

Maximum Recovery of Pesticides from Alfalfa and Related Material

by GEORGE A. ROOT
*Division of Chemistry
California State Department of Agriculture
Sacramento, California*

This laboratory is required to make many analyses on alfalfa treated with pesticides in the field at unknown concentrations, the time between application and analysis also unknown in most cases. The following work was done to find the method that consistently extracted the greatest amount of pesticide from the hay in a reasonable time. We have been mainly concerned with DDT and parathion, but some work was done on other commonly used pesticides. The samples used were random ones brought into the laboratory.

Experimental

The following methods of extraction were tried:

1. Sample tumbled 15 minutes with petroleum ether.
2. Sample tumbled 15 minutes with benzene.
3. Sample tumbled 15 minutes with acetonitrile.
4. Sample tumbled 1 hour with acetonitrile.
5. Sample Omni-mixed for 5 minutes with 2:1 benzene-isopropanol.
6. Sample Omni-mixed for 2 minutes with acetonitrile.

7. Sample Omni-mixed for 5 minutes with acetonitrile.
8. Sample extracted in Soxhlet apparatus for 18 hours with ethyl ether.
9. Sample refluxed for 1 hour in benzene.
10. Sample refluxed for 1 hour in acetonitrile.

All of these except refluxing have been reported in the literature(1). The solvent ratio to sample was in all cases 10 to 1. After extraction, all samples were filtered and cleaned up by the method of Mills et al(2). They were then injected into an electron-capture detector gas chromatograph using a mixed Dow 200-QF-1 column.

The following table shows the recoveries of pesticides on various samples extracted as described. The numbers in the table under extraction method correspond to those in the text. Cored samples are pieces of alfalfa not more than 1 1/2 inches long. Ground samples had all been ground in a Wiley mill to less than 20 mesh.

TABLE

<u>Material</u>	<u>Extraction Method</u>	<u>Condition of Sample</u>	<u>Amount of Pesticide Found in Parts Per Million</u>
			DDT
A(dry alfalfa)	1	Cored	1.2
		Ground	2.4
	2	Ground	2.8

TABLE (cont.)

<u>Material</u>	<u>Extraction Method</u>	<u>Condition of Sample</u>	<u>Amount of Pesticide Found in Parts Per Million</u>
DDT			
A(dry alfalfa)	3	Cored	1.35
		Ground	2.6
	4	Cored	1.9
		Ground	3.6
	5	Cored	1.2
	6	Cored	0.9
	7	Cored	1.2
	8	Cored	3.4
		{Ground	3.8
		{Ground	0.1 additional*
	10	Cored	3.9
DDT			
B(dry alfalfa)	3	Cored	1.1
	8	Ground	1.6
	10	Cored	3.0
Parathion			
C(dry alfalfa)	7	Cored	7.1
	9	Cored	5.6
	10	Cored	7.8

*Extracted in Soxhlet for additional 3 hours

TABLE (cont.)

<u>Material</u>	<u>Extraction Method</u>	<u>Condition of Sample</u>	<u>Amount of Pesticide Found in Parts Per Million</u>		
			Parathion		
D(dry alfalfa)	9	Cored	14.0		
		Ground	18.5		
	10	Cored	17.4		
		Ground	20.0		
			Methyl Parathion	Parathion	
E(dry alfalfa)	4	Ground	1.3	8.25	
	7	Ground	1.3	10.0	
	8	Ground	1.35	7.0	
	9	Ground	0.8	4.4	
	10	Ground	1.35	11.2	
			Kelthane	Toxaphene	
F(alfalfa meal)	3	Ground	0.7	5.6	
	6	Ground	0.6	4.2	
	8	Ground	1.3	6.0	
	10	Ground	1.5	6.8	
			Kelthane	Tedion	DDT
G(almond hulls)	3	Ground	2.3	4.5	0.9
	6	Ground	2.2	3.5	0.8
	8	Ground	3.5	4.2	0.9
	10	Ground	3.6	4.9	1.0

TABLE (cont.)

<u>Material</u>	<u>Extraction Method</u>	<u>Condition of Sample</u>	<u>Amount of Pesticide Found in Parts Per Million</u>
			Toxaphene
H(seed screenings)	4	Ground	7.5
	7	Ground	10.
	8	Ground	13.
	9	Ground	15.
	10	Ground	15.

Discussion

It is apparent in all cases that maximum extraction is obtained when the sample is ground and refluxed in acetonitrile. Other methods of extraction reach or approach this level in some samples but never exceed it. Soxhlet extraction is consistently near the maximum found by refluxing while tumbling and Omni-mixing give erratic recoveries.

Conclusion

We believe that grinding to less than 20 mesh and then refluxing in acetonitrile for an hour is the preferred method for extraction of pesticides in alfalfa and similar material.

References

1. W. O. Wheeler and D. E. H. Frear, Residue Reviews 16, 86 (1966)
2. P. A. Mills, J. Onley. and R. Gaither, J.A.O.A.C. 46, 186 (1963)