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Sinnhuber R O, Yu T C & Yu Te C. Characterization of the red pigment formed in the 2-thiobarbituric acid determination of oxidative rancidity. *Food Res.* **23**:626-34, 1958.
[Dept. Food and Dairy Technology, Oregon State Coll., Corvallis, OR]

Pure crystalline 2-thiobarbituric acid (TBA) pigments prepared from rancid fish oil, sulfadiazine, and malonaldehyde were analyzed. Elemental analysis, absorption spectrophotometry, and paper chromatography all suggest that the pigments are identical and are the condensation product of one molecule of malonaldehyde with two molecules of TBA. The molecular formula of the pigment was proposed. [The *SCI*® indicates that this paper has been cited in over 160 publications since 1961.]

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"The Seafoods Laboratory at Astoria, Oregon, situated near the mouth of the Columbia River, is an experiment station of Oregon State University. Our function was to provide technical assistance and carry on research relative to the fishing industry. Our laboratory was housed in an old abandoned warehouse generously donated by the county. The furnishings and equipment, of the do-it-yourself variety, were somewhat less than adequate!

"One of the problems facing the fishing industry was rancid off-flavors or oxidation deterioration. A simple and sensitive method was needed to monitor the condition of raw and frozen fish and their storage stability. A similar problem confronted the dairy industry and Patton *et al.*[1] suggested that the 2-thiobarbituric acid (TBA) test could be used to measure oxidized flavor. A red color results which they thought was the reaction of TBA with malonaldehyde. We adopted a similar procedure for fishery products which proved to be highly sensitive.[2] We assumed that malonaldehyde found in oxidized fishery products was responsible.

"In order to confirm this assumption, our team proceeded to isolate and identify the red pigments prepared from oxidized fish oil. However, there was a major hurdle—malonaldehyde, a highly unstable compound, was unavailable as a standard and its preparation was not described. This obstacle was overcome when we noted an advertisement by Kay-Fries Chemicals for a new organic intermediate, 1,1,3-3-tetraethoxypropane (TEP), which on acid hydrolysis would yield malonaldehyde. Securing some TEP, and with addition of a few drops of acid and TBA, an intense red color formed and to our surprise even some beautiful dark purple-black crystals resulted. We were ecstatic! From this point the solution required some precision analytical work, which proved difficult in our rather primitive laboratory. The most sophisticated instrument at our disposal was a Beckman Model D spectrophotometer, and our most advanced laboratory technique was paper chromatography. We eventually succeeded in identifying the red TBA pigment and the molecular formula and structure of the pigment was proposed, which was confirmed many years later by mass spectrometry.

"The TBA method has since been adopted by many researchers for the quality evaluation of many food products and lipid oxidation of biological material.

"Our paper was the only one to identify the TBA pigment; researchers, thereafter, relied on the molecular extinction coefficient to quantify their TBA results. Also, the method could be run on the intact sample, without oil extraction. These two factors probably account for its wide acceptance. In 1976, the senior author was invited by Noboru Matsuo and the Japan Oil Chemists' Society to conduct a seminar on TBA reactions at Tokyo, Japan. A review article was subsequently published.[3]

"This paper, although highly cited, did not bring about compliments from the university. On the contrary, it is now rather amusing that the manuscript was rejected for publication by the university appointed reviewer, who was inexperienced in this subject, on the grounds that the work was inconclusive."

1. **Patton S & Kurtz G W.** 2-Thiobarbituric acid as a reagent for detecting milk fat oxidation. *J. Dairy Sci.* **34**:669-74, 1951.
2. **Yu T C & Sinnhuber R O.** 2-Thiobarbituric acid method for the measurement of rancidity in fishery products. *Food Technol.* **11**:104-8, 1957.
3. **Sinnhuber R O & Yu T C.** The 2-thiobarbituric acid reaction, an objective measure of the oxidative deterioration occurring in fats and oils. *J. Jpn. Oil Chem. Soc.* **26**:259-67, 1977.