

# STANDARDS

## A PROGRESS REPORT ON STANDARDS FOR LABORATORY REAGENT- GRADE WATER

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ater is the most common component of all biological research and testing. However, excluding proprietary knowledge, almost nothing is known about the impact of water purity on modern laboratory applications. For as long as anyone can recall, standards for laboratory water have been so flawed and misleading there has been no practical way to compare results or keep score. Now, as momentum is building for reform, one practical new standard has recently been introduced and at least one more is in the pipeline.

### Historical Background

Historically, only two consensus standards have been widely applied to laboratory water purification. One is the National Committee for Clinical Laboratory Standards (NCCLS) C3-A3 Guideline, *Preparation and Testing of Reagent Water in the Clinical Laboratory*. This standard is used primarily by clinical laboratories, because the College of American Pathologists (CAP), which monitors clinical laboratories for proficiency, strongly recommends it. Research and reference laboratories have relied on the other standard, American Society for Testing and Materials (ASTM) International D1193, *Standard Specifi-*

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**Editor's note:** This article, presented at ULTRAPURE WATER Portland, raises some concerns by the author about the process and manner in which standards used to govern water treatment are developed. The comments made within the article are based on the author's involvement in this area. While different from the technical papers that we normally publish, we feel this subject is worth the consideration of our readers.

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cation for Reagent Water. However, both standards are awash with inconsistencies and lack requirements for meaningful validation. Critiques of these standards are easily accessible on the internet and should be of great interest to every laboratory (1, 2).

*United States Pharmacopeia 25-National Formulary (USP 25-NF)*, published by the United States Pharmacopeial Convention Inc., and ISO 3696, published by the International Organization for Standardization, are frequently mentioned in relation to laboratory water, but they do not really apply. *USP 25-NF* is primarily concerned with pharmaceutical safety issues, not reagent-grade purity, and ISO 3696 states, "It [3696] is not applicable to water for organic trace analysis, to water for the analysis of surface active agents, or to water for biological or medical analysis" (3, 4).

As a regulatory fig leaf, C3-A3 may serve a purpose; however, as a tool for clinical laboratory improvement, it misses the mark. C3-A3 does not state unequivocally where in a purification system measurements are to be made. Moreover, the guideline's recommendations for controlling water quality depend almost exclusively on the measurement of resistivity, determined only once a day. There are no controls for organic contamination, which is likely to be far more harmful to clinical laboratory testing than traces of salt. And control of microbiological contamination is based on weekly viable plate counts, which are notoriously inaccurate and take at least a week to develop (5).

Furthermore, C3-A3 Type I water is required to have a 10 megohm-cm lower limit for resistivity (referenced to 25 °C), without regard to the important differences between water purification systems that use cold-technologies (e.g., ion exchange, carbon sorption, and ultrafiltration) and those that use distillation. Distilled water with a resistivity of 10 megohm-cm is likely to be quite pure; conversely, 10 megohm-cm water from a cold-technology system is certain to be quite impure and the system may be failing or poorly designed.

The ASTM International maintains that D1193 Type I "has virtually nothing in it" and requires its use to determine the

precision and bias of all ASTM D-19 (Water) Committee methods standards<sup>a</sup>. And a high percentage of research and reference laboratories have been led to believe that water produced by so-called, "D1193 Type I systems" is essentially *high-purity water*. However, D1193 is nothing more than a vague *process specification*, not a reagent water *product specification*. Paragraph 1.2 states, "The method of preparation of the various grades of reagent water determines the limits of impurities . . ." and there is no explicit requirement for the monitoring of product water. In fact, D1193 does not even require maintenance of the specified purification processes. D1193 Type I water is so poorly defined that it has to be an inconsistent unknown. And in April of 2001, the American National Standards Institute (ANSI) declined to accept D1193 as an ANSI standard, in spite of the fact that ASTM International is an ANSI accredited standards developer organization.

### The Consensus Process

Consensus organizations can be big businesses and concerned with raising money to ensure that they remain in business, even those that have not-for-profit status. Consensus organizations aggressively market their imprimaturs and give every impression their standards are scientific and technically sound. According to Morris Brooke, ASTM International's General Counsel, "When a consensus is reached among interested and affected parties within a technical committee operating under due process, that's as good as it gets. There is no meaningful place to *appeal* from there" (6). In contrast, Galileo was struggling with a consensus standard when he made a less sanguine observation, "In questions of science, the authority of a thousand is not worth the humble reasoning of a single individual." Galileo's words transcend the centuries – the consensus process will always be fundamentally political, not scientific. An unscientific and technically unsound standard does not get better as it gains wider acceptance, it simply becomes more destructive.

**The imprimatur.** Does the imprimatur

of a nationally or internationally recognized consensus organization add any assurance that a standard is technically sound and unbiased? Not really, and the condition of C3-A3 and D1193 serves as proof. In fact, a number of consensus organizations, including ASTM International, argued as much before the United States Supreme Court nearly 20 years ago in a case where a small number of individuals had used the consensus process to unfairly eliminate competition for their business interests<sup>b</sup>. The gist of the argument made by the consensus organizations was that their standards are written and approved by *independent* committees, subcommittees, and working groups – consensus organizations only serve to facilitate this process. However, the Supreme Court noted: “[A consensus standard organization] wields great power in the Nation’s economy, and when it cloaks its subcommittee officials with the authority of its reputation, it permits those agents to affect the destinies of businesses and thus gives them the power to frustrate competition in the marketplace.”

#### Who joins consensus committees?

There are many reasons why an individual, or an organization, would voluntarily join a consensus committee; however, while altruism undoubtedly motivates some, others join for different reasons.

Consensus organizations require large numbers of members to lend authority to their standards, and it is common practice to encourage membership by providing prerequisites that offset the costs. The *résumé factor* is routinely enhanced by providing members with free copies of standards, listing members in directories (e.g., reference labs, expert witnesses, lecturers), and providing specialized services. Such ostensibly benign practices have serious consequences when combined with another ostensibly benign practice, requiring members to vote in order to retain their membership.

In combination, these practices load consensus organizations with large numbers of voters, who are unlikely to read the documents on which they vote. However, their votes can amplify decisions made by a mere handful of persons into what appears to be thunderous approval by a huge committee of supposedly *interested and affected parties*. The fact that D1193 was overwhelmingly approved in 1999, despite serious editorial errors and internal inconsistencies, implies that a very high percentage of D-19 members must not

have read it<sup>c</sup>.

A significant number of persons join consensus organizations as a means of protecting, or promoting, their special interests, or the special interests of an employer. These persons are typically well informed, perhaps among the best informed, and it is natural that their opinions are respected and their suggestions welcomed. They are also the most likely to be in a position to appreciate, and manipulate guideline details. And, they are the most likely to be able to justify the great expense involved with drafting standards and attending in-person committee meetings – where agendas are set and the serious politics takes place<sup>d</sup>. Millions upon millions of dollars are at stake in the laboratory water purification market; it would be naive to believe that special interests make no effort to influence the outcome of pertinent consensus standards.

**Controlling special interests.** Consensus organizations consider special interests to be legitimate interests, and why not? Of course, consensus organizations have regulations prohibiting more than one member of a special interest voting block from voting. On paper these regulations look good; however, there can be a gulf between theory and practice. If a membership committee does not have the resources to confirm the current employment of a member, or prospective member, let alone perform independent thorough background investigations for possible conflicts of interest, what good are the regulations<sup>e</sup>? Membership committees may consist of one or two active members, who volunteer their time and have virtually no budget. If a new, or existing, member lists his or her home address on an application and claims to have an independent interest in a committee, what is the membership committee supposed to do? And how can the membership committee determine the voting links between members who work for different divisions of large corporations?

In 2001, an outside audit of the D-19 membership roster turned up so many examples of multiple voters for special interest voting blocks that D-19 was compelled to recount an entire full committee ballot, including over 50 ballot items<sup>f</sup>. As a result of the recount, a new standard for laboratory water, which had been defeated in the first count, gained enough votes to continue the approval process. Perhaps in response to this debacle, ASTM International changed the way membership rosters can be

accessed – now, it is much more difficult to perform an independent audit.

Officers of committees, subcommittees, and working groups have enormous power to influence an outcome and who is to say if these officers represent special interests, directly or indirectly. A special interest as uncomplicated as *pride of authorship* can insert tremendous bias into the consensus process. Officers can set agendas and control the tone of discussions. And, the paid staff of consensus organizations are naturally eager to please the officers of a committee. There are many little things staff can do to be helpful, such as alerting an officer to early voting returns. Negative votes carry so much weight that making one or two calls to persons, who would vote negative with a little encouragement, can prevent the progress of a standard.

The argument that special interests will compete with one another to produce unbiased, correct standards simply does not hold water; there are many reasons for special interests to find common cause. Distorting a standard to promote the sale of an exceptionally profitable class of product is likely to have all the manufacturers of that class of product joining forces. It is also easy to understand how special interests might agree not to interfere with one another’s strategies.

The most recent changes to the D1193 table are an indication that checks and balances are not working. The text of D1193 explains that water from Type I systems (water produced by cold-technology systems) is likely to contain considerably more total organic carbon (TOC) contamination than water from Type II systems (water produced by distillation). Cold-technology systems are known to release, or pass, TOC contamination; whereas, distillation removes most TOC contaminants and should not add any (7, 8)<sup>g</sup>. Feeding a Type II system with water from a Type I system will reduce TOC contamination significantly; whereas, the reverse is not true. Yet, in 1999, manufacturers of cold-technology water purification systems lobbied successfully to reduce the TOC value in the Type I column of the D1193 table to match the value in the Type II column (50 ppb). This tweaking of the table amounted to nothing less than a marketing coup, because the table is all that most laboratories will ever see of D1193 (see the Promoting Consensus Standards section). Presented without context, the tweaked table gives the impression that Type I

water purification systems are superior to Type II systems, which is not the case. The systems are not comparable and each is better suited for some applications than others.

#### Hurdles to Participation

As mentioned, the serious business of passing consensus standards takes place during committee meetings. When barriers to participation increase, so does the likelihood that meetings will be controlled by special interests. These days, few people have the funds or the time to travel to two D-19 meetings a year, which many find to be inconveniently timed and located<sup>b</sup>. Attendance at recent D-19 meetings has been so low that it has been a common practice for officers to desperately solicit proxies in order that the 40-member quorum (20 members with 20 proxies) for the meeting of the D-19 Committee could be achieved<sup>d</sup>. Lists of persons available to carry proxies were posted.

On paper, consensus organizations encourage the use of proxies; however, in practice, notably where challenges to D1193 have been involved, certain proxies have not been counted (9). According to the ASTM Regulations (Section 9.5), no individual may hold or exercise proxies for more than one member in any committee, and soliciting proxies is prohibited. D-19 Bylaws defer to the ASTM regulation on proxies; however, D-19 officers and staff have interpreted the Regulations to mean that members must personally select and contact the individual to carry their proxies, without third-party assistance (10). The proxy rules tend to transfer power to the relatively small number of members who attend meetings.

**Natural selection within consensus organizations.** Unfortunately for the consensus process, the best and brightest of water experts, especially those associated with universities and laboratories, are unlikely to appreciate the rough and tumble politics of consensus organizations, where a vote does not necessarily have to be grounded in fact or reason. They drop out or avoid involvement altogether.

**Promoting consensus standards.** ASTM International D5196, *Standard Guide for Biomedical Grade Water*, is no more awash with inconsistency than D1193 (12). Yet, D1193 is perhaps the most widely cited laboratory water standard in the world and D5196 is essentially unknown. The difference is that

consensus standards like C3-A3 and D1193 have been promoted and D5196 has not. There is nothing inherently wrong with promoting a consensus standard; however, it does provide a means whereby special interests can add, or amplify, mischief, and a well-established standard is a more inviting target for special interest manipulation. CAP essentially requires all clinical laboratories to reference C3-A3 and ASTM International requires D1193 to be cited in all ASTM standards requiring purified water – standards that are referenced by the U.S. Environmental Protection Agency and the Department of Defense, among others<sup>k</sup>.

Standards are essentially legal documents, and a condensed version of a standard should be viewed skeptically, particularly when it is included in a catalog or other promotional material. Given an inch, competitive strategists (i.e., marketers) can take a mile. Tables, which supposedly summarize limits for contaminants in the various types of water specified by C3-A3 and D1193, have been so extensively promoted by manufacturers and distributors of water purification systems that it is likely the great majority of laboratories citing C3-A3 or D1193 have never seen the actual documents. The summary tables are routinely juxtaposed with advertisements for water purification systems that are rated by reference to the systems or types of water specified in the standards. And a consumer could be forgiven for leaping to the mistaken conclusion that a system rated as *Type I*, for example, would consistently produce water that satisfies the supposed limits in the summary tables. However, there is no factual basis for this conclusion. Certainly, the texts of C3-A3 and D1193 do not support such a conclusion. There is no simple, black box solution for purifying water – laboratories must understand the technologies used by their water purification systems and know how to properly maintain and monitor the systems. It is puzzling that NCCLS and ASTM International have not objected vigorously to the practice of summarizing C3-A3 and D1193, because their tacit approval lends legitimacy to a practice that seems bent on deception.

#### Progress

As increasing numbers of laboratories become aware of the shortcomings of C3-A3 and D1193 and come to grips with the limitations of the consensus process, the pace of progress has also increased.

**A/H LabWater-1.** By making it possible to develop, promote, and disseminate scientific materials inexpensively, the internet has enabled the creation of an alternative standard for laboratory water. The A/H Labwater-1 Standard, *Standard for Laboratory Reagent-Grade Water*, departs from tradition by taking the approach that specifications for laboratory reagent-grade water should derive their validity from scientific reason and widespread voluntary approval by laboratories, not from authoritarian fiat (14). The Standard uses a matrix of process and product parameters to permit the orderly specification of a range of reagent-grade waters. This novel approach provides anyone with a need to communicate, document, or specify the quality of reagent-grade laboratory water with the option of selecting the specification that best suits the requirements for a given application. The approach also permits the extension of the matrix in future versions of the Standard without redefining grades of water and without making specifications based on the current matrix obsolete. Furthermore, specifications based on the A/H Labwater-1 Standard are not vulnerable to special interest mischief, because they are laboratory driven and require validation. The A/H Labwater-1 Standard is currently available free of charge.

**NCCLS** has announced plans to replace C3-A3 with a new Guideline, C3-A4. The indications are that C3-A4 will describe practical, effective specifications for clinical laboratory water in a frank and technically accurate manner. This is a sea change and evidence for the momentum gained by the reform movement.

**ASTM International.** D-19 officers have circulated copies of the critique that details the deficiencies of D1193 to the full committee and requested suggestions for improving D1193 (1). This is a positive development and may pave the way for alternative standards. Another hopeful sign is that ASTM is experimenting with internet conferencing as a means of encouraging greater participation in standards development. ■

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#### Endnotes

<sup>a</sup>"In Committee D-19, Type I reagent water is the required matrix when performing a round robin to determine the precision and bias of a test method. Why? Because Type I is so pure. In other words, because Type I has virtually nothing in it, the test you are performing will not be adversely affected by the solvent (water) you use. . . . Without it [Type I water] the vast majority of Committee D19 test methods [thousands] would be little better than 'cookbook' chemistry." – *ASTM International D19 (Water) Committee Chairperson writing to the ASTM International Committee On Technical Committee Operations, August 28, 2001.*

<sup>b</sup>American Society of Mechanical Engineers, Inc. v. Hydrolevel Corp., 456 U.S. 556 (1982), Certiorari to the United States Court of Appeals for the Second Circuit No. 80-1765, Argued January 13, 1982, Decided May 17, 1982 – <http://laws.findlaw.com/us/456/556.html>.

<sup>c</sup>The D-19 Committee has more than 400 members, according to the ASTM Web site, [www.astm.org](http://www.astm.org) on Nov. 1, 2002.

<sup>d</sup>The D19.90 (Executive) Committee Minutes for the June 29, 2000, meeting include a report by the

chairman of the Membership Committee in which he reported that the great majority (80%) of those members who do not attend meetings cite an inability to justify the time or expense as their reasons for not attending.

<sup>e</sup>The D19.90 (Executive) Committee Minutes for the January 18, 2001, meeting include a report by the chairman of the Membership Committee in which he reported that only 57% of D-19 members had returned classification forms and that he classified the remaining 153 members with the help of the D-19 Chairperson. One third of the members were classified as manufacturers, one-third as users, and one-third as general interest.

<sup>f</sup>ASTM International Ballot D19-0101.

<sup>g</sup>"Measuring Resistivity Is Not Enough – Water at the theoretically pure limit of 18.2 megohm-cm may still contain high concentrations of neutral organic contaminants which may adversely affect your analytical methods and cause analyses to fail. Most water purification systems exhaust their capacity to remove dissolved organics before they lose their capacity to remove ions." – *A10 Brochure*, Millipore Corp. (1996).

<sup>h</sup>The D-19 Winter Meeting is held shortly after year-end holidays in Cocoa Beach, Fla., and the Summer Meeting is held shortly after Memorial Day in June, rarely in proximity to a major hub airport.

<sup>i</sup>The minutes of the D-19 Committee Meeting for June 2002 indicate that only 14 members and 2 proxies were present.

<sup>j</sup>ASTM D5196 is cited in only one ASTM standard, D1924, and may be dropped when D1924 is next revised. Repeated calls by the D-19 Committee for users of the standard to come forward in support of its continuation have not yielded a single response of which the author is aware.

<sup>k</sup>"*Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type—of Specification D1 193." – *Form and Style for ASTM Standards* (for years, including the most current edition, issued in September, 2002, Section A12.2) (13).

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