- (3) Bowker, A. H., Goode, H. P., "Sampling Inspection by Variables," Mc-Graw-Hill, New York, 1952.
- (4) Byer, A. J., Abrams, Dorothy, Food Technol. 7, 185-7 (1953).
- (5) Cochran, W. G., Cox, G. M., "Experimental Designs," Wiley, New York, 1957.
- (6) Dawson, E. H., Harris, B. H., U. S. Dept. Agr. Bull. 34, 108-12 (1951).
- (7) Duncan, D. B., Biometrics 11, 1-42 (1955).

- (8) Eisenhart, Churchill, *Ibid.*, **3**, 1–21 (1947).
- (9) Food Technol. 13, 733-6 (1959).
- (10) Hogue, D. V., Briant, A. M., Food Research 22, 351-7 (1957).
- (11) Kramer, A., Food Technol. 14, 576– 81 (1960).
- (12) Kramer, A., Ditman, L. P., *Ibid.*, **10**, 155–9 (1956).
- (13) Murphy, E. F., Covell, M. R., Dinsmore, J. S., Jr., Food Research 22, 423-9 (1957).
- (14) Peryan, D. R., Ind. Quality Control 6, 11 (1950).

- (15) Tompkins, M. D., Pratt, G. B., Food Technol. 13, 149-52 (1959).
- (16) Tukey, J. W., Proc. Am. Soc. Quality Control 5, 189-97 (1951).
- (17) Wiley, R. C., Briant, A. M., Fagerson, I. S., Sabry, J. H., Murphy, E. F., *Food Research* 22, 192-205 (1957).

Received for review August 18, 1960. Accepted December 12, 1960. Scientific article A845, Contribution 3136, Maryland Agricultural Experiment Station, Department of Horticulture.

PESTICIDES AND FOOD FLAVO

Influence of Herbicides on Flavor of Processed Fruits and Vegetables

F. J. McARDLE and A. N. MARETZKI

Pennsylvania Agricultural Experiment Station, University Park, Pa.

R. C. WILEY and M. G. MODREY

Maryland Agricultural Experiment Station, College Park, Md.

As a part of regional research on flavor effects of pesticides, 28 herbicides were applied to major processing crops. Manufacturers' suggested rates were used with all chemicals, and some were applied in excess of the suggested rate to increase effectiveness of weed control. Flavor evaluation of processed products by experienced taste panels indicated that 11 herbicides reduced product flavor scores; two produced slight off-flavors when applied at their suggested rates; three produced slight off-flavors when applied in excess; 17 of the chemicals studied did not reduce flavor scores of any products treated. The flavor changes observed were of low magnitude and might not have been detected by a consumer panel.

 \mathbf{F} LAVOR CHANGES in processed fruits and vegetables caused by the use of pesticides (insecticides, fungicides, and herbicides) on growing crops have been noted frequently during the past decade (1-3). These may result from application of the pesticide to the crop during its growing season or from an accumulation in the soil of pesticide residues from past seasons.

A study of the influence of pesticides on the flavor of fresh and processed fruits and vegetables was initiated on a regional basis in 1954 at the agricultural experiment stations in the northeast region. The effects of herbicides on the flavor of processed fruits and vegetables were studied cooperatively at The Pennsylvania State University and The University of Maryland.

Procedure

Herbicides were applied to major processing crops grown on horticultural farms at the Pennsylvania and Maryland stations. Manufacturers' suggested rates and methods of application were followed when this information was furnished for the chemical. Rates for

Table I.	Taste Panel Flavor Evaluation of Herbicide-Treated Crops	
	Elayor Compared to Standar	4

Herbicide	Food Product	Applica- tion Rote, Lb./Acre	Crop Years	Better	Equal Number	Poorer of Tests	Slight off- flavor
ACP 103	Corn, canned	1.5	2		3	1	
	Total				- 3	1	
ACP M 118	Lima beans, canned	3.0	1	1	3		
	,	4.5	2		6		
	Total			1	-9		
ACP M 119	Lima beans, canned	2.0	1	1	1		
		3.0	2	_1	_5		
	Total			2	6		
ACP M 622	Tomatoes, canned	10.5	1				
	Total				2		
Atrazine	Corn, canned	2.0	1		4		
D 402 A	Total		1		4	1	
Benzac 103 A	Corn, canned	1.5	1		1	1	
<u>.</u>	Total	1.0	1	1	1	1	
Uniorazine	Lima Deans, canned	4.0	2	1	Ę	2	
	Corn. canned	12.0	1	-	1	1	
	Total			2	7	3	
CIPC (Chloro-	Beets, canned	3.0	2	_	4	-	
IPC)	Carrots, canned	6.0	1	1	1		
	Lima beans, canned	4.0	1	1	1		
	Cuturel Guran	6.0	2	1	5		
	Spinach, frozen	5.0	1		2		
	Tomatoes, canned	6.5	1		2		
	Total		_	3	17		

	Table	I. (contin	ved)	Flavor	Compare	ed to Sta	ndard
		Applica- tion	_	Better	Eaual	Poorer	Slight off- flavor
Herbicide	Food Product	Rate, 1b./Acre	Crop Years		Numbe	of Tests	
Crag-Sesone	Strawberries, frozen	1.5 3.0 6.0	2 2	1	2 1 2		
Dalapon	Total Potatoes, stored, baked	6.0	1	1	5		
Diuron	Total Corn, canned	1.5	2		$\frac{1}{3}$		
Emid	Corn, canned	1.5	2	1	2	1	
Endothal	Beets, canned	6.0 9.0	1 3	1	2 3 10	1	
EPTC	l otal Beets, canned Strawberries, frozen	6.0 1.5 3.0 6.0	2 2 2 2	1 1 1 1	$ \begin{array}{c} 13 \\ 3 \\ 1 \\ 1 \\ 2 \\ \end{array} $		
FW-450	Total Beets, canned Total	10.0	2	3	$\frac{7}{3}$	$\frac{1}{1}$	
Monuron (Karmex-W) (Telvar)	Beets, canned Corn, canned Spinach, frozen	0.75 0.3 1.5 3.5	1 1 2 2	1	2 2 3 4	1	
Natrin	Total Tomatoes, canned	9.0	2	1	11 _4		
Neburon (Kloben)	Lima beans, canned	3.0 4.0 6.0	2 1 2	1	4 2 1 4	1 1	1 1
	Tomatoes, canned	4.5 7.0	2		4	2	1
	Strawberries, frozen	1.0 2.0 4.0	2 2 2		2 1 2	1	
Niagara (5521)	Total Lima beans, canned	6.0 9.0	1 2	1	19 1 4	5 1 1	3
Premerge (Dinitro) (DNBP)	Total Corn, canned Lima beans, canned	3.0 4.0 6.0 13.3	3 1 2 1	1 2	5 3 2 8 4	2	1
	Tomatoes, canned	10.0 11.5	1 1		2 2		
Randox (CDAA)	Total Beets, canned Tomatoes, canned	9.0 8.0 9.0	2 1 2	3	21 3 2	1 1 1	_1
Salt	Beets, canned	200 400	1 2	1	8 1 4	3	1
Simazine	Corn, canned	2.0 3.0	1 2	1	5 2 3	1	
Stoddard Solvent	Tomatoes, canned Total Carrots, canned	3.0 100 gal./acre	2 1		$ \frac{6}{11} $	$\frac{2}{3}$	
2,4-D Amine	Total Corn, canned	0.75 2.0	2 1		2 3 3	1	1
Trietazine	Total Corn, canned	4.0	1		-6 2 2	_1	1
	Lima beans, canned	4.0 8.0	1 2	1	1 5		
URAB	Total Corn, canned Total	2.25	1	2	$\frac{10}{2}$		
Vegadex (CDEC)	Beets, canned Corn, canned	6.0 6.0 9.0	3 1 2	1	6 2 3		
	Tomatoes, canned Total	9.0	2		$\frac{4}{19}$		

other chemicals were based on earlier published reports. Application rates in excess of that suggested or previously reported for a chemical were used on some crops in an attempt to obtain better weed control. When a single application rate was used for a chemical on one crop, it was the rate suggested by the manufacturer or previously reported for use on that crop. When two rates were used for a chemical on one crop, the suggested rate and an application in excess of the suggested rate were used. When three application rates were used for a chemical on one crop, they were $1/2 \times$, $1 \times$, and $2 \times$ the suggested rate. The products were harvested and processed by commercial methods at the station pilot plants. Products from untreated check plots were processed and used as standards for flavor comparison. After an 8-week storage period the processed foods were presented to experienced taste panels at each of the stations.

The taste panel methodology employed was that designed for the regional study of the effects of pesticides on the flavor of fruits and vegetables (5). Samples were presented to the judges in sets of six, including an unknown standard sample which had received no herbicide treatment. The judges were asked to compare the flavor of these six samples to the standard, which was a seventh and known sample, and to place each in one of five categories on the NE-15 score sheet (4). Mean scores for each sample were calculated from values previously assigned to the categories. A minimum of 40 individual judgments constituted a panel test. These individual judgments were averaged to obtain the mean score, which became the sample score. The mean score of the untreated standard sample was also thus obtained, since it was presented as an unknown. Treatment and standard means scores were compared statistically for differences.

Results and Discussion

A summary of the flavor evaluation of herbicide-treated crops is given in Table I. Results are the combined data from Pennsylvania and Maryland panels, except for the frozen strawberries from Neburon, EPTC, and Crag-Gesone treated plots, which were analyzed only by the Pennsylvania panel. The data are presented as the number of panel test results which terminated in the various flavor categories when the treated samples were compared to the standard sample. The score sheet category headed "definite off-flavor, not acceptable" was eliminated from the table, since no sample means scores were sufficiently low to place them in this category.

On the basis of data summarized in

Table I the herbicides studied may be divided into two groups.

Group I. Herbicides which produced tests rated poorer than the standard sample. This group included ACP 103, Benzac 103A, Chlorazine, Diuron, Emid, FW 450, Neburon, Niagara 5521, Randox, Simazine, and 2,4-D Amine.

Group II. Herbicides for which no tests scored lower than the standard sample. Included in this group were ACPM 118, ACPM 119, ACPM 622, Atrazine, Chloro-IPC, Crag-Sesone, Dalapon, Endothal, EPTC, Monuron, Natrin, Premerge, salt, Stoddard Solvent, Trietazine, URAB, and Vegedex.

Of the six slight off-flavor tests encountered, two occurred in sweet corn which had been treated with chemicals (Neburon and 2.4-D Amine) at their suggested rate of application. The remaining four slight off-flavor tests occurred in products which had been treated with chemicals (Neburon, Niagara, and Randox) applied at concentrations higher than their suggested rates. However, these chemicals also yielded products with flavor mean scores lower than the standard sample when treatments were at the suggested rate of application.

Treatments in excess of the suggested levels were not included for six of the chemicals listed in Group II. These six herbicides (ACPM 622, Atrazine, Dalapon, Natrin, Stoddard Solvent, and URAB) were somewhat favored by this bias.

The extent to which these herbicide flavor changes would influence consumer acceptance of the processed products cannot be determined from these data. Since experienced taste panels were employed for these analyses, the judges were undoubtedly more sensitive to flavor changes than most consumers would be. Thus, some of the flavor effects observed might not be detected by a consumer panel. Although none of the sample mean scores was sufficiently low to be considered unacceptable in flavor, any flavor impairment of processed foods is undesirable and should be controlled as closely as possible.

Acknowledgment

The field plot layouts and herbicide

applications were performed by C. J. Noll of the Pennsylvania Agricultural Experiment Station, and by L. P. Ditman and A. A. Duncan of the Maryland Agricultural Experiment Station.

Literature Cited

- (1) Dawson, E. H., Gilpin, G. L., Kirk-patrick, M. E., Weigel, C. A., J. Agr. Food Снем. **1,** 399 (1953)

- (2) Gilpin, G. L., Parks, A. B., Reynolds, Howard, *Ibid.*, 5, 44 (1957).
 (3) Greenwood, M. L., Tice, J. M., J. Agr. Research 78, 477 (1949).
 (4) Murphy, E. F., et al., J. AGR. FOOD CHEM. 7, 214 (1961).
 (5) Wiley, P. C. Parent, A. M. F.
- (5) Wiley, R. C., Bryant, A. M., Fager-son, I. S., Sabry, J. H., Murphy, E. F., Food Research 22, 192 (1957).

Received for review August 18, 1960. Accepted December 12, 1960. Part of a Northeast Regional Project (NE-15), a cooperative study involving agricultural experiment stations in the northeast region and supported in part by funds of the U.S. Department of Agriculture. Journal paper 2480, Pennsylvania Agricultural Experiment Station.

ESSENTIAL OILS Treatment of Compositional Data for the Characterization of Essential Oils. Determination of Geographical Origins of Peppermint Oils by Gas Chromatographic Analysis

D. MORISON SMITH and LEO LEVI Food and Drug Laboratories, Department of National Health and Welfare, Ottawa, Canada

Mentha piperita and M. arvensis oils have been analyzed by gas-liquid partition chromatography. Compositional criteria thus established are utilized for determination of geographical origins, recognition of biochemical relationships governing formation of oil in the plant, evaluation of manufacturing processes, and detection of subtle adulterations. Data and their treatment should prove of value not only to processors of essential oils, but also in characterization, analysis, and quality control of complex natural and synthetic compositions produced by the food, drug, and cosmetic industries.

IL OF PEPPERMINT is one of the few essential oils whose production and processing have assumed great commercial importance in many countries. Of all flavors competing for man's taste that of peppermint has long proved and remains one of the most popular. From less than 2000 pounds to well over 2 million pounds per year, such is the remarkable development of the peppermint oil industry in the United States since the early 19th century. Production has more than doubled during the last two decades (3).

To the food industry, oil of peppermint is an essential raw material for flavoring a wide range of consumer products, particularly baked goods, confectioneries, and alcoholic liqueurs. Large quantities are also used by the pharmaceutical industry to mask objectionable tastes or modify the nauseating, griping effects of many medicinals.

Canadian Food and Drug Regulations permit use of both Mentha piperita and Mentha arvensis oils of the required flavoring strength for these purposes (13) and are in this respect considerably

broader in scope than those applying in the United States, where a ruling of the U. S. Department of Agriculture forbids mint oil to be designated as peppermint oil, and the U.S. Federal Food and Drugs Act requires that preparations containing M. arvensis oil must be labeled "flavored with corn mint" or "flavored with field mint" (20).

This situation requires some explanatory comments regarding the botanical classification of peppermint. According to Guenther (18), the plants used in