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CENTRALIZED VETERINARY LABORATORY QUALITY ASSURANCE STUDY - I

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Final Report

Prepared for:

UNITED STATES ARMY HEALTH SERVICES COMMAND (HSVS)
Fort Sam Houston, Texas 78234

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This report covers intralaboratory and interlaboratory aspects of a complete veterinary food testing laboratory quality assurance program for Health Services Command (HSC). Each CONUS HSC veterinary food testing laboratory was surveyed to determine the status of existing intralaboratory quality assurance programs. Results of this survey indicate that all participating laboratories are currently conducting comprehensive programs in the area of evaluation of equipment. However, considerable variance exists between laboratories in methods

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employed for the evaluation of precision of analytical results, the evaluation of accuracy of analytical results, the analysis of intralaboratory quality assurance data, and the application and interpretation of results obtained by statistical analysis of intralaboratory quality assurance data. There was insufficient interlaboratory data with which to derive an objective estimate of basic confidence levels associated with each analytic procedure. Therefore, a modified delphi method was employed to derive relative estimates of these levels. These relative estimates were combined linearly with workload data for the eleven most commonly performed chemical analyses. The resulting vector is a basis for the allocation of analytical quality control resources in a manner which provides the greatest potential gain in overall laboratory quality assurance level per unit of quality control resource expended.

SUMMARY

This report covers both intralaboratory and interlaboratory aspects of a complete veterinary food testing laboratory quality assurance program for the Health Services Command (HSC).

Each Continental United States HSC veterinary food testing laboratory was surveyed to determine the status of existing intralaboratory quality assurance programs. Results of this survey indicate that all participating laboratories are currently conducting comprehensive programs in the area of evaluation of equipment. However, considerable variance exists between laboratories in methods employed for the evaluation of precision of analytical results, the evaluation of accuracy of analytical results, the analysis of intralaboratory quality assurance data, and the application and interpretation of results obtained by statistical analysis of intralaboratory quality assurance data.

There was insufficient interlaboratory data with which to derive an objective estimate of basic confidence levels associated with each individual analytic procedure. Therefore, a modified delphi method was employed to derive relative estimates of these levels. These relative estimates were combined linearly with workload data for the eleven most commonly performed chemical analyses. The resulting vector provides an estimate of the relative potential overall gain in quality assurance levels associated with each procedure. Specifically, those procedures with the lower initial confidence levels offer the greater potential marginal gain in overall laboratory quality assurance level.

On the basis of a content analysis of existing intralaboratory quality assurance programs and the results obtained from the subjective evaluation of basic confidence levels, a central quality assurance program is proposed which complements existing intralaboratory programs. The basic central quality assurance program is outlined in the attached chart.

OUTLINE OF A CENTRALIZED QUALITY ASSURANCE (CQA) PROGRAM

	Quality Assurance Area	CQA Task	Frequency	Category of Personnel Required
A.	Evaluation of equipment	1. Perform on-site surveys of intralaboratory programs	1. Annually	Senior Vet Lab Off (VLO) Senior Lab Analyst
		2. Conduct random spot checks of specific items	2. "	
		3. Report on findings	3. "	
B.	Evaluation of the precision of analytical results	1. Review split sample and recycling procedures during on-site visits	1. Annually	Senior VLO Senior Analyst
		2. Evaluate precision on interlaboratory level	2. With each interlab sample	
C.	Evaluation of the accuracy of analytical results	1. Conduct an interlaboratory quality assurance sample program	1. Ongoing	Senior VLO Senior Analyst Lab Technician
D.	Evaluation of the current state of the art	1. Conduct ongoing literature and methodology review	1. Ongoing	Senior VLO Senior Lab Analyst Lab Technician
		2. Provide technical data to HSC area labs	2. "	
		3. Evaluate methodology during on-site visits	3. Annually	
E.	Evaluation of sample control	1. Review and evaluate laboratory procedures for identification, storage, routing, and pre-analysis preparation of both routine and quality assurance samples during on-site survey.	1. Annually	Senior VLO Senior Analyst

Preface

This study is specifically oriented to the US Army Continental United States veterinary food testing laboratory activities. However, the approach and techniques developed and recommended herein have broader applicability and are potentially useful to other military and civilian laboratories in their quality control efforts.

TABLE OF CONTENTS

1. INTRODUCTION.

a. Purpose.

This study was conducted for the purpose of developing a complete centralized laboratory quality assurance program for the five Continental United States (CONUS) Veterinary Food Testing Laboratories of the United States Army Health Services Command (HSC). Once developed and implemented, this program is designed to provide the veterinary laboratory personnel with a continuing source of information with which they may better evaluate their laboratory test reliability status. In addition, this program is intended to provide HSC with a data base for evaluation of interlaboratory test reliability. Thus, the scope of this study includes both intra- and interlaboratory quality assurance procedures for the five CONUS HSC Veterinary Food Testing Laboratories.

b. Background.

(1) As a result of the 1973 Army reorganization, the five CONUS Area Medical Laboratories became the responsibility of the United States Army Health Services Command. The veterinary division of each of these laboratories conducts procurement quality requirements testing in support of the Department of Defense (DOD) subsistence procurement program as well as wholesomeness testing of government-owned foods. Since 1970, the combined procurement and surveillance phases of this program have accounted for more than eighteen billion pounds of subsistence annually.[1] Army laboratory findings directly influence the disposition of these subsistence items and, therefore, even small analytic errors have the potential for resulting in large public health and/or economic impact.

(2) Prior to the formation of HSC, interlaboratory quality assurance (technical supervision) for these laboratories was conducted by the Veterinary Division of the Walter Reed Army Institute of Research (WRAIR), Walter Reed Army Medical Center, Washington, D.C. In addition, each laboratory conducted an independent intralaboratory quality assurance program following general guidelines provided by WRAIR.

2. OBJECTIVE. To survey existing intralaboratory procedures and to identify the associated HSC-level interlaboratory tasks required for accomplishing the following:

a. Evaluation of equipment.

b. Evaluation of the precision of the analytical procedure.

c. Evaluation of the accuracy of the analytical procedure.

d. Statistical methods (such as control chart procedures) for evaluating results.

e. Evaluation of the present state-of-the-art.

f. Evaluation of the control of samples within the laboratory.

3. METHODOLOGY.

a. Overview.

The basic approach to this study was to perform a comprehensive survey of existing intralaboratory programs within the HSC system. This survey data was then combined with laboratory workload data for the purpose of describing the levels at which a centralized HSC quality assurance program should operate. The rationale for this approach is as follows:

(1) For each procedure to be included in the program, there exists some basic confidence level[2] which is independent of quality assurance measures. This basic confidence level is a partial function of externalities such as analyst experience.[3] These externalities, while not precisely quantifiable in a completely objective sense, are real and must be considered in any formulation representing overall confidence in an analytic procedure.

(2) Graphically (See Figure 1) the basic confidence level is shown as the intercept on the Y axis. As quality control measures are applied to a procedure, the confidence level tends to increase until it asymptotically approaches an upper limit as N becomes larger.

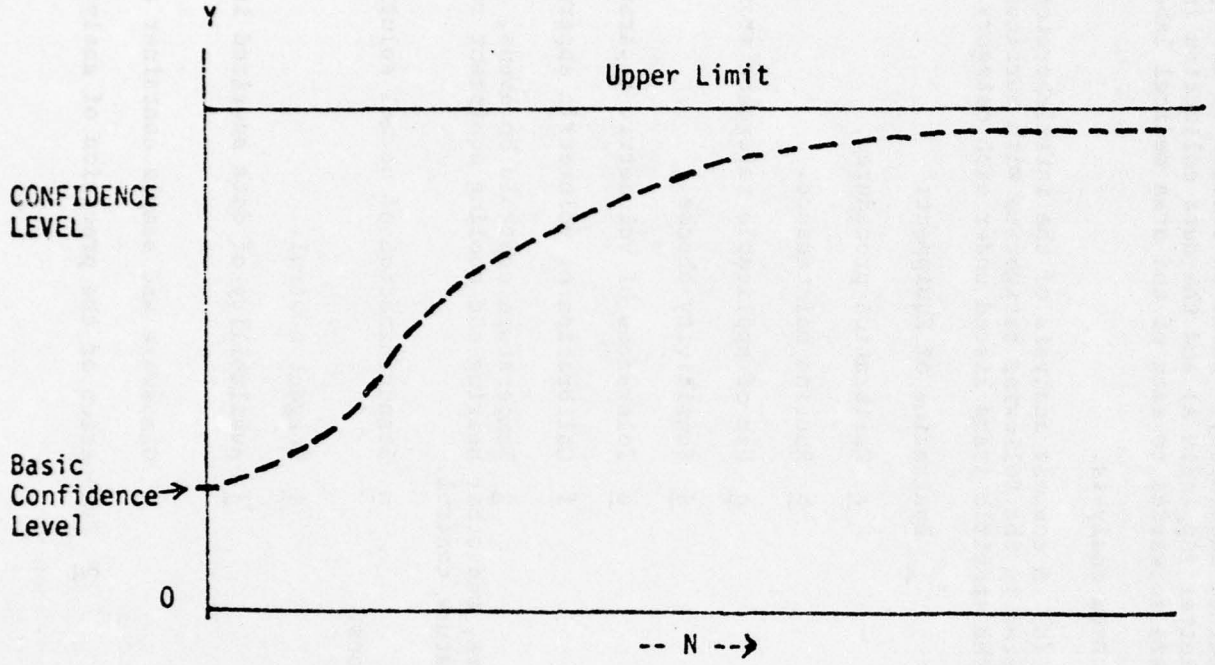
(3) Having thus described an implicit function of the basic confidence level and the number (N) of quality assurance procedures, it becomes possible to select a level of quality assurance on the basis of marginal return per incremental increase in N. More specifically, as the slope of the confidence level curve increases, the incremental return per unit of N (quality assurance procedures) increases. It follows that a quality control program should be most heavily weighted toward providing greater levels of quality control for those procedures with higher marginal return.

b. Procedures.

(1) Data Collection.

(a) Much of the data necessary for the conduct of this study was extracted from existing records and reports. This

FIGURE 1



was accomplished during visits to HSC Headquarters, to the Veterinary Division of WRAIR, and to the individual laboratories.

(b) Certain additional data for this study was provided by supervisory personnel of the area medical laboratory veterinary services. In order to obtain this information, data collection instruments were designed and evaluated in a pretest phase that was conducted with the Veterinary Department, Fort Sam Houston Area Medical Laboratory. Based on results of this pretest phase a cover letter (Appendix A) and the data collection instruments (Appendix B) were forwarded to each of the area medical laboratories.

(2) Data analysis.

(a) A content analysis of the intralaboratory response data was conducted in the following categories with particular attention given to the specific items listed under each category.

1 Evaluation of Equipment:

a Calibration procedures.

b Routine maintenance.

c Use of applicable reference standards.

d Sensitivity checks.

e Tolerances of volumetric apparatus.

f Calibration of volumetric apparatus.

g Temperature controls on ovens, refrigerators, incubators, and other heating and cooling equipment requiring precise temperature control.

h Standardization of normal solutions and control solutions.

i Reagent control.

j Availability of data acquired in equipment evaluation.

k Glassware and sample container control.

2 Evaluation of the precision of analytical results:

a Use of duplicate samples.

b Recycled samples.

c Sample control procedures.

3 Evaluation of the accuracy of analytical results:

a Extra laboratory source samples.

b Control samples.

c Primary standards employed.

4 Statistical analysis of quality assurance data:

a Frequency.

b Criteria employed.

c Use of control charts.

5 Interpretation of results obtained by statistical analysis of quality assurance data:

a Control limits employed.

b Allowable variation between duplicates.

(b) Workload data was extracted from medical laboratory activities reports and used with American Society of Clinical Pathologists (ASCP) weights to obtain an ordered scaling of the most commonly performed chemical analyses.

(c) Subjective response data regarding the relative degrees of indeterminate error associated with each of the most commonly performed chemical analyses was analyzed by a modified delphi procedure.[4] (See Appendix C). The resulting ordinal scale of procedures was converted to an interval scale by the method of scaling successive intervals.[5]

(d) Finally, the resulting interval scale was linearly combined with the frequency of performance scale to obtain a combined inclusion vector of interlaboratory quality assurance procedures.

(e) Testing capability data was provided by each laboratory and is summarized in Appendix D. This information defines

the potential scope of a "complete" CONUS Army interlaboratory sampling program, which could include up to 210 different procedures to be individually sampled.

4. FINDINGS.

a. Intralaboratory quality assurance activities (as reported by each laboratory).

(1) Evaluation of equipment.

(a) Fort Meade, Maryland. Instrumentation is calibrated following manufacturers recommended procedures. Accuracy of instrumentation is maintained by use of appropriate reference standards. Analytical balances and weighing devices are calibrated at the beginning of each weighing session, then verified at the end of each session. Periodically, sensitivity is determined as recommended by the manufacturer. Annually, a complete service check of each balance is performed by a professional service technician. Volumetric apparatus, which conforms to National Bureau of Standards (NBS) tolerances, is purchased as dictated by the procedure being used; also, standardization of this type of equipment is verified by "in-house" calibration. Ovens, refrigerators, incubators, and other heating and cooling equipment requiring specific temperatures are recorded in a daily log. Normal solutions and control samples are standardized against primary standards using official procedures. All raw data acquired in performing the calibrations and standareizations are permanently recorded for future reference.

(b) Fort McPherson, Georgia. Quarterly and annual maintenance of equipment is performed by laboratory maintenance personnel. Service contracts are maintained for the balances and gas chromatographs. Laboratory personnel make daily temperature checks of refrigerators, freezers, incubators and waterbaths. Balances, pH meters, and gas chromatographs are calibrated with approved standards.

(c) Fort Sam Houston, Texas. All equipment requiring calibration, such as centrifuges and waterbaths, are calibrated at least annually by the laboratory maintenance section. All repairs and calibrations are entered on DA Form 2409 for all major equipment items and maintained on file by the maintenance section. Quarterly, semiannual, and annual inspections are performed as required.

(d) St. Louis, Missouri. Routine inspection and maintenance of laboratory equipment is performed by Medical Maintenance technicians. Preventive maintenance is performed for equipment

quarterly, semiannually, and annually, depending on the type, in accordance with HSC Regulation 750-1. Daily records are kept on 3x5 and 5x7 cards of temperature for incubators and water baths, and drying ovens by department technicians. Standardization of burettes and pipettes is performed quarterly or when new ones are put in use. These records are kept in a special log book.

(e) Fort Baker, California. The most frequently used equipment is evaluated on a routine basis. pH meters are calibrated with each use using standard buffer solutions as well as making inter-instrument comparison. Background profiles are run on the gas chromatograph on a monthly basis and known external and internal standards are utilized with each run to assure proper functioning of the instrument. Analytical balances are calibrated monthly using NBS Class S Standard weights and daily temperature control charts are maintained on ovens, freezers and refrigerators. In addition, a Tem-Scribe records permanent graphs of any temperature variation in the incubators. There are also various pieces of equipment, such as the -60° C freezer used for storage of live rabies virus, which have a built in alarm system in the event of malfunction or power failure.

(2) Evaluation of the precision of analytical results.

(a) Fort Meade, Maryland. Tests are performed in duplicate; results that exceed established standard deviations are repeated until acceptable values are obtained.

(b) Fort McPherson, Georgia. Duplicate determinations are performed and reported results must be within specified variances as stated by the official methods.

(c) Fort Sam Houston, Texas. Most tests are performed in duplicate and test results of duplicates are required to be within a specified range of variance.

(d) St. Louis, Missouri. All samples are run in duplicate and are recycled approximately once per week.

(e) Fort Baker, California. There exist established limits for variation between duplicate results for routine tests. These limits are based upon the evaluation of precision in previous laboratory results, and vary according to the inherent accuracy of the method and the characteristics of the product. These limits are:

Fat: .03% milk, buttermilk, evaporated milk, skim milk, yogurt

.05% cottage cheese, ice cream, sour cream, whipping cream

.50% meat products

Total Solids or Moisture:

.03% milk

.10% cottage cheese

.50% meat products

Salt: .02- .03% products with 2% or less salt

.05% products greater than 2% salt

In the testing of nonroutine samples, the limits are determined by extrapolation from values previously established for similar analyses performed on products of comparable physical and chemical characteristics.

(3) Evaluation of the accuracy of analytical results.

(a) Fort Meade, Maryland. Control samples, with established test values and standard deviations, are run concurrently with test samples. Accuracy of the test system is evaluated in terms of results obtained on the control sample.

(b) Fort McPherson, Georgia. Depending upon the test, results are compared to a standard or control. The external Quality Control (QC) program from Walter Reed also assists in the evaluation of accuracy.

(c) Fort Sam Houston, Texas. Analytical results of the test sample are compared to the results of a standard or control sample.

(d) Fort Baker, California. Two commercial products, Spam and Similac, are used as quality control standards in meat and dairy chemistry, respectively. Duplicate tests of these products are run by each technician every time a procedure is performed. These results give a check against systematic errors, and allow an evaluation of the quality of work performed by each technician. If the results of a quality control test do not fall within the

established acceptable values, the most probable cause for error is determined and corrected, and the samples retested. Solutions of known composition are used as controls whenever possible. National Bureau of Standards reagents are used as standards in analyses for sugars while Environmental Protection Agency pesticide standards are used in gas chromatography. Other controls routinely employed are reagent blanks, quality tests on reagents (e.g., testing for peroxides in ethyl ether), frequent standardization of solutions with primary standards, purification of reagents when necessary, and frequent routine regeneration of dessicant.

(4) Statistical analysis of quality assurance data.

(a) Fort Meade, Maryland. Control charts are maintained that represent mean values and upper/lower confidence levels on internal control samples.

(b) Fort McPherson, Georgia. Logs are maintained of all quality control test samples; however, no statistical analysis is performed other than "eyeballing" the results. Judgmental analyses are made by the technician and supervisor. The data maintained is such that a "true" statistical analysis could be made at any time.

(c) Fort Sam Houston, Texas. Subsistence items used as control samples for each test procedure are procured in case lots. Examples of such items are Similac for butterfat test controls and potted meat as a salt test control. Initially a minimum of 20 tests are conducted on each new lot of control item to determine the mean, standard deviation, and standard error for that product. With the accumulation of additional test data, the statistics on the product are revised.

(d) St. Louis, Missouri. Quality control charts are maintained weekly: Deviation between two successive results on the same sample are calculated and graphed. Those deviations falling outside the upper and lower allowable limits are investigated to ascertain the problems involved with the test.

(e) Fort Baker, California. Statistical analysis has been used to determine the acceptable values for quality control standards and to determine the validity of Spam and Similac for quality control use. The composition of the products used as controls must not vary significantly from can-to-can if the control is to be useful. A one-way classification test was performed on Spam and Similac and the can-to-can variation was found to be not significant. The "true" values for Spam and Similac were determined by taking an

average. Inasmuch as random errors cause equal deviation from both sides of the mean, it was assumed that systematic errors had been eliminated due to the use of the controls discussed previously. The composition of Similac given by the manufacturer was used to determine the expected values for percent butterfat and total solids. This determination compared favorably with laboratory results. The mean and the standard deviation for Spam and Similac have been calculated, using all but outlying results obtained over roughly one month's time. Twice the standard deviation, which is a 95% confidence interval, is being used as the acceptable range. Daily quality control results are plotted and evaluated for deviations from this confidence interval, any outlying results being immediately suspect due to the probability of their occurance. In addition to the methods and products used to evaluate quality control for procedures on routinely received samples, all procedures performed on a nonroutine basis are evaluated for accuracy and precision through the use of applicable standards and calibration equipment.

(5) Interpretation of results obtained by statistical analysis of quality assurance data.

(a) Fort Meade, Maryland. As laboratory data accumulates, the degree of scatter and displacement from the accepted reference values becomes evident, necessitating corrective action.

(b) Fort McPherson, Georgia. Judgmental determinations are used. The official methods (AOAC, Standard Methods) state the allowable variation between duplicates and between technicians.

(c) Fort Sam Houston, Texas. Graphs are constructed and posted for each analytical procedure for which a control has been devised. On the graph is plotted the mean value and two standard deviations above and below the mean. Each time the specific analytical procedure is performed, a control sample is included along with the test samples. The test value of the control sample is plotted on the graph. The graph permits the detection of situations in which the analytical procedure is out of control or when there is a trend for the results to be above or below the mean value.

b. Vector of most commonly performed chemical analyses is as follows:

Procedure	Relative Value
SNF	.0019
pH	.0069
Cryoscope	.0094
Protein (Meat)	.0161
Acidity	.0230

Moisture (Meat	.0514
Protein (Dairy)	.0625
Salt	.0634
Soxhlet	.1786
Moisture (Dairy)	.1808
R-G (Mojonnier)	.4059

c. Vector of subjective response data is:

Procedure	Relative Level
Cryoscope	.0835
pH	.0839
Moisture (Meat)	.0873
Acidity	.0881
Moisture (Dairy)	.0886
R-G (Mojonnier)	.0897
Protein (Dairy)	.0913
Protein (Meat)	.0919
Soxhlet	.0959
SNF	.0976
Salt	.1024

d. Combined inclusion vector is:

Procedure	Relative Level
pH	.0454
Cryoscope	.0464
SNF	.0497
Protein (Meat)	.0540
Acidity	.0555
Moisture (Meat)	.0693
Protein (Dairy)	.0769
Salt	.0829
Moisture (Dairy)	.1347
Soxhlet	.1372
R-G (Mojonnier)	.2478

5. DISCUSSION.

a. Evaluation of equipment. Each participating laboratory is conducting a comprehensive program for accomplishing the maintenance, calibration, standardization and related quality assurance activities. Records of these activities are routinely maintained by each laboratory. Instructions and guidance for this program are contained in technical letters[6] provided by the Veterinary Division of WRAIR. However, the

individual laboratory retains full responsibility for internal standards and reagents with no formal interlaboratory comparability checks.

b. Evaluation of precision. A major difference between existing intralaboratory precision evaluation programs is that some laboratories are currently expending a substantial amount of resources in their intralaboratory control sampling efforts while others rely heavily upon the criteria of satisfactory agreement between duplicates. In the development of a centralized HSC quality assurance program it is important to recall that parallel duplicates by the same analyst require the application of a "total error" concept. Each analytic procedure is an intricately designed series of events, each of which is a potential source of analytic error. The combination of independent analytic error associated with each step and the analytic error due to interaction effects between steps all combine to form "total error."[7] In addition, consideration must be given to the question of whether the analytic error is random or systematic in nature. Clearly, the "satisfactory agreement" criteria alone does little to assist the laboratory analyst in defining or identifying the type or source of analytic error.

c. Evaluation of accuracy.

(1) As Youden[2] has pointed out, "Good agreement between duplicates is a necessary but not a sufficient condition for a good procedure." Thus, any complete quality assurance program must contain provisions for evaluating accuracy as well as precision. Existing intralaboratory accuracy testing is somewhat constrained by the limited availability of reference standards and "known" control samples. Therefore, the need for an ongoing interlaboratory accuracy sample program is clear. The composition of each interlaboratory quality assurance sample is of course a matter for judgemental determination by the analysts in charge of the program. However, the potential gain in overall quality assurance levels associated with each type of analytic procedure to be conducted is quantifiable and can serve as the basis for determining allocation of laboratory resources within the CQA program. A recommended vector of interlaboratory procedures (see para 4a(5)(d)) was developed for this purpose. This vector is a basis for the allocation of analytical quality control resources in a manner which, on a subjective basis, provides the greatest potential gain in overall laboratory quality assurance level per unit of quality control resource expended. In other words, rather than conducting a quality assurance program that includes all 210 procedures, this vector permits use of a lesser number of interlaboratory samples while still maintaining an acceptably high level of overall laboratory quality assurance. It should be emphasized that this recommended

inclusion vector was derived from subjective data and, therefore, should be critically evaluated and readjusted as objective interlaboratory data becomes available. Also, this vector includes only those most commonly performed chemical analyses. Clearly, this list may change with changing workload requirements, and should be reconstructed accordingly. Further, those analyses not currently included in the vector should be included in the interlaboratory program on a less frequent basis (semiannually or annually) as resources permit.

(2) The subjective response basis for determining basic confidence levels (Matrix A) was designed around the concept that, because all laboratories perform the same basic procedures in the same manner, there should be a fairly high level of subjective agreement as to which procedures were inherently more reliable than others. Results of the Matrix A responses are summarized by laboratory in Table 1. Using a rank order statistic[8] at the .05 level, the lowest column sum must be less than 12 and the highest must be greater than 48 for statistical significance. As shown in the table, the level of interlaboratory agreement is too small to produce the required significance levels. A detailed attempt to isolate and identify these lab-associated factors would be an interesting project but is not within the scope of this study.

d. Evaluation of statistical analyses.

(1) Some laboratories are currently conducting comprehensive and meaningful statistical analyses of intralaboratory sampling data while others rely upon less quantitative procedures. In a system in which only random errors exist (and small ones at that), there need be no particular concern with the application of statistics to routine quality assurance data. However, in our laboratory system where total analytic error is usually assignable by both source and type, statistical techniques are very useful for separating the assignable and random causes of analytic error and for detecting the presence of these assignable causes.[7] Further, because a system of chance causes is inherent in the nature of any process or procedure, there is usually an identifiable pattern of variation. When this pattern is stable, the process or procedure is said to be in "statistical control." Variation outside of this pattern will have an assignable cause and, in many cases, the assignable cause can be determined and corrected. Thus, on an intralaboratory level, statistical analyses assist the laboratory analyst in routine surveillance (control charting) and in identifying sources and types of analytic error when the process or procedure is out of "statistical control."

(2) A widely used graphic display technique for interlaboratory test results was devised by Dr. W. J. Youden.[9] This graphic

Table 1

SUMMARY OF MATRIX "A" RESPONSES BY LABORATORY

Number of Procedures

	1	2	3	4	5	6	7	8	9	10	11
FSHT	2	3	5	1	6	7	10	8	9	4	11
Ft Meade	1	5	7	4	10	5	11	9	3	4	7
Ft McPherson	5.5	4.5	7	1	7.5	7.5	7.5	7.5	3	4	11
St Louis	4.3	1	5.7	4.3	7.7	7.7	4.3	7	4.7	5.3	7.7
Ft Baker	7	3	4	3	8	10	5	6	1	2	9
	19.8	16.5	28.7	13.3	39.2	37.2	37.8	37.5	20.77	19.3	45.7

display technique allows participants to compare their values with those from the Central Quality Assurance (CQA) laboratory and also gives some indication of what is wrong with the procedure if the results are not acceptable. A statistical background is not required to understand the graph.

(a) Each laboratory analyzes two samples for the same constituent.

(b) The CQA also analyzes a series of these samples.

(c) The two median lines X and Y (See Figure 2) drawn from the ordinate and abscissa represent the means of reference (CQA) laboratory values for the two samples.

(d) The standard deviation of CQA laboratory values leads to construction of the circle.

(e) A line (W) drawn at 45 degrees through the intersection of the median lines divides lower left and upper right quadrants. Two additional lines (s and t) are drawn along the circumference of the circle parallel to the 45 degree line.

(f) The pairs of values from participants for the two samples are used to plot additional points on the graph.

(3) Interpretation of results:

(a) Plotted points within the circle are within $\pm$ 3 standard deviations of the CQA lab.

(b) Plotted points outside the circle, but between the parallel lines, are from laboratories which reported high values or low values for both samples. Consistently high or low values suggest a systematic error.

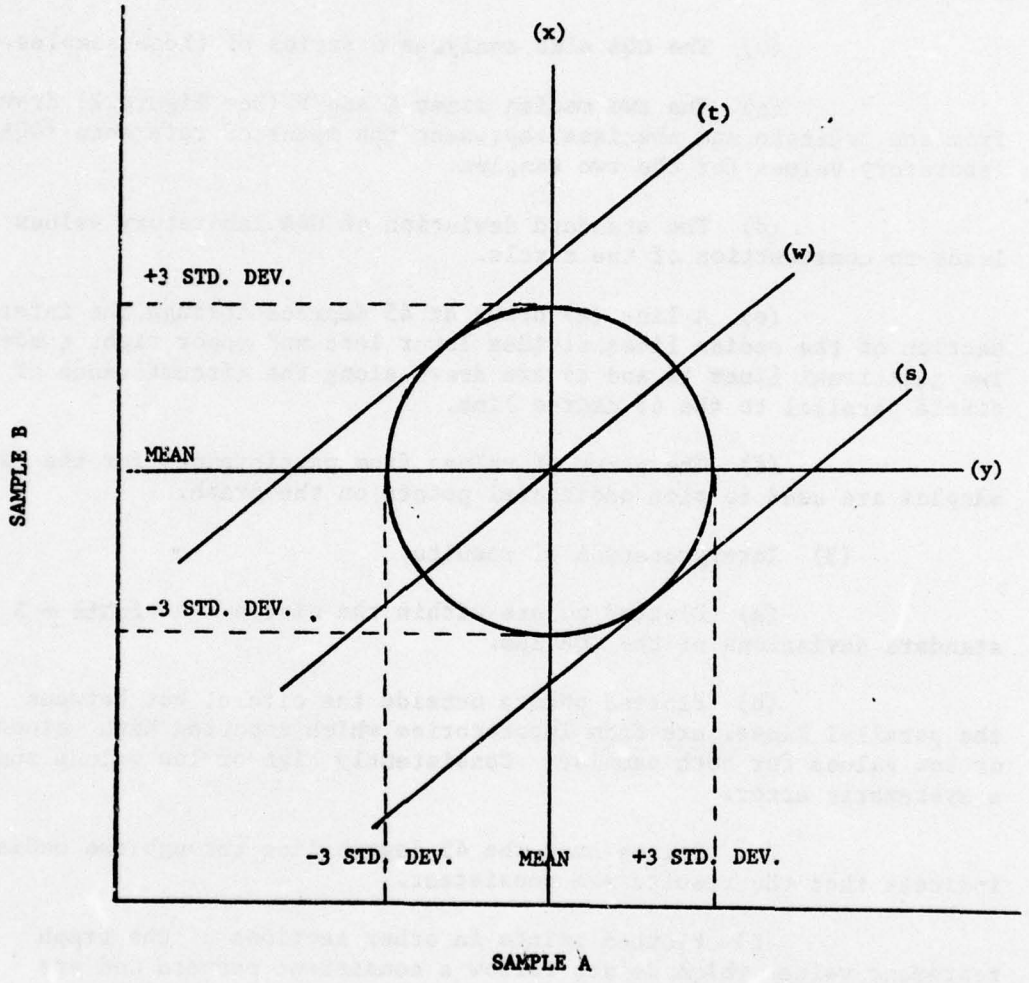
(c) Points near the 45 degree line through the median indicate that the results are consistent.

(d) Plotted points in other sections of the graph represent values which do not follow a consistent pattern and are attributable to random error.

(4) Another advantage to this technique is that it requires only basic statistics such as the mean and standard deviation.

e. Evaluation of the current state-of-the-art. The preceding sections of this report have dealt at some length with the topics of

FIGURE 2


(x)
(t)
(w)
(s)
(y)
+3 STD. DEV.
MEAN
-3 STD. DEV.
SAMPLE B
-3 STD. DEV.
MEAN
+3 STD. DEV.
SAMPLE A

control sampling and statistical analysis of results. In order to put these topics in their proper perspective it seems important at this point to emphasize that statistical analyses only reflect the existing levels of quality control. Quality control is, and must remain, a primary function of the individual laboratory analyst. Strict adherence to current published methodologies is essential to the consistent production of acceptable laboratory results and, therefore, must continue to be the most important area for evaluation.

f. Evaluation of the control of samples within the laboratory. It is axiomatic that results obtained by laboratory analysis are no better than the sample presented for analysis. Thus, the proper identification, storage, intralaboratory routing, and preparation for analysis of both routine and quality assurance samples is essential to the success of an intralaboratory quality assurance program. Each laboratory is conducting an effective sample control program. It should be observed, however, that the Veterinary Divisions of the various laboratories do not have complete autonomy in these matters and, consequently, the procedures employed for accomplishing sample control vary from laboratory to laboratory.

6. CONCLUSIONS.

a. Evaluation of equipment. Intralaboratory programs for accomplishing this requirement are complete and, therefore, should be incorporated into the centralized HSC program as an essentially independent set of activities which receive interlaboratory attention for comparability purposes only.

b. Evaluation of precision. A large amount of potentially useful intralaboratory precision data is available for analysis and review. A CQA laboratory would be in the best position to conduct an ongoing evaluation of this data and to correlate it with associated interlaboratory data.

c. Evaluation of accuracy. Existing intralaboratory programs should be supplemented by an interlaboratory sampling program. In the absence of reliable interlaboratory data, the inclusion vector shown in paragraph 4a(5)(d) above, should serve as the initial basis for this program.

d. Evaluation of statistical analyses. Statistical techniques are currently not universally applied throughout the system and, therefore, interlaboratory comparisons on historical data is difficult. While the application of statistical techniques and control charting is not universal, there is a sufficiently large number of analyses which are well suited to these techniques. Thus, agreement on standard

practices is feasible and would aid the CQA activity in performing the interlaboratory comparability determinations.

e. Evaluation of the present state-of-the-art. The system currently relies heavily upon the Veterinary Division of WRAIR for accomplishing this requirement.

f. Evaluation of sample control. Because of the individual variations between laboratory policies and the lack of uniformity is laboratory configurations it follows that there is probably not one "best" method for accomplishing sample control which is applicable to all laboratories.

7. RECOMMENDATIONS.

a. Evaluation of equipment. Recommend that a central quality assurance (CQA) activity conduct an annual on-site survey of the intralaboratory equipment evaluation programs. This on-site survey should include, but not be limited to, the specific items listed in paragraph 2b(2)(a) of this report. A complete and detailed evaluation of each item in this category is not practical and, therefore, it is recommended that specific items within the category be selected at random for detailed spot-check evaluation during the survey. Results of these surveys should be made available promptly and should serve as guidelines for interlaboratory comparability determinations in this area.

b. Evaluation of precision. It is recommended that each laboratory conduct both duplicate analyses and recycling programs which are formally evaluated and recorded for review. While many systems are available for displaying these results, it is recommended that the existing control chart techniques be continued. Results of these programs should be evaluated by a CQA team during on-site visits.

c. Evaluation of accuracy.

(1) Recommend that the CQA activity initiate a formal interlaboratory sampling program based on the inclusion vector shown in paragraph 4a(5)d and that the vector be recomputed when current interlaboratory data becomes available.

(2) Because most microbiological analyses are not routinely included in the interlaboratory sampling program, it is recommended that these procedures receive particularly close evaluation during on-site surveys by the CQA representatives. Initially, the CQA activity at Fort Sam Houston should serve as the sole source for interlaboratory samples. However, as the interlaboratory program progresses, it is recommended that a rotating source concept be employed in which each participating laboratory alternates responsibility for preparation and submission of

interlaboratory samples. This program would be coordinated and directed by the CQA activity at FSH. Data analysis and reporting of survey results should be accomplished by the CQA Lab at FSH.

d. Evaluation of statistical analyses. Recommend that existing control chart techniques for the analysis and display of intralaboratory data be continued and expanded to include all laboratories in the system. Recommend that interlaboratory data be displayed on a graphic format such as that discussed in paragraph 5d(2) of this report.

e. Evaluation of the current state-of-the-art. Recommend that a central quality assurance activity assume prime responsibility for conducting a current literature review (both military and civilian) program and for providing current state-of-the-art information to each of the participating area labs. The availability and application of this material at the intralaboratory level should then be evaluated by the CQA team during on-site visits.

f. Evaluation of sample control. Recommend that a CQA team review and evaluate the various intralaboratory procedures for identification, storage, routing and pre-analysis preparation of both routine and quality assurance samples during on-site surveys.

g. Finally, although no objective estimates of personnel requirements for implementation and operation of the CQA program are available, it is recommended that the program be supervised by a senior veterinary laboratory officer. In addition, the expertise of a senior laboratory analyst is considered essential to the program. Further, the part-time assistance of one or two laboratory technicians will be required (especially during the phase of the program in which the CQA acts as sole source for interlaboratory quality assurance samples). Therefore, it is recommended that this program be implemented in phases during which the specific personnel and other laboratory resource requirements may be objectively evaluated. Specifically, it is recommended that the interlaboratory sampling portion of the CQA program be initiated within the existing capabilities of the FSH area laboratory and that, concurrently, work should begin on the development of SOPs and reporting formats for accomplishing the complete quality assurance program outlined above.

REFERENCES

1. Army Medical Department Annual Chart Book, Department of the Army, Office of the Surgeon General, Directorate of Resources Management, FY 1974, page 81.

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4. Brown, B., et al., The Delphi Method, Rand Corporation, Santa Monica, California, 1969.

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8. Kramer, A. and Twigg, B. A., Fundamentals of Quality Control for the Food Industry, AVI Publishing, Westport, Connecticut, 1966.

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APPENDIX

A

DEPARTMENT OF THE ARMY
ACADEMY OF HEALTH SCIENCES, UNITED STATES ARMY
FORT SAM HOUSTON, TEXAS 78234

AHS-DCS

SUBJECT: AHS Veterinary Laboratory Quality Assurance Study

Commander, Area Medical Laboratory, Ft Meade, MD 20755
Commander, Area Medical Laboratory, Ft McPherson, GA 30330
Commander, Area Medical Laboratory, Ft Sam Houston, TX 78234
Commander, Area Medical Laboratory, 12th & Spruce St., St Louis MO 63139
Commander, Area Medical Laboratory, Ft Baker, CA 94965

1. The Health Services Command has requested that the Health Care Studies Division of the Academy of Health Sciences conduct a study of HSC area medical laboratory veterinary food testing quality assurance programs. Both intra- and interlaboratory quality assurance procedures will be examined during the course of this study.

2. Much of the data considered necessary for the conduct of this study can and is being extracted from existing records and reports. Certain additional data for this study can best be provided by supervisory personnel of the area medical laboratory veterinary services. Therefore, it is requested that the information referenced in subparagraphs a through d, below, be compiled and forwarded to the Health Care Studies Division, Academy of Health Sciences, US Army, ATTN: MAJ Armstrong, Fort Sam Houston, TX 78234, not later than 29 April 1974.

a. Briefly outline your current intralaboratory quality assurance procedures for accomplishing the following:

(1) Evaluation of equipment.

(2) Evaluation of the precision of analytical results.

(3) Evaluation of the accuracy of analytical results.

(4) Statistical analysis of quality assurance data.

(5) Interpretation of results obtained by statistical analysis of quality assurance data.

AHS-DCS
SUBJECT: AHS Veterinary Laboratory Quality Assurance Study

b. Workload data for the veterinary department during the four quarters ending December 1973 (copies of DA Form 3222 will suffice).

c. Attached as Inclosure 1 is a list of analytic procedures. You are requested to indicate which of the listed procedures your laboratory can and cannot currently perform. For each of the listed procedures which your laboratory cannot currently perform, you are requested to check, by category, additional resources required by your laboratory for performance of the procedure.

d. Attached as Inclosure 2 is a matrix listing commonly performed analytic procedures. The veterinary department is requested to complete the matrix on the basis of observations made in your laboratory.

3. In the event that questions arise regarding information requested above, please contact MAJ Tommy S. Armstrong, AUTOVON 471-3331 or 3116.

FOR THE SUPERINTENDENT:

2 Incl
as

APPENDIX

B

INSTRUCTIONS--MATRIX A

Even when all determinate error due to identifiable sources such as method error, analyst error, and instrumental error have been isolated and removed, replicate analyses using a given method will not consistently give the same values. Such variation is due to indeterminate error, also known as random, chance, or uncontrollable error. This indeterminate error is the criterion variable by which you are asked to compare each of the procedures listed on the left side of Matrix A with each of the procedures listed at the top of Matrix A.

If, on the basis of your intralaboratory quality assurance results, there is more indeterminate error associated with the procedure on the left than with the procedure at the top, enter a <u>one</u> in the corresponding matrix square.

Conversely, if there is more indeterminate error associated with the procedure at the top than with the procedure on the left, enter a <u>zero</u> in the corresponding matrix square.

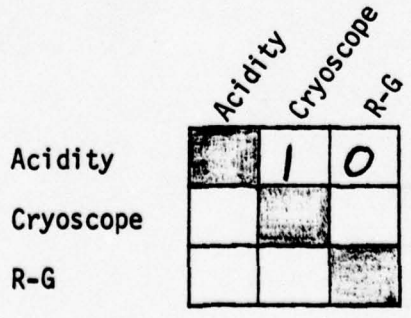
If your existing intralaboratory quality assurance data is inconclusive in this matter, it is requested that the evaluations of indeterminate error be made on a subjective basis.

<u>Example</u>:

Comparing Acidity (on left) with each procedure on top:

(a) More indeterminate error associated with Acidity than Cryoscope--Enter <u>1</u>.

(b) Less indeterminate error associated with Acidity than R-G--Enter <u>0</u>.



Incl 2

MATRIX A

	Acidity	Cryoscope	R-G(Mojonnier)	pH	Protein(Dairy)	Protein(Meat)	SNF	Soxhlet	Moisture(Dairy)	Moisture(Meat)	Salt
Acidity											
Cryoscope											
R-G(Mojonnier)											
pH											
Protein(Dairy)											
Protein(Meat)											
SNF											
Soxhlet											
Moisture(Dairy)											
Moisture(Meat)											
Salt											

NAME _____

GRADE _____

POSITION _____

LABORATORY _____

APPENDIX

C

APPENDIX C

TABLE 1. SUMMARY OF MATRIX A RESPONSES

Procedures	RESPONSES							
	1	2	3	4	5	6	7	8
1	0	1	0	4	6	7	2	6
2	4	2	0	0	0	2	5	2
3	6	4	5	7	2	5	7	3
4	3	0	8	1	1	0	0	2
5	9	5	6	8	6	7	6	7
6	4	6	5	8	7	8	5	9
7	10	9	0	5	5	4	9	4
8	8	7	2	6	10	7	6	5
9	2	8	1	3	7	2	2	0
10	3	3	1	4	8	3	3	1
11	6	10	8	9	3	10	10	8

APPENDIX C

TABLE 2. RESPONSE DATA EXPRESSED AS FREQUENCIES PER CATEGORY OF RESPONSE (Table is now nxm.)

Procedures	RESPONSE CATEGORIES										
	0	1	2	3	4	5	6	7	8	9	10
1	2	1	1	0	1	0	2	1	0	0	0
2	3	0	3	0	1	1	0	0	0	0	0
3	0	0	1	1	1	2	1	2	0	0	0
4	3	2	1	1	0	0	0	0	1	0	0
5	0	0	0	0	0	1	3	2	1	1	0
6	0	0	0	0	1	2	1	1	2	1	0
7	1	0	0	0	2	2	0	0	0	2	1
8	0	0	1	0	0	1	2	2	1	0	1
9	1	1	3	1	0	0	0	1	1	0	0
10	0	2	0	4	1	0	0	0	1	0	0
11	0	0	0	1	0	0	1	0	2	1	3

APPENDIX C

TABLE 3. RELATIVE CUMMULATIVE FREQUENCY FOR EACH ROW WITH COLUMN NUMBER 10 OMITTED WHICH WILL BE THE UNIT COLUMN VECTOR (This is an nx(m-1) table.)

Procedures	0	1	2	3	4	5	6	7	8	9
1	2/8	3/8	4/8	4/8	5/8	5/8	7/8	1	1	1
2	3/8	3/8	6/8	6/8	7/8	1	1	1	1	1
3.	0	0	1/8	2/8	3/8	5/8	6/8	1	1	1
4	3/8	5/8	6/8	7/8	7/8	7/8	7/8	7/8	1	1
5	0	0	0	0	0	1/8	4/8	6/8	7/8	1
6	0	0	0	0	1/8	3/8	4/8	5/8	7/8	1
7	1/8	1/8	1/8	1/8	3/8	5/8	5/8	5/8	5/8	7/8
8	0	0	1/8	1/8	1/8	2/8	4/8	6/8	7/8	7/8
9	1/8	2/8	5/8	6/8	6/8	6/8	6/8	7/8	1	1
10	0	2/8	2/8	6/8	7/8	7/8	7/8	7/8	1	1
11	0	0	0	1/8	1/8	1/8	2/8	2/8	4/8	5/8

APPENDIX C

TABLE 4. CONVERSION OF TABLE 3 INTO DECIMAL EQUIVALENTS

Procedure	DECIMAL CUMULATIVE FREQUENCIES									
	0	1	2	3	4	5	6	7	8	9
1	.250	.375	.500	.500	.625	.625	.875	1.000	1.000	1.000
2	.375	.375	.750	.750	.875	1.000	1.000	1.000	1.000	1.000
3	0	0	.125	.250	.375	.625	.750	1.000	1.000	1.000
4	.375	.625	.750	.875	.875	.875	.875	.875	1.000	1.000
5	0	0	0	0	0	.125	.500	.750	.875	1.000
6	0	0	0	0	.125	.375	.500	.625	.875	1.000
7	.125	.125	.125	.125	.375	.625	.625	.625	.625	.875
8	0	0	.125	.125	.125	.250	.500	.750	.875	.875
9	.125	.250	.625	.750	.750	.750	.750	.875	1.0000	1.000
10	0	.250	.250	.750	.875	.875	.875	.875	1.000	1.000
11	0	0	0	.125	.125	.125	.250	.250	.500	.625

APPENDIX C

TABLE 5. STANDARD NORMAL DEVIATES FOR THE VALUES SHOWN IN TABLE 4

Procedures	STANDARD NORMAL DEVIATES										Row Sum	Row Mean
	0	1	2	3	4	5	6	7	8	9	Σ	$\bar{Y}$
1	-.675	-.319	.0	.0	.319	.319	1.151	3.49	3.49	3.49	11.265	1.127
2	-.319	-.319	.675	.675	1.151	3.49	3.49	3.49	3.49	3.49	19.313	1.931
3	0	0	-1.151	-.675	-.319	.319	.675	3.49	3.49	3.49	9.319	.932
4	-.319	.319	.675	1.151	1.151	1.151	1.151	1.151	3.49	3.49	13.41	1.341
5	0	0	0	0	0	-1.151	0	.675	1.151	3.49	4.165	.417
6	0	0	0	0	-1.151	-.319	0	.319	1.151	3.49	3.49	.349
7	-1.151	-1.151	-1.151	-1.151	-.319	.319	.319	.319	.319	1.151	-2.496	-.250
8	0	0	-1.151	-1.151	-1.151	-.675	0	.675	1.151	1.151	-1.151	-.115
9	-1.151	-.675	.319	.675	.675	.675	.675	1.151	3.49	3.49	9.324	.932
10	0	-.675	-.675	.675	1.151	1.151	1.151	1.151	3.49	3.49	10.909	1.091
11	0	0	0	-1.151	-1.151	-1.151	-.675	-.675	0	.319	-4.484	-.448
Column Sum Σ	-3.615	-2.820	-2.459	-.952	.356	4.128	7.937	15.236	24.712	30.541	73.064	
Column Mean $\bar{X}$	-.329	-.256	-.224	-.087	.032	.375	.722	1.385	2.247	2.776	.664 $\bar{\bar{X}}$ Column Mean of Mean	.664 $\bar{\bar{Y}}$ Row Mean of Mean

TABLE 5. (Contd)

These are the X_{jg} values to be used in the following computations:

(1) For each row of Table 5

$$\bar{X}_j = \frac{\sum_{g=1}^{m-1} X_{jg}}{m-1}$$

1. 1.127
2. 1.931
3. 0.932
4. 1.341
5. 0.417
6. 0.349
7. -0.250
8. -0.115
9. 0.932
10. 1.091
11. -0.448

TABLE 5. (Contd)

(2) For each column

$$tg = \frac{\sum_{j=1}^{n} X_{jg}}{n}$$

0. -0.329

1. -0.256

2. -0.224

3. -0.087

4. 0.032

5. 0.375

6. 0.722

7. 1.385

8. 2.247

9. 2.776

TABLE 5. (Contd)

(3) Compute grand mean $\bar{t}$

0.664

(4) Compute

$$A = \sum_{g=1}^{m-i} (tg-\bar{t})^2 = 11.158$$

(5) For each row

$$B_j = \sum_{g=1}^{m-i} (X_{jg}-\bar{X}_j)^2$$

$B_1 = 25.937$

$B_2 = 26.040$

$B_3 = 30.295$

$B_4 = 13.659$

$B_5 = 13.552$

$B_6 = 13.816$

$B_7 = 6.512$

$B_8 = 7.402$

$B_9 = 20.395$

$B_{10} = 19.127$

$B_{11} = 2.976$

TABLE 5. (Contd)

(6) Scale values are:

$$S_j = \bar{t} - \bar{X}_j \sqrt{\frac{A}{B_j}}$$

(Acidity)	S_1	-0.074
(Cryoscope)	S_2	-0.598
(R-G)	S_3	0.100
(pH)	S_4	-0.546
(Protein--Dairy)	S_5	0.286
(Protein--Meat)	S_6	0.351
(SNF)	S_7	0.991
(Soxhlet)	S_8	0.805
(Moisture--Dairy)	S_9	-0.020
(Moisture--Meat)	S_{10}	-0.168
(Salt)	S_{11}	1.531

TABLE 5. (Contd)

(7) Perform linear transformation

$$S_j^1 = 1 + 0.1\, S_j \text{ for } j=1,\cdots,9$$

		Transformed Interval Scale
(Acidity)	S_1'	.9926
(Cryoscope)	S_2'	.9402
(R-G)	S_3'	1.0100
(pH)	S_4'	.9454
(Protein--Dairy)	S_5'	1.0286
(Protein--Meat)	S_6'	1.0351
(SNF)	S_7'	1.0991
(Soxhlet)	S_8'	1.0805
(Moisture--Dairy)	S_9'	.9980
(Moisture--Meat)	S_{10}'	.9832
(Salt)	S_{11}'	1.1531

		Ordered Transformed Interval Scale
(Cryoscope)	S'_2	0.9402
(pH)	S'_4	0.9454
(Moisture--Meat)	S'_{10}	0.9832
(Acidity)	S'_1	0.9926
(Moisture--Dairy)	S'_9	0.9980
(R-G)	S'_3	1.0100
(Protein--Dairy)	S'_5	1.0286
(Protein--Meat)	S'_6	1.0351
(Soxlet)	S'_8	1.0805
(SNF)	S'_7	1.0991
(Salt)	S'_{11}	1.1531

APPENDIX

D

ANALYTICAL PROCEDURES	Is the Vet. Dept. Currently Capable of Performing This Procedure?		Category of Additional Resources Required		
	YES	NO	Personnel	Equipment	Lab Space
Absorbence		3	3		3
Absorbency and absorbency ratio		3	3		3
Absorbtivity		3	3		3
Acidity					
Added water					
Adulteration of color		3,5	3	3,5	3
Alkalinity of ash					
Alpha-monoglyceride		2,3,4	3	3	3
Ammonium chloride		3,5	3	3,5	3
Anaerobic Spores					
Anti-caking agent particle test		3,4,5	3	3,4,5	3
Arsenic, atomic absorption		2,3,4,5	2,3	2,3,4,5	2,3
Ascorbic acid		2,3,4	2,3	4	2,3
Ash					
Ash, insoluble					
Ashed sediment		3	3	3	3
Atomic absorptivity		2,3,4,5	2,3	2,3,4,5	2,3
Available carbon dioxide		3	3	3	3

Key: 1 - Ft Meade
2 - Ft McPherson
3 - Ft Sam Houston
4 - St. Louis
5 - Ft Baker

	YES	NO	Personnel	Equipment	Lab Space
Benzoic acid					
Bloom gelometer		1,2,3, 4,5	3	1,2,3, 4,5	3,5
Bostwick consistometer		1,2,3, 4,5	3	1,2,3 4,5	3,5
Brix					
Brix-acid ratio		3	3	3	3
Cadmium, atomic absorption		2,3,4,5	2,3	2,3,4,5	2,3
Caffeine		2,3,4	2,3,4	3	2,3
Calcium		4	4		
Calcium stearate		2,3,4	2,3,4	3	3
Calcium sulfate		2,3,4	2,3,4	3	3
Carbohydrates		2,3	2,3	2,3	2,3
Carbon dioxide (Orsat)		3	3	3	3
Corotenoid color		3,4	3,4	3	3
Centers content		3,4,5	3,4	3,5	3
Chlorides (as sodium chloride)					
Chlorides in alcohol--soluble matter		2,3,4	3,4	2,3	3
Chocolate solids content		2,3,4	2,3,4	3	2,3
Cider vinegar (qualitative)		3,5	3	3,5	3
Citrates		2,3,5	2	5	
Citric acid		2,3	2		
Citrus oil		2,3,4		2,4	
Clump count		3	3	3	3
Coating content		2,3	3	2,3	3
Cocoa content		2,3,4	2,3,4	3	3

	YES	NO	Personnel	Equipment	Lab Space
Coconut content		3,4	3,4	3	3
Cold test		3	3	3	3
Cold water extract		2,3,4	3,4	3	3
Coliform (presumptive) duplicate plates					
Color comparison		3	3	3	3
Color reflectance		1,3,5	3	1,3,5	3
Copper		3,4	4		
Cottonseed oil (qualitative)		2,3,4	2,3	2,3,4	2,3
Crude fiber		3	3	3	3
Cube test		3,4	3,4	3	3
Densitometry		2,3	3	2,3	3
Density		2,4	4	2,4	
Dextrose (copper reduction)		2,3,4	2,3,4	3	3
Dextrose (paper chromatography)		2,3,4	3,4	2,3	2,3
Diastatic activity		3	3	3	3
Direct microscopic count					
Disintegration		3,4,5	3,4,5	3,5	3,5
Dispersibility		3,4,5	3,4	3,5	3
E. Coli					
Emulsion stability		3,4	3,4	3	3
Ethyl vanillin		3,4	3,4	3	3
Extractable color		3,4,5	4,5	5	
Extraneous matter		3	3	3	3

	YES	NO	Personnel	Equipment	Lab Space
Fat (acid hydrolysis)					
Fat (Babcock)		3,5		3,5	3
Fat (Roese-Gottlieb)					
Fat (Soxhlet)					
Fat acidity		3	3	3	3
Fat stability		3,4	3	3,4	3
Filter test		3,4,5	3	3,4,5	3
Flame photometry		2,3,4,5	3	2,3,4,5	2,3
Foam test		1,3,4,5	3,4	1,3,5	3
Free and suspended pulp		3,4,5	3,4	3,5	3
Free fatty acids					
Gel strength		1,2,3, 4,5	3,5	1,2,3, 4,5	3
Gelatin liquefiers		3,4,5	3,4	3,5	3
Glucose (copper reduction)		2,3,4	3,4	2	2,3
Glucose (paper chromatography)		2,3,4	3,4	2,3	2,3
Granular size		3,5	3	3,5	3
Headspace					
Heating test		3,5	3	3,5	3
Heavy metals (qualitative)		3,4	3,4	3,4	3
Homogenization efficiency		4,5	5	4,5	
Inactivation of catalase		3,4	3	3	3
Iodine value		3	3		3
Insoluble solids		3,4	3,4	3	3
Invert sugar (copper reduction)		2,3,4	2,3,4	2,3	2,3
Iron		2,3,4	3,4	2,4	2,3

	YES	NO	Personnel	Equipment	Lab Space
Lactose		2,3,4	2,3,4	2,3	2,3
Lead, atomic absorption		2,3,4,5	3,4	2,3,4,5	2,3
Leakage of fat		3,4,5	3,4	3,5	3
Linolenic acid		2,3,4,5	2,3,4	2,5	3
Lipoid phospherus		2,3	2,3		3
Liquefiers		3,4,5	3,4	3,5	3
Maggott determination		2,3,4	4		
Magnesium		2,3	3	2	2,3
Magnesium, atomic absorption		2,3,4,5	3	2,3,4,5	2,3
Maltose		2,3,4	3,4		3
Malt vinegar (qualitative)		3,4,5	3,4	5	3
Manganese, atomic absorption		2,3,4,5	3,4	2,3,4,5	2,3
Melting point		3	3	3	3
Methylene blue test		3	3	3	3
Milkfat (Babcock)		3,5		3,5	3
Milkfat (Roese-Gottleib)					
Milk protein (Kjeldahl)					
Milk solids					
Milk solids non-fat					
MIL-STD-900 (thermophiles, flat sours, etc.)		4	4		
Moisture (air oven or toluene distillation)					
Mold count (Howard)		3		3	
Monoglyceride		3,4	3,4	3,4	3
Monosodium glutamate		3,4	3,4	3	3

	YES	NO	Personnel	Equipment	Lab Space
Niacin		3,4,5	3,4,5	3	3
Nitrogen					
Non-fat dry milk					
Non-fat salt-free solids					
Non-volatile ether extract (Soxhlet)					
Non-volatile methylene chloride extract of pepper (Soxhlet)		3,4	3,4		3
Non-volatile solids		2	2		2
Optical density		2,3	3	2	3
Optical rotation		2,3,5	3	2,3,5	2,3
Over-run		3	3	3	3
Oxygen (Orsat)		3	3	3	3
Particle size (screening)					
Pasteurization efficiency (Phosphatase)					
Peroxidase activity		3,4	3,4	3	3
Peroxide value					
pH value					
Phosphatase activity					
Phosphorus					
Phosphorus pentoxide		2,3	3	2,3	3
Piperine		2,3,4,5	2,3,4	3,5	2,3
Potassium iodide		3,5	3	3,5	3
Potassium oxide (flame photometry)		2,3,4,5	2,3,4	2,3,4,5	2,3
Pouring test		1,3,5	3	1,3,5	3

	YES	NO	Personnel	Equipment	Lab Space
Propyl gallate		2,3,4	2,3,4	2,3	3
Propylene glycol		2,3,4	2,3,4	2,3	3
Protein (Kjeldahl)					
Protein reducing substances					
Psychrophilic count					
Pungency		3,5	3	3,5	3
Purity (monosodium glutamate)		2,3,4,5	3,4	2,3,5	3
Recoverable oil		2,3,4,5	3,4	3,5	3
Reducing sugars as dextrose		2,3,4	2,3,4		2,3
Refractive index		2,3	3	2	3
Resazurin test		3,4	3,4	3	3
Residual carbon dioxide		3,4	3	3	3
Residual phosphatase					
Residual plate count on containers					
Riboflavin		1,2,3, 4,5	2,3,4	1,5	2,3
Rope spore count		3,4	3,4		3
Salmonella					
Salt (total chlorides as sodium chloride)					
Scorched particles		3	3	3	3
Screening (sieve test)					
Screening test for entrapped orange granules		3,4,5	3,4	3,4,5	3
Sediment					
Sifting (sieve test)					
Sirup density		3,4,5	3,4	4,5	3

	YES	NO	Personnel	Equipment	Lab Space
Smoke point		3	3	3	3
Sodium (flame photometry)		2,3,4,5	3,4	2,3,4,5	3
Sodium benzoate		2,3	2,3	2,3	2,3
Sodium bicarbonate		3	3		3
Sodium bisulfite content		3,4,5	3,4	5	3
Solids, salt-free					
Solids, total					
Solid fat index values (per temperature)		1,2,3, 4,5	2,3,4	1,2,3, 4,5	2,3,5
Solubility		3	3		3
Solubility, hydrolyzed protein		3,4	3,4		3
Solubility index		3,5	3	5	3
Solubility of entrapped orange oil granules		1,2,3, 4,5	2,3,4	1,2,3,5	2,3
Solubility performance test		3,4,5	3,4	5	3
Soluble solids		3	3		3
Soy oil (Epstein-Harris)		2,3,4,5	2,3,4	2,3,4,5	3
Soy oil (AOCS)		2,3,4,5	2,3,4	2,3,4,5	3
Specific gravity					
Spore count					
Stability, AOM		3,4	3	3,4	3
Stability of emulsion		3,4	3,4	3	3
Standard plate count					
Staphylococci MPN					
Starch (copper reduction)		2,3,4	2,3,4		3

	YES	NO	Personnel	Equipment	Lab Space
Sucrose (copper reduction)		2,3,4	2,3,4	2	2,3
Sucrose (polarization)		2,3	3	2	3
Sulfate		3	3	3	3
Sulfated ash		3	3	3	3
Sulfide (qualitative)		3	3		3
Sulfite		3	3	3	3
Sulfur dioxide		3,4	3,4	3	3
Sulfurous acid		2,3,4	3	3,4	3
Sweat test		1,3,4,5	3,4	1,3,5	3
Tenderometer reading		1,2,3, 4,5	3,4	1,2,3, 4,5	3
Test weight per 32 fluid ounces		3	3	3	3
Thiamine		1,2,3, 4,5	2,3,4	1,2,5	2,3
Thiobarbituric acid		2,4	4	2	
Tin, atomic absorption		2,3,4,5	3,4	2,3,4,5	3
Titer test (solidifying point of fatty acids)		2,3	3	2	3
Titratable iodine					
Total solids					
Total sugars (copper reduction)		2,3,4	2,3,4		2,3
Total sulfites		2,3	2,3	2,3	2,3
Transmittance		3	3		3
Vacuum					
Vanillin or ethyl vanillin		1,3,4	3,4	1,3	3
Viscosity (Stormer)		2,3,4,5	3,4	2,5	3
Vitamin A		2,3,4	3,4	2	3

	YES	NO	Personnel	Equipment	Lab Space
Vitamin B1 (thiamin)		1,2,3,4	2,3,4	1,2	2,3
Vitamin C (ascorbic acid)		2,3,4	2,3,4		2,3
Volatile acids		2,3	2,3	2,3	3
Volatile and non-volatile ether extract		3	3		3
Volatile esters		2,3,4,5	2,3,4	2,5	2,3
Volatile oil		2,3,4	2,3,4	2	2,3
Water-insoluble solids (jams)		3,4	3,4		3
Whey protein nitrogen		3,4	3,4		3
Whipping test		3			3
Wrapper adherance		1,2,3,4	3,4	1,2,3	3
Yeast and mold counts					
Zinc, atomic absorption		2,3,4,5	3	2,3,4,5	3

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