

RESEARCH NOTES

AN EXTRACTION PROCEDURE FOR MALATHION IN FRUIT PASTES

A request to evaluate Malathion residues in a banana paste manufactured locally was received from the Department of Agriculture of the Commonwealth of Puerto Rico. The reason for checking for Malathion was that the company placed a cheese cloth impregnated with Malathion above the drying bananas to kill fruit flies. Extraction by carbon tetrachloride of the banana paste following the standard procedure¹ quickly proved futile probably because the paste has large quantities of sugar which is not soluble in non-polar solvents such as carbon tetrachloride.

Consequently, several polar solvents, methanol, isopropyl alcohol, acetone, acetonitrile and water, were tried in combination with carbon tetrachloride for extraction, and then the polar solvent was washed out with water leaving the carbon tetrachloride with the Malathion in it. A procedure using methanol was found most successful as the other four polar solvents formed intractable emulsions.

The procedure was as follows: Paste (200 g.) in small pieces was blended for 1 minute with methanol (ACS grade, 200 ml.). Carbon tetrachloride (ACS grade, 200 ml.) was added, and the slurry was blended for 2 minutes more. The slurry was filtered through a mat of glass wool using a beaker to force sufficient liquid through the mat and into a separatory funnel (1000 ml.). Distilled water (ca. 400 ml.) and saturated sodium chloride solution (10 ml.) were added to the separatory funnel which then was shaken for 1 minute. The layers in the funnel were allowed to separate, and the lower carbon tetrachloride layer was drained into another separatory funnel (500 ml.). Distilled water (ca. 150 ml.) and saturated sodium chloride solution (2-3 ml.) were added to the second separatory funnel, and the funnel was shaken for 1 minute. The layers in the funnel were allowed to separate, and the lower layer was drained through a layer of anhydrous sodium sulfate into a graduated cylinder until sufficient extract was obtained (usually 100 ml.). At this point the Malathion is in carbon tetrachloride, and the usual method of analysis² can be initiated.

With this procedure, banana, guava and sweet potato pastes were analyzed for recovery of added Malathion. Grapefruit paste was found amenable to the usual carbon tetrachloride extraction³, and recovery studies also were made on it. Table 1 indicates the amount of Malathion

¹ Sutherland, G. L., *Analytical Methods for Pesticides Plant Growth Regulators and Food Additives*, Vol. II, Academic Press, New York, p. 283, 1964.

² *Ibid.*, p. 290, d, iii.

³ *Ibid.*

added to paste sample, the amount recovered using the above extraction procedure followed by the usual method of analysis, and the percent of the malathion recovered.

From table 1 it can be seen that percent recoveries were excellent at the higher levels and adequate at the 1 p.p.m. level. Possibly the lower recovery at the lower level could be overcome by a somewhat longer hydrolysis period than the 1 minute of the usual procedure. During preparation of a

TABLE 1.—*Recovery of Malathion from fortified fruit pastes*

Paste	Item	Amount of Malathion added in parts per million			
		0	1	4	10
Banana	P.p.m. recovered	0.0	0.58	4.4	9.9
	Percent recovery	—	58	110	99
Guava	P.p.m. recovered	0.0	.67	3.6	9.7
	Percent recovery	—	67	90	97
Sweet potato	P.p.m. recovered	0.0	.67	4.1	9.3
	Percent recovery	—	67	103	93
Grapefruit	P.p.m. recovered	0.0	.80	3.5	9.4
	Percent recovery	—	80	88	94

standard curve, it was noticed that 1 minute and 15 or 20 seconds of shaking for the hydrolysis raised the 1 p.p.m. value by almost 20 percent over the 1-minute period while having little effect on the higher concentrations.

The recoveries on the banana paste exposed to the Malathion showed essentially no Malathion in excess of the added Malathion indicating that Malathion from the cheese cloth had not contaminated the bananas used in these samples of paste. The excellent recoveries in the other pastes indicate that the method of extraction is generally useful for fruit pastes.

James A. Singmaster III
Central Analytical Laboratory