

## RESEARCH PAPER RP1196

Part of Journal of Research of the National Bureau of Standards, Volume 22,  
April 1939

## SOLUBILITY OF COLORED GLAZES IN ORGANIC ACIDS

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## ABSTRACT

The purpose of this study was to determine primarily which, if any, of the glazes tested constituted a health hazard because of lead or other toxic oxides which might be dissolved from them and, secondarily, to develop suitable test procedures for future control work.

A conference of representatives of the U. S. Potters Association, the Food and Drug Administration of the U. S. Department of Agriculture, and the National Bureau of Standards was held, and a series of accelerated and simulative service tests were outlined. These tests were made on specimens from six cooperating manufacturers.

It was shown that the amount of lead extracted by pouring hot distilled vinegar (5-percent acetic acid) or a 0.5-percent solution of citric acid into a cup and leaving it to cool in contact with the glaze for  $\frac{1}{2}$  hour would be as much or more than the lead normally extracted by jams and fruit juices. With the same or a similar test procedure, a citric-acid solution of 10-percent strength may be required to indicate how much lead might be taken up by lemonade or vinegar.

The results indicate also that glazes of only one color among those tested constitutes a probable health hazard. However, two other glazes from one manufacturer were found to be marginal, and a third may cause trouble if indicated corrective measures, such as a preliminary acid wash, should be neglected by the manufacturer.

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## I. INTRODUCTION

Accounts of the earliest investigations and regulations regarding the toxicity of lead in ceramic glazes have been summarized by Mellor<sup>1</sup> and by Koenig.<sup>2</sup>

More recently, investigations in Massachusetts<sup>3</sup> established that water containing as little as 0.1 part per million of lead produced symptoms of poisoning, and Jackson and Jackson<sup>4</sup> found that 1.7 parts per million of lead in cider gave acute symptoms of poisoning to the consumers. Considerable helpful information on the contamination of foods may be found in a report by G. W. Monier-Williams,<sup>5</sup> and W. P. Mason<sup>6</sup> stated that in the writer's opinion, no water to be

<sup>1</sup> J. W. Mellor, *The durability of pottery frits, glazes, glasses, and enamels in service*, Trans. Eng. Ceram. Soc. **34**, 113 (1934-35).

<sup>2</sup> J. H. Koenig, *Lead frits and fritted glazes*, Ohio State Eng. Exp. Sta. Bul. 95 (July 1937).

<sup>3</sup> Wade Wright, C. O. Sappington, and Eleanor Rantoul, *J. Ind. Hygiene* **10**, 234 (1928).

<sup>4</sup> M. C. N. Jackson and L. N. Jackson, *The Lancet*, **223**, 717 (1932).

<sup>5</sup> Report on Public Health and Medical Subjects No. 29 of the Ministry of Health, London (1925).

<sup>6</sup> Examination of Water (J. Wiley & Sons, Inc., New York, N. Y.).

used for drinking purposes should contain lead compounds in quantity greater than what would correspond to 0.5 part per million of metallic lead. Recently the U. S. Department of Agriculture established the tolerance of 0.025 grain of lead per pound of food (equivalent to about 3.57 parts of Pb per million parts of food), based upon investigations made by the U. S. Public Health Service. There is reason to believe, however, that a tolerance for "lead" in solid foods may be too lenient for application to lead ingested in solution as the salt of an organic acid. Therefore, the tolerance of 0.5 part per million in drinking water should not be lost sight of.

The present investigation was occasioned by a specific case of nausea believed to have been caused by tea flavored with lemon juice and drunk from a green cup, which came to the attention of the Food and Drug Administration. The matter was referred to the National Bureau of Standards, and a conference, attended by representatives of the U. S. Potters Association, the Food and Drug Administration, and the National Bureau of Standards, was held on May 21, 1938. A series of tests, to be made in cooperation with six manufacturers, was outlined, and the results of these tests are here reported.

## II. PURPOSE

The purpose of the work was threefold: (a) to develop test procedures for the treatment of the ware and for the analysis of the dissolved materials; (b) to correlate the results of relatively rapid and standardizable laboratory tests with those obtained under service conditions; and (c) to determine if any of the representative glazes submitted might constitute a health hazard.

## III. MATERIALS AND METHODS

Bowls, fruit dishes, and cups were submitted by the 6 cooperating companies, and usually 1 dozen specimens of each shape and color were available. Four colors (tangerine, yellow, bright green, and cobalt blue) were submitted by each manufacturer, but additional colors and shades were submitted by some. The maximum number from one concern was 20 colors.

A number of test methods were tried, but all except those given in table 1 were discarded. The discarded methods included heating on a steam bath or holding at "room temperature." Both involve temperature conditions difficult, if not impossible, to duplicate under ordinary conditions. The symbol accompanying each test was adopted for convenience in designating that test in the figures.

Unless otherwise designated in the table, the lead dissolved was determined by extraction with diphenylthiocarbazone, called "the dithizone method."<sup>7</sup> In the case of fruit juices and jams it was necessary to ignite and take up the residue in acid. Determination by precipitation as the sulfide with H<sub>2</sub>S followed customary chemical procedure. The amount of lead present was approximated by colorimetric comparison with standards containing 0.5, 1, 2, 4, and 6 parts of Pb per million parts of solution. If more than 6 parts per million were present in the test solution, an aliquot portion was used.

<sup>7</sup> The procedure used was essentially that in *Method for determining lead in food*, J. Assn. Official Agr. Chem. 17, 123 (1934), separate copies no longer available, and in *Method for determining lead in canned food*, J. Assn. Official Agr. Chem. 18, 315 (1935). Copies are obtainable from the Association (Box 540, Benjamin Franklin Station, Washington, D. C.).

TABLE 1.—Test methods

[Methods 1, 2, 10, 14, 15, and 16 are primarily for laboratory work; methods 3 to 9, inclusive, and 11 to 13, inclusive, were designed to simulate conditions in service, i. e., home or restaurant use]

Test number	Symbol	Method	Test number	Symbol	Method
1	{WV 1/2 H <sub>2</sub> S	A cup or fruit dish, into which 150 ml of boiling white vinegar (about 5-percent acetic acid) had been poured, was covered with a 3-liter beaker to protect it from drafts. After 1/2 hour the vinegar was removed to a Pyrex flask and subjected to the H <sub>2</sub> S test for lead.*	9	{WV RT 2 da	Same as test 7, except that the specimens were held at room temperature 85°±5° F.
	2		{WV 1/2	Same as test 1, except that lead is determined by the dithizone method.†	
3		{PJ 70° 2 da	A cup containing 150 ml of unsweetened pineapple juice was kept for 2 days in a room maintained at 70° F.		11
	4	{GFJ 70° 2 da		Same as test 3, except that unsweetened grapefruit juice was used.	12
5		{1/2 CAc 70° 2 da	Same as test 3, except that 0.5 percent of citric acid was used to simulate lemonade.		13
	6	{BV 70° 2 da		Same as test 3, except that brown vinegar (4.4-percent acidity) was used.	14
7		{WV 70° 2 da	Same as test 3, except that distilled vinegar (5-percent acidity) was used.		15
	8	{WV 40° 2 da		Same as test 7, except that the specimens were held 2 days in a refrigerator at 40° F.	16

\* This test may give too high values for lead because other metals, such as copper, which may be present also, are precipitated. The test was suggested to the industry for control and development work shortly after the conference held in May 1938 and became known as the National Bureau of Standards Tentative Test. It is described in the National Bureau of Standards Technical News Bulletin 255, July 1938.

† The dithizone extraction, followed by electrolysis, is a specific test for lead, except in the presence of bismuth. The dithizone method was used in all of the tests except Nos. 1, 10, 14, and 15.

Comparison standards prepared with vinegar could be used for about 2 hours at most. In this connection, it was found that both test and comparison solutions prepared with acetic acid or citric acid were not satisfactory. The nature of the precipitate imparted a "smoky" color to the solutions and made comparisons difficult or impossible. The condition was corrected by diluting these acids with a 0.25-percent solution of gelatin. Resultant test liquids and standards containing up to 6 parts of Pb per million parts, could be compared satisfactorily. Furthermore, the standards remained constant in color for as much as 2 days.

Copper was determined by "carbamate extraction", using sodium diethyldithiocarbamate, and the procedure followed by the U. S. Bureau of Fisheries.<sup>8</sup> The values are believed to be significant to 0.1 part per million. The following method, giving results significant to 1 part per million, also was used. A 150-ml portion of test solution was evaporated to dryness, ignited, evaporated with HNO<sub>3</sub>, and treated with 0.5 ml of NH<sub>4</sub>OH in a porcelain crucible. The shade of blue obtained was compared with those of standards prepared in the same way.

No test was developed for antimony, because it was evident from results for the yellow glazes that a total of not more than 0.5 part of

<sup>8</sup> E. J. Coulson, J. Assn. Official Agr. Chem., 20, 178 (1937).

material per million parts of reagent was dissolved by the severest tests.

Blank determinations for lead and for copper indicated that the white vinegar used in the tests (table 1) contained 0.3 and 1.0 part per million, respectively, and that the brown vinegar contained not more than 0.1 part of lead per million parts of vinegar.

#### IV. RESULTS AND CONCLUSIONS

The results are presented in figures 1 to 7, inclusive, and some of the values are plotted in several graphs to facilitate comparison and

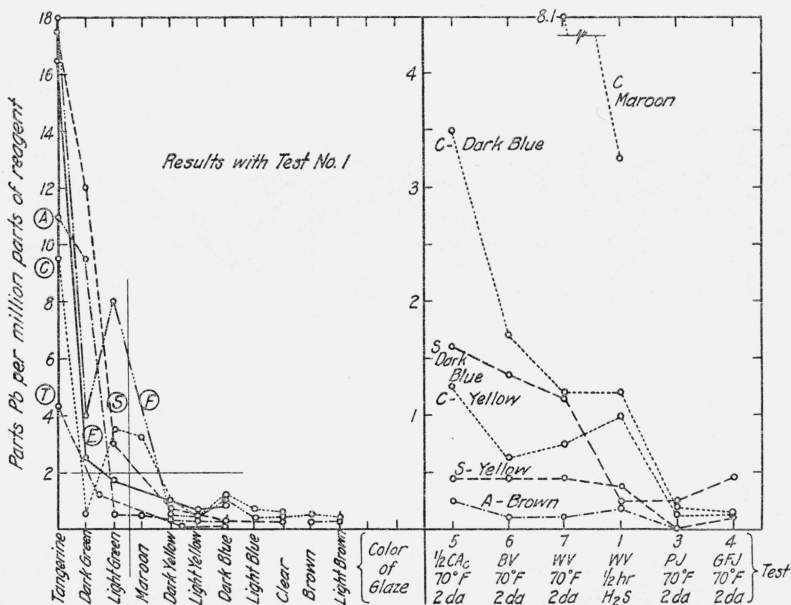


FIGURE 1.—(Left) effect of test 1 (see table 1) on glazes in 11 colors from 6 manufacturers; (right) effect of tests 3 to 7, inclusive, on glazes of relatively low solubility according to test 1.

In this figure, as in the others, the letters A, C, E, F, S, and T refer to the manufacturers of the ware tested.

correlation with the various factors involved. Each plot represents the average of at least two determinations, unless otherwise stated.

Data obtained with test 1 on cups in various colors from the cooperating manufacturers are presented in figure 1 (left). Values for some intermediate shades of yellow, brown, blue, and gray (or "white") are omitted, but in no case did they exceed 0.5 part per million. With the exception of one manufacturer's maroon glaze, the only specimens giving up more than 2 parts per million of Pb were the tangerines (reds) and the greens.

Some of the dark blues and yellows showed solubilities equal to or in excess of 1 part per million. These colors, together with the maroon mentioned and a brown, were subjected to tests 3, 4, 5, 6, and 7. The results are shown in figure 1 (right), and values from test 1 are repeated for comparison.

The combined data of figure 1 show that: (a) glazes of high resistance to solution under the conditions of test 1 (not over 1 part per million of Pb) will also present very little, if any, hazard to health, even if "lemonade" (test 5) or distilled vinegar (test 7) is left in contact with them for as much as 2 days; (b) with some colors (for example, dark blue and maroon) contact with vinegar or a citric-acid solution of lemonade strength for 2 days may be dangerously more severe than the tentative test (No. 1) indicates; and (c) the limited time available for this investigation should be concentrated on the tangerine and green glazes.

Accordingly, the specimens of tangerine and green glazes from two concerns were subjected to the series of tests indicated in figure 2.

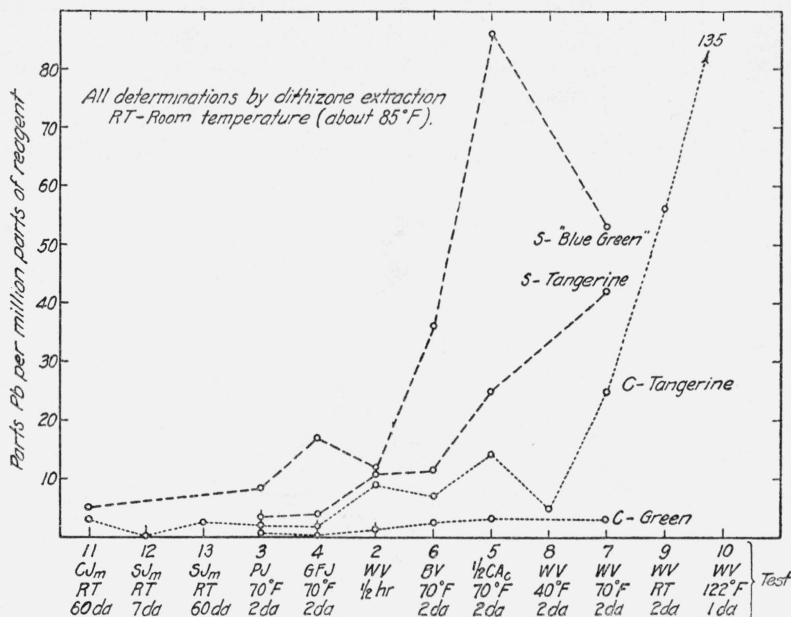


FIGURE 2.—Effect of accelerated test 2 and simulated service tests 3 to 13, inclusive, on tangerine and green glazes from two manufacturers.

The first five tests, when reading from left to right, show that their use in the home with fruit juices and jams would be less severe than in laboratory test 2,<sup>9</sup> while tests 6, 5, 7, and 9 (representing home use with lemonade and with vinegar) would be more severe, and the glazes, under these conditions, might be unsafe. For example, a vinegar containing 25 parts of lead per million would have a little over 1 mg of lead in 1.5 fluid ounces. Correspondingly, an 8-ounce tumbler of lemonade would have 6 mg of lead, and in the references cited it was stated that ingesting 1 mg daily produced symptoms.

Tests 8, 7, 9, and 10 were made to show the effect of variation in temperature. They show that vinegar may take up only 5 parts per million during 2 days in a refrigerator but 25 parts in a room when the climate is moderate, and over 50 parts in summer heat. Test 10

<sup>9</sup> Test 2 results were plotted here, instead of test 1, to make the method of analysis common for all tests.

indicates what might be expected if such glazes were used, for example, in warming ovens.

Based on the results described, tests 1, 2, 5, 7, and 16 were selected as representative of both laboratory and service tests. Five tangerine and six green glazes, subjected to these treatments, are represented by the graphs in figure 3. Plotted in the general order of relative severity, the values show first of all that this order is not the same for the two colors, but that more lead is dissolved from green glazes by "lemonade" than is dissolved by white vinegar under the same conditions. Figure 3 shows also, in the large majority of cases and test for test, that the green glazes are more reluctant to give up lead than are the tangerines. The indication of figure 2 that the tentative test (No. 1 or 2) may not be nearly as severe as "home conditions" (Nos. 5 and 7) is substantiated. Also, the substitution of a 0.5-percent citric-acid

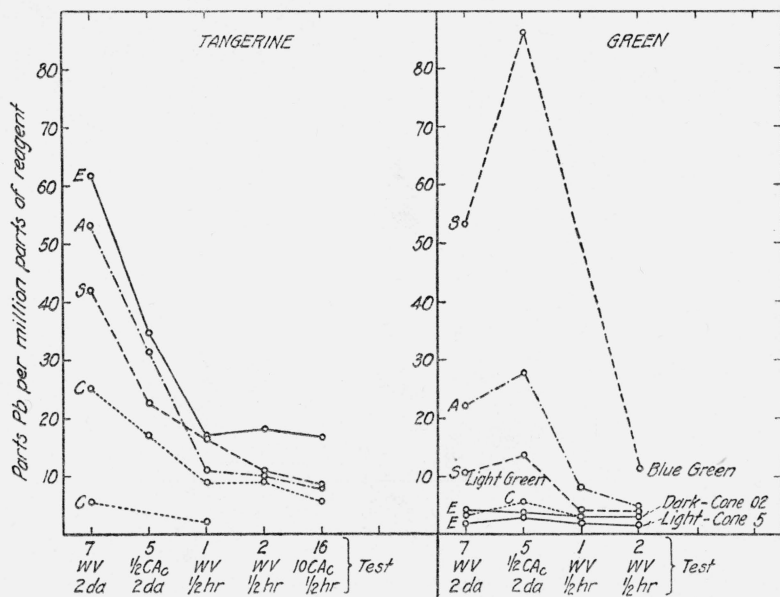


FIGURE 3.—Effect of accelerated tests 1, 2, and 16 and service tests 5 and 7 on tangerine glazes (left) and on green glazes (right) from four manufacturers.

solution for distilled 5-percent acetic-acid vinegar in tests 1 and 2 produces roughly comparable results with the tangerine glaze.<sup>10</sup>

Two additional points of interest are brought out by the data in figure 3 (right), namely, the effect of the stain on the solubility of a base glaze and the effect of the glaze temperature. The first is illustrated by the graphs for a "blue-green" and a "light-green" glaze. The only known difference between these glazes is that the latter contains only half of the identical stain used in the former. Also, the clear, or "base glaze," to which the stain had been added gave up only 0.6 part per million of lead in test 5. The second point is illustrated by the plots identified as "dark-cone 02" and "light-cone 5." Naturally, the compositions of the two base glazes, and probably

<sup>10</sup> Citric-acid solutions are more desirable than vinegar or acetic-acid solutions for laboratory work, because solutions of known strength can be prepared and maintained more conveniently.

also those of the stains, differed, but the results show that glazing at the lower temperature need not materially lower the resistance to solution.

The possibility of substituting citric-acid solution for vinegar was investigated further, as shown by the information in figure 4. Because of the shortage of cups, test results given in the larger graph were obtained with fruit saucers. They show that differences obtained with tests 1 and 14 are not significant.

Test 16 was then made on tangerine, maroon, and blue cups from one manufacturer to determine the strength of citric acid required in an accelerated test to dissolve glaze comparable to vinegar or lemonade in contact with the glaze for 2 days at 70° F (test 7). The data are plotted in figure 4 (right). Graphs 4C1, 4C2, and 4C5 are for three tangerine cups from the fourth shipment. Cups 4C1 and 4C2 were

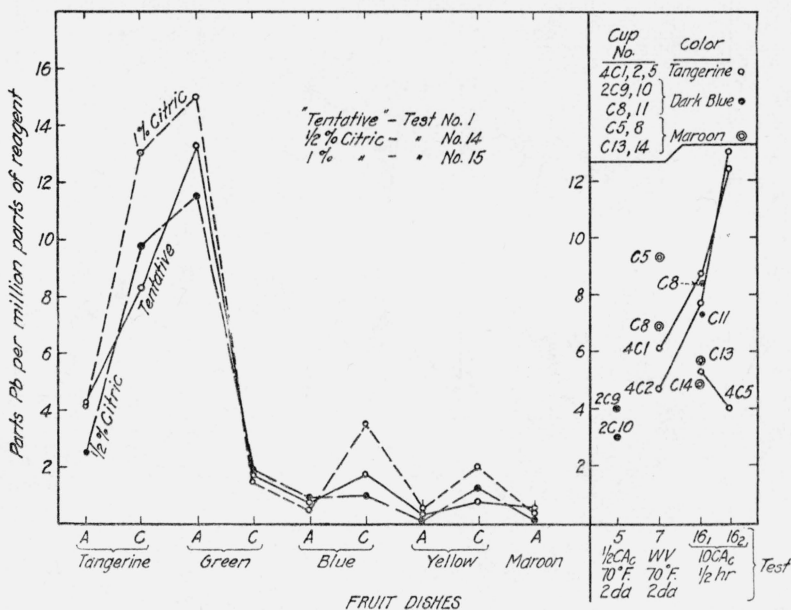


FIGURE 4.—Relative results with tests 1, 14, and 15 (left) on nine glazes in five colors; and (right) relative results with 10-percent citric-acid solution, test 16, and simulated service tests 5 and 7.

Test 16 was made twice on cups 4C1 and 4C2, after having made test 7, and twice on the previously untested cup, 4C5.

subjected to test 7 and then twice to test 16. Cup 4C5 was subjected twice to test 16. The indication is that a solution containing at least 10 percent of citric acid, when substituted for vinegar in the tentative method in order to obtain values comparable to those of test 5 or 7, will give higher values for some colors (for example, dark blue) and lower values for others (for example, maroon).

The values for individual cups given in figure 4 (right) lead to the question—what variations were found among supposedly duplicate cups? Typical examples are given in table 2. Results with tangerine cups were especially erratic. One of the factors which may be responsible for this is difference in heat treatment. It was thought

this could be illustrated by testing the glaze on the inside and on the outside of several cups. The graphs in figure 5 (left) show that a significant difference is to be found on the same cup, and therefore differences between cups are not surprising.

Another factor to be considered in the testing of commercial ware with a specified amount of reagent (in this case, 150 ml per cup) is the variable surface exposed because of differences in design. Some glazes were represented by the "regular", or approximately hemispherical cup, by a straight-sided cup having the shape of an inverted

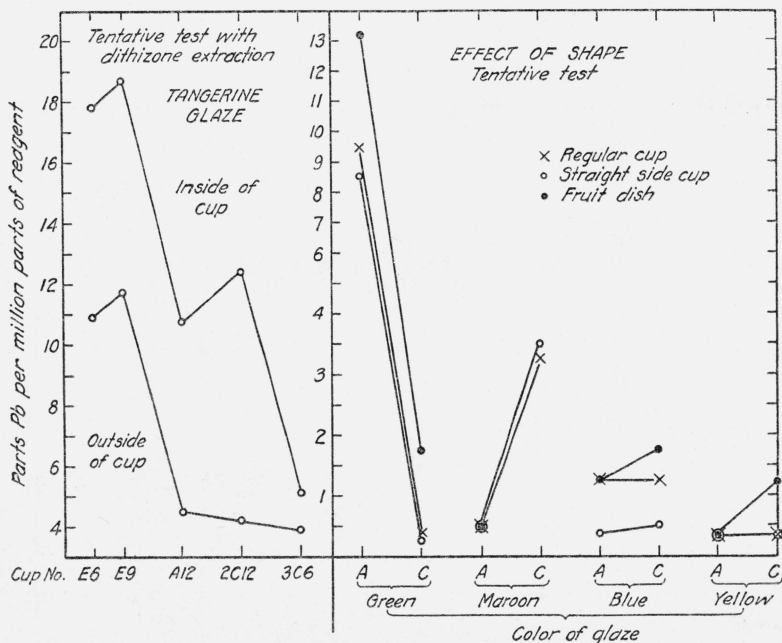


FIGURE 5.—(Left) relative solubility on inside and on outside of five cups from three manufacturers; (right) effect of shape of specimen (and consequent variation in surface exposed to the 150 ml of reagent) on results with the tentative test 1.

and truncated cone (frustum), and by fruit dishes. The tentative test (No. 1) was conducted on each shape, using 150 ml of vinegar for each shape. The results (fig. 5, right) show that the greater surface exposed by the fruit dish will usually give slightly higher values for lead dissolved, but the differences introduced by the shapes tested have little significance in the tentative test.

In addition to the above, three bowls in green glaze were subjected to tests 1, 5, and 7, using 325 ml of reagent because the capacity of the bowls was in the ratio of 325:150 to the capacity of comparable cups. The results for cups and for bowls were as follows: Test 1 gave 7 and 8 parts of lead per million; test 5 gave 26.0 and 29.1 parts per million; and test 7 gave 24.5 and 19.0 parts per million, respectively. This shows that similar shapes in various sizes will give comparable results if proportionate amounts of reagent are used.



TABLE 2.—Variations in solubility among different specimens of the same glaze

Manufacturer	Test number	Tangerine		Yellow		Green		Blue	
		Cup	Lead extracted <sup>a</sup>	Cup	Lead extracted <sup>a</sup>	Cup	Lead extracted <sup>a</sup>	Cup	Lead extracted <sup>a</sup>
C	1	C18	12	C6	1	C1	0.5	C6	1.5
C	1	3C1	12	C7	1	C2	<0.5	C7	1.0
C	1	3C3	9	2C11	1	2C11	>3	2C12	1.0
C	1	3C6	6	2C12	1	2C12	3		
C	2	2C11	11.1						
C	2	2C12	12.4						
C	2	3C5	5.9						
C	2	3C6	5.1						
C	5	2C7	13.8	2C9	0.5	2C9	5.9	2C9	4.0
C	5	2C8	20.0	2C10	1.0	2C10	5.6	2C10	3.0
A	1	A1	11	A4	<0.5	A4	10	A4	1.0
A	1	A4	9	A5	<0.5	A5	9	A5	0.5
A	1	A11	12			A12	>7		
A	2	A11	11.6			A6	6.0		
A	2	A12	10.0			A8	6.9		
S	7	S3	44.5	S2	0.5	S2	48.0	S2	1.2
S	7	S4	40.2	S3	0.4	S3	58.0	S11	1.1

<sup>a</sup> Values are in parts of lead (Pb) per million parts of reagent.

<sup>b</sup> The number before the letter indicates the shipment; thus, 3C1 in this column means tangerine cup 1 of the third shipment from manufacturer C.

An appreciable difference had been found in the amount of lead indicated by H<sub>2</sub>S precipitation, and by dithizone extraction, for some green and also some tangerine glazes. The precipitation of tin might account for the difference in the case of the tangerine glaze. However, the difference was found with a glaze reported as containing only Pb, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, and UO<sub>2</sub>—and uranium sulfide is not precipitated in weak acid solutions according to textbooks on the subject. This was checked by passing H<sub>2</sub>S through vinegar containing a known amount of uranium. A slight discoloration was produced, much less than that required to account for the differences actually found. Since there is no reason to believe that the slight amounts of uranium which might be ingested would be toxic, the matter was not investigated further.

The case of nausea mentioned in section I, which followed the drinking of tea from a new, green-colored cup may have been caused by copper. It was readily demonstrated that the differences in results for tests 1 and 2 could be explained by the precipitation of CuS. The data given in figure 6 were obtained for four cups, and also for four fruit dishes on each of which test 1 was conducted three times, that is, the acid treatment of test 1 was used, and analytical methods of tests 1 and 2 were made on aliquot portions of the same liquid.

It is evident from figure 6 that the solubility of the green glaze on the fruit dishes, so far as extraction of lead and copper are concerned, had been decreased materially by the first acid treatment. This interesting and important phenomenon was investigated further, and the results are plotted in figure 7. Cups glazed in tangerine, from four manufacturers, were subjected four times to test 1, and duplicate sets were washed with hot water and soap between each test to remove

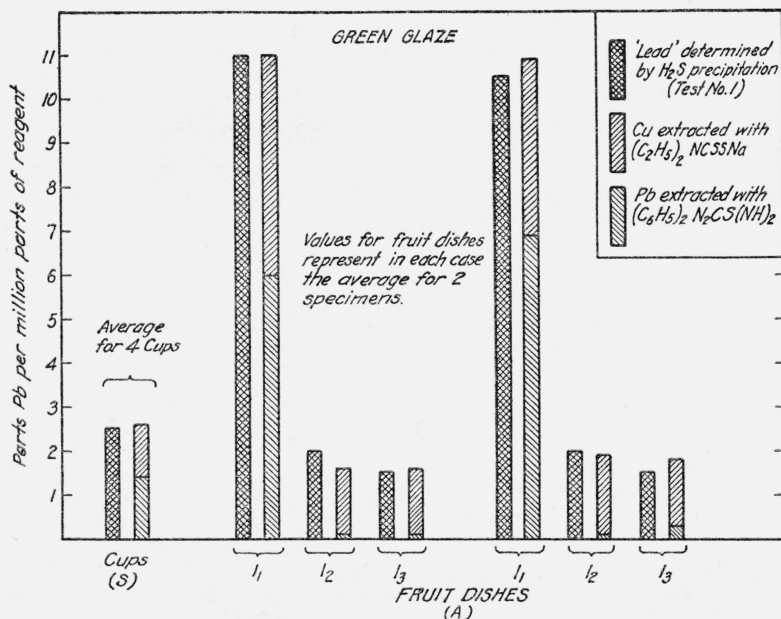


FIGURE 6.—Amounts of copper and of lead making up the “lead” dissolved during test 1, as indicated by  $H_2S$  precipitation.

The test was made on previously untested cups, and repeated twice on the fruit dishes.

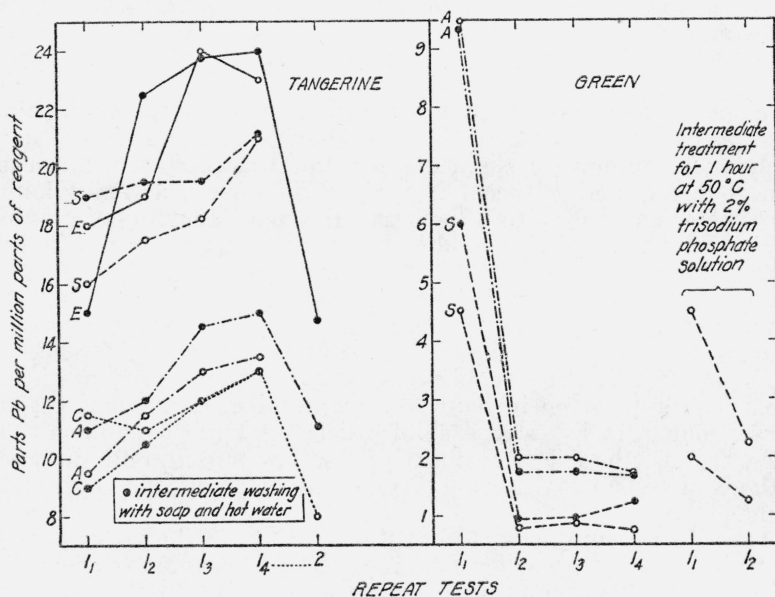


FIGURE 7.—Results obtained by repeating test 1 on tangerine cups from four manufacturers (left) and on green cups from two manufactures.

One series of cups was permitted to dry in the air between tests, a duplicate series was washed with soap and hot water. Also, the dithizone extraction for Pb (test 2) and the  $H_2S$  precipitation was made, on the same solution from the fourth test, for three samples of tangerine glaze.

The intermediate treatment with trisodium phosphate was made to simulate cleansing in a washing machine. The upper and lower pairs of values are on cups of slightly different color from manufacturer S, and from a separate shipment.

any film of colloidal silica which might have formed. Also, in three cases, the dithizone extraction was made, after this fourth test, on an aliquot portion of the same solution. The washing had no marked effect on the solubility. The graphs (fig. 7, left) indicate also that the susceptibility of the tangerine glazes to acid attack will increase with continued use, but that this increase (by the  $H_2S$  test) may not, necessarily, mean a proportionate increase in the lead taken up by the acid (dithizone extraction). As mentioned previously, the difference in the amount of dissolved lead, as determined by these two methods, has not been accounted for.

Figure 7 (right) shows the results of four successive tests by method 1 on green glazes from two manufacturers, and again with and without intermediate washing. The first acid treatment has reduced the amount of "lead" extracted ( $H_2S$  test) to not over 2 parts per million, supporting the results given in figure 6. It will be recalled that test 1 is a comparatively mild treatment compared with tests 5 and 7. Therefore, these two tests were repeated on two cups (Brand A). For test 5 the lead extracted dropped from 26.0 to 6.5 parts per million; for test 7, from 24.5 to 5.9 parts per million. It was further suggested that a treatment in an alkaline bath, such as ware is subjected to in commercial washing machines, might break down the glaze sufficiently to counteract the effect of the acid wash. Repeat tests were made on cups with an intermediate treatment in 2-percent trisodium phosphate solution at  $50^\circ C$  for 1 hour. The results (fig. 7, right) show that the alkali had little, if any, effect.

## V. DISCUSSION

This discussion deals primarily with the extent to which the results obtained have met the purpose of the work as outlined in section II.

The "tentative test" (No. 1, table 1) appears to be satisfactory with regard to possibility of duplication in different laboratories, as illustrated by the following:

*Case 1.*—A sample of cups tested at the National Bureau of Standards lost 3+, 3-, 2.5, and 2.5 parts of Pb per million parts of reagent, respectively. Cups in a duplicate sample submitted to another laboratory were reported as losing between 2.5 to 3 parts.

*Case 2.*—Average values obtained at the Bureau for four glazes from one manufacturer were: Tangerine 4+, green 1+, yellow <0.5, and blue <0.5. Values obtained by the manufacturer in the plant laboratory were: >3, >1, <0.8, and <0.8, respectively.

That determinations on individual specimens of a sample differ may reasonably be explained by actual differences in the glazes, as shown by the data in figure 5 (left) and by similar differences obtained with the other test methods.

Correlation of results obtained with laboratory tests (Nos. 1, 2, 14, and 15) and those designed to represent conditions in home use (Nos. 3 to 13, incl.) show that the former group of tests adequately determine the lead which might be taken up by fruit juices and jams. They do not, however, give a satisfactory measure of the lead which might be found in weak acid solutions, such as vinegar or lemonade, after 2 days of contact at ordinary temperatures (tests 5 and 7); test 16 was more severe for some colors and less so for others.

The proposed "solubility limit" in the "tentative test" of not over 2 parts of Pb per million parts of reagent should be considered as purely arbitrary until a decision on the matter is made by the proper State or Federal agencies. It is believed desirable, also, to substitute a solution containing 0.5 percent of citric acid and 0.25 percent of gelatin for the white, distilled vinegar (nominally 5-percent acetic acid) at present specified for the tentative test.

The H<sub>2</sub>S test for lead and the ammonia test for copper are believed to give values accurate within  $\pm 0.5$  part per million for solutions containing not over 6 parts. The H<sub>2</sub>S will precipitate also, in weakly acidic solutions, any copper, antimony, or tin which may be present, and this fact must, of course, be taken into consideration when evaluating results. For example, figure 6 shows that the precipitate obtained after the second and third treatments of a typical green glaze was almost entirely copper sulfide.

Whether one glaze will constitute a health hazard and another will not is still a moot question. Nevertheless, it may be said of the typical glazes covered in this investigation that, with one and possibly three exceptions, they very probably will not, provided the manufacturer exercises a modicum of care in their preparation. The first exception is the tangerine glaze from which organic acids extract quantities of lead detrimental to health, as judged by the best available medical records. Furthermore, this condition does not improve with continued use and may actually be aggravated. The two possible exceptions are the dark blue and the maroon, for which values are shown in figure 1. Whether or not these glazes will improve with repeated use was not determined.

Although a green glaze precipitated this study, the results of the investigation indicate that its use is not dangerous under certain conditions. There is reason to believe that the lead and copper dissolved by weak acids from many green glazes as they come from the kiln are a health hazard. Fortunately, this high solubility appears to be a surface condition. The results in figure 6 (which were, incidentally, obtained with the least resistant of the green glazes "in production") show that a preliminary acid wash will reduce the lead absorbed during test 1 to a few tenths of 1 part per million and, probably, to not over 6 parts per million during the severe conditions of tests 5 and 7. This does not seem to warrant much concern, but it would seem preferable to use copper-free stains for the green glazes. These glazes do constitute a potential danger, and every precaution should be exercised to assure their harmlessness to the user.

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The authors acknowledge the assistance of the U. S. Potters Association and the six cooperating companies for material furnished and for information regarding their manufacture, and to W. B. White of the Food and Drug Administration and R. R. Sayers of the U. S. Public Health Service for helpful suggestions.

WASHINGTON, January 20, 1939.