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Spray Residue Work in Missouri

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SUMMARY

Water alone in the wash section of an underbrush flood-type washer reduced the lead content of the spray residue by as much as 50% when the original load did not exceed .050 grain of lead per pound of fruit.

Kolofog and also certain oils greatly increase the difficulty of removing spray residue. The use of lime in sprays greatly increases the ease of removal of both lead and arsenic even where Kolofog or oils have been used.

When neither Kolofog nor oil had been used with arsenate of lead, 1.2% hydrochloric acid in the underbrush flood-type washer reduced the lead content from .080 to below tolerance limits at about 60°F.

Where oil had been used with each cover spray, 1.2% hydrochloric acid plus 4 pounds of dry Vatsol to 100 gallons did not reduce the lead content from .100 to within tolerance limits at 65°F and 30 seconds exposure. To successfully lower the lead and arsenic content of the residue on such fruit it was necessary to heat the wash solution to 100-110°F. Where Kolofog and oil both had been used, it was necessary to increase the time of contact as well as heat the solution containing Vatsol.

Home-made flotation washers are effective in satisfactorily reducing light to medium residues, especially where oil or Kolofog has not been used.

Adequate provision should be made for rinsing the fruit with fresh water after it leaves the wash solution in a flotation washer.

It was possible to reduce the arsenic content of the residue to, or near, zero and yet leave an appreciable residue of lead.

Zinc is definitely an interfering substance in the determination of lead by the diphenylthiocarbazone (dithizone) method.

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In 1903 the British Royal Commission on Arsenical Poisoning recommended that the maximum amount of arsenic (calculated as arsenic trioxide), which food products could carry should be less than 1/100 of a grain of arsenic trioxide per pound of solid, or per gallon of liquid, food. In enforcing the British Food and Drugs regulations, the British authorities and courts have generally been guided by the above recommendation of the Royal Commission.

Great Britain, in 1925, declared an embargo on fruit carrying over .01 grain of arsenic trioxide per pound of fruit. This resulted in a temporary embargo on such fruit for interstate commerce in the United States. The final outcome was the establishment of a tolerance limit for arsenic in food. For the year 1926 it was thought that the tolerance limit of .01 grain per pound of fruit could be met by subjecting fruit to the simple procedure of wiping. Wiping, however, did not always prove effective in lowering the arsenic calculated as arsenic trioxide in the spray residue to below .01 grain per pound of fruit. Accordingly the United States Department of Agriculture in 1927 set the tolerance limit for arsenic trioxide at .025 grain per pound of fruit. The limit was then lowered annually as follows:

Year	*Grain of Arsenic Trioxide Per Pound of Fruit*
1928	.020
1929	.017
1930	.015
1931	.012
1932	.010

Since 1932 the limit of .01 grain of arsenic trioxide per pound of fruit has remained the same and will continue in force for 1937. This limit is now known as the world tolerance limit.

A tolerance limit for lead was first established in 1933. The limit that year was placed at .020 grain of lead (calculated as metallic lead) per pound of fruit. In 1934 the limit was lowered to .019. Since 1935 the limit has remained the same; namely, .018 grain per pound of fruit.

As long as there was a tolerance limit for arsenic only, and that limit was relatively high, fruit growers in Missouri experienced little difficulty in meeting the requirements. By 1930, however, when the tolerance limit became .015 grain of arsenic trioxide per pound of fruit, most growers found it necssary to resort to some method of cleaning the fruit. Some were able to reduce the residue below tolerance limits by wiping or brushing the fruit; but the majority found it necessary to employ the more effective dilute hydrochloric acid wash method.

FLOTATION WASHER

A flotation washer was constructed by the Department of Horticulture in 1930. This was constructed according to plans outlined in a U. S. Department of Agriculture pamphlet entitled "Removal of Spray Residue from Apples and Pears" by H. C. Diehl, W. F. Pentzer, A. L. Ryall and B. D. Ezell. The dimensions as specified were adhered to, hence the capacity of the washer was about 60 bushels per hour at a speed which required 1½ minutes for fruit to pass through the washer. This machine was used to clean fruit successfully in 1931, 1932, 1933 and 1934.

With the flotation washer slowed until it required two minutes for the fruit to pass through, the residues of lead and arsenic were brought within the tolerance limits when the original load was as high as .100 grain of lead per pound of fruit. A wash solution of one per cent hydrochloric acid was used. In the flotation washer time of contact with the wash solution was so great that injury to the fruit was likely to occur, since the fruit was not so thoroughly turned and rinsed. A contact agent was easily used in the flotation washer and no antifoam was necessary. A contact agent, however, wasn't necessary in order to reduce residues below tolerance limits on fruit grown here at Columbia.

The flotation washer functioned quite satisfactorily except for the warping of some of the elevator slats. It was often necessary to trim and adjust the warped slats. Teeth of sprocket wheels were invariably too large. These were ground down until the chains ran smoothly.

Such a flotation washer when well cared for should be good for at least four or five sasons. Care should be exercised in seeing that the elevator chains are lifted free of the wash tank and rinse tank at the end of each run. At the end of the washing season the tanks should be drained and thoroughly rinsed. Washers should be properly housed at all times, but espcially so during the winter.

FLOOD TYPE WASHER

During the season of 1935 the newest type of underbrush flood washer was employed. In passing through this machine fruit was in contact with the wash solution 30 seconds. When a contact or wetting agent was used, a defoaming agent was also necessary. Passing fruit through this machine when only plain water was in the washing section resulted in a marked improvement in appearance of the fruit. Water alone, in the underbrush flood type washer, at a temperature of

78°F, reduced the lead content of the spray residue by as much as 50% when the original load did not exceed .050 grain of lead per pound of fruit. The arsenic trioxide content of the spray residue was reduced in about the same proportion with the time of contact 30 seconds.

When neither oil nor Kolofog had been used with arsenate of lead, 1.2% hydrochloric acid in the underbrush flood type washer reduced the lead content from .080 to below tolerance limits at about 60°F. Where the lead content of the residue was .100 or more, the above hydrochloric acid wash did not reduce the lead to within tolerance limits. Where oil had been usd with each cover spray, 1.2% hydrochloric acid plus 4 pounds of dry Vatsol to 100 gallons did not reduce the lead content from .100 to within tolerance limits at 65°F and 30 seconds exposure. To successfully lower the lead and arsenic content of the residue on such fruit, it was necessary to heat the wash solution to 100-110°F. Where Kolofog and oil both had been used, it was necessary to increase the time of contact as well as heat the solution containing Vatsol. The use of lime with Kolofog greatly reduced the difficulty of cleaning.

SPRAY RESIDUE DETERMINATIONS

Cooperative work in cleaning fruit and determining spray residue with the fruit growers of the State by the Department of Horticulture was initiated in 1931. Cooperating with the State Food and Drug Department in 1931, the Department of Horticulture received 136 samples of fruit for arsenic trioxide determinations in the spray residue. Only 13 of these samples or about 9.6 per cent contained arsenic trioxide in excess of the tolerance limits before cleaning. The highest arsenic trioxide content of the residue on any sample was .020. A few of the samples were cleaned by commercial brush machines. The results are given in Table 1.

Method of Cleaning	Arsenic Trioxide Content of Spray Residue before Cleaning	Arsenic Trioxide Content of Spray Residue after Cleaning
Brushed	.011 grain per pound of fruit	.008 grain
Brushed	.006 grain per pound of fruit	.005 grain
Brushed	.012 grain per pound of fruit	.009 grain
Brushed	.011 grain per pound of fruit	.007 grain

Jonathan constituted the greater proportion of the samples recived for spray residue determinations in 1931. The crop was light in 1932 and no samples were received from growers. Again in 1933 the crop was sufficiently heavy to obtain samples from the various fruit districts of the state. The crop was again light in 1934, but in 1935 a still greater number of samples was obtained. Table 2 gives the data obtained.

Table 2.—Data Obtained in Cleaning Fruit.

Year	Number Cover Sprays	Variety	Method of Cleaning	Spray Residue Before Cleaning		Spray Residue After Cleaning		Per cent of Residue Removed	
				Lead	As_2O_3	Lead	As_2O_3	Lead	As_2O_3
1933	3	Jonathan	Washed	.010	.009	.005	.003	50	66
1933	3	Jonathan	Washed	.012	.007	.010	.004	16	43
1933	3	Duchess	Flotation washer .9% HCl, 4 min.	.101	.039	.008	.007	92	82
1933	3	Duchess	Flotation washer .9% HCl, 4 min.	.097	.057	.002	.005	98	91
1933	3	Pumpkin Sweet	Flotation washer .9% HCl, 4 min.	.074	.032	.004	.004	94	87
1933	4	Duchess	Flotation washer .8% HCl, 3 min.	.049	.027	.004	.003	92	89
1933	4	Pumpkin Sweet	Flotation washer .8% HCl, 1¾ min.	.021	.054	.002	.002	90	96
1933	4	Wolf River	Flotation washer .8% HCl, 1¾ min.	.038	.025	.011	.004	71	84
1933	5	Jonathan	Washed	.050	.013	.005	.003	90	77
1933	5	Winter Banana	Flotation washer .8% HCl, 1¾ min.	.038	.032	.003	.006	92	81
1933	5	Winter Banana	Flotation washer .8% HCl, 1¾ min.	.027	.014	.004	.003	85	78
1933	5	Jonathan	Washed, 38 lbs. R.M.S. to 300 gal. water	.010	.006	.008	.004	20	33
1933	5	Jonathan	Brushed	.010	.010	.005	.005	50	50
1933	5	Jonathan	Brushed	.035	.011	.025	.008	28	27
1933	5	Jonathan	Brushed	.075	.069	.050	.062	33	10
1933	6	Jonathan	Brushed	.070	.050	.037	.039	47	22
1933	6	Jonathan	Brushed	.060	.048	.020	.035	66	27
1933	6	Jonathan	Brushed	.050	.049	.040	.042	20	14
1933	6	Jonathan	Brushed	.040	.061	.035	.040	12	34
1933	6 + oil	Jonathan	Brushed	.060	.050	.020	.035	66	30
1933	6 + oil	Jonathan	Brushed	.075	.069	.050	.062	33	10
1933	6 + oil	Jonathan	Brushed	.070	.048	.037	.039	47	19
1933	6 + oil	Jonathan	1% HCl, flood washer, 30 sec.	.170	----	.035	----	79	--
1933	6	Jonathan	2 gal. HCl to 100 gal. water	.050	.060	.015	.018	70	70
1933	6	Jonathan	2 gal. HCl to 100 gal. water + Vatsol + Cocoanut oil	.100	.072	.030	.025	70	65
1933	6	Jonathan	2 gal. HCl to 100 gal. water	.070	.040	.020	.026	71	35
1933	6	Maiden Blush	Washed in 1% HCl	.060	----	.015	----	75	--
1933	6	Maiden Blush	Washed in 1% HCl, towel dried	.060	----	.018	----	70	--
1933	6	Maiden Blush	Washed in 1% HCl, towel dried	.060	----	.018	----	70	--
1933	6	Maiden Blush	Washed in water, towel dried	.060	----	.042	----	30	--
1933	6	Maiden Blush	Washed in water, wiped	.060	----	.040	----	33	--
1935	2	Payne's Late Keeper	Flood washer 1.2% HCl at 59° F.	.012	.004	.004	.000	66	100
1935	3 + lime	Jonathan	Flood washer .8% HCl at 60° F.	.044	.018	.012	.005	73	72
1935	4	Jonathan	Flood washer .5% HCl in cistern water	.025	.021	.018	.006	28	71
1935	4	Winesap	Flood washer 1.2% HCl + Vatsol + defoamer	.072	.031	.012	.006	83	81

TABLE 2. (Continued)

Year	Number Cover Sprays	Variety	Method of Cleaning	Spray Residue Before Cleaning		Spray Residue After Cleaning		Per cent of Residue Removed	
				Lead	As_2O_3	Lead	As_2O_3	Lead	As_2O_3
1935	5 + oil	Jonathan	Brushed	.062	.018	.044	.012	29	33
1935	6	Rome	Flood washer 1.2% HCl at 59° F.	.072	.020	.014	.000	80	100
1935	6	Payne's Late Keeper	Flood washer 1.2% HCl at 59° F.	.060	.019	.008	.000	87	100
1935	6	Grimes	Flood washer 1.2% HCl at 59° F.	.160	.032	.030	.013	81	59
1935	6	Golden Delicious	Flood washer 1.2% HCl at 59° F.	.120	.040	.034	.013	72	67
1935	6	Jonathan	Flood washer 1.2% HCl at 75° F.	.080	.020	.012	.003	85	85
1935	6	Maiden Blush	Flood washer, plain water, 78° F.	.015	.008	.010	.002	33	75
1935	6	King David	Flood washer, plain water, 78° F.	.050	.026	.026	.013	48	50
1935	6	McIntosh	Flood washer, plain water, 78° F.	.022	.016	.015	.008	32	50
1935	6 + lime	Red June	Flood washer, plain water, 78° F.	.050	.041	.015	.021	70	49
1935	6	Jonathan	Flood washer 1% HCl, 60° F.	.030	.009	.012	.005	60	44
1935	6	Jonathan	Flood washer 2% HCl, 70° F.	.020	.008	.012	.000	40	100
1935	6 + oil + lime	Jonathan	Flood washer 1% HCl, 60° F.	.070	.047	.004	.003	94	94
1935	6 + oil + Kolofog	Jonathan	Flood washer .86% HCl, 75° F., 30 seconds	.070	.048	.050	.018	28	62
1935	6 + oil + Kolofog	Jonathan	Flood washer 1.17% HCl, 75° F., 30 sec.	.070	.048	.052	.016	26	66
1935	6 + oil + Kolofog	Jonathan	Flood washer 1.17% HCl + NaCl at 75° F., 30 seconds	.070	.048	.051	.013	27	73
1935	6 + oil + Kolofog	Jonathan	Flood washer 1.5% HCl, 95° F., 45 sec.	.070	.048	.027	.009	61	81
1935	6 + oil + Kolofog	Jonathan	Flood washer 2% HCl, 95° F., 45 seconds	.070	.048	.018	.006	74	87
1935	6 + oil + Kolofog	Ben Davis	New Underbrush flood washer 1% HCl, 75° F., 30 seconds	.240	.060	.030	.010	87	83
1935	7 + Kolofog	Jonathan	New Underbrush flood washer 1.5% HCl, 95° F.	.240	.075	.024	.009	90	88
1935	7 + soap + kerosene	Jonathan	Flood washer 1.5% HCl + salt, 95° F., 45 seconds	.245	----	.028	.008	88	--
1935	8	Jonathan	Flood washer 1.5% HCl, 95° F.	.160	.055	.018	.006	89	89
1935	8	Jonathan	Flood washer 1% HCl, 70° F.	.176	.050	.042	.016	76	68
1935	8	Jonathan	Flood washer 1.2% HCl, 64° F.	.144	.044	.024	.012	83	73
1935	9 + oil	York	Flood washer 1.2% HCl + Vatsol + defoamer, 64° F.	.100	.032	.034	.017	66	47
1936	2	Benoni	1% HCl, 80° F.	.072	.024	.011	.009	85	62

The data in Table 3 shows, as one would expect, the increase of lead and arsenic in the spray residue with an increase in the number of cover sprays. The great capacity of certain oils to cause lead to be retained on the surface of the fruit is brought out in these tables. Kolofog seems only a little less effective in causing retention of lead. On the other hand, the use of lime with lead arsenate, even when Kolofog was also applied, is shown to generally reduce the residue of lead and arsenic and when used with calcium arsenate reduced the residue of arsenic.

The effect of lime on spray residue is well illustrated by a large, well loaded Red June tree which, in 1935, received five cover sprays containing lead arsenate (3 lbs. in 100 gallons of water). Slaked lime was added to the last two sprays at the rate of 6 pounds to 100 gallons. August 21, 1935 the spray residue on this fruit contained .041 grain of arsenic trioxide per pound of fruit and about .050 grain of lead per pound of fruit. After passing this fruit through an underbrush flood type washer with tap water as the wash solution, the residue on the washed fruit contained .021 grain of arsenic trioxide and .015 grain of lead per pound of fruit. The lead content of the residue was therefore reduced about 70 per cent by using tap water only in the wash section.

The growing season of 1935 was unlike those of 1931 and 1933 in that the temperature was not so high and the rainfall was much greater. In spite of heavy rainfall in the early and latter part of the 1935 growing season, the arsenic trioxide and lead loads were generally high. Many growers were forced to use strong hydrochloric acid solution (1.75%) at a temperature of 110°F with the addition of an agent making for better contact of the solution with the fruit in order to reduce the lead load to below tolerance limits. The time of contact was also necessarily increased, but even then difficulty was often experienced in successfully reducing the lead content of the residue.

Inspection of the data in Table 2 discloses the fact that in some cases it was possible to reduce the arsenic content of the residue to, or near zero at the same time the lead content remained appreciable. At first this was thought to be due to some error made in the course of the determinations, but repetition failed to disclose such error. It was not possible to make determinations on duplicate fruit samples, however. Lead determinations were always made in duplicate on the same fruit sample and under the above conditions repeated a number of times. Arsenic trioxide determinations were always made in quadruplicate.

The Missouri apple crop was light in 1936. In some sections the fruit was very small due to the abnormally dry season. Few samples were, therefore, obtained for washing and for spray residue determination.

TABLE 3.—DATA TABULATED ACCORDING TO THE NUMBER OF COVER SPRAYS AND THE YEAR

Year	No. of Cover Sprays	Variety	Section	Spray Residue	
				Lead	As_2O_3
1933	2	Mixed Sample	East	.002	.002
1933	3	Jonathan	East	.012	.007
1933	3	Duchess	Central	.097	.057
1933	3	Duchess	Central	.101	.039
1933	3	Pumpkin Sweet	Central	.074	.032
1933	3	Jonathan	West Central	.005	.004
1933	3	Jonathan	West Central	.005	.008
1933	3	Jonathan	West Central	.010	.009
1933	4	Grimes	Northwest	.020	.016
1933	4	Jonathan	East	.010	.006
1933	4	Jonathan	East	.018	.008
1933	4	Jonathan	West Central	.010	.013
1933	4	Jonathan	West Central	.012	.013
1933	4	Jonathan	West Central	.020	.005
1933	4	Jonathan	West Central	.023	.007
1933	4	Jonathan	West Central	.020	.016
1933	4	Duchess	Central	.049	.027
1933	4	Wolf River	Central	.038	.025
1933	5	Jonathan	Central	.050	.013
1933	5	Winter Banana	Central	.038	.032
1933	5	Winter Banana	Central	.027	.014
1933	5	Maiden Blush	Central	.060	----
1933	5	Maiden Blush	Central	.047	----
1933	5 + oil	Jonathan	Central	.170	----
1933	5	Jonathan	Northwest	.075	.069
1933	5	Jonathan	Northwest	.070	.050
1933	5	Jonathan	Northwest	.060	.048
1933	5	Jonathan	Northwest	.070	.040
1933	5	Jonathan	Northwest	.100	.072
1933	5	Jonathan	Northwest	.050	.060
1933	5	Jonathan	Northwest	.035	.061
1933	5	Jonathan	Northwest	.040	.040
1933	5	Jonathan	Northwest	.050	.049
1933	5	Jonathan	Northwest	.010	.010
1933	5	Jonathan	East	.010	.006
1933	6 + lime	Jonathan	Central	.008	.007
1933	6 + lime	Delicious	Central	.008	.008
1933	6 + lime	Grimes	Central	.005	.004
1933	6	Jonathan	Northwest	.050	.049
1933	6	Jonathan	Northwest	.060	.050
1933	6	Jonathan	Northwest	.075	.069
1933	6	Jonathan	Northwest	.070	.048
1933	7	Jonathan	Central	.012	.007
1933	7	Jonathan	Central	.015	.008
1933	7	Jonathan	Central	.018	.008
1933	7	Jonathan	Central	.035	.012
1935	2	Payne' Late Keeper	Central	.012	.004
1935	2	Golden Delicious and Red York	Central	.010	.000
1935	2 + lime	Mixed Sample	East	.006	.000
1935	2 + oil	Jonathan	Northwest	.176	.049
1935	2 + oil	Jonathan	Northwest	.176	----
1935	3 + lime	Mixed Sample	East	.015	.005
1935	3	Mixed Sample	East	.015	.004
1935	3	Jonathan	West Central	.032	.019
1935	3	Jonathan	West Central	.044	.018
1935	3 + oil	Jonathan	West Central	.120	.043
1935	4	Jonathan	Central	.025	.021
1935	4	Jonathan	West Central	.032	.020
1935	4	York & Delicious	East	.009	.002
1935	4 + lime	Jonathan	East	.040	.012
1935	4 + lime	Jonathan	East	.026	.009
1935	4 + lime	Jonathan	East	.012	.004
1935	4 + lime	Grimes	East	.021	.006
1935	4 + lime	Jonathan	East	.027	.008
1935	4 + lime	Jonathan	East	.025	.007
1935	4 + lime	Willow Twig	East	.027	.009
1935	4 + lime	Willow Twig	East	.027	.012
1935	4	Jonathan	Northwest	.136	.035
1935	4	Winesap	Northwest	.072	.031
1935	4 + Kolofog	Jonathan	Northwest	.152	.030
1935	5	Jonathan	Northwest	.160	.032
1935	5	Jonathan	Northwest	.062	.018
1935	5	Winesap	Northwest	.064	.019
1935	5	Ben Davis	Northwest	.030	.016

TABLE 3. (Continued)

Year	No. of Cover Sprays	Variety	Section	Spray Residue	
				Lead	As_2O_3
1935	5	Jonathan	Northwest	.160	.051
1935	5 + oil	Jonathan	Northwest	.184	.072
1935	5 + oil	Jonathan	Northwest	.176	.069
1935	6	Rome	Central	.072	.020
1935	6	Payne's Late Keeper	Central	.060	.019
1935	6	Grimes	Central	.160	.032
1935	6	Golden Delicious	Central	.120	.040
1935	6	Jonathan	Central	.080	.020
1935	6	King David	Central	.050	.026
1935	6	Jonathan	West Central	.030	.009
1935	6 + oil	Ben Davis	West Central	.240	.060
1935	6 + lime	Jonathan	East	.070	.047
1935	6	Jonathan	East	.020	.008
1935	6	King David	East	.022	.009
1935	6 + lime	Golden Delicious	East	.019	----
1935	6 + lime	Golden Delicious	East	.018	----
1935	6 + lime	Golden Delicious	East	.018	----
1935	6 + lime	Golden Delicious	East	.015	.000
1935	6 + lime	Golden Delicious	East	.021	.004
1935	6 + lime	Golden Delicious	East	.018	.010
1935	6 + lime	Golden Delicious	East	.027	.009
1935	6 + lime	Golden Delicious	East	.027	.011
1935	6 + lime	Golden Delicious	East	.024	.011
1935	6 + lime	Golden Delicious	East	.012	.007
1935	6 + lime	Golden Delicious	East	.012	.005
1935	6 + lime	Golden Delicious	East	.016	.007
1935	6 + lime	Golden Delicious	East	.015	.003
1935	6 + lime	Golden Delicious	East	.012	.001
1935	6 + lime	Golden Delicious	East	.010	.002
1935	6 + lime	Golden Delicious	East	.014	.003
1935	6 + lime	Golden Delicious	East	.012	.001
1935	6 + lime	Golden Delicious	East	.015	.000
1935	6 + lime	King David	East	.027	.004
1935	6 + lime	Jonathan	East	.015	.003
1935	6 + lime	Jonathan	East	----	----
1935	6 + lime	Golden Delicious	East	.012	.004
1935	6 + lime	Jonathan	East	.018	.005
1935	6 + lime	Golden Delicious	East	.012	.000
1935	6 + lime	Golden Delicious	East	.030	.002
1935	6 + lime	Jonathan	East	.017	.000
1935	6 + lime	Golden Delicious	East	.024	.010
1935	6	Jonathan	Northwest	.024	.015
1935	6	Jonathan	Northwest	.176	.057
1935	7	Jonathan	East	.024	.012
1935	7 + Kolofog	Jonathan	West Central	.240	.075
1935	8	Jonathan	East	.072	.013
1935	8	Jonathan	Northwest	.160	.055
1935	8	Jonathan	Northwest	.176	.050
1935	8	Winesap	Northwest	.144	.044
1935	8 + oil	Winesap	Northwest	.240	.080
1935	8 + oil	Winesap	Northwest	.240	.080
1935	9	Jonathan	Southwest	.160	.044
1935	9	Jonathan	Southwest	.176	.080
1935	9 + oil	Jonathan	Southwest	.176	.080
1935	9 + oil	Jonathan	Southwest	.110	.075
1935	9 + oil	Jonathan	Southwest	.240	.080
1935	9 + oil	Payne's Late Keeper	Southwest	.048	.019
1935	9 + oil	York	Southwest	.100	.032
1935	10	Jonathan	Northwest	.240	.085
1935	10 + oil	Jonathan	Northwest	.240	.080
1935	10 + oil	Jonathan	Northwest	.240	.080
1936	2	Benoni	Central	.072	.024
1936	6 + lime + oil	Willow Twig	East	.168	.049
1936	6 + lime + soybean flour	Willow Twig	East	.164	.033
1936	6 + lime + Rawleigh Dip	Willow Twig	East	.164	.048
1936	6 + lime + tar soap	Willow Twig	East	.160	.024
1936	6 + lime + oil	Willow Twig	East	.176	.056
1936	6 + lime + oil	Willow Twig	East	.152	.035
1936	7 + lime + oil	Willow Twig	East	.160	.037
1936	7	Jonathan	Northwest	.176	.063
1936	7 + Rawleigh Dip	Jonathan	Northwest	.144	.058
1936	7 + soybean flour	Jonathan	Northwest	.080	.057
1936	7	Jonathan	Northwest	.128	.055

TABLE 3. (Continued)

Year	No. of Cover Sprays	Variety	Section	Spray Residue	
				Lead	As_2O_3
1936	7 + tar soap + kerosene	Jonathan	Northwest	.160	.055
1936	7 + oil + soybean flour	Jonathan	Northwest	.144	.057
1936	6 + Oxo Bordeaux	Gano	Southwest	.160	.067
1936	6 + Oxo Bordeaux	Gano	Southwest	.144	.038
1936	6 + oil	Gano	Southwest	.168	.069
1936	7 + lime + Paris Green	Gano	Southwest	.036	.016
1936	7 + Alorco Cryolite	Gano	Southwest	.064	.023
1936	7 + Phenothiazine	Gano	Southwest	.022	.009
1936	8 + DX67	Gano	Southwest	.024	.010
1936	8 + lime + Paris Green	Gano	Southwest	.176	.063

RECOMMENDATIONS

Growers in Missouri who have sprayed thoroughly and according to the recommended schedule will find the arsenic trioxide and lead content of the spray residue on their fruit in excess of the tolerance limits. This necessitates removal of the excess spray residue before offering the fruit for interstate commerce.

Many means have been used to remove excess spray residue from fruit, but the use of some kind of solvent has been found most effective. The two solvents which have been used more than any others, and have, so far, been found most satisfactory are dilute hydrochloric acid and sodium silicate solution. Dilute hydrochloric acid has proved more satisfactory in the Middle West.

In preparing to wash fruit, one should first learn the amount of residue present. This will vary with the number of cover sprays, concentration of spray materials, rate and date of application, and weather conditions. Some variation between different varieties may be found where the same spray schedule has been followed.

Where the lead content of spray residue is not greater than .100 grain per pound of fruit a dilute hydrochloric acid solution, made by adding 3 gallons of commercial concentrated hydrochloric acid to 100 gallons of water, may reduce the residues of lead and arsenic below tolerance limits. In case oil or Kolofog sprays have been used without the addition of lime, and where a waxy coat has developed on the fruit, the concentration of hydrochloric acid mentioned above will probably not prove effective. For spray residues with more than .100 grain of lead per pound of fruit and not over .160 grain, it is possible to remove such lead residues by using a wash solution containing commercial hydrochloric acid at the rate of 5 gallons to 100 gallons of water, providing again that oil or Kolofog sprays have not been employed and excessive wax formation has not taken place.

To effectively remove spray residue where oil or Kolofog sprays have been used, or where the fruit has become waxy, it will be necessary to raise the temperature of the wash solution to 100°F or slightly higher. This greatly increases the efficiency of removal. When lime is used with Kolofog, ease of removal of spray residue is greatly increased. The wetting agent, Vatsol, added at the rate of 8 pounds to 100 gallons of wash solution increases the efficiency of the wash. When Vatsol is used in the flood type washer, foaming is very troublesome and a foam breaker is necessary. Foam breakers, however, may injure the fruit. With increasing temperature of the wash solution above 100°F, there is an increasing tendency to injure the fruit.

In the flood type of washer, time of exposure to the dilute hydrochloric acid may be varied. With average spray residues in Missouri, 20 to 30 seconds may be sufficient. With residues difficult to remove, it may be necessary to increase the time to 45 seconds. In case a flotation type of washer is used, the time of contact between the fruit and the wash solution must be further increased. The exact time necessary for the fruit to be in the wash solution, however, can be determined only by trial. Each variety and each lot with different spray applications may require a different length of time.

Fruit is much more easily cleaned if washed immediately after picking. Wax formation takes place on the surface of apples on standing. The wax causes removal of spray residue to be much more difficult. Wax formation takes place more rapidly at higher temperatures. When temperatures at picking time are relatively high, it is all the more important to wash the fruit at once.

In general 100 gallons of wash solution should be sufficient to wash 1000 bushels of fruit. It is then advisable to drain the washer, rinse it, then recharge the wash section with a fresh dilute hydrochloric acid solution. Strong hydrochloric acid readily attacks iron, steel, stone, and concrete and should be stored and measured out preferably in glass containers.

SPECIFIC DIRECTIONS FOR WASHING FRUIT WHERE LEAD ARSENATE ONLY HAS BEEN USED AS THE STOMACH POISON

1. Have the spray residue on the fruit under consideration determined. Where all varieties of a given orchard have received the same number of spray applications of the same materials at about the same time and the same rate, the residue is not likely to vary greatly with the variety.

2. With the lead content not over .050 grain per pound of fruit and where neither oil nor Kolofog has been used, it may be possible to satisfactorily lower the spray residue below tolerance limits by using a one-half of one per cent hydrochloric acid wash solution. An exposure of 30 seconds of the fruit to the wash solution in an underbrush flood type washer should be sufficient, especially when the fruit is washed immediately after picking. A one-half of one per cent hydrochloric acid solution may be prepared by adding the concentrated commercial hydrochloric acid to water at the rate of 1½ gallons of the concentrated acid to 100 gallons of water.

Where lead arsenate is the only arsenical which has been used, it is usually safe to assume that the arsenic content, calculated as arsenic trioxide, of the residue is below the tolerance limit of .01 grain per pound of fruit when the lead content of the residue is below the tolerance limit of .018 grain per pound of fruit.

3. With the lead content of the residue not over .100 grain per pound of fruit, where the temperature of the wash solution is not less than 75°F and where neither oil nor Kolofog has been used, it may be possible to satisfactorily lower the residue below tolerance limits by using a one per cent hydrochloric acid solution for the wash with an exposure of the fruit to the wash solution of 30 seconds in an underbrush flood type washer and 1¾ minutes in a flotation washer. A one per cent solution of hydrochloric acid may be prepared by adding the concentrated commercial acid at the rate of 3 gallons to 100 gallons of water.

4. With higher residues, or where either oil or Kolofog has been used, it may be necessary to increase the acid concentration of the wash solution to 1.5% or 1.75%. Where oil or Kolofog or both have been used and the lead content of the residue is above .100, it may be necessary to heat the solution to as high as 110°F, in addition to using the higher concentrations of hydrochloric acid wash solution. Wash solutions of higher temperatures are much more effective than the same solutions at lower temperatures in reducing spray residues.

Where lime is used in the cover sprays, especially the later ones, the residue is much more easily reduced.

The strength of the wash solution should be checked at intervals, especially where fruit with a high spray residue is being washed. When the acid is found below the strength required, by referring to the following table the amount of concentrated hydrochloric acid necessary to produce any desired concentration of the wash solution may be readily determined.

For the determination of the strength of the hydrochloric acid wash, the following equipment may be used: One 10 cc bulb pipette; one 10 cc measuring pipette, graduated in .1 cc; a clean pint milk bottle or any clean wide mouthed bottle of colorless, clear glass; standard sodium bicarbonate solution containing 23 grams of sodium bicarbonate in 1000 cc of water. The standard sodium bicarbonate solution should contain enough methyl orange indicator to give a yellow color. This equipment is inexpensive.

TABLE 4.—VOLUME OF CONCENTRATED ACID REQUIRED TO MAKE 100 GALLONS OF THE DILUTE HYDROCHLORIC ACID WASH SOLUTION

Per cent of acid by weight required in wash	28% HCl, 18° B.			32% HCl, 20° B.			35% HCl, 22° B.		
	Gals.	Qts.	Pts.	Gals.	Qts.	Pts.	Gals.	Qts.	Pts.
.1	0	1	1	0	1	½	0	1	⅓
.2	0	3	0	0	2	1	0	2	⅔
.3	1	0	1	0	3	1½	0	3	1
.4	1	1	1	1	1	0	1	0	1
.5	1	3	0	1	2	½	1	1	1½
.6	2	0	1	1	3	1	1	3	0
.7	2	2	0	2	0	1½	1	3	1
.8	2	3	1	2	2	0	2	1	½
.9	3	1	0	2	3	½	2	2	1
1.0	3	2	1	3	0	1	2	3	1
1.2	4	1	0	3	3	0	3	1	1½
1.5	5	2	1	4	2	1½	4	1	½
1.75	6	1	0	5	1	1¾	5	0	0
2.00	7	0	1	6	1	0	5	3	0

To determine the strength of the acid, fill the bulb pipette with clean solution from the acid tank of the washer, drawing it into the pipette by suction. Let the excess acid flow out until the top of the liquid column is even with the mark on the upper part of the pipette; then let the measured acid flow slowly into the clean, wide-mouthed bottle, rotating or shaking the bottle gently as the bicarbonate solution is carefully allowed to flow in. At the point where the color of the acid changes from red to yellow, note the number of cubic centimeters of the sodium bicarbonate solution which have been run into the bottle from the pipette. From this number the strength of the acid is read directly. For example, if 7.2 cc of the sodium bicarbonate were required to produce the color change, then the strength of the acid wash solution is .72%. To make such a solution to a concentration of one per cent would require enough acid to make a concentration of 1.00—.72=.28% or approximately .3% which is sufficiently accurate. Reading from the table this will require 9 pints of the 28% acid; 7½ pints of the 32% acid or 7 pints of the 35% acid. To be more accurate, however, 28/30 of the above amounts would be required, making 8½ pints of the 28% acid; 7 pints of the 32% acid and 6.5% of the 35% acid.

Home-made flotation washers may be constructed cheaply and are simple in operation. Plans for such a washer should be obtained and construction started fully three weeks before actual washing operations are to begin. Home-made flotation washers are effective in removing light to medium residues, especially where oil or Kolofog has not been used. For the heavier residues and those otherwise more difficult to remove, flotation washers may not be effective. If they are used under such conditions, the speed must be reduced to such an extent that their capacity is appreciably lowered. The greater time of contact resulting from reduced speed makes injury to the fruit more likely. Adequate provision should be made for rinsing the fruit with fresh water after it leaves the wash solution in a flotation washer.