

A Critique of ASTM Standard D1193

Standard Specification for Reagent Water

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Foreword

In the mid seventeenth century, Galileo was struggling with a consensus standard when he said, "In questions of science, the authority of a thousand is not worth the humble reasoning of a single individual."^b Galileo's words transcend the centuries – the consensus process will always be fundamentally political, not scientific. An unscientific or technically unsound standard does not get better as it gains wider acceptance, it simply becomes more destructive. ASTM International's D1193 – Standard Specification for Reagent Water is a classic example of just such a situation.

D1193 appears to be a surprisingly vague, poorly edited, error-filled, and internally inconsistent *process*^c specification (not a reagent water *product*^d specification), which does not require any monitoring or maintenance of the specified purification systems. This critique of D1193 has been evolving with input from many interested parties since January of 1998 and its purpose is to serve as a banner around which the loyal opposition to D1193 can rally their forces in order to bring about a change. This foreword places D1193 in perspective, explains why it is so important for anyone who uses reagent-grade water to become familiar with the issues, and suggests how a scientifically and technically sound alternative standard might be developed.

An Overview of D1193

The significance of reagent-grade water — Reagent-grade water is widely used; however, it is absolutely essential to laboratories engaged in researching and testing biological systems (this includes medical laboratories), which represent the large majority of laboratories in general. Of all the purified chemicals used by laboratories, reagent-grade water is among the most difficult to purify and the most likely to be used in *concentrated* form (as a solvent). Traces of contamination in reagent-grade water are a much greater problem than traces of contamination in a reagent-grade salt, which would typically be diluted many times. Much of what laboratories study and measure can be significantly affected by trace levels of contaminants in reagent-grade water. And if laboratories cannot trust the consistency and purity of their reagent-grade water, can they trust their results.

How pure is reagent-grade water? — Potable tap water is pretreated to make it acceptable for human consumption; however, compared to reagent-grade water, tap water remains a veritable soup of contaminants. The purity of reagent-grade water will depend heavily on the efficiency of subsequent stages of purification. Salts are the easy part. Purification systems must also cope with microorganisms and an inconceivable array of organics, produced naturally and as the result of human activity (including pharmaceuticals). The challenge for laboratories is to select effective systems, or components, and apply them to their best advantage with effective monitoring. But standards impact the choices laboratories make, directly and indirectly. Standards affect the availability and market share of commercial water purification systems and components. Standards influence what laboratories are told and learn about water purification technologies. And standards

^b Quoted in Arago, *Eulogy of Galileo* (1874)

^c Process parameters relate to the process (system) used to produce reagent-grade water.

^d Product parameters relate to the reagent-grade water product.

dictate how laboratories report reagent-grade water purity. So how pure is reagent-grade water? The honest answer is that poor standards have made it impossible to even guess. All that can be said with certainty is that reagent-grade water is not nearly as pure as it could be with greater awareness and very little effort.

The Emperor's New Robe – D1193 in perspective — D1193 has been ASTM International's only reagent water specification for nearly 50 years, and it is the most widely cited specification for reagent water in the world.^e Informal surveys suggest that a majority of scientists believe Type I water, a term coined by D1193, to be the accepted standard of water purity for nearly every discipline. Most laboratory water purification systems have probably been purchased on the basis of Type I claims. However, as this critique will make clear, there is a gulf between the perception of D1193 and its reality. In fact, D1193 brings to mind Hans Christian Andersen's allegorical tale, *The Emperor's New Robe*.^f

D1193 – what you see is *not* what you get — It appears that almost no one has seen, let alone read, the actual text of D1193. What nearly everyone has seen are liberally reformatted copies of a table in D1193, which have been widely disseminated in catalogues and brochures. This table lists limits for certain physical and chemical parameters of four (Type I-IV) reagent waters; however, the significance of this table is unclear. The table could be left out of D1193 and never missed – it has no title, figure number, or explanatory legend, and the text of D1193 makes no reference to it. Furthermore, the text of D1193 clearly specifies the four types of reagent water according to the processes by which they are produced and states, "The method of preparation of the various grades of reagent water determines the limits of impurities . . ." (Section 1.2). In other words, the text amounts to a stand-alone *process specification* that does not require the table.

How is the D1193 table perceived? — The preponderance of evidence indicates that the overwhelming majority of users and manufacturers of Type I water systems clearly share a common view of what the D1193 table *does not represent* – it is not a required D1193 *product specification* for reagent water. The Type I column in the table lists limits for five parameters; electrical resistivity/conductivity, total organic carbon, sodium, chloride, and silica. Yet, manufacturers of laboratory-scale Type I systems do not attempt to equip their systems with means to measure the five parameters. But both manufacturers and users of Type I systems are comfortable describing the water produced by these systems as "Type I water," even though the five parameters are essentially never measured. It is widely accepted that the cost of the test instruments, materials, and support staff, required to make the five measurements accurately and frequently enough to maintain control within the D1193 table limits, would be prohibitive for all but very large-scale installations – in a word, *impractical*.^g The test instruments alone are likely to cost more than \$35,000.

^e According to ASTM research, D1193 is accepted by the American Institute of Aeronautics and Astronautics (AIAA), the Department of Defense (DOD), the International Organization for Standardization (ISO) and the National Aeronautics and Space Administration (NASA).

^f Hans Christian Andersen, *The Emperor's New Clothes*, 1837 <http://hca.gilead.org.il/emperor.html>

^g The D1193 Task Group Chairman confirmed that D1193 does not require validation of product water in response to a direct question by Dr. Gibbs at the D1193 Task Group Meeting in Louisville, KY (06/13/99) and again at the ASTM International Meetings held in Tucson, AZ (06/27/00).

The views of users and manufacturers diverge sharply with regard to what the table *does represent*. Most users appear to view the table as a sort of *suggested typical analysis* of the product water. Whereas, manufacturers view it as a *sales tool*, which some embrace and others avoid for reasons relating to potential liability.

What manufacturers know — Manufacturers know that the D1193 table cannot possibly be a *real* product specification, because it does not come with any instructions. No one would consider measuring each of the five Type I parameters continuously for laboratory-scale systems, and batch testing is not an option because the resistivity limit is so high. Therefore, the table must be subject to interpretation; however, in the absence of any instructions, one interpretation is as valid as the next. Type I water could be almost anything – water that meets some or all of the limits, part or all of the time, at the same or different points in the production process. And as one considers the details associated with measuring the parameters, one quickly appreciates just how enigmatic the D1193 table is. Resistivity serves as an excellent example.

The D1193 table limit of $\geq 18.0 \text{ M}\Omega\text{-cm}$ is so close to the $\sim 18.3 \text{ M}\Omega\text{-cm}$ resistivity for absolutely pure water that even a sophisticated resistivity instrument (cell, cable, meter, etc.), rigorously cleaned and calibrated, can give “bogus” results.^{h, i} Would laboratories pay for sophisticated resistivity instruments? Could laboratories justify the time required to perform what would amount to a research project in order to make the measurements? And what would be the point of equipping systems with extremely accurate resistivity instruments. After all, it is an accepted fact that organics and microorganisms, which are undetected or poorly detected by resistivity testing, are the Achilles’ heel of Type I systems.^{j, k} Manufacturers of laboratory-scale, Type I systems are unlikely to interpret the D1193 table in the manner that would handicap sales, so one should take the 18.0 $\text{M}\Omega\text{-cm}$ limit with a grain of salt.

But the issue of resistivity instrument accuracy is of far less significance than the issue of where in a Type I system resistivity should be measured. If perfectly pure water is exposed to perfectly clean air, the resistivity of the water will drop very rapidly to less than 1 $\text{M}\Omega\text{-cm}$, because CO_2 will dissolve to form ions. Therefore, if Type I water were required to have a resistivity of $\geq 18.0 \text{ M}\Omega\text{-cm}$ at the time of use, there could be no practical way to actually use it. In fact, it does not even make technical sense to measure resistivity at the output of a Type I system. Type I systems produce high-resistivity water by means of ion exchange and the most effective point at which to measure resistivity is immediately following this stage of purification. Measuring resistivity at this point will

^h Carr G. *Instruments – Evaluating the limitation of current technologies and improvements in future analytical methods*. *Ultrapure Water* 2001; 18(2):24-32.

ⁱ Resistivities are referenced to 25° C.

^j “When it comes to ultrapure water, what you can't see can often hurt you. Your system's resistivity meter may indicate that your product water is at the theoretically “pure” limit of 18.2 megohm-cm, but don't be fooled! In spite of high resistivity levels, your water can still contain high concentrations of organic contaminants which cannot be detected by resistivity measurements alone.” – *Waterline*, Millipore Corporation 1995 [Millipore is a well-known manufacturer of Type-I systems.]

^k “To illustrate the problem of organic compounds, Millipore Corp's Jon DiVincenzo explains that Simply dissolving sugar in 18 megohm-cm water can raise the TOC values to 1000 parts per billion (ppb) without reducing the specific resistance of the water.” – Brush M., *Water, water, everywhere*. *The Scientist* 1998; 12(12):18-20

give the highest possible values and provide the earliest indication when the stage(s) responsible for removing ions begin to fail. Stages of purification that follow ion exchange are likely to decrease the resistivity of the water stream and make the measurement less sensitive as an indicator of system failure. Filtration, UV or ozone treatment, recirculating pumps, etc. reduce the resistivity of high-resistivity water. Even a short length of flexible plastic tubing leading to a dispenser nozzle is likely to be permeable to CO₂ (fluorocarbons are especially permeable). Of course, making the decision to measure resistivity somewhere upstream from the output of a system means the resistivity of the water actually delivered may be many millions of ohms-cm lower than the displayed value.

Because manufacturers of Type I systems are certain to pay close attention to standards that could potentially impact the sale and use of their products, they must also know that the D1193 table is essentially arbitrary: 1) What would be the source of hard data, because users have not been making useful measurements; 2) The table is open to hopelessly broad interpretation; and 3) The parameters and limits for Type I water (as well as Types II-IV) have changed repeatedly, making it impossible to keep score. In March of 2002, ASTM International reduced the TOC limit for Type I water from 100 ppb to 50 ppb, without bothering to change the version date of D1193 from 1999, and made the change retroactive to October of 2001. Apparently, ASTM believes that changes made to the table do not impact the technical content of D1193 (see the *Critique* heading below – section 1.1).

What is Type I water? — There is no basis for believing that the Type I process, as defined by D1193, will necessarily produce water that meets the Type I limits in the D1193 table (regardless of how the limits may be interpreted). According to D1193, Type I Water *shall* be prepared using cold-technology purification components (e.g., mixed-bed ion exchange resins, 0.2 micron membrane filters; etc.) – components that are certain to exhaust, wear out, and contribute contaminants to the *purified* water. Microorganisms are known to flourish in exchange and sorption beds, where they produce a mind-boggling variety of byproducts that enter the water stream. And, as sorption and exchange beds exhaust, they do not simply become slightly less efficient – they release concentrated pulses of the weakly bound contaminants they accumulated from the time they were first placed in service. The purity of water produced by Type I systems will be unpredictable, unless the systems are extensively monitored and carefully maintained ; however, D1193 does not include requirements or guidance for monitoring or maintenance.

How could D1193 happen? — The analogy between *The Emperor's New Robe* and D1193 is apt. In Hans Christian Anderson's tale, the swindlers took advantage of the emperor's power and hubris. They let it be known that the robe they pretended to weave from the emperor's real gold and silver would be invisible to all those who were crooked or unpardonably stupid. Anyone who spoke out was the subject of ridicule, or worse. ASTM International is a powerful organization that has 32,000 members, offers 10,000 standards, and is accredited by the American National Standards Institute (ANSI) as a Standards Developer Organization. ASTM International is also a supremely self-confident organization.

“ASTM International's Mission Statement: 1) To be the foremost developer and provider of voluntary consensus standards, related technical information, and services having internationally recognized quality and applicability that promote public health and safety, and the overall quality of life. . . .” – James A. Thompson, President of ASTM International, *ASTM International: The Global Vision* ASTM Standardization News, July 2002

"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." – Morris (Mo) Brooke, ASTM International's general counsel, *In Praise of the Fat Lady ASTM Standardization News*, June 2000

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.† ñ *ASTM International D19 (Water) Committee Chairperson writing to the ASTM International Committee On Technical Committee Operations, August 28, 2001*

"*Purity of Water* – Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type _ of Specification D1193." – Form and Style for ASTM Standards (for years, including the most current edition, issued in March, 2001)

And those who have challenged D1193 over the years may not have been publically branded as crooks or unpardonably stupid, but it would be safe to say that they were most definitely disparaged.

Pssst, the emperor is naked — In the *Emperor's New Robe*, the deception unraveled when an innocent child spoke out. Early in 2001, ASTM International submitted D1193 for approval as an ANSI standard. ANSI has over 15,000 standards and lists 1000+ companies, 30+ government agencies, and 270+ other organizations as members. ANSI approval would have doubled or tripled the consensus behind D1193, making it even more difficult to challenge and greatly expanding its sphere of influence. But ANSI received numerous objections, many citing a previous version of this Critique, and D1193 was not approved. The effect has been to legitimize concerns about D1193. Now, it is acceptable to speak out.

A Fresh Approach Is Required

D1193 is beyond salvage — ASTM International has argued that D1193 cannot be withdrawn because its four grades of water, especially Type I, are so thoroughly woven into the argot of water purification and the fabric of a broad range of standards, contracts, and regulations.^{l, m} Though the significance of this argument is deeply disturbing, the logic is compelling. And the same argument

^l According to ASTM International research, D1193 is cited in approximately 1000 ASTM International standards.

^m "ASTM Standards are used by thousands of individuals, companies, and agencies. Purchasers and sellers incorporate standards into contracts; scientists and engineers use them in laboratories; architects and designers use them in plans; government agencies reference them in codes, regulations, and laws; and many others refer to standards for guidance." – ASTM Annual Book of Standards (for years, including the most current edition, Volume 11.01, 2002)

applies to correcting D1193. The problems with D1193 are so systemic that making meaningful corrections would effectively result in a new standard, which could not be published under the same title or standard number without causing utter chaos. Few of the myriad references to D1193 include mention of the version, probably because ASTM International has discouraged doing so.ⁿ The only solution to this dilemma is to declare D1193 obsolete, leave it on the books for continuity, and start fresh.

The objective — The objective is to provide standards that will permit laboratories to communicate, document, or specify the quality of reagent-grade water in an orderly, practical, and effective fashion. Standards should derive their validity and value from sound, scientific reason, hard evidence, and widespread voluntary approval, not from authoritarian fiat. The language of the standards must be unambiguous. And the great majority of laboratories, regardless of specialty, must be able to perform, or obtain, the testing required to use the standards, without incurring costs that are perceived to be excessive in relation to benefits. In other words, the standards must provide practical benefits. The importance of voluntary adherence cannot be overemphasized; only when a standard is widely used and data honestly gathered and reported will it yield a maximum benefit.

What should be specified? — Reagent-grade water specifications must use *product* and *process* parameters, because there is so much practical synergy in the combination. The spectrum of contaminants in reagent-grade water depends to a large extent on the process used to purify it. And measuring a limited number of product water parameters will serve to control the effectiveness of the process, while providing hard evidence for the actual purity of the water. Excluding process parameters would ignore the most easily obtained information about the purity of the product water and require the measurement of many more product parameters in order to achieve the same degree of control. However, insofar as possible, only the global parameters of purification systems should be specified in order to avoid a geometric increase in detail and the potential for meddling by special interests. Product parameters should also be global, unless a specification is intended for a highly specialized purpose. The use of global product parameters will achieve the greatest control for the lowest cost and with a minimum of record keeping.

A range of limits should be offered for each product parameter. As limits for product parameters become more difficult to achieve, the cost associated with measuring and achieving them increases rapidly. Providing a range of limits for product parameters permits laboratories to select the parameters and limits for the parameters that represent the most practical, yet effective, specification for a given application. Providing ranges for limits also reduces any advantage that special interests might gain from influencing the selection of limits. Organizing the limits and parameters into a matrix would make it possible to describe each possible specification as a short, readable, alphanumeric string (e.g., R10-TOC020-LAL-005), so there would be no confusion. This approach would also permit the future extension and maintenance of standards, without making the experience gained from using an older version obsolete or irrelevant.

Should a specification include application notes? — A specification for reagent-grade water must include application notes to assure that its purpose is clearly understood and it is properly applied. The notes should avoid prescribing methods, because there are often many acceptable

ⁿ "Do not include the year of issue when designating referenced documents unless there is a technical reason for requiring a particular revision." – Form and Style For ASTM Standards (for years, including the most current edition, issued in September, 2002, Section A6.3)

ways to achieve a given end and the technologies for purifying water and measuring parameters are evolving very rapidly. However, it can be very useful to provide warnings of known hazards. The value of a road sign that warns of a bump in the road is not diminished because the sign does not describe the many possible ways to cope with the bump, or because drivers may choose from multiple routes.

Why The Critique Is Detailed

The editors and scientific experts, who have contributed to writing this Critique, accepted the challenge to be thorough and detailed for three reasons:

- 1) D1193 is essentially a legal document and an incorrectly used word, or what might seem like an insignificant editorial error, can create large loopholes and great confusion;
- 2) In order to avoid the possibility that D1193 will be reapproved with only minor changes, it is important to leave no doubt about the depth and breadth of its deficiencies; and
- 3) Laboratories and manufacturers, who are defending themselves in regulatory or contract disputes, want as much detail as possible.

Critique

Important: The published text of D1193 is shown in black, with the exception of the five key sections that are all there really is to the "specification", which are shown in red. Note how little red text there is and watch for the astonishing paragraph (6.2) near the end. Invalid sections of the table are shown in purple. Comments and corrections are contained within square brackets, [], and shown in blue.

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Beginning of D1193 . . .

ASTM Designation: D1193 - 99^{e1}
Federal Test Method Standard No. 7916

Standard Specification for Reagent Water¹

This standard is issued under the fixed designation D1193; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

^{e1} Note – An editorial change was made to 1.1 in October 2001

1. Scope

1.1 This specification covers requirements for water suitable for use in methods of chemical analysis and physical testing. *[Define "requirements", "suitable", "chemical analysis" and "physical testing". Does D1193 apply only to methods of chemical analysis and physical testing and not to all the other many uses for reagent water (e.g., synthesis, biological media, etc.) Four grades are specified: [It appears that the following table is not part of the specifications (see below.)]*

¹ This specification is under the jurisdiction of ASTM Committee D-19 on Water and is the responsibility of Subcommittee D19.02 on General Specifications, Technical Resources, and Statistical Methods.

Current edition approved Feb 10, 1999. Published March 1999. Originally issued as D1193 - 51 T. Last previous edition D1193 - 91.

Limit	Type I	Type II	Type III	Type IV
Electrical conductivity, max, $\mu\text{S}/\text{cm}$ at 25°C [Note: The meters used on most Type I systems are not capable of sufficient accuracy to determine that water produced by the systems are within the 0.056 $\mu\text{S}/\text{cm}$ limit.]	0.056 [Should be 0.0555. Rounding to 0.056 causes a discrepancy – 17.8 M Ω -cm.]	1.0	0.25	5.0
Electrical Resistivity, min, M Ω -cm at 25°C [Note: The meters used on most Type I systems are not capable of sufficient accuracy to determine that water produced by the systems are within the 18 M Ω -cm limit.]	18.0 [D5391 includes no precision and bias data and few, if any, instruments may be capable of this measurement on a routine basis]	1.0	4.0	0.2
pH at 298 K (25°C) [Unless D5128 is added to the list of methods, Sections 2 & 7, the conductivity of Type IV water must be exactly 5.0 $\mu\text{S}/\text{cm}$, the lower limit of D1293 and the upper limit for this table.]	Δ	Δ	Δ	5.0 to 8.0
Total organic carbon (TOC), max, $\mu\text{g}/\text{L}$	50 [Note: This value was decreased in March of 2002 from “100” to “50”, as an “editorial” change.]	50	200	n/a
Sodium, max, $\mu\text{g}/\text{L}$	1	5	10	50 [The stated upper limit of D6071 is 40]
Chlorides [Chloride – “chlorides” suggests molecular species], max, $\mu\text{g}/\text{L}$	1	5	10	50 [The stated upper limit of D5542 is 24]
Total silica, max, $\mu\text{g}/\text{L}$	3 [D 4517 has not been tested below 35, where the bias was $\pm 20\%$. The non-ASTM reference has an SD of 6% at 25 and the limit of detection is 2.5.]	3 [See Type I column]	500	n/a

^A The measurement of pH in Type I, II, and III reagent waters has been eliminated from this specification because these grades of water do not contain constituents in sufficient quantity to significantly alter the pH.

^B EU = Endotoxin Units.

[One can easily conclude, as most manufacturers and users of water purification systems have, that D1193 is a *process* specification that does not require the measurement of the parameters in the table:

- The paragraph immediately following the table, Paragraph 1.2, states, “The method of preparation of the various grades of reagent water determines the limits of impurities . . .” – there is no mention of the table.
- Nowhere in D1193 is it unequivocally stated that the table is part of the D1193 reagent water specifications;

- c) The parameters and limits of the D1193 table appear to be arbitrary and impractical for the great majority of applications that require reagent water.
- d) If the table were part of the D1193 specifications for reagent water, D1193 would be expected to indicate how frequently the parameters must be measured and at exactly what points in purification systems the measurements must be made or the samples for measurements taken – it does not; and
- e) There is no evidence that most laboratories, even those performing validation of ASTM standards that require the use of Type 1 water, are capable of measuring the parameters in the D1193 table accurately enough to be certain their water is within the table limits at all times.

The resistivity of water produced by the Type I process is shown as both 18.0 or 17.8 M Ω -cm (the resistivity calculated by converting 0.056 μ S/cm); it cannot be both? The small difference is significant, because the difficulty of producing high-resistivity water and measuring the resistivity becomes much more difficult as one approaches the theoretical limit of \sim 18.3 M Ω -cm (referenced to 25° C). Even very costly meters, which have been carefully cleaned and calibrated, may be unable to distinguish between water samples that have resistivities of 18 and 17.8 M Ω -cm (referenced to 25° C) over the range of temperatures and flows that are likely to be encountered in typical laboratory water systems.

The manner in which the TOC value for Type I water has been chosen demonstrates the essentially arbitrary, political nature of the table. About 1996, interests with a stake in selling Type I water systems began lobbying to reduce the TOC value for Type I water substantially below the value for Type II water. The version of D1193 that was in print at that time had been approved in 1991 and the TOC values for Type I and Type II waters were published as 100 ppb and 50 ppb respectively. There are many reasons why deionized, filtered, Type I water would be expected to contain substantially more organic and microbial contamination than distilled, Type II water, and some of these reasons are described in the body of D1193 (see Section 5). Yet, the proponents for reducing the TOC value for Type I water prevailed in *unofficial, non-binding* D1193 Task Group meetings, without providing any supporting data. In 1997 the Task Group recommended to the D19.02 Subcommittee that the TOC value for Type I water be changed to 25 ppb. And the Subcommittee signed off on it and submitted this value to the full committee in the form of a letter ballot – without any supporting evidence. The ballot failed, in part because a handful of D19 Committee members objected to the low TOC value. So the chairman of the Task Group, Verity Smith, offered a purely political compromise, which he described in his March 15, 1998 cover letter for the next ballot of D1193.

“The new submittal includes the reduction in the TOC maximum from 100 to 50 micrograms per liter. The previous ballot reduced this content to 25 which was interpreted by some to be 2.5 micrograms per liter. There were several negatives based upon the 2.5 level and a couple of negatives based on the fact that Type I reagent grade water generally speaking does not have a requirement for TOC even as low as 100 micrograms per liter. For this reason, the submittal is based upon a reduction from 100 to 50 with the hope that this will satisfy all of those concerned for the present time.”

The change that Mr. Smith recommended be made to the table was approved, without any concessions to the conflicting text of D1193. The opposition had been asked not to oppose the change, in exchange for placing the development of a new, more effective standard on a fast track. Then, the version of the standard that was published did not include the approved change and

nobody noticed – for years. Early in 2001, the error was discovered and brought to the attention of ASTM's Committee on Standards by the Chairman of D19.02.03.08, Erich Gibbs, Ph.D. At that time, Dr. Gibbs underscored the fact that there was no scientific evidence for either the 100 ppb value or the 50 ppb value and that, in his opinion, the issue should be debated and brought to the attention of the full D19 Committee. At the D19 Meetings in January of 2002, the Chairman of D19, Natalie Perkins, ruled that a motion dealing with the subject was out of order and referred the motion to ASTM Headquarters for review. Then, in March of 2002, the TOC value for Type I water was reduced from 100 to 50, effective retroactively to October of 2001 – without bothering to change the version date of D1193 from 1999, without scientific evidence for the change, without any public discussion, and without the advice or consent of D19 Committee members. Apparently, ASTM believes that changes made to the table do not impact the technical content of D1193.

10.6.4 *Editorial Changes*—Editorial changes may be made at any time without a requirement for letter ballot.

10.6.4.1 Editorial changes are of two types: (1) those which introduce no change in technical content, but correct typographical errors, modify editorial style, change nontechnical information, or reduce ambiguity, and (2) those which correct typographical errors in substance (essential information that could be misused). In the latter case, the year designation of the standard is changed. — Regulations Governing ASTM Technical Committees, May 2001

The reason for including sodium and chloride in the table cannot be that these ions are of greater importance than many other ions that are likely to cause interferences with specific applications. Before a TOC parameter was included in the D1193 table, sodium and chloride may have been included for the purpose of interpreting resistivity measurements by providing a crude indication of the total concentration of dissolved solids. Thus, water with a resistivity of 1 M Ω -cm, might contain dissolved carbon dioxide (770 ppb CO₂), as the result of having been in contact with air, or it might contain solids (439 ppb of NaCl). However, a TOC measurement involves actually measuring the carbon dioxide concentration, so there does not appear to be any rationale for including sodium and chloride in the table at this time.

Silica does not distill, but it is weakly dissociated in water and is among the first contaminants to be displaced from exhausting mixed-bed ion exchange columns. Most laboratories that are concerned about silica (and/or boron) install systems that combine resistivity testing with passive redundant mixed-bed design (i.e., several identical beds connected in series) as a practical way of monitoring and controlling bed exhaustion – they do not measure silica directly. Silica is of particular interest to laboratories that are involved with applications that are silica specific (e.g., diatom research, etc.) or silica sensitive (e.g., high-pressure steam plants, electronics fabrication plants, etc.); however, these laboratories represent a very small cohort of all laboratories. There appears to be no evidence that traces of silica cause non-specific interference for the great majority of reagent water applications. And, there are many other possible contaminants, which are not included in the D1193 table, that are far more likely to cause non-specific interferences and are of more interest to the majority of laboratories.

a) The issue of silica was raised at the May 13, 1999 ASTM D19.02 Meeting in Louisville, KY. Members were asked for references supporting the contention that silica caused non-specific interferences in laboratory experiments/tests. The challenge was repeated at the January 17, 2000 ASTM Meeting in Cocoa Beach, FL. No references have been provided.

b) At the May 13, 1999 ASTM D19.02 Meeting, Louisville, KY, Erich L. Gibbs, Ph.D. reported that he had performed an extensive Dialog search (over 20,000 journals from 1989>1999) to locate any articles dealing with silica contamination as a source of interference in laboratory tests/experiments. None were found.

c) At the May 13, 1999 ASTM D19.02 Meeting, Louisville, KY, Erich L. Gibbs, Ph.D. announced that his company had conducted an e-mail survey of over 23,000 bioscientists around the world in search of any evidence that silica contamination was a source of non-specific interference in laboratory applications. The only reports were for specific interferences (i.e., the growth of diatoms etc.).

d) On March 7, 1996 Dr. David Jeffers, Chairholder of the National Committee for Laboratory Standards reported to the Working Group on Reagent Water that an NCCLS survey of literature was unable to find references to support the statement, "Silica adversely affects most enzyme determinations, and trace metal and electrolyte analyses" (Section 9.5 C3-A2 Preparation and Testing of Reagent Water in the Clinical Laboratory: Approved Guideline – Second Edition, 1991) Therefore, the wording was changed to, "Silicates or colloidal silica can interfere with certain assays [which are unspecified]." (Section 6.1.4 of C3-A3 Preparation and Testing of Reagent Water in the Clinical Laboratory: Approved Guideline – Third Edition, 1997).

Proponents of D1193 argue passionately that the measurement of silica is critically important to D1193 and should be an absolute prerequisite for the approval any alternative ASTM reagent water specification. They do not appear to appear to appreciate the fact that the vast majority of laboratories that claim to be using Type I - III reagent water do not measure silica or see any need to do so.]

Microbiological contamination - When bacterial [bacteria?] levels need to be controlled, [What exactly is meant by "controlled"] reagent grade types should be further classified as follows: [According to the Form and Style for ASTM Standards manual, the definition of "classification" is as follows: "classification, *n*—a systematic arrangement or division of materials, products, systems, or services into groups based on similar characteristics such as origin, composition, properties, or use." D1193 is a *process specification*; how does the user *control* bacteria levels by further *classifying* a specified process with limits on bacteria counts and endotoxins? The only way to *control* bacteria levels is to alter the process.]

The limits are as follows:

	Type A	Type B	Type C
Maximum heterotrophic bacteria count [What about autotrophs or oligotrophs, the later being the most likely to be present in purified water? Method F1094 is likely to understate the number of viable microorganisms by 100-1000 times.]	10/1000 ml [10/L]	10/100 ml [100/L]	100/10 ml [10K/L]
Endotoxin, EU/ml ^B	<0.03	[<]0.25	n/a

^B EU = Endotoxin Units.

["Bacteria count" must be defined. Plate counts only detect those organisms capable of growth on the nutrient selected and under the physical conditions chosen for incubation. The referenced method, F1094, typically underestimates the total numbers of viable organisms in the sample by 100- to 1000-fold. (McAlister MB, Kulakov LA, Larkin MJ, Ogden KL. *Microbials - Analysis of bacterial contamination in different sections of a high-purity water system. Ultrapure Water 2001; 18(1):18-26.*) Even if a plate count were accurate, it would not provide a useful indication of the growth a microorganisms in a water purification system, because the great majority of microorganisms will live in biofilms, attached to surfaces. And it is a relatively simple matter to kill (ozone, UV, etc.) or filter free-floating microorganisms. Most of the microbiological contamination in purified water will be cellular debris, not viable cells.

The Type A endotoxin level of <0.03 could be consistent with 300-3000 gram negative bacteria, or their cell walls, per ml. This might be construed to be an admission by ASTM that most of the microbiological contamination in reagent water will be byproducts, not viable cells. In any case, such high levels of endotoxin are inconsistent with high-purity water. A well designed water purification system should produce water with endotoxin levels of <0.001 EU/ml. Perhaps the 0.03 value was the lowest level of EU detection available (the gel-clot method) when this section of D1193 was last reviewed; however, the lower limit for the detection of endotoxins was 0.001 EU/ml in 1999, when D1193 was reappraised. An endotoxin level of 0.25 EU/ml matches the USP 24 NF-19 requirement for *Water for Injection*, which is not intended to be used as reagent water, and would be consistent with 2500-25,000 gram negative bacteria, or their cell walls, per ml.

Fungi and algae are likely to inhabit water purification systems, and the cell walls of many of these contain (1→3)-β-D-Glucan, which can produce a falsely elevated LAL test.]

1.2 The method of preparation of the various grades of reagent water determines the limits of impurities [ASTM is saying that the purity of the four grades of water is determined by the methods of preparation. This statement makes it possible for users and manufacturers of water purification systems to meet D1193 specifications for reagent water without measuring any parameters of the product water.] **and shall be as follows:**

1.2.1 Type I grade of reagent water shall be prepared by distillation [There are many different types of distillation (e.g., fractional, vapor compression, continuous, etc.) **or other equal process** [Define "equal process". As D1193 and other ASTM Standards emphasize, distillation is very different from other processes of water purification. Since distillation involves phase changes with at least two net changes of phase, one might conclude that the only other "equal process" might involve freezing and thawing. It is certainly hard to imagine any cold-technology, such as filtration, ion exchange, or sorption, as being even remotely equivalent to

distillation.], followed by polishing [The term, "polishing", is a colloquialism that lacks a clear definition. The use of a colloquialism seems inappropriate in D1193. Treating distilled (or other equal process) water with ion exchange may trade a substantial amount of organic and microbiological contamination for a trace amount of inorganic salts, a fact that is recognized in paragraph 5.1.] with a mixed bed of ion exchange materials [The type and quality of the resins is critical and should be defined. A mixed bed containing two, or more, different anion resins and no cation resins would not be effective. A poor balance of anion and cation resins would give a poor result. Resins that have been used for heavy metal recovery would not be acceptable, etc.] and a 0.2- μm membrane filter.

[Why specify a membrane filter, since other types of filters will also filter to 0.2- μm and lower? Would a finer filter be unacceptable? Is it to be understood that Type I water systems cannot utilize any elements besides the combination of an ion exchange bed and membrane filter, both of which must follow a pretreatment by "distillation or equal process"? In that case recirculation and the use of UV or ozone, which are widely used in systems that claim to meet Type I specifications, would be precluded?] Feedwater [Feed Water sp?] to the final polishing step must have a maximum conductivity of 20 $\mu\text{S}/\text{cm}$ at 298K [What is the point of referencing Kelvin degrees throughout D1193?] (25°C). [What is the relevancy of this sentence, since the definition of "distillation or equal process" is so vague? The only apparent purpose of the sentence is to limit the salt content of the feed water for the final mixed bed of exchange resin. However, the salt content would not be expected to affect the quality of the final product water, provided the size and flow for the final mixed bed of exchange resin have been properly optimized.]

1.2.2 Type II grade of reagent water shall be prepared by distillation using a still designed to produce a distillate having a conductivity of less than 1.0 $\mu\text{S}/\text{cm}$ at 298 K (25°C). [Why not say, "... shall have a conductivity of less than 1.0 $\mu\text{S}/\text{cm}$ (referenced to 25°C) and be prepared by distillation." The existing sentence is very confusing; a) Because the operating conditions are not described, a still could meet this requirement even if it were *designed* to produce distillate having a conductivity of less than 1.0 $\mu\text{S}/\text{cm}$ at 298 K (25°C) only while being fed with perfectly pure water and operating under nitrogen, and b) The sentence would permit a user to *operate* a still *designed* to produce distillate having a conductivity of less than 1.0 $\mu\text{S}/\text{cm}$ at 298 K (25°C) under conditions that produce water with a much higher conductivity?] Ion exchange, distillation, or reverse osmosis and organic adsorption [It is virtually impossible to distinguish the relative components of adsorption and absorption, so the term "sorption" is preferred. Why include electrodeionization (EDI) for Type III and IV water and not here?] may be required prior to distillation if the purity [What purity? What is the definition of "purity?"] cannot be attained by single distillation [What is the definition of "single distillation" – the use of a single still (vapor compression?, fractional?, continuous?, . . .), the use of a still that has a single plate of distillation, . . . ?]

Note - Because distillation is a process commonly relied upon ["Upon should be replaced with the word, "on". Does the use of the word, "relied", imply that users can actually rely on a still to produce high-purity water without any performance testing?] to produce high purity water [Define "high-purity water"], the levels specified for Type II reagent water [What is the meaning of "levels" in this context? Should "levels" be replaced with "limits"?] were selected to represent the minimum quality of water that a distillation process should produce. [This is the closest that D1193 comes to referring to the table; however, the use of the word, "should" supports the general impression that the limits in the table are not a required part of the D1193 specification. ASTM seems to be stating that it has intentionally made distillation appear to be less effective than it actually is, and the justification is nonsensical, "... distillation is a process commonly relied upon to produce high-purity water ..." Well-designed stills can easily produce water with

a resistivity above 1 MΩ-cm and it is unlikely that most Type I systems routinely produce 18 MΩ-cm water.]

1.2.3 Type III grade of reagent water shall be prepared by distillation, ion exchange, continuous electrodeionization reverse osmosis [“Continuous electrodeionization” (CEDI) is a term apparently coined by, and used almost exclusively by, the Vivendi/USFilter Company, which has been instrumental in supporting D1193 – “electrodeionization” (EDI) would be the preferred term. Why is EDI mentioned under Type III and Type IV only? If EDI is not *ion exchange*, then does it not stand to reason that “ion exchange” and “electrodeionization” should be defined and the language throughout this Standard changed to reflect the differences between the two? The phrase, “continuous electrodeionization reverse osmosis” is nonsensical, perhaps it was meant to read, “continuous electrodeionization, and reverse osmosis.”], or a combination thereof, followed by polishing with a 0.45-μm membrane filter. [It would appear that the meaning of the term, “polishing”, is not the same here as it was in 1.2.1. Why is a membrane filter required, why not any type of 0.45 μm? Would a finer filter be unacceptable?]

1.2.4 Type IV grade of reagent water may [The use the word, “may”, here amounts to saying that the user may produce Type IV water any way he chooses? Thus, D1193 does not even really include a Type IV process specification.] be prepared by distillation, ion exchange, continuous electrodeionization reverse osmosis [“Continuous electrodeionization” (CEDI) is a term apparently coined by, and used almost exclusively by, the Vivendi/USFilter Company, which has been instrumental in supporting D1193 – “electrodeionization” (EDI) would be the preferred term. Why is EDI mentioned under Type III and Type IV only? If EDI is not *ion exchange*, then does it not stand to reason that “ion exchange” and “electrodeionization” should be defined and the language throughout this Standard changed to reflect the differences between the two? The phrase, “continuous electrodeionization reverse osmosis” is nonsensical, perhaps it was meant to read, “continuous electrodeionization, and reverse osmosis.”], electro dialysis, or a combination thereof.

1.3 The choice of one of the various grades may be designated by the method or by the investigator. [What is the purpose of this apparently empty statement? Does it mean that agencies, companies, or organizations cannot designate a grade of water for certain applications? What is meant by “choice” in this context – a method is not going to make a *choice*? What is meant by “the method” – any little change in the way an application is performed can result in a different *method*? “Investigator” of what? – this is the only use of the word, “investigator” in D1193 and its use here is puzzling. Does “Investigator” refer to a laboratory inspector or to a researcher and, if so, why are all the other classes of individuals who work in laboratories excluded? Why permit a *method* or *investigator* to choose only *one* grade of water, perhaps more than one grade would work equally well for a given application.]

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water² [This test method is only to be used between 5 and 0.200 $\mu\text{S}/\text{cm}$ and cannot be used to measure the resistivity levels shown in the table for Type I water.]

D 1129 Terminology Relating to Water²

D 1293 Test Methods for pH of Water² [This test method is not applicable below 5 $\mu\text{S}/\text{cm}$ and therefore cannot be used to measure the pH parameter listed in the table for Type IV water, with the possible exception of the very special case of Type IV water that happens to have a conductivity of exactly 5 $\mu\text{S}/\text{cm}$, not higher or lower.]

D 4453 Practice for Handling of Ultra-Pure Water Samples²

D 4517 Test Method for Low-Level Total Silica in High-Purity Water by Flameless Atomic Absorption Spectroscopy³ [D 4517 has not been tested below 35 $\mu\text{g}/\text{L}$, where the bias was -20%. Furthermore, the non-ASTM method that is referenced in the footnote has an SD of 6% at 25 and the limit of detection is 2.5, which suggests that it will not be useful for determining a level of 3 or lower.]

D 4779 Test Method for Total, Organic, and Inorganic Carbon in High Purity Water by Ultraviolet (UV) or Persulfate Oxidation, and Membrane Conductivity Detection²

[Analyzers that use this method cost tens of thousands of dollars and no method of determining TOC by less costly means is cited.]

[D5128 Standard Test Method for On-Line pH Measurement of Water of Low Conductivity² – this method must be added to this list in order to determine the pH parameter listed in the table for Type IV water.]

D 5391 Standard Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample² [This test method includes no precision and bias data, and there is reason to believe that instruments capable of routinely determining resistivities of 18 $\text{M}\Omega\text{-cm}$ (referenced to 25°C) have yet to be manufactured.⁴]

D 5542 Test Methods for Trace Anions in High Purity Water by Ion Chromatography² [This specified range for chloride in this test method is 1-24 $\mu\text{g}/\text{L}$ and the bias exceeds 10% above 5 $\mu\text{g}/\text{L}$. This method cannot be used to measure the 50 $\mu\text{g}/\text{L}$ value listed in the table for Type IV water.]

D 5997 Test Method for On-Line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate Oxidation and Membrane Conductivity Detection² [Analyzers that use this method cost tens of thousands of dollars and no method of determining TOC by less costly means is cited.]

D 6071 Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atomic Absorption Spectroscopy² [The specified range for this test method is 1-40 $\mu\text{g}/\text{L}$ of sodium and the error rises unacceptably outside of this range. This method cannot be used to measure the 50 $\mu\text{g}/\text{L}$ value listed for Type IV water in Table 1.]

[D 6317 Standard Test Method for Low Level Determination of Total Carbon, Inorganic Carbon and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection² – This method should be added to the list; however, under ideal conditions, the overall percent standard deviation at 50 $\mu\text{g}/\text{L}$ of organic carbon is approximately 10% and at 100 $\mu\text{g}/\text{L}$ of organic carbon it was approximately 7%. It is likely that the errors would have been much greater had real-world samples, involving complex matrices, been measured.]

F 1094 Test Methods for Microbiological Monitoring of Water Used for Processing Electron and Microelectronic Devices by Direct Pressure Tap Sampling Valve and by the Presterilized Plastic Bag Method⁵ [This method is likely to underestimate the number of viable microorganisms present in purified water by 100 -1000 times.]

² Annual Book of ASTM Standards, Vol 11.01.

³ Determination of Trace Silica in Industrial Process Waters by Flameless Atomic Absorptions [Absorption] Spectrometry, Judith Rawa and Earl Henn, Analytical Chemistry, Vol. 51 No3, March 1979.

⁴ Annual Book of ASTM Standards, Vol 11.02.

⁵ Annual Book of ASTM Standards, Vol 10.04.

3. Terminology

3.1 *Definitions* - For definitions used in this specification refer to Terminology D 1129.

4. Significance and Use

4.1 Different analytical methods and industrial processes require water of different purities. [This is an empty statement that is not necessarily true – many different methods and industrial processes require essentially the same purity of water.] Also, different types of contaminants affect these methods and processes differently. [What point is ASTM trying to convey? Why not say that the choice of a grade of water, best suited for a particular method or process, will depend on a balance between the profile of impurities in the water and the cost of producing the water.] This specification is intended to provide the user with a choice of different grades of water having different purities as described in Section 1. It is intended to satisfy the requirements for normal laboratory procedures, but does not necessarily apply to the large-scale production of pure water or for specific applications. [Are users of D1193 to conclude that the specified processes only apply when the product water is used in *normal* applications and when the purification systems are *small-scale*. What is meant by “normal”, “small-scale”, and “apply”? Because D1193 does not require any testing and provides for no enforcement, exactly how does it *apply* to anything?] Different grades of water may be covered by other ASTM specifications or guides, or by the requirements of other standards organizations. [This is a meaningless and unnecessary statement.]

5. Use and Application

[This section includes arguments that contradict Section 1.2. It also includes compelling reasons for requiring the validation of reagent water, so why has ASTM omitted all mention of validation from D1193? The section is more a disjointed collection of loose ends than an explanation of how to use or apply D1193.]

5.1 The method of preparing Type I reagent water may add organic contaminants to the water by contact with the ion-exchange materials. [In fact, it is very likely to do so. It is also likely that a Type I system (as specified by D1193) will fail to remove much of the organic material from the source water, because the phrase “distillation or equal process” in section 1.2.1 is so vague.] It should be noted also that the method [“Method” – perhaps the words, “Type I process” would be more appropriate.] may or may not remove non-ionized dissolved gases. [The Type I process is very unlikely to remove non-ionized dissolved gases effectively, because there is no requirement to use technology that would perform this function.]

5.1.1 The dissolved or particulate organic contamination would ["Would" – the use of this word seems inappropriate.] normally range from 30 to 50 µg/L. [Is this sentence referring to Type I water? In any case, some important punctuation is missing – is the phrase referring to *dissolved contamination or particulate organic contamination* or is it referring to *dissolved, or particulate, organic contamination*? What does the word, "normally", mean in this context? And to what does the 30-50 µg/L concentration refer – TOC, 100° C dry weight of organics, . . . ?] The concentration of nonionized [sic – this word is hyphenated in 5.1.] dissolved gases may exceed 5 mg/L. [What is the purpose of this sentence – of course, the concentration of non-ionized, dissolved gases may exceed 5 mg/L, because there is no requirement to use technology that would remove this type of contamination. Reading between the lines for the intended meaning, it would appear that this paragraph is at complete odds with paragraph 5.1, the table, and the literature. The Type I process does not include limits that would in any way control the amount of organic impurities in the product water!]

5.2 [What types of water does this paragraph apply to – Type I or all types?] The quality [What is the definition of "quality"?] of the effluent water depends upon the type, age, and method of regeneration of the ion exchange materials. ["Effluent" from where – does effluent water refer to water leaving an ion exchange bed or the system product water? Which materials – the final mixed ion exchange bed in Type I, Type III, and Type IV processes or ion exchange materials used in pretreatment stages?] Likewise, ["Likewise" – poor choice of a word, because this sentence describes a very different phenomenon than the previous sentence.] the flow rate through the ion exchange resin bed will change the conductivity of the effluent water. [Perhaps this sentence could be reworded – "Altering the rate of water flow through an ion exchange bed, **above or below** the rate for optimum ion exchange, will impact the ion content of the water exiting the bed."] The instructions of the manufacturer of the resins or the resin cartridge bed should be followed. [What if the instructions are inappropriate? ASTM has no control over the manufacturer's instructions, yet it is recommending that these instructions be followed. This sentence would seem to be at odds with the ASTM definition of a specification.]

5.3 [What is the point of emphasizing the possible water soluble contaminants in membrane filters, since many other components of water purification systems are likely to bleed significant quantities of water soluble organic and inorganic material into the water stream?] The use [Under 1.2.1 and 1.2.3 the use of a membrane filter is required; therefore this sentence should read, "The required use . . ."] of the membrane filter in the preparation of Type I and Type III water may add a small ["Small" – too vague.] amount of organic components [Strained wording – "... of organic material.. "] to the water first produced. ["First produced" – too vague.]

5.3.1 Some [What is meant by "some" – *Some* filters could be intentionally contaminated and contain over 80% by weight of water soluble contaminants.] membrane filters contain as much as 8 mass % [8% by weight?] of soluble components [The use of the word, "components" suggests that some of the filter structure, and integrity, will dissolve. Soluble in what?] resulting from the manufacturing process [And design?].

5.3.2 If the contamination of the water [What water?] by the organic component [What "organic component"?] is of significance to the test [What "test"?], Type II water should be used or the membrane [Section 1.2.2 does not state that a membrane filter cannot be used after distillation to produce Type II water] should be rinsed by discarding the first 10 ml of water produced per square centimetre ["Centimetre" – is this the approved ASTM spelling?] of filter area [What is the point of stating how much water must pass through the membrane, if the rest

of this sentence applies?], and until a test [What “test”?] for the organic components [“Organic components”?] shows them to have been reduced to less than the specified level. [“Specified level” – D1193 does not specify levels (limits?) for the bleed of organic *components* (contaminants?) from membrane filters.]

5.4 [Does the inclusion of Section 5.4, 5.4.1, 5.4.2, and 5.4.3 indicate that Type I, Type III, and Type IV water are likely to contain unacceptable levels of: organics, including surface active organics; pyrogens; silica; and particulates?] Type II grade of reagent water should be sterile and pyrogen-free as produced [Is this statement a requirement or a statement of fact? If it is a statement of fact, it is not true. If it is a requirement, almost no water purification systems will be able to meet it.] and generally may [“Generally may” – what does this mean?] be used whenever freedom [“Freedom” – does this mean zero, none?] from organic or biological contaminants is desirable. [When is it not *desirable* to have a minimum of organic or biological contamination?] However, the method of storage and handling of the water [What water?] may itself [What does “itself” mean in this context – this word is unnecessary and confusing.] result in contamination. [This statement seems pointless – it is possible to contaminate any type of water, even sea water, during storage or handling.]

5.4.1 Type II water should be pyrogen-free [Is this statement a requirement or a statement of fact? If it is a statement of fact, it is not necessarily true.], but must be tested in conformance with the requirements of the current edition of U.S.P. if proof is needed. [What “requirements” – there should be a more specific reference, because USP does not have any requirements for ASTM Type II water.]

5.4.2 The description [What “description” – the totality of D1193 or Section 1.2.2?] of Type II reagent water is intended [What exactly does “intended” mean in this context? This statement suggests that ASTM realizes it may have failed to *characterize* the product of distillation.] to characterize the product of distillation [What is meant by “product of distillation” – Type II water or some other entirely different product of distillation? In any case, this statement is a non sequitur, because nowhere in D1193 is the user precluded from following the distillation step that is described for the Type II process (1.2.2) with additional steps of purification.]

5.4.3 Distilled water [Type II water?] is often [But not always?] specified when freedom from moles [The word, “moles”, is defined as meaning small particles – are large particles and minute particles acceptable?] and silica is required [“Freedom” would suggest total absence; however, D1193 contains no product limits for particulates and the table has the same 3 µg/L silica value for both Type I and Type II water. “Required” – by whom?], or when surface-active organics are proven to be a problem [What type of “problem”? “Proven” how and under what circumstances?], and when alternative processes have been found to be inadequate. [Why would it not be acceptable for a user to specify distillation even if other processes are found to be adequate?]

5.5 Types I, II, and III reagent water should be protected from atmospheric contamination [What is the definition of “atmospheric contamination” in this context? Would it not be more appropriate to use the term, “ambient air”?] and from solution [This is a very strained use of the word, “solution”, and results in permitting contamination by anything insoluble from the container and tubing.] of container and tubing [Is pipe considered to be tubing?] materials. [What about all the other sources of contamination – is there no need to protect Types I, II, and III water from all sources of contamination?]

5.5.1 Extreme [Extreme? – is this meant to imply clean room conditions?] care must be exercised in handling samples [Samples of what – reagent water or samples related to an application?] when making an analysis [“Analysis” – what type of analysis?]. Sample containers and tubing should be made of fluorocarbon, titanium, tantalum, block tin [The inclusion of tin in this list is a throwback to archaic technology. Fluorocarbons are not recommended when samples are obtained for resistivity measurements.], quartz, 18-8 stainless steel, polyethylene, or other material [Materials?] proven to be sufficiently resistant to chemical attack so as not to cause contamination [Materials in this list are not necessarily acceptable for transferring samples of water.] in [“In” – this is an inappropriate use of the word, “in”.] the intended use [There is no point to including a partial list of supposedly acceptable materials, only to go on to say that any acceptable material is *acceptable*?]. Practice D 4453 should be consulted.

5.6 [This statement is misleading and probably should read, “Contaminants in the ambient air, which dissolve to produce ions, will rapidly increase the conductivity of exposed water, so enclosed, in-line probes must be used”] Because atmospheric gases and impurities rapidly recontaminate exposed water, in-line electrodes should be employed for determining the electrical conductivity [And resistivity?] of reagent water Types I, II, and III. [Why are these two unrelated sentences present in the same paragraph?] The measurement of pH in Type I, II, and III reagent waters has been eliminated from this specification because the values would be a function of the instrumentation. [Does this mean that anyone who has reported the pH of water that has a resistivity in excess of 1 MΩ-cm has been in error – this statement is incorrect.]

5.7 Since freedom [Total absence?] from biological contaminants may be important [If it is *significant* (see continuation of this sentence), it is certain to be *important*.] in the test procedure [What “test procedure”?] using any of the reagent waters specified [D1193 has not specified any “waters”, except indirectly through the specified processes.], a classification [“Classification” – this is not the correct word?] of bacterial levels [“Bacterial levels” – what does this mean?] is included [“Included” – included where?] and should be specified [What does it mean to *specify a classification of bacterial levels*?] if it is of significance to the test being performed. [“Bacterial levels” and “biological contaminants” are not synonymous, so one would not expect to insure purity with respect to biological contaminants by controlling bacteria levels.]

5.7.1 To obtain sterile water [In Section 5.4, ASTM states that Type II water should already be sterile.], any of the types of reagent water listed in this section [Section 5.7 and 5.7.1 does not list any type of reagent water. Does this mean that the reader must read back over Section 5 for any references to “types of reagent water”?] may be produced, bottled [Why bottled? Bottled in what? Bottled and sealed?], and heated to 394 K (121°C) for 20 min. This procedure is most easily carried out by autoclaving at 103 kPa (15 psi) for 20 min. [Why are metric and US units being mixed together. If it is appropriate to use “psi”, why not give 394 K in Fahrenheit? If the containers are not autoclaved, they must be heated in a pressurized atmosphere or the containers must be sealed and capable of withstanding in excess of 15 psi at 121° C. Is this paragraph intended to be a process specification for “Sterile Water”, which would be distinct from water produced by the Type I - IV processes?]

5.8 *The following requirement* [“Requirement” – this paragraph is only a recommendation or comment.] *is beyond the requirements* [Very circular wording – “..... requirement is beyond the requirements ...”.] *of the general specifications.* [What “general

specifications"? The use [User?] of reagent grade water should recognize that analytes [The use of the word "analytes" is incorrect. The correct word should be "contaminants"? A contaminant is only an "analyte" when it is known and being measured.] may exist in water that meets the criteria listed in Section 1 ["Criteria listed in Section 1" - what does this mean?], but these analytes may interfere with the use of the water.[Is this statement saying that the user should be aware that contaminants in the water produced by the processes specified in Section 1 may interfere with *the use of the water* or may interfere with *the applications in which the water is used*?] If levels of other analytes ["Analytes"?] are important ["Important" – in what regard?], it is the user's responsibility to specify their limits. [Is this paragraph intended as a disclaimer of Section 1.2? If it is, why not position it under Section 1.2?]

6. Requirements

6.1 Reagent water shall conform to the requirements specified in Section 1. [Does this sentence mean that the rest of D1193 is not part of the specification?]

6.2 [This paragraph is the coup de grace for D1193!] Additional requirements [This could be taken to mean the modification of much of the language in D1193.] concerning specific contaminants [Including those not mentioned in D1193?] or methods of preparation [Including those not mentioned in D1193?] may be included in this specification [". . . **included in this specification** . . ." By not continuing to say, *in order to create a customized version and provided the changes are noted*, ASTM is permitting virtually anyone to modify **this specification**, this actual D1193 specification.] **by mutual** [Two persons or groups?] **agreement** [Written, verbal, or unspoken?] **between the parties concerned**. [Any person with an interest in D1193 is potentially a "concerned party". This paragraph can be interpreted to mean that nobody knows what D1193 may, or may not, be at any given moment in time, because virtually anyone can be changing it at any time. At the very least, this paragraph permits anyone to claim to be meeting D1193, regardless of the process they use to produce their water.]

7. Test Methods

7.1 *Electrical Conductivity and Resistivity* - Refer to Test Methods D 1125 & D 5391 [See Section 2.1.]

7.2 *pH* - Refer to Test Methods D 1293 [& D 5128 (See Section 2.1.)]

7.3 *Silica* - Refer to Test Method D 4517 [and Determination of Trace Silica in Industrial Process Water by Flameless Atomic Absorption Spectrometry³. (See Section 2.1.)]

7.4 *Sodium* - Refer to Test Method D6071 [See Section 2.1.]

7.5 Chlorides⁶ - Refer to Test Method D 5542 [There should not have been a footnote here. It was crossed out in the approved draft and the error appears to have gone unnoticed by those in charge of D1193. (See Section 2.1.)]

7.6 *TOC* - Refer to Test Methods D 4779, D 5997 [&D6317 (See Section 2.1.)]

7.7 *Endotoxins* - Refer to LAL Test Method⁶

7.8 *Microbiological Contamination* - Refer to Test Methods F 1094. [\[See Section 2.1.\]](#)

⁶ Published in U.S. Pharmacopeia by The U.S. Pharmacopeia Convention, Inc.

8. Keywords

8.1 laboratory analysis; reagent; water

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... End of D1193