www.trjfas.org
ISSN 1303-2712
Turkish Journal of Fisheries and Aquatic Sciences 12: 1-5 (2012) DOI: 10.4194/1303-2712-v12_1_01

PROOF

Comparison of Fresh and Dried Flathead Grey Mullet (*Mugil cephalus*, Linnaeus 1758) Caviar by means of Proximate Composition and Quality Changes during Refrigerated Storage at 4±2°C

Ufuk Çelik[1,*], Can Altınelataman[1], Tolga Dinçer[1], Deniz Acarlı[2]

[1] Ege University, Fisheries Faculty, 35100, Izmir, Turkey.
[2] Çanakkale Onsekiz Mart University, Gokceada School of Applied Sciences, 17760, Çanakkale, Turkey.

* Corresponding Author: Tel.: +90.232 3115229; Fax: +90.232 3747450;
E-mail: ufuk.celik@ege.edu.tr

Received 22 April 2011
Accepted 01 June 2011

Abstract

The aim of this research was to compare the differences in shelf life and resistance to decomposition of fresh and dried caviar produced from flathead grey mullet (*Mugil cephalus*, Lin. 1758) caught from Homa Lagoon, which is located 20 km northeast of Izmir Bay, Turkey. The proximate compositions of caviars were determined as follows: moisture 50.2±0.15 %, crude fat 13.1±0.23 %, protein 25.7±0.43 %, ash 1.48±0.03 % and carbohydrate 9.59±0.10 %, for fresh caviar and moisture 26.3±0.53 %, crude fat 13.3±1.51 %, protein 41.8±0.56 %, ash 4.68±0.11 % and carbohydrate 14.0±0.05 % for dried caviar. The proximate composition values of dry and fresh caviars were significantly different ($p<0.05$). To determine the refrigerated shelf life, chemical quality control analysis using Total Volatile Basic Nitrogen (TVB-N), Trimethylamine (TMA) and Thiobarbituric Acid (TBA), pH and color values were measured. Both fresh and dried samples were found to be spoiled end of the 8th day of refrigerator storage, based on their TMA-N and TVB-N values.

Keywords: Grey mullet, Color measurement, Caviar, TBA, TMA, TVB-N.

Buzdolabında 4±2°C'de Muhafaza Edilen Taze ve Kurutulmuş Kefal (*Mugil cephalus*, Linnaeus 1758) Havyarının Yaklaşık Kompozisyon ve Kalite Değişimlerinin Karşılaştırılması

Özet

Bu araştırmanın amacı Türkiye'de İzmir Körfezinin kuzey doğusunda ve merkeze 20 km mesafede bulunan Homa Dalyanından avlanmış kefallerden *(Mugil cephalus*, Lin. 1758) elde edilmiş taze ve kurutulmuş havyarların raf ömrü ve bozulma direnci farklılıklarının karşılaştırılmasıdır. Yaklaşık kompozisyon değerleri sırasıyla su şekilde tespit edilmiştir: taze havyar için, nem % 50.2±0.15, ham yağ %13.1±0.23, protein % 25.7±0.43, kül % 1.48±0.03 ve karbonhidrat % 9.59±0.10 ve kurutulmuş havyar için, nem % 26.3±0.53, ham yağ % 13.3±1.51, protein % 41.8±0.56, kül % 4.68±0.11 ve karbonhidrat %14.0±0.05. Kurutulmuş ve taze havyarların yaklaşık kompozisyonlarının belirgin olarak birbirinden farklı olduğu tespit edilmiştir ($p<0.05$). Buzdolabı raf ömrünü tespit etmek için, kimyasal kalite kontrol analizlerinden Toplam Uçucu Bazik Azot (TVB-N), Trimetilamin (TMA) ve Tiyobarbutirik asid (TBA) pH ve renk ölçümleri yapılmıştır. Hem taze hem de kurutulmuş örneklerde TMA-N ve TVB-N değerlerine göre, buzdolabı muhafazasının 8. gününde bozulma tespit edilmiştir.

Anahtar Kelimeler: Kefal, Renk ölçümü, Havyar, TBA, TMA, TVB-N.

Introduction

Caviar is the most popular and well-known processed edible fish roe product in wide demand all around the world. The term "caviar" refers to some of the processed fish roes, and is an expensive product high in nutrients, particularly the B vitamins (Altug and Bayrak, 2003). Sturgeon caviar is one of the most exclusive and expensive fishery products and this is increasing because of increasing interest and tremendous over-fishing of commercial sturgeon species (Wolf *et al*., 1999). There are more than 20 species of sturgeon harvested for caviar (Al-Holy *et al*., 2005). Mullets have gained importance for caviar production in recent years, especially with increased demand from France, Italy and the United States. The word 'bottarga' is used to refer to grey mullet caviar in the traditional gourmet food literature and it is generally consumed dried with spaghetti, salad and wine. Bottarga can also be smoked. Smoked grey mullet caviar is also a good appetizer. Same product is named as "avgotaracho" in Greece. Rogdakis (1994) has described avgotaracho process as; the whole ovaries dissected from the fish, washed with

water, salted with natural sea salt, dried under the sun and submerged in melted natural wax. This is one of the five products in the category of fresh fish, mollusks, crustaceans and products with protected designation of origin and geographical indication by laws of Greek Government and the European Union (Katselis *et al*., 2005). To obtain a good caviar a great deal of skills are required; nowadays different procedures exist, from the more traditional to the industrial ones; in the latter drying is conducted in rooms with controlled humidity and temperature. The final product can be sold as whole ovaries under vacuum packaging, or grated in jar. Bottarga has an amber color and its unique chewy mouth feel is due to the peculiar lipid composition, rich in wax esters (Bledsoe *et al*., 2003). Restriction Fragment Length Polymorphism (RFLP) analysis of 16s rDNA segments of different mullet species such as *Mugil cephalus*, *Liza aurata*, *Liza ramada* and *Liza saliens* has been done to establish the origin of fish roe samples (Klossa-Kilia *et al*., 2002). There are limited studies in terms of proximate composition and quality changes of mullet roes (bottarga). Scano *et al*., (2008) used the high resolution ^{13}CNMR spectroscopy together with HPLC and GC techniques to investigate the lipid extracts of different commercial samples of bottarga produced in Sardinia. Rosa *et al*., (2009) studied the lipid composition and oxidative stability of mullet (*Mugil cephalus*) raw roes and cured products (bottarga) to find lipid modifications due to manufacturing procedures.

This study is the first investigation to focus on the proximate composition and quality changes of the refrigerated bottarga with the terms "fresh" and "dried" refer to "roe" and "caviar" respectively.

Materials and Methods

Samples

The research material, female flathead grey mullet (*Mugil cephalus*, Lin. 1758) samples were obtained from the Homa lagoon which was located about 20 km northeast of Izmir bay. Two different samplings were done in summer season in the middle of August. For each sampling 6 individual female fish were selected with the average weight of 1409±210 g and 1512±110 g. First 6 samples were used to process caviar using 'dry-salting' method. After 4 days, 6 new fish were selected which's roe were removed with cold sea water for evaluating the fresh group. 12 female mullets (54±4 cm length) were used in the current study. After second sampling, the removed roe and dried caviars were placed in a polystyrene box with ice (ice/roe ratio 2/1) and transported to the laboratory (in 30 min) with using cooler truck.

Caviar Processing

Caviar process was carried out by using 'dry-salting' method in the processing area, which is located near the main building of the Fisheries Faculty pilot plant of Ege University (Turkey) in the lagoon. Blood and other wastes on the roe were removed with cold sea water (Special care has been given to avoid harvesting caviar until after approximately 15 minutes of cooling the fish). Then the surface moisture of the roe was removed with the help of a clean cloth. They were put in polystyrene boxes with one layer of salt and one layer of roe. Non-processed rock salt, which was obtained as clean from a salt processing plant near the lagoon was used in the study. According to the size of the caviars, they were let in salt, for small sizes (between 200g-300g) for 2.5 hours and for big sizes nearly 6 hours (for 300g and over). During this period, they were pressed one time by fingers in a palm for five minutes and afterwards they were dipped into tap water and covered with a wet cloth for 4-4.5 hours. After that, caviars were left standing in a dry cool place (approximately 20^{o}C) to be dried and they were placed on shelves under 250g wooden blocks to be shaped into the traditional bottarga. Product was turned over every two hours. When salt concentration exceeded and the excess salt was removed using a clean cloth. Caviars were expected to lose total moisture of 10% approximately.

Determination Of Proximate Composition

Dry matter was determined by drying the samples at 105^{o}C to a constant weight (AOAC, 1990). Crude protein content was calculated by converting the nitrogen content determined by the Kjeldahl method (6.25xN) (AOAC, 1990). Fat was determined by using the method described by Bligh and Dyer (1959). Ash was determined by using the method of Ludorff and Meyer (1973). Carbohydrate content of each sample was determined by difference. The analyses of the pooled samples were all carried out in triplicate.

Determination Of Quality Changes During Refrigerated Storage

Fresh and dried caviar samples were stored at a refrigerator (at 4±2°C). During storage, sampling was carried out at 1st, 4th and 8th day to determine chemical quality changes. Total volatile basic nitrogen (TVB-N) was determined according to the method of Vyncke (1996). Thiobarbituric acid (TBA) was determined according to the method proposed by Tarladgis *et al*. (1960). The pH value was recorded using a Hanna 211 model pH meter (Cluj-Napoca, Romania), the glass electrode being applied directly in the homogenate (5g of fish/5ml distilled water).The homogenate was prepared by using an Ultraturax homogenizer (Yellow line, DI 25 Basic, Staufen, Germany). The Ultraturax was dipped into the solution and used for 1 min.

Color Measurement Characteristics

All color measurements were carried out using a Spectro-pen® (Dr. Lange, Dusseldorf, Germany), a colorimeter measuring the visible spectral range (400 to 700 nm) at intervals of 10 nm, by the method of Schubring (2002). The colorimeter operates on the spectral method described in DIN 5033 (Deutsches Institut für Normung, CIE 1995) using the 45/ 0º circular viewing geometry, the sample is illuminated with polychromatic light encircling it at an angle of 45º, with the optical unit observing the reflected light from a horizontal angle (0º) towards the sample surface. A 10º standard observer and a D65 illuminant were used. The PC-software "Spectral – QC" for Windows (Spectral-QC Operating Instructions Version 3.6, Dr. Bruno Lange GmbH & Co. KG, 4/2002, Dusseldorf, Germany) was used for data processing. Before measuring each lot, the colorimeter was calibrated against a white standard (LZM 229). For each batch, 3 samples were taken. Dried and fresh caviar samples were minced separately in a Kitchen Aid KPMS Professional meat grinder (St. Joseph, Michigan, USA), equipped with 2 cm grinding blades and a metallic screen with 4 mm diameter circular holes. The pooled mince was placed in a plastic Petri dish and the color was measured on homogenates prepared from samples. Attention was paid for the surface to be smooth and the mince to be a nearly uniform color. The color measurement was repeated ten times using different parts of this surface. In the CIE Lab system (CIE, 1995) where L^* denotes lightness on a 0 to 100 scale from black to white; a^* denotes redness index, ranging from red (+) to green (-), and b^* denotes yellowness index, ranging from yellow (+) to blue (-) (Schubring, 2002).

Statistical Analysis

The SPSS (SPSS, 1999, Version 9.0. Chicago, IL, USA) program was used to test the differences between mean values of the different analysed parameters. Differences between means were analyzed by one-way analysis of variance (ANOVA) followed by either the Tukey or Duncan multiple comparison test, when a significant difference was detected between the days of storage ($P<0.05$). To compare the groups an independent sample-T test was used to determine the differences. The results were presented as means ± SD with the significance level set at $p< 0.05$ under varying storage periods.

Results and Discussion

Proximate composition values of fresh caviar samples were: moisture 50.2±0.15%, crude fat 13.1±0.23%, crude protein 25.7±0.43%, ash 1.48±0.03% and carbohydrate 9.59±0.10%. On the other hand, the dried caviar samples had significantly lower moisture and higher protein contents. This was expected due to drying effect on evaporating water partially out of the product resulting in an increase in dry weight. The percentage values were: moisture 26.3±0.53%, crude fat 13.3±1.51%, crude protein 41.8±0.56%, ash 4.68±0.11% and carbohydrate 14.0±0.05% as wet weight basis. In terms of dry matter in both samples, only salt content made difference for caviar. Caprino *et al*. (2008) have reported moisture, total lipid, crude protein and ash in caviar of fish that fed with soybean oil+fish oil combination as; 54.41%, 11.04%, 25.39% and 3.79% respectively.

The pH values of the samples during the storage can be seen in Table 1. The pH values of both fresh and dried samples ranged between 5.79 and 5.96 and did not changed significantly with time. TMA-N and TVB-N results for the fresh and dried samples can be seen in Table 2.

TMAO is an osmoregulating agent in salt water fish muscle but does not exist in large amounts in roe. Dried caviar showed reduced levels of TMA due to the limited content of water and TMAO at the start. Despite its limited amounts TMA also can be used as a secondary parameter of spoilage for caviar. In fresh fish, TMA-N values should be close to 1 mg N/100g and in spoiled samples it is more than 8 mg N/100g (FAO, 1986). TMA-N values obtained on the first day in refrigeration were 5.56±0.4 mg N/100g in fresh samples and 11.6±1.2 mg N/100g in dried samples. On the other hand, while TMA-N values of fresh caviar samples significantly increased ($p<0.05$) during the period of 8 days, a significant decrease occurred ($p<0.05$) for dried samples. On the 8th day of storage, fresh samples were spoiled based on TMA-N analysis but the dried samples improved in terms of this parameter. Therefore, TMA-N is not found suitable for measuring quality changes of dry caviar products since bacterial activity could be decreased depending on low water content. At low temperatures, denaturation of protein is reduced by inhibition of some enzymes and microorganisms, which are normally present in caviar. TVB-N should capture all the volatile N containing compounds during decomposition. Therefore, this parameter is accepted as a spoilage index for fish and seafood (FAO, 1986). FAO has indicated that samples with TVB-N value less than 25 mg N/100g are 'perfect quality', samples with up to 30 mg N/100g are 'good quality', samples

Table 1. pH values (means ± standard deviation) of fresh and dried caviar during refrigerated storage

Periods	pH	
	Fresh	Dried
Day 1	5.79±0.01^{a1}	5.86±0.00^{a1}
Day 4	5.83±0.00^{a1}	5.82±0.01^{a1}
Day 8	5.96±0.00^{b}	5.88±0.01^{b2}

Different superscript letters within each column represent significant differences ($p<0.05$) between days of storage; different numbers within each row represent significant differences between fresh and dried caviar (n=3).

with up to 35 mg N/100g are 'marketable quality' and the samples with TVB-N value more than 35 mg N/100g are indicated as 'spoiled' (Schormuller, 1968; Ludorf and Meyer, 1973). According to TVB-N values for both type of caviars spoiled on day 8.

The TBA index is a widely used indicator for the assessment of the degree of lipid oxidation (Nishimoto *et al*., 1985). At the beginning of the storage period, TBA values were 0.55±0.01 and 3.23±0.80 mg malonaldehyde/kg, respectively in fresh and dried samples (Table 3)**.** The changes in TBA values were negligible for the 8 days period. At the end of the storage while both kinds of caviar samples were spoiled according to the TVB-N values, no spoilage was detected according to the TBA test. Significant differences were observed in TBA values between fresh and dry samples with higher values representing dried products indicating the effect of drying process on lipid oxidation. Rosa *et al*. (2009) have stated that the PUFA oxidative stability in mullet roe products was influenced by different parameters, such as manufacturing procedures, storage, and physical state of the matrix.

The values of the color parameters are shown in Table 4.

Values of a* and b* for dried caviar slightly increased (except for b* value of fresh caviar) while the value of L* for the same samples dropped during the considered period of storage. These changes are not found statistically important during storage for the 8 day period and no other clear changes were observed.

Color measurement is one of the important parameters in processed fish products because of consumers' association with a natural and characteristic caviar color. Table 4 shows changes in the color values of dried and fresh mullet caviar during refrigerated storage. A significant increase and decrease occurred ($p<0.05$) in the L∗ values of fresh samples and dried samples, respectively. Therefore, while the color of the fresh samples changed as lighter, the color of the dried samples became darker during the storage. The a* and b* values also supported the results of L* parameter with an exception of b values of fresh samples indicating the darkness of dried samples was contributed by the increasing level of these parameters. Significant differences were also observed ($p<0.05$) in terms of L*, a* and b* values between the groups (fresh and dried) at each sampling day.

Bekhit *et al*., (2009) reported varying L*, a* and b* values for the roes of different fish species such as barracuda, hake and blue whiting. Among their reported results L* values of roe for barracuda

Table 2. TMA-N and TVB-N values (means ± standard deviation) of fresh and dried caviar during refrigerated storage

Periods	TMA-N (mg/100g)		TVB-N (mg/100g)	
	Fresh	Dried	Fresh	Dried
Day 1	5.6±0.4 ^{a1}	11.6±1.2 ^{a2}	12.5±0.6 ^{a1}	28.3±3.3 ^{a2}
Day 4	9.0±0.2 ^{b1}	9.9±1.1 ^{b2}	19.5±0.42 ^{b1}	29.9±1.4 ^{a2}
Day 8	17.3±0.2 ^{c1}	7.5±0.8 ^{c2}	36.4±1.2 ^{c1}	47.0±2.3 ^{b2}

Different superscript letters within each column represent significant differences ($p<0.05$) between days of storage; different numbers within each row and criteria represent significant differences between fresh and dried caviar (n=3).

Table 3. TBA values (means ± standard deviation) of fresh and dried caviar during refrigerated storage

Periods	TBA (mg malonaldehyde/kg)	
	Fresh	Dried
Day 1	0.55±0.01 ^{a1}	3.23±0.80 ^{a2}
Day 4	1.14±0.04 ^{b1}	2.58±0.03 ^{b2}
Day 8	1.24±0.03 ^{c}	2.64±0.03 ^{b2}

Different superscript letters within each column represent significant differences ($p<0.05$) between days of storage; different numbers within each row represent significant differences between fresh and dried caviar (n=3).

Table 4. Color parameters values (means ± standard deviation) of fresh and dried caviar during the storage

	L*		a*		b*	
	Fresh	Dried	Fresh	Dried	Fresh	Dried
Day 1	49.6±2.9 ^{a1}	32.3±1.1 ^{a2}	8.8±0.7 ^{a1}	6.6±0.5 ^{a2}	38.7±4.1 ^{a1}	15.0±1.2 ^{a2}
Day 4	44.9±2.4 ^{b1}	28.1±2.0 ^{ab2}	8.9±1.2 ^{a1}	9.4±0.8 ^{b2}	36.2±4.3 ^{a1}	17.6±1.5 ^{a2}
Day 8	52.5±3.7 ^{c1}	26.2±1.0 ^{b2}	7.2±0.6 ^{b1}	10.1±0.3 ^{b2}	39.3±2.7 ^{a1}	19.3±0.4 ^{b2}

Different superscript letters within each column represent significant differences ($p<0.05$) between days of storage; different numbers within each row represent significant differences between fresh and dried caviar on the same day for L*(lightness), for a*(redness) and for b*(yellowness) (n=10).

(*Thyrsites atun*), warehou (*Seriolella brama*) and hake (*Merluccius australis*), a* values of roe for barracuda were closed to our findings, while they usually observed lower b* values for all analysed samples.

Conclusion

This study represents first report on proximate composition and quality changes of fresh and dried grey mullet roe at refrigerated temperature. Despite the increasing levels of TMA for fresh roe samples during storage, a decrease occurred for dried samples. TMA might have changed to other compounds during spoilage for dry samples. Therefore, this parameter was not found suitable for monitoring spoilage for particularly dried caviar samples. TBA analysis results are negligible for during storage for both groups indicating low risk for all for monitoring oxidation process. The lightness of fresh samples significantly increased during storage while decreased for dried samples. Further studies are required to investigate the effect of drying process on TMA and color values of fish roe. Although TVB-N values seems to be a good spoilage indicator for both dried and fresh samples, studies in combination with sensory analysis are required to draw a better conclusion. Therefore, further investigation is suggested to include sensory and microbial quality changes during refrigerated storage of bottarga. Vacuum packaging technique and other preservatives can also be used to increase its shelf life in refrigerator.

References

Al-Holy, M., Wang, Y., Tang, J., Rasco, B. 2005. Dielectric properties of salmon (*Oncorhynchus keta*) and sturgeon (*Acipenser transmontanus*) caviar at radio frequency (RF) and microwave (MW) pasteurization frequencies. Journal of Food Engineering, 70: 564-570. doi:10.1016/j.jfoodeng.2004.08.046

Altug, G., Bayrak, Y. 2003. Microbiological analysis of caviar from Russia and Iran. Food Microbiology, 20: 83–86. doi:10.1016/S0740-0020(02)00090-4

AOAC. 1990. Official Methods of Analysis of the Association of Analytical Chemist, 15th edition, Washington DC, USA: 66-88.

Bekhit, A.E.A., Morton, J.D., Dawson, C.0., Sedcole, R. 2009. Optical properties of raw and processed fish roes from six commercial New Zealand species. Journal of Food Engineering, 91: 363-371. doi:10.1016/j.jfoodeng.2008.09.005

Bledsoe, G.E., Bledsoe, C.D., Rasco, B., 2003. Caviars and fish roe products. *Critical Reviews* Food Science and Nutrition. 43: 317–356. doi: 10.1080/10408690390826545

Bligh, E. G., Dyer, W. J. 1959. A rapid method of total lipid extraction and purification. Canadian Journal of Biochemistry and Physiology, 37(8), 911–917. doi: 10.1139/o59-099

Caprino, F., Moretti, V.M., Bellagamba, F., Turchini, G.M., Busetto, M.L., Giani, I., Paleari, M.A., Pazzaglia, M. 2008. Fatty acid composition and volatile compounds of caviar from farmed white sturgeon (*Acipenser transmontanus*). Analytica Chimica, 617: 139-147. doi:10.1016/j.aca.2008.02.005

CIE, 1995. Industrial colour difference evaluation, CIE Publication 116, ISBN 3 900 734 60 7 Vienna, Austria. doi: 10.1002/9780470175637

FAO, 1986. FAO Food and Nutrition paper manuals of food quality control food analysis: quality, adulteration, and tests of identity. Food and Agriculture Organization of the United Nations. Rome, Italy.

Katselis, G., Koutsikopoulos, C., Rogdakis, Y., Lachanas, T., Dimitriou, E., Vidalis, K. 2005. A model to estimate the annual production of roes (avgotaracho) of flathead mullet (*Mugil cephalus*) based on the spawning migration of species. Fisheries Research, 75: 138-148. doi:10.1016/j.fishres.2005.03.013

Klossa-Kilia, E., Papasotiropoulos, V., Kilias, G., Alahiotis, S. 2002. Authentication of Messolongi (Greece) fish roe using PCR–RFLP analysis of 16s rRNA mtDNA segment. Food Control, 13: 169–172. doi:10.1016/S0956-7135(01)00097-4

Ludorf, W., Meyer, V. 1973. Fische und Fischerzeugnisse. Paul Parey Verlag: Hamburg.

Nishimoto, S., Ohtani, B., Kajiwara, H., Kagiya, T. 1985. Correlation of the crystal structure of titanium dioxide prepared from titanium tetra-2-propoxide with the photocatalytic activity for redox reactions in aqueous propan-2-ol and silver salt solutions", Journal of the Chemical Society, Faraday Transactions 1, 81: 61-68. doi: 10.1039/F19858100061

Rogdakis, Y., 1994. Avgotaracho Messolonghiou: a seafood product with designation of origin and protection. Fishing News, 153: 90–97 (in Greek).

Rosa, A., Scano, P., Melis, M.P., Deiana, M., Atzeri, A. and Dessi, M.A. 2009. Oxidative stability of lipid components of mullet (*Mugil cephalus*) roe and its product "bottarga". Food Chemistry, 115(3): 891-896. doi:10.1016/j.foodchem.2009.01.002

Scano, P., Rosa, A., Cesare Marincola, F., Locci, E., Melis, M.P.and Dessì, M.A., Lai, A. 2008. ^{13}C NMR, GC and HPLC characterization of lipid components of the salted and dried mullet (*Mugil cephalus*) roe "bottarga", Chemistry and Physics of Lipids 151(2): 69–76. doi: 10.1016/j.chemphyslip.2007.10.001

Schormuller, J. 1968. Handbuch der Lebensmittel Chemie, Band 3/2 Teil, Trierische Lebensmittel Eier, Fleisch, Fisch, Buttermilch, Springer-Verlag: Berlin, Germany: 872-878.

Schubring, R. 2002. Influence of freezing/thawing and frozen storage on the texture and colour of brownshrimp (*Crangon crangon*). Archiv für Lebensmittelhygiene, 53: 34–36.

Tarladgis, B. G., Watts, B. M., Younathan, M. S., Dugan, L. Jr. 1960. A Distillation method for the quantitative determination of malonaldehyde in rancid foods, Journal American Oil Chemistry Society, 37(1): 44-48. doi:10.1007/BF02630824

Wolf, C., Hübner, P., Lüthy, J. 1999. Differentiation of sturgeon species by PCR-RFLP. Food Research International, 32(10): 699-705. doi:10.1016/S0963-9969(99)00150-7

Vyncke, W. 1996. Comparison of the official EC method for the determination of total volatile bases in fish with routine methods. Archiv für Lebensmittelhygiene, 47: 110-112.