribed in titrator instructions and read value from titrator. Look up percent salt in solution from titrator calibration chart.

5. Compute the amount (grams) of salt in the original 10 gram sample by multiplying the titrator %salt reading by the grams of water in the diluted sample. The % of salt is then calculated by dividing the grams salt by 10 and multiplying the result by 100.

$$\%Salt = \left[\frac{gramssalt}{10}\right] x100$$

Example: A 10 gram sample was determined to have %60 moisture and therefore contributed 6 grams moisture to the diluted sample giving a total of 96 grams (90 + 6) solution. A titrator reading of 0.3% would mean there was 0.288 grams of salt in the solution (0.003 x 96). The percent of salt in the 10 gram sample would then be 2.88% ([0.288/10]x100).

$$\%Salt = \left[\frac{0.288g}{10}\right] x100 = 2.88\%$$

COMPUTE WATER PHASE SALT CONTENT

Water Phase Salt can be calculated by using either percent or grams of salt and moisture from the analysis.

$$WPS = \left[\frac{\%Salt}{\%Salt + \%Moisture} \right] X100$$

or

$$WPS = \left[\frac{gSalt}{gSalt + gMoisture} \right] X100$$

Example: Using the example above where a10 gram sample of smoked fish was found to have 60% moisture and 2.88% salt (0.288 g salt and 6.0 g moisture in a 10 gram sample)

$$WPS = \left[\frac{2.88\%Salt}{2.88\%Salt + 60\%Moisture} \right] x100 = \frac{2.88}{62.88} x100 = 4.58\%$$

or

$$WPS = \left[\frac{0.288gSalt}{0.288gSalt + 6.0gMoisture} \right] x100 = \frac{0.288}{6.288} x100 = 4.58\%$$

Notes on Procedures

a. Osterize® brand food blenders used with small Oster® plastic Mini Blending containers work best because the sample is contained during mixing. Glass jars might break during blending, do not hold a glass jar while blender is operating. Oster® makes heavy plastic one cup blending jars for this purpose.

b. Any clean container will suffice for hot water extraction but they must be salt free (conducting the extraction in a blender jar is most convenient).

c. Clean paper towels will suffice for filter paper if a Quantab© test is run to insure that they are salt free.

d. Use a wash (squirt) bottle of distilled water to add the last few milliliters of water to the salt sample.

e. Boiling tap water can be used for extraction in place of distilled water as long as it is shown to be salt free. An easy way to heat a few hundred milliliters of water is with a microwave oven. When doing frequent samples an electric tea pot is handy.

f. Be sure to save the sample in the original blending container (refrigerated with lid on) until the results are accepted.

g. If the salt content of diluted solution is so high that the titrator strip goes off scale, simply dilute the solution, test with another titrator strip, and then multiply the result by a the appropriate dilution factor.

The minimum investment for salt and moisture analysis should be less than $350, including the cost of a small carousel type microwave oven. At 80 cents per titrator, 20 cents for two sample pads, plus a few cents for miscellaneous supplies, the cost per test will run about $1.00 each.

USE OF SALT CHARTS AND GRAPHS

Use Chart I to estimate how much salt must be in raw brined fish (55% to 80% raw brined moisture) to give 3.5% WPS in finished product (40% to 80% final moisture).

CHART I
RAW BRINED FISH SALT CONTENT NEEDED
TO ACHIEVE 3.5 % WATER PHASE SALT (WPS)
AT VARIOUS FINAL MOISTURE CONTENTS

	MOISTURE CONTENT OF RAW BRINED FISH					
Final moisture	55%	60%	65%	70%	75%	80%
40%	1.09	0.96	0.85	0.72	0.60	0.46
45%	1.33	1.18	1.03	0.69	0.74	0.59
50%	1.63	1.44	1.27	1.09	0.91	0.72
55%	1.99	1.76	1.55	1.33	1.11	0.88
60%		2.18	1.91	1.61	1.36	1.09 *
65%			2.36	2.03	1.69	1.35
70%				2.54	2.12	1.69
75%					2.72	2.18
80%						2.90

To predict final WPS in a finished product you must first conduct a salt and moisture test of the raw brined or salted fish and know the final moisture content of the product you wish to produce. For example, a raw brined fish with 80% moisture and 1.09% salt (see

line with * at the end) will have 3.5% WPS when dried to 60% final moisture. Anything less than 1.09 % salt will not produce 3.5% WPS in a product which is finished to 60 % moisture.

Also, testing moisture and salt after brining and prior to smoking gives information useful in predicting how much water (shrinkage) must be removed to achieve any final WPS level. Shrinkage is (100 - %yield).

% y = %Yield needed to give a final WPS with any raw %salt and %solids.

% solids = 100 - % moisture

$$\%Yield = \%Salt\left(\frac{100}{\%WPS}(-1)\right)(+\%Solids)$$

$$\%Yield = \%Salt\left[\frac{100}{\%WPS}(-1)\right]+\%Solids$$

$$\%Yield = \%Salt\left[\frac{100}{\%WPS}(-1)\right]+(100-\%moisture)$$

To estimate approximate final yield necessary to produce any desired final WPS based on salt and moisture content of raw brined fish.

%Yield = %Yield needed to give a desired final product WPS with any %Salt and %Solids from raw brined fish prior to smoking. %Yield = 100 - %Weight Loss in the smokehouse.

First brine fish, then measure %moisture and % Salt content and enter desired WPS into the equation. The result will be the yield in the smokehouse necessary to give the desired WPS.

For Example: After brining, 300 pounds of raw fish (brined weight) is tested to have 2% Salt and 75% Moisture. It will need to be smoked (dried) to 240.42 pounds (80.14 % Yield) to have a WPS content of 3.5%. This assumes that no solids (e.g. fat & oil) are baked out of the fish during smoking. To be accurate this equation assumes that all weight loss is from moisture evaporation (drying).

$$\%Yield = \%Salt\left[\frac{100}{\%WPS}(-1)\right] + (100 - \%moisture)$$

$$= 2.0\left[\frac{100}{3.5}(-1)\right] + (100 - 75)$$

$$= 2.0(28.57 - 1) + 25$$

$$= 2.0(27.57) + 25 = 55.14 + 25 = 80.14$$

Quantab© Accuracy and Storage

Quantab© chloride salt titrators are very accurate but will deteriorate over time due to exposure to moisture and light. Be sure to follow the manufacturers recommendations for storage. Moisture proof storage might be obtained by vacuum sealing the titrator jar between uses or by storing in a larger tightly sealed jar containing some desiccant like that which is used to dry flowers and can be purchased in many discount stores.

While the accuracy of the titrators may be high, there are several sources of error including:

Variability between samples
Variability within a sample (inadequate mixing)
Weighing error (a scale which weighs only to 1/10th gram might read 10.0 g while the sample actually weighs somewhere between 9.95 and 10.05 g.

The accuracy of a jar of titrators can be checked by preparing a standard salt solution and keeping it on hand in a tightly sealed glass container. Pick a salt concentration in the middle of the titrator range and weigh out the appropriate amount of salt and water.

Example: To make up a standard test solution of 0.304% salt, add exactly 2 grams of salt to 655.9 grams of distilled water. The formula for this calculations is:

$$(0.00304)x(X) = 2.0 grams$$

X = total weight of standard solution

$$X = \frac{2.0}{0.00304} = 657.9 grams$$

The weight of the final solution containing 2.0 grams of salt is 657.9 grams so the weight of the water in the solution must be 655.9 grams (657.9 minus 2.0 = 655.9). The easy way to do this is to zero (tare) a container on a scale capable of weighing to the nearest 1/10 gram or better, add 2.0 grams salt, zero the balance again, then add exactly 655.9 grams of distilled water. Use an eye dropper or small squirt bottle for better accuracy. If more that 655.9 grams water is inadvertently added, simply recalculate the exact salt percentage by dividing the grams salt by the total grams of solution. The larger the amount of test standard made, the higher the degree of accuracy due to weighing error.

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