

# Twenty-five Years of FEMA Vanilla Research

A. S. Filandro, Vice President of Virginia Dare Extract Co. and President-elect of FEMA,  
and  
R. W. Koch, Chairman of the Board of Food Materials Corporation and Past President of  
FEMA

It began on the morning of May 19, 1954, at the Hotel Biltmore in New York City. There, at a Chemists' Breakfast meeting held during the Flavor & Extract Manufacturer's Association (FEMA) annual convention, a group of flavor chemists proposed a fresh approach to the problems of pure vanilla. There has been a growing dissatisfaction with the arbitrary and non-specific character of most vanilla analytical methods and the lack of correlation between flavor quality and analytical values. The proposal, which was accepted by the FEMA Board of Governors, called for a radical change in vanilla research: the establishment of a fund to finance a program of research to be conducted by independent scientific institutions not connected with the industry.

The program would be guided by the Scientific Research Committee of FEMA. It would seek new methods for determining the purity of vanilla products, elucidate the flavor components in vanilla, and help develop standards for vanilla extract and related products. The Vanilla Bean Association soon agreed to assist the program financially and materially. In 1955, FEMA and VBA worked out a mechanism by which an assessment of four cents per pound of vanilla beans was collected to form the research fund. In later years, the Malagasy Republic, the world's largest supplier of vanilla beans, also contributed substantially to the fund.

The first main thrust of the research project was conducted by the Boyce Thompson Institute for Plant Research, Yonkers, New York. A

6-month exploratory program surveyed the existing information, studied extraction methods, and broke new ground in the then-novel field of chromatography. It is interesting to note that this research program, fairly elaborate for those days, cost the heady sum of \$6,000!

A characteristic of research is that it reveals to you how little you know, and it leads to more

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## FEMA Vanilla Research

research. The project at Boyce Thompson Institute eventually burgeoned into about five years of studies, with Burchfield and Prill as the principal researchers. Ten publications and several new methods for establishing the authenticity of vanilla extract emerged from this work. Some of the methods, such as the barium number suggested as an adjunct to the traditional lead number for quantifying pure vanilla content, did not withstand the test of time. More fruitful was the characterization of vanilla extract by paper chromatography, which eventually became the basis of a standard Association of Official Analytical Chemists (AOAC) analytical procedure for vanilla. The new test was dramatically effective in revealing adulteration in some commercial tenfold (so-called) vanilla extracts, which otherwise appeared quite normal when examined by classical analytical procedures. Authentic vanilla chromatograms gave a characteristic fluorescent pattern under ultraviolet light, and the addition of foreign botanicals could be discerned by abnormalities in the pattern.

The analysis of vanilla for amino acids by paper chromatography was also pioneered by Boyce Thompson. Twelve amino acids formed the principal pattern, and it was again demon-

strated that several so-called tenfold extracts then on the market were woefully deficient in bean content. Many of these extracts, whatever their composition, soon vanished from the scene.

The distribution of organic acids in vanilla extract was measured by gradient elution chromatography. Here again, the test's practical significance was immediately demonstrated; a commercial sample of a certain concentrated vanilla was found to contain an amount of malic acid which was completely atypical for authentic vanilla. Although the test was a useful one, the lengthy and tedious nature of the gradient elution method inhibited its full utilization.

The gas chromatography performed in the 1950s at Boyce Thompson would be considered crude by today's standards. Even so, it was established that Tahiti vanilla could be distinguished from Bourbon vanilla by means of its gas chromatographic pattern. It was also found that extracting Bourbon beans in the presence of alkali altered the GLC pattern sufficiently to distinguish it from a normal extraction.

FEMA's Scientific Research Committee, led by Dr. David Jorysch, kept in close touch with the progress of the Boyce Thompson research. Collaborative studies in the laboratories of FDA

## FEMA "Seed Money" Program

FEMA has long supported general industry research through its general funds. However, wherever a "special interest" problem has arisen, the Association has provided "seed" money, management, and technical personnel from industry ranks. FEMA policy for "seed" money has been described as follows:

"New projects, in the aggregate, may not exceed ten thousand dollars per year in costs; that they shall be renewable for one year only, subject to approval by the Board of Governors; that following such a renewal a new project would have to become self-sustaining."

Once these funds are expended and if, in the Board's judgment, the future financing should be from the specific firms rather than from general funds, then solicitations are made to members for the continuation of the project.

This policy has been applied in the past to develop self-sustaining research programs funded by members with special interests in BVO/SAIB, vanilla plant maintenance, and botanicals.

As previously indicated—considerable funds from specifically interested companies and groups have funded vanilla research for many years. It is interesting to note that while vanilla is the #1 flavor in the U.S., its flavor industry concern is not widespread enough to allow general FEMA support. The FEMA total membership has approximately 100 firms, while those firms with significant vanilla interests number only about 20.

There is certainly no question in anyone's mind that one or two companies could easily fund this current vanilla research; but the deeper and underlying question is whether general support for any and all such projects—not only vanilla—could be found when the costs involved may be many times greater.

and FEMA members were run to perfect the paper chromatographic procedures as rapid screening methods for foreign botanicals.

A second phase of the research project got underway in December 1958 when these procedures were begun to be put into forms acceptable to AOAC. Dr. Jack Fitelson, the director of Fitelson Laboratories and an AOAC associate referee, was a key coordinator in working out the collaborative studies which led to many of these methods becoming recognized as official AOAC procedures. In 1960, Dr. William Stahl suggested improving the single-dimensional paper chromatographic screening method by making it a two dimensional chromatogram. The two-dimensional chromatogram helped detect some foreign botanicals which were not readily visible in the one dimensional chromatogram. The modification became an official AOAC method in 1962.

For the third major phase of its vanilla research, FEMA began considering the investigation of the agronomy of vanilla. It was realized that a basic problem was the feast-or-famine supply variability inherent in the crop cycles with resultant undesirable fluctuations in the quality, price, and availability of pure vanilla.

Dr. Thomas Theis of the USDA Experimental Station in Puerto Rico was contacted. He proposed projects aimed at increasing the basic knowledge of vanilla agriculture, hybridizing

varieties of vanilla for greater vigor and root rot resistance, and the possible mechanization of culture to eliminate the laborious hand pollination of the vanilla orchid. A contract was signed with the USDA Experimental Station July 1, 1959, and a Vanilla Agronomy Committee headed by Dr. A. S. Wendt was formed to direct the project. The program included several ob-

jectives: collection and testing of new varieties for resistance to root rot, multiplication of the desirable clones, the development of improved hybrids, and testing fruit-setting plant growth hormones to replace hand pollination.

Given the long time required for the vanilla plant to mature, the project was necessarily a lengthy one. Much useful information on vanilla cultivation was gathered, but the basic problem, root rot disease, remained unsolved at the conclusion of the program in 1971. Without an answer to this key question, there appeared to be little prospect that vanilla would ever become a viable economic crop in Puerto Rico, and the USDA and FEMA agreed to terminate the project. In 1974, the remaining collection of vanilla hybrids and vines was transplanted to the Pacific Tropical Botanical Garden in Hawaii, where they are still available to interested botanists.

The conclusion of the Boyce Thompson work did not signal an end to innovative analytical methods by the Scientific Research Committee. A thin layer chromatographic procedure to detect the addition of foreign aromatic compounds was developed and was included as an official method in the AOAC Tenth Edition (1965). The following year, a simplified ultraviolet method for the determination of vanillin was approved as an official method. A more rapid Wichmann Lead Number method, utilizing an EDTA titration, became official in 1967. George Bowden's work on a better GLC method for determining vanilla organic acids, utilizing trisilyl derivatives, was adapted into a routine procedure. It was considerably faster than the older gradient elution method and became an official method in 1969. Richard Potter and others developed a method of detecting the addition of synthetic aromatic chemicals to vanilla extract using gas liquid chromatography. The method was extremely sensitive, detecting additions as low as a few ppm. This procedure for measuring the non-vanillin vanilla volatiles was approved as an official AOAC procedure, the Direct Gas

Chromatographic Method, in 1971. Thus, most of the characterizing tests for vanilla extract appearing in the current AOAC Book of Methods are either substantially revised or did not exist at all twenty years ago.

The work of the FEMA Technical Committee (as the Scientific Research Committee is now called) has been successful in leading to a federal standard of identity for vanilla and in eliminating much of the adulteration with foreign botanicals and aromatics which plagued the vanilla industry 25 years ago. But the task is not complete. Current activity centers on the collection of analytical data on the vanilla crops of today which have been found to have somewhat different analytical constants than the crops of 15 or 20 years ago, and on a new method for differentiating natural vanillin from synthetic vanillin by means of carbon isotope ratios according to Bricout's method, initially examined by the Committee in 1975. Another of the recent projects has measured the inorganic constituents of vanilla, establishing that the metallic constituents of vanilla extract do not have any public health significance in the quantities present. Another project has measured the range of vanillin:potassium ratios found in different varieties of vanilla as an aid in characterization, building upon the work done in this area by the BATF's Glenn Martin.

The inherent composition of a natural product such as vanilla may change over the years, and the growing sophistication of would-be adulterators may lead to novel attempts at "stretching" vanilla. The methods of analysis must therefore be periodically updated to reflect current conditions and to maintain their effectiveness.

A survey of the FEMA membership is now in progress soliciting support for the vanilla research program for coming years. Substantial support has been received, but response from the rest of the FEMA membership is required in order to gauge the extent of the current interest in this program.