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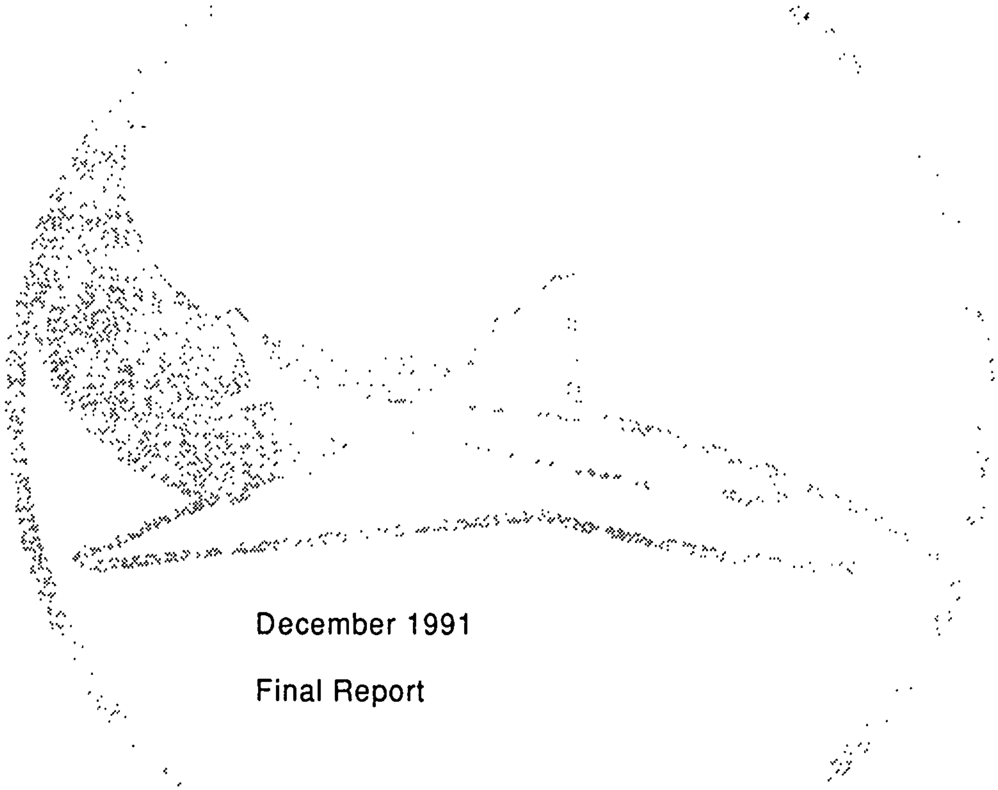
FAA Technical Center
Atlantic City International Airport
N.J. 08405

Feasibility of Systematic Recycling of Aircraft Halon Extinguishing Agents



December 1991

Final Report

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16. Abstract

This study was performed to determine the feasibility of recycling Halon 1301 extinguishing agent for support of the United States civil aircraft fleet until 2020. Research for this study centered around known relationships of refrigerants; scientific principles of Avogadro, Dalton, Henry, Raoult; and natural physical properties of Halon 1301. Results of this study demonstrate that recovery of Halon 1301 to military specification purity requirements is practical. Agent recovery efficiency of 98 percent (minimum) can be expected; and the recovered agent can meet applicable purity requirements, except for a maximum noncondensible gas content of 10 pounds per square inch partial pressure at 70 degrees Fahrenheit. Support of current and future United States domestic civil fleet until 2020 and beyond requires less than 5 percent of the projected 1994 Halon 1301 bank.

17. Key Words
Halon, Recovery, Extinguishing Agent, Recycling, Aircraft Fire Safety

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PREFACE

This work was conducted under FAA Contract DTFA03-90-C-00039 by Walter Kidde Aerospace, Inc. during the one year period from September 1990 through August 1991.

The work of this contract addresses Halon 1301 requirements for support of the U.S. civil commercial aviation fleet in two parts. First, the ten year period from 1991 to 2000 for present fleet and ten year new production aircraft are examined. These results are used as the basis for the second forecast, demonstrating feasibility for support of the U.S. civil commercial aviation fleet from 2000 through 2020 using recycled Halon 1301.

Completion of this feasibility study was made possible by the cooperation and efforts of project team members towards accomplishment of a common goal.

Walter Kidde Aerospace, Inc., McDonnell Douglas, and DuPont Chemicals combined efforts toward establishing the feasibility of Halon 1301 reclamation and the possibility of using recycled Halon 1301 to fulfill the present and future Halon requirements of the U.S. domestic aircraft fleet from now until 2020 and beyond.

Technical and analytical support in the characteristics and physical properties of Halon 1301 were provided by Mr. Dan Moore and the Chestnut Run Laboratories of DuPont Chemicals and were instrumental in guiding research efforts which resulted in first-time solutions toward Halon recycling to agent mole purity levels better than those specified in Military Specification, MIL-M-12218C, for Halon 1301 which requires 99.6 percent mole purity.

Aircraft fleet data and event analysis was extracted from the McDonnell Douglas computer data base files by Mr. Thomas Peacock who supplied critical information needed for determining present and predicted future Halon needs of the domestic fleet.

Mr. Al Thornton of Great Lakes Chemical Corporation provided information on Halon 1301 recovery efforts at the manufacturer level. Mr. John Isenberg of ICI provided information on water solubility characteristics within Halon 1211.

During the course of this study, a visit was made to Canadian Airlines International for the purpose of photographing and reporting on a unique Halon 1301 recovery system developed by Mr. James Hamilton, Ground Support Engineering, of Canadian Airlines International. Canadian Airlines International provided permission to present the photographs and a system description in this study.

Mr. Gere D. Coffey, Publisher of AIR TRANSPORT WORLD Magazine, granted permission to use an article published in the May 1990 issue entitled, "AIRBUS, 10,000 JETS BY CENTURY'S END", written by Arthur Reed, which further illustrated world aircraft fleet growth.

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EXECUTIVE SUMMARY

Halon 1301 used for fire suppression in aircraft can be recovered from aviation fire extinguishers, conditioned, and non-condensible contaminants removed at greater than 98 percent recovery efficiency. This is also true for non-aviation Halon sources.

Recovery methods are now available for conditioning of Halon 1301 to all requirements of Military Specification MIL-M-12218C except that for the requirement of 1.5 percent fixed gases in the vapor phase. Military Specification MIL-M-12218C requires Halon 1301 to be of 99.6 percent mole purity, exhibit a boiling point of -57.75 degrees Centigrade at one atmosphere pressure, contain 1.5 percent or less fixed gases in the vapor phase of the storage container (.22 psi partial pressure at 25 degrees Centigrade), exhibit a water content of 10 parts per million or less, exhibit an acidity content of 3 parts per million or less, and contain no more than .5 grams per liter high boiling impurity. Present recovery methods can achieve less than 10 psi partial pressure fixed gas content in the vapor phase which has been demonstrated to be adequate for containers which previously required "pure" Halon 1301. This does not apply to refrigeration systems which use Refrigerant 13B1 (Halon 1301). Refrigeration systems require near ZERO fixed gas content to operate efficiently.

A basis for purification of these compounds incorporates standard refrigeration components. Assembly of mechanical filters, water and hydrocarbon removal modules can be performed rapidly at material costs of approximately $1,300.00. All required materials are generally available from suppliers of industrial refrigeration and fluid process component suppliers.

Efficient recovery, purification and non-condensible gas removal for Halon 1301 used in aviation and non-aviation fire suppression involves thermal treatment of recovered extinguisher charge at temperatures of approximately -80 degrees Fahrenheit. Development of unique thermal conditioning equipment for this purpose has been achieved and successfully demonstrated.

Mixed Halons, such as Halon 1211 and Halon 1301, do NOT form azeotropes. Hence, these mixtures can be separated by fractional distillation means using the same thermal treatment as used for recovery and non-condensible gas removal.

Support of the current and future U.S. civil aircraft fleet until the year 2020 and beyond using recycled Halon 1301 is feasible. Elimination of attempted Halon recovery through vapor side venting by using a modular Halon recovery system will increase recovery efficiency from approximately 70 percent to 98 percent. In addition, the recovered agent will conform to all requirements of MIL-M-12218C except for a maximum non-condensible gas content of 10 psi. Modular Halon 1301 recovery is available which returns 98 pounds out of every 100 pounds agent input. Less than 5 percent of the projected 1994 Halon 1301 bank is required in order to attain this goal. Continued production of Halon 1301 until 2000 may reduce this percentage due to an increase of banked Halon 1301 within this time period.

Equations and process models have been developed for charging fire extinguishers with Halon 1301/nitrogen mixtures which compensate for non-condensible gas weight in order to obtain correct agent charge weight. These equations use single temperature, pressure and weight data during charging which makes them unique. The filter-drier conditioning process, used in pressure transfer applications, permits agent recovered from one extinguisher to be used in another. Prior to development of this process, the extinguishing agent would either not be recovered or would be lost to the atmosphere. This conditioning process ensures improvement in agent purity while not decreasing recovery efficiency.

INTRODUCTION

PURPOSE

The purpose of this study is to investigate and report on practical and feasible methods for the recycling of Halon 1301 to support present and future domestic civil aircraft until 2020 and beyond if needed. Within this purpose is included the economics of recycling Halon 1301 at three different levels of recycling efficiency.

BACKGROUND

This study, prompted by concern over a dwindling atmospheric Ozone layer coupled with the desire to maintain flight safety, is aimed at reducing unnecessary Halon 1301 discharge into the atmosphere by exploring whether recycling Halon 1301 which contains non-condensible gas and other contaminants is possible. Halon 1301 is widely used in aircraft for cargo, powerplant, auxiliary power unit and several other areas for fire suppression protection and it remains the agent of choice for maintaining the present level of aircraft flight safety. The Montreal Protocol and EPA actions toward prohibiting atmospheric release of ozone-depleting substances also prompted this study since Halon 1301 must be removed from aircraft fire extinguishers during periodic pressure vessel testing and service.

Only one conceptual approach was used during the development of the processes presented within the feasibility study. Many "classic solution" options were considered; however, an approach involving commonly available components and well established technical relationships appeared most practical due to the long term availability of replacement parts and the cost effective nature of those parts.

Since Halon 1301, as well as other Halons, is a refrigerant and has been used in "super-cold" refrigeration systems since 1957, it was obvious that it should be able to be recovered as other refrigerants are recovered and removed of unwanted air. By using standard refrigeration components and technical processes known since 1885 a system was developed which enabled fixed gases, moisture, and oil contaminated Halon 1301 to 99.6 percent mole purity with less than 10 psi fixed gas content within the vapor phase.

OBJECTIVE

The objective of this study was to determine the feasibility of Halon 1301 recovery and the portion of the Halon 1301 bank required to supply recycled Halon 1301 for protection of the U.S. Civil Aviation Fleet until 2020. Other objectives of this report were to determine recovery efficiency which could be practically realized, quality control measures to be used for recycled Halon 1301, and techniques for removal of contaminants likely to be found within Halon 1301 so that it could meet the requirements of MIL-M-12218C.

1. A SUBSTANTIATED ESTIMATE OF THE AMOUNT OF HALON 1301 BANKED IN THE FIXED SYSTEMS OF THE CURRENT DOMESTIC FLEET

1.1 GENERAL

An estimate of the total amount of Halon 1301 currently banked in the domestic civil fleet was compiled. These data are presented in Tables 1, 2, and 3. Table 1 provides Halon 1301 amounts by aircraft manufacturer, aircraft model, and engine model. Each entry includes the amount of Halon 1301 contained in a single aircraft, the number of aircraft of that type in the civil fleet, and the subtotal amount of Halon 1301 required for that type of aircraft in the civil fleet. Table 2 summarizes the data by aircraft manufacturer and model only. Table 3 further summarizes the data into the total amount of Halon 1301 used in the civil fleet by each separate aircraft manufacturer.

The tables include only those aircraft models which use Halon 1301 as the primary fire extinguishing agent. Aircraft which use other extinguishing agents, such as Halon 1211, were not listed. Included would be the Fokker models F-100, F-28, and similar aircraft.

1.2 SURVEY RESULTS

Preparation of the estimate for Halon 1301 banked in the fixed systems of the current domestic fleet was performed by Mr. Robert A. Dickson. This estimate, as recorded by Mr. Dickson those aircraft operating under FAR.121, FAR.125 or FAR 135 consisting of Domestic and/or Flag Air Carriers, Supplemental/Scheduled Cargo Air Carriers, Commercial Operators (scheduled intrastate only), and Commuter Air Carriers.

Operators in these categories report data monthly to their FAA District Office. For the estimate, aircraft models and quantities were obtained from the June 1990 issue of FAA's "Air Carrier Aircraft Utilization and Propulsion Reliability Report". Aircraft quantities have been adjusted to reflect new aircraft deliveries for the second half of 1990.

The number of containers per aircraft and the quantity of Halon 1301 per container is based on best available information. Each specific part number container has a nominal agent charge with a charging tolerance of plus a fraction of a pound - minus zero pounds. For the estimate, the nominal agent charge was used.

For the most part, the estimate is based on the "as delivered" extinguishing system configuration. Some older aircraft may have been modified - such as the addition of an APU, and engine retrofit, or conversion from Halon 1211 to Halon 1301; however, detailed information on these modifications is not available. Modifications that were considered in this estimate are:

- 767's with added cargo firex agent quantity for extended range (EROPS).
- DC-8-60 Series re-engined with CFM-56 engines.
- CV 340/440's re-engined with Allison 501 or RR Dart turboprop engines.
- Light twins originally certified for private/business operation with engine firex added for commercial operation under FAR 135

Regarding lavatory fire protection, it is known that a few carriers use a central agent container capable of being directed to any one of several lavatories for "total flooding" type protection. Detailed information on the number of aircraft equipped with this system is not available. For the purpose of the estimate, it was assumed that all lavatories use a small automatic fire extinguisher (Potty Bottle) protecting the waste container. Since the number of lavatories varies with aircraft interior arrangement and the mix between first/business/coach classes, the estimate used a figure of one lavatory for approximately every 30 passengers in a medium density seating configuration.

The survey revealed a current total of 5,468 aircraft in the civil fleet which have 217,773 pounds of Halon 1301 banked in their fixed systems. As a group, the two largest users of Halon 1301 are aircraft manufactured by Boeing and McDonnell Douglas. Boeing manufactured aircraft, which consist of 2,362 units, or 43 percent of the total number of aircraft, use 112,804 pounds of Halon 1301, or 52 percent of the total amount of Halon 1301 currently found in the civil fleet. There are also 1,294 McDonnell Douglas manufactured aircraft, which comprise 24 percent of the total number of aircraft in the civil fleet. McDonnell Douglas aircraft require 64,870 pounds of Halon 1301, or 30 percent of the total amount of Halon 1301 in the civil fleet. The remaining 3,656 civil fleet aircraft, or 33 percent of the total number, are produced by 18 different manufacturers. These aircraft require a collective total of 38,533 pounds of Halon 1301, or 28 percent of Halon 1301 banked in civil fleet aircraft.

Within Table 1, when comparing installed agent amounts between similar aircraft designs from different manufacturers, it should be noted that some aircraft companies practice a more conservative approach to equipping aircraft with fire extinguishing agent. For example, aircraft manufactured by McDonnell Douglas do not combine Halon bottle applications so that bottles designated to be used on two engines could also be used to provide some cargo coverage. This is one reason why McDonnell Douglas aircraft of narrow body design (DC-8) require more agent than narrow body design Boeing (707) aircraft.

TABLE 1. HALON 1301 AMOUNTS BY AIRCRAFT AND ENGINE MODELS

	A/C Mfgr	A/C Model	Eng. Model	No. in Civil Fleet	Halon 1301 lbs/unit	Halon 1301 lbs/model
1.	Aeorspatiale	Airbus A310	PW JT9D & PW 4152	21	161.00	3381.00
2.	Aeorspatiale	Airbus A320	CFM 56	12	50.25	603.00
3.	Aerospatiale	Airbus A300	CF6-50	67	161.50	10850.50
4.	Aerospatiale	AS 350D	Lycoming LTS-101	4	10.00	40.00
5.	Aerospatiale	ATR42/72	PWC PW120, PW121	73	23.82	1739.00
6.	Beech Aircraft	1900	PWC PT6A	119	4.20	499.80
7.	Beech Aircraft	200	PWC PT6A	12	5.00	60.00
8.	Beech Aircraft	58	Continental 105	8	5.00	40.00
9.	Beech Aircraft	99	PWC PT6A	37	5.00	185.00
10.	Boeing	707-100, -300; 720	PW JT3D	27	19.00	513.00
11.	Boeing	727-100, -200	PW JT8D	1112	21.95	24408.40
12.	Boeing	737-100, -200	PW JT8D	377	9.20	3468.40
13.	Boeing	737-300, -400, -500	CFM 56	416	16.20	6739.20
14.	Boeing	747-100, -200, -300	PW JT9D	172	238.25	41339.00
15.	Boeing	747-400	PW 4056	12	275.25	3303.00
16.	Boeing	757-200	RB211-535	57	94.35	5377.95
17.	Boeing	757-200	PW 2037	63	103.75	6536.25
18.	Boeing	767-200	GE CF6-80A	28	124.30	3480.40
19.	Boeing	767-200	PW JT9D	19	120.70	2293.30
20.	Boeing	767-200ER	GE CF6-80A	17	179.30	3048.10
21.	Boeing	767-200ER	PW JT9D	11	175.70	1232.70
22.	Boeing	767-300	GE CF6-80A	6	149.30	895.80
23.	Boeing	767-300ER	CF6-80C2	39	208.30	8123.70
24.	Boeing	767-300ER	PW 4060	6	224.10	1344.60

TABLE 1. HALON 1301 AMOUNTS BY AIRCRAFT AND ENGINE MODELS - Cont'd

A/C Mfgr	A/C Model	Eng. Model	No. in Civil Fleet	Halon 1301 lbs/unit	Halon 1301 lbs/model
25. British Aerospace	ATP	PWC 124	3	24.07	144.42
26. British Aerospace	Jetstream 31	TPE 331	160	8.40	1344.00
27. Britten-Norman	BN2A, BN2B	Allison 250 B17, Lycoming LT10-540	15	5.00	75.00
28. Cessna	402	Continental TSIO520	113	5.00	565.00
29. Cessna	441	Garrett TPE 331	3	5.00	15.00
30. Cessna	550	PW JT15D	7	8.00	56.00
31. Convair	440	R-2800	16	40.50	648.00
32. Convair	580/640	Allison [illegible], RR DART 542	32	36.50	1168.00
33. de Havilland	DHC-6	PWC PT6A	66	2.74	180.84
34. de Havilland	DHC-7	PWC [illegible]	40	29.50	1180.00
35. de Havilland	DHC-8	PWC PW120	70	21.50	1505.00
36. Dornier	DO 228	TPE 331	41	6.00	246.00
37. Embraer	EMB-110	PWC PT6A	52	4.20	218.40
38. Embraer	EMB-120	PWC PW118	138	14.75	2035.50
39. Fairchild	SA 226	TPE 331-3	47	5.00	235.00
40. Fairchild	SA227	TPE 331-11	200	5.00	1000.00
41. Fokker	F-100	RR TAY620	16	10.25	164.00
42. Fokker	F-28	RR SPEY 555 or TAY650	48	10.00	480.00
43. Grumman	G73 Mallard	PW PT6A, PW R-1340	10	10.00	100.00
44. Gulfstream	G159	RR DART 529 or RR SPEY 511	7	21.75	152.25
45. Learjet	25	GE CJ610	2	10.00	20.00
46. Learjet	35A	TFE 731	2	10.00	20.00
47. Lockheed	L-100	501D	20	76.00	1520.00
48. Lockheed	L-188	501D	32	42.00	1344.00

TABLE 1. HALON 1301 AMOUNTS BY AIRCRAFT AND ENGINE MODELS - Cont'd

A/C Mfgr	A/C Model	Eng. Model	No. in Civil Fleet	Halon 1301 lbs/unit	Halon 1301 lbs/model
49. Lockheed	L1011-100, 200, 250	RR RB211-22, -524B	90	49.40	4446.00
50. Lockheed	L1011-500	RR RB211-524B	11	194.40	2138.40
51. McDonnell Douglas	DC-10 Series 10	GE CF6-6, -50	117	128.00	14976.00
52. McDonnell Douglas	DC-10 Series 30	GECF6-6, -50	49	293.00	14357.00
53. McDonnell Douglas	DC-10 Series 40	PW JT9D	21	263.00	5523.00
54. McDonnell Douglas	DC-8 Series	PW JT3D	10	34.60	346.00
55. McDonnell Douglas	DC-8 Series 60	PW JT3D	73	35.10	2562.30
56. McDonnell Douglas	DC-8 Series 70	CFM 56	83	44.00	3652.00
57. McDonnell Douglas	DC-9 Series 10, 15, 30, 40, 50	PW JT8D-1, -7, -9, -11, -15, -17	508	17.80	9042.40
58. McDonnell Douglas	MD-11	GE CF6-80C2	5	293.00	1465.00
59. McDonnell Douglas	MD-80 and DC-9-80	JT8D-209, 217, 219	428	30.25	12947.00
60. Mitsubishi (Nihon)	YS-11	RR DART 542	21	4.50	94.50
61. Piper Aircraft	PA 23/31	Lycoming 105	85	5.00	425.00
62. Piper Aircraft	PA 34	Lycoming D540, Continental TSO 360	7	5.00	35.00
63. Piper Aircraft	PA31	PWC PT6A	9	13.00	117.00
64. Saab	340	GE CT7	105	10.90	1144.50

TABLE 2. HALON 1301 AMOUNTS BY AIRCRAFT MODEL

	Aircraft Mfgr	Aircraft Model	No. in Civil Fleet	Halon 1301 Lbs/Model
1.	Aerospatiale	Airbus A300	67	10851
2.	Aerospatiale	Airbus A310	21	3381
3.	Aerospatiale	Airbus A320	12	603
4.	Aerospatiale	AS 350D	4	40
5.	Aerospatiale	ATR42/72	73	1739
6.	Beech Aircraft	1900	119	500
7.	Beech Aircraft	58	8	40
8.	Beech Aircraft	99/200	49	245
9.	Boeing	707	27	513
10.	Boeing	727	1112	24408
11.	Boeing	737	793	10208
12.	Boeing	747	184	44642
13.	Boeing	757	120	11914
14.	Boeing	767	126	21119
15.	British Aerospace	ATP	3	144
16.	British Aerospace	Jetstream 31	160	1344
17.	Britten-Norman	BN2A, BN2B	15	75
18.	Cessna	402	113	565
19.	Cessna	441	3	15
20.	Cessna	550	7	56
21.	Convair	440	16	648
22.	Convair	580/640	32	1168
23.	de Havilland	DHC-6	66	181
24.	de Havilland	DHC-7	40	1180

TABLE 2. HALON 1301 AMOUNTS BY AIRCRAFT MODEL - Cont'd

	Aircraft Mfgr	Aircraft Model	No. in Civil Fleet	Halon 1301 Lbs/Model
25.	de Havilland	DHC-8	70	1505
26.	Dornier	DO 228	41	246
27.	Embraer	EMB-110	52	218
28.	Embraer	EMB-120	138	2036
29.	Fairchild	SA226/SA227	247	1235
30.	Fokker	F-100	16	164
31.	Fokker	F-28	48	480
32.	Grumman	Mallard/Widgeon	10	100
33.	Gulfstream	G159	7	152
34.	Learjet	35/36	4	40
35.	Lockheed	L-100	20	1520
36.	Lockheed	L-188	32	1344
37.	Lockheed	L1011	101	6584
38.	McDonnell Douglas	DC-10	187	34856
39.	McDonnell Douglas	DC-8	166	6560
40.	McDonnell Douglas	DC-9	508	9042
41.	McDonnell Douglas	MD-11	5	1465
42.	McDonnell Douglas	MD-80	428	12947
43.	Mitsubishi (Nihon)	YS-11	21	95
44.	Piper Aircraft	PA 23/31/34	92	460
45.	Saab	340	105	1145
			Total Aircraft	**Total Agent**
			5468	217,773 Pounds Halon 1301

TABLE 3. HALON 1301 AMOUNTS BY AIRCRAFT MANUFACTURER

	Aircraft Mfgr	No. in Civil Fleet	Halon 1301 Lbs per Mfgr
1.	Aerospatiale	177	15048
2.	Beech Aircraft	176	785
3.	Boeing	2362	112804
4.	British Aerospace	163	1488
5.	Britten-Norman	15	75
6.	Cessna	[illegible]	636
7.	Convair	[illegible]	1816
8.	de Havilland	176	2866
9.	Dornier	41	246
10.	Embraer	190	2254
11.	Fairchild	247	1235
12.	Fokker	64	644
13.	Grumman	10	100
14.	Gulfstream	7	152
15.	Learjet	4	40
16.	Lockheed	153	9448
17.	McDonnell Douglas	1294	64870
18.	Mitsubishi (Nihon)	21	95
19.	Piper Aircraft	92	460
20.	Saab	105	1145

2. A SUBSTANTIATED ESTIMATE OF THE YEARLY DISCHARGE OF HALON 1301 BANKED IN THE DOMESTIC CIVIL FLEET [1]

2.1 GENERAL

Mr. Tom Peacock of McDonnell Douglas Aircraft approached the task of estimating the yearly Halon 1301 discharged by the civil fleet with an assessment of the bottle discharge reported in three data bases, one public and two proprietary. Only U.S. registered carriers were included. The data obtained was then used in Section 3 of this report to estimate Halon 1301 release from aircraft over the ten year period 1991 through 2000.

Estimates of fleet sizes were obtained from public, Douglas Aircraft, and Walter Kidde Aerospace sources. The requirement to use multiple data sources in this study results in some disagreement concerning fleet size numbers. However, the detailed analyses produced deviation values within 4 percent.

2.2 ANNUAL INSERVICE HALON 1301 CONSUMPTION

Estimating the yearly discharge of Halon 1301 from fixed systems of the domestic civil fleet was done by sampling historical records of U.S. registered aircraft operations for the 24 month period July 1988 through June 1990. These records were processed to provide the best estimate of agent discharge totals. The total events were then analyzed by mode of halon release, aircraft type and model, and average number of airplanes in the fleet during the time period. Factors derived relating these parameters were then applied in Section 3 to the estimated U.S. fleet aircraft during the ten year period 1991 through 2000.

From Table 9, Section 3.3, total Halon 1301 estimated consumption is 138,343 pounds between 1991 and 2000. If Class D compartments are redesignated and operated as Class C this consumption is estimated to increase by 31,672 pounds to 170,015.

From these numbers an annual estimated average consumption of 13,834 pounds results for the current civil fleet (17,002 if Class D to C compartment election is made). Emissions for aviation testing and for hydrotest inspection requirements are addressed in Sections 2.6 and 2.7 and are not included in this estimate.

2.3 FLEET RECORD SAMPLING

From analysis of "reported" agent discharge occurrences, an inferential estimate of the "actual" discharges occurring, and projection of these events over the 1991 to 2000 period were developed by Mr. Tom Peacock of the McDonnell Douglas Aircraft Company.

In recognition of the fact that all discharges are not captured by reporting media, it was decided that the best prediction of usage could be determined by estimating the number of discharges that occurred rather than the number of discharges that are captured by the reporting media.

An accepted practice of estimating a population's size (e.g., the number of fish in a lake) is to take independent samplings and draw an inference of the population's size by reference to sample sizes

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[1] Estimates developed by Mr. Tom Peacock of McDonnell Douglas Corporation, Douglas Aircraft Company, 3855 Lakewood Blvd., Long Beach CA 90846-0001, under contract to Walter Kidde Aerospace, Inc., as a work structured toward specific goals required within this feasibility study.

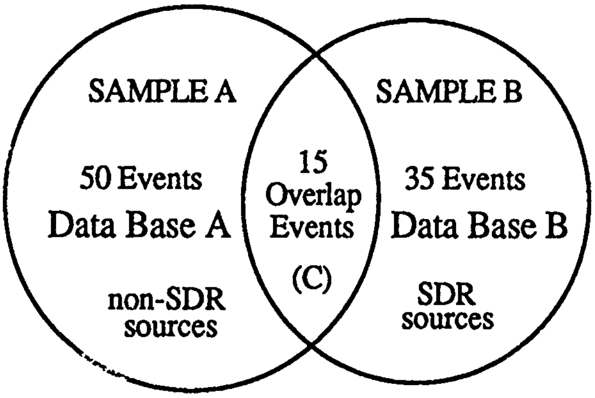
and the relative overlap/underlap between these samples. The larger the size of each sample and overlap between them, the less likely that the population unaccounted for is extensive. Conversely, the less sample overlap between the given samples, the greater the likelihood that there are occurrences in the population that neither sample captured and that the population is more expansive than the arithmetic sum of the samples.

Mathematically, the product of the sample sizes divided by the overlap between them infers the population's true size.

Douglas Aircraft Company maintains an extensive Safety Information System (SIS) data base that captures, among other things, fire extinguisher discharge reports. SIS exists as a singular data base which draws information from a broad group of sources (samples), including the FAA's Service Difficulty Report (SDR) system. Since this study is targeted at discharge volume in the U.S., the SDR source is geographically ideal. The non-SDR and SDR sources were therefore selected as samples "A" and "B" in order to estimate the "real" population of agent discharges.

This concept is shown graphically in Figure 1 as a Venn diagram. Figure 1 shows 50 Sample A (non-SDR reported) events, 35 Sample B (SDR reported) events, and 15 overlapping (non-SDR and SDR reported) events. The product of Sample A and Sample B divided by the overlap, as previously described, is 116.7 probable total events.

Douglas Aircraft Halon Bottle Events
July 1 1988 - June 30 1990



$$\text{Estimate} = \frac{50 \text{ X } 35}{15} = 116.7 \text{ Total Events}$$

FIGURE 1

All data base records were electronically searched, but the ultimate selection, interpretation, and event counting was performed manually. This process was rigorous and required many rechecks which confirmed final data set validity.

Users and maintainers of the data bases have learned that although all events are not recorded in every data base, a high confidence estimate of the total set of events can be made by using accepted probability theory (reference 1). This Venn diagram process and approach, presented in Figure 1, was verified using computer simulation models.

Data base screening for this study was performed using key words such as "fire", "extinguisher", "firex", "bottles", "fire warning", "cabin", "APU" (auxiliary power unit), "engine" and "cargo".

An event is defined as a single reportable situation or occurrence outside that of "normal" safe aircraft operation. For this study those situations relate to Halon usage or predicted usage.

The number of halon bottle discharges in the engine nacelle, APU compartment, cargo compartment and cabin (potty and handheld 1211 portables - mostly 1211 portables) were counted and are recorded in Table 4. There were no reports of leakage, discharge for bottle service, or for container retirements. All discharges were in response to a system warning, or observance of a fire, or were due to pneumatic system overtemperature. Thermal events may be considered "accidental" but are included in predictions because they do occur, albeit infrequently, and can be expected to occur in the future. The estimated number of events is expected to be on the conservative side since discharge is assumed even if verbal description was not explicit. For example, if an engine was shut down in response to a fire warning and it was not stated (it sometimes was) that both bottles were fired, it was assumed that only one of the bottles was actually fired unless the description went on to say that the fire light stayed on which would require the crew to fire the second bottle. The same assumption was made for cargo discharges. A cabin bottle discharged into electrical equipment was assumed to be Halon 1211 if agent type was not reported.

TABLE 4. Data Base B (SDR Sources)
Bottle Discharges Counted
July 1988 - June 1990

MODEL	CABIN	APU	ENGINE	CARGO
DC 8	2	0	13	0
DC9/MD80	7	4	8	0
DC10	4	5	1	1
B707	0	0	2	0
B727	10	16	23	1
B737	4	31	6	0
B747	9	4	27	17
B757	5	0	0	5
B767	5	2	0	2
A300	2	0	2	3
A320	0	0	0	1
BAC111	0	0	2	0
L1011	16	2	2	0
F28	0	1	0	0
TOTAL	64	65	86	30

Within the cabin designated column recorded discharges consist of one potty bottle discharge and 63 Halon 1211 handheld portable fire extinguishers. All discharges within APU, engine and cargo columns are Halon 1301 fire extinguishers.

All zeros in Table 4 are not necessarily the result of event non-occurrence where occurrence was possible. There are instances where equipment is either not installed or is installed on an insignificant number of airplanes which makes an event occurrence to be impossible or extremely unlikely. For example, the data base shows no discharges into DC-9/MD-80 cargo area because this equipment is not installed on these aircraft. The same is true for most narrow body aircraft. The DC-8 and B-707 does not have APU equipment and only a few have been fitted in the after market.

Fleet records adjusted by statistical methods outlined above are depicted in Table 5. Decimals associated with the factors were carried through. These figures are carried into the following phase of the analysis.

TABLE 5. Estimated Bottles Discharged
July 1988 - June 1990

MODEL	CABIN	APU	ENGINE	CARGO
DC 8	6.67	--	43.2	--
DC9/MD80	23.3	13.3	26.6	--
DC10	13.3	16.6	3.33	3.33
B707	--	--	21.6	--
B727	33.3	53.3	76.6	--
B737	13.3	103.0	20.0	--
B747	30.0	13.3	89.9	56.6
B757	16.6	0	0	16.6
B767	16.6	6.67	0	6.67
A300	6.67	0	6.67	10.0
A320	0	0	0	3.33
BAC111	0	0	6.67	--
L1011	53.3	6.67	6.67	0
F28	0	0	0	--
TOTAL	213.0	216.2	301.2	96.5

ESTIMATED GRAND TOTAL = 827 BOTTLES

2.4 FLEET RECORD ANALYSIS

Values in Table 5 were next brought into Table 6 where the fleet record event data was put on the basis of events per airplane and per engine. The average existing fleet size values shown for the sampled period were estimated using numbers from multiple public sources, available Flying Time indexes, Flight International annual census and Air Transport World annual census. These sources contained conflicting data in some areas so representative values were chosen. Within the Boeing 747 data group the data base reveals a higher number of discharges which are attributed to an increased frequency of false alarms.

Table 6 depicts a wide variation in individual model discharge frequencies on both per engine and per airplane bases. This factor in addition to a problem of transferring data into the next usage resulted in grouping the data to a "per airplane" basis within the broad classes of wide and narrow body aircraft.

TABLE 6. Halon Usage Analysis July 1988 - June 1990

			BOTTLES DISCHARGED								
			ENGINE			APU		CARGO		CABIN	
AIRCRAFT TYPE	AVG FLEET	TOTAL ENGINES	TOTAL	PER ENG.	PER PLANE	TOTAL	PER PLANE	TOTAL	PER PLANE	TOTAL	PER PLANE
NARROW BODY											
F28	47	94	0	--	--	3.33	.071	--	--	0	--
BAC111	9	18	6.67	--	--	0	--	--	--	0	--
DC9/MD80	965	1930	26.6	.0138	.0276	13.3	.0138	--	--	23.3	.024
B727	1108	3324	76.6	.023	.069	53.3	.048	--	--	33.3	.030
B737	706	1412	20	.0142	.028	103	.146	--	--	13.3	.019
B757	149	298	0	--	--	0	--	16.6	.111	16.6	.111
A320	10	20	0	--	--	0	--	3.33	.33	0	--
DC8	164	656	43.2	.066	.263	--	--	--	--	6.67	.041
B707	29	116	21.6	.186	.745	--	--	--	--	--	--
TOTAL	3187	7868	194.67			172.93		19.93		93.17	
WIDE BODY											
DC10	183	549	3.33	.0061	.0182	16.6	.091	3.33	.0182	13.3	.073
B747	192	768	89.9	.117	.468	13.3	.069	56.6	.295	30	.156
B767	95	190	0	--	--	6.67	.070	6.67	.070	16.6	.175
A300	72	144	6.67	.046	.093	0	--	10	.139	6.67	.093
L1011	114	342	6.67	.0195	.059	6.67	.059	0	--	53.3	.468
TOTAL	656	1993	106.57			43.24		76.6		119.87	

Notes:
1. Fleets estimated using several information sources, freighters included.
2. Discharges are for a two year period, July 1988 through June 1990.
3. Entries designated as -- indicate no system installed.
4. Entries designated as 0 indicate no report of incident.
5. Most of the 164 DC8 aircraft have been converted to freighters.
6. F28 and BAC111 aircraft are converted to freighters.

Freighters have been included because they have engines and APUs. They do not help fill in the other two modes of discharge because they do not have passenger cabins nor are they required to have extinguishers in lower cargo spaces. There was one event in the data base where both engine bottles and a single extinguisher serving a hazardous material container in upper cargo were all discharged as a precaution during an emergency evacuation. The populations used in Table 7 were only the aircraft experiencing the mode of discharge employed in the calculation. The factors calculated in Table 7 were then carried into Table 9 in Section 3. Table 10 provides an overall summary of Halon use.

TABLE 7. Summary Of Halon Usage Analysis

NARROW BODY

Engines	$\frac{\text{195 bottles}}{\text{3187 a/c 2 yrs}}$	=	.0306	$\frac{\text{bottles}}{\text{aircraft year}}$	
APU	$\frac{\text{173 bottles}}{\text{2994 a/c 2 yrs}}$	=	.0289	$\frac{\text{bottles}}{\text{aircraft year}}$	
Cabin	$\frac{\text{93.2 bottles}}{\text{3158 a/c 2 yrs}}$	=	.0148	$\frac{\text{bottles}}{\text{aircraft year}}$	(Halon 1211 - See Table 9)
Cargo	$\frac{\text{19.9 bottles}}{\text{159 a/c 2 yrs}}$	=	.0626	$\frac{\text{bottles}}{\text{aircraft year}}$	

WIDE BODY

Engines	$\frac{\text{106.6 bottles}}{\text{656 a/c 2 yrs}}$	=	.0813	$\frac{\text{bottles}}{\text{aircraft year}}$	
APU	$\frac{\text{43.2 bottles}}{\text{656 a/c 2 yrs}}$	=	.0329	$\frac{\text{bottles}}{\text{aircraft year}}$	
Cabin	$\frac{\text{120 bottles}}{\text{656 a/c 2 yrs}}$	=	.0915	$\frac{\text{bottles}}{\text{aircraft year}}$	(Halon 1211 - See Table 9)
Cargo	$\frac{\text{76.6 bottles}}{\text{656 a/c 2 yrs}}$	=	.0584	$\frac{\text{bottles}}{\text{aircraft year}}$	

Note: Class D cargo compartments in narrow bodies, Class E in freighters accounted for.

2.5 HALON 1301 - ESTIMATED EMISSION FOR AVIATION TESTING

Fire suppression systems in domestic aircraft are usually certified by test using applicable agent and plumbing design. This test is usually performed in some "nominal" condition either in flight or on the ground. During the test, one or more fire extinguisher bottles are discharged which generates Halon concentration within the compartment airstream. This mixture of air and Halon from the discharge is detected by various means and instruments.

Walter Kidde Aerospace keeps all certification records, for a period of time, indicating exact quantities of agent used for suppression system testing. Typically, six to fifteen system discharges are required to optimize discharge nozzle location and size for proper agent distribution which effects efficient fire suppression. These tests are usually performed on each unique model of aircraft or on aircraft where significant design modifications have been incorporated. Testing is normally performed only once per aircraft model which helps in minimizing agent lost to testing.

There are specific requirements for minimum agent concentration and duration.[2, 3, 4] (reference 10)

Typical aircraft fire suppression system testing involves between 6 to 15 system discharges. This is true whether agent concentration testing is being performed in an engine or cargo or APU compartment. Small compartments require fewer discharges due to their typically requiring fewer nozzle locations as determined by airflow through the firezone and firezone geometry. Large, high airflow, firezone volumes which require 15 to 27 outlet nozzle locations typically require additional discharges in order to optimize agent distribution and application for the purpose of minimizing agent weight requirements.

Since APU or cargo compartments are generally of the same design and airflow dynamics for each aircraft of a given model, only one model need be tested. Each engine on a multi-engine aircraft is usually the same or similar enough such that concentration certification for one engine is adequate for all engines of that type when used in similar installations.

Typical engine agent concentration certification using Halon 1301 will consume approximately 150 pounds of agent. Cargo system testing can consume 270 pounds in a single discharge. From agent concentration testing performed by Walter Kidde Aerospace during the past three years on MD-11 engine compartments with CF6-80C2 and PW4000 engines, Beech Starship, Seastar, 747SP APU compartments, Bell Boeing V-22 APU, A330/A340 APU, EA6B with PW 408 engine and other aircraft it is estimated that approximately 1335 pounds of Halon 1301 is expended per year for aircraft certification test purposes.

2.6 HALON 1301 - ESTIMATED EMISSION FOR CURRENT HYDROTEST INSPECTION REQUIREMENTS[5]

Banked within fixed aviation systems are approximately 109 tons of Halon 1301. The requirement (reference 2) that all aircraft pressure vessels be hydrostatically inspected every five years requires, on average, 20 percent of this 109 ton quantity be dumped each year if not otherwise recovered.

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[2] The FAA definition of "adequate level" is found in Technical Development Report No. 403, page 14, which states that the minimum relative concentration at all pickup locations for 0.5 seconds is 15 percent concentration relative to air for Halon 1301 (CF_3Br).

[3] FAR 25.1195(b) states, "It must be shown by either actual or simulated flight tests that under critical airflow conditions in flight the discharge of the extinguishing agent in each designated fire zone...will provide an agent concentration capable of extinguishing fires in that zone and of minimizing the probability of reignition."

[4] FAR 25.1195(c) states "The fire-extinguishing system for a nacelle must be able to simultaneously protect each zone of the nacelle for which protection is provided."

[5] Estimate developed by Walter Kidde Aerospace based upon data contained in Sections 1 and 2 of this study.

This is a demand of 22 tons per year. As the fixed aviation systems Halon 1301 increases so does this demand due to hydrotest requirements.

From estimations presented in Section 3.4, the total Halon 1301 requirements for new aircraft, years 1991 to 2000, are 479,886 pounds increasing to 547,560 pounds if Class C redesign and operation of Class D compartments occurs.

The maximum Halon 1301 requirement of 547,560 pounds for new production aircraft, 1991 - 2000, results in an average annual demand of 54,756 pounds of Halon 1301. In 5 years this same amount will be required by current regulations to be removed for hydrotest inspection of fire extinguisher containers.

FAA Order 8000.40B, MAINTENANCE OF PRESSURE CYLINDERS IN USE ARE AIRCRAFT EQUIPMENT, dated 18 October 1991 (reference 2), reinstituted five year hydrostatic pressure test interval requirements for all aircraft pressure vessels. This requirement had previously been cancelled by FAA Order 8000.40A dated 17 July 1983.

Average annual Halon 1301 demand due to inspection requirements outlined in FAA Order 8000.40B, which requires fire extinguisher bottles (pressure vessels) pressure testing at 5 year intervals, will be approximately 54,756 pounds per year, beginning in 1996. This approximation assumes an average linear relationship based on projections from one-tenth the projected (547,560 pounds) 1991-2000 new production aircraft Halon 1301 requirement. This worst-case requirement does not consider that agent removal for hydrotesting and inspection could be recovered at 98% efficiency by a Halon 1301 recovery system.

An average of 43,555 pounds of Halon 1301 per year, or one-fifth the existing bank of 217,773 pounds, would be required if fire extinguishers on existing fleet aircraft, including commuter aircraft, were to undergo hydrostatic test inspection during the next five years. However, in 1996, due to the five year limit, all fire extinguisher bottles placed on aircraft manufactured during 1991 must also be hydrostatically inspected. Aircraft newly produced in 1991 have an initial agent requirement of approximately 54,756 pounds, therefore the 1996 agent demand due to hydrotest requirements is projected to increase to a minimum of 98,311 pounds which is the sum of the current average fleet and 1991 manufactured aircraft agent requirements (43,555 pounds plus 54,756 pounds). *Note: The 43,555 figure for existing fleet aircraft has been averaged over a five year period. It is not realistic to expect that actual annual demand of Halon 1301 will be 43,555 pounds. More likely, hydrostatic pressure testing on existing fleet fire extinguishers will be deferred as long as possible so that the actual 1996 demand for Halon 1301 could greatly exceed 98,311 pounds.*

Continued fleet growth, which is estimated at 3.5 percent to 4 percent per year beyond 2000, also increases the demand for Halon 1301 due to the 5 year hydrostatic testing requirement. The demand increase will be realized beginning in 2005 and will continue to increase at an annual 3.5 to 4 percent rate.

3. ESTIMATE THE ANNUAL HALON 1301 NEEDS FOR NEWLY PRODUCED AIRCRAFT OVER THE NEXT TEN YEARS [6]

3.1 GENERAL

As in Section 2, the required use of multiple data sources resulted in disagreement about fleet size and growth prediction. In some cases the fleet is divided and predictions are made using different classifications. This sometimes generates apparent population inconsistencies when these numbers are compared to other cases. However, analyses demonstrate less separation with overall values being within a 4 percent deviation level, which is an acceptable level of precision required of this study. Fleet growth values have historically and generally followed GNP values.

3.2 FLEET SIZE PREDICTION FOR U.S. DOMESTIC CIVIL FLEET 1991-2000

Data for prediction of future civil fleet size was produced from a published Douglas marketing analysis (reference 3) adjusted and updated using unpublished proprietary estimates of market requirements, market shares and model/market splits. Table 8 presents data from the above adjustments and updated information. Population within groupings is different here than in Table 6 because some large narrow body aircraft are moved into the "medium" category and because freighter population changes within the prediction. This study was performed using two periods of interest, historical records and future projections. A different data base for each category was available and accessible. Because the project scope did not allow extensive data base fractioning and reformation, it was more logical and convenient to divide the historical data base into wide and narrow fuselage categories and to divide the future projection data base into seating capacity categories. Therefore, large, narrow body aircraft fit into one category in the historical treatment and into another category in the future projections. However, overall aircraft count is consistent. Operating fleet predictions are annual and then summed into a total of integrated aircraft years in order to obtain estimates of related fleet Halon consumption over the ten year period.

TABLE 8. Domestic Civil Fleet Size Estimates By Year

Aircraft Type	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	Integrated Aircraft Years
Narrow Body											
Passenger	2359	2399	2441	2490	2536	2564	2608	2645	2674	2703	25419
Freighter	222	228	234	240	246	258	270	282	294	305	2579
Medium & Wide Body											
Passenger	1216	1272	1334	1397	1465	1532	1613	1683	1770	1843	15125
Freighter	200	209	219	228	239	252	265	278	291	305	2486

Freighters are included because of their commonality of systems, engines, and APUs with passenger aircraft. They are not used in the calculation of cargo or cabin discharges because they are largely operated as Class E aircraft which do not require extinguishing systems. Combi's have not been considered because they are not in service in the U.S. domestic fleet to a degree significant to this study. Where Combi's are operated their use is of a somewhat unique and specialized nature.

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[6] Estimates developed by Mr. Tom Peacock of McDonnell Douglas Corporation, Douglas Aircraft Company, 3855 Lakewood Blvd., Long Beach CA 90846-0001, under contract to Walter Kidde Aerospace, Inc., as a work structured toward specific goals required within this feasibility study.

3.3 INSERVICE HALON CONSUMPTION

Table 9 combines fleet size aspect with estimated bottle discharge frequency and bottle size representative of aircraft type and protection mode. Foreign produced aircraft in U.S. registered operator service are included.

Total Halon 1301 release from 1991-2000 for each protected aircraft area is presented. The rates per year are one-tenth the total amounts. Ten year Halon 1301 consumption is forecast at 138,343 pounds. Ten year Halon 1211 consumption is forecast at 4572 pounds. Values in parentheses for passenger installation on narrow body aircraft is that amount of Halon 1301 increase for fleet maintenance for re-designation and operation of Class D cargo compartments as Class C.

TABLE 9. Inservice Fleet Halon Consumption 1991 - 2000

	Narrow Body		Med & Wide body		Estimated Total Consumption
	Passenger	Freighter	Passenter	Freighter	
Aircraft Years	25419	2579	15125	2486	
Engine Installations (1301)					
Discharged Bottles/Airplane Year	.0306	.0306	.0813	.0813	
Bottle Size, lbs	14.25	14.25	19.6	19.6	
Usage, lbs	11084	1125	24101	3961	40271
APU Installations (1301)					
Discharged Bottles/Airplane Year	.0289	.0289	.0329	.0329	
Bottle Size, lbs	3	3	5	5	
Usage, lbs	2204	224	2488	409	5325
Cargo Installations (1301)					
Discharged Bottles/Airplane Year	.0623	–	.0584	–	
Bottle Size, lbs	20	–	105	–	
Usage, lbs	(31672)	[3213]	92747	[15244]	92747
	TOTAL HALON 1301 (If Class D - - C)				138343 (170015)
Cabin Installations (1211)					
Discharged Bottles/Airplane Year	.0175	–	.0915	–	
Bottle Size, lbs	2.5	–	2.5	–	
Usage, lbs	1112	–	3460	–	4572
	TOTAL HALON 1211				4572

Average bottle size in Table 9 was determined by examining fleet distribution, population, and educated estimates based on engines expected to be used and their historical agent requirements. Estimates are conservative with regard to average bottle size.

FAR Part 25, Paragraph 25.857 details the requirements for each compartment classification. Class C requires agent whereas Class D and Class E do not.

Values in brackets within the freighter heading for cargo compartments represent additional Halon 1301 protection on freighter aircraft for re-designation and operation from Class E to operation as Class C. This quantity of 18,457 is forecast due to warning system malfunctions primarily with an intermediate quantity of false fire warnings. There are no events in the data base of actual cargo fires in either passenger or freighter fleets.

Effects of narrow body passenger aircraft Class D (no extinguishing required) compartment re-designation to Class C are estimated to be an annual release of 31,672 pounds of Halon 1301. This number is determined by using Table 7 consumption factors as associated to aircraft years with respective bottle sizes and affected aircraft. This discharge, while providing fire protection if needed, can be expected to result from non-fire alarm occurrences.

3.4 PREDICTED NEW PRODUCTION HALON REQUIREMENT FOR THE U.S. COMMERCIAL CIVIL FLEET 1991-2000

Newly produced passenger aircraft must be equipped with fire protection systems. Table 10 presents a summary of new Halon needs which satisfy estimated new production from 1991 to 2000 in the United States. These are data derived from proprietary fleet projection needs and market share estimates for various sizes of aircraft. The projection includes U.S. production forecast for foreign markets. The McDonnell Douglas study does not include commuter aircraft. New aircraft production in that study are estimated to require larger quantities of engine protection agent because the higher bypass air higher technology engines have greater nacelle airflows.

TABLE 10. Halon Required to Equip New U.S. Domestic Production Aircraft 1991-2000 Excluding Commuter Fleet (Based Upon Approximately 4,500 Aircraft)

	Engine (1301)	APU (1301)	Cargo (1301)	Cabin (1211)
PASSENGER AIRCRAFT				
Narrow Body	75393	3717	15642	27094
If Class C Mandated Add ( )			(40658)	
Wide Body	66894	21786	287122	26531
FREIGHTERS	6910	2422	---------	365
If Class C Elected Add ( )			(27016)	
SUB-TOTALS	**149,197**	**27,925**	**302,764**	**53,990**

TOTAL HALON 1301 = 479, 886 pounds (547,560 if Class C)
TOTAL HALON 1211 = 53,990 pounds

NOTES:

(1) Most new small and medium freighter demand is met by conversion of passenger aircraft which do not need new extinguisher bottles.
(2) Values shown are pounds of agent.
(3) Class C values add if mandated on narrow body and if elected on freighters for lower cargo areas.
(4) Add an additional 5,768 pounds if two Class A rated portable bottles are elected on small and medium freighters and three on large freighters

Projections within Table 10 consider that most small and many medium to large freighter aircraft requirements will be satisfied by conversion of existing passenger aircraft. These conversion aircraft will carry over supplies of Halon for engine and APU protection. Although the freighters can operate as Class E, it can be expected that many new and converted small freighters will need Halon for lower cargo protection only by election. Many freighter aircraft carry portable extinguishers. Allowances may be made for these portable extinguishers by adding the number indicated in the notes of Table 10. Accounting for required or elected Class C cargo compartments in newly produced aircraft can be made by adding values indicated to the respective totals.

3.5 EFFECTS OF REGULATORY CHANGES

Some effects on Halon requirements due to near term regulatory changes are shown in Tables 9, 10 and 11. The possible change associated with conversion of narrow body lower cargo compartments from Class D to Class C has the largest effect on Halon consumption of any other regulatory change anticipated in the near future. Over 31,000 pounds (Table 9, 170,015 minus 138,343) of increased Halon discharge from 1991-2000 is estimated to result from such a change. Almost all of these discharges are due to non-fire conditions. Table 11 provides a detailed description, by aircraft model, of agent requirement for conversion of cargo areas of the current civil fleet from Class D to Class C.

TABLE 11. Cargo Class D To C Conversion

Aircraft	Largest Compartment	Agent Reqd/Bottle	No in Fleet Year End 1990	Agent Reqd
A-320	900 cu. ft.	18.5	11	204
B-727	1000 (est)	20.56	1040	21382
B-737-100/200	505	13.22	756	9994
B-737-300/400/500	786	17	88	1496
B-757	1090	(two 33 lbs in some a/c, use 1)	197	6501
DC-9	717	Use 14.6 lb bottle	448	6541
DC-10		Use 165 lb system	102 (est)	16830
MD-80	464	Use 14.6 lb bottle, 3 head	437	6380
L-1011		Use 165 lb system	59	9735
F-28	500 (est)		45	309
TOTAL				**79372 lbs**

Assume: One shot system and coverage for largest compartment to 6 percent with one bottle (or 20.56 lb/1000 cu ft) if bottles are not known to be approximately compatible with another model or available bottle; or an existing system available for the aircraft. Freighters go Class E. B-757 entries cover some Class D currently flying. Some B-757 aircraft already exist as Class C, and other B-757 aircraft are being converted to freighters.

The FAA recently issued FAA Order 8000.40B which is a policy change requiring more frequent testing of pressurized cylinders on aircraft by imposing a limit on their service times between inspections to intervals conforming to strict government criteria. This policy change was motivated by failures of higher pressure cylinders which were not used as Halon containing fire extinguisher pressure vessels. Nevertheless, the change affects Halon containing fire extinguishers and in particular those containing Halon 1301. The policy change was made with no provision for compliance time. In response to industry appeal, the FAA currently has held enforcement of this new policy in abeyance for an indefinite period. The effects on Halon consumption are not associated with aircraft production or operational usage. A short term effect of fleet compliance to new policy implementation will be a need for additional Halon extinguishers to replace bottles that suddenly become overdue for inspection.

The large increase in Halon bottle reinspections and transport for reinspection due to the policy change will likely increase atmospheric Halon release. The associated inefficiencies of an increased frequency of bottle discharge and refill using unimproved methods will result in an increase in the source of Halon discharge.

A longer term effect will likely be a significant increase in Halon discharged to atmosphere due to transportation, testing and reinspection of the bottles. An increased need to ship the bottles in a short compliance period will likely result in more frequent bottle discharges to atmosphere to allow shipment as regular cargo rather than as hazardous cargo in a pressurized condition.

Another proposal currently under consideration by regulatory agencies would require recurrent flight attendant fire fighting training to be accomplished with actual charged extinguishers (and agent) which they would be expected to use in an actual emergency. Realism of this nature, while improving the fire fighting skills of flight attendants, would result in an increased release of Halon 1211 to atmosphere.

3.6 COMMUTER FLEET HALON 1301 ANNUAL REQUIREMENTS 1991 - 2000[7]

This estimate is from 3 categories, regional/commuter air carriers, commercial operators and business aviation. Table 12 lists the annual needs for newly produced commuter aircraft of those three categories from 1991 to 2000.

The estimates are influenced by current production rates and future predictions by industry officials and analysts. The following is a brief summary of market comments and forecasts for each category of aircraft.

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[7] Preparation of this estimate was performed by Mr. Robert A. Dickson. Mr. Dickson is a retired employee of Walter Kidde Aerospace whose aviation field experience was most valuable in preparing this estimate. Included in this section is a brief summary of market comments by Mr. Dickson based on his investigation. The investigation included a review of various publications and solicitation of expert opinion on the future market for each category of aircraft. The General Aviation Manufacturers Association (GAMA) was contacted and a copy of their General Aviation Statistical Data Book was obtained for a reference. A list was compiled of all multi-engine aircraft in production and under development that would be likely candidates to fill market needs.

TABLE 12. Commuter Fleet Annual Halon 1301 Requirements for New Aircraft

Year	Commuter Fleet Approximate Population	Halon 1301 Requirements (pounds)	Hydrostatic Test Requirements
1991	590	3,516	
1992	663	3,954	
1993	682	4,109	
1994	697	4,210	
1995	721	4,369	
1996	681	4,169	3,516
1997	683	3,955	3,954
1998	657	3,890	4,109
1999	633	3,605	4,210
2000	611	3,502	4,369
		39,280	20,158

Total 10 year need: 29.72 tons

3.6.1 REGIONAL/COMMUTER AIR CARRIERS

During the 80's most of the major airlines in the U.S. have acquired outright or have established working relationships with smaller airlines that feed their route systems. This trend is expected to continue and expand as the majors give up small, unprofitable cities, allowing their regional/commuter affiliates to pick up that service.

The majority of aircraft in operation are 19 passenger turboprops. There is a market for replacement aircraft of this type, as well as a need for larger aircraft for longer route segments dubbed "Regional Jet". This market is dominated by foreign manufacturers. Beech and Fairchild have a share of the 19 passenger turboprop business, but have yet to come up with anything to compete with their foreign competitors in larger capacity aircraft (50 to 75 seats). Fairchild has stretched its Metroliner to accommodate 25 passengers and plans to offer a jet powered version in the late 90's. A modest number of these aircraft are included in the estimate.

3.6.2 COMMERCIAL OPERATORS

Included in this category are air taxis, charter operators and generally anyone other than a scheduled air carrier who carries persons or cargo for hire. Aircraft types are light twins, small jets and helicopters.

The market forecast for this category is mildly optimistic with companies such as Beech, Cessna and Piper continuing to produce light twin piston and turboprop aircraft at rates at or slightly higher than '89 and '90, particularly for the late '90's. Mid size helicopters should enjoy good sales through the '90's for use by air ambulances, law enforcement, executive shuttles and commercial application such as oil platform support.

3.6.3 BUSINESS AVIATION

The sale of business aircraft has been in a decade-long slump. In spite of the current economic situation, most experts predict growth in sales throughout the '90's, particularly in small business jets. Reasons for this optimism are the growing market in European and Pacific Rim nations. If the U.S. dollar remains depressed, manufacturers such as Beech, Cessna, Gulfstream and Learjet should benefit.

Also, both Cessna and Swearingen are developing low cost, low maintenance small business jets using the Williams/Rolls Royce FJ44 fanjet engine. Market experts predict that aircraft of this type will replace the demand for light twin turboprops in the mid to late '90's.

3.6.4 COMMUTER FLEET SUMMARY

In the estimate, it is assumed that these multi-engined aircraft would be equipped with a fire extinguishing system regardless of the type of usage for which the aircraft is certified. Also, where agent quantity was unknown, it was assumed that the relatively small piston, turboprop and turbojet engines used in these aircraft would require only 2.5 lbs. of agent for protection. This is similar to the amount of agent required for this type of power plant in current production aircraft.

3.7 HALON DEMAND SUMMARY 1991 - 2000

Table 13 summarizes Halon 1301 demand for previous categories detailed in tables 9 thru 12, inclusively. Halon 1301 demand illustrated in Table 13 does not consider recovery of that agent lost due to hydrotest inspection requirements. Recovery of that agent which would otherwise be lost because of hydrotest inspection at a 98 percent available reuse efficiency rate should significantly reduce Halon 1301 demand.

TABLE 13. Halon 1301 Demand Summary
1991 - 2000

Category	Estimated Total Consumption (Pounds)	Class Conversion	Hydrotest Consumption
Inservice fleet usage engine installations	40,271		Existing Fleet 1991 - 2000 engine + APU + cargo
Inservice fleet usage APU installations	5,325		436,000
Inservice fleet usage cargo installations	92,747		
If Class D - C	(31,672)		79,372
Freighter	[18,457]		
Existing fleet Class D - C retrofit/conversion		79,372	New 1991 to 1995 fixed systems hydro 1996 - 2000
Engine installations (new aircraft)	149,197		
APU installations (new aircraft)	27,925		
Cargo installations (new aircraft)	302,764		
If Class D - C		(40,658)	273,780
Freighters E - C		(27,016)	
Commuter Fleet (new aircraft)	39,280		20,158
Testing	13,350		
	720,988	147,046	809,309

4. HALON 1301 CONTAMINANTS AND CONTAMINATION REMOVAL TECHNIQUES

4.1 GENERAL

In order to make an accurate determination of the contaminants likely to be encountered in Halon 1301 recovery processes, samples from ten separate origins of reclaimed Halon 1301 were examined.

Halon 1301 removal from aviation type fire extinguisher containers is accomplished by using the charging port which is part of the fire extinguisher assembly. There are primarily three extinguisher seal designs. Eutectic solder, non-hermetic and hermetic.

Solder types are usually similar to the eutectic "potty bottle" design illustrated as Figure G-10 on page G-8 of Appendix G. Removal of agent from this type of extinguisher design requires that the outlet be placed into a hose clamping assembly and the end of the hose heated, usually by heated air, to the eutectic solder melting temperature. The tube clamp prevents the agent from escaping; however, if the fit is poor due to bending of the tube or other injury, some of the agent charge could escape.

An important point must be made about the "potty bottle" designs. "Potty bottles" do not fall under the category of pressure vessels addressed by FAA Order 8000.40B. "Potty bottles" and similar Halon-only containers, classified as "disposable fire extinguishers", are low pressure containers which do not require hydrotest inspection. The most probable reasons for recovering agent from a "potty bottle" would be physical damage to the bottle or a low, below specification, weight condition.

Non-hermetic fire extinguishers typically use a plug type fitting in the charging port similar to Figures G-5 thru G-8 on pages G-5 and G-6. Some plug type fittings use a Schrader valve where others use a "turn-to-open" valve as illustrated in Figure G-8. A few bottle designs use a check valve arrangement. Unless the fill plug or charging port is damaged, the extinguisher charge may be accessed and removed. In situations where the charge port is damaged or not otherwise accessible it may be possible to mount the extinguisher into a retaining fixture, design an appropriate adaptor, and recover the agent through the main discharge outlet following actuator or squib detonation. This recovery technique is risky and therefore should not be required. The use of main outlet discharge for agent charge recovery should not be necessary in any extinguisher design. The effects of detonation byproducts from squib actuation for the purpose of Halon 1301 recovery are not known, therefore, evaluation of filtration methods for removal of these byproducts has not been performed. It is conceivable that a mechanical puncturing device for recovery of agent through main outlet diaphragms could be designed which would achieve the goal of Halon 1301 recovery without requiring squib actuation.

Charge recovery from a hermetic design is similar to that from a non-hermetic design except the charging port fill plug (usually a stainless steel port and plug are used) is sealed to the lip of the port by a weld. Figure G-1 illustrates a hermetic fill plug and charge port. Recovery of charge involves cutting the weld area away using a port tool supplied by the manufacturer followed by attaching a charge port adapter as illustrated in Figure G-3. Recovery of extinguisher contents is achieved by turning the stem on the adapter which opens the seat between the fill plug and charging port. Again, the use of the main outlet discharge is not necessary.

Four of the ten samples, designated as samples A - D, were obtained from the aviation bank of Halon 1301. One sample each was obtained from the following aviation service centers.

Aerospace International
7115 Northwest 41st Street
Miami, Florida 33166

Casp Aerospace Inc.
103 Gunn Avenue
Pointe Claire, Quebec
Canada H9R-3X2

HRD Aerosystems, Inc.
27811 Hopkins Ave., Suite 4
Valencia, California 91335

C. B. Enterprises
20138 State Road
Cerritos, California 90701

Each of the above aviation service centers uses different Halon 1301 recovery methods. Samples were taken from cylinders used exclusively for storing Halon 1301 recovered from many different fire extinguishers. Therefore, these cylinders, due to blending characteristics of Halon 1301, contain some "average" level of contamination obtained from many different fire extinguisher containers.

Four samples of non-aviation Halon 1301, designated as Samples E - H, were obtained from aged, partially discharged, industrial cylinders. Fenwal and Walter Kidde cylinders supplied one sample each and the other two were taken from Ansul cylinders.

The above eight samples were analyzed for the following by DuPont Laboratories at their Chestnut Run facility in Wilmington, Delaware:

A. Moisture as parts per million by weight
B. High boiling residue (HBR) as percent by volume including oils and other related hydrocarbons
C. Impurities such as:
    FC-12 DiChlorodiFluoromethane
    FC-13 ChlorotriFluoromethane
    FC-22 ChlorodiFluoromethane
    FC-23 TriFluoromethane
    FC-114 1,2-DiChloro-1,1,2,2-tetrafluoroethane
    (Halon 1211) FC-12B1 BromoChlorodiFluoromethane
    (Halon 1202) FC-12B2 DiBromodiFluoromethane
    FC-22B1 BromodiFluoromethane

Analysis of Halon 1301 samples should checked for compliance with Halon 1301 standards contained in Military Specification MIL-M-12218C which is included as Appendix D. Page D-2 contains Table 1 of MIL-M-12218C which lists Halon 1301 requirements.

Key items are purity (99.6 percent minimum), water (moisture) content (10 parts per million maximum), and high boiling residue (HBR), which is usually oil or other similar compounds, is listed on page D-2 as high boiling impurities.

The compounds listed above are referred to as Other Halocarbons on page D-2 of MIL-M-12218C. This is another key item, but only if sample analysis reveals a sum total higher than .4 mole percent, which is 4000 parts per million (ppm). The Halon 1301 manufacturing process requires dealing with several Fluorocarbon (FC) compounds as byproducts. It is not unusual for any one of the listed FC compounds to be present at 700 or 800 ppm as long as the sum total of all FC compounds does not exceed 4000 ppm (.4 percent).

FC-12B1 is Halon 1211 and FC-12B2 is Halon 1202.

TABLE 14. Samples A - D Impurity Test Results

DuPont Analytical Results
WR-9302 (WR - Work Request)
AR-2530 (AR - Analytical Request)
(VP - Vapor Pressure)

Sample No.	VP PSIG 24 Deg C	HBR Percent Vol	Average Moisture PPM
A	207	Trace	75
B	308	0.05	8
C	360	0.10	23
D	202	0.10	20

GC Results in PPM by Peak Area

	Probable Impurities	Sample A	Sample B	Sample C	Sample D
	FC-23	46	97	24	17
	FC-13	56	411	32	40
	FC-22	796	75	248	236
	FC-12	32	---	30	146
	FC-22B1	---	---	<10	<10
(Halon 1211)	FC-12B1	136	---	<10	112,000
(Halon 1202)	FC-12B2	---	---	250	54
(Halon 1301)	FC-1301	99.8%	99.9%	99.9%	88.8%
	Unknowns	151	11	29	65

DuPont analysis of the four non-aviation samples resulted in the following data:

TABLE 15. Samples E - H Impurity Test Results

DuPont Analytical Results
WR-9302 (WR - Work Request)
AR-2621 (AR - Analytical Request)

Sample No.	VP PSIG 24 Deg C	Average Moisture PPM
E	.01	36
F	.01	10
G	<0.01	17
H	<0.01	25

GC Results in PPM by Peak Area

	Probable Impurities	Sample E	Sample F	Sample G	Sample H
	FC-23	17	85	98	72
	FC-13	81	210	24	91
	FC-22	97	48	325	48
	FC-12	116	10	854	376
	FC-22B1	---	---	---	---
(Halon 1211)	FC-12B1	---	---	---	---
(Halon 1202)	FC-12B2	---	---	---	---
(Halon 1301)	FC-1301	99.96%	99.96%	99.85%	99.94%
	Unknowns	79	26	159	8

In addition to the preceding analyses, collective samples were taken from twenty aviation fire extinguishers at the Walter Kidde Aerospace, Inc. Service Center in Wilson, North Carolina. The twenty Halon 1301 fire extinguishers had previously been serviced by six other service centers as identified by means of service date and servicing agency labeling on the fire extinguishers (not including the four service centers that provided reclamation samples.) A total quantity of 150 pounds of Halon 1301 from these 20 aircraft fire bottles from six service centers, designated as Sample I, was transferred into an evacuated cylinder. A second quantity of Halon 1301, designated as Sample J, was obtained from fire extinguisher containers serviced only by the repair station at Walter Kidde Aerospace, Wilson, N.C. Analysis performed by DuPont of these two groups of samples is presented in Table 16.

TABLE 16. Samples I - J Impurity Test Results

DuPont Analytical Results
WR-9302 (WR - Work Request)
AR-2584 (AR - Analytical Request)

Sample No.	VP PSIG 24 Deg C	Average Moisture PPM
I	<.01	22
J	.04	8

GC Results in PPM by Peak Area

	Probable Impurities	Sample I	Sample J
	FC-23	13	6
	FC-13	88	79
	FC-22	60	60
	FC-12	21	19
	FC-22B1	9	10
(Halon 1211)	FC-12B1	10	12
(Halon 1202)	FC-12B2	10	32
(Halon 1301)	FC-1301	99.98%	99.97%
	Unknowns	---	88 Possible FC-114

During the development of a Halon recovery system by ATOCHEM, more than 100 separate containers of Halon 1301 were analyzed. Information supplied by ATOCHEM in a market information release contained the following:

> Average Water Content: 22 PPM. 85 percent of the samples had a water content >10 PPM, 4.5 percent of the samples had a water content > 50 PPM @20^{o} C and 4 percent of the samples contained oil traces. No trace of acidity was found.

Correlation of types and quantities of contaminants found in Halon 1301 samples analyzed by ATOCHEM, in general, agrees closely with similar contaminant levels observed within field samples analyzed by DuPont for this project. The major single significant difference between the data groups was the detected presence of Halon 1211 in Sample D. In fact, ATOCHEM data analysis did not mention any significant levels of foreign halocarbon contamination in any of their samples. Techniques for removal of excessive foreign halocarbon contamination from mistakenly mixing other halocarbon compounds with Halon 1301 are discussed in Section 4.4. Large or small quantities of other halocarbons mixed with Halon 1301 is not detrimental to either compound at normal normal container handling and storage temperatures.

4.2 SOLID PHASE RESIDUE CONTAMINATION REMOVAL

Particulate contamination within Halon 1301 should not be visible to normal, unaided eyesight. MIL-M-12218C (Appendix D, Page D-2) refers to suspended matter or sediment as "none visible". The following discussion addresses controlling suspended matter or sediment within Halon 1301 and cites a typical example of why such controls are important. In the future recycled Halon 1301 will become increasingly prevalent. If installation of extended discharge and long duration inerting suppression systems increase, due to extended range aircraft operation, control of particulate matter in Halon 1301 will become increasingly important to prevent mechanical blockage of the 200 to 300 micron orifices typically used in such systems.

Halon 1301 used in applications for extended discharge inerting should be clean and free of any particulates larger than approximately 20 to 30 microns because of the possibility of bleed orifice blockage in those systems. Some designs for extended discharge within the Walter Kidde Aerospace, Inc., product line utilize a valve-in-bottle, built-in variable pressure regulator feeding an orifice assembly which contains passage diameters of 200 microns. Halon 1301 used for charging this type of fire extinguisher should have no particulates exceeding the 20 to 30 micron size for proper and reliable operation of internal devices.

Insoluble, solid phase, residue contamination found in Halon 1301, during the reclamation process, can be removed by mechanical filtration. In agent reclamation tests for this feasibility study, an aluminum housed bronze element mechanical filter was used. The filter's bronze element used in the tests had a 10 micron nominal rating. Sealing was accomplished using Buna-N rubber O-rings. Working pressure of the referenced filter unit assembly is 3000 psi. The bronze element is easily removed and cleaned by reverse flow methods. The filter assembly, which is a standard assembly, cost $225.00, yet as effective as similar lower working-pressure units which were more expensive. It is available from Fluid Conditioning Products of Lititz, Pa. The part number for the filter assembly is PN 10011-10. For mechanical filtration to the 5 or 2 micron level, the Fluid Conditioning Products part number is PN 10011-05 or PN 1011-02, respectively. For other agents, O-RINGS within the assembly are easily replaced preserving elastomer compatibility.

These mechanical filters can be used anywhere in the Halon recovery flow path; however, filtering at the Halon recovery system input has the advantage of preventing particulates from clogging and damaging valves and other system components. Additionally, filtering agent output from bulk tank storage containers prevents the possibility of charging fire extinguishers with storage tank debris not normally removable by evacuation methods. Locating these mechanical filters following replaceable core type drier stages also prevents possible desiccant debris contamination.

Analytical determination of insoluble solid phase residue can be accomplished using a high pressure filter holder and appropriate filter media. A filter holder incorporating a backing plate and membrane filter elements of cellulose esters material in the 2 micron to 5 micron range, demonstrated good results. The filter membranes are available from Millipore as Catalog No. SSWP 047 00. The membranes were used in a high pressure filter holder obtained from Fisher Scientific as catalog number 09-753. By measuring and recording membrane weight prior to use and again following the transfer of some mass of Halon 1301, a contaminant loading per-pound figure may be determined.

4.3 MOISTURE AND HYDROCARBON (OIL) REMOVAL FROM HALON 1301

Removal of moisture and hydrocarbons from Halon 1301 can be accomplished using standard refrigeration filter-drier units. Both SPORLAN and ALCO inline catch-all type filter-driers and replaceable core type driers were used in this feasibility study to evaluate moisture and hydrocarbon removal capability. When used in a specific process geometry, selected standard refrigeration components demonstrated contamination removal of moisture and hydrocarbons from Halon 1301 to MIL-M-12218C specification in a single pass. Unless severely contaminated, as determined by visual means, the geometry and flow dynamic relationship depicted in Figure 2 will process Halon 1301 to meet MIL-M-12218C requirements of less than 10 parts per million for moisture and less than .5 grams per liter for oils as listed on page D-2 of Appendix D. Standard industrial refrigeration moisture indicators can be used as a guide for excessive loading and excessive water contamination levels. The specific location of the moisture indicators shown in Figure 2 permit replacement of shell cores before the second Drier Stage reaches saturation.

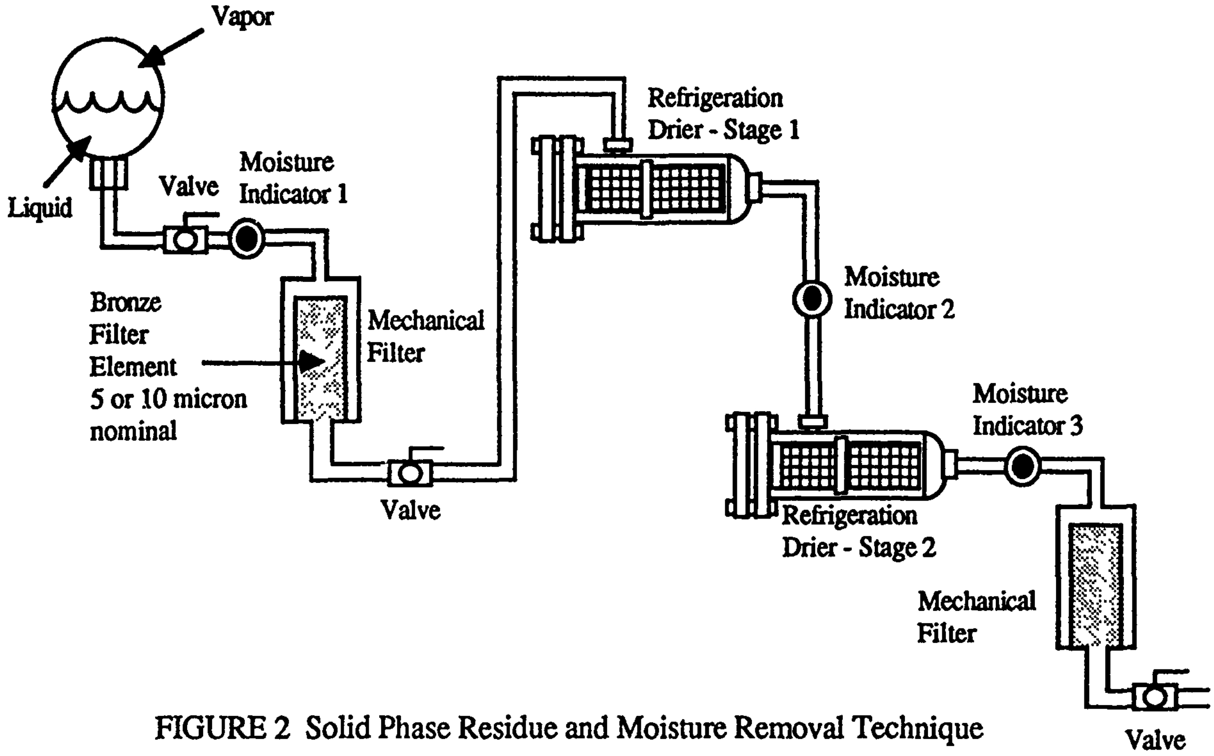
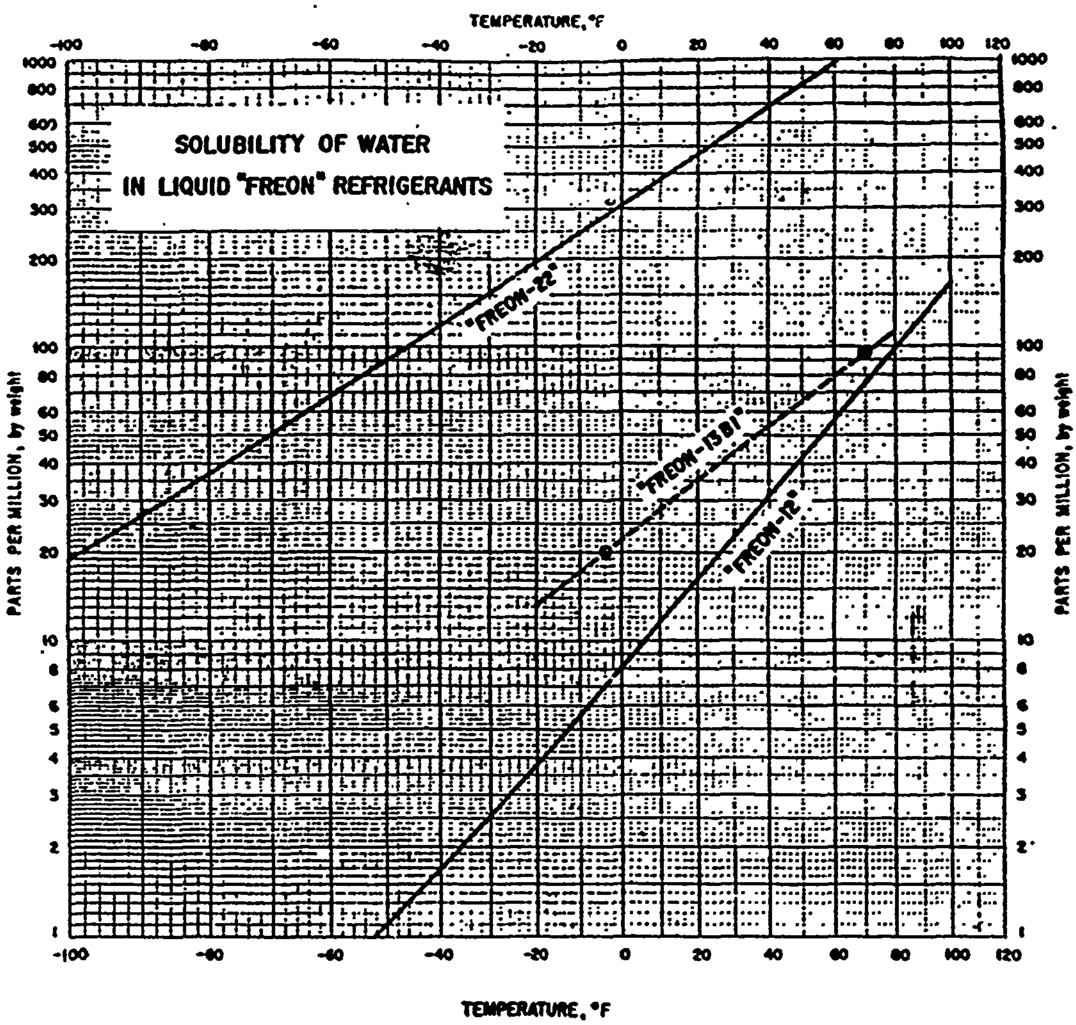


FIGURE 2 Solid Phase Residue and Moisture Removal Technique

Demonstrated results at liquid agent flow rates of 6 to 8 pounds per minute achieved moisture reduction from 8 ppm to 2 ppm and 22 ppm to 4 ppm as detailed in Section 5.6.

4.3.1 DESCRIPTION OF THE DETECTION AND FILTER-DRIER PROCESS

Application technique used within the filter-drier-indication process is critical to both proper system operation and final product purity. Initial mechanical filtration and visual moisture indication (Figure 2) provides for in-line sampling of feed stream product conditions. Using initial, visual sight glass, moisture indicator devices prior to agent conditioning facilitates discontinuation of agent recovery operations whenever severe contamination situations are found to exist. Moisture indicator positioning at the recovery system input is also required because water solubility within refrigerants increases with increasing refrigerant temperature (Halon 1301 is a refrigerant designated as 13B1). Therefore; the warmer the agent flow stream, the higher the required moisture concentration within the stream must be to effect an indication. By placing moisture indicators within the "suction" side of a recovery system the indicator is exposed to temperatures either at or below ambient which facilitates moisture detection at lower water contamination levels. Figure 3, illustrates moisture solubility relationships for several compounds as a function of temperature.



Solubility of Water in Liquid "Freon" Refrigerants.
FIGURE 3

As earlier illustrated in Figure 2, a first stage mechanical filter following initial moisture indication screens out basic solid phase contaminants.. A C-413 filter-drier pair or preferably a replaceable core type, as illustrated, with appropriate core elements performs initial moisture and hydrocarbon removal and follows the mechanical filter. First stage treatment is followed by a second moisture indicator which serves as a safeguard indicating the need to replace desiccant cores.

Agent flow proceeds from first stage treatment to a second moisture indicator and then to a replaceable core type Sporlan C-967 or C-966P or larger second stage containing two RCW-48 elements which further remove moisture. This arrangement also provides for absorbing contamination overflow from first stage treatment without sacrificing recovered agent purity. Second stage treatment is followed by a final moisture indicator and mechanical filter or in-line refrigeration strainer.

Physically positioning all filter-drier elements in a horizontal or slight output end upward attitude increases contaminant removal efficiency due to turbulence and due to vaporization chill effects. Halon 1301 moisture removal ability increases as temperature decreases due to the decreased ability of the refrigerant (Halon 1301) to retain water. Hence, water becomes more available to be adsorbed by desiccating materials. Halon 1301 used in fire extinguishers must not contain more than 10 parts per million water due to the possibility of discharge blockage from ice formation during discharge

4.4 REMOVAL OF FOREIGN HALOCARBONS

Halocarbons whose boiling points are warmer than approximately -55 degrees Centigrade can be removed by fractional distillation methods. Mixtures of Halon 1301, Halon 1211, Halon 1202, and Halon 1011 do not form Azeotropes. These Halocarbons, whose boiling points significantly differ, can be separated by controlled temperature vapor phase evacuation methods.

A pressure vessel containing a mixture of Halon 1301 and Halon 1211, when maintained at temperatures between -10 and -30 degrees C will provide vapor phase Halon 1301 which can be removed from the top of the vessel using a FRICK "BLU-COLD®" vapor recovery unit. If the temperature of the vessel is maintained at -10 degree Centigrade, all of the Halon 1301 can be removed to terminal pressure of 12 psia. Liquid which remains in the lower portion of the vessel will be Halon 1211 or other high boilers which may be transferred to a separate holding tank for further identification by GC or boiling point.

Any non-azeotropic liquid phase mixture of refrigerants can be separated by this manner; however, the closer the boiling point is of the mixed components, the less definitive will be the separation due to contribution of vapor state partial pressures.

4.5 ACID REMOVAL

Acids found within Halon 1301 will be in the aqueous phase, therefore effective moisture removal will also result in effective acid removal. Although Halon 1301 thermally breaks down into hydrogen bromide with minor hydrogen fluoride at 370 degrees Centigrade and free bromine at 390 degrees Centigrade, there is no conceivable rationale which justifies exposing Halon 1301 to this temperature within a pressure vessel. Additionally, there are no known charged pressure vessels used in any aviation applications which could endure this thermal level without thermally relieving through either relief devices, main outlet disc rupture or catastrophically rupturing.

4.6 NITROGEN REMOVAL

Nitrogen used for discharging Halon 1301 over a wide temperature range is a contaminant that should be removed in order to properly charge some fire extinguishers (such as potty bottles) to within specification parameters.

Removal of nitrogen charging gas allows for conditioned agent to be handled in standard shipping containers designed for shipment and storage of Halon 1301. Removal of nitrogen from Halon 1301 also allows the agent to be used for charging aviation fire extinguishers such as "potty bottles" and some cargo fire extinguishers designed for Halon 1301 without nitrogen superpressurization. Fixed gas partial pressure of up to 10 psi in the vapor phase of reclaimed Halon 1301 does not present enough overpressure contribution to require pressure vessel redesign in vessels which previously contained Halon 1301 without nitrogen superpressurization. Efficient recovery equipment is available to achieve better than 99.6 percent pure Halon 1301 at 98 percent recovery efficiency with less than 10 psi vapor phase partial pressure, therefore operation and design of "Halon 1301-only" fire extinguishers can remain unchanged.

The use of cold temperatures to facilitate nitrogen removal takes advantage of two natural properties of Halon 1301. The first takes advantage of Henry's Law which indicates a decrease of solubility of nitrogen in Halon 1301 with decreasing temperature due to resulting pressure decrease. The second takes advantage of the vapor pressure function of Halon 1301 (equation (1)). The vapor pressure function of Halon 1301 reveals a pressure of 13.18 psia at -60° C. At -65° C the vapor pressure is 10.3 psia which indicates that the Halon/Nitrogen mixture could be vented to atmospheric pressure without sacrificing high loss of Halon 1301.

The vapor pressure function of Halon 1301 is expressed as (reference 4):

$$\mathrm{VP1301} = 10^{\left(7.32506 - .00438339(T) - \frac{1795.678}{T} - \frac{44295}{T^2} + .000003125(T^2)\right)} \qquad (1)$$

T in degrees Rankine

VP1301 in psia

Efficiencies associated with Halon 1301 loss during venting of a cold mixture of nitrogen and Halon 1301 is discussed in Section 5 of this report.

4.7 ESTIMATING NITROGEN WEIGHT IN HALON 1301 CYLINDERS

During the course of this feasibility study it became necessary to determine the nitrogen weight within a container where both liquid Halon 1301 and nitrogen were present. Henry's Law (presented in Section 5.2.1 as Equation (4)) models the weight of nitrogen <u>dissolved</u> within Halon 1301 as partial pressure per mole fraction. Henry's Law is valid over a temperature range of from -40 degrees Fahrenheit to +150 degrees Fahrenheit. Henry's relationship is valid provided the container is not at a condition of "liquid full" due to fill density, nitrogen pressurization and temperature.

$$\mathrm{N2w} = \left[0.00148148 \left(\frac{P_t - V_p}{T_r}\right) - 1.94213 \times 10^{-9} \times \left(\frac{C_w}{C_v}\right)^{2.67} \times P_t^{\,3.10} \right] C_v \qquad (2)$$

P_t = measured cylinder pressure in psia

V_p = Vapor Pressure of Halon 1301 (psia) at measured cylinder temperature

T_r = cylinder temperature in degrees Rankine

C_w = net weight of cylinder contents in pounds (Halon 1301 + nitrogen)

C_v = internal cylinder volume in cubic inches

N2w = nitrogen weight in pounds in cylinder

Equation (2) is applicable at nitrogen partial pressures of 1 to 800 psi, a temperature range of 0°F to 130°F, and a fill density up to 75 pounds per cubic foot of Halon 1301.

Fill density (also see Appendix A) is defined as the total weight of agent per unit internal volume of the container. For example, if 150 pounds of Halon 1301 are placed into a 2 cubic foot (internal volume) container, the fill density would be 150 pounds divided by 2 cubic feet or 75 pounds per cubic foot. <u>Total weight</u> of the charging material is considered regardless of its state as liquid or gas. Fill density has no relationship whatever to percent liquid fill unless a specific temperature and nitrogen charge are referenced.

Table 17 compares estimated results obtained using Equation (2), Equation (3) (page 36) and with tables provided by DuPont and results from a DuPont computer program entitled "SUPER".

TABLE 17. Nitrogen Estimate Comparisons

Container Volume (70^oF) Nitrogen Pressure and Halon 1301 Charge	Pounds of Nitrogen in Container Dupont Tables	SUPER Program	N2w Equation	Est N2 Equation
630 cu in., 625 psig, 21 lbs	0.6916	0.6849	0.6730	0.740
2500 cu in, 360 psig, 105 lbs	1.0900	0.9956	1.0200	1.120
60 cu in, 600 psig, 1 lb	0.0649	0.0677	0.0660	0.067
630 cu in, 800 psig, 21 lb	0.9760	0.9670	0.8900	1.050

Table 17 results indicate that the N2w approximation can validly determine nitrogen weight within Halon 1301/nitrogen mixtures. Maximum error using the N2w equation is within eight percent of measured nitrogen charge weight (nitrogen weight listed as DuPont tables) at high fill densities and pressures; however, typical error is less than three percent of measured nitrogen charge weight. Equation (2) was derived from relations of Halon 1301 and nitrogen as modeled by the DuPont "SUPER" program. Conservative adjustments were then made so that fire extinguisher overcharging would be avoided. This was considered necessary since overcharging could cause thermal relief of the fire extinguisher at less than maximum operating temperatures. Equation (2), as can be seen from data in Table 17, is slightly conservative. The intention of the N2w equation is to serve as a Halon 1301 weight adjustment when charging fire extinguishers with Halon 1301 containing nitrogen.

Consider the first entry in Table 17, a 630 cubic inch fire extinguisher charged with 21 pounds Halon 1301 pressurized to 625 psig. According to Equation (2) nitrogen weight is .673 pounds. Hence the Halon 1301 and nitrogen charge total is 21.673 pounds. Since aviation fire extinguishers contain a data charge plate label listing both Halon and nitrogen charge weights, or total charge weight, it is a simple matter of subtraction to calculate full charge specification nitrogen weight within the container.

As an example, consider Halon 1301 supplied from a 400 psig container for charging a 630 cubic inch fire extinguisher requiring 21 pounds Halon 1301 charge weight. A charging procedure for obtaining correct agent charge weight could be as follows:

> Charge the fire extinguisher to a net indicated weight of 21 pounds of Halon 1301 and nitrogen from the 400 psig Halon 1301 source. Close the charging valve and measure extinguisher liquid side temperature and charging line pressure. This step is necessary since the quantity of nitrogen being transported into the fire extinguisher being charged will vary depending upon quantity of Halon in the source cylinder, temperature of the source cylinder (Henry's Law), and whether or not the syphon tube (if so equipped) within the supply bottle begins to encounter vapor phase flow. The relationship between nitrogen contained in the liquid and vapor phases of Halon 1301 is one of mole fractions. Therefore, as liquid Halon 1301 is removed from a nitrogen pressurized cylinder the quantity of nitrogen transferred per pound of Halon decreases. For this example assume that the measured temperature is 75 degrees Fahrenheit and the indicated pressure is 320 psig. Using Equation (2), N2w is calculated as .18 pounds where:
>
> Pt = 320 + 15 = 335 psia
> Vp = 229 psia
> Tr = 460 + 75 = 535 degrees Rankine
> Cw = 21 pounds (charge weight)
> Cv = 630 cubic inches

Hence, to compensate for nitrogen weight contained within the transferred agent, the fire extinguisher should be charged to 21.18 pounds using that particular Halon 1301 source.

After pressurizing the fire extinguisher to 625 psig using additional nitrogen, the total charge weight should be approximately 21.673 pounds.

Equation (3), given below, can also be used for estimating nitrogen content; however, Equation (3) applications are limited to a temperature range of 65 to 75 degrees Fahrenheit and a fill density of 45 to 65 pounds per cubic foot (refer to Appendix A for the definition of fill density). Although the application range of Equation (3) is limited, it is easier to use.

$$\text{Est. N2} = 2.78 \times 10^{-6} (P_t - 199) \times C_v \qquad (3)$$

Where:

P_t = Measured cylinder pressure in psig
C_v = Internal cylinder volume in inches

Equation (3) is derived from converting nitrogen density in Halon 1301 pressure charged vessels between 300 and 600 psi and 60 to 80 degrees Fahrenheit to pounds per cubic inch rather to pounds per cubic foot. The constant 2.78×10^{-6} is an average weight fraction per weight component per cubic inch of nitrogen and Halon 1301 within application parameters.

5. HALON 1301 RECLAMATION - RECOVERY TECHNIQUES

5.1 GENERAL

Because of moisture, nitrogen, mixed halocarbons, hydrocarbons, acids and particulates that may be present in aviation fire extinguishers, the contents of Halon 1301 fire extinguishers should not be directly transferred into shipping type storage containers. In particular, at ambient temperatures of 60 to 80 degrees Fahrenheit, nitrogen partial pressures in some aviation fire extinguisher containers is 625 psi. This produces total container pressures of 825 psi at 70 degrees Fahrenheit. This nitrogen content must be properly and safely dealt with to avoid excessive loss of agent when transferring Halon 1301 from an aviation fire extinguisher into pressure vessel designs of lower test and design pressure.

Older, 4BA400 and 4BW500, Halon 1301 storage vessels contain Superior 1250X packless diaphragm valves equipped with a spring loaded relief valve designed to nominally operate at 450 psig (reference 5). Halon 1301 must not be placed into these older containers if the nitrogen partial pressure at 70 degrees Fahrenheit is greater than 5 psi because these containers, at fill densities (fill density is defined as total weight of agent per unit internal volume of the container, refer to Appendix A) beyond 20 pounds per cubic foot, develop internal pressures of 435 psig at 130 degrees Fahrenheit due to the vapor pressure characteristics of Halon 1301 as a pure substance. Twenty pounds per cubic foot fill density corresponds to 40 pounds in a 150 pound nominal capacity container and 67 pounds in a 250 pound nominal capacity container. These vessels, because of the 450 psig automatic relief operation, must be maintained in a manner that prevents accidental thermal relief or discharge.

Newer, 4BA400 and 4BW500, vessels contain Superior 1250X packless diaphragm valves equipped with a spring loaded relief valve designed to operate at 600 psig. At 70 degrees Fahrenheit Halon 1301 placed into these containers must contain no more than 45 psi nitrogen partial pressure. This applies at any fill density up to the maximum allowable fill of 75 pounds per cubic foot. It also applies to the one ton storage containers 110A800W. The DOT 110A800W one ton bulk cylinders are equipped with two Superior 1031X valves, fusible relief plugs thermally functioning at a nominal 160 degrees Fahrenheit, and spring loaded relief valves designed for a nominal relief of 500 psig (reference 5).

DOT regulated containers used for storage of Halon 1301 may not be charged beyond that condition where it would be possible for the container to become liquid full at or below a temperature of 130 degrees Fahrenheit (reference 6).

Halon 1301 liquid density is a temperature dependant function as described in Appendix C on page C-2. Halon 1301 at -76 degrees Fahrenheit (-60 degrees Centigrade) exhibits a liquid density of 124.98 pounds per cubic foot. This density (volume which a given weight of Halon 1301 occupies) decreases to 97.79 pounds per cubic foot at 70 degrees Fahrenheit (21 degrees Centigrade). As the temperature continues to increase density decreases. At 134 degrees Fahrenheit (56.7 degrees Centigrade) liquid density becomes 75.44 pounds per cubic foot. This demonstrates that 75.44 pounds of Halon 1301 placed into a one cubic foot container at 70 degrees Fahrenheit (21 degrees Centigrade) would occupy approximately 76 percent of the container volume as liquid phase Halon 1301. The vapor space within the container contains vapor phase Halon 1301, therefore all of the 75.44 pounds is not liquid. This is also why 76 percent of the container volume is occupied as liquid and not 77 percent as the ratio of 75.44 divided by 97.79 would indicate. At a temperature of 134 degrees Fahrenheit, however, the entire 1728 cubic inches of the container volume would be occupied by liquid phase Halon 1301. The addition of nitrogen to Halon 1301 decreases its liquid density due to the solubility of nitrogen within liquid phase Halon 1301. This relationship is referred to as Henry's Law and is described in detail in Appendix C.

From the preceding discussion, it can be concluded that attempting to store Halon 1301 containing even moderate levels of nitrogen into the above storage vessel configurations could cause loss of agent in the event of increased storage container temperature.

Definitions for fill density and percent liquid fill as relating to Halons will be found in Appendix A.

Moisture is a second major concern. When Halon 1301 moisture content increases to 90 ppm at 70 degrees Fahrenheit, the corrosive nature and hydrolyzing action of the mixture is significant. This combination causes rapid deterioration of carbon steels used for construction of standard shipping containers for the 4BA, 4BW, 110A800W and 110A3000W classifications. This hydrolyzing action, although present, does not significantly affect stainless steels used in modern aviation fire extinguisher construction. However, moisture level beyond approximately 25 ppm in aviation fire extinguisher systems can mechanically inhibit proper system performance due to freezing and cold temperature Halon Hydrate formation. Halon Hydrate consists of small, solid phase particles which are created by a combination of water, Halon 1301, and low temperature. At cold temperatures of -40 degrees Fahrenheit or below and when sufficient water is present, Halon 1301 forms solid white crystalline particles which vary in size. In a cold discharge stream, these particles can clog small nozzles and small inline filters.

Following is a discussion of recovery techniques for Halon 1301 that have demonstrated acceptable product purification. Acceptable purification is considered to be an output product conforming to those requirements of MIL-M-12218C except for containing a partial pressure up to 10 psi partial pressure of nitrogen or non-condensible dry gas. The requirement of 1.5 percent by volume in the vapor phase for Halon 1301 specified by MIL-M-12218C (Appendix D) reflects a partial pressure of approximately 3.5 psi at 77 degrees Fahrenheit (25 degrees Centigrade) in a container filled to 75 pounds per cubic foot. It is probable that this extremely low fixed gas content was originally intended for refrigeration applications and not fire suppression. Fixed gas content in a refrigeration system seriously reduces the coefficient of performance. However, fire extinguisher containers which specify Halon 1301 without nitrogen are designed to certain material stress parameters at container test pressures determined by temperature and service pressure rating. Fixed gas content up to 10 psi partial pressure at 77 degrees Fahrenheit does not adversely affect the discharge, operation, or stress conditions within "Halon 1301 only" pressure vessels. It is suggested that MIL-M-12218C could be changed so that Halon 1301 with a fixed gas content up to 10 psi partial pressure at 77 degrees Fahrenheit is acceptable for fire suppression operations. This change would result in Halon only fire extinguisher vessels charged to 75 pounds per cubic foot experiencing a pressure of 473 psig at 135 degrees Fahrenheit instead of the 465 psig for Halon 1301 containing 3.5 psi partial pressure fixed gas content. At a temperature of 200 degrees Fahrenheit a pressure of 1461 psig is developed in a Halon only vessel whereas 1493 psig is developed if a 10 psi partial pressure content at 77 degrees Fahrenheit is considered. These pressure increases are well within design safety margins for "Halon 1301 only" fire extinguishers.

Section 5.2 addresses basic pressure transfer as a recovery means for Halon 1301. This method should not be used to recover Halon 1301 or any other agent because recovery efficiencies are typically less than 70 percent. Contaminants at levels which are intolerable can also be transferred in an unconditioned transfer stream. In addition to the preceding negative effects are those associated with possible transfer hose moisture which can add to moisture already present.

Section 5.3 addresses loss of Halon 1301 associated with near ambient temperature venting as an attempt to remove excess nitrogen for the purpose of storing into lower pressure vessels. Halon 1301 loss associated with this method can exceed 50 percent.

Section 5.4 addresses costs associated with a pressure transfer treatment system to purify Halon 1301 as it is transferred from a larger to a smaller pressure vessel. When Halon 1301 or any Halon agent MUST either be transferred or lost to the atmosphere and it cannot be recovered by any other means, it is best to use a method which provides some level of agent cleanup. The method presented in Section 5.4 is key to a successful, cost effective and efficient Halon recovery when used with specific refrigeration techniques.

Section 5.5 describes a system allowing greater than 98 percent recovery efficiency while maintaining Halon output purity conforming to MIL-M-12218C (Appendix D) standards except for 2 to 10 psi nitrogen partial pressure content in the vapor phase.

5.2 RECLAMATION OF HALON 1301 UTILIZING BASIC PRESSURE TRANSFER

The contents of most aviation fire extinguisher bottles containing Halon 1301 fire extinguishant cannot be transferred directly into storage containers normally used for bulk storage of Halon 1301. Some aviation fire extinguisher bottles contain nitrogen partial pressure levels of 625 psi at 70 degrees Fahrenheit which results in a total pressure of approximately 825 psig. Some, or preferably, all nitrogen must be removed either during an agent recovery process or prior to agent storage. Additionally, because an accurate agent charge weight must be known when charging aviation fire extinguisher bottles, it is preferred that as much nitrogen as possible be removed in order to eliminate compensating for errors associated with Halon-Nitrogen mixture charging.

Discussion of approximating nitrogen content within a vessel containing Halon 1301 and charging with Halon 1301/nitrogen mixture is presented in Section 4.8. Equations describing these physical properties are in Appendix C.

Discussion of nitrogen solubility mechanisms within liquid Halon 1301 as effected by nitrogen super pressurization is discussed later in this section.

Pressure transfer methods for Halon 1301 reclamation can be performed using many techniques. Because Halon 1301 is a stable compound it is conceivable to directly transfer contents between fire extinguisher containers since long term storage, under proper housekeeping conditions, is not detrimental to the compound. However, charging fire extinguisher containers by this method could result in significant loss of agent. Transferring agent from a charged extinguisher into an empty, evacuated extinguisher is not recommended and should not be practiced. If the direct transfer method must be performed, the receiving fire extinguisher should be evacuated prior to charging by vacuum pump to 1.0 psi or less in order to minimize moisture accumulation and agent loss.

Figure 2 illustrated a method for using basic pressure transfer techniques and obtaining "conditioned" Halon 1301 which, except for non-condensible dry gases, should meet the requirements of MIL-M-12218C (Appendix D) unless the agent is highly contaminated. The refrigeration components comprising the parts list have a working pressure rating of 500 psi. The design minimum burst pressure of the core shell is above 2000 psi. These components should be examined internally whenever replaceable cores are changed. These components can withstand transient pressures to the 825 to 900 psi levels required by some aviation fire extinguishers; however, by designing a basic pressure transfer system with an empty, small tank (630 cubic inch suggested), located between the mechanical filter and first drier stage to act as a ballast, pressures within the conditioning system will not exceed transient pressures of 600 psi. The valves may be packless type needle valves. Valves may contain elastomers for packing seals. Halon compatible materials such as PEEK^{TM}, BUNA-N, NITRILE and NEOPRENE. TEFLON will work; however, with long term exposure the valve will become difficult to actuate due to swelling of the Teflon seals. This swell effect will decrease during periods of non-use.

Attempted recovery of Halon 1301 from aviation type fire extinguisher designs by pressure transfer methods into storage, shipping or bulk tanks cannot be performed without nitrogen venting. This venting will result in approximately 40 percent or greater agent loss depending on temperature. Standard Halon 1301 storage tanks or shipping vessels must NOT contain a nitrogen partial pressure component beyond 45 psi in the vapor phase at 70 degrees Fahrenheit at 75 pounds per cubic foot fill density. Older shipping containers using 450 psi relief valves must NOT contain a nitrogen partial pressure component beyond 5 psi in the vapor phase at 70 degrees Fahrenheit and 75 pounds per cubic foot fill density. Nitrogen content beyond those partial pressures will cause relief devices on the respective containers to actuate at approximately 130 degrees Fahrenheit.

Approximately 0.40 pounds (calculated from Equation N2W, Appendix C) nitrogen weight is required to pressurize 150 pounds Halon 1301 contained within a 2 cubic foot shipping container (4BW500) to 245 psig. One 630 cubic inch aviation fire extinguisher containing 22 pounds Halon 1301 pressurized to 625 psig at 70 degrees Fahrenheit contains .68 pounds nitrogen. Upon using pressure transfer methods to transfer liquid contents from a 630 cubic inch fire extinguisher to a 4BA or 4BW vessel design, approximately .35 pounds nitrogen will be transferred with the liquid. Available liquid phase Halon 1301 from a typical 630 cubic inch spherical aviation fire extinguisher at 70 degrees Fahrenheit is nominally 18.5 pounds. Without venting excess nitrogen, only ONE 630 cubic inch fire extinguisher can be recovered at 88 percent efficiency into a standard 150 pound shipping/storage Halon 1301 cylinder. Any attempt to transfer additional nitrogen charged Halon 1301 into the cylinder can result in loss of agent should the container become excessively warm or be stored in a 120 to 130 degree Fahrenheit ambient temperature. Pressure within any Halon 1301 container is characterized by Halon 1301 vapor pressure, fill density and nitrogen partial pressure which is non-condensible gas content. Additionally, a pressure vessel is only EMPTY if it is evacuated to 0.00 psia which is not realistic in practice. A pressure vessel containing a pressure of 1.0 psia of other than Halon 1301 molecules will result in an additional partial pressure of approximately 1.67 psi at 70 degrees Fahrenheit when charged to 75 pounds per cubic foot fill density.

Container pressure indication by gauge measurement at a specific measured temperature beyond the calculated vapor pressure of Halon 1301, less 14.7 due to atmospheric reference, is usually attributed to that pressure from non-condensible gases. Helium and nitrogen are two non-condensible gases used for charging Halon 1301 fire extinguishers. Helium is used for leak detection purposes in hermetic container designs and is NOT soluble in liquid phase Halon 1301. However, nitrogen is soluble in liquid phase Halon 1301 according to Henry's Law. Henry's Law, presented below as HX, is a function of temperature in degrees Fahrenheit. Henry's function represents nitrogen as psi per mole fraction contained within the Halon 1301 liquid portion in a charged fire extinguisher.

$$HX = 4763.868266 + 3.94643566 \times T - 0.0162423503 \times T^2 - .00044459 \times T^3 \qquad (4)$$

where T is in degrees Fahrenheit. Solving the equation at a temperature of 70 degrees Fahrenheit results in a figure of 4808.037 with units expressed in psi per mole fraction (reference 4).

This expression is correctly interpreted as follows: With both components maintained at a temperature of 70 degrees Fahrenheit, nitrogen pressure required to cause one pound mole (28 pounds mass) of Nitrogen to be contained within the liquid phase of one pound mole of Halon 1301 (148.9 pounds mass) is a nitrogen partial pressure figure of 4,808 psi beyond that of the natural vapor pressure of Halon 1301.

Henry's Law explains why nitrogen is transferred with liquid phase Halon 1301 during pressure transfer from one charged container to another. Calculation of the exact quantity of nitrogen which is contained within the transfer stream depends on fill density, temperature, agitation during transfer, and nitrogen partial pressure components. This calculation involves partial fraction relationships based on mole fraction quantities of the mixture involved. It is best performed by computer methods.

Recovery of Halon 1301 at ambient temperatures from charged fire extinguishers is best accomplished by direct transfer into other fire extinguisher assemblies through an equipment arrangement similar to that shown in Figure 2. Relationships exist which make this a practical solution. Where a Halon 1301 recovery system is not available, this method is far better than attempting to perform agent transfer into a vessel which requires ambient venting in order to obtain a liquid charge.

5.3 LOSS OF HALON 1301 THROUGH ATMOSPHERIC VENTING

Use of standard shipping or industrial containers requires venting Halon 1301 below 245 or 360 psig as applicable, prior to transfer of agent into storage. Table 18, below, tabulates losses associated with ambient temperature venting of a nitrogen charged fire extinguisher or container.

TABLE 18. Halon 1301 Losses Associated with Ambient Temperature Venting

Initial Pressure: 625 psig
Initial Temperature: 70°F Average (58°F to 85°F)

Final Pressure psig	Percent Loss
625	0.0
600	1.0
500	5.2
400	10.3
300	16.6
250	24.9
200	36.0

The above figures were obtained from venting numerous bottles of various agent fill densities and geometries. They represent average losses when venting from the vapor side to achieve nitrogen removal. Any particular situation could have up to 10 percent additional loss. In some cases in the above examples, because of chilling from the venting procedure, the bottle internal pressure increased as much as 30 psig above the final indicated internal pressure. Halon 1301 losses can be reduced if the bottle is first chilled prior to venting.

Table 19 demonstrates results obtained when a typical fire extinguisher was vented at a vessel temperature of -80°F

TABLE 19. Agent Loss While Venting Containers at -80°F

Initial Pressure: 625 psig
Initial Temperature: 70°F Average (58°F to 85°F)
Chill Pressure: 290 psig
Chill Temperature: -80°F

Final Pressure psig	Percent Loss
290	0
200	1
100	2
000	4

When the fire extinguisher was returned to 71°F the internal pressure was measured to be 210 psig, which is clear indication that chilling the agent is a significant factor in achieving a high level of recovery efficiency due to reduced Halon 1301 vapor pressure.

5.4 COSTS ASSOCIATED WITH PRESSURE TRANSFER RECOVERY TECHNIQUES

The treatment method for Halon 1301 illustrated in Figure 2 can be used in direct liquid transfer and recovery which permits ambient temperature venting to be avoided.

Cost breakdown for the items used in Basic Transfer System depicted in Figure 2 is:

TABLE 20. Basic Transfer System Cost Breakdown

Valves (Needle or Ball) If Ball Parker SA-B6LPKC-BN-SSP is suggested which is stainless steel with 3/8 tubing lok fittings	$195.00
Sporlan C-966P or C-967 Shell (2 required)	$220.00
Fluid Conditioning Products PN 10011-5 Mechanical 5 micron filter (2 suggested)	$500.00
Gauge 0 - 600 psig	$17.50
Sporlan SA-13 Moisture Indicator (3 suggested)	$24.00
Miscellaneous other Plumbing 3/8 SS tubing, fittings and mounting hardware	$225.00

Costs listed above for construction of the Figure 2 Solid Phase Residue and Moisture Removal System represent typical investment. Actual cost (except for mechanical filters) may be approximately 5 percent to 10 percent less depending on area and quantity. Typical construction cost sums to $1,181.50. Labor cost for mounting, based on $45.00 per hour for 30 hours is $1,350.00.

The configuration of Figure 2 will process approximately 6,000 pounds Halon 1301 (or Halon 1211) with an average system input water content of approximately 30 ppm and deliver Halon with less than 10 ppm Halon output stream water content. Replacement parts to renew system performance consist of four RCW-48 elements which could cost $70.00 System service time, including replacing cores, inspection of shell interior and cleaning mechanical elements is approximately 80 minutes. These averages predict a service cost (based on labor + burden rates of $45.00 per hour) of $70.00 + $60.00 or $130.00. This results in a process cost (based on 6000 pound Halon throughput) of approximately $0.02167 (2.167 cents) per pound of Halon. Based on estimated system component service life (other than RCW-48 Cores) of 250,000 pounds Halon, replacement time of 55.6 hours at $45.00 per hour, construction labor hours (30) at $1,350.00 and initial investment of $1,251.50, per-pound estimated life operating cost is $0.02167 (consumables) + $0.0154 (replacement parts and labor) + $0.0100 (initial cost) which yields a process cost based on 250,000 pound transfer lifetime of $.0471 (4.71 cents) per pound. This amount, plus bottle service time, is the cost involved to remove water and particulate debris from Halon 1301 (Halon 1211 and 1202 included) while transferring the contents into a second vessel or into a Halon recovery system. This cost does not include labor to recover the Halon, which is the major cost.

Purity and quality verification remains to be performed. Laboratory verification equipment for this consists of a gas chromatograph, a Karl Fischer titrator and related accessory equipment which total approximately $20,000.00. Because analysis of one sample requires approximately one hour, it is advantageous to sample on a bulk basis.

A unique, although somewhat labor intensive, Halon recovery system used by Canadian Airlines International is presented in Appendix B. The cost to implement this recovery system which achieves 89 percent to 92 percent recovery efficiency is approximately $2000.00

5.5 ACHIEVING HIGH EFFICIENCY HALON 1301 RECLAMATION INCLUDING REDUCTION IN FIXED GAS NITROGEN CONTENT

A Halon recovery system has been developed outside the work of this feasibility study which utilizes components of Figure 2 and refrigeration treatment for fixed gas removal. This operating and fully functional Halon recovery system shown below in general block diagram form as Figure 4 achieves better than 98 percent recovery efficiency and delivers, at its output, Halon 1301 conforming to MIL-M-12218C except for approximately 2 to 10 psi nitrogen partial pressure in the vapor phase.

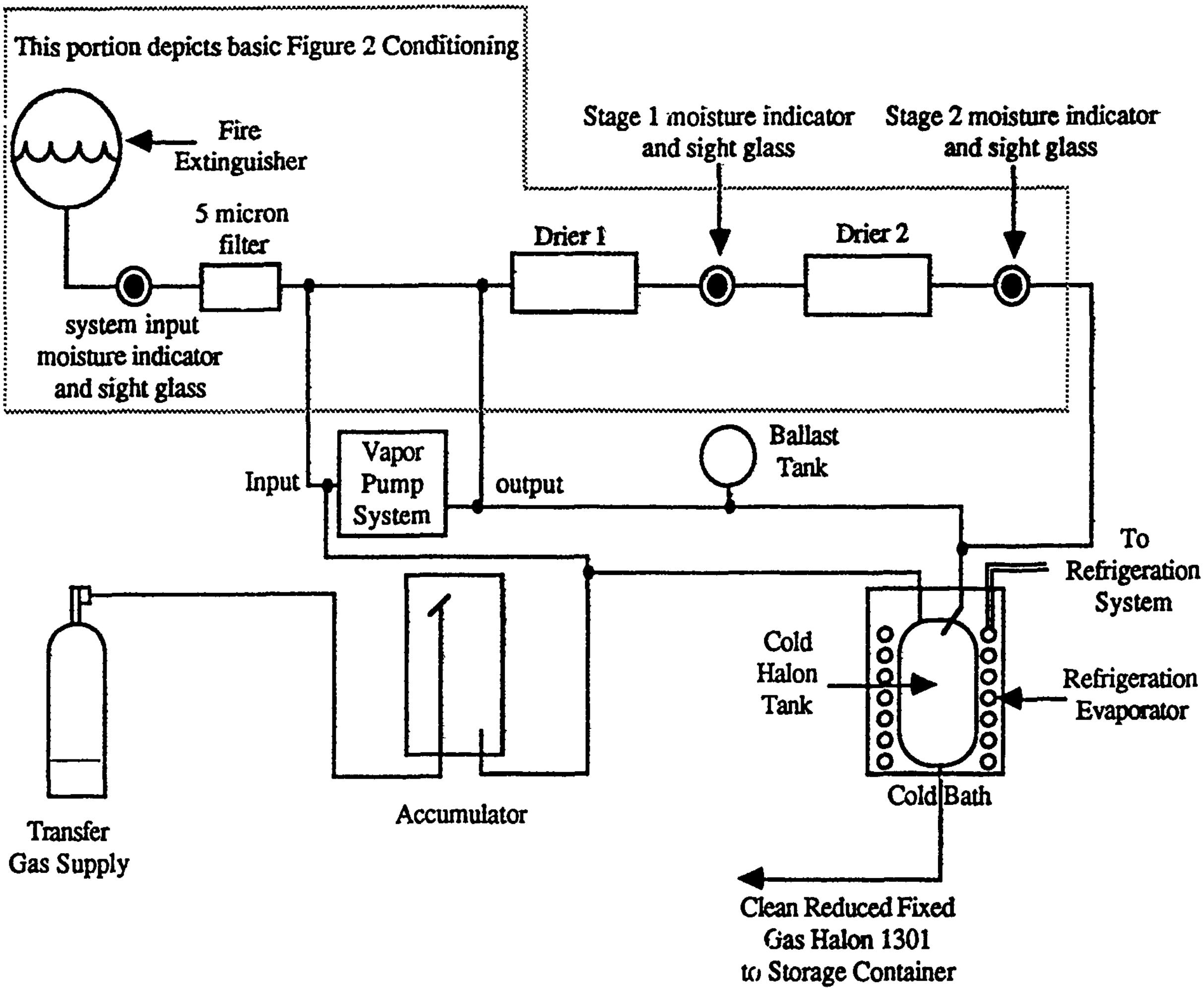


FIGURE 4. General Flow Diagram of REACH® High Efficiency Halon Recovery System Developed by Walter Kidde Aerospace

A unique refrigeration system has been developed which operates at approximately -80 degrees Fahrenheit to allow non-condensible fixed gases to be removed from Halon 1301 with less than 2 percent agent loss. This high recovery efficiency is possible because Halon at -80 degrees Fahrenheit exhibits a vapor pressure of 11.8 psia which is 2.9 psi below atmospheric pressure. The significant component is that of Dalton's Law (refer to Appendix A, page A-1) which describes how gases combine as partial pressures when mixed to a total pressure. The inverse of this physical relationship is also valid. When two gases leak from a mixture they leak in proportion to their molecular weights and individual partial pressures as well as their specific volumes and temperature relationships. Therefore, where the partial pressure of Halon is 8 psi and the partial pressure of nitrogen is 250 psi, the nitrogen will leave the vessel at a significantly increased rate over that of the Halon 1301 vapor.

Figure 4 will next be described in six basic steps initially assuming that the entire system has been evacuated and the refrigeration system is operating at approximately -80 degrees Fahrenheit (-64 degrees Centigrade).

First, a fire extinguisher is connected to the input in a liquid side down configuration. The charge fitting is opened which allows the agent (liquid phase) to flow through all components previously described in the Figure 2 configuration and then into the cold tank as shown in Figure 4. This flow takes place at a rate of approximately 8 liquid pounds per minute beginning at approximately 600 psig and gradually decreasing to approximately 300 psig which is the end of liquid phase flow.

Second, at approximately 250 psig a vapor recovery pump system is switched on which evacuates the fire extinguisher to approximately 4 psia. This action removes approximately 99.4 percent of the entire contents of the fire extinguisher including Halon 1301 with all contaminants including nitrogen. All contents are then pumped through the filter system into the cold tank. As the contents enter the cold tank a pressure accumulator permits bleed over in order to prevent excessive pressure build up due to fixed gas content. The fire extinguisher is then replaced with another and the process repeated until the nominal 150 pound liquid capacity of the cold tank is reached.

Third, at liquid capacity in the cold tank and at a temperature of at least -67 degrees Fahrenheit (-55 degrees Centigrade) the accumulator is sealed off from the cold tank and the cold tank is vented to approximately 40 psig. At this pressure the vapor pumping system pumps vapor from the cold tank into a smaller ballast tank until pressure within the cold tank has reached approximately 13 psia. This further removes fixed gases with less than 1.5 percent agent loss since the significant vapor content is pumped into a second storage vessel.

Fourth, upon reaching approximately 13 psia in the cold tank, which contains approximately 150 pounds of clean and gas free Halon 1301, the vapor side of the tank is slowly pressurized from the small ballast and from the accumulator. Additional nitrogen or other transfer gas is added from the purge side of the accumulator which pushes out the heavier Halon vapor into the cold tank under pressure. This serves to condense out additional Halon 1301 which had previously escaped into the accumulator during recovery. As long as the cold tank is not mechanically shaken or otherwise agitated, very little nitrogen will enter into solution with the Halon 1301 because of decreased solubility and saturation of the immediate top layer of Halon which does not get transferred out of the cold tank. Transfer gas pressure is between 300 to 325 psig. Internal tank contouring assists in preventing vortex motion effects which could cause immediate adsorption of nitrogen into liquid phase Halon 1301.

Fifth, upon achieving an appropriate low level condition all transfer process valves are closed and the cold tank vented of transfer gas to its initial low pressure state.

Sixth, the recovery system resets itself and the cycle is ready to begin again.

Halon tested from the output of this system met all requirements of MIL-M-12218C except for a very small residual partial pressure of Nitrogen as discussed above. Samples analyzed by DuPont indicated mole purity of 99.9+ percent and moisture level of 5 ppm. This Halon 1301 was equivalent to virgin manufactured Halon in purity and well below the MIL-M-12218C moisture criteria of 10 ppm. Halon entering the system contained typical moisture levels of 22 ppm and traces of hydrocarbons (oils).

5.6 RESULTS OF SYSTEM TESTS FOR PURIFICATION

Halon 1301 samples taken from twenty fire extinguishers at Walter Kidde Aerospace, Inc. Service Center, I and J (Table 16, page 29) were processed through the Halon Recovery System described above. Each sample, after recovery, was respectively designated as K and L. Samples K and L were analyzed by DuPont to determine contaminant levels. The results are presented in Table 21, which also includes Table 16 data for comparison purposes. Liquid flow through the Halon

Recovery System components was eight pounds per minute, and, for th's particular sampling, mechanical filtration was not utilized.

TABLE 21. Samples I - L Impurity Test Results

DuPont Analytical Results
WR-9302 (WR - Work Request)
AR-2584 (AR - Analytical Request)

Sample No.	HBR Percent Vol	Average Moisture PPM	
I	<.01	22	input to recovery system
K	<.01	04	output from recovery system
J	0.04	08	input from recovery system
L	0.04	02	output from recovery system

GC Results in PPM by Peak Area

	Probable Impurities	Sample I	Sample K	Sample J	Sample L
	FC-23	13	9	6	4
	FC-13	88	85	79	74
	FC-22	60	50	60	60
	FC-12	21	20	19	21
	FC-22B1	9	9	10	10
(Halon 1211)	FC-12B1	10	10	12	12
(Halon 1202)	FC-12B2	10	10	32	30
(Halon 1301)	FC-1301	99.98%	99.98%	99.97%	99.97%
	Unknowns	---	---	88	79 probable FC-114

It is significant to note the following with regard to the data in Table 21:

- No mechanical filtration was used in order to evaluate raw effects of the refrigeration components.
- Data in Table 16 represents system performance following the processing of 500 pounds of Halon 1301 from field returned fire extinguishers.
- Data in Table 16 demonstrated significant moisture removal without quality deterioration of the agent. Moisture levels were reduced in Sample I from 22 ppm to 4 ppm, significantly within the requirements of MIL-M-12218C.
- GC results indicated significant stability of the treatment system.
- Nitrogen partial pressure of Sample I was measured as 135 psi. Nitrogen partial pressure of Sample K was measured as 2 psi at 70 degrees Fahrenheit.
- Nitrogen partial pressure of samples J and L was 396 psi and 2 psi respectively
- Overall recovery efficiency of both samples was 96 percentas defined by physical science relationships of mass conservation.

5.7 FINANCIAL OUTLOOK - RECLAMATION TO MIL-M-12218C vs COST AND SUPPLY OF NEW MANUFACTURED HALON 1301

Cost of commercially available Halon 1301 reclamation systems vary, at the time of this study, from approximately $3,000.00 to $250,000.00. This extreme variation is due to philosophy of design and ability of designed equipment to process Halon 1301, at some given rate, to some purity level, at various non-condensible gas partial pressure content at some efficiency level. Halon recovery system manufacturers and those actively pursuing Halon recovery designs include TEAM Aer Lingus, Dublin, Ireland; Walter Kidde Aerospace, Inc., Wilson, North Carolina, ATOCHEM, located near Paris, France; HTL Division of Pacific Scientific, Duarte, California; Applied Ecological Systems, Hazelhurst, Georgia; and a Germany company named Vulcan.

Aircraft fire suppression systems utilizing Halon 1301 require agent purity to maintain known physical properties which existed at the time of aircraft certification. It is the physical properties of Halon 1301, in most applications combined with nitrogen, which provides an effective fire suppressing action at extreme conditions demanded in aircraft service. Aircraft designs are, or in most cases were, certified using KNOWN agent quality conforming to MIL-M-12218C. Concentration measurements were calibrated against known mole purity Halon 1301. As previously discussed, some fire suppression designs utilize extended discharge valve systems. Any change in mass flow rate due to out-of-spec Halon 1301 can cause loss of agent concentration from discharge freeze-up because of excess moisture or change in boiling point. In addition, today's extended range operation (EROPS) aircraft designs make it most imperative that all Halon be tested and certified by proven methods to meet applicable requirements of military standards prior to use in aircraft fire extinguisher installations. This should also be the requirement for Halon used in inhabited spaces since toxic contaminants may accumulate in unclean Halon.

As of July 1991, a mini-bulk quantity purchase of Halon 1301 (approximately 12,000 pounds) cost approximately $2.05 per pound plus an excise tax of $0.2466 for a total cost of $2.2966 per pound. The excise tax increases to $0.2505 per pound in 1992 and $0.2650 per pound in 1993 according to the OMNIBUS BUDGET RECONCILIATION ACT OF 1989.

This act imposes new taxes on Ozone depleting chemicals. The effect this act has on Halon 1211 and Halon 1301 is presented below in Table 22.

TABLE 22. Tax Imposed Per Pound Of Material Purchased - U.S. Dollars

YEAR	92	93	94	95	96	97	98	99
HALON 1211	0.25	0.25	7.95	9.30	10.65	12.00	13.35	14.70
HALON 1301	0.25	0.25	26.50	31.00	35.50	40.00	44.50	49.00

For this cost example a purchase cost of $100,000.00 is assumed for a Halon recovery system. For analytical equipment, including training, a figure of $20,000.00 is assumed. In consideration of a quantity of 250,000 pounds, removal of moisture, particulates, foreign hydrocarbons and other contaminants will cost approximately $0.0471 per pound for the treatment system which includes replacement cores and maintenance.

Vapor recovery pumps require weekly checks for crankcase oil level. Vapor recovery pumps and filter/treatment elements are the only items requiring frequent service. Refrigeration system components are generally service free. Cost analysis is based on 10 year book, straight-line depreciation, which is typical for equipment purchases of this amount. Analytical equipment maintenance is also included.

Initial investment :	$100,000.00 Halon Recovery System $20,000.00 Lab Equipment
Electrical Consumption: (Operating)	Assume 8 kw per hour worst case for machine and options, if installed.
(Standby mode at -25°C):	Assume 1 kw per hour.
Annual operating hours:	2000 Hence an overall average throughput of 125 pounds per hour based upon an annual recovery of 250,000 pounds. This is average considering bottle handling and cool down time.

Annual cost of filter-drier components and maintenance to a treatment system: ($0.0471 per pound for 250,000 pounds) (based on 6,000 pound drier capacity.)	$11,775.00
Annual cost of VRU operation maintenance and estimated Preventive Maintenance service at 0.5 hour per week at $45.00 per hour.	$1,170.00
Electrical energy system operating cost:	
2080 hours at 8kw per hour at $0.07 per kw	$1,164.80
6686 hours at 1kw per hour at $0.07 per kw	$468.02
Annual Halon Recovery System depreciation	$10,000.00
Annual Lab Equipment depreciation	$2,000.00
Annual Lab Equipment calibration and service	$1,000.00
Heat exchange solution annual replacement, maintenance checks for specific gravity shift (.25 hour per week, 20 gal fluid at $100.00)	$685.00
	$28,262.82
Agent recovery from each bottle is automatic. Recovery system operation typically switches off upon completing evacuation of fire extinguisher. Small bottles, however,of up to 8 pound agent charge will be recovered in about 2 minutes which does not permit significant dedication of work time to other tasks. Labor is estimated at 1040 hours annually for bottle handling as related to recovery system usage. (1040 @ $45.00/hr.) The estimated annual cost per pound is $0.3003 for an installation recovering 250,000 pounds per year.	$46,800.00 $75,082.82

For an installation recovering 12,000 pounds per year, process cost per pound is as follows:

Annual cost of Filter Drier components and maintenance to treatment system: ($0.0471 per pound for 12,000 pounds) (based on 6,000 pound drier capacity).	$565.20
Annual cost of VRU operation maintenance and estimated Preventive Maintenance service at 0.3 hour per week at $45,00 per hour.	$702.00

Electrical energy system operating cost:	
2080 hours at 8kw per hour at $0.07 per kw	$1,164.80
6686 hours at 1kw per hour at $0.07 per kw	$468.02
Annual Halon Recovery System depreciation	$10,000.00
Annual Lab Equipment depreciation	$2,000.00
Annual Lab Equipment calibration and service	$1,000.00
Heat exchange solution annual replacement, maintenance checks for specific gravity shift (.25 hour per week, 20 gal fluid at $100.00)	$685.00
	$16,585.02
Agent recovery from each bottle is automatic. Recovery system operation typically switches off upon completing evacuation of fire extinguisher. Small bottles, however,of up to 8 pound agent charge will be recovered in about 2 minutes which does not permit significant dedication of work time to other tasks. Labor is estimated at 500 hours annually for bottle handling as related to recovery system usage. (500 @ $45.00/hr.) The estimated annual cost per pound is $3.257.	$22,500.00
	$39,085.02

As can be seen from these two models, it is not yet cost effective for small volume users of Halon 1301 or other agents to operate a Halon recovery system of this investment amount. If Halon 1301 rises in cost at 10 percent per year, the 1992 cost (including excise tax) could be $2.5016 per pound and the 1993 cost could be $2.7271 per pound. In 1994 the cost could be $29.2285 per pound. The 1994 virgin manufactured Halon 1301 cost is far above any reasonable recovery cost.

Figure 5 illustrates recovery cost, based upon the above example, as dollars per pound versus pounds per year recovered. Figure 5 also illustrates projected cost of virgin manufactured Halon per year in dollars per pound versus manufacturing year.

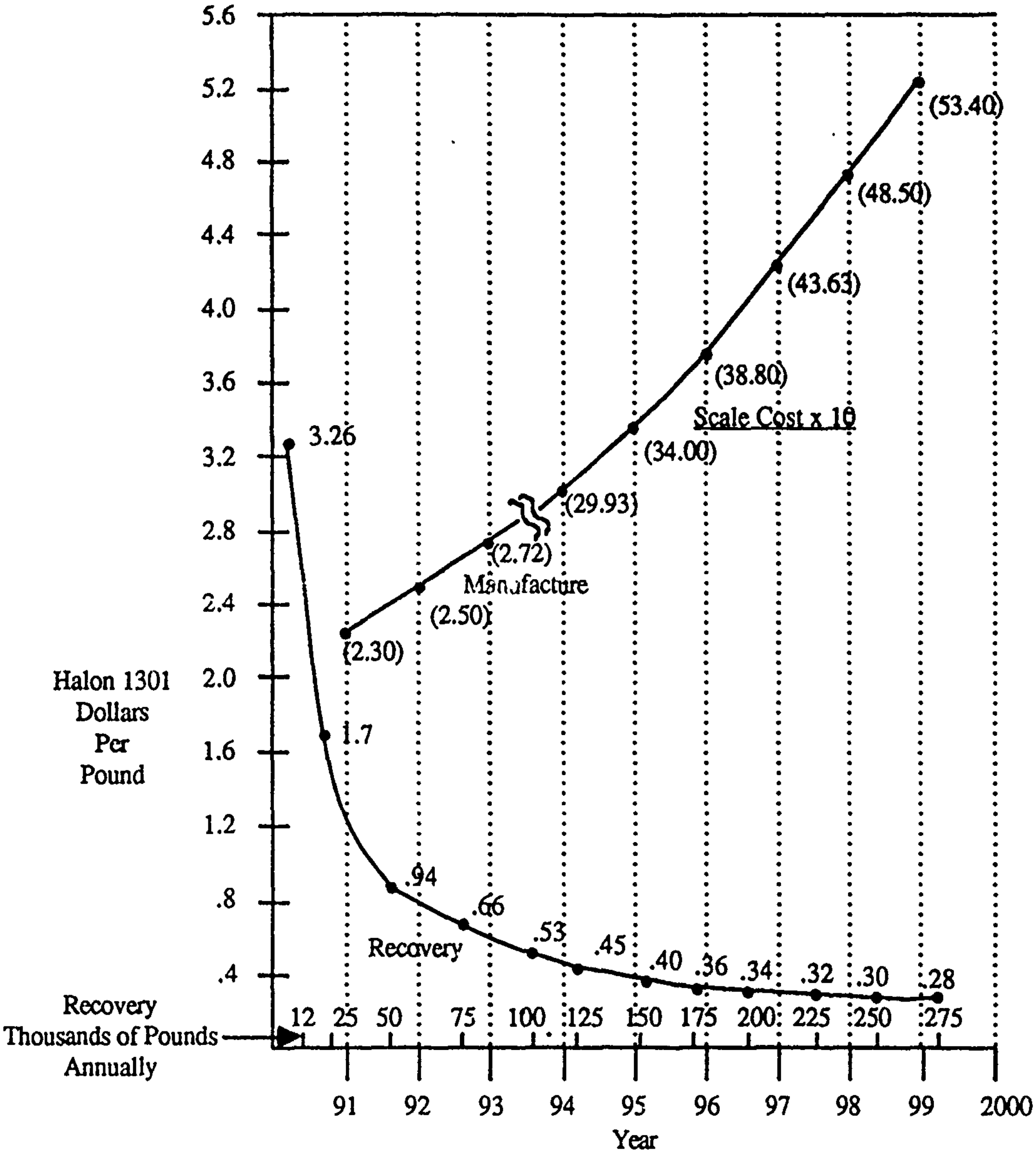


FIGURE 5. Comparison of Recovery vs Manufacturing Costs of Halon 1301 into the Future

5.8 LEVEL III RECOVERY - COMPLETE RE-REFINING HALON 1301

Complete re-refining of Halon to a virgin Halon 1301 state requires a system capable of vaporization, distillation, and filtration of Halon 1301 as well as total fixed gas removal which would require extremely cold temperatures, evacuation and pumping. This system would be prohibitively expensive to build and maintain. Since equipment is available at a relatively modest cost which can process Halon 1301 to military specification purity levels except for 2 to 10 psi fixed gas partial pressure, it is unreasonable to construct a re-refining system.

Halon 1301 does not form azeotropic mixtures with contaminants which are likely to be encountered in civil aviation fire extinguisher containers. When mixed with FC32 Halon 1301 does form an azeotrope at approximately 20 percent FC32. However, FC32 is not currently manufactured and has not been produced for approximately 15 years. Some Halon 1301 systems use methanol (methyl alcohol) for moisture control in fire extinguishers. This is removable by carbon and sieve techniques. If methanol is found in a sample of Halon 1301 such that the 99.6 mole purity condition is not satisfied, that Halon 1301 can be blended with 99.8 or 99.9 mole percent Halon 1301 to conform to military specification requirements.

This study found no Halon 1301 contaminants which could not be removed using standard refrigeration filtration core media such as carbon or alumina-molecular sieve. Excessive contaminants could conceivably pass through one of the refrigeration type conditioners; however, a second treatment using "renewed" cores would produce clean Halon.

Severely rusty or heavy particulate loading with oils or other mixed Halons can also be handled with the following proper treatment sequence:

First, remove particulates with a 5 or 10 micron element as discussed in Section 4.2.

Second, Filter the Halon through carbon core filters as discussed in Section 4.3 for oil removal.

Third, filter the Halon through water adsorbing core filters as discussed in Section 4.3.

Fourth, pass the Halon mixture into a Halon recovery system capable of thermal treatment for con-condensible fixed gas removal and separation of Halons, such as the REACH system designed by Walter Kidde Aerospace described in Section 5.5. Operation of the system at -60 degrees Centigrade for fixed gas removal followed by operation of the system at -20 degrees Centigrade will allow removal of Halon 1301 from the mixture with less than .4 percent of remaining halocarbon contamination.

During the course of this study no contaminants were found which could not be removed from the reclaimed Halon 1301. Since Halon 1301 is a stable compound, except in the presence of alkali earth metals such as sodium, there is no reason to suspect that a given mass of Halon would be classified as unusable due to contamination. When stored in carbon steel containers Halon 1301 and liquid phase water will generate undesirable corrosion byproducts such as bromide salts, free bromine ions and acids. These contaminants can be removed with the water as described in Section 4.5.

6. QUALITY CONTROL PROCEDURES FOR HALON RECLAMATION TECHNIQUES

6.1 GENERAL

Reclamation of Halon 1301, to be successful, must be based on strict housekeeping guidelines. As with charging fire extinguisher containers, lines must be clean and containers into which the agent is to be placed should be evacuated to at least 1 psia prior to use. Halon from any reclamation activity, as a minimum, must be checked for residue, boiling point, boiling point range 5 percent to 85 percent distilled, and moisture. Mole purity can be verified using a Gas Chromatograph costing approximately $10,000.00. Water content can be verified using a variety of methods. An automated Karl-Fischer device for water determination, depending upon automation level, costs approximately $7,000.00. Test methods for Halon 1301 quality determination described in MIL-M-12218C are clear and serve as good instruction for using Gas Chromatograph and other methods. Initial check of product, using a Goetz bulb (Centrifuge tube), for boiling point and boiling point range 5 percent to 85 percent distilled will serve as a good indicator for determining if GC results are reasonable.

Recovery efficiency is directly affected by quality control measures. One boiling point and boiling point range check requires approximately one half pound of Halon 1301. One Karl Fisher titration requires at least one quarter pound of Halon 1301 and usually more. Analysis by Gas Chromatograph requires several milliliters, however, this is based upon how the sample is obtained. This illustrates the importance of sampling reasonably large quantities of Halon 1301 for quality determination. Quality determination on a 22 pound sample requires approximately 1 pound of agent or 4.5 percent of the sample. This immediately reduces recovery efficiency to a maximum of 95.5 percent. Quality determination on a 2000 pound sample requires approximately 0.05 percent of the sample, which, assuming an overall 98 percent recovery efficiency, yields an overall recovery efficiency of 97.95 percent.

6.2 INCOMING AGENT QUALITY DETERMINATION

It is not practical to test agent from each fire extinguisher container for quality. Halon recovery equipment must be designed to accept reasonable contamination. Treatment methods outlined in Section 4, using standard refrigeration components, can be configured to remove a considerable quantity of contamination, except that of mixed Halons. This type of cross contamination must be separated by distillation techniques. One Halon recovery design presently available on the market allows for the distillation (vapor separation) of Halon 1301 from other halocarbons. This system, designated as REACH®, by Walter Kidde Aerospace, Inc., operates nominally at -60 degrees Centigrade. However, the REACH® system can also operate at any temperature between -70 and +20 degrees Centigrade. By operating the system at -60 degrees Centigrade, which allows high efficiency recovery of Halon 1301 and fixed gas separation, and then warming the cold tank of reduced fixed gas content Halon 1301 to approximately -20 degrees Centigrade, the Halon 1301 can be removed as a vapor which the higher boiler remains in the liquid phase. This separation technique of non-azeotropic mixtures is explained in Section 4.4.

Recovered Halon agent, following non-condensible gas removal, should be placed into cylinders, such as 150, 250, or 2000 pound containers. A sample taken from the cylinder should be analyzed for purity and moisture. Purified agent reclaimed into 150 pound, 250 pound or larger cylinders that is found to meet MIL-M-12218C requirements can then be transferred to a 2000 pound or mini-bulk 16,000 pound tank. It is possible, although unlikely, that recycled agent within a 2000 pound or smaller cylinder may not meet moisture or purity requirements. However, by analyzing a second sample from that particular cylinder, following treatment through filtration and filter techniques as shown in Figure 2, moisture and hydrocarbon content, if previously beyond specification, should be well within specification limits. This agent, provided it now meets MIL-M-

12218C requirements, can be transferred through the filter drier into a larger storage vessel without the excess nitrogen it previously contained.

Contamination from mixed Halons, if found to be beyond 0.4 percent, must be separated using distillation techniques within Halon recovery systems which offer such features, such as the REACH System by Walter Kidde Aerospace. Use of GC methods and Karl Fischer titration as outlined in MIL-M-12218C, or equivalent, should be used for quality determination of reclaimed Halon 1301 *prior* to use in *any* fire extinguisher charged with that agent.

Nitrogen pressure component in a charged fire extinguisher or other pressure vessel containing Halon 1301 can be adequately determined by measuring fire extinguisher pressure using a calibrated pressure gauge such as an ASHCROFT 0 - 1500 psig test gauge, Grade 3A, 1/4 percent accuracy with 5 psi subdivisions. This particular gauge costs approximately $300.00. Following pressure measurement a temperature measurement is made at the outside of the container near the center of the approximated liquid region. Use this measured temperature to calculate Halon 1301 vapor pressure. Subtract the calculated pressure from measured pressure to reveal the approximate nitrogen pressure component.

Halon 1301 containing a nitrogen pressure component greater than 5 psi cannot safely be stored in shipping containers normally used for transport if 450 psig relief valves are used. Halon 1301 containing greater than 40 psi nitrogen content cannot safely be stored in shipping containers normally used for transport if 500 psig or 600 psig relief valves are used. This specifically addresses DOT container classifications 4BA, 4BW, 110A800W, and 110A3000W.

6.3 PURITY DETERMINATION OF RECYCLED HALON 1301

Gas chromatography is a standard technique for measuring purity and identifying foreign materials in gasses and volatile liquids. A specific procedure is detailed in Mil Standard MIL-M-12218C and in DuPont Laboratory Test Method H0265.165.01.CW(P), both of which are appended to this feasibility study report as Appendix D and Appendix E, respectively.

Operating a gas chromatograph according to the routine procedures described can be done by a trained technician. Training is often available, at minimal cost, from the instrument supplier. Equipment necessary to perform GC purity measurement is available for approximately $10,000.00. Considering an investment of this magnitude for recovery equipment and previously mentioned liabilities, this additional cost is minimal.

Determination of moisture in Halon 1301 may be conducted by a variety of techniques including the phosphorus pentoxide method, infrared absorption, electrolytic moisture analysis, or by use of a piezoelectric analyzer. The standard method is Karl Fischer titration. While it is possible for a skilled chemist to assemble and use an orthodox titration apparatus, automatic titration specifically developed for Karl Fischer moisture determinations are sold by many laboratory supply companies. They range from $5,000.00 to $8,000.00 depending upon speed, sensitivity, and degree to which they are automated. These Karl Fischer titrators can be operated routinely by a trained technician.

6.4 NON-CONDENSIBLE GAS CONTENT WITHIN HALON 1301

It is feasible and has been demonstrated by the REACH® Halon recovery system design, that non-condensible gas removal to a pressure content below 10 psi is practical. This procedure allows for reclaimed Halon 1301 to be used in all fire extinguisher designs and it also allows for accurate agent charge weight determination without calculation and without compensating for non-condensible gas weight content. Evacuation methods combined with thermal treatment to -96 degrees

Fahrenheit permit non-condensible gas content removal to less than 1 psi partial pressure content. It is important to note here that preparation of any cylinder into which recovered Halon is to be placed MUST include evacuation to at least 1 psia. This action prevents additional non- condensables from being added to Halon placed into the cylinder. An unevacuated cylinder contains approximately 3 psi partial pressure oxygen. These pressure components ADD to any non-condensible partial pressure components remaining from a recovery process.

6.5 MOISTURE WITHIN RECLAIMED HALON

A second reason for evacuation of reclaimed agent storage containers, and possibly for preliminary dry nitrogen purge, is one of moisture.

Table 23[8] illustrates that a 26.7 cubic foot shipping container (2000 pound) containing an air temperature of 70 degrees Fahrenheit at a relative humidity of 50 percent at atmospheric pressure contains 0.015219 pounds of water. This water weight constitutes 7.6 ppm in 2000 pounds Halon 1301. This quantity ADDS to that water contained with the reclaimed agent. Reclaimed agent with water content of 5 ppm placed into such a container would indicate 12.6 ppm water content when a sample is removed and tested.

Note that the container filled with 1000 pounds of Halon results in a water content of 20.2 ppm due to moisture within the container.

---

[8] Table 22 information on moisture provided by Mark Sweval of Great Lakes Chemical Company.

TABLE 23. CONTRIBUTION OF MOISTURE TO HALON BY HUMID AIR

Water in a Cubic Foot of Air at Various Temperatures and Relative Humidity

Temp. °F	Relative Humidity 10	20	30	40	50	60	70	80	90	100	Temp. °C
	Grains	Grains	Grains	Grains	Grains	Grains	Grains	Grains	Grains	Grains	
-10	.028	.057	.036	.114	.142	.171	.200	.228	.256	.285	-23.3
0	.048	.098	.144	.192	.240	.289	.331	.385	.433	.481	-17.8
10	.078	.155	.233	.310	.388	.486	.543	.621	.598	.776	-11.7
20	.124	.247	.370	.434	.619	.741	.864	.986	1.112	1.235	-6.7
30	.194	.387	.580	.774	.968	1.161	1.354	1.648	1.742	1.936	-1.1
32	.211	.422	.634	.845	1.056	1.268	1.479	1.690	1.903	2.113	0
36	.237	.473	.710	.946	1.183	1.420	1.656	1.893	2.129	2.366	1.7
40	.285	.570	.855	1.140	1.424	1.709	1.994	2.279	2.564	2.849	4.4
45	.341	.683	1.024	1.366	1.707	2.043	2.390	2.731	3.073	3.414	7.2
50	.403	.816	1.223	1.630	2.038	2.448	2.853	3.261	3.658	4.076	10.0
55	.486	.970	1.455	1.940	2.424	2.909	3.394	3.879	4.364	4.849	12.8
60	.574	1.149	1.724	2.298	2.872	3.447	4.022	4.596	5.170	5.745	15.6
62	.614	1.228	1.843	2.457	3.071	3.685	4.299	4.914	5.528	6.142	16.7
64	.658	1.313	1.969	2.625	3.282	3.938	4.594	5.250	5.907	6.563	17.0
66	.701	1.402	2.105	2.804	3.504	4.205	4.906	5.607	6.303	7.009	18.9
68	.748	1.498	2.244	2.992	3.740	4.488	5.236	5.984	6.732	7.480	20.0
70	.798	1.596	2.394	3.192	3.990	4.789	5.585	6.384	7.182	7.980	21.1
72	.851	1.702	2.552	3.403	4.254	5.105	5.956	6.806	7.657	8.508	22.2
74	.907	1.813	2.720	3.626	4.533	5.440	6.346	7.253	8.159	9.066	23.3
76	.966	1.931	2.898	3.862	4.628	5.792	6.758	7.724	8.890	9.655	24.4
78	1.028	2.055	3.083	4.111	5.138	6.166	7.194	8.222	9.249	10.277	25.6
80	1.093	2.187	3.280	4.374	5.467	6.560	7.854	8.747	9.841	10.934	26.7
82	1.163	2.325	3.486	4.650	5.813	6.976	8.138	9.301	10.463	11.625	27.8
84	1.238	2.471	3.707	4.942	6.176	7.414	8.649	9.885	11.120	12.326	28.9
86	1.313	2.625	3.938	5.251	6.564	7.877	9.189	10.502	11.814	13.137	30.0
88	1.394	2.787	4.181	5.576	6.968	8.362	9.756	11.150	12.543	13.997	31.1
90	1.479	2.958	4.437	5.916	7.395	8.874	10.363	11.832	13.311	14.780	32.2
92	1.589	3.133	4.707	6.278	7.844	9.413	10.982	12.551	14.120	15.639	33.3
94	1.663	3.327	4.990	6.654	8.317	9.980	11.644	13.307	14.971	16.824	34.4
96	1.763	3.626	5.288	7.050	8.813	10.576	12.338	14.101	16.263	17.676	35.6
98	1.867	3.734	5.601	7.468	9.336	11.203	13.070	14.937	16.804	18.611	35.7
100	1.977	3.953	5.930	7.906	9.883	11.860	13.836	15.813	17.789	19.768	37.8

NOTE: 1 grain = 1/7000 lb.

$$\frac{\textit{grains/ft}^3\textit{(chart)}}{7} \times \frac{\textit{Container volume, ft}^3}{\textit{lb. Halon in container}} \times 1000 = \textit{ppm moisture in Halon due to moist air}$$

Example: Temperature = 70 F,° Rel Humidity = 80%, Container Volume = 0.2 ft^3 to hold 17 lb. Halon

From the chart, $\text{grains/ft}^3 = 6.384$

Then: $\frac{6.384}{7} \times \frac{.2}{17} \times 1000 = 10.7$ ppm moisture could be added to the Halon due to moist air in the container prior to filling

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7. AN ESTIMATE OF THE AMOUNT OF HALON 1301 THAT MIGHT BE AVAILABLE FROM RECYCLE OF AGENT FROM NON-AVIATION SOURCES (reference 9)

7.1 GENERAL

In order to develop an estimate of Halon 1301 that might be available from recycle from non-aviation sources, two main factors must be examined: (1) quantity of Halon 1301 produced, or is projected to be produced, to a certain point in time, and (2) an estimate of Halon 1301 that has been expended, or is projected to be expended at that point. Once total Halon 1301 supply is determined, the amount that is dedicated to aviation use is subtracted from the total. This should provide a reasonable estimate of the total amount of Halon 1301 used in non-aviation sources.

7.2 HALON 1301 AVAILABILITY ESTIMATES

From Table 2 in Section 1, current U.S. aviation fixed systems bank of Halon 1301 is estimated at approximately 109 tons (98.9 tonnes). According to the Taylor/Wagner report (reference 9), the total amount of Halon 1301 that could be available at the end of 1993 is 57,870 tonnes; therefore, world-wide availability of Halon 1301 from other than U.S. aviation sources is 57,870 minus 98.9 tonnes (109 tons) or 57,771 tonnes. From Section 3.7 Table 13, 1991 - 2000 aviation requirements account for approximately 1.47 percent (935 tons, 848 tonnes) of the total Halon 1301 bank. This 1.47 percent includes new production, class change impact, current fleet and hydrotest demand. The figure of 57,870 tonnes was derived as follows: 158,977 tonnes of new Halon 1301 is estimated to have been produced since 1970 and 96,738 tonnes is estimated to have been expended through fires, testing/training, leakage, venting to the atmosphere, etc. leaving a net Halon 1301 bank of 57,870 tonnes. Projecting ahead to the end of 1999 when production of new Halon 1301 is projected to cease, the Taylor/Wagner report predicts a world-wide Halon 1301 bank amount of 60,497 tonnes. The McCullough report provides Halon 1301 bank estimates only through the end of 1989. At that time McCullough estimated a Halon 1301 bank amount of 67,165 tonnes vs. a 1989 estimate of 41,818 tonnes by Taylor/Wagner. Even though McCullough states that a worst-case scenario for Halon 1301 emissions is considered, the figures for the cumulative bank total of Halon 1301 in the Taylor/Wagner report appear more conservative.

7.3 CONCLUSIONS

Even though the percentage of Halon 1301 produced for aviation purposes is small, it is supplied to a relatively small concentration of end-users compared to Halon 1301 produced for non-aviation purposes; therefore, it is likely that most of the aviation Halon 1301 could be made available for recycling. Halon 1301 that is produced for non-aviation purposes is used in a myriad of different configurations ranging from hand-held fire extinguishers to elaborate under-floor and above-floor fire protection systems. Because of the large and diverse number of end users for non-aviation Halon 1301 systems, the percentage available for recycling is projected to be much lower than in the aviation industry.

Repair and servicing of pressure vessels used for aviation is controlled by the airworthiness authority, FAA, CAA and JAA. It is therefore feasible to carefully control both recovery and quality control of the recovered product.

Support of the U.S. civil fleet of aircraft utilizing recycled Halon 1301 until and beyond 2020 is addressed in Section 9.

8. NON-AVIATION HALON 1301 RECLAMATION

8.1 GENERAL

Halon 1301 reclamation from industrial or commercial non-aviation type fire extinguishers generally involves larger quantities of agent with lower nitrogen concentration than that found in aviation fire extinguishers. However, reclamation from marine and ordnance Halon 1301 fire extinguishers may involve dealing with nitrogen pressure as high as 750 psig at 70 degrees Fahrenheit.

Halon 1301 found in non-aviation pressure vessels is often of a higher purity level than found in aviation fire extinguishers because of decreased handling and number of recharge cycles. Aviation fire extinguisher containers (Halon 1301) are generally smaller than industrial and commercial fire extinguisher containers. Aviation fire extinguisher containers are typically charged at 40 to 65 pounds per cubic fill density. Commercial and industrial Halon 1301 fire extinguisher containers are generally larger, lower pressure containers typically charged to higher fill densities and generally contain greater agent quantities, usually from 150 to 1000 pounds, than aviation fire extinguisher containers. Fixed gas pressure in industrial and commercial fire extinguisher containers is typically 360 psig whereas aviation systems use 625 or 825 psig. Aviation cargo applications typically use 300 or 360 psig. Larger agent quantities, provided that moisture content is well controlled, will be more tolerant to the same quantity of a contaminant than that of a smaller quantity of Halon 1301. This means that moisture from a charging hose open to the atmosphere prior to charging will have a greater effect on a smaller quantity of agent than it will on a larger quantity. Although commercial and industrial fire extinguisher containers are constructed from different materials, Halon 1301 is a stable compound under clean conditions. Therefore, the material from which the container was constructed is insignificant as long as the container and the Halon 1301 are clean and dry. For example, in Section 4, Table 13, samples E - H are from industrial sources. Analysis of those samples revealed a water content of from 10 to 36 ppm while FC-1301 mole content was 99.96 percent to 99.85 percent. Average impurity levels in samples E - H were clearly less than that found in aviation Halon 1301 samples. Data from ATOCHEM on analysis of contamination found within aviation samples corresponded favorably to contaminant levels as experienced in analytical work performed by DuPont for the present study.

8.2 CONTAMINATION REMOVAL FROM NON-AVIATION HALON 1301 SOURCES

Halon 1301 used in non-aviation sources will typically be found in cylindrical design pressure vessels constructed from either carbon steel or aluminum. They usually will contain a syphon tube. Water contamination in Halon 1301 beyond 90 ppm will create a corrosive action on the syphon tube at the liquid interface level. Water can also cause corrosion effects on the container which could produce a safety hazard. If corrosion effects on the syphon tube are so severe that it is unusable, the cylinder must be inverted in order to effectively recover the Halon 1301 contents.

Contamination of a non-chemically reactive nature may be removed from Halon 1301 by those procedures as described in Section 4 of this study. Complete recovery treatment of the Halons is addressed within Section 5.

If aluminum or carbon steel pressure vessels are found to contain a high degree of moisture contamination, then multiple passes through refrigeration type filter driers are required. Disposable refrigeration filter driers are very effective in removing water and other contaminants from Halon

1301. The use of disposable type filter driers greatly facilitates replacement when exposed to severely contaminated Halon 1301 or after being used to capacity.

8.3 NON-AVIATION HALON RECLAMATION PROCESSES AND EFFICIENCIES

Non-aviation Halon 1301 fire extinguisher assemblies typically utilize working pressures near 360 psig as compared to aviation fire extinguisher pressures of 600 psig to 825 psig or higher. Because of this decreased nitrogen content, slightly more efficient Halon 1301 recovery can be realized. In some cases, Halon 1301 may be used from 500 pound or larger industrial containers. Figure 6 illustrates a disposable filter-drier method for using Halon 1301 directly from industrial containers for charging purposes. Nitrogen weight MUST be compensated for as detailed within this section when the direct transfer method is used.

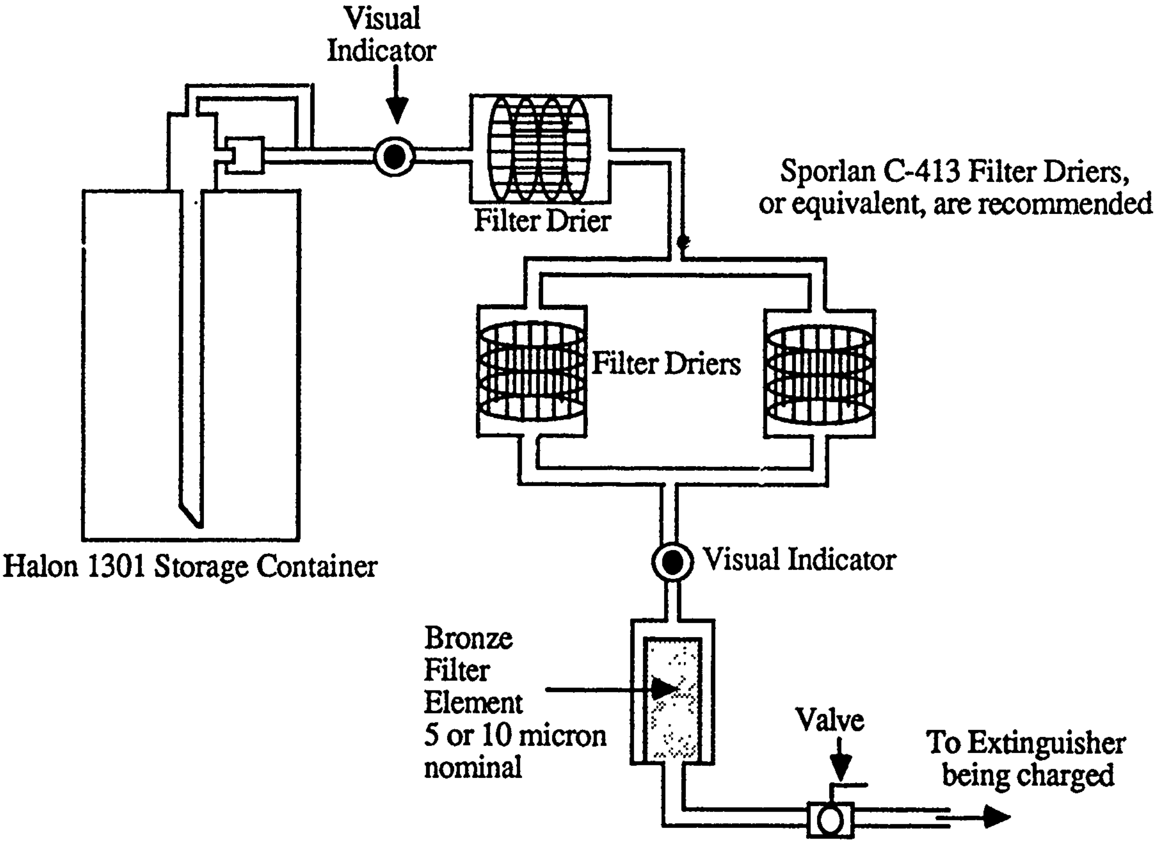

FIGURE 6. Direct Transfer Method

This direct transfer method, which may alternatively be achieved by using replaceable core components as illustrated in Figure 2, must also include appropriate agent quality determination as discussed in the following example. This quality or purity check should be performed on an untreated sample of the agent. One sample from each location is sufficient to determine water content and purity. If water content in an untreated sample is near saturation level in Halon 1301, as illustrated in Figure 3, all desiccant elements should be replaced upon reaching the end of liquid agent transfer from the container. Also, in cases of near saturation water content, the initial moisture indicator may become fully hydrated and require replacement. Location of a mechanical filter following treatment will remove any loose desiccant debris should it occur.

Transfer of Halon 1301 from large industrial/commercial storage containers may be performed using the previously described direct transfer method without consuming the time and energy required to process the entire container contents through a Halon recovery system to achieve fixed gas separation. This technique for using Halon 1301 directly from industrial or commercial fire extinguisher containers is described as follows:

An initial sample is taken from the storage container for analysis. A second sample is subsequently taken following treatment as depicted in Figure 6. Even if moisture levels up to 90 ppm are indicated in the initial sample, moisture levels less than 10 ppm will be realized at the output of the system. If present, liquid water will float on the surface of the Halon 1301 at temperatures below 145° F, therefore any water in the liquid phase would not be encountered until almost all of the Halon has been transferred out of the storage container. The nitrogen content within the storage vessel provides sufficient pressure to transfer the agent into the fire extinguisher. Nitrogen weight compensation is approximated by using Equation (2) in the following manner:

$$N2w = \left[0.00148148 \left(\frac{Pt - Vp}{Tr} \right) - 1.94213 \times 10^{-9} \times \left(\frac{Cw}{Cv} \right)^{2.67} \times Pt^{3.10} \right] Cv \qquad (2)$$

Charge the fire extinguisher to the specified agent charge weight plus agent charge tolerance. Measure pressure and temperature in the fire extinguisher. Into Equation (2) enter this measured pressure, Halon vapor pressure at the measured container temperature, agent charge weight, internal volume of the container being charged, and the container temperature in degrees Rankine. Calculation results will be the weight, in pounds, of the nitrogen contained within the fire extinguisher.

$$\text{Est. N2} = 2.78 \text{ X } 10^{-6} (P_t - 199) \text{ X } C_v \qquad (3)$$

For applications involving near ambient conditions (60 to 80 degrees Fahrenheit), Equation (3) can be used.

After calculating container nitrogen weight using Equation (2), add this adjustment weight of Halon in order to obtain total supply weight of nitrogen and Halon. If an adequate volume of liquid Halon is not available from a supply cylinder or extinguisher, any available Halon liquid source will work provided the new Halon 1301 source pumping pressure is sufficient to complete container charging.

After performing this adjustment, especially when using virgin Halon for the adjustment specification, calculate or look up the new weight, pressure, and temperature condition. The Halon charge weight, within tolerance range, should now equal the total container charge weight less the calculated nitrogen weight. For Example, if the final contents weight were to be 22.53 pounds and the nitrogen weight as calculated by Equation (2) is 0.28 pounds, then the agent charge component is 22.53 - .28 or 22.25 pounds. This 22.25 pounds is within fill tolerance of 22.00 +.25, -.00 pounds for a 630 cubic inch aviation fire extinguisher. The .28 pounds of nitrogen corresponds to a vessel pressure of 385 psia or 360 psig. Further pressurization of this vessel to 600 psig at 70° F will result in a correct nitrogen weight of .64 pounds.

In the foregoing manner, Halon can be used directly from large industrial/commercial containers without the need for recycling the entire contents.

The remaining vessel contents, upon utilizing all or most of the available liquid, may be recovered by connection to a high efficiency Halon recovery system.

8.4 QUALITY CONTROL PROCEDURES FOR NON-AVIATION HALON

There are no known conditions that would require differentiating between non-aviation and aviation sources of Halon 1301 in respect to quality control procedures. Long term storage of Halon 1301 in aluminum or carbon steel pressure vessels, although providing a greater susceptibility to corrosive effects created by excess moisture, does not cause deterioration of the Halon 1301 compound. Agent processing from aluminum or carbon steel vessels can process in the same manner as Halon 1301 from aviation fire extinguishers constructed of stainless steel materials.

9. UTILIZATION OF RECLAIMED HALON 1301 IN SUPPORT OF PRESENT AND FUTURE CIVIL FLEET REQUIREMENTS UNTIL 2020 AND BEYOND

9.1 GENERAL

Halon 1301 is the single most significant fire suppressant in aviation. The agent's suppression efficiency, low toxicity and effectiveness between -80 to +300 degrees Fahrenheit make it ideal for use throughout the aviation industry. More data and dynamic behavior properties are known about Halon 1301 than any other fire extinguishing agent.

Halon 1301 can be efficiently recycled, conditioned to within general MIL-M-12218C specifications, and placed into storage for future aviation use. Halon 1301 remains indefinitely stable when properly handled and stored.

9.2 HALON 1301 - PRESENT AND FUTURE CIVIL AIRCRAFT DEMAND, 1991 - 2000

Projected total Halon demand for present and future U.S. civil fleet needs from 1991 through 2000 can be derived from data contained in Tables 9, 10, 11, and 12. Table 13, Section 3.7 summarizes data contained in Tables 9 through 12, inclusively. Table 24 presents this summary on an annual breakdown basis. The annual breakdown assumes equal annual demand where ten year projections were previously made. Hydrotest inspection demand is based on the assumption that all Halon 1301 in the existing fixed systems bank, transport plus commuter fleets, estimated at approximately 109 tons (Table 2) will be replaced due to hydrotest inspection requirements over the next 5 year period at an average annual rate of 21.8 tons per year.

Table 24 also lists Halon 1301 demand based upon recovery of that Halon that would otherwise be lost because of hydrotest inspection at an overall recovery efficiency of 96 percent. The 96 percent rate considers an initial 98 percent recovery efficiency (reclaimed to MIL-M-12218C purity standards) and losses resulting from quality control procedures and handling, leakage, etc.

The "New Aircraft" column in Table 24 includes the Halon 1301 demand for new transport and commuter aircraft. This column is derived from the addition of data from Tables 10 and 12, the annual averages of 47,989 pounds based on the total 10 year projection from Table 10 of 479,886 pounds and annual commuter projections from Table 12.

The "Cargo Reclassification" column in Table 24 considers retrofit of existing aircraft (79,372 pounds from Table 11) averaged over the three year 1991 through 1993 period (26,457 pounds per year) and, from Table 10, the amount of Halon required to equip new production aircraft from 1991 through 2000 (40,658 + 27,016). Years 1991 through 1993 represent the sum of retrofit and new production aircraft Halon demand during that period, which are 26,457 pounds plus 6,767 pounds per year or 33,224 pounds per year.

The "Hydro" column in Table 24 is the worst case Halon 1301 demand due to hydrotest inspection for both transport and commuter fleets. It results from:

a) Existing fleet hydrotest inspection requirements based on approximately 109 tons averaged over a period of 5 years at 21.8 tons per year.

b) Hydrotest of the agent added to the existing fleet in years 1991 through 1993 due to class change (D to C). Because of the 5 year hydrotest inspection requirement, that retrofit will require hydrotest in 1996, 1997, and 1998. This results in an additional 26,457 pounds added to the existing 43,600 amount for a period of three years and every 5 years thereafter.

c) Additionally, beginning in 1996 is the amount of agent which must be removed for hydrotest inspection on aircraft produced in 1991. That amount is 10 percent of the projected 547,560 pound total (Table 10) or 54,756 pounds. This results in a sum of the prior annual 43,600 pound requirement plus 26,457 pounds for retrofit (Table 11) plus 54,756 pounds from new production plus 3,516 pounds from commuter fleet aircraft produced in 1991 (Table 12) for a total requirement in 1996 of 128,329 pounds. Hydrotest requirement considerations for 1997 create the same 128,329 base plus the increase in commuter fleet requirements to 3,954 pounds for a total 1997 demand of 128,767 pounds. Sums for the remaining years are calculated in the same manner.

The "Discharged Bottle Consumption" column in Table 24 reflects worst case consumption predicted by Table 9, including class change impact of 170,015 pounds averaged over 10 years for an average annual consumption of 17,002 pounds.

The "Sub Total without Recovery" column in Table 24 is the sub total requirement if no recovery of Halon 1301 occurs during hydrotest inspection. The column "Sub Total with Recovery" is the sub total requirement if 96 percent of that agent which would be lost during hydrotest inspection is recovered and made available for reuse. Implementation of Halon 1301 recovery at an efficiency of 96 percent could potentially save 776,940 pounds of agent during the 1991 to 2000 time period.

TABLE 24. Total Halon 1301 Demand
U.S. Civil Domestic Fleet 1991 - 2000

Year	New Aircraft	Cargo Reclassification	Hydro	Discharged Bottle Consumption*	Subtotal Without Recovery	Subtotal With Recovery
1991	51,505	33,224	43,600	17,002	145,331	103,475
1992	51,943	33,224	43,600	17,002	145,769	103,913
1993	52,098	33,224	43,600	17,002	145,924	104,068
1994	52,199	6768	43,600	17,002	119,569	77,712
1995	52,358	6767	43,600	17,002	119,727	77,871
1996	52,158	6768	128,329	17,002	204,257	81,060
1997	51,944	6768	128,767	17,002	204,409	80,792
1998	51,879	6767	128,922	17,002	204,570	80,805
1999	51,594	6768	102,566	17,002	177,930	79,466
2000	51,491	6768	102,725	17,002	177,986	79,370
	519,169	147,046	809,309	170,015	1,645,472	868,532

* includes cargo discharge due to compartment reclassification within existing fleet

Variation in numbers within the columns is due to all previously outlined considerations of transport plus commuter fleet Halon 1301 requirements. In some cases sums of columns or rows may not exactly add up to previously stated quantities. These small errors, typically 10 to 20 pounds out of 100,000, are due to rounding to the nearest integer.

9.3 HALON 1301 REQUIREMENT FOR SUPPORT OF THE CIVIL FLEET UNTIL 2020.

Prior to estimating this requirement several factors from the preceding work elements must be determined.

Factor 1: Inservice Halon 1301 consumption from 2000 to 2020, due to inservice discharge, averages the 1991 baseline of 8.5 tons per year plus additional discharge due to growth.

Factor 2: Projection of Halon consumption from 1991 to 2000 due to 5 year hydrostatic testing requirements is based upon (1) the existing bank in the current civil fleet of 109 tons (Table 2, Section 1.1) increasing to 149 tons due to the Class D to Class C change, (2) this 149 tons increasing again to 423 (*add Table 10, 547,560 if Class C*) tons by the year 2000 due to new aircraft production, and (3) the commuter fleet (Table 12) increasing the requirement by an additional 19.64 tons to 443 tons. Hydrostatic test of the existing 109 tons necessitates a 22 ton replacement requirement per year. Hydrostatic testing of the next 40 tons (class change impact) requires an additional 8 tons per year. The next increase of 274 tons (423 minus 149) results from new production requirements 1991 - 2000.

Factor 3: The Halon 1301 demand in Factor 1 includes Class D to Class C redesignation and operation.

Factor 4: Retiring aircraft are replaced and as aircraft retire, basic replacement aircraft occurrences of non-fire events are assumed to be approximately the same as previous, similar installation occurrences.

Factor 5: Numbers of aircraft increase at an annual rate of approximately 3.5 percent per year with a proportionate increase in Halon consumption.

Halon projected demand from 2001 to 2020 is based upon an increase of 3.5 percent (reference 11 Airbus Article, Appendix F) compounded annually. Long term market predictions indicate a growth, based upon 20 year global averages, of approximately 3.6 percent. Passenger traffic growth (base case) is predicted as 4.1 percent.

Table 25 lists Halon 1301 and Halon 1211 projected demand for the 2001 through 2020 time period. The demand includes new aircraft fleet growth and increases in consumption. The estimates are listed beginning with a base line at the year 2000. Each year a factor of 3.5 percent is used in an attempt to model fleet growth with a parallel Halon 1301 consumption from one year to the next. From Table 10 an average of 54,756 pounds of Halon 1301 is obtained by dividing the greatest need for the ten year projection by ten to obtain an annual average consumption. In a similar fashion, average annual demand for Halon 1211 based on Table 10 is 5,399 pounds. Projected average annual demand for Halon 1301 from one year to the next is then calculated using a base line of 54,756 pounds per year for new production, 80,931 pounds per year due to hydrotest inspection requirements, plus an average Halon 1301 usage consumption of 17,000 pounds per year (458 pounds per year of Halon 1211 from Table 9).

For Halon 1301 applications these demands sum to 152,687 pounds or 76.34 tons at year 2000 as an average demand. The average demand for Halon 1211 is 5,857 pounds or 2.93 tons. These baseline numbers are then compounded on a 3.5 percent annual rate to project requirements over the next 20 years. For example, to determine total estimated Halon 1301 consumption for the fifteenth year past 2000 (year 2015) multiply 76.34 times (1 + .035) raised to the 15th power which yields 127.90 tons. Each annual projection is then summed (an arithmetic compound series) from 2001 through 2020 to obtain total, twenty year, Halon 1301 demand.

TABLE 25. Halon Projected Demand 2000 - 2020
Requirements in U.S. tons

YEAR	New Production Halon 1301	Hydrotest Halon 1301	Consumption Halon 1301	Halon 1211 Combined	
2000	27.38	40.46	8.50	2.93	Baseline
2001	28.31	41.87	8.80	3.03	
2002	29.92	43.34	9.11	3.14	
2003	30.97	44.86	9.42	3.25	
2004	32.05	46.43	9.75	3.36	
2005	33.17	48.05	10.10	3.48	
2006	34.33	49.74	10.45	3.60	
2007	35.53	51.48	10.81	3.73	
2008	36.78	53.28	11.20	3.86	
2009	38.06	55.14	11.58	4.00	
2010	39.40	57.07	12.00	4.13	
2011	40.78	59.07	12.41	4.28	
2012	42.20	61.14	12.84	4.43	
2013	43.68	63.28	13.29	4.58	
2014	45.21	65.49	13.76	4.74	
2015	46.79	67.78	14.24	4.91	
2016	48.43	70.16	14.74	5.08	
2017	50.13	72.61	15.25	5.26	
2018	51.88	75.15	15.79	5.44	
2019	53.70	77.78	16.34	5.63	
2020	55.57	80.51	16.91	5.83	
TOTAL 2001 - 2020	844.27	1,224.69	257.29	88.89	

9.4 HALON 1301 DEMAND OF THE CIVIL AVIATION FLEET 1991 UNTIL 2020 VS AVAILABLE AGENT PROJECTIONS

Table 26 summarizes Halon 1301 and Halon 1211 demand as previously projected from Tables 24 and 25.

TABLE 26. Halon Demand 1991 - 2020 (Tons)
U.S. Civil Fleet (Transport and Commuter)

	WITHOUT HYDROTEST HALON 1301	WITH HYDROTEST HALON 1301	HALON 1211
1991 thru 2000 Including Class Change Impact	418	823	30
2000 thru 2020 Including Class Change Demand	1,102	2,327	89
TOTAL (Tons)	1,520	3,150	119

It is important to note that the following discussion refers to metric tons (tonnes). One metric ton (tonne) equals 2,204.6 avoirdupois pounds (ton). The previous discussion referred to Halon weight in units of 2,000 avoirdupois pounds per ton. With reference to Section 7.0, McCullough estimated the 1989 Halon 1301 bank to consist of 67,165 tonnes (74,036 tons) vs. 41,818 tonnes (46,096 tons) as reported by Taylor/Wagner.

According to Taylor/Wagner, 57,870 tonnes (63,790 tons) of Halon 1301 would be in the Halon 1301 bank at the end of 1993. This amount minus the 99 tonnes (109 tons) currently in the civil aviation fleet yields 57,771 tonnes (63,681 tons) available for reclamation (if not used for other purposes).

It is projected that the civil fleet demand until 2020 will be 1,379 tonnes (1,520 tons) or 2,858 tonnes (3,150 tons) if the 5 year hydrotest inspection demand is considered. Out of the total 57,771 tonnes of Halon 1301 theoretically available, it is then very conceivable that the civil fleet could be supported far beyond 2020 using reclaimed Halon 1301.

Support of the civil aviation fleet until 2020 is estimated to demand approximately 4.95 percent of the present Halon 1301 bank (considering a worst case demand which includes the 5 year hydrotest inspection requirement without considering recycling). The 4.95 percent figure is based on Halon 1301 production ending in 1993. Halon 1301 production continuing until 1999 would reduce the percentage of Halon 1301 required from the bank as needed for the civil aviation fleet.

Considering the practicality of recovering Halon 1301 and realizing that a reusable agent recovery efficiency of from 96 to 98 percent is possible, utilizing Halon recovery and conditioning systems could considerably reduce the current and future Halon bank demand. From Table 24, the 1991 through 2000 hydrotest inspection agent loss is projected to be 405 tons. Assuming a 96 percent agent recovery efficiency, a savings of 388 tons could be realized. Agent savings from 2001 through 2020 could be 96 percent of 1,225 or 1,176 tons. Total savings from 1991 through 2000 could therefore be 1,419 tonnes (1,564 tons) which would decrease the demand on the Halon 1301 bank by approximately 2.46 percent (demand would be reduced from 4.95 percent to 2.49 percent).

Figure 7 on page 66 and Figure 8 on page 67 illustrate Table 24 and Table 25 data in flow chart form.

9.5 HALON 1211 DEMAND BY THE CIVIL AVIATION FLEET VS. AVAILABLE AGENT PROJECTIONS

With reference to Appendix A of the Taylor/Wagner report, the estimated Halon 1211 bank size was 83,824 tonnes as of 1990

If Halon 1211 were to remain as the agent of choice for aviation portables, the 1211 civil fleet requirements until 2020 would be assured. It is probable, however, that Halon 1211 will be replaced by substitute streaming agents in the near future which offer equivalent fire suppression qualities with less environmental impact..

Projected requirements of 108 tonnes (119 tons) is less than 0.2 percent of the total amount of available Halon 1211.

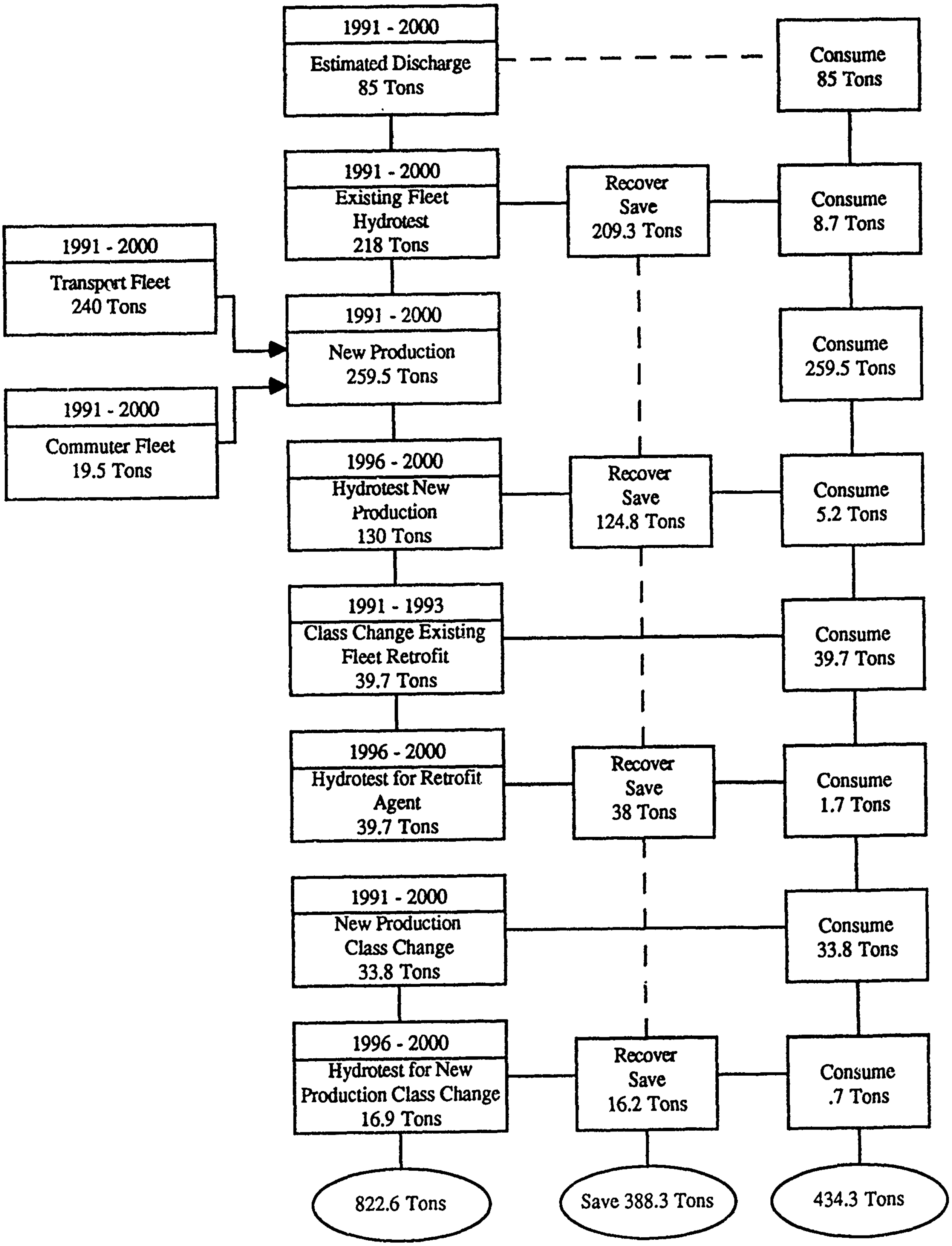

FIGURE 7. Illustrated Representation of Halon 1301 Usage and Demand
1991 - 2000 From Table 24

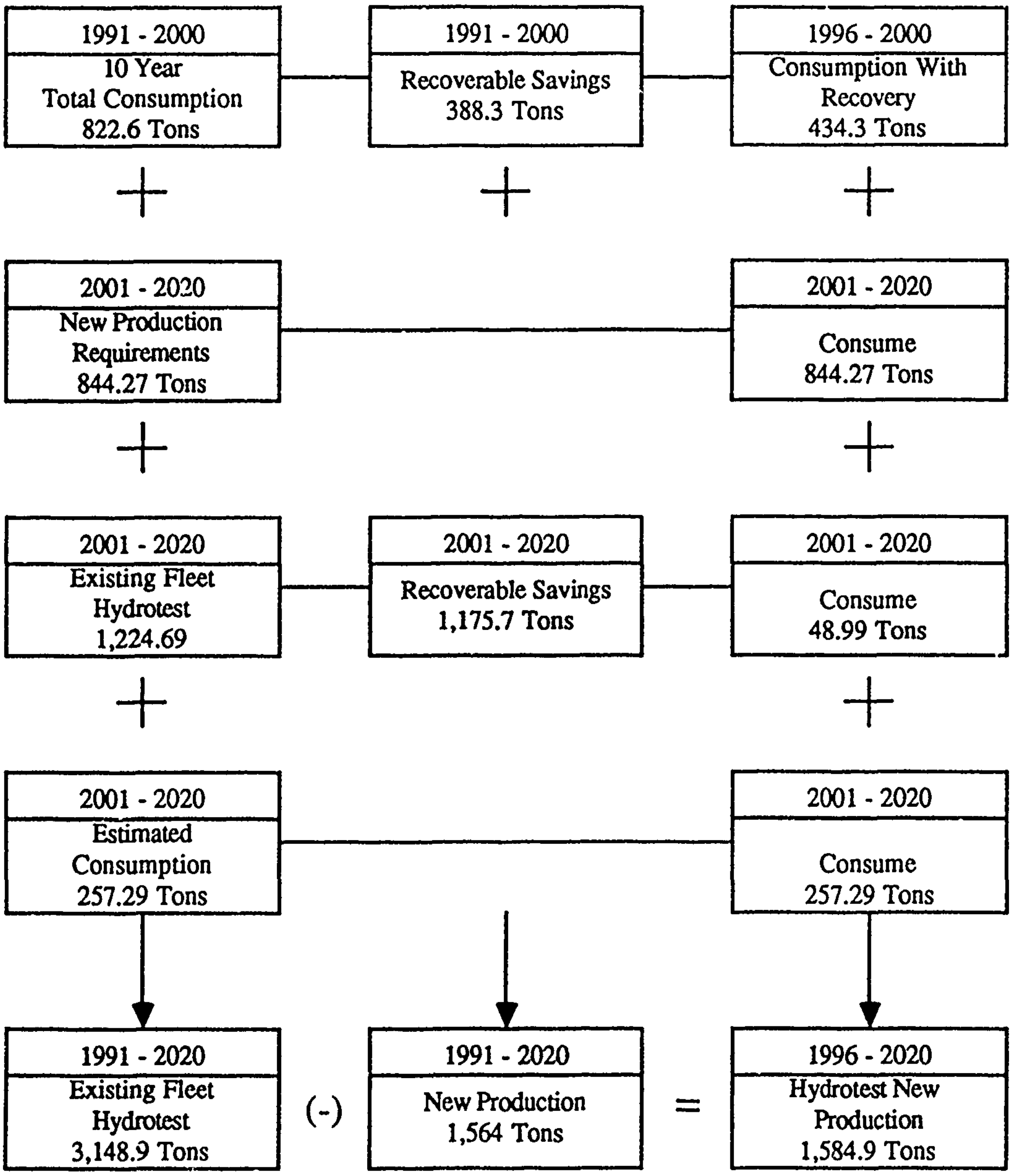

FIGURE 8. Illustrated Representation of Halon 1301 Usage and Demand, 1991 - 2000 and 2001 - 2020 From Table 25.

10. FIRE SUPPRESSION EFFECTIVENESS - RECYCLED VS. VIRGIN HALON 1301

10.1 GENERAL

Halon 1301, bromotrifluoromethane, is a stable compound manufactured by introducing bromine into trifluoromethane superheated vapor at certain conditions. This process produces and maintains a "fully-filled" molecular form. All available positions of the Trifluoromethane (CHF_3) molecular form remain closed due to hydrogen atom replacement/substitution. The hydrogen atom is broken away and replaced with Bromine. This forms $CBrF_3$.

Fire Suppression using Halon 1301 is effected by chemical action. Although Halon 1301 behaves as a refrigerant during discharge and demonstrates an ambient temperature vapor density of approximately 5.5 times the density of air, the primary fire suppressive action is of a chemical nature. The $CBrF_3$ molecule, in a vapor state, reacts with the oxidizing interface at the fuel/air boundary. The $CBrF_3$ molecules, following thermal (heat) input, dissociates into CF_3 and a free Br radical. The Br radical reacts with hydrogen (H) at the oxidizing interface forming HBr (Hydrogen Bromide).

The Hydrogen Bromide (HBr) combines with available Hydroxyl (OH) radicals to form water (H_2O) and free Bromine (Br) radicals. This Bromine radical chain of events of molecular re-combination and dissociation produces a very efficient fire suppressive action and is labeled the Free Radical Theory.

Given that there are 6.023×10^{23} molecules of Halon 1301 per gram mole reference of the agent. One pound weight of Halon 1301 contains approximately 1.83×10^{24} molecules of $CBrF_3$.

A second theory, the Ionic Theory, relates oxygen atom electron affinity to the fire-fuel-oxidizer role. In this theory, elemental oxygen combines with available electrons to activate the oxygen atom prior to reaction with the fuel. The Bromine atom from the $CBrF_3$ becomes a significantly larger target for the capture of these available electrons. The $CBrF_3$, therefore, reduces the likelihood of a given oxygen atom participating in the reaction chain.

10.2 RECYCLED HALON 1301 FIRE SUPPRESSION EFFECTIVENESS

Bromotrifluoromethane effects fire suppression by chemical means. Quality of Halon 1301, according to MIL-M-12218C, is determined in part by the percent of $CBrF_3$ as determined by gas chromatography or similar methods. A requirement of 99.6 percent minimum purity, according to MIL-M-12218C results in a reference level of fire suppression effectiveness.

Recycled Halon 1301 can be processed, using conventional equipment, to remove contaminants within 99.6 mole percent purity levels. This has been demonstrated using several different techniques.

The primary factor that permits conventional recovery methods to be used is the stability of Halon 1301 in current storage methods. Halon 1301 remains stable as $CBrF_3$ unless heated to above 600 degrees Fahrenheit (316 degrees Centigrade) relative to normal storage conditions, exposed to alkaline earth elements in large quantities, or stored in severely contaminated carbon steel vessels with approximately 10,000 ppm or greater water content. Those conditions which would render Halon 1301 unusable as recovered are highly unlikely and highly improbable to be encountered.

Halon 1301, reclaimed to 99 plus percent mole purity levels will be as effective in fire suppression as virgin manufactured Halon 1301 from the aspect of agent and fire scenarios. However, the use of reclaimed Halon 1301 in fire suppression systems is a function of physical purity rather than fire efficiency.

Halon 1301 reclaimed from fire extinguisher bottles must not only meet mole purity treatments, it must also meet moisture and particulate criteria. This is necessary in order to maintain discharge reliability performance within the varied system configurations found in aviation applications.

Moisture within reclaimed Halon 1301 can be removed sufficiently to meet MIL-M-12218C standards using conventional means. Particulates can be mechanically removed as demonstrated in Section 4.

Based upon demonstrated capabilities in reclaiming Halon 1301 to MIL-M-12218C quality levels, there will be no difference in fire fighting performance of reclaimed Halon 1301 and virgin manufactured Halon 1301.

11. SAFETY ISSUES OF RECLAIMED HALON 1301

As discussed in Section 10, Halon 1301 can be reclaimed using conventional means to within the purity levels of MIL-M-12218C excluding, specifically, dissolved gas content such as dissolved helium or nitrogen. However, a small amount of those gasses, from 2 to 10 psi total partial pressure, is neither detrimental to the reclaimed agent nor should it prohibit reuse of the agent for fire extinguisher charging.

The single most significant safety issue is corrosion and toxicity. Halon 1301 can remain an effective fire suppressant while exhibiting a corrosive behavior in the presence of moisture. In a similar manner, Halon 1301 can become contaminated with metallic salts, oils, acids and other halocarbons which may increase the toxicity of post-discharge byproducts.

Reclamation of Halon by Level I methods, as described in Section 4, should remove moisture and contamination to acceptable levels. The only cautions here are those associated with overloading the filter drier, mechanical filters, and transferring excess nitrogen into a storage vessel that is not designed to be used as a nitrogen pressurized fire extinguisher. Level I methods will not remove mixtures of halocarbons. Thermal treatment is required to accomplish foreign halocarbon removal.

Thermal treatment to -80 degrees Fahrenheit or colder will allow fixed gas separation while minimizing Halon 1301 loss. Thermal treatment must be performed only on the Halon 1301 and nitrogen charge and not on the fire extinguisher pressure vessel. Sudden chilling of pressurized vessels to extremely low temperatures (-80 degrees Fahrenheit) is dangerous and must be avoided since thermally induced stress risers and material embrittlement will occur.

The use of pressure transfer, Level I, methods without mechanical filtration and drier implementation is not recommended under any circumstances because analysis of Halon 1301 field samples revealed mixed halocarbons, oil traces and high water levels.

Halon 1301 reclaimed by Figure 2 or similar processes, with proper techniques, filtration and filter drier integrity, will be identical, from a safety viewpoint, to virgin Halon 1301.

12. ADDITIONAL ISSUES OF HALON 1301 RECYCLING

12.1 GENERAL

Halon 1301, bromotrifluoromethane, CF3Br, is a colorless, water-white liquid with a slight ether-like odor in high concentration. In concentrations used for fire extinguishing applications Halon 1301 is odorless.

As of this report date, Halon 1301 is the agent of choice for aircraft fixed systems. It is easily liquefied by compression which classifies it as a liquified compressed gas. Halon 1301 is normally stored and handled under saturated conditions such that the liquid and vapor coexist in equilibrium.

TABLE 27.

PHYSICAL PROPERTIES OF DUPONT HALON 1301 (reference 5)

Molecular Weight	148.93
Boiling Point at 1 atm. °F	-71.95
°C	-57.75
Freezing point °F	-270
°C	-168
Critical Temperature °F	152.6
°C	67.0
Critical Pressure, psia	575
atm	39.1
Critical Volume, cu. ft. per lb.	0.0215
Critical Density, lb per cu. ft.	46.5
gm per cu. cm.	0.745
Specific Heat, Liquid (Heat Capacity) at 77°F, BTU per lb per °F	0.208
Specific Heat, Vapor, at constant pressure (1 atm) 77°F, BTU per lb per °F	0.112
Heat of Vaporization at 1 atm, BTU per lb	51.08
Thermal Conductivity of Liquid at 77°F BTU/(hr) (ft) (°F)	0.025
Viscosity, Liquid at 77°F, centipoise	0.15
Viscosity, Vapor at 77°F, 1 atm, centipoise	0.018
Surface Tension at 77°F, dynes per cm	4
Refractive Index of Liquid at 77°F	1.238
Relative Dielectric Strength at 77°F (Nitrogen = 1)	1.83
Solubility of halon 1301 in Water at 1 atm, 77°F, Wt%	0.03
Solubility of Water in Liquid Halon 1301 at 77°F, Wt%	0.0095

Isothermal pressure decrease will cause the liquid to flash into vapor with characteristic refrigeration effects. Handling of Halon 1301 can be made quite safe and simple by understanding the physical properties of the agent and proper handling techniques.

12.2 MAJOR EQUIPMENT USED DURING THIS FEASIBILITY STUDY

Two pumps, a FRICK BLU-COLD® Vapor Recovery Unit Model 10003 and GETZ HALON 1301 liquid pump, were evaluated during this feasibility study. Both performed well during use.

The FRICK BLU-COLD VRU® (Vapor Recovery Unit) retails for approximately $3,180.00. It is available from FRICK, 345 West Main Street, Waynesboro, Pennsylvania 17268. This vapor pump has a design compression ratio of 23 to 1, an oiled crankcase operating at pump pressure, and an oil separator. Maximum input pressure is 300 psig. Overload at the input will result in excess oil being captured by the Halon which removes oil at excessive rates from the crankcase. Use and evaluation of two FRICK BLU-COLD® pumps in the development of a Halon recovery system by Walter Kidde Aerospace demonstrated that a weekly visual check of oil crankcase level at the pump's sight glass was adequate. Low oil level results in increased cycle frequency at 230 to 260 psig output pressure loading. Crankcase lubrication is supplied by 030 refrigeration oil (Calgon C-4), viscosity 300 SUS.

The GETZ Halon pump, for pumping liquid phase Halon 1301, is a triple diaphragm pump which retails for approximately $3950.00. The pump is available from GETZ Manufacturing, 1723 SW Adams Street, Peoria, Illinois 61602. This pump, which can be used for fire extinguisher charging, is also effective for transferring Halon 1301 from 150 pound or 250 pound cylinders into 2000 pound or larger storage cylinders. During evaluation a maximum transfer rate of 18 pounds per minute was obtained. It is quite difficult to maintain liquid state delivery with this pump. Many pounds of agent can be lost during cylinder exchange if the hose end valves are accidentally opened. However, the pump is relatively easy to use, transport and service. Maximum input pressure for this pump is 500 psig.

12.3 FEASIBILITY OF FIELD RECYCLING

While field recovery of Halons is feasible, it is labor intensive and has a relatively low (80 percent to 90 percent) recovery efficiency. Cold bucket methods, such as those shown in Appendix B, can be used for field reclaim. Halon, which would otherwise be lost to the atmosphere, can be relieved of excess nitrogen by taking advantage of Dalton's Law and reduced nitrogen solubility at the lower temperatures. Dalton's Law at low temperatures is the more significant function beyond that of Henry's Law which effects removal of non-condensables at low temperatures. Upon non-condensible gas removal to less than 15 or 20 psig at -60 degrees Fahrenheit the remaining agent can be warmed and pumped into a larger vessel.

If electric power is available a FRICK® VRU or other appropriate pump can be used to evacuate the fire extinguisher into a chilled vessel, being careful not to deliver high pressure liquid phase agent into the VRU, to slightly below atmospheric pressure thereby increasing recovery efficiency beyond 90 percent.

12.4 MIL-M-12218C NON-CONDENSIBLE GAS SPECIFICATION

MIL-M-12218C addresses non-condensables as 1.5 percent maximum content in the vapor phase by volume without temperature or fill density reference. If the specification were changed to read: "10 psi maximum pressure indication at 70 degrees Fahrenheit beyond temperature indicated natural vapor pressure of 99.6 percent mole purity Halon 1301 when contained at a 75 pound per cubic foot fill density", there would result a maximum vessel pressure of 210 psig at 70 degrees Fahrenheit. Halon from such a vessel, when used for charging fire extinguisher containers, would result in a 6.0 psi maximum vapor phase partial pressure within the container being charged due to Henry's Law.

12.5 GUIDELINES FOR EVALUATING HALON RECLAMATION SYSTEMS

Evaluating Halon reclamation systems can be broken down into the following primary categories:

12.5.1 RECOVERY EFFICIENCY:

Normalize particular design for the recovery system being evaluated so that error associated with system contained agent charge is minimized. This can be performed by temperature, weight and pressure status within the component parts of the recovery system being evaluated.

Evacuate a test container to 2 psia (use absolute pressure gauge) and weigh container on a scale with a resolution accuracy of 0.002 (0.005 maximum) pounds. Make certain that all caps and fittings are weighed and that the test container is clean on the outside. This is WEIGHT 1.

Charge the container using the same scale calibrated to a known accuracy of 0.002 (0.005 maximum) pounds. Record the charge weight within the test container as AFTER removal of the charging line. This is WEIGHT 2.

Pressurize the test container using DRY nitrogen to a pressure know to be correct within at least 5 psi (preferably within 1 psi). Disconnect the charging line and weigh again. This is WEIGHT 3.

Recover the agent from the container using the recovery system being evaluated and weigh the container again. BE SURE THAT THE TEST CONTAINER FITTING IS TIGHT BEFORE REMOVING FROM RECOVERY SYSTEM HOSE CONNECTION. Weigh the container AFTER removing and drying any moisture that may have condensed on the test container surfaces. This is WEIGHT 4.

Percent charge removal from the test container can now be calculated as follows:

$$\left[1 - \left[\frac{(\text{WEIGHT 4} - \text{WEIGHT 1})}{(\text{WEIGHT 3} - \text{WEIGHT 1})} \right] \right] \text{X } 100 \qquad (4)$$

Evacuate the test container to 2 psia, weigh (WEIGHT 5), and check agreement of WEIGHT 5 with WEIGHT 1. ANY difference is charge loss or an error.

Process the charge as delivered from the test container through the recovery system according to the manufacturer's instructions to achieve output agent purity level conforming to MIL-M-12218C except for a maximum of 10 psi NCG content at 70 degrees Fahrenheit.

Recover agent from the output of the recovery system and place it back into the ORIGINAL test Container. Prior to weighing it, assure that the test container is dry and clean on all exterior surfaces. This is WEIGHT 6. Recovery efficiency can now be calculated as:

$$\frac{(\text{WEIGHT 6})}{(\text{WEIGHT 2})} \text{ X } 100 \qquad (5)$$

A recovery system design developed by Walter Kidde Aerospace called "Reach" attains an overall minimum recovery efficiency of 98 percent.

12.5.2 HALON 1301 RECOVERY IN PROCESS WATER DETECTION:

There are no suitable methods for detection of water to 10 ppm for Halon 1301 within a Halon recovery System. Water solubility within Halon is a function of temperature. Water indication devices typically used within refrigeration systems may be used as "indicators" within Halon recovery systems if they are placed in appropriate stream locations within those systems. Water solubility within all Halon compounds increases with increasing temperature; therefore, moisture indication devices which incorporate hydration indicator color change processes must be placed within the recovery system such that the lowest practical recovery stream temperatures, as determined by comparing solubility functions of the agent with those of the detection device, are realized during recovery. Water solubility in Halon 1301, saturation limit, varies from 95 ppm at 70 degrees Fahrenheit to 22 ppm at 0 degrees Fahrenheit. This usually means placing these devices at the suction or input side of the recovery system. These devices do not provide immediate indication upon encountering stream laden moisture content. In a Halon recovery system where media is not continuously circulating the actual indication due to color change delay can be masked by incoming agent flow. This results in being unable to obtain the full color change range for a given agent quantity. Interpreting these color change devices also relies on the operator's color acuteness and lighting in the area which may or may not be repeatable from day to day. Placing refrigeration type moisture indicators at pump output locations where compression heat is added to the recovery flowstream will increase the amount of moisture required to effect a color change indication.

Halon 1301 generally follows the moisture indication characteristics of Freon 12. These characteristics are:

Liquid Line Temperature	75°F	100°F	125°F
"DRY" indication	Below 5	Below 10	Below 20
"CAUTION" indication	5-15	10-30	20-50
"WET" indication	Above 15	Above 30	Above 50

Stream Moisture Content in PPM

The above data is from Sporlan Bulletin 70-10 on "See All" moisture and liquid indicators. These indicators provide good "indication" for stream moisture content. That is, a steady indication of dark green usually means that the sample, when analyzed by Karl Fischer titration, will be found to be below 10 ppm in moisture content.

12.5.3 HALON 1211 WATER SOLUBILITIES

Mr. John Isenberg of ICI supplied the following information on water solubility in Halon 1211.[9]

TABLE 28.

Temp	Percent Wt/Wt	Mole Fraction
+4°C	.007705	.0007068
+6°C	.007707	.0007070
+8°C	.007709	.0007070
+10°C	.007709	.0007071

Corresponding ppm values for each temperature above are: 77.05, 77.07, 77.07, and 77.09.

The equation presented in the text for water solubility in Halon 1211 is:

$$W_2 = 7.67620 \times 10^{-3} + 1.29997 \times 10^{-5} (T) - 1.78723 \times 10^{-6}(T2) + 8.12270 \times 10^{-8}(T3)$$

W_2 is Weight percent/Wt

T is Temperature degrees Centigrade

As can be seen by such a relationship, the saturated water content at 25°C is 81.5 ppm.

Due to the flat nature of the water solubility curve for Halon 1211 it is reasonable to expect adequate water content indication in Halon 1211 to be 20 ppm or less using refrigeration type moisture indicators.

12.5.4 AGENT PURITY DETERMINATION:

The most suitable and practical method for agent purity determination is that of Gas Chromatography (GC). Boiling point and boiling point range, 5 percent to 85 percent distilled, is satisfactory as an "indicator" for agent purity prior to GC testing. This is similar to that of moisture and water content indication by color change methods. Halons can be recovered to meet applicable MIL specification requirements, except for up to 10 psi non-condensible gas content at 70 degrees Fahrenheit. A 1 percent contamination level of Halon 1211 in Halon 1301 shifts the Halon 1301 boiling point at 5 percent distilled by .55 degrees Centigrade. (This is based upon boiling point shift as modeled by Raoult's law which has been shown to be true for non-azeotropic mixtures of Halons.) Atmospheric pressure change of 25mm Hg and other contaminants can account for this .5 degree shift. MIL-M-12218C requires a purity of 99.6 percent minimum. This requires a boiling point measurement and indication accuracy of .23 degrees Centigrade which requires a resolution significantly greater than that obtainable through conventional means.

12.5.5 NON-CONDENSIBLE GAS DETERMINATION:

Recovered agent can be adequately checked for 10 psi maximum NCG content at 70 degrees Fahrenheit (21 degrees Centigrade) by using a calibrated pressure gauge of 1/4 percent accuracy. The pressure gauge should have a scale range of 0 to 500 psig and be graduated in 2 psi or finer

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[9] This information was taken from a text entitled, "Halogenated Hydrocarbons, Solubility/Miscibility with Water", authored by Al Horvath, published by Marcel Dekker, New York. Copyright 1982.

increments. Agent vapor pressure may be calculated or found in applicable tables. Vessel temperature may be measured by using a surface contact thermocouple, measuring at three locations within the suspected liquid region and averaging the three temperature readings. A measured pressure which exceeds the 10 psi maximum temperature adjusted pressure (based on ideal gas law relationships) would be cause for designating that lot of Halon to be used in applications requiring additional nitrogen charge.

12.5.6 AGENT RECOVERY RATE:

All recovery equipment manufacturers should provide agent recovery rates which address typical or average operation. This rate should reference the time required from valve opening on the fire extinguisher from which the agent is being recovered until the fire extinguisher is removed from the recovery system. In order to be meaningful, the recovery rate should reference an efficiency level. This rate should be determined by dividing recovered agent weight by the amount of time that the fire extinguisher remained attached to the recovery system. Nitrogen weight component should not be included in the calculation since it is not used in the efficiency calculation.

12.6 RECLAIMED HALON CERTIFICATION - IS MIL SPEC TOO STRINGENT?

Boiling point and boiling point range testing can detect, with a high degree of certainty under controlled temperature test conditions, Halon 1301 and Halon 1211 contaminated with high boilers at a practical level of 1.5 percent. This results in purity minimums of 98.5 percent compared to MIL-M-12218C requirements of 99.6 percent. Moisture can be detected, under controlled temperature conditions, to 10 ppm in Halon 1301 and 20 ppm in Halon 1211 using standard refrigeration moisture indicators.

Agent that is dry, within reasonable limits, and that is pure within 98.5 percent may be adequate for use in aviation applications. Testing of such a scope is beyond this feasibility study.

It is possible to recover to MIL spec purity levels. It now becomes a question of methodology to determine purity. Boiling point and boiling range methods will release more agent to the atmosphere than GC methods; however, these tests provide a reasonable indicator of expected GC results. GC methods are more expensive; although, the end result is directly quantitative. A GC result of 99 percent and a boiling point 5 degrees above or below normal would be cause for retest. Likewise, a boiling point within .5 degrees Centigrade of normal does not agree with a GC result of above 100 percent (due to obvious over-injection errors) or below 98 percent. As long as Halon agents are recycled, standards will be available for equipment calibration.

Moisture determination in Halon compounds, although feasible by color change indication is risky. Subtle shift in color may mean a water content of 11 ppm or 9.8 ppm. Is 11 ppm too high? If it could cause discharge malfunction in an extended discharge inerting system it simply is not worth the risk. Perhaps two methods should be used. Initial check by color change methods to obtain a ballpark indication prior to performing Karl Fischer titration. In this manner obvious errors would be detected such as K-F titration indicating 30 ppm with no color change being noted in the indicator. The converse could also be true, a definite green to chartreuse color shift (using Sporlan 40-10) indicates moisture levels well above 10 ppm in Halon 1301. A Karl Fischer titration result of 5 ppm on the same sample would be cause enough to rerun the test.

With the proper equipment, basic house keeping skills and technical common sense, Halon recovery is practical.

13. CONCLUSIONS

1. Support of the U.S. Domestic Civil Fleet until 2020, considering worst case Halon 1301 accidental discharge, 5 year hydrotest inspection and fire extinguisher retirement, with no recycle of Halon 1301, is projected to require a total of 3,149 tons or approximately 4.95 percent of the 1993 Halon 1301 bank. Recovery of that agent removed for hydrotest inspection purposes from 1991 through 2020, approximately 1,564 tons, reduces demand on the 63,681 ton Halon 1301 bank by 2.46 percent, from 3,149 tons to 1,585 tons. This 1,585 ton requirement includes new production, agent loss due to discharge for fire and false alarm, and increased consumption from class change impact. This is based on 96 percent of all agent removed for hydrotest purposes being usable and available for recharge. The estimate of using a 96 percent minimum reusable quantity of agent removed from a typical Halon 1301 fire extinguisher, after quality determination, is practical and is currently attainable.

2. There is no difference in fire protection effectiveness or safety of use between recycled Halon 1301 and virgin manufactured Halon 1301 provided the recycled Halon 1301 meets specification requirements of MIL-M-12218C with an allowable 2 to 10 psi non-condensible gas content at 77 degrees Fahrenheit (25 degrees Centigrade). Halon 1301 can currently be recovered and conditioned at 98 percent recovery efficiency, using standard refrigeration techniques, to the purity requirements of MIL-M-12218C except for 2 to 10 psi non-condensible gas content at 70 degrees Fahrenheit.

3. Non-aviation Halon 1301 will be no different with regard to reclamation processing than Halon 1301 presently banked in aviation systems.

4. Certification of reclaimed Halon 1301 for reuse is best performed using a Gas Chromatograph for purity determination and a Karl Fischer Titrator for moisture determination.

5. Approximately 109 tons of Halon 1301 is banked within fixed systems of the current U.S. Civil Aviation Fleet.

6. Estimated U.S. Civil Fleet Halon 1301 consumption due to discharge from 1991 - 2000 is approximately 69 tons. The impact of compartment class change and operation increases Halon 1301 consumption due to discharge by 16 tons from passenger aircraft and by 9 tons from freighter aircraft. The impact of the 5 year hydrotest inspection requirement increases discharge by an additional 22 tons per year from 1991 through 1995. Fleet growth from 1991 to 2000 increases Halon 1301 discharge due to the 5 year hydrotest inspection requirement from the 22 ton per year requirement to an average requirement of 40 tons per year beginning in 1996.

7. In support of newly produced aircraft from 1991 to 2000 approximately 240 tons of Halon 1301 will be required. If compartment class redesignation and operation from Class D to Class C occurs an additional 34 tons of Halon 1301 will be required. Commuter fleet requirements for this same ten year time period is estimated at 19.64 tons for new aircraft and 10 tons due to hydrotest inspection..

8. It is estimated that there will be 57,771 tonnes (63,547 tons) of Halon 1301 in the world bank by the end of 1993 which could be available for recycling in support of the U.S. Domestic Civil Fleet.

9. Excluding false alarm discharge and assuming all Halon 1301 removed for hydrotest inspection is reclaimed at a realizable efficiency of 96 percent, Halon 1301 required to support the U.S. civil fleet until 2020 is approximately 1,476 tons or 2.32 percent of the estimated world bank of Halon 1301 in 1994 of 57,771 tonnes (63,680 tons).

14. REFERENCES

1. An Introduction to Probability Theory and Its Applications, William Feller, Vol. I, 2nd Edition, John Wiley & Sons, Inc.

2. U.S. Department of Transportation, Federal Aviation Administration Order 8000.40B; Subj: Maintenance of Pressure Vessels in use as Aircraft Equipment.

3. "Outlook for Commercial Aircraft 1988 - 2002", McDonnell Douglas, Feb. 1989

4. Equation taken from DuPont computer program "SUPER" which is used for calculating pressure-temperature characteristics of Halon 1301 and nitrogen.

5. DuPont FE-2A Manual "Handling and Transferring DuPont Halon 1301 Fire Extinguishant".

6. DOT 49CFR 173.304, Chapter 1.

7. Estimate developed by Walter Kidde Aerospace, Inc. based upon data from References 1 and 2 and requirements set forth in U.S. DOT, FAA Order 8000.40B dated 10/18/90. This order rescinded Order 8000.40A.

8. Halogenated Hydrocarbons, Solubility/Miscibility With Water, Al Horvath, Marcel Dekker, New York. Copyright 1982.

9. Data relating to total Halon 1301 production and availability used for this report is taken from two publications: (1) *Halon Bank Management - A Rationale To Evaluate Future World Supplies by Gary Taylor of Taylor/Wagner Inc. and Temothy Leah of Environment Canada* and (2) *The Past and Future of Halons: An Industry Perspective by Archie McCulloch of ICI Chemicals & Polymers Ltd.*

10. FAA Report FAA-DS-70-3 ("Criteria for Aircraft Installation and Utilization of an Extinguishing Agent Concentration Recorder") states that in engine nacelles with several fire zones separated by an adequate firewall, each fire zone may be treated individually with respect to the simultaneous requirement.

11. "Airbus: 10,000 Jets by Century's End", Arthur Reed, Air Transport World Magazine, May 1990.

APPENDIX A

GLOSSARY

APU	Abbreviation used in the airline industry for Auxiliary Power Unit. A small turbine powered generator which supplies electrical energy to an aircraft for ground operation and engine start-up.
ATA	Abbreviation for Air Transport Association.
Azeotrope	Azeotrope, or Azeotropic mixture, as involved with Halon fire suppressing substances, refers to a common phase liquid state mixture of two or more substances, in this context Halons, which behaves like a single substance in that the vapor produced by partial evaporation of liquid has a fixed composition as all liquids in the mixture remain present. The mixture must be of constant boiling point character throughout its distillation range and exhibit a maximum or minimum boiling point as compared with that of other mixtures of the same substances.
Boiling Point	Liquid temperature at which vapor pressure from boiling liquid phase is slightly greater than surrounding total pressure. Generally approximated by noting temperature at which bubbles of vapor are formed within liquid body as its temperature is gradually increased. Precise determination of boiling point temperature is more complicated and requires special methods due to liquid superheating effects, dissolved gasses, and other properties.
Boiling Point Range, 5% to 85% Distilled	Volumetric reference for determining boiling point stability through a specified liquid evaporation range. For non-azeotropic mixtures the boiling point temperature will change as components of the mixture vaporize at different rates. Generally, low boilers vaporize first which results in the boiling point increasing in temperature as liquid phase quantity decreases. A method for approximating contaminants within refrigerants by change in boiling point as 100cc vaporizes from a liquid quantity of 95cc to 15cc. These relationships generally follow Raoult's Law.
Dalton's Law	The partial pressure of a gas or vapor (as in vapor pressure) in a perfect gaseous mixture is equal to its mole fraction in the mixture multiplied by the total pressure. On a molecular basis, within a gas mixture the molecules of gas of each kind exert the same pressure as they would if they were present alone, and that the total pressure is the arithmetic sum of the partial pressures exerted by the different gases in the mixture. This relationship was discovered by John Dalton (1766-1844) in 1805
DOT	Abbreviation for Department of Transportation.
Event	As used in reporting aircraft situations, an event is defined as a single reportable situation or occurrence outside that of "normal" safe aircraft operation.
FAA	Abbreviation for Federal Aviation Administration
Fill Density	As relating to the charging of Halon containing fire extinguisher, fill density is defined as the total weight of agent (i.e., Halon 1301) per unit internal volume of the container. For example, if 150 pounds of Halon 1301 are placed into a 2 cubic foot container, the fill density would be 150 pounds divided by 2 cubic feet or 75 pounds per cubic foot. This considers the TO-

	TAL weight of substance regardless of its existence as liquid or vapor phase. The fill density has no relationship whatever to percent liquid fill unless a specific temperature and nitrogen charge are referenced.
Fill Ratio	The ratio of the amount of material actually contained (or to be contained) in a vessel to the amount which could be contained if the vessel were completely full of liquid is termed "fill ratio". Since this term is dependant upon agent liquid density, a reference temperature must also be stated. At 70 degrees Fahrenheit, Halon 1301 has a liquid density of 97.790 pounds per cubic foot, therefore the fill ratio expressed as percent, is very nearly numerically equal to the fill density. Halon 1301 has a density of 100.0 pounds per cubic foot at 60.85 degrees Fahrenheit.
Filter-Drier	A device commonly used within the refrigeration industry to remove particulate matter and water from refrigerants. Usually constructed of a steel shell, containing fiberglass or similar media for filtration purposes, carbon for hydrocarbon adsorption, and alumina and molecular sieve materials for water capture.
FIREX	Acronym for FIRE EXtinguisher
Halon	Generic term which represents those halogenated hydrocarbons used for the purpose of fire suppression. In this report Halon refers specifically to Halon 1211 and Halon 1301.
Halon 1211	BromoChlorodiFluoromethane, or commonly referred to as BCF as trademarked by Imperial Chemical Industries Limited for their liquefied gas fire suppressant.
Halon 1301	Bromotrifluoromethane. A liquified gas fire suppressant known for its low toxicity, high suppression efficiency, and wide temperature range of application. Properties of this agent are presented in Section 12.
Henry's Law	A physical property. The solubility of a gas in a liquid is greatly increased by an increase in pressure. At low pressures it is directly proportional to the pressure of the gas. If the gas is a mixture, the solubility of each substance in the gas mixture is separately proportional to the partial pressure component of each gas. Properties of this nature are usually defined in terms as PSI per MOLE FRACTION. Which means that one MOLE of subject gas will become dissolved into one MOLE of subject LIQUID phase substance at a given temperature and pressure. This law was discovered in 1803 by British chemist William Henry (1774-1836). The equation for nitrogen solubility within Halon 1301 and 1211 will be found in the list of equations in APPENDIX C.
MOLE	A unit quantity in chemistry. An amount of a substance in grams (gram mole) or pounds (pound mole) which corresponds to the sum of the atomic weights of all the atoms composing the molecule. When calculations are made dealing with gas solubilities within liquids and boiling point modification based upon Raoult's Laws or similar chemical properties all calculations MUST be performed using MOLE relationships. One gram MOLE of any substance within the realm of Classical Physical Chemistry contains one Avogadro Number of molecules which is $6.02252 \text{ X } 10^{23}$ molecules plus or minus approximately 1 percent. This means that 149.9 grams (one gram mole) of Halon 1301 contains approximately the same number of molecules as 28 grams (one gram mole) of nitrogen.

Nitronic 40	A 21-6-9 stainless steel typically comprised of 21 percent chromium, 6 percent nickel, 9 percent manganese with trace components of carbon, phosphorus, Sulfur, and silicon (1 percent). Nitronic 40 is the preferred material of choice for aircraft fire extinguishers since it offers superior formability, weight savings, strength, fatigue life, and stress-strain characteristics significantly beyond those of the 300 series stainless steels.
Non-azeotropic	Refers to a common phased liquid state mixture of two or more substances, in this context Halons, which does NOT behave like a single substance in that the vapor produced by partial evaporation of liquid demonstrates different property and composition from the mixture of liquids. The vapor composition is directly related to liquid state composition and changes as the liquid mixture changes.
Percent Liquid Fill	The percentage of the container volume occupied by liquid at a specified temperature is called the "percent liquid fill". For liquefied compressed gases, the total weight of material is distributed between the liquid phase and vapor phase according to temperature, fill density, and liquid and vapor densities. The percent liquid fill (for pure Halon 1301 only without dissolved gas content) is calculated from equations found in APPENDIX C.
Percent of Water Capacity	Water capacity is a common means of designating the capacity of a pressure vessel, and is expressed as water weight required to fill subject vessel. Percent water capacity is simply the ratio of agent weight to be put into subject vessel to the weight of water which can be put into the same vessel. Water density is temperature dependant, so the percent of water capacity varies with temperature. Water density at 60 degrees Fahrenheit is 62.2995 pounds per cubic foot which is reference condition for Department of Transportation regulation.
PPM	Abbreviation for Parts Per Million. Usually as referring to a population or concentration of components OTHER than those associated with a pure substance. One part per million is a distance of one foot in a million feet or 189.394 miles or (in a ppm by weight reference) one drop of water (.05 grams water) in 50,000 grams, or 110.23 pounds, of Halon 1301. In a ppm by volume reference, one ppm is one drop of water in 1.765733 cubic feet or at 70 degrees Fahrenheit 3051.19 cubic inches (174.28 pounds) Halon 1301.
Raoult's Law	A French Chemist, Francois Raoult (1830-1901) in 1883 discovered that weight-molar freezing point lowering produced by different solutes is the same for a given solvent. This useful discovery has many implications. Raoult's Law, derived from this discovery states that the vapor pressure of a substance, in equilibrium with a solution containing the substance, is equal to the product of the mole fraction of the substance in the solution, and the vapor pressure of the pure substance at the temperature of the solution. This law is NOT applicable to MOST solutions but is often a close approximation for non-azeotropic mixtures of closely similar substances, and in particular for substances present in high concentration.
SDR	Abbreviation for Service Difficulty Report. A reporting system and format established and used by the FAA.
SIS	Abbreviation for Safety Information System. The name of a McDonnell Douglas maintained, Proprietary Data Base that captures aircraft incident data and event reports.

Ton	Represents a U.S. weight of 2,000 avoirdupois pounds, commonly called a short ton.
Tonne	Represents a metric ton weight of 2,204.5 pounds.
Vapor Pressure	The pressure characteristic at any given temperature of a vapor in equilibrium with its liquid or solid form.

APPENDIX B

PHOTOGRAPHIC DATA REPORT
OF
A PUMPLESS HALON 1301
RECOVERY AND RECYCLING
SYSTEM USED AND DEVELOPED
BY
CANADIAN AIRLINES INTERNATIONAL

APPENDIX B FIGURES

PHOTOGRAPHIC DATA REPORT
OF
A PUMPLESS HALON 1301
RECOVERY AND RECYCLING
SYSTEM USED AND DEVELOPED
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CANADIAN AIRLINES INTERNATIONAL

03 JANUARY 1991

PREPARED BY	APPROVED BY
Mark D. Mitchell Senior Project Engineer	Robert E. Glaser Vice President Engineering

TABLE OF CONTENTS

1.0 INTRODUCTION

This report offers a photographic review of a unique and low cost practicle method of recovering up to a theoretical maximum of 96% of Halon 1301 from Aerospace type fire extinguishers. Although the apparatus and methods demonstrated here limit recovery mass to a practicle limit of approximately 30 pounds per cylinder (using a 678 cubic inch scuba tank), the method is valid for larger quantities where a larger recovery cylinder would be used. This report contains in Appendix A Canadian Airlines International Engineering Report number 99-26R-1066. Appendix B contains the Walter Kidde Aerospace, Inc., visitation report to CAI on 08 and 09 November 1990.

2.0 PURPOSE

This report illustrates that Halon 1301 can be recovered from Aerospace fire extinguishers using chilling methods to burp off Nitrogen from a Halon 1301 solution containing nitrogen.

3.0 MATHEMATICAL MODELS

The equations presented in this document are approximations and are not absolutely correct and do not represent ZERO ERROR models of the natural phenomena portrayed by them. The approximations are adequate for the purposes intended. The equations are conservative (relative to empirical tabulations) such that the vapor pressure (which is from DuPont) equation is, at worst case, no more than 1.7% HIGHER than actual. This equates to a maximum error of 10 psi at the Halon 1301 critical temperature of 153 degrees F. This type of agreement between model, experimental and actual data is considered excellent.

Within the pressure ranges encountered in Aerospace fire extinguishers, at near-ambient temperatures which are 360 to 1000 psig, the nitrogen density relationship (page 11 of 17 in the CAI Engineering Report) is as much as 3.2% in error at 1000 psig. This error references data from "TABLES OF THERMAL PROPERTIES OF GASES", US Dept. of Commerce, NBS Circular 564, November 1, 1955. This is; however, acceptable since the fire extinguisher bottles are charged by NOMINAL AGENT weight and pressurized to a specified charge pressure at temperature.

The Nitrogen weight approximation equation is designed for use with Halon 1301 charge tolerances in mind. REMEMBER, the equation works with specific density properties of Halon 1301 and Nitrogen in BOTH, VAPOR and LIQUID, phases of Halon 1301. Attempting to "try" the equation using input data that could not possibly exist in the "real world" will result in erroneous results. Below is a table comparing three calculation methods for determining the nitrogen content of various Halon 1301 and nitrogen conditions. The columns are designated as follows:

DuPont -- Known nitrogen content from Dupont tables.
Super -- Calculated nitrogen content from a DuPont computer program named "super".
CALC -----Calculated from the N2w equation presented in Jim Hamilton's report.

Container Volume (70 deg F)	Pounds of Nitrogen in Container		
Nitrogen pressure and Charge	DuPont TABLES	Super PROGRAM	CALC EQUATION
630 cu in, 625 psig, 21 lbs	0.6916	0.6849	0.673
2500 cu in, 360 psig, 105 lbs	1.0900	0.9956	1.020
60 cu in, 600 psig, 1 lb	0.0649	0.0677	0.066
630 cu in, 800 psig, 21 lbs	0.9760	0.9670	0.890

4.0 USE OF MODEL FOR FIRE EXTINGUISHER CHARGING

When charging a Halon 1301 fire extinguisher it is necessary to accurately charge them with agent. Excessive agent will result in above normal pressures and cause premature thermal relief which is unacceptable. Charging below nominal agent weight is unacceptable since this sacrifices discharge concentration safety margin and suppression efficiency.

Charging a fire extinguisher with Halon 1301 containing Nitrogen is performed as follows:

FIRST: Charge to NOMINAL specification agent weight as indicated by the fire extinguisher data plate.

SECOND: Record pressure, weight and temperature of the fire extinguisher.

THIRD: Calculate the nitrogen content weight using the equation given for N2w. Consider the following example:

Volume of FIREX	Charge Weight	Pressure	Temperature
630 cu in	18 pounds	291 psig	83 deg F

From this data the 291 psig becomes 291+14.7 = 305.7 psia

The 83 deg F becomes 83 + 460 = 543 degrees R (Rankine).

Equations for calculating the following will be found on page 10 of 17 within the Canadian Airlines International Engineering Report. This report is in Appendix A.

Vapor Pressure of Halon 1301 at 83 deg F is 254 psia.

Entering the above data into the N2w equation:

Tp=305.7 psia   Vp=254 psia   TR=543   Cw=18   Cv=630

We then yield a nitrogen content of .085 pounds or 39 grams.

This amount is less than 1% of the charge weight. In this container the charge tolerance of +.25 / -0 pounds compensates for the .085 pounds of nitrogen which would otherwise be agent if no nitrogen were present. In this example the container can be charged to maximum specification agent weight without calculating nitrogen content. It is wise, however; to check the nitrogen content in each case. Generally, a total pressure below 300 psig at 70 degrees F or higher contains a nitrogen content that could be ignored.

5.0 PRACTICALITIES

The previous example demonstrates that a 50 psi partial pressure of nitrogen is, for practicle purposes, negligible with regard to Halon 1301 Aerospace fire extinguisher charging. If a 50 psi or less Nitrogen partial pressure exists in a container of recycled Halon 1301, weight compensation can be made by simply charging to maximum charge tolerance without the need of calculation for nitrogen weight. Of course, vapor pressure - temperature tables for Halon 1301 or performing the vapor pressure calculation will be required in order to determine nitrogen partial pressure content.

Container volume as well as partial pressure determines nitrogen weight component in a fire extinguisher pressure vessel. Below is a table for a 50 cubic inch and 630 cubic inch fire extinguisher that lists nitrogen weight component versus partial pressure. This table demonstrates significance of both volume and partial pressure.

VOLUME CU IN	PARTIAL PRESSURE NITROGEN PSI	AGENT CHARGE POUNDS	NITROGEN WEIGHT
50	100	2.0	0.013
50	400	2.0	0.048
630	50	18.0	0.085
630	100	18.0	0.170
630	400	18.0	0.660

FIGURE B-1 Standard 678 Cubic Inch SCUBA Tank With Fittings

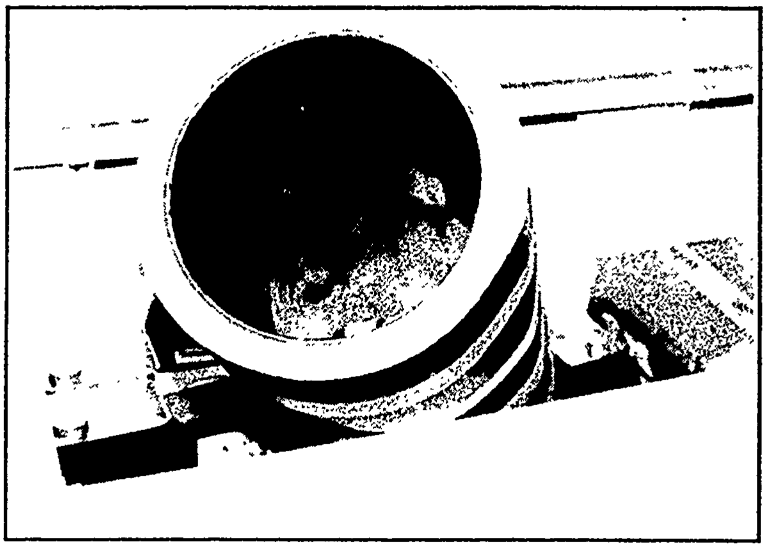

FIGURE B-2 347 Stainless Steel Insulated Enclosing Cylinder from Freezer for Dry Ice and Alcohol Bath

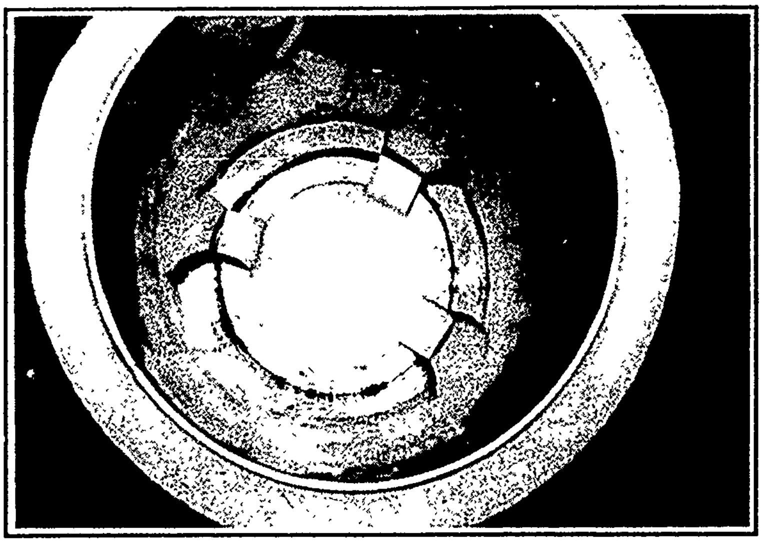

FIGURE B-3 Enclosing Cylinder with Lower Insulating Material Installed

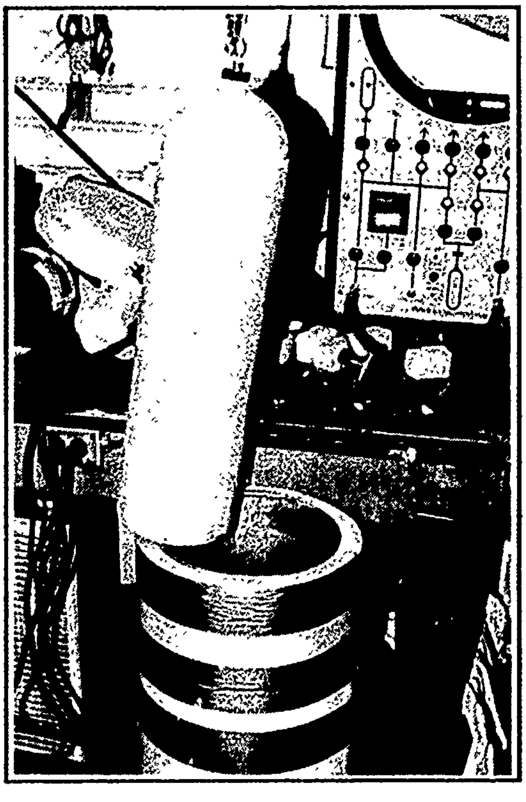

FIGURE B-4 SCUBA Tank Positioned Prior to Placement into Enclosing Cylinder

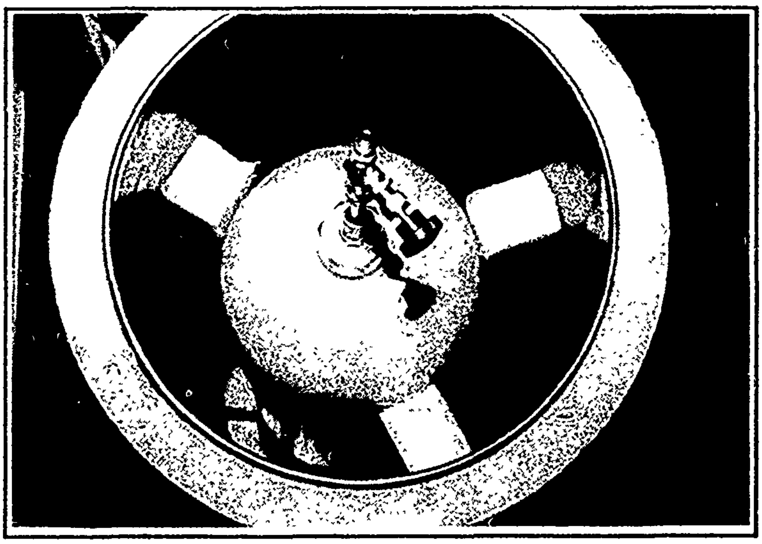

FIGURE B-5 SCUBA Tank in Cylinder with Later Insulation Retainers

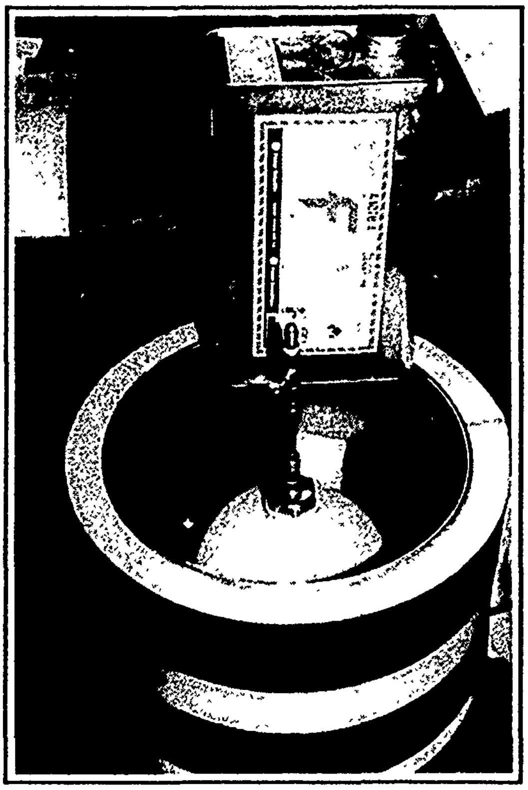

FIGURE B-6 Preparing to Add 4 Liters of Denatured Alcohol - 85% Ethanol, 15% Methanol

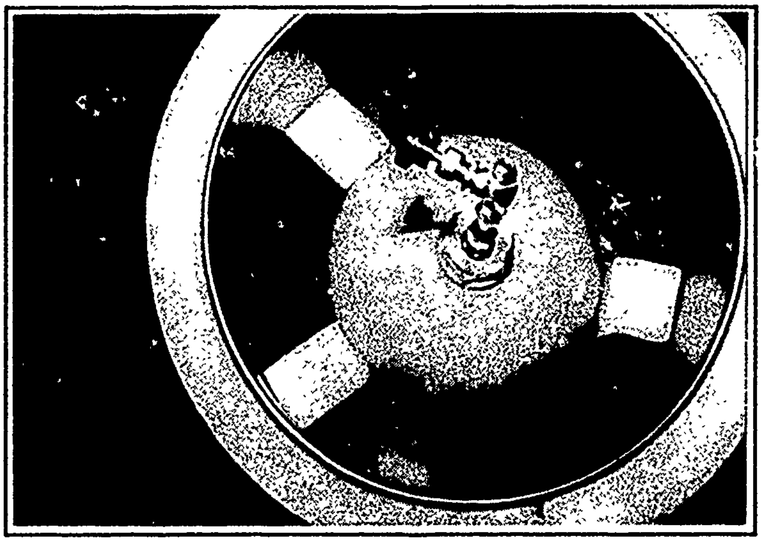

FIGURE B-7 Two Supports Float to Surface After Adding Some Alcohol

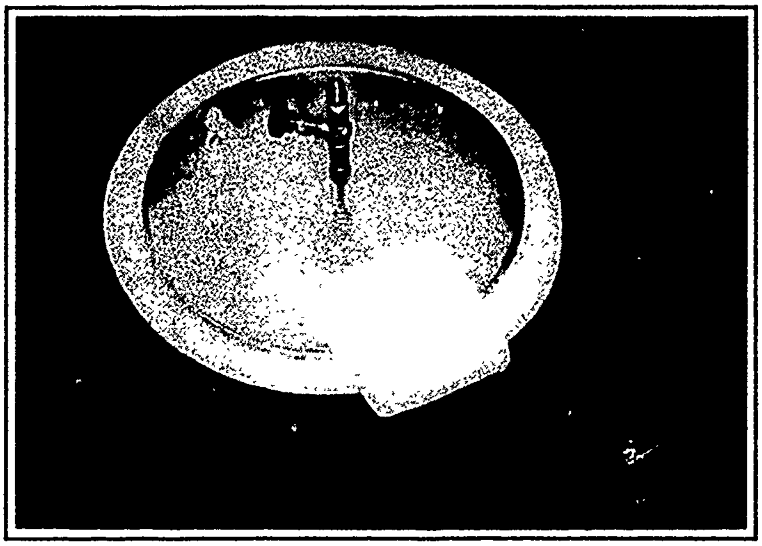

FIGURE B-8 Dry Ice is Added Cautiously to Prevent Burp Out

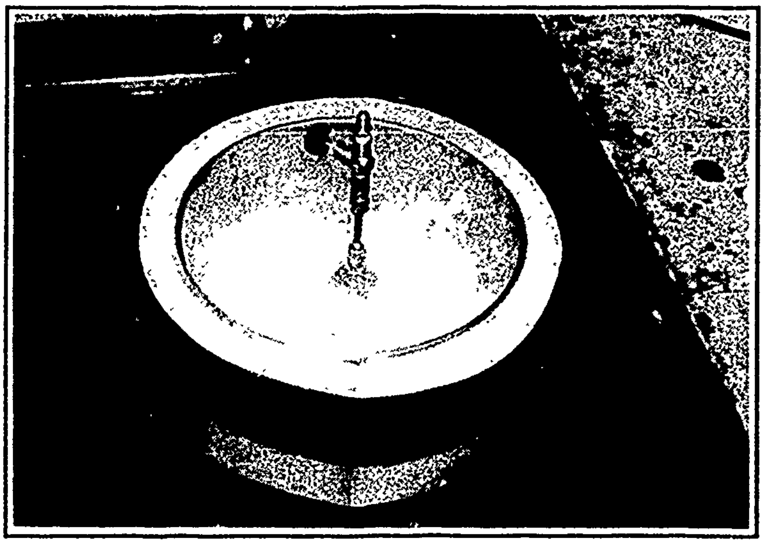

FIGURE B-9 As Heat Transfer Stabilizes Agitation Decreases. About 5 Minutes Following First Group of Bricks.

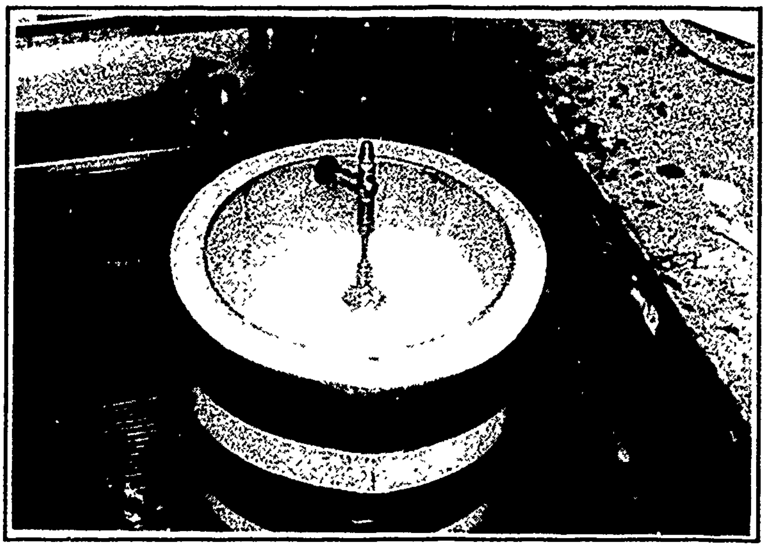

FIGURE B-10 About 10 minutes after the Addition of the First Dry Ice Brick

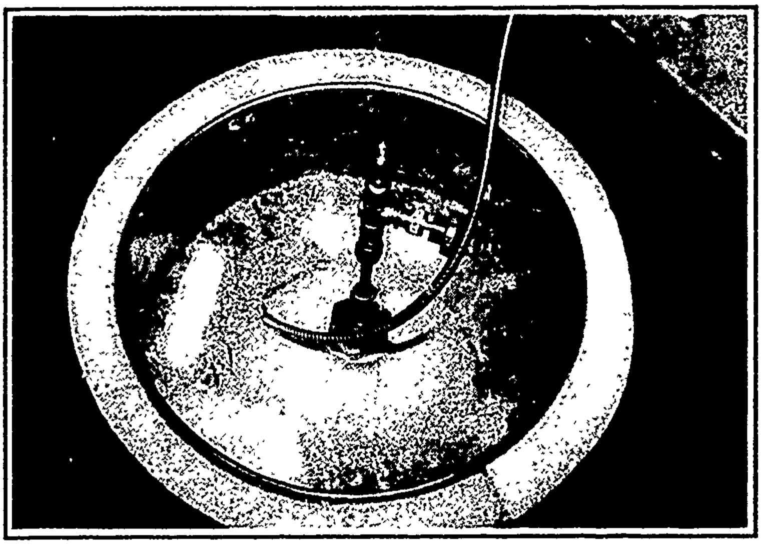

FIGURE B-11 15 Minutes After First Dry Ice Bricks Are Added A Temperature Probe Is Inserted

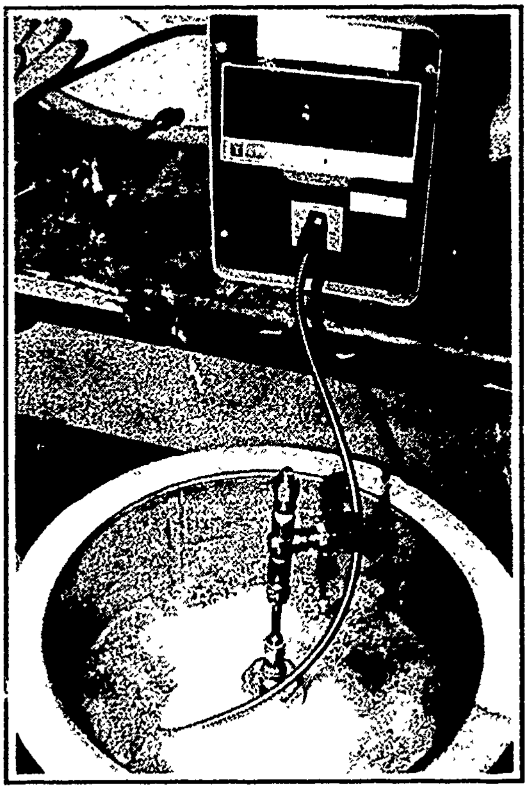

FIGURE B-12 As Dry Ice Sublimes and Solution Cools The Liquid Level Changes

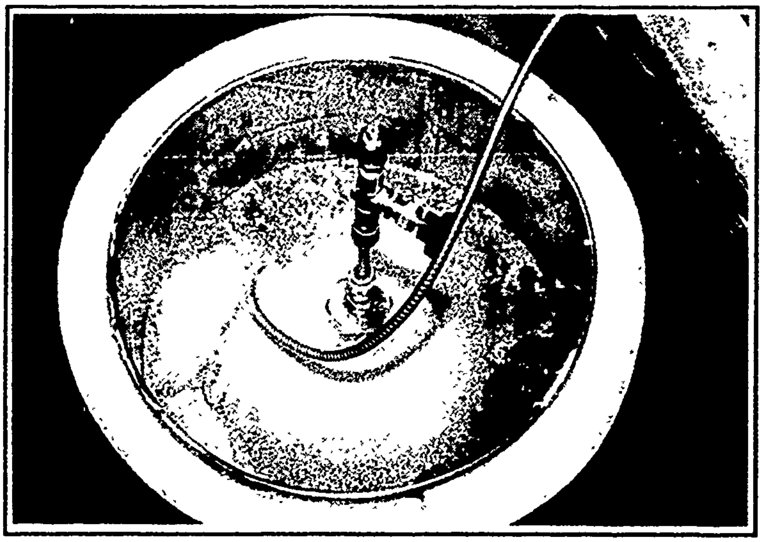

FIGURE B-13 About 30 Minutes Following Initial Dry Ice Introduction SCUBA Tank is at -80 Degrees Centigrade

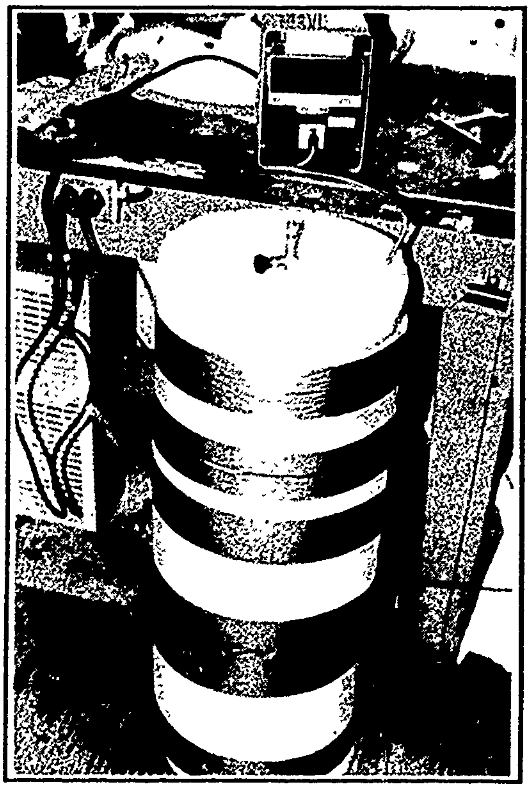

FIGURE B-14 347 Stainless Steel Insulated Enclosing Cylinder With SCUBA Is Capped to Decrease Heat Loss and Help Hold SCUBA

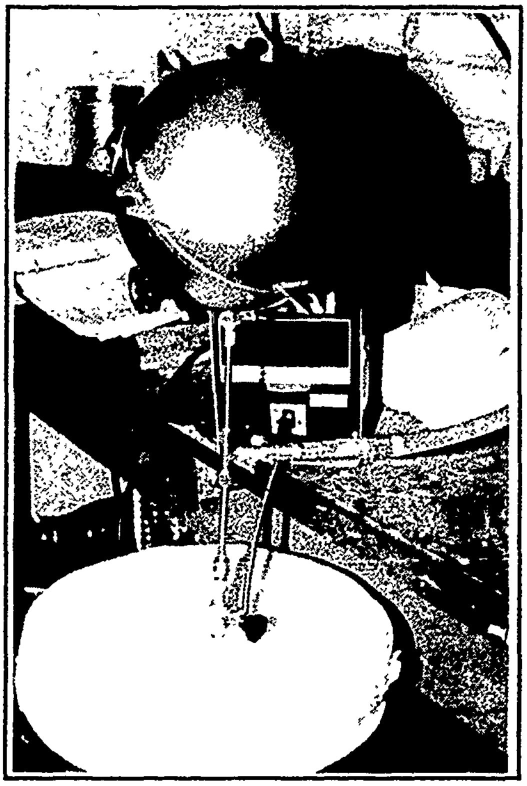

FIGURE B-15 Fire Extinguisher is Connected (224 Cu In) For Recovery into SCUBA

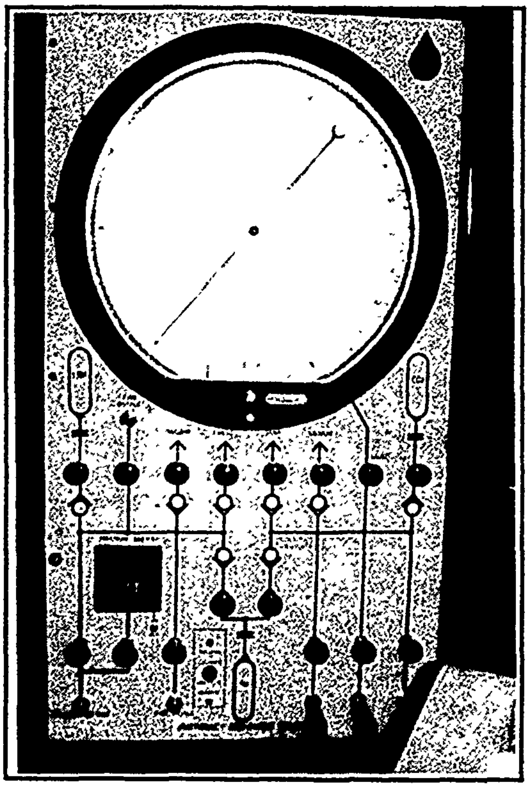

FIGURE B-16 Canadian Airlines International Designed Control Panel For Recovery and Recharge

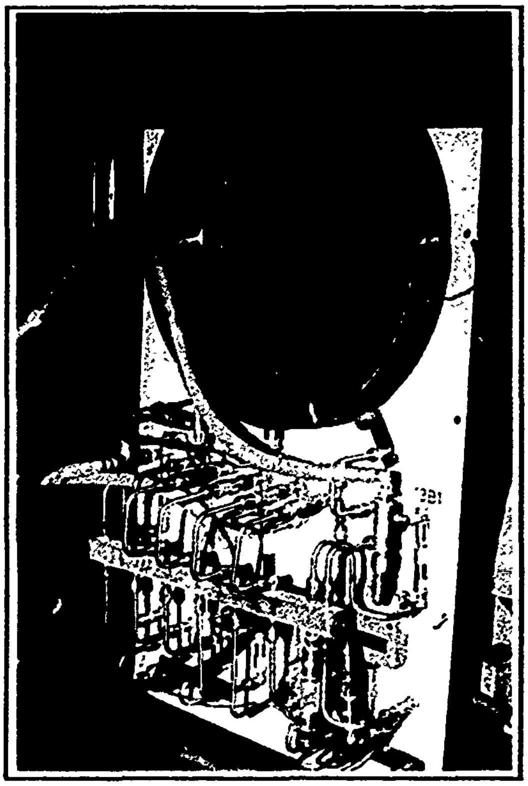

FIGURE B-17 Interior of Control System Illustrating Simplicity and Practicality of Design

FIRST APPENDIX

Canadian Airlines International
Engineering Report 95-2[illegible] [illegible]066

CANADIAN AIRLINES INTERNATIONAL

ENGINEERING REPORT

DATE: 01 Feb 90 **REPORT NUMBER:** 99-26R-1066 **PAGE:** 1 of 17

ISSUE DATE: 29-Nov-90 **ISSUE:** A

SUBJECT: HALON 1301 RECOVERY SYSTEM

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CANADIAN AIRLINES INTERNATIONAL ENGINEERING REPORT

DATE: 01 Feb 90 **REPORT NUMBER:** 99-26R-1066 **PAGE:** 2 of 17

ISSUE DATE: 29-Nov-90 **ISSUE:** A

SUBJECT: HALON 1301 RECOVERY SYSTEM

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CANADIAN AIRLINES INTERNATIONAL ENGINEERING REPORT

DATE: 01 Feb 90 **REPORT NUMBER:** 99-26R-1066 **PAGE:** 3 of 17

ISSUE DATE: 29-Nov-90 **ISSUE:** A

SUBJECT: HALON 1301 RECOVERY SYSTEM

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DATE:	01 Feb 90	**REPORT NUMBER:**	99-26R-1066	**PAGE:** 5 of 17
ISSUE DATE:	29-Nov-90.		**ISSUE:** A	
SUBJECT:	HALON 1301 RECOVERY SYSTEM			

1.0 OBJECTIVE:

To prevent the discharging of Halon 1301 to atmosphere. To reclaim and reuse Halon 1301.

2.0 BACKGROUND:

Halon 1301 is currently used in the Engine, APU, and Cargo compartment fire extinguishers. Periodically, these units are returned for maintenance. Depending on the degree of maintenance required, the unit may have to be discharged. At present, all units serviced in-house are discharged to atmosphere.

The fire extinguishers are filled in the following manner:

.01 Place evacuated extinguisher on weigh scale.

.02 Fill, through gravity feed, the required amount by weight of Halon 1301.

.03 Pressurize Halon 1301 to 600 psig with Nitrogen.

3.0 SEPARATION OF HALON 1301 FROM HALON/NITROGEN MIXTURE:

To reclaim the Halon from a filled extinguisher, the Nitrogen must first be removed. This can be accomplished by liquifying the Halon and venting the Nitrogen to atmosphere.

At normal room temperature and atmospheric pressure, Halon 1301 exist as a gas. Liquefaction will occur at pressures in excess of the vapour pressure at room temperature 198.9 psig or at temperatures below the boiling point at 1 atmosphere (-57:8°C).

The extinguishers generally come back for service with internal pressures in excess of 198.9 psig. At these pressures, the Halon is liquified. The remaining contents of the cylinder consist of Nitrogen gas mixed with Halon vapour.

Initially, in-house attempts were made to remove the bulk of the Nitrogen from the liquified Halon by venting the cylinder down to the vapour pressure of Halon at room temperature. Although successful, the recovery rate of the Halon was below 60%. This procedure allowed for too much of the Halon to be vaporized and subsequently vented off with the Nitrogen. Recovery of the Halon while under pressure was not considered practical.

The alternative approach was to reduce the temperature of the Halon/Nitrogen mixture to below the boiling point of Halon (-57.8°C) at atmospheric pressure. This was done in the following manner:

Apparatus: (Ref. Figure 1)

1. A standard 3000 psig, 678 cubic inch aluminium scuba tank.
2. A water tight insulated cylinder, 11" ID X 30.25" deep with lid. (cooling container for scuba tank)
3. Approximately 5 gallons of denatured alcohol.
4. Approximately 18 lbs of dry ice (used with alcohol to cool scuba tank to -80°C).
5. A vacuum source (to evacuate air from required plumbing and cylinders).
6. A one way check valve (for venting scuba tank to atmosphere).
7. 0 to 500 psig gauge (to monitor scuba tank pressure).
8. Miscellaneous hardware (valves, hose, and fittings).

Procedure:

1. Submerge an evacuated aluminium scuba tank in a bath of denatured alcohol and dry ice (or liquid nitrogen in place of dry ice). Leave only the valve stem showing. Allow tank to cool for approximately 20 minutes.

2. Take aircraft fire extinguisher and shake. Connect extinguisher to top of reclaiming tank. The extinguisher must be oriented with the valve on the bottom. In this orientation, the pressurized nitrogen will help push the liquid Halon out.

   Evacuate shop air from interconnecting hardware. Discharge the contents of the fire extinguisher into the supercooled aluminium tank.

3. Heat the extinguisher to 30°C with a hot air blower. This will help push the remaining Halon out.

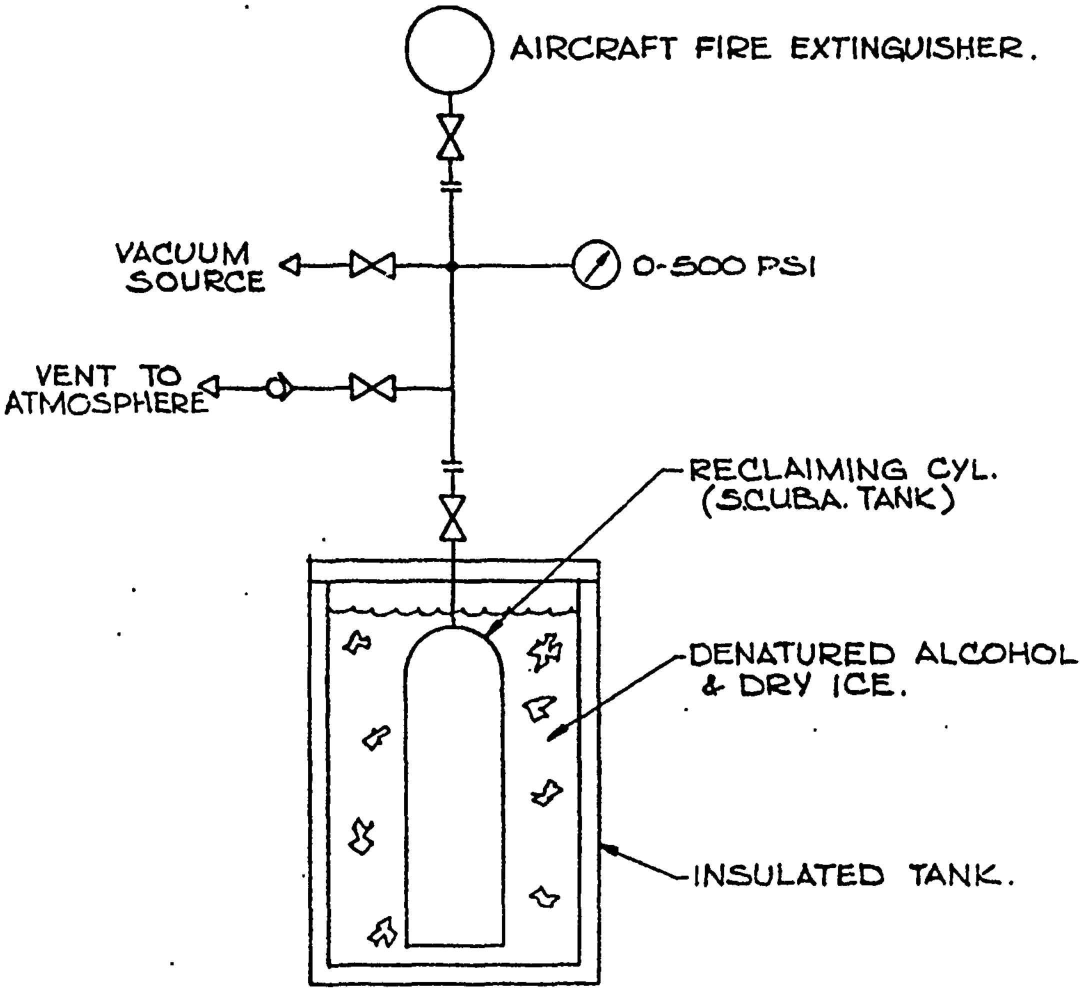
   Allow 15 minutes for the Halon to condense in the supercooled tank. The denatured alcohol/dry ice mixture will cool the contents of the aluminium tank to minus 80°C. At this temperature, Halon 1301 is a liquid at atmospheric pressure.

4. Discharge remaining contents of aircraft extinguisher to atmosphere. Remove extinguisher.

5. Through a one-way check valve, vent the top of the reclaiming tank to atmosphere.

   Venting the reclaiming tank after each discharge will allow for numerous extinguishers to be discharged into the aluminium tank with minimal resistance.

The solubility of Nitrogen into liquid Halon is a function of pressure as well as temperature. By condensing the Halon and by venting the top to atmospheric pressure, the bulk of the Nitrogen will escape.



HALON RECOVERY SYSTEM FIG. 1

(THIS PAGE REVISED
By M. MITCHELL)
N^2 15 JAN 91

4.0 FORMULAS FOR DETERMINING RATIO OF HALON TO NITROGEN

The following formulae were derived by Mark Mitchell of Walter Kidde Aerospace, Inc. Walter Kidde has been commissioned by the United States Federal Aviation Authority to investigate and prepare a report on the recovery and reuse of Halon for aircraft use.

Nitrogen weight in Halon/Nitrogen mixture:

The following formula is relevant for cylinder pressure above the vapour pressure of Halon between 0°F and 130°F.

$$N_2w = (0.00148148 \times (Tp - Vp) / (T_R) - 1.94213 \times 10^{-9} \times (Cw/Cv)^{2.67} \times Tp^{3.10}) \times Cv$$

Where,
N_2w = Weight of Nitrogen (lbs)
Tp = Cylinder Pressure (Psia)
Vp = Vapour Pressure at Temperature according to Halon vapour pressure equation (Psia)
T_R = Temperature of cylinder in Degrees Rankine
Cw = Net weight of contents (lbs)
Cv = Internal cylinder volume (in^3)

Halon Vapour Pressure: (FROM DUPONT PROGRAM)

$$Vp = 10^{\left(\frac{-44295}{T_R^2} - \frac{1795.678}{T_R} + 7.322506 - .00438339 \times T_R + 3.1258 \times 10^{-6} \times T_R^2\right)}$$

Nitrogen Density:

Density of N_2 = Do X.($T_{R}o$ / T_R) X (P / Po)

Where,
T_{Ro} = 492 °R
T_R = Nitrogen Temperature (°R)
Po = 14.696 Psia.
P = Nitrogen Pressure (Psia)
Do = .07807 lb/ft^3

1 lb. = 453.6 grams

5.0 HALON RECOVERY

Halon was recovered from two aircraft fire extinguishers.

The recovery was conducted on November 8, 1990 at Canadian Airlines Operation Centre in Vancouver, B.C.

#1 Aircraft Fire Extinguisher:
(224 in^3 Apco unit)

Evacuated Weight = 2060 grams
Evacuated to minus 12 psig

Internal Volume = 224 in^3

The extinguisher was filled and charged in the shop:

Halon 1301 charge = 1610 grams
Nitrogen charge = 120 grams

-Total Weight = 3790 grams

The extinguisher was than discharged to supercooled evacuated reclaiming tank.

Weight of ext. after discharge = 2180 grams

Weight of Halon and Nitrogen remaining in extinguisher at 85 psig = 120 grams

30 grams Nitrogen (calculated)
90 grams Halon (calculated)

After 15 minutes, the recovery tank was vented to 2 psig. 2 psig is the spring load on the one way check valve.

#2 Aircraft Fire Extinguisher:
(72 in^3 Apco unit)

This extinguisher required reclaiming because of a slow leak through the pressure gauge.

Internal Volume = 72 in^3
Initial Pressure = 293 psig
Initial Weight = 1415 grams
Final Weight = 1125 grams
Final Pressure = 15 psig
Final Weight at -12 psig = 1119 grams
Weight Change = -278 grams Halon (calculated)
-12 grams Nitrogen (calculated)

Available Components:

Halon 1301 = 281 grams (calculated)
Nitrogen = 15 grams (calculated)

Lost in extinguisher:

Halon 1301 = 3 grams (calculated)
Nitrogen = 3 grams (calculated)

Note: An unknown amount of Halon/Nitrogen gas unintentionally escaped through a loose extinguisher fitting.

Reclaiming Tank:

Aluminium Recovery Tank:
(678 in^3)

Reclaiming tank is vented to 2 psig at -80°C.

Initial Weight of Tank = 14650 grams
Final Weight of Tank = 16360 grams
Pressure of Tank at 70 F = 218 psig
Nitrogen Weight in Tank = 16 grams (calculated)
Halon Weight in Tank = 1694 grams (calculated)

Recovery Rate:

Total Halon Recovered = 1693.25 grams

Sum weight of Halon in Aircraft Fire Extinguishers before discharging
= 281 +1610 = 1891 grams

Recovery Rate = 1693.25/1891 = 90%

6.0 CONTAMINATION OF LIQUID HALON BY REMAINING NITROGEN GAS

Formulae taken from Dupont Halon 1301 Technical Bulletin B-29E.

Tank Pressure at 70°F = 218 psig

1. Hx = Pn / Xn

Hx = Henry's Law constant, psi per mole fraction.
Pn = Partial pressure of N_2 in vapour phase, psia.
Xn = Composition of Nitrogen in liquid phase, mole fraction.

2. Pn = Pt - (1 - Xn) X (P1301)

Pt = Total system pressure, psia
P1301 = Vapour pressure of Halon, psia

3. Combining (1) & (2)

Xn = (Pt - P1301) / (Hx - P1301).

For 70 F, 218 psig:

P1301 = 213.7 psia
Pt = 232.7 psia
Hx = 4808
Xn = 0.0041355 mole fraction

4 Xn = Ni / n

Where Ni is the number of moles of component i in the mixture and n is the total number of moles in the mixture.

$CBrF_3$:
Molecular weight = 148.9 g

N_2:
Molecular weight = 28.013 g

Wt % of N_2 in solution is:

(.004136 X 28.013) / (.004136 X 28.013 + (1-.004136) X 148.9)) X 100 = 0.07813% (by weight)

Purity of new Halon 1301 as stated in Dupont sales specifications:

Purity (% by weight) = 99.70 Min

Provided that contamination of reclaimed Halon is limited to Nitrogen only, reclaimed Halon has the potential of being (100 - 0.07813) = 99.92 % pure by weight.

Gas Chromatography of the Reclaiming Tank contents showed the following:

Gas (from top of tank) = 94 % Halon Vapour by volume
6 % Nitrogen gas by volume

Liquid (from bottom of tank) = 99.5 % Halon 1301 by volume

Note: Maximum detectability range of Gas chromatographer is .5 % by volume.

Assuming the gaseous sample of the liquid acts as an ideal gas, the purity of the liquid Halon by weight is:

$$\frac{.995 \times 148.9}{.005 \times 28.013 + .995 \times 148.9} \times 100 = 99.9\%$$

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7.0 CONCLUSION:

Halon 1301 can easily and effectively be reclaimed from aircraft extinguishers and reused in aircraft extinguishers.

A minimum of 90 % agent recovery can be expected using a pumpless system.

Purity of reclaimed Halon 1301 has the potential of being 99.92 % pure by weight.

As further verification, gas chromatography was conducted on the reclaimed Halon. The Halon was found to be a minimum of 99.5% pure by volume (99.9% pure by weight).

NOTE: Agent recovery could be improved by:

a) incorporating an inline vapour pump to evacuate extinguisher.

b) closing extinguisher valve after initial discharge into Reclaiming Tank. Vent Reclaiming Tank. Close vent and discharge remaining extinguisher contents into Tank. Repeat as necessary.

SECOND APPENDIX

Walter Kidde Aerospace, Inc.
Visitation Report
Canadian Airlines International
November 08 and 09, 1990

Visitation to Canadian Airlines International **Project 1503-100**

Contact: Mr. James Hamilton, P.E.
Engineer
Ground Support Engineering

Subject: Pumpless Halon 1301 Recovery System using Dry Ice and Denatured Alcohol (85% Ethanol 15% Methanol)

Date of Visit: 08 and 09 November 1990

SUMMARY of VISIT:

Actual recovery procedures demonstrated that a minimum of approximately 92% agent recovery can be expected using a pumpless technique for the recovery of Nitrogen pressurized Halon 1301 from aircraft fire extinguishers. The technique involves the use of an insulated enclosing cylinder, a standard 678 cubic inch 3000 psig aluminum scuba tank, approximately 5 gallons denatured alcohol, and approximately 18 pounds dry ice.

Discussion with Mr. James Hamilton and Mr. Ken Goosen, P.E., Director, Central Engineering, revealed that beginning in June 1989 Canadian Airlines International had terminated removing fire extinguishers for the specific purpose of hydrostatic testing. The revised policy is that NO fire extinguisher will be removed for hydrostatic testing unless it visually appears to be damaged, low in pressure or does not meet appropriate specification weight tolerance when weight checked. Of course, if any abnormal condition were noted visually the firex would be removed and repaired. Upon removal for ANY reason that required discharge the firex would be hydrotested if overdue. Please see the attached letter dated 29 May 1989 file 99-2600 Canadian Airlines. FAA order 8320.10 waives the 5 year hydrostatic test until such time as the firex is discharged or otherwise indicates need for such service.

HALON 1301 RECOVERY SETUP DETAILS

The aluminum scuba tank was placed into the insulated enclosing cylinder (insulated using 1.5 inch closed cell foamed polymer on exterior surfaces except the bottom) atop an insulating pad of foamed polymer material. Radially located about this bottom insulating pad were three short rectangular columns which centered the pad in the bottom of the enclosing cylinder (constructed of welded stainless steel) and formed guides for centrally locating the scuba tank within the cylinder. Following scuba tank insertion into the cylinder additional polymer material was inserted from above to form radial stiffening elements between the scuba tank and the interior sidewall of the cylinder.

Once the scuba tank was secured within the cylinder approximately 16 liters of 15% denatured alcohol was poured into the cylinder followed by the introduction of approximately 8 kilograms of dry ice. A top fabricated of foamed polymer was the placed to seal the scuba tank within the cooling solution. In approximately 30 minutes the solution and scuba tank were at -112 degrees F (-80 deg C).

HALON 1301 FIREX RECOVERY DETAILS

Two firex units were recovered. A 224 cubic inch Apco unit and a 72 cubic inch Apco unit that developed a leak at the gauge glass edge.

Initial weight of evac (-12 psig) scuba: 14650 gms (32.288 lbs)

Initial weight of evac (-12 psig)
224 cu in firex : 2060 gms (4.541 lbs)
Halon 1301 Charge weight (+1610 gms) : 3670 gms (8.089 lbs)
Nitrogen Charge weight (+120 gms) : 3790 gms (8.353 lbs)

Upon initial transfer into the recovery scuba tank the firex chilled to a low temperature. Upon removal at 87 psig, while cool, the firex weighed 2395 grams.

Prior to re-connecting to the recovery system some line loss occurred since the connection plumbing was evacuated to -12 psig to remove unnecessary moisture from entering the system.

Upon warming to approximately 70 deg F (21 deg C) the pressure increased to 143 psig. Upon re-connection to the recovery scuba, heating using a hot air gun to approximately 90 deg F and discharging into the scuba a disconnect weight of 2180 and pressure of 84 psig resulted. This action recovered an additional 188 grams or 11.6% halon 1301.

Weight following recovery into scuba : 2180 gms (4.805 lbs)
Pressure following removal from scuba : 84 psig

Halon lost by venting the 84 psig from the 224 cu in firex was 90 grams plus 30 grams nitrogen. This 90 gram loss of 1301 represents 5.6%. Initial recovery efficiency indicated is 94%.

Prior to recovery of the second firex the recovery scuba was vented to a pressure of 2 psig.

A second 72 cu in firex, Apco PN 829700-5, that had a leak through the edge of the gauge glass following charging was also recovered.

This second firex was heated using a hot air gun while still connected to the recovery scuba.

Initial pressure : 293 psig
Initial weight : 1415 gms (3.119 lbs)
Final weight : 1125 gms (2.480 lbs)
Final pressure : 15 psig
Final weight at -12 psig : 1119 gms (2.467 lbs)
Weight change : -278 gms Halon 1301
: -012 gms Nitrogen
Available components Halon 1301 : 281 gms
Nitrogen : 15 gms
Lost in firex Halon 1301 : 3 gms
Nitrogen : 3 gms

Indicated recovery efficiency of this firex is 98%.

Following removal the recovery vessel was vented to 2 psig.

The recovery vessel was removed, heated to ambient, dried and weighed.

Initial weight of evac scuba : 14650 gms (32.288 lbs)
Final weight of scuba : 16360 gms (36.057 lbs)
Pressure of Scuba at 70 deg F (21 C) : 218 psig
Nitrogen weight in scuba : 16.0 gms (0.035 lbs)
Halon weight in scuba : 1694 gms (3.703 lbs)
Available Halon : 1891 gms (4.168 lbs)
Realized recovery efficiency : 89.6 %

During removal and re-connection of the first firex some halon was lost which would not have been lost if the firex were heated without being removed first. During recovery of the second firex some leakage developed at the bottle fitting. This leakage, since the bottle contained primarily halon vapor and liquid, resulted in an abnormally low overall recovery.

It is interesting to note that 24 hours following the recovery the solution of denatured alcohol and cylinder remained at approximetly -112 degrees F. Dry ice bricks could still be seen in the bottom of the cylinder. By bleeding the liquid side into the scuba halon stream temperatures assist in cooling the recovery tank. Stream temperatures are dependant on downstream pressure, which in this case, vary from 2 at the beginning of recovery to some higher pressure as determined by the size of the recovered firex. Halon stream temperature at 50 psig is approximately -6 degrees F.

RECOVERY SYSTEM HARDWARE

The recovery system basics used by CAI consist of:

1. Enclosing Container 347 SS 11.00 Dia X 30.25 X .025 wall
2. Some type of insulating material for tank that is resistant to attack from denatured alcohol.
3. Denatured Alcohol 85% Ethanol 15% Methanol Approx 5 Gal
4. Dry Ice Bricks Approx 18 pounds
5. Scuba Tank DOT 3AL 3000 · 678 Cu In
   Source: Luxfer USA Limited
   5005 La Mart Dr.
   Suite 201 PO Box 56100
   Riverside, Ca. 92517
   Telephone: 714-684-5110 Fax: 714-351-0790
6. Needle Valve 3000 psi standard 1/4 inch NPT female X 2
7. Scuba Tank Adapter Reducer Bushing - This must be fabricated
   Reducer from 1.031-14 UNS scuba thread
   to 1/4 - 18 NPT for 1/4 pipe to AN adapter.
8. Three - 1/4 NPT to AN adapters
9. Two short 1/4 inch SS tubing lengths to connect from scuba to valve and from valve to firex.
10. O-Ring -- MS 28775-214

Additional parts that may be needed are:

Fill fitting adapters for a variety of firex charging ports.
A vacuum pump either electric or manual for gross moisture removal.

GENERAL CHARGING PRACTICES OF CANADIAN AIRLINES INTERNATIONAL

CAI does not perform hydrostatic testing at their facility. This service is performed by an outside contractor.

Upon return from hydro test or prior to charging firex containers are dried using a hot air gun, evacuated to -12 psig, purged with dry nitrogen twice to 600 psig, evacuated to -12 psig and then charged using 1301 and nitrogen.

Information on some items are attached.

A formal report with photographs will be forthcoming.

APPENDIX C

EQUATIONS FOR MODELING PHYSICAL PROPERTIES

of

HALON 1301 AND 1211

Equations listed within this appendix are well known in the aerospace fire suppression industry. Original source for some equations are unknown; however, the equation remains valid due to demonstrated application measurement data. Primarily, equations addressing Halon 1301 are from E.I. DuPont Halon 1301 program "SUPER" for calculating Pressure-Temperature characteristics of nitrogen-charged Halon 1301, from DuPont manual FE-2A "Handling and Transferring DuPont Halon 1301 Fire Extinguishant", DuPont "KINETIC" Technical Bulletin B-4, and selected pertinent equations from Walter Kidde Aerospace, Inc. in-house modeling of aerospace fire extinguisher system discharge dynamics.

Equations modeling Halon 1211 physical characteristics are from Imperial Chemical Industries (ICI) Mond Division "BCF®"[C1] , Halon 1211, Systems Design Manual. Physical property data for Halon 1211, included in this appendix, is also from the ICI manual.

Equations developed during the course of this feasibility study are respectively noted.

The following equations and relationships are approximations of natural physical phenomena of subject compounds. While some representations are quite accurate and agreement between measured and calculated results may appear to have zero error, it must not be assumed that the following relationships are exact representation of the physical property being modeled.

The following equations approximate physical properties of Halon 1301. Equations immediately below are from the DuPont computer program "SUPER".

T: Temperature in degrees RANKINE (degR)
degrees R = degrees Fahrenheit + 459.67

TF: Temperature in degrees FAHRENHEIT

LD: Liquid density in pounds per cubic foot at specific temperature

VP1301: Vapor pressure of Halon 1301 in PSIA at specific temperature

HX: Henry's solubility function in PSI per Mole Fraction for calculating nitrogen solubility in LIQUID phase Halon 1301 at a specific temperature and Nitrogen partial pressure content.

[C1] BCF is the trademark of Imperial Chemical Industries Limited for their liquefied gas fire fighting agent.

Halon 1301 liquid phase density at 99.6 percent mole purity or higher (ZERO non-condensible dry gas content) in equilibrium with the vapor phase between -250 degrees Fahrenheit and +152.5 degrees Fahrenheit is calculated as follows:

$$LD = 46.5 + .039808 \cdot (612.17 - T) + .82202 \cdot (612.17 - T)^{.500} + 9.306269 \cdot (612.17 - T)^{.333333} + (9.22 \times 10^{-7}) \cdot (612.17 - T)^2$$

Typical values are:

-70° F: LD = 124.06 lb/cu ft
0° F: LD = 112.46 lb/ cu ft
+70° F: LD = 97.77 lb/cu ft

Halon 1301 liquid phase vapor pressure at 99.6 percent mole purity or higher (ZERO non-condensible dry gas content) in equilibrium with the vapor phase between -250 degrees Fahrenheit and +152.5 degrees Fahrenheit is calculated as follows (Equation (1)):

$$VP1301 = 10^{\left(7.32506 - .00438339(T) - \frac{1795.678}{T} - \frac{44295}{T^2} + .000003125(T^2)\right)}$$

Typical values are:

-70° F: 15.46 psia
0° F: 71.12 psia
+70° F: 213.61 psia

Nitrogen solubility (Henry's Law) in liquid Halon 1301 between -40 degrees Fahrenheit and +152.5 degrees Fahrenheit is calculated as follows:

$$HX = 4763.868266 + 3.94643566\,(TF) - .0162423503\,(TF^2) - .00044459\,(TF^3)$$

Typical values are:

-40° F: 4608 PSI/Mole Fraction
0° F: 4764 PSI/Mole Fraction
+40° F: 4867 PSI/Mole Fraction
+130° F: 4026 PSI/Mole Fraction

The numeric value resulting from this calculation is PSI per MOLE Fraction. Interpretation of the units describe nitrogen partial pressure required in PSI to effect X moles of nitrogen into Y moles of Halon 1301. At 70 degrees Fahrenheit the numeric value of 4808 PSI/MOLE Fraction means that 4808 psi nitrogen partial pressure is required to infuse 148.9 pounds (one pound mole) of Halon 1301 liquid with 28.02 (one pound mole) pounds of nitrogen.

The following equations are from DuPont's FE-2A manual, "Handling and Transferring DuPont Halon 1301 Fire Extinguishant".

$$\text{Percent Liquid Fill} = \frac{FD - DV}{LD - DV} \times 100$$

Since both liquid density and vapor density are temperature dependent, the percent liquid fill varies with temperature. If percent liquid fill is specified it must be referenced to a specific temperature.

Nitrogen partial pressure in the vapor phase within a Halon 1301 nitrogen pressurized fire extinguisher may be calculated as:

$$\bar{P}_n = P_t - (1 - X_n)\ VP1301$$

During superpressurization of Halon with nitrogen, some nitrogen becomes dissolved in liquid phase Halon 1301 according to Henry's Law. Henry's Law Constant, PSI per Mole Fraction, relates nitrogen concentration in the liquid phase as Mole Fraction and is defined as:

$$HX = \frac{\bar{P}_n}{X_n}$$

Nitrogen mole weight/mole in liquid phase Halon 1301 is calculated in the form:

$$X_n = \frac{P_t - VP1301}{HX - VP1301}$$

The result of X_n using $P_t = 640$ psia, VP1301 = 213.3 psia and HX = 4808 (70°F) is 0.09287. This result multiplied by nitrogen molecular weight of 28 yields 2.6. This 2.6 factor is the weight of nitrogen in pounds which can be physically dissolved into one liquid phase pound mole (148.9 pounds weight) of Halon 1301 at 70° Fahrenheit exposed to a nitrogen pressure of 640 psia.

The following equation, EQUATION (2) in the text, was developed as one result of this project. It became necessary to know approximate nitrogen weight in a vessel at any single given near-ambient temperature when the vessel contained some Halon 1301 and some nitrogen as indicated by measured container pressure.

The following, denoted as N2w is valid from nitrogen partial pressures of 1 to 800 psi and over a temperature range of 0° F to 130° F. The approximation maintains an acceptable error of approximately 6 percent (maximum) nitrogen weight up to fill densities of 75 pounds per cubic foot.

$$N2w = \left[0.00148148 \left(\frac{Pt - Vp}{Tr} \right) - 1.94213 \times 10^{-9} \times \left(\frac{Cw}{Cv} \right)^{2.67} \times Pt^{3.10} \right] Cv$$

Table 16 in the text lists values for nitrogen weight obtained by this approximation vs. other methods.

The following, EQUATION (3) from the text, gives a rough approximation of nitrogen weight required to pressurize a vessel of C_V cubic inches at 70° F when the vessel is approximately 50 percent full of liquid Halon 1301.

$$\text{Est. N2} = 2.78 \times 10^{-6} (P_t - 199) \times C_V$$

Where:

P_t = Measured cylinder pressure in psig
C_V = Internal cylinder volume in cubic inches

HALON 1211 RELATIONSHIP EQUATIONS AND PHYSICAL PROPERTIES:

Formula: $CBrClF_2$ (BromoChlorodifluoromethane)

Molecular Wt: 165.4

Boiling Point: -4.0°C (24.8°F)

Freezing Point: -160.5°C (-256.9°F)

Latent Heat of Vaporization: 58.3 BTU/lb

Liquid Density: 114 lb/Ft^3 @ 68°F

Superheated Vapor Density, 1Atm. 68°F: 2.3 ft^3/lb

Specific Volume (Superheat 1 Atm. 68°F): .4348 lb/ft^3

Critical Pressure: 595 psia

Critical Density: 44.4 lb/ft^3

Critical Temperature: 153.8°C (308.84°F)

Vapor Pressure (from Walter Kidde conversion of ICI equation):

$$VP1211 = 10^{\left[5.199 - \frac{1736.81}{(TF + 405.941)}\right]}$$

TF = Temperature in degrees Fahrenheit
VP1211: Vapor pressure psia

Nitrogen solubility at 75.2°F

$S = 2.35 \times 10^{-3} (P - 38.8)$
P = Total Pressure psia
S: Nitrogen in liquid phase, percent weight/weight 1211

APPENDIX D

MIL-M-12218C
26 October 1977
SUPERSEDING
MIL-M-12218B
31 March 1961

MILITARY SPECIFICATION
MONOBROMOTRIFLUOROMETHANE (LIQUIFIED) TECHNICAL GRADE FOR FIRE EXTINGUISHER

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

1. SCOPE

1.1 This specification covers technical grade monobromotrifluoromethane for specialized fire extinguisher applications.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

FEDERAL

RR-C-910 - Cylinders, Compressed Gas: ICC 4BA, ICC 4BW, and ICC 4E.

UU-T-81 - Tags, Shipping and Stock

MILITARY

MIL-V-2 - Valves, Cylinder, Gas (for Compressed or Liquefied Gases) General Specifications for.

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

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.

MIL-STD-129 - Marking for Shipment and Storage.

MIL-STD-1411 - Inspection and Maintenance of Compressed Gas Cylinders.

(Copies of specifications and standards required by contractors 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.

DEPARTMENT OF TRANSPORTATION

Title 49 - Code of Federal Regulations.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing office, Washington, DC 20402.)

3. REQUIREMENTS

3.1 Description. The monobromotrifluoromethane shall be furnished in a liquefied form in a container. The monobromotrifluoromethane shall be technical grade with a minimum purity of 99.6 percent and shall conform to the requirements of Table I when tested with the applicable method.

TABLE I.

	Property	Requirement
1)	CF_3BR, Mole Percent, Minimum	99.6
2)	Other Halocarbons, Mole Percent, Maximum	0.4
3)	Acidity ppm (by weight), Maximum	3.0
4)	Halogen Ion	Pass Test
5)	Water Content, Percent by Weight, Maximum	0.001
6)	Fixed Gases in Vapor Phase of Shipping Cylinder, Expressed as Air, Percent by Volume	1.5
7)	Boiling Point, ^{o}C at 760mm Hg	-57.75
8)	Boiling Range, ^{o}C, 5 to 85 percent distilled	0.3
9)	High Boiling Impurities, grams/100 ml, Maximum	0.05
10)	Suspended Matter or Sediment	None Visible

3.2 Containers. The monobromotrifluoromethane shall be supplied in Government-furnished containers in accordance with RR-C-910 for 400 psi maximum service pressure and filled in accordance with CFR 49. When specified (see 6.2), cylinders shall be furnished by the contractor and shall be in accordance with RR-C-910 for 400 psi maximum service pressure and shall be equipped with valves in accordance with MIL-V-2/22 and filled in accordance with CFR 49. When specified (see 6.2), monobromotrifluoromethane shall be supplied in contractor-owned DOT approved containers. The cylinders shall be inspected and maintained in accordance with MIL-STD-1411 for fluorocarbon cylinders. The fluid capacity of the fully charged cylinders shall be 150 pounds plus or minus 1/2 pound of monobromotrifluoromethane.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specifications where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Component and material inspection. The contractor is responsible for insuring that components and materials used are manufactured, examined, and tested in accordance with referenced specifications and standards as applicable.

4.2 Classification of inspections. The inspection requirements specified herein are classified as follows:

(a) Quality conformance inspection (see 4.3).
(b) Inspection of packaging (see 4.5)

4.3 Quality conformance inspection.

4.3.1 Lot. A lot shall consist of all cylinders filled from each integral source in 1 working day.

4.3.2 Sampling. Sampling for examination and tests shall be from filled cylinders and shall be in accordance with MIL-STD-105, Inspection Level 5.

4.3.3 Examination. Sample cylinders selected in accordance with 4.3.2 shall be examined for the following defects. AQL shall be 1.5 percent defective. Presence of one or more defects shall be cause for rejection of the lot.

101. Monobromotrifluoromethane not supplied in cylinders as specified.
102. Cylinders not inspected and maintained in accordance with MIL-STD-1411.
103. Tags missing or not as specified.
104. Weight of charge not as specified.

4.3.4 Test. The monobromotrifluoromethane in the container selected in accordance with 4.3.2 shall be tested for quality conformance as specified in 4.4.1 through 4.4.8. AQL shall be 1.5 percent defective. Presence of one or more defects shall be cause for rejection of the lot.

4.4 Test Methods.

4.4.1 Assay. The percentage of monobromotrifluoromethane shall be determined by gas-liquid chromatography. The components of the sample are separated and the area of each peak is measured. The monobromotrifluoromethane content of the sample is found by comparing the area of its peak with the total area of all components.

4.4.1.1 Apparatus. The following special apparatus is required to determine the percentage of monobromotrifluoromethane:

(a) Gas chromatograph, equipped with a 1 mv recorder, and thermal conductance detector.
(b) Column: 3 meters x 1/8 inch (3.175 mm) o.d. thin wall stainless steel tubing, packed with 80 to 100 mesh Porapak Q or equivalent.
(c) Gas sampling valve, 5 ml volume.

4.4.1.2 Reagents. The carrier gas shall be a commercial grade of helium. The column packing shall consist of a standard solution, e.g., 20 percent (wt/wt) practical hexadecane, on 80 to 100 mesh Porapak Q or equivalent.

4.4.1.3 Procedure.

(a) Install the column and adjust the temperature of the column oven to 80°C, the injection port to 160°C, and the detector block to 100-110°C. Temperature should be programmed to rise 8°C/min. to a maximum of 180°C.

(b) Adjust the helium flow to 20 ml/min.

(c) Adjust the detector voltage to 8 volts and allow the instrument to stabilize.

(d) Take the sample from the liquid phase (inverted cylinder). Flush the sample loop and sample valve for approximately 2 minutes before sampling.

(e) Rotate the gas sampling valve to transfer the sample into the chromatographic system and note the time.

(f) Close the sample cylinder valve.

(g) Allow the sample to elute, for approximately 18 minutes, attenuating as necessary to make the peak heights a convenient size. Under proper instrument settings, the monobromotrifluoromethane should elute after about 5 minutes.

4.4.1.4 Calculations. The percent of monobromotrifluoromethane shall be calculated as follows:

$$\% \, CF_3Br = \frac{A\,(CF_3Br) \times 100}{A_S}$$

Where: (a) $A\,(CF_3Br)$ = Area of monobromotrifluoromethane peak multiplied by recorder range setting.
(b) A_S = Sum of the relative peak heights.

Monobromotrifluoromethane percent less than that specified in Table I shall constitute failure of this test.

4.4.2 Acidity. A large sample shall be vaporized in the presence of distilled water. The acidity of the solution shall be determined by titration or by a pH indicator.

4.4.2.1 Sodium hydroxide titration.

4.4.2.1.1 Reagents.

(a) Sodium hydroxide, 0.01 N solution, standardized against reagent grade potassium acid phthalate.

(b) Methyl red indicator, 0.1 percent solution

4.4.2.1.2 Procedure. Place 10 ml of a crushed ice - distilled water slurry in a 250 ml glass stoppered Erlenmeyer flask and add 50 grams of monobromotrifluoromethane to the slurry. Place the stopper in the flask loosely and swirl the flask gently from time to time until the ice is completely melted. Add 1 drop of methyl red indicator, and if a reddish color remains, titrate to a yellow endpoint with 0.01 N sodium hydroxide solution. Run a crushed ice - distilled water blank (no monobromotrifluoromethane) along with the sample.

CAUTION: Perform the above procedure in a hood.

4.4.2.1.3 Calculations. The ppm acid halides, as HBr, shall be calculated as follows:

$$\text{ppm Acid Halides} = \frac{(A-B) \times N\ NaOH \times 0.0809 \times 10^6}{\text{weight of sample (grams)}}$$

Where: (a) A = ml NaOH for sample
(b) B = ml NaOH for blank

4.4.2.2 Acidity by Gramercy Indicator.

4.4.2.2.1 Apparatus.

(a) Fritted glass sparger of coarse porosity, contained in a 100 ml glass scrubbing bottle provided with inlet and outlet tubes.
(b) Neoprene connecting tubing.
(c) Wet test meter (0.1 cu ft/rev)
(d) Needle valve control, No. 55-660, Matheson Co., or equivalent.

4.4.2.2.2 Reagent. Gramercy Universal Indicator with color chart, Fisher Scientific Company, Catalog No. 11-508-4, or equivalent.

4.4.2.2.3 Procedure. Prepare neutralized distilled water by adding 0.4 ml of Gramercy Universal Indicator solution to 100 ml of distilled water, and titrate with 0.01 N sodium hydroxide until the water shows a pH of 7.0 when compared to the Gramercy Color Chart. Add 50 ml of the neutralized water to the glass scrubbing bottle fitted with glass gas sparger. Attach a needle valve control to the sample cylinder, and connect the cylinder, inverted, to an empty safety trap. Connect the safety trap outlet to the scrubbing bottle inlet. Connect the scrubbing bottle outlet to the inlet of the wet test meter. Slowly open the needle valve and pass 20 liters of sample through the scrubber at a flow rate of approximately 500 ml/min. Turn off the needle valve and disconnect the sample cylinder from the scrubbing bottle. Transfer 10 to 12 ml of water solution to a clean test tube. Add 0.3 ml of Gramercy Universal Indicator solution and swirl. Read the Ph of the solution by comparison with the Gramercy Color Chart. Report the pH reading. No observable change in pH indicates an acidity of less than 3.0 ppm.

4.4.3 Qualitative Test for Halogen Ion. A sample treated with an alcoholic solution of silver nitrate. The presence of halogen ions will cause the formation of insoluble silver halide and the development of turbidity.

4.4.3.1 Apparatus.

(a) Fitted glass gas sparger of coarse or "A" porosity, contained in a 100 ml glass scrubbing bottle provided with inlet and outlet tubes.
(b) Neoprene tubing
(c) Wet test meter (0.1 cu ft/rev).
(d) Needle valve control, No. 55-660, Matheson Company, or equivalent.

4.4.3.2 Reagents.

(a) Methyl alcohol, absolute.
(b) Silver nitrate, saturated solution in methyl alcohol.

4.4.3.3 Procedure. Add 10 ml of methyl alcohol into the scrubber assembly and add 3 to 4 drops of saturated alcoholic silver nitrate solution. Attach a needle valve control to the sample cylinder. Connect the sample cylinder in the upright position to an empty safety trap with neoprene tubing. Connect the outlet of the safety trap to the inlet of the scrubbing bottle assembly with neoprene tubing. Connect the outlet of the scrubbing bottle assembly to the inlet of the wet test meter. Slowly open the needle valve and pass about 2 liters of sample through the scrubber at a flow rate of approximately 100 ml/min. Turn off the needle valve and disconnect the scrubber assembly from the sample cylinder and the wet test meter. Examine the contents of the scrubber visually for the presence of turbidity. Report halide present if any turbidity develops. The appearance of any turbidity shall constitute failure of the test.

Note: The approximate ratio of methyl alcohol to monobromotrifluoromethane must be maintained. An excess of sample will cause the silver nitrate to precipitate, giving a false turbidity reading as halide.

4.4.4 Water content. Monobromotrifluoromethane shall be tested for water content. The analysis may be conducted by the phosphorus pentoxide method, by infrared absorption, by an electrolytic moisture analysis, or by a piezoelectric analyzer. The accuracy of the results and the standard method shall be by orthodox Karl Fischer method. Water content greater than specified in Table I shall constitute failure of this test.

4.4.5 Air in the vapor phase. Monobromotrifluoromethane may be tested for air in the vapor phase in the original container by the determination of gases not absorbable in perchloroethylene, or by gas chromatography.

4.4.5.1 Perchloroethylene absorption. Monobromotrifluoromethane may be tested for air in the vapor phase in the original container by the determination of gases not absorbable in perchloroethylene using water as a sealant. The test may be conducted by the determination of gases not absorbable in perchloroethylene using mercury as a sealant or by the determination of gases remaining when extinguishant is frozen. The determination of gases not absorbable in perchloroethylene using water as a sealant shall be the standard.

4.4.5.1.1 Apparatus.

(a) Absorption vessel, Lab Glass Co. No. LG-11119, or equal.
(b) Leveling bulb and connecting tubing.
(c) Perchloroethylene reagent: Specific gravity at 59°/39° F, 1.628-1.632; no free halogens; 0.001 maximum free acid as HCI; 0.00007 maximum residue on evaporation; distill completely between 248° and 252° F at 760 mm Hg; 31° F maximum cloud point.
(d) Silicone solution: 1 part "Siliclad" (Scientific Glass Co.) in 100 parts of water.

4.4.5.1.2 Procedure

(a) Wash the absorption vessel thoroughly and rinse in distilled water. Coat the inside with the silicone solution. Let stand 5 minutes and then discard the solution. Rinse again with distilled water and dry at 212° F for 10 minutes.
(b) When cool, ad 20 ml of distilled water and fill with perchloroethylene.
(c) Add sufficient water to the leveling bulb to give a water seal.
(d) Collect a 100 cc sample from the vapor space of the monobromotrifluoromethane cylinder directly into the absorption vessel. A slight positive pressure is needed to prevent entry of extraneous air.
(e) Tilt the vessel in such a way as to remove all the water lock which separated the perchloroethylene from the gas. Allow the perchloroethylene to flow into the upper chamber of the vessel and gently agitate to aid in the absorption of the gas.
(f) Return the vessel to an upright position and allow the water layer to flow back into the burette section.
(g) Adjust the leveling bulb so that the liquid layers are at the same level.
(h) Read the percent of nonabsorbable gas from the burette.
(i) A blank run is made with a sample from the liquid phase of the cylinder and the result subtracted from the analysis of the vapor phase.

The presence of air in the vapor phase in excess of 1.5 percent by volume shall constitute a failure of this test.

4.4.5.2 Gas chromatography. Monobromotrifluoromethane may be tested for the concentration of air in the vapor phase by gas chromatography. A concentration of air in excess of the percent by volume listed in Table I shall constitute failure of this test.

4.4.6 Boiling point, boiling range and high boiling impurities.

4.4.6.1 Apparatus.

(a) Goetz phosphorus tube, 100 ml capacity.
(b) Thermometer, National Bureau of Standards certified for the temperature range anticipated.
(c) Carborundum crystals, 20 mesh.

4.4.6.2 Procedure.

(a) Fill the Goetz tube with 100 ml of the sample, add two or three crystals of carborundum, and suspend thermometer in the sample.
(b) Suspend the tube in a medium (air or water) held at a minimum temperature of 90^{o} F (50^{o} C) above the expected boiling point.
(c) As soon as the thermometer is constant after 5 ml of the sample has been distilled, record the readings as the initial observed boiling point.
(d) Record as the end point the temperature reached when 85 ml of sample has been evaporated. Calculate the boiling range.
(e) Transfer the tube and the remaining 15 ml of sample to a second medium maintained at a temperature 50^{o} F (27.8^{o} C) above the boiling point.
(f) After 30 minutes, record the residue material as the percentage of high boiling impurities.
(g) A boiling point correction factor will be applied for atmospheric pressures other than 760 mm Hg.

A corrected boiling point other than than specified in Table I, a corrected boiling range in excess of that specified in Table I, or a percentage of high boiling impurities more than that specified in Table I, shall constitute failure of this test.

4.4.7 Suspended matter and sediment. Examine visually for any suspended matter or sediment. Observation of any suspended matter or sediment shall constitute failure of this test.

4.5 Inspection of packaging. The packing and marking of the cylinders shall be examined to determine compliance with the quality assurance provisions of MIL-C-52752.

5. PACKAGING

5.1 Packing. The level of packing shall be Level A, Level B, or Commercial as specified (see 6.2).

5.1.1 Level A. Packing shall be in accordance with the Level A requirements of MIL-C-52752.

5.1.2 Level B. Packing shall be in accordance with the Level B requirements of MIL-C-52752.

5.1.3 Commercial. Packing shall be in accordance with the Level C requirements of MIL-C-52752.

5.2 Marking. Marking shall be on a label secured to the full cylinder or on a wired tag which conforms to UU-T-81. The tag will be wired to the neck of the cylinder. Marking shall include care weight and weight of the contents.

5.2.1 Military packing. Military marking for military levels of protection shall be in accordance with MIL-STD-129

5.2.2 Commercial packaging. Marking shall be by any means which provides legibility and shall include the National Stock Number (NSN) or part number; nomenclature; quantity and unit of issue; contract or delivery order number; and address.

6. NOTES

6.1 Intended use. The monobromotrifluoromethane is intended for use as a specialized fire extinguisher fluid.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number, and date of this specification
(b) When other than Government-furnished containers are to be used (see 3.2)
(c) When procurement shall be made on a cost per unit basis in accordance with MIL-STD-1411 Inspection and Maintenance of Compressed Gas Cylinders.
(d) Level of packing required (see 5.1)

Custodians:
Army - ME
Navy - AS
Air Force - 68

Review activity:
DLA - GS

User activities:
Navy - MC, MS

Preparing activity:
Army - ME

Project 6830-0088

MIL-M-12218C
AMENDMENT 1
24 December 1981

MILITARY SPECIFICATION

MONOBROMOTRIFLUOROMETHANE (LIQUIFIED) TECHNICAL GRADE FOR FIRE EXTINGUISHER

This amendment forms a part of Military Specification MIL-M-12218C, dated 26 October 1977, and is approved for use by all Departments and Agencies of the Department of Defense.

PAGE 1

2.1, under SPECIFICATIONS, FEDERAL delete:

"UU-T-81 - Tags, Shipping and Stock."

PAGE 2

2.1, under SPECIFICATIONS, MILITARY delete:

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

under STANDARDS, MILITARY add:

"MIL-STD-147 - Palletized Unit Loads."
"MIL-STD-1188 - Commercial Paging of Supplies and Equipment."

Add:

"PUBLICATIONS

AFR 71-4
TM 38-250
NAVSUPPUB 505
MCOP 4030.19
DLAM 4145.3
- Preparation of Hazardous Materials for Military Air Shipment."

PAGE 11

4.5, delete all and substitute: "Inspection of packaging. Cylinders packed for shipment shall be examined for compliance with the requirements of Section 5 of this specification.

5, delete entire Section 5 and substitute:

"5. PACKAGING

"5.1 General requirements. In addition to the requirements detailed below, packaging for military air transport shall comply with Manual AFR 71-4/TM 38-250/NAVSUPPUB 505/MCOP 4030.19/DLAM 4145.3. No contact preservatives shall be applied to cylinders, valves, or other cylinder components.

"5.2 Packing. Packing shall be level A, level B, or Commercial, as specified (see 6.2).

"5.2.1 Level A. Cylinders shall be palletized in accordance with MIL-STD-147, load type IX.. Strapping shall be finish B.

"5.2.2 Level B. Cylinders shall be palletized as specified in 5.2.1 except that strapping shall be finish A.

"5.2.3 Commercial. Cylinders shall be packed in accordance with the requirements of MIL-STD-1188.

"5.3 Marking. Marking for level A or B shall be in accordance with MIL-STD-129, marking for commercial shall be in accordance with MIL-STD-1188."

"5.3.1 Special marking for Navy cylinders. Unless otherwise specified (see 6.2) cylinders for the US Navy shall have the following additional special marking:

CAUTION

a. Decomposition products (e.g., HBr, HF) produced if monobromotrifluoromethane is released into a very hot fire may be toxic to personnel and corrosive to equipment.

b. Exposure to monobromotrifluoromethane in the presence of elevated adrenaline levels (which would be likely during usage of fire extinguishing agents) could produce cardiac arrhythmia in some personnel."

PAGE 12

6.2(d) delete "5.1" and substitute "5.2"

Add "6.2(e) When special marking for Navy is not required (see 5.3.1)."

Custodians:
Army - ME
Navy - AS
Air Force - 68

Review activity:
DLA - GS

User activities:
Navy - MC, MS

Preparing activity:
Army - ME

Project 6830-0106

DU PONT

FREON® Fluorocarbon Products

TEST METHOD

APPENDIX E

DuPont Laboratory Test Methods
for
Halon 1301

HALON 1301

DETERMINATION OF PURITY

METHOD NO. H0265.165.01.CW(P)

E. I. du Pont de Nemours & Company (Inc.)
Du Pont Chemicals

Method No. H0265.165.01.CW (P)
First Revision: December 2, 1986
Page 1 of 20

HALON 1301

Determination of Composition by Gas Chromatography

I. Principle

Halon 1301 fluorocarbon composition is determined by program temperature gas chromatography using a Chromosorb 102 column. Separated component concentrations are quantified and documented using the Hewlett-Packard Model 3350A Laboratory Automation System.

II. Applicability

This method is applicable to the routine quantitative determination of volatile impurities in all grades of "Freon"* 13B1 fluorocarbon.

III. Limitations

This method does not address impurities other than those listed under Specific Application. There are no known interferences to the method.

IV. Sensitivity, Precision and Accuracy (Specific Reference 2)

A. Sensitivity

The method's sensitivity was determined to be 0.0001 weight percent for all calibration components.

B. Precision

1. Single Operator

The average analysis ($\bar{X}$), standard deviation (σ) and 95% confidence limits (95% CL) established for the method's single operator precision are as follows:

---

*Registered U.S. Patent Office

1. Single Operator (Continued)

Components	$\bar{X}$	σ	95% CL
	Percentages (w/w)		
"Freon" 14	0.0099	± 0.0002	± 0.0005
"Freon" 23	0.0098	± 0.0002	± 0.0004
"Freon" 13	0.0120	± 0.0001	± 0.0003
FC-218 (perfluoropropane)	0.0132	± 0.0024	± 0.0057
FC-125 (pentafluoroethane)	0.0147	± 0.0012	± 0.0027
Halon 1301	99.8740	± 0.0120	± 0.0028
"Freon" 22	0.0115	± 0.0014	± 0.0033
"Freon" 12	0.0142	± 0.0005	± 0.0013
FC-22B1 (bromodifluoromethane)	0.0101	± 0.0004	± 0.0010
FC-12B1 (bromochlorodifluoromethane)	0.0105	± 0.0009	± 0.0022
"Freon" 11	0.0112	± 0.0014	± 0.0032
FC-12B2 (dibromodifluoromethane)	0.0110	± 0.0013	± 0.0031

The above data were calculated from eight replicate analyses of one synthetic sample performed by one analyst over an eight-hour period.

2. Multiple Operator

The average analysis ($\bar{X}$), standard deviation (σ) and 95% confidence limits (95% CL) established for the method's multiple operator precision were as follows:

Components	$\bar{X}$	σ	95% CL
	Percentages (w/w)		
"Freon" 14	0.0091	± 0.0003	± 0.0008
"Freon" 23	0.0093	± 0.0004	± 0.0009
"Freon" 13	0.0119	± 0.0004	± 0.0009
FC-218 (perfluoropropane)	0.0111	± 0.0024	± 0.0054
FC-125 (pentafluoroethane)	0.0139	± 0.0015	± 0.0034
Halon 1301	99.8720	± 0.0064	± 0.0145
"Freon" 22	0.0110	± 0.0001	± 0.0003
"Freon" 12	0.0161	± 0.0028	± 0.0062
FC-22B1 (bromodifluoromethane)	0.0100	± 0.0005	± 0.0012
FC-12B1 (bromochlorodifluoromethane)	0.0129	± 0.0018	± 0.0040
"Freon" 11	0.0110	± 0.0013	± 0.0028
FC-12B2 (dibromodifluoromethane)	0.0112	± 0.0015	± 0.0032

The above data were calculated from sixteen replicate analyses of one synthetic sample performed by four analysts over a four-day period.

IV. Sensitivity, Precision and Accuracy (Specific Reference 2) (Continued)

C. Accuracy

	Percentages (w/w)	
Components	Standard Concentrations	Average Errors
"Freon" 14	0.0100	± 0.0009
"Freon" 23	0.0100	± 0.0007
"Freon" 13	0.0120	± 0.0001
FC-218 (perfluoropropane)	0.0100	± 0.0011
FC-125 (pentafluoroethane)	0.0130	± 0.0009
Halon 1301	99.8800	± 0.0020
"Freon" 22	0.0110	± 0.0000
"Freon" 12	0.0140	± 0.0021
FC-22B1 (bromodifluoromethane)	0.0100	± 0.0001
FC-12B1 (bromochlorodifluoromethane)	0.0100	± 0.0029
"Freon" 11	0.0100	± 0.0010
FC-12B2 (dibromodifluoromethane)	0.0100	± 0.0012

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Gas chromatograph, thermal conductivity detector, Model No. HP-5890, Hewlett Packard Co., Valley Forge, PA 19842. (Gas chromatographic data are reduced automatically using a Hewlett-Packard Model 3350A Laboratory Automatic System.)

2. Recorder, Omni Scribe, Cat. No. 55423-069, VWR Scientific, Philadelphia, PA 19101.

3. Gas chromatographic column, 60-80 mesh Chromosorb-102 (styrene-divinylbenzene copolymer), in an 8-meter 1/8 in. o.d., s/s column, configured for a Hewlett-Packard 5890 gas chromatograph. Prepacked columns are purchased from Supelco, Inc., Bellefonte, PA 16823.

4. Gas sampling valve, s/s, 6-port (attach a calibrated 1 mL, s/s, gas sampling loop to the valve), Cat. No. SS43Y6FS2, Wilmington Valve and Fitting Co., Wilmington, DE 19720.

   Attach the gas sampling valve (inlet side/vent) to a s/s, small volume, vacuum manifold, equipped with a pressure-vacuum gauge (Ashcraft-Dresser gauge, Cat. No. 25-1009-S-02L, range 30 in. Hg-0-60 psig, McArdle-Desco Corp., Wilmington, DE 19720), a vacuum pump capable of 10^{-3} mm Hg vacuum and three s/s toggle valves

V. Special Apparatus (Equivalent apparatus may be substituted) (Continued)

(Cat. No. SS-1GM4, Wilmington Valve and Fitting Co.) located between the vacuum manifold and

- the vacuum pump (Valve A)
- the 1/8 in. o.d., s/s tube from the gas sample loop vent (Valve B)
- The atmosphere (Valve C)

5. Hypodermic syringes, gas tight and liquid types of appropriate volumes.
6. Vacuum manifoid system, capable of 10^{-3} mm Hg vacuum.
7. Serum bottle stopper septum, Cat. No. 03-225-5, Fisher Scientific Co., King of Prussia, PA 19406.
8. Cylinder, 900 mL capacity, with 3/8 in. spud (ICC-4B-240ET), E. F. Britten Co., West Cranford, NJ 07016. Equip the cylinder with a Superior valve, Cat. No. 1014C, Superior Valve Co., Washington, PA 15301.

VI. Reagents (Reagent grade except as noted)

Calibration Reagents

1. "Freon" 14 fluorocarbon, E. I. du Pont de Nemours and Co. (Inc.).
2. "Freon" 23 fluorocarbon, E. I. du Pont de Nemours and Co. (Inc.).
3. "Freon" 13 fluorocarbon, E. I. du Pont de Nemours and Co. (Inc.).
4. FC-218 (perfluoropropane), Cat. No. 11430-6, Specialty Chemicals, Gainsville, FL 32602.
5. FC-125 (pentafluoroethane), Cat. No. 10074-3, Specialty Chemicals.
6. Halon 1301, E. I. du Pont de Nemours and Co. (Inc.).
7. "Freon" 22 fluorocarbon, E. I. du Pont de Nemours and Co. (Inc.).
8. "Freon" 12 fluorocarbon, E. I. du Pont de Nemours and Co. (Inc.).
9. FC-22B1 (bromodifluoromethane), may be obtained by custom synthesis from Specialty Chemicals.
10. FC-12B1 (bromochlorodifluoromethane), Cat. No. 11111-2, Specialty Chemicals.

VI. <u>Reagents</u> (Reagent grade except as noted) (Continued)

11. "Freon" 11 fluorocarbon, E. I. du Pont de Nemours and Co. (Inc.).

12. FC-12B2 (dibromodifluoromethane), Cat. No. 10060-2, Specialty Chemicals.

Gas Chromatographic Reagents

13. Helium, C&P Department Specification L-114, Union Carbide Corp., Linde Division, South Plainfield, NJ 07080.

VII. Special Safety Considerations

A. Product Hazards

Halon 1301 is a compressed liquefied gas, which can cause frostbite if discharged on the skin. Avoid contact with the person. For information, refer to MSDS #2040FR.

B. Procedure Hazards

All of the chemicals, except "Freon" 11 fluorocarbon and FC-12B2, listed under Calibration Reagents (VI) are compressed liquefied gases, which will cause frostbite, if discharged on the skin. Avoid contact with the person.

Halogenated liquid organic compounds (e.g. "Freon" 11 fluorocarbon and FC-12B2) dissolves natural oils. Avoid contact with the skin.

Hypodermic syringes must be handled with great care in order to preclude the possibility of accidental puncture or injection. Shield hypodermic syringe needles when in use.

VIII. Procedure

A. Operating Conditions

Instrument Conditions

Gas Chromatograph:
  Type = Hewlett-Packard
  Model No. = 5890

Carrier gas:
  Type = Helium
  Flow rate, mL/min. = 30

Injection port:
  Temperature, °C = 200

A. Operating Conditions (Continued)

Column:
Length, meters = 8
Material of construction = S/S
o.d., in. = 1/8
Solid support = Chromosorb-102
Mesh size = 60-80
Temperature limit, °C = 240

Column temperature:
Initial temperature, °C = 75
Pre-program hold, min. = 8
Rise rate, °C/min. = 10
Final temperature, °C = 200
Post-program hold, min. = 9.5

Detector:
Type = Thermal conductivity
Temperature, °C = 200

Miscellaneous:
Sample size, mL = 1
Chromatographic time, min. = 30

Component Data

Calibration Components	Retention Times, Minutes	Concentration Ranges Investigated, % (w/w)	Response Factors
Air	2.12	0.090	-
"Freon" 14	2.56	0.020	0.8049
"Freon" 23	4.93	0.020	0.5407
"Freon" 13	7.09	0.020	0.4913
FC-218	10.70	0.020	0.1951
FC-125	11.23	0.020	0.8224
Halon 1301	11.86	99.690	1.0000
"Freon" 22	15.24	0.020	0.5987
"Freon" 12	17.62	0.020	0.6938
FC-22B1	19.33	0.020	0.8629
FC-12B1	21.44	0.020	0.6745
"Freon" 11	23.58	0.020	1.3135
FC-12B2	25.14	0.020	0.7877

VIII. Procedure (Continued)

B. Calibration

Standard Preparation

1. Obtain a sample of the highest purity Halon 1301, which shows no impurity peaks for the calibration components, when analyzed using the chromatographic conditions described in VIII-A and the most sensitive attenuator setting. Reserve this sample for standard preparations.

2. Determine the "area percent" purity of all calibration reagents (exclude Halon 1301) by analyzing each reagent using the chromatographic conditions described in VIII-A. Record component purities to the nearest 0.01 percent on the standard preparation chart (Appendix 1). Use these purity percentages for standard preparation.

3. Evacuate a clean, dry, 900 mL capacity, sample cylinder to ca. 10^{-3} mm of mercury, on a vacuum manifold system.

4. Close the cylinder valve and return the manifold to atmospheric pressure.

5. Remove the cylinder from the vacuum manifold.

6. Weigh (± 0.1 g) the cylinder and record the weight.

7. Cap the cylinder valve with a serum bottle stopper-septum.

8. Calculate the weight of Halon 1301 required to 90 percent liquid-fill the sample cylinder (Step 3), using the following equation:

$$W = C \times P \times D$$

Where: W = weight of Halon 1301, grams

C = cylinder capacity, mL

P = fill percentage expressed as a decimal = $\frac{90}{100} = 0.9$

D = Halon 1301 liquid density at 25°C = 1.538 g/mL

Record Halon 1301 weight to the nearest 0.1 gram.

B. Calibration (Continued)

9. Individually and in turn, calculate the weight of each component to be added to the Halon 1301 for standard preparation. Use the following equation for these calculations:

$$G = \frac{A}{P}$$

Where: G = actual weight of component to be added to the standard, gram
A = standard component weight desired, gram
P = component purity (Step 2) expressed as a decimal (% purity ÷ 100)

Record component weights to the nearest 0.0001 g. Enter these weights on the Standard Preparation Chart (Appendix 1).

10. Individually and in turn, convert standard LIQUID component weights to liquid volumes for standard introduction purposes. Use the following equation for these calculations:

$$V_L = \frac{G \times 1{,}000}{D}$$

Where: V_L = Standard LIQUID component volume, µL

G = LIQUID standard weight, gram (Step 9)

D = LIQUID standard density at 25°C, g/mL

1,000 = Conversion factor, mL to µL

Record LIQUID component volumes to the nearest microliter (µL). Enter these volumes on the Standard Preparation Chart (Appendix 1). Use these volumes for standard preparation.

11. Individually and in turn, convert standard GASEOUS component weights to gas volumes at 25°C and one atmosphere pressure, for standard preparation. Use the following equation for these calculations:

B. Calibration (Continued)

$$V_G = \frac{G \times 82.054 \times 298.18 \times 1,000}{P \times M}$$

Where: V_G = standard GASEOUS component volume, µL

G = GASEOUS standard weight, gram (Step 9)

P = pressure = 1 atmosphere

M = GASEOUS component molecular weight, grams/mole

82.054 = gas constant, mL atmos/°K mole

298.18 = 25°C expressed as °K

1,000 = conversion factor, mL to µL

Record GASEOUS component volumes to the nearest microliter (µL). Enter these volumes on the Standard Preparation Chart (Appendix 1). Use these volumes for standard preparation.

12. Individually and in turn, add LIQUID standard components to the evacuated, tared, serum capped, standard cylinder (Step 7) as follows:

    a. Fill and flush a clean, dry, appropriate volume, liquid, microliter syringe several times with the LIQUID standard under consideration. Discard the rinsings.

    b. Fill the syringe to a volume greater than that to be delivered.

    c. Hold the syringe vertically with the needle pointed up.

    d. Carefully push the needle through a facial tissue to catch expelled liquid.

    e. Tap the syringe barrel gently with the finger to permit air bubbles to rise to the top of the barrel.

    f. Slowly depress the plunger until the plunger tip is aligned with the volume scribe line desired (Step 10).

    g. Remove the facial tissue from the needle, wiping away liquid droplets in the process.

    h. Carefully insert the needle point into a soft rubber septum.

B. Calibration (Continued)

i. Weigh (± 0.0001 g) the microliter syringe and its contents (Step 12-h). Record the weight.

j. Open the cylinder (Step 7) valve.

k. Uncap the microliter syringe, insert the needle through the cylinder septum, and inject the syringe content into the cylinder.

l. Remove the microliter syringe from the cylinder septum and reinsert the needle into its soft rubber septum.

m. Close the standard cylinder valve (Step 12-j).

n. Weigh (± 0.0001 g) the microliter syringe. Record the weight.

o. Calculate the weight of LIQUID standard component added to the cylinder using the following equation:

gram LIQUID standard added = gram (Step 12i-Step 12n)

Record the LIQUID standard weight on the Standard Preparation Chart (Appendix 1).

13. Individually and in turn, add GASEOUS standard components to the evacuated, tared, standard cylinder (Step 12-m) as follows:

a. Clamp the GASEOUS standard cylinder (valve down) to a ring stand.

b. Adapt a Nupro needle valve (Cat. No. SS-2F-7, Wilmington Valve and Fitting Co.) to the cylinder valve (Step 13-a).

c. Attach a 6-inch length of tight-fitting Tygon tube to the needle valve.

d. Attach a 6-inch length of glass tube to the Tygon tube discharge end.

e. Clamp the glass tube to a ring stand and position the discharge end a few millimeters from the bottom of a 250-mL beaker, which contains ca. 100 mL of tap water.

f. Check to ensure the needle valve (Step 13-b) is closed.

B. Calibration (Continued)

g. Open the GASEOUS standard cylinder valve (Step 13-a).

h. Open the needle valve and adjust gas flow to vigorous bubbling.

i. Allow the standard to bubble for ca. 1-2 minutes to completely purge the tubing (Tygon and glass) of air.

j. Open the standard cylinder valve (Step 12-m).

k. Inject a clean, dry, appropriate volume, gas-tight, hypodermic syringe (plunger in the full discharge position) into the purged Tygon tube (Step 13-i).

l. Fill and discharge the hypodermic syringe slowly.

m. Repeat Step 13-1 twice.

n. Fill the gas-tight syringe to the desired volume scribe line (Step 11).

o. Remove the filled syringe from the Tygon tube (Step 13-n).

p. Immediately insert the needle through the cylinder septum (Step 12-j) and inject the syringe content into the cylinder.

q. Immediately remove the hypodermic syringe from the cylinder septum.

r. Record the GASEOUS standard weight delivered on the Standard Preparation Chart (Appendix 1).

s. Close the GASEOUS standard cylinder valve (Step 13-g).

t. Close the standard cylinder valve (Step 13-j).

u. Disconnect the needle valve/Tygon tube/glass tube assembly from the GASEOUS standard cylinder valve.

14. Remove the serum bottle stopper-septum from the standard cylinder valve (Step 7).

15. Evacuate another clean, dry, 900-mL capacity cylinder to ca. 10^{-3} mm of mercury on a vacuum manifold system.

16. Repeat Steps 4 and 5.

B. Calibration (Continued)

17. Weigh (± 0.1 g) the evacuated cylinder (Step 16). Record the weight.

18. Add the calculated weight of Halon 1301 (Step 8) to the tared cylinder (Step 17).

19. Weigh (± 0.1 g) the filled cylinder (Step 18) and record the weight.

    a. If content weight (grams, Step 19-Step 17) exceeds the desired weight (Step 8), vent the cylinder until the correct weight is achieved. Proceed to step 20.

    b. If content weight (grams, Step 19-Step 17) is less than the desired weight (Step 8), add additional Halon 1301 until the desired weight is achieved. Proceed to Step 20.

20. Connect (valve up) the standard cylinder (Step 13-t) to the Halon 1301 cylinder (Step 19) with the valve down. Use the smallest length and volume connector possible.

21. Fully open the Halon 1301 cylinder valve (Step 20).

22. Fully open the standard cylinder valve (Step 20) and allow the Halon 1301 to transfer to the standard cylinder.

23. When the Halon 1301 transfer is complete (termination of audible flow), <u>immediately</u> close both cylinder valves.

24. Disconnect the cylinders from the connector.

25. Shake the standard cylinder (Step 24) vigorously for 60 ± 5 seconds.

26. Weigh (± 0.1 g) the Halon 1301 cylinder (Step 24). Record the weight on the Standard Preparation Chart.

27. Calculate the weight of Halon 1301 transferred to the standard cylinder using the following equation:

    grams Halon 1301 = grams (Step 19-Step 26)

    Record the Halon 1301 actual weight on the Standard Preparation Chart (Appendix 1).

B. Calibration (Continued)

28. Calculate individual standard component weight concentrations in the standard as follows:

$$\text{ppm component} = \frac{W \times 10^6}{G}$$

Where: W = component weights, g (Appendix 1)

G = total standard weight, g (Appendix 1)

10^6 = parts per million factor

Record individual component concentrations to the nearest 0.1 ppm.

Determination of Component Response Factors (Manual Procedure)

29. Connect the standard cylinder (Step 25), valve down, to a ring stand.

30. Close all toggle valves on the vacuum manifold.

31. Make certain the gas sampling valve's inlet valve (Cat. No. SS-1RS4, Wilmington Valve and Fitting Co.) is closed, then position the gas sampling valve to the sample purge position.

32. Attach a cylinder adapter, to which has been attached a valve (Cat. No. SS-4VFA, Wilmington Valve and Fitting Co.) adapted to a 1/8 in. s/s, flare fitting.

33. Connect the standard cylinder adapter (Step 32) to one end of a 1/8 in. s/s tube.

34. Loosely connect the free end of the 1/8 in. s/s tube to the gas sampling valve's inlet valve.

35. Open the standard cylinder valve.

36. Open the cylinder adapter valve and allow standard gas to purge through the 1/8 in. s/s tube for 20 ± 1 seconds, then tightly connect the tube to the gas sampling valve (Step 34).

37. Open the vacuum pump, toggle valve (A), and evacuate the manifold to 30 in. Hg vacuum (gauge).

38. Close the vacuum pump toggle valve (A).

B. Calibration (Continued)

39. VERY CAREFULLY crack open the gas sampling valve's inlet valve and allow calibration gas to flow into the gas sampling valve, until manifold pressure reaches 0 psig.

    NOTE: If the valve is opened too quickly, the gas pressure (>> 60 psig) surge will probably damage the gauge.

40. IMMEDIATELY close the gas sampling valve's inlet valve.

41. Repeat Steps 37, 38, 39 and 40.

42. Repeat Step 41.

43. Carefully crack the vacuum pump toggle valve (A) to reduce manifold pressure to exactly 0 psig, then immediately close the toggle valve (A).

44. Simultaneously introduce (gas sampling valve, Step 43) the standard into the chromatograph and start the recorder.

45. Analyze the standard (Step 25) using the chromatographic conditions described in VIII-A.

46. Chromatograph the standard, adjusting individual component attenuator settings to maximize peak heights. Record the attenuator settings on the chromatogram.

47. Determine all standard component areas, individually and in turn, as follows:

    a. Draw a baseline between the two peak minima. (Ideally, peak minima should be in the same plane.)

    b. Measure the peak height (mm) perpendicular to the baseline to the peak maximum. Record the height to the nearest 0.5 mm.

    c. Measure the peak width (mm) at one-half the peak height. Record the width to the nearest 0.5 mm.

    d. Calculate the peak area using the following equation:

$$C_A = H \times W \times A$$

Where: C_A = component peak area, mm^2

H = component peak height, mm (Step 47-b)

W = component peak width, mm (Step 47-c)

A = component attenuator setting (Step 46)

B. Calibration (Continued)

Record component areas to the nearest mm^2.

48. Calculate the chromatogram's total area as follows:

$$\Sigma_A = C_A + C_B + C_C + \ldots\ldots C_Z$$

Where: Σ_A = total chromatogram area, mm^2

C_A = Component A peak area, mm^2 (Step 47-d)

C_B = Component B peak area, mm^2 (Step 47-d)

C_C = Component C peak area, mm^2 (Step 47-d)

C_Z = last component peak area, mm^2 (Step 47-d)

Record the chromatogram's total area to the nearest mm^2.

49. Calculate all standard component area concentrations, individually and in turn, as follows:

$$\text{ppm C} = \frac{C_A \times 10^6}{\Sigma_A}$$

Where: ppm C = component area concentration, ppm

C_A = component peak area, mm^2 (Step 47-d)

Σ_A = total chromatogram area, mm^2 (Step 48)

10^6 = part per million factor

Record component area concentration to the nearest 0.1 ppm.

50. Calculate all standard component response factors, individually and in turn, as follows:

$$F = \frac{S}{C}$$

Where: F = response factor

S = actual component concentration in the standard, ppm (Step 28)

C = component area concentration from the chromatogram, ppm (Step 49)

B. Calibration (Continued)

Record component response factors to the nearest 0.0001 unit on the Standard Preparation Chart (Appendix 1). Use these response factors for sample calculations.

C. Sampling

Not applicable.

D. Sample Analysis

1. Analyze the sample using the chromatographic conditions described in VIII-A and the sampling procedure described in VIII-B, 29 through 44.
2. Chromatograph the sample, adjusting component attenuator settings to maximize peak heights. Record the attenuator settings on the chromatogram.

E. Calculations

1. Determine all sample component peak areas individually as described in VIII-B-47. Record component areas to the nearest mm^2.
2. Calculate the chromatogram's total area as described in VIII-B-48. Record total area to the nearest mm^2.
3. Calculate all sample component area concentrations individually as described in VIII-B-49. Record component area concentrations to the nearest 0.1 ppm.
4. Individually and in turn, apply response factor corrections to each sample component area percentage to obtain the true component composition in the sample. Use the following equation for these calculations:

$$\text{ppm } C_W = A \times F$$

Where: ppm C_W = actual sample component concentration

A = sample component area concentration, ppm, Step 3

F = component response factor, VIII-B-50 or Appendix 1

Report sample component concentrations to the nearest 0.1 ppm.

IX. Quality Control

1. Run the calibration standard beweekly or at any time questions arise concerning the method's performance.

2. Use A Shewhart plot to ensure measurement reliability.

X. Comments

None.

XI. References

"'Freon' Fluorocarbons Properties and Applications," Technical Bulletin G-1, E. I. du Pont de Nemours & Co (Inc.), "Freon" Products Division, Wilmington, DE 19898, 1985.

XII. Appendix

1. Standard Preparation Chart

2. Computer Conditions Printout

3. Sample Chromatogram - Computer Printout

Appendix 1

Standard Preparation Chart

Components	Component Quantities mL or µL	Component Quantities g	Component Purity, %	Actual Amount Of Component Added, g	Component Conc. In Std., %	Response Factors
"Freon" 14						
"Freon" 23						
"Freon" 13						
FC-218						
FC-125						
Halon 1301						
"Freon" 22						
"Freon" 12						
FC-22B1						
FC-12B1						
"Freon" 11						
FC-12B2						
			Totals			

Appendix 2

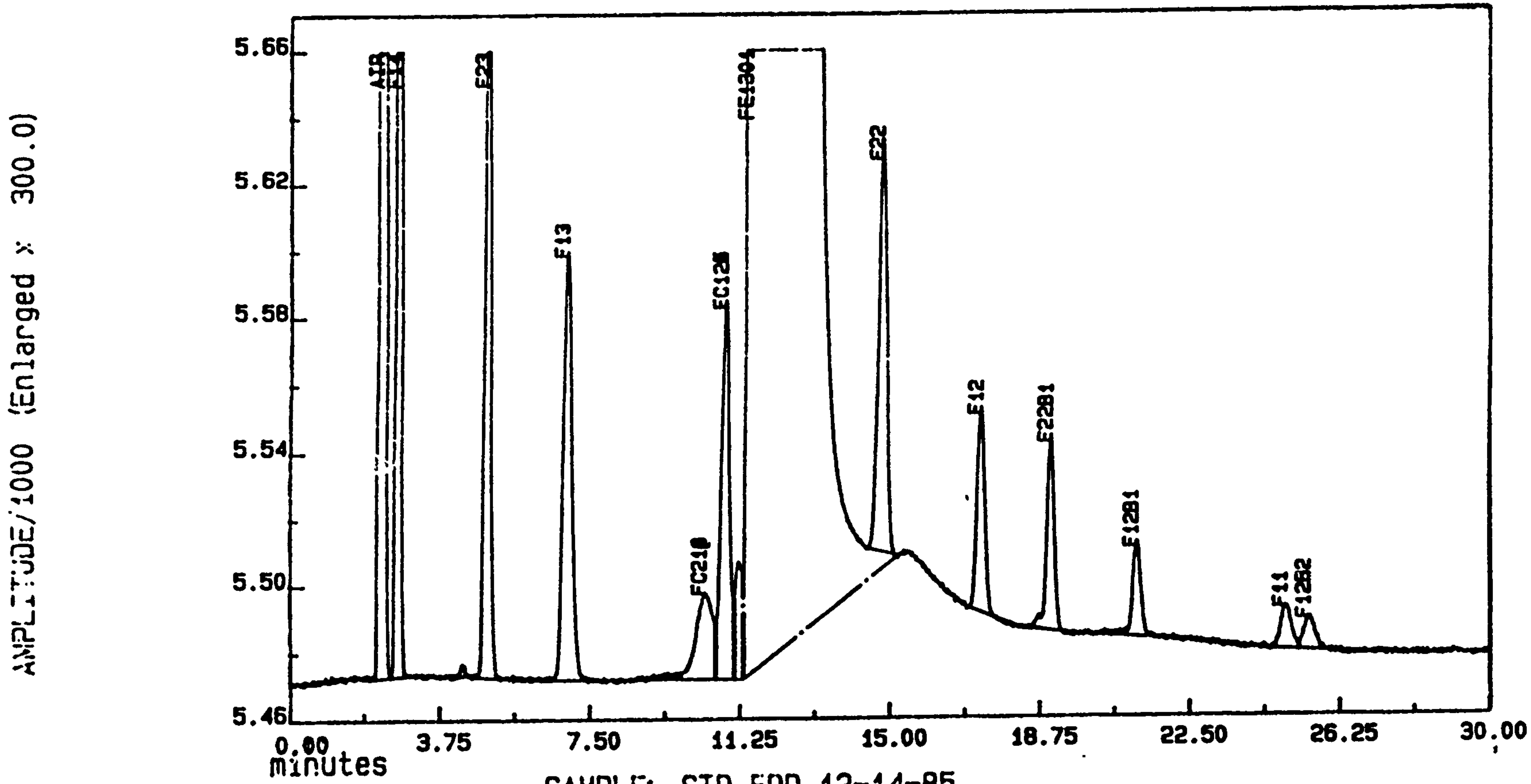
Computer Conditions Printout

```
METHOD HEADER

    Method        /DATA/LOOP/METHOD/F1301C13.MTH
    Created       Thu Jun  6  1985  9:01:43 am
    Updated     : Sun Nov 16  1986  2:34 48 am
    Instrmnt    : A/D 13
    Calc Type     NORM

COMMENT FIELDS

    Comment 1 :
    Comment 2 :
    Comment 3 .
    Comment 4

DATA INPUT

    Runtime              : 35.00                  Number Of Peaks    25
    Delay Start          : 0.00                   Minimum Area     · 10.0
    Slope Sens.            .100                   Bunch Mode       . AUTO
    Bunch Start          : 2.00

DATA ANALYSIS

    Area Calc     YES

USER PROGRAMS

    Post Run    :
    Dlc Proc
    Parm File   :
    Overlap       YES

PEAK IDENTIFICATION

    Ref Window             1.000                  Non-Ref Window   · 10.000
    Resp Factor Unkn       1.00000                Minimum Id Level . 0.0
    Dead Volume Time     · 0.00

TIMED EVENTS

           Time   Event  Value        S EQN          Relay #
          ------  -----  --------  ---------------   -------
   -       11.50  TS

CALIBRATION FILE

    File Name

CALIBRATION TABLE

           Time    Factor      Amount                    Peak Name                 Type Grp
          ------ ---------- ----------  --------------------------------------- ---- ---
   1        2.12 3.0232E-05 1.0000E-06  AIR
   2        2.56    .804944 9.7000E-03  F14
   3.       4.93    .540730    .010500  F23
   4        7.09    .491261    .012000  F13
   5.      10.70    .195064    .010000  FC218
   6.      11.23    .822451 9.7000E-03  FC125
   7       11.86   1.000000  39.896000  FE1301                                      R
   8       15.24    .598727    .011500  F22
   9       17.62    .693761 9.7000E-03  F12
   10      19.33    .862919 9.7000E-03  F22B1
   11.     21.44    .674518 9.7000E-03  F12B1
   12      23.58   1.313501    .010300  F11
   13      25.14    .787716 8.9000E-03  F12B2
   14      38.00   0.000000 1.0000E-06  UNK                                         R

REPORT SPECIFICATION

    Fmt File    · /DATA/LOOP/FORMAT/MEDIUM AREA.FMT
    Rpt Units     WGT%
```

```
REPORT DEVICES

         Report Device Number Copies
         ------------- -------------
   1.          L2              1
```

Appendix 3

Sample Chromatogram - Computer Printout



SAMPLE: STD EDD 12-14-85
ANALYZED: Sat Dec 14, 1985 8:39:50 am
RESULT: /DATA/LOOP/RESULT/SF1301C13 574.RES

DU PONT

FREON® Fluorocarbon Products

TEST METHOD

FREON® PRODUCTS

DETERMINATION OF MOISTURE

METHOD NO. F3200.135.01.CWP

"FREON"* PRODUCTS

Determination of Water by Electrolysis (Moisture Mite)

I. Principle

A fixed mass flow rate of "Freon" fluorocarbon gas is passed through an electrolytic cell, which consists of two closely-spaced, parallel platinum wires coated with phosphorous pentoxide (P_2O_5). A constant potential (d.c. voltage) is applied to the cell. Sample moisture is absorbed on the phosphorous pentoxide and simultaneously electrolyzed in the cell. The current developed in the cell is directly proportional to the amount of water electrolyzed, as described by Faraday's Law. The sample's moisture content is read directly from the instrument's meter.

II. Applicability

The method is generally applicable to all materials with vapor pressures > 1 atmosphere at ambient temperatures, provided the materials are neither reactive with phosphorous pentoxide nor electrolytically unstable under test conditions.

III. Limitations

Only gaseous materials should be introduced into the electrolytic cell. Introduction of liquid samples into the electrolytic cell will wash the phosphorous pentoxide film from the electrodes and desensitize the cell.

Samples which contain oil must not be analyzed by this method because the oil coats and desensitizes the cell.

Samples which contain rust or other particulates must not be analyzed by this method because of the potential for a "short circuit" and permanent damage to the cell.

See General Applicability.

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

To be determined.

---

* Registered U.S. Patent Office

B. Precision

1. Single Operator

The average analysis ($\bar{X}$), standard deviation (s) and 95% confidence limits (95% CL) established for the single operator precision of the method were as follows:

	$\bar{X}$	s	95% CL
Moisture, ppm (w/w)	To be determined.		

The above data were calculated from xxx replicate analyses of a standard sample analyzed by one technician over a xxx day period.

2. Multiple Operator

The average analysis ($\bar{X}$), standard deviation (s) and 95% confidence limits (95% CL) established for the multiple operator precision of the method were as follows:

	$\bar{X}$	s	95% CL
Moisture, ppm (w/w)	To be determined.		

The above data were calculated from xxx replicate analyses of a standard sample analyzed by xxx technician(s) over a xxx day period.

C. Accuracy

To be determined.

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Moisture Mite Analyzer, MEECO, Warrington, PA 18976.
2. Sample delivery tube, Fluoroflex "Teflon"* hose, 18 inches, Cat. No. R-2200200-4-L, Briggs Rubber Products Co., New Castle, DE 19720.
3. Gas pressure regulator, Model No. 2930. Thermco Instrument Co., La Porte, IN 46350.

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* Registered U.S. Patent Office

VI. Reagents (Reagent grade except as noted)

1. "Freon" 12 fluorocarbon, E. I. du Pont de Nemours & Co. (Inc.)

VII. Special Safety Considerations

A. Product Hazards

Compressed, liquefied "Freon" fluorocarbons (e.g., "Freon 12", "Freon" 22, "Freon" 114, etc.) will cause frostbite if discharged on the skin. Avoid contact with the person. For more information, refer to the specific MSDS for that product.

B. Procedure Hazards

Phosphoric acid and its solutions, used for cell regeneration (XI-B-4), are corrosive to body tissue. Avoid contact with the skin. In the event of accidental contact, flush the affected area with copious amounts of water.

VIII. Procedure

A. Operating Conditions

1. Turn the instrument power switch to the ON position.

2. Set the selector switch to the STAND-BY position.

   NOTE: Moisture Mites are normally stored with the power switch ON and the selector switch in the STAND-BY position, in order to ensure the electrolysis cell is dry and ready for use. A small residual current may produce a scale reading between 0 and 1 part per million background moisture on some instruments. Disregard this reading.

B. Calibration

1. Isolate and analyze, either by Karl Fischer titration (XI-B-2) or by Infrared Spectroscopy (XI-B-3), the moisture content in a series of specification grade "Freon" 12 fluorocarbon cylinders and record their moisture contents. Isolate cylinders with moisture contents of ca. 5 ppm and 20 ppm, respectively. Reserve these cylinders as Moisture Mite calibration standards.

2. Remove the flare plug from the Moisture Mite's speed coupler.

3. Connect a delivery tube to a 20 ppm moisture, "Freon" 12 standard cylinder valve, with a speed coupler.

4. Position the cylinder to deliver liquid sample.

B. Calibration (Continued)

5. Ensure that both the vaporizer needle and bypass valves are closed.

   NOTE: The vaporizer needle valve should not be opened before the sample line is purged. This sequence will preclude the introduction of "tramp" moisture into the electrolytic cell.

6. Open the standard cylinder valve.

7. Open the bypass valve and purge the sample line of air and moisture for 30 seconds, then close the bypass valve. The sample line is now liquid-full.

   NOTE: Sample vaporization MUST occur in the vaporizer, because error will be introduced into the analysis if vaporization occurs upstream of the vaporizer. "Freon" 22 moisture analyses are particularly susceptible to premature sample vaporization.

8. Set the standard flow rate to that specified (XII-1) for the chlorofluorocarbon ("Freon" 12) under study, by adjusting the vaporizer control valve.

   NOTE: Do NOT permit the flow rate to exceed the flowmeter's range, because liquid fluorocarbon could flow into the electrolytic cell and desensitize the cell.

9. Set the selector switch to the appropriate position (XII-1) for both the fluorocarbon and moisture range under study.

10. Allow the moisture Mite scale reading to equilibrate according to the following schedule:

Moisture, ppm	
Moisture Range	Equilibrium Reading
0 - 10	± 0.1
0 - 100	± 1.0

   NOTE: Instrument equilibrium usually requires 10-15 minutes.

11. Read and record the standard's moisture content. Go to Step 23.

12. Set the selector switch to the STAND-BY position.

13. Close the vaporizer control valve.

B. Calibration (Continued)

14. Close the standard cylinder valve.

15. IMMEDIATELY open the vaporizer bleed valve to vent the sample line.

NOTE: The delivery tube is liquid-full and must be vented immediately in order to preclude the possibility of hydrostatic pressure buildup with potentially violent pressure release.

16. Close the vaporizer bleed valve, after the sample line has vented.

17. Disconnect the 20 ppm moisture, "Freon" 12 standard cylinder from the delivery tube.

18. Connect a 5 ppm moisture, "Freon" 12 standard cylinder to the delivery tube.

19. Repeat Steps 4 through 16.

20. Disconnect the 5 ppm moisture, "Freon" 12 standard cylinder from the delivery tube.

21. Disconnect the sample line from the Moisture Mite and connect the flare plug to its speed coupler.

22. Replace the flare plug in the Moisture Mite's speed coupler.

23. If Moisture Mite analyses [Steps 11 and 19(11)]:

    * Agree within ± 2 parts per million of the standard's moisture content, the instrument is deemed satisfactory and may be used for sample analyses.

    * Do NOT agree within ± 2 parts per million of the standards moisture content, the instrument requires maintenance (X) and must NOT be used for sample analyses.

C. Sampling

Not applicable.

D. Sample Analysis

1. Connect the sample cylinder to the Moisture Mite as described in VIII-B-2 through 5.

2. Open the cylinder valve.

D. Sample Analysis (Continued)

3. Repeat VIII-B-7.

4. Set the sample flow rate to that specified (XII-1) for the material under analysis, by adjusting the vaporizer control valve.

   NOTE: Do NOT permit sample flow to exceed the flowmeter's range, because liquid fluorocarbon could flow into the electrolytic cell and desensitize the cell.

5. Repeat VIII-B-9 and 10.

6. Read and record the sample's moisture content.

7. Repeat VIII-B-12 through 16.

8. Disconnect the sample cylinder from the delivery tube.

9. Repeat VIII-B-22.

E. Calculations

Not applicable.

IX. Quality Control

1. Analyze a low moisture standard on the Moisture Mite daily or at any time questions arise concerning the analyzer's performance.

2. ,se appropriate CUSUM/PQM procedures established in the Control Laboratory to ensure product quality.

X. Comments

1. Moisture Mite Corrective Maintenance

If Moisture Mite readings exceed the ± 2 parts per million moisture tolerance specified in VIII-B-23, proceed as follows:

a. Apply a high voltage (160 v) to the cell, using the Simpson instrument.

b. If the high voltage treatment does not restore cell function, replace the cell with a new or resensitized one.

c. See XI-B-4 for the cell regeneration procedure.

X. Comments (Continued)

2. If "Freon" 14 fluorocarbon is to be analyzed for moisture, the pressure regulator, sample delivery tube and Moisture Mite must be connected to the sample source and purged slowly for 8 hours to pacify and equilibrate the apparatus before sample analysis.

3. "FARADAY'S LAWS OF ELECTROLYSIS -- The law that: (1) for any electrolyte the amount of electrolysis is proportional to the quantity of electrical current which is passed through a solution; (2) for the same quantity of current passed through solutions of various electrolytes, the amounts liberated at the electrodes are directly proportional to chemical equivalents of the electrolytes. The quantity of current required to deposit one chemical equivalent weight is one FARADAY which is equal to 96,500 coulombs" (XI-A-1)

XI. References

1. N. E. Lange, "Handbook of Chemistry", 10th Ed., McGraw-Hill Company, New York, 1961, pp. 1732.

XII. Appendix

1. Mositure Mite Analytical Settings

APPENDIX 1

MOISTURE MITE ANALYTICAL SETTINGS

Products for Analysis	Selector Switch Position	Sample Flow Rate, mL/min
"Freon" 12	"Freon" 12	50
"Freon" 13	"Freon" 22	70
"Freon" 13B1	"Freon" 22	70
"Freon" 14	"Freon" 22	70
"Freon" 22	"Freon" 22	70
"Freon" 23	"Freon" 22/"Freon" 12	90
FC-31	"Freon" 12	50
FC-32	"Freon" 22	70
"Freon" 12/"Freon" 114	"Freon" 12/"Freon" 114	50-70 (dependent on composition)
"Freon" 14/Nitrogen	"Freon" 12	90
"Freon" 114	"Freon" 114	70
"Freon" 115	"Freon" 12	50
"Freon" 116	"Freon" 12	60
"Freon" C318	"Freon" 114	70
"Freon" 500	"Freon" 12	50
"Freon" 502	"Freon" 22	70
Air	"Freon" 22	70
Nitrogen	"Freon" 22	70
Difluoroethane	"Freon" 12	92

DU PONT

[illegible]

TEST METHOD

E. I. du Pont de Nemours & Company (Inc.)
Du Pont Chemicals

Method No. F3200.037.01.CW
First Revision: October 8, 1985
Page 1 of 14

"FREON"* PRODUCTS

Determination of Boiling Range
Determination of Halides by Paper Test
Determination of Particulate Matter
Determination of Soluble Residue, Goetz Bulb Method

I. Principle

A measured amount of sample is evaporated from a Goetz bulb under controlled bath temperature conditions. The following analyses are conducted during and after sample evaporation:

- The sample's boiling range is the corrected temperature range observed and recorded during the distillation interval between 5 mL and 85 mL of collected distillate.

- Halides are detected visually during sample evaporation, using halide indicator paper, which develops a color when contacted by volatile "halides" such as hydrogen chloride, chlorine or phosgene.

- Particulate matter is determined visually by inspection of the Goetz bulb residue, after complete sample evaporation.

- Soluble residue is determined visually by inspection of the Goetz bulb residue 30 minutes after complete sample evaporation.

II. Applicability

This method is applicable to the routine Goetz bulb determination of the following characteristics -- boiling range, halides by paper test, particulate matter and soluble residue, either individually or in combination -- in neat "Freon" fluorocarbons, "Freon" fluorocarbon blends, "Freon" fluorocarbon azeotropes and mixtures with other compounds and individual "Dymel"* aerosol propellants and propellant blends.

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* Registered U. S. Patent Office

III. Limitations

Owing to the high volatility of many fluorocarbons, it is important that the time elapsed between sample addition into the Goetz bulb and analysis start (boiling) must be minimized, in order to reduce halide impurity losses and also to allow for thermometer temperature equilibration, prior to taking the 5 mL boiling range temperature reading.

Experience has shown the necessity to adhere rigidly to the water bath temperatures specified in the method. Failure to do so will produce erratic boiling ranges and soluble residue results.

Halide analyses must be run in areas devoid of hydrogen chloride, chlorine and phosgene, in order to preclude interferences by these substances.

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

Not required.

B. Precision

1. Single Operator

Not required.

2. Multiple Operator

Not required.

C. Accuracy

Not required.

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Goetz centrifuge tube, 100-mL, graduated, Cat. No. 21123-009, VWR Scientific, Philadelphia, Pa. 19101.
2. Barometer, Fortin type, Cat. No. 13142-857, VWR Scientific.
3. Thermometer, graduated in 0.1°C subdivisions, NBS calibrated, Precision Thermometer and Instrument Co., Southhampton, Pa. 18966.

Thermometer temperature ranges: - 60°C to 5°C
- 5°C to 30°C
25°C to 60°C

V. Special Apparatus (Equivalent apparatus may be substituted) (Continued)

4. Constant temperature water baths thermostatically controlled to ± 1°C.

5. Steam bath

6. Filter paper, Whatman No. 1, Cat. No. 28451-006, VWR Scientific.

7. Carborundum boiling stones, Cat. No. 1590-D30, Thomas Scientific, Philadelphia, Pa. 19105. Grind stones to ca 20 mesh.

VI. Reagents (Reagent grade except as noted)

1. Diphenylamine, Cat. No. EK-00105-5, VWR Scientific.

2. Dimethylaminobenzaldehyde, Cat. No. EK-00095-5, VWR Scientific.

3. Methanol, anhydrous, Cat. No. EK-13032-3, VWR Scientific.

4. Halide test paper.

   Prepare test papers as follows:

   a. Weigh (± 0.01 g) 0.50 g of diphenylamine (DPA) into a 100 mL volumetric flask.

   b. Weigh (± 0.01 g) 0.50 g of dimethylaminobenzaldehyde (DMAB) into the 100 mL volumetric flask.

   c. Fill the flask to volume with methanol and mix thoroughly.

   d. Soak several sheets of Whatman No.1 filter paper in the DPA/DMAB solution (Step 4c).

   e. Dry the papers in hydrogen chloride-chlorine-phosgene free air.

   f. Cut the dry paper (Step 4e) into 0.25 in. x 1.25 in. strips.

   g. Store the dry paper strips (Step 4f) in tightly sealed, amber, screw-capped bottles.

VII. Special Safety Considerations

A. Product Hazards

Compressed, liquefied "Freon" fluorocarbon gases (e.g., "Freon" 12, "Freon" 22, etc.) can cause frostbite if discharged on the person. Avoid contact with the skin.

All "Freon" fluorocarbons dissolve natural oils. Avoid contact with the skin.

A. Product Hazards (Cont.)

Evaporation of "Freon" fluorocarbons should be conducted in a hood, in order to minimize fluorocarbon concentrations in air.

B. Procedure Hazards

Diphenylamine and dimethylaminobenzaldehyde are toxic aromatic amines. Avoid inhalation of their vapors or contact with the skin. In the event of accidental contact, flush the affected area with copious amounts of water.

Methanol is a toxic and flammable substance. Avoid vapor inhalation, contact with the skin and ignition sources.

VIII. Procedure

A. Operating Conditions

None.

B. Calibration

None.

C. Sampling

None.

D. Sample Analysis

Analytical Test Conditions

Select the PRODUCT/ANALYSIS/WATER BATH TEMPERATURE from Appendix I.

1. Adjust the water bath to the initial temperature (± 1°C) specified.
2. Add 2 carborundum chips to a clean, dry, 100 mL, Goetz bulb.
3. Secure the Goetz bulb in the initial evaporation bath specified.
4. Immerse only the cylindrical tip of the Goetz bulb in the evaporation bath.
5. Fill the Goetz bulb with liquid sample, as rapidly as possible, to the 100 mL scribe mark. Proceed immediately to the sections(s) "Determination of Boiling Range" and/or "Determination of Halides by Paper Test" and run concurrently if both analyses are required.

D. Sample Analysis (Cont.)

Determination of Boiling Range

6. Immediately position and clamp the appropriate temperature range calibrated thermometer in the sample so that the bottom of the thermometer's mercury bulb is aligned with the Goetz bulb's 5 mL scribe mark.

7. Continuously observe the thermometer temperature until the reading stabilizes.

8. Read and record the temperature (± 0.1°C) after 5 mL of sample has evaporated (the liquid level in the Goetz bulb is at the 95 mL scribe mark).

9. Continue sample evaporation until vigorous boiling subsides.

10. Reposition the Goetz bulb/thermometer assembly in the final evaporation bath (Appendix I).

11. Continue sample evaporation.

12. Read and record the temperature (± 0.1°C) after 85 mL of sample has evaporated (the liquid level in the Goetz bulb is at the 15 mL scribe mark).

13. Continue sample boil-off until visual evaporation stops.

14. Allow the Goetz bulb to remain in the final evaporation bath for 30 additional minutes.

15. Remove the Goetz bulb from the thermostated bath and place in a Goetz bulb support rack.

16. Read and record both the barometric pressure (± 1 mm Hg) and room temperature (± 0.5°C).

Determination of Halides by Paper Test

17. Immediately suspend a halide test paper in the Goetz bulb (Step 5) opening. Make certain that the test paper does NOT contact the liquid sample, only its distilling vapor.

18. After the sample has evaporated completely (Step 15), inspect the test paper for color.

19. Report test result as follows:

    a. If color is absent, report result as NO HALIDES DETECTED.

    b. If a yellow color is present to any degree, report result as HALIDES PRESENT.

D. Sample Analysis (Cont.)

Determination of Particulate Matter

20. After the sample has evaporated completely (Step 15), examine the empty Goetz bulb for the presence of dirt, rust or other particulate contamination.

21. Report test result as follows:

    a. If particulate matter is absent, report result as NO PARTICULATE MATTER DETECTED.

    b. If particulate matter is present, report results as follows:

        (1) SAMPLE SUBSTANTIALLY FREE OF PARTICUALTE MATTER.

        (2) SAMPLE CONTAINS PARTICUALTE MATTER.

Determination of Soluble Residue

22. After the sample has evaporated completely (Step 15), examine the empty Goetz bulb for the presence of soluble residue.

23. Report test result as follows:

    a. If no organic or oily residue is noted in the Goetz bulb, report result as NO SOLUBLE RESIDUE DETECTED.

    b. If organic or oily residue is noted in the Goetz bulb, report result as follows:

        (1) If the residue appears as a light film in the Goetz bulb, report results as a TRACE OF SOLUBLE RESIDUE PRESENT.

        (2) If a significant amount of soluble residue is found in the Goetz bulb, measure the amount present volumetrically and report residue volume to the nearest 0.05 mL.

E. Calculations

Calculate the Boiling Point/Range Temperature(s) for Fluorocarbons Boiling at or Below 4°C and for "Freon" 11 Samples as Follows

1. Apply a temperature correction to the barometric pressure (Appendix II or III) reading obtained in VIII-D-16. Record (± 0.001 mm Hg) the barometric pressure correction value.

E. Calculations (Cont.)

2. Calculate the corrected barometric pressure as follows:

$$P = B - C$$

Where: P = corrected barometric pressure, mm Hg

B = observed barometric pressure, mm Hg (VIII-D-16)

C = correction value, mm Hg (Step 1)

Record (± 1 mm Hg) the corrected barometric pressure.

3. Calculate the 5 mL boiling point temperature using the following equation:

$$BP_5 = T + C1 + C2$$

Where: BP_5 = 5 mL boiling point, °C

T = observed boiling temperature, °C (VIII-D-8)

$C1$ = thermometer temperature correction, °C (Step 1)

$C2$ = barometer/temperature correction factor, °C (Appendix II or III)

Report (± 0.1°C) the 5 mL corrected sample boiling point temperature.

4. Calculate the 85 mL boiling point temperature using the following equation:

$$BP_{85} = T + C1 + C2$$

Where: BP_{85} = 85 mL boiling point, °C

T = observed boiling temperature, °C (VIII-D-12)

$C1$ = thermometer temperature correction, °C (Step 1)

$C2$ = barometer/temperature correction factor, °C (Appendix II or III)

Report (± 0.1°C) the 85 mL corrected sample boiling point temperature.

E. Calculations (Continued)

Calculate the Boiling Point/Range Temperature(s) for "Freon" Fluorocarbons with Boiling Points $\geq$ 4°C as Follows (Exclude "Freon" 11)

5. Calculate the boiling point temperature correction factor using the following equation:

$$C = 0.00012 \times (760-P) \times (273.18 + B)$$

Where: C = boiling point correction factor, °C

P = observed barometric pressure, mm Hg (VIII-D-16)

B = observed boiling temperature, °C (VIII-D-8 or 12)

Record (± 0.01°C) the boiling point temperature correction.

6. Calculate the boiling point temperature using the following equation:

$$BP = B + C + C1$$

Where: BP = product boiling point, °C

B = observed boiling temperature, °C (VIII-D-8 or 12)

C = boiling point correction factor, °C (Step 5)

C1 = thermometer temperature correction, °C (Appendix II or III)

Report (± 0.1°C) the corrected sample boiling temperature.

7. Calculate the sample's boiling range as follows:

$$Br = T_2-T_1$$

Where: T_2 = corrected BP after 85 mL of sample distilled, °C (Step 6)
T_1 = corrected BP after 5 mL of sample distilled, °C (Step 6)

Report (± 0.1°C) the sample's boiling temperature range.

IX. Quality Control

Use appropriate CUSUM/PQM procedures, as established in the Control Laboratory, to ensure individual product quality.

X. Comments

1. Soluble residue (VIII-D-22,23) may be classified as either organic or oily residue, by placing the Goetz bulb (VIII-D-15) in a steam bath for 5 minutes. If residue remains in the Goetz bulb after the 5 minute steam-heating period, it is assumed to be oil.

2. The designations particulate matter and soluble residue used in this method apply only to data obtained by the Goetz bulb technique.

XI. References

None

XII. Appendix

1. Product/Analysis/Water Bath Temperature Selection Chart.

2. Barometric Pressure Correction at Different Temperatures

3. Temperature Corrections at Different Barometric Pressures

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APPENDIX I

Product/Analysis/Water Bath Temperature Selection Chart

"Freon" Product	Analyze For					Water Bath Temperature
	Halide	Boiling Range	Particulate Matter	Soluble Residue	Boiling Point °C	
"Freon" 12/ Propane	x		x	x		Room temperature until vigorous boiling has subsided; transfer to 34°C bath.
"Freon" 12/ "Freon" 11/ Isobutane	x		x	x		Room temperature until vigorous boiling has subsided, transfer to + 54°C bath.
"Freon" 12/ "Freon" 11	x		x	x		Room temperature until vigorous boiling has subsided, transfer to + 54°C bath.
"Freon" 12/ "Freon" 11S	x		x			Room temperature until vigorous boiling has subsided, transfer to + 54°C bath.
"Freon" 12/ "Freon" 114	x		x	x		Room temperature until vigorous boiling has subsided, transfer to + 34°C bath.
"Freon" 11	x	x	x	x	23.8	+ 54°C
"Freon" 11B	x		x			+ 54°C
"Freon" 11S	x		x			+ 54°C
"Freon" 12	x	x	x	x	-29.8	Room temperature for boiling range; transfer to 0°C bath for soluble residue.

APPENDIX I (Cont.)

Product/Analysis/Water Bath Temperature Selection Chart

"Freon" Product	Analyze For					Water Bath Temperature
	Halide	Boiling Range	Particulate Matter	Soluble Residue	Boiling Point °C	
"Freon" 22	x	x	x	x	-40.8	Room temperature for boiling range, transfer to 0°C bath for soluble residue.
"Freon" 113			x	x	47.6	Submerge the Goetz bulb to neck in + 54°C bath.
"Freon" 114	x	x	x	x	3.6	+ 34°C
"Freon" 114B2	x		x	x	47.5	Steam bath to 35 mL of Goetz bulb with tip immersed in water
"Freon" 115	x	x	x	x	-38.7	Room temperature for boiling range, transfer to 0°C bath for soluble residue
"Freon" 501	x	x	x	x	-42.1	Room temperature for boiling range, transfer to 0°C bath for soluble residue.
"Freon" 502	x	x	x	x	-45.6	Room temperature for boiling range, transfer to 0°C bath for soluble residue.
"Freon" T-E Solvent			x	x		Steam bath to 35 mL of Goetz bulb with tip immersed in water

APPENDIX II

Barometric Pressure Correction at Different Temperatures

Temp. °C	Millimeters of Mercury 750	751	752	753	754	755	756	757	758	759
20	2.440	2.443	2.446	2.449	2.452	2.455	2.458	2.461	2.464	2.467
20.5	2.500	2.504	2.507	2.511	2.514	2.518	2.521	2.525	2.528	2.532
21	2.560	2.564	2.568	2.572	2.576	2.580	2.584	2.588	2.592	2.596
21.5	2.625	2.629	2.632	2.636	2.639	2.643	2.646	2.650	2.653	2.657
22	2.690	2.693	2.696	2.699	2.702	2.705	2.708	2.711	2.714	2.717
22.5	2.750	2.753	2.756	2.759	2.762	2.765	2.768	2.771	2.774	2.777
23	2.810	2.813	2.816	2.819	2.822	2.825	2.828	2.831	2.834	2.837
23.5	2.870	2.874	2.877	2.881	2.884	2.888	2.891	2.895	2.898	2.902
24	2.930	2.934	2.938	2.942	2.946	2.950	2.954	2.958	2.962	2.966
24.5	2.990	2.994	2.998	3.002	3.006	3.010	3.014	3.018	3.022	3.026
25	3.050	3.054	3.058	3.062	3.066	3.070	3.074	3.078	3.082	3.086
25.5	3.110	3.114	3.118	3.122	3.126	3.130	3.134	3.138	3.142	3.146
26	3.170	3.174	3.178	3.182	3.186	3.190	3.194	3.198	3.202	3.206
26.5	3.230	3.235	3.239	3.244	3.249	3.254	3.258	3.263	3.267	3.272
27	3.290	3.295	3.300	3.305	3.310	3.315	3.320	3.325	3.330	3.335
27.5	3.350	3.355	3.360	3.365	3.370	3.375	3.380	3.385	3.390	3.395
28	3.410	3.415	3.420	3.425	3.430	3.435	3.440	3.445	3.450	3.455
28.5	3.475	3.480	3.484	3.489	3.493	3.498	3.502	3.507	3.511	3.516
29	3.540	3.544	3.548	3.552	3.556	3.560	3.564	3.568	3.572	3.576
29.5	3.600	3.605	3.609	3.614	3.618	3.623	3.627	3.632	3.636	3.641
30	3.660	3.665	3.670	3.675	3.680	3.685	3.690	3.695	3.700	3.705
30.5	3.720	3.725	3.730	3.735	3.740	3.745	3.750	3.755	3.760	3.765
31	3.780	3.785	3.790	3.795	3.800	3.805	3.810	3.815	3.820	3.825
31.5	3.840	3.845	3.850	3.855	3.860	3.865	3.870	3.875	3.880	3.885
32	3.900	3.905	3.910	3.915	3.920	3.925	3.930	3.935	3.940	3.945

(CONTINUED ON NEXT PAGE)

APPENDIX II (Cont.)

Barometric Pressure Correction at Different Temperatures

Millimeters of Mercury									
760	761	762	763	764	765	766	767	768	Temp. °C
2.470	2.474	2.478	2.482	2.486	2.490	2.494	2.498	2.502	20
2.535	2.539	2.542	2.546	2.549	2.553	2.556	2.560	2.563	20.5
2.600	2.603	2.606	2.609	2.612	2.615	2.618	2.621	2.624	21
2.660	2.664	2.667	2.671	2.674	2.678	2.681	2.685	2.688	21.5
2.720	2.724	2.728	2.732	2.736	2.740	2.744	2.748	2.752	22
2.780	2.784	2.788	2.792	2.796	2.800	2.804	2.808	2.812	22.5
2.840	2.844	2.848	2.852	2.856	2.860	2.864	2.868	2.872	23
2.905	2.909	2.913	2.917	2.921	3.925	2.929	2.933	3.937	23.5
2.970	2.974	2.978	2.982	2.986	3.990	2.994	2.998	3.002	24
3.030	3.034	3.038	3.042	3.046	3.050	3.054	3.058	3.062	24.5
3.090	3.094	3.098	3.102	3.106	3.110	3.114	3.118	3.122	25
3.150	3.155	3.159	3.164	3.168	3.173	3.177	3.182	3.186	25.5
3.210	3.215	3.220	3.225	3.230	3.235	3.240	3.245	3.256	26
3.275	3.280	3.284	3.289	3.293	3.298	3.302	3.307	3.311	26.5
3.340	3.344	3.348	3.352	3.356	3.360	3.364	3.368	3.372	27
3.400	3.406	3.409	3.414	3.418	3.423	3.427	3.432	3.436	27.5
3.460	3.465	3.470	3.475	3.480	3.485	3.490	3.495	3.500	28
3.520	3.525	3.530	3.535	3.540	3.545	3.550	3.555	3.580	28.5
3.580	3.585	3.590	3.595	3.600	3.605	3.610	3.615	3.620	29
3.645	3.650	3.654	3.659	3.663	3.668	3.672	3.677	3.681	29.5
3.710	3.714	3.718	3.722	3.726	3.730	3.734	3.738	3.742	30
3.770	3.775	3.799	3.784	3.788	3.793	3.797	3.802	3.806	30.5
3.830	3.835	3.840	3.845	3.850	3.855	3.860	3.865	3.870	31
3.890	3.895	3.900	3.905	3.910	3.915	3.920	3.925	3.930	31.5
3.950	3.955	3.960	3.965	3.970	3.975	3.980	3.985	3.990	32

APPENDIX III

Temperature Corrections At Different Barometric Pressures

Barometric Pressures mm	Barometric Pressures in.	Temperature Corrections Fluorocarbons, BP $\leq$ 4°C °C	Temperature Corrections "Freon" 11 °C
745	29.330	0.05	0.45
746	29.370	0.02	0.42
747	29.409	- 0.01	0.39
748	29.449	- 0.04	0.36
749	29.488	- 0.07	0.33
750	29.527	- 0.10	0.30
751	29.567	- 0.13	0.27
752	29.606	0.16	0.24
753	29.645	- 0.19	0.21
754	29.685	- 0.22	0.18
755	29.724	- 0.25	0.15
756	29.764	- 0.28	0.12
757	29.803	- 0.31	0.09
758	29.842	- 0.34	0.06
759	29.882	- 0.37	0.03
760	29.921	- 0.40	0.00
761	29.960	- 0.43	- 0.03
762	30.000	- 0.46	- 0.06
763	30.039	- 0.49	- 0.09
764	30.078	- 0.52	- 0.12
765	30.118	- 0.55	- 0.15
766	30.157	- 0.58	- 0.18
767	30.197	- 0.61	- 0.21
768	30.236	- 0.64	- 0.24
769	30.275	- 0.67	- 0.27
770	30.315	- 0.70	0.30
771	30.354	- 0.73	- 0.33
772	30.393	- 0.76	- 0.36
773	30.433	- 0.79	- 0.39
774	30.472	- 0.82	- 0.42
775	30.512	- 0.85	- 0.45
776	30.551	- 0.88	- 0.48
777	30.590	- 0.91	- 0.51
778	30.630	- 0.94	- 0.54
779	30.669	- 0.97	- 0.57

NOTES:

- * THE FLUOROCARBON, BP $\leq$ 4°C TEMPERATURE CORRECTIONS TABULATED ABOVE ARE COMPRISED OF TWO FACTORS. THE TEMPERATURE CORRECTION INCLUDES A CORRECTION FOR SUPERHEATING EFFECTS AND A CORRECTION FOR BOILING POINT VARIANCE FROM STANDARD BAROMETRIC PRESSURE.

- * THE "FREON" 11 TEMPERATURE CORRECTIONS ARE FOR BOILING POINT VARIANCE FROM STANDARD BAROMETRIC PRESSURE.

DU PONT

"FREON" Fluorocarbon Products

TEST METHOD

"FREON" PRODUCTS

DETERMINATION OF FREE ACIDITY

METHOD F3200.075.01.CW(P)

E. I. du Pont de Nemours & Company (Inc.)
Du Pont Chemicals

Method No. F3200.075.01.CW(P)
First Revision: December 2, 1988
Page 1 of 10

"FREON"* PRODUCTS

Determination of Free Acidity - Electrical Conductivity Method

I. Principle

Ionizable acid impurities in "Freon" products are extracted into deionized water of measured, very low conductivity. Any measured increase in aqueous conductivity is attributed to the sample acidity (ionic strength). Calculations are made relating sample conductivity to free acidity, calculated as hydrochloric acid, in the sample.

II. Applicability

The method is applicable to the routine conductimetric determination of ionizable acidity in neat "Freon" fluorocarbons, "Freon" fluorocarbon blends and most fluorocarbon azeotropes and mixtures with other compounds.

III. Limitations

Aqueous conductivity is a measure of an aqueous solution's ability to carry current between two electrodes, per unit time. Any substance which ionizes when dissolved in water will interfere with the determination, producing erroneously high results.

The test must be run in an area where the air is free from acidic (e.g. carbon dioxide, hydrogen chloride, etc.) or alkaline (e.g. ammonia, etc.) vapors. These substances ionize in aqueous solution, increase the sample's apparent ionic strength and interfere with the analysis.

"Freon" 11 samples spiked with phosgene gave very low phosgene recoveries when analyzed by the aqueous conductivity method, owing to its very slow hydrolysis rate in deionized ("conductivity") water. Phosgene as well as most other latent acid precursors are not quantifiable by this procedure.

All equipment used in this analysis must be thoroughly clean, ion-free and dedicated for conductivity analysis only, in order to minimize ionic contamination.

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* Registered U.S. Patent Office

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

No study has been made of the method's sensitivity. The method's sensitivity is estimated to be ca. 1 part per billion (ppb), based on a minimum reproducible electrical conductivity of 0.05 µmho and 150 grams of sample, at 18°C.

B. Precision

1. Single Operator

The average analysis ($\bar{X}$), standard deviation (s) and 95% confidence limits (95% CL) established for the single operator precision of the method are as follows:

	$\bar{X}$	s	95% CL
Free acidity, %	To be determined		

The above data were calculated from a standard analyzed by xxx analyst(s) over a period of xxx.

2. Multiple Operator

The average analysis ($\bar{X}$), standard deviation (s) and 95% confidence limits (95% CL) established for the multiple operator precision of the method are as follows:

	$\bar{X}$	s	95% CL
Free acidity, %	To be determined		

The above data were calculated from xxx replicate analyses of xxx sample performed by xxx analysts over a period of xxx.

C. Accuracy

To be determined.

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Conductivity bridge, Model No. 910-2C, The Foxboro Co., Foxboro, MA 02035.
2. Conductivity cell, 0.1 cell constant, Model No. 910-0.1T, The Foxboro Co.

V. Special Apparatus (Equivalent apparatus may be substituted) (Cont'd)

3. Heat gun, Varitemp, Cat. No. 26405-30, VWR Scientific, Philadelphia, Pa. 19101.

4. Graduated cylinder, polypropylene, 25-mL, Cat. No. 24774-036, VWR Scientific.

5. Graduated cylinder, polypropylene, 100-mL, Cat. No. 24774-069, VWR Scientific.

6. Polypropylene tube, 1/4 in. i.d., Cat. No. TR-6400-32, Cole-Palmer Instrument Co., Chicago, Ill. 60648.

7. Gas dispersion tube, polyethylene, 1/4 in. i.d. x 12 in., 70 µm porosity, Cat. No. TR-6614-20, Cole-Palmer Instrument Co.

8. Bottle, polyethylene, wide mouth, screw capped, 250-mL, Cat. No. 16125-060, VWR Scientific. Calibrate and mark the bottles at the 20 mL (grad. cyl.) volume.

9. Hypodermic syringes of appropriate volume to satisfy procedural need.

10. Brushes, soft, appropriate sizes to clean plasticware used in the analysis.

11. Beaker, polyethylene, 250 mL, Cat. No. 13890-080, VWR Scientific.

12. Water purification system, 3 modules with pump, Nanopure* II, Barnstead, Cat. No. 26302-300, VWR Scientific.

VI. Reagents (Reagent grade except as noted)

1. Hydrochloric acid, 0.01 N standard in SDA-2B ethyl alcohol (RH 525.000.01.CW). Store the standard in separate 50-mL serum capped vials.

2. "Freon" 11, special (analyzed to have an aqueous conductance of < 0.5 µmho/cm)

3. Distilled and deionized water, aqueous conductivity ≤ 0.5 µmho/cm (X-1), prepared immediately prior to use by passing distilled water through a water demineralizer (V-12) and analyzing it with a conductivity bridge (V-1, 2). DO NOT STORE DEIONIZED WATER FOR USE IN THIS ANALYSIS.

4. RBS-35 cleaner, Cat. No. P127951, VWR Scientific.

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*Registered U.S. Patent Office

VII. Special Safety Considerations

A. Product Hazards

Compressed, liquefied "Freon" fluorocarbon gases (e.g. "Freon" 12, "Freon" 13B1, "Freon" 23, etc.) can cause frostbite if discharged on the person. Avoid contact with the skin.

All "Freon" fluorocarbons dissolve natural oils. Avoid contact with the skin. For more information, refer to the specific MSDS for that product.

B. Procedure Hazards

Evaporation of "Freon" fluorocarbons should be conducted in a hood, in order to minimize fluorocarbon concentrations in air.

Hypodermic syringes must be handled with care in order to preclude the possibility of accidental puncture or injection. Shield hypodermic syringe needles when in use.

VIII. Procedure

A. Operating Conditions

1. Equipment Cleaning

USE ONLY PLASTIC LABWARE FOR ELECTRICAL CONDUCTIVITY MEASUREMENTS. ALL EQUIPMENT USED IN THIS PROCEDURE MUST BE CLEANED ACCORDING TO THE FOLLOWING PROCEDURE BEFORE USE (X-2).

a. Wash (brush) all labware with an aqueous solution of RBS-35 cleaner.

b. Rinse thoroughly with warm tap water.

c. Rinse thoroughly with distilled water.

d. Rinse thoroughly with deionized water (VI-5).

e. Store cleaned labware, upside down, on clean, dry paper towels.

2. Conductivity Bridge/Cell

a. Turn the conductivity bridge switch to the STANDBY position.

b. Adjust SETPOINT knob to position 18.

c. Rinse the conductivity cell thoroughly with deionized water.

d. Store the conductivity cell in a polyethylene beaker, which contains deionized water (X-2), when not in use.

B. Calibration

1. Add special "Freon" 11 fluorocarbon (X-3) (to the 20-mL calibration mark) of a clean, screw-capped, polyethylene (PE) bottle.

2. Immediately cap the bottle and shake contents vigorously for 30 ± 1 seconds.

3. Discard the "Freon" 11 fluorocarbon.

4. Repeat Step 1.

5. Immediately add 100 mL (graduate cylinder) of deionized (DI) water to the PE bottle (Step 4).

6. Immediately cap the bottle and shake the contents vigorously for 120 ± 5 seconds.

7. Rinse the conductivity cell with DI water, and shake it "dry."

8. Uncap the PE bottle (Step 6) and dip the conductivity cell into the water layer. Use whatever meter RANGE setting provides the highest sensitivity.

9. Read and record the conductivity value to the nearest interpretable unit scale value (see below).

   - Black scale (0.0-2.0), report results ± 0.01 µmho
   - Green scale (0-20), report results ± 0.1 µmho
   - Red scale (0-200), report results ± 1 µmho

10. Remove the conductivity cell from the solution (Step 8), rinse with DI water and store in a PE beaker containing DI water.

11. Immediately cap the PE bottle (Step 8).

12. Fill and flush a clean, dry, 50 microliter syringe several times with 0.01N, standard, hydrochloric acid in SDA-2B ethanol. Discard the rinsings.

13. Fill the microliter syringe to a volume of 30 µL.

14. Hold the syringe vertically with the needle pointed up.

15. Carefully push the needle through a facial tissue to absorb expelled liquid.

16. Tap the syringe barrel gently with the finger to permit air bubbles to rise to the top of the barrel.

B. Calibration (Cont'd)

17. Slowly depress the plunger until the plunger tip is alligned exactly with the 20 µL scribe mark.

18. Remove the facial tissue from the needle wiping away liquid droplets in the process.

19. Immediately uncap the PE bottle (Step 11).

20. "Inject" the syringe (Step 18) content into the PE bottle. Touch-off the solution on the needle tip in the DI water.

21. Immediately cap the bottle (Step 20).

22. Shake the bottle and its contents vigorously for 120 ± 5 seconds.

23. Repeat Step 7.

24. Uncap the PE bottle (Step 23) and dip the conductivity cell into the water layer. Use whatever RANGE setting provides the highest sensitivity.

25. Read and record the conductivity value to the nearest interpretable unit scale value (Step 9).

26. Repeat Step 10.

27. Calculate the calibration factor using the following relationship:

    a. Calculate the nanograms (ng) of hydrochloric acid used in the standard from the following equation:

$$\text{ng HCL} = \frac{V \times N \times MEQ \times 10^9}{10^3}$$

Where: ng HCl = nanograms of HCl in the standard

V = HCl volume, µL (Step 17)

N = HCl normality (VI-1)

MEQ = HCl milliequivalent weight = 0.036461

10^9 = conversion factor, ng/g

10^3 = conversion factor, µL/mL

Record results to the nearest unit.

B. Calibration (Cont'd)

b. Calculate the calibration factor as follows:

$$\text{ng HCl}/\mu\text{mho} = \frac{A}{(C2-C1)}$$

Where: ng HCl/µmho = calibration factor
A = ng HCl in standard (Step 27a)
C1 = blank conductivity, µmho (Step 9)
C2 = standard conductivity, µmho (Step 25)

28. Repeat steps 1-27 at least six times.

Record results to the nearest 0.01 unit. Use the average of the six calibration factors for sample calculations.

C. Sampling

Volatile Samples

1. Repeat VIII-B-1 through 11.
2. Weigh (± 0.01 g) the capped sample cylinder. Record the weight.
3. Remove the cylinder valve cap and isolate.
4. Attach a PE gas dispersion tube to the sample cylinder valve.
5. Clamp the cylinder (valve down) on a ring stand.
6. Uncap the PE bottle (Step 1) and lower the gas dispersion tube (Step 4) into the water, until it is a few millimeters from the bottom of the bottle, then clamp the cylinder to maintain that position.
7. Open the cylinder valve so that ca. 150 grams of sample are added to the bottle over ca. a 60-minute period.
8. Heat the sample cylinder/gas dispersion tube/PE beaker moderately, with a Varitemp heat gun in order to preclude moisture condensation and/or ice formation on or in the equipment.
9. Close the cylinder valve and immediately remove the gas dispersion tube from the bottle.

C. Sampling (Cont'd)

10. Immediately cap the bottle.

11. Remove the gas dispersion tube from the cylinder valve.

12. Recap the cylinder with its original cap (Step 3).

13. Weigh (± 0.01 g) the capped cylinder. Record the weight.

14. Calculate sample weight as follows:

    grams sample = grams (Step 2 - Step 13)

15. Proceed to VIII-D-1.

Liquid Samples

16. Repeat VIII-B-1 through 11.

17. Weigh (± 0.01 g) the PE bottle (Step 16). Record the weight.

18. Uncap the PE bottle and immediately add ca. 150 grams of sample to the bottle.

19. Immediately recap the PE bottle.

20. Weigh (± 0.01 g) the PE bottle (Step 19). Record the weight.

21. Calculate the sample weight as follows:

    grams sample = grams (Step 20 - Step 17)

22. Proceed to VIII-D-1.

D. Sample Analysis

1. Shake the bottle and its contents vigorously for 120 ± 5 seconds.

2. Repeat VIII-B-7 and 8.

3. Read and record the conductivity value to the nearest interpretable unit scale value (VIII-B-9).

4. Repeat VIII-B-10.

E. Calculations

1. Calculate the sample's free acidity using the following equation:

$$\text{ppb HCl} = \frac{(C2-C1) \times F}{W}$$

Where: ppb HCl = free acidity
C1 = blank conductivity, µmho/cm (VIII-D-3)
C2 = sample conductivity, µmho/cm (VIII-D-3)
F = calibration factor, ng HCl/µmho (VIII-B-27b)
W = sample weight, grams
- volatile samples (VIII-C-14)
- liquid samples (VIII-C-21)

Report results to the nearest part per billion.

IX. Quality Control

1. Run the calibration standard at least once per week, or at any time questions arise concerning the method's performance.

2. Use appropriate CUSUM/PQM procedures to ensure "Freon" fluorocarbon product quality.

X. Comments

1. If the DI water conductivity is > 0.5 µmho/cm, and the Nanopure II water system appears normal, reclean the apparatus and remeasure the DI water's aqueous conductance. If DI water conductivity values > 0.5 µmho/cm are still obtained, replace the ion exchange cartridges.

2. The conductivity cell should be stored in DI water. DI water will absorb ionizable gases (III) which will increase its conductivity. In order to minimize errors resulting from cell contamination, storage water should be changed whenever its conductivity exceeds 1.0 µmho/cm. During equipment start-up, the cell should be rinsed in freshly deionized water as often as required to remove adsorbed ions.

3. The special "Freon" 11 added to the PE bottle is used to provide a vapor blanket to exclude carbon dioxide and other gaseous contaminants from entering the bottle when the cap is removed.

4. Parts per billion = $\frac{\text{nanograms}}{\text{grams}} = \frac{\text{grams} \times 10^9}{\text{grams}}$

X. Comments (Cont'd)

5. The Foxboro Co. Model 910-2C conductivity bridge specifies the use of a 0.1 cm cell. No cell-constant factor is required in the calculation because the instrument is electronically self-compensated for cell constant.

XI. References

1. The Foxboro Co., Catalog No. 683, 1982.

2. The Foxboro Co., Technical Bulletin TI 612-112, 1982.

XII. Appendix

None

DU PONT

"FREON" Fluorocarbon Products.

TEST METHOD

E. I. du Pont de Nemours & Company (Inc.)
Chemicals & Pigments Department

Method No. F3200.523.01.CW
First Revision: August 9, 1989
Page 1 of 5

"FREON"* PRODUCTS

Determination of Halide Ion
Visual, Turbidimetric Procedure

I. Principle

A sample is dissolved in methanol. Methanolic silver nitrate is added to the solution to precipitate halide ion. The turbidity developed by silver halide formation is determined visually.

Test chemistry is defined by the following chemical equation:

$$AgNO_3 + HX \rightarrow AgX + HNO_3$$

X = Halide ion.

II. Applicability

This qualitative method is applicable to the routine, visual, turbidimetric, determination of halide ion in neat "Freon" fluorocarbons, "Freon" fluorocarbon blends and most fluorocarbon azeotropes and mixtures with other compounds.

III. Limitations

The approximate ratio of methanol to "Freon" sample specified in the Procedure must be maintained. An excess of "Freon" sample can cause silver nitrate to precipitate from solution and produce a false-positive halide test.

Other ions, such as cyanide ion, also react with silver ion to produce insoluble silver compounds and would interfere with the determination. These ions are not normally present in "Freon" products.

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

Not applicable.

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* Registered U.S. Patent Office

B. Precision

1. Single Operator

Not applicable.

2. Multiple Operator

Not applicable.

C. Accuracy

This procedure has proven to be a satisfactory qualitative test for halide ion in "Freon" fluorocarbons.

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Midget bubbler with fritted inlet tube, 30-mL, Cat. No. LG-6893-102, Labglass Co., Vineland, NJ 08360.

2. Nupro needle valve, Cat. No. SS-4MG, Wilmington Valve and Fitting Co., New Castle, Del. 19720.

   a. Adapt a two-inch length of 1/4-in od, s/s tube to the outside of the valve and clamp (wire) an 8-in. length of Neoprene tube to it.

   b. Adapt a one-inch length of 1/4-in od, s/s tube to the inlet side of the valve and adapt the other end to a 1/4-in., s/s, female flare fitting.

VI. Reagents (Reagent grade except as noted)

1. Methanol, absolute, E. I. du Pont de Nemours and Co.

2. Silver nitrate, methanolic, saturated (RS075.000.01.CW)

VII. Special Safety Considerations

A. Product Hazards

1. Compressed, liquefied "Freon" fluorocarbon gases (e.g. "Freon" 12, "Freon" 22, etc.) can cause frostbite if discharged on the person. Avoid contact with the skin.

2. All "Freon" fluorocarbons dissolve natural oil. Avoid contact with the skin.

B. Procedure Hazards

Methanol is a toxic and highly flammable substance. Avoid contact with the person and ignition sources.

VIII. Procedure

A. Operating Conditions

None

B. Calibration

None

C. Sampling

Volatile Samples

1. Add 10 mL (grad cyl) of methanol to the midget bubbler receiver.
2. Add 3-4 drops of saturated methanolic silver nitrate solution to the midget bubbler receiver and swirl the receiver to mix the contents thoroughly.
3. Assemble the midget bubbler.
4. Clamp the assembled midget bubbler to a ring stand.
5. Clamp the sample cylinder, upright, to another ring stand.
6. Connect the needle valve assembly (VI-2) to the sample cylinder valve.
7. Connect the Neoprene tube to the midget bubbler (Step 4).
8. Place the sample cylinder ring stand (Step 5) on a balance with the midget bubbler assembly adjacent to it.
9. Check to ensure that the needle valve is closed.
10. Open the cylinder valve.
11. Open the needle valve and slowly "sparge" 11 ± 1 grams of sample into the methanolic silver nitrate solution.
12. Close the cylinder valve. Allow the needle valve to remain open until bubbling, exit the fritted tube, stops.
13. Close the needle valve.
14. Disassemble the apparatus.
15. Proceed to VIII-D-1.

C. Sampling (Cont.)

Liquid Samples

16. Add 5 mL (grad cyl) of methanol to a test tube.

17. Add 3-4 drops of saturated methanolic silver nitrate solution to the test tube and swirl the tube to mix the contents thoroughly.

18. Add 5 mL (grad cyl) of sample to the test tube.

19. Stopper the test tube and shake the contents vigorously, for 15 seconds.

    NOTE: Vent pressure in the test tube.

20. Proceed to VIII-D-1.

D. Sample Analysis

Qualitative Determination of Halide Ion

1. Visually examine the sample solution for turbidity.

    NOTE: Turbidity is present if the sample solution appears "milky" (cloudy) to any degree.

2. Report test results as follows:

    a. If turbidity is absent report results as NO HALIDE DETECTED.

    b. If turbidity is present report results as HALIDE PRESENT.

E. Calculations

None

IX. Quality Control

Use appropriate CUSUM/PQM procedures to ensure individual product quality.

X. Comments

None.

XI. References

None

XII. Appendix

None

APPENDIX F

Airbus: 10,000 Jets by Century's End

The following article, written by Arthur Reed, was originally published in the May 1990 issue of Air Transport World Magazine. It is reprinted in this feasibility study with the permission of Airbus Industrie and Air Transport World Magazine.

FORECAST

Airbus: 10,000 jets by century's end

European consortium anticipates that it will take 20% of narrowbody sales and 45% of widebodies over the next 20 years. *By Arthur Reed.*

London—Airbus Industrie, in its most recent forecast of the market for jet airliners, says that the prospects for aircraft manufacturing over the coming 20 years, "remain encouraging."

The world's airlines are at the height of a buying circle, with more than 3,000 aircraft ordered but not yet delivered. A large percentage of the recent order wave was for narrowbody aircraft, confirming earlier forecasts for the replacement peak of first-generation jets.

The surge in widebody orders was influenced largely by continued traffic growth, coupled with increasing congestion problems on the ground and in the air. As a result of the latter, it could be predicted that widebody aircraft would be increasingly in demand during the 1990s and beyond. "As a consequence, average aircraft size, which now stands at 167 seats, will increase to 230 seats by the end of the forecast period," the forecast said.

Growth of 5%, 7%

RPK growth over the next 20 years is expected to be in the range of 5% a year, while it predicted that freight tonne-kms will increase at a worldwide rate two percentage points higher. This growth will create a global jet fleet of 10,000 aircraft by the turn of the century and almost 13,000 by the year 2008.

Airbus said it considers that the current order flow will not continue at the rate that the industry enjoyed during recent years. Nevertheless, 12,000 new aircraft will be delivered to the airlines through to the year 2008, to satisfy traffic growth and to replace aircraft that will have reached the end of their structural or economic lives. Deliveries will be split almost equally between narrowbody and widebody aircraft.

"Despite strong variations in the airlines' annual order volumes, the average yearly delivery rate for new civil-jet aircraft will be in the order of 500–600 aircraft for the next 20 years. This is equivalent to a total investment value of around $700 billion, in 1989 terms."

Airbus Industrie remains confident that the air-transport industry has a healthy future. But it points out that the industry is undergoing structural changes that make any traffic and capacity forecast beyond a 10-year period "somewhat uncertain."

A widespread range of traffic growth rates was noticeable in different parts of the world. Replacement of old equipment and related capital requirements will affect North America more than other regions. Deregulation or liberalization tendencies might change the airlines' competitive environment and marketing behavior and growing system constraints will put physical limits on the future expansion of flight frequency.

Regional air-traffic development and the demand for aircraft would be subject to the following influencing factors:

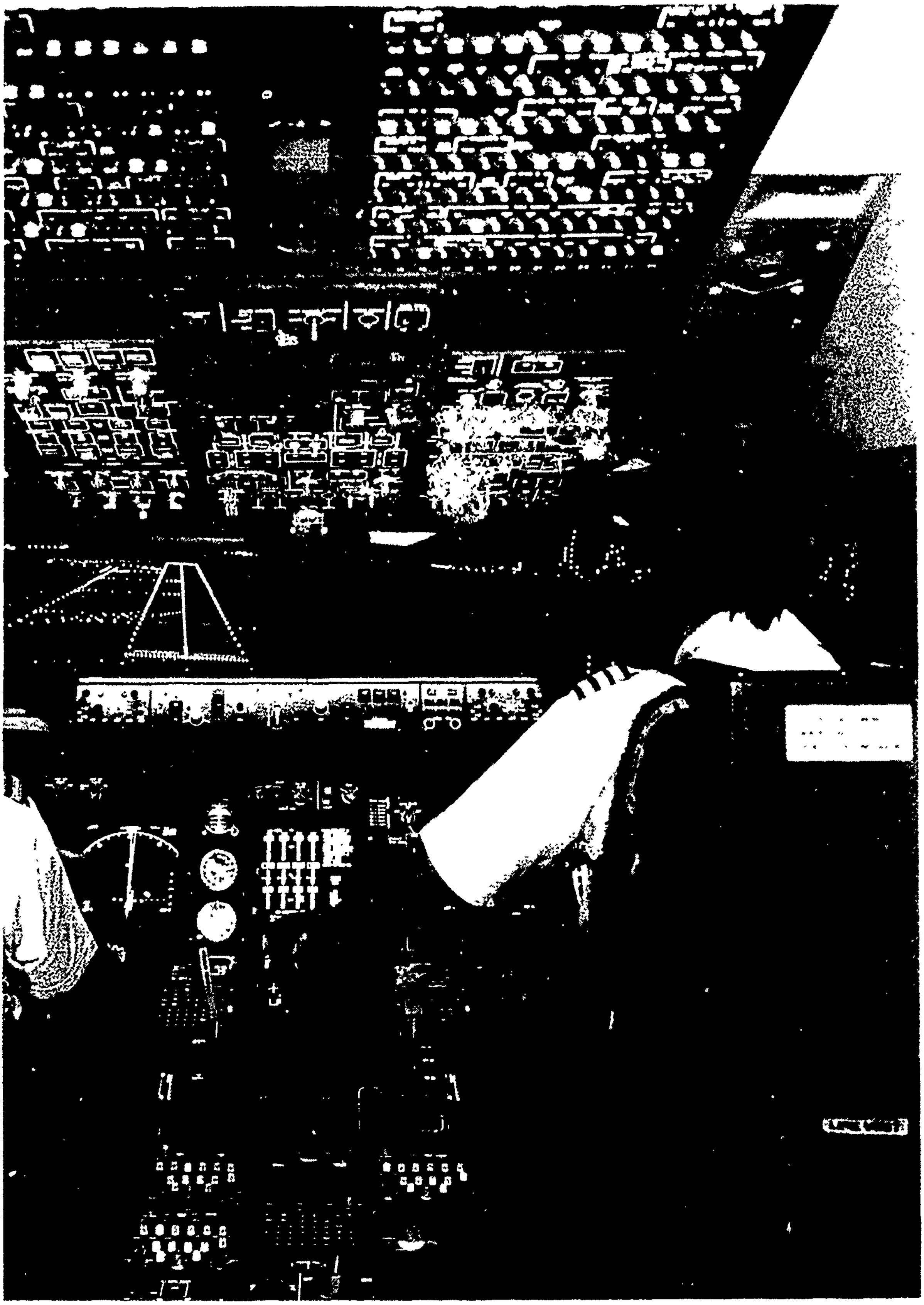
- Europe, excluding the U.S.S.R. and East European countries: Average traffic growth; liberalization; system capacity and operational constraints; environmental ac-

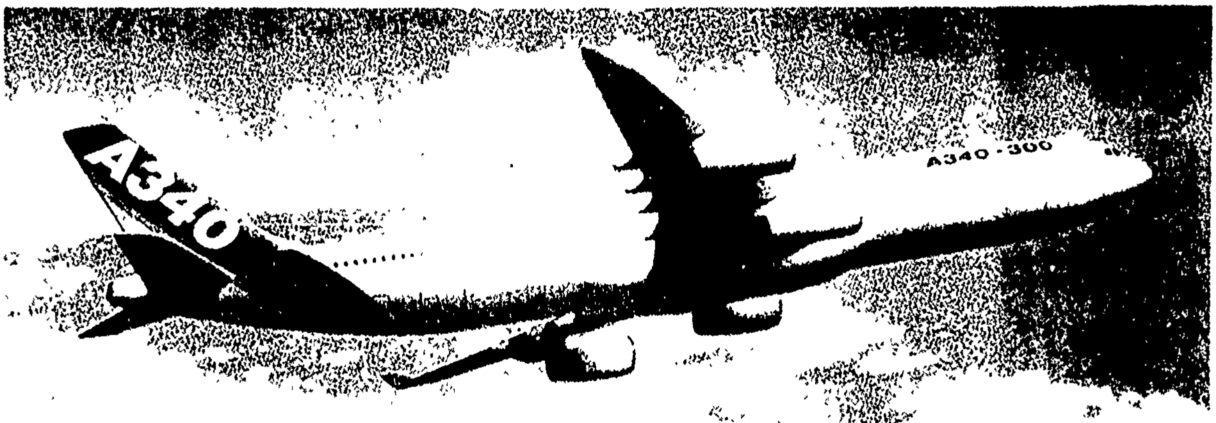
Air Transport World 5/90

Jet aircraft delivery forecast

Region	1989–1998	1999–2008	Total deliveries
≤130 seats			
Europe	273	81	354
North America	654	230	884
Asia/Rest of World	191	176	367
Sub-total	1,118	487	1,606
131–170 seats			
Europe	466	221	687
North America	622	427	1,049
Asia/Rest of World	353	271	624
Sub-total	1,441	919	2,360
171–230 seats			
Europe	292	227	519
North America	611	563	1,174
Asia/Rest of World	200	260	460
Sub-total	1,103	1,060	2,153
231–340 seats			
Europe	305	563	868
North America	453	647	1,100
Asia/Rest of World	473	676	1,149
Sub-total	1,231	1,886	3,117
341–430 seats			
Europe	132	125	257
North America	195	278	473
Asia/Rest of World	313	706	1,021
Sub-total	640	1,110	1,751
≥431 seats			
All regions	43	107	150
Total Europe	1,468	1,217	2,685
Total North America	2,535	2,145	4,680
Total Asia/Rest of World	1,573	2,198	3,771
Total	5,576	5,560	11,136
Deliveries: Leasing companies	710	360	1,070
Deliveries: All civil jet aircraft	6,286	5,920	12,206

Source: Airbus Industrie





The 4-engine A340 will take Airbus into the very long-range market for the first time. The company foresees higher-than-average growth in this segment.

ceptability of aircraft, young existing fleet.

- North America, below average traffic growth; creation of megacarriers; yield-management systems; noise limitations, high age of existing fleet; capacity demand dominated by replacement.
- Asia/Pacific, Middle East, above average traffic growth, creation of new routes, young existing fleet, limited operating infrastructure, capacity demand dominated by traffic growth.
- Rest of the world—High growth potential; financial constraints; air-transport growth constrained by the availability of trained personnel; no operational limitations.

Airbus based its forecast on a number of global and industry parameters. It assumed a real increase in gross domestic product (GDP) in the order of 2.9% annually, following a cyclical pattern, a continuing trend of real fare decreases sustained by liberalization, increased competition and cost-cutting measures within the airline industry.

The forecast said that world population will increase by 1% per year over the next 10 years and people of all ages will gain air-travel experience, increasing their willingness to continue flying. After having satisfied their needs for basic consumer products, people's preferences are turning more and more to leisure activities, many of which include air travel.

Corporate-travel reduction

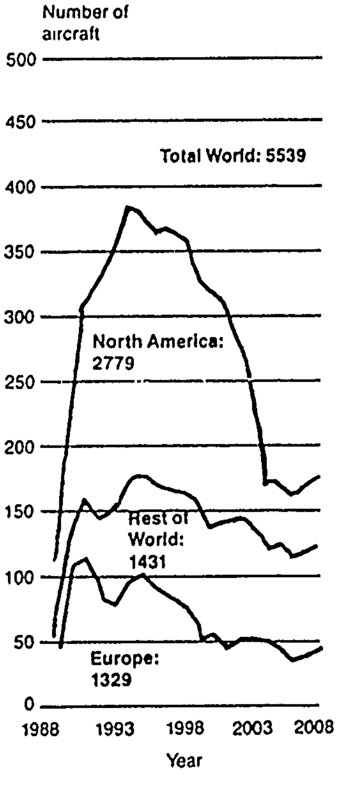
More shorter excursions will be made in addition to annual holidays. By the turn of the century, some 80% of air travelers will make their journeys for reasons not entirely related to business. New means of telecommunications could replace some intro-corporate travel, while on short-haul distances, high-speed trains will complement air transport in a few selected markets. This will allow airports to allocate slots for medium and long-haul air services.

The significance of the relationship between macroeconomic factors and the demand for air transport is undisputed, Airbus claimed. Economic organizations and forecasting institutes expect worldwide expansion of real GDP in the order of 2.5%-3% annually, with fewer cyclical variations than in the past.

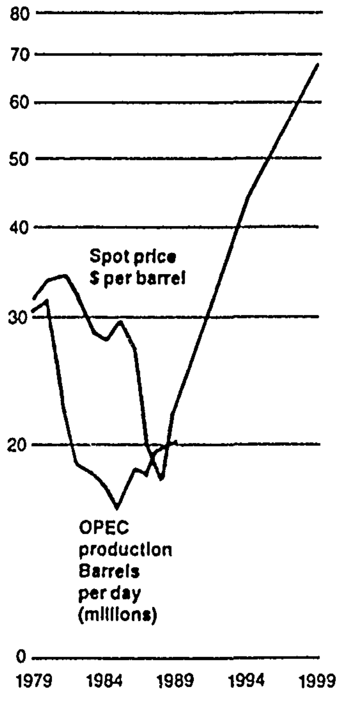
Positive developments include controlled inflation in industrialized countries, real interest rates remaining roughly at today's levels, less-regulated airline environments promoting productivity improvements and the recovery of the employment pattern.

Adverse factors are uncertainties and fluctuations in exchange rates not entirely related to economic developments, debt servicing in developing countries, energy prices increasing at a faster rate than world inflation, increasing collaboration and mergers between airlines supporting re

Jet aircraft retirements



Crude oil spot price and OPEC production



Sources: IMF, OECD, DRI

duced competition and fare increases and increasing government involvement in international-trade activities leading to more protectionism.

Between 70% and 80% of today's worldwide GDP is consumed. Diverging developments can be expected in the future in Asia, where in the case of the Four Tigers—Hong Kong, Korea, Singapore and Taiwan—consumption might increase at the expense of investment. Other Asian countries then might continue the drive toward industrialization. As a result, the share of the seven major industrialized countries of the world's GNP is expected to show a slightly downward tendency. By the mid-1990s, the traffic carried by all Asian airlines combined is expected to surpass that of Europe.

In North America, airlines no longer expect future traffic to grow as a function of GDP; it will be more sensitive to the evolution of fare levels, with the increasing application of yield-management systems permitting the airlines to set maximization of revenue as the prime industry target. Therefore, the quality of the passengers will be of greater importance to the airlines than the sheer quantity of passengers and goods to be transported.

In Europe, air-traffic liberalization will mean increased competition between the airlines, with fare reductions and a higher level of flight frequency attracting the traveling public. However, the possibility exists that from 1992 onward, intra-European air travel might be defined as domestic travel, becoming subject to value-added tax, with ticket prices going up correspondingly.

Should this occur, airports would see revenues from tax-free sales decreasing sharply. "Airport authorities may then look for other sources of revenue, including increased handling fees, which will be passed on to the passenger in the form of higher ticket prices. Monopolistic pricing of prime slots at hub or congested airports is likely to push landing fees up.

"It is possible, therefore, that potential cost and fare reductions may be compensated for by an increase in taxes and fees. In a no-fare-reduction case, traffic expansion would reduce to 4.3% from 4.9% otherwise."

Belly-cargo capacity rise

On air cargo, the Airbus forecast sees the types of passenger aircraft to be introduced during the 1990s leading to a cargo-capacity increase of some 7%. Along with a worldwide GNP forecast of 2.9%, future cargo traffic was expected to expand at a rate of around 7.9% a year, compared with a historical growth of around 10%-11% over the previous 19 years.

Turning to the operating environment, the forecast said that in the absence of any significant change in current administrative policies, the risk exists that future air traffic will encounter some sort of self-limitation that will reduce the forecast traffic growth by one percentage point a year.

By the year 2000, there will be 24 frequency-limited airports in Europe, 59 in North America and 12–15 in Asia. These airports handle some 55% in all passenger movements. The 24 airports in Europe have runway capacity of some 4.6 million movements a year. But in 1989, they handled almost 4.2 million movements, leaving only marginal opportunities for future growth. However, a 20% improvement in capacity by the turn of the century, which could be achieved principally by a better organization of resources, could increase frequency by 2.5% annually.

Passenger traffic development

ICAO world and regions (billions RPK)
Scheduled and non-scheduled operations

	1978	1988	1998	2008
Europe (excl. USSR)	262	419	683	1,031
North America	425	753	1,158	1,709
Latin America	49	82	153	258
Africa	28	43	69	110
Middle East	28	48	82	136
Asia/Pacific	130	315	712	1,367
Total world	932	1,660	2,858	4,611

Airbus expects to collect 45% of the world's widebody-aircraft sales over the next 20 years, with the A330 as one of its prime contenders.

Long-term market predictions

20-year global averages

	Europe	North America	Asia/Pacific	World average
Passenger traffic growth (base case)	4.8%	4.1%	7.4%	5.3%
Aircraft productivity growth (speed, utilization, trip distance, load factor)	0.4%	0.5%	1.4%	0.7%
Required capacity growth	4.4%	3.6%	6.0%	4.6%
Provided by				
— frequency growth	2.8%	2.1%	4.2%	2.9%
— aircraft size growth	1.6%	1.5%	1.8%	1.7%

In North America, airport limitations have reached "critical dimensions." The only new airport being developed is at Denver; the capacity of some others cannot be expanded because of geographical limitations. In total, the U.S. airport system is expanding its capacity, which will enable it to handle a growing passenger volume of 3.5%-4% through to the turn of the century. "Operating limitations as a result of locally imposed noise rules and curfews are developing into a major threat to the long-term expansion of aircraft movements in North America," Airbus stated. Larger aircraft are the only viable short-term solution to congestion problems.

Re-emergence of rail technology is leading to improved train services that, in many cases, should be considered as a complement to air travel. If the total air-trip time for a given sector was equivalent to the total train-trip time, giving no advantage in using the aircraft, the expected diversion of potential air-traffic demand to surface transport could be on the order of 50%.

The future—to 2008—need for new-aircraft capacity depends on traffic-growth expectations and the replacement of around 5,500 aircraft currently in service. Some 2,700 of these aircraft are in North America, 1,300 in Europe and 1,400 in the rest of the world. At the end of 1989, 85% of the world's passenger fleet was being operated by the 100 largest airlines, 25 of which possessed 50% of the world's fleet. "As potential launch airlines," Airbus commented, "these carriers play a decisive part in the definition of new and derivative versions of current aircraft designs."

Leasing had become a "major phenommenon" in the aircraft business. Although the leasing companies have been in business since the mid-1970, they did not start to buy new aircraft until the early 1980s. The major companies now are developing into a third force in the aircraft business, along with the manufacturers and the airlines. In the course of the past few years, more than 20% of all new aircraft had been ordered by leasing companies.

Lessors boost flexibility

The presence of the operating-lease companies provided increased flexibility to the airlines and enhanced business opportunities and long-term production stability for the aircraft and engine makers. In view of the relative youth of this new force in the industry, these benefits were accompanied by increased levels of uncertainty.

Airbus figures show that while 6.6% of 400 orders for new jet airliners came from lessors in 1984, the figures had changed to 27% of 600 by 1986, to 22.3% of 1,100 by 1988 and to 16.7% of 1,700 last year.

Total deliveries forecast by Airbus over the next 20 years were: In the 130-seat category, 1,118 aircraft in 1989–98, 487 in 1999-2008, making 1,605 in all; 131–170 seats, 1,441 and 919, making 2,360; 171–230 seats, 1,103 and 1,050, making 2,153; 231–340 seats, 1,231 and 1,886, making 3,117; 341–430 seats, 640 and 1,110, making 1,751; 431 seats-plus, 43 and 107, making 150. Grand total, 11,136.

At the end of February, Airbus had 1,375 firm orders from 89 customers for its family of airliners, with 575 delivered and a backlog of 300. The orders break down as 619 A300/A310s from 72 customers, 562 A320/A321 from 27 customers and 194 A330/A340s from 21 customers. David Jennings, VP-marketing, said here that the European consortium believes that it will take 20% of all narrowbody sales and 45% of all widebody sales.

The consortium is evaluating "X" versions of its A330/A340 designs, according to senior execs who are visiting here. All options are open for both the twin-engine A330 design and the 4-engine A340 design, with changes in payload/range and in capacity being considered as part of an overall philosophy that Jennings said could lead eventually to a family of derivatives.

"We have already exposed these ideas to a number of airlines around the world," he added. "These are proposals; none of them is certain at the moment."

APPENDIX G

TYPICAL AVIATION FIRE EXTINGUISHER CONFIGURATIONS
and
MARINE FIRE EXTINGUISHER

APPENDIX B FIGURES

APPENDIX G

TYPICAL AVIATION FIRE EXTINGUISHER CONFIGURATIONS

and

MARINE FIRE EXTINGUISHER CYLINDER

Charge recovery from fire extinguisher bottles containing Halon 1301, nitrogen and/or helium is performed by connecting to an appropriate outlet on the bottle. In many cases this outlet is the fill or charge fitting through which the bottle was last charged. In some cases, such as ball valve operated Marine or Ordnance Vehicle bottles, agent recovery is possible through the main outlet valve by incorporating the charging adapter through which the bottle was initially charged.

Aviation fire extinguisher bottle designs are basically of four types. Those are hermetic, non-hermetic, inerting and fusible plug or cap.

Hermetic designs are, as the name implies, of all welded construction. These fire extinguishers typically contain Halon 1301, nitrogen super pressure and 4 to 6 psi helium partial pressure for leak detection purposes. Total charge pressure at 70 degrees Fahrenheit can be 825 psig or higher. Access to bottle charge within these designs is usually the fill fitting. Typical design of fill plug and fitting geometries provide an inner cylindrical skirt on the plug which rotates inside the bottles fill fitting inside diameter. Upon completion of bottle charging these two concentric cylinders are fused together at their outside circular edges, by TIG weld, forming a hermetic seal. Access to bottle charge after welding is accomplished by using a tool to remove fusion weld material which formed the hermetic seal between bottle fitting and fill plug. Upon weld removal fill plug is grasped with an appropriate wrench to verify rotational freedom. Upon confirming removal of fusion material an appropriate fill fitting adapter (as seen in Figure G-3) is placed over fill fitting and locked in place. Sealing between bottle fill fitting and adapter is accomplished by O-ring seal. A hose is connected to adapter output fitting and fill plug rotated, using the stem on the adapter, which opens fill plug from fill fitting releasing bottle charge for recovery. The bottle is then inverted to recover liquid first. Two hermetic fire bottle designs are shown in Figures G-1 and G-2. The fill fitting with welded fill plug can be seen (Figure G-1 or G-2) as a small cylindrical protrusion about 1 inch in length terminating into either a square or parallel, flat edge geometry, as on the dual outlet cargo container. Thermal (over temperature) relief of bottles using this type fill fitting design is through the main outlet disc.

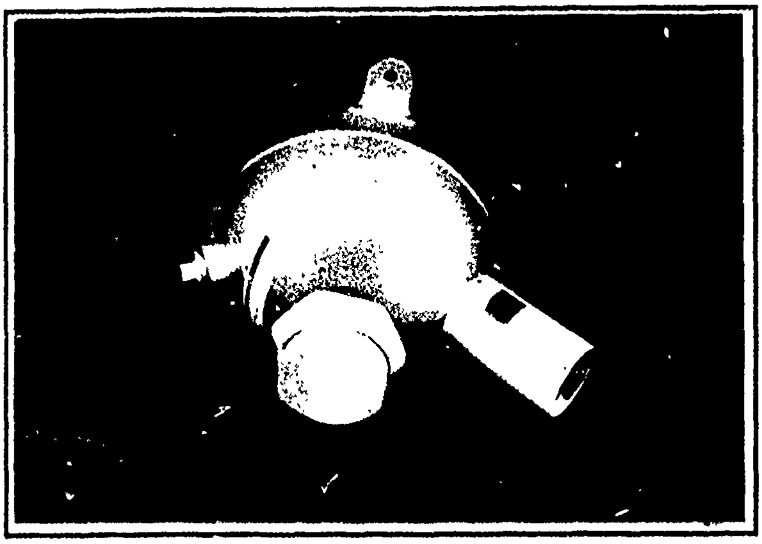


FIGURE G-1

86 Cubic Inch Hermetic Aviation Firebottle
Pressure Gauge, Fill Fitting and Single Outlet

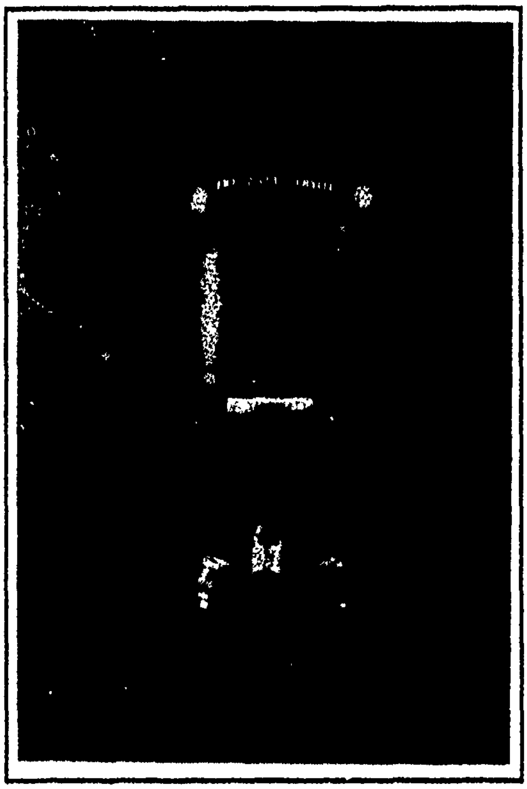


FIGURE G-2

2500 Cubic Inch Hermetic Aviation Firebottle
Pressure Gauge (near center bottom), Fill Fitting (near top right)
and Dual Outlet Configuration

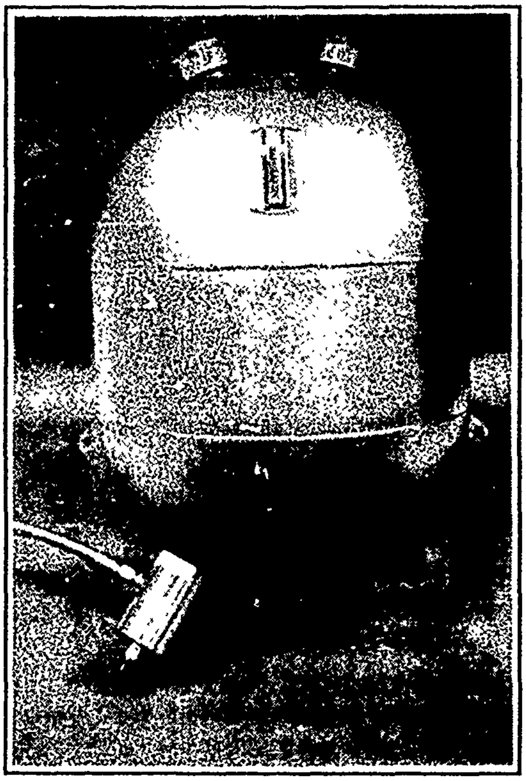


FIGURE G-3

2170 Cubic Inch Hermetic Aviation Firebottle Charging Adapter Attached To Fill Fitting
Illustrates Inverted Bottle And Stem For Opening Fill Fitting

The typical adapter is shown in Figure G-3 attached to a hermetic bottle similar to the bottle shown in Figure G-2. There are approximately seven different configurations used for fill fittings on hermetic bottle designs. Each configuration is specific for customer or aircraft manufacturer. Recovery from all types of hermetic aviation fire extinguisher designs requires that adapters be readily available from each manufacturer for use on their particular bottles fill fitting.

FIGURE G-4

378 Cubic Inch Hermetic Aviation Firebottle Pressure Gauge (right center), Fill Fitting (top of bottle) and Dual Outlet Configuration

Figure G-4 illustrates a hermetic design whose fill and safety relief fitting serves a second purpose. That purpose is one of thermal relief, hence the nomenclature fill and safety. The fill fitting can be seen as the smaller cylindrical protrusion near top center. This particular fill fitting contains at its end an anti-recoil cap to vent contents without generating thrust. Another type adapter is used for recovery of agent from this fire extinguisher; however, the method is the same in each case. The internal mating geometry of the adapter merely changes to meet a particular fill fitting design.

Figures G-5, G-6, and G-7 that follow, illustrate three aviation container configurations of a non-hermetic design. In each design charging and recovery of charge is performed by way of a standard one quarter inch male flare connection located under the removable anti-recoil cap. The anti-recoil cap is the small cylindrical protrusion about one half inch in diameter and one inch in length. Just under the cap a nine sixteenths hex will be seen which is the fill and safety relief fitting. By attaching a standard one quarter inch high pressure hose with quarter inch female AN flare nut to the fill and safety fitting and loosening fitting using a nine sixteenths open end wrench bottle charge is recovered.

Figure G-8 illustrates fill and safety fitting and anti-recoil cap used on most non-hermetic designs. This type fill fitting contains a small burst disc design geometry at the seat end. Disc burst pressure is selected by several factors and can be designed for practically any pressure range from 1200 psig to 2700 psig or higher. No adapter is required for servicing a fire bottle of this design. The 55 cubic inch Nitronic 40 container pictured with the fill and safety was used during the feasibility study for sample collection and transportation to DuPont for analysis.

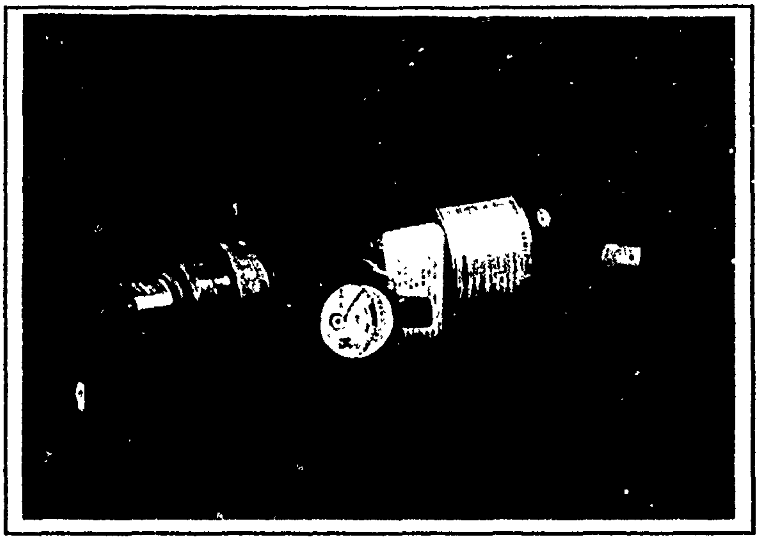


FIGURE G-5

65 Cubic Inch Non-Hermetic Aviation Firebottle,Pressure Gauge (front left end), Fill & Safety Fitting (far right end) and Single Outlet Discharge Head (far left)

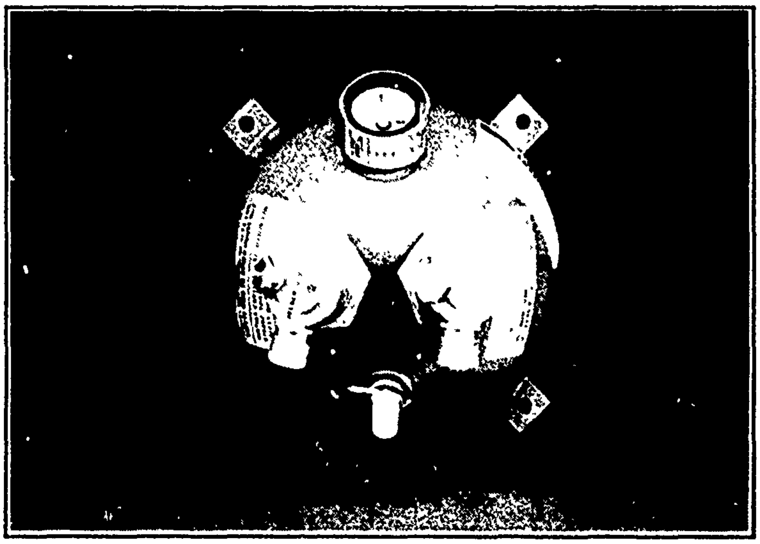


FIGURE G-6

378 Cubic Inch Non-Hermetic Aviation Firebottle, Pressure Gauge (top), Fill & Safety Fitting (bottom) and Dual Outlet Discharge Heads (center)

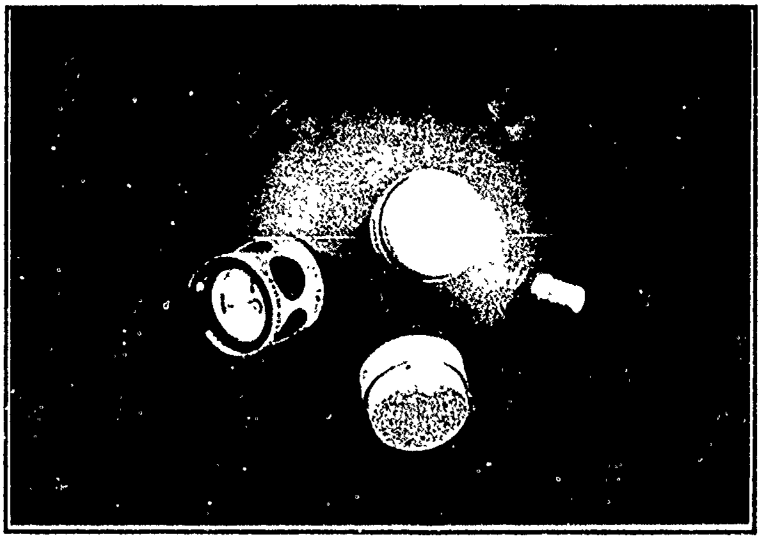


FIGURE G-7

224 Cubic Inch Non-Hermetic Aviation Firebottle, Pressure Gauge(left), Fill & Safety Fitting (right) and Safety Capped Dual Outlet Necks (center)

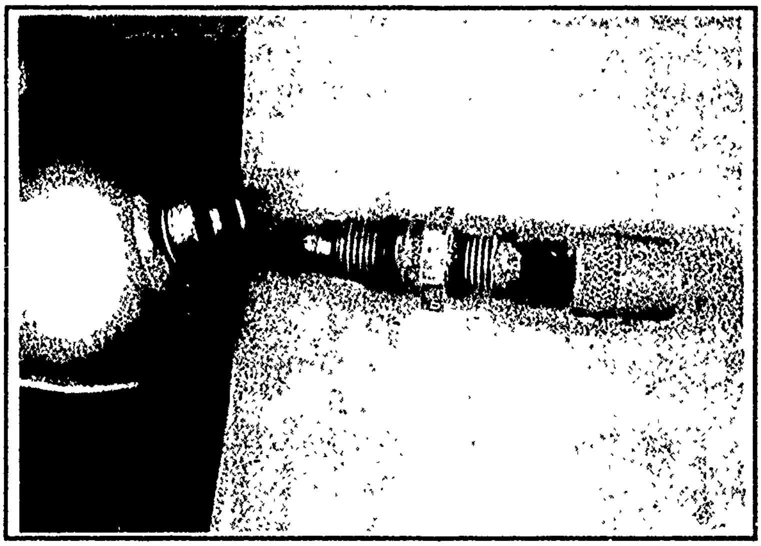


FIGURE G-8

Fill and Safety Fitting With Anti-Recoil Cap

Some aircraft use inerting bottles which contain Halon 1301 only. Typical inerting bottle design incorporates a heater which is energized during flight. Thermal output increases Halon 1301 vapor pressure causing a spring loaded relief valve to open. As long as liquid agent remains, Halon vapor will be bled from the bottle into the compartment being protected. Heater cutoff removes thermal input from Halon 1301 resulting in reduced Halon 1301 vapor pressure. Vapor pressure reduction allows relief valve to reseat conserving agent until needed again. One type of inerting bottle design used on the F-16 is shown in Figure G-9.

This particular design is normally filled immediately prior to flight. During manufacture it is necessary to charge bottle with Halon 1301 to verify proper operation. This Halon 1301 is now being recovered at greater than 96% efficiency. Recovery from this type bottle is more difficult than from a Nitrogen pressurize bottle due to the bottles design prohibiting access to liquid phase agent. All agent must be removed as vapor phase.

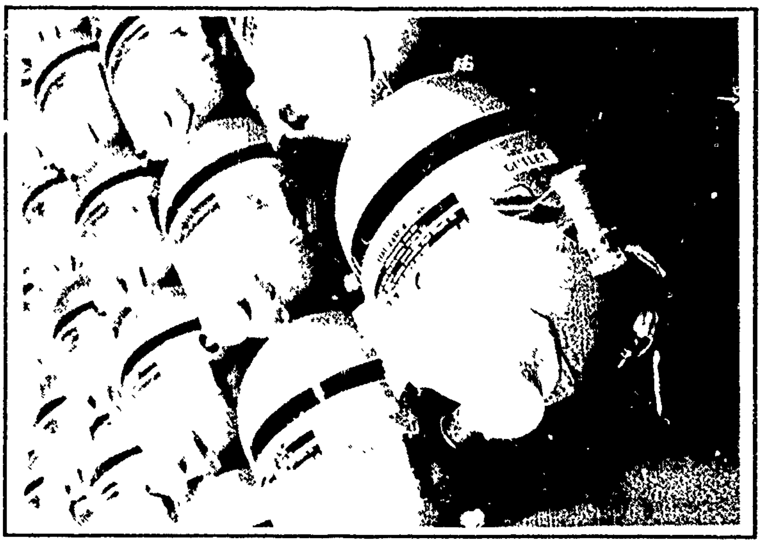


FIGURE G-9

315 Cubic Inch Halon 1301 Inerting Bottle Heater And Relief Valve Assembly

Figure G-10 is a lavatory fire extinguisher, which is disposable following discharge, for waste bin or similar applications. This extinguisher design utilizes a fusible cap design retained to the directional tubes by eutectic solder. In the presence of heat at the tube end, of approximately 172 degrees Fahrenheit, as would be in the case of a wastepaper bin fire, pressure due to Halon 1301 vapor pressure blows of the end cap allowing escaping agent to extinguish fire. These designs are completely sealed by solder at plug ends and inert atmosphere brazed otherwise. Charge is presently verified by weight check. Should weight check indicate charge loss below minimum acceptable level, contents (approximately 100 grams Halon 1301) are discharged and extinguisher is disposed of. Hydrotest requirements of FAA Order 8000.40B do not apply to disposable, low pressure fire extinguisher containers such as the "potty bottle". Discussion of this topic can be found in Section 4.1. Agent recovery from this eutectic solder design is discussed on page 25, Section 4.1.

FIGURE G-10

Typical Disposable Lavatory Fire Bottle

Figure G-11 illustrates a typical Non-aviation (Marine) cylindrical fire bottle. These fire bottles are typically 35 pound or 55 pound cylinders containing 35 or 55 pound Halon 1301 charge pressurized to 360 psig nominal charge at 70 degrees Fahrenheit using dry helium. Charging and charge recovery from these cylinders is through the main valve. Recovery is performed using the same adapter as that used for charging.

Ordnance Halon cylinders are similar except for main outlet valve design. Ordnance cylinders are typically pressurized to 650 psig or 750 psig at 70 degrees Fahrenheit. Recovery from Ordnance fire extinguishers is also performed through main valve also using the charging adapter.

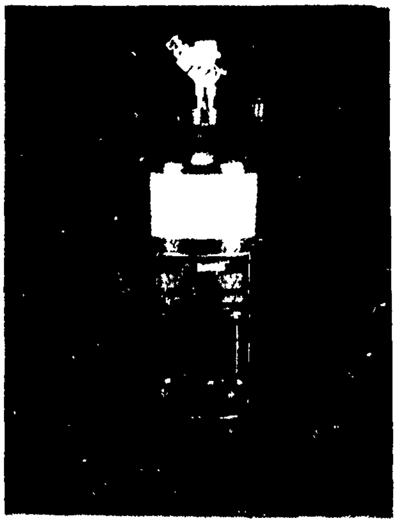


FIGURE G-11

Typical Non-Aviation Fire Extinguisher Configuration
Used For Marine And Ordnance Applications

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