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# START





MICROCOPY RESOLUTION TEST CHART NATIONAL BUREAU OF STANDARDS-1963-A

MICROCOPY RESOLUTION TEST CHART NATIONAL BUREAU OF STANDARDS-1963-A



### Further Studies on the Removal of Spray Residues from Eastern-Grown Apples<sup>1</sup>

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#### INTRODUCTION

Studies on the removal of spray residues have been carried on with apples grown in the Shenandoah-Cumberland Valley during the four seasons 1934 to 1937, inclusive. The results for the first two seasons have been presented in a previous publication (6),<sup>2</sup> and the results for the last two seasons are presented herein. The purposes of these investigations were to determine the effect of certain modifications of the spray program on residue removal and the relative effectiveness of various washing treatments in removing the residue as influenced by spray treatment, maturity or ripeness of the fruit, and other factors.

At the time these investigations were conducted the regulatory tolerances for spray residues were 0.018 grain of lead and 0.010 grain of arsenious oxide (As<sub>2</sub>O<sub>3</sub>) per pound of fruit. Since then the tolerances have been raised to 0.050 grain of lead and 0.025 grain of As<sub>2</sub>O<sub>3</sub> per pound. The new standards will make it possible for some additional

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growers to avoid washing entirely and for others to use less severe washing treatments. In the investigations reported herein, even when only five cover sprays were used the lead residues were in excess of the present lead tolerance, showing that cleaning would be necessary even with fairly simple spray programs. Consequently, the change in the tolerances has not materially affected the value of the results. As indicated previously (6), it is not possible to formulate precise directions for washing apples after a given spray practice to meet any particular residue tolerance, because of the great variability in the spray-residue load resulting from many uncontrollable factors, such as growth conditions of the trees and thoroughness and time of spray For this reason the results have been presented and applications. discussed largely from the standpoint of the relative effects of the various treatments and not of meeting any particular residue tolerances. The determination of these fundamental principles forms a basis by which spray-residue problems of the individual growers may be solved.

#### REVIEW OF LITERATURE

Since this work was started there have been reported a number of experiments in which both the lead and the arsenic determinations have been presented and the ratios of lead (Pb) to arsenious oxide (As<sub>2</sub>O<sub>3</sub>) have been or may be computed. For the lead arsenate spray material

(PbHAsO<sub>4</sub>) this ratio is 2.09 (2).

Hartzell and Wilcoxon (7) determined the lead and the arsenic on apples at harvest. The ratios on 20 duplicate samples (4 apples only per sample) ranged from about 3 to about 30 and averaged 9.1. These investigators concluded that the arsenic weathered off more rapidly than the lead. However, the great variability in the ratios between the duplicate samples would indicate large sampling or analytical errors.

Robinson and Hatch (16) determined the solubility of the lead and the arsenic of lead arsenate in hydrochloric acid, sodium silicate, and other solutions. In the acid the lead and the arsenic were dissolved in the same proportion in which they existed in the lead arsenate, whereas in sodium silicate the lead was slightly more soluble than the arsenic. However, in the spray residues after washing with hydrochloric acid solutions they found the ratio of remaining lead to remaining arsenic to be higher than the ratio in the spray material when waxy or oily apples were used but not when nonwaxy apples were used.

Overley et al. (13) reported lead and arsenic determinations on apples after various spray and washing treatments. They stated that "the ratio of lead to arsenic, which exists in lead arsenate itself, is not maintained in washed fruit." They obtained ratios of lead to arsenious oxide ranging from approximately 1 to more than 10. The ratios did not vary consistently with any spraying or washing treatment, and in some instances the ratios of duplicate lots differed greatly. This would indicate that the variations were probably due largely to analytical or sampling errors.

McLean and Weber (11), using various spray treatments and varieties, obtained ratios at harvest of 2.3 to 3.6 (average 3), and, after washing the fruit with hydrochloric acid (HCl) solutions at room

temperatures, ratios that ranged from 3.1 to 6.3 (average 4.1).

Pentzer (15), with several varieties and spray treatments, obtained average ratios for the residues at harvest essentially the same as that for the spray material. The ratios after washing with HCl solutions were somewhat higher (about 2.5) but probably not significantly different from that for the original spray material. Residues after washing were frequently very low, so that a slight error in the determination of either the lead or the arsenic would result in a large error in the ratio.

Analyses given by Hough (10) for York Imperial, Ben Davis, and Gano with various lead arsenate spray treatments indicated ratios at harvest of 1.6 to 2.1 (average 1.9). After washing with various hydrochloric acid solutions and some sodium silicate solutions, the ratios averaged the same as at harvest with somewhat greater variation, but with no consistent relation to the spraying or the washing treatments.

Weber et al. (19) presented data indicating that the ratios at harvest for various spray treatments ranged from 4.5 to 6.6 (average 5.6). After washing with a hydrochloric acid solution the ratios ranged from 3 to 5 (average 4). They concluded that the arsenic weathered from the fruit to a greater extent than did the lead. On the other hand, the results indicated that a somewhat greater proportion of

lead was removed during washing.

Working with York Imperial and Stayman Winesap apples in Pennsylvania, Frear and Worthley (4) obtained ratios of 3.56 when no sticker was used and 2.17 and 2.24 when skim-milk powder and fish oil were used, respectively. They stated that the ratio was greatest when no modifying agent was used. On the other hand, in similar spray treatments reported in an earlier publication (3), they obtained corresponding ratios of 3.1, 2.9, and 3.9, indicating that the ratio was greater when fish oil was used than when no modifying agent was used.

Ellenwood et al. (1) reported analyses of apples from four spray treatments in which lead arsenate was used. The average ratio of lead to arsenious oxide on apples from these sprays was 2.55 at harvest (before washing) and 1.61 after washing with 1 percent acid. Their results indicate a somewhat greater removal of lead than of

arsenic during washing.

Fahey and Rusk (2) determined the ratio of lead to arsenious oxide on apples and leaves from various spray plots. Analyses were made immediately after each cover-spray application and after the residues had weathered during a season of very heavy rainfall (average of 0.17 inch per day) and one of very light rainfall (average of 0.08 inch per day). The average ratio for 248 samples did not differ significantly from that for the original spray material. They considered that the high ratios obtained by early investigators probably were due to inadequate samples or to unreliable methods of analysis.

Pearce and Avens (14) found that the ratio of lead to arsenic was somewhat higher than the theoretical (2.2 to 3) at harvest and that it was slightly higher after washing than before (2.4 and 2.5 after as compared with 2.2 before). The ratio was less with oil in the sprays

than without,

Cassil and Moulton <sup>3</sup> determined the arsenic as well as the lead on certain lots of York Imperial apples used in these investigations and

<sup>\*</sup> Cassil. C. C., and Moulton, C. H. Unpublished data for January 1937.

obtained from spray plots 1 and 2 (table 1) in 1936. At harvest an average Pb/As<sub>2</sub>O<sub>3</sub> ratio of 2.2 with a standard deviation of 0.25 was obtained. Apples from these spray plots were washed with an acid solution alone at room temperature and with an acid wetting-agent solution at 100° F. For 24 determinations the mean ratio after washing was 2.4 with a standard deviation of 0.3. There were no consistent differences due to spraying or washing treatments. These results indicate ratios slightly higher than in the original spray material, particularly after washing, but the variability was such that the difference was not statistically significant or great enough to be of much practical importance.

From this review of the literature there appears to be considerable disagreement as to the ratios of lead to arsenious oxide that obtain at harvest and after washing. However, ratios that do not differ greatly from that in the lead arsenute spray material were found by a majority of the investigators (1, 2, 10, 14, 15, 16) . Some, as Frear and Worthley (3, 4) and Overley et al. (13), in most instances obtained ratios approximating the theoretical but in other instances obtained ratios that were distinctly divergent. The variations were not consistent with any particular type of spraying or washing treatments. Only in the case of Hartzell and Wilcoxon (7) and McLean and Weber (11) were the ratios rather consistently at variance with the theoretical. It is difficult to understand why these investigators should have obtained such high ratios when other investigators using similar types of spraying and washing treatments do not obtain them. general, the results seem to indicate that, with the present (1940-41) tolerances (0.050 and 0.025 grain per pound, respectively, for lead and arsenious oxide), apples sprayed with lead arsenate would carry lead and arsenic residues approximately in the same proportion as the tolerances, so that washing treatments that are effective for lead would be about equally so for arsenic. For this reason lead only was determined in the investigations herein reported. The consequent saving in time permitted more extensive removal investigations than would have been possible if both lead and arsenic had been determined.

#### MATERIAL AND METHODS

The apples for these investigations were obtained from spray plots in an orchard near Martinsburg, W. Va. Stayman Winesap, York Imperial, and Delicious were used in 1936, and Stayman Winesap, York Imperial, and Winesap in 1937.

The spray treatments are outlined in table 1. These consist of 5 and 7 cover sprays of lead arsenate with and without bordeaux mixture and with and without mineral oil emulsion in the second-brood cover sprays, and in one instance (treatment 5) with mineral oil emulsion in the second and third cover sprays. Each spray treatment was replicated on 2 plots of 1 or more (usually 2) trees per plot.

The cumulative rainfall for the growing seasons is shown in figure 1. The apples were harvested during the commercial harvest season, and in some instances an early and a late picking were also made.

The washing treatments outlined in table 2 were used. Certain of these washing treatments were used in both seasons and with all lots

<sup>4</sup> See footnote 3.

of apples. Others were limited in their application to certain lots and seasons, as indicated in the tabulation of results (see tables 3 to 8).

The samples for chemical analysis consisted of duplicate lots of 30

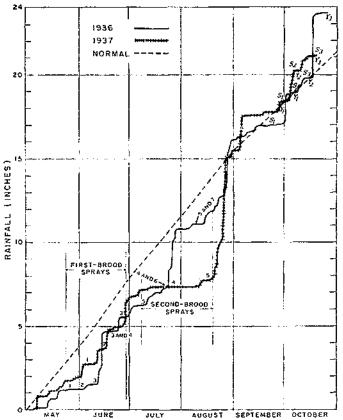


FIGURE 1. - Cumulative rainfall during the growing seasons of 1936 and 1937 at United States Weather Bureau stations nearest to experimental plots. Numbers 1 to 7 on curves indicate cover-spray applications.  $S_1$ ,  $S_2$ , and  $S_3$  and  $Y_1$ ,  $Y_2$ , and  $Y_3$  represent first, second, and third picking of Stayman Winesap and York Imperial apples, respectively.

apples from the replicate spray plots. Lead only was determined by the mush-dithizone-electrolytic method described by Wichmann et al. (20).

Table 1.—Outline of treatments for apples used in experiments on removal of spray residue

Date of applies-		7	Materials (	for spray is	rentment No	-	
tion and cover spray No.	1	5	3	-5	ñ	6	7
1936							
3 (June 19) 4 (July 21) 5 (Aug. 8) -cover test; 1 (May 26) 2 (June 3) 3 (June 10) 4 (June 19) 6 (July 21)		LA+B LA+B LA+B LA+B+MO LA+B+MO			LA+B BA+B+MO LA+B+MO LA+B LA+B	LA+B LA+B LA+B LA+B	LA+B LA+B LA+B LA+B LA+B+M LA+B+M
7 (Aug. 8) 1937	·- ·				•••••	LA+B	LA+B+M
5-cover (est: 1 (June 4) 2 (June 15) 3 (June 20) 4 (July 27) 5 (Aug. 16)	LA+B	LA+B LA+B LA+B LA+B+MO LA+B+MO		LA+B LA+B LA+B LA+MO LA+MO LA+MO	. 4444		

 $<sup>^{-1}</sup>$  Abbreviations and quantity per 100 gallons: LA = lead arsenate, 3 pounds; B = bordeaux mixture (2-4-100); MO=mineral oil crudsion, 1 gallon.

Tables 2.- Outline of apple-washing treatments

		Concen- tration of	Additional materia	1			
Wash- ing treat- ment	Machine	hydro- chloric neid in washing solution	Kind	Cancen- tration	Temper- ature of solution	Time in washing solution	Years
		Percent		Percent	• <sub>F</sub> ,	Seconds	
A1	Flotation.	0.5			60-70	60	1936; 1937 1936.
B-1 B-2	do	1.5			60-70	60	1930: 1937
B-3	(ip	1.5	• • • • • • •		100	60	1936.
33-3. 13-1	do	1.5	Wetting agent (Vatsoft	1.00	60-76	άΰ	1936.
B-5	do	L5 :	do	1.00	100	ĞÕ	1936: 1937
2-1	Floor	1,5			60-70	35	1936; 1937
(*.2	. do	1.5			100	35	1936; 1037
C-3,	.,.do	1,5	Wetting agent (Vatsol)+ antifonm (De Gras),	. 25	100	3.5	1937.
C-1	.do '	1.5	Mineral oil .	1.00	100	35	1937.
D-1	Floorl-brush	1, 5			60-70	35	1936; 1937
3.2	(lo	1.5			160	35	1936; 1937
D-3	do	1.5	Writing agent (Vatsol)- - antifoam (De Gras).	. 25	160	35	1937.
1)-4	do.,	1.5	Mineral oil.	1.00	100	35	1936; 1987

<sup>1</sup> Not washed.

#### RESULTS

The general data are shown in tables 3, 4, and 5 for Stayman Winesap, York Imperial, and Delicious, respectively, in 1936, and in tables 6, 7, and 8 for Stayman Winesap, York Imperial, and Winesap, respectively, in 1937. Some of the average results from spray treatments 1 and 2 on Stayman Winesap and York Imperial are also presented in figures 2 and 3.

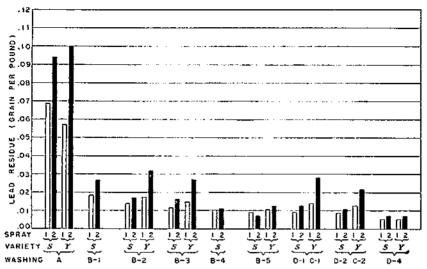


FIGURE 2.—Lead residues at harvest and after various washing treatments on Stayman Winesap (S) and York Imperial (Y) 1936. (See tables 1 and 2 for descriptions of spray and washing treatments.)

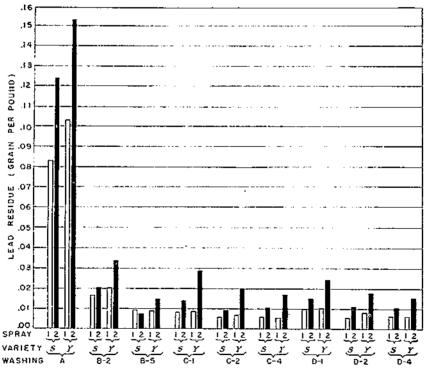


FIGURE 3.—Lead residues at harvest and after various washing treatments on Stayman Winesap (S) and York Imperial (Y) 1937. (See tables 1 and 2 for descriptions of spray and washing treatments.)

Table 3.—Lead residue on Stayman Winesap apples in relation to spraying and washing treatments, time of picking, and delay before washing, 1936

[Results expressed as grain per pound of fruit and percent of residue before washing]

Delay before wash-	Spray	treat nt <sup>1</sup>									Lead	residu	after	washin	g treat	ment ²	_				4.1			
ing and No. and date of picking	No.	Rep- li- cate		1	1	J-1	В	3–2	E	i <b>-3</b>	I	1-4	В	i <b>-5</b>	С	-1	С	-2	D	-1	D	-2	l	 )- <b>4</b>
No delay:  1 (Sept. 23)  2 (Oct. 5)  3 (Oct. 15)  13-day delay at room temperature:  2 (Oct. 5)	$ \left\{ \begin{array}{c} 1 \\ 2 \\ 3 \\ 5 \\ 6 \\ 7 \\ 1 \\ 2 \\ \end{array} \right. $	\{ \bar{b} a b a b a b a b a b a b a b a b a b a	Grain 0.067 0.073 0.097 0.084 0.065 1.000 0.088 0.082 0.077 0.081 0.073 0.068 0.074	Per- cent 100 100 100 100 100 100 100 100 100 10	Grain 0.020 .017 .028 .025 .030 .023 .023 .013 .034 .032		Grain 0. 017 . 014 . 024 . 020 . 015 . 013 . 020 . 014 . 021 . 017 . 017 . 011 . 012 . 020 . 014 . 013 . 017 . 017 . 017 . 011 . 012 . 020 . 020 . 020	Per- cent 25 19 25 24 20 20 20 16 23 22 21 15 23 15 27 32 32 33	Grain 0. 015 . 013 . 020 . 015 . 013 . 011 . 018 . 014 . 017 . 016 . 014 . 011 . 009 . 020 . 016 . 012 . 011 . 011 . 011	Per-cent 222 18 211 18 18 16 18 19 19 15 16 18 26 28 29 27	0.012 -009 -012 -018 -015 -011 -012 -011 -009 -014 -010	Per- cent 16 14 12 11 19 18 14 15 15 17 11	Grain 0.010 .009 .009 .008 .010 .007 .007 .016 .013 .008 .009 .008 .009 .008 .009 .008 .009 .008 .009 .008 .009	Per- cent 15 12 9 14 12 7 8 8 17 16 10 11 11 13 6 8 17 10 11	0.011 012 .018 .014 .013	*****	Grain 0.009 .011 .012 .014 .010 .009	Per- cent  12 17 13 17 13 11	Grain  0.011 .008 .014 .012 .015 .012 .011 .019 .009 .007 .019	Per-cent  15 12 14 14 15 14 15 12 13 14 12	Grain 0.009 0.008 0.012 0.09 0.11 0.009 0.006 0.05 0.014		Grain 0.007 0.007 0.004 0.007 0.008 0.006 0.006 0.006 0.006	Per-cent

 $<sup>^1</sup>$  For details of spray treatments, see table 1; a and b refer to tree replicates.  $^2$  For details of washing treatments, see table 2.

Table 4 .- Lead residue on York Imperial apples in relation to spraying and washing treatments, time of picking, and delay before washing, 1936 [Results expressed as grain per pound of fruit and percent of residue before washing]

Delay before washing and	Spray me	treat- nt <sup>1</sup>						Lea	d residu	e after w	ashing tr	eatment i	1					
No. and date of picking	No.	Repli- cate	1	1	В	-2	P	J <b>-</b> -3	В	-5	c	-1	C	'-2	D-1	D-2	E	)-4
No delay:	<b>1</b>	{ n } b	Grain 0,076 .083	Percent 100 100 100	0.016	Percent 21 16	0.017	Percent 22 17	0.012	Percent 16 14	Grain	Percent	Grain	Percent	Grain	Grain	Grain	Percent
2 (Oct. 15)	1 2 6	h	. 108 . 105 . 061 . 053 . 104 . 096	100 100 100 100 100 100	.027 .024 .021 .014 .035 .029	25 23 34 26 33 31	. 026 . 023 . 017 . 013 . 029 . 025	24 22 28 24 28 26	.010 .013 .011 .010 .013 .012	12 18 19 12 13	0.015 .013 .028 .028	25 24 27 29	0. 014 . 011 . 021 . 022	23 21 20 23	0.009	0.006	0.007 -004 -007 -007 -006 -004	1
3 (Oct. 26)	$egin{pmatrix} 7 \\ 1 \\ 2 \end{bmatrix}$	{	.056 .049 .070 .070	100 100 100 100 100	.013 .017 .028 .026	23 35 40 37	.012 .011 .022 .019	21 22 31 27	.010 .008 .010 .012	18 16 14 17					.019	.014	.006	
2 (Oct. 15)  13-day delay at room temperature:	$\left\{egin{array}{c} 1 \ 2 \end{array} ight.$	{ a b a a b	. 061 . 053 . 104 . 096	100 100 100 100 100	. 027 . 019 . 053 . 048	44 36 50 50	.022 .018 .045 .043	36 34 43 45	.012 .013 .018 .016	20 24 17 17								
2 (Oct. 15)	$\left\{egin{array}{c} 1 \ 2 \end{array} ight.$	{ a b a b b b	.061 .053 .104 .096	100 100 100 100	. 030 . 025 . 064 . 074	49 47 61 78	. 026 . 025 . 054 . 057	43 47 51 60	.014 .012 .021 .019	23 23 20 20							117, 11 12. 11 20 1 12. 12 20 1 18. 18. 18. 18. 18. 18. 18. 18. 18. 18.	

For details of spray treatments, see table 1; a and b refer to tree replicates.
 For details of washing treatments, see table 2.

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Table 5.—Lead residue on Delicious apples in relation to spraying and washing treatments, 1936

[Results expressed as grain per gound of fruit and percent of residue before washing]

Date of picking		trent-	Le	ad residue after w	eshing treatmen	L 2-
	No.	Rep- liente	Λ	B-2	B-3	13-5
Sept, 22	1 2	0.8 G.B	Grain Porcent 0.068 100 .081 100 .135 100 .113 100	Grain   Percent   20   20   19   .026   19   .024   21	Grain   Percent   0.012   18   .011   18   .021   16   .022   10	Grain   Percent   0.007   11   .008   13   .012   9   .010   15

 $<sup>^{\</sup>rm t}$  For details of spray treatments, see table 1; a and b refer to tree replicates.  $^{\rm t}$  For details of washing treatments, see table 2.

Table 6.—Lead residue on Stayman Winesap apples in relation to spraying and washing treatments, time of picking, and delay before washing, 1937

[Results expressed as grain per pound of fruit and percent of residue before washing]

Delay before wash-	Sp trenti	ray nent !									Lead re	esidue	ifter wi	shing	treatme	ent 2					-			
ing and No. and date of picking	No.	Rep- licate	-	A	В	-2	В	i-5	C	2-1	O	-2	c	-3	o	<b>-</b> 1	D	-1	α	-2	D	-3	D	)-4
No delay:	1	{ a b	Grain 0.069 .083 .120	Per- cent 100 100 100	Grain 0.013 .018 .022		Grain 0.008 009	Per- cent 12 11	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent
2 (Oct. 8)	$  \begin{cases}    2 \\                             $	{	.120 .104 .077 .090 .137 .111 .067 .074	100 100 100 100 100 100 100 100	.022 .016 .016 .018 .020 .021 .021 .022 .047	18 15 21 20 15 19 31 30 28	.005 .006 .009 .010 .005 .007 .013 .010	6 12 11 6 6 19 14 13	0,008  .009  .015  .013	10 10 11 11 12	0.006 .006 .009 .009	8 7 7 8	0.005 .005 .007 .006	6 6 5 5	0.006 .006 .010 .011	8 7 7 10	0.00S .012 .014 .015	10 13 10 14	0.005 .007 .012 .010	6 8 9 9	0.007 .008 .009 .008	9 9 7 7	0.007 .007 .010 .012	9 8 7 11
3 (Oct. 18)	{ I 2	{ b a b a b }	. 154 . 074 . 068 . 103 . 104	100 100 100 100 100	.038 .015 .013 .018 .018	25 20 19 17 17	.021 .017 .011 .008 .007 .007	13 11 15 12 7 7	The state of the s															
2 (Oct. 8) S-day delay at room temperature:	$\left\{egin{array}{c} 1 \\ 2 \end{array}\right.$	{ b a b b	.077 .090 .137 .111	100 100 100 100	.016 .018 .027 .024	21 20 20 20 22	.006 .009 .010 .007	8 10 7 6												/# _# /4, #				
2 (Oct. 8)8-day delay at 32° F.:	1 2	{ b a b b	.077 .090 .137 .111	100 100 100 100	.016 .016 .027 .023	21 18 20 21	.007 .009 .009 .007	9 10 7 6																
2 (Oct. 8)	1 2	{	.077 .000 .137 .111	100 100 100 100	.015 .012 .021 .019	19 13 15 17	.008 .009 .009 .007	10 10 7 6																

<sup>1</sup> For details of spray treatments, see table 1; a and b refer to tree replicates.

<sup>&</sup>lt;sup>2</sup> For details of washing treatments, see table 2.

Table 7.—Lead residue on York Imperial apples in relation to spraying and washing treatments, time of picking, and delay before washing, 1937 [Results expressed as grain per pound of fruit and percent of residue before washing]

Delay before wash-	Spi treatn									L	ead resi	due aft	er wasl	ing tr	eatmen	ts 7—								
ing and No. and date of picking	No.	Rep- licate			В	-2	В-	-5	r	-1	c	-2	C	-3	c	4	D	<b>-1</b>	D	-2	D	-3	D	- <b>4</b> .
No delay:	1	{ a b	Grain 0.098 .080	100 100	Grain 0. 020 . 019	20 24	Grain 0.013 .008	13 10	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent	Grain	Per- cent
2 (Oct. 8)	$\begin{bmatrix} & 2 \\ & 1 \\ & 2 \end{bmatrix}$	{ a b a b { a b }	.155 .157 .103 .104 .142 .164	100 100 100 100 100	.033 .031 .020 .021 .037	21 20 19 20 26 18	.012 .010 .007 .011 .015	8 6 7 11 11 9	0.010 .008 .028 .029	10 8 20 18	0,007 .007 .020 .019	7 7 11 12	0.006 .006 .016 .015	6 6 11 9	.006	21 6 13 10	0, 011 . 010 . 024 . 025	11 10 17 15	0.008 .009 .021 .015	8 9 15 9	0.009 .008 .022 .017	9 8 15 10	0.007 .007 .013 .018	7 7 9 11
3 (Oct. 18)	$\begin{vmatrix} & 3 \\ & 4 \\ & 1 \\ & 2 \end{vmatrix}$	{ b n h { b a b } { a }	.113 .110 .204 .212 .112 .092 .158	100 100 100 100 100 100 100	.026 .025 .088 .055 .019 .017	23 23 43 26 17 18 23	.013 .008 .051 .038 .013 .010	12 7 25 18 12 11 8																
3-day delay at room temperature: 2 (Oct. 8). 8-day delay at room	$\left\{egin{array}{c} 1 \\ 2 \end{array}\right.$	{ a b a b { a b b }	. 134 . 103 . 104 . 142 . 164	100 100 100 100 100	.043 .021 .023 .046 .042	20 22 32 26	.015 .012 .012 .013 .012	11 12 12 9 7												*****				*****
temperature;	$ \begin{vmatrix} 1 \\ 2 \end{vmatrix}$	{ b a b b	. 103 . 104 . 142 . 164	001 001 001 001	.020 .015 .045 .038	19 14 32 23	.010 .010 .016 .013	10 10 11 8	1															
8-day delay at 32° F.: 2 (Oct. 8)	1 2	{ b a b	. 103 . 104 . 142 . 164	100 100 100 100	.019 .019 .036 .030	18 18 25 18	.012 .011 .018 .015	12 11 13 9																

For details of spray treatments, see table 1; a and b refer to tree replicates.
 For details of washing treatments, see table 2.

Table 8.—Lead residue on Winesap apples in relation to spraying and washing treatments, 1937

[Results expressed as grain per pound of fruit and percent of residue before washing]

Date of picking	S <sub>i</sub> trest	orny ment !	Lead reside	or after washing to	rentment 2—
	No.	Repli- cate	Α	B-2	B-5
Oct. 8	1 2	d # d # d	Grain   Percent   0.080   100   100   101   100   100   100   138   100	Grain   Percent   0.012   15   16   16   16   16   17   17   17   17	Orain   Percent   0.007   0.005   5   0.005   4   0.008   6

<sup>1</sup> For details of spray treatments, see table 1; a and b refer to tree replicates.

1 For details of washing treatments, see table 2.

All of the data pertinent to certain phases of the investigation were separated from these tables and treated statistically by an analysis of variance, and the discussion is based on the statistical significance of the differences as estimated by such analyses. In most cases the comparisons were based on percentages of the residues before washing rather than on the actual residues.

#### COMPARISON OF VARIETIES

#### RESIDUES AT HARVEST

Data relative to the residues at harvest on apples of the different varieties are presented in table 9; some additional data for Stayman Winesap and York Imperial at the different pickings were available but are not presented in the table. A statistical analysis of all the data for York Imperial and Stayman Winesap for the two seasons showed that York Imperial carried considerably more residue than Stayman Winesap, the difference being particularly marked in the 1937 season. As the York Imperial apples were considerably smaller, it seemed probable that the difference in residue was due mainly to difference in size. Ellenwood et al. (1) concluded that size of fruit was the principal cause of differences between varieties.

TABLE 9 .- Relation of variety and weight of apples to lead residue at harvest, 1986 and 1937

[Results expressed as grain per pound of fruit]

Variety and year	Ī 	Spra m	y	Lre	eat-	Average weight of apples in	Lead residu	e at harvest
variety and year		No.			Rep- iente	sample (S <sub>i</sub> )	£2, 2	$R_{7}^{3}$
Stayman Winesap:		1	1	{	a b a b	Grums 123 133 120 157	Ornin 0.073 .065 .100 .088	Grain 0, 087 , 083 , 117 , 130
A Verage			;			133	. 0815	
11037	{	1 2		{	n b n h	166 143 164 177	. 077	. 120 . 122 . 199 . 183
Average						160	. 1038	, 154
Average, both years	ļ		1			147	. 0927	. 131
York Imperial: 1936		1 2	į	{	11 11 11	107 119 90 101	. 061 . 063 . 104	. 065 . 061 . 101 . 097
Average						106	.0785	
1937		1	:	{	n h n h	99 95 112 127	, 103	. 10; . 10; . 15; . 201
Average	١					108	. 1282	. 140
Average, both years			]			107	. 1034	
Delicious:		1 2	:	{	n h n b	119 119 119 120	. 066 . 061 . 135 . 113	. 077 . 071 . 150 . 141
Average	-		i			122	.093\$	. 111
Wingsap: 1937		3	ŀ	{	n b a b	3-4 119 141 126	, 101 , 134	. 110 . 117 . 150 . 168
A verage	.		1			132	. 1132	. 144

For details of spray treatments, see table 1; a and b refer to tree replicates.
 Based on actual size of apples in the sample.
 Based on apples of 100-grain size (about 2); inches in diameter). K=adjustment factor of 0.84 (see ρ, 15). Adjustment for size when S<sub>2</sub>=100:

$$R_2 = R_1 + \left(\frac{S_1 - S_2}{S_2} \cdot K \cdot R_1\right)$$

(b) for  $S_1 < 100$ ,

$$R_2 = R_1 - \left(\frac{S_2 - S_1}{S_1} \cdot K_1 R_1\right)$$

As reported previously (5), it was observed that apples from replicate spray plots of the same variety frequently differed greatly, but not as much as in the previous report, and, contrary to the earlier report, with few exceptions when there was a difference in the size of the apples the sample with the larger apples had the lower residue. Heald et al. (8) reported a similar relation with washed samples; they found an average of 30.8 percent more arsenieal residue on the smaller apples of 12 duplicate samples in which the apples in the sample with the larger apples averaged 17.6 percent larger by weight. A decrease in residue with increased size of apples is to be expected, since the larger apples would have a smaller surface area per pound.

In the present study there were 16 pairs of unwashed samples from duplicate spray plots in which there was at least a 5-gram difference between the samples from the duplicate plots in the average size of the apples. There was a significant average increase in residue on the samples with the smaller apples. The difference averaged 0.0048±0.0023 grain per pound for each 10-gram difference in the average size of the apples. As this difference would no doubt vary with the size of the load, it might be expressed better on a percentage basis. On this basis the average increase in residue was highly significant and amounted to 0.84±0.30 percent on the sample with the smaller apples for each 1-percent difference in the size of the apples in the samples. Using this as an adjustment factor, the residues were computed on the basis of the apples weighing 100 grams, which are equivalent to those of about 2.5 inches in diameter. On this basis the average difference between replicates was reduced, as shown in table 9.

An analysis of variance of the adjusted values showed that York Imperial apples had highly significantly less residue than Stayman Winesap apples. This finding is in agreement with the results of Frear and Worthley (4), who found that more lead per unit of surface area was deposited on Stayman Winesap than on York Imperial fruit. When corrected for size in 1936 the Delicious also had significantly more residue than the York Imperial apples. Delicious and Stayman Winesap did not differ significantly. The interaction of sprays with varieties indicated that the addition of mineral oil emulsion to the late cover sprays increased the residue on Delicious to a greater extent than on the other varieties. This is in accord with earlier results (6, p. 12). in which it was noted that Delicious had heavier residues than the other varieties treated when mineral oil emulsion was used in the late sprays, but not with other types of spray treatments. When adjusted for size, Winesap apples averaged significantly less residue than Stayman Winesap in 1937.

#### RESIDUES AFTER WASHING

As the residues on the different varieties differed at harvest, and as a rather high correlation between residues at harvest and after washing has been reported (6, p. 16), they might be expected to differ correspondingly after washing. In order to measure the ease of residue removal from the different varieties, it seemed desirable to express the results as percentages of the original residues. This was done in table 10 for the two washing treatments used for all varieties during both seasons. These results show a highly significant difference between York Imperial and Stayman Winesap, with the average percent of the original residue retained by the York Imperial apples 35 percent greater than that retained by Stayman Winesap. The York Imperial apples retained a significantly higher percentage of the original residue

than Delicious did in 1936 and than Winesap did in 1937. Stayman Winesap apples did not differ appreciably from Delicious in 1936 but retained a higher percentage of residue than Winesap in 1937.

Table 10.—Relation of variety to ease of lead-residue removal [Results expressed as percent of residue before washing]

	Spra	y treut-	11	lesidue afte	r washing	trentment:	<u> </u>
Variety and year	m	ent i			Vi	riety aver	uge
	No.	Repli- ente	R-2	B-5	1936	1037	Both years
Stayman Winesap:			Percent	Percent	Percent	Percent	Percent
1936	$  \begin{cases}                                  $	1 1 1 1	20 20 20 16	14   12   7	14.6		]
1937	1 2	} 6 h 8 h	21 20 15	S 12 11 6	}	13.8	14. 2
York Imperial:		י י	19	6	,	' i	
1936	$\begin{bmatrix} 1 \\ 2 \end{bmatrix}$	{ ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ;	34 26 33 31	18 19 12 13	23.3	<b></b>	]
1937	{ 1 } 2	{	19 20 26 15	7  11  11  21	}	15. [	19, 2
Delicious:	,	,	20			!	
1936,	1 2	1 h h h	21   19	11   13   9	15,4		
Winesap;	. [	· "	21.	9 []		i	
1037	$\left\{egin{array}{c} 1 & rac{1}{2} & rac{1}$	1 h h	15 16 16 17	0   5   4   6		11.0	

For details of spray treatments, see table 1; a and b refer to tree replicates.
For details of washing treatments, see table 2.

These results with York Imperial are in agreement with those of Frear and Worthley (3) and Hough (9), who reported that York Imperial apples were harder to clean than other varieties.

## RELATION OF TIME OF PICKING TO RESIDUES AT HARVEST AND EASE OF RESIDUE REMOVAL

With delay in harvesting, the residue in grain per pound should decrease both because of the increased weight of the fruit and because of the possible loss of residue from weathering. On the other hand, the ease of removal might decrease because of the development of wax that might tend to cover the residue and thus protect it from the washing solution. In order to determine the extent to which the residue decreases with delay in harvesting and the increased difficulty of removal, three pickings of Stayman Winesap and York Imperial apples were made approximately at 10-day intervals.

The results at different harvesttimes are presented in table 11. There was no reduction in residue during the first interval and only about 12 percent reduction during the second interval. There was a gradual but hardly significant increase in the average size of the apples used in the samples at the different pickings. In some instances there was an apparent decrease in size with time. This would indicate that

representative samples were not obtained at each picking, which may account in part for the lack of decrease in residue. That apples of approximately the same average size showed no decrease in residue between the first and the second picking would also indicate that little or no weathering occurs. With Stayman Winesap in both years and York Imperial in 1937, about 2 inches of rain fell between the first and second pickings (fig. 1) and only 1 inch between the second and third pickings, yet there was a greater loss of residue during the second period. With York Imperial in 1936, 1 inch of rain fell between the first and second pickings and 4 inches between the second and third pickings, yet the loss of residue was nearly the same in both periods. would indicate that rainfall has little tendency to wash the residue from the apples after it has been on for some time. Weber et al. (19) observed that one heavy rain will remove by washing more residue than an equal amount of rainfall coming as several small rains. the other hand, Frear and Worthley (4) found no consistently greater loss of residue when rain fell than during periods free from rain, and they concluded that the weathering away of spray deposits was a negligible factor compared with per-unit losses due to fruit growth. Moreover, Ellenwood et al. (1) reported higher residues in a season of heavy precipitation (1935) than in one of lower precipitation (1936). The results presented herein are in accord with those of Frear and Worthley (4) and Ellenwood et al. (1).

Table 11.—Relation of time of picking to lead residues at harvest.
Residue results expressed as grain per pound of fruit; size of apple indicated by weight in grangle.

		y trent- ent l	First	vicking	Second	picking	Third	picking
Variety and year	No.	Repli- ente	Weight per apple	Residue	Weight per apple	Residue	Weight per apple	Residue
Stayman Winesap: 1936	$\begin{bmatrix} 1 \\ 2 \end{bmatrix}$	n b	Grams	Grain 0, 067 . 073 . 097	Grams 123 133 120	Grain 0.073 .065 .100	Grums 153	Grain 0.068
1937	1 2	} b { n { b } a } b	168 163 144 153 172	. 084 . 069 . 083 . 120 . 104	157 166 143 154 177	.088 .077 .090 .137 .111	176 153 186 165	. 074 . 068 . 103 . 104
Average		- <b></b>		.087		. 093		. 079
York Imperiat:	1 2	{ a b { a b }	96 103 98 09	.076 .083 .108	105 122   98 106	, 061 , 053 , 104 , 006	107 115 106 118	. 056 . 049 . 070 . 070
1937	1 2	1      1)     11     1)	92 161 127 110	.008 .080 .155 .157	99 95 112 127	. 103   104   142   164	96 98 105 101	. 112 . 092 . 158 . 134
A verago			TT (TT 2	108		, 103	· · · ·	. 093
Average, both varieties			124	.097	127	. 098	130	. 086

<sup>1</sup> For details of spray treatments, see table 1; a and b refer to tree replicates.

As indicated previously, the case of residue removal is best indicated by the percentage of the original residue remaining after washing treatments. The data relative to the effect of picking time on residue removal are presented on this basis in table 12. A statistical exami-

nation of these data showed that the varieties responded differently to time of picking. The difficulty of residue removal increased with delay in picking in the case of York Imperial but not with Stayman Winesap. There is a greater tendency for the waxy coating on York Imperial apples to become soft and greasy with late harvest and during ripening than there is on Stayman Winesap, and this may account for the difference in the response of the two varieties.

The use of a wetting agent in a heated washing solution (B-5) greatly increased the residue removal but was of greater benefit with the late-picked York Imperial apples than with the early-picked lots. The use of oil in the second-brood cover sprays had no effect on the relative case of residue removal from early- and late-picked apples.

Table 12. Relation of time of picking to case of lead-residue removal [Results expressed as percent of residue before washing]

	S		trent-	Lend residue v? indicated picking after washing treatment?							
Variety and year		_			H-2		H-5				
· · · · · · · · · · · · · · · · · · ·	Nn.		RepH- rate	First picking	Second picking	Third picking	First picking	Second picking	Third picking		
Stayman Whesap			۲.	Percent	Percent	Percent :	Percent	Percent	Percent		
	{	$egin{array}{c} 1 & \{ 2, \} \end{array}$	a   b   a   b	25 19 25 21	20 20 20 16	23	15 12 9	14 12 7 8	12 9		
1937	1	1 . 2 .	n l b n l h	10 22 18 15	21 20 15 19	20 19 17 17	12 11 7	11 11 12	15 12 1		
Average				21	19	201	10	to	10		
York Imperial:		,									
1936,	{	1 .{ 2 {	n b a	21 10 25 23	34 26   33   31	23 ° 35 40 37	16   1   9   12	18 19 12 13	18 16 14 17		
1937	{	1 { 2 ;{	d b H b	20 1 21 21 20	19 20 26 18	17 ( 18 23 :	13 10 8 8	7 11 11 9	(2 11 8 14		
Average				21	2ft	25	11	19			
Average, both varieties :				21	22	21	11	- 11	12		

<sup>7</sup> For details of spray treatments, see table 1° a and 5 refer to tree replicates, 7 For details of washing treatments, see table 2.

#### RELATION OF DELAY BETWEEN HARVEST AND WASHING TO EASE OF RESIDUE REMOVAL

The development of wax on the fruit proceeds after harvest and is relatively rapid at higher temperatures. Increased difficulty in residue removal has been reported with delay after harvest. In 1936 Stayman Winesap apples picked on October 5 and placed at 32° F. on October 6 were washed on October 7 and 8, at which time similar unwashed samples were placed at room temperature (about 60°) to be washed after 2 weeks. York Imperial apples picked on October 15 were placed in 32° storage. The no-delay (at harvest) washing treatments were given after 1 week at 32°, at which time unwashed samples were left at room temperature and washed approximately 1 and 2 weeks later. The results are given in the first part of table 13.

Table 13. - Relation of delay between harvest and washing to lead-residue removal [Results expressed as percent of residue before washing]

And the second s		migracos addito	Lead residue after indicated delay and washing treatment 2—														
Year and variety	Spray treat- ment 1					Delay at 32° F.			Delay at room temperature for -								
			No delay 3		for 8 days			3 days		7 and 8 days			13 days				
	No.	Repli-	B-2	В-3	В 5	B · 2	13 - 1	B-5	B-2	B-3	B-5	B-2	В-3	B-5	B-2	В-3	B-5
1936: Stayman Winesap	[1 [2	fa 1b fa b	Percent 20 20 20 20 16	Percent 18 17 18 18	Percent 14 12 7 8	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent 27 32 32 33	Percent 26 28 28 20 27	Percent 18 17 10 11
Average			]14	17	10				er generaliser er storgen		ست بدید دروست دروست				31	27. 5	14
York Imperial	${1 \brace 2}$	fa 1b 1a (b	31 26 33 31	28 24 25 26	18 19 12 13							44 36 50 50	36 34 43 45	20 24 17 17	49 47 61 78	43 47 51 60	23 23 20 20
Average			31	26.5	15.5							45	39. 5	19. 5	59	50	21.5
1937: Stayman Winesap	${                                    $	{a h {a b	21 20 15 19		12 11 6 6	19 13 15 17		10 10 7 6	21 20 20 20 22		8 10 7 6	21 18 20 21		9 10 7 6			
Average			19		9	16	The second secon	8	21	1.22.42	8	20		8 .			***************************************
York Imperial	$\left\{egin{matrix} 1 \\ 2 \end{smallmatrix} \right.$	{n {b {n {b	19 20 26 18		7 11 11 9	18 18 1 25 18		12 11 13 9	20 22 32 26		12 12 9	19 14 32 23		10 10 11 8			
Average	erika Vidoji		21		9. 5	20		11	25		10	22		10	Same price desired.		

For details of spray treatments, see table 1; a and b refer to tree replicates.

For details of washing treatments, see table 2.

The no-delay lots were washed as soon as possible after harvest, but this was generally after several days at 32° F., as indicated on p. 18. The delayed lots were given additional delay as indicated.

As in table 12, the data in table 13 are presented as percentage of the original residue remaining and show that a delay of approximately 2 weeks at room temperature greatly increased the difficulty of removal. The effect of delay was much greater on York Imperial apples, the coating of which tends to become more greasy with ripening, than on Stayman Winesap. After approximately 2 weeks' delay there was an average of 79 percent more residue on York Imperial and 56 percent more on Stayman Winesap than when they were washed as soon as possible after harvest. A delay of only 1 week resulted in an intermediate (42 percent) increase in residue on York Imperial. Thus, a delay of 1 week had about as much effect on York Imperial as a delay of 2 weeks had on Stayman Winesap. The type of spray treatment also influenced significantly the effect of delay on case of residue removal. A delay of approximately 2 weeks resulted in 89 percent more residue after washing on apples from spray treatment 2, in which mineral oil emulsion was added to the late cover sprays, whereas the increase was only 52 percent when mineral oil emulsion was not used (spray treatment 1). The effect of delay was much less when a detergent or wetting agent was used, as there was 78 percent more residue when washed with heated acid only (B-3) after 2 weeks' delay than when washed immediately, but only 38 percent more residue when a heated acid wetting-agent wash (B-5) was used.

Because of the great influence of delay on the 1936 results, it was thought advisable in the 1937 experiments to study the effect of shorter periods of delay and to determine whether storage at 32° F. could be used to prevent the adverse effects of holding at room temperatures. Delays of 3 and 8 days at room temperature and 8 days at 32° F, were used, and the results are given in the lower part of table 13. The effect of delay was much less pronounced in 1937 than in 1936. In 1937 the delays had no discernible effect when a heated acid wetting-agent solution (B-5) was used. Holding the apples for 8 days at 32° did not increase the difficulty of residue removal, even when they were washed with acid alone (B-2). delay of 8 days at room temperature did not increase the difficulty of residue removal any more than a delay of 3 days at room temperature. Delays of 3 and 8 days at room temperature resulted in a significant increase in residue over immediate washing and delay at 32° when washed with acid alone at room temperature (B-2). This increase averaged 17 percent. As in 1936, the lots sprayed with a mixture to which mineral oil emulsion was added to the late cover sprays gave a somewhat greater increase with delay (28 percent) when washed with the same solution. The interaction of variety with delay did not indicate any significant difference in the varietal response to delay in 1937 such as was found in 1936.

The cause of the difference in response to delay in the two seasons is not apparent. It may be that climatic conditions in 1937 stimulated an early development of wax, so that little further wax development occurred during the delay periods. The heavier residues at harvest in 1937, and generally the greater difficulty of cleaning as compared with 1936, would tend to support this assumption.

#### RELATION OF SEASON TO SPRAY RESIDUE AT HARVEST

The residues at harvest averaged 0.032 grain per pound (42 percent) greater in 1937 than in 1936 on comparable lots of Stayman Winesap and York Imperial. The total rainfall during the growing season (May 1 until the apples were picked in October) was practically the same for the two growing seasons (fig. 1), so that the rainfall does not appear to be associated with the greater weathering that apparently occurred in 1936. Nor can the difference be attributed to differences in the size of the apples, as in 1937 they averaged as large as or larger than in 1936.

### RELATION OF SPRAY TREATMENTS TO RESIDUES AT HARVEST AND EASE OF RESIDUE REMOVAL

The addition of mineral oil to the last two of five cover sprays (spray 2 versus spray 1) increased the average residue at harvest for the various lots in 1936 and 1937 from 0.0768 to 0.1135 grain per pound, an increase of 48 percent. This is in close agreement with the 49-percent increase reported (6, p, 14) for the 1934 and 1935 seasons.

Certain additional spray treatments were applied in 1936 to Stayman Winesap apples. The addition of mineral oil emulsion to first-brood cover sprays (covers 2 and 3) (spray 5 versus spray 1) resulted in a 14-percent increase in residue at harvest. On the basis of such limited evidence this difference is not significant. When no lime (bordeaux mixture) was used in the last two of five cover sprays (spray 3 versus spray 1), the residue was increased 27 percent. Again the evidence is not sufficient to demonstrate that the difference was

statistically significant.

Additional spray treatments were applied in 1937 to both Stayman Winesap and York Imperial. The addition of mineral oil emulsion to the last two of five cover sprays resulted in a 48-percent increase in residue when bordeaux (2-4-100) was used in all cover sprays (spray 2 versus spray 1) and 102 percent when it was not used in the last 2 sprays (spray 4 versus spray 3). Omitting the bordeaux (spray 3 versus spray 1) did not increase the residue at harvest when oil emulsion was not used in 1937, but the results for Stayman Winesap only in 1936 indicated a 27-percent increase. When mineral oil emulsion was used in the last two cover sprays the omission of the bordeaux (spray 4 versus spray 2) resulted in a 33-percent increase in residue. This is in general agreement with the results for 1935 (6), when the residue was 54 percent higher with bordeaux omitted from the last four of seven cover sprays of lead arsenate with oil emulsion in the last four covers.

The case of residue removal as shown by the percentage of the original residue remaining after washing may also be influenced by the types of spray and of washing treatments. Although the addition of mineral oil emulsion to late cover sprays increased the lead residues at harvest, the percentage of the original residue removed was also greater, particularly with the more effective washing treatment (B-5) and with the Stayman Winesap variety. Thus, with the more effective washing treatments the residue in grain per pound on oil-sprayed lots was as low as or lower than on comparable lots without oil.

On the other hand, when no oil was used the omission of bordeaux (lime) (spray 3) from the late cover sprays did not significantly increase the lead residues at harvest, but as a lower percentage of the original residue was removed, the residues on such lots were appreciably higher after washing than they were on comparable lots (spray 1) in which bordeaux was used throughout. The omission of bordeaux from late cover sprays of lead arsenate and oil (spray 4) not only increased the residues at harvest but also reduced the percentage removal when washed, particularly on the York Imperial apples, so that residues after washing were much higher with this type of spray treatment than they were with any of the others. This type of spray treatment is more common in the Pacific Northwest. The higher residues with the same number of sprays in the Pacific Northwest and the greater difficulty in removal compared with the central and eastern regions have been attributed to greater weathering because of rainfall in the central and eastern regions. These results indicate that the differences may be due rather to the difference in the type of spray program generally used in the different regions, since rainfall apparently has very little weathering effect, whereas type of spray treatment may have a great effect.

# Relation of Washing Treatments to Lead-Residue Removal influence of acid concentration

Lots from the different spray treatments on Stayman Winesap in 1936 were washed with 0.5 percent HCl at room temperature in a flotation machine (fig. 2 and table 3, washing treatment B-1). After this relatively simple washing treatment an average of 27.2 percent of the original residue remained. The washing treatment reduced the residue on the apples from all of the spray treatments to appreciably below the present lead tolerance of 0.05 of a grain per pound of fruit. This percentage of residue remaining is in fairly close agreement with the average of 31.5 percent obtained in 1934 but considerably lower than the 46 percent average reported for 1935 (6, p. 29).

When the apples were washed with 1.5 percent acid (B-2) instead of 0.5 percent acid (B-1) there was 28.2 percent less residue remaining than when they were washed with the weaker solution. Similar benefits of 37.5 and 30.5 were reported (6, p, 29) for the 1934 and 1935 seasons, respectively.

#### INFLUENCE OF TEMPERATURE OF WASHING SOLUTION

Various lots of apples were washed with 1.5 percent HCl with the washing solution at room temperature (60° to 70° F.) and at 100°, in a flotation washer for 60 seconds (treatments B-2 and B-3), in a flood washer for 35 seconds (treatments C-1 and C-2), in a flood-brush washer for 35 seconds (treatments D-1 and D-2), and with an acid wetting-agent solution in a flotation machine for 60 seconds (treatments B-4 and B-5). In 34 comparisons with acid alone in a flotation machine there was an average of 23.4 percent of the original residue remaining after washing at room temperature (B-2) and 20 percent after washing at 100° (B-3). Thus the percentage residue was 14.5 percent lower when the apples were washed with the heated solution. The benefit from heating was considerably greater with an acid wetting-agent solution in a flotation machine (B-4 versus B-5) than with acid alone. This comparison was possible only with Stayman Winesap

in 1936, when the benefit from heating the acid wetting-agent solution (B-4) versus B-5) averaged 22.6 percent, as compared with only 12.3 percent for comparable lots with acid alone (B-2) versus B-3). These results are in agreement with those for the two previous seasons (6, p.21), in which an average benefit of 26.3 percent was found from heating acid wetting-agent solutions compared with a benefit of 12.8 percent from heating an acid solution.

With 18 and 20 comparisons there was an average benefit from heating an acid solution of 21.3 percent in a flood washer (C-1 versus C-2) and 22.6 percent in a flood-brush machine (D-1 versus D-2). The benefit from heating an acid solution was apparently greater in flood or flood-brush machines than in flotation machines, in which the benefit from heating was only 14.5 percent, but the lots were not entirely comparable with those used in the flood and flood-brush machines.

#### COMPARISON OF TYPES OF WASHING MACHINES

For most of the washing treatments a flotation-type machine was used, but certain lots were given various washing treatments in flood and flood-brush machines. Some of these treatments were similar to those used in the flotation-type washer except that the time of exposure to the washing solution was shorter than in the flotation machine.

In 1936 the percentage of the residues remaining averaged 22 percent lower with Stayman Winesap and 17 percent lower with York Imperial when washed in a flood (C-1 and C-2) instead of in a flotation machine (B-2 and B-3). In 1937 this difference (B-2 versus C-1) averaged about 43 and 32 percent for Stayman Winesap and York Imperial, respectively. The most effective washing treatment for a flotation machine consisted of a heated acid wetting-agent solution (B-5), and the most effective and practical treatment in flood-type machines consisted of a heated acid solution to which a very light mineral oil (viscosity about 50 Saybolt seconds) was added (C-4 and It is of interest, therefore, to compare these treatments in the muchines to which they are adapted. In 1936 the heated acid-oil combination in a flood-brush machine (D-4) was considerably more effective than the heated acid wetting-agent combination (B-5) in a flotation machine (fig. 2). However, in 1937 there was no significant difference between the acid-oil combination in a flood (C-4) or floodbrush (D 4) machine and the acid wetting-agent solution (B-5) in the flotation machine (fig. 3).

Stayman Winesap apples in 1936 averaged 13 percent less residue when washed in a flood-brush instead of in a flood machine. However, there was no significant difference between the machines in 1937, when both Stayman Winesap and York Imperial apples were used.

The results are in general agreement with those of Hough (10), who stated that there was no practical difference in efficiency of the three types of washers when washing fruit that carried residues not especially difficult to remove, but that the flood-brush machine was more effective for lead-oil-sprayed apples.

#### EFFECT OF ADDITION OF WETTING AGENT TO ACID WASHING SOLUTION

Stayman Winesap apples in 1936 were washed with the acid solution at room temperature and at 100° F, both with (B-4 and B-5) and without (B-2 and B-3) the addition of 1 percent of a wetting agent

(Vatsol). The results in table 3 and figure 2 show that when the wetting agent was used the percentage of residue after washing averaged 30.5 percent lower than when no wetting agent was used. As reported previously (6, p. 23), the addition of the wetting agent to the acid solution increased its effectiveness for all types of spray treatments used and at all temperatures of the wash solution. However, in agreement with the earlier results (6, p. 23), the wetting agent was more effective in heated solutions (B-5 versus B-3) than in cold solutions (B-4 versus B-2) and with oil-sprayed than with non-oil-sprayed lots, and was less effective with lots in which the bordeaux (lime) was omitted from the late cover sprays. These results are in general agreement with those of Schrader and Haller (17) in which additional wetting agents were used.

With York Imperial and Delicious apples the wetting agent was used only in heated acid solutions in 1936. The residue percentages remaining after washing were 39 and 41 percent lower on York Imperial and Delicious, respectively, when the acid wetting-agent solution (B-5) was used than when acid alone (B-3) was used. The response to the wetting agent was much greater when the apples were from oil-sprayed lots than from non-oil-sprayed lots. With York Imperial washed at harvest the average benefit was 25 percent with non-oil-sprayed lots, whereas it was 51 percent with oil-sprayed lots. The response was also greater when the apples were ripened before washing (54 percent benefit) than when they were washed immediately after harvest (41 percent benefit).

When only 0.25 percent of a wetting agent was added to a flood-type washer (treatment C-3) or to a flood-brush machine (treatment D-3) it caused considerable foaming that was difficult to control with De Gras autifoaming agent. This acid wetting agent combined with the antifoaming agent was no more effective than acid alone (C-2 and D-2) when used in a heated solution with Stayman Winesap

and York Imperial apples in 1937 (tables 6 and 7).

During the 1937 senson the wetting agent was used in the flotation-type machine only in heated acid solution (B-5), and acid alone was used only at room temperature (B-2). The average benefit from both heating the solution and adding the wetting agent (B-5 versus B-2) averaged 53 percent for all lots in 1937 and 52 percent for those in 1936. These results are in good agreement with the 51 and 43 percent reported (6, p. 24) for the 1935 and 1934 sensons, respectively. The agreement is particularly good, since the spraying and certain other treatments, such as delay before washing, differed considerably in the different seasons, and these factors have been shown to influence the response to wetting agents.

#### EFFECT OF ADDITION OF MINERAL OIL TO ACID WASHING SOLUTION

The addition of a very light mineral oil has been found to increase the effectiveness of heated acid washing solutions in flood-type washers on apples grown in the Pacific Northwest (18). This treatment (C-4 and D-4) was used in flood or flood-brush machines with certain of these lots of apples (figs. 2 and 3 and tables 3, 4, 6, and 7). The results with mineral oil have not been consistent. In 1936 Stayman Winesap apples (table 3) carried 32 percent less residue when oil was added to the acid solution (D-4) than when the acid

was used alone (D-2) in a flood-brush machine. Limited results with York Imperial in 1936 (table 4) indicated an average benefit of 39 percent, which, however, occurred only in the oil-sprayed lot. On the other hand, in 1937, the use of oil in the washing solution did not significantly influence residue removal from either Stayman Winesap or York Imperial apples when washed in either a flood or a flood-brush machine. Results for 1935 indicated a benefit from oil in the washing solution only when the apples were sprayed with oil emulsion (6, p. 24).

#### RELATION OF WASHING TREATMENTS TO KEEPING QUALITY OF APPLES

The York Imperial variety is rather subject to cracking or checking of the skin of the fruit while still on the tree. This growth checking is very similar to heat injury from heated washing solutions, except that when it does not occur too near to harvest the growth cracks may be healed over. In the 1936 season considerable injury of this kind occurred near harvest on York Imperial apples grown near Hancock, Md. A study was made with these apples to determine whether the checking was increased by washing and whether washing

increased decay or shriveling in apples with growth checks.

A lot of apples was sorted into three classes according to degree of checking, and samples of each class were washed as indicated in table 14. As was to be expected, the loss in weight during storage and the extent of shriveling increased with increased severity of checking. The amount of decay was also much greater in apples with severe checking, but there was no consistent difference between apples with slight to medium checking and those with no checking. The washing treatments had no apparent effect on the severity of shriveling or the amount of decay in these lots, and it did not significantly affect the firmness of the apples.

Table 14.— Relation of washing with acid and with sodium silicate to the shriveling and keeping quality of York Imperial apples stored at 32° F. from Oct. 24, 1936, to May 27, 1937

	i		Condition at end of storage period							
Washing treatment and skin checking at start	Total apples in sample	Loss in weight during	Shriv	celing						
	sample	storage	None to slight	Medium to severe	Deeny	Pressure test				
Not washed: None Slight to medium Severa Wushed with HCU	Number 188 244 159	Percent 3. 1 4. 3 7. 6			Percent 1, 1 3, 3 21, 4	Pounds				
None Slight to medium Severe Washed with sodium silicate <sup>1</sup>	104 286 133	3. 3 4. 6 8. 0	89. 7 80. 8 17. 3	3, 0 14, 3 63, 2	7.3 4.9 19.5	15.3				
None. Slight to medium Severe	139 250 148	3.3 5.3 9.8	92. 8 62. 8 7. 4	5, 0 34, 8 74, 4	2.2 2.4 18.2	14.4				

 $<sup>^4</sup>$  Washed with 1.5 percent HCl  $\pm$  1 quart of Aresket wetting agent to 100 gallons  $\pm$  De Gras antifoam at 100° F. for 30 seconds in flood-type washer.  $^4$  Washed with sodium silicate 60 pounds to 100 gallons at 110° F. for 20 seconds in flood-type washer.

Data on the effect of temperature of the washing solution on heat injury and keeping quality of York Imperial apples are presented in It has been reported (13) that sodium silicate solutions can be used at higher temperatures than acid solutions without injury to the fruit; consequently the temperatures of the sodium silicate solution used extend somewhat higher than those of the acid wetting-agent At the inspection of the apples on February 5 the results did not show any marked difference between the two types of solutions in the extent of injury (checking or checking and shriveling) at the same temperatures (110° and 115° F.). Even at the lowest temperature used (100° for 30 seconds) there was apparently an increase in the amount of skin checking as compared with the unwashed lot. With the acid wetting-agent solution there was a gradual increase in the amount of checking with increased temperature. On the other hand, when the apples were washed with sodium silicate solution there was no apparent increase in checking with increased temperature except at the highest temperature (125°). These York Imperial apples after being held at 32° until May 27, considerably beyond their main marketing period, were then post-ripened for 1 week at 70°. Severe decay developed in all lots, but it was not consistently greater in washed than in unwashed lots. Nor was there any consistent relation between the temperature or type of the washing solutions and the percentage of the apples with decay and scald. Although the washed lots were all less firm (as indicated by pressure test) than the unwashed lots, the differences are of questionable significance and of no practical value, as all lots were sufficiently firm for marketing purposes.

Table 15.—Relation of washing solutions and temperatures to keeping quality of York Imperial apples stored at 32° F. from Oct. 27, 1936, to Feb. 5, 1937, and May 27, 1937, and post-ripened at 70° until June 3, 1937

	95.50	ü	Con	dition	Feb. 5,	1037		Condition June 3, 1937					
Washing solution	Temperature	Total opples	Sound	Checked	Checked and shriveled	Decayed	Sound or	Deenyed	Sealded	Shriveled	Break-down	Pressure test	
None  ## Non	9 F. 100 105 110 115 110 115 120 125	No. 137 124 120 137 130 132 145 145 131	Pat. 36.0 17.0 14.5 13.8 1.5 6.0 10.3 8.8 3.7	Pct. 64.0 81.6 82.4 85.5 04.7 02.0 86.9 82.4 79.3	Pcl. 0 0 7 3.8 7.4 14.1	Pct. 0 .8 3.1 0 .7 0 1.4 3.0	Pct. 24.8 21.0 39.0 20.5 23.8 10.7 12.4 25.5 11.6	Pct. 71.6 75.0 52.3 75.9 08.5 71.2 79.3 68.3 81.5	Pct. 3.0 4.0 7.0 6.9 5.3 5.1 0	Pct. 0 0 0 8 3.59 1 4.9	Pct. 0 5 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	Lbs. 10. 0 15. 1 15. 0 15. 2	

<sup>1.6</sup> percent HCl+wetting agent (1 quart to 100 gallons)+sufficient antifoam to control the foam. Exposure to wash solution for 30 seconds in flood-type washer (old solution used 1 day, commercial ran), i.B. W. sodium silicate (95 pounds to 100 gallons)+soap (M pound to 100 gallons)+light mineral oil (1 gallon to 100 gallons). Exposure to washing solution for 30 seconds in flood-type washer (fresh solution).

Additional studies on the relation of washing treatments to keeping quality of York Imperial and Stayman Winesap apples were made in 1937, and the results are presented in tables 16 and 17. The York Imperial apples (table 16) were held beyond their main marketing season but showed very little decay when removed from 32° F. storage. However, the decay developed rapidly at room temperature, so that

about 57 to 87 percent of the fruit was decayed after 2 weeks of postripening. There was no consistent relationship between the amount of decay in washed and unwashed lots or between lots washed at the different temperatures. In the flood-brush machine there was some indication that storage scald increased with the temperature of the acid washing solution but decreased with the temperature of the acid-oil combination. The percentage of scald was also high when the apples were washed with an acid solution only, at room temperature, in the flotation machine. The loss of weight in these apples during storage and post-ripening (table 16) was not large and apparently did not differ consistently with the various washing treatments. The loss in unwashed fruit averaged slightly less than in the washed lots, and the loss was somewhat greater in the lots washed with acid and oil than in those washed with acid alone. These differences might have been accentuated had the fruit been stored under conditions more favorable for moisture loss. Under such conditions Marshall et al. (12) found that the addition of mineral oil to a hydrochloric acid washing solution resulted in a substantial increase in moisture loss. The riponess of the apples as indicated by pressure test did not differ significantly among these lots.

Results with similar washing treatments on Stayman Winesap apples are shown in table 17. The various washing treatments did not have any apparent influence on the development of decay or the loss in weight of these apples. Nor was there any consistent difference in scald development except possibly a reduction in scald when the apples were washed with heated solutions at the higher temperatures used, particularly when oil was added to the acid solution. This relationship was indicated by the York Imperial apples also. The washing treatments had no effect on the ripeness of the apples as

indicated by pressure test.

#### DISCUSSION

At the time these investigations were conducted the regulatory tolerances for lead and arsenious oxide (As2O3) were 0.018 and 0.010 of a grain per pound of fruit. Thus the ratio of lead to arsenious oxide in the tolerances (1.8) was lower than the ratio in the lead arsenate spray material (2.1). It was assumed that this ratio in the spray residue deposit remained the same as in the original spray material, both at harvest and after washing with hydrochloric acid or sodium silicate solutions. If this were so, then any washing treatment that effectively removed the lead to below its tolerance would also be effective for the arsenic. For this reason lead only was determined in these experiments, since it seemed more important and could be determined more accurately. More recently (August 1940) the Federal lead tolerance was raised to 0.050 of a grain per pound and the arsenic tolerance to 0.025 of a grain of As2O3 per pound of apples or pears, so that the ratio of lead to arsenic in the tolerance was slightly less than this ratio in the spray material. (See discussion of ratio of lead to arsenic on pp. 2 to 4.)

Varietal differences in the retention of residue have been reported. These have been based generally on the apples representing the average size for the varieties. Practically the smallest size packed must meet the tolerance, and regulatory samples usually consist of the smallest size apples. On this basis the size of apples in the samples

Table 16.—Relation of washing treatments to keeping quality of York Imperial apples stored at 32° F. from Oct. 29, 1937, to Apr. 15, 1938, and then post-ripened at 70° until Apr. 29, 1938

Washing treatment					Condition on indicated dates										
Solution <sup>1</sup>	Time	Tem-		Tem-	Machine	Total apples in samples	Loss in	weight	Sot	and	Dec	ayed	Scal	ded 2	Pressure test
	ture			Apr. 15	Apr. 29	Apr. 15	Apr. 29	Apr. 15	Apr. 29	Apr. 15	Apr. 29	Apr. 29			
Not washed	60 60 30 30	65 100 100 100 110 110 120 120	Flotationdo Flood-brushdo dodo do dodo do dodo		Percent 1. 6 1. 8 1. 9 1. 8 1. 7 2. 0 1. 8 2. 1 1. 7 2. 4	Percent 2. 7 3. 1 3. 8 3. 7 3. 2 4. 1 3. 2 3. 9 3. 3 3. 6	Percent. 93. 7. 93. 5. 74. 8. 93. 9. 92. 2. 86. 7. 85. 0. 90. 9. 72. 5. 96. 3	Percent 25. 5 3. 2 2. 4 13. 6 8. 6 9. 4 6. 7 5. 9 11. 0 24. 5	Percent 0 0 1.6 2.0 .6 3.5 .6 .6 2.0 3.1	Percent 56. 6 74. 1 87. 4 69. 4 78. 3 78. 4 81. 1 77. 8 68. 2 57. 7	Percent 6.3 6.5 23.6 4.1 7.2 9.8 14.4 8.5 25.5	Percent 17. 9 22. 7 10. 2 17. 0 13. 1 12. 2 12. 2 16. 3 20. 8 17. 8	Pounds 14.8 15.6 15.3 15.1 15.4 14.8 15.2 15.4		

HCl at 1.5 percent. Oil at 1 gallon per 100 gallons.
 Packed without oiled paper.

Table 17.—Relation of washing treatments to keeping quality of Stayman Winesap apples stored at 32° f'. from Oct. 10, 1937, to Jan. 3, 1938, and then post-ripened at 70° until Jan. 10, 1938

						Condition Jan. 10, 1938							
Solution †	Time	Tem- pera- ture	Machine	Total apples in sam- ples	Loss in weight	Sound	De- cayed	Scald- ed 2	Pres- sure test				
None Do Do HCl Do Do HCl Do HCl Do HCl Do HCl Holl HCl HCl HCl HCl HCl HCl HCl HCl DCl To HCl	30 30 30 30 30	65 160 160 160 110 110 120 120	Flotation. do flooti brush do do do do do do do	Number 117 95 99 95 96 98 112 91 86 102	Percent 1.0 2.0 1.0 1.7 1.7 1.5 2.2 1.6	83. 8 86. 3 88. 9 79. 0 92. 9 93. 0 93. 2 96. 7	Percent 0.8 0 2.0 1.0 1.0 0 1.1 0 2.9	Parcent 15.4 13.7 9.1 29.0 6.1 7.0 6.8 2.2 0	Pounds 11.3 11.1 11.3 11.1 11.3 11.1 11.6 11.6				

<sup>1</sup> HCl=1.5 percent; olf. 1 gallon to 100 gallons. 2 Packed without oiled paper.

from different varieties probably would not vary greatly, even though

the average size would. In comparing varieties, therefore, it would seem desirable to base the comparison on apples of the same size. On this basis, apples of the York Imperial variety retained less residue at harvest than did Stayman Winesap and Delicious, whereas on the basis of the average size the York Imperial apples retained more

residue.

The results emphasize the interrelation of a number of factors, such as variety, season, spray treatments, maturity and ripeness of the fruit, and type of washing treatment in the removal of lead spray residues. For example, varieties may respond differently to spray treatments; the use of mineral oil emulsion in the cover sprays increased the residue on Delicious to a greater extent than on certain other varieties. Furthermore, the effectiveness of a washing treatment may vary with the condition of the fruit and the type of spray treatment applied. The addition of a wetting agent to an acid washing solution, for example, increased the effectiveness of the solution to a greater extent with riper fruit and with fruit that was sprayed with oil emulsion than with unripe fruit or fruit not sprayed with oil emulsion.

#### SUMMARY AND CONCLUSIONS

Several varieties of apples were given different spray treatments during two seasons. Pickings were made at different times, and different washing treatments were given all lots. Lead determinations were made on washed and unwashed samples. Certain lots were held in storage after the various washing treatments, to determine the effect of the treatments on storage quality.

The variation in lead residues among tree replications was less than reported previously (5) and was due largely to differences in the size of the apples. For each 1-percent increase in weight per apple there was an average decrease in residue of about 0.8 percent, both

before and after washing, on replicate lots. York Imperial apples averaged much smaller than Stayman Winesap apples, and because of their small size they gave higher residue values at harvest. apples of equal size there were lower residue values on York Imperial than on Stayman Winesap and Delicious and lower on Winesap than on Stayman Winesap.

After washing, York Imperial apples retained a greater percentage of the original residue than Stayman Winesap, Delicious, or Winesap, and Stayman Winesap retained somewhat more than Winesap.

Growth of the apples and weathering apparently had very little effect on the reduction of residue during the harvest period. evidence is presented that indicates that rainfall has little tendency to wash the residue from the apples, particularly during the harvest period.

The percentage of the residue removed by washing was not affected by delay in the harvesting of Stayman Winesap apples but was reduced by delay in the harvesting of York Imperial apples.

The general effect of delay between harvest and washing was to increase the difficulty of residue removal but varied with season, variety, spray treatment, washing treatment, temperature at which the apples were held during the delay period, and length of the delay The waxy conting on York Imperial apples tends to become greasy during ripening, and the effect of delay (ripening) on residue removal was greater in York Imperial than in Stayman Winesap The difficulty of residue removal was increased more by delay when the apples were sprayed with mineral oil emulsion in the late cover sprays than when no oil emulsion was used, possibly because of a stimulating effect of oil sprays on wax development during ripening. The effect of delay was less when a wetting agent or detergent that would tend to remove some of the waxy coating was used in the washing solution. A delay of I week at 32° F, did not increase the difficulty of residue removal.

The addition of mineral oil emulsion to the second-brood cover sprays of lead arsenate and bordeaux mixture increased the residue at harvest nearly 50 percent. However, the percentage of the original residue removed by the washing treatments was greater in the lots sprayed with mineral oil emulsion, so that with the more effective washing treatments (heated acid wetting-agent solutions) the residue remaining on oil-sprayed lots after washing was as low as or lower than that on non-oil-sprayed lots.

The addition of mineral oil emulsion to first-brood cover sprays did not significantly increase the residue at harvest or its removal.

The omission of bordeaux mixture (2-4-100) from the secondbrood cover sprays of lead arsenate did not significantly increase the residue at harvest but did increase the difficulty of residue removal, so that a considerably heavier residue remained after washing. omission of bordeaux mixture from second-brood sprays of lead arsenate and mineral oil emulsion greatly increased the lead residue at harvest and reduced the percentage of residue removed by washing, so that the residues after washing were much higher with this type of spray treatment than when mineral oil emulsion was used with bordeaux or when bordeaux was used without oil emulsion.

Sixty different lots of apples representing different varieties and spray treatments have been washed with 0.5 percent HCl in a flotation machine with an exposure of 1 minute to the acid at room temperature. The mean percentage of the original residue remaining during three seasons (1934, 1935, and 1936) was 33.6±1.2 (standard error).

Increasing the acid concentration to 1.5 percent resulted in an average benefit or additional removal of about 32 percent of the residue present after washing with the weaker concentration, leaving a residue of about 23 percent of the original residue (unwashed).

Heating the 1.5 percent acid solution to 100° F, resulted in nearly 15 percent greater effectiveness and left a residue of about 20 percent of the original residue. A much greater benefit from heating was obtained with a solution of acid and wetting agent than with acid alone.

Heating an acid solution to 100° F. in a flood or flood-brush machine resulted in a considerably greater benefit than in a flotation

machine.

With the same acid concentration a flood machine with an exposure of 35 seconds was considerably more effective than a flotation machine with an exposure of 60 seconds. With the most effective washing treatment for each type of machine there were no very large or consistent differences in the effectiveness of the different types of machines. Nor was there any large or consistent difference between flood and flood-brush machines.

In a flotation machine the addition of 1 percent of a wetting agent to the acid solution greatly increased the effectiveness of the wash for all types of spray treatments and for all varieties of apples.

The wetting agent was more effective in heated solutions than in cold solutions, with oil-sprayed than with non-oil-sprayed lots, and with bordeaux-sprayed than with non-bordeaux-sprayed lots.

In a flood-type machine the addition of a wetting agent, at the concentration possible and with an antifoam agent, did not increase

the effectiveness of the washing treatment.

The addition of a light mineral oil to heated acid solutions in flood-type machines has generally increased the removal of lead from the apples when oil emulsion was used in the cover spray but has not been consistently effective when oil emulsion was not used in the sprays.

Washing treatments using heated solutions did not increase the amount of spoilage in York Imperial and Stayman Winesap apples during storage. Also, such washing treatments did not affect the

firmness of the fruit or appreciably increase the wilting.

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