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PROCEDURES FOR QUALITY ASSURANCE OF POLYETHYLENE BOTTLES

AND NITRIC ACID AMPOULES USED FOR TRACE-METAL ANALYSES OF

WATER-QUALITY SAMPLES

By Linda C. Friedman, Fred E. King, Hanford J. Miller, and LeRoy J. Schroder

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PROCEDURES FOR QUALITY ASSURANCE OF POLYETHYLENE BOTTLES AND NITRIC ACID AMPOULES USED FOR TRACE-METAL ANALYSES OF WATER-QUALITY SAMPLES

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ABSTRACT

Sporadically high lead and cadmium concentrations in water samples, collected and analyzed by the U. S. Geological Survey, were found to have been caused by contamination from nitric acid ampoules used to preserve the samples. The loss of valuable data and of many man-hours of work indicated the need for a formal materials-testing program.

Data collected from analyzing ampouled nitric acid and from analyzing leachates of the polyethylene bottles, used to collect trace-metal samples, are used to establish a quality assurance program for these items. Operating characteristic curves indicate simple testing of one or two ampoules or bottles per box is insufficient.

To reduce costs, the sampling scheme developed requires initially analyzing combinations of bottles and ampoules, using data from Dodge-Romig double-sampling tables. If a second sample is needed, the second portion of the double sample can be selected and analyses continued, or, using Dodge-Romig single-sampling tables, bottles and ampoules can be selected and only analyzed for those ions which appear to have yielded some contamination.

INTRODUCTION

Materials used in sample collection must be adequate, and of uniform or known quality to have effective analytical data quality assurance. In 1978, in spite of an analytical quality assurance program, sporadically high lead and cadmium values were noted in water samples collected by the Water Resources Division (WRD) of the U.S. Geological Survey. Because many analyses were on samples from sites with a previous history of low lead and cadmium concentrations (including samples from public water supplies), contamination of the sample during or after collection seemed likely.

The most probable source of contamination was either the polyethylene bottles used to collect the samples or the ampouled acid used to preserve the samples. Polyethylene bottles were rinsed routinely with nitric acid by the two analytical laboratories of the WRD, but were not tested prior to distribution to the field. A few ampoules were usually analyzed by the laboratories upon receipt of a new shipment; however, no formal materials testing program was in effect. Loss of valuable analytical data plus the loss of time spent in the collection and analysis of the original samples and in the man-hours spent during investigation of the source of contamination, indicated the need for a formal materials-testing program.

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INVESTIGATION INTO CONTAMINATION

Initial analyses of bottles and ampoules used in collection of the samples indicated that the contamination had been caused by the ampouled acid. Although the source of ampouled acid had provided assay results showing low trace metal concentrations, discussion with the company indicated that the assay had been made prior to ampouling. It seemed probable that contamination had occurred during ampouling or that the ampoule container itself was the contaminant.

It also appeared that, as the initial sporadic results had indicated, concentrations of lead and cadmium added to the samples were not uniform. For example, analyses of five replicate samples by atomic absorption spectrometry (AAS) showed concentrations of lead ranging from 14 to 350 μg/L and concentrations of cadmium ranging from 3 to 35 μg/L.

The outside surfaces of 40 ampoules from the contaminated lot were rinsed with deionized water into 250 mL polyethylene bottles, the rinse water diluted to approximately 250 mL and the solutions analyzed using inductively coupled plasma (ICP) emission spectrometry. Only one solution contained a concentration of cadmium which was greater than 1 μg/L (but less than 2 μg/L) and only two solutions contained concentrations of lead which were greater than 10 μg/L (but less than 20 μg/L).

After discarding this rinse water, ampoules were broken at their neck and contents of each diluted to approximately 250 mL in the polyethylene bottles. Analyses of these acid solutions by ICP showed much greater concentrations of cadmium and lead (fig. 1); only two solutions contained less than 1 μg/L cadmium and only four solutions contained less than 10 μg/L lead. The cadmium concentration was usually (but not always) approximately one-tenth the lead concentration. Data from the ICP analysis of other trace metals showed that concentrations were low; although one solution contained copper and two contained barium slightly in excess of 10 μg/L, beryllium, cobalt, copper, iron, lithium, manganese, molybdenum, strontium, vanadium, and zinc were all below analytical detection levels.

A blue ring around the neck of the ampoule (painted to aid in the uniform breaking of the scored ampoule) seemed a likely source of the contamination. Therefore, three 2-mL ampoules and two 1-mL ampoules were broken above the ring and the contents added to five 250-mL polyethylene bottles and diluted to approximately 250 mL. Although concentrations of lead and cadmium (table 1) were considerably less than had been found when ampoules were broken at the blue ring (fig. 1), slight contamination was still apparent.

Because it was probable that the blue ring was the chief source of contamination, new lots of ampoules were purchased without the blue ring. All remaining ampoules in the contaminated lot were discarded.
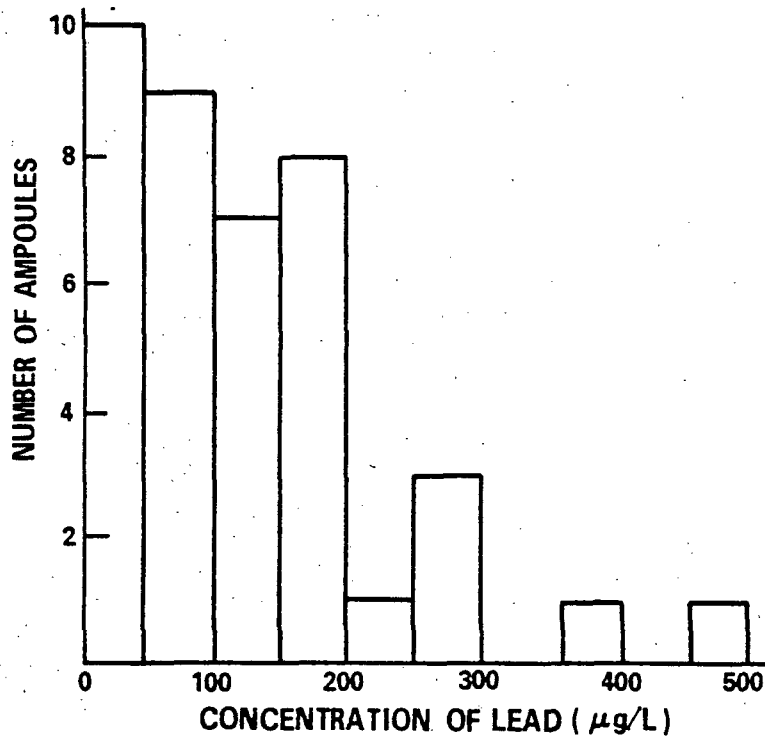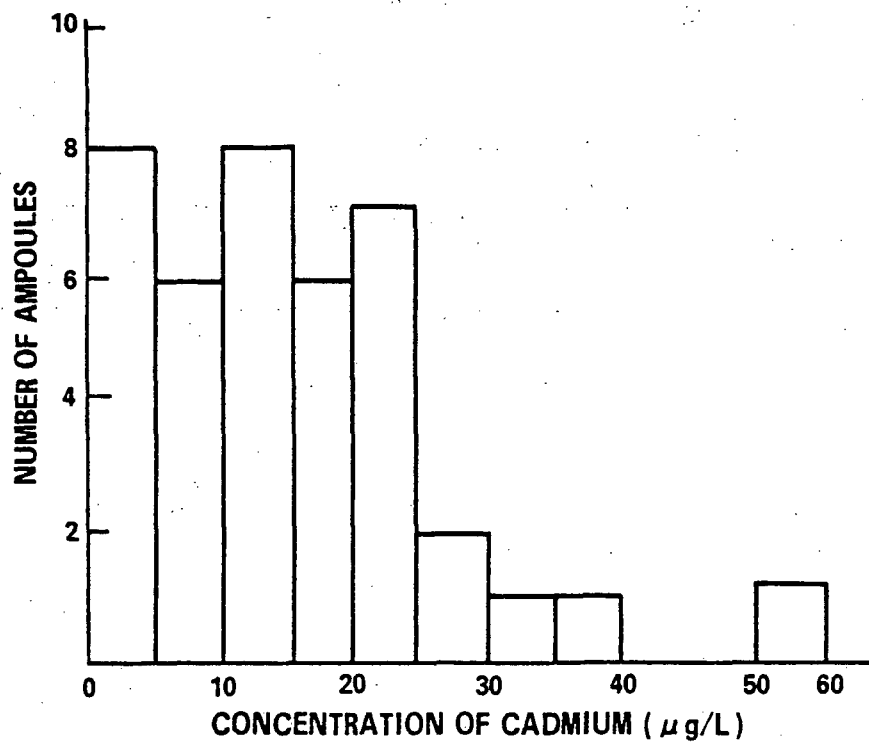
2

Figure 1.—Summary of lead and cadmium analyses of contents
    of 40 "contaminated" nitric acid ampoules,
    diluted to 250 mL.

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Table 1.—Analysis of lead and cadmium in five nitric-acid ampoules.

Size of ampoule[a] (mL)	Pb (µg/L)	Cd (µg/L)
1	18.1	1
1	26.7	2.7
2	18.3	2.7
2	10	1
2	10	1

[a]Ampoules were broken above blue ring, and contents diluted to 250 mL.

PRELIMINARY DATA

The two WRD laboratories began an examination of the newly purchased ampoules. Each laboratory followed a slightly different inspection procedure.

The Denver Central Laboratory selected one ampoule per box from 36 boxes of 1-mL and 36 boxes of 2-mL nitric acid ampoules. Contents of each ampoule were diluted in volumetric flasks to 25.0 mL using deionized water, and the solution was analyzed for barium, beryllium, calcium, cadmium, cobalt, copper, iron, lithium, magnesium, manganese, molybdenum, sodium, lead, silica, strontium, vanadium, and zinc using inductively coupled plasma (ICP) emission spectrometry. In addition, using a hydride-generation atomic absorption spectrophotometric method, three of the solutions made from the 1-mL ampoules and three of the solutions made from the 2-mL ampoules were analyzed for arsenic and three of each were analyzed for selenium. Three of each were also analyzed for mercury by flameless atomic absorption spectrometry.

To preserve trace-metal samples, using the assumption that 2-mL ampoules would be diluted to 500 mL and 1-mL ampoules would be diluted to 250 mL, concentrations of trace metals at these dilutions were computed. One ampoule showed copper above the level indicated in table 2; two showed zinc above the level indicated; and one showed three ions, copper, lead, and strontium, above the level indicated.

The Atlanta Central Laboratory selected a minimum of two ampoules from each box of 1-mL and 2-mL ampoules and diluted each, in acid-rinsed polyethylene bottles, to approximately 250 mL, using deionized water. Six hundred thirty four solutions were analyzed for lead, using chelation-extraction plus atomic absorption spectrometry. Of these, 162 solutions were also analyzed for cadmium; 84 for cadmium, nickel, arsenic, and mercury; 100 for cadmium, copper, nickel, cobalt, and arsenic; and 78 for cadmium, copper, nickel, cobalt, arsenic, and mercury. All analyses were done by chelation-extraction plus atomic absorption spectrometry except for arsenic, which was done by hydride-generation atomic absorption spectrometry, and mercury, which was done by flameless atomic absorption spectrometry. Five ampoules showed contamination above the levels specified in table 2.

Table 2.—Nitric acid ampoules as a source of contamination: Summary of data from Atlanta and Denver Central Laboratories.

Constituent	Number of ampoules analyzed	Percent of ampoules contributing contamination above levels specified	
		Percent	Level (μg/L)
Arsenic	268	1	3[a]
Barium	72	0	10
Beryllium	72	0	1
Cadmium	496	0	1
Cobalt	250	1	1
Copper	250	1	2
Iron	72	0	10
Lead	706	1	2[b]
Lithium	72	0	1
Manganese	72	0	1
Mercury	168	0	0.2
Molybdenum	72	0	1
Nickel	262	0	4[c]
Selenium	6	0	1
Strontium	72	1	1
Vanadium	72	0	1
Zinc	72	3	10

[a] 6 percent of the ampoules are greater than 2 micrograms per liter.

[b] 3 percent of the ampoules are greater than 1 microgram per liter.

[c] 4 percent of the ampoules are greater than 2 micrograms per liter.

Although polyethylene bottles were not the source of the high lead and cadmium concentrations observed in 1978, they were clearly a possible source of trace-metal contamination. The bottles were investigated in a separate study (Schroder and Taylor, 1980). Ninety-seven 500-mL polyethylene bottles were randomly selected from 180 cases of 216 bottles per case, and leached, using nitric acid solutions ranging from 0.2 percent to 5.0 percent (by volume).The leachates were analyzed for barium, beryllium, cadmium, cobalt, copper, iron, lead, lithium, manganese, molybdenum, strontium, vanadium, and zinc using ICP emission spectrometry. One bottle gave a positive result for one ion tested (manganese). No difference was noted between rinsing with deionized water and rinsing with nitric acid prior to leaching.

FACTORS CONSIDERED

Theoretically, each polyethylene bottle could be tested prior to issuance to field personnel; however, the cost of such a complete program would be prohibitively high. To reduce this cost, it was clear that development of a sampling program was necessary, in which the quality of the lot is inferred from data collected during examination of the sample. A sampling program was also necessary to determine the quality of the ampouled acid, since there is no way to test all ampoules without destroying the lot.

Obviously, a program to test bottles and ampoules together would be cheaper to operate than two separate testing programs. However, in rejecting a lot because of contamination shown by a bottle-ampoule combination, it was recognized that it might be important to determine whether contamination was due to bottles, ampoules, or a combination of bottles and ampoules. Contamination from both bottles and ampoules was considered to be less likely, since the probability of occurrence of a compound event is the product of the probabilities that each of two independent subevents will occur (Grant and Leavenworth, 1972); thus, if there is a 10 percent chance that each lot (bottle and ampoules) is bad, there is a 1 percent chance that both are bad.

To ensure that the sample represents the lot, each ampoule or bottle in the lot must have an equal chance of being chosen. Because random sampling of each lot (Wiesen , 1974) would give a representative sample, it was determined that random sampling techniques must be used.

A program in which testing is done on one or two ampoules per box was considered; however, to control risk factors involved in using data from a sample to indicate the quality of a lot, such a system would rarely be appropriate. This conclusion can be illustrated from operating characteristic (OC) curves, since OC curves give the best descriptions of sampling plans (Miller and Freund, 1977). The curves can be drawn using a binomial distribution to approximate the hypergeometric distribution, which actually represents the sampling distribution, since sample sizes are small (less than one tenth) compared to lot sizes. Because a risk of ten percent probability of accepting a defective lot is commonly used (Wiesen, 1974), it was used to examine the OC curves.

In the trivial case where each box of ampoules is considered as a separate lot, a sample size of one (fig. 2) gives a 10 percent chance of accepting a lot in which 90 percent of the lot is defective (able to contaminate above specified levels). A sample
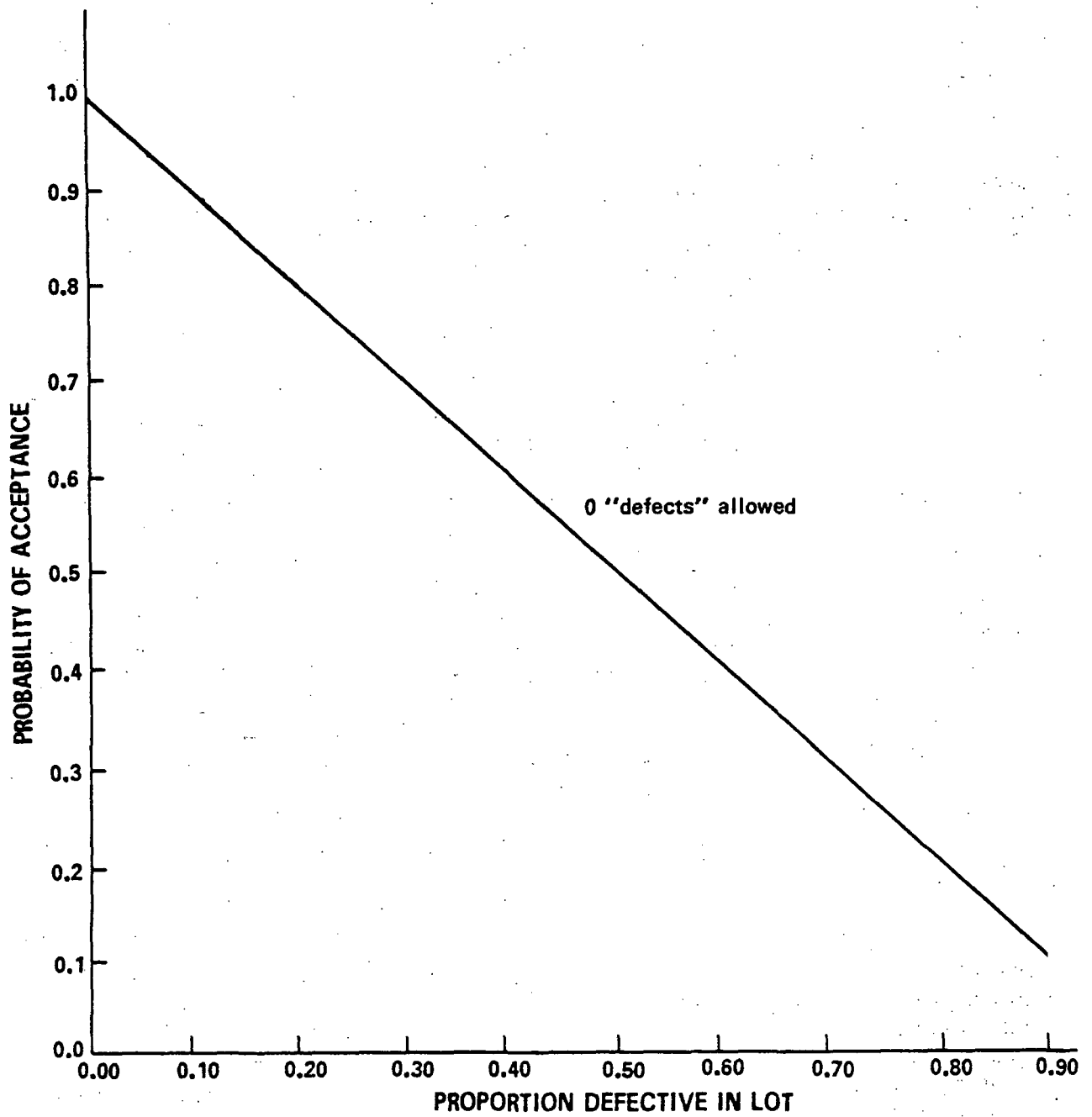
Figure 2.--Operating characteristic curve, sample size 1.

size of two (fig. 3) gives a 10 percent chance of accepting a lot in which about 68 percent of the lot is defective. In both cases, no ampoules could be rejected without rejecting the lot (box).

If, more realistically, the lot is considered to be all ampoules (or bottles) in all boxes received at one time from one manufacturing lot, the OC curve (and associated probabilities of acceptance) will change according to the number of boxes received, if a constant number of ampoules per box are selected. Since the number of ampoules per box has varied slightly in the past (between 100 and 144), it was recognized that this could be significant even when the lot size is held constant.

A more important factor is that the operating characteristic curve will change, depending on the number of ampoules allowed to be rejected. Figure 4, for example, depicts several OC curves for a sample size of 72. Examining the figure, it is apparent that, if no ampoules are allowed to be rejected without rejecting the lot, there is a 10 percent chance of accepting a lot in which 3 percent of the lot is defective. However, if 1 ampoule is allowed to be rejected without rejecting the lot, there is a 10 percent chance of accepting a lot in which 5 percent of the lot is defective; while, if 5 ampoules are allowed to be rejected without rejecting the lot, there is a 10 percent chance that 13 percent of the lot is defective. Similarly, for a sample size of 36 (the sample size of 2-mL ampoules selected by the Denver Central Laboratory), the OC curve (fig. 5) indicates that, if no ampoules are allowed to be rejected, there is a 10 percent chance of accepting a lot in which 6 percent of the lot is defective; while, if only 1 ampoule is allowed to be rejected without rejecting the lot, the probability changes to a 10 percent chance that 11 percent of the lot might be defective.

It should be noted that, although there is a chance (for example, 10 percent) that a certain percentage (for example, 11 percent) of the lot is defective, this does not mean that percentage is defective. If only 0.1 percent of the lot is defective, then no more than 0.1 percent is defective. Unless every item is sampled, the exact percentage of defective items in the lot will remain unknown; only probabilities can be determined and such probabilities will depend on the sample size.

Both acceptable quality level (AQL) plans and lot tolerance percent defective (LTPD) plans were considered; the former are used to indicate the maximum defects which are satisfactory for the process average (expected average number of contaminated samples), while the latter are used to indicate levels which are unsatisfactory and should cause refection (Juran and others, 1974). LTPD plans were felt to be preferable, since they were less likely to allow lots of quality much poorer than the AQL to be selected (Mundel, 1979).

SELECTION OF SAMPLE

The selection of sample sizes indicated below is based on both the Dodge-Romig double and single sampling tables developed for the Bell Telephone System (Dodge and Romig, 1959). A ten percent chance that five percent of the bottles and ampoules may add trace metal combinations above the levels specified in table 3 is allowed. Based on the preliminary data, a process average of 1.01 to 1.50 percent is used to

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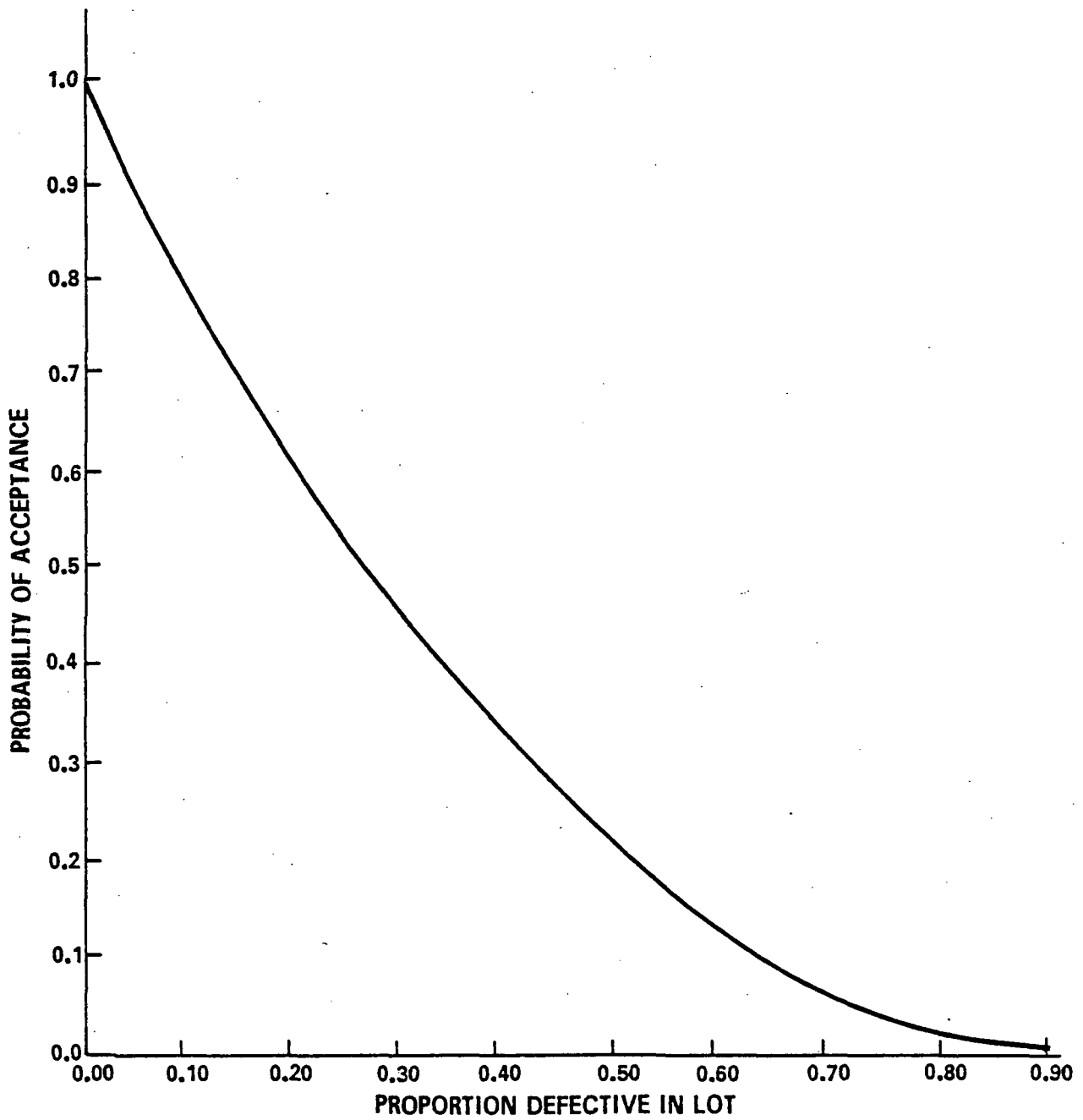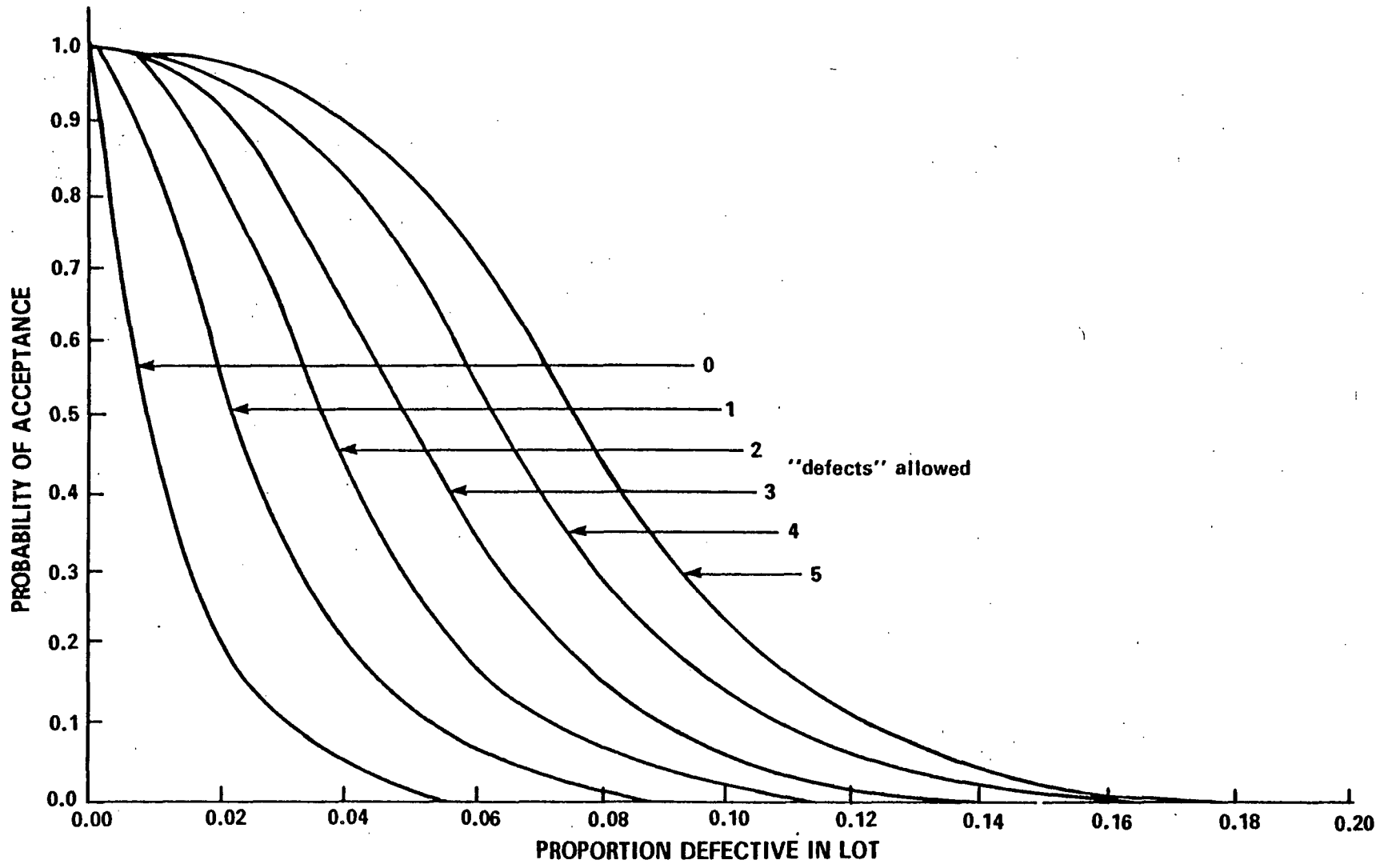Figure 3.--Operating characteristic curve, sample size 2.

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Figure 4.--Operating characteristic curve, sample size 72.

PROBABILITY OF ACCEPTANCE

PROPORTION DEFECTIVE IN LOT

"defects" allowed

0

1

2
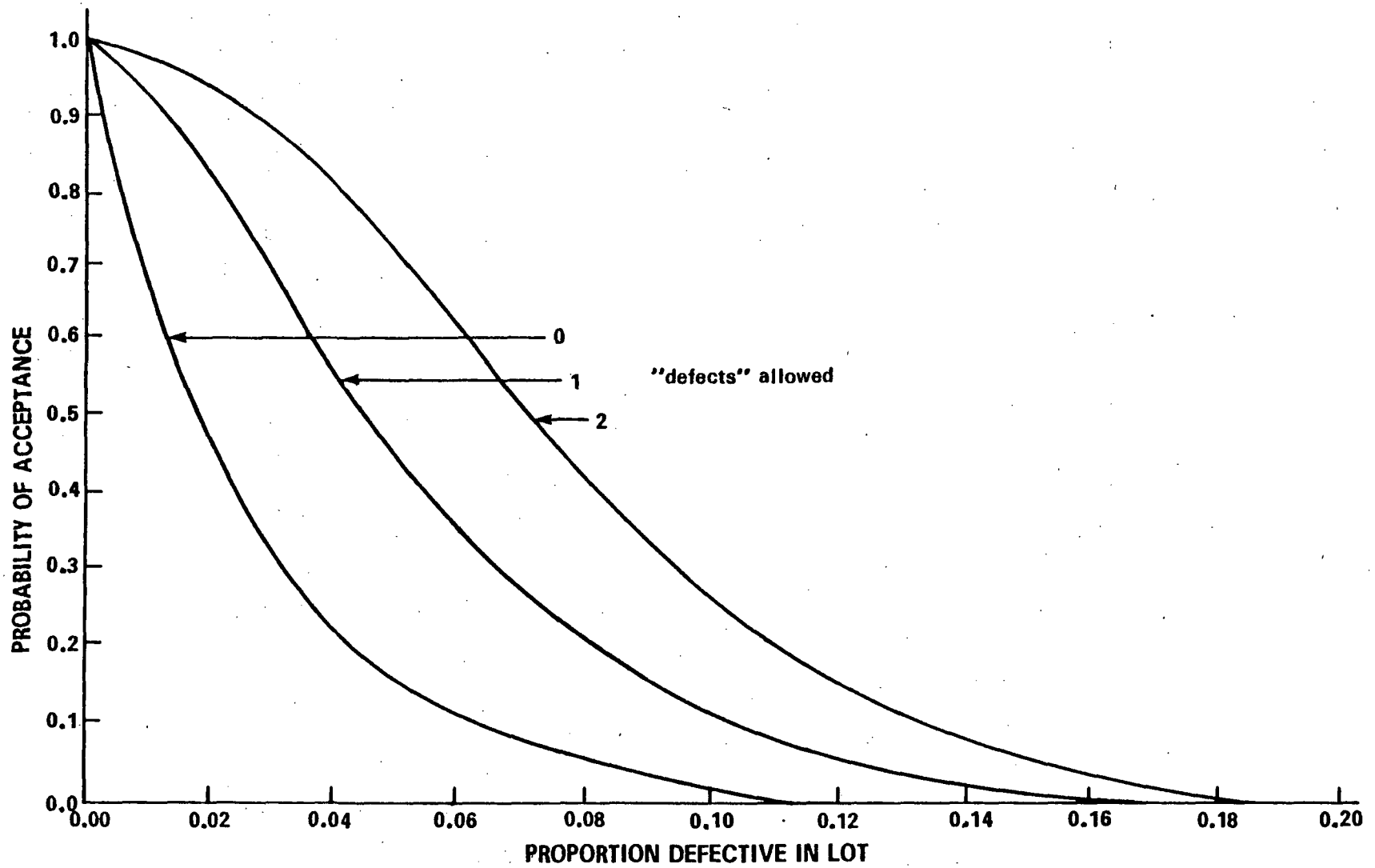
Figure 5.--Operating characteristic curve, sample size 36.

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Table 3.--Maximum contamination levels allowed in water samples

Constituent	Maximum contamination[a] (µg/L)
Aluminum	60
Antimony	1
Arsenic	3
Barium	10
Beryllium	1
Cadimium	1
Chromium	6
Cobalt	2
Copper	6
Iron	10
Lead	6
Lithium	3
Manganese	1
Mercury	0.2
Molybdenum	6
Nickel	4
Selenium	1
Silver	3
Strontium	1
Vanadium	4
Zinc	10

[a]Concentrations are based on addition of 2-mL ampoules to 500-mL samples and addition of 1-mL ampoules to 250-mL samples.

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select the initial sample size from the double sampling tables; it is recognized that this estimate of process average may be in error and have to be revised, especially since ampoules were tested for varying types of ions and neither ampoules or bottles were tested for all ions indicated in table 3.

For more cost-effective testing, nitric acid ampoules and polyethylene bottles used for collection of trace metal water samples should be purchased in similar lot sizes (10,000 to 20,000; 20,000 to 50,000; or 50,000 to 100,000), at similar times. Although initial testing is on a polyethylene bottle-acid ampoule combination, the normal Dodge-Romig sampling scheme has been modified so an independent bottle plus ampoule test may be made, if a second sample is necessary. The sampling scheme is outlined in figure 6 for lot sizes of between 20,000 to 50,0000. To allow greater flexibility, lot sizes may vary between 10,000 and 100,000; however, it should be recognized that it will usually be more difficult to get a random sample from large lots (between 50,000 and 100,000), but less expensive to test them.

Maximum contamination concentrations (table 3) were chosen by considering the lowest concentration levels routinely reported by the WRD and by examining both the analytical detection level of the method chosen for analysis (table 4) and the preliminary data (table 2). To avoid rejections of lots because trace metal concentration was near the analytical detection level, a minimum of three times the detection level was used for the maximum concentrations allowed in the test samples. These allowable concentrations were further increased if the preliminary data indicated that a large number of solutions probably would contain concentration in excess of three times the detection level.

Initially, 120 ampoules and 120 bottles should be randomly selected from each lot. Each incoming carton in the lot should be assigned a number. If the lot is systematically packed within each carton, then each row, column, and level should be assigned a number and four-digit random numbers (obtained from a table, programmable calculator, or computer) used to select carton, row, column, and level. If the lot is not systematically packed, the top section of each carton should be assigned a 1, the middle section a 2, and the bottom section a 3; within each section the numbers 1 through 9 should be assigned beginning in the upper left area of the section and ending in the lower right area (see fig. 7), and three-digit random numbers used to select carton, section, and area.

Each of the 120 ampoules and 120 bottles should then be assigned a number (from 001 to 120), and three-digit random numbers used to assign each ampoule to a bottle. Four teflon-bottle blanks, prepared as described under the section, Recommended analysis, should be assigned the numbers 121, 122, 123, and 124, and three-digit random numbers used to determine the order in which all of the bottles will be analyzed.

If not more than two bottle-ampoule combinations yield trace metal concentrations in excess of those in table 3, both the lot of polyethylene bottles and the lot of acid ampoules should be accepted. If more than 14, 15, or 16 bottle-ampoule combinations for lot sizes of 10,000 to 20,000, 20,000, to 50,000, and 50,001 to 100,000 respectively, are found to contain concentrations in excess of those in table 3, the combination of these particular lots of bottles and ampoules must be rejected. If it is desirable to determine whether the bottles alone or ampoules alone would cause the rejection, further testing must be done on both bottles and ampoules.
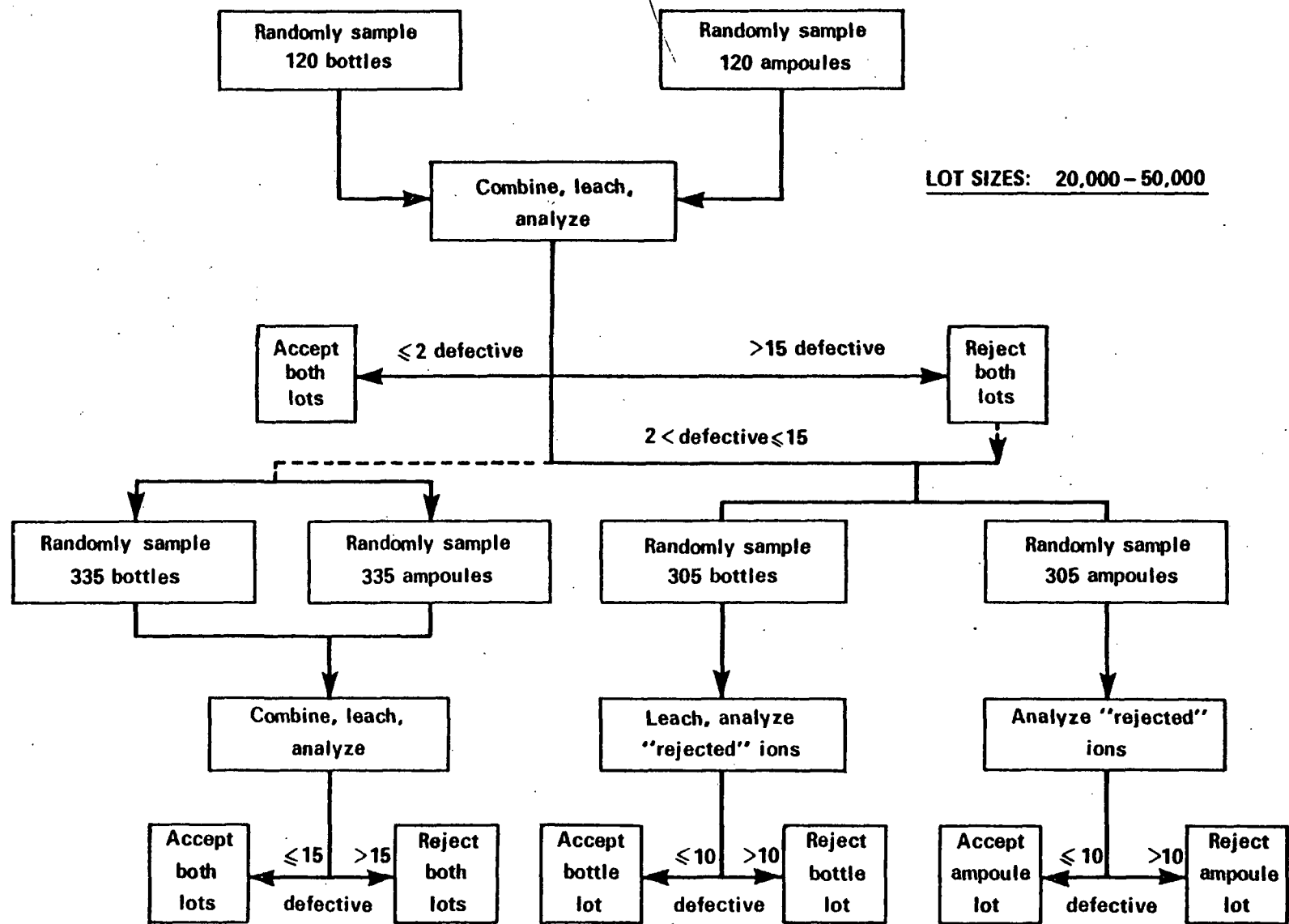
Figure 6.—Selection of bottles and ampoules to be tested for trace-metal contamination.

Table 4.—Maximum concentrations allowed in test samples

Constituent	Maximum concentration (μg/L)[a]	Analytical method to be used	Analytical detection level (μg/L)
Antimony	5	AAS (hydride)	1
Arsenic	15	AAS (hydride)	1
Barium	50	ICP	2
Beryllium	5	ICP	0.5
Cadmium	5	ICP	1
Chromium	30	AAS	10
Cobalt	10	ICP	3
Copper	6	ICP	10
Iron	50	ICP	3
Lead	30	ICP	10
Lithium	15	ICP	4
Manganese	5	ICP	1
Mercury	1	AAS (flameless)	0.2
Molybdenum	30	ICP	10
Nickel	20	AAS (20 mL, chelation-extraction)	5
Selenium	5	AAS (hydride)	1
Silver	15	AAS (20 mL, cheltion-extraction)	5
Strontium	5	ICP	0.5
Vanadium	20	ICP	6
Zinc	50	ICP	3

[a]Since only 100 mL of water is used in the leach test, any contamination found may be expected to be about five times the concentration which would be found in a 500-mL water sample collected for trace metal analysis.

Top

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        11              12              13

        14              15              16

        17              18              19
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Middle

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        21              22              23

        24              25              26

        27              28              29
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Bottom

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        31              32              33

        34              35              36

        37              38              39
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Figure 7.—Assignment of numbers to be used in random number selection.

If more than two, but not more than 14, 15, or 16 bottle-ampoule combinations, depending on lot size, yield results which are greater than the concentrations specified in table 3, a second sample must be taken using one of the two approaches following.

Bottles and ampoules are randomly selected, 285 (for lot sizes between 10,000 and 20,000), 305 (for lot sizes between 20,001 and 50,000), or 355 (for lot sizes between 50,001 and 100,000). Bottles and ampoules are tested independently only for those ions which gave results in excess of the limits in table 3. Considering each lot independently, the lot of bottles or lot of ampoules is rejected, if more than 9, 10, or 12 (for sample sizes of 285, 305, or 355, respectively) bottles or ampoules are found to contain concentrations in excess of those in table 3; otherwise, the lot is accepted.

Alternatively, 310, 335, or 360 (depending on lot size) bottles and ampoules are randomly selected. Bottles and ampoules are randomly combined as indicated, and analysis is done for all ions listed in table 3. Both lots are rejected, if more than 14, 15, 16 (for sample sizes of 310, 335, or 360) bottle-ampoule combinations exceed tabulated concentrations; otherwise, both lots are accepted.

RECOMMENDED ANALYSIS

After being randomly chosen, all bottles to be tested are rinsed, prior to testing, with deionized water; 100 mL deionized water plus contents of the acid ampoule randomly selected for that bottle are added to each bottle. The concentrations of any contaminants in these bottles would be five times higher than in samples collected for trace-metals analysis. Four blanks are prepared, using Teflon (fluorinated ethylene propylene) bottles and caps, which have been precleaned by filling with a ten percent (by volume) nitric acid solution; allowed to sit for 24 hours; and rinsed three times with deionized water (Schroder and Taylor, 1980). One hundred mL of deionized water and 2 mL of fresh, double-distilled or ultrapure grade nitric acid are added to the four Teflon bottles.

Bottles should be shaken for one hour per day for two weeks [1] using a wrist-action shaker. The order in which polyethylene and Teflon bottles are to be analyzed should be randomly determined as indicated previously.

To reduce costs the concentration of barium, beryllium, cadmium, cobalt, copper, iron, lead, lithium, manganese, molybdenum, strontium, vanadium, and zinc should be simultaneously determined, using ICP emission spectrometry. The average concentration of each element found from analyzing the Teflon bottles is used to determine blank-corrected concentrations. If any blank-corrected concentration exceeds a maximum concentration in table 4, the bottle-ampoule combination should be considered rejected, and testing on that bottle-ampoule combination should not be continued. For any one solution analyzed, a maximum of one rejection is counted, even if several of the concentrations in table 4 are exceeded.

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[1] The 2-week time limit is chosen to represent the time in which a routine water sample would be expected to be in contact with the bottle and acid prior to analysis.

Concentrations of aluminum and chromium should be determined using atomic absorption spectrometry (AAS). Concentrations of nickel and silver should each be determined by chelating 20-mL of each solution, standard, and blank, with ammonium pyrrolidine dithiocarbamate (APDC) solution, extracting with methyl isobutyl ketone (MIBK), and analyzing by AAS. Antimony, arsenic, and selenium should be analyzed by hydride-generation AAS and mercury by flameless AAS. Bottle-ampoule combinations should be considered rejected if blank-corrected concentrations are above those indicated in table 4.

If a second set of samples must be taken from the lot, the same criteria for rejection should be used. However, if the option is used to test bottles and ampoules separately, ultrapure acid should be added to the 100 mL of deionized water in each bottle and the bottles leached for two weeks prior to analysis; and ampouled acid should be added to 100 mL deionized water in clean glass containers and may be analyzed immediately. Single element techniques (such as AAS) may be substituted for multi-element techniques (such as ICP); although bottles and ampoules are analyzed separately using this option, the cost may be less since analysis is for only those ions which have shown some contamination.

SUMMARY

Sporadically-high lead and cadimum concentrations in water samples collected and analyzed by the U. S. Geological Survey were found to have been caused by contamination from nitric acid ampoules used to preserve the samples. The contam inated lot of ampoules was discarded and data from trace metal analysis of new lots of ampouled nitric acid were studied. Concentrations of strontium, lead, copper, and zinc slightly in excess of 1, 2, 2, and 10 ug/L, respectively, were noted in one study in which 72 ampoules were analyzed for 17 constituents; 4 of the ampoules showed some contamination. Six hundred thirty four ampoules were analyzed for between one and seven trace metals in a separate study; cobalt, lead, and arsenic were found in concentrations above 1, 2, and 3 ug/L, respectively; 5 ampoules showed some contamin ation.

The data from analysis of the ampouled acid plus data from analysis of nitric-acid leachates of polyethylene bottles were used to develop a sampling scheme in which polyethylene bottles and nitric-acid ampoules would be tested simultaneously. Consideration of OC curves indicated that interpretation of lot quality, when one or two items per box were tested, would depend on lot size, number of items per box, and number of items allowed to be rejected without rejecting the lot. To control the risk factors involved in using data from the sample to approve or reject a lot, a procedure was developed in which the first stage of the Dodge-Romig double-sampling plan was used to select bottles and ampoules. The normal Dodge-Romig double-sampling procedure was modified, so that if a second sample was needed, the second portion of the double sample could be taken, or a single-sampling plan could be used to select separate sets of bottles and ampoules to be analyzed for only those ions which failed to meet the criteria used in testing the initial combined sample.

The process average used to determine sample size was based on preliminary data. As more data are developed, the estimate of process average may need to be revised, and the size of sample required to maintain the same risk factor reduced or increased.

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