* **Sample dehydration**
* **Analysis environment**

CONTAINER CLEANLINESS

The cleanliness level of sample containers, process fluids, and the surfaces of analysis equipment are all external influences affecting the validity of particulate contaminant analysis. The manner in which a crucial item is cleaned is not nearly as important as having complete confidence in its cleanliness.

Using new, or "surgically clean" bottles as fluid sample containers is totally inadequate. Bottles free from live microorganisms may contain a high level of organic and inorganic particulate matter. Individual particles below approximately 40 micrometres cannot be seen with the naked eye. However, the fact that particles or soil are not visible is no assurance of a container's cleanliness level.

One fact worth remembering is that effective cleaning of any component depends not only on separating and dislodging particulate matter from its surfaces, but also on the cleanliness level of the rinsing fluid. Experimental studies have shown that, under ordinary conditions, an ultrasonic bath with a power level of at least 10 watts per sq. in. of bath area is necessary to detach particulate matter from most surfaces. Ultrasonic wave transmission increases when detergents are added to the fluid. Detergents are also helpful in removing oil films and soil from the surfaces to be cleaned.

As a guide for those wanting to establish a **cleaning procedure** for sample containers, the procedure used by the FPRC/OSU may be of help --

1. Dump contents of sample container, rinse container with ether, and arrange containers into batches of 20.
2. Fill ultra-sonic unit with hot water and 50 mL of ZZ Chemical Cleaner.
3. Insert bottles and let soapy water run into them.
4. Cover the ultrasonic with a sound suppressant top and operate ultrasonic for 5 min.
5. Drain water from bottles, and rinse soap from the tank and bottles.
6. Rinse bottles once for 5 min. by filling the ultrasonic and bottles with clean hot water and 10 mL of reagent grade acetic acid to neutralize the alkaline effects of the cleaner.
7. After the ultrasonic rinse, flush each bottle with "soft" tap water; then, drain and turn upside down in drying rack.
8. Rinse thoroughly with alcohol and then with ether.
9. After the ether evaporates (fumes are no longer exiting from the mouth of the bottle), spray a plastic square with ether and let it dry.
10. Cover the bottles with the sprayed side of the plastic; put a rubber band around the bottle neck and cap.
11. Continue until 20 bottles are completed. Keep the bottles upside down at all times until the opening is covered with plastic.

Evaluating Container Cleanliness

The **cleanliness level of a sample container** can be evaluated by using the following procedure:

1. Fill the container to 50 (plus/minus 5) percent with super clean fluid having a cleanliness level less than one particle per mL greater than 5 micrometres (in 10 mL of fluid there should not be any particles greater than 10 micrometres).
2. Replace the film covering and cap. Shake the container vigorously.
3. Using an automatic particle counter calibrated per ISO Standard 4402, determine the number of particles per millilitre of fluid greater than 10 micrometres.
4. Multiply the particle count by the ratio -- (fluid volume added to the sample container volume divided by the total sample container volume).

The number obtained in Step 4 is the container cleanliness level in particles greater than 10 micrometres per mL of container volume.

ISO Quality Assurance

ISO Standard 3722, for qualifying and controlling cleaning methods for fluid sample containers, ignores the technique by which the containers are cleaned. The qualifying proce-

dure is based upon the assumption that, if a sufficient number of consecutive clean sample containers produced by a given process are inspected and all of them meet a specified cleanliness level, there is a high level of confidence that the process will continue to produce a like product. The method also states that if samples from an outgoing flow of clean sample containers are selected at random, any deviation from the accepted cleanliness value will most likely reflect a change in the process.

The flow chart shown in Fig. 2-1 illustrates the different aspects of the qualifying procedure. The flow chart indicates that, until a cleaning process is qualified, each container must be 100 percent inspected using the evaluation procedure described. After qualification, periodic inspection of the containers as specified in the ISO document is permitted.

The **Required Cleanliness Level (RCL)** specified in the standard should be selected so

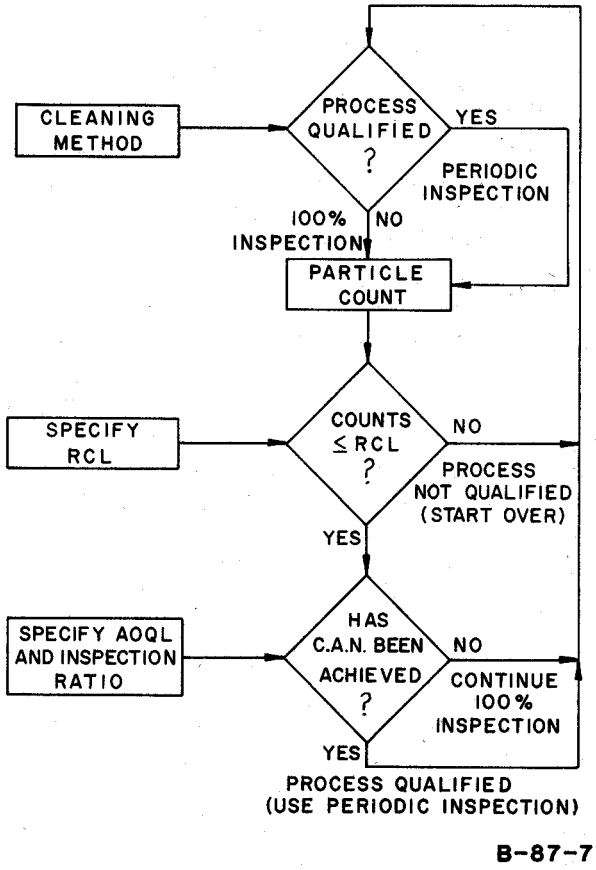

Fig. 2-1. ISO Container Qualification Flow Chart.

that two orders of magnitude separate the sample container cleanliness from the anticipated fluid contamination level. An **RCL** of 1.5 particles greater than 10 micrometres per mL of container volume is a practical value to achieve and maintain. The RCL Selection Nomograph shown in Fig. 2-2 was designed to aid in the selection of the RCL value.

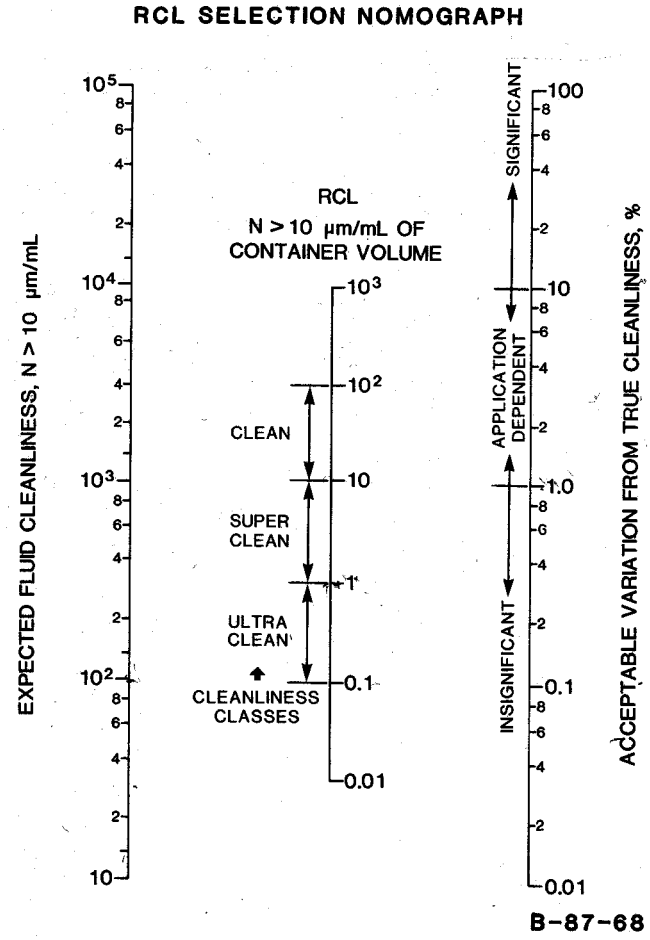

Fig. 2-2. Required Cleanliness Level for Sample Containers.

To establish a value for the **Average Outgoing Quality Level (AOQL)**, trade-offs are generally made with the **Inspection Ratio (IR)**, as shown in Table 2-1. If a decision can be made that as many as 1 percent of the samples can be in error, then a 1 percent **AOQL** is selected. Regardless of the value of the **AOQL**, the actual percent of defective containers may be zero. A 1 percent **AOQL** is generally a satisfactory value to use.

Table 2-1. Consecutive Acceptance Numbers for Specific Inspection Ratios and Average Outgoing Quality Levels.

B-88-194

INSPECTION RATIO	A.O.Q.L. VALUES				
	10%	5%	2%	1%	1/2%
1 in 5	–	13	35	70	115
1 in 10	10	21	55	103	210
1 in 20	14	29	68	115	310

The IR requires production considerations as well as confidence in the cleaning process. A facility that produces about 50 containers per day probably cannot afford a high sampling ratio of, say, 1 in 5, which would require 10 checks per day. A ratio of 1 in 20 would be preferable.

Once the IR and the AOQL of the process have been selected, the **Consecutive Acceptance Number (CAN)** -- the number of consecutive acceptable containers produced before the process is qualified -- can be established by using Table 2-1 or by applying the Dodge Romig Nomograph as shown in Fig. 2-3. This nomograph is the basis for the ISO sample container cleanliness certification.

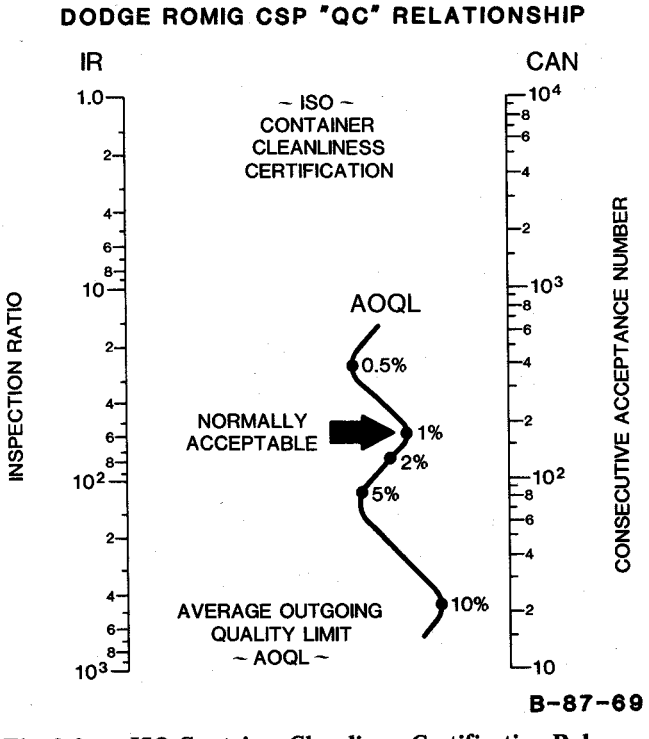

Fig. 2-3. ISO Container Cleanliness Certification Relationship.

FLUID SAMPLING

Fluid sampling can create a major source of error in particulate contaminant analysis. The vagaries of many sampling methods in the past have contributed to a general lack of confidence in practice -- a situation that still prevails in some sectors of the field. Much work has been accomplished in recent years to elevate sampling from an art to a science. This section will disclose this scientific approach to sampling.

A **sample** is a finite part (**a specimen**) of a whole -- or, more specifically, a representative collection of particles drawn from a statistical population. **Sampling**, of course, is the process of taking a sample. A sampling method which does not provide a representative fluid sample for the entire system can yield misleading and possibly dangerous results. There are two basic types of samples -- **static** and **dynamic**.

Static Sampling

Static samples are drawn from a fluid body at rest and suffer from the following problems:

- **Concentration gradients exist.**
- **Vertical segregation of particles by size and density exists.**
- **Horizontal particle gradients exist.**
- **No single point "average" sample extraction can be made.**
- **Fluid particle size distribution continually changes.**

Static samples taken from the bottom of a vessel may have value for chemical and analysis purposes. For example, trace amounts of precipitated constituents or contaminants can be detected through gravity separation methods. Such samples, however, are basically worthless for particulate contaminant analysis.

Dynamic Sampling

Dynamic samples are taken from fluid in motion. The basic types of dynamic sampling are laminar and turbulent flow sampling. Interest in laminar flow sampling has its origin from sedimentation studies in open channels and has no value for particulate contaminant analysis. This fact is especially true when it is realized that closed conduit fluid systems seldom, if ever, exhibit true