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STORABILITY INVESTIGATIONS OF WATER

VOLUME II. ANNOTATED BIBLIOGRAPHY

AEROJET LIQUID ROCKET COMPANY

PREPARED FOR
AIR FORCE ROCKET PROPULSION LABORATORY

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STORABILITY INVESTIGATIONS OF WATER
VOLUME II - ANNOTATED BIBLIOGRAPHY

AEROJET LIQUID ROCKET COMPANY
SACRAMENTO, CALIFORNIA

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DECEMBER 1973

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The objective of this program is to gather data that will permit the Air Force to assess the long-term storage characteristics of water with respect to biological growth, galvanic corrosion, and changes in composition of the water, so that the feasibility of long-term storage of water for use as a transpiration coolant can be determined. Eleven metallic materials of construction were included in the program: 301 (cryoformed), 304L, 347, A-286 (aged), and 17-4 PH (aged) stainless steels; 1100-0, 2014-T6, 2219-T87, and		

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6061-T6 aluminums; Inconel 718 (aged); and 6Al-4V titanium (STA). Twelve nonmetallic materials were included in the program: Tetrafluoroethylene (TFE), Fluorinated Ethylene Propylene (FEP), Chlorotrifluoroethylene (CTFE), Polypropylene, Polycarbonate (Lexan), TFE/FEP sprayed laminate, Buna N, Viton, Butyl rubber, Ethylene Propylene rubber, Silicone rubber, and Krytox 240 AC, a fluorinated grease. Four categories of tests were used to obtain the data necessary to assess the long-term storage characteristics of water: (1) Basic Fluid/Material Compatibility Tests, (2) Galvanic Couple Measurements, (3) Biological Growth Tests, and (4) Long-Term Storage Tests. Two types of water were used in the investigation: Oxygen-saturated, deionized, filtered; and oxygen-free, deionized, filtered. An extensive literature survey and critical review were conducted in conjunction with formulating the experimental portion of the program. In addition, a complete analysis was made on a LAR coolant tank in which water had been stored for 2.5 years.

The non-aluminum based alloys were found to be suitable candidates for the long-term storage of water, particularly with regard to formation of particulate matter. The aluminum-based alloys all exhibited minor degrees of corrosion and displayed a tendency to form particulate matter in the water. The galvanic couples containing aluminum specimens exhibited significant attack of the aluminum. Of the synthetic rubber materials, the silicone rubber exhibited the least change in characteristics during storage. The fluorinated plastics and polypropylene maintained their characteristics during the storage tests, while Lexan exhibited a tendency toward embrittlement. Microorganisms were found to have an insignificant effect on material compatibility. However, their presence enhanced the clogging tendencies in filters.

Five-year storage tests have been initiated in 304, A-296, and 17-4 PH stainless steels, Inconel 718, and 6Al-4V titanium (STA) tanks using cold-sterilized oxygen-free and oxygen-saturated water.

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Section 1

UNIFORM CORROSION

1. CORROSION AND FOULING OF ADMIRALTY, ALUMINUM, AND STEEL TUBES IN OPEN RECIRCULATING COOLING WATER SYSTEMS. Joerg Gutzeit. Mater Protect, v 4, no 7, July 1965, p 28-34.

Details are given of a test program conducted to establish economic justification for the proposed substitution of Al or steel for admiralty brass in cooler and condenser tube service. Tube corrosion and fouling were studied under heat transfer conditions over periods of eight months in two water systems. For comparison, change in over-all heat transfer coefficient of two plant-size coolers with admiralty tubes was followed. Results indicate that good heat transfer rates and service lives can be obtained with steel and Al tubes in a well treated water system. Accumulation of corrosion and fouling products increases the pressure drop through steel and Al tubes and can, in some cases, significantly reduce the economic advantages. 11 ref. (M18--53820.)

Section 1

2. (French) CORROSION OF LOW-ALLOY STEELS IN DEMINERALIZED WATER AT 25 C. AND 360 C. INFLUENCE OF OXYGEN. H. Coriou, L. Grall, M. Pelras and A. Perez. Corrosion Anti-Corrosion, v 12, no 8, Dec. 1964, p 358-270.

Feasibility of use of carbon and low-alloy steels in nuclear reactor construction investigated. Comparative corrosion tests made on a mild steel, several low-alloy grades and an 18/10 stainless, in deoxygenated as well as in oxygen-saturated demineralized water. The carbon steel was also tested in the same medium saturated variously with A, N, and O. Results in terms of weight loss and morphology and nature of oxide film. 10 ref.

(M18--21025)

Section 1

3. (French) STUDY OF AQUEOUS CORROSION IN NUCLEAR ENERGY. H. Coriou. Paper no. 135-136, Aug. 1965, 42 p. Centre Belge D'Etude de la Corrosion, 24 Rue des Chevaliers, Brussels 5, Belgium.

Aqueous corrosion in the nuclear field is concerned with chemical treatment of uranium ores, functioning of reactors and treatment of irradiated combustibles. Aqueous corrosion of Al, Mg, Zr and their alloys, stainless steel and Ni alloys are also studied. Poor corrosion behavior is found on the part of pure Al, Mg and Zr in demineralized water and vapor as compared with commercial grades of the same metals. A multiplicity of corrosion phenomena, such as temperature, radiation, pH, heat treatment, surface condition, impurities and solution composition, rather than a single ascertainable factor, seem to be responsible for this result. The beneficial effects of coupling aluminum with stainless steel are shown, as well as the above-mentioned effects for each metal. The results of irradiation and various types of cathodic protection, as well as intergranular corrosion and transgranular fissuration, are explored. The problem of welding silicon steels in nuclear assemblies is solved by the development of a 17% Cr, 14% Ni, 4% Si, 0.020% carbon alloy and additions of 3.3% Si in 18-8 stainless steels are found helpful in preventing stress corrosion in chloride environments. (M18--44942.)

Section 1

4. (Pamphlet) POLARIZATION MEASUREMENTS ON ASTM TYPE 6061-T6 ALUMINUM ALLOY IN THREE ONTARIO MINE SHAFT WATERS. G. J. Biefer. Technical Bulletin no TB-73, Aug. 1965, 16 p. Department of Mines and Technical Surveys, Mines Branch, Ottawa.

Cathodic polarization measurements carried out on 6061-T6 Al alloy in three Ontario mine waters of different acidities and compositions provided a rapid estimate of relative corrosion rates even though these rates were too low to be readily measured by conventional weight-loss methods. Results showed that corrosion rates in water from the Helen iron ore mine were much lower than those in waters from the two other mines. This correlated with the corrosion fatigue behavior of 6061-T6 alloy in the same waters. 10 ref.

(M18--44772.)

Section 1

5. **THE EFFECT OF ALLOYING ELEMENTS ON THE CORROSION OF ALUMINUM.**
K. F. Lorking. Australasian Corrosion Eng, v 9, no 10, Oct. 1965,
p 13-17.

The effect of the alloying elements Cu, Zn, Mg, Ag, Si, Cr, and Mn on the corrosion of the corresponding Al binary alloy had been determined over a wide pH range in water and alkali and in 0.1N solutions of the anions chromate, benzoate, chloride sulfate and phosphate. The effect of the anion was found to be specific although chromate inhibited corrosion of all alloys. Alloying elements may be divided into two groups--those causing a significant but small increase in weight losses due to corrosion, and those such as Zn, Ag and Cu which cause a marked increase in corrosion weight losses.

10 ref.

(M18--40674.)

Section 1

6. EVALUATION OF METALS IN DEEP OCEAN ENVIRONMENTS. John J. DeLuccia, Water Protection, v 5, no 8, Aug. 1966, p 49-51.

Over 200 metallic specimens, which were attached to a submersible test unit and exposed to deep ocean environments at 5640 ft for 123 days, have been evaluated for corrosion effects. Specimens were either heat treated, welded, painted, stressed or coupled. Materials included Al-Cu alloys, Al-Mn alloys, Al-Mg alloys, Al-Mg-Si alloys, Al-Zn-Mg alloys, Mg and dissimilar metal couples with 7975 Al, Be-Cu alloys, stainless steels, high-strength steels and Ti alloys. Differences between deep exposure corrosion and existing marine corrosion data were not extreme in most cases. Corrosion rates on Be-Cu alloys are only half as great as in shallow sea water. Epoxy coatings showed no loss of adhesion or gloss and no blistering, cracking or peeling.

(M18--53926.)

Section 1

7. OXIDE DISSOLUTION AND ITS EFFECT ON THE CORROSION OF 1100 ALUMINUM IN WATER AT 70 C. Shiro Mori and J. E. Draley. Electrochem Soc J, v 114, no 4, Apr. 1967, p 352-353.

Metal corrosion rates, as determined by measurements of weight gain, metal loss by corrosion and the Al content of the dissolved corrosion product have been analyzed for 1100 Al specimens exposed to water at 70 C. Results indicate that boehmite is formed as a corrosion product and covered by a thin layer of bayerite. The porous bayerite is the only one of the two compounds which dissolves.

9 ref.

(M18--72168.)

Section 1

8. (Russian--RZ) INVESTIGATION OF THE CORROSION OF ALUMINUM IN WATER AT HIGH TEMPERATURES. M.I. Tsypin, I.L. Rozenfel'd, Yu.P. Ol'khovnikov and S.V. Vizhekhovskaya. Trudy Gos N-I Proektn Inst Splavov Obrabotki Tsvetn Met, no 24, 1965, p 103-123.

The corrosion behavior of AOO brand 99.99% pure Al was investigated. Distilled water having a pH 5.5-6.3 was used as a corrosive medium and tests were carried out in large and small autoclaves. The specimens were previously etched in a 10% aqueous NaOH solution at 50 to 60 C., bleached in a 50% HNO₃ solution, washed in water, dried and degreased in ethyl alcohol. The oxidation kinetics of Al at 100 to 300 C was studied by weighing. At 200 to 225 C. the oxidation is parabolic; at 100 to 150 C. a general corrosion of Al takes place, the surface becoming covered by a thin film. At 200 C. a gray film is formed, which in due course gets darker. At 225 C. small blisters appear on the surface of Al, which finally merge into corrosion pits. At 250 C. an intense precipitation of white products of corrosion occurs. The structure of the film of the products of Al corrosion in water in different testing conditions was studied in light and electron microscopes. Isometric and acicular crystals, the products of inter-crystalline corrosion, could be observed on the external surface of the film. The internal side of the layer is a surface without a structure. The concept of the two-layer oxidation of the film is confirmed. The film of the products of corrosion consists of a basic, boehmite layer ($\gamma = \text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$) and a thin, optically structureless layer. The basic product of the reaction of Al with H₂O at 100 to 300 C. is boehmite. The structure of the film, which determines the diffusion rate of ions at a given temperature, should be considered the principal factor restricting corrosion. (M18--58048.)

Section 1

9. Corrosion of Aluminum in Water. D. N. Fultonberg.
NASA Contractor Report, Oct. 1967, (CR-899), 76 p.

Tests were performed in autoclave systems and in pumped loops to determine the corrosion and hydrogen generation of Al when subjected to conditions typical of those anticipated in a tungsten water-moderated reactor (TWR). These tests, the equipment used and the results obtained are discussed. It was concluded that the H₂ generation could be excessive under certain conditions and that a broader program would be necessary to predict it with some confidence. 11 ref. (35 0102.)

Section 1

10. Aluminum (Corrosion). Hugh P. Godard. Paper from 'The Corrosion of Light Metals'. 1967, 3-218 (Met. A., 6806-720062).

Detailed coverage on the corrosion of Al includes an analysis of: the nature and growth rate of the surface oxide film in air and water; the general corrosion behavior of Al alloys as influenced by environmental factors such as water, temperature, movement, surface-to-volume ratio, heat capacity of the surface, impurities and surface finish; the influence of Cr, Cu, Fe, Mg, Ni, Si, Ag, Sn, Ti, Zn, Sb, Bi and Pb on Al corrosion; galvanic, crevice, pitting, intergranular, stress, cavitation and impingement corrosion; atmospheric corrosion in rural, marine and industrial areas; corrosion in pure and sea water and in soils; corrosion of Al by building materials such as concrete and mortars, asbestos cement shingles and wood; corrosion behavior of welded, brazed and soldered joints; corrosion in transportation and storage and by specific chemicals and foods; cleaning Al surfaces and preventive measures, including material selection, cathodic protection with sacrificial anodes, Al-clad products or impressed current, organic, inorganic and metallic coatings, the use of inhibitors, alternation of the environment and the use of nonmetallic linings. 383 ref. (35 0519.)

Section I

11. An Electrochemical Study of Aluminum Corrosion in Boiling High Purity Water. R. A. Legault and J. E. Draley. Corrosion, Dec. 1967, 23, (12), 365-370 (in English).

An equation relating current and electrochemical potential has been derived on the basis of a physical model of the corrosion process. In the model, the total potential drop from metal to solution is the sum of potential contributions arising from the interfacial reactions at the metal-oxide and oxide-solution interfaces and that which derives from the transport of charged species through the film. On the basis of some simplifying assumptions, a derived equation has been fitted successfully to experimental data. Assigning the value usually attributed to α for the H_2 reaction, $1/2$, optimum values of the constants were found to vary reasonably with time. If a value of α is not assigned, the equation can be fitted to experimental data over a wide range of values for the three constants. Polarization measurements alone are not sufficient to determine unequivocal values for the constants, hence a determination of the mechanism of the aqueous oxidation of Al will require additional independent measurements. (35 0722.)

Section 1

12. USE OF ELECTROCHEMICAL POLARIZATION TECHNIQUES FOR CORROSION STUDIES IN AQUEOUS SYSTEMS AT HIGH TEMPERATURE AND PRESSURE. Wilde, B. E. (General Electric Vallecitos Nucleonics Lab., Pleasanton, Calif.). pp 68-72 of Proceedings of the First Joint Aerospace and Marine Corrosion Technology Seminar, Los Angeles, California, July 10-12, 1968. Houston, Tex.; National Association of Corrosion Engineers (1969).

From 1st Joint Aerospace and Marine Corrosion Technology Seminar, Los Angeles, Calif. See CONF-680732.

Methods of conducting in situ electrochemical studies of corrosion at temperatures up to 317°C in reactor cooling systems are described. The technique can be used to obtain accurate rate/time data in high resistance aqueous media down to corrosion rates of 10^{-4} mils per year. Data are included on 304 stainless steel corrosion rates in water.

(16417.)

Section 1

13. THE DISSOLUTION OF METAL CORROSION PRODUCTS IN DEIONIZED WATER AT 38°C (100°F). Johnson, Barbara A. (Lewis Research Center, Cleveland). Nucl. Appl. Technol.; 8: 50-61 (Jan 1970).

The dissolution of the corrosion products of metals in deionized water at 38°C (100°F) is discussed. Copper, tungsten, cadmium, and Mallory-1000 give soluble corrosion products. Mention is made of 304 stainless steel, 1100 aluminum, titanium, tantalum, silver, and boron steel, all of which showed no soluble corrosion products. The significance of soluble corrosion products in reactor cooling water is discussed. Data were obtained by chemical analyses of the water rather than from physical measurement of the test specimen.

(4718.)

Section 1

14. (ANL-7417, pp 79-83) SURFACE PHENOMENA. (Argonne National Lab., Ill.).

Research progress is reported on electrochemical processes in oxidation, aqueous corrosion of Al 1100, low-pressure oxidation and growth morphology, and spectral line shifts in electron probe microanalysis.

(25638.)

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Section 1

15. (ORNL-3994, pp 88-94) ELECTROCHEMICAL KINETICS AND ITS APPLICATION TO CORROSION. (Oak Ridge National Lab., Tenn.).

Further studies on the inhibition of corrosion of iron and steel showed that inorganic substances of the general formula XO_4^{n-} exert a noncathodic effect that greatly facilitates the passivation process. A mechanism is proposed, which accounts for the complex electrochemical behavior of iron in the presence of adsorbed anionic inhibitors. Studies with a rotating disk electrode assembly on the dissolution of Zr in HF-HCl and HF-HNO₃ mixtures showed that at potentials below the pitting potential in chloride media the rates of dissolution in the two solutions are identical. This result suggests that neither Cl⁻ nor NO₃⁻ ions exert a specific effect on the dissolution of the passive oxide layer by HF. Other experiments in HF-H₃PO₄ solutions disclosed a reduction in dissolution rate by several orders of magnitude; PO₄³⁻ ion inhibited the dissolution reaction by HF in a manner similar to the SO₄²⁻ ion. The diffusion coefficient of HF in certain solutions of interest was determined by the open-end capillary method. The rate of the anodic dissolution reaction of aluminum in alkaline NaCl solutions was measured as a function of pH, electrode potential, and time by use of a rotating disk electrode assembly. Observed changes in the rate of the dissolution reaction with time may be interpreted in terms of the kinetics of formation and dissolution of a porous aluminum oxide layer on the metal surface. This hypothesis was verified by electron micrographs of the oxide layers. Experiments were initiated on electrochemical aspects of the formation and growth of pits on titanium in chloride solutions. No significant effect of solution velocity on the initiation and growth processes was observed up to approx 3500 rpm. Breakdown potentials observed on application of constant

Section 1

14. (ANL-7417, pp 79-83) SURFACE PHENOMENA. (Argonne National Lab., Ill.).

Research progress is reported on electrochemical processes in oxidation, aqueous corrosion of Al 1100, low-pressure oxidation and growth morphology, and spectral line shifts in electron probe microanalysis.

(25638.)

17<

Section I

15. (cont.)

current were found to depend to some extent on the initial thickness of the passive oxide layer, although the final pitting potential was relatively unaffected. Addition of sulfate ions increased the breakdown potentials but hardly affected the pitting potential. Growth kinetics of single pits were investigated potentiostatically. Polarization curves of Ti and some of its alloys were measured in chloride solutions at temperatures up to 200°C by use of a Ti loop facility equipped for electrochemical studies. In the presence of a sufficient quantity of chloride ions, Ti alloys exhibited a pitting potential, which in some cases is low enough to allow pitting attack to proceed spontaneously. Increase of temperature profoundly affects the value of the pitting potential; plots of pitting potential as a function of temperature are reproducible and show clearly which alloys are superior in tendency towards pitting attack. Electrochemical aspects of the corrosion of types 5454 and 6061 Al alloys were studied in 1M NaCl at 150°C in Ti loop facilities. Changes in the polarization curves of anodic and cathodic processes occurring at the Al-electrolyte interface with pH provided a kinetic explanation for the existence of a minimum corrosion rate in chloride media. An enhanced rate of the cathodic process on the 6061 alloy accounts for its greater corrosion rate at any pH and for its susceptibility to pitting attack; this catalysis may be attributable to the Cu content of the 6061 alloy.

(1864.)

Section 1

16. (ORNL-4034) MATERIALS COMPATIBILITY AND CORROSION STUDIES FOR THE ARGONNE ADVANCED RESEARCH REACTOR. Griess, J. C.; English, J. L. (Oak Ridge National Lab., Tenn.). Nov. 1966. Contract W-7405-eng-26. 42p. Dep. mn. CFSTI \$3.00 cy. \$0.65 mn.

A material compatibility and corrosion investigation was conducted to determine the extent of corrosion to be expected in certain parts of the Argonne Advanced Research Reactor (AARR). The areas of concern were the Be reflector, the Al beam tubes and the stainless steel fuel element cladding, all of which are exposed to the primary coolant. All experiments were conducted in deionized water with a specific resistivity of 1 million ohm-cm or greater. The Be was free of localized attack, and neither crevices nor contact with stainless steel or Al produced adverse effects. The ratio of Be surface area to volume of water in the AARR is $2.3 \text{ cm}^2/\text{liter}$ and the Be reflector would be expected to corrode at a rate between 1.1 and 2.8 mils/yr, either of which would be acceptable. The corrosion of 6061-T6 Al, the material from which the beam tubes will be made, results in the formation of an insulating layer of corrosion products. Test results indicate that if the surface temperature of the beam tubes in the AARR can be maintained at 200°F or less, the rate of corrosion, and consequently the rate of corrosion-product buildup on the surfaces, will be low enough to prevent excessive temperatures in the beam tube walls. Tests showed that in Type 304 stainless steel after a 1000-hr exposure at a heat flux of $4.0 \times 10^6 \text{ Btu/hr-ft}^2$, numerous shallow (0.2 mil deep) cracks were present on the cooled surface. In a comparable test in which the heat flux was $2.1 \times 10^6 \text{ Btu/hr-ft}^2$ (hot-spot heat flux for 100 Mw operation of AARR) and the exposure time was 2000 hr, no cracks were found. In view of the failures experienced in stainless steel fuel-element cladding in both pressurized and boiling water reactors, it is recommended that further investigation into this type of cracking be carried out.

Section 1

17. (NAA-SR-Memo-10178) THE WATER CHEMISTRY OF HERMETICALLY-SEALED BOILING WATER REACTORS. Haroldsen, G. O. (Atomics International, Canoga Park, Calif.). Aug. 10, 1964. Contract AT-11-1-GEN-8. 52p. Dep. mn. CFSTI \$3.00 cy, \$0.50 mn.

To provide a basis for establishing the requirements of the water purification system for a hermetically-sealed boiling water reactor (such as COMPACT or its predecessor, SNAP 4), an extensive literature survey is made of corrosion rates and of corrosion-product deposition rates in 300-series stainless-steel systems containing high-purity, high-temperature water. After a stable oxide film is formed, the rate of corrosion remains low, generally reported as 5 to 10 mg/dm²mo. Within reasonable limits, dissolved oxygen, dissolved hydrogen, pH, and temperature do not significantly affect the rate, which is already low. Although much has been written about crud deposition and scale formation, the information is largely qualitative, and the data do not allow firm, quantitative estimates to be made. It is clear, however, that maximum water purity minimizes deposition. Based on presently available information, it appears that fouling in the COMPACT reactor, over its intended core life of 10,000 hours, can be precluded by maintaining the level of impurities at about 10 ppb. It is probable that a ten-fold increase in this amount could be tolerated, but experimental verification is required. It is feasible that a mixed bed deionizing system, to purify the total condensate flow and a 0.5% side stream from the reactor core, could reduce the impurity concentration to 10 ppb. Here again experimental verification is needed. Less well established is the basis for designing a deionizing system with a 10,000 hour life that can remove both filterable and dissolved impurities.

(18089.)

Section 1

18. ALUMINUM FOR HIGH-TEMPERATURE WATER-COOLED REACTOR SERVICE: CORROSION CONSIDERATIONS. R. L. Dillon (General Electric Co. Hanford Atomic Products Operation, Richland, Wash.). June 7, 1961. Contract AT(45-1)-1350. 12p. Dep. (mn); OTS.

A review is presented of the corrosion of Al and its alloys in high-temperature steam and water. The concept of corrosion regulation by dissolution of corrosion products is discussed and applied to pressurized-water and boiling-water reactors, and predictions of Al corrosion are compared with in-reactor experience. The development of Al alloys and powder metallurgy products with decreased corrosion properties is discussed. In-reactor demonstration of Al corrosion behavior and the economic incentives for Al development are discussed.

922 (HW-69876).

Section 1

19. OXIDE DISSOLUTION IN CORROSION OF ALUMINUM CLADDING ON NUCLEAR REACTOR FUEL ELEMENTS. D. R. Dickinson (Battelle Memorial Inst., Richland, Wash.). Corrosion, 21: 19-27 (Jan. 1967).

The effect of aluminum oxide dissolution on aluminum corrosion in high-temperature deionized water is discussed. Such dissolution promotes corrosion by removing protective oxide and is especially significant in nonisothermal systems, such as aluminum-clad fuel elements in nuclear reactors cooled by pressurized water, where the water cannot be kept saturated with dissolved oxide. It is hypothesized that resistance to dissolution lies principally in mass transfer from oxide-water surface into bulk water. Dissolution rates could be estimated by use of general equations for mass transfer if oxide solubility was known as a function of temperature. Dissolution rates thus calculated were in good agreement with in-reactor corrosion rates of aluminum-clad fuel elements in high-temperature deionized water, which were previously measured at Hanford. Fair agreement was obtained with measurements in recirculating out-of-reactor loops. It was concluded that mass transfer of dissolved oxide into the water may be the principal factor controlling the corrosion rate of aluminum-clad fuel elements in many nuclear reactor systems using high-temperature pressurized deionized water as coolant.

(13920.)

Section 1

20. **CORROSION STUDIES FOR AQUEOUS REACTORS AND CHEMICAL PROCESSES.**
J. C. Griess, J. L. English, L. L. Fairchild, and P. D. Neumann
(Oak Ridge National Lab., Tenn.).

Corrosion testing in support of the High Flux Isotope Reactor was completed. Dynamic and static tests were conducted on Al alloys and Be in water adjusted to a pH of 5.0 at 50 and 100°C. The corrosion of neither metal was sufficient to preclude its use in the construction of the reactor. Tests with other materials, including plastics, were conducted to determine their suitability in reactor components. Corrosion tests in various chloride-containing solutions of direct interest to the Transuranium Processing Facility were completed; most tests were of the behavior of Ta and Zr. Both materials have adequate corrosion resistance to the process solutions, but both absorb corrosion-product H which, in sufficient quantities, produces embrittlement. The corrosion resistance of metals and alloys was examined in gaseous and aqueous environments of interest to various fuel element re-processing schemes. In addition, the corrosion resistance of Type 304 ELC stainless steel was determined in calcining waste products with and without additions of glass-forming chemicals. 30671 (ORNL-3789(p.122-5))

Section 1

21. MATERIALS DEVELOPMENT, CORROSION, AND HEAT TRANSFER STUDIES,
(General Electric Co. Hanford Atomic Products Operation, Richland,
Wash.).

Zircaloy-2 samples exposed to 540°F water in the ETR G-7 loop (7.6×10^{13} nv fact) corroded roughly 15 times the rate of comparison coupons exposed out-of-reactor. Oxidation rates for thoria dispersion-hardened nickel were determined. Oxidation of crystal bar zirconium and Zircaloy-2 in 600°C CO was ten times lower than in steam at the same temperature. Comparisons of the corrosion of various nickel and iron alloys in deoxygenated 3000 psi, 550°C steam with data from oxygenated systems indicated little effect from 3 to 4 ppm O₂. In tests of aluminum alloys in 330°C deionized water, corrosion rates increased 50 to 100% as the ΔT in the loop increased from 8 to 55°C. Radiation effects on aluminum alloy corrosion were determined in the H-1 loop. Oxidation and deuterium absorption of Zircaloy-2 and Zircaloy-4 in D₂O at pD10 LiOD were determined. Studies were continued on the mechanism by which zirconium is charged with hydrogen in excess of solubility. The ninth pressure tube to be discharged from the PRTR for destructive testing was examined. 8752 (HW-78388(p.9.1-4))

Section 1

22. CORROSION OF SOME SPECIAL ALUMINUM ALLOYS FOR REACTOR CONSTRUCTION IN WATER UP TO 100°C. H. Spindler, W. H. Hauthal, and D. Rettig. (Zentralinstitut für Kernphysik, Rossendorf, Ger.). Kernaenergie, 7: 291-9 (May 1964). (In German)

Pure aluminum, a number of aluminum-nickel-iron alloys, and a number of aluminum-silicon-nickel alloys were submerged in ultra-pure water at 25, 50, and 100°C for 30 days and examined for corrosion phenomena. The results obtained at 25°C differed from those obtained at the two higher testing temperatures while the latter two did not differ significantly. Silicon inhibits the corrosion rate, regardless of concentration. Hydration of the oxide layer formed in water increases with increased exposure time; after the entire layer hydrated, the action of the galvanic microelements between aluminum and alloying metals becomes evident. Hydration rate is affected by exposure time, temperature, and composition. 32093.

Section 1

23. CORROSION OF ALUMINUM IN WATER. M. Kawasaki, S. Nomura, H. Itami, Y. Kondo, T. Kondo, N. Ito, and C. Akutsu (Japan Atomic Energy Research Inst., Tokyo). June 1962. eep.

Dynamic corrosion of aluminum was studied under conditions corresponding to those prevailing in reactors. When commercial grade pure aluminum is corroded in flowing water at 50 to 70°C, the weight of the aluminum specimen will increase with time at low flow rates, and decrease with increasing flow rate. The change of weight of the specimen is also affected by water purity and conditions of the surrounding atmosphere. The total amount of corrosion from chemical analysis does not always parallel the change of weight of the specimen, and pitting (about 10 microns in diameter) is also observed to occur in many specimens. It can therefore be concluded that it is difficult to estimate the corrosion amount only from the change of weight of the specimen. In several cases the relation between the properties of surface oxide film and corrosion reaction in flowing water was established. With the further objective of developing aluminum alloys that are corrosion-resistant in hot water, some studies on the corrosion mechanism of aluminum alloys in hot water and steam were conducted. In hot water, aluminum is corroded easily with higher purities of the metal, and according to Draley, this is due to the penetration of hydrogen into the grain boundaries. Various methods were used to ascertain his mechanism, and in particular, hydrogen evolution at the grain boundaries was directly observed in a windowed autoclave. It was also established by x-ray and electron-microscopic methods that the transformation and structural change of the surface oxide film modifies the kinetics of corrosion of aluminum in steam around 350°C. 18698 (JAERI-1035).

Section 1

24. Fundamentals of the Corrosion of Super-Pure Aluminum in Water. Ketil Videm. Proc. Third Internat. Congress on Metallic Corrosion, Moscow, 1966, 1967, 521-533; discussion, 530-532 (Met. A., 7001-720024) (in English).

Factors affecting the movement of ions through a passive film were investigated. The corrosion-rate and anodic-polarization curves were determined for super-pure Al in continuously purified water; the a.c. capacity of the exposed specimens and their behaviour during anodizing were studied. Data for the protective properties of the barrier oxide; relations between current, barrier-thickness, and potential; determination of barrier voltage; stability of the barrier oxide; the unpolarized corrosion of Al; and influence of O in the water are reported. The effect of electric field strength on ionic transport processes, significance of constants in the rate equation, magnitude of the dielectric constant function of the bulk oxide during corrosion, corrosion of active Al, and attack at high temp. are discussed. 13 ref. (35 0590)

Section 1

25. Corrosion of Metals in Distilled Water at 250°C. A. V. Byalobzhesky and G. M. Anurova. Zashchita Metallov, Mar.-Apr. 1971, 7, (2), 112-117 (in Russian).

The corrosion of Fe, Ni, Cr, Ta, Mo, Cu, and various alloys in distilled water under pressure in an autoclave at 250°C was studied by chromatographic, gravimetric, and X-ray methods. All the metals tested underwent a certain amount of corrosion under these conditions, H being evolved in each case, although the part played by the evolving H in the corrosion mechanism was only a minor one. The main corrosion process was the formation of an oxide or hydroxide film, which was then decomposed by the electrolyte. Formulae representing the progress of the corrosion processes are derived.

(35 0839.)

Section

26. Effects of Deep Ocean Organisms on Constructional Materials.
James S. Muraoka. Proceedings of the First Joint Aerospace and Marine Corrosion Technology Seminar, 1969, 115-116 (Met. A., 7006-72 0137) (in English).

Steels, Cu alloys, Al alloys, plastic rods, rope and wood panels were exposed on the sea floor at depths of 2400 and 6800 ft for 4-36 months. The steels and Cu alloys corroded uniformly over their surfaces. Stainless steels 316, 316L, 20Nb and 17Cr-7Ni-0.7Ti-0.2Al were not corroded, but 405, 410, 430, 301 and 304 showed pitting, edge and crevice corrosion. 2219-T81, 3003-H12 (Alclad), 5454-H32, 5456-H321, 5083-H113, 6061-T6, 7178-T6 and other Al alloys all showed pitting, edge and crevice corrosion. Ni alloys, Ni-Fe-Cr-825 and Ni-Mo-Cr-'C' were not corroded, nor were Ti alloys.- M.J.R.

(35 0632.)

Section 1

27. Studies on Boehmite Forming Process. II. - Effect of Water Purity on the Formation and the Corrosion Resistance of Boehmite Films. Hiroyuki Yamada and Tsuneyoshi Okamoto, J. Japan Inst. Light Metals, June 1971, 21, (6), 405-412 (in Japanese).

Cf. preceding abstract. Boehmite films formed in city water were thinner and less protective than those formed in deionized water. This was attributed to the appreciable silicate content of the former. The films formed in deionized water were independent of the resistivity of the water, providing that its value was $\times 10^5 \Omega$ cm. The blackness of films formed in city water was apparently related to its Si and Ca content. The results were obtained from electron microscope electron diffraction, and microprobe analyser studies. 8 ref. (35 0175.)

Section 1

28. Transfer of Steel Corrosion Products to Water and Their Deposition on the Surfaces of Structural Materials under Static Conditions. A. I. Gromova, I. K. Murozova, V. V. Gerasimov, A. V. Zhenikhova, and V. A. Kucheryaev. Teploenergetika, June 1970, (6), 54-56 (in Russian).

The transfer of corrosion products from stainless and pearlitic steels to water, and their deposition, was studied in an autoclave at 300°C and 87 kg/cm². The internal surface of the autoclave was made of stainless steel Kh18N10P or steel St. 20. The rate of deposition of the corrosion products onto specimens of Ti, Zr, and Ni alloys, placed in the autoclave, was measured by a radioactive method. The rate of transfer from stainless steel to de-aerated water was more than two orders of magnitude lower than from steel St. 20. The rate of transfer increased on raising the O content of the water. The rate of deposition onto the Ti alloy was greater than onto the Zr alloy. The results are presented graphically and discussed. (35 0134.)

Section 1

29. The Influence of Small Amounts of Impurities in Water Corrosion of Aluminum. G. Ito, S. Ishida, M. Kato, T. T. Nakayama, Aluminum, Dec. 1970, 46, (12 815-819 (in German)).

The increase in weight or loss of metal was measured when Al (commercial quality, degreased sheet; the purity of the metal has little influence on its corrosion) was kept at 25, 35, or 50°C for up to 40 contact, unstirred, with pure water (resistance $> 10^6 \Omega \text{ cm}$) or into water to which Cl^- (from HCl or metallic chlorides), SO_4^{2-} (from H_2SO_4 or metallic sulphates), NO_3^- (from metallic nitrates), OH^- , CO_2^{2-} , HCO_3^- , CrO_4^{2-} , PO_4^{3-} , borate ion, and F^- had been added to concentrations of 10^{-4} to 10^{-8} M. Changes in the composition of water have considerable effects on the corrosion; these depend more on the anions than on the cations. The pH value has some influence but this is largely caused by the anions present. According to the activity anions can be separated into two groups, the first causing general corrosion of Al similar to pure water, the second inhibited corrosion with increasing concentration. An attempt is made to explain this and a hypothesis is offered for the role played by ions on pitting of Al. 13 ref. (35 0624.)

Section 1

30. Leaching Action of High Purity Water Plastics and Other Materials, Knoedler, E. L. and Goodson, J. B.; Corrosion, Vol 16, No. 8, 1965.

Laboratory tests were made to determine the leaching effects of high purity water on plastics and other materials used in building or lining of high purity water storage tanks. Specifically tested were enamels used in painting interiors of condensate storage tanks, materials used for filter tubes, plastic and metal materials of construction, and miscellaneous coating and linings materials. Materials tested include polyethylene, epoxy resins, phenol-formaldehyde resins, cotton, cellulose acetate, glass fiber, mineral rubber with inert filler mixture, asphalt clay-cement mixture, polyvinyl over sprayed zinc and aluminum, sprayed zinc and aluminum, hydraulic cement, commercially pure aluminum, and coal tar enamels. 4.6.5

Section 1

31. Performance of Aluminum Alloys Under Desalination Conditions.
R. A. Legault and W. J. Bettin. Mater. Prot. Performance,
Mar. 1971, 10, (3), 9-12 (in English).

Five Al alloys, 1100, 3003, 5052, 5554 and 6061 were tested in de-aerated sea water at 250°F for two weeks to discover their ability to withstand desalination conditions. Only 5052 Al performed satisfactorily enough to suggest its use in desalination applications. 15 ref.

(35 0911.)

Section 1

32. Behavior of Metals in Desalination Environments: Third Progress Report. LaVern Rice, Charles F. Schrieber and F. H. Coley. Proceedings NACE 26th Conference, 1970, 589-596 (Met. A., 7108-72 0171) (in English).

Potentiostatic anodic polarization experiments were performed on two low-alloy steels and four stainless steels in a test facility at a sea-water treating plant. Results show that the corrosion rates for the A242 Cu-bearing steel alloy are substantially lower than those for the 1010 mild steel in short-term tests at O concentrations of 100 ppb. Above this level, there is no difference in corrosion rates for the two alloys. The pitting of 410, 304 and 316 stainless steels appears to be predictable from the critical pitting potential as determined from an anodic polarization curve and the open-circuit potential of the material in the same environment. Pitting is noted on susceptible test materials with surface areas as small as 8.5 cm². Parameters such as pH, temp. and dissolved O affect the open-circuit potential and the critical pitting potential. 27 ref. (35 0971.)

Section 1

33. Observations of Hot Saline Water Corrosion of Aluminum Alloys.
A. C. Fraker and A. W. Ruff, Jr. Corrosion, Apr. 1971, 27, (4),
151-156 (in English).

Corrosion films formed on Al in hot salt water (100-177°C) after short-time tests of 30s-1h were studied by transmission electron microscopy, and diffraction. It was concluded from observation of 6061, 5454, 2024, Al-1.52 Mn and Al-1.66 Ni alloys that one controlling factor in the initiation and growth of the outer film of the duplex oxide film is the alloy composition. The outer film formed earlier on alloys containing Cu and the film growth was also more rapid on these alloys. Additions of Mg, Mn and Ni retarded this outer film formation but with Ni additions, pitting attack was more severe and deposition of Cu from solution was increased. The formation and growth of the outer film was slowest on the 5454 alloy which was also the most resistant alloy tested. 12 ref. (35 1283.)

Section 1

34. Corrosion of Aluminum Alloys in High Purity Water in the Range 150°C-340°C, D. F. MacLennan; Corrosion, Vol 17, No. 5, 1961.

Examination of the cross-section of the film on aluminum and its alloys, while attached to the metal, reveals that the second phase particles play an important part in the corrosion pattern. The 2S aluminum contains at least two types of second phase particles, one of which corrodes more rapidly while the other corrodes more slowly than the surrounding aluminum. The majority of second phase particles in the 157 alloy corrode more slowly than the surrounding aluminum.

Section 1

35. Oxide Dissolution in Corrosion of Aluminum Cladding on Nuclear Reactor Fuel Elements. D. R. Dickinson; Corrosion, Vol 21, No. 1, (1965)

Effect of aluminum oxide dissolution on aluminum corrosion in high temperature deionized water is discussed. Such dissolution promotes corrosion by removing protective oxide and is especially significant in non-isothermal systems, such as aluminum-clad fuel elements in nuclear reactors cooled by pressurized water, where the water cannot be kept saturated with dissolved oxide. Hypothesis is advanced that resistance to dissolution lies oxide-water surface into bulk water; dissolution rate can be estimated by use of general equations for mass transfer, if oxide solubility is known as a function of temperature. Dissolution rates thus calculated were in good agreement with in-reactor corrosion rates of aluminum-clad fuel elements in high temperature deionized water, which had been previously measured at Hanford. Fair agreement was obtained with measurements in recirculating out-of-reactor loops. It is concluded that mass transfer of dissolved oxide into the water may be the principal factor controlling corrosion rate of aluminum-clad fuel elements in many nuclear reactor systems using high temperature pressurized deionized water as coolant.

Section 1

36. Polarization Studies of Aluminum Alloys in Water at 200C and 300C
J. H. Greenblatt and A. F. McMillan, Corrosion, Vol 19, No. 4,
(1963).

Specimens of commercial 2S aluminum and two special alloys containing iron and nickel were polarized anodically and cathodically at a number of different current densities at 200°C and 300°C. Weight gains were obtained and the potentials relative to the stainless steel autoclave were measured by an interrupter method. The weight gain data indicated that the polarizing current is being carried by electronic conduction.

The potential-time curves for anodic polarization indicate differences between 2S aluminum and the alloys in that greater polarization is obtained with the latter. These curves also indicate that the impressed current decreases the film resistance. In all cases the potential reached a plateau value with time and this time was shorter for the alloys.

The potential-time curves for cathodic polarization also show plateau values but the rise to a plateau value is in the opposite sense to the applied current. With increasing cathodic polarization the plateau values occur at more negative values of the potential. This latter trend is in the same direction as the applied polarizing current. This apparently is explained in terms of the build-up of the aluminum oxidation potential which acts in a sense opposite to the applied current. Again the time to reach plateau values was shorter for the alloys.

Voltage-current curves were also obtained on specimens left overnight (approximately 17 hours) at two different anodic polarizing currents. These curves indicated differences between 2S aluminum and the alloys; these differences are discussed in terms of the semiconducting properties of the oxide film.

Section 1

36. (cont.)

The observations made on the differences in the properties of the oxide films on the materials examined as revealed by potential and polarization curves are discussed as to their significance in determining corrosion resistance.

21

Section 1

37. Corrosion Aspects of Iron, Austenitic Stainless Steel and Inconel in High Temperature Water, C. F. Cheng; Corrosion, Vol. 20, Nov. 11 (1964).

The available data dealing with corrosion of iron, austenitic stainless steel and Inconel in high temperature water are presented. The mechanism of iron corrosion and the effect of water conditions such as pH, dissolved oxygen, dissolved hydrogen and velocity are reviewed. Maintaining the pH close to 10.5 is beneficial to pressurized water-cooled reactors for all three metal systems. Dissolved oxygen in sufficient quantity may be desirable for carbon steel, but it will release a high concentration of chromates in austenitic stainless steel and Inconel systems. Dissolved hydrogen may have a slight inhibiting effect during the early stages of corrosion of austenitic stainless steel, but has little effect on carbon steel and Inconel. In oxygen-free water, carbon steel produces a brown-black film of magnetite, austenitic stainless steel a dull film of magnetite and spinel (R_3O_4); and Inconel a dark tarnished film of NiO, spinel (R_3O_4) and R_2O_3 .

Section 1

38. Effect of Oxygen Concentration and pH on Electrode Potential of Stainless Steels and Operation of Microcouples, Korovin, Yu. M., Ulanovskii, I. E., Corrosion, Vol 22, (1966)

Using Russian steels of 13 Cr and 18-9 analyses in general experiments are conducted varying pH and oxygen concentrations in sea water to determine the relative effect of these variables on corrosion rates in crevices. Effect of oxygen concentrations in the range 1-9 mg/l and pH 8.3 to 2.3 are reported.

Changes in pH were found to strongly and changes in oxygen concentration weakly influence the corrosion rate. Authors postulate that anodic passivity is effected after application of tenths of a milliampere per square centimeter whereas when the pH in the anodic space is lowered, critical current density required for passivation increases 40-50 fold.

Heat treatment of the steel does not influence electrode potentials when solution pH is lowered.

Section 1

30. Corrosion Behavior of Nickel-Chromium-Iron Alloy 600 in Borated Pressurized Water Reactor Environments, Van Rooyen, D., Copson, H. R., Berry, W. E.: Corrosion, Vol 25, No. 5, (1969)

Corrosion tests were run for periods up to 5000 hrs in borated water at 600°F (316°C) in a high velocity loop on strip, tubes, and weldments of Alloy 600. During the first 1000 to 2000 hrs of exposure, corrosion rate differences depending upon surface preparation and in the case of weld metal on composition were observed. However, by 3000 to 5000 hrs, the corrosion rates of all samples tested had decreased to around 1.0 mg/dm² per month, or to rates of scale formation of about 1.4 mg/dm² per month. Approximately half the scale was adherent, and half was lost to the stream. Comparison with earlier tests in borate--free water seemed to indicate that the addition of boric acid increased corrosion somewhat during the initial exposure periods, but this was a transient effect, and corrosion rates decreased with time.

Section 1

40. Initial Corrosion of Aluminum Alloys in High Temperature Water, Greenblatt, J. H.; Corrosion, Vol 19, No. 9 (1963).

The corrosion rates of commercial 2S aluminum and two iron- and nickel-containing alloys have been obtained by measuring the thickness of the oxide film on specimens after exposure to high temperature water for periods of 1 minute to 7 hours. Measurements were made on suitably polished and mounted cross sections with a filar micrometer eye-piece. The rates from thickness measurements were compared to rates obtained from weight gain data and the activation energies were determined for both types of rate data. The rate data and activation energy values are compared with previously reported values and the resulting differences discussed.

Section 1

41. Influence of Oxygen on High Temperature Aqueous Corrosion of Iron; Ruther, W. E. and Hart, R. K., Corrosion, Vol. 29, No. 4 (1963).

The corrosion of pure iron in distilled water was investigated over a range of temperature (50-315°C) and oxygen content of the water (0.1-540 ppm).

A uniform brown-black coating formed in water of low oxygen content at 260°C. The corrosion rate was low after the initial period.

Severe pitting of the iron was encountered in water containing intermediate concentrations of oxygen over the entire range of temperature. The average metal loss rates were also much higher than that obtained in a low oxygen environment.

A thin protective film formed when adequate oxygen was present. No significant pitting was noted. The total corrosion at 30 days' exposure was lower for samples in a 260C-540 ppm oxygen test than for those with very little oxygen at the same temperature.

The protective film forming ability of iron in a pitting oxygen-water environment was improved by alloying, particularly with chromium and carbon. The improved corrosion performance of some mild and low alloy steels over pure iron at intermediate oxygen concentrations was also noted.

The thin films formed on pure iron in adequately oxygenated water were composed of $\text{-Fe}_2\text{O}_3$ (hematite) as contrasted with the Fe_3O_4 (magnetite) coatings produced in deoxygenated water. Distinctive growth habits for the various iron oxides were found on the outer surfaces of the protective coatings.

Section 1

42. Reybrouck, G. and H. Van de Voorde, "Simple Method of Testing the Corrosiveness of Disinfectants", Zentralbl. Bakteriologie, Parasitenkunde, Infektionskrankheiten, Abt. 1: Orig., 217 (1), 128-31 (1971). (C.A., 15, 67458x (1971)).

Corrosiveness is expressed as the weight loss (mg) per dm^2 area of specimen per day at unspecified temp. The most corrosive disinfectant was Lugol soln. (5% I_2 in 10% KI) which was tested with Al, Cu, steel, and stainless steel. Other disinfectants tested, 0.3% chloramine, 0.5% chlorhexidine gluconate, 3% chloroxylenol and terpineol, and 0.9% iodophore, were similar in corrosiveness to H_2O .

Section 1

43. Static Tests of Corrosion Inhibitors for Aluminum and Carbon Steel, R. B. Richman, BNWL-29, Pacific Northwest Laboratories, Richland, Washington. February 1965.

Uniform corrosion losses and pitting tendencies were measured for carbon steel and aluminum samples exposed for as long as 9-1/2 months at ambient temperatures in static, non-refreshed solutions of 38 different inhibitor mixtures. Only two candidates, sodium hexametaphosphate, and sodium nitrite plus sodium silicate, provided inhibition comparable to that obtained with sodium dichromate. The tests were conducted with concentration levels of 100 ppm, although in-practice 1-2 ppm of sodium dichromate is effective.

Section 2

MICROBIOLOGICAL CORROSION

1. MICROBIOLOGICAL CORROSION--SULPHATE REDUCING BACTERIA & THEIR CORROSIVE INFLUENCE ON METALS. Brajendra Nath Tripathi. J Sci Ind Res, v 23, no 9, Sept. 1964, p 379-389.

Taxonomy and morphology of bacteria, sulfate metabolism, conditions for the natural growth of bacteria, habitat, corrosive locations and damage. The detection of corrosive locations and the mechanism of corrosion reaction including cathodic depolarization, anodic stimulation, electrochemical cell action and the chemical action of ecological materials. Forms of corrosion, rate of penetration, presence of Fe^{2+} in the medium, effect of anchorage and effect of galvanic current. Methods for protection against bacterial corrosion include: treatment of metal surface, cathodic protection and chemical inhibition. 196 ref. (M18--18099.)

Section 2

2. DIRECT EVIDENCE FOR THE CATHODIC DEPOLARIZATION THEORY OF BACTERIAL CORROSION. Warren P. Iverson, Science, v 151, no 3713, Feb. 25, 1966, p 986-988.

Cathodic depolarization of mild steel by *Desulfovibrio desulfuricans* was demonstrated with benzyl viologen used as an electron acceptor. Direct measurement of the cathodic depolarization current indicated a maximum current density of 1 micro amp/cm². Al alloys are also cathodically depolarized by the organism. 7 ref. (M18--45652.)

Section 2

3. **MICROBIAL CORROSION OF METALS.** J. McDougall. Anti-Corrosion Methods Mater, v 1, no 3, Aug. 1966, p 9-13.

Mechanisms of anaerobic and aerobic corrosion of ferrous and non-ferrous metals by micro-organisms are reviewed. Corrosion can be brought about by a direct effect of the bacteria on electrochemical reactions, by the production of corrosive chemicals and by the production of differential aeration and concentration cells on the metal surface. Bacteria may prevent formation of a protective scale with resultant pitting; attack is especially severe if the micro-organisms produce acids and alkalis. Incrustations by Fe bacteria in cast iron pipes, anaerobic corrosion of cast iron and steel pipes and tanks by desulphovibrio, acid corrosion of steel by thiobacillus and bacterial nitrate reduction as a corrosion process are discussed. Control measures include changing environmental conditions, the use of protective coatings and cathodic protection. 69 ref. (M18--55694.)

Section 2

4. CORROSION OF MILD STEEL BY ACTIVELY GROWING CULTURES OF SULFATE-REDUCING BACTERIA. THE INFLUENCE OF FERROUS IRON. G. H. Booth, Pamela M. Cooper and D. S. Fickerley. Brit Corrosion J, v 1, no 9, Nov. 1966, p 345-349.

The rate of corrosion of mild steel has been studied in the presence of actively growing pure cultures of a number of different strains of sulfate-reducing bacteria using a culture medium containing sufficient ferrous iron to prevent the accumulation of free sulfide ions in the culture and hence to prevent the direct interaction between the metal and sulfide ions to give partially protective films. This was only partially successful in so far as some patchy film formation was experienced. Nevertheless, it was found that all the bacteria induced corrosion to approximately the same extent and that corrosion-time curves were essentially linear. There was no relationship observable between the rate of corrosion and the hydrogenase activity of the bacteria. *Desulfotomaculum orientis*, an organism previously thought to be relatively innocuous, was found to show hydrogenase activity in the conditions used and to be almost equally aggressive as *Desulfovibrio desulfuricans*. 15 ref. (M18--65995.)

Section 2

5. **MECHANISM OF MICROBIOLOGICAL CORROSION OF ALUMINUM ALLOYS.**
H. G. Hedrick, M. G. Crum, R. J. Reynolds and S. C. Culver.
Electrochem Technol, v 5, no 3-4, Mar. -Apr. 1967, p 75-77.

Several hypotheses of possible mechanisms of microbiological corrosion of Al alloys are discussed: the micro-organisms remove mineral inhibitors from the medium in their growth process and thereby stimulate metal corrosion, the micro-organisms produce corrosive materials by the oxidation and transformation of hydrocarbon substrates, the micro-organisms establish microcenters of galvanic activity on the metal surfaces, the micro-organisms remove electrons from the surface of the metal by acting as electron mediators. Results of experiments to demonstrate microbiological corrosion on aircraft structural Al alloys under laboratory conditions are presented. 7075 and 2024 Al alloys were placed in a deionized-water fuel environment, with and without a surfactant added, and some were inoculated with cultures of micro-organisms reported as fuel contaminants and species of organisms capable of corroding structural Al alloys. 9 ref. (M18--71690.)

Section 2

6. A POSSIBLE ROLE FOR SULFATE REDUCERS IN THE CORROSION OF ALUMINUM ALLOYS. Warren P. Iverson. Electrochem Technol, v 5, no 3-4, Mar.-Apr. 1967, p 77-79.

Serious corrosion of Al alloys in integral fuel tanks and associated fuel system components has been a problem in turbine-powered aircraft utilizing hydrocarbon jet fuels. The corrosion has been associated with microbial contamination of the fuels. Most of the corroded fuel tanks were topcoated with a Buna-N phenolic resin, a material which appears to be quite susceptible to microbial attack. In order to simulate topcoating deterioration and corrosion in the laboratory, several test tanks were fabricated from the same Al alloy and were coated with various topcoatings. Evidence was obtained for the presence of sulfate-reducing bacteria in pits of an epoxy-topcoated 7178 Al alloy tank, and in tubercles and pits in a similar tank coated with Buna-N phenolic resin. The sulfate-reducers suggest the presence of anaerobic conditions in the Al corrosion pit which could result in an oxygen concentration-cell effect. 12 ref. (M18--71691.)

Section 2

7. **ALUMINUM CORROSION PROCESSES IN MICROBIAL CULTURES.** Gordon C. Blanchard and Charles R. Coucher. Electrochem Technol, v 5, no 3-4, Mar. -Apr. 1967, p 79-83.

The growth of micro-organisms for 30 and 90 days in media containing 0-0.8 m molar KNO_3 as the only nitrogen source for growth caused significantly greater corrosion of alloys 7075 and 2024 than was observed in controls. Micro-organisms failed to cause corrosion, even after 90 days in BH medium (12m molar KNO_3), although the nitrate concentration after 20 days had decreased to a noninhibitory concentration (0.1 m molar) for corrosion. All nitrate concentrations inhibited corrosion by the media alone. Micro-organisms reduced some of the nitrate to nitrite during the first 20 days' incubation on 12 m molar KNO_3 media; nitrite increased from 0 at zero time to 2 m molar after 20 days. However, chemical corrosion tests showed that nitrite was not an effective inhibitor of aluminum alloy corrosion. Nitrite at 10 times the concentration of CaCl_2 caused no inhibition of CaCl_2 corrosion of alloys 7075 and 2024. Nitrate, however, was a very effective inhibitor of CaCl_2 corrosion in this concentration range. The 90-day high nitrate growth media were found to contain corrosive anionic and cationic components upon fractionation by ion exchange chromatography. These corrosive components were demonstrated by Sephadex chromatography to be large molecules with molecular weights greater than 5000. The lack of corrosion by micro-organisms after the 90-day incubation in BH medium (12 m molar KNO_3) was believed to be due to the formation of corrosion-inhibiting products. 30 ref.

(M18--71692.)

Section 2

8. Bacteria as a Cause of Corrosion. ----. Corrosion Prevention and Control, Feb. 1968, 15, (2), 21-23.

Special ref. is made to studies demonstrating (i) the ability of certain bacteria to remove H or electrons from Fe and Al surfaces and (ii) the setting-up of anodic/cathodic couples in fuel tanks by the action of, for instance, anaerobic sulphate-reducing organisms growing under an aerobic-bacteria slime.

(35 0589.)

Section 2

9. Effects of Marine Organisms. James S. Muraoka. Mach. Design, 18 Jan. 1968, 40, (2), 184-187.

Ship-bottom fouling is one of the oldest biological problems known to mariners. The various forms of animal and plant life that cause shallow-water and deep-ocean biodeterioration are described. The effects of deep-ocean environment on test specimens of metals and alloys were studied, producing data on fouling and also valuable information on the related problem of corrosion. Results are available on Cu-Ni alloys, stainless steel (316, 316L, 20-Cb, 17Cr-7Ni-0.7Ti-0.2Al, 405, 410, 430, 301 and 304), Al alloys (2219-T81, Alclad 3003-H12 5454-H32, 5456-H321, 5083-H113, 6061-T6, 7039-T6, 7079-T6, and 7178-T6); Ni alloys (Ni-Fe-Cr-825, Ni-Mo-Cr-'C', Ni 200); and Ti alloys. Marine fouling on submerged surfaces is effectively retarded by application of toxic Cu or Hg-containing paints. Recently developed organometallics such as bis(tributyl) tin oxide show great promise for use against fouling organisms. (35 0387.)

Section 2

10. **Anaerobic Corrosion of Aluminum by Sulphate-Reducing Bacteria.**
A. K. Tiller and G. H. Booth. Corrosion Sci., July 1968, 8, (7),
549-555 (in English).

An investigation was carried out to determine whether the corrosion and polarization of Al, in cultures of sulphate-reducing bacteria, is closely parallel to the behaviour of steel in similar circumstances. Hydrogenase-active strains of sulphate-reducing bacteria in batch cultures are effective cathodic depolarization agents for the anaerobic corrosion of Al; hydrogenase-negative organisms are inactive. Al corrosion rates a hundred times greater than those in sterile controls could occur, compared with 5 times for Fe in similar conditions. For Fe, some stimulation of corrosion is attributed to the accumulation of FeS precipitated during the organism growth. The corrosion of Al can be explained on the same mechanism as the anaerobic corrosion of steel. 14 ref. (35 1175.)

Section 2

11. Aluminum Corrosion Processes in Microbial Cultures. Gordon C. Blanchard and Charles R. Goucher. Electrochem. Technol., Nov.-Dec. 1967, 5, (11-12), 556-558.

The results of a recent study on the effects of nitrate and nitrite on the corrosion of Al and mild steel by CaCl_2 solutions and by microorganisms are presented. The comparative corrosivity to 7075 Al alloy by microbial products is shown. The corrosion of mild steel by bacteria cultures is similarly shown. Results indicate that, if corrosion in a medium of neutral pH in the absence of bacteria is considered, NO_3 is a good inhibitor of Al corrosion and, depending on concentration, will either accelerate the effects or produce no effect on mild steel corrosion. Nitrite is a good inhibitor of mild steel corrosion and, depending on concentration, either has no effect or accelerates Al corrosion. (35 023.)

Section 2

12. A New Technique for Estimating the Efficiency of Bacterial-Growth Inhibitors, and Its Application in Prevention of Biological Corrosion of Metals. E. Lagarde and A. Malderez. Corrosion Traitements, Protection, Finition, Aug.-Sept. 1967, 15, (6), 275-280 (in French).

The cultures are incubated in a thermostatically controlled chamber connected, via stopcocked lines, with an upper vessel containing a sterile medium which is introduced to the lower chamber when the culture-growth rate becomes logarithmic. This state is maintained by controlled addition of the medium, and balancing withdrawals with the H_2S evolved. Arrangements for inert-gas agitation and introduction of the inhibitors are incorporated, the efficiencies of the latter being determined from the H_2S curves. Tests with various sulphate-reducing bacteria and amine- and $Na_2Cr_2O_7$ -based inhibitors are discussed. (35 0688.)

Section 2

13. Microbiological Corrosion. Warren P. Iverson. Chem. Eng.,
23 Sept. 1968, 75, (2), 242, 244 (in English).

The nature of corrosion-producing microorganisms found in soil, the conditions under which they grow and their role in the corrosion of buried structures, such as pipelines, are described. The corrosive effects associated with other bacteria, algae and fungi are also noted. The probable role of some of these agents in the corrosion of Al fuel tanks is assessed. Three mechanisms of microbial corrosion are discussed: metabolic production of materials with an excess of H ions; formation of localized concentration gradients of electron acceptors--concentration cells; removal of H₂ and electrons through direct enzymatic action, or cathodic depolarization. Inhibitors, coatings, cathodic protection and placement of structures in a bed of well drained gravel as methods of preventing or controlling microbial corrosion are evaluated.

(35 0025)

621 <

Section 2

14. **The Biodeterioration of Materials: An Appraisal.**
H. J. Hueck. (Proc. Internat. Symposium on Biodeterioration of Materials. Southampton, Sept. 1968, 1969, 6-12 (Met. A., 6905-72 0075) (in English).

A historical introduction is followed by a systematic approach to biodeterioration and a classification of the processes involved. Corrosion of Fe by sulphate-reducing bacteria, fouling of ship's hulls by barnacles and algae, and the clogging of pipelines by iron bacteria are among the subjects discussed. 16 ref.

(35 0602.)

Section 2

15. Mechanisms of Microbial Corrosion. Warren P. Iverson.
(Proc. Internat. Symposium on) Biodeterioration of Materials,
Southampton, Sept. 1968, 1969, 28-43 (Met.A., 6905-72 0075)
(in English).

The mechanisms by which micro-organisms may enhance corrosion through the formation of corrosion products, O concentration cells, and cathodic depolarization are surveyed. 66 ref. (35 0602.)

Section 2

16. Microbiological Corrosion. Warren P. Iverson. Corrosion Prevention and Control, Feb. 1969; 16, (1), 15-19 (in English)

Characteristics of aerobic and anaerobic bacteria capable of producing H_2SO_4 or sulphide environments are reviewed, together with cathodic-polarization and other mechanisms involved. Protective measures are discussed. 46 ref. (35 0489.)

Section 2

17. Microbiological Aspects of Metal Corrosion. John McDougall.
Australasian Corros. Eng., Nov. 1969, 13, (11), 13-16 (in English).

Microbiological aspects of metal corrosion are considered with particular ref. to the species of bacteria and their environment. Corrosion of ferrous and non-ferrous materials is discussed.

(35 0595.)

Section 2

18. Microbiological Corrosion. Ted A. Graek. Proceedings 2nd Western States Corrosion Seminar, 1968, 245-248 (Met. A., 7001-72 0003) (in English).

Corrosion caused by micro-organisms can be classified as that due to microbiological accumulations, that caused by reduction in the pH of the water and that caused by the action of sulfate-reducing bacteria. The presence of the bacteria can be detected by inoculating water samples into various kinds of culture media and examining the water microscopically. Control mechanisms include the use of biocides to kill microbiological growth, of filters to physically remove the organisms, of Cl for the oxidation of organic matter and of plastic coated or cement coated pipe.

(35 0054.)

Section 2

19. Microbiological Corrosion of Aluminum. H.G. Hedrick.
Mater. Prot., Jan. 1970, 9, (1), 27-31 (in English).

Microbiological corrosion of Al alloys becomes significant when structural components, such as aircraft fuel tanks, are corroded. Experiments on four aircraft structural Al alloys exposed to microbiological attack are described, and results of investigations to determine the effects of microbiological corrosion on Al alloys and to explain one postulated mechanism are presented. The alloys included four series, 7178-T651, 2024-T351, 7079-T651 and 7075-T651. 28 ref.

(35 0468.)

Section 2

20. The Oxidation of Aqueous Ferrous Sulphate by Thiobacillus Ferrooxidans. D. G. Macdonald and R. H. Clark. Can. J. Chem. Eng., Dec. 1970, 48, (6), 669-676 (in English).

The Fe oxidizing bacteria, thiobacillus ferro-oxidans, were grown in an aqueous ferrous sulphate solution in batch and continuous culture vessels and the dependency of the specific growth rate on ferrous sulphate concentration, temp. and pH are shown. The effect of carbon dioxide concentration on cell growth is also considered. The phenomenon of wall growth is shown to be an important characteristic of the organism which must be considered in any batch or continuous culture experiments. The role that it may have in the biological leaching process is also indicated. 26 ref.

(35 0927.)

Section 2

21. Role of Metallic-Ion Concentrating Micro-Organisms in the Corrosion of Metals. W. B. Engel. Proc. 25th Conf., Nat. Assoc. Corros. Eng., 1970, 588-596 (Met. A., 7012-72 0279) (in English).

Interactions and relationships of specific metals (2024 Al, 7075 Al, Mg, Cu and Ti) with bacteria isolated from aircraft fuel tanks and other related environments were investigated. Objectives of investigation were to determine if these micro-organisms could corrode different metallic cations. A survey was conducted with these organisms to determine the effect 20 metals had on interfacial pellicle formation and the degree of concentration of these metal ions at the fuel/water interface. Physiochemical changes produced by these pellicles that could lead to a corrosive environment were investigated. Study of several metal ions was made to determine their rate of concentration in a minimal and kerosine/mineral salt medium by pellicle and nonpellicle cells. 30 ref.

(35 1396.)

Section 2

22. Sea-Floor Microbiological Corrosion. D. B. Hunter and E. C. Fischer. Proc. 25th Conf., Nat. Assoc. Corros. Eng., 1970, 597-608 (Met. A., (in ENGLISH)).

Basic studies on the physiology of marine micro-organisms isolated from deep ocean sediments indicate that anomalies in the corrosive properties of the ocean floor may be the result of metabolic activity. Analyses of sediment cores show the presence of an average bacterial population of 10 million organisms/gram of sediment. Isolated pure cultures of these bacteria demonstrate the ability to produce H_2S gas, as well as anerobic environments at the sediment/water interface. When cultured at high hydrostatic pressures, O uptake is increased along with increases in the biological activity of environmental O.

(35 1397.)

Section 2

23. Microbiological Corrosion of Aluminum Alloys. H.G. Hedrick.
Proc. 25th Conf., Nat. Assoc. Corros. Eng., 1970, 609-619
(Met. A., 7012-72 0279) (in English).

Presents results of investigations to explain the mechanism of microbiological corrosion of Al alloys (7178, 2024, 7079, 1100, and 7075). The results of studies of examining microbial utilization of minor metal constituents in these alloys are reviewed. Experiments on four alloys exposed to microbial action are described. A hypothesis on the role of Mg and Zn is explored. Results of experiments to support the hypothesis that the progress and extent of microbiological corrosion of Al alloys are functions of metabolic requirements for pure metals are presented. 27 ref.

(35 1398.)

111

Section 2

24. Anaerobic Corrosion of Mild Steel by Desulfovibrio. Warren P. Iverson. Proc. 25th Conf., Nat. Assoc. Corros. Eng., 1970, 620-623 (Met. A., 7012-72 0279) (in English).

Brief review of cathodic depolarization theory of anaerobic corrosion by sulfate reducers is presented. Evidence indicates that cathodic depolarization of steel by desulfovibrio does occur. Phosphate appears to be the final electron acceptor as indicated by formation of iron phosphide by desulfovibrio growing in contact with steel. The low rates of corrosion by desulfovibrio do not appear to account for the extensive anaerobic corrosion found in the field. Other possible mechanisms are discussed. 32 ref.

(35 1399.)

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Section 2

25. **Microbial Corrosion of Buried and Immersed Metal.** J. D. Miller and A. K. Tiller. Microbial Aspects of Metallurgy, 1970, 61-105 (Met.A., 7109-72 0197) (in English).

Sulphate-reducing bacteria can cause severe corrosion of unprotected ferrous metal underground structures, particularly in wet clay soils of about neutral pH. The problem is minimized by using noncorroding materials, constructing nonaggressive surroundings such as chalk or sand, using biocides, employing cathodic protection or applying protective coatings. Detailed information is given on the mechanism of anaerobic corrosion, the characteristics which produce aggressive bacterial action and polarization curves for steels exposed to these soils. An appendix gives a microbiological examination method for detecting sulphate reducing bacteria in soil or water.

48 ref.

(35 1109.)

Section 2

26. Corrosion of Mild Steel by Nitrate Reducing Bacteria. D. D. Mara and D.J.A. Williams. Chem. and Ind., 22 May 1971, (21), 566-567 (in English).

Anaerobic corrosion by hydrogenase-positive nitrate-reducing bacteria was studied by exposure of mild-steel specimens to a semi-continuous culture of *Escherichia coli* under N at 25°C. Corrosion rates were similar to those reported for sulphate-reducing bacteria under the same conditions, and were relatable with the nitrate reduction. The product was a loose flocculent film easily removed by washing. Control experiments with a hydrogenase-negative denitrifying organism showed negligible attack.

(35 1147.)

1147

Section 2

27. Clapp (William F.) Labs., Inc., Duxbury, Mass. ANAEROBIC BACTERIAL CORROSION OF METAL IN SEA WATER AT ELEVATED HYDROSTATIC PRESSURES Final Report, 1 May 1968 - 31 May 1971. Charles A. Willingham 31 Jul. 1971 30 p refs (Contract N00014-68-C-0365) (AD-727221. Rept-14254; NR-137-770) Avail: NTIS CSCL 11/6

The effects of hydrostatic pressures on the anaerobic corrosion of ingot iron, E.C. Grade aluminum, and 316-type stainless steel by three marine sulfate-reducing bacterial cultures were examined. Depending upon the period of exposure, all three cultures were capable of producing a greater amount of iron corrosion at 200 atmospheres than at one atmosphere. At 20 C, all cultures were sterilized by a hydrostatic pressure of 600 atmospheres, although incubation of one culture (BS-1) at 40 degrees allowed limited growth and corrosion of iron at this pressure. Increase in the amount of available nutrient medium in a comparable pressure system produced a significant increase in the corrosion of iron. Weight loss of aluminum and stainless steel specimens was little affected by the presence of a bacterial system. Aggressiveness of all three cultures to iron at one and 200 atmospheres when molecular hydrogen was present in the bacterial/metal system was significantly reduced. Little change in any corrosion of aluminum or stainless steel was noted when also exposed to a system with hydrogen. Interaction of the hydrogen present in the culture medium with the iron surface to produce sufficient atomic hydrogen for bacterial metabolism was indicated as the cause of unusually low corrosion rates but good bacterial growth. Type of corrosion attack on iron was most often uniform; when corrosion of aluminum or stainless steel occurred, attack was a shallow, pitting type. No intergranular cracking type of corrosion was observed.

(N72-12474#)

Section 3

STRESS CORROSION

1. **A STUDY OF THE MECHANISMS OF STRESS CORROSION CRACKING IN THE IRON-NICKEL-CHROMIUM ALLOY SYSTEM IN CHLORIDE ENVIRONMENTS.**
Quarterly Report, December 1, 1969-March 31, 1970. Staehle, R. W.
(Ohio State Univ. Research Foundation, Columbus). 24 Jun 1970.
Contract AT(11-1)-2069. 62p Dep. NTIS.

Results of studies are reported on work using circulating autoclaves, weight loss in boiling $MgCl_2$ solutions, transient dissolution, morphology of sensitized stainless steel, and work on the partial current dissolution of Fe-Cr-Ni alloys. Results of autoclave studies show that the performance of the higher nickel alloys continued to improve as the $Cl^- + O_2$ is lowered into the 1 to 5 ppm range. At 10 ppm $Cl^- + 10$ ppm O_2 Incoloy 800 cracks easily; at concentrations below this cracking is progressively less likely. Weight loss data for 51 alloys exposed up to $154^\circ C$ were carried out for six months. In general, the Fe-Cr-Ni base alloys exhibit weight loss behavior typical of film covered materials where the exponent of time in $\Delta w = at^n$ is in the range of 0.05 to 0.40. Only the alloys containing the platinum element exhibit higher exponents. Grains from sensitized stainless steels were examined in the scanning electron microscope after they had been removed by chemical attack. The results defined the general distribution of chromium-depleted regions. Work on the time-dependence of reactions for film growth in acid and neutral solution demonstrated that certain portions of the kinetic behavior follow well-known inverse logarithm behavior. Results of the studies on Fe-Ni electrodes show that the specific rate constants for Fe and Ni dissolution in a given alloy are the same, providing the alloys are dissolving in the activated region of potentials where Tafel behavior applies.

Section 3

2. **Intergranular Stress-Corrosion Resistance of Austenitic Stainless Steels in Water-Oxygen Environments: Accelerated Test Procedure.**
F. E. Wilde and J. E. Weber. Brit. Corrosion J., Jan. 1969, 4, (1), 42-46 (in English). (x).

A study to distinguish between the influence of gross plastic deformation and heat-treatment on the intergranular stress-corrosion cracking behaviour of a variety of austenitic stainless steels using 100 ppm dissolved O as an accelerator, is described. A system for studying the stress-corrosion behaviour of materials, loaded in uniaxial tension in water at 289°C, is described. The experimental variables studied included heat-treatment, C content, and alloy composition. Results for a number of steels are presented. Solution annealed material with no plastic deformation other than that associated with stressing to yield showed no evidence of intergranular stress corrosion cracking after exposure to water at 289°C + 100 ppm O; and the cracking is not promoted by the presence of oxidizing cations, e.g. Cr⁶⁺. Sensitized austenitic materials (C 0.03 wt.-%) exhibit intergranular stress corrosion cracking in the above environment even in the presence of high Si content, and C not Si and P controls susceptibility. Duplex alloys (ferrite + austenite) are immune to the corrosion even after heat-treatment corresponding to sensitization in austenitic materials, and immunity is independent of the nature of the ferrite stabilizer employed. Effects in welded sections are considered. 21 ref. (35 0487.)

Section 3

3. **Effect of Some Environmental Conditions on Stress Corrosion Behavior of Ni-Cr-Fe Alloys in Pressurized Water.** H. R. Copson and G. Economy. Corrosion, Mar. 1968, 24, (3), 55-65 (in English).

Stress corrosion tests have been run in pressurized water at 600 F. (316 C.) using double U-bends and double bent beam specimens. The tests were conducted in autoclaves for up to 3000 hr. The composition of the gas phase above the water and the pH were controlled. The materials tested were commercial heats of Inconel 600, Inconel 625, Incoloy 800 and stainless steel 304, 304L and 347; some Inconel alloy 600 weldments; and some experimental laboratory compositions. Several heat treatments were included. No attack was observed in specimens stressed at 90% of room temperature yield strength. No attack was observed when H₂ constituted the gas phase. No attack was observed when crevices were absent. The combined action of a high stress, a high O₂ concentration and a crevice did promote intergranular attack and cracking in the crevice area in all alloys tested. The severity of the attack increased with the O₂ concentration. Many specimens heated for 2 hr at 1250 F. (655 C.) showed deeper attack than annealed specimens. Solution heat treatment, low C content or the presence of carbide stabilizers did not eliminate the attack. The behavior of weld metal deposits was not significantly different from that of base plate. Variations in composition within the normal range of alloy 600 were unimportant. Differences in behavior among the several alloys tested were minor compared with those from environmental effects. 30 ref. (35 1032.)

Section 3

4. **Effects of Composition on the Susceptibility of Austenitic Stainless Steels to Stress Corrosion Cracking in Chloride-containing High Temperature Water.-I. Stress-Corrosion Cracking of the AISI 304, 304 L, 316 and 347 Steels.** Goro Ito, Tadao Ishihara, and Yoshihiko Shimizu. Trans. Nat. Research Inst. Metals (Japan), 1967, 9. (2), 87-94 (in English).

A comparison was made of susceptibility of several commercial austenitic stainless steels to stress-corrosion cracking in high-temp. water contg. a low concn. of Chloride. The effects of dissolved O or Chloride ion on the crack formation were also studied, with particular ref. to the relationship between stress-corrosion cracking and pitting corrosion. Soln.-treated type 316 steel was most resistant to cracking at temp. of 300 and 350°C. The susceptibility of the other steels was almost the same. The sensitizing heat-treatment markedly increased the susceptibility of all the steels to cracking, with the 316 type again being the most resistant. It was demonstrated that pitting corrosion is a cause of crack initiation, and that the more susceptible the steels are to cracking the more liable they are to pitting. Crackings in boiling 42% MgCl₂ soln. were always transgranular and initiated from a pit, in contrast to the high-temp. water tests.

11 ref. - (35 008.)

Section 3

5. **STRESS-CORROSION CRACKING OF AUSTENITIC STAINLESS STEELS IN CHLORIDE SOLUTIONS.** Takaaki Shimose, Akira Takamura, Kenji Mori and Kazutoshi Shimogori. Jap Inst Metals Trans, v 6, no 2, Apr. 1965, p 83-87.

Investigations were carried out to determine the effects of stress, chloride concentration, temperature and O_2 on the stress-corrosion cracking susceptibility of 18-8 and 18-13 Mo stainless steels. The tests were made using autoclaves with the chloride-ion concentration between 0 and 300,000 ppm at 132 to 250C. The stress-corrosion cracking susceptibility was found to increase with an increase of the chloride-ion concentration and/or temperature. At temperatures below 150 C., no stress-corrosion cracking was observed except in boiling 42% $MgCl_2$ solutions. Above 180 C., the susceptibility was greater in the vapor phase than in the liquid phase. In air atmosphere, the 18-8 stainless cracked at 180 C. and at 30,000 ppm chloride-ion concentration in the liquid phase and at 30 ppm in the vapor phase but at 250 C. it cracked at 10 ppm in the vapor phase. The addition of O_2 into the autoclaves promoted stress-corrosion cracking; at 180 C. the 18-8 stainless steel cracked at 200 ppm chloride-ion concentration in the liquid phase and at 10 ppm in the vapor phase. The 18-13 Mo stainless steel was observed to be more susceptible than the 18-8 steel in the vapor phase and less susceptible in the liquid phase in dilute chloride solutions but in the boiling 42% $MgCl_2$ solution both fields were almost equally susceptible. These results suggest the presence of definitive threshold stresses for the stress-corrosion cracking which depend on the chemical composition and corrosive environment such as temperature and chloride and O_2 content. It was also observed that in dilute chloride solutions, all cracks originated in pits formed in both vapor and liquid phases and that a complete exclusion of O_2 eliminates the occurrence of stress-corrosion cracking as well as pitting corrosion. 5 ref.

(M18--16420.)

Section 3

6. PROBLEMS AND SOLUTIONS OF AGGRESSIVE WATERS IN COOLERS AND CONDENSERS.
R. H. Banfield. Corrosion Tech, v 12, no 5, May 1965, p 15-18.

The results of an investigation of failure in Al "once-through" coolers are discussed and the effects of the various chemicals in the water outlined. Al coolers clad with Al-1% Zn on the water side also failed due to the effects of galvanic action, cathodic protection proving inadequate though sprayed-on Zn coatings gave increased protection. Stress-corrosion cracking occurred in austenitic steel coolers necessitating replacement by mild steel and the use of soft water with slight acidity but low chloride content. Corrosion was still observed but was minimized by addition of nonnodular calcium carbonate scale plus periodical mechanical reaming out of the tubes. 6 ref. (M18--25973.)

Section 3

7. (Pamphlet) STRESS-CORROSION CRACKING OF ALUMINUM ALLOYS. J. D. Jackson, and W. K. Boyd. Feb. 15, 1965, 13 p. DMIC Memo no 202, Battelle Memorial Institute, Columbus, Ohio 43201.

Al alloys most often associated with stress-corrosion cracking in actual service are 2014, 2024, 2219, 7075, 7079, and 7178 alloys and cast alloys of Ternalloy 7, 40E, 195 and 220 compositions. The tensile stresses necessary to initiate stress-corrosion cracking are either applied (static loads, press fits or fasteners) or residual (from heat treatment or machining). The prevention of failure requires a basic knowledge of the phenomenon and of the relative resistance of the various alloys and the effect of grain structure on cracking susceptibility. Application of this knowledge and use of good practices in heat treatment, working, forming, machining and design can reduce the incidence of stress corrosion. The application of compressive stresses, as by shot peening, or the use of coatings offers an effective means of minimizing or at least delaying stress-corrosion cracking. Laboratory investigations of stress-corrosion cracking often involve C-ring or tensile specimens exposed by alternate immersion in 3-1/2% NaCl solution. 23 ref.

(M18--26171.)

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Section 3

8. STRESS CORROSION CRACK PROPAGATION IN STAINLESS STEEL. L. Fairman.
Iron Steel, v 38, no 8, July 1965, p 371-375.

A research survey on nature, cause and mechanism of propagation of stress corrosion cracks in austenitic stainless steels. Published theories, beginning with 1944, are reported and compared. Methods of stress-corrosion testing and applicable equipment are included. 47 ref. (M18--31605.)

Section 3

9. CORROSION AND STRESS CORROSION OF 5000-SERIES Al ALLOYS IN MARINE ENVIRONMENTS. R. B. Niederberger, J. L. Basil and G. T. Bedford. Corrosion, v 22, no 3, Mar. 1966, p 68-73.

Studies involving sea water exposure were conducted with plain and welded samples of alloys 5083, 5086, 5154, 5356 and 5456. Several tempers and sheet gages were included, with exposure times up to five years for stressed and seven years for unstressed samples. General corrosion damage was mild, characterized by scattered shallow pitting. No stress corrosion cracking was encountered in standard mill tempers. However, sensitization of materials by holding one week at 212 F. to simulate long-time natural aging did result in stress-corrosion cracking at the weld fusion line of welded and stressed 5456-H24 samples within one year. Unstressed, sensitized samples of the same alloy showed no more than normal scattered pitting.

5 ref. (M18--43252.)

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Section 3

10. (Japanese) STRESS-CORROSION CRACKING OF AUSTENITIC STAINLESS STEELS IN HIGH-TEMPERATURE WATER. Masaomi Okazaki, Shoji Onishi, Jun Nakamura and Katsuyasu Fujino. Jap Inst Metals J, v 29, no 4, 1965, p 340-345.

Austenitic stainless steels have been generally used to build atomic reactors. These steels can suffer stress-corrosion cracking under certain conditions of stress and corrosion environment. Marine atomic reactors are especially in danger of hastened occurrence of stress-corrosion cracking as a result of the repetitious wetting and drying of the reactor wall by cooling water. Various autoclave tests established the following: wetting and drying of specimens accelerates the occurrence of stress-corrosion cracking; additions of Nb and solution treatment reduce the corrosion rate; and dissolved oxygen, chloride ions of about 5 ppm in water and surface contamination of the specimen augment stress-corrosion cracking. 5 ref. (M18--49375.)

Section 3

11. (Pamphlet) FRACTURE BEHAVIOR OF TITANIUM ALLOYS IN AQUEOUS ENVIRONMENT. N. G. Feige and T. Murphy. ASM Tech Rep no W 6-3.2, 1966, 21 p. American Society for Metals, Metals Part, Ohio.

Cantilever-beam, low cycle-high strain, three-point-slow-bend and four-point-slow-bend fatigue tests have been used to show the reduced fracture toughness of some Ti alloys in aqueous environments. There is a threshold level of strain which must be imposed on the notch area to initiate the rupture of the oxide film and allow exposure of metal for chemical reaction with the environment. The level is the energy to activate slip in the metal lattice. The mode of fracture determines the susceptibility of reaction of material with the environment. The attack can appear in the presence of an aqueous environment and can be modified by the addition of known inhibitors including an oxidizing agent, a reducing agent, neutral salts, a halide and a base. Both anodic and cathodic currents, when externally applied, affect the initiation and, to a limited extent, the propagation of cracking. Susceptibility to this stress corrosion can be eliminated by prevention of any one of the steps in the crack-formation process. Prevention of film rupture can best be approached by techniques that prevent large blocks of atoms from slipping and rupturing the protective film. In the case of Ti-7Al-2Cb-1Ta and Ti-8Al-1Mo-1V, heat treatments improve cross slip and reduce compound precipitation. Cathodic applied currents can be used to prevent dissolution of the metal when exposed and immediately reform the oxide film. The most desirable approach would be the addition of minor alloy modifications which would stop the corrosion tunneling reaction without

Section 3

11. (cont.)

degradation of mechanical properties. Nonsusceptible Ti alloys such as Ti-6Al-4V, Ti-4Al-3Mo-1V and Ti-7Al-2-1/2 Mo are available and minor process changes can be imposed to reduce susceptibility in other alloy systems such as Ti-8Al-1Mo-1V. 14 ref.

(M17--46707.)

87<

Section 3

12. (French) STRESS CORROSION OF AUSTENITIC STAINLESS STEELS.
J. Hochmann. Mec Elec, no 191, Sept. 1965, p 79-84.

Describes theories of stress corrosion, conditions under which it occurs in austenitic stainless and methods of prevention particularly in chloride solutions. Discusses superiority in this respect of a sand-blasted surface over a mechanically polished or pickled surface, both in grades in which austenite is very stable (18% Cr-20% Ni) and in those having a metastable austenite (18% Cr-10% Ni), and the role of compressive stress assumed created by sandblasting. Effects of workhardening on resistance to stress corrosion are noted. Increase in resistance afforded by raising Ni content to 45-50% and by massive additions of Si (3-4% Si) is discussed. Corrosion behavior is reported of two steels especially developed for corrosive service conditions, one an austenoferritic grade, Uranus 50, containing 0.05% C, 0.5-1.0% Mn, 0.5-1.0% Si, 20% Cr, 8% Ni, 2.5% Mo and 1.5% Cu; and Uranus S, containing about 0.02% C, 1.5% Mn, 17.5% Cr, 14% Ni and 3.5% Si. Examines hypotheses concerning reasons for beneficial effect of Si. 37 ref.

(M18--45165.)

585

Section 3

13. (Japanese) EFFECTS OF CHLORIDE ION AND DISSOLVED OXYGEN ON STRESS-CORROSION CRACKING OF AUSTENITIC STAINLESS STEEL IN HIGH-TEMPERATURE WATER. Kazumu Sugimoto, Tatsuo Maekawa and Masaru Kagawa. Corrosion Eng. v 14, no 4, Apr. 1965, p 11-14.

An extensive study was conducted over wide ranges of chloride and O_2 concentrations. Corrosion cracking of AISI Type 304, 304L and 347 steels was detected at considerable contents of O_2 (0.1 ppm) and Cl^- (100 ppm) in 300 hr at 300 C. However, corrosion cracking of AISI 316 steel was not detected at higher contents in 300 hr. Cracking susceptibility of Type 304 steel was increased by sensitizing heat treatment. 5 ref.

(M18--35569.)

Section 3

14. Stress-Corrosion Cracking of Welded Aluminum Alloys. Fred H. Meyer, Jr. and Richard Turley. Proceedings of the First Joint Aerospace and Marine Corrosion Technology Seminar, 1969, 44-47 (Met. A., 7006-72 0137) (in English).

Stress-corrosion susceptibility tests were carried out on seven Al alloys, 2014-T6, 2021-T8E31, 2024-T81, 2218-T87, X7002-T6, 7039-T64, and 7106-T6. Specimens were tested in synthetic seawater either unwelded, as-welded or after postweld treatments involving only artificial aging or solution heat treatment and artificial aging. Results show that all seven alloys have satisfactory stress-corrosion resistance in the basic unwelded condition. In the as-welded temper, all alloys are satisfactory except 2014-T6, 2021-T8E31 and 2024-T81, which should be avoided. 2014-T6 and 7106-T6 are not satisfactory after postweld artificial aging, and 2014-T6 sheet and plate, 2021-T8E31 plate, 2219-T87 sheet, 7039-R64 sheet and plate and 7106-T6 are not recommended in the solution heat treated and artificially aged postweld condition. -M.J.R. (35 0625.)

Section 3

15. (Japanese) STRESS CORROSION CRACKING OF STAINLESS STEEL HEAT EXCHANGER TUBES IN HIGH TEMPERATURE WATER. Shoji Onishi and Jun Nakamura. Corrosion Eng. v 14, no 8. Aug. 1965, p 8-13.

Austenitic stainless steels are generally used for atomic reactor heat exchangers, but stress corrosion cracking under condition of high-temperature water (650 to 1100 C.) has been reported. The results obtained from some experiments to study the stress corrosion cracking of stainless steel heat exchangers in high-temperature water are: cold drawing of stainless steel tubes and existence of chloride ion and dissolved oxygen in water accelerate the occurrence of stress corrosion cracking; seal welding by Tig method between the tube plates and the tubes accelerates the crack initiation; solution treatment of cold drawn stainless steel tubes decreases the sensitivity to stress cracking; the eddy current method is effective for nondestructive inspection of stress-corrosion cracking in expanded parts of stainless steel heat exchangers. 5 ref. (M18--47817.)

Section 3

16. STRESS-CORROSION CRACKING OF ALUMINUM ALLOYS. F. H. Haynie and W. K. Boyd. IMIC Rep 228, July 1, 1966, 44 p.

The mechanisms involved and the theory of stress corrosion cracking of Al alloys are discussed. Experimental techniques described include specimen configuration and loading of tubing and forged cylinders, plate, bar, rod and welded specimens. The following corrosion tests are applicable to Al alloys: alternate immersion in NaCl, salt spray exposures, continuous immersion, electrolytic tests and atmospheric exposure. Stress corrosion cracking behavior of wrought strain-hardened, 2XXX-wrought heat treatable, 6XXX-wrought heat treatable and 7XXX-wrought heat treatable Al alloys is discussed. Stress corrosion cracking may be prevented by correct material selection, reduction of tensile stresses, and protection of material from corrosive environment. 125 ref. (M18--54987.)

Section 3

17. SENSITIVITY TO STRESS CORROSION AND INTERGRANULAR ATTACK OF HIGH-NICKEL AUSTENITIC ALLOYS. H. Coriou, L. Grall, C. Mahieu and M. Pelas. Corrosion, v 22, no 10, Oct. 1966, p 280-290.

High-Ni alloys and various analyses of Types 304L, 316 and 347 as well as European commercial steels of similar analysis were tested in high temperature (350 C.) demineralized water and steam (650 C.) for varying periods up to five months. High-Ni austenites were susceptible to intergranular stress-corrosion cracking which was different from the transgranular cracking known to occur in austenitic stainless steels. Tests in 650 C. steam show cracking of high-Ni alloys increases with slight plastic deformation while 18-10 and 20-25 stainless steels were unaffected. Severe intergranular attack was observed in the high-Ni alloys subjected to highly oxidizing acid media. It is possible that a structural element in these susceptible alloys is responsible. Pb or oxygen contamination of test water may have contributed to results reported, which are at variance with numerous test and service results of the same material in like environments. Pb could not be detected in experiments performed later to check influence of this contaminant; oxygen content was 0.003 mg/l, equal to the sensitivity of the analytical method. 27 ref. (M18--58513.)

Section 3

18. Effect of Contaminants on Resistance to Stress Corrosion Cracking of Ni-Cr Alloy 600 in Pressurized Water, Copson, H. R., and Dean, S. W., Corrosion, Vol 21, No. 1 (Jan. 1965).

Numerous tests have shown that Alloy 600, a 76 nickel-15 chromium-7 iron alloy, has excellent corrosion resistance in pressurized high temperature water. The present tests were undertaken to determine the influence of possible contaminants in 600°F (316°C) pressurized water on corrosion behavior, using both single and double U-bend specimens. The double U-bend provided a combined stress and crevice specimen. Contaminants were sodium fluoride, air, lead powder, lead oxide, a petroleum hydrocarbon, and a mixture of lead powder and the hydrocarbon. Contamination and aeration were much in excess of any condition likely to be encountered in pressurized water reactors. Under certain conditions, some contaminants induced stress corrosion cracking.

Section 3

19. Influence of Environment on Crack Propagation Characteristics of High-Strength Aluminum Alloys, J. H. Mulherin. Paper from 'Stress Corrosion Testing', 1967, 66-83.

The stress corrosion behavior of four alloys of Al has been investigated. Using cantilever-loaded fatigue-cracked specimens, the crack propagation characteristics of 7178-T6, 2014-T6, 2024-T351, and 7075-T6 Al alloys were determined. The results are interpreted in terms of fracture mechanics parameters. Where susceptibility to stress corrosion exists, the rate of crack propagation is dependent upon stress intensity and environment. A method is also presented to obtain threshold values of stress intensity to stress corrosion attack with minimal experimental effort. The aqueous environments used were immersion in distilled water, immersion in 3-1/2% sodium chloride, and alternate immersion in salt solution and air. The cantilever technique is evaluated and the results compared with other testing methods.

7 ref.

(35 0299.)

Section 3

20. Critical Species in Stress Corrosion Phenomena. E. N. Pugh and A. R. C. Westwood. Paper from 'Stress Corrosion Testing', 1967, 228-247.

Consideration has been given to the identification of the critical species in several stress corrosion systems. It is shown that in the α -brass/ aqueous ammonia system, cupric complex ions of the type $\text{Cu}(\text{NH}_3)_n^{2+}$ play a controlling role in the cracking process. Complex ions are also found to constitute the critical species in the embrittlement of silver chloride in certain aqueous environments. In the case of materials such as stainless steels and Mg and Al alloys, which undergo stress corrosion cracking in chloride environments, the critical species may be the chloride ion itself or metal-chloride complexes. Attention is given to both the role of the critical species in the mechanisms of failure and the practical significance of these findings to stress corrosion testing. It is suggested that more attention to the chemistry of environments which cause stress corrosion cracking, with particular regard to the identification of the critical species, could be of significant practical value. 51 ref. (35 0309.)

Section 3

21. Accelerated Crack Propagation (in Titanium) in Liquid Environments.
I. Roy Lane, Jr. Paper from 'Titanium for the Chemical Engineer',
1 Apr. 1968, 45-49 (Met. A., 6807-72 0068).

The use of Ti in a chemical environment should be based on a consideration of the susceptibility of the material to low stress-level crack propagation. These considerations include structural design, stresses in the structure, the environment to which it will be exposed, and the nature of the Ti alloy to be used. A curve of nominal bend stress to fracture vs time for Ti-7Al-2Cb-1Ta alloy, subjected to the cantilever-beam stress-corrosion in salt water, is plotted. The effect of notch acuity on the same alloy is shown. The effect of sheet thickness on the nominal breaking stress of Ti-8Al-1Mo-1V, Ti-4Al-3Mo-1V and Ti-6Al-4V alloy is compared. The Ti-Al effect on crack propagation in liquid environments is under study. The relationship between the formation of Ti_3Al and crack growth sensitivity in sea water is shown for quenched and aged Ti-Al alloys. 11 ref.

(35 0602.)

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Section 3

22. A Metallurgical Look at Stress Corrosion of Aluminum Alloys. Carson L. Brooks. Paper from 'Proceedings of the Air Force Materials Laboratory Conference on Corrosion of Military and Aerospace Equipment'. Nov. 1967, 738- 760 (Met. A., 6809-72 0102) (in English).

Stress corrosion of Al alloys may be defined as a complex interaction of sustained tension stress and corrosion attack resulting in cracking and premature brittle failure of an otherwise ductile material. The three factors which work in combination to cause failure are considered: a sustained tension stress, a proper combination of chemical environment, and a proper metallurgical condition of the metal. Various stress corrosion specimens, jigs and fixtures for testing are illustrated. Specific test data, showing highest sustained stress (ksi) at which test specimens of 2014-T6, 2024-T3 or T4, 2024-T6 or T8, 2219-T8, 7075-T6 or T73, 7079-T6, and 7178-T6, of different orientations, did not fail in a 3-1/2% NaCl alternate immersion test are listed.

(35 0827)

Section 3

23. A New Solution Potential Measurement for Predicting Stress Corrosion Performance of 2219 Aluminum Alloy Products. R. L. Horst, Jr., E. H. Hollingsworth, and W. King. Paper from 'Proceedings of the Air Force Materials Laboratory Conference on Corrosion of Military and Aerospace Equipment'. Nov. 1967, 762-797 (Met.A., 6809-72 0102) (in English).

A new electrochemical test for evaluating the stress corrosion performance of 2219 alloy products in the T851 and T87 tempers is described. The test requires only a simple measurement of solution potential in a mixture of abs. methyl alcohol and carbon tetrachloride. It is rapid, requiring less than 1 hr, and has the outstanding advantage of providing differences of nearly 200 mv between borderline and resistant conditions of stress corrosion susceptibility, as contrasted to differences of less than 20 mv in conventional NaCl-H₂O₂ solution. The test is suitable for routine use in a plant control laboratory to establish the stress corrosion performance of 2219 alloy products as either an alternate or supplement to the 30 day alternate immersion test in 3-1/2% sodium chloride solution. (35 0828.)

Section 3

24. Intergranular Stress Corrosion Cracking of Austenitic Stainless Steels in Oxygenated High Temperature Water, Armijo, J. S., Corrosion, Vol 24, No. 10, (1968).

A bellows arrangement for controlled high temperature, high pressure, stress corrosion tests of thick specimens has been used to study the intergranular stress corrosion cracking of Type 304 stainless steel and Incoloy 800 in oxygenated (100 ppm) 550°F (288°C) water. Complete intergranular fracture of sensitized Type 304 stainless steels occurs after 10.4 ± 4.4 hours exposure at stress levels of 14,000 to 17,000 psi. Cracking of Incoloy 800 requires longer exposures at high stress. Crevices are not necessary to initiate cracking of sensitized alloys.

Section 3

25. Corrosion Fatigue and Stress-Corrosion Cracking in Aqueous Environments. D. E. Piper, S. H. Smith, and R. V. Carter. Metals Eng. Quart., Aug. 1968, 8, (3), 50-63 (in English).

The primary objective of this study was to learn how to increase the crack-propagation resistance of high-strength alloys that are susceptible to stress-corrosion cracking and to rapid corrosion fatigue. The secondary objective was to learn how to use these alloys in structures intended for aerospace or oceanspace environments that require fail-safe design. The approach was through quantitative characterization tests based on the prior existence of a sharp crack which is the prerequisite for stress-corrosion cracking in Ti alloys. Test conditions were amenable to analysis by stress-intensity factor or linear elastic fracture mechanics methods. The investigation was concerned mainly with Ti alloys, although some steels and Al alloys were evaluated for purposes of comparison. The evaluation of crack propagation resistance was based on four types of testing: fracture toughness, both plane stress and plane strain; stress-corrosion cracking, both plane stress and plane strain; corrosion fatigue; and tensile property, to determine the validity of fracture data. 10 ref. (35 1120.)

Section 3

26. Metals and (Marine) Corrosion. B. F. Brown. Mach Design, 18 Jan. 1968, 40, (2), 165-173.

Requirements of materials for marine environments include strength, density, low cost, fabricability, procurability, and resistance to fracture and corrosion. The general problem of fracturing is divided into two groups: one, purely mechanical (fracture toughness based on monotonic loading, and fatigue); and a second group, which consists of corrosion fatigue, stress-corrosion cracking, and H₂ embrittlement. Fracture toughness experience with Ti-7Al-2½ Mo, Ti-6Al-2Mo and Ti-6Al-4V is shown. The fatigue crack growth rate and critical flaw size for D6AC, 18 Ni maraging, 12 Ni maraging, HY-130, HY-80, A302B, A201B, 4335 and T-1 steels were measured in air, with loads at the proportional limit and full stress reversal. The stress-corrosion cracking behavior of Ti-6Al-2Cb-1TA-0.8Mo, Ti-6Al-4V, Ti-6Al-2Mo and Ti-7Al-2½ Mo Ti alloys in salt water was determined. Stress is not a requisite for crevice corrosion, pitting, dealloying, and general corrosion by cracking in sea water. The comparative localized attack (crevice corrosion and pitting) of the following marine alloys as a function of sea water flow conditions is shown: 2219, 2014, 2024, 7178, 7079, 7075, 3003, 6061, X 7002, 5052, 5154, 1100, 5456, 5086, and 5083 Al alloys; 90/10 CuNi; 70/30 CuNi; Monel; Ni; stainless steels; Hastelloy C; and Ti. (35 0386.)

Section 3

27. Effect of Microstructure on the Fracture Properties of Titanium Alloys in Air and Salt Solution. R. E. Curtis and W. F. Spurr. ASM Trans. Quart., Mar 1968, 61, (1), 115-127.

The environmental fracture properties of Ti-6Al-4V and Ti-4Al-3Mo-1V have been related to microconstituents formed during commercial-type heat treatment operations. Titanium-6Al-4V containing equiaxed grains was susceptible to stress-corrosion cracking as demonstrated by extensive slow crack growth in 3.5% NaCl solution as well as by a low threshold for stress-corrosion cracking. Electron fractography and direct observation of secondary stress-corrosion cracks identified transgranular cleavage in the susceptible α phase. Treatments which transformed equiaxed α to an acicular morphology and which precipitated a large volume fraction of β increased the stress-corrosion threshold and fracture toughness. Thin-foil studies related the superior properties of the acicular morphology to a reduced α grain size and more effective dispersion of the ductile β phase. Differences in the fracture properties of Ti-4Al-3Mo-1V and Ti-6Al-4V are quantitatively related to the intensity of the stress field surrounding a dislocation pile-up. In Ti-6Al-4V, coplanar slip across large α grains can intensify the stress component normal to the fracture plane to a level which initiates transgranular cleavage cracks. The salt solution is thought to reduce the applied stress necessary to initiate the brittle failure by reducing the fracture stress. 27 ref.

(35 0517.)

Section 3

28. Effects of Heat Treating Environmental Conditions on the Stress-Corrosion Cracking Resistance of Several Titanium Alloys. D. G. Howe and R. J. Goode. Paper from 'Applications Related Phenomena in Titanium Alloys'. 1968, 189-201 (Met. A., 6809-72 0097) (in English)

The effects of heat treating environmental conditions on the stress corrosion cracking resistance of several Ti alloys have been studied: Ti-8Al-1Mo-1V, Ti-7Al-1Mo-1V, Ti-6Al-4V, and Ti-7Al-2.5Mo. Pre-cracked cantilever type specimens were heat treated in either an inert atmosphere or in vacuum and tested in a 3.5% salt water solution. A comparison of the stress intensity required for failure in salt water with that required for 'dry' mechanical fracture indicates that all of these alloys become essentially insensitive to the aqueous environment when vacuum solution-annealed and He gas-cooled. Similar heat treatments in A or He developed a wide range in level of stress corrosion cracking resistance for these alloys; the level was dependent upon the temperatures used. The environmental conditions associated with aging treatments at 1200°F. following the solution anneal appear to have little effect on the stress corrosion cracking resistance for these alloys; the level was dependent upon the temperatures used. Comparison of the stress corrosion cracking data with H₂ content before and after treatment indicates a close connection between stress corrosion cracking sensitivity and H₂ content in the material even at very low H₂ levels. These results suggest that H₂ contained in the metal may play an important role in the stress corrosion cracking process of Ti alloys. 8 ref.

(35 0736.)

Section 3

29. Corrosion of Aluminum in Water. Yoshio Yamazaki and Hiromitsu Harimoto. J. Metal Finishing Soc. Japan, 1969, 20, (2), 68-74 (in Japanese).

Oxide growth on 1100 Al in water at 30-100°C was examined. Boehmite and a thicker film of bayerite are initially formed below 84°C, but at higher temp. the film consists of boehmite only. The rate of film formation was a max. at 60°C although thicker bayerite films were formed at lower temp. Pitting was caused by compound particles in the Al substrate and the concentration cell formed by O dissolved in water. Pitting ceased when these compounds were separated from the substrate by dissolution of the substrate, so that all pits remained microscopic in size. Al was coloured black below 50°C owing to the roughness of the surface (caused by corrosion); white rings which developed around pits were attributed to the change in pH brought about by the pitting action. Many fine particles consisting of a secondary Al phase were observed on films formed at 60-80°C, and the specimen colour (brownish-grey) was attributed to these particles. (35 0841.)

Section 3

30. FRACTURE TOUGHNESS AND STRESS-CORROSION CRACKING OF SOME TITANIUM ALLOY WELDMENTS. Huber, R. W.; Goode, R. J.; Judy, R. W. Jr. (Naval Research Lab., Washington, D.C.). Weld. J. (N.Y.), 46: 439s-47s (Oct. 1967).

Fracture toughness and stress-corrosion cracking characteristics have been determined for a variety of 1 in. thick titanium alloy plates and welds. The drop-weight tear test (DWTT) and a bend-bar K_{Ic} test were used in the fracture toughness studies. A precracked cantilever bend specimen was used for the stress-corrosion cracking studies. The properties of as-deposited and heat-treated gas metal-arc, electron beam, and a few plasma arc welds are compared with those of as-received and heat-treated base metal plate of similar chemical composition. By comparing the DWTT and K_{Ic} results with a fracture toughness index diagram developed for 1 in. thick titanium alloy plate, predictions can be made of the amount of prefracture plastic strain required to cause the welds to fracture. The results of a study on the stress-corrosion cracking resistance of as deposited and heat-treated gas metal-arc welds and as-deposited electron beam welds in water containing 3.5% NaCl were also compared to those obtained for the base metal. The critical stress intensity parameter for stress-corrosion cracking to occur, K_{Isc} , was determined for as-deposited and heat-treated gas metal-arc welds and as-deposited electron beam welds in water containing 3.5% NaCl. The results indicate that for many alloys considerable differences in resistance can be expected between the base metal and the weldment. For some of the alloys, improvements were made in the stress-corrosion cracking resistance and fracture toughness of the weldments with postweld heat treatments or in some cases by heat treating the base metal prior to welding. Application of post-welding heat treating procedures to a

Section 3

30. (cont.)

large structure may be difficult; therefore, the fracture toughness and stress-corrosion cracking resistance of the as-deposited weld are considered highly critical aspects in the failure-safe design and fabrication of large, welded structures. (2619.)

1475

Section 3

31. CORROSION OF CARBON STEELS AND STAINLESS STEELS IN PRESSURIZED WATER AT HIGH TEMPERATURES. Special Report No. 9. Warzee, M.; Sonnen, C.; Berge, Ph. (Societe d'Etudes, de Recherches et d'Applications pour l'Industrie, Brussels (Belgium)). July 6, 1967. Contract 087-1-TEEB(RD). 40p. (EUR-3388). Dep. CFSTI.

Work performed under United States-Euratom Joint Research and Development Program.

A carbon steel grade and two stainless steel grades, AISI 410 and AISI 304, have been submitted to static and dynamic corrosion tests in pressurized water at 200 to 350°C. The effects of renewal and circulation of water, surface state of the metal, pH, and oxygen content of the solution were also examined. The oxidation mechanism of steels in high temperature pressurized water has been investigated. It may be observed that the oxides formed in this medium, either on Cr or Cr-Ni alloyed steels or on unalloyed steels, appear to be formed of a double layer; the outer layer may in some cases be composed of scattered crystals. These crystals are magnetite, whatever the steel grade, and develop from ferrous hydroxide in solution. The protective character of this external layer plays an important part in the corrosion kinetics of steels in water. The most severe corrosion cases have been observed under dynamic conditions, in constantly purified water at pH 7. Under these conditions, solubility of ferrous hydroxide is very high at the test temperatures, and its accumulation is prevented, by constant purification and by high flow rates, so that a deposit of magnetite on the surfaces submitted to corrosion is impossible. The increase of the pH up to 10, diminishing considerably the solubility of ferrous hydroxide, is favorable to deposition. The direct effects of temperature on the transformation of ferrous hydroxide into magnetite partially explains the fouling of fuel elements in PWR reactors.

Section 3

32. STRESS CORROSION AND HYDROGEN EMBRITTLEMENT PROPERTIES OF 17-4 PH IN 600°F WATERS. Suss, Henry (Knolls Atomic Power Lab., Schenectady, N.Y.). Apr. 1967. Contract W-31-109-eng-52. 64p. Dep. CFSTI.

Stressed 17-4 PH specimens were exposed for 700 days to 600°F hydrogen-ammoniated water and to (cyclic) hydrogen-ammoniated and aerated water. The susceptibility of the alloy aged at 900°F to stress corrosion in 600°F aerated water was confirmed. No such susceptibility was evident in the hydrogen-ammoniated water. The susceptibility of the 900°F aged material to hydrogen embrittlement in both water conditions was shown. This susceptibility was evident only with an applied stress of 165,000 psi for air melt and 100,000 psi for consumable electrode melt materials. No stress corrosion attack of the alloy aged at 1025°F or 1100°F in either environment was indicated. The one failure of each 1025°F aged and 1100°F aged, with applied stresses close to yield strength, has been attributed tentatively to hydrogen embrittlement. The consumable electrode heats appeared more susceptible to hydrogen embrittlement than air melt material. The alloy in the three aged conditions (900°F, 1025°F and 1100°F) was subject to secondary aging resulting in increase in hardness up to 4 R_C units and 20,000 psi in tensile strength--with some loss in ductility. Additional studies are recommended to establish the significance of the secondary aging on the mechanical, impact, stress corrosion, and hydrogen embrittlement properties of 17-4 alloy aged at 1025 and 1100°F.

Section 3

33. Stress Corrosion Properties of High Strength Precipitation Hardening Stainless Steels, Carter, C. S., Farwick, D. G., Ross, A. M. and Uchida, J. M., Corrosion, Vol 27, No. 5 (1971).

The plane-strain fracture toughness K_{Ic} and stress corrosion threshold K_{Isc} (in 3.5% aqueous sodium chloride solution) have been determined for the following high strength, precipitation hardening steels: 17-7 PH (RH 950, TH 1050), PH 15-7Mo (RH 950, TH 1050), AM 355 (SCT 850, SCT 1000), AM 362 (H 900, H 1000), AM 364 (H 850, H 950), 17-4 PH (H 900, H 1000), 15-5 PH air melted and vacuum melted (H 900, H 1000), PH 13-8Mo (H 950), and Custom 455 (H 950). Correlations of K_{Isc} with service performance and smooth-specimen test data are discussed.

Section 3

34. Use of Precracked Specimens in Stress Corrosion Testing of High Strength Aluminum Alloys, Hyatt, M. V., Corrosion, Vol 26, No.11, (1970)

Resistance to stress corrosion cracking of 10 high strength aluminum alloys in a variety of heat treatment conditions has been measured using precracked double cantilever beam (DCB) specimens. A new technique is described, and stress corrosion crack growth rates for the alloys tested are presented as a function of the plane-strain stress intensity K_I . Crack growth rates for alloys in the T3 and T6 tempers showed both K_I -independent and K_I -dependent behavior, whereas alloys in the more resistant tempers showed only K_I independent behavior over the K_I range studied. Double cantilever beam specimen data correlated with established trends from smooth specimens tested by alternate immersion in 3.5% NaCl solution. From the crack growth rate data and the speed and simplicity with which it is obtained, it is concluded that the DCB specimen will be highly useful for (1) comparing and rating alloys, (2) developing new alloys and heat treatments, (3) comparing the effects of environments, (4) achieving or ensuring product uniformity, and (5) studying mechanisms of cracking.

Section 3

35. A STUDY OF THE MECHANISM OF STRESS CORROSION CRACKING IN THE IRON-NICKEL-CHROMIUM ALLOY SYSTEM. Quarterly Report, March 17, 1966-June 16, 1966. Staehle, R. W. (Ohio State Univ. Research Foundation, Columbus). Aug. 30, 1966. Contract AT(11-1)-1319. 56p. Dep. mn. CFSTI \$3.00 cy. \$0.50 mn.

Stress corrosion cracking experiments are reported in dilute chloride environments at 500°F and in boiling MgCl₂ environments. Data are reported in terms of % unbroken vs time. Studies on effect of surface preparation are reported. Results of these studies show that various surface preparations greatly affect mean cracking times and can cause significant differences in data scatter. A study of the effect of alloy composition on annealing twin frequency was completed and final results show generally that increasing the Ni and Cr increases the frequency of annealing twins. Results are reported from a study of tensile properties as a function of temperature and alloy composition. A potentiokinetic polarization study was completed for the ternary Fe-Cr-Ni alloy system and results are summarized graphically. Figures presenting concepts in the theory of stress corrosion cracking are included.

1860 (COO-1319-42)

Section 3

36. CORRELATIONS BETWEEN SENSITIZATION AND STRESS CORROSION CRACKING OF 300 SERIES STAINLESS STEELS. Progress Report, January-March 31, 1964. R. C. Brayer, G. Zuromsky, and W. P. Chernock (Combustion Engineering, Inc. Nuclear Div., Windsor, Conn.). Apr. 1964. Work Performed under United States- Euratom Joint Research and Development Program. Contract AT(30-1)-3256. 34p. (EURAE-1073).

Tests of Types 304 and 347 stainless steel in both the solution-annealed and sensitized conditions were initiated in pressurized water containing 15 ppm dissolved oxygen and 25 ppm chloride ion. The unstressed samples indicated no attack after exposure to this environment for 180 hours at 190°C. These samples have been stressed to half the yield point and are in test under the same environmental conditions. Preparation of internally pressurized tubular samples is nearing completion.

24125 (CEND-3256-209)

Section 3

37. STRESS CORROSION CRACKING EVALUATION OF SEVERAL FERROUS AND NICKEL ALLOYS - Humphries, T. S. and Nelson, E. E. NASA Report No. TM X-64511, April 2, 1970.

The stress corrosion cracking characteristics of H-11 steel, Hastelloy C, Inconel 718, Waspaloy, music wire, and stainless steels Arde (cryoformed) low silicon 301, AISI 303, AISI 304, Armco 21-6-9 are described. Three types of specimens (round tensile, flat tensile, and C-ring) were employed and the main test environment was alternate immersion in 3.5 percent salt solution. The results of the test indicated that music wire, Hastelloy C, Inconel 718, Waspaloy, and stainless steels Arde 301, AISI 303, AISI 304, and Armco 21-6-9 in most forms and heat treat conditions are resistant to stress corrosion cracking in environments containing moist chlorides. However, another investigator has reported both service and laboratory stress corrosion cracking of cryoformed 301 stainless steel under different conditions. H-11 steel is susceptible to stress corrosion cracking in both moist chloride environments and semi-industrial atmospheres.

Section 3

38. Analysis of Stress Corrosion Testing of 2014-T651 Aluminum, Using Weibull Techniques. Mitchell O. Locks. Corrosion, Sept. 1971, 27, (9), 386-389 (in English).

The results are analysed of alternate immersion testing of samples of Type 2014-T651 Al in a 3.5% NaCl bath under 10000 lb/in² tensile stress, during approx. one month of continuous testing to determine the distribution of the times until the noticeable appearance of stress corrosion cracking (SCC). Weibull distributions were used because a wide range of failure phenomena could be covered. Five different groupings or broad categories of 2014-T651 Al specimens from three different manufacturers were used based upon the source (manufacturer), the specification to which the sheets were prepared, and the method of preparation. A wide variation of results occurred from one grouping to another. Characteristic lifetimes were as low as 70 h and as high as 1085 h. Both increasing failure rate phenomena, such as early failure of all specimens in a group, and decreasing failure rates were exhibited. 11 ref. -AA (35 0188.)

Section 3

39. Intergranular Corrosion and Corrosion Cracking of Stainless Steels in Aqueous Media. V. P. Pogodin, V. L. Bogoyavlensky and V.P. Sentyurev. 1970, Pp 422 (Met. A., 7103-72 0068) (Book.-Russian).

Intergranular corrosion and stress-corrosion behavior are reported for austenitic, ferritic and martensitic stainless steels in pure water, steam, and solutions of salts, acids and alkalis. The corrosion-testing conditions include those prevailing in the water- and steam-cooled reactors, power stations and in the chemical industry. Analyses are given of the basic factors affecting corrosion behavior, such as media composition, composition and structure of steels, heat treatment, stresses and radiation effects. Corrosion preventive methods are reviewed, including alloying the steels with elements such as C, Ti, Nb, Ta, Cr, Ni, N, Mo, Si, V, W, Mn and B. - A.F.S. (35 0304.)

Section 3

40. On the Existence of a Threshold Stress for Corrosion Cracking in Titanium Alloys in Salt Water. B. F. Brown, J. Mater., Dec. 1970, 5, (4), 786-791 (in English).

The reason for the uncertainty over the question of genuine threshold stress (or stress intensity) for propagation of a stress-corrosion crack is discussed. An experimental method is described from which electrochemical data provide evidence of the existence of such a threshold for Ti alloys. An experiment is described which confirms by an independent method the foregoing conclusion; the significance of the conclusion is discussed. -AA

(35 0494.)

Section 3

41. Preventing Stress Corrosion Failures in Susceptible Aluminum Alloys. H. L. Craig, Jr. and F. E. Loftin. Mater. Prot. Performance, Mar. 1971, 10, (3), 17-20 (in English).

Discusses the mechanism of stress-corrosion cracking, susceptibility factors and prevention of stress corrosion in Al alloys 2014, 2024, 2219, 5456, 7075, 7079 and 7178. Emphasis is placed on avoiding failure by approaches based on a knowledge of metallurgical structures, environment and stress distribution. -AA

(35 0912.)

1185

Section 3

42. An Accelerated Stress-Corrosion Test for High-Strength Ferrous Alloys. A.H. Freedman. J. Mater., June 1970, 5, (2), 431-466 (in English).

An accelerated laboratory test was developed for evaluating the susceptibility of high-strength ferrous alloys to stress corrosion in a sea-coast environment. Single-edge-notched and fatigue-cracked specimens are tension loaded in a NaCl solution (200 g/l. distilled water), and the threshold stress-intensity factor for stress corrosion (K_{ISCC}) is determined. Identical specimens were tension loaded in racks exposed at the seacoast, and their K_{ISCC} values were measured to act as standards for evaluating the accelerated test. The accelerated test requires a max. test time of 100 h. Test times are one to three orders of magnitude shorter than those required for similar specimens in a sea-coast environment. The acceleration of test time is produced by the aggressive corrodent, the presence of a crack and the plane-strain loading conditions. The steels investigated were H-11, 4340, 18Ni maraging, 410 SS, 17-4PH, AM 355 and 304 SS. Weld fusion zones and synthetic heat affected zones of the 4340, AM 355 and 18 Ni maraging steels were also tested. Some tests were also conducted on Inconel 718 to determine applicability of the test to Ni-base alloys. Twenty of the 23 combinations of material, heat treatment and welding conditions that were tested showed good to excellent agreement between the K_{ISCC} values obtained in seacoast and in accelerated tests. 18 ref. (35 1359.)

Section 3

43. Embrittlement of Ti-7Al Binary Alloy in Sea Water.
Joseph L. Cavallaro and Roy C. Wilcox. Corrosion, Apr. 1971,
27, (4), 157-163 (in English).

The effect of the precipitation of α_2 (Ti₃Al) particles on the stress-corrosion cracking (SCC) susceptibility of Ti-7Al binary alloy in seawater was determined by the notched, cantilever beam, stress-corrosion test. This alloy was aged in the $\alpha + \alpha_2$ region up to 500 h following α , β , and α - β solution-annealing treatments. Ageing of the α -annealed material caused an increase in stress-corrosion susceptibility for ageing times up to 120 h at 1200°F (649°C). Ageing for 500 h produced a significant recovery of SCC resistance, indicating dependence of SCC on the size of the α_2 precipitate particles. The extent of susceptibility was increased by increasing grain size of the alloy. The stress-corrosion fracture path was found to be transgranular for both equiaxed and Widmanstätten microstructures with cracking primarily by cleavage. Mixed ductile and cleavage fracture was observed in the fast fracture (air) zone. A correlation is shown between impact toughness in air and fracture stress in seawater. 16 ref. -AA

(35 1284.)

Section 3

44. Stress Corrosion Cracking of High Strength Steels and Titanium Alloys in Chloride Solutions at Ambient Temperature, Peterson, M. H., Brown, R. F., Newbegin, R. L. and Groover, R. E. Corrosion Vol 22, (1966) Paper presented at the 22nd Conference, National Association of Corrosion Engineers, Miami Beach, Florida, April 18-22, 1966.

By using a precracked cantilever-loaded test specimen, a relatively rapid and economical method of studying stress corrosion cracking in chloride solutions was developed. The method permits testing sensitivity of specimens to SCC in a fraction of the time required for smooth specimens.

Both high strength steels and titanium alloys proved susceptible in flowing sea water. AISI 4340 heat treated to strengths up to 225 ksi showed stress intensity increasing as the crack grew. Stress cracking of Ti-8Al-1V occurred at a final velocity of 1/4 in per minute.

Cathodic protection of high strength steel was not found to be a reliable means of preventing SCC because of close potential control required. At potentials created by a steel-zinc couple, it cracked at a stress intensity much lower than was the case for a freely corroding specimen.

Section 3

45. Exfoliation and Stress-Corrosion Characteristics of High Strength, Heat Treatable Aluminum Alloy Plate, paper presented at a joint conf. of North Central and Northeast Regions, National Association of Corrosion Engineers, October 2-4, 1967 at Columbus, Ohio, by B. W. Lifka, D. O. Sprowls, J. G. Kaufman, Corrosion Vol 23, (1967)

Corrosion tests in laboratory accelerated environments and in seacoast and inland industrial atmospheres have been conducted on specimens from 1-3/8 in thick hot rolled plate of several, high strength, heat treatable, aluminum alloys, and tempers.

Alloy-tempers 2020-T651, 2024-T851, 2219-T851, and 7075-T7351 were shown to be highly resistant to stress-corrosion cracking. No failures occurred with specimens stressed as high as 75 percent of the respective yield strengths, even in the short-transverse direction, in any environment studied. Alloy-tempers 7001-T7551 and 7178-T7651 also were resistant in longitudinal and longtransverse directions; but showed some susceptibility to high sustained stresses acting in the short-transverse direction. The 'threshold' stress for these two alloy-tempers, below which no failure is expected in 3.5 percent NaCl alternate immersion or in the atmosphere, is estimated at 27 ksi or 40 percent of the average short-transverse yield strength measured on these plates. In contrast, alloys 7075 and 7079 in the T651 temper showed some susceptibility to stress-corrosion cracking in the longitudinal and long-transverse directions at high sustained stresses, 75 percent of the yield strength and were susceptible in the short-transverse directions at a stress of 15 percent of the yield strength. Previous investigations have placed the threshold stress for 7075-T651 and 7079-T651 plate at 7 ksi or about 10 percent of the short-transverse yield strength.

Section 3

45. (cont.)

All alloy-tempers were highly resistant to exfoliation with the exception of 7079-T651, which showed a very mild degree of susceptibility and 7075-T651 which showed medium susceptibility.

Section 3

46. Effect of pH on Sea Water Stress Corrosion Cracking Behavior of the Ti-7Al-2Cb-1Ta Alloy, Litvin, D. A., Hill, B. Corrosion, Vol. 26, No. 3, (1970).

The purpose of this investigation was to determine the effect of varying the pH of a sea water environment on the stress corrosion cracking behavior of the Ti-7Al-2Cb-1Ta alloy. Various pH levels of 1.0, 3.0, 5.0, 7.4, 9.0, 11.0, and 11.6 were studied by additions of HCl and NaOH to the as-received sea water (pH = 7.4). The titanium specimens were beta-fabricated (rolled 1900°F, 1038°C), and tested using a cantilever weight test. It was found that an increase in the pH level of the sea water from 1.0 to 7.4 decreased the stress corrosion susceptibility of the Ti-7Al-2Cb-1 Ta alloy. In all cases, the mode of cracking was transgranular; yet, differences in the angle of cracking through the titanium platelet structure were observed. A distinct low ductility zone on the fracture surface of the sea water samples was noted, but no correlation could be made between the size of this zone and the pH of the solution. Photomicrographs of the specimens are included, along with macrographs of the fractured surfaces.

Section 3

47. A New Solution Potential Measurement for Predicting Stress Corrosion Performance of 2219 Aluminum Alloy Products, Horst, Jr. R. L., Hollingsworth, E. H., King, W.; Corrosion, Vol 23, No. 5, (1969)

A new electrochemical test for predicting the stress corrosion performance of 2219 alloy products in the T851 and T87 tempers is described. The test requires only a simple measurement of solution potential of an unstressed specimen in a mixture of absolute methyl alcohol and carbon tetrachloride. It is rapid, requiring less than an hour and has the outstanding advantage of providing differences of nearly 200 millivolts between borderline and resistant conditions of stress corrosion susceptibility, as contrasted to differences of less than 20 millivolts in conventional NaCl-H₂O₂ solution. The test offers an attractive possibility for evaluating stress corrosion performance of 2219 products quickly as an alternate or supplement to the conventional 30-day alternate immersion exposure in 3-1/2% sodium chloride solution. The mechanism of the reaction between 2219 and this organic electrolyte suggests that a similar test can be developed for other aluminum alloys.

Section 4

PITTING AND INTERGRANULAR CORROSION

1. CORROSION BEHAVIOUR OF ALUMINUM IN FRESH WATERS WITH SPECIAL REFERENCE TO PIPELINES. F. F. Booth, G. A. W. Murray and H. P. Godard. Brit Corrosion J, vl, no 2, Sept. 1965, p 80-86.

A review is given of work carried out on the long-term behavior of Al in fresh waters and the methods developed for predicting service behavior. The corrosion behavior of Al in waters is discussed and it is shown that it corrodes by pitting. The effect of pitting on tensile properties, on bursting strength of pipe, and on the hydraulic efficiency of pipe is described. It is shown that the most important consideration is that of prevention of perforation by pitting. The mechanism of pit initiation and pit growth is described. The methods used for determining the corrosivities of waters are given. The alloys in general use in fresh waters are given, together with their chemical compositions. Applications are mentioned.

15 ref.

M18--41162.

Section 4

2. ALUMINUM IN FRESH WATERS. Winifred A. Bell and Hector S. Campbell. Brit Corrosion J, v 1, no 2, Sept. 1965, p 72-79.

The type of corrosion affecting Al depends on water composition. Deep pitting requires O₂, chloride, carbonate hardness and Cu; the higher chloride contents encourage more general corrosion. High carbonate hardness reduces the number of pits formed; sulfates have a similar effect and also increase pit depth. Except in waters of very high Cu content, attack is generally less at elevated temperatures. Different alloys differ only slightly in resistance to attack. On superpurity Al, pits are slow to form but subsequently penetrate at the same rate as in Cu-Zn type. The best form of protection for Al is cladding with Al-1 1/4% Zn. This prevents pitting of N3 sheet or tube even in highly corrosive hard waters. Spraying with Al-Zn or anodizing is effective. Treatment to form boehmite films gives some protection but is ineffective in the more corrosive waters. 18 ref.

(M18--41161.)

Section 4

3. (Translation--Brutcher no 6388) STUDY OF PITTING CORROSION IN STAINLESS STEEL. PT. 1. EFFECT OF SURFACE CONDITION OF STEEL ON PITTING CORROSION. Y. Adachi. Tetsu-To-Hagane, v 50, no 4, 1964, p 565-567.

Method for rapid appraisal of the resistance of stainless steels and alloys to pitting corrosion, with special consideration of the effect of surface condition. Composition of materials studied. Experimental arrangement and procedure. Surface conditions of specimens: polished; etched; passivated; etched plus passivated. Relationship between voltage and testing time for Cr-Ni-Mo stainless steel in 3% NaCl. Number of corrosion pits per unit area as a function of current density. Voltage in relation to testing time for as-polished specimens. Effect of passivating treatment upon onset time and intensity of corrosion pitting in Cr-Ni stainless with and without Mo, Ti, Cb additions. (M18--22658.)

Section 4

4. Studies on Pit Formation on Aluminum in (Pure and) Artificial (Solute-Containing) Waters. Kinya Horibe. J. Japan Inst. Light Metals, Nov. 1968, 18, (11), 579-585 (in Japanese).

The pitting behaviour of 99.89% Al in pure and contaminated waters was examined; in pure water it was particularly sensitive to small amounts of FeSi and other impurities in the metal. The effects of adding various impurities (1 to 10 ppm) to pure water were studied by comparison with results for pure water. Neutral or acid salts did not cause pit formation; alkaline salts (except 3-5 ppm Na carbonate) inhibited pit formation; no macropitting was observed in any of the solutions studied.

(35 0753)

Section 4

5. PITTING OF TITANIUM ALLOYS IN SALINE WATERS. Posey, F. A.; Bohlmann, E. G. (Oak Ridge National Lab., Tenn.). Desalination, 3: 369-79(1967).

Electrochemical aspects of the corrosion of Ti and some of its alloys were studied in flowing, high-temperature salt solutions at temperatures up to 220°C in a Ti loop facility. Measured polarization curves showed that a pitting potential exists at sufficiently high potentials for all the alloys. Increase of temperature greatly affected the pitting potentials of Ti alloys. Plots of pitting potential as a function of temperature were reproducible and characteristic of each grade of Ti alloy and show clearly which alloys are superior in tendency towards pitting attack. The results suggest an upper temperature limit of usefulness for many Ti alloys without risk of severe localized attack. Alloys of Ti which contain Mo show promise for application in high-temperature saline waters. (41010.)

Section 4

6. (Symposium--Russian--RZ) CORROSION OF ALUMINUM AND TITANIUM IN GAPS.
A. I. Golubev. I.B. Ulanovskii and Yu.M. Korovin. Paper from "Korroziya
Met i Splavov", Metallurgiya, Moscow, no 2, 1965, p 351-358.

The processes of decrease in the concentration of oxygen in gaps and the corrosion of Al, Ti and Al- and Ti-base alloys were investigated. In sea water the oxygen concentration in gaps on Al and Ti alloys sharply decreases, which leads to the formation of differential aeration pairs. The value of pH decreases in the gap on Al under the effect of the pairs. No change in pH on Ti was observed; the breakdown of Ti in gaps was therefore insignificant.

(RZ--61472) M18--60787.

Section 5

VARIOUS TYPES OF CORROSION

1. (Book) CORROSION AND ITS PREVENTION IN WATERS, G. Butler and H.C.K. Ison. 1966, 281 p. Reinhold Publishing Corp., 450 Park Ave., New York 22, N.Y.

The principles and phenomena of metallic corrosion and the means of prevention and control of corrosion problems arising from the contact of metals with both natural and treated water are studied. Corrosion reactions with Fe, cast iron, wrought iron, austenitic Ni irons, low-alloy steels, stainless steels, Al and its alloys, Be, Cd, Co; Cr, Cu and its alloys, Pb, Mg, Mn, Mo, Ni and its alloys, Nb, Sn, Ti, W, U, Zn, Zr and the noble metals are considered separately, taking into account the influence of mechanical and metallurgical factors and variations in corrosion severity due to water temperature and flow rate. Methods of corrosion prevention, including water treatment and inhibition, the use of metallic and nonmetallic coatings and the application of cathodic protection are considered.

(M18--52507.)

Section 5

2. (French) PROBLEMS OF AQUEOUS CORROSION IN THE FIELD OF NUCLEAR ENERGY.
H. Coriou. Corrosion Anti-corrosion, v 13, no 2, Feb-Mar. 1965, p 55-71.

Discussion of: corrosion behavior in water of Al, Mg, Zr, U and their alloys, Ni-base alloys and stainless steel in their various roles as sheathing, structural and fuel materials in water-cooled and moderated nuclear reactors; corrosion mechanisms; and preventive measures.

(M18--35284.)

Section 5

3. (Pamphlet) APPLICATIONS OF STEELS FOR DESALINATION PLANTS.
Richard T. Jones. ASM Tech Rep no C6-20.6, 1966, 23 p (+ 21 plates).
American Society for Metals, Metals Park, Ohio.

Corrosion performance of carbon steel, 15% Cr steel, 17% Cr steel, AISI types 304 and 316 stainless steels is studied in various environments associated with distillation-type seawater desalination plants. Samples are tested for 90 and 757 days; in raw and deaerated seawater having low or moderate turbulence; at temperatures from 85 to 200°F. The corrosion rate is calculated from weight loss; depth of penetration of pitting is measured. Results are tabulated and illustrated. No evidence of stress corrosion is noted resulting from Erichsen cups in stainless steel. Results of service tests of Types 304 and 316 stainless steel evaporator tubes in initial evaporation stage are reported. Factors affecting corrosion are: deposition of seawater solids; temperature, 230 to 270°F.; dissolved oxygen in seawater; cathodic protection offered by carbon steel top and bottom tank sheets; addition of Mo to 18-8 stainless steel. Suggestions are made regarding environmental conditions under which carbon and stainless steels will give suitable service. 18 ref.

(M18--61538.)

Section 5

4. Corrosion of Aluminum. --. Light Metal Age, Apr. 1968, 26, (3-4), 15-16 (in English).

Recent research on stress and galvanic corrosion of Al includes work on: the corrosion rate of alloy 1100 in water; mechanisms of stress corrosion cracking in 7075-T6, 2219-T851 and pure Al-4Cu in NaCl and Na₂SO₄ solutions; an accelerated stress corrosion test for Al alloys involving immersion in 1% NaCl-2% K₂Cr₂O₇ solution at pH 4 and 60 C. for ring specimens of 2024, 2219 and 7075 Al alloys; the exfoliation and stress corrosion characteristics of high-strength heat treatable Al alloy plate; the threshold stress levels which cause stress corrosion cracking in synthetic seawater as determined from alternate immersion tests of 2014-F6, X2021-T8E31, 2024-T81, 2219-T87, X7002-T6, 7039-T64, and 7106-T6 alloys; crack initiation during stress corrosion as studied on tuning fork specimens of 2219 and 7075 specimens exposed to NaCl-AlCl₃-HCl solution at pH 1; the role of dislocations in stress corrosion cracking; corrosion effects of Al-Cu and Al-Ni couples in oxygenated distilled water and Al alloys in oxygenated copper sulfate solution; and the effect of a deionized water rinse after sulfuric acid-sodium dichromate treatment on the adhesive-bonding characteristics of 2024 Al. A new encapsulated rivet is available which is coated with tiny capsules of zinc chromate primer or a polysulfide sealant. When the rivet is clinched, the primer capsules rupture to provide a corrosion resistant film.

(35 0922.)

Section 5

5. (A/CONF.28/P/243) CORROSION IN AQUEOUS SYSTEMS. J. E. Draley (Argonne National Lab., Ill.), J. A. Ayres, W. E. Berry, E. Hillner, and S. P. Rideout. 16p.

Prepared for the United Nations Third International Conference on the Peaceful Uses of Atomic Energy, 1964.

The corrosion of reactor materials is reviewed. An evaluation of in-reactor corrosion behavior is followed by consideration of the corrosion of materials by type, and efforts to develop new alloys. Major attention is focussed on the pressurized water reactor, the boiling water reactor, and the steam superheat reactor. The primary emphasis is on corrosion problems which relate to the economic production of nuclear power.

(32081.)

Section 5

6. THE AQUEOUS CORROSION OF REACTOR METALS, J. N. Wanklyn and P. J. Jones (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Nucl. Mater., 6: 291-329 (Aug. 1962). (In English)

The literature of the corrosion of metals used in high and low-temperature water-cooled reactors is reviewed and technological and scientific advances in this field are assessed. The review extends to the latter part of 1961. (3431.)

Section 6

GALVANIC CORROSION

1. GALVANIC CORROSION CHARACTERISTICS OF ALUMINUM ALLOYED WITH GROUP IV METALS. D.S. Keir, M. J. Pryor and P. R. Sperry. Electrochem Soc J, v 114, no 8, Aug. 1967, p 777-782.

The electrochemical behavior of Al, alloyed with selected Group IV elements, has been investigated in 0.1 N sodium chloride solution. Of the group IV elements studied, only Sn had a major effect on Al; Al-Sn alloys carried surface oxide films of very low ionic resistance, exhibited potentials more active than -1.0 v on the hydrogen scale and gave very large galvanic currents when coupled to mild steel cathodes. The conditions permitting entry of a group IV alloying addition from the metal into the surface oxide film have been partly clarified. 13 ref.

M18--79962.

Section 6

2. (USNRDL-TR-68-26) ELECTROCHEMICAL CORROSION STUDIES OF GALVANICALLY COUPLED SNAP-21 MATERIALS. Kubose, D. A.; Cordova, H. I. (Naval Radiological Defense Lab., San Francisco, Calif.). Jan. 15, 1968. 42p. Dep. CFSTI.

Electrochemical corrosion rate measurements on materials used in the SNAP-21 radiosotopically-fueled power system were made in seawater at room temperature. The materials examined included aluminum, copper, Hastelloy C, Hastelloy X, nickel, 304 stainless steel, tantalum, titanium-621 alloy and uranium-8% molybdenum alloy. The normal corrosion rate of each material was measured by means of galvanostatic polarization techniques. A galvanic series of the materials in seawater was determined and the galvanic currents between galvanically coupled materials were measured with a zero-resistance ammeter circuit. The effect of galvanic coupling of construction materials of the SNAP-21 system does not materially change the containment time of the ⁹⁰Sr fuel in the corrosive seawater environment. (28737)

Section 6

3. (WCAP-1844) THE GALVANIC BEHAVIOR OF MATERIALS IN REACTOR COOLANTS. D. G. Sammarone (Westinghouse Electric Corp. Atomic Power Dept., Pittsburgh). Aug. 1961. 68 p.

Approximately 50 different couples between dissimilar materials used in nuclear steam generators designed by WAPD were evaluated for galvanic corrosion at temperatures ranging from 140 to 575°F in solutions of boric acid, boric acid and lithium hydroxide, lithium hydroxide, and in demineralized water with no additives. Couples other than those containing 2S aluminum, 4340 c/s, boronated stainless steel, boral, and nickel plated 80Ag-15In-5Cd with a 550°C heat treatment for 1 hour in vacuo were found to present no problems under the test conditions used. Cells containing either 2S aluminum or 4340 c/s were found to produce an excessive amount of galvanic corrosion at all but a few test conditions. The heat treatment used for nickel plated 80Ag-15In-5Cd control rod alloy significantly affected its resistance to galvanic corrosion. Galvanic corrosion results for cells containing boronated stainless steels and boral were inconclusive. Polarity reversals were found for two galvanic cells at high temperature. Since most cells were found to result in an insignificant amount of galvanic corrosion, effects due to temperature could not be detected.

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Section 6

4. (Italian) CORROSION RESISTANCE OF STAINLESS STEELS IN AQUEOUS SOLUTIONS. Pt. 2. Acciaio Inossidabile, v 34, no 1, Jan-Mar. 1967, p 27-32.

Pitting, interstitial and galvanic corrosion phenomena in stainless steels are explained by taking into account their microstructure and chemical composition, stresses, acidity and temperatures. Special attention is given to corrosion caused by couples creating galvanic cells acting as corrosion sites. Galvanic couples studied include stainless steel coupled to graphite, Ti, Inconel, Monel, bronze, Cu, Fe, Pb, Al and Zn. The severity of the corrosion in steels due to these couplings is indicated. (M18--78399)

Section 7

STERILIZATION: BOOKS, BIBLIOGRAPHIES, REVIEWS AND GENERAL INFORMATION

1. Sykes, G., Disinfection and Sterilization, 2nd ed., E. and F.N. Spon Ltd., London (1965)

This book is a comprehensive treatise on disinfection and sterilization. It is organized into six parts: (1) Theory of Disinfection and Methods of Testing, 102 pp., (2) Methods of Sterilization, 141 pp., (3) Air Disinfection and Sterilization, 36 pp., (4) Disinfection of Viruses, 18 pp., (5) Chemical Disinfectants, 116 pp. and (6) Preservation and Preservatives, 31 pp. Part I is composed of four chapters covering: (1) introduction, (2) the theory and mode of action of disinfection, (3) methods of testing disinfectants and (4) methods of testing antiseptics. Part II is composed of five chapters covering: (1) sterilization by heat, (2) radiation sterilization, (3) sterilization by filtration, (4) sterilization by gases and vapors, and (5) treatment by cold, desiccation and cellular disintegration. Parts III and IV are each single chapters covering air disinfection and sterilization and disinfection of viruses, respectively. Part V contains five chapters covering: (1) phenols, soaps, alcohols and related compounds, (2) dyes, (3) surface active compounds, (4) the halogens and (5) heavy metals. Part VI is a single chapter on preservation and preservatives. The references cited are authored by more than 1500 different investigators.

Section 7

2. Lawrence, C. A. and S. S. Block, *Disinfection, Sterilization, and Preservation*, Lea and Febiger, Philadelphia (1968)

This book is a recent comprehensive treatise on the subject containing contributions by 48 authorities and covering nearly 800 pages of text. It is organized into seven parts containing a total of 46 chapters. Part I, Introduction, contains two chapters: (1) Historical Review, pp. 3-8 and (2) Definition of Terms, pp. 9-12. Part II, Mode of Action, is comprised of four chapters: (1) Principles of Antimicrobial Activity, pp. 15-31, (2) Bacterial Resistance and Dynamics of Antibacterial Activity, pp. 32-43, (3) Physical Factors Influencing the Activity of Antimicrobial Agents, pp. 44-62 and (4) Thermal Destruction of Microorganisms, pp. 63-105. Part III, Methods of Testing, contains nine chapters: (1) Introduction to Antimicrobial Testing Methods, pp. 109-113, (2) Methods of Testing for Sterility and Efficacy of Sterilizers, Sporicides and Sterilizing Processes, pp. 114-132, (3) Method of Testing Disinfectants, pp. 133-158, (4) Methods of Testing Sanitizers and Bacteriostatic Substances, pp. 159-178, (5) Methods of Testing Antiseptics, pp. 179-193, (6) Method of Testing Virucides, pp. 194-206, (7) Methods of Testing Fungicides, pp. 207-217, (8) Methods of Testing Protozoocides and Anthelmintics, pp. 218-220 and (9) Safety Evaluations on Antimicrobial Chemicals, pp. 221-233. Part IV, Antiseptics and Disinfectants, is comprised of 16 chapters: (1) Alcohols, pp. 237-252, (2) Acid-Anionic Surfactant Sanitizers, pp. 253-256, (3) The Bis-Phenols, pp. 257-277, (4) Chlorine and Chlorine Compounds, pp. 278-304, (5) Fungistatic and Fungicidal Compounds, pp. 305-320, (6) Heavy Metals Other than Mercury and Silver, pp. 321-328,

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(cont.)

(7) Iodine, pp. 329-347, (8) Mercurials-Inorganic and Organic, pp. 348-371, (9) Oligodynamic Metals, pp. 372-400, (10) Phenolic Compounds, pp. 401-429, (11) Quaternary Ammonium Surface-Active Disinfectants, pp. 430-452, (12) The Salicylanilides and Carbanilides, pp. 453-468, (13) Silver Compounds, pp. 469-475, (14) Virucidal Agents, pp. 476-488, (15) Antiprotozoan and Anthelmintic Compounds, pp. 489-503, and (16) Mycobactericidal Agents, pp. 504-516. Part V, Surgical and Hospital Disinfection, contain three chapters, pp. 517-554. Part VI, Antimicrobial Preservatives and Protectants, contains five chapters, pp. 555-651. Part VII, Chemical and Physical Sterilization, is comprised of seven chapters: (1) Disinfection of Drinking Water, Swimming Pool Water and Treated Sewage Effluents, pp. 655-668, (2) Gaseous Sterilization, pp. 669-685, (3) Spacecraft Sterilization, pp. 686-702, (4) Sterilization by Heat, pp. 703-740, (5) The Destruction of Microorganisms by Ionizing Irradiation, pp. 741-760, (6) Sterilization by Ultraviolet Radiations, pp. 761-777 and (7) Sterilization Filtration, pp. 778-791.

Section 7

3. Bernarde, M. A., Disinfection, Dekker, New York (1970), 466 pp. \$24.50. (C. A., 73, 91231x (1970)).

Contains a review by W. B. Hugo on "Mechanisms of Disinfection", pp. 31-60, with 53 refs. (C.A., 73, 76007k (1970)); a review by A. Prokop and A. E. Humphrey on "Kinetics of Disinfection", pp. 61-83, (C.A., 73, 62675q (1970)); a review by P.M. Borick and R. E. Pepper on "The Spore Problem", pp. 85-102, with 61 refs. (C.A., 73, 76008m (1970)); a review by K. Grossgebauer on "Virus Disinfection", pp. 103-48, with 77 refs. (C.A., 73, 76009n (1970)); and a review by C. W. and M. K. Bruch on "Gaseous Disinfection", pp. 149-206, with 164 refs. (C.A., 73, 76010f (1970)).

4. "Sterilization- A Selected Bibliography from the Literature Retrieval System, Space Biology Branch", NASA-TM-X-55457, NASA, Goddard Space Flight Center, Greenbelt, Md., March 1966. (N66-24690).

An annotated bibliography is presented with topical headings. These headings include: (1) clean rooms; (2) decontamination; (3) sterilization methods, i.e., air filtering, chemicals, and heat; (4) methods and statistics for detection and monitoring of microorganisms; (5) spacecraft sterilization; and (6) survival, i.e., viability of microorganisms.

Section 7

5. Le Doux, F. N., "Bibliography: Codes, Procedures, Specifications and Reports Relating to Contamination Control", NASA-TM-X-63982, Goddard Space Flight Center, Greenbelt, Md., June 1970. (NASA Accession No. X70-16650)

The bibliography contains 382 citations and covers the following subject areas: (1) specifications for acids and detergents, (2) cleanliness, (3) cleaning, (4) clean room and work stations, (5) contamination, (6) decontamination, (7) manufacturing, (8) miscellaneous, (9) particle count analyses, (10) passivation, (11) packaging, and (12) water. Eight citations deal specifically with water. Many more citations are applicable to the control of contamination (particulate, soluble, and microbial) in water systems.

Section 7

6. "Fungusproofing: Industrial Processing Series, Vol. 1, Report Bibliography, Dec. 1943-Jul. 1970", AD-720202, DDC-TAS-70-83-1, Defense Documentation Center, Feb. 1971 STAR, 2, (14), 2207, N71-26638)

The bibliograph is the first volume of a two volume set on Fungusproofing in a series of bibliographies on Industrial Processing. It includes 44 annotated references and covers the period from January 1953 to January 1971. Corporate author-monitoring agency, subject, title, personal author, and report number indexes are included.

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Section 7

7. "Water: Purification and Decontamination, A DDC Bibliography, July 1952-August 1969", DDC-TAS-71-30, Defense Documentation Center, Alexandria, Va., June 1971 (AD-725610, N72-11103).

The bibliography consists of 76 annotated references to reports which were selected from the Defense Documentation Center's data bank covering the period from January 1953 through December 1970. The reports analyze the purification, distillation and decontamination of water. It includes the recycling of closed ecological systems for the potability of water by the use of ion exchange, filtration, disinfecting agents, and coagulation. The indexes are corporate author-monitoring agency, and subject.

Section 7

8. Sykes, G., "Methods and Equipment for Sterilization of Laboratory Apparatus and Media", *Methods Microbiol.*, 1, 77-121 (1969). (C.A. 76, 22595u (1972))

A review. Discussed are methods of sterilization (stn.), mechanisms of stn., stn. by heat, radiation stn., stn. by gases and vapors, filtration, chem. stn., and applications to various materials. 57 refs.

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Section 7

9. Pearce, A. S., "Water Treatment", Rep. Progr. Appl. Chem., 54, 670-80 (1969). (C.A., 74, 146114q (1971)).

A review with 117 refs. The purification of sea water, removal of biol. matter and sterilization, ion exchange, filtration and removal of colloidal and particulate matter, waterside corrosion and industrial water treatment, and anal. and sampling methods are discussed.

Section 7

10. Kinman, R. N. and H. A. Faber, "Review of the 1969 Literature on Waste Water and Water Pollution Control. Waste Water Treatment. Disinfection", J. Water Pollut. Contr. Fed., 42 (6), 949-52 (1970). (C.A. 73, 69576S (1970)).

The use of a no. of disinfectants was reported in 1969. Emphasis on compds. contg. different forms of Cl was obsd. in this review. Also considered were radiation treatment, chloramine compds. for the inactivation of viruses, including uv treatment for polio virus. Lab. and field studies are reported by using a no. of the disinfectants mentioned. A fully automatic disinfection plant for treating waste water contg. pathogenic organisms also appeared during 1969. Findings concerning the physiol. effects of I on a human population are mentioned. 22 refs.

Section 7

11. Dos Santos, J.L.A., "Sterilization by Chemical Substances in the Gaseous State", Rev. Port. Farm (Portugal), 20 (1), 8-33 (1970). (C.A., 73, 133941m (1970)).

A review of the use of HCHO, ethylenimine, β -propiolactone, and propylene and triethylene glycols and the industrial applications and limitations of ethylene oxide are presented. 12 refs.

Section 7

12. "Contamination Control Handbook", NASA-CR-61264, NASA-SP-5076
Sandia Corp., Albuquerque, N. Mex., NASA Order H-13245A,
Feb. 1969 (NASA Microfiche N69-28593 and N70-13566)

Technical information and data on contamination control are assembled and include the following topics: contamination control in product design, gases, and liquids; and microbial, airborne, and surface contamination control. Sections are also presented on radiation, clean packaging, and maintaining product and personnel cleanliness. The experience described was gained in the aerospace community, and is presented as a technology which can be utilized in pharmaceutical, electronic, and other industries.

Section 7

13. "Contamination Control Handbook", NASA SP-5076, National Aeronautics and Space Administration, Washington, D.C. (1969).

Section 6.1 - Characteristics of Microbial Contamination.

Groups generally considered as microorganisms include the viruses, rickettsiae, bacteria, fungi, protozoa, and algae. Microorganisms may differ over a wide range of dimensions. The viruses are the smallest of all biological entities and they vary generally from 8 to 10 millimicrons (μ) to 250 to 300 μ . The viruses are obligate intracellular parasites, i.e., they require a living host cell. The rickettsiae are similar to the bacteria in many respects; they are about the size of the smallest bacteria. The rickettsiae are also obligate intracellular parasites. The bacteria may occur as single cells but in most cases they occur in clumps, chains, filaments, plates, or compact aggregates. The individual cells may be rod-shaped (cylindrical), coccoid (spherical), or spiral-shaped. The bacteria vary in size from less than 1μ to filamentous forms as long as 100μ . The molecular oxygen requirement of the bacteria varies from aerobic organisms (which require molecular oxygen) to the obligate anaerobic organisms (for which molecular oxygen is toxic). On occasion, certain bacteria form heavy walls (spores), and in this state they are very resistant to adverse environmental conditions. The fungi are a heterogeneous group made up of such diverse forms as yeasts, molds, mildews, and mushrooms. The yeasts are somewhat larger (5 to 10μ) than most bacteria. Molds generally grow in filaments, or hyphae, which in turn comprise the body of the organism, the mycelium. The mold hyphae have diameters of 2 to 200μ but may have lengths of many millimeters. Mold spores are considerably larger (4 to 100μ) than the bacterial spores. The protozoa are found in water or in the animal body. Protozoa are 1μ or larger in size

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and highly susceptible to drying. Certain parasitic protozoa form resistant cysts, but these are not nearly as resistant as the spores of bacteria and molds. The algae vary widely in size from the microscopic blue-green algae to the macroscopic brown algae (seaweed). The algae live and grow in water and on moist surfaces. The smallest algal cells are approximately 1 to 2 μ in diameter. Some algae, such as the diatoms, form mineral shells (calcium carbonate) and may be very resistant to harsh environmental conditions.

All microorganisms share a set of physiological and nutritional requirements which are necessary for growth and reproduction. Autotrophic organisms are very complex and complete in their enzymatic properties, but they are sometimes extremely simple in their nutritional requirements. Autotrophs grow and reproduce when supplied with carbon dioxide (or carbonate) and a few simple inorganic salts. They obtain their energy either from light (prototrophs) or from the oxidation of inorganic compounds (chemotrophs). Heterotrophic organisms must be supplied with an organic carbon source. The heterotrophs vary widely in their nutritional requirements; from an organism requiring a single organic compound plus a few simple salts to one which requires such additional growth factors as metabolites and vitamins. Obligate intracellular parasites such as all of the viruses (plant, animal, and bacterial), the rickettsiae, and perhaps a few of the bacteria must be cultured in living cells; i.e., they cannot be grown in a cell-free medium.

Section 7

14. "Contamination Control Handbook", NASA SP-5076, National Aeronautics and Space Administration, Washington, D.C. (1969)

Section 6.2- Control of Microbial Contamination

The control of microbial contamination entails three primary principles: deactivation, removal, and isolation. The deactivation (killing) of microorganisms by an antimicrobial or lethal agent generally follows an exponential form similar to that observed in unimolecular chemical reactions, i.e. the log of the surviving number of organisms decreases approximately linearly with time.

Three basic methods of deactivation are normally used: heat, chemicals, and radiation. Of these three, only the application of heat has the practical potential for rendering a mass sterile. Two heating methods are commonly used: (1) a moist heating such as autoclaving at 121°C for 20 minutes and (2) dry heating such as in a hot-air oven at 160°C for 2 hr. Variations in the time and temperature for both of the methods are possible. Chemical sterilization may employ either gaseous or liquid sterilants. The common gas sterilants are ethylene oxide, propylene oxide, formaldehyde, methyl bromide, and beta-propiolactone with the first being generally preferred. Typical ethylene oxide sterilization procedures call for a concentration of greater than 400 mg/l., relative humidity of 40-50%, temperatures of 49-60°C, and times of 3-24 hrs. Some of the classes of liquid sterilants include, phenolic compounds, alcohols, iodophors, quaternary ammonium compounds, and chlorine compounds. Their efficacies are highly variable for different species and conditions of use. Sterilization by radiation may be based on the use of UV, X-rays, or gamma-rays. The former has the highest bactericidal efficiency but very little penetrating power. Microorganisms vary widely in their radiation sensitivity.

(cont.)

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The removal of microorganisms is basically limited to liquids, solutions, and gases and generally involves filtration. Filters of many types and pore sizes are available. Microfiber filters can remove practically all biological particles 1 micron or greater in size.

Three basic methods of isolating sterile articles from a nonsterile environment include: (1) using biobarriers, such as sealed plastic films, containers, etc., (2) placing in a room, hood, etc. that is kept "particle free" by air filtration, and (3) isolating by air pressure gradients so only outflow from the sterile area is possible.

Section 7

15. Dwyer, J. L., "Control Techniques in High Purity Water Production", Bull. Parenteral Drug Ass., 26 (6), 267-75 (1968). (C.A., 73, 59193a (1970)).

Process treatment techniques for removing contaminants from high-purity water are outlined. They include distn., chem. pptn., settling, adsorption, ion exchange, filtration, and chem. disinfection. Contaminants to be excluded are generalized into 4 categories and are given with detection methods; (1) ionic, as the electrolytes, Na, Fe, Ca, Cl^- , SO_4^{2-} , and PO_4^{3-} detected by elec. cond., or elemental anal.; (2) dissolved nonelectrolytes; org. materials as lignins, tannins, detergents, and biodecompn. products, detected by spectrophotometric methods, colorimetry, or absorption-extraction procedures; (3) suspended particles, silt, pipe scale, settled dust; detected and identified by membrane filtration methods; (4) microbiol.: bacteria, algae, and protozoa, detected by cultures and microscopy. A typical high-purity water installation and its problems are discussed.

Section 7

16. Phillips, C. R., "Gaseous Sterilization", Army Biological Labs., Fort Detrick, Md. In NASA, Washington Spacecraft Sterilization Technology, pp. 231-257 (1966). (N67-14777).

Ethylene oxide, formaldehyde, and beta-propiolactone are identified as the compounds used most frequently in gas sterilization applications, and the advantages and disadvantages of each are assessed. Based on a literature survey, other gases which are considered to have bactericidal properties are discussed. These include propylene oxide, ozone, methyl bromide gas, chloropicrin gas, epichlorohydrin and epibromohydrin, ethylene imine, glycidaldehyde, and peracetic acid. An extensive bibliography is included.

Section 7

17. Crivellaro, G. B., "Sterilization Techniques in the Pharmaceutical Industry", *Farmaco. Ed. Prat.*, 25 (5), 277-90 (1970). (C.A., 73, 38453v (1970)).

The various sterilization processes currently employed in the pharmaceutical industry are listed and discussed. These processes include: Various heat sterilization procedures, sterile filtration techniques, uv irradiation, ionizing radiation, sterilization by chem. means (ethylene oxide, formaldehyde, β -propiolactone, peracetic acid, and others). In particular, the most recent methods, such as sterilization by gas and irradiation, are considered.

Section 7

18. Lacoux, P., "Sterile Demineralized Water for Modern Techniques", Tech. Eau Assainissement, No. 288, 61-3 (1970). (C.A., 74, 102918a (1971)).

Many advances in medicine, pharmacology, electronics, and space exploration are based on the availability of water of great purity and total sterility. Procedures and materials satisfactory for the prepn. of such water are presented including an outline for process automation.

102918a

Section 7

19. Roper, W. D., "Effects of Decontamination, Sterilization, and Thermal Vacuum on Spacecraft Polymeric Products", NASA-CR-103426, JPL-TR-32-1411, Jet Propulsion Lab., Contract NAS 7-100, 30 June 1969 (STAR, 1 (18), 3396, N69-31924)

An investigation of 21 polymeric products was made to establish their suitability for use in spacecraft hardware. Each polymeric product, which included several material categories, was given sequential exposure to the following three environments: ethylene oxide-Freon 12 decontamination, thermal sterilization, and long-term thermal vacuum exposure. The first exposures were made in accordance with a sterilization specification. The environmental conditions for the third exposure were 500 h at 135°C and 10^{-6} torr. The purpose was to evaluate the total effect of all these exposures on the products tested. Compatibility ratings were assigned to each product tested. Of the products evaluated, 55% were rated as compatible, 30% marginal and 15% non-compatible with the three exposures.

Section 7

20. Gundermann, K. O. and S. Glueck, "Survival of Bateria on Surfaces and Possibilities for Influencing Survival. III. Effect of Various Light Conditions and Prior Disinfection in the Use of Plastics", Arch. Hyg. Bakteriöl., 154 (5), 480-7 (1971). (C.A., 75, 106533q (1971)).

The survival time of bacteria on PVC, polyethylene, polystyrene, acrylonitrile-styrene copolymer, cellulose acetate, polyacetal, polypropylene, polycarbonate, formaldehyde-melamine copolymer, alkyd resin, phenolic resin, and acrylic resin revealed a bactericidal effect of some plastics (alkyd and phenolic resins) and an unfavorable effect on germs of some others (PVC, polyacetyl). Additives in the plastics affected the bactericidal properties, and daylight had a bactericidal effect. A phenolic disinfectant had a considerable after effect on polyethylene, polystyrene, polypropylene, polycarbonate, phenolic resin, and acrylic resin and had a small aftereffect on PVC and polyacetal.

Section 8
THERMAL STERILIZATION

1. Craven, C. W., J. J. McDade, and J. O. Light, "Sterilization and Quarantine Parameters for Consideration During the Design of Planetary Vehicles", JPL, Calif. Inst. Tech., Pasadena, Calif. In NASA, Washington Spacecraft Sterilization Technology, pp 43-50 (1966). (1367-14766).

The tentative policy adopted as a constraint on all launches for Mars is outlined, and the known sources of contamination are summarized. Capsule sterilization procedures, based on chemical decontamination and exposure to dry heat, have been selected as optimum, and steps taken to insure the development of sterilizable materials and components are discussed. Although heating to 135°C for 24 hours has proved adequate, the possibility is mentioned that a lower temperature for a longer time may be selected for the flight programs. Sterilization is aimed at the inactivation of all microorganisms, in particular the fungi and bacteria; problems connected with their destruction are discussed. It is expected that all subsystems, including the various science payloads, will be subjected to one chemical decontamination cycle (ethylene oxide), and one dry heat sterilization cycle as part of the flight acceptance test before the start of final assembly. Capsule assembly and sterilization procedures are depicted.

Section 8

2. Bruch, C. W., "Dry-Heat Sterilization for Planetary-Impacting Spacecraft", NASA, Washington, D.C. In NASA, Washington Spacecraft Sterilization Technology, pp. 207-229, (1966). (N67-14776).

The dry-heat cycle of 135°C for 22 hours being investigated for the sterilization of planetary spacecraft is described herein. A historical approach of how that particular cycle was derived is presented. After an analysis of some of the environmental factors that influence dry-heat sterilization, various mathematical considerations of the available data are examined. By use of activation energy calculations, it is shown that some components might suffer less damage with high-temperature short-time cycles. It is also necessary to consider the integration of the lethality that occurs during the comeup time and cooldown time of the heating process with the lethality that occurs at the desired holding temperature.

Section 8

3. Hopkins, G. H., "Effect of Heat on the Physical Properties of Rubber Closures". Bull. Parenteral Drug Ass., 22 (4), 181-6 (1968). (C.A., 13, 48525j (1970)).

The effects of dry and moist heat of sterilization on 24 reinforced natural, neoprene, and butyl rubber closure compns. were studied. The tensile strength, durometer hardness, and elongation of the compns. were evaluated before and after heat treatment. Most rubbers withstood autoclave sterilization better than the dry heat method. When dry heat is necessary, short exposure to higher heat is better than long exposure to lower heat.

Section 8

4. Fajers, C. M. and L. G. Holmlund, "Corrosion and Corrosion Inhibition During Cleaning, Decontamination, Packing, Autoclave Sterilization, and Sterile Storage of Instruments in Medical Praxis Including an Ultrasonic Standardization Method", Scand. Corros. Congr., Proc., 5th (1968), The Danish Corrosion Centre: Copenhagen, Denmark (1969). (C.A., 74, 24665z (1971)).

A concn. of 35 ppm of a volatile amine (e.g., cyclohexylamine) in the autoclave chamber prevents corrosion of surgical instruments during sterilization. The inclusion of the amine in the storage envelope prevents corrosion during subsequent storage.

Section 8

5. Brierly, J. A. and S. E. Podlaseck, "A Parametric Study to Determine Time-Temperature-Vacuum Relationships for Sterilization of Terrestrial Spores", Phase 1, Summary Rpt. Period Ending 18 April 1969, NASA-CR-99627, Martin Marrietta Corp., Baltimore, Md., Contract NAS9-9261, April 1969 (STAR, 7 (12), 2033, N69-23883)

Spores of *Bacillus* sp. G2, *Bacillus subtilis* var niger and *Clostridium sporogenes* were exposed to temperatures of 25, 70, 90 and 110°C at atmospheric or vacuum (10^{-6} Torr) pressure for periods of 2 and 20 hours. No effect of vacuum pressure was noted for spore survival at 25°C. At 70°C *Bacillus* sp G2 and *B. subtilis* var niger spores were not affected at atmospheric pressure and *C. sporogenes* spores demonstrated slight susceptibility. D-value 13.81. At the vacuum pressure *B. subtilis* spores were not affected, but *Bacillus* sp G2 spore D-value was 4.31 and *C. sporogenes* spore D-value was 4.48. *C. sporogenes* had little resistance to 110°C with D-values of 4.34 at atmospheric pressure and 4.51 at vacuum pressure. It may be stated that generally vacuum pressure (10^{-6} Torr) increases the susceptibility of spores to temperature.

Section 8

6. Brierley, J. A., "A Parametric Study to Determine Time-Temperature-Vacuum Relationships for Sterilization of Terrestrial Spores", Phase 2, Summary Report, NASA-CR-101701, Martin Marietta Corp., Denver, Colo. Contract NAS9-9261, June 1969 (STAR, 7 (16), 2884, N69-29751)

The results of a parametric study to determine time-temperature-vacuum relationships for sterilization of terrestrial bacterial spores are presented. Spores of *Bacillus* sp G2, *Bacillus subtilis* var niger, and *Clostridium sporogenes* were exposed to temperatures of 100°, 105°, 110°, 115° and 120°C at vacuum (10^{-6} torr) and atmospheric pressure for periods of 1, 4, 8 and 16 hours. The data were used to determine the D-values for the test spores at the test and control conditions. The vacuum increased the rate of spore death for *Bacillus* sp. G2 and *B. subtilis* spores at all test temperatures. The spores of *C1 sporogenes* were more resistant to heat within the vacuum at 100°, 105°, and 110°C, but they showed no detectable heat resistance within the vacuum at 115° and 120°C. The data indicated that temperatures above 110°C, even in the vacuum environment need to be used for sterilization.

Section 8

7. Brady, F. and D. DiStefano, "Sterilization Liquid Propulsion Part 1," Final Rpt. 5 Oct. 1966-31 March 1968, NASA-CR-106366, MCR-68-119-PT-1, Martin Marietta Corp., Denver, Col. Contracts NAS7-100; JPL-951709, Aug. 1968 (STAR, 7, 4660, N69-40843)

The program involved the exposure of an assembled and fueled bi-propellant liquid propulsion system to the ethylene oxide (ETO) and heat sterilization requirements specified. After exposure the system was fired for 280 sec. The program included a design and component selection phase during which the propulsion system design was evolved. A second phase involved the procurement of components for both a component test series and for assembly into the complete system. The third phase of the program, carried on in parallel with the design phase was a materials investigation. The fourth phase of the program involved the assembly and test of the complete propulsion system. The components underwent 12 heat sterilization cycles along with functional tests to measure degradation. Corrections or modifications were made as required to allow system testing.

Section 8

8. Lukens, S. C., "Sterilizable Liquid Propulsion System, Part 2", Final Rpt., 1 April 1968-9 May 1969, NASA-CR-106380; MCR-68-119-PT-2, Martin Marietta Corp., Denver, Corp., Contracts NAS7-100; JPL-951709, Sept. 1969 (STAR, 7, 4661, N69-408844)

The program was continued to gather additional information resulting from a second series of sterilization exposure cycles and additional materials compatibility tests. Insofar as possible the propulsion system was reassembled to the initial configuration following evaluation of the effects of the initial exposure and test firing. It was then exposed to three-30-hr. dry heat sterilization cycles and refired for an additional 280 sec. The initial portion of the program included a materials testing activity and a components test activity wherein early test results could be factored into the design and assembly of the complete propulsion module.

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Section 8

9. "Ecology and Thermal Inactivation of Microbes in and on Interplanetary Space Vehicle Components", Public Health Service, Cincinnati, Ohio, NASA Order R-36-015-001. See following series of quarterly reports.

- 9.a. , First Quarterly Progress Report, 1 Apr.-30 June 1965, NASA-CR-64834, July 1965. (N65-33537)

Several batches of *Bacillus globigii* spores have been produced.

Several plating media were compared to determine their ability to permit germination and good outgrowth of *B. globigii* spores. Tryptone glucose extract agar was selected as the medium of choice as a result of these comparisons.

- 9.b. , Second Quarterly Report, July. 1- Sept. 30, 1965, NASA-CR-69345, Oct. 1965. (N66-15381)

Data are presented indicating that *Bacillus globigii* spores are recoverable from experimentally contaminated blocks of balsa wood by maceration in a Waring Blendor. A technique has been developed whereby spores introduced into acetone soluble plastics may be recovered quantitatively by means of dissolution and entrapment in Seitz filter pads. In connection with the development of a satisfactory model system for insoluble components, a method has been developed which provides quantitative information on the toxicity of plastics. This technique utilizes the concept of the direct surface agar plate which has found wide application in the field of surface contamination.

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- 9.c. , Fourth Quarterly Progress Report, Jan. 1- Mar. 31, 1966, NASA-CR-74711, April 1966. (N66-24988).

The thermal resistance of *Bacillus Globigii* spores dried on paper strips and encapsulated in Lucite rods has been determined at a dry heat exposure temperature of 125°C. The $D_{125^{\circ}\text{C}}$ value for these spores dried on paper strips was found to be 1.72 hours with a 95% confidence interval of 1.61 to 1.83 hours. In duplicate experiments performed with these spores encapsulated in Lucite, the $D_{125^{\circ}\text{C}}$ values were: experiment 1, 3.08 hours, 95% confidence interval of 2.55 to 3.61 hours: experiment 2, 3.44 hours, 95% confidence interval of 2.98 to 3.89 hours.

- 9.d. , Fifth Quarterly Progress Report, Apr. 1 - June. 30, 1966, NASA-CR-76539, July 1966. (N66-32145).

D values for dry heat exposure temperatures of 115°C and 135°C were determined for *Bacillus globigii* spores encapsulated in plastic and dried on paper strips. These values are plastic 115°C. $D = 15.4$ hours: 135°C. $D = 1.4$ hours; paper 135°C. $D = 16.1$ minutes. The slope of the Decimal Reduction Time curve for spores encapsulated in Lucite and obtained by plotting the logarithms of D for 115, 125, and 135°C against temperature was calculated to be 18.5°C (33.3°F).

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- 9.e. , Sixth Quarterly Report, Jul. 1 - Sept. 30, 1966, NASA-CR-80484, Oct. 1966. (N67-13129).

The dry heat resistance of *Bacillus subtilis* var. *niger* spores encapsulated in Lucite rods at an inoculum of approximately 1×10^8 spores per gram and exposed to temperature of 105, 120, and 160°C was determined. The D values and their corresponding 95% confidence intervals were found to range respectively: $D_{105} = 1.2$ to 1.3 days, 95% C.I. of 1.1 to 1.5 days; $D_{120} = 5.9$ to 6.2 hours, 95% C.I. of 5.0 to 6.8 hours; $D_{160} = 4.6$ to 4.8 minutes, 95% C.I. of 3.8 to 4.9 minutes. A decimal reduction time curve was constructed employing the D values from all temperatures studied to date. The slope of this curve over the temperature range of 105°C to 160°C yielded a value of $z_D = 20.8$ Centigrade degrees.

- 9.f. , Quarterly Progress Report, Oct. 1- Dec. 31, 1966, NASA-CR-82514, Jan. 1967. (N67-19086).

Methods have been developed for incorporating and recovering *B. subtilis* var. *niger* spores in epoxy resin and preliminary data indicate that D value in comparison to Lucite may be twice as great in this type of plastic. A system has been developed for measuring the thermal resistance of spores on stainless steel mated surfaces and preliminary data indicate that D values will be much less for spores so located than for spores encapsulated in Lucite. Measurements of the thermal resistance of spores adjusted to various levels of water activity prior to encapsulation in Lucite indicate that water activity may be significant consideration in establishing dry heat sterilization processes.

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- 9.g. , Quarterly Report, Jan. 1-Mar. 31, 1967, NASA-CR-85238, QR-8, April 1967. (N67-30005).

The dry heat resistance of *Bacillus subtilis* var. *niger* spores in and on various materials was determined. D values for spores encapsulated in epoxy and exposed to temperatures of 115, 125, and 135°C were found to be respectively: $D_{115} = 16.1$ hours, $D_{125} = 5.3$ hours, $D_{135} = 1.9$ hours. The z_D value for spores in epoxy was 21.4 Centigrade degrees with a 95% C.I. of 20.8 to 22.1 Centigrade degrees. D values for spores trapped between stainless steel surfaces mated at 12 and 150 inch-pounds and exposed to 135°C were found to range respectively: D_{135} for 12 inch-pounds = 7.9 to 8.1 minutes and D_{135} for 150 inch-pounds = 22.0 to 25.5 minutes. Inoculated Lucite powders that were stored for two weeks at relative humidities of 20, 40, 60, and 80%, and then formed into rods and exposed to 135°C were found to have D values of 73.5, 88.7, 67.4, and 36.0 minutes, respectively.

- 9.h. , Quarterly Progress Report, Apr. 1- June. 30, 1967, NASA-CR-86616, QPR-9, July 1967. (N67-32086).

Dry heat resistance of spores was determined at controlled water activities. The D_{135} values for *Bacillus subtilis* var. *niger* spores encapsulated in lucite exposed to water activities are reported. Another system employing inoculated stainless steel strips exposed to a water activity demonstrated the protective effect of moisture on dry heat resistance. The thermal resistance of spores on stainless steel was comparable to that of epoxy encapsulated spores. In addition, D values were determined for mated surface units at 150 inch-pounds torque.

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- 9.i. , Quarterly Progress Report, Jul. 1- Sept. 30, 1967,
NASA-CR-90097, QPR-10, Oct. 1967. (N68-10628).

The dry heat resistance of *Bacillus subtilis* var. *niger* spores on stainless steel strips was determined. D values at 115°, 125°, and 135°C were obtained and a z_D value calculated. In addition, a thermal death time curve for spores trapped between mated surfaces at 150 inch-pounds of torque was plotted from D values reported in the Ninth Quarterly Report of Progress. A z_D value was determined from the curve and reported. Finally, the effect of water activity on the thermal resistance of spores was investigated. In this system, spores were dried on glass and exposed to various water activities prior to heating. Results from these experiments substantiated earlier findings indicating that intermediate water activities increase dry heat resistance of spores.

- 9.j. , Quarterly Progress Report, Oct. 1-Dec. 31, 1967,
NASA-CR-92720, QPR-11, Jan. 1968 (N68-16109).

Techniques are described for measuring the dry heat resistance of *B. subtilis* var. *niger* spores in and on various materials. The D and z values are presented for these spores in the various test systems, in which the D value is the rate of death of an organism at a given temperature, and the z value is a measure of the change in rate of destruction with temperature. The systems tested included spores located on steel and paper strips, spores between stainless steel washers mated together under 150 in.-lbs and 12 in.-lbs. of torque, and spores encapsulated in methylmethacrylate and epoxy plastics. An explanation of the differences in heat resistance as related to the materials in or on which spores are located is offered. The results indicate that the dry heat resistance of *B. subtilis* var. *niger* are influenced by the initial moisture content of the spores, the rate of spore desiccation during heating, and the equilibrium relative humidity of the system.

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9.k. , Quarterly Progress Report, Jan. 1 - Mar. 31, 1968,
NASA-CR-94681, QPR-12, March 31, 1968. (N68-24369).

Previous dry heat resistance studies of *Bacillus subtilis* var. *niger* spores impregnated in filter paper indicated that a wet heat kill mechanism was involved as evidenced by a z_D of 12.9°C. Because sufficient water remained in the system to effect a wet heat system during the drying process, lyophilization was investigated as an improved drying method. D and z values for spores on paper dried by lyophilization are reported. An unusually high z_D value (32.0°C) was previously reported for spores located between steel washers mated at 150 inch-pounds of torque. An explanation for this high value is presented which is based on the slow loss of spore moisture to the hot gaseous environment in the TDT tube during heating. Sealing the mated surface units with epoxy resin prevented loss of moisture and resulted in a z_D value of 21.9°C which was comparable to that observed for the same spores heated in lucite, epoxy, and on stainless steel strips. The rate at which dried spores absorb water from a humid environment and the effects of such absorption on thermal resistance was also studied. Results indicated that the spores did absorb water and that the thermal resistance was increased in proportion to the increased spore moisture content. Also developed was a dry heat exposure system employing spores embedded in epoxy resin. This system will be used to determine the effect of a_w on z values. Results were comparable to those reported for spores embedded in lucite.

Section 8

- 9.1. , Quarterly Progress Report, July 1-Sept. 30, 1968,
NASA-CR-98510;QPR-14, Oct. 1968 (STAR, 7, 414, N69-13436)

Results of continuing research on dry heat inactivation of bacterial spores and the influence of spore moisture content on z values are reported. Lyophilization techniques for drying spores in thermal inactivation studies were discounted due to leakage around the vial stopper. Test use of 8 by 50 mm borosilicate glass tubes sealed in commercial tin cans was evaluated and found to be worthy of further study. Three spore crops of *B subtilis var niger* were produced and compared to the 1965 spore suspension which used stainless steel strips as a vehicle. All three had a heat-resistance either equal to or greater than the original crop. Insonation methods for eluting dry spores from the glass tubes were found to be the most efficient. Z values were obtained for spores equilibrated at relative humidities of less than 10%, spores embedded in epoxy containing added water, and spores suspended in water little difference in the values was noted regardless of the initial moisture.

- 9.m. , Quarterly Progress Report, 1 Oct. - 31 Dec. 1968,
NASA-CR-100820; QPR-15, Jan. 1969 (STAR, 7, 2235, N69-24762).

Bacillus subtilis var. niger spores were imbedded in an emulsion of epoxy and various amounts of water in order to observe the effects of epoxy encapsulation on their heat resistance. Heat inactivation experiments at temperatures between 110° to 135°C showed that water located in the epoxy resin influenced the moisture content of the encapsulated spores and significantly increased their dry heat resistance. The length of storage of heated spores at refrigeration temperatures did not effect subsequent assay or recovery.

Section 8

- 9.n. , Quarterly Progress Report, 1 Jan - 31 March 1969,
NASA-CR-101471; QPR-16, April 1969 (STAR, 7, 2671, N69-28642)

Moisture effects on spore heat resistance are investigated, and parameters governing water uptake and loss from spores were studied. Efforts were concentrated on (1) determination of spore moisture, content during various phases of spore preparation process, (2) determination of moisture content of the can-matrix system used during heat resistance determinations, (3) effect of moisture in the environment on spore water content, (4) influence of moisture on the thermal destruction of *Bacillus subtilis* var. *niger*, and (5) effect of spore lyophilizates on the z_D of *Bacillus subtilis* var. *niger*.

- 9.o. , Quarterly Progress Report, 1 April-30 June 1969,
NASA-CR-106007; QPR-17, Aug. 1969 (STAR, 7, 4115, N69-38410)

Studies are centered upon the establishment and the evaluation of a rapid procedure for preparing spores for dry heat inactivation, obtaining ancillary data on water sorption with the spores of *B. Substilis* var *niger*, and identifying isolates from Apollo 10. A statistical application to thermal death time of the spores is included.

- 9.p. , Quarterly Progress Report, 1 Jul-30 Sept 1969, NASA-CR-107933;
QPR-18, Nov. 1969 (STAR, 8, 1185, N70-19091)

Processes involved with thermal inactivation determinations were examined. Emphasis was placed on: (1) heat penetration studies of the heat exposure system and modification to accommodate stainless steel cups in lieu of tubes used in previous studies, (2) comparison of D values as obtained from plate-count and most-probable number data, and (3) studies on the rate of cross contamination between cups in the test system. Cultures from Apollo 11 were examined for growth characteristics, colony appearance, and pigment production. Tables of various culture properties data are included.

Section 8

- 9.q. , Quarterly Progress Report, 1 Oct. -31 Dec. 1969,
NASA-CR-109406; QPR-19, March 1970 (STAR 8, 1962, N70-24447)

The results of thermal inactivation research are presented. Accomplishments reported include: (1) preliminary studies on the inactivation of *B. subtilis* var. *niger* in the range of mean concentrations of 1 million to 0.01 test organisms per sample, (2) determined the amount of water in the system when spores were exposed to heat, and (3) characterized 1247 isolates from Apollo 12.

- 9.r. , Quarterly Progress Report, 1 Jan.-31 Mar. 1970, NASA-CR-111387, QPR-20, June 1970 (STAR 2, 151, N71-11073)

Thermal inactivation determinations of *Bacillus subtilis* var. *niger* spores at 125 C under 0.25 micrograms of water per ml of headspace air were analyzed for the ecology and thermal inactivation of microbes in and on interplanetary space vehicle components. An inactivation curve was established using experimental designs which statistically identified the within-run and between-run variations. Experiments using the same conditions except with 100 micrograms of water per ml of headspace air were also performed. Inactivation curves for *B. subtilis* var. *niger* spores at 125 C with 0.25 microgram of water per ml headspace air when the spores were dried in distilled water were compared with similar data using phosphate buffer as the suspending fluid before drying. The dry-heat resistance of two strains of *B. subtilis* spores was established to have a high wet-heat resistance.

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- 9.s. , Quarterly Progress Report, 1 Apr.-30 Jun.
1970, NASA-CR-113870; QPR-21, Sept. 1970 (STAR, 8, 4257,
N70-41163)

Experiments were conducted, involving thousands of spore populations, to identify the nature of the thermal inactivation curve of *B. subtilis* var. *niger* spores under 2.6, 10, and 100 micrograms of water per ml of headspace air. The spores were suspended in 95% ethyl alcohol, diluted in sterile double distilled water, and dispensed in 0.01 ml amounts into stainless steel cups to give about 1 million spores per cup. The cups were placed in tin cans and dried in a vacuum oven for 90 minutes at 46 to 50 C at 1.5 inch Hg pressure. Spore survivors from cups containing less than 10 spores per cup were assayed by sonifying the cups in peptone water and plating. Cups were scored for growth or no growth and the most probable numbers of survivors per cup calculated from these data. Typical thermal inactivation curves are given.

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9.t. , Quarterly Progress Report, 1 Oct.-31 Dec. 1970,
NASA-CR-121727; QPR-23, March 1971 (N71-34054)

A continuing investigation in identifying the thermal inactivation curve of *B. subtilis* var. *niger* spores is reported. Several experiments were conducted to determine the nature of the curve under 0.25, 2.6, 10, 100, and 500 micrograms of water per ml of headspace. The conventional plate count method was used in assaying spore survivors ranging from 1 million to less than 10 spores per cup. The data obtained are given along with discussions as to their significance. In addition, the results of tests conducted on the hardware and equipment to be used for the evaluation of a terminal sterilization process for unmanned landers are presented.

Section 8

10. Planetary Quarantine Program, Sandia Corp., Albuquerque, N. Mex., NASA Order H-13245A, NASA Order R-09-019-040, NASA Order W-12853. See following series of quarterly reports.

- 10.a. , Quarterly Report for Period Ending 30 Sept. 1968, NASA-CR-97462; QR-10, 30 Sept. 1968 (STAR, 7, (1), 14, N69-10213)

A preliminary copy of the NASA Contamination Control Handbook was reviewed for errors and correctness of technical content. Mathematical sampling models were developed to identify bacterial species and to quantitatively estimate microbial distribution on Apollo missions. To better understand the nature of particle behavior in controlled environments, three dissemination methods were designed: mechanical mixing, spinning, and acoustical generation of tagging inert particles with spores. A users' manual for the planetary quarantine lunar information system was written. Dry heat survival studies at three different temperatures levels were conducted to aid in generating bioengineering parameters necessary for achieving planetary quarantine objectives.

- 10.b. , Quarterly Report for Period Ending 31 Dec. 1968, NASA-CR-99345; QR-11, Dec. 1968 (STAR, 7, (7), 1100, N69-18046)

Information on clean packaging materials was reviewed and assembled into standard handbook format. Models for estimating spacecraft bioburdens were developed for use in areas associated with manned lunar missions. A recirculating downflow unit was developed and tested for wash efficiency with aluminum oxide particles. Possible effects of lunar contamination by terrestrial organisms from Apollo systems impacting on lunar surface were estimated as well as the likelihood of retrieving any organism during the following Apollo mission. Sterilization modeling and support experimentation for generating bioengineering parameters of planetary quarantine sterilization continued. Based upon model predictions, heating at reduced levels for five to ten days evolved as effective spacecraft sterilization method.

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- 10.c. , Quarterly Progress Report, Period Ending 31 March 1969,
NASA-CR-100815; QPR-12, March 1969 (STAR, 7, 2248, N69-248865)

The current status of the NASA Contamination Control Handbook and the development of the contamination control training course outline are described. A vertical laminar air flow room was constructed for studying the deposition of particles from the air flow in a laminar-flow clean room. The problem of generating a continuous and uniform aerosol has been solved in this facility. Two different aerosols were used in a set of experiments: one contained 10 aluminum oxide particles tagged with *Bacillus subtilis* var. *niger* spores, the other contained individual *Bacillus subtilis* var. *niger* spores. Also briefly described are the programming of a reduced version of a lunar quarantine information system and the development of a kinetic sterilization model that is physically based and consistent with all known forms of survival data.

- 10.d. , Quarterly Progress Report for Period Ending 30 June 1969,
NASA-CR-103823; QR-13, 30 June 1969 (STAR, 7, 3520, N69-33761)

A planetary quarantine computer system for lunar programs was rendered in a reduced version, programmed, and checked out. Work on a kinetic death sterilization model for spore heat inactivation is described. Another model study dealt with the quantification of qualitative microbial sampling. Procedures, materials, and a microscopic particle counter were examined for use in bioburden experiments and modeling. In a thermo-radiation sterilization study, the complementary effects of simultaneous gamma radiation and heat were determined on various spores and on *Streptococcus faecium*. An estimate was completed of the transfer of microorganisms from the surface of the Apollo spacecraft-lunar module adaptor to the lunar module during launch.

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- 10.e. , Quarterly Progress Report, Period Ending 31 Dec. 1969,
NASA-CR-113668; QR-15, Dec. 1969 (STAR, 8, 4060, N70-40306)

Techniques for planetary quarantine and bacterial spore inactivation are discussed. Subjects presented are: (1) bacterial spore inactivation modeling, (2) thermoradiation sterilization, (3) bioburden experimentation, and (4) computerized identification scheme.

- 10.f. , Quarterly Progress Report, Period Ending 31 March 1970,
NASA-CR-111386; QR-16, March 1970 (STAR 9, 150, N71-11066)

Research progress is reported on the following topics: (1) the effect of dry heat sterilization in bacterial spore inactivation modeling, (2) modeling of thermoradiation inactivation of spores, (3) a computerized identification scheme, (4) a general approach to estimating and predicting bioburdens, (5) the effectiveness of thermoradiation of *B. Subtilis*, (6) fabrication and testing of a microbiological air samples, and (7) spore inactivation by ultraviolet radiation.

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- 10.g. , Quarterly Progress Report, Period Ending 30 June 1970, NASA-CR-110046; QPR-17, 30 June 1970 (STAR, 8, 3480, N70-35931)

Experimental activities in thermoradiation sterilization fell into three primary categories. First, further low temperature work was undertaken. Of particular significance was thermoradiation sterilization of *B. subtilis* var. *niger* spores at 60 C with simultaneous gamma radiation at 8 Krads/hr. The D value for this combined environment was found to be 6 hours. The D value in dry heat alone is in excess of 53 hours at 60 C. The second area of work was embedded spores. Spores were embedded in methyl-methacrylate and then exposed to thermoradiation environments at 105 C and varying dose rates. A synergism apparently comparable percentage-wise to that found for surface contamination was observed. Thus one D value for embedded spores at 105C and 12 Krads/hr was essentially the same as the dry heat D value for exposed organisms at 105 C without the radiation added. Finally, brief studies were undertaken by other organizations to investigate any deleterious effects of thermoradiation on typical electronic devices and materials and to determine the feasibility of building a relatively inexpensive thermoradiation chamber large enough to handle a Viking spacecraft

- 10.h. Quarterly Progress Report, 30 Sept. 1970, NASA-CR-111309; QPR-18, Sept. 1970 (STAR, 9, 799, N71-16022)

Activities included an appraisal of the overall thermoradiation program along with considerable experimentation in dry heat, radiation and thermoradiation inactivation of *B. subtilis* var. *niger*. Heat resistance and dose rate sensitivity experiments at 95 C revealed promising results at that temperature. At a dose rate of 12 Krads/hour, the D value was two hours. A 10 log population reduction could then be accomplished at 95 C with a total dose of less than 250 Krads in

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(cont.)

20 hours. Good confirmation was also obtained in the dose rate sensitivity of radiation inactivation of room temperature. High rate gamma radiation inactivation at room temperature required twice the total dose that low radiation required. A preliminary analysis of the radiation burden for a Mars Lander derived a safe upper bound of about 6300 krads of radiation encountered post launch.

10.1. , Quarterly Progress Report, Period Ending 31 Dec. 1970, NASA-CR-116420; QPR-19, Dec. 1970 (STAR, 9, 985, N71-18056)

The following activities are discussed: (1) oxygen dependency in radiation inactivation of organisms at elevated temperatures; (2) formation of a model for thermoradiation inactivation of spores that includes a term representing breakage; (3) a behavioral model for spore inactivation as a function of water activity; and (4) a computerized bioassay identification system for Apollo 10, 11, 12 and 14 spacecraft biological data.

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- 10.j. , Quarterly Report for Period Ending 31 Mar. 1971,
NASA-CR-121921; QR-20, Mar. 1971 (N71-36161).

Data covering relative humidity sensitivity of thermoradiation at 95 C, inactivation of naturally occurring spores in soil (PHS Phoenix stock) and the effect of extremely high radiation dose rates on *Bacillus subtilis* var. niger are reported. Results are given in tables and graphs.

- 10.k. , Quarterly Report for Period Ending 30 Jun. 1971,
NASA-CR-119314; QPR-21, 30 Jun. 1971 (N71-30662).

During this period, emphasis was placed on evaluating the problem of the recently discovered hardy soil spores in spacecraft sterilization. As a result, it was recommended that sterilization cycles be based on the estimated actual distribution of resistance among organisms. Other activities reported include: thermoradiation experiments and modeling, the effects of relative humidity on particle adhesion to surfaces, removal of organisms from dust particles, and bacterial spore inactivation.

- 10.l. , Quarterly Report for Period Ending 30 Sept. 1971,
NASA-CR-122848; QPR-22, Sept. 1971 (N72-10076)

A quantitative means was developed to investigate the sensitivity of current spacecraft sterilization plans to variations in D-values. A quantitative expression was derived to represent the distribution of D-values among a population of naturally occurring organisms. An investigation was made of (1) the inactivation of both *Bacillus subtilis* var. niger spores and Cape Kennedy soil spores at elevated temperatures below 125 C. The relation between standard survival experiments with bacterial spores in soils and results obtained on spacecraft surfaces is discussed. Sporicidal properties of aqueous formaldehyde can be increased by elevating the temperature.

Section 8

11. Environmental Microbiology as Related to Planetary Quarantine, Minnesota Univ., Minneapolis, Minn. Grant NGL-24-005-160. See following series of semiannual progress reports.

- 11.a. , Semiannual Progress Report, NASA-CR-99761; SAPR-1, Dec. 1968 (STAR, 7 (8), 1272, N69-18490)

Progress in five research projects is summarized in this report. Four of the projects are concerned with behavior and survival of the *Bacillus subtilis* var. *niger* spores; the last project deals with the detection of low levels of microbial contamination on surfaces by chemical methods and determination of the relative percentages of living and dead cells. The behavior and survival of the spores was experimentally studied at temperatures below 60°C in controlled air flow, and under conditions of heat-fixing at 80°C. The effects of humidity, location, surface finish, and separator thickness on the heat destruction of the spores when located between mated surfaces were also investigated. Experimental procedures, equipment, and results are presented for each study.

- 11.b. , Semiannual Progress Report, 1 Dec. 1968 - 31 May 1969, NASA-CR-113669, SAPR-2, June 1969 (STAR 8, 4060, N70-40307).

Environmental microbiology as related to planetary quarantine is discussed. Subjects presented are: (1) survival of bacillus spores at temperatures below 60 degrees C, (2) survival of bacillus spores in a controlled air stream, (3) effect of humidity, location, surface finish, and separator thickness on heat destruction of bacillus spores, and (5) detection of low levels of microbial contamination on surfaces by chemical approaches.

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11.c. , Semiannual Progress Report, Jun. 1- Nov. 30, 1969,
NASA-CR-110431, SAPR-3, Dec. 1969. (N70-30770-N70-30775).

The survival rates of bacterial spores as a function of relative humidity in sealed plastic containers at 22°C and 45°C are reported. It was found that significant die-away of Bacillus subtilis var. niger spores occurs at relatively low temperatures (45-60°C). This die away is most pronounced at a humidity of greater than 90% but is significant at humidities of approximately 50% or lower. The same effect was noted for natural spores but with considerably longer D-values. Glass strips, when compared to stainless steel, appear to greatly accelerate the die-away at >50% RH.

Analysis of experimental results show (1) Treatment humidity has a much larger effect on D-values in an open system than it does in a mated-surface system. (2) In the open system, both conditioning and treatment humidity affect D-value, y-intercept, and intercept ratio (IR). (3) Both the D-value and y-intercept must be considered in a discussion of microbial survival. (4) The mated-surface system shows a greater variability than does the open system. (5) The spores near the edge of a mated surface tend to have smaller D-values than spores at the center of the plate.

Tests show that heating in a dry-gas environment (<0.03% RH) results in D-values considerably lower than those obtained by heating in an ambient environment (34-36% RH at 22°C or 0.65-0.86% at 110°C). A D-value ratio of approximately 1:4 was noted in almost every trial. A comparison of dry nitrogen and dry air did not reveal any difference between the two gases.

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11.d.

Semiannual Progress Report, June 1970, NASA-CR-122088, SAPR-4, June 1970. (N71-36467).

The long term survival rates of *Bacillus subtilis* var. *niger* spores on glass and stainless steel surfaces, under temperatures less than 90°C and varying humidity conditions, are investigated. The effects of dry heat and chemical approaches in a closed system are also investigated. Attempts were made to measure box-to-box variations at several temperature-humidity conditions. Results indicate little change in box to box conditions; under temperature and humidity conditions, it was found that there is a significant change in survival rate with time. Experiments were carried out in distilled and buffered distilled water. Little difference was observed in the survival rates between the two waters.

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- 11.e. , Semiannual Progress Report, Jun, 1 - Nov. 30, 1970,
NASA-CR-119638, SAPR-5, Dec. 1970. (N71-31601).

The relationship between environmental microbiology and planetary quarantine is discussed. Subjects presented are: (1) survival of microbial spores under several temperature and humidity conditions, (2) detection of low levels of microbial contamination of surfaces by chemical approaches, (3) dry heat destruction rates of microorganisms on surfaces.

- 11.f. , Semiannual Progress Report, NASA-CR-123326, SAPR-6,
June 1971. (N72-11075).

The experiments carried out to determine the effects of temperature and relative humidity on the survival rate of *Bacillus subtilis* var. *niger* spores are reported. The experiments were conducted in environmental chambers at temperatures of 75 and 90°C. Data are also included on the survival characteristics of the spores suspended in sucrose solutions at 90°C with water activities of 0.99, 0.9, and 0.85.

Section 9

STERILIZATION BY FILTRATION

1. Koesterer, M. G., "Guidelines on Preparing Filtered Water for the LAR System", Program Information Release, PIR-1R61-70-117, General Electric, Missiles and Space Div., Philadelphia, Pa., Nov. 6, 1970.

This document provides guidelines and recommendations for an improved method for preparing filtered, sterile water for the LAR System. The recommended method involves the use of the millipore filtration/sterilization approach wherein 0.45 or 0.22 micron pore size filters are utilized. The operating instructions are given by reference to: "Sterilizing, Filtration, and Sterility Testing", Application Data Manual, ADM-20, Millipore Filter Corp., Bedford, Mass. Acceptable equipment are indicated to be available from: (1) Millipore Filter Corp., Bedford, Mass. and (2) Gelman Instrument Co., Ann Arbor, Mich.

Section 9

2. Pistolesi, D., "Highly Pure Water. Primary Material of Industrial Interest", *Inquinamento (Italy)*, 12 (3), 37-41 (1970). (C.A., 73, 91139y (1970))

An app., Millipore Super Q, which meets the requirements for ultrapure H₂O, particularly in the pharmaceutical and microelectronic fields, is described. It comprises 4 modules, chosen for their efficiency in their particular functions, which are in order, a glass fiber prefilter, an activated C absorber, a mixed bed deionizer, and a membrane ultrafilter. The resistivity of the output is monitored. From normal tap water a Super Q can produce 75 l./hr of H₂O with a resistivity of 18 megohm/cm. Approx. 600 l./hr can be produced from a purer feed. Five membrane ultrafilters are available with pore sizes between 0.22 and 1.2 μm.

Section 9

3. Mulvany, T. G., "Sterilization of Industrial Water", Process Biochem., 5 (9), 25-8 (1970). (C.A. 73, 123419h (1970))

Sterilization of large vols. of water by controlled filtration is an acceptable alternative to sterilization by heat or other methods. The filtration system is able to assure abs. removal of all bacteria under any possible condition of flow and pressure fluctuation. The means of the efficiency of the filter (the bubble point test) at any time during this operation, and this information is analogous to a temp. measurement made during heat sterilization. The filter can operate over extended periods (up to several weeks) without the possibility of organisms growing or breaking through to the downstream sterile side. The economics of operating a high-flow rate filtration system, in the same order as heat sterilization, with rapid filtration as an advantage, give rise to significant savings in production processes.

Section 9

4. Saunders, L. (Lorch, Walter F.), "Pyrogen-Free Water", Ger. Offen. 2,001,940, 13 Aug. 1970. (C.A., 73, 91240z (1970)).

Pyrogen-free H₂O, useful for the prepn. of medicinal injection solns., was prepd. by directing deionized H₂O of 10 M Ω-cm through a cylindrical app. contg. a multi-plate membrane filter and cation and anion exchange zones sterilized by ⁶⁰Co-γ irradiation of 2.5 megarad. Ion exchangers used were macroreticular cation and anion exchange resin IR 200 and XE 238, resp. No growth in the sterility test was obsd. after 400 l. H₂O passage at 80-120 l./hr and 7 bar. Pyrogen activity was 0.73 and 0.82° after 150 and 400 l. passage, resp.

Section 9

5. Decker, H. M., L. M. Buchanan, et al, "Filter Applications for Spacecraft Sterilization Program", Army Biological Labs., Fort Detrick, Md. In NASA, Washington Spacecraft Sterilization Technology, pp. 259-272 (1966). (N67-14778).

Filtration as a method of removing bacteria is discussed in relation to the five categories of filters used in biological air cleaning. These are roughing filters; medium efficiency filters; high efficiency filters; ultra-high efficiency filters, also classified as high efficiency particulate air filters; and filters for complete filtration. Test results of filters in each category are tabulated, and it is shown that the higher the efficiency, the greater the pressure drop across the filter. Also discussed is the development of a portable dioctyl phthalate test apparatus, for determining the effectiveness of installed filters in removing 0.3 micron particulate material from air. Methods of air cleaning by washing and scrubbing are reviewed, along with such techniques as the electrostatic precipitator, heat sterilization, and ultraviolet light treatment.

Section 9

6. Hauslein, R. H., "Ultrafine Filtration of Bulk Fluids", Chem. Engr. Progr., 67 (5), 82-8 (1971). (C.A., 75, 25033z (1971)).

A review of ultrafine filtration as applied to removal of very fine particulate and microbiological particles from bulk fluids.

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Section 9

7. "Microbial Contamination in Spacecraft Water System", Final Rpt. NASA-CR-108336, Aircraft Porous Media, Inc., Glen Cove, N. Y., Contract NAS9-9027, 9 Mar 1970, (STAR 8 (1c), 1780, N70-23897)

A study was conducted to determine the degree of microbial back contamination to be expected from the waste water to the potable water in the Apollo Command Module water system. The study shows that a bacteria removal filter is needed immediately upstream of the potable water tank or immediately upstream of the potable water outlet.

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Section 10

CHEMICAL STERILIZATION

1. Koesterer, M. G., "Sterilization Procedure and Sterility Test Protocol for the IAR Water Containment System", Program Information Release, PIR-1R61-70-115, General Electric, Missiles and Space Div., Philadelphia, Pa., Nov. 4, 1970.

This document defines operating procedures for sterilizing the IAR water containment system (water bottle, tubing, and associated components) by treatment with ethylene oxide and defining the efficacy of sterilization by culturing samples and controls for microbiological life forms. The sterilization procedure entails exposing the components and control spore strips to a mixture of 12% wt ethylene oxide and 88% wt dichlorodifluoromethane at a nominal concentration of 450 mg ethylene oxide per cubic foot and 40-50% relative humidity in a closed chamber of plastic (PVC) film for a period of 12-16 hours. The efficacy of the sterilization is defined by culturing samples of the components rinse water supply, the used rinse water, the rinse water millipore filtering membranes, and exposed control spore strips in appropriate culturing media at appropriate conditions of temperature and time.

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Section 10

2. Daley, D. J. and J. L. Sliger, "Development of Parametric Data for the Establishment of an Ethylene Oxide Cycle for the Decontamination of Spacecraft", Final Report, NASA-CR-12 764, Becton, Dickinson and Co., Raleigh, N.C. in cooperation with JPL, Contract NAS7-100; JPL-952169, July 1971 (N71-34057).

A decontamination chamber with the capability of maintaining set parameters of temperature, relative humidity, pressure and gas concentration was designed and fabricated. After establishing proper operation of the chamber, a sufficient number of cycles were conducted to gain some insight into the operation of the chamber and the effects of varying parameters and cycle phases on the efficiency of sterilization of spacecraft-type materials. The test pieces included glass, plastic, and stainless steel strips, capillary tubing, and open and Morton-capped test tubes, which were inoculated in all but one test series with spores of *Bacillus subtilis* var. niger. Morton-capped and capillary tubes were the most difficult test pieces to decontaminate. Stainless steel strips were the easiest test pieces to decontaminate. With respect to the individual parameters investigated, relative humidity appeared to have the most pronounced effect on sterilizing efficiency. An improved spore kill was observed when relative humidity was increased from 30% to 50% in the 50 C cycles. Effects of gas concentration, time and temperature might have been manifest with further testing.

Section 10

3. Tortora, G. D., "Synergistic Effects in Sonochemical Sterilization", Ph.D. Thesis, St. John's University (1970). (Diss. Abst. Int. B, 31 (6), 3587 (1970)).

The synergistic effects observed during sterilization of Bacillus subtilis var. niger ATCC 9372, Bacillus stearothermophilus ATCC 7953, and Serratia marcescens ATCC 4003 by the combined action of ethylene or propylene oxide with high intensity airborne sound waves (34.8 kc/sec) were investigated. Distance of the sample from the sound source was one inch. Effects of concentration of gaseous sterilant, time of exposure, and temperature were investigated. The antibacterial activity of propylene oxide was found to increase sharply with increase in concentration for sonochemically treated spores. The effects of increase in time of exposure were more pronounced at the lowest concentration of propylene oxide. An increase in temperature resulted in an increase in the synergistic effect due to the combined action of large amplitude acoustic waves and gaseous sterilant. Sonochemical sterility was achieved with spores of both B. subtilis var. niger and B. stearothermophilus under the following experimental conditions: propylene oxide concentration, 1000 mg/liter; acoustic intensity, 162 db; contact time, 100 min; temperature, 40 C; and relative humidity, 40%. In the tests in which S. marcescens served as the test organism, sonochemical sterility was attained under the following experimental conditions: propylene oxide concentration, 1000 mg/liter; acoustic intensity, 162 db; contact time, 5 min; temperature, 30 C; and relative humidity, 40%. Accelerated gas diffusion is suggested as the basic mechanism contributing to sonochemical sterilization.

Section 10

4. Imperial Chemical Industries Ltd., "Ethylene Oxide-Containing Compositions as Bactericides or Sterilizing Agents", Fr. Demande 2,043,586, 21 May 1969. (C.A., 75, 144021d (1971)).

Nonflammable ethylene oxide (I) sterilizing agents were prepd. from 3-8:1 $\text{CF}_3\text{CF}_2\text{Cl}$ (II)-I mixts. An 84.7:15.3% II-I mixt. was as useful as a conventional 88:12% CCl_2F_2 -I one; a given I concn. was used at much lower pressures with II than with CCl_2F_2 .

Section 10

5. Long, E. T., "Sterilization with Ethylene Oxide", U.S. Patent 3,498,742, 6 May 1970. (C.A., 73, 18481z (.970)).

A portable sterilizing app. in which a mixt. of 10-20% ethylene oxide and 90-80% CO₂ or methane halide is used as sterilant is described.

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Section 10

6. Velecky, R. and K. Brazdova, "Sterilization with Ethylene Oxide",
Cesk. Hyg., 16 (1), 39-42 (1971). (C.A., 75, 1730y (1971)).

A com. 1:9 mixt. of ethylene oxide and CO₂ (Etoxen) killed the spores
of 5 common pathol. microorganisms, and plastic surgical materials, textiles,
paper, etc. (latex excepted) were not damaged during sterilization.

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Section 10

7. McDonald, R. L. (Sybron Corp.); "Sterilization of Articles by Ethylene Oxide", Ger. Offen. 1,492,494, 8 Oct. 1970. (C.A., 74, 79612k (1971)).

The sterilization process included the partial evacuation of the chamber, raising the relative humidity to 20-50% and maintaining the temp. at 38-50° while treating the contents with a mixt. of 20% ethylene oxide (I) in an inert diluent gas. After the required sterilization time, the chamber was evacuated to remove residual I, then sterile air was introduced to return to ambient pressure before removal of the sterile contents.

Section 10

8. Rendell-Baker, L. and R. B. Roberts, "Safe Use of Ethylene Oxide Sterilization in Hospitals", *Anesth. Analg.*, 49 (6), 919-21 (1970). (C.A., 74, 67657r (1971)).

Safe sterilization of rubber and plastic with ethylene oxide (I) requires 3-4 hr exposure at 49-57° and over 50% relative humidity followed by adequate aeration to eliminate I, ethylene glycol and ethylene chlorohydrin. Aeration time can be cut from 7 days at room temp. to 12 hr by aerating at 50-60° in a properly constructed aerator. γ -Ray sterilized polyethylene should not be resterilized with I because large quantities of ethylene chlorohydrin form which are difficult to remove.

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Section 10

9. Doyle, J. E., A. W. McDaniel, K. L. West, J. E. Whitbourne and R. R. Ernst, "Ethylene Oxide Resistance of Nondessiccated and Dessiccated Spores of *Bacillus Subtilis* var. *niger* Hermetically Sealed in Various Plastic Films", *Appl. Microbiol.*, 20 (5), 793-7 (1970). (C.A., 74, 39711k (1971)).

The following films, in decreasing order of effectiveness, protected *B. subtilis* spores from sterilization by ethylene oxide: mylar-polyethylene laminate, phenoxy, cellophane-polyethylene laminate, nylon, and polyethylene or polyvinyl chloride. Dessiccated spores sealed in the various polymeric films were much more resistant to ethylene oxide sterilization than nondessiccated spores.

Section 10

10. Struppe, H. F., "Effect of Protective Agents on Ethylene Oxide Sterilization", *Gesundheitsw. Desinfek.*, 62 4, 49-53 (1970). (C.A., 73, 76033r (1970)).

Considerable variation was obsd. in the resistance of different preps. of organisms and even of duplicates of the same prepn. to gas sterilization with ethylene oxide. Methods are given for the removal of both water-sol. and water-insol. protective substances from these preps. It was concluded that: (1) ethylene oxide sterilization should only be used when a thermal procedure is not feasible; (2) the material should be clean or thoroughly washed with water to remove protective substances and prevent too high a density of organisms; and (3) fluid preps., gas-tight hollow objects, or objects which contain capillaries or narrow channels should not be sterilized with ethylene oxide.

Section 10

11. Schumann, R. L. and H. Sonnenschein, "Sterilizing Materials in Plastic Bags with a Gas (Ethylene Oxide)", U.S. Patent 3,490,863, 20 Jan. 1970. (C.A., 73, 7243x (1970)).

Materials (usually in plastic bags) are placed in a chamber and sterilized with a gas (such as ethylene oxide). The system provides means to dispense the sterilizing liq. from an aerosol container through a flash evaporator into the evacuated chamber contg. the articles, means for evacuating the chamber, means for stopping the flow of gas when a predetd. concn. (usually differential pressure) is reached, means to time the length of the sterilizing cycle, means for exhausting the remaining liq. in the aerosol container, and means for flushing the sterilizing gas from the chamber.

Section 10

12. Moreno Lopez, D.M., "Ethylene Oxide Handling Technique", *Galenica Acta*, 20 (5-6), 185-93 (1969). (C.A., 73, 28860m (1970)).

Prolonged exposure to ethylene oxide in a modified autoclave at a 450 mg per l. and 54.4° was proved effective for sterilizing objects subject to damage by steam or dry heat. Test organisms were *Bacillus subtilis*, *B. cereus*, *B. stearothermophilus*, and *Clostridium asporogenes*.

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Section 10

13. Kereluk, K. and R. S. Lloyd, "Ethylene Oxide Sterilization: A Current Review of Principles and Practices", American Sterilizer Co., Erie, Pa. (1969), 75 pp. (C.A., 73, 48533k (1970)).

No abstract available.

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Section 10

14. Cleary, D. J., "Residual Effects of Ethylene Oxide Sterilization", Bull. Parenteral Drug Ass., 23 (5), 233-6 (1969). (C.A., 73, 8594n (1970)).

A review was presented on the problems of lowering residual ethylene oxide and its derivs. to nontoxic levels in sterilized materials.

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Section 10

15. Vashkov, V.I., "Chemical Sterilizing Agents", Wiss. Z. Humboldt-Univ. Berlin, Math.-Naturwiss. Reihe, 13 (6), 999-1002 (1969). (C.A., 74, 139934d (1971)).

Based on investigations carried out at several hospitals a survey is given of the clinical applications of a series of chem. sterilizing agents, and the properties and suitability of the agents for different areas of medical work are discussed. Gaseous and liq. β -propiolactone is noted as a highly effective sporicide, esp. suitable for sterilization of biol. material. A 25% increase of the antibacterial activity of ethylene oxide, frequently used as a sterilizing agent in combination with freons or CO₂, is noted in a 1:1.44 mixt. of ethylene oxide and MeBr.

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Section 10

16. Kunenzel, W., H. Jacobi and E. Zillgith, "Sterilization Experiments with Gaseous β -Propiolactone", *Wiss. Z. Humboldt-Univ. Berlin, Math.-Naturwiss. Reihe*, 18 (6), 1003-6 (1969). (C.A., 74, 139939; (1971)).

For the production of gaseous β -propiolactone (I) and evaluation of its antibacterial activity, a small app. for lab. use was constructed of which a detailed description is given. The obtained concns. of I were calcd. at different intervals at 100% rel. humidity and 37°, the max. concn. 9-10 mg/l. being attained after 120 min. The sterilization test was carried out by exposing 1 cm² pieces of paper, each contaminated with 8.9×10^7 spores of *Bacillus subtilis*, to I for increasing periods of time. The min. sterilization time, at which all the contaminated samples were sterile, was 240 min.

Section 10

17. Pierce, A.C. (Allied Chemical Corp.), "Trimethylene Oxides for Vapor-Phase Sterilization", British Patent 1,194,507, 10 June 1970. (C.A., 73, 91241a (1970)).

Concs. of 0.1 ml trimethylene oxide per l. killed 100% of test samples of *Staphylococcus aureus* after 4 hr exposures at 50% and 90% relative humidity at room temp. Comparable bactericidal effects were noted using 1-chloro- and 1-methyltrimethylene oxide vapor and 1,2-bis(trifluoromethyl)-2,3,3-trifluoro-trimethylene oxide. The comds. are useful as fumigants for sterilizing hospital rooms, laboratories, animal quarters, medical equipment, fabrics and plasters.

Section 10

18. Krzywicka, H., and E. Groniowska-Golecka, "Formaldehyde as a Disinfectant", *Probl. Tech. Med. (Warsaw, Pol.)*, 2 (2), 169-76 (1971). (C.A., 76, 517t (1972)).

Elimination of bacterial activity was obtained within 60 min using 0.8% aq. formaldehyde (50-00-0) solns. on *Staphylococcus aureus*, *Escherichia coli*, *Proteus vulgaris*, and *Pseudomonas aeruginosa*, and within 90 min using 6.8% aq. solns. on the *Bacillus subtilis*, *B. cerus*, *B. stearothermophilus*, and *Clostridium sporogenes*. In a gaseous formaldehyde (0.25 mg/l., 22°), sterilizer 99% sterilization was obtained for *S. aureus*, *E. coli*, *Proteus vulgaris*, and *Pseudomonas aeruginosa* after 90 min exposures, for *Mycobacterium tuberculosis* after 1 hr 40 min., and for *Bacillus subtilis* after 7 hr 30 min. The inner, middle parts of CATHETHERS were still CONTAMINATED even after 48 hr in the formaldehyde chamber.

Section 10

19. Karaki, I. and T. Hattori, "Sterilization of an Alcohol Fermentor by Formalin", Hakkō Kyōkaishi, 28 (7), 228-34 (1970). (C.A., 74, 139466c (1971)).

Sterilization of the fermentor for alc. production with formalin was tested. Treatments of a 100 Kl. fermentor with 166 ml formalin for 20, 60, and 120 min were effective against bacteria, fungi, and wild yeasts, resp. Against the usual contaminants in mash, treatment for 90 min with 332 ml formalin was effective. Corrosion of the iron fermentor was not a problem; it was the same as that with tap water.

Section 10

20. Krzywicka, H., "Disinfectant Activity of Peracetic Acid on Spores of Bacteria", *Rocz. Panstw. Zakl. Hig.*, 21 (6), 595-9 (1970). (C.A., 74, 108606u (1971)).

Disinfectant activity of peracetic acid was studied on the spores of *Bacillus cereus*, *B. stearothermophilus*, *B. subtilis*, *Clostridium perfringens*, *C. sporogenes*, and *C. tetani*, using the detn. of the phenol coeff. as well as the modified AOAC method for testing the activity. *B. cereus* was the most resistant, being killed by 3 min exposure to 0.3% and by 90 min exposure to 0.01% peracetic acid. In practice, it is recommended that 0.5% peracetic acid and 10 min exposure be used.

Section 10

21. Muecke, H. and M. Sproessig, "Properties of Peracetic Acid",
Wiss. Z. Humboldt.-Univ. Berlin, Math.-Naturwiss. Reihe, 18 (6),
1167-70 (1969). (C.A., 74, 124717m (1971)).

Aq. soln. (0.2%) of AcOOH were used for disinfection. The sterilizing effect on spores, fungi, viruses, and bacteria, the corrosive behavior, and the stability of the solns. at different pH-values and temps. were studied. The properties of 100% and 40% AcOOH are reported.

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Section 10

22. Trujillo, R. and N. Laible. "Reversible Inhibition of Spore Germination by Alcohols", Appl. Microbiol., 20 (4), 620-3 (1970). C.A., 74, 1431f (1971)).

Low levels (0.003-2.0%) of alcs., such as EtOH, iso-PrOH, isoamyl alc., and octanol, inhibited spore germination of *Bacillus subtilis* and *B. pumilus*. The extent of germination was dependent on the concn. of alc. present in the germination medium. The concn. of alc required for complete inhibition of germination decreased as its mol. wt. increased. Removal of the alc. from the environment allowed germination to proceed, indicating that the inhibition was reversible.

Section 10

23. Juers, R. (Kommanditgesellschaft Argentox Gesellschaft fuer Ozon-Verfahrenstechnik m.b.H. und Co.), "Disinfection of Water by Ozone", Ger. Offen. 1,909,758, 4 Feb. 1971. (C.A., 74, 79461k (1971)).

Water of musty odor and yellow color was disinfected by adding 1 mg $O_3/l.$ and, after 5 min reaction time, appropriate amts. of $FeSO_4$ or $NaHSO_3$ solns. for removal of excess O_3 to give clear water of fresh odor suitable for swimming pools.

Section 10

24. Sokolova, N. F., I. I. Troitskii and T. M. Rybalko, "Hydrogen Peroxide and Potassium Permanganate for Purification of Water of Spore Forming Bacteria", Tr. Tsent. Nauch.-Issled. Dezinfek. Inst., 1969 No. 20, 172-8. (C.A., 74, 115771x (1971)).

H_2O_2 in 40 mg/l. dosage has weak sporicidal properties. $KMnO_4$ (I) in 1-3 mg/l. dosage also increased the purification effectiveness of water from spores with its chlorination with partially basic $Ca(OCl)_2$ (II) and coagulation with $Al_2(SO_4)_3$ (III). Waters were purified more effectively from spore forms with addn. of reagents in the following order: III-I-II. By using Al oxychloride and I, the purification effectiveness with II decreased.

Section 10

25. Cooke, A. H., "Chlorination of Water as Carried Out by the Metropolitan Water Board (London)", Chem. Ind. (London), 1971 (6), 164-9. (C.A., 74, 102923y (1971)).

Treatment of river and well waters, control of chlorination, relative germicidal efficiencies of Cl and Cl compds., disinfection of water mains, and Cl and enteroviruses are discussed.

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Section 10

26. Malpas, J. F., "The Use of Chlorine for Water Disinfection in Industry", Chem. Ind. (London), 1971 (4), 111-15. (C.A., 74, 102924z (1971)).

The use of Cl for water disinfection in the food and papermaking industry, cooling water, reuse of sewage effluents, and the effect of Cl on corrosion are discussed. 15 refs.

227 <

Section 10

27. Bruce, R. A., "Specialized Halogen Generator, Patent Application", NASA-Case-XIA-8913, 9 Oct 1969 (STAR, 8 (19) 3474, N70-35681)

An apparatus is described for generating controlled quantities of a halogen gas as needed in a water purifying system. The apparatus is designed to eliminate bacteria from contamination water and render it safe for human consumption. The chlorine generator is for use in a closed integrated life support system on board manned spacecraft for sterilizing potable water and maintaining stowed water in a sterile condition.

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Section 10

28. Dvir, J. and L. Halpern, "Prolonged Storage of Potable Waters in Covered Reservoirs", *Terres Eaux*. 1970, No. 65, 23-5. (C.A., 74, 102925a (1971)).

Surface and well water with concns. of free Cl in the 0.2-2 mg/l. range stored from 6 months to 2 years at temps. of 6-38° in 10 covered concrete reservoirs of 500 m³ and one of 10,000 m³ showed no changes in compn. or characteristics during storage. The reservoirs had been cleaned and disinfected with Cl before filling with chlorinated water. Tablets of Ca(OCl)₂ were introduced periodically. During the first 2 months, the Cl demand was 3 mg/l. which then fell to 0. The stored water was periodically analyzed from bacteriol, biol., and phys.-chem. standpoints. Potable water can be stored for long periods in covered concrete reservoirs if the free Cl is maintained in the 0.2-2 mg/l. range.

Section 10

29. Sokolova, N. F., I. I. Troitskii and N. K. Zhuchkova, "Effectiveness of Chlorine Dioxide as Disinfectant Against Spore Forming Bacteria in Water", Tr. Tsentr. Nauch-Issled. Dezinfe. Inst., No. 20, 179-84 (1969). (C.A., 75, 25168x (1971)).

Disappearance intensity and disinfection effectiveness against spore forming anthracoid bacteria culture strain 96 in water increased with increase of ClO_2 and contact time. For increasing the sporicidal effect, ClO_2 dosage should be increased and not the contact time with it. ClO_2 is ~5 times more active than Cl^1 .

200<

Section 10

30. Ruetschi, R. and R. Gehring (Schweizerische Sodafabrik), "Aqueous Solutions of Chlorine Dioxide for Sterilizing Water", Swiss Patent 498,045, 15 Dec. 1970. (C.A., 74, 79460j (1971)).

A method is described for the prepn. of ClO_2 in soln. by using a min. of 5% Na chlorite and a min. of 5% HCl in a molar excess of 250-300% with respect to the NaClO_2 , and in the presence of at least one catalyst. During use of the ClO_2 for disinfection, the pH is maintained between 7.0 and 7.4. As an example, 5% NaClO_2 soln. contg. 0.2% Na persulfate is mixed in a 1:1 ratio with 6% HCl and allowed to react at room temp. for approx. 10 min. The formation of ClO_2 exceeds 90%. By using 7.5% NaClO_2 and 9% HCl, the formation of ClO_2 exceeds 95%.

Section 10

31. Fielding, G. H., R. A. Meihol, W. H. Echols and R. L. Dimmick, "Disinfection of Resistant Spores with Hypochlorous Acid", Amer. Chem. Soc., Div. Water, Air Waste Chem., Gen. Pap., 2 (2), 88-9 (1969). (C.A., 74, 98711h (1971)).

Nylon-cotton and wool fabrics were contaminated with the highly resistant spores of *Bacillus subtilis* var *niger*. With Ca hypochlorite solns. maintained in the form of HClO by means of pH control, the spores on fabrics were killed at about the same rate as found by others for spores freely suspended in water. To minimize fabric degradation and metal corrosion, which can result from protracted action of hypochlorites, citrate was added as a slow-acting reducing agent. In such solns., the half-life of HClO can be selected within wide limits; one min was a convenient figure. These solns. killed spores as rapidly as the stable solns., but, since no detectable HClO was detectable after a few min, their corrosivity was low. Using either the stable or the citrate-contg. solns., disinfection has been performed on men, their outer clothing, underwear, socks, and shoes; on a wide variety of construction materials; and on some elec. and electronic items, all without noticeable damage.

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Section 10

32. Sokolova, N. F. and E. I. Moloshavaya, "Disinfection of Water with Some Active Chlorine Compounds", *Wiss. Z. Humboldt- Univ. Berlin, Math.-Naturwiss. Reihe*, 18 (6), 1159-60 (1969). (C.A., 75, 9761e (1971)).

The bactericidal activity of $3 \text{ Ca}(\text{ClO})_2 \cdot 2 \text{ Ca}(\text{OH})_2 \cdot \text{H}_2\text{O}$ (I), N-chlorosuccinimide (II), dichlorodimethylhydantoin (III), and Na and K salt of dichloroisocyanuric acid (IV, V) was tested against *Escherichia coli* and pathogenic bacteria in aq. soln. The compds. are useful for ground water disinfection. Increasing org. substance content and pH value decrease the activity of II and III while I, IV, and V is scarcely affected. Animal expts. showed only low toxicity and no cumulative effects.

Section 10

33. Karalekas, P. C., Jr., L. N. Kuzminski and T. H. Feng, "Recent Developments in the Use of Iodine for Water Disinfection", J. N. Engl. Water Works Ass., 84 (2), 152-88 (1970). (C.A., 15, 9716u (1971)).

Following a review of I literature, death kinetics of several genera of bacteria on exposure to I (1 mg/l. at 3-4°) are presented. The times in sec for 99.99% cell destruction at pH 5.0, 7.0, and 9.0, resp., were: Escherichia coli K 12; 54, 81, and 210; Aerobacter aerogenes (Enterobacter aerogenes) PRL-R3: 63, 58, and 70; Pseudomonas aeruginosa ATCC 7700: 120, 110, and 180; Salmonella senftenberg 775 W: 110, 140, and 280; Streptococcus faecalis ATCC 882: 180, 340 (by extrapolation), and 1000 (assume continuing latter trend of survivor curve); and Staphylococcus aureus 150: 45, 50, and 320 (by extrapolation). 32 refs.

Section 10

34. Layton, R.F., "A New Method of Water Disinfection Involving the Oxidation of Iodide Ion with Monochloramine", Ph.D. Thesis, University of Cincinnati, 1970. (Diss. Abstr. Int. B, 32 (1), 252 (1971))

The theoretical considerations concerning the oxidation of iodide ion with monochloramine to develop a new method for disinfection of water have been studied. From the data collected this method of disinfection of water and wastewater systems seems very promising. The kinetics, stoichiometry, and completeness of this reaction to form hypiodous acid are all favorable to increase the potential usefulness and low cost of this disinfectant scheme. The important variables of pH, temperature, concentration of disinfectant, and concentration of organisms were all quantitatively studied in this work and illustrate various conclusions.

Conventional "chloramine" treatment is a poor disinfection procedure at best, and almost useless at worst. Speculation would lead one to predict that many water systems using this procedure are biologically unsafe.

The addition of iodide ion to these monochloramine systems decreases the time required for 99.99% kill of the test organisms used by a factor ranging from 6 to 10.

Increases in pH ranging from 7 to 9.5 had very little effect on the biocidal properties of the monochloramine-iodide ion system but as was previously known decreased greatly the killing ability of the monochloramine alone. From a practical standpoint, pH values of 8 to 9.5 are very common in most water distribution systems, and the addition of iodide ion would increase notably the ability to adequately disinfect water supplies under these conditions.

The reaction stoichiometry for the iodide ion-NH₂Cl system was shown to be 1:1 with the following reaction involved:



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(cont.)

In general, the addition of iodide ion to a "chloramine" treated water appears to be a low-cost, easy-to-handle system that would require little more than rudimentary addition of the iodide ion prior to entry of the water into the distribution system.

The stoichiometry and mechanism of leuco crystal violet as a redox indicator for monitoring hypiodous acid in the presence of "chloramines" and excess iodide ion has also been demonstrated. The hydride-transfer mechanism involved with this indicator has been shown to be applicable for analysis of other compounds.

An outcropping of this research has been the development of a new, sensitive, colorimetric procedure for ozone in both air and water samples. Data were generated to show the applicability of leuco crystal violet to ozone and its probable reaction mechanism.

Section 10

35. Hurst, N. S. and P. L. Schwartz, "Evaluation of the Hypoiodous Iodine Tablet", Rpt. No. NMFRL-VOL-XXI/No-7, Navy Medical Field Research Lab, Camp Lejeune, N.C., April 1971 (AD-723561)

Investigations were made to determine the feasibility of utilizing hypoiodous acid tablets (HOI) as an agent for microbial decontamination of water. The paper presents results of studies of the palatability, disinfectant capability and iodine content of the HOI tablets stored at 4°C, ambient conditions, and 29°C with a relative humidity of approximately 85%, tablets stored at 29°C with a relative humidity of approximately 85% turned yellowish-green, and the time required for a tablet to dissolve was increased. Storage conditions influenced the iodine content, coloration and solubility of the HOI tablets.

2078

Section 10

36. Taylor, S. L., L. R. Fina and J. L. Lambert, "New Water Disinfectant: An Insoluble Quaternary Ammonium Resin-Triiodide Combination That Releases Bactericide on Demand", *Appl. Microbiol.*, 20 (5), 720-2 (1970). (C.A., 74, 45370q (1971)).

Strongly basic anion-exchange resins form stable, water-insol. combinations with triiodide ions. The combinations have remarkable antibacterial properties: 3.0×10^5 *Escherichia coli* cells per ml were killed when passed through a 3.8 g column of com. available resin treated with triiodide (vol. 4 ml after treatment). In an attempt to deplete the resin-triiodide complex, 1.14×10^9 *E. coli* cells in 15 l. were passed through the column with no significant loss of effectiveness. The antibacterial capabilities of the resin-triiodide columns ranged from 10^6 *Salmonella typhimurium* per ml to 1.1×10^4 *Streptococcus faecalis* per ml. *Staphylococcus aureus* and *Pseudomonas aeruginosa* were also tested and killed at concns. of 1.8×10^4 and 1.3×10^5 per ml. resp. The cells were not filtered from the water. They emerged from the column in nonviable form. This was demonstrated by using ^{14}C -labeled bacteria. The irreversible nature of the antibacterial action was revealed when attempts to wash the damaged cells did not restore viability.

Section 10

37. Ilic, M., "Disinfectant Effect of Iosan on Nonsporogenic Bacteria", *Mikrobiologija*, 7 (1), 87-91 (1970). (C.A., 74, 84391s (1971)).

Iosan, a prepn. from the iodophor group, at a concn. of 0.025% kills *Escherichia coli* and *Staphylococcus aureus* within 5 min and *Proteus* and *Pseudomonas aeruginosa* within 10 min at 18-20°. By addn. of 20% serum to the bacterial suspension, the disinfectant effect of Iosan is decreased by ~40 times. *Pseudomonas aeruginosa* is the most resistant while *E. coli* is the most sensitive to Iosan.

Section 10

38. Salkin, Nicolas, "Germicidal Acidic Composition for Removing Scale and Disinfecting Equipment", Fr. Demande 2,029,396, 27 Nov. 1970. (C.A., 75, 9883w (1971)).

In order to simplify the procedure and to gain time, the removal of scale and the disinfection of equipments, tanks etc. in the food industry, particularly in dairies, breweries, etc. is carried out simultaneously by using germicidal acidic compns. contg. at least 20% acid and 0.01-5.00% of free iodine. Thus, 12.5 g of Antarox Vrozo, an iodophore prepn. contg. 20% free iodine was mixed with 17.5 g of a nonylphenol condensate with 15 moles. ethylene oxide, and the mixt. added to a soln. of 970 g 51.5% phosphoric acid.

2-10-

Section 10

39. Granimar, A. G., "Bactericidal Detergent Compositions", Fr. 2,032,967, 27 Nov. 1970. (C.A., 75, 22959v, (1971)).

The title compns. were prepd. from solns. of ICl_3 and anionic and (or) nonionic surfactants in H_2O and (or) in org. solvents. Thus, ICl_3 6, Na dodecylbenzenesulfonate and Na dodecyltri(oxyethyl) sulfate 4, and H_2O 90% gave a tasteless soln. that was storage-stable in the presence of metals.

2.115

Section 10

40. Theisig, F., H. Schmitt, and J. Brettner. "2, 4, 6-Tris (chloromethyl)-1,3,5-hexahydrotriazine as Microbicidal Additive for Technical Products", Ger. Offen. 1,961,995, 30 April 1971. (C.A., 15, 34489e (1971)).

The title compd. (I) was used to prevent decomn. of tech. products, e.g. paper, coating materials, and adhesives by bacteria and fungi. Thus, a dispersion dye contg. 0.1% I was totally protected against infections by bacteria and fungi, e.g. *Aspergillus niger*, *Chaetomium globosum*, *Penicillium funiculosum*, *Penicillium citrinum*, and *Pullularia pullularis*.

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Section 10

41. Albright, C. F., et al., "Development of an Electrolytic Silver-Ion Generator for Water Sterilization in Apollo Spacecraft Water Systems: Apollo Applications Program", Final Rpt., NASA-CR-65738, Garrett Corp., AiResearch Div., Contract NAS9-3541, June 1967 (STAR, 7 (4), 611, N69-14494)

An electrolytic water sterilizer was developed for control of microbial contamination in the Apollo spacecraft. Individual units are self-contained and require no external power or control. The small size (2.5-in. diameter by 4 in. long) light weight (0.6 lb) and absence of interface requirements make it possible to incorporate such sterilizers at various desirable locations in the potable water system or the waste water system. The sterilizer produces silver ions in concentrations of 50 ppb to more than 200 ppb in the water flow system, the desired concentration being adjusted to the average water flow rate. After installation, no maintenance is required. The unit can be neglected with no damage to the cell or the system, since it becomes self-limiting if water flow is shut down. An external shunt is provided for on-off function and monitoring of current flow. Probable life expectancy is 9000 hr without a change of batteries. Laboratory tests under simulated conditions have demonstrated essentially complete kill of *Staphylococcus aureus* and *Escherichia coli* within 8hr using initial bacterial concentrations greater than 5×10^5 organisms per ml.

Section 10

42. Albright, C. F. and J. B. Gillerman (U.S. NASA), "Electrolytic Cells for Water Management System", U.S. Patent 3,457,801, 15 Dec. 1970. (C.A., 74, 91072s (1971)).

A metal ion method of bactericidal control by the oligodynamic effect maintains potable water for extended periods. Ag ions are generated in a centrally located Ag cathode. The housing around the cell permits flows up to 7 ml/min over the anode. An elec. control system consists of an Ag oxide battery (100 mA hr, 4.5V) and resistor (1 Mohm) which limits the current. The Ag ions are released according to Faraday's law $Q = 0.001 i$, when $i =$ current (μ A). Thus at 10 ml/min and a desired concn. of 100 ppb the required amperage is 14.9 μ A. Fluid flow prevents deposition of Ag ions on the cathode. The cathode is Al Ag-plated 0.001 in. thick and the anode is 0.001 pore Ag. Tetrafluoroethylene may be used as a support material. In a test of the system Escherichia coli and Staphylococcus aureus were killed in a flowing system within 24 hr. Ag ions in concns. below 50-100 ppb may be safely ingested. This system provides a continuous flowing stream of Ag ions to provide purification and disinfection of water for space applications.

2.115

Section 10

43. Cliver, D. O., et al., "Biocidal Effects of Silver", Final Tech. Rpt., NASA-CR-108338, Wisconsin Univ. Food Research Inst., Madison, Wis., Contract NAS9-9300, Feb. 1970 (STAR 8 (10), 1778, N70-23888)

An investigation to determine if silver ions can kill or inactivate microbial and viral agents in very pure water is reported. The results are to be applied in the design of future spacecraft water systems. Salts of silver were employed in many of these experiments, but silver ions generated by an electrolytic apparatus were used when possible. Silver was assayed by neutron activation analysis. Bacteria employed as experimental models were selected on the basis of tests of previous spacecraft water systems. Viruses were chosen simply to represent a broad variety of agents.

Section 10

44. Cliver, D. O., et al., "Biocidal Effects of Silver", Final Tech. Rpt., NASA-CR-114978, Wisconsin Univ., Madison, Wis., Contract NAS9-9300, Feb. 1971 (STAR, 9 (13), 2031, N71-24436)

The ability of silver ions to kill or inactivate microbial and viral agents in very pure water and possible application to the design of future spacecraft water systems is discussed. Salts of silver were employed in many of these experiments, but silver from an electrolytic ion generator was used when possible. Fairly extensive results of sampling previous prototypes and spacecraft water systems were available to guide the choice of test organisms. Many of the bacteria were laboratory or prototype strains, but some had actually been isolated from water systems. The viruses were selected very arbitrarily to represent several groups. There had been no previous effort to detect viruses which might be present in spacecraft water systems, but at least two of the groups represented here are detected fairly frequently in community wastewater.

Section 10

45. Shaydorova, V. V., S. V. Chizhov, Yu. Ye. Sinyak, A. A. Ballod, and N. A. Sokolova, "Producing and Evaluating the Effectiveness of Silvered Filters for Decontaminating and Preserving Water", translation of Space Biol. and Med. (USSR), 5 (5), 54-60 (1971), Joint Publications Research Service, Washington, D.C., 22 Dec. 1971 (N72-14072).

The experimental characteristics are given for the contact method of decontaminating water using silver reduced on the surface of activated charcoals and ion exchange resins using ascorbic acid, hydroquinone and formaldehyde. It was established that the dynamics of dissolving of ionic silver from silvered surface is dependent on the chemical nature of the sorbent and on the method for reducing the silver. In this method the decisive role is played by impurities present on the sorbent surface. A filter consisting of a silvered KU-2 x 8 cationite, reduced by hydroquinone, and AG-5 activated charcoal exhibited a stable rate of washing of silver from the surface.

Section 10

46. Stroganov, N. S., "Organotin Compounds Used in Control of Plankton Growth", JPRS-39292; TT-66-35714, Joint Publications Research Service, Washington, D.C., Translation from Dokl. Akad. Nauk SSSR (Moscow), 170 (5), 1189-91 (1966).

The algi- and zoocidal properties of organotin compounds were investigated to determine the feasibility of using these compounds for water purification in industrial enterprises. Results show that organotin inhibits the viability of algae and daphnia even at a concentration of 0.02 mg/liter. It is concluded that organotin compounds have an advantage over mineral tin because they prove toxic for planktonic organisms at substantially lower concentrations.

Section 10

47. Good, Hans, "Organotin Disinfectant", Ger. Offen. 2,045,337, April 1971. (C.A., 75, 121428c (1971)).

The disinfectant is a 0.05-10 wt.% soln. in H₂O, alc. or aq. alc. of a mixt. contg. a trialkyltin hydroxide or its carboxylic ester (I) 1-10, a quaternary ammonium compd. (II) 0.05-100, a lower aliphatic dialdehyde (III) 10-200, salicylic acid or its salts 0.1-10, and \geq 10 parts by wt. iso-PrOH. I is preferably Bu₃Sn benzoate, II is dimethyl(dichlorobenzyl)ammonium chloride, and III is glyoxal or glutaraldehyde. An emulsifier such as an alkylphenyl polyethylene glycol ether (1-20 parts) may be added. A typical compn. is H₂O contg. I 0.05, II 0.5, glyoxal 0.4, iso-PrOH 0.4, emulsifier 0.09, Na salicylate 0.03, glycerol 0.25, and EtOH 1.3 wt.%.

Section 10

48. Loncin, M., J. A. Kozulis and P. D. Bayne, "n-Octyl Gallate: A New Beer Microbiological Inhibitor", Amer. Soc. Brew. Chem. Proc., 89-101 (1970). (C.A., 74, 98167k (1971)).

n-Octyl gallate (I) and n-heptyl p-hydroxybenzoate (II) are both good beer sterilants. Because of the chem. nature of I, higher concns. can be used relative to II with increased antimicrobial activity. Application problems in the brewery are minimized compared with II because of the higher soly. of I. I showed no evidence of hydrolysis in beer at room temp. and 100°, and it had no influence on beer flavor and odor compared with untreated controls. Storage trials showed no spoilage in 3 months.

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STERILIZATION BY RADIATION

1. Silverman, G., "Survey of Certain Nonthermal Methods of Decontamination and Sterilization", Mass. Inst. Tech., Cambridge, Mass. In NASA, Washington Spacecraft Sterilization Technology, pp. 193-206, (1966). (N67-14775).

The feasibility and limitations inherent in using ionizing radiations for spacecraft sterilization are discussed. The types of radiation for inactivating microorganisms are classified into particle radiations and electromagnetic radiations: for practical considerations, these are limited to fast electron, X-rays, gamma rays, and ultraviolet light. The biological effects of ionizing irradiation are examined, and the radiation resistivities of several biological systems are given. Additional factors to be considered in this sterilization technique are identified as the dose rate, the presence of oxygen and organic materials, protective compounds, physiological state, water content, and temperature during irradiation. Microwave and dielectric heating processes and the use of lasers are considered briefly.

Section 11

2. Glasson, D. H. (Aquatron Corp. (Aust.) Pty. Ltd.), "Sterilization by Ultraviolet Radiation", Australian Patent 292,563, 10 July 1969. (C.A., 73, 38545b (1970)).

An app. is described for the pre- and post-bottling sterilization of pharmaceuticals by uv radiation. Bottles passing along a continuous conveyor are filled from a bulk reservoir and capped. Bottles, caps, reservoir and filling tube are of uv transparent material such as polyethylene. Uv tubes situated at 3 points irradiate the empty bottles, the reservoir and filling tube, and the caps. The capped tubes are checked for sterility by measurement of their transparency to uv radiation.

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Section 11

3. Moore, P. W., "Polyethylene Ampuls as Containers for Radiation-Sterilized Parenteral Solutions", Australas. J. Pharm., 51 (608), S57-S62 (1970). (C.A., 74, 6364w (1971)).

Irradn. sterilization of distd. water for parenteral solns. in polyethylene ampuls is discussed. Water irradiated in polyethylene differed in H_2O_2 , dissolved O_2 , and oxidizable matter content and in pH from water sterilized in glass containers, but no differences in the biol. effects of the solns. were found. Problems arising from interaction between water and polyethylene are unlikely to result from irradn. sterilization.

Section 11

4. Rauch, I., "Water Disinfection by UV Irradiation", Ger. Offen. 1,937,126, 11 Feb. 1971. (C.A., 74, 79462m (1971)).

Water is disinfected by uv irradiation at 2537 \AA in a plant consisting of poly (vinyl chloride) tubes impermeable to uv fitted with quartz lamps in the tube axis and with rectangular connections to the feed pipes to give eddy motion. The lamps can be exchanged during operation.

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MISCELLANEOUS STERILIZATION METHODS

1. Zhuk, E. G., "Water Disinfection with a Pulsed Electric Discharge", Zh. Mikrobiol., Epidemiol. Immunobiol., 48 (1), 99-103 (1971). (C.A., 74, 102926b (1971)).

Water inoculated with 2×10^6 - 2×10^{10} bacteria/l. was treated with discharges until no count was obtainable in 0.1 ml; 1.2-1.7 sec were necessary for bacteria (*Escherichia coli*, *Staphylococcus aureus*) and 7.6-16.8 sec for spores (STI strain, anthrax). The increase of *E. coli* from 1×10^6 to 1×10^{10} cells/l. required only triple amt. of energy for complete destruction (19 and 57 impulses, resp.) while the STI spores needed for 1×10^6 and 1×10^7 spores/l. 75 and 155 impulses, resp. Peat dust 1000 and blood serum protein 750 mg/l. have a protecting effect, raising the necessary no. of impulses for 2×10^7 *E. coli*/l. from 22 to 35 and 165, resp. The destruction of *E. coli* occurred at 5 J/ml, that of spores at 22 J/ml.

Section 12

2. Urusov, A., R. Bretosh, and L. Rudenko, "Disinfection of Waste Waters by Submerged Electric Spark Discharge", *Myas. Ind. SSSR*, 41 (2), 22-3 (1971). (C.A., 75, 24941c (1971)).

Waste waters from a Donetsk meat packing plant contg. $< 4.7 \times 10^6$ bacteria/ml water were effectively sterilized to 100-200 bacteria/ml by high voltage spark (3kVA, 80,000 V secondary winding) discharges between electrodes placed 4 mm apart; the app. and method were less effective in the sterilization water contg. spores. A water contg. 500,000 spores/ml showed a residual spore content of 7000/ml after 550 discharges: more powerful discharges produced by a 20 kVA transformer were less bactericidal.

Section 12

3. Hiler, E. A. and W. M. Lyle, "Electrophoretic Clarification of Water", Water Resources Inst., Texas A and M Univ., College Station, Tex., PB Rep. No. 194566 (1970). (C.A., 15, 9763g (1971) and USGRDR, 70 (23), 123 (1970)).

The ease of automation with an electrophoretic system does not justify the high cost of water treatment by this method. Water purification by electrochem. means overcame this problem and was successful both operationally and economically. As a result of the exptl. testing, a sample design of a small semiautomated electrochem. water system is offered which incorporates electrochem. flocculation, settling, and electrochem. disinfection operations.

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Section 12

4. Palmer, C.H.R. and T. D. Whittet, "Removal of Pyrogenicity from Solutions of Purified Pyrogens and Tap Waters Using Ion-Exchange Resins", Chem. Ind. (London), 1971 (13), 341-4. (C.A., 74, 115762v (1971)).

The redn. of pyrogenicity in solns. of pure pyrogens and in tap water by ion exchange, esp. by anion exchange, is demonstrated. The effectiveness of the ion-exchange resin depends on the nature of the pyrogen, but strongly basic ion-exchange resins were esp. useful for tap water.

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5. Sidorenko, G. I., et al., "The Use of the Combined Effect of Gamma-Radiation and Chlorine for Sterilization of Water, JPRS-21975 (Del.) Translated from *Gigiena; Sanit.*, 28; No. 9, 97-8 (Sept. 1963) (Nucl. Sci. Abst. 18, 5697)

The combined use of gamma radiation and chlorine in water sterilization was studied. Sterile water samples were inoculated with *B. coli* and *B. anthracoides*. One set of samples was chlorinated, another irradiated with 25,000 r Co^{60} gamma, and a third both chlorinated and irradiated with doses equivalent to the controls. With the combined treatment, an equivalent effect was obtained with one-third the chlorine and one-tenth the radiation dose.

BIOLOGICAL INDICATORS, SENSORS, AND DETECTION

1. Irons, A. S., et al, "Development of a Biological Indicator for Dry-Heat Sterilization", JPL, Calif. Inst. Tech., Pasadena, Calif. In NASA, Washington Spacecraft Sterilization Technology, pp 177-189 (1966). (167-14774).

The indicator system will consist of a spore tablet, a sealed carrier or container, and a handling procedure designed to prevent inadvertent contamination of the space hardware being assayed. It will be capable of discerning, biologically, the attainment of the conditions considered necessary to sterilize space hardware. Sterilization of the indicator will not, in itself, prove that sterility of the lander capsule has been achieved; it will merely verify that the particular process being utilized was successfully applied. The process used to produce sterility must have been previously shown to be reliable and predictable as to the probability of attaining sterility.

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Section 13

2. Miller, W. S., "The Types of Biological Indicators Used in Monitoring Sterilization Processes", Becton, Dickerson and Co., Raleigh, N.C., 23 Nov. 1970. (STAR, 9 (2), 168, N71-11192)

Several types of biological indicators were reviewed. The preliminary work which preceded their selection and the bases on which a particular indicator was evaluated are given. Information is provided on the importance of preparation method in order to obtain consistent indicator performance. Studies of a number of factors influencing organism resistance indicate that method of preparation can result in resistance differences greater than those normally seen between common indicator species. The critical points for the valid use of biological indicators include correlation of resistance with that of the most resistant organism found on the materials before sterilization, certified and adequately controlled sterilization cycles, uniformity in indicator production and comprehensive sterility test programs for both indicators and products.

21515

Section 13

3. Doyle, J. E., "Sterility Indicator with Artificial Resistance to Ethylene Oxide", Bull. Parenteral Drug Ass., 25 (2), 98-104 (1971). (C.A., 74, 146312c (1971)).

Artificial resistance to ethylene oxide was developed by suspending microorganisms in a mixt. of a plasticizer with a film former. The degree of resistance was 3 times that of freshly harvested spores of *Bacillus subtilis* var niger and the viability and resistance were stable for at least 2 years under ambient conditions.

2/2/75

Section 13

4. Ernst, R. R. (Sybron Corp.), "Biological Sterility Indicator",
U.S. Patent 3,585,112, 15 June 1971. (C.A., 75, 85115n (1971))

Division of U.S. 3,346,464. The indicator consists of a semipermeable envelope permeable to sterilizing media, H₂O, and growth media but impermeable to bacteria in liq. or gases. A selected amt. of an unincubated test organism, a growth medium, and a dye indicator are sealed in the envelope. For testing multiple types of sterilizing media, multiple types of test organisms are used, each of which is sufficiently resistant to at least 1 of the sterilizing media consisting of steam, dry heat, radiation, and gaseous agents.

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Section 13

5. Geating, J. A., and F. P. Rudek, "Bacteria Sensor for Reprocessed Water-Microbiology Research, Design and Fabrication", AMRL-TR-68-173, General Electric Co., Philadelphia, Pa., Contract F33615-67-C-1564, Feb. 1969 (AD-691471; STAR, 7, 4511, N69-41123)

The results of the developmental research leading to the design and fabrication of a short-time electronic sensor to monitor the bacteriological quality of reprocessed water aboard spacecraft are reported. The basic sensing capability is furnished by a Coulter Counter that selectively detects and counts particles of bacterial size. Detection is accomplished by comparing the number of bacteria-size particles in the reprocessed water sample at some point in time with a particle count at some future point in time. i.e., following the establishment of conditions necessary to allow growth and multiplication of bacteria. A significant difference between the two counts strongly implies bacterial replication, and therefore the presence of viable organisms in the raw reprocessed water.

6. "Development of Bacterial Sensing Instrumentation for Spacecraft Water Systems", Final Rpt., NASA-CR-114779, Aerojet-General Corp., El Monte, Calif., Contract NAS9-10432, 13 Nov. 1970 (STAR, 2 (4), 615, N71-14270)

The porphyrin initiated chemiluminescence technique forms the basis of an instrument which was developed for monitoring water sterility in spacecraft water storage and supply systems. The instrumentation permits differentiation between living and dead organisms by comparing the chemiluminescent signals of incubated and unincubated water samples; a higher signal for the incubated sample is used to indicate nonsterility of the water supply. The system selected for the breadboard prototype is both sensitive and rapid and has the potential of being developed into a compact lightweight instrument capable of operation in a zero gravity environment.

Section 13

7. Wilkins, J. R., "Chemiluminescent Bacterial Sensor", in NASA-SP-261, pp 541-545, Langley Research Center, 17-18 Nov. 1970 (STAR, 9 (10), 1498 N71-20993)

The purpose of the chemiluminescent bacterial sensor experiment during the 90-day test was to determine: (1) if the sensor could rapidly detect gross contamination of recovered water and (2) the correlation between sensor response and viable counts. The results showed good correlation between a strong sensor response and viable counts on eight occasions. In 60 tests, sensor response was strongly positive, and the plate counts were either negative or below the level of sensor sensitivity or were not performed. Because the sensor responds to living or dead cells, these results suggest that the recovered water may have contained: (1) dead bacterial cells, (2) porphyrins leached from cells, or (3) substances unrelated to bacteria which triggered a positive response. In general, the sensor was able to detect gross contamination rapidly, required minimum crew participation, and was easy to operate.

Section 13

8. Sharpley, J. M., "Detection of Microbial Contaminants in Space System Water Supplies", Final Rpt. April 1967- June 1968, AMRL-TR-68-67, Sharply Labs., Inc., Fredericksburg, Va., Contract F33615-67-C-1597, Dec. 1968. (STAR 7 (19), 3519, N69-34221)

The feasibility of detecting microorganisms in water by using declining flow rates through a membrane filter has been determined. Results obtained indicate that microorganisms in water may be detected by the decrease in flow rates through a sub-micron filter used under standard conditions. Decreases in flow rates shown by bacteria are appreciably greater than those caused by inert materials. Results from this study indicate that differential filtration rates may be used as a device for the physical detection of microorganisms in potable water in space system water supplies.

1.574

Section 13

9. "Detection and Enumeration of Microbiological Contaminants in Water Used for Processing Electron and Microelectronic Devices, Standard Methods for", ASTM-F-60-68, American Society for Testing Materials, Philadelphia, Pa.

These methods, involving both culturing and microscopic approaches, cover the detection and enumeration of microbiological contaminants including bacteria, yeasts, fungi, molds, algae, and protozoa which may be found in electron device-processing waters. No attempt is made in these methods to establish "acceptable" levels of microbiological contamination since tolerances to such contaminants in industrial systems and processes vary widely.

Section 13

10. Bryce, A. J., "Identification of a Recurring Bacterial Contaminant in a Spacecraft Watering System", NASA-CR-73431, General Electric Co., Philadelphia, Contract NAS2-1900, 11 Aug. 1970 (STAR, 8 (18), 3301, N70-343398)

The recurring bacterial contaminant found in the biosatellite water system was identified as *Ps. boreopolis*. The nature of the organism precluded its control without the employment of strict sterile procedures or chemical agents throughout the spacecraft manufacture and test cycle. Additional treatment of the water system by halogenation or other means would be required to control the contaminating organism.

Section 14

MICROBIOLOGICAL BARRIERS AND STERILE INSERTION

1. Phillips, G. B., "Microbiological Barrier Techniques", Army Biological Labs., Fort Detrick, Md. In NASA, Washington Spacecraft Sterilization Technology, pp. 105-35 (1966). (N67-14770).

The types of microbiological barrier techniques and equipment that could be useful in solving sterilization problems are defined and illustrated. The five stages of microbiological contamination control are listed as (1) recognize and define the problem; (2) establish contamination control criteria; (3) employ approaches and techniques of control; (4) use microbiological testing and surveillance and (5) analyze results and certification procedures. The importance of each is discussed. Microbiological barrier systems are classified according to purpose, size, and degree of contamination, and specific examples of each are given. Recommended sterilization and decontamination agents for each barrier application are listed. Advantages and disadvantages are assessed, with emphasis focused on heat, vapors and gases, liquid decontaminants, and radiation. Recommended conditions of use for such agents are summarized, and certain desirable minimum features for the microbiological barriers are listed.

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Section 14

3. Taylor, D. M., et al., "A Feasibility Study of Liquid Sterile Insertion", NASA-CR-111095, Avco Corp., Contract NAS7-100, 21 July 1969 (STAR, 9 (1), 10, N71-10382)

The development of a concept for liquid sterile insertion into a previously sterilized spacecraft and a test program to evaluate the feasibility of such a system are summarized. The system concept developed had two criteria: (1) a method of reducing system reliability requirements by verifying the probability of sterility of the filtrate prior to actual insertion into the sterile spacecraft, and (2) a method to verify the probability of sterility without assaying the filtrate.

2/1<

Section 14

2. Arnett, J. C., et al., "Design Feasibility Study of Sterile Insertion Techniques", Final Tech. Rpt., NASA-CR 101632, Martin Marietta Corp., Denver, Col. NASW-1621, Oct. 1967, (STAR, 1, (16), 3058, N69-29726)

The objective was to develop a number of design concepts for sterile insertion. These concepts use heat sealing of plastic films to permit aseptic penetration of a container with a sterilized interior. The study had two major portions: one was devoted to the development of the design concepts for the sterilization canister entry port, the service bag, and the heat-sealing and cutting tools; the second was devoted to the materials evaluation and selection of the plastic film for use as the barrier over the entry port. As a result of this study, six feasible design concepts for sterilization-canister entry ports, with corresponding service bags and service-bag mounting methods, were conceived. Three concepts used external plastic barriers; three (including the final recommended design) employ internal plastic barriers. In addition, four designs for heat-sealing tools and five designs for cutting tools were evolved.

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Section 15

WATER STORAGE SYSTEMS AND CONTAINERS

- Rosenwasser, S. N., "Contamination Analysis - ID13005-1 Water Expulsion System Qualification Test Assembly", Catalog No. PDL 104544, Serial No. MP 51,864, Douglas Aircraft Co., Inc., Missiles & Space Systems Div., Sept. 30, 1970 and "Expulsion System Storage Tests", MDC Report E0234, McDonnell-Douglas Corp., Sept. 4, 1970.

Two water expulsion systems, a qualification test unit and a flight unit, were serviced with deionized, sterile water and placed in storage for approximately 4-1/2 and 2 months, respectively, to evaluate the effect of prolonged storage on water quality. The wetted surfaces consisted mostly of 17-4 PH stainless steel but included some 303 S.S., beryllium copper, nickel and an Inconel. Water samples taken at approximately 1-month intervals from the qualification unit showed no measurable change in dissolved metal ion concentrations or in filtration characteristics with a 1.2 micron filter. Post test inspection of both units showed evidence of deposits and corrosion in the qualification unit but not in the flight unit. Analyses of the solid deposits showed Cu, Fe, and Ni to be major components and the presence of Cr, Be, and Al. Analyses of "new" water and the stored water from the two units for dissolved metal ions, resistivity, pH, and flow decay of 1.2 micron filters and porous discs were made and provided the following results.

	<u>New Water</u>	<u>Water from Flight Unit (~2-month storage)</u>	<u>Water from Qualification Unit (~4-1/2 month storage)</u>
Dissolved metal ions, ppm	~0.083	< 0.85	< 2.05
Specific resistance, ohm/cm	952,000	234,000	86,000
pH	6.0	8.5	9
Flow time, minutes			
8.1 ml H ₂ O through 1.2μ filter, 0.10 cm ² area, ~7 psid		0.42	0.67
14.85 ml H ₂ O through 1.2μ filter, 0.10 cm ² area, ~7 psid		0.82	1.42
100 ml H ₂ O through 1.2μ filter, 0.40 cm ² area, ~7 psid	2.57	4.19	9.27
200 ml H ₂ O through 1.2μ filter, 0.40 cm ² area, ~7 psid	5.17	24.28	118.00
Effect on porous disc permeability, % change (initial value 1.4 x 10 ⁻¹¹ in. ²)	-2	0	0

On the basis of these data, it was recommended that the beryllium copper be replaced in the system by 17-4 PH S.S. to decrease galvanic corrosion tendencies. Continued storage tests were suggested to substantiate the "fix".

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Section 15

2. "Expulsion System Supplementary Storage Test", MDC Report E023, Appendix I, McDonnell Douglas Astronautics Co. - West, Huntington Beach, California, Contract F04701-68-C-0034, 29 Dec. 1970.

The flight proof water expulsion unit, ID13005-1, previously subjected to a $4\frac{1}{2}$ -month storage test was retested with filtered, sterile water for an additional 83-day storage period after replacing the beryllium copper components with 17-4 PH S.S. and the Inconel with 302 S.S. Post test inspection of the wetted surfaces showed only minor signs of corrosion (light stains and small spots). Microprobe analyses of the stained areas showed the presence of Fe, Ni, Cr, Cu, and O. Electron microscopic examination showed some evidence of surface pitting in the stained areas on the piston. Analyses of the water stored in the unit showed the dissolved metal concentration had increased from ~ 0.083 to 0.32 ppm, the specific resistance decreased from $952,000$ to $240,000$ ohm/cm and the pH increased from 6.0 to 6.5 . Filter tests of the stored water through 1.2 micron filters showed no significant clogging tendencies at throughputs up to 150 ml/cm² of filter surface area.

21/7/70

Section 15

3. Unclassified excerpts from a McDonnell Douglas Astronautics Co. report concerning water expulsion systems for reentry vehicles. Presumably based on work under "Reentry Vehicle Technology and Observables Program", Contract FO4701-68-C-0034.

Expulsion System Coolant. The water coolant is required to meet the following specifications (as defined by MDAC drawing 1D12811):

Resistivity (minimum)	1 megohm/cm
pH	6.5-7.5
Sterility	<10/ml aerobic count
Particulate Matter (maximum)	
0.5-2.0 microns	2/ml
2.0-5.0 microns	0.1/ml
5.0 microns	0.91/ml

These requirements are met by purchasing commercial distilled water, double deionizing, filtering through 1, 0.45, and 0.22 micron filters, sterilizing by heating in a suitable container for a minimum of 1 hr at 250°F, vacuum filling the water reservoir of the unit, and sterilizing the loaded unit for 1 hr at 250°F. The procedure is referenced to MDAC drawing 1D12901.

Coolant Contamination Tests. Four 8-week tests were conducted to determine the type and amount of contamination which could occur during storage and the effect of it on flow through small passages similar to those in nose tips. The tests consisted of immersing coupons of the reservoir material (17-4 PH S.S., 2-in dia x 1/8-in thick, 30 rms finish, 1.9 surface to water volume ratio) in the specification water contained in Pyrex glass. Test conditions for the four tests were as follows: Test 1, water purposely contaminated by operator's hands and apparatus sealed and stored at room temperature; Test 2, apparatus left open to atmosphere for 1 week prior to sealing and storing as in Test 1; Test 3, apparatus sealed and stored at 110°F; and Test 4, apparatus sealed and stored at room temperature. Water samples were removed at 2-week intervals and analyzed. The test results are summarized in the following table.

3. (cont.)

	Test 1 Time, wk.		Test 2 Time, wk.		Test 3 Time, wk.		Test 4 Time, wk.		Original water					
	4	6	4	6	2	8	2	8						
pH	6.8	7.0	7.8	8.0	44	48.1	77	208	177	134	113	72.7	147	2,000
Specific Resistance, kilohm/cm	>10 ⁸	10 ⁸	10 ⁷	10 ⁶	10 ²	10 ⁴	0	10 ²	10 ²	0	10 ²	0	10 ³	0
Bacteria, count/ml*	1	0.7	1.1	1.1	0.2	0.3	0.4	0.8	0.8	0.4	0.4	0.4	0.4	0.1
Dissolved metals, ppm	<3	<3	<4	<4	<3	<3	<4	<4	<4	<1	<3	<1	<3	<0.22
Size of particulate, microns														

Nose Tip Flow Decay after
8-wk storage

Significant (≥25%)

Negligible

Negligible Negligible

* Bacteria identified as the iron-ingesting species *Siderocapsa*. All samples believed to have been inadvertently contaminated at the 2-week sampling period and Test 2 apparently recontaminated at the 6-wk sampling period.

3. (cont.)

	Test 1 Time, wk.		Test 2 Time, wk.		Test 3 Time, wk.		Test 4 Time, wk.		Original Water					
	2	4	2	4	2	8	2	8						
pH	6.8	7.0	7.8	8.0	44	48.1	77	208	177	134	113	72.7	147.	2,000
Specific Resistance, kilohm/cm	>10 ⁸	10 ⁸	10 ⁷	10 ⁶	10 ²	10 ²	10 ⁴	0	10 ²	0	10 ²	0	10 ³	0
Dissolved metals, ppm	1	0.7	1.1	1.1	0.2	0.3	0.4	0.8	0.4	0.4	0.4	0.4	0.4	0.1
Size of particulate, microns	<3	<3	<4	<4	<3	<3	<4	<4	<4	<1	<3	<1	<3	<0.22
Note: Tip Flow Decay after 8-wk storage					Significant (~25%)			Negligible		Negligible		Negligible	Negligible	

* Bacteria identified as the iron-ingesting species *Siderocapsa*. All samples believed to have been inadvertently contaminated at the 2-week sampling period and Test 2 apparently recontaminated at the 6-wk sampling period.

Section 15

4. Koesterer, M. G., "Results of Sterility Tests on Water Sample Recovered from IAR Water Tank after 16 Weeks Storage", Program Information Release, PIR-1R61-71-105, General Electric, Missiles and Space Div., Philadelphia, Pa., Feb. 26, 1971 and Waite, R., "Tank #1, Blow-Down #2", Memorandum to J. Richardson, General Electric, Re-Entry and Environmental Systems Div., Philadelphia, Pa., March 1, 1971.

These two documents show the effects of ~16-weeks of storage on the sterility, pH, and specific resistance of water stored in a IAR water containment system. The first document shows the water to have been sterile after the storage period based on four culturing tests in Trypticase Soy Broth at 32°C for 7 days. The second document shows the water decreased in pH from 6.5 to 6.2 and the specific resistance decreased from 440,000 to 270,000 ohms/cm during storage.

Section 15

5. Dorfman, D. L., "Development of Standard Procedure for Packaging and Packing of Battery Water", Evaluation Report No. 3232, Industrial Test Laboratory, Philadelphia Naval Shipyard, Philadelphia, Pa., Feb. 1959 (AD-210783)

Eight methods of packaging battery water were evaluated. Four of these methods basically consisted of polyethylene film liners in fiberboard or metal outer containers. Three consisted of molded or blown polyethylene inner containers with fiber, wood, or metal overpacks. One method was an organic plastisol-lined metal pail. The evaluations included storability tests, container shock resistance, and ease of handling. Storability tests at 130°F for a period of at least 3 months showed the water to be within impurity and pH specifications (Federal Specification O-W-41a; Water, Battery, 19 Apr. 1954) after the storage in all containers except the one with the organic plastisol lining (pH decreased to 5.3 and surface blistering occurred). Free fall drop tests showed the container having molded or blown polyethylene inner containers performed satisfactorily while those with polyethylene film liners failed.

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Section 15

6. Townsend, D. E., "Check Test of Canteen, Cold-Weather", Report of Test-Project No. ATB 3-380, USCONARC, U.S. Army Arctic Test Board, Fort Greely, Alaska, 18 May 1960. (AD 237748).

The subject canteen is a vacuum insulated vessel of one quart capacity and constructed of inner and outer shells of stainless steel. It features a non-metallic mouthpiece, a protecting plastic cap, and a nesting type metal cup. Tests revealed no incidents of water, soup or hot beverages becoming rancid, stale, or spoiled. There was no rusting, flaking or chipping of the canteen or cup during the conduct of the tests.

Section 15

7. Kapil, A. L., "Non-Corrodible Dual-Purpose Water Container", GARD Final Report 1479, General American Research Div., General American Transportation Corp., Niles, Ill., Contract No. DAHC 20-68-C-0123, Feb. 1970 (AD 720219, ALRC Library No. 720379).

This report describes the design, fabrication and testing of a 14-gallon, non-corrodible, dual-purpose water container for use in fallout shelters. It consists of a blow molded linear polyethylene cube with two injection molded, ABS side frames. Its major features are: (1) the container is manufactured as a seamless, leak-proof unit, (2) cubical shape allows increased space utilization and stability in stacking, (3) molded hand-holes simplify lifting and stacking, (4) the top is removable for converting empty containers into commodes, (5) container material is approved by the Food and Drug Administration for use in contact with potable water, (6) container material is immune to attack by sodium hypochlorite (Clorox) used as a water disinfectant and is pigmented to prevent ultra-violet degradation, (7) container material will not support combustion, (8) water is easily removed by means of a small, plastic pump provided, (9) minimum hand production labor is needed, and (10) no packing is required so containers can be banded together on pallets for shipping purposes. Compression tests with multiple strain gages, over extended periods of time, show the cube and frames to be structurally sound for stacking up to five-high indefinitely.

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Section 16

WATER STANDARDS AND SPECIFICATIONS

1. "Reagent Water, Standard Specification for", ASTM-D-1193-70, American Society for Testing Materials, Philadelphia, Pa.

This specification covers requirements for water suitable for use in methods of chemical analysis and physical testing. Two grades of reagent water are specified: (1) Type I reagent water may be prepared by the distillation of a water having an electrical conductivity, maximum, of 20 micromhos/cm at 25°C followed by polishing with a mixed bed of ion-exchange materials and (2) Type II grade reagent water may be prepared by single discillation. Reagent water shall conform to the following requirements:

	<u>Type I</u>	<u>Type II</u>
Total matter, max., mg/liter	0.1	2.0
Electrical Conductivity, max., micromhos/cm at 25°C	0.1	5.0
Consumption of $KMnO_4$	pass test	pass test

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Section 16

2. Morris, J. C., et al, "Water Quality Standards for Long-Duration Manned Space Missions", Report of an ad hoc Panel, Space Science Board, National Academy of Sciences, Sept. 1967

This report presents the recommendations of the Panel on physical, chemical, and biological standards for reclaimed water intended for human consumption on spacecraft. The recommended physical standards are: (1) turbidity (Jackson units), not to exceed 13; (2) color (platinum-cobalt units), not to exceed 15; (3) taste, none objectionable; (4) odor, none objectionable and (5) foaming, none persistent more than 15 sec. The recommended chemical standards (max. values, mg/liter) are: As, 0.5; Ba, 2.0; B, 5.0; Cd, 0.05; Cr (hexavalent), 0.05; Cu, 3.0; Pb, 0.2; Se, 0.05; Ag, 0.5; Cl^- , 450; F^- , 2.0; $\text{SO}_4^{=}$, 250; NO_3^- and NO_2^- (as nitrogen), 10.0; chemical oxygen demand (dichromate method), 100.0; and total solids, 1000. Biological quality was of particular concern to the Panel; accordingly, a positive sterilizing procedure, probably involving heat treatment, was strongly recommended. The biological standards goal should be essential sterility; the realistic criterion being a max. of 10 viable microorganisms per ml. Recommended procedures for assessing sterility are discussed.

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Section 16

3. "Water, High Purity and Distilled, Specification for", MSC-SPEC-C-20A, Manned Spacecraft Center, NASA, Houston, Texas, May 5, 1967

This specification establishes the minimum requirements for high purity water and distilled water used in spacecraft applications such as cleaning, flushing and cooling. The requirements are as follows:

	<u>High Purity Water</u>	<u>Distilled Water</u>
Conductivity @ 25°C, $\text{ohm}^{-1} \text{cm}^{-1}$	1.0×10^{-6}	2.0×10^{-6}
pH	6.00-7.50	6.00-7.50
Particulates, max. no. per 500 ml fluid		•
0-50 microns	Unlimited*	
50-75 microns	100	
75-100 microns	10	
>100 microns	0	
Nonvolatile Residue, mg/100 ml	0.2 max.	1.0 max.
Halides, ppm	1.0 max.	1.0 max.
Surface Tension @ 20°C, dynes/cm	72.72 ± 1.0	72.72 ± 1.0
Silting	No evidence	

* Unlimited means that particles in this size range are not counted; however any obscuring of the filter grid lines shall be cause for rejection.

Section 16

4. "Water, High Purity (Potable), Specification for", MSC-SPEC-C-21A, Manned Spacecraft Center, NASA, Houston, Texas June 19, 1967

This specification establishes the usage requirements for water used on board spacecraft system and subsystems and Aerospace Ground Equipment/Ground Service Equipment (AGE/GSE). These requirements are:

Electrical Conductivity @ 25°C (max.)*	$1 \times 10^{-6} \text{ ohm}^{-1} \text{ cm}^{-1}$
pH @ 25°C*	6.5-7.5
Surface Tension @ 20°C*	72.72 \pm 1.0 dynes/cm
Total Solids (max.)*	2 mg/l
Sterility	Free of viable organisms
Taste and Odor*	None at threshold odor no. of 1
Turbidity*	1.0 unit
Color, true*	0 units
Particulates, number/50 ml fluid	
0-10 microns	Unlimited**
10-25 microns	875
25-50 microns	100
50-100 microns	50
> 100 microns	0
Ionic Species (Heavy Metals), mg/l, (max.)	
Cd	0.01
Cu	1.0
Cr	0.05
Fe	0.3
Pb	0.05
Mn	0.05
Hg	0.002
Ni	0.05
As	0.05
Zn	0.05

* Max. allowable limit is not applicable following addition of high purity water with MSC approved bacteriocidal material.

** Unlimited means that particles in this size range are not counted; however, any obscuring of the filter grid lines shall be cause for rejection.

Section 16

5. "Sampling of High Purity (Potable) Water, Procedure for," MSC-PROC-C-104, Manned Spacecraft Center, NASA, Houston, Texas, June 19, 1967

This procedure establishes the method for sampling high purity (potable) water used in onboard spacecraft systems and subsystems and Aerospace Ground Equipment/Ground Service Equipment of the Apollo spacecraft. The sample shall be used for microbiological and chemical analysis.

2.0 -