

MIL-P-27407A
28 November 1978

SUPERSEDING
MIL-P-27407 (USAF)
4 February 1964

MILITARY SPECIFICATION
PROPELLANT PRESSURIZING AGENT HELIUM

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers the requirements for two types of helium. Helium furnished under this specification shall be Bureau of Mines helium pursuant to the Code of Federal Regulations, Title 30 CFR 602.

1.2 Classification. Helium shall be of the following designated types and grades as specified (6.2).

Type I - Gaseous Helium

Grade A - Pressurizing Helium

Grade B - Respirable Helium

Type II - Liquid Helium

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

Military

MIL-C-45662 Calibration System Requirements.

MIL-C-52752 Cylinders, Compressed Gas, Packaging of.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Air Force Rocket Propulsion Laboratory (LKCP), Edwards AFB CA 93523, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 9135

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STANDARDSMilitary

MIL-STD-10L	Color Code for Pipelines and for Compressed Gas Cylinders.
MIL-STD-129	Marking for Shipment and Storage.
MIL-STD-172	Color Codes for Containers of Liquid Propellants.
MIL-STD-1411	Inspection and Maintenance of Compressed Gas Cylinders.

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other Publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

National Bureau of Standards

Research Paper No. 1965	Measurement of Water in Gases by Electrical Conduction in a Film of Hygroscopic Material and the Use of Pressure Changes in Calibration.
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(Application for copies should be addressed to the National Bureau of Standards, Washington, D.C. 20234.)

Bureau of Mines

Report of Investigations 7444	An Improved Method and Apparatus for Analysis of Impurities in Helium.
Information Circular IC 8367	Computing Volume of Cylindrical Steel Containers at 10 to 10,000 psia.

(Application for copies should be addressed to the Publications Distribution Section, Bureau of Mines, 4800 Forbes Street, Pittsburgh, Pennsylvania 15213.)

Code of Federal Regulations

30 CFR 602	Purchase of Helium by Federal Agencies and Their Contractors.
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49CFR 170-179

Department of Transportation Rules
and Regulations for the Transporta-
tion of Explosives and Other Dan-
gerous Articles.

(Application for copies should be addressed to the Superintendent of Docu-
ments, U.S. Government Printing Office, Washington, D.C. 20402.)

American Society for Testing and Materials (ASTM)

E 29-73

Recommended Practices for Indicating
Which Figures are to be Considered
Significant in Specified Limiting
Values.

(Application for copies should be addressed to the American Society for
Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

3. REQUIREMENTS

3.1 Chemical and Physical Properties.

3.1.1 Type I Helium. Unless otherwise specified (6.2 and 6.3.1), Type I helium shall conform to the limits listed in Table I when tested according to the applicable test methods (4.5).

3.1.2 Type II Helium. Impurity limits for liquid helium are not specified since sufficient technical data and analytical procedures are not available to warrant a definitive quantitative specification. Acceptability by the procuring activity shall be based on contractor or supplier good processing techniques and certification of conformance to the filtration requirement 3.1.3 and helium assurance test 4.6 specified herein.

3.1.3 Filtration.

3.1.3.1 Type I. Unless otherwise specified, Type I helium need not be filtered (6.2).

3.1.3.2 Type II. Unless otherwise specified, Type II shall be delivered to the final shipping container through a 10 micron stainless steel filter (6.2).

3.2 Limiting Values. The following applies to all specified limits of this specification. For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM Recommended Practice E 29-73, indicating which place of figures is to be considered significant in specified limiting values.

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TABLE I, REQUIREMENTS

TYPE I, (GASFOUS)			
COMPOSITION	LIMIT		TEST METHOD
	GRADE A	GRADE B	
Helium percent by volume - min.	99.995	99.997	4.5.1
Dev. Point - not higher than	-78°F	-78°F	
Maximum Allowable Impurities ppm by volume			
Water	9	9	4.5.2.1
Hydrocarbons (as methane)	5	1	4.5.2.2
Oxygen	3	3	4.5.2.3
Nitrogen + Argon	14	5	4.5.2.4
Neon	23	23	4.5.2.4
Hydrogen	1	1	4.5.2.4
Carbon Dioxide	1		4.5.2.5
Carbon Monoxide	1		4.5.2.5
Total Allowable Impurities	50		
TYPE II (LIQUID) SEE 3.1.2			

3.3 Filling Pressure. Each gaseous helium container (cylinder, tube bank car or tube bank truck) shall be filled to the allowable filling pressure in accordance with the pressure-temperature conversion chart contained in Table II, based on the rated service stamped on the cylinders and the ambient temperature, and DOT regulations. The net content in standard cubic feet (SCF) of each container shall be the gas volume corrected to 70°F (21°C) and 14.7 psia as shown in Bureau of Mines Circular IC 8367, when tested in accordance with 4.5.3.1.

3.4 Leakage. Containers and valves shall not leak when tested in accordance with 4.5.3.2.

3.5 Rejected Filled Container. Filled containers rejected as a result of a test failure shall be processed in accordance with the following processing requirements.

<u>Test Failure</u>	<u>Processing Requirement</u>
(a) Total Hydrocarbons (4.5.2.2)	3.5.1
(b) Oxygen (4.5.2.3)	3.5.1
(c) Other gases (4.5.2.4)	3.5.1
(d) Carbon Dioxide and Carbon Monoxide (4.5.2.5)	3.5.1
(e) Moisture (4.5.2.1)	3.5.1
(f) Filling pressure (4.5.3.1)	3.5.2
(g) Leakage (4.5.3.2)	3.5.3

3.5.1 Impurity. Containers rejected for impurity content shall be discharged to zero psig pressure and processed in accordance with 5.2.1 before refilling.

3.5.2 Filling Pressure. A container having more than the maximum allowable filling pressure shall be discharged to the acceptable range as specified in 3.3. Recharged containers shall be retested as freshly filled containers. Cylinders recharged shall not be selected as a test sample specified in 4.3.2.

3.5.3 Leakage. If leakage occurs through the valve or at the junction between the valve and the container, the container shall be discharged to zero psig and valve reinstalled, repaired or replaced in accordance with 5.2.1. The container shall then be dried in accordance with 5.2.1 before refilling. Leakage through the container wall shall be cause for rejection and the container shall be discharged and processed in accordance with 5.2.1

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial

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Table II - Pressure - Temperature Conversion Chart*

Settled Temp °F	Container Service Pressure (expressed in psig)						
	1800	2000	2200	2365	2400	2490	2640
-50	1393	1549	1704	1755	1840	1930	2046
-48	1400	1556	1711	1763	1849	1940	2056
-46	1407	1563	1719	1772	1858	1948	2066
-44	1414	1571	1728	1780	1867	1958	2076
-42	1421	1579	1737	1789	1876	1967	2086
-40	1427	1587	1746	1798	1885	1977	2096
-38	1434	1594	1752	1806	1894	1986	2106
-36	1441	1602	1761	1815	1903	1996	2116
-34	1448	1609	1769	1823	1912	2005	2126
-32	1455	1617	1779	1832	1921	2014	2136
-30	1462	1624	1787	1840	1931	2024	2146
-28	1468	1632	1796	1849	1940	2033	2156
-26	1475	1640	1804	1858	1949	2043	2166
-24	1482	1647	1812	1866	1958	2052	2176
-22	1489	1655	1821	1875	1967	2061	2186
-20	1495	1662	1829	1883	1976	2071	2196
-18	1502	1670	1837	1892	1985	2080	2206
-16	1509	1677	1846	1900	1994	2090	2216
-14	1516	1685	1854	1909	2003	2099	2226
-12	1523	1692	1862	1917	2012	2108	2236
-10	1530	1700	1870	1926	2021	2118	2246
- 8	1536	1708	1879	1934	2030	2127	2256
- 6	1543	1715	1887	1943	2039	2136	2266
- 4	1550	1723	1895	1951	2048	2146	2276
- 2	1557	1730	1904	1960	2057	2155	2285
0	1564	1738	1912	1969	2066	2165	2295
2	1570	1745	1920	1977	2075	2174	2305
4	1576	1753	1928	1985	2084	2183	2315
6	1584	1760	1937	1994	2093	2193	2325
8	1591	1768	1945	2003	2102	2202	2335
10	1598	1775	1953	2011	2111	2212	2345
12	1604	1783	1962	2020	2120	2221	2355
14	1611	1790	1970	2028	2129	2230	2365
16	1618	1798	1978	2037	2138	2239	2375
18	1625	1805	1986	2045	2147	2249	2384
20	1631	1813	1994	2054	2156	2258	2394
22	1638	1820	2003	2062	2165	2267	2404
24	1645	1828	2011	2071	2174	2277	2414
26	1652	1835	2019	2079	2183	2286	2424
28	1659	1843	2027	2088	2192	2295	2434
30	1665	1850	2036	2096	2201	2304	2444
32	1672	1858	2044	2104	2210	2314	2453
34	1679	1865	2052	2113	2219	2323	2463
36	1686	1873	2060	2121	2228	2332	2473
38	1692	1880	2069	2130	2237	2342	2483
40	1699	1888	2077	2138	2246	2351	2493
42	1706	1895	2085	2147	2255	2360	2503
44	1713	1903	2093	2155	2264	2370	2513
46	1719	1910	2102	2164	2273	2379	2522
48	1726	1918	2110	2172	2282	2388	2532
50	1733	1925	2118	2181	2291	2397	2542
52	1740	1933	2126	2189	2300	2407	2552

Table II - Pressure -Temperature Conversion Chart* (Continued)

Settled Temp °F	Container Service Pressure (expressed in psig)						
	1800	2000	2200	2265	2400	2490	2640
54	1746	1940	2134	2197	2324	2416	2562
56	1753	1948	2143	2206	2338	2425	2571
58	1760	1955	2151	2214	2346	2434	2581
60	1767	1963	2159	2223	2355	2444	2591
62	1773	1970	2167	2231	2364	2453	2601
64	1780	1979	2175	2240	2373	2462	2611
66	1786	1985	2184	2248	2382	2472	2621
68	1793	1993	2192	2257	2391	2481	2630
70	1800	2000	2200	2265	2400	2490	2640
72	1807	2007	2208	2273	2409	2499	2650
74	1814	2015	2216	2282	2418	2508	2660
76	1820	2022	2225	2290	2427	2518	2669
78	1827	2030	2233	2299	2436	2527	2679
80	1834	2037	2241	2307	2445	2536	2689
82	1841	2045	2249	2316	2454	2545	2699
84	1847	2052	2257	2324	2462	2555	2709
86	1854	2060	2265	2332	2471	2564	2718
88	1861	2067	2274	2341	2480	2573	2728
90	1867	2074	2282	2349	2489	2582	2738
92	1874	2082	2291	2358	2498	2592	2748
94	1881	2089	2298	2366	2507	2601	2757
96	1888	2096	2306	2374	2516	2610	2767
98	1894	2104	2315	2383	2525	2619	2777
100	1901	2112	2323	2391	2534	2629	2787
102	1908	2118	2331	2400	2543	2638	2797
104	1915	2127	2339	2408	2551	2647	2806
106	1921	2134	2347	2416	2560	2656	2816
108	1928	2141	2355	2425	2569	2665	2826
110	1935	2149	2364	2433	2578	2675	2836
112	1941	2156	2372	2442	2587	2684	2845
114	1948	2164	2380	2450	2596	2693	2855
116	1955	2171	2388	2458	2605	2702	2865
118	1962	2179	2396	2467	2614	2712	2875
120	1968	2186	2404	2475	2623	2721	2884
122	1975	2194	2413	2484	2632	2730	2894
124	1981	2201	2421	2492	2640	2739	2904
126	1988	2208	2430	2501	2649	2748	2914
128	1995	2215	2437	2509	2658	2758	2923
130	2001	2223	2445	2517	2667	2767	2933
132	2008	2231	2454	2526	2676	2776	2943
134	2015	2238	2462	2534	2685	2785	2953
136	2022	2246	2470	2543	2694	2794	2963
138	2028	2253	2478	2551	2703	2804	2972
140	2035	2261	2486	2559	2712	2813	2982
142	2042	2268	2494	2568	2720	2822	2992
144	2048	2275	2502	2576	2729	2831	3002
146	2055	2283	2511	2585	2738	2841	3011
148	2062	2290	2519	2593	2747	2850	3021
150	2069	2298	2527	2601	2756	2859	3031

* From CGA Specification G-9.1

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laboratory unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements (6.2).

4.1.1 Points of Inspection. When helium containers are filled from production plant systems or other storage facilities for delivery to destination by any mode of transportation, quality conformance tests shall be conducted on the container contents prior to leaving the site of filling or shipment (6.2).

4.2 Classification of Tests. The inspection and testing of helium shall be classified as quality conformance tests

4.3 Quality Conformance Tests Quality conformance tests shall consist of:

- (a) Individual tests 4.3.1
- (b) Sampling tests 4.3.2

4.3.1 Individual Tests. Each container of helium shall be subjected to the following tests

- (a) Filling Pressure 4.5.3.1
- (b) Leakage 4.5.3.2

4.3.2 Sampling Tests. Type I helium shall be selected in accordance with 4.3.2.1 and tested for the requirements of Table I as specified in 4.5. Type II helium shall be selected in accordance with 4.3.2.2 and tested for the requirements of 3.1.2 as specified in 4.6.

4.3.2.1 Sampling Plan - Type I

4.3.2.1.1 Lot.

4.3.2.1.1.1 Containers. When helium gas cylinders or tubes are filled, each set of cylinders or tubes charged on the same manifold at the same time shall constitute a lot. Tubes that are interconnected shall be considered as one lot for the purpose of this specification. The number of samples per lot shall conform to Table III.

TABLE III - TEST SAMPLES

<u>Lot Size</u>	<u>Number of Samples</u>
2 to 10	1
11 to 40	2
41 to 70	3
71 and up	4

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4.3.2.1.1.2 Contractor Sample. Samples taken by a commercial vendor shall consist of not less than 4 cubic feet of helium obtained in any suitable sampler. Unless otherwise specified, quality conformance tests shall be made upon each sample taken directly from the shipping containers. When required, an equivalent sample quantity shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance tests specified herein.

4.3.2.2 Sampling Plan - Type II. Each filled container of Type II helium shall constitute a lot.

4.4 Test Conditions.

4.4.1 Temperatures. Temperatures shall be measured using a mercury-in-glass thermometer or thermocouple having scale divisions of one degree Fahrenheit. Thermometers or thermocouples used for measuring temperatures during filling of containers shall be in contact with the outside surface of the container.

4.4.2 Pressure. Pressure shall be measured by means of precision Bourdon tube gages or any other laboratory type pressure measuring device accurate to plus or minus 10 psi and having scale increments of 20 psi or smaller.

4.4.3 Calibration. Test equipment shall be calibrated as applicable in accordance with MIL-C-45662.

4.5 Test Methods - Type I

4.5.1 Helium (percent by volume). The helium percent shall be found by determining the aggregate impurities by the methods described in succeeding sections. The percent helium is the value obtained when this amount, expressed as volume percent, is subtracted from 100.

4.5.2 Impurities

4.5.2.1 Moisture - Type I Helium. The sample shall be tested for moisture content in accordance with one of the following methods. The electrolytic method described in 4.5.2.1.1 shall be the reference method.

(a) Electrolytic moisture apparatus (hygrometer) operated on a range no greater than ten times the specified maximum moisture content.

(b) Frost (dew) point method.

(c) Electrical conductivity method in accordance with NBS Research Paper RP 1865.

(d) Piezoelectric sorption hygrometer operated on a range no greater than ten times the specified maximum moisture content.

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(e) Aluminum oxide capacitor equipped analyzer operated on a range no greater than ten times the specified maximum moisture content.

4.5.2.1.1 Electrolytic Method.

4.5.2.1.1.1 Procedure. Connect the sample container to a pressure regulator which is attached to the electrolytic moisture apparatus. Open the sample container valve and adjust the pressure to the apparatus in accordance with the manufacturer's recommended value. Allow sufficient time for the indicated moisture content to become stable and read the value obtained while using the most sensitive scale setting possible for the moisture content of the sample. Report the moisture content in terms of ppm by volume.

4.5.2.1.1.2 Equipment. The test equipment, or its equivalent, shall consist of the following:

- (a) Regulator, helium diffuser resistant, metal diaphragm.
- (b) Stainless steel tubing, maximum OD of 1/8 inch.
- (c) Apparatus, electrolytic, moisture, Beckman Cat. No. 179C2, Consolidated Electrodynamics Corp. Model No. 26-35C.

4.5.2.2 Total Hydrocarbons. A total hydrocarbon analyzer meeting the requirements of 4.5.2.2.1 shall be used for the determination of hydrocarbons in helium.

4.5.2.2.1 Total Hydrocarbon Analyzer. The total hydrocarbon analyzer shall be operated on a method such that all hydrocarbons in the sample stream shall be reduced to a common reference. The common reference shall be quantitatively analyzed and indicated in ppm by volume as methane equivalent. The sensitivity of the analyzer shall be 0.2 ppm or 20% of the specified maximum, whichever is greater. The meter or recorder shall have a span of 5 times the specified maximum or less.

4.5.2.3 Oxygen. Cylinder-filled helium samples or samples for container-filled helium shall be connected to the inlet of a specific oxygen analyzer which operates on an electrolytic, galvanic cell (e.g., polarography), heat of reaction principle of measurement, or any other applicable principle. Calibration of the analyzer may be performed with accurate trace oxygen standards, or integrally in accordance with Faraday's Law. The sensitivity of the instrument, when calibrated, shall be 0.2 ppm or 20% of the specified maximum, whichever is greater. The meter or recorder shall have a span of 5 times the specified maximum or less. In cases of dispute, the galvanic cell shall be used as the referee.

4.5.2.4 Other Gaseous. The sample shall be tested for other gaseous trace impurities using a gas chromatograph in accordance with the method described in 4.5.2.4.1. The analyzer shall be capable of separating and determining each component. Appropriate impurity concentration techniques may be used to attain sensitivity.

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4.5.2.4.1 Procedure. Assemble a gas chromatograph and calibration system as shown, or equivalent to that shown in Figure 1. Insure that the apparatus is free from helium leaks by pressurizing to normal operating pressures and testing each connection with leak detecting solution. After any leaks are eliminated it may be necessary to purge the apparatus for several days in order that all residual air be removed from the system. After the recorder baseline becomes stable, and replicate helium analyses indicate a constant impurity level, the gas chromatograph is ready for sample analysis. Connect the sample to the sampling valve and adjust the sample flow to 100 milliliters (ml) per minute (measured at the rotameter). Allow the sample to purge the valve for several minutes and then inject the sample into the chromatograph column. Several replicate injections may be necessary in order to obtain a reproducible impurity level. Read and record the indicated peak height or peak area for each trace impurity. The first peak appearing on the chromatograph is a pressure surge followed in order by peaks for neon, hydrogen, oxygen-argon, nitrogen, methane, and carbon monoxide (see Figure 2). Oxygen-argon normally appear as a single peak, and the oxygen portion shall be determined in accordance with 4.5.2.3. Carbon monoxide may be determined by this method or by the method described in 4.5.2.5, however, the method described in 4.5.2.5 shall be the referee in cases of dispute. Compare the peak heights or areas of each impurity to calibration curves prepared in accordance with 4.5.2.4.2. Maximum accuracy and instrumentation stability is obtained if the gas chromatograph is maintained in a continuous operational condition.

4.5.2.4.2 Apparatus Calibration. Connect the outlet of the dilution flask to the gas sampling valve adjacent to the chromatograph as shown in Figure 1. Adjust the helium flow rate to 100 ml per minute and inject replicate samples until the indicated impurity levels are constant. Connect a vessel containing a reagent grade contaminant selected for instrument calibration to the gas inlet valve. Purge the valves for 5-10 seconds with the contaminant and then turn off the purge. Immediately inject the contaminant into the dilution flask and start the timer. Inject samples of the diluted contaminant into the gas chromatograph through the sampling valve at specific time intervals (i.e., every 5 minutes). Record the accumulated time, in minutes, and peak height or area of the contaminant peak corresponding to each injection. Continue the injection sequence until the size of the contaminant peak is equal to the background level. Repeat the above procedure for each contaminant listed in 4.5.2.4.3. Calculate the contaminant concentration at each injection time using the following formula.

$$\log C = \log C_0 - \frac{tV}{2.303U}$$

C_0 = volume injected in ml $\times 10^6$ divided by the volume of the dilution flask in ml; V = flow rate in ml per minute; t = accumulated time in minutes; C = concentration in ppm by volume at time = t ; U = volume of the dilution flask in milliliters. Subtract each background contaminant level from its total observed during the calibration sequence and plot this difference in peak height, or area, versus the calculated concentration. Use this curve

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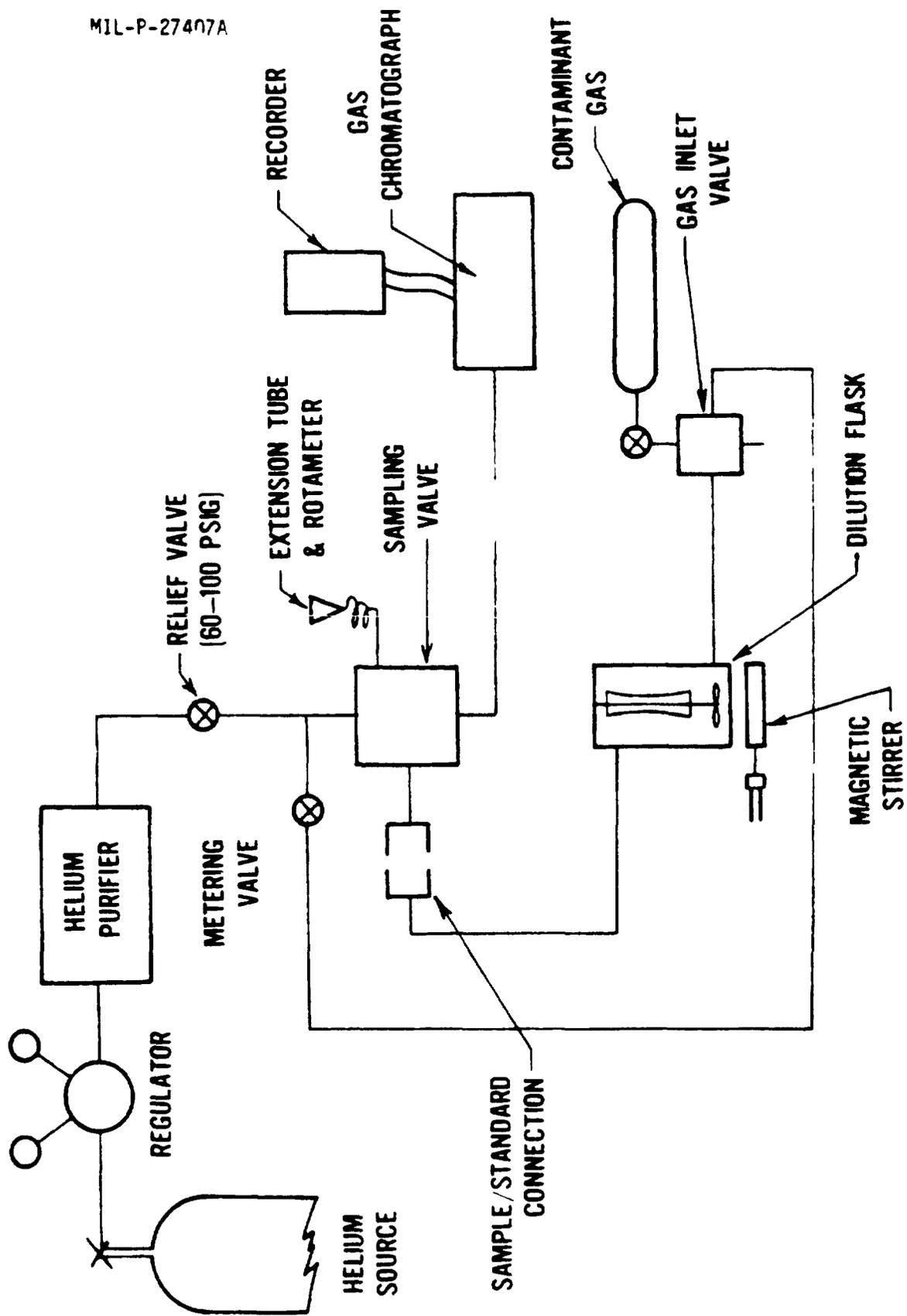


FIG. 1, OTHER GASEOUS IMPURITIES APPARATUS

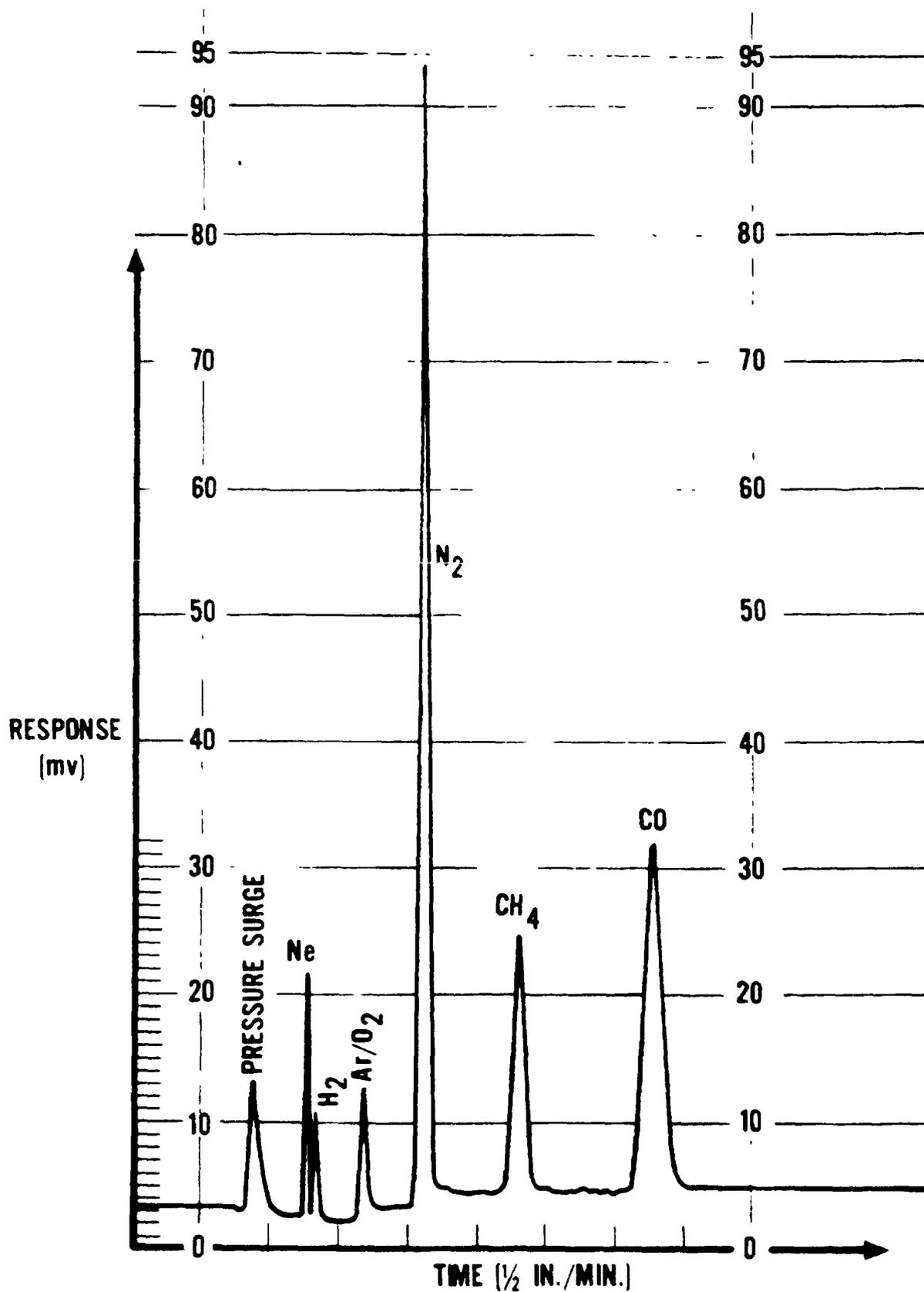


Figure 2. A TYPICAL CHROMATOGRAM OF IMPURE HELIUM

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to determine the contaminant concentrations in the sample. Calibrate at least weekly or just prior to use when the instrument is used less frequently.

4.5.2.4.3 Reagents and Equipment. The following reagents and equipment shall apply as test conditions of 4.5.2.4.

(a) Reagents.

- (1) Hydrogen, 99% min. gaseous
- (2) Neon, 99% min. gaseous
- (3) Oxygen, 99% min. gaseous
- (4) Argon, 99% min. gaseous
- (5) Nitrogen, 99% min. gaseous
- (6) Methane, 99% min. gaseous
- (7) Carbon Monoxide, 99% min. gaseous (optional)

(b) Equipment:

- (1) Gas chromatograph, equipped with an ultrasonic or a helium ionization detector, or other detector of equivalent sensitivity.
- (2) Recorder, potentiometric, 1 mv span, 1 second FS pen speed, or equivalent.
- (3) Integrator, digital or mechanical (optional).
- (4) Purifier, A purifier may be constructed from 10 feet of 3/8 inch C.D. copper tubing packed with activated coconut charcoal (ca 40/60 mesh) and maintained at liquid nitrogen temperature during apparatus use.
- (5) Valve, sampling, 1 cc sample loops with purgeable valve housing; Carle Model No. 2014, or equivalent.
- (6) Valve, gas inlet, 0.5-1 cc sample loop; Carle Model No. 2018, or equivalent.
- (7) Flask, flow dilution, 250 cc volume, magnetically stirred, Varian Aerograph Co. Model No. 02-000382-00, or equivalent.
- (8) Stirrer, magnetic variable speed.
- (9) Regulator, metal diaphragm, diffusion resistant.

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- (10) Cylinder, containing helium gas, 99.999% min.
- (11) Stopclock, 1 hour capacity, 1 second divisions, or equivalent.
- (12) Column, 15 feet long by 1/8 inch C.D. Stainless Steel, packed with 40/50 mesh 5A Molecular Sieves; activate the Molecular Sieves by purging the column at 350°C with 99.995% helium gas for 4 hours. For analysis the column is maintained at 90°C and with a helium flow rate of approximately 25 ml per minute. Any equivalent column or columns which are capable of performing the required separations for the complete analysis, including the separation of oxygen and argon, are acceptable. In cases of dispute, the 40/50 mesh 5A Molecular Sieves column shall be used as the referee.
- (13) Tube, extension, 6 feet long by 1/16 inch O.D.
- (14) Rotameter, 0-250 cc/min. range, or equivalent.

4.5.2.4.4 Alternate calibration curves may be prepared by analyzing, in the chromatograph standard samples obtained from the Bureau of Mines¹, qualified commercial standards, or synthetically-prepared mixtures analyzed in accordance with the mass spectrometer method as specified in RI 7444.

4.5.2.5 Carbon Dioxide plus Carbon Monoxide Content. The carbon dioxide plus carbon monoxide content shall be determined by using either a non-dispersive infrared (I.R.) which can distinguish between CO and CO₂ or a gas chromatograph equipped with an Ethyl vinyl benzene polymer bead column ("Porapak Q", "Chromosorb 102", etc.), and a detector capable of measuring directly each of the above components. If a less sensitive detector is used, appropriate preconcentration of the carbon dioxide and carbon monoxide may be accomplished using a trap filled with ethyl vinyl benzene polymer beads maintained at the temperature of liquid nitrogen. The gas chromatograph used shall be capable of separating and determining the carbon dioxide and carbon monoxide content of the sample with a sensitivity of 0.2 ppm or 20% of the specified limit, whichever is larger. The meter or recorder shall have a span of 5 times the specified limit or less. In cases of dispute, the non-dispersive I.R. shall be the referee method.

4.5.3 Container.

4.5.3.1 Filling Pressure. Test the appropriate number of cylinders and tubes, according to 4.3.2.1.1, for proper filling pressure by attaching a Bourdon tube test gage, or its equivalent, to the valve outlet and by attaching a thermocouple or thermometer to the container wall. If a thermometer is used, apply tape or putty to the bulb to protect it from alien temperatures, putty shall not be applied between the bulb and the container wall. Stabilize the container temperature to ambient temperature. Refer to the pressure-temperature conversion chart, Table II, and the rated service pressure stamped on the cylinder to determine the proper filling pressure.

¹ Helium calibration gas standard is available from The Bureau of Mines Helium Operations, Box H 4372, Herring Plaza, Amarillo TX 79101.

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Open the container valve and observe the internal gage pressure. Some small cylinders and semitrailer tubes can be filled to 10 percent excess if the requirements of 49 CFR 173.302 (c) are satisfied. Tube bank cars may be filled to 10 percent excess if they meet the requirements of 49 CFR 173.314 (d) (1) Note 1.

4.5.3.2 Leakage. Test each container for leakage by brushing a leak detecting solution over the valve and at the junction between the valve and the container. Valve seat leakage shall be tested by applying the leak-detection fluid sparingly across the outlet of the valve. Only leak-detection fluid that leaves no residue shall be used on the outlet. When any cylinder of a tube bank car or truck shows visible evidence of damage, the piping, valves and fittings shall be tested for leakage and the cylinder shall be hydrostatic tested as required by DOT (ICC) regulations.

4.6 Test Methods - Type II.

4.6.1 Liquid Purity Assurance. All helium contaminants with a concentration greater than 1.0 ppm are in the solid phase when helium is in the liquid state. No liquid helium sampling method has been developed that will give a valid representation of solid content in bulk liquid helium storage containers or transfer equipment. Liquid helium purity can only be assured by.

(a) Filtering.

(b) Strict control of the procedures used to transfer and transport the helium

4.6.1.1 Equipment Control. Procedures used for insuring contaminant free containers will, as a minimum, cover the following:

(a) Provisions for the periodic cleaning of shipping containers (such as warming and purging based on time and type of service),

(b) Provisions for insuring the maintenance of a positive helium pressure (relative to ambient) in the shipping containers,

(c) Provisions for insuring that filters will be isolated before they warm to the point where solid contaminants will vaporize and contaminate the shipping containers,

(d) Provisions for insuring that contaminants do not enter the shipping container during transfer equipment connect operations.

4.6.2 Helium Assurance Test. The requirement for insuring that the loaded fluid is liquid helium can be satisfied by:

(a) Analyzing the shipping container vent gas (thermal conductivity, mass spectrometer, etc. . .). or

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(b) Demonstrating that the temperature of the loaded fluid is below the hydrogen triple point (13.8°K).

4.7 Preparation for Delivery Inspection. The preservation, packaging, packing and marking for shipment and storage of helium shall be inspected to determine compliance with the requirements of Section 5.

4.8 Rejection and Retest. When any sample of helium fails to conform to the requirements specified herein, the entire lot shall be rejected.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. Helium furnished under this specification is regulated by the Code of Federal Regulations (CFR), Title 49 CFR 170 to 179. All containers and packaging to be shipped shall comply with the requirements of this regulation. Gas cylinders shall also be packed in accordance with MIL-C-52752. The product shall be packaged in containers and unit quantities as specified by the procuring activity (6.2).

5.2 Preparation of Containers. The contractor shall prepare the containers as follows (6.2):

5.2.1 Gas Cylinders. The cylinders shall be inspected, maintained, and filled in accordance with MIL-STD-1411 for helium cylinders, except that paragraph 5.3.2.3.2 shall not be used to clean Type I, Grade B cylinders.

5.2.2 Gas Tube Banks. Each tube bank car or tube bank trailer for initial service or refill shall be processed as specified for standard cylinders in accordance with 5.2.1, where applicable. When tubes have sufficient residual pressure (greater than 15 psig at 70°F) but doubt exists regarding the quality of the residual, a tube or tubes may be sampled and tested following purging of the manifold with approximately one cubic foot of gas from the tube or tubes in question. When insufficient prefilling pressure (less than 15 psig at 70°F) is evident, the tubes shall be evacuated in accordance with 5.2.2.1 or purged in accordance with 5.2.2.2 until tests show that they will fill with the grade of helium ordered.

5.2.2.1 Evacuation. Tube bank cars or trucks shall be evacuated to 100mm Hg or less pressure and pressurized to 10 psig with helium and the process repeated until tests show that they will fill with the grade of helium ordered.

5.2.2.2 Purging. If capabilities exist to recover purge helium, the tube bank cars or trucks can be purged with helium instead of being evacuated. Purging will continue until tests show that the tubes will fill with the grade of helium ordered.

5.2.3 Liquid Cylinders. Qualifications, maintenance, and use of liquid cylinders (Dewar type) shall conform to requirements of 49 CFR 173.34, and any other DOT requirements, and as follows:

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5.2.3.1 Cleaning and Repair. Unless otherwise provided for in the contract of purchase order, all cylinders shall be visually inspected for the presence of water, rust, scale, oil film, or other foreign matter, sufficient insulation and vacuum, or physical damage (6.2). Any insulation damage, vacuum depletion, or physical damage which would endanger safe transportation of the product shall be repaired prior to reuse. If evidence of internal contamination is found, the cylinders shall be recleaned by a suitable method to remove the contamination prior to use.

5.2.3.2 Filling. Cylinders shall be filled through a filter (3.1.3) to the filling density specified by DOT.

5.3 Marking.

5.3.1 Containers. In addition to marking required by the contract or order, unit containers and shipping containers shall be marked in accordance with MIL-STD-129 or DOT regulations. In addition, an identification tag and container color code shall be used.

5.3.2 Identification Tag. Unless otherwise specified in the contract or order, an identification tag impervious to climatic conditions shall be wired to the outlet port of each container and shall contain the following information: Product name, specification number with revision letter, type designation, FSN number, quantity, name of manufacturer, name of contractor (if different from manufacturer), date of manufacture, and lot identification number (6.2).

5.3.3 Cylinder Color Code. Unless otherwise specified by the procuring activity, each DOD owned cylinder shall be color coded in accordance with MIL-STD-101 for Type I, or MIL-STD-172 for Type II, and the exact name identification to be marked on the outside of the cylinders shall be "Gaseous Helium", "Respirable Helium", or "Liquid Helium" as appropriate (6.2). Any other name identification shall be obliterated by removing or overpainting.

6. NOTES

6.1 Intended Use. Type I, Grade A helium is intended for pressurization of rocket engine propellant systems, space vehicles and associated ground support equipment, welding and other shielding gas operations. Type I, Grade B helium is intended for use as a respirable breathing gas for divers during deep excursions into the oceans. Type II helium is intended for use as a cryogenic coolant in specialized test and measurement equipment.

6.2 Ordering Data. Purchasers should select the preferred options permitted herein and include the following information in procurement documents:

- (a) Title, number, type, grade, and date of this specification.

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(b) On drawings, specifications, standards, publications, or procurements dated prior to this specification, reference to helium conforming to MIL-P-27407, 4 February 1964, shall be interpreted to mean Type I, Grade A of this specification.

(c) Method of shipment, type and capacity of containers (5.1).

(d) The basis for procurement of Type I should be the cubic foot based upon normal atmospheric conditions (70°F and 14.7 psia). Type II should be procured by liters or pounds.

(e) When inspection requirements are to be performed by other than the supplier (4.1).

(f) When variation in points of inspection is granted (4.1.1).

(g) When waiver of quality conformance tests on each sample is granted, such as when Bureau of Mines helium has not been repackaged by the supplier (4.3.2.1.1.2).

(h) When variation in sampling method is granted (4.3.2.1) or (4.3.2.2) as applicable.

(i) When filtration of Type II is not required or a different size of filter is required (3.1.3).

(j) When filtration of Type I is to be performed through a 10 micron stainless steel filter (3.1.3).

(k) When disposition of rejected product has to be specified (3.5).

(l) When cleaning and repair schedule is required for leased or Government-owned containers (5.2).

(m) When instructions for disposition of rejected cylinders are required (5.2.1).

(n) When cleaning and repair provisions are other than as specified (5.2.3.1).

(o) When identification tag is to be other than as specified (5.3.2).

(p) When the container color code is to be waived (5.3.3).

(q) When complete testing is not required (3.1 and 6.3.1).

6.2.1 Contract Data Requirements. Two copies of test report, Data Item Description DI-T-3733, signed by the contractor's representative, listing values obtained in all tests (quantitative values where method provides) shall accompany each shipment delivered to consignee.

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6.3 Specification Exceptions.

6.3.1 Impurity Testing. It shall be possible to order Type I helium without requiring testing for all or any of the impurities listed in Table I, providing that the contract or order states the nature and scope of the specification relaxation, and the manufacturer is willing to provide such a material. There can be no guarantee that the price of any such "relaxed" grade will be lower than for Type I helium.

6.3.2 Container Handling. Filling of Bureau of Mines containers by a commercial helium supplier shall be in accordance with these specifications. Bureau of Mines regulations shall be adhered to during all operations.

6.4 Supercession Data. This specification includes the requirements of:

MIL-P-27407 (USAF), 4 Feb 1964
MSFC-364C (NASA), 15 Sep 1969

Custodians:
Air Force - 12

Preparing Activity:
Air Force - 12

Review Activities:
Air Force - 19, 68

Civil Agency Interest:
NASA

User Activities:
Navy-SH

(Project 9135-0098)

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