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Moisture in seafood has come under increasing federal scrutiny since the U.S. Food and Drug Administration (FDA) established the FDA Office of Seafood in 1991. A foremost priority of this new FDA branch was to address economic fraud issues, with the addition of water to seafood of primary focus. Currently, FDA maintains a zero tolerance for added water to seafood. The scallop industry became the initial focal point for policy enforcement due to common industry handling and processing practices which result in scallop moisture uptake. Due to similar processing practices, the shrimp industry will likely come under tighter federal scrutiny.

Due to an interim agreement reached between FDA and the scallop industry, all scallops are marketed either as scallops or scallop products, with moisture content determining the product identity (FDA 1992). Scallops with moisture levels below 80% may be labeled and marketed as scallops, while scallops with moisture levels between 80-84% must be labeled as "scallop products." Scallops over 84% moisture are deemed adulterated by FDA and, therefore, unmarketable. To stay in compliance with this regulation, scallop processors must constantly monitor moisture content of scallops from receiving, through processing and distribution. Multiple sampling and analysis for moisture using a private lab is expensive and not timely, with results not available for one to two days. The time factor creates a large problem for processors, who need moisture levels at time of processing in order to continue operations by packing in appropriately labeled containers. The processor needs a tool to provide real-time moisture data for process control.

Prior to the FDA interim agreement, an automated moisture analyzer was introduced to the scallop industry by Virginia Sea Grant as a tool to monitor scallop moisture levels for in-house quality control checks. This automated unit, the Ohaus MB 200, uses an infrared heating element to rapidly drive-off moisture, providing moisture results in 15-18 minutes. The balance requires programming to run on automatic mode, which dries the sample at a specific temperature until a selected rate of drying is reached. These drying parameters were established for scallops by Virginia Sea Grant through multiple testing against the recognized standard method by the Association of Official Analytical Chemists (AOAC) International. Many industry members have invested in this instrument allowing them on-line process control to maintain product uniformity and regulatory compliance.

For regulatory compliance, inspection agencies traditionally collect samples and send them to their respective laboratories for analysis using standard AOAC methods. However, federal National Marine Fisheries Service (NMFS) inspectors began using the Ohaus instrument for regulatory inspections, and basing compliance on those results. The Ohaus MB 200 moisture balance is not a recognized standard method for moisture determination, and to date, no one has performed a structured collaborative study to determine if this balance could be considered for acceptance as a standard method. A collaborative study was conducted to determine the accuracy, repeatability and reproducibility of the Ohaus moisture balance compared with the AOAC International method.

Collaborative Study

This collaborative study was conducted in 1997. Four scallop and four shrimp meat samples (representing blind duplicates) were submitted to 8 collaborating laboratories, each experienced in scallop and/or shrimp testing, for the determination of moisture using the Ohaus MB 200 infrared moisture balance as described in the method. The samples represented unprocessed scallops and shrimp with low moisture (76-80%) and phosphate (2.5% sodium tripolyphosphate) processed product with high moisture (82-86%) content. Samples were prepared according to AOAC food processor method 983.18A (Official Methods of Analysis 1990, 15th ed., 1st supplement, AOAC, Arlington, VA). Identical samples (approximately 100 g units) of both scallop and shrimp meat types were vacuum packed, frozen, coded, then distributed to collaborating laboratories. Collaborators were instructed to completely thaw meat samples under refrigeration, open vacuum sealed bags, transfer each sample into mixing bowl of a small food processor, cover, then re-mix sample for 15 seconds prior to moisture analysis. A single moisture analysis was performed on each sample. Identical blind duplicate samples of both scallop and shrimp meat types were also submitted to four FDA regional laboratories where the samples were analyzed in duplicate for moisture by AOAC method 950.46 (Official Methods of Analysis 1990, 15th ed., AOAC, Arlington, Virginia). The FDA regional labs functioned as control analysts in this study.

Ohaus MB 200 Method

Moisture is removed from the sample by using a temperature controlled infrared heater. Weight loss is determined by electric balance readings before and after drying and is converted to % moisture content by an internal microprocessor.

For scallops and shrimp, the MB 200 is used in the AUTO DRY MODE with meat samples dried at 180^{o} C to a rate of .05g in 60 seconds (180 /0.5g/60s). Test set-up procedures are available from Ohaus Corp., 29 Hanover Road, Florham Park, New Jersey, 07932. Samples are prepared in a food processor as previously described. A 10-11g sub-sample of homogenate from food processor is thinly and evenly spread over entire surface of a tarred aluminum pan liner. The pan liner with sample is then replaced onto the balance platform. The hood of the moisture balance is closed, and the start button is pressed to initiate the drying process. When the test is completed, the stop indicator will light, an audible signal will sound, and the upper display will read test complete. Percent moisture will appear on the lower display. Allow platform to cool before running another test.

Results

Results were analyzed by statistical methods outlined by Youden and Steiner (1975). Statistical values used for comparison include the mean, standard deviation, within-laboratory repeatability and between-laboratory reproducibility. Outliers (labs) were identified by both the Cochrans maximum variance test and the Grubbs extreme value (mean) test (Table 1). Within lab error was probable for lab # 8 in the Ohaus group (scallop, high and shrimp, low) and lab #4 in the AOAC group (shrimp, low). Results and summary statistics for the collaborative study are given in Table 2 for the Ohaus balance, and Table 3 for the AOAC method. Statistics for each product tested include the mean (X), range and standard deviation (s). Overall statistical summaries were calculated with and without outliers, which are shown in bold type. Further, all precision estimates exclude values from outlying laboratories.

Results from this study show varying levels of agreement between the two moisture methods tested. When looking only at the overall means from both collaborative groups, the two methods seem to be comparable for all four products. However, when focusing on the precision estimates, differences become apparent. The repeatability value is the number that the difference between duplicate analyses (range) by the same lab, same sample and same method cannot exceed if the method is used properly under repeatability conditions. Within laboratory repeatability was demonstrated for all products within the AOAC method group, and all but two products within the Ohaus method group. Scallop (high) and shrimp (high), both from laboratory 5 (Table 2) had ranges beyond their respective repeatability value. Between laboratory reproducibility was also observed in the AOAC group for all products. Reproducibility between labs in the Ohaus group was also observed in all products except two. The difference between determinations from lab 6 and 8 (Table 2) for scallop (low), and that between labs 4 and 5 for shrimp (low) were beyond the values calculated for reproducibility. In general, when comparing precision estimates of two methods, the lower of the two for a given product tested indicates more precision.

For ease in further comparing results, information from Tables 2 and 3 have been combined and presented in Table 4. The precision estimates calculated from the AOAC method data are all considerably lower that those calculated from the Ohaus method data. This indicates that the AOAC method is a more precise method for moisture determination.

Though the standard AOAC method demonstrated more precision than the Ohaus balance in this study, acceptance of the Ohaus as a rapid method for moisture determination for industry use should not waver. This study indicated good correlation between the methods (means), while also demonstrating possible limitations for the Ohaus balance. Most deviation occurred between-lab determinations. Since sample homogeneity was apparently not a factor (observing summary statistics), sample preparation, or re-mixing the sample in this study, and varying levels of operator experience, may cause varying results when using the Ohaus balance. The Ohaus balance remains a very effective, and relatively inexpensive tool for industry use for monitoring moisture, but should be backed-up by the standard AOAC method for regulatory compliance issues.

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Table 1. Outliers identified by the Cochran maximum variance test and the Grubbs extreme value (mean) test for moisture determinations by the Ohaus rapid method and by AOAC standard method. Outlying laboratories are indicated by number with respect to product tested and method followed.

Method	Product tested	Cochran	Grubbs
Ohaus	Scallop, high	8	8
	Shrimp, low	8	
AOAC	Scallop, low		1
	Shrimp, low	1	1
	Shrimp, high	4	

Table 2. Collaborative results for the determination of moisture (%) in blind duplicate samples of scallops and shrimp by the Ohaus infrared balance.

Coll.	Blind dup.	Scallops (low)	Summary stats. X	range	s	Scallops (high)	Summary stats. X	range	s	Shrimp (low)	Summary stats. X	range	s	Shrimp (high)	Summary stats. X	range	s
1	1	81.0	—	—	—	85.0	—	—	—	83.8	—	—	—	83.8	—	—	—
	2	80.6	80.8	0.40	0.283	85.8	85.4	0.80	0.566	83.2	83.5	0.60	0.424	83.7	83.7	0.100	0.071
2	1	82.1	—	—	—	87.2	—	—	—	84.5	—	—	—	85.4	---	---	---
	2	82.1	82.1	0.00	0.000	87.3	87.2	0.10	0.071	85.1	84.8	0.60	0.424	85.5	85.4	0.100	0.071
3	1	82.1	—	—	—	86.5	—	—	—	84.5	—	—	—	85.2	---	---	---
	2	81.8	81.9	0.30	0.212	86.7	86.6	0.20	0.141	84.5	84.5	0.00	0.000	85.3	85.2	0.100	0.071
4	1	82.3	—	—	—	87.6	—	—	—	84.9	—	—	—	85.7	---	---	---
	2	81.4	81.8	0.90	0.636	87.5	87.5	0.10	0.071	84.9	84.9	0.00	0.000	85.6	85.6	0.100	0.071
5	1	81.7	—	—	—	85.2	—	—	—	83.5	—	—	—	85.3	---	---	---
	2	81.8	81.7	0.10	0.071	87.1	86.1	1.90	1.344	82.2	82.8	1.30	0.919	84.1	84.7	1.200	0.849
6	1	82.8	—	—	—	87.5	—	—	—	84.8	—	—	—	85.7	---	---	---
	2	82.5	82.7	0.30	0.212	87.6	87.5	0.10	0.071	84.7	84.7	0.10	0.071	85.9	85.8	0.200	0.141
7	1	81.2	—	—	—	87.3	—	—	—	83.2	—	—	—	85.4	---	---	---
	2	80.6	80.9	0.60	0.424	87.2	87.2	0.10	0.071	84.6	83.9	1.40	0.989	84.9	85.1	0.500	0.354
8	1	81.2	—	—	—	**85.5**	**—**	**—**	**—**	**83.6**	**—**	**—**	**—**	85.6	---	---	---
	2	80.0	80.6	1.20	0.849	**77.4**	**81.4**	**8.10**	**5.728**	**80.0**	**81.8**	**3.80**	**2.548**	84.9	85.2	0.700	0.495
			X	**range**	**s**		**X**	**range**	**s**		**X**	**range**	**s**		**X**	**range**	**s**
Overall N=16			81.6	2.80	0.780		86.1	10.20	2.495		83.9	4.90	1.314		85.1	2.20	0.684
Excluding outliers N=14			-------		------		86.8	2.60	0.879		84.2	2.70	0.867		-----		-------
Precision Est.		Labs=8				Labs=7				Labs=7				Labs=8			
Repeatability*			1.20				1.56				1.57				1.05		
Reproducibility**			2.20				2.52				2.45				1.96		

*Number that the difference between duplicate analyses (range) by same lab, same sample and same method cannot exceed if the method is used properly under repeatability conditions.
**Number that the difference between a single determination by one lab and a single determination by another lab cannot exceed if the method is operating under reproducibility conditions.
Outliers for a given laboratory and product tested are presented in bold type.
x=mean, s=standard deviation.

Table 3. Collaborative results for the determination of moisture (%) in blind duplicate samples of scallops and shrimp by standard AOAC method.

Coll.	Blind dup.	**Scallops (low)**	Summary stats. X	range	s	**Scallops (high)**	Summary stats. X	range	s	**Shrimp (low)**	Summary stats. X	range	s	**Shrimp (high)**	Summary stats. X	range	s
1	1	**78.7**	**—**	**—**	**—**	86.5	—	—	—	84.6	—	—	—	**83.1**	**—**	**—**	**—**
	2	**78.6**	**78.6**	**0.10**	**0.071**	86.1	86.3	0.40	0.283	84.6	84.6	0.00	0.000	**83.7**	**83.4**	**0.600**	**0.424**
2	1	81.2	—	—	—	87.0	—	—	—	84.2	—	—	—	85.2	---	---	---
	2	81.4	81.3	0.20	0.141	87.1	87.1	0.10	0.071	84.5	84.3	0.30	0.212	85.1	85.1	0.100	0.071
3	1	81.6	—	—	—	87.2	—	—	—	84.6	—	—	—	85.4	---	---	---
	2	81.7	81.6	0.10	0.071	87.1	87.1	0.10	0.071	84.8	84.7	0.20	0.141	85.3	85.3	0.100	0.071
4	1	81.3	—	—	—	87.0	—	—	—	**88.1**	**—**	**—**	**—**	85.3	---	---	---
	2	81.4	81.3	0.10	0.071	87.1	87.0	0.10	0.071	**84.7**	**86.4**	**3.40**	**2.404**	85.3	85.3	0.000	0.000
			x	**range**	**s**		**x**	**range**	**s**		**x**	range	**s**		**X**	range	**s**
Overall N=8			80.7	3.00	1.298		86.9	1.10	0.383		85.0	3.90	1.260		84.8	2.30	0.883
Excluding outliers N=6			81.4	0.50	0.186		-----	------	------		84.5	0.60	0.173		85.3	0.30	0.103
Precision Est.		Labs=3				Labs=4				Labs=3				Labs=3			
Repeatability*			0.28				0.43				0.41				0.16		
Reproducibility**			0.57				1.15				0.58				0.31		

*Number that the difference between duplicate analyses (range) by same lab, same sample and same method cannot exceed if the method is used properly under repeatability conditions.
**Number that the difference between a single determination by one lab and a single determination by another lab cannot exceed if the method is operating under reproducibility conditions.
Outliers for a given laboratory and product tested are presented in bold type.

Figure 4. Direct comparison of average moisture (%) determinations for shrimp and scallops and precision estimates generated from collaborative study comparing the Ohaus moisture balance with the standard AOAC method. Outliers have been excluded from these comparison values.

	Scallops (low moisture)	Scallops (high moisture)	Shrimp (low moisture)	Shrimp (high moisture)
Repeatability*				
Ohaus method	1.57	1.22	1.06	1.58
OAOC method	0.44	0.26	0.18	0.42
Reproducibility**				
Ohaus method	2.55	2.23	1.98	2.47
AOAC method	1.20	0.57	0.32	0.59
Mean Moisture (%)				
Ohaus method	86.82	81.57	85.15	84.15
AOAC method	86.88	81.43	85.27	84.55

*Number that the difference between duplicate analyses (range) by same lab, same sample and same method cannot exceed if the method is used properly under repeatability conditions.
**Number that the difference between a single determination by one lab and a single determination by another lab cannot exceed if the method is operating under reproducibility conditions.

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