ON-SITE ACIDIZING FLUID ANALYSIS SHOWS HCL AND HF CONTENTS OFTEN VARIED SUBSTANTIALLY FROM SPECIFIED AMOUNTS

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ABSTRACT

A quality control survey of 162 acidizing fluids revealed the following problems:

1. Acid concentrations were often too high or too low.
2. Frequently, fluids were not thoroughly mixed.
3. In some cases, fluids contained incompatible additives.

A field test kit and conventional laboratory analyses were used to determine the acid concentrations in fluids from 44 acid jobs done in Southern California during the last four years. On 41% of the jobs, the acid concentration of at least one fluid varied more than 30% from the specified value. The quality of the fluids from five service companies were surveyed; however, just two companies did 77% of the jobs.

Analyses of iron content in the acids showed that 78% of the fluids contained less than 200 ppm iron. The average iron content was 180 ppm.

The test kit assembled for this survey permits rapid well-site analysis by people who do not have formal training in chemistry. The total analysis time is about 2 minutes each for HCl and HF and 5 minutes for the iron analysis. The concentrations of HCl and HF are determined volumetrically by using constant volume dispensing bottles rather than a buret. A novel method is used to titrate for HF directly. A commercially available kit is used to measure the iron content of the fluids.

The high percentage of jobs where acid concentration varied more than 30% from the specified value suggests that analysis of acid concentrations is a necessary part of any acidizing program. The test kit described here permits the simple and rapid analysis required for a successful quality control program.

I. INTRODUCTION

Acidizing fluids, and the additives that comprise them, are the product of research and field experience. Although laboratory tests contribute to the design of acidizing fluids, field tests and experience determine the final fluid composition. Once a fluid appears to work, it, or minor variations of it, will be used routinely in a specific field or area of the country. Familiarity with a specific fluid, as it is mixed and used routinely, can give the false impression that prepa-

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ration and use of these fluids are easy. In reality, these fluids are complex mixtures which contain several critically active chemicals. One fluid may contain HCl, HF, acetic acid, a corrosion inhibitor, anti-sludging agent, foaming agent, solvent or mutual solvent, sequestering agent and others.[1,2,3] Each chemical contributes a specific function and must be compatible with others. The presence of these additives in proper concentration is always critical.[4]

Therefore, we initiated a quality control program for acids used in southern California. Our objective is to obtain the fluids we specify by:

a. Checking acid quality on the job to prevent the use of poor quality fluids.
b. Defining quality control problems so they can be solved.
c. Giving the acid suppliers more incentive to control the quality of their fluids.

This paper presents the results of a four year quality control survey and a description of the kit we used to analyze the fluids.

II. DESCRIPTION OF THE ACID QUALITY TEST KIT

The test kit, shown in Fig. 1, permits rapid analysis of HF, HCl and iron concentrations. Plastic labware is used almost exclusively in the kit to avoid the inconvenience of breakage in the field.

The HCl and HF concentrations are determined by titration; however, samples are titrated by using constant volume dispensing bottles rather than a buret. These bottles accurately and reproducibly dispense 1 ml quantities of the titrating solution. Therefore, the analytical result is a concentration range rather than a discrete concentration. This range spans 0.4% for HF, and 0.91% for HCl in the absence of HF. For example, in the HF titration, each milliliter of titrating solution represents 0.4% of HF. If six 1 ml quantities of the solution are required to reach the endpoint, the HF concentration is known to be between 2.0 and 2.4%. A chart, Fig. 2, is provided with the kit to give HCl and HF concentrations from the quantities of titrating solutions used. Values given by this chart must be corrected for sample density by a method described later in this text.

Iron concentrations are determined by using a kit that is commercially available from Hach Chemical Co. (Model IR-18A). Samples are diluted and neutralized with sodium acetate before the analysis.

Therefore, the kit contains three tests that permit rapid analysis of acidizing fluids. Approximate analysis time for each test is:

HCl - 2 minutes
HF - 2 minutes
Fe - 5 minutes

Although these tests yield valuable information about the quality of acidizing fluids, it is also important to observe the appearance of each sample. A sample that meets specifications according to our three analyses, may still be damaging if it contains unwanted solids or sludge, or if additives, such as a corrosion inhibitor are left out. Although a test for the presence or absence of corrosion inhibitor has been reported, we did not include it in our quality control survey of acidizing fluids.[5,6]

Conventionally, acid concentrations are based only on the aqueous phase. However, solvents are sometimes present in acidizing fluids in concentrations up to 10%. Therefore, if the whole sample is analyzed, the analytical result must be adjusted for the presence of solvent. For example, if the sample were specified to contain 10% HCl and 10% solvent, the correct analytical result would be 9.0% HCl because the aqueous phase comprised 90% of the sample.

A. HF Analysis

The test kit employs a novel volumetric method for determining fluoride ion concentration directly. Common volumetric analyses of HF in mud acid are indirect. These techniques measure the total acidity from both HCl and HF, and then subtract the HCl acidity determined by titration for the chlorides. Therefore, the result obtained for fluoride concentration depends on the assumption that the chloride content is entirely accounted for by the HCl added to the treating fluid. Although this is usually a good assumption, excess chlorides due to contamination will cause the HF concentration to appear lower than it actually is. To eliminate the uncertainty introduced by this possibility, we chose a direct method of analysis.

We developed a direct method using 0.05M thorium nitrate [$Th(NO_3)_4 . 4H_2O$] as a titrating solution. Although the fluoride specific ion electrode provides an excellent direct analysis for fluoride, we decided the simpler volumetric analysis is more appropriate for rapid field testing. Two thorium nitrate methods are described in the literature.[7,8] In both methods, thorium tetrafluoride (ThF_4) precipitates during the titration; at the endpoint, the excess thorium complexes with the indicator to cause the yellow to purple-red color change. These methods require strict pH control for accurate analysis. Rowley and Churchill report that a pH of 2.9 to 3.4 gives results that are stoichometrically correct, with a pH of 3.0 giving the most positive end point.[9]

The method used in our quality control survey permits analysis throughout a broader pH range, further simplifying the titration for rapid use in the field. A mixture of ethanol and ethylene glycol monobutyl ether (EGMBE) in a sodium acetate/acetic acid buffer permits analysis within a pH range of 4.2 through 2.8. Without the ethanol/EGMBE mixture, the indicator, Alizarin Red S, begins its acid-base color change from yellow to red at pH 3.7. Therefore, without this mixture, at pH values of 3.7 and higher, the endpoint is at least partially obscured by the initial red-orange color; also, at a pH of 4.2, the end point is premature by about 20%. The presence of ethanol and EGMBE preserves the initial yellow at a pH as high as 5.5, permitting a much clearer endpoint. Further, the presence of these liquids eliminates most of the error due to premature endpoint. At a pH of 4.2 the end point was 4.4% premature instead of 20% and at a pH of 3.7 it was just 1.8% premature. The effect of pH on the titration accuracy is given in Table 1.

Table 1 also shows the buffering capacity of the buffer-indicator solution. Mixtures with acid strengths equivalent to 8.28% HCl - 2.5% HF through 15.1% HCl - 2.5% can be titrated with a high degree of accuracy. Mixtures with lower acid strength are titrated with at least 95.6% accuracy. Mixtures with higher acid strengths yield indistinct end points upon titration.

Because the titration yields a concentration range rather than a discrete value, a slightly premature endpoint is unlikely to significantly affect the test kit result. For example, the titration of a 2.5% HF - 8.3% HCl solution would yield 2.46% HF. Both the actual and the determined values fall within the same concentration range determined by the kit, 2.4 - 2.8%.

In addition to permitting analysis over a broad range of acid strengths, the buffer-indicator solution prevents endpoint interference by iron III. Iron III is almost always present in acid samples and can be complexed to prevent interference.

Therefore, 1 gm/L of 1,2 (cyclohexylene dinitrilo)-tetraacetic acid (CDTA), an iron complexing agent, is added to the buffer-indicator to prevent this interference.

With all additives included, the composition of the buffer-indicator solution is:

2.0M aqueous sodium acetate	27.2 vol %
EGMBE	54.4
Ethanol	13.6
CDTA	0.1 (wt% 1 gm/L)
Glacial acetic acid	4.48
Alizarin Red S - 1% in ethanol	0.23

In practice, 5 ml of the diluted acid sample (diluted 1:5) is added to a mixture of 10 ml buffer-indicator solution and about 10 ml distilled water. The titrating solution, 0.05 M Th $(NO_3)_4$ is added in 1 ml increments until the yellow to purple-red endpoint is reached. In this system, each 1 ml increment titrates 0.4% HF. The "uncorrected HF concentrations" correlating with a wide range of $Th(NO_3)_4$ volumes used is shown on the left part of the chart in Fig. 2. These values are called uncorrected because they do not account for the density changes due to the presence of HCl and HF. Instead they yield a value for wt % HF based on the density of distilled water. Therefore, the values obtained from Fig. 2 must be divided by the sample density. The density is influenced primarily by HCl and to a lesser degree by HF in the range of concentrations encountered most in the field.

Fig. 3 shows the effect of HCl and HF concentration on fluid density in terms of incremental changes above 1.000 gm/ml for distilled water.[10,11] The sample density is taken to be equal to the sum of the increments from both HCl and HF added to the unit density of distilled water. The density of HCl solutions are plotted against the wt % HCl in the sample. This value is obtained by a method described later in the text. The densities of HF solutions are plotted against the volume of 0.05 M thorium nitrate required to reach the endpoint. In both cases, the midpoint of the concentration range determined by the test kit is used to obtain the density value. The uncorrected HF concentration is then divided by the density to yield the corrected value. For example, consider the values of HCl concentration and amount of $Th(NO_3)_4$ to be 6.8 - 8.3% and 5 - 6 ml, respectively. The density from Fig. 3 is 1.044. Therefore the corrected HF concentration is 1.92 - 2.30% HF instead of the uncorrected range of 2.0 - 2.4%.

The thorium nitrate used in this analysis emits a low level of radioactivity. The 0.05M solution, bottled in plastic, emits 0.08 millirems per hour of radioactivity over background radiation at the surface of the bottle. Background radiation was about .02 mrem/hr. No special control or monitoring is required at this level of radiation.[12] Procedures usually used in handling toxic chemicals are appropriate; these include use of gloves while doing the analyses to avoid contact of the material with skin. The California Radiation Control Regulations, published by the State of California, specify the maximum levels of radiation dosages in uncontrolled areas is:[13]

a. 2 millirems in any one hour
b. 100 millirems in any 7 consecutive days
c. 500 millirems in one year

In comparison, direct contact with a bottle of .05 $Th(NO_3)_4$ solution for 40 hours results in a dosage of 3.2 mrems. Of course, during actual use, direct contact will be limited to time periods on the order of minutes per week.

B. HCl Analysis

The HCl concentrations are determined by titration with sodium hydroxide (NaOH). Again, as in the HF analysis, 5 ml of the diluted acid sample (diluted 1:5) is titrated in 1 ml increments. The titrating solution is 0.25N aqueous NaOH that contains the endpoint indicator methyl red. Because this analysis actually yields a total acid concentration, including acid present as both HCl and HF, the HCl concentration is determined by subtracting the HF concentration from the total acid concentration. This subtraction must be done using molar concentrations rather than wt %. Fig. 2 shows the results of this subtraction for a wide range of HF and HCl concentrations. The diagonal lines on Fig. 2 have a slope equal to the ratio of sodium hydroxide to thorium nitrate solution normality, or 1.25. A value for HCl concentration can be obtained simply from the volumes of HF and HCl titrating solution required to reach their respective endpoints. If the acid were not specified to contain HF, the HCl concentration is obtained by taking the volume of HF titrating solution required to be zero.

For example, suppose the acid contained no HF and required 17-18 ml of the total acid titrating solution. The HCl concentration in wt % is obtained by moving the horizontal line for 0 ml HF titrating solution to the space between 17 and 18 ml total acid titrating solution; then, moving upward between the diagonal lines to the top of the scale, the wt % HCl is given as between 15.5 and 16.4. If the acid contained HF, a similar procedure is followed where the first horizontal line is chosen to coincide with the amount of HF titrating solution used. An example of an analysis which found 2.0-2.4% HF and 6.8-8.3% HCl is given by the shaded bars on the chart itself. These wt % HCl values are uncorrected for density and must be divided by the sample density. The density is obtained using Fig. 3 and the method previously described.

Acids occasionally contain acetic acid or citric acid to prevent secondary deposition of iron III. These acids will also be titrated with sodium hydroxide. One percent acetic acid is equivalent to 0.588% HCl, while 1% citric acid is equivalent to 0.52% HCl.

C. Iron Analysis

Iron concentrations were determined by using a commercially available colorimetric test kit, Hach Chemical Co., Model 1R-18A. The acid sample is diluted 1 to 500 prior to the analysis. This dilution effectively eliminates interferences due to colored substances present in most acids. Because the analysis must be done in a solution of pH 4-7, sodium acetate is added to the diluted solution until the pH is adjusted in this range. To check the accuracy of this procedure we analyzed dilute acid solutions with known iron concentrations of 95 and 200 ppm. The averages of four trials each were 96.8 ± 4.1 ppm and 202 ± 2.8 ppm iron, respectively.

III. RESULTS OF THE QUALITY CONTROL SURVEY

The field test kit previously described, and in a few cases, conventional laboratory analyses were used to determine the acid and iron concentrations in fluids sampled from 44 jobs in Southern California. A total of 162 acidizing fluids were sampled. The quality of fluids from five service companies were compared; however, just two companies did 77% of the jobs. The quality control survey revealed the following problems:

a. Acid concentrations were often too high or too low.
b. Frequently, fluids were not thoroughly mixed.
c. In some cases, fluids contained incompatible additives.
d. Fluids almost always contained iron, averaging 180 ppm in iron concentration.

Because of the uneven representation of service in companies in the survey, and its long duration, we infer no conclusions about their relative performances.

A. Acid Concentrations Were Often Too High or Too Low

The analytical results are interpreted in terms of the number of jobs where an acid concentration in one fluid deviated 30% or more from the specified concentration. This ±30% window was chosen because it seems:

1. Broad enough to include minor deviations in acid concentrations, which are inevitable in field applications.
2. So broad that any fluid with an acid concentration deviating outside this range could be considered unacceptable.

Therefore, the use of this window permits us to determine the frequency of jobs affected by receiving a quantity, usually at least 2000 gal, of an unacceptable fluid.

As previously mentioned, the field test yields a concentration range rather than a discrete value. To avoid a misleadingly negative interpretation of the data, the value of the range closest to the specified value was used as the analytical result. For example, if an HF solution specified to be 3.0% was found to contain 1.92 - 2.30% HF, we considered it to be deviated "23% or more" from the specified value, even though it could be deviated by 36%.

In almost all cases, acids were sampled by dipping a container a few feet below the fluid level in the transport tank after the fluid had been mixed. The results of both HCl and HF analyses show that in 18 of 44 jobs surveyed, or 41%, at least one of the solutions varied more than 30% from the specified values. Table 2 shows the frequency and range of variation of the worst fluid on each of the 44 jobs. On almost all jobs surveyed, acid was delivered in tank transports. Usually more than one transport was required. Each transport, or in some cases, each compartment within one transport contained a separately mixed acid solution. Therefore, several separate mixtures were used on each job. In all, 162 fluids were analyzed. On most jobs, each of these mixtures was analyzed to determine the frequency of jobs where at least one fluid could be considered unacceptable.

During our survey, the percentage of jobs where one fluid varied outside the 30% window decreased during the second and third years of the survey and increased during the fourth year. The first year, 1978, 4 of 7 jobs had at least one fluid that varied greater than 30%. In the following two years, 26% and 28% of the jobs had at least one unacceptable fluid. During those two years, 24 and 7 jobs were surveyed, respectively. During the last year of the survey 3 of 6 jobs had fluids varying outside the window.

Although these results show a high percentage of jobs had at least one unacceptable fluid, a lower percentage of the 162 individual fluids deviated outside the window. The deviation of acid concentrations is shown in Figs. 4 and 5. Fig. 4 shows that 18% of the HCl concentrations varied outside the 30% window while 35% of the HF solutions were unacceptable, as shown in Fig. 5. The deviations occurred both above and below the specified values with similar frequency.

Mancillas and Burroughs found that concentrated acid received from the chemical manufacturers can sometimes contain varying amounts of HCl.[14] They found, in one case, that 20°Be' HCl which should have contained 31.45% HCl, actually contained 24% HCl. Although the frequency of such variations was not reported, it is possible that some of the variation we observed in field acids were due to variations in the concentrates used to prepare them.

B. Frequently, Fluids were Not Thoroughly Mixed

Often fluids contained additives in the proper concentrations but were not thoroughly mixed. Poor mixing of acidizing fluids was also observed as a problem by Mancillas and Burroughs in 1974.[15] We found it necessary to mix fluids for about 15-30 minutes by injecting air through them or by circulating them through pumps. Agitation of the fluid in the transport compartment while driving to location was not adequate. In one case, after the fluid was transported on the road 25 miles, the concentration of HF at the top of the tank was 0.64%, while at the bottom it was 11.4%. After mixing for 20 minutes by air injection, the HF concentration was uniform and near the specified value of 3.0%.

In addition to the problem of acid settling to the bottom of mixtures, many organic additives float to the top. These include solvents added to the acid solution and surfactants, such as the corrosion inhibitor. Often, samples from the top of a tank were much more concentrated in the organic additives than samples from the bottom.

C. In Some Cases, Fluids Contained Incompatible Additives

Acidizing fluids often contain several surfactants with specific functions. On several occasions, although additives were present in their specified concentrations, they were incompatible. Fig. 6 shows a mixture exhibiting phase separation because of a chemical incompatibility. The mixture should have been one phase, containing 5% EGMBE as a mutual solvent. However, the corrosion inhibitor, a cationic surfactant, formed a complex with the anionic anti-sludging agent, and partitioned into the top phase which contained a mixture of 10% HCl and EGMBE. The lower phase also contained 10% HCl and a smaller amount of EGMBE. The four mixtures in Fig. 6 show the effect of this phase separation on the acid pumped downhole. The mixture on the left was taken from the tank before the job. The other three mixtures were taken from the wellhead during pumping. These three samples arranged left to right represent acid pumped at the beginning, middle and end of the job. They show an increasing amount of EGMBE in the acid as the job progressed, indicating the phase separation occurred to a significant extent in the tank. The first acid pumped contained just 2.7% EGMBE determined by N.M.R. spectroscopy. The mixture at the right contains a small amount of particulates identified as mixture of the anti-sludging agent, corrosion inhibitor and EGMBE by I.R. spectroscopy. In this case, the lower phase contained enough corrosion inhibitor that was not complexed to provide adequate protection of the pipe.

Although we encountered this formulation just once in our survey, it had been used several times in that field. Subsequently, an alternative additive mixture was used.

We also found the anti-sludging agent tended to destabilize solvent-acid emulsions and adversely affect foam stability of foamed acid mixtures. In one case, a foamed acid solution that contained the specified amounts of foaming agent and anti-sludging agent, did not produce a foam or froth when beaten for two minutes in an egg beater. A synthetic acid mixture that did not contain the anti-sludging agent but was otherwise identical to the field mixture readily foamed when beaten, producing a drain half-life of about 4 minutes in a 500 ml graduated cylinder.

D. Fluids Always Contained Iron, Averaging 180 ppm Fe

Other authors have suggested that iron III in acidizing fluids will be reduced to iron II as it contacts iron pipe.[16,17] Therefore, if they are correct, iron III initially present in acid will not precipitate as $Fe(OH)_3$ as acid spends in the formation. Also, the concentration of iron in the acid may be minor compared to the iron dissolved from corrosion products in the pipe and minerals in the formation. However, we have not investigated the reduction of iron during injection so we cannot specify a maximum iron content based on the prevention of formation damage. Instead, to be conservative, we requested that iron concentrations be kept below 100 ppm.

Although almost all acids we sampled contained iron, the average iron concentration decreased from 455 ppm in 1978 to 114 ppm in 1981. Further, the percentage of jobs where at least one fluid contained more than 200 ppm iron decreased from 81% to 0 from 1978 to 1981. This is due in part to the increased use of lined transport tanks during the last two years.

IV. CONCLUSIONS

A. The acid test kit permits rapid on-site analysis of acidizing fluids.

B. On-site analysis has prevented the use of poorly mixed fluids.

C. The quality control survey has defined the following problems:

1. Acid concentrations were often too high or too low.
2. Frequently, fluids were not thoroughly mixed.
3. In some cases, fluids contained incompatible additives.

D. Acid almost always contained iron, averaging 180 ppm Fe.

E. Routine analysis of acid concentrations is a necessary part of any acidizing program.

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NOMENCLATURE

°Be' = degrees Baume'
M = molar (moles per liter)
mrem/hr = millirems per hour
ρ_{sample} = density of the acid sample, gm/ml

$\Delta\rho_{HCl}$ = incremental change in density due to the presence of HCl, gm/ml
$\Delta\rho_{HF}$ = incremental change in density due to the presence of HF, gm/ml

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TABLE 1: Effect of acid strength on buffer pH and HF analysis.

Acid Composition X % HCl - 2.5% HF	Acid normality	pH of Sample with Buffer	Accuracy of Analysis, %
X = 14.9	5.40	2.80	100
11.9	4.70	3.10	100
8.1	3.52	3.70	98.2
3.9	2.35	4.20	95.6
0	1.25	4.45	90.3

TABLE 2: Frequency and extent of variation from specified values.

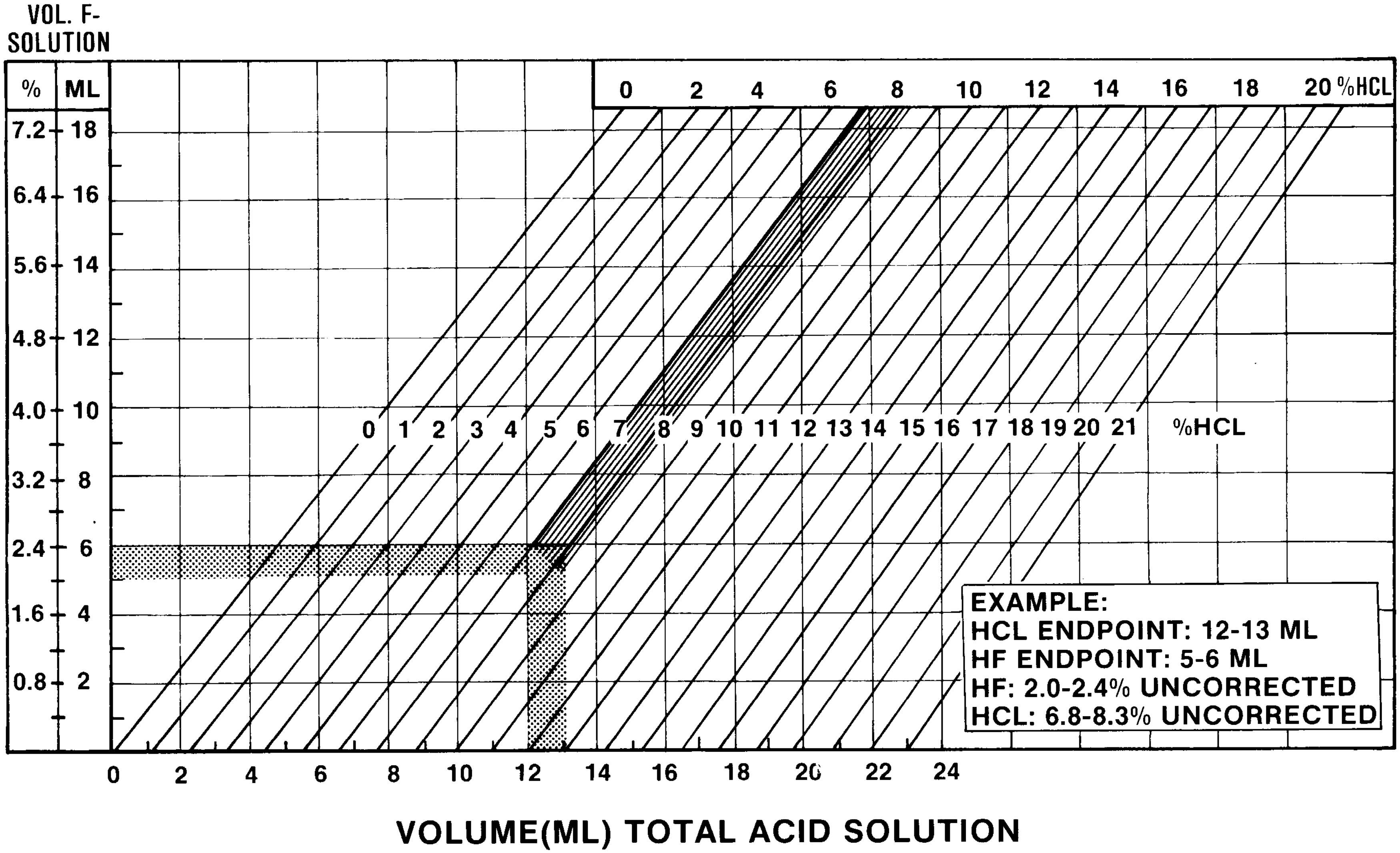
	Frequency of Variations (Worst Fluid per Job)	
Extent of Variation from Specified Values (%)	No. of Jobs	Cumulative % of Jobs Varying ≥ Indicated Value
<10	11	100.0%
≥10	7	75.0
15	4	59.1
20	4	50.0
25	0	40.9
30	4	40.9
40	4	31.8
50	4	22.7
60	2	13.7
70	1	9.1
80	2	6.8
90	0	2.3
100	1	2.3


HF TEST BUFFER-INDICATOR 2 SHOTS (10 ML) PER TEST
1 ml PER SHOT TOTAL ACID TEST
ACID QUALITY TEST KIT
HACH
IRON TEST
FIELD ACID SAMPLE
HF TEST 1 ML PER SHOT

FIGURE 1

FIGURE 2

UNCORRECTED ACID CONCENTRATIONS



EXAMPLE:
HCL ENDPOINT: 12-13 ML
HF ENDPOINT: 5-6 ML
HF: 2.0-2.4% UNCORRECTED
HCL: 6.8-8.3% UNCORRECTED

FIGURE 3

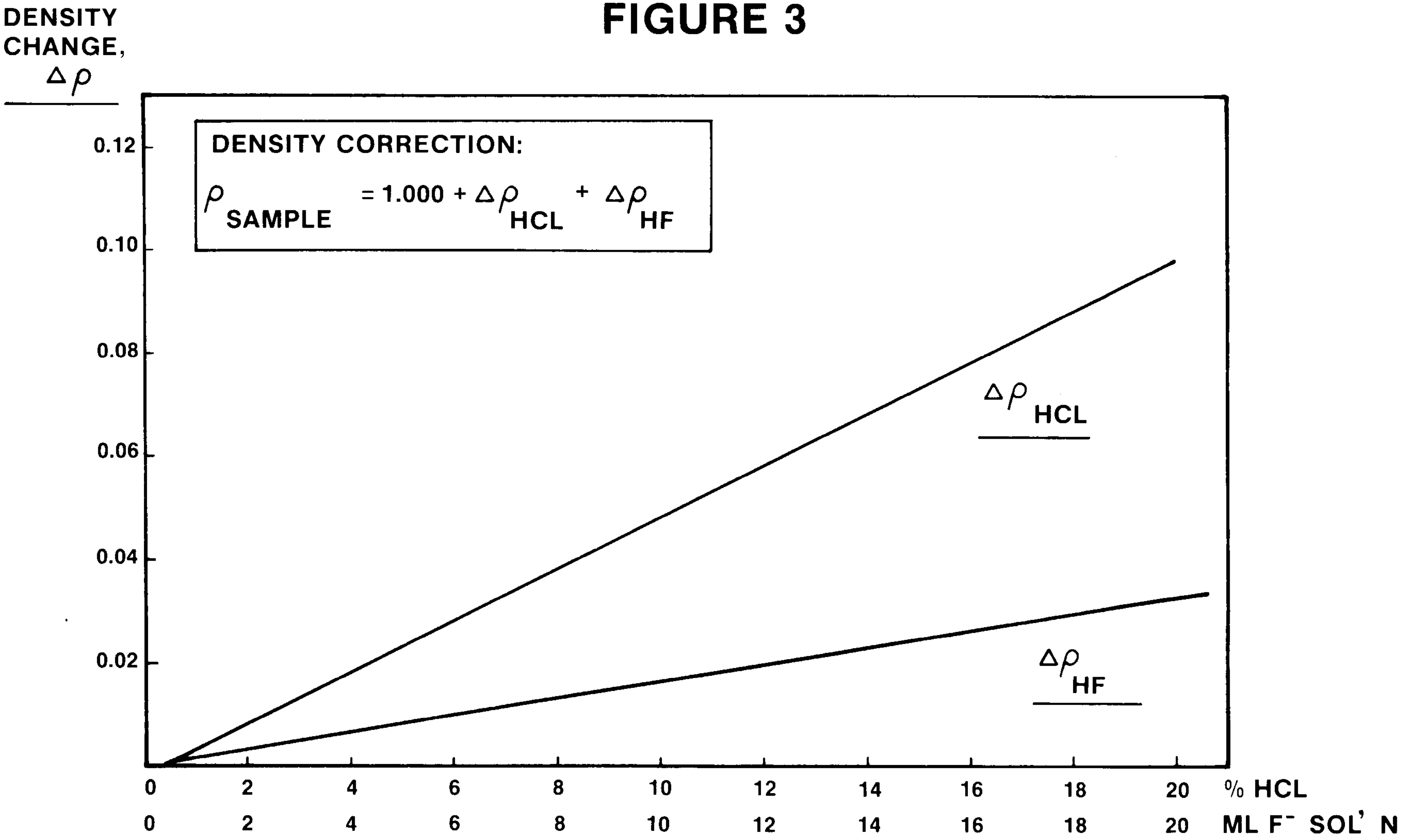

DENSITY CHANGE, $\Delta\rho$
DENSITY CORRECTION:
$\rho_{SAMPLE} = 1.000 + \Delta\rho_{HCL} + \Delta\rho_{HF}$
0.12
0.10
0.08
0.06
0.04
0.02
$\Delta\rho_{HCL}$
$\Delta\rho_{HF}$
0 2 4 6 8 10 12 14 16 18 20 % HCL
0 2 4 6 8 10 12 14 16 18 20 ML F⁻ SOL' N

FIGURE 4: HCL CONCENTRATIONS

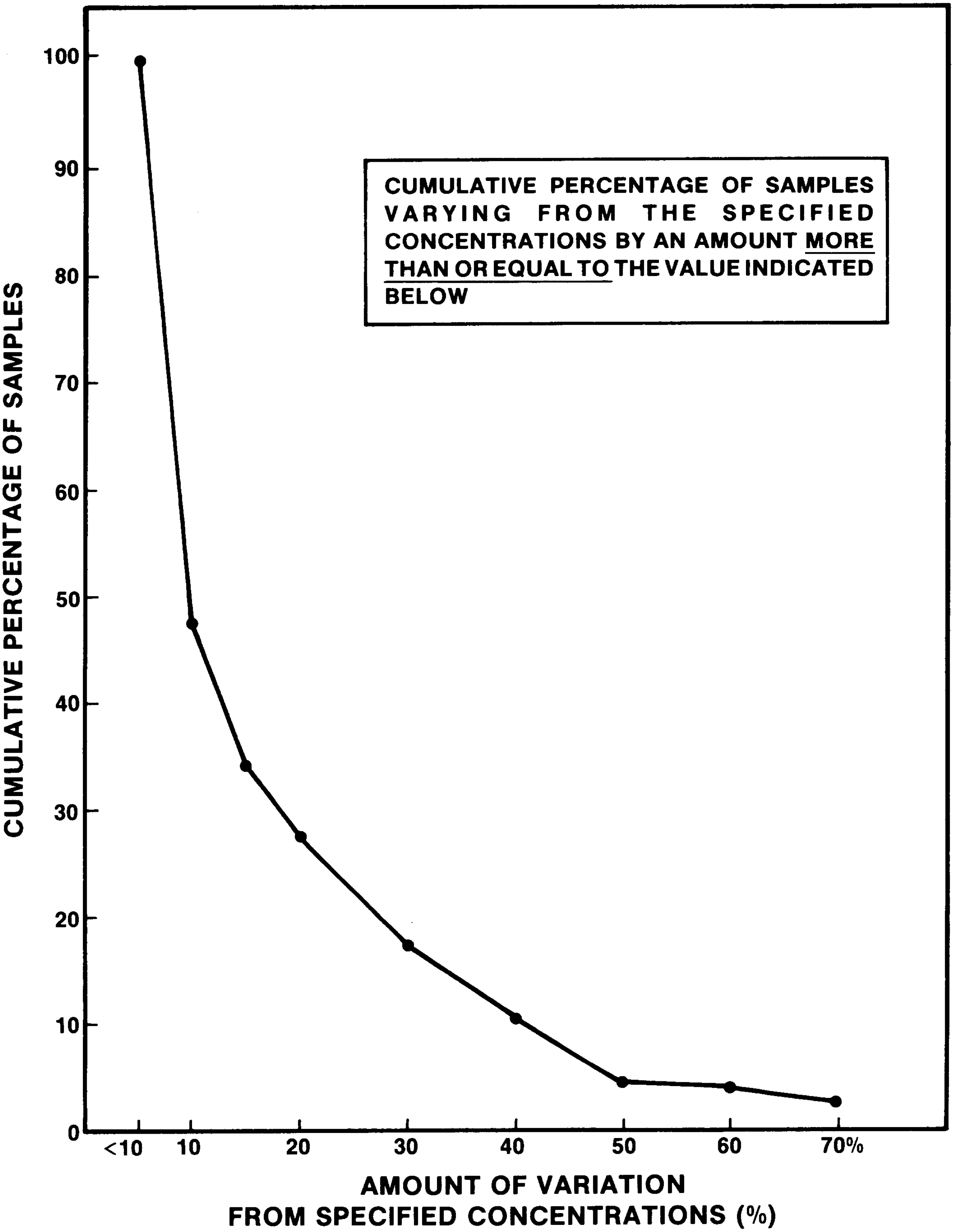


FIGURE 5: HF CONCENTRATIONS

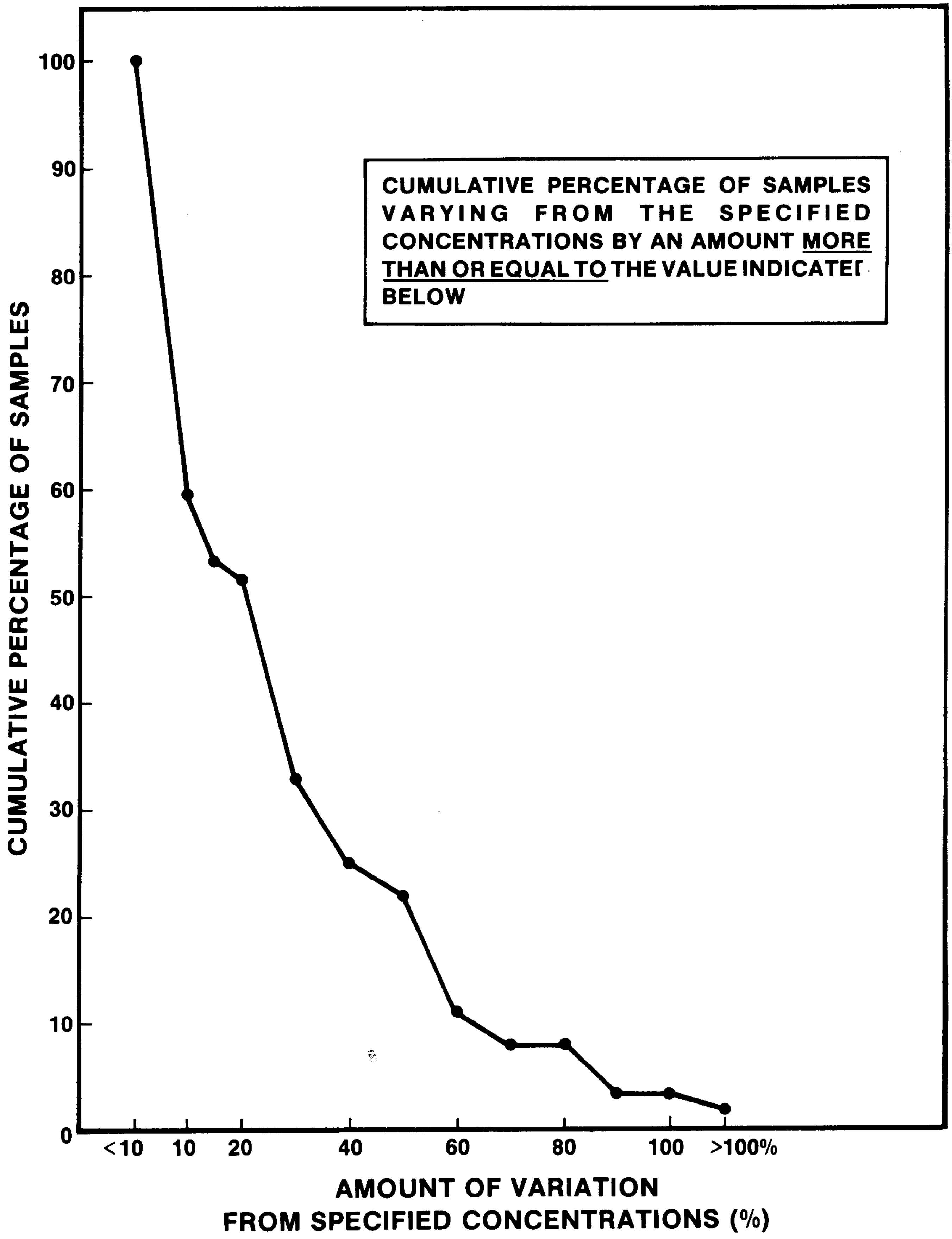


FIGURE 6

CHEMICAL INCOMPATIBILITY