QUALITY CONTROL MANUAL OF THE U.S. GEOLOGICAL SURVEY'S NATIONAL WATER QUALITY LABORATORY

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INTERIM QUALITY CONTROL MANUAL

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U.S GEOLOGICAL SURVEY'S NATIONAL WATER QUALITY LABORATORY

By Berwyn E. Jones

ABSTRACT

Quality-control practices have been established for the operation of the U.S. Geological Survey's National Water Quality Laboratory. These practices specify the ways in which samples are preserved, shipped, and analyzed in the laboratory. This manual documents the practices that are currently (1987) used in this laboratory. It is termed an "interim" manual because it is intended to serve as the basis for the development of a more complete and detailed quality assurance plan for the laboratory, to be made possible by the expansion of staff, operations, and data-processing capability, which is currently underway.

INTRODUCTION

This manual describes the current (1987) quality control practices and standards of the U.S. Geological Survey's National Water Quality Laboratory (NWQL). It is designated an interim manual because the organization, staffing, and data management system of the NWQL are all in a state of rapid change and development. The intent of the manual is to document the current state of quality control in the NWQL in order to serve as a guide to current practices as well as a basis for the development of a more definitive quality control program in the near future.

The purpose of a laboratory quality control manual is to document the standard operating procedures that are used to ensure that the data the laboratory produces are of high, uniform and well-defined quality. Although the quality control program of the NWQL is operated by a quality control group, which reports directly to the chief of the NWQL, a successful quality control program requires active and enthusiastic participation by all analysts and supervisors in the NWQL. Review and technical oversight of this program will be a responsibility of the Branch of Quality Assurance (currently being organized).

In all cases, the quality assurance manual of Friedman and Erdmann (1982) is governing where it contains applicable standards.

QUALITY CONTROL PRACTICES

1. SAMPLING

1.1 Sample Collection

Sample collection is performed by personnel of the U.S. Geological Survey engaged in water-resources investigations throughout the country. Quality assurance for sampling operations is the responsibility of the Office of Water Quality at the U.S. Geological Survey's headquarters in Reston, Virginia.

1.2 Containers

Sample containers are furnished by the NWQL to all U.S. Geological Survey field offices, on request. The type of container to be used for each determination is specified in the water quality laboratory services catalog (hereinafter, referred to as the Catalog), issued annually as a U.S. Geological Survey Open-File Report (Feltz and Anthony, 1984). The NWQL does not normally accept samples in nonstandard containers. The cleaning requirements for containers are described in the Catalog; exact laboratory protocols for cleaning containers are set forth in a bench-level protocol of the logistical-support section of the NWQL. All bench-level protocols are on file in the quality control office of the NWQL, and are also located in the office of the appropriate section chief and in the laboratory where the process is actually performed. Quality control checks are performed on all lots of containers to assure freedom from contamination. These checks are performed according to methods described by Friedman and others (1980), and Grant and Leavenworth (1972).

1.3 Preservatives

Sample-preservation chemicals also are supplied by the NWQL. The formulations of preservatives are specified in periodic Office of Water Quality technical memoranda, and are described in the catalog in most instances. Preservatives are prepared in numbered lots either in the NWQL or by contractor laboratories. Exact specifications are contained in bench-level protocols by the logistical-support section of the NWQL, and are available from the quality control office of the NWQL. Each lot of preservative is sampled for testing according to protocols outlined in the chapter, "Materials Evaluation," of the report by Friedman and Erdmann (1982). Test data files are maintained in the logistical-support section.

1.4 Field Blanks and Spikes

Protocols for field blanks and field-spiking of organic samples with surrogate compounds are determined by each field project's quality assurance plan. Field spiking with surrogate compounds requires special arrangement with the NWQL to avoid double spiking.

1.5 Sample Documentation and Shipping

Analytical requests to the NWQL are submitted on a standardized central laboratories analytical services request form (U.S. Govt. Printing Office 679-483 1983; see figure 1). This form uniquely identifies the sample, the office requesting the analysis, the determinations requested, and any special sample information relating to safety hazards or sample-matrix problems.

Certain samples are required to be shipped in ice to maintain a temperature of 4 degrees Celsius. These requirements are listed in the Catalog.

2. SAMPLE HANDLING

2.1 Logging Samples

Samples and the accompanying request forms are received by the log-in group. Each sample and its request form is assigned an unique NWQL identification number that encodes the date of receipt, the sample type, and a serial number. The various containers received for each sample are noted, and each container is routed to the laboratory section that performs the appropriate determinations. Irregularities detected by the log-in group in sample shipping and preservation are noted on the request form. The request form is routed to the automated data-processing (ADP) section, which enters all sample information and analytical requests into the NWQL's main database. This database is the NWQL's principal record-keeping system, and is permanently maintained by a professional staff of data-processing personnel. Data-processing quality assurance measures are described in this manual. The database serves as the repository for all analytical data. After entry into the database, the request forms are converted daily to a series of laboratory workload requests. As results of individual determinations are reported to the ADP section by analysts, they are compiled into sample-analysis reports in this database. After completion of all requested determinations for any individual sample, the analysis is submitted to a data-checking program, which is described in this manual, and released to the requestor when all quality control criteria are met. If all criteria cannot be met, appropriate messages describing problems that could not be solved are appended to the analysis report.

2.2 Blind Quality Control Sample Insertion

The log-in group inserts blind quality-control samples into each day's workload on a schedule determined by the quality control group. These samples are repackaged and labeled to be indistinguishable from the samples submitted by field offices. Blind samples are currently used in all determinations for which standard reference water samples (SRWS) are available.

Special Handling (Circle as appropriate and explain in record 5)

Hazardous material

Site Type (circle one)

SW - Surface Water
GW - Ground Water
ME - Meteorological
LK - Lake
ES - Estuary
SP - Spring
SS - Special Source

Field ID

Station Name | Field Office | Project | Collector | Phone (FTS)

File Deposition* (Circle one)

Q - WATSTORE
X - Lab File

For Laboratory Use Only

Record 1 — Sample identification

Station ID or Unique Number* | Project Account #

1 9 Year* | Month* | Day* | Time* (Begin Date) | Month | Day | Time (Composite End Date) | State Code* | District/ User Code* | County Code

Record 2 — Analysis level codes and schedules

Sample Medium** | Geologic Unit | Analysis Status** (H or 9) | Analysis Source** | Hyrdologic Condition** | Sample Type** | Hydrologic Event**

Schedule #1 | Schedule #2 | Schedule #3 | Schedule #4 | Schedule #5

Record 3 — Laboratory codes to be added to (A) or deleted from (D) above schedules

Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D

Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D | Code | A/D

Record 4 — Field values to be added to analysis.

WATSTORE/ Lab Code	Value	Rmk Code	QA Code	Meth Code	WATSTORE/ Lab Code	Value	Rmk Code	QA Code	Meth Code	WATSTORE/ Lab Code	Value	Rmk Code	QA Code	Meth Code
00027/ 83 Collecting Agency					82398/1201 Sampling Method Code					00061/ 61 Discharge, Instantaneous (cfs)				
72019/ 312 Depth to Water (BLS) (ft)					00020/ 65 Air Temperature (°C)					00010/ 64 Water Temperature (°C)				
00095/ 21 Specific Conductance (umhos)					00400/ 51 pH, Field					00025/1167 Barometric Pressure (mm Hg)				
00300/ 25 Dissolved Oxygen (mg/L)					00410/ 2 Alkalinity, Field (mg/L)					/				
/					/					/				

Records 5, 6 — Comments (limit to 138 characters)

Record 5

Record 6

Total number of sample bottles for this request: _____

* — Mandatory for acceptance for laboratory analysis
** — Mandatory for storage in WATSTORE

U.S. GOVERNMENT PRINTING OFFICE 679-483 1984
Revised 10/84

Figure 1.--Central Laboratories Analytical Services Request Form.

2.3 Holding Times

The maximum holding times from date of sampling required to be met by the NWQL are contained in the official parameter-code dictionary, which is maintained in the NWQL computer. They are summarized below:

Acidity, alkalinity, pH, and specific conductance	7 days
Nutrients	8 days
Anions and low-level major ions	28 days
Trace metals and major cations	42 days
Dissolved and total solids	42 days
Phenols, total	7 days
Methylene blue-active substances	14 days
Volatile organic chemicals	14 days
Acid-base/neutral extractables:	
Extraction	2 working days after receipt
Chromatography	45 days
Pesticides:	
Extraction	2 working days after receipt
Chromatography	45 days

In certain instances, it becomes necessary to proceed with an analysis even though the holding time has been exceeded. This must be clearly documented in the analysis report sent to the requestor. Such analyses are not included in the U.S. Geological Survey's national water information system database.

2.4 Sample Storage

Samples undergoing analysis are in the custody of the analytical section that performs the analysis until all determinations have been completed. Custody of completed samples reverts to the logistical-support section, which stores them in a warehouse area in the NWQL building. When warehoused samples are needed for reanalyses, they are retrieved by the logistical-support section and returned to the analytical section along with the analysis request.

Organic samples which require refrigeration are stored in the organic section.

Samples are stored for 30 days (minimum) after delivery of data reports to the requestor's office. Unless a reanalysis is requested during these 30 days, the sample may be released for disposal. Samples are retained as long as available space permits; usually about 6 months.

3. REAGENTS, GLASSWARE, AND LABORATORY SERVICES

3.1 Reagent Specifications

The specifications for reagent purity for each method of analysis are part of the bench-level protocol for the individual method. In general, American Chemical Society (ACS) reagent-grade or chromatographic-grade chemicals are required for all processes, unless the individual method requires a chemical with higher purity. Only in cases where ACS reagent-grade chemicals are not available through any source are less pure chemicals permitted.

All reagent preparations are clearly labeled as to the contents, concentration, date of preparation, and initials of preparer. Unstable preparations are prepared on a regular schedule as specified in the official method of analysis. All new reagent lots and preparations are tested by using them in the analysis of blanks and standards before they are used in actual sample analyses. New reagent-preparation dates are noted in logbooks.

3.2 Glassware Specifications

All volumetric glassware are class A. The washing of analytical glassware for each type of analysis is specified in the bench-level protocol for the method and also is maintained in the bench-level protocol of the glassware-washing group. It is the responsibility of the analytical section chief to ascertain that the glassware-washing protocol is correct. The chief of the logistical-support section is responsible for determining that the glassware-washing protocol is accurately followed.

3.3 Laboratory Services

The deionized-water system consists of a sand filter, a softener, a carbon filter, a reverse-osmosis unit, and a final mixed-bed deionization unit. The water produced by this system is used for rinsing glassware and for other nonreagent uses.

Reagent chemicals, calibration standards and sample dilutions for inorganic analysis are prepared using water that has been passed through an additional mixed-bed deionizer. This doubly deionized water must have a specific resistance of at least 12 megohm-centimeters.

For organic analysis, laboratory deionized water is further purified by distillation and carbon filtration.

4. INSTRUMENT CALIBRATION AND MAINTENANCE

Instrument maintenance is a continuing process that is a regular part of the duties of each analyst in the NWQL. A logbook is maintained for each instrument. Analysts are required to enter into this logbook a description of all maintenance procedures at the time they are performed. Supervisors regularly review these logbooks for completeness and adequacy, as well as to apprise themselves of any patterns of excessive maintenance that may indicate the need for equipment replacement.

Specific periodic maintenance and calibration checks are performed on schedule as indicated below:

4.1 Analytical Balances

Balances are professionally serviced and calibrated semiannually using class S weights.

4.2 Atomic Absorption Spectrometers

Burner heads and nebulizer chambers are cleaned daily. Optimization of nebulizers and burner position is checked daily. Photomultipliers and lamps are replaced as required based on daily checks of energy readings and lamp currents.

4.3 Conductivity Meters

Conductivity meters are calibrated daily using the procedure described by Friedman and Erdmann (1982).

4.4 Continuous-Flow Automated Analyzers

Pump tubing is changed on a preset schedule that is part of the bench-level protocol for each different methodology. Optical alinement of colorimeters is optimized periodically on a regular schedule and each time the lamp is changed.

4.5 Gas Chromatographs

Injection-port septa are changed after each autosampler run (a maximum of 50 injections). Chromatographic columns are replaced when column resolution begins to deteriorate. Major repair and maintenance is performed according to manufacturers' specifications.

4.6 Mass Spectrometers

Sources are cleaned or replaced whenever spectral background indicates the need. Major repair and maintenance is performed according to manufacturers' specifications and schedules.

4.7 pH Meters and Electrodes

pH meters are calibrated daily using commercially-prepared, National Bureau of Standards-traceable buffer solutions. Buffer solutions are used once and discarded. At least three different pH solutions (4.00, 7.00, and 10.00 or equivalent) are used. Measurements of all three buffers must be correct within the readability of the instrument after calibration.

5. INSTRUMENT STANDARDIZATION PROCEDURES AND FREQUENCIES

Procedures and frequencies of standardization are specified in all U.S. Geological Survey standard methods of analysis. Daily or more frequent standardization is the norm, except in the case of certain internal-standard

procedures. In those cases, the existing standardization factor is checked at least daily, and accepted only if the accuracy specified in the standard method or bench-level protocol is met.

5.1 Preparation of Standard Solutions

All reagents used in the preparation of standards are the highest-purity materials practically available and meet all requirements specified in the standard method. In the case of materials that are not available in a suitable purity such as ACS reagent or primary-standard grade, it is necessary to obtain an independent measure of accuracy, such as comparison to a second source of material or analysis of the standard by an independent method.

Fresh standards are prepared as frequently as specified by the standard method. When a fresh standard is prepared, it is checked by comparison to the previously-prepared standard and, when possible, by independent methodology or by cross-analysis in another laboratory. All standard preparations are labeled with contents, concentration, date of preparation, and initials of preparer. The date of preparation and initials of preparer also are entered in the logbook.

5.2 Frequency of Standardization

Standardization is performed daily or more often as required, except in the case of certain internal-standard techniques such as gas chromatography/mass spectrometry; in those cases the standardization is checked daily and accepted only if the method-specific criteria are met.

5.3 Number of Standards

With few exceptions, a five-point standardization curve is used in all methods. In cases where a specific instrument is programmed to accept fewer than five standards, the remaining standards are used as check samples to verify the curve between the calibrating points.

5.4 Periodic Verification of Standardization

The standardization is verified initially by analyzing all of the standardizing solutions as samples. During a series of analyses, the standardization is rechecked periodically by analyzing one standard solution as a sample after, at most, every 20th sample and at the end of the series of analyses. Insofar as possible, the standard solution is chosen to be similar in concentration to the samples being analyzed. The result must meet the precision criteria specified in the standard method or in the bench-level protocol.

5.5 Periodic Standard-Reference-Material Check

The precision and accuracy of the method is verified by the analyst by analyzing a standard-reference material, if one is available for the parameter being determined, every 20th sample. The result must meet the method-specific precision and accuracy criteria. In the case of SRWS, the

usual criterion is 1.5 interlaboratory standard deviations from the interlaboratory mean value. In a few cases, a more stringent criterion may be used if the particular method for determining the parameter in question is more precise than the methods used in determining the interlaboratory values. For example, this is the case in some methods designed for the analysis of precipitation samples, where constituent concentrations are small.

6. ANALYTICAL PROCEDURES

6.1 Standardized Methods of Analysis

All regular methods of analysis offered by the NWQL are contained in the current editions of manuals, such as those by Fishman and Friedman (1985) and Wershaw, Fishman, Grabbe, and Lowe (1982). Newer methods that are pending inclusion in a forthcoming edition may be used after having been approved by the chief of the methods development and technology-transfer program. Such methods are on file in the office of the chief for methods development and technology transfer. Each approved method is identified by a specific laboratory code, and data produced by these methods may be included in the U.S. Geological Survey's National Water Information System database. The description of each method in this set of manuals includes data on the expected precision of the method, where available. Newer methods that are pending inclusion in a forthcoming edition of these manuals may be used after having been approved by the chief of the methods development and technology transfer program.

6.2 Bench-level Protocols

For each standard method commonly used in the NWQL, there also is a written protocol that details the specific application of the method using current equipment and facilities. This protocol is much more specific than the standard method and serves as an aid to the memory of the operator, as a training guide to new operators, and as a documentary record of the details of application of the method. The protocol may be updated with the agreement of the operator's immediate supervisor, but the update must be filed with the quality control officer as a matter of record.

6.3 Nonroutine Methods of Analysis

So-called "special analyses" may be performed using minor modifications of the approved methods. So-called "custom analyses" are those that require major modifications of existing U.S. Geological Survey methods or other methods not previously approved for use in the NWQL. The results of special and custom analyses are transmitted to the requestor via a letter report. The letter-report documents the exact modifications, or method, used to produce the results reported. Because precision and accuracy statements similar to those contained in the standard methods are not available for special or custom methods, it is necessary to include additional information on the estimated precision and accuracy of special and custom analyses. This typically is done by including in the letter report the results for the analysis of spiked samples, reference materials,

or duplicate analyses, or all three, in the letter report. The letter report also contains a statement indicating that the methods used for special and custom analyses are not completely documented, and that the data need to be used with caution.

7. OPERATOR/SECTION INTERNAL QUALITY CONTROL--INORGANIC

7.1 Instrument Logbook

Each analyst maintains a logbook that contains notations concerning all instrument maintenance and repair procedures, preparation of new reagents and standards (check data verifying the accuracy of these solutions), daily standardization or calibration data, daily standard reference sample values, and other instrument parameters that document the validity of the data. Logbooks are regularly reviewed by supervisors for completeness and to obtain information concerning the condition of the equipment and any problems with the analysis.

7.2 Standard Reference Materials

Each analyst analyzes standard reference materials (SRM), such as SRWS, immediately after calibrating each instrument and periodically throughout every set of determinations with a frequency of about every 20th sample, depending on the characteristics of the determination being performed. These values are recorded in the logbook and, when possible, on a quality control chart.

The SRM values must meet certain specifications of precision and accuracy in order for the analyst to proceed with the analysis. For SRWS, this specification is 1.5 interlaboratory standard deviations from the interlaboratory mean. Where SRWS are not available and other SRM are used, a precision specification is set by the supervisor and included in the bench-level protocol for the method.

7.3 Standardization Check Samples

Every 20th sample, a standardization check sample, which is usually one of the standardization solutions, is analyzed to check for drift in the calibration. The specification of precision for this check varies with the precision of the method and is included in the bench-level protocol for each method.

7.4 Data Report

Data are reported to the ADP section on computer-generated worksheets. Each analyst is responsible for reviewing his/her own work sheets and then signing and dating each page. These work sheets are filed as a permanent legal record of the data. SRM values and standardization checks are included on these work sheets to validate the data. Cross reference to the logbooks provides complete documentation.

7.5 Training

All on-the-job training is the direct responsibility of the section chief. This responsibility may be delegated only to senior professional personnel. In the unlikely event that a new employee has to be trained by someone other than a senior professional, the training always is personnally observed by the section chief.

Training always is conducted from printed operating procedures including standard methods, instrument manuals, the NWQL's safety handbook (Skinner and others, 1983), and bench-level protocols prepared by the supervisor. The operator is supplied with copies of all pertinent procedural manuals by the section chief. Before an employee is certified to perform a given process, a set of quality control samples is analyzed to verify that the process is being performed to U.S. Geological Survey standards.

8. OPERATOR/SECTION INTERNAL QUALITY CONTROL--ORGANIC

8.1. Instrument Logbook

Each analyst maintains a logbook that contains notations concerning all instrument-maintenance procedures, preparation and testing of all new reagents and standards, daily calibration or standardization data, and daily instrument parameters that document the validity of data. Logbooks are regularly reviewed by supervisors for completeness and to obtain information concerning the condition of equipment and skills of the analysts.

8.2 Sample Documentation

Within the section, a separate computer log is maintained that lists the date and the operator who performed each step of the analytical process. The turnaround times for processes, such as sample extraction, are carefully monitored.

8.3 Surrogate Compounds

Surrogate compounds are added to all samples for all gas-chromatographic determinations. Recovery data are collected for quality control studies. On letter-reported analyses, surrogate-compound recoveries are disclosed to the requestor as a quality control statistic. When duplicate samples are available, reanalysis of samples with unacceptably low surrogate-compound recoveries will be performed. Plans are being developed to report surrogate-compound recoveries for all analyses.

8.4 Spike Recoveries

A spiked distilled-water sample is added to every set of 10 or fewer samples. The percentage recovery of the spikes is tabulated for future use as an acceptance criterion for individual data sets and as a precision statement for the methods manual. For the organochlorine, organophosphorus, and triazine pesticides, each spike does not contain all the compounds determined, but the set of spiked solutions, analyzed in rotation, does.

For volatile organic compounds, chlorophenoxy herbicides, and carbamate herbicides, all compounds are contained in a single spiking solution. Only the surrogate compounds are spiked for the acid-base/neutral extractable-compound schedules at present.

8.5 Data Validation

All gas-chromatographic computations are performed using a Hewlett-Packard Model 3357* data system, which is online for all gas chromatographs. Computations are checked by the analyst and reviewed by a second analyst.

For two-column analyses, the lesser of the two determined values is reported. The greater value is assumed to be interfered by an unresolved impurity.

Identification of a peak in a chromatogram requires: (1) The retention time of the peak must be within a "window" of 0.1 minute of the standard peak on both columns; (2) any discernible peak shift within that window must be duplicated by the surrogate-compound peak; and (3) the peak sizes in the two-column chromatograms must be in proper proportion to each other, unless an obvious unresolved interfering peak is noted.

8.6 Training

All on-the-job training is the direct responsibility of the section chief. This responsibility may be delegated only to senior professional personnel. In the unlikely event that a new employee has to be trained by someone other than a senior professional, the training always is personnally observed by the supervisor.

Training always is conducted from printed operating procedures including standard methods, instrument manuals, the NWQL's safety handbook (Skinner and others, 1983), and bench-level protocols prepared by the supervisor. The employee is provided copies of all pertinent procedural manuals. Before an employee is certified to perform a given process, a set of quality-control samples is analyzed to verify that the process is being performed to U.S. Geological Survey standards.

9. LABORATORY AND EXTERNAL QUALITY CONTROL

9.1 Laboratory Blind-Sample Program

The log-in group inserts SRWS and other SRM as blind quality control samples on a daily schedule determined by the quality control office, to verify the precision and accuracy of inorganic parameters. The data from these analyses are plotted on quality-control charts, which are reviewed monthly by the quality control officer, by the senior staff of the NWQL, and by the analysts. The criterion for accuracy of these parameters is plus or minus 1.5 interlaboratory standard deviations from the interlaboratory mean.

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*The use of the brand name in this report is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.

Failure to meet this standard results in re-evaluation of the day's analyses; repeated failure may result in shutdown of the determination until the cause of the difficulty is determined and corrected.

Quality control samples for organic analyses are not currently available in the SRWS program, but are being developed by the Branch of Quality Assurance and the NWQL quality control office; the quality control office will distribute the samples as soon as they are available.

9.2 External Blind-Sample Program

The Branch of Quality Assurance, which is completely independent of the NWQL, submits blind reference samples that are prepared by mixing various SRWS. These samples are submitted through field offices, who include them as part of regular sample shipments. An average of one sample per day for each determination covered by the SRWS program is submitted. The data are analyzed by a quality assurance chemist and are compiled into semiannual reports, which are widely circulated throughout the U.S. Geological Survey. In addition, a weekly report to the NWQL chief provides timely warning of any problems as they develop.

9.3 External Performance-Audit Samples

The NWQL's policy is to participate in all appropriate interlaboratory standard-sample-analysis programs. The NWQL regularly participates in the U.S. Geological Survey's SRWS performance evaluation tests, the U.S. Environmental Protection Agency's "Water Supply" and "Water Pollution" performance-evaluation sample programs, and Canada's Centre for Inland Waters' "Long Range Transport of Atmospheric Pollutants" (LRTAP) program round-robin analyses. Results of these programs are carefully reviewed by staff and management of the NWQL, and by headquarters personnel to determine whether any corrective actions are required.

9.4 Data Review

Completed samples are subjected to a computerized review for internal consistency of data, such as cation-anion balance, specific conductance concentration relations, and dissolved-ion total-ion concentrations. The tests are specified in Friedman and Erdmann (1982). Samples which satisfy these checks are released to the requestor through the U.S. Geological Survey's computer network. Samples that do not pass these checks are referred to the quality control office for inspection and resolution of the problems, and request for reanalysis of selected parameters as deemed necessary. Some computer-rejected samples can be explained as special cases not expected to meet ordinary criteria; analyses of these samples are released without further inquiry. Other samples require extensive efforts to resolve analytical or data-transcription problems, or labeling errors by the log-in group or field personnel.

After data are released, 30 days are allowed for review of data by the requestor. During this time, additional reanalysis may be requested on the basis of the requestor's review of the analysis for hydrologic sense. After this time, the sample may be discarded.

10. DATA VALIDATION AND REPORTING

10.1 Data Entry

Nearly all data are entered manually into the computer database by data-entry operators. As yet, relatively few instruments are online. Data are entered, checked for errors, and reviewed again by a second operator.

10.2 Data Validation

Completed samples are reviewed as described in section 9.4 above. Every possible effort is expended to ensure that every analysis produced by the NWQL is correct. As a final check, one sample data file from each workfile is compared to the original values entered into the database before data are transmitted. Punch verification is planned.

10.3 Data Reporting

Data reports for regular analyses involving standardized tests are compiled in the NWQL's central computer and are delivered to the requestor via the U.S. Geological Survey's Distributed Information System. It is the responsibility of the requestor to enter all appropriate data into the U.S. Geological Survey's National Water Information System.

10.4 Data Archiving

To comply with U.S. Geological Survey policy, all original data records must be permanently filed. NWQL records such as logbooks and workfiles containing original results are retained. The NWQL database is retained on disk or tape for a period of several years at present; in the new NWQL information-management system now being installed, provision is being made for permanent tape archiving of data and accompanying quality assurance information.

10.5 Computer and Database Security

Incremental backup of all active files is done twice each day, at noon and at close of business. Global backups are performed weekly. Incremental backup tapes are saved for 2 weeks. Global backup tapes are saved for 3 weeks. Tapes are stored in a room away from the computer.

Combination locks are used to restrict access to the computer room. Combinations are changed monthly. Databases are all password protected with different levels of access available depending on the nature of the individual need.

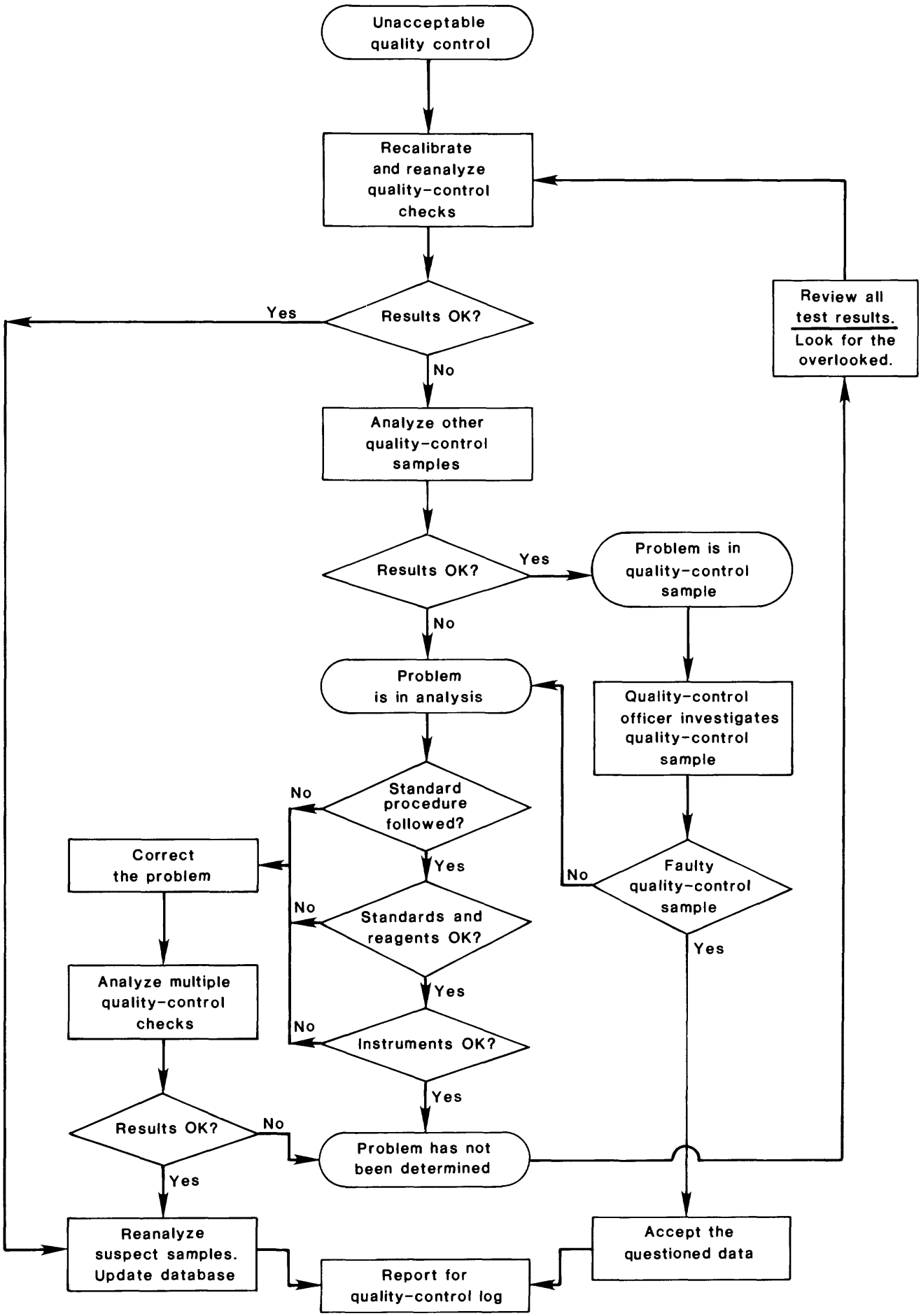

Figure 2.-- Corrective-action sequence Inorganic analysis.

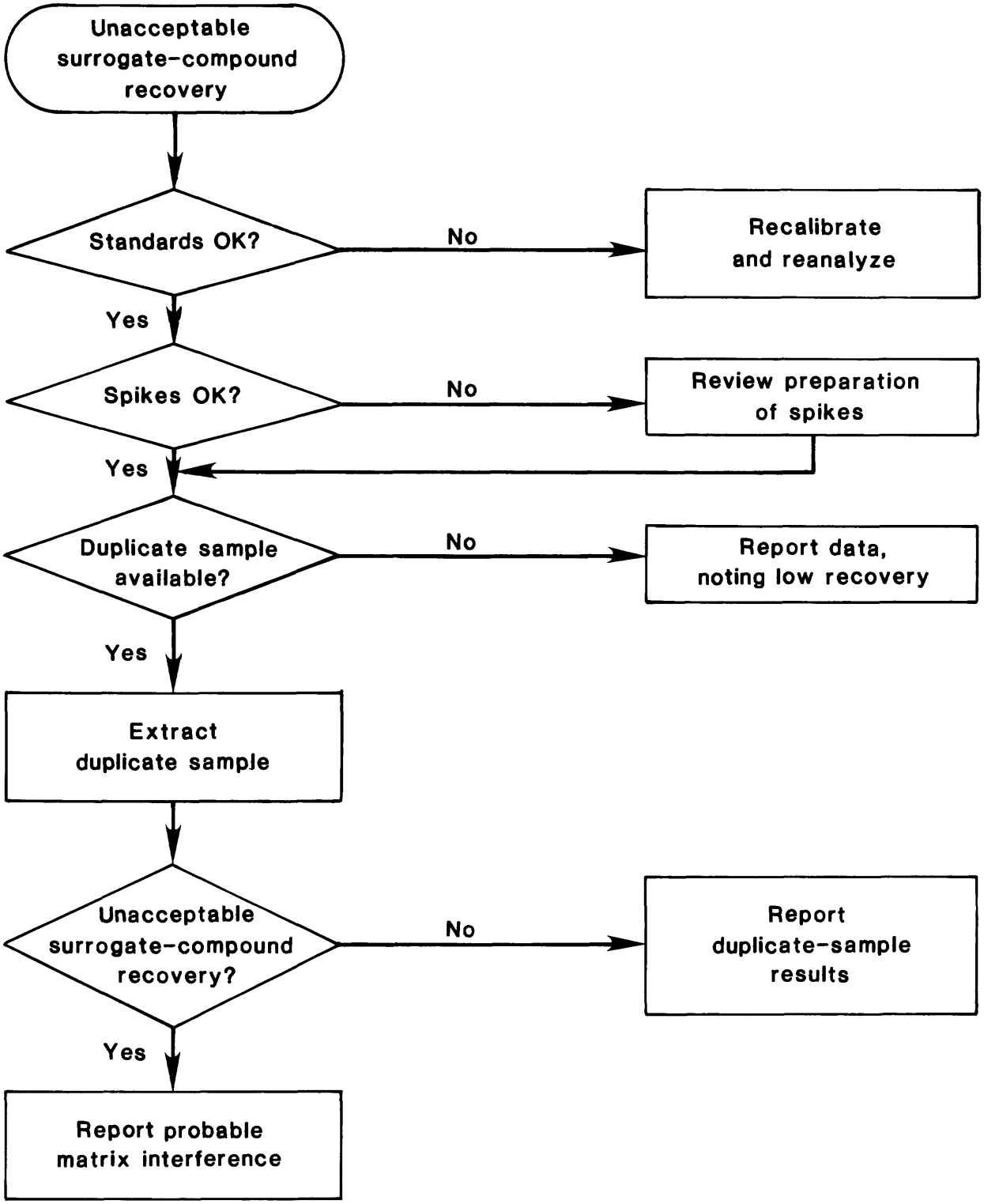

Figure 3.-- Corrective-action sequence Organic analysis.

11. CORRECTIVE ACTION

11.1 Inorganic Analyses

Corrective action is required whenever the result of a quality control blind check sample is rejected. A flowchart showing the sequence of corrective actions usually required is shown in figure 2.

11.2 Organic Analyses

Corrective action is required whenever a surrogate-compound recovery is outside the allowable ranges. At present, this action includes: (1) Rechecking the calibration of the instrument; (2) reviewing spiked sample recoveries in the same analytical series; and (3) reviewing sample-preparation steps for any discrepancy. A flowchart showing the sequence of corrective action usually required is shown in figure 3.

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