D-ZERO
COLD VALVE

D-Zero Engineering Note: 3740.510-EN-139

C.H. Kurita

January 20,1988

Approved: G. T. Mulholland

Abstract

The cold valve used for the D-Zero calorimeters was originally designed to be a dual-purpose valve, serving as a fill/drain valve at the bottom of each vessel and, with a pilot valve attached, as a relief valve at the top of each vessel. However, because of a conflict which arose on the matter of using a non-coded relief valve on an ASME coded vessel, the use of the cold valve has been limited to serving as only a fill/drain valve for the argon.

Valve Design

While the original design of the cold valve has been modified several times, the internal structure has basically remained the same. Most changes have occurred in the design of the valve body and the plug. Two bellows assemblies and various guide cylinders allow for the motion of the piston and plug which creates a sealing surface with the valve seat. Actuation of the valve is accomplished by applying helium pressure to one of the two ports, dependent on whether the valve is to be in the "open" or "close" position. See Fig. 1 for the valve design.

The desired leak rate of the valve is $1x10^{-7}$ std atm cc/sec. In order to achieve this low leak rate at liquid argon temperature (87K), various design modifications were made to the Kel-F plug until cold testing of the valve in liquid nitrogen showed a sufficiently low leak rate. Another major modification made was the replacement of large body flanges, with a piston guide machined in one, with an all-welded construction and a separate lantern ring to guide the piston. The internal structure of the valve has remained basically the same despite the aforementioned modifications. Fig. 2 shows the design of the original prototype.

For current design details, see the "Cold Valve Drawings" listed in Table 1.

Completion of the fabrication and testing of one valve has prompted suggestions to facilitate these processes in the future. One suggestion made was to ensure that seamless tube was used for the piston guide cylinder to avoid any "hang-ups" with the brass guide rings. Due to the tight fit between the seal ring, piston rod and the inside diameter of the bellows, piston guide, a modification of the seal ring and welding assembly was proposed. See Fig. 3 for details. While both the warm and cold testing of the valve demonstrated satisfactory leak rates, close examination of the plug revealed a permanent deformation of the lower lip, and the appearance of a white line which indicates that the material had yielded at some point in the testing prodcedure. An investigation is currently being conducted to get a better understanding of what happened to the Kel-F plug. When complete, the results will be appended to this engineering note.

Cold Valve Test

After fabrication of the valve was completed, the valve was cycled warm approximately seventy-five times to check the "precision running fit" of the brass rings in the piston guide cylinder. The valve was then taken to C-Zero where it was both warm and cold tested by E. Ramirez and C. Pallaver.

The leak rate of the valve was measured using the flange assembly shown in Fig. 4, with glass teflon seals on the vacuum side. With a vacuum applied, helium was sprayed on this valve/flange interface and no leak of the glass teflon seals was detected. The valve was leak checked warm using the set-up in Fig. 5 (without the LN_2). The leak detector sensitivity was 3.5×10^{-10}. By applying a "close" port pressure of 28 psig to achieve a vacuum reading of 18 microns, the leak rate was measured to be 2.45×10^{-7} std atm cc/sec. However, when helium was sprayed on the plug/seat interface through the exhaust port of the valve, a leak was detected. The "close" port pressure was then increased to 40 psig, bringing the vacuum reading down to 5 microns. The leak rate was then measured to be 1.4×10^{-7} std atm cc/sec, and when sprayed with helium, no leak was detected. See Fig. 6.

The valve was then slowly cooled down by spraying nitrogen on it, and no change was observed. The valve was taken off of the leak detector and left only on the roughing pump with a vacuum reading of 20 microns. LN2 was sprayed on the valve until most of the valve was immersed in LN2. While cold, the data found on the following page was taken.

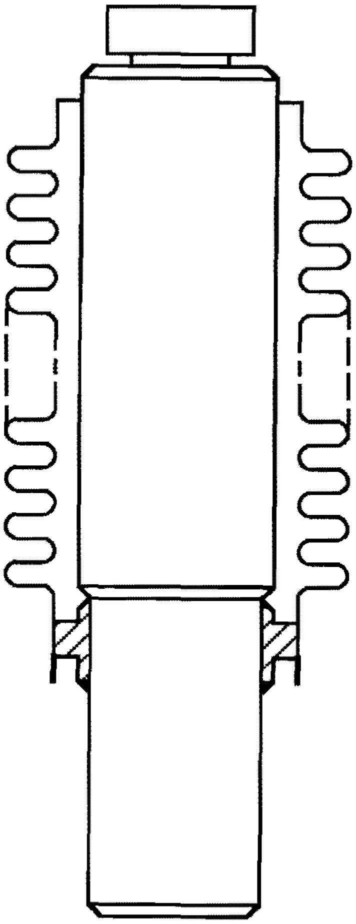


FIG. 3 SUGGESTED MODIFICATIONS

CONDITION	"CLOSE" PORT PRESSURE(PSIG)	VACUUM (MICRONS)	LEAK DETECTOR (STD ATM CC/SEC)	HE LEAK
rough pump only	48	18		
rough open/throttle partially open	50		6.3×10^{-8}	no
no rough pump/full leak detector	50		2.3×10^{-8}	no
open/reset	80	500		
open/reset	86	40		
open/reset	100	18		
rough open/throttle partially open	100	14	2.38×10^{-7}	no

**The valve was then cycled 5 times, pumping down to 100 microns each time.

rough open/throttle partially open	100	25	2.45×10^{-7}	no

The measured leak rates were determined to be sufficient for the application involved, although they fell just short of the original desired leak rate. This original leak rate had been set with the intention of using the valve as a relief valve, and were therefore more rigid than those necessary for a fill/drain valve.

See Fig. 7.

Actuation

The valve is actuated by using helium pressure. Applying 100 psig to the "close" port of the valve and none to the "open" port moves the piston plug assembly into the position where the Kel-F seal is compressed against the seat of the valve. Reversing the pressure by applying 100 psig to the "open" port and none to the "close" port forces the piston plug assembly to move away from the valve seat allowing the fluid to pass through the valve and out the exhaust. The schematic for the actuation set-up is shown in Fig. 8. In the event that the primary helium source should be depleted, a pressure switch senses the pressure drop and in turn activates a secondary source set at a lower pressure.

Failure modes

Two possible ways in which the cold valve could fail might be improper functioning of the bellows or inadequate sealing at the plug/seat interface. With respect to the potential bellows problem, the inclusion of guide cylinders around the bellows should prevent the allowance for any squirm due to overpressure. The possibility of improper functioning because of the build up of dirt which may have passed through the valve in the bellows is very small because of the limited access of argon to the bellows.

If the valve were to leak at all, the fluid would pass into the argon fill/drain line which is valved off at the other end, near the dewar. Leakage into this closed line would not cause any damage and the possibility of some liquid boiling and causing a rise in the line pressure would activate the reliefs installed on the line.

Installation

The cold valve is to be installed through nozzle N3V and welded into N3P of the CC cryostat. See Fig. 9 and DWG. 3740.214-MD-255078 for installation details. Precautions shall be taken to ensure that the valve exhaust is properly lined up with the exhaust pipe on the vessel before installation. The current drawing shows two welds which hold the valve in place. The possible addition of a drain guard ring at the top weld to help prevent residue from passing through the valve when the vessel is drained would modify this drawing. Details of such a ring have not yet been determined. The argon spill trough which surrounds the bottom of the vessel shall be designed to allow accessibility to N3V. Therefore, if the valve plug or any other part of the internal structure needs to be repaired or replaced, the necessary steps involved include penetration of the argon spill trough, removal of the flange on N3V, and the grinding of the weld which holds the top plate and outer cylinder of the valve together. The entire internal structure of the valve is attached only to this top plate, and hence, removal of this single plate constitutes removal of the entire valve (except the outer cylinder and bottom plate).

After being installed in the vessel, it is necessary that the valve be leak checked again (warm only) to ensure proper functioning. While the set-up for such a test has not yet been determined, the main component would be the design of a cover that would fit over the valve opening inside of the vessel. The seal between this cover and the valve must be vacuum tight in order that a vacuum could be drawn on the plug/seat interface to measure the leak rate of the valve.

When testing of the valve is complete, the valve must be protected from dirt that may fall into it during the period in which the rest of the calorimeter components are assembled. This protective cover should be easy to remove after the modules are in place.

Any additional comments on the installation procedure shall be appended to this report when they have been finalized.

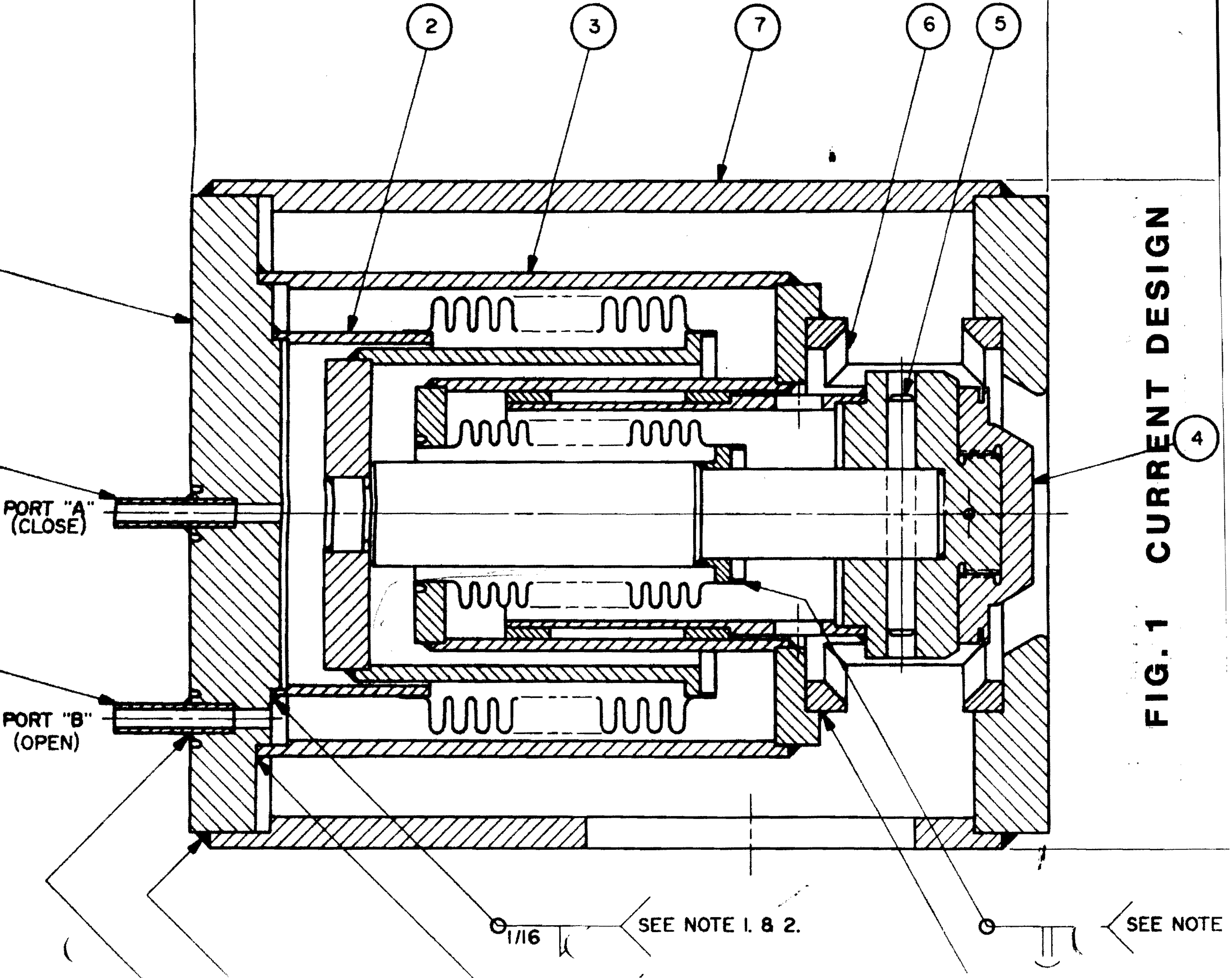


FIG. 1 CURRENT DESIGN

ORIGINAL PROTOTYPE

REV.	DESCRIPTION	DRAWN / APPD.	DATE / DATE
A	ADDED VCR TYPE FTG'S. ADDED NOTES 1 & 2	TP	12/4/85

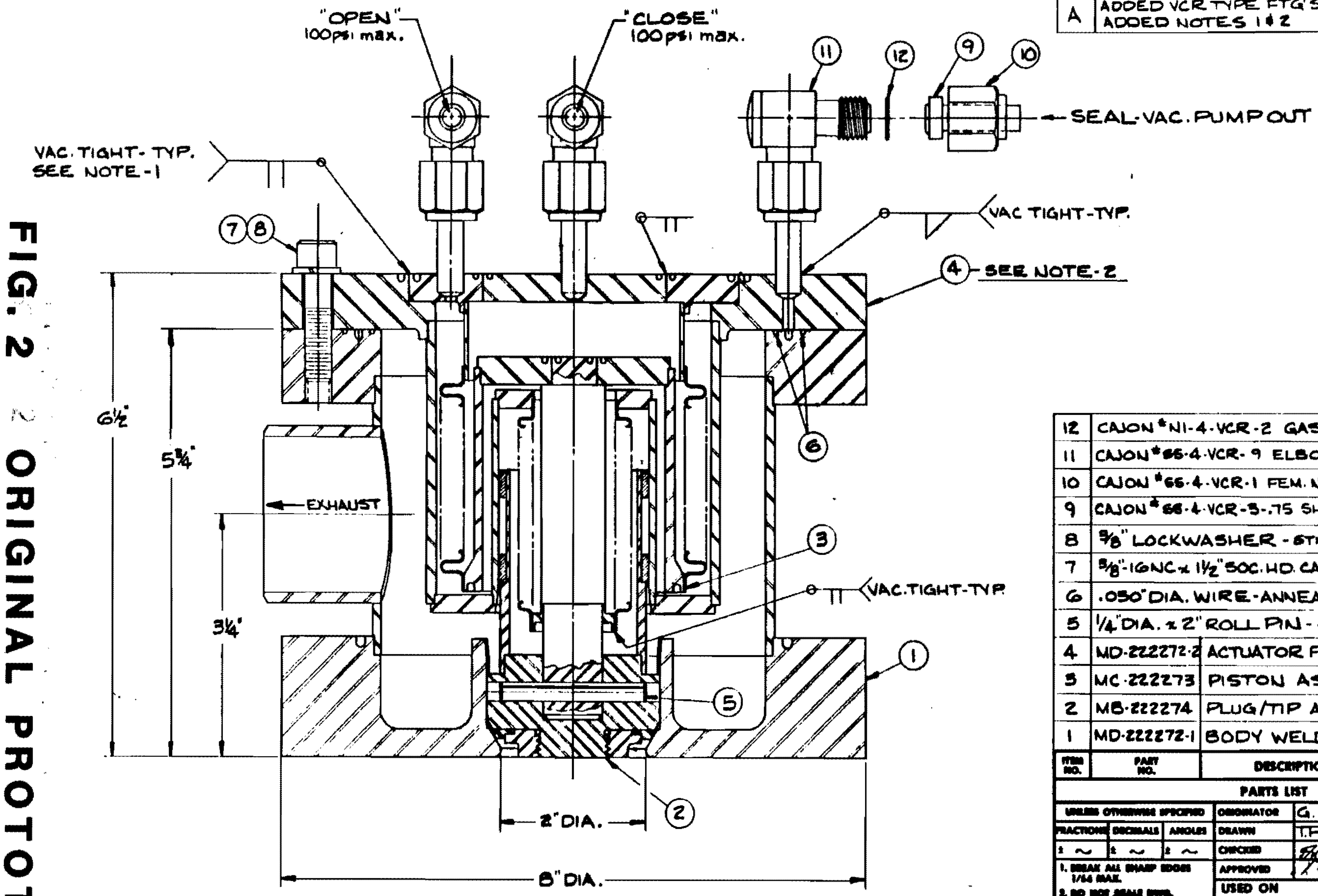


FIG. 2 ORIGINAL PROTOTYPE

NOTES:

1) BEFORE WELDING, ALIGN FITTING PORTS AS SHOWN.

2) EXHAUST ARROW ON ITEM 4 TO BE INSTALLED IN LINE WITH EXHAUST PIPE.

ITEM NO.	PART NO.	DESCRIPTION OR SIZE	QTY. REQ.
12	CAJON # NI-4-VCR-2 GASKET		6
11	CAJON # SS-4-VCR-9 ELBOW		3
10	CAJON # SS-4-VCR-1 FEM. NUT (¼" TUBE)		6
9	CAJON # SS-4-VCR-3-.75 SHORT GLAND		6
8	⅜" LOCKWASHER - STNL'S.		16
7	⅜"-16NC x 1½" SOC. HD. CAP SCR. - STNL'S		16
6	.050" DIA. WIRE-ANNEALED COPPER		A/R
5	¼" DIA. x 2" ROLL PIN - STNL'S.		1
4	MD-222272-2	ACTUATOR FLG. ASS'Y.	1
3	MC-222273	PISTON ASS'Y.	1
2	MB-222274	PLUG/TIP ASS'Y.	1
1	MD-222272-1	BODY WELDMENT	1

PARTS LIST

UNLESS OTHERWISE SPECIFIED					
FRACTIONS	DECIMALS	ANGLES	ORIGINATOR	G. MULHOLLAND	
± ~	± ~	± ~	DRAWN	T. PARKER	11-15-85
			CHECKED		11-22-85
			APPROVED		12-4-85

1. BREAK ALL SHARP EDGES 1/64 MAX.
2. DO NOT SCALE DWG.
3. DIMENSIONING IN ACCORD WITH ANSI Y14.5 STD'S.
✓ MAX. ALL MACHINED SURFACES

USED ON D0

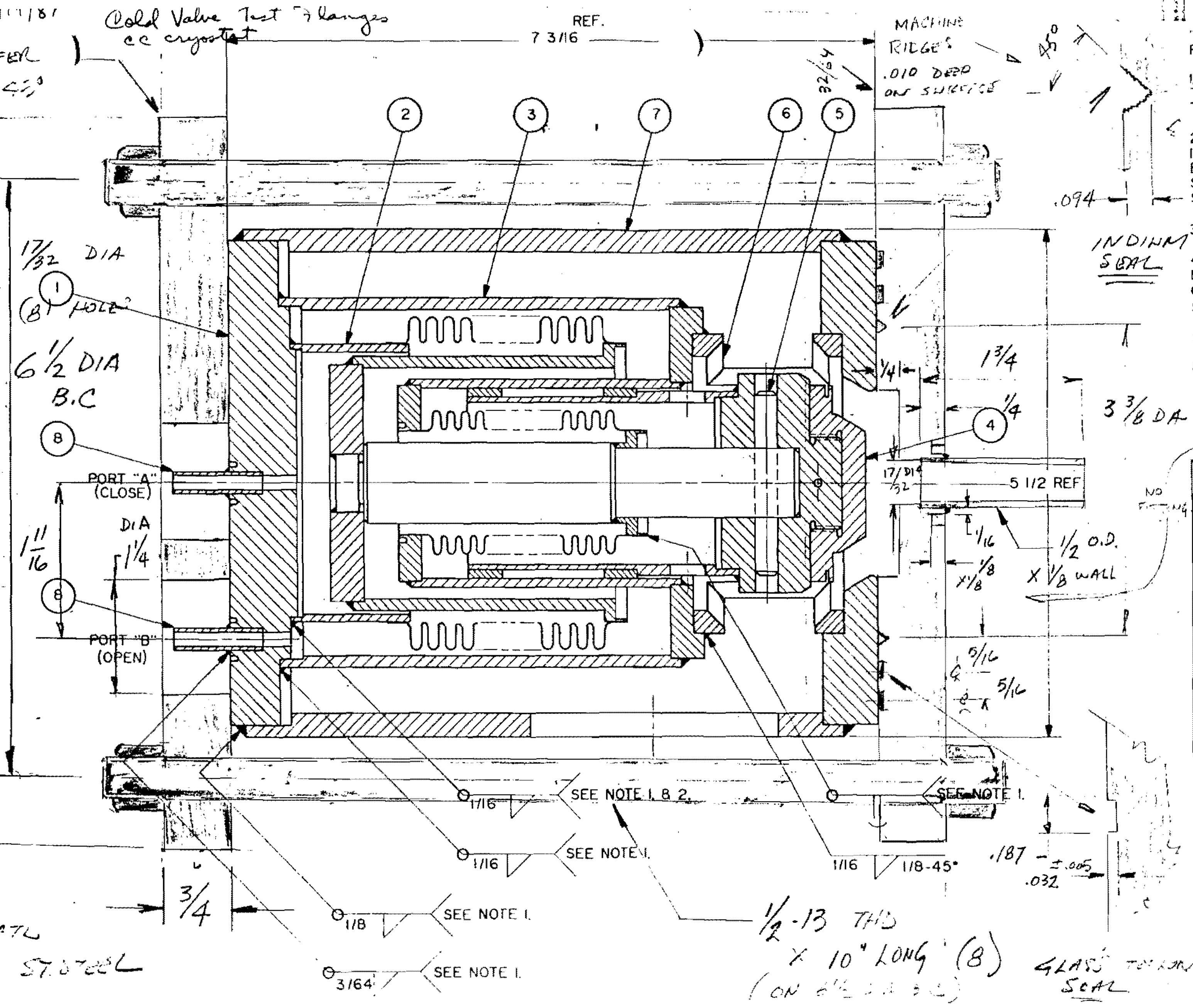
MATERIAL: AS NOTED

FERMI NATIONAL ACCELERATOR LABORATORY
UNITED STATES DEPARTMENT OF ENERGY

DØ
ARGON COLD VALVE
ASSEMBLY

SCALE	FILMED	DRAWING NUMBER	REV.
1" = 1"		3740.510-MC-222271	A

EMR 11/?/87 Cold Valve Test Flanges cc cryostat



8" ROUND OR SQUARE

NOTE ALL MATL 304 STEEL

FIG. 4 TEST FLANGE ASSEMBLY

"VACUUM TE
FOR COLD V.
} ASSEM
ASSY.

1. WELD ITEM 2 (PISTON ACT
TO ITEM 1 (TOP PLATE, VALV
THRU PORT "A".

2. WELD ITEM 3 (PISTON GUI
ITEM 1 (TOP PLATE, VALVE B
BELLOWS (ON PISTON GUIDE
SEAL RING, PISTON ROD (ON
SUB-ASSY.). PUMP THRU POF

3. ASSEMBLE ITEMS 4, 5 &

4. WELD ITEM 7 (VALVE BOD
ITEM 1 (TOP PLATE, VALVE B
ON VALVE BODY BOTTOM PL
SIDE PORT ON VALVE BODY

5. WELD ITEM 8 (1/4 O.D. ×
TUBING) INTO PORTS "A" &
PLATE, VALVE BODY). 2"

8	1/2 DIA TUBE	TU
7	MC-224660	VA
6	MC-224659	LA
5	———	SP
4	MC-224658	PI
3	MC-224657	PI
2	MC-224656	PI
1	MC-224655	T
ITEM NO.	PART NO.	

UNLESS OTHERWISE SPECIFIED

FRACTIONS	DECIMALS	ANGLES
± 1/64	± .005	± 1°

1. BREAK ALL SHARP EDGES 1/64 MAX.
2. DO NOT SCALE DWG.
3. DIMENSIONING IN ACCORD WITH ANSI Y14.5 STD's.

√ MAX. ALL MACHINED SURFACES

COLD VALVE TEST SET-UP

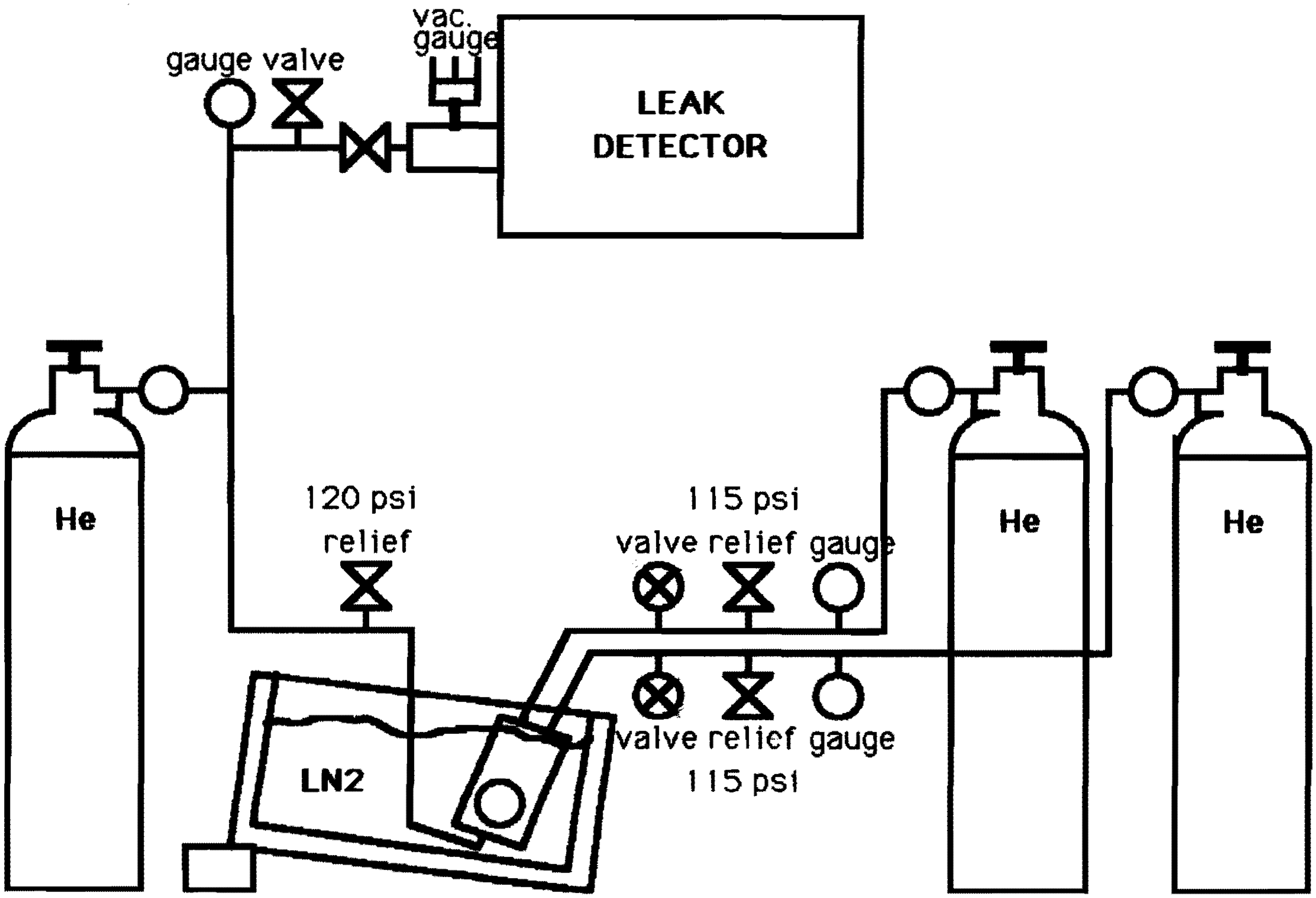
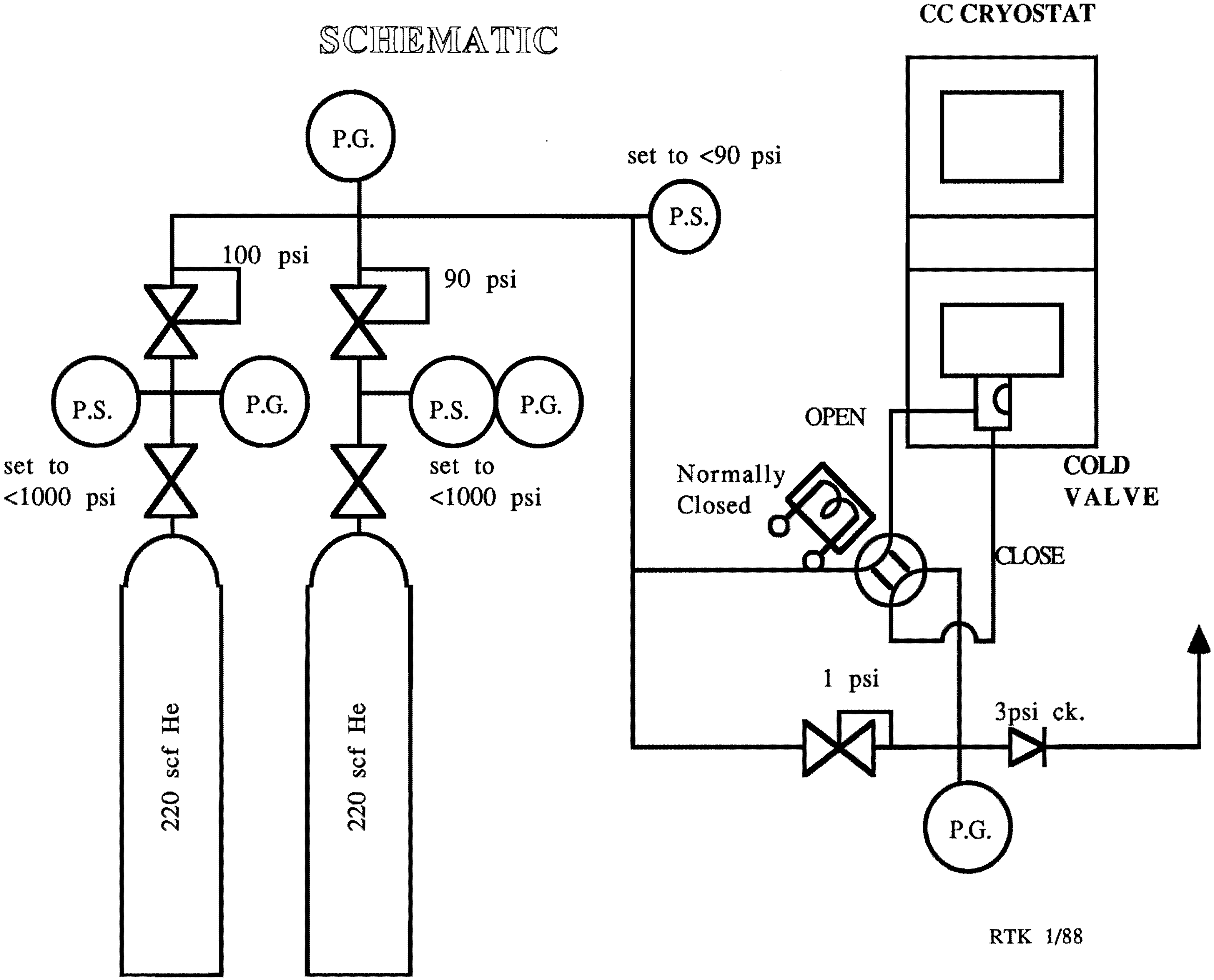


FIG. 5 TEST SET-UP

COLD VALVE ACTUATION SCHEMATIC



RTK 1/88

FIG. 8 COLD VALVE ACTUATION

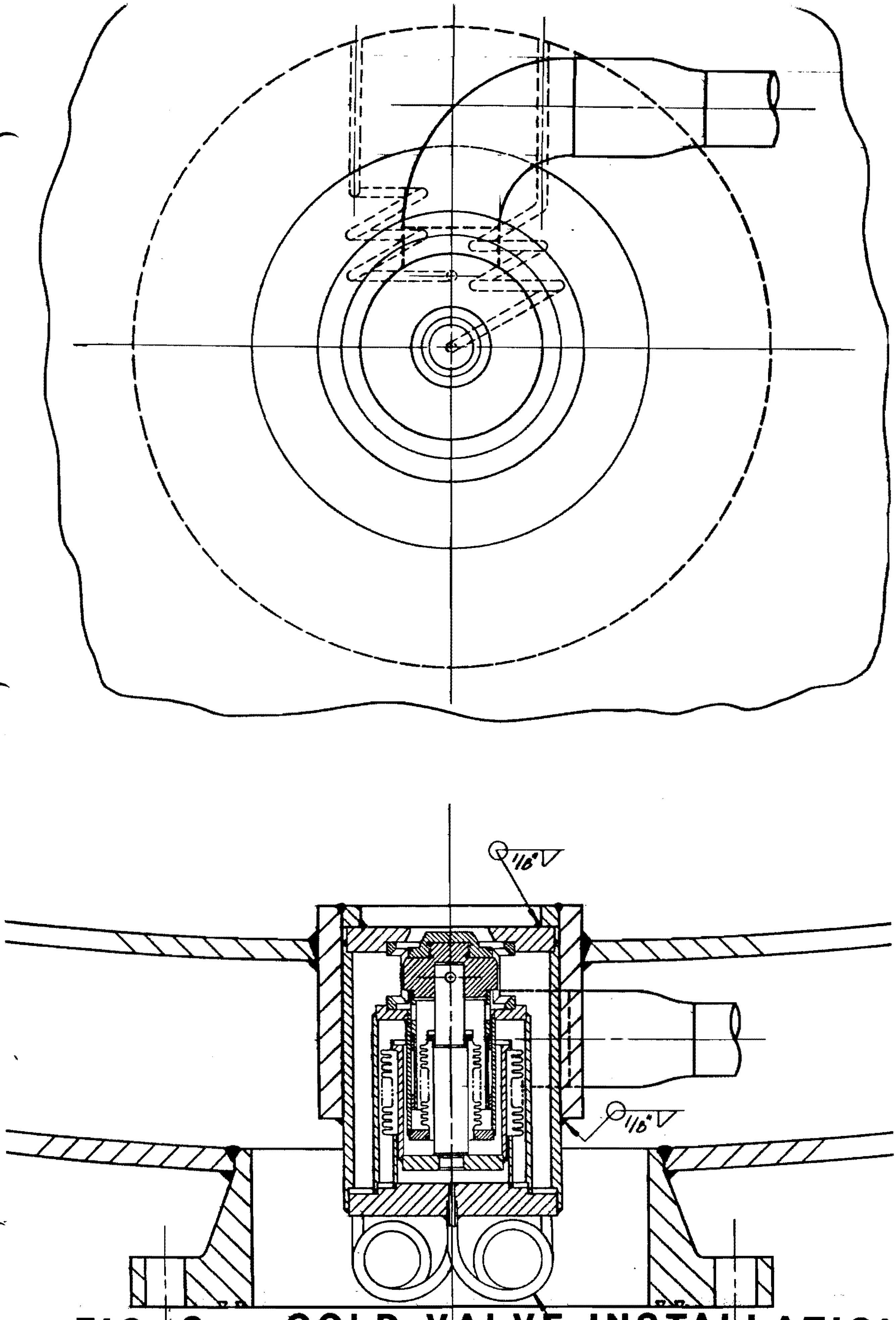


FIG. 9 COLD VALVE INSTALLATION

"COLD VALVE DRAWINGS"

DWG. NUMBER	TITLE
3740.510-MB-222285	D0 Detector 1 1/2" O.D. "Pathway" Bellows Argon Relief Cold Valve
3740.510-MB-222286	D0 Detector 3 5/8" O.D. "Flexonics" Bellows Argon Relief Cold Valve
3740.510-MC-224654	D0 Detector - Cold Valve Assembly - Central Calorimeter Cryostat
3740.510-MC-224655	D0 Detector - Top Plate, Valve Body - Cold Valve Assembly
3740.510-MC-224656	D0 Detector - Piston Actuator Sub-Assy. - Cold Valve Assembly
3740.510-MC-224657	D0 Detector - Piston Guide Sub-Assy. - Cold Valve Assembly
3740.510-MC-224658	D0 Detector - Piston & Plug Sub-Assy. - Cold Valve Assembly
3740.510-MC-224659	D0 Detector - Lantern Ring - Cold Valve Assembly
3740.510-MC-224660	D0 Detector - Valve Body Sub-Assy. - Cold Valve Assembly
3740.510-MB-224661	D0 Detector - Plate Actuator - Piston Actuator Sub-Assy.
3740.510-MB-224662	D0 Detector - Rod, Piston - Piston Actuator Sub-Assy.
3740.510-MB-224663	D0 Detector - Seal Ring, Piston Rod - Piston Actuator Sub-Assy.
3740.510-MB-224664	D0 Detector - Sleeve, Actuator - Piston Actuator Sub-Assy.
3740.510-MB-224665	D0 Detector - Cuff, Bellows - Piston Actuator Sub-Assy.

TABLE 1

DWG. NUMBER	TITLE
3740.510-MB-224666	DO Detector - Top Plate, Cylinder - Piston Guide Sub-Assy.
3740.510-MB-224667	DO Detector - Cylinder, Piston Guide - Piston Guide Sub-Assy.
3740.510-MB-224668	DO Detector - Bottom Plate, Piston Guide - Piston Guide Sub-Assy.
3740.510-MB-224669	DO Detector - Sleeve, Piston Guide - Piston Guide Sub-Assy.
3740.510-MB-224670	DO Detector - Piston - Piston & Plug Sub-Assy.
3740.510-MB-224671	DO Detector - Sleeve, Piston - Piston & Plug Sub-Assy.
3740.510-MB-224672	DO Detector - Ring, Piston - Piston & Plug Sub-Assy.
3740.510-MB-224673	DO Detector - Plug, Piston - Piston & Plug Sub-Assy.
3740.510-MB-224674	DO Detector - Bottom Plate, Valve Body - Valve Body Sub-Assy.
3740.510-MC-224675	DO Detector - Cylinder, Valve Body - Valve Body Sub-Assy.

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Approved:

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The desired leak rate of the valve is 1×10^{-7} std atm cc/sec. In order to achieve this low leak rate at liquid argon temperature (87K), various design modifications were made to the Kel-F plug until cold testing of the valve in liquid nitrogen showed a sufficiently low leak rate. Another major modification made was the replacement of large body flanges, with a piston guide machined in one, with an all-welded construction and a separate lantern ring to guide the piston. The internal structure of the valve has remained basically the same despite the aforementioned modifications. Fig. 2 shows the design of the original prototype.

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The valve was then slowly cooled down by spraying nitrogen on it, and no change was observed. The valve was taken off of the leak detector and left only on the roughing pump with a vacuum reading of 20 microns. LN2 was sprayed on the valve until most of the valve was immersed in LN2. While cold, the data found on the following page was taken.

CONDITION	"CLOSE" PORT PRESSURE(PSIG)	VACUUM (MICRONS)	LEAK DETECTOR (STD ATM CC/SEC)	HE LEAK
rough pump only	48	18		
rough open/throttle partially open	50		6.3×10^{-8}	no
no rough pump/full leak detector	50		2.3×10^{-8}	no
open/reset	80	500		
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open/reset	100	18		
rough open/throttle partially open	100	14	2.38×10^{-7}	no

**The valve was then cycled 5 times, pumping down to 100 microns each time.

rough open/throttle partially open	100	25	2.45×10^{-7}	no

The measured leak rates were determined to be sufficient for the application involved, although they fell just short of the original desired leak rate. This original leak rate had been set with the intention of using the valve as a relief valve, and were therefore more rigid than those necessary for a fill/drain valve.

See Fig. 7.

Actuation

The valve is actuated by using helium pressure. Applying 100 psig to the "close" port of the valve and none to the "open" port moves the piston plug assembly into the position where the Kel-F seal is compressed against the seat of the valve. Reversing the pressure by applying 100 psig to the "open" port and none to the "close" port forces the piston plug assembly to move away from the valve seat allowing the fluid to pass through the valve and out the exhaust. The schematic for the actuation set-up is shown in Fig. 8. In the event that the primary helium source should be depleted, a pressure switch senses the pressure drop and in turn activates a secondary source set at a lower pressure.

Failure modes

Two possible ways in which the cold valve could fail might be improper functioning of the bellows or inadequate sealing at the plug/seat interface. With respect to the potential bellows problem, the inclusion of guide cylinders around the bellows should prevent the allowance for any squirm due to overpressure. The possibility of improper functioning because of the build up of dirt which may have passed through the valve in the bellows is very small because of the limited access of argon to the bellows.

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Installation

The cold valve is to be installed through nozzle N3V and welded into N3P of the CC cryostat. See Fig. 9 and DWG. 3740.214-MD-255078 for installation details. Precautions shall be taken to ensure that the valve exhaust is properly lined up with the exhaust pipe on the vessel before installation. The current drawing shows two welds which hold the valve in place. The possible addition of a drain guard ring at the top weld to help prevent residue from passing through the valve when the vessel is drained would modify this drawing. Details of such a ring have not yet been determined. The argon spill trough which surrounds the bottom of the vessel shall be designed to allow accessibility to N3V. Therefore, if the valve plug or any other part of the internal structure needs to be repaired or replaced, the necessary steps involved include penetration of the argon spill trough, removal of the flange on N3V, and the grinding of the weld which holds the top plate and outer cylinder of the valve together. The entire internal structure of the valve is attached only to this top plate, and hence, removal of this single plate constitutes removal of the entire valve (except the outer cylinder and bottom plate).

After being installed in the vessel, it is necessary that the valve be leak checked again (warm only) to ensure proper functioning. While the set-up for such a test has not yet been determined, the main component would be the design of a cover that would fit over the valve opening inside of the vessel. The seal between this cover and the valve must be vacuum tight in order that a vacuum could be drawn on the plug/seat interface to measure the leak rate of the valve.

When testing of the valve is complete, the valve must be protected from dirt that may fall into it during the period in which the rest of the calorimeter components are assembled. This protective cover should be easy to remove after the modules are in place.

Any additional comments on the installation procedure shall be appended to this report when they have been finalized.

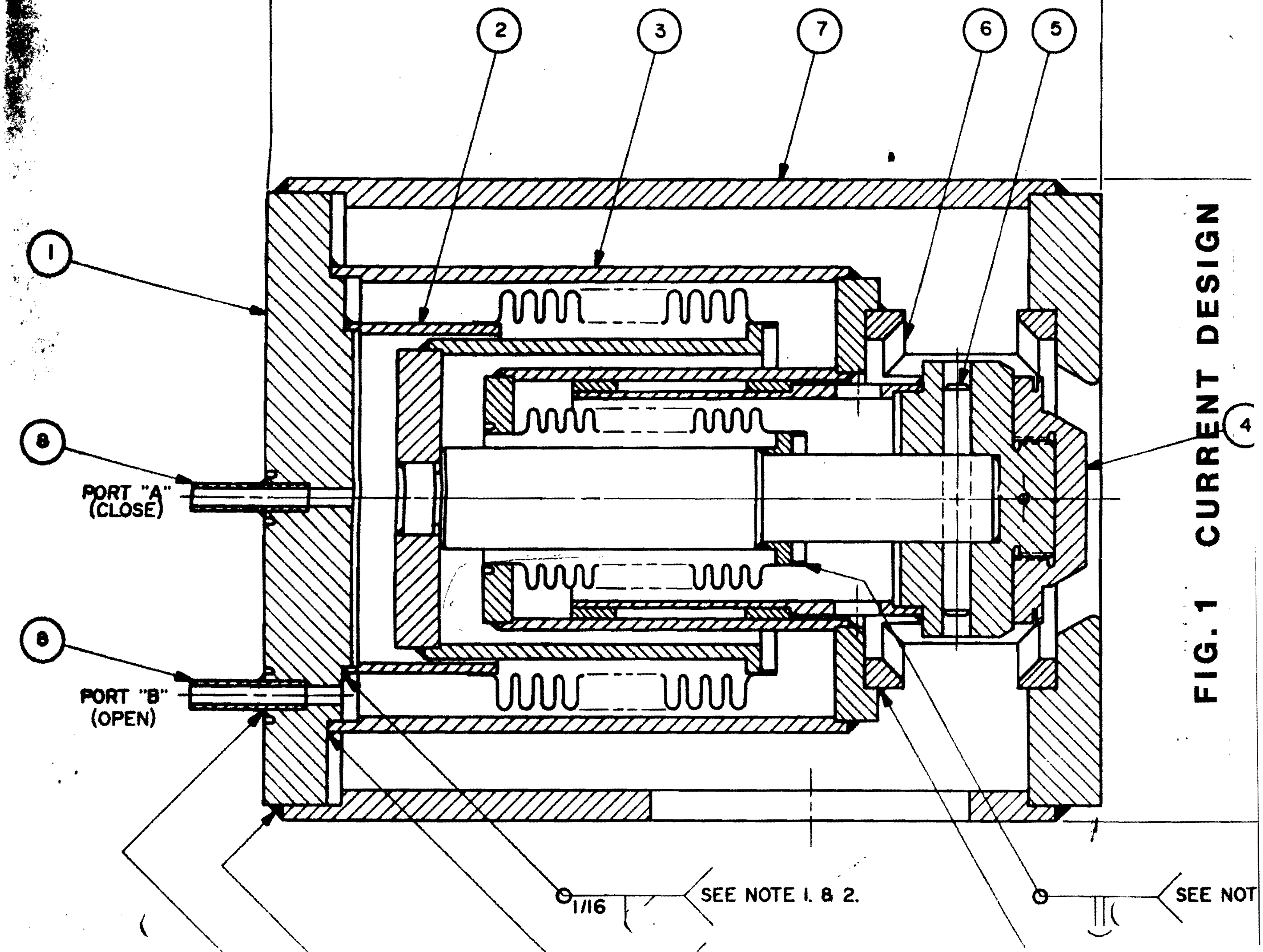

FIG. 1 CURRENT DESIGN

ORIGINAL PROTOTYPE

REV.	DESCRIPTION	DRAWN APPR.	DATE DATE
A	ADDED VCR TYPE FTG'S. ADDED NOTES 1 & 2	TP	12/9/85

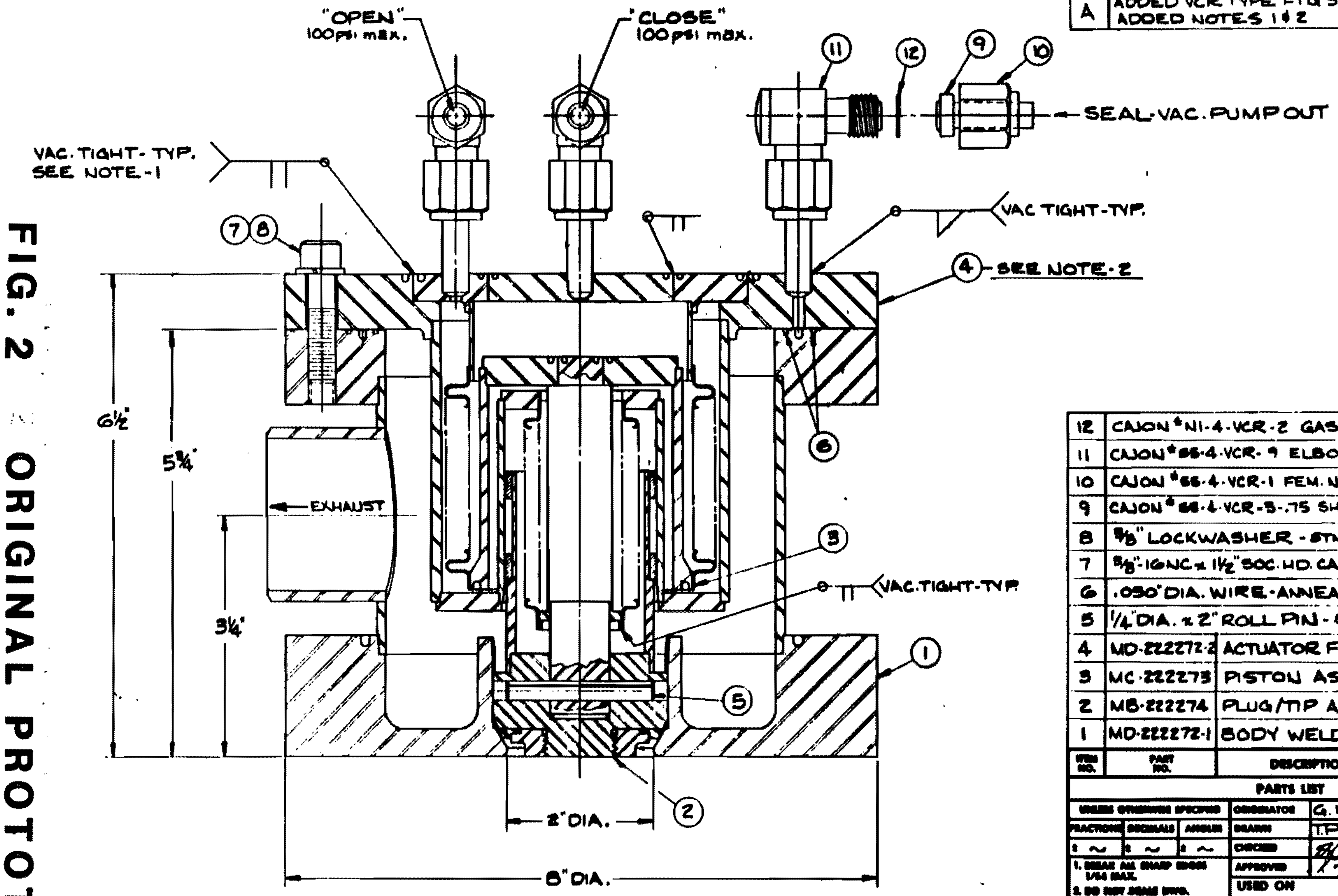

FIG. 2 ORIGINAL PROTOTYPE

NOTES:
1) BEFORE WELDING, ALIGN FITTING PORTS AS SHOWN.
2) EXHAUST ARROW ON ITEM 4 TO BE INSTALLED IN LINE WITH EXHAUST PIPE.

ITEM NO.	PART NO.	DESCRIPTION OR SIZE	QTY. REQ.
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11	CAJON #SS-4-VCR-9 ELBOW		3
10	CAJON #SS-4-VCR-1 FEM. NUT (¼" TUBE)		6
9	CAJON #SS-4-VCR-3-.75 SHORT GLAND		6
8	⅜" LOCKWASHER - STNL'S.		16
7	⅜"-16NC × 1½" SOC. HD. CAP SCR - STNL'S		16
6	.050" DIA. WIRE-ANNEALED COPPER		A/R
5	¼" DIA. × 2" ROLL PIN - STNL'S.		1
4	MD-222272-2	ACTUATOR FLG. ASS'Y.	1
3	MC-222273	PISTON ASS'Y.	1
2	MB-222274	PLUG/TIP ASS'Y.	1
1	MD-222272-1	BODY WELDMENT	1

PARTS LIST

UNLESS OTHERWISE SPECIFIED			ORIGINATOR	G. MULHOLLAND	
FRACTIONS	DECIMALS	ANGLES	DRAWN	T. PARKER	11-19-85
± ~	± ~	± ~	CHECKED		11-22-85
			APPROVED		12-9-85

1. BREAK ALL SHARP EDGES 1/64 MAX.
2. DO NOT SCALE DWG.
3. DIMENSIONING IN ACCORD WITH ANSI Y14.5 STD'S.
✓ MAX. ALL MACHINED SURFACES

USED ON

MATERIAL: AS NOTED

FERMI NATIONAL ACCELERATOR LABORATORY
UNITED STATES DEPARTMENT OF ENERGY

DØ
ARGON COLD VALVE
ASSEMBLY

SCALE	FILMED	DRAWING NUMBER	REV.
1"=1"		3740.510-MC-222271	A

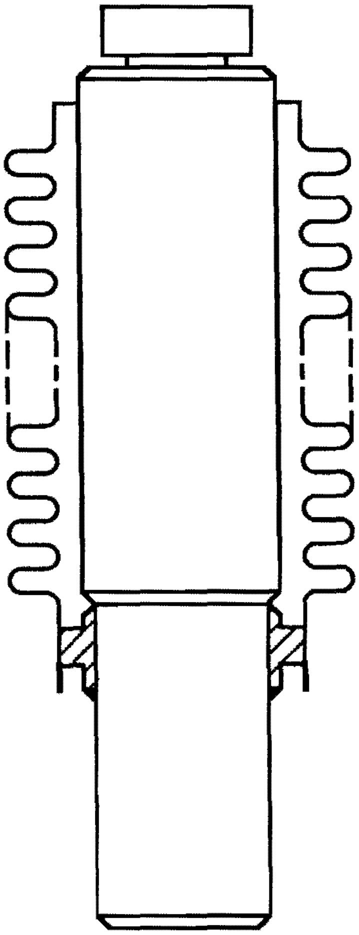


FIG. 3 SUGGESTED MODIFICATIONS

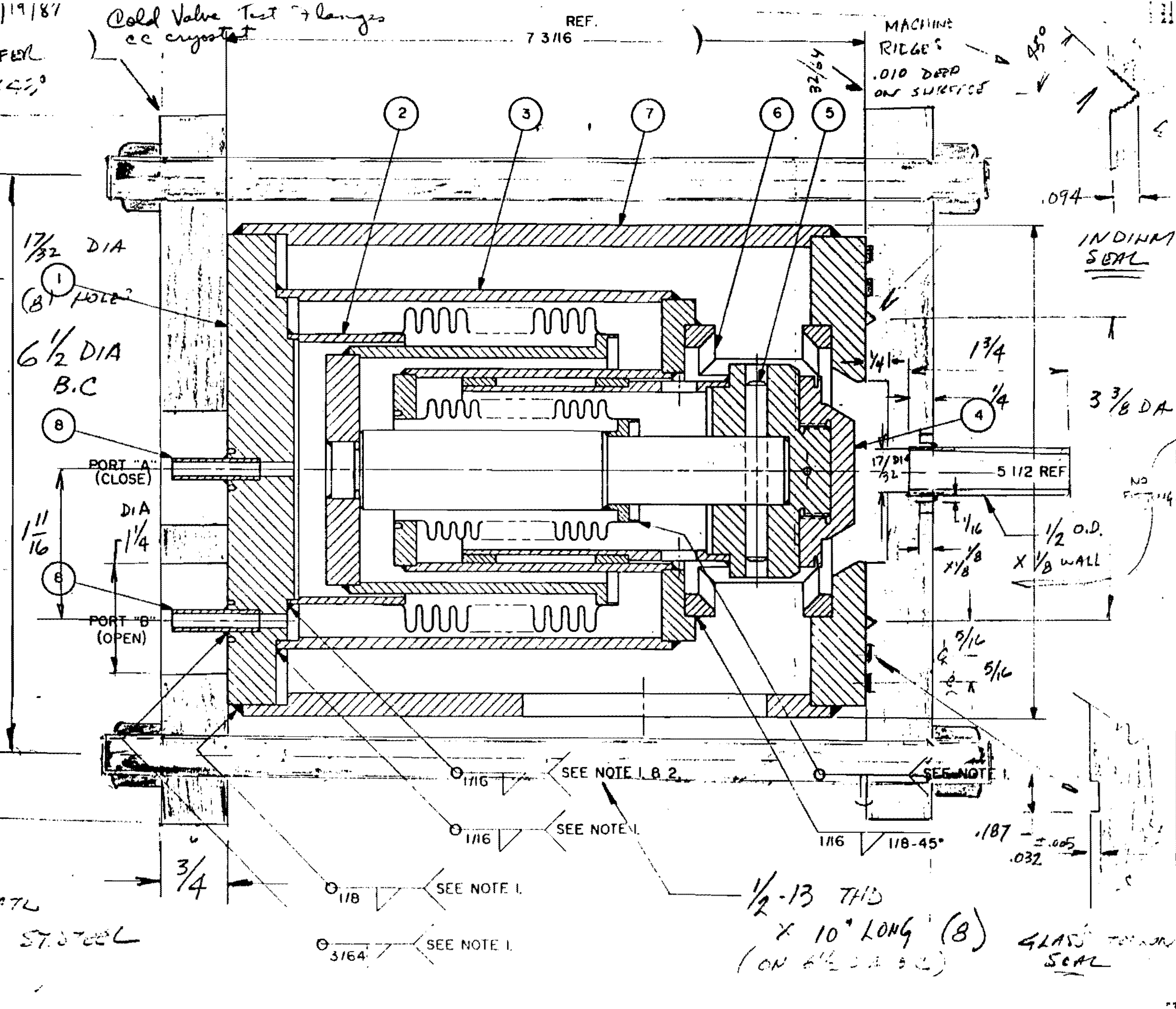


FIG. 4 TEST FLANGE ASSEMBLY

"VACUUM TE
FOR COLD V.

ASSEM
ASSY.

1. WELD ITEM 2 (PISTON ACT
TO ITEM 1 (TOP PLATE, VALV
THRU PORT "A".

2. WELD ITEM 3 (PISTON GU
ITEM 1 (TOP PLATE, VALVE &
BELLOWS (ON PISTON GUIDE
SEAL RING, PISTON ROD (ON
SUB-ASSY.). PUMP THRU POR

3. ASSEMBLE ITEMS 4, 5 &

4. WELD ITEM 7 (VALVE BOD
ITEM 1 (TOP PLATE, VALVE &
ON VALVE BODY BOTTOM PL
SIDE PORT ON VALVE BODY

5. WELD ITEM 8 (1/4 O.D. ×
TUBING) INTO PORTS "A" &
PLATE, VALVE BODY). 2"

8	~~1/2 DIA TUBE~~	TU
7	MC-224660	VA
6	MC-224659	LA
5	———	SP
4	MC-224658	PI
3	MC-224657	PI
2	MC-224656	PI
1	MC-224655	T
ITEM NO.	PART NO.	

UNLESS OTHERWISE SPECIFIED

FRACTIONS	DECIMALS	ANGLES
± 1/64	± .005	± 1°

1. BREAK ALL SHARP EDGES 1/64 MAX.
2. DO NOT SCALE DWG.
3. DIMENSIONING IN ACCORD WITH ANSI Y14.5 STD's.

√ MAX. ALL MACHINED SURFACES

COLD VALVE TEST SET-UP

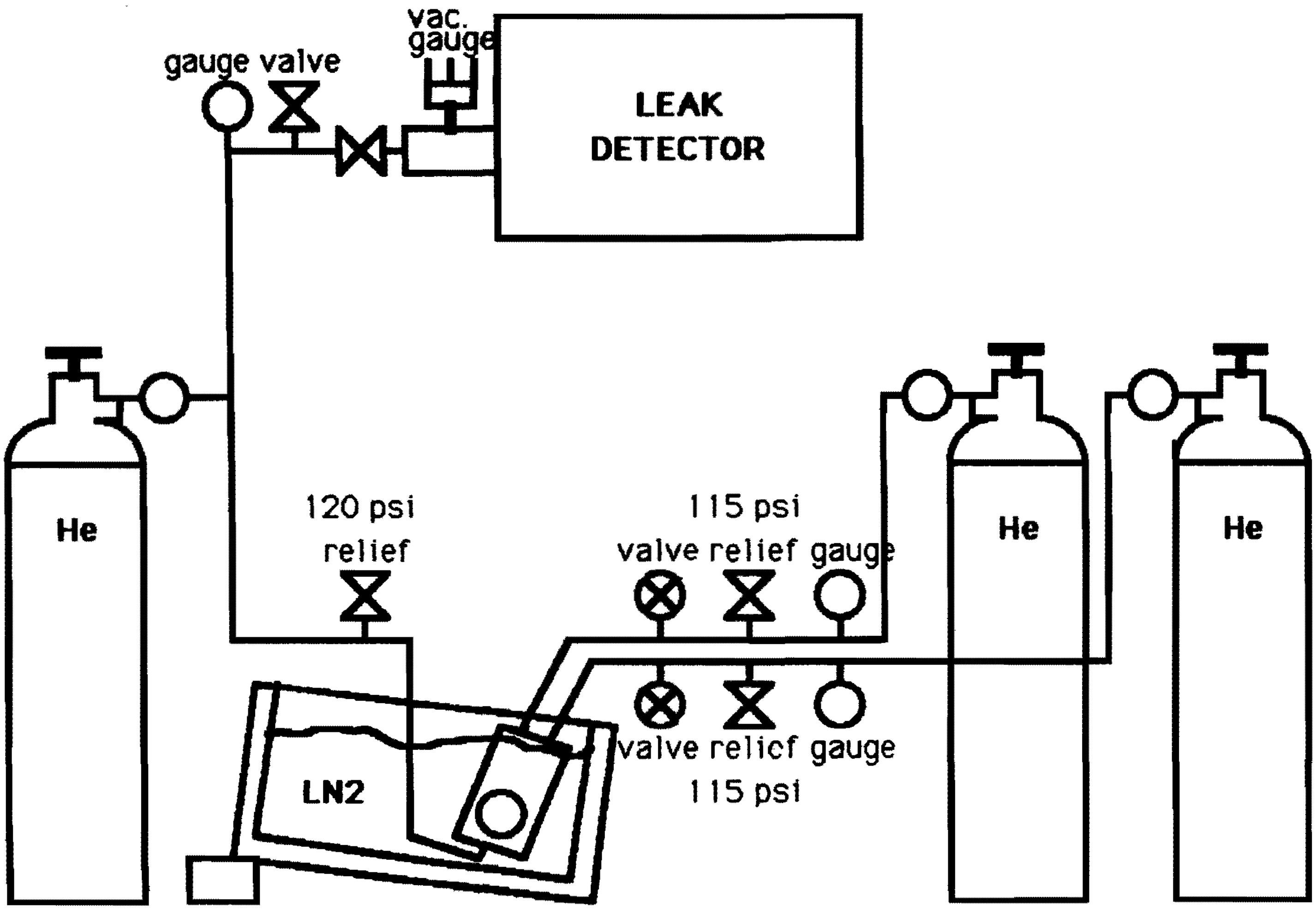
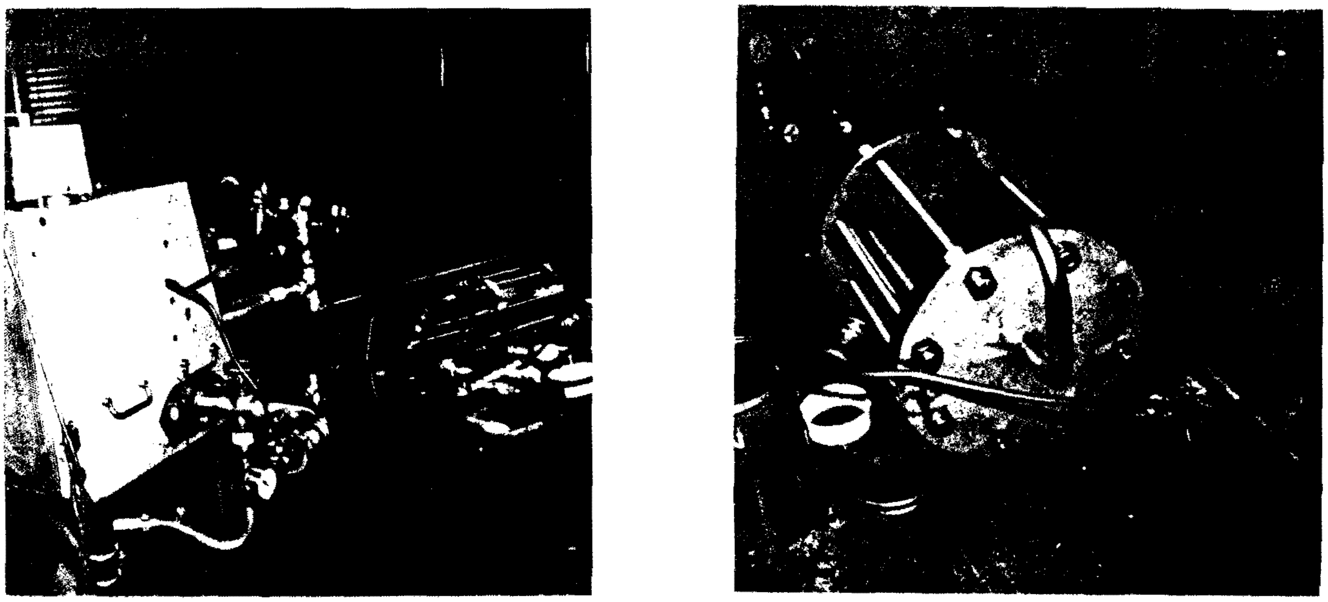


FIG. 5 TEST SET-UP

12/2/87 COLD VALVE FOR CC
CRYOSTAT D0

a)



12/2/87 COLD VALVE WARM TEST

b)

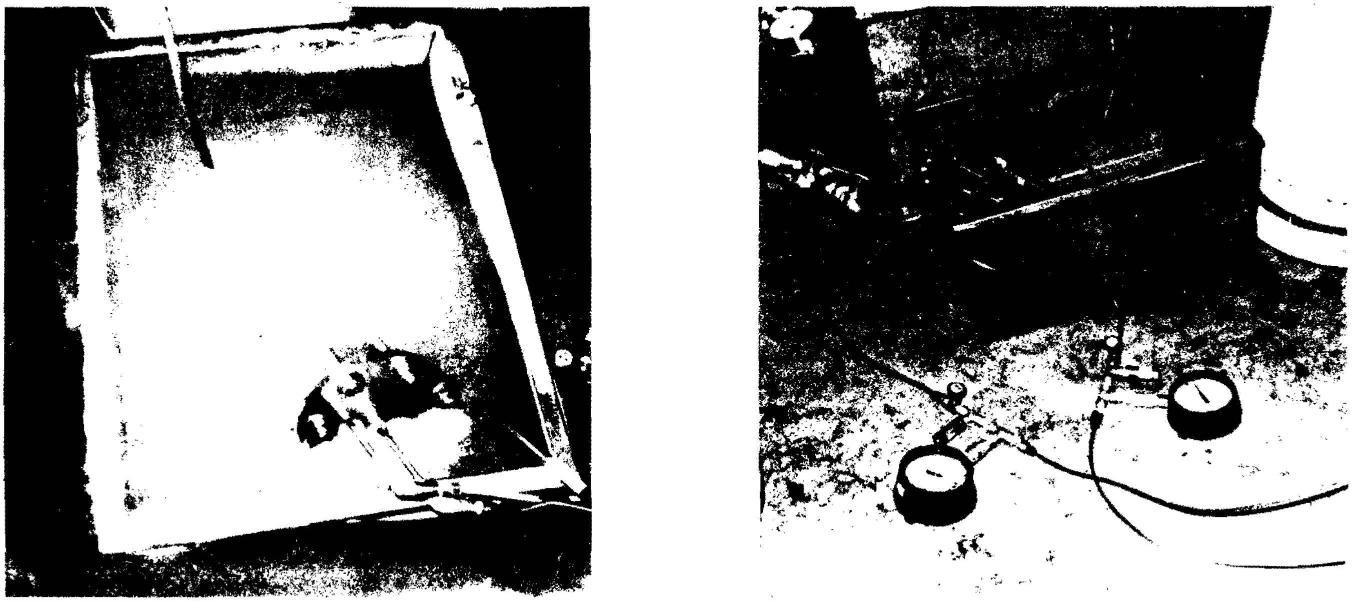
12/2/87 COLD VALVE TEST FLANGES

c)

FIG. 6

12/2/87 COLD VALVE TEST SET-UP

a)



12/3/87 COLD VALVE TEST

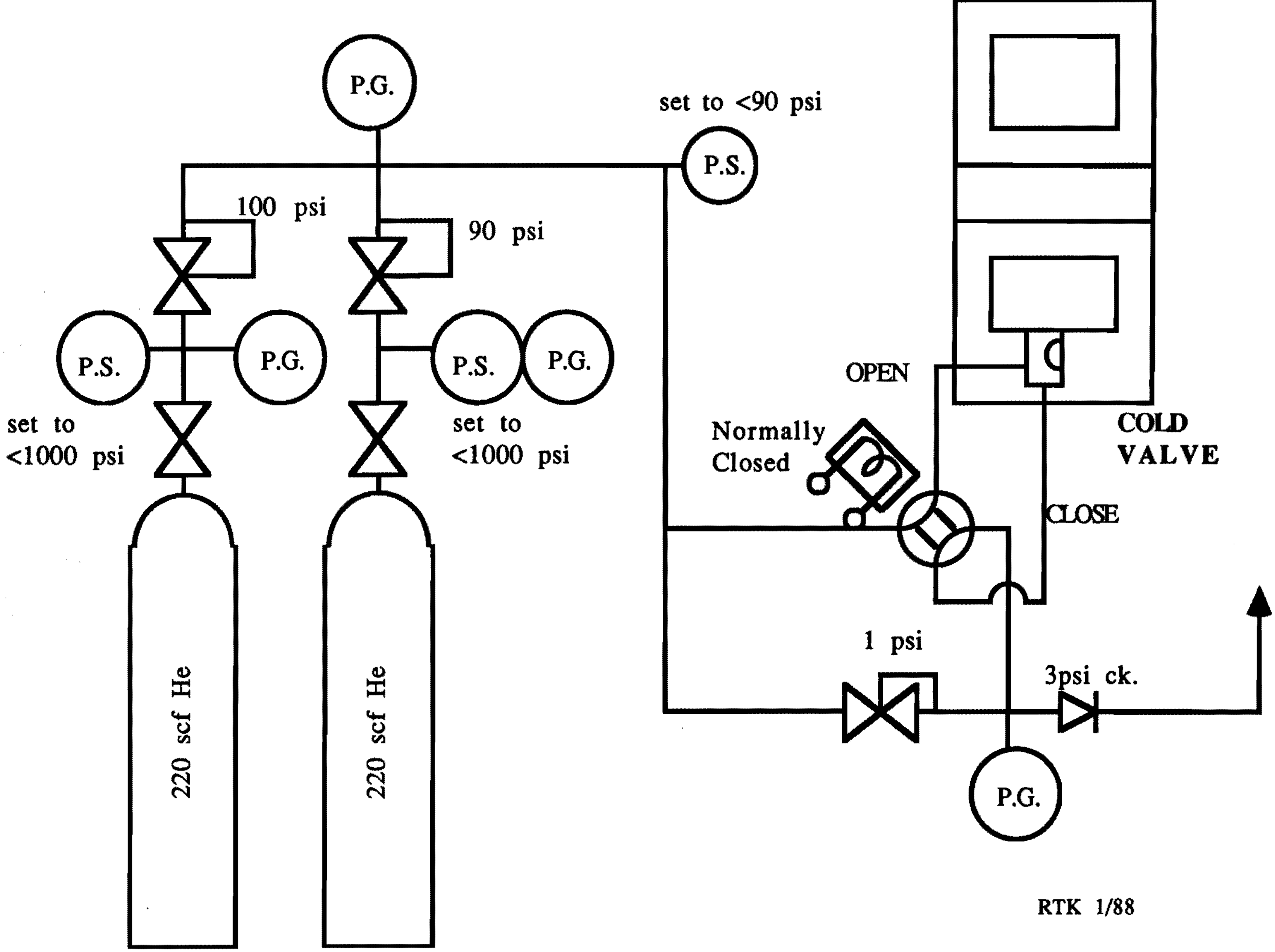
b)

12/2/87 COLD VALVE TEST SET-UP

c)

FIG. 7

COLD VALVE ACTUATION SCHEMATIC



RTK 1/88

FIG. 8 COLD VALVE ACTUATION

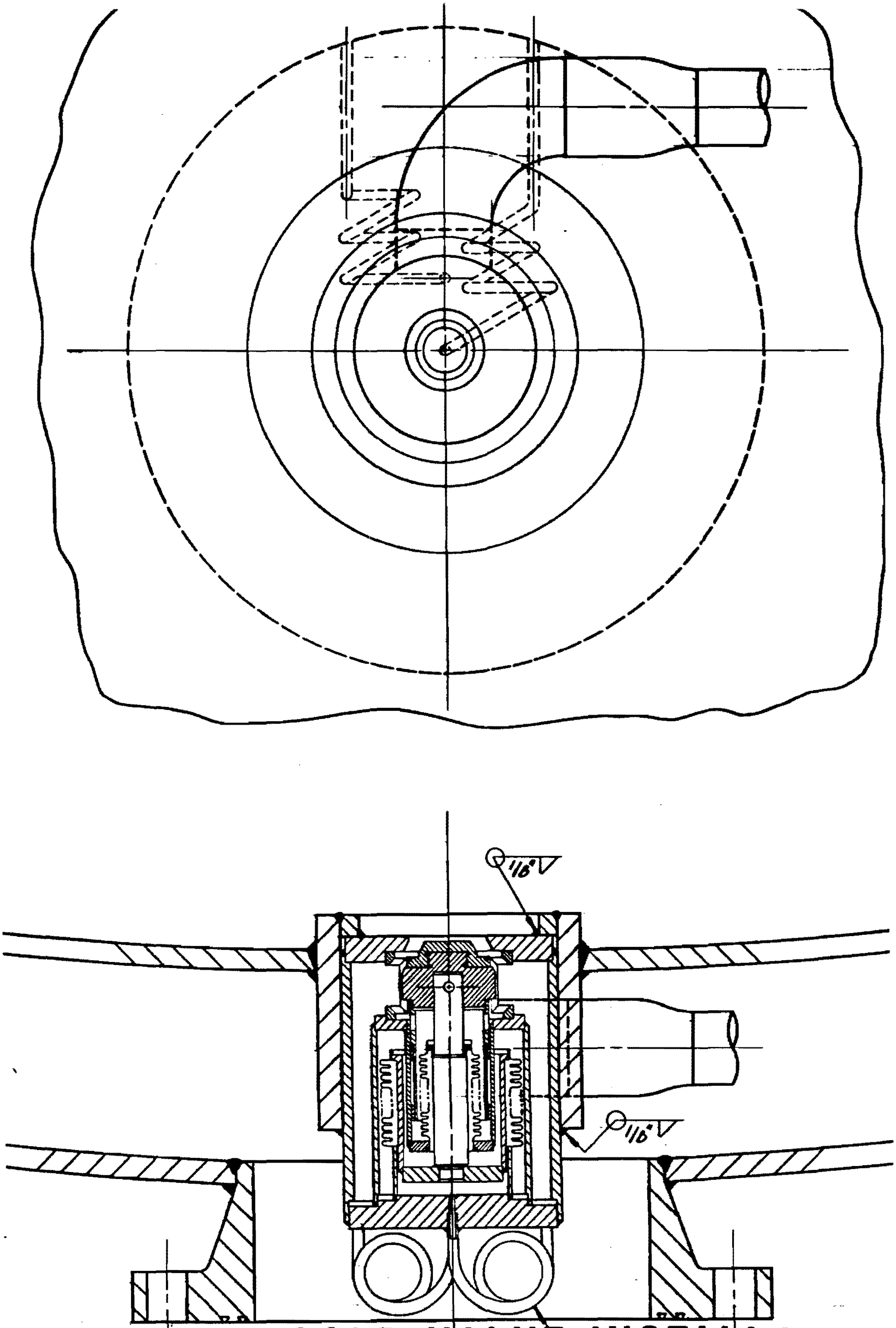


FIG. 9 COLD VALVE INSTALLATION

"COLD VALVE DRAWINGS"

DWG. NUMBER	TITLE
3740.510-MB-222285	D0 Detector 1 1/2" O.D. "Pathway" Bellows Argon Relief Cold Valve
3740.510-MB-222286	D0 Detector 3 5/8" O.D. "Flexonics" Bellows Argon Relief Cold Valve
3740.510-MC-224654	D0 Detector - Cold Valve Assembly - Central Calorimeter Cryostat
3740.510-MC-224655	D0 Detector - Top Plate, Valve Body - Cold Valve Assembly
3740.510-MC-224656	D0 Detector - Piston Actuator Sub-Assy. - Cold Valve Assembly
3740.510-MC-224657	D0 Detector - Piston Guide Sub-Assy. - Cold Valve Assembly
3740.510-MC-224658	D0 Detector - Piston & Plug Sub-Assy. - Cold Valve Assembly
3740.510-MC-224659	D0 Detector - Lantern Ring - Cold Valve Assembly
3740.510-MC-224660	D0 Detector - Valve Body Sub-Assy. - Cold Valve Assembly
3740.510-MB-224661	D0 Detector - Plate Actuator - Piston Actuator Sub-Assy.
3740.510-MB-224662	D0 Detector - Rod, Piston - Piston Actuator Sub-Assy.
3740.510-MB-224663	D0 Detector - Seal Ring, Piston Rod - Piston Actuator Sub-Assy.
3740.510-MB-224664	D0 Detector - Sleeve, Actuator - Piston Actuator Sub-Assy.
3740.510-MB-224665	D0 Detector - Cuff, Bellows - Piston Actuator Sub-Assy.

TABLE 1

DWG. NUMBER	TITLE
3740.510-MB-224666	DO Detector - Top Plate, Cylinder - Piston Guide Sub-Assy.
3740.510-MB-224667	DO Detector - Cylinder, Piston Guide - Piston Guide Sub-Assy.
3740.510-MB-224668	DO Detector - Bottom Plate, Piston Guide - Piston Guide Sub-Assy.
3740.510-MB-224669	DO Detector - Sleeve, Piston Guide - Piston Guide Sub-Assy.
3740.510-MB-224670	DO Detector - Piston - Piston & Plug Sub-Assy.
3740.510-MB-224671	DO Detector - Sleeve, Piston - Piston & Plug Sub-Assy.
3740.510-MB-224672	DO Detector - Ring, Piston - Piston & Plug Sub-Assy.
3740.510-MB-224673	DO Detector - Plug, Piston - Piston & Plug Sub-Assy.
3740.510-MB-224674	DO Detector - Bottom Plate, Valve Body - Valve Body Sub-Assy.
3740.510-MC-224675	DO Detector - Cylinder, Valve Body - Valve Body Sub-Assy.

D-ZERO
COLD VALVE
APPENDIX-A

D-Zero Engineering Note: 3740.510-EN-139-A

R. T. Krasa
March 1, 1988

Approved: ______________________

Plug Deformation Investigation

After cycling of the cold valve under 100psi air pressure it was noticed that, although performance was not affected, the lip of the plug had yielded permanently. This prompted a review of the design criteria for the specified groove(see fig. 1). It is believed that the safety factor used to specify the original plug dimensions was sufficient under cold cycling conditions. However, the yield strength of the Kel-F warm(300K) is approximately 4.5 times smaller than at cold(77K) test conditions. Therefore, the original safety factor was inadequate to prevent permanent yielding at room temperature.
This prompted a recalculation of yield strengths. The plug load was taken as the force provided by the pneumatic force generated at maximum pressure. A thinner, shallower groove was selected to provide a safety factor greater than two at room temperature. Warm and cold cycling revealed no signs of permanent yield. Warm and cold leak checking with a sensitivity of 1.9×10^{-10} He std. cc/sec. exposed no evidence of a leak.

The new plug design was approved and drawing number 3740.510-MB-224673 was altered. The new plug design will be used in the drain/fill Cryostat Cold Valve.

Dirt Guard Design

The "Dirt Guard" for the Cold Valve will consist of a piece of 6" I.P.S. sch. 10 pipe welded to the top of the cylinder in which the Cold Valve is mounted. Its purpose is to protect the Cold Valve from any matter heavy enough to settle to the bottom of the cryostat, for example, a stray nut or large U_3O_8 particles.

Design Criteria

The pipe should be tall enough to prevent as much collected sediment as possible from being carried into the drain/fill lines, but short enough to preclude interference with module installation, ect.. A length of $2^1/_2$ inches was chosen making the Dirt Guard approximately equal in height to the heaters.

What is the largest particle of U_3O_8(most likely the least dense solid particle sediment) that can be picked up and carried into the drain/fill lines?

Model:

Two flow rates will be used, 5000 gal/8 hours(expected maximun drain flow rate) and 5000 gal/ 6 hours. The results given from the larger flow rate can be taken as an **absolute** maximum value while the results from the lower flow rate can be taken as a normal conditions maximum value.

Assumptions:

1) a surface boundary of equal flow rates near the cold valve approximates a sphere.
2) turbulence and vortex flow effects are not addressed.

Steps used to determine the largest particle of U_3O_8 that can be expected in the cryostat fill drain line(fig. 2):

A) Calculate the area for a sphere with a radius equal to the distance from the center of the dirt guard opening to the corner of intersection of the vessel and Cold Valve.
B) Subtract the area lost from the sphere due to the Cold Valve and Module intersections with the sphere. Use the result as the Effective Flow Area.
C) Convert gal./hour to in.^{3}/sec..
D) Divide Flow Rate by Effective Flow Area to get Flow Velocity at the surface boundary.

Terminal Velocity refers to the maximum speed at which the liquid can flow past the particle. A particle with a Terminal Velocity greater than the Flow Velocity will remain at the bottom of the cryostat as sediment, since gravitational influence will not be completely overcome by force due to the flow. Therefore, by matching Flow velocity with Terminal Velocity, the maximum probable exiting particle size can be determined.

E) Use Table 1 to select the appropriate particle size for a given Flow Velocity.

Conclusion:

No spherical particle originating in the cryostat greater than 64 microns in diameter, with a density equal to or greater than that of U_3O_8(8300 kg/m^3) should be found in the drain/fill lines of the cryostats. It is possible, although unlikely that particles will fall from above directly into the Cold Valve due to the flat surface approximately 7" above the valve.

Very few, if any, particles 69 microns in diameter subject to the above constraints can be expected in the drain/fill line.

Kel-F Data

All data used in the design of the Cold Valve plug using Kel-F 81 plastic is attached.

Additional Cold Valve Drawings

3740.214-MD-255078 -COLD VALVE INSTALLATION ASSEMBLY

3740.214-MB-255.80 -SLEEVE

Vacuum Vessel Cold Valve Port Cap

3740.214-MB-255438 -BOTTOM PLATE

3740.214-MB-255437 -BOTTOM FLANGE RING

COLD VALVE PLUG DESIGN CHANGES

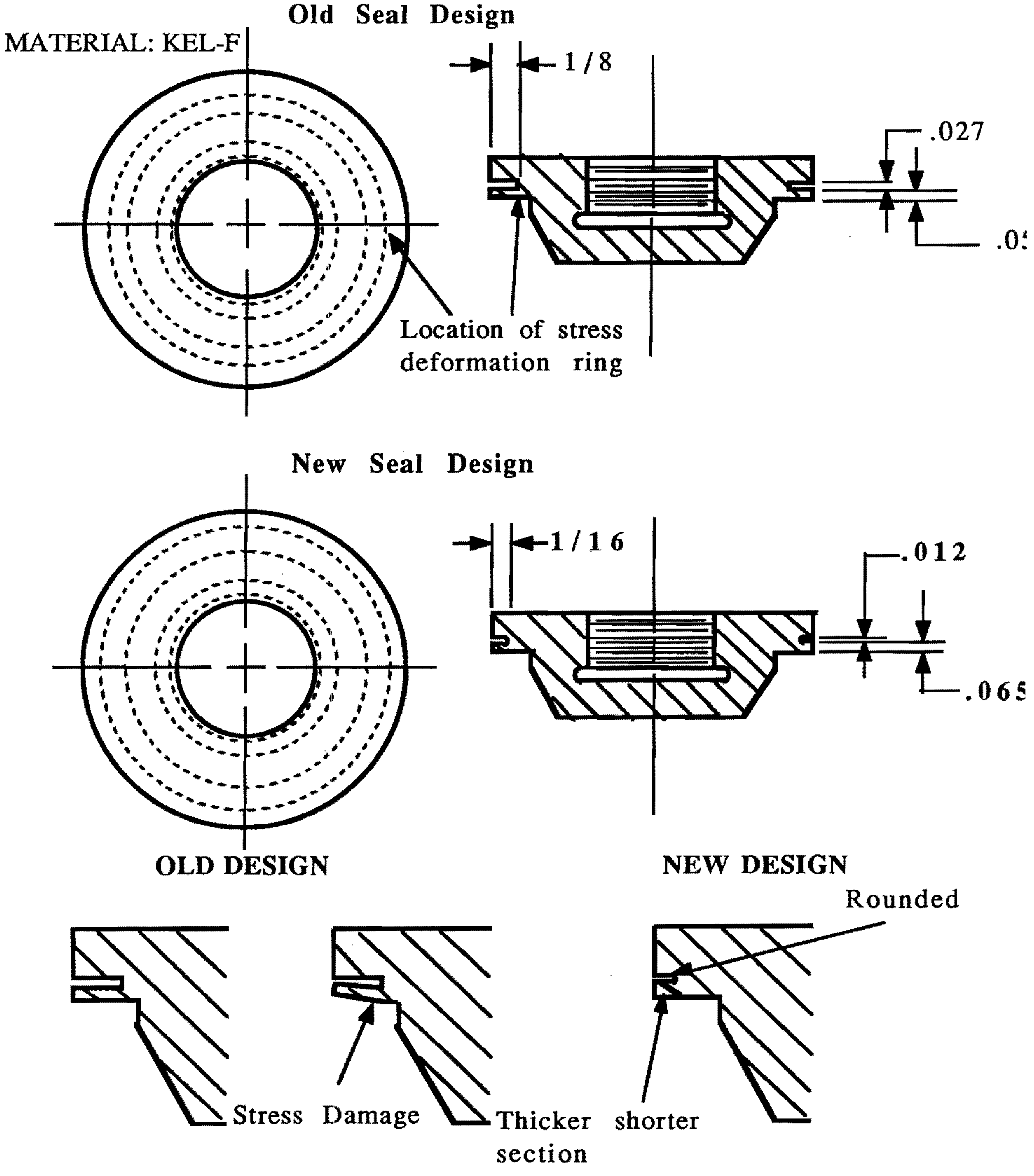


figure 1

Calculations:

$$\frac{X\text{ gal.}}{Y\text{ hours}} \times \frac{1\text{ hour}}{3600\text{ sec.}} \times \frac{.003785\text{ m}^3}{1\text{ gal.}} \times \left(\frac{100\text{ cm}}{1\text{ m}}\right)^3$$

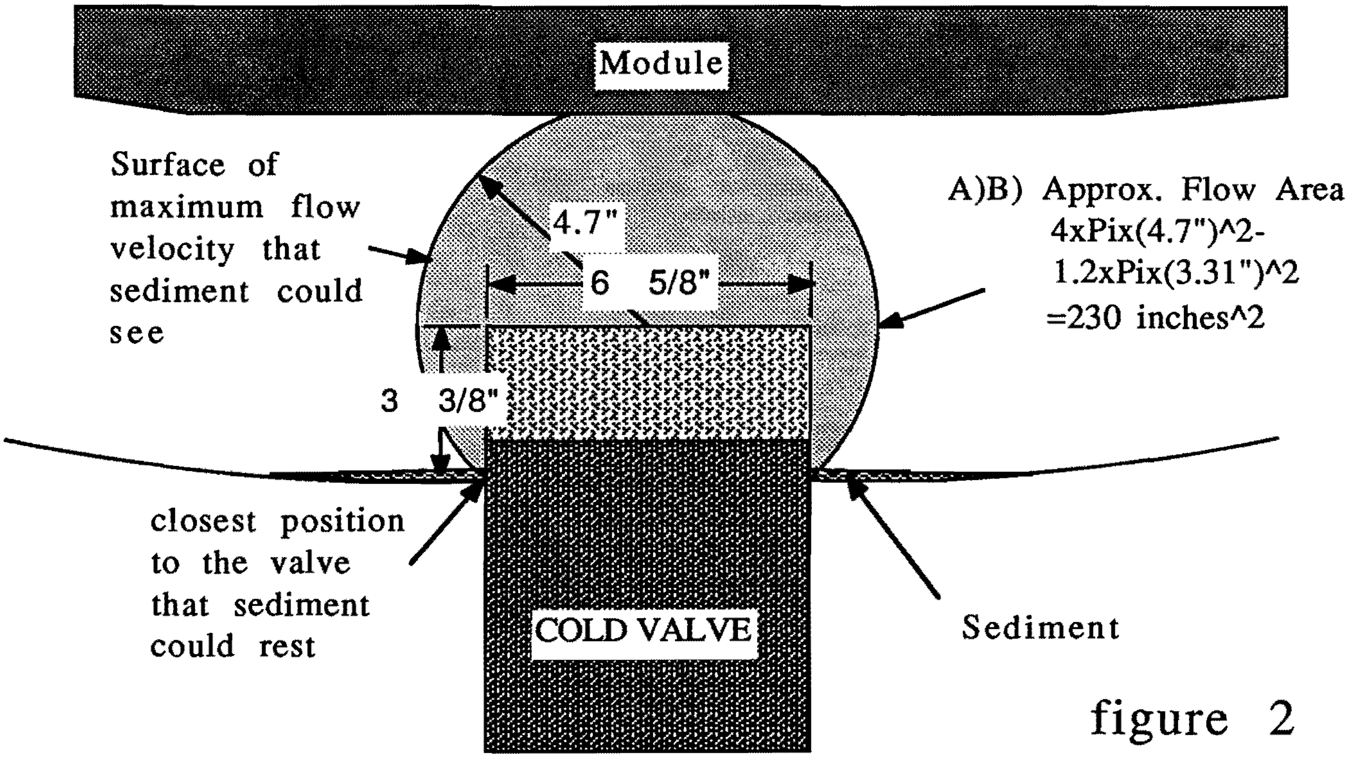
$$\times \left(\frac{1\text{ in.}}{2.54\text{ cm}}\right)^3 = \boxed{\frac{X\text{ gal.}}{Y\text{ hours}} \times \frac{.06416\text{ hr. in.}^3}{\text{gal. sec.}}}$$

C) $$\frac{5000\text{ gal.}}{8\text{ hours}} \times \frac{.06416\text{ hr. in.}^3}{\text{gal. sec.}} = \frac{40.1\text{ in.}^3}{\text{sec.}}$$

$$\frac{5000\text{ gal.}}{6\text{ hours}} \times \frac{.06416\text{ hr. in.}^3}{\text{gal. sec.}} = \frac{53.5\text{ in.}^3}{\text{sec.}}$$

D) $$\frac{40.1\text{ in.}^3}{\text{sec.}} \times \frac{1}{230\text{ in.}^2} \times \frac{2.54\text{cm}}{1\text{ in.}} = \boxed{.44\text{ cm/sec}}$$

$$\frac{53.5\text{ in.}^3}{\text{sec.}} \times \frac{1}{230\text{ in.}^2} \times \frac{2.54\text{cm}}{1\text{ in.}} = \boxed{.59\text{ cm/sec.}}$$

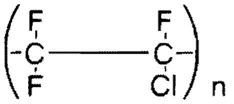


A)B) Approx. Flow Area
$4\text{xPix}(4.7")^2-$
$1.2\text{xPix}(3.31")^2$
$=230\text{ inches}^2$

figure 2

Introduction

"Kel-F 81" Plastic is the homopolymer of chlorotrifluoroethylene.



$$\left(-\overset{F}{\underset{F}{C}}-\overset{F}{\underset{Cl}{C}}- \right)_n$$

This polymer structure imparts the properties that make "Kel-F 81" Plastic a truly unique engineering thermoplastic. A complete family of powdered and pelletized polymers is covered by the "Kel-F 81" Plastic product line. These products are designed for the production of high-performance parts by the various conventional thermoplastic processing techniques currently used in the plastic industry.

Since "Kel-F 81" Plastic is a versatile material whose properties may be modified over a wide range depending upon fabrication conditions, careful specification of pertinent properties for a given application is advisable. To meet the needs of the application, 3M offers a complete set of literature on "Suggested Guidelines" for the:

- Injection molding of "Kel-F 81" Plastic
- Compression molding of "Kel-F 81" Plastic
- Machining of "Kel-F 81" Plastic
- Finishing (Machining and Heat Treatment) of "Kel-F 81" Plastic
- Extrusion of "Kel-F 81" Plastic

This Engineering Manual describes the unique combination of physical and mechanical properties, nonflammability, high optical transparency, chemical resistance, near zero moisture absorption and excellent electrical properties that cannot be found in any other thermoplastic fluoropolymer with a useful temperature range of —240° C (—400° F) to +200° C (400° F).

"Kel-F 81" Plastic Molding Powders are suitable for processing into parts that meet the following government and industrial specifications:

MIL-P-46036
MIL-P-21470
LP-385C
AMS-3645
AMS-3650
FDA, Regulations under paragraph CRF 177.1380

"Kel-F 81" Plastic, following 3M processing guidelines, also meets Marshall Space Flight Center Specification MSFC-SPEC-106, Testing Compatibility of materials for liquid oxygen systems.

Applications

The following list provides an idea of the extraordinary versatility of this tough, durable fluoroplastic:

Valves: Valve seats, seals and stems of molded or machined Kel-F have been used very successfully for many years.

Seals: Lip, o-ring, v-rings and special construction seals are fabricated for valves, compressors and pumps.

Films: Food packaging, pharmaceutical packaging, optical recording, electroluminescent display panels to take advantage of its excellent permeation resistance.

Gaskets: Pressure gaskets, diaphragms, liquid gauge shields are fabricated from Kel-F for use in fluid handling systems.

Bearings: Including sleeve and thrust, are fabricated where high wear is a problem.

Stock shapes: Molded sheets, solid rods and rings, as well as extruded rod and tubing, are available through fabricators.

General Properties

TABLE 1:
GENERAL PHYSICAL & MECHANICAL PROPERTIES
OF "KEL-F 81" PLASTIC

Property	ASTM Method	Units and Typical Values*		
Zero Strength[1] Time (ZST)	D-1430	200-420 seconds		
Melting Point, Tm	DTA	SI	210-215 C	
		English	410-420 F	
Specific Gravity	D-792	2.08-2.185		
Tensile Strength @ Break	D-638	SI	25 C 125 C	40 MPa 4 MPa
		English	77 F 258 F	5725 psi 510 psi
Elongation @ Break	D-638	SI	25 C 125 C	150% 400%
		English	77 F 258 F	150% 400%
Tensile Modulus	D-638	SI	25 C	14 x 10^2 MPa
		English	77 F	207 x 10^3 psi
Compressive Modulus	D-695	SI	25 C	1350 MPa
		English	77 F	180 x 10^3 psi
Compressive Strength	D-695	SI	25 C	38 MPa
		English	77°F	55 x 10^2 psi
Flexural Modulus	D-695	SI	25 C	1250 MPa
		English	77°F	180 x 10^3 psi
Flexural Strength	D-695	SI	25°C	60 MPa
		English	77°F	8500 psi
Izod Impact (notched)	D-256	SI	25°C	265 Kj/m
		English	77°F	5 ft. lb/in
Hardness, Shore D	D-2240	SI	75-80	

poisson's ratio 0.44

(*Not for specification purposes.)

"Kel-F 81" Plastic may well be described as crystallizable, but never completely crystalline. The degree of crystallinity in any "Kel-F 81" Plastic molded part is a function of its thermal history. It is this controllable variation in the amount of crystallinity which gives rise to so many of the unique properties of this plastic.

It is common to speak of the "quick quenched" material as "amorphous" and of the "slow-cooled" material as "crystalline." At best, these terms are relative, since only thin parts can be quenched rapidly and completely enough to inhibit crystal growth. In all cases, some crystal nuclei develop, particularly when thicker molded or extruded parts are cooled rapidly.

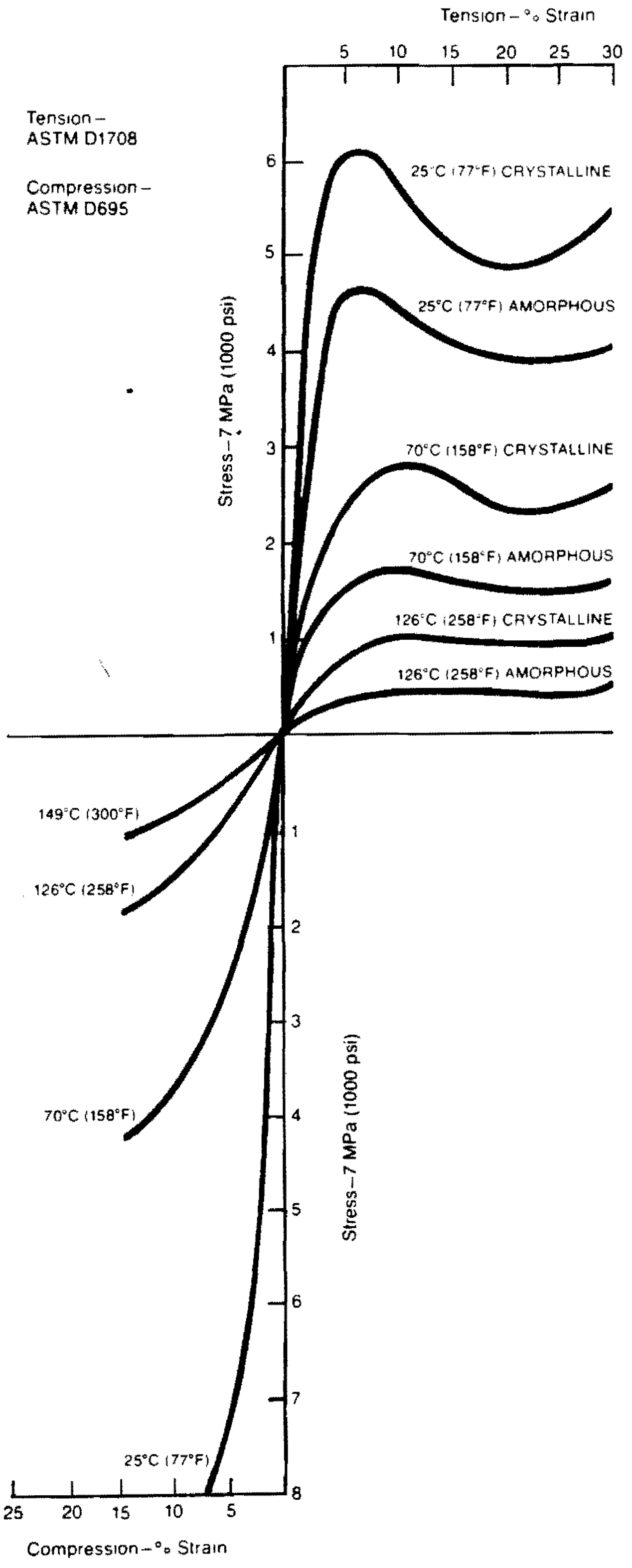
At higher degrees of crystallinity, "Kel-F 81" Plastic is a denser, less transparent material with higher tensile modulus and lower elongation. It has greater resistance to the penetration of liquids and vapors and a 200°C use temperature. "Amorphous" forms of the plastic, on the other hand, are less dense, more elastic, optically clear, and exceptionally tough. To maintain these characteristics of the amorphous state for a specific application, the upper use temperature should be limited. Spherulitic domains can develop on prolonged aging at 150-200°C (300-400°F).

The combination of properties discussed in this manual is not available in any other engineering plastic, and makes "Kel-F 81" Plastic an extremely versatile material with a unique balance of mechanical, chemical and electrical properties as shown in Table I.

[1] Zero Strength Time (ZST) is a simple test (described in detail in ASTM D-1430) designed to give approximation of relative molecular weight. It involves placing a notched (0.047 0.001" x 0.062 0.003" cross section at the notch) and weighted (7.5g) test specimen in an oven at 250± 2°C and measuring the time (in seconds) to reach zero strength (melt or break). For the specific details please refer to the ASTM test procedure.

High Compressive Strength

FIGURE I
"KEL-F 81" PLASTIC
STRESS/STRAIN RELATIONSHIPS



The very high compressive strength of the "Kel-F 81" Plastic polymer is indicative of the low "cold-flow" characteristics shown in Figure I. The "Kel-F 81" Plastic polymer has good "elastic memory." The permanent deformation, that which remains after removal of a force, is only 4 to 5% of the deformation caused by application of a load of 60 MPa (8,500 psi). The high compressive strength and good resistance to cold flow of the "Kel-F 81" Plastic polymer qualifies molded parts for use as valve sealing material and as structural members.

Thermal Properties

TABLE II
THERMAL PROPERTIES

Property	ASTM Method	Units and Values	
Thermal Conductivity	C 177	SI	0.15 W/(m K)
		English	1.0 BTU in/hr ft^2 °F
Specific Heat	DSC	SI	900 J/(kg K)
		English	.215 BTU/lb °F
Heat of Fusion	DSC	SI	1200 J/(kg K)
		English	2.85 BTU/lb °F
Deflection Temperature	D 648	SI	.46 MPa 126°C 1.82 MPa 70°C
		English	66 psi 258°F 264 psi 167°F
Limiting Oxygen Index	D 2863	SI	100%
Vertical Burn Rating	UL 94		VE-O
Resistance to Heat, Continuous Service		SI	200°C
		English	400°F

The physical properties of "Kel-F 81" Plastic (Table I), the thermal properties (Tables II and III) and the low coefficient of thermal expansion (Figure II) indicate that "Kel-F 81" Plastic can be put to practical use between −240°C (−400°F) and +200°C (+400°F). Under certain conditions, "Kel-F 81" Plastic has seen service at temperatures close to absolute zero, −275°C (−420°F).

TABLE III
CRYOGENIC MECHANICAL PROPERTIES[a]
"KEL-F 81" PLASTIC
(ASTM D-1430, TYPE L, GRADE I, CLASS C)

Property	Temperature °C	(°F)	Typical Values	
Tensile:[b] Ultimate Strength, psi (MPa)	−252	(−420)	29,000	(200)
	−129	(−200)	21,800	(150)
	+ 25	(+ 75)	5,600	(39)
Elongation, %	−252	(−420)	5	
	−129	(−200)	9	
	+ 25	(+ 75)	140	
Modulus of Elasticity psi (MPa)	−252	(−420)	1,260 x 10^3	(8,680)
	−129	(−200)	800 x 10^3	(5,500)
	+ 25	(+ 75)	220 x 10^3	(1,520)
Notched Izod Impact Strength[c] ft-lbs/in. Notch (J/M)	−252	(−420)	7.6	(14)
	−196	(−320)	7.1	(13)
	+ 25	(+ 75)	7.6	(14)

[a] R.E. Mowers: Cryogenic Properties of PCTFE, Air Force Contract No. AF04 (611)-6354 (1962)

[b] Amorphous polymer, 40% crystallinity

[c] Amorphous polymer, 60% crystallinity

U3O8 in Argon(1bar,87K)

Particle Diameter Microns	Terminal Velocity cm/sec.	Particle Diameter Microns	Terminal Velocity cm/sec.	Particle Diameter Microns	Terminal Velocity cm/sec.	Particle Diameter Microns	Terminal Velocity cm/sec.
0.01	1.4443E-08	30	0.129986711	77	0.856323565	124	2.2207507406
0.02	5.7772E-08	31	0.138796921	78	0.878710166	125	2.2567137306
0.03	1.2999E-07	32	0.147895991	79	0.901385625	126	2.29296558
0.04	2.3109E-07	33	0.15728392	80	0.924349944	127	2.3295062887
0.05	3.6107E-07	34	0.166960709	81	0.947603122	128	2.3663358568
0.06	5.1995E-07	35	0.176926356	82	0.97114516	129	2.4034542843
0.07	7.0771E-07	36	0.187180864	83	0.994976057	130	2.4408615711
0.08	9.2435E-07	37	0.19772423	84	1.019095813	131	2.4785577172
0.09	1.1699E-06	38	0.208556456	85	1.043504429	132	2.5165427227
0.1	1.4443E-06	39	0.219677541	86	1.068201904	133	2.5548165876
0.2	5.7772E-06	40	0.231087486	87	1.093188239	134	2.5933793118
0.3	1.2999E-05	41	0.24278629	88	1.118463432	135	2.6322308954
0.4	2.3109E-05	42	0.254773953	89	1.144027485	136	2.6713713384
0.5	3.6107E-05	43	0.267050476	90	1.169880398	137	2.7108006407
0.6	5.1995E-05	44	0.279615858	91	1.19602217	138	2.7505188023
0.7	7.0771E-05	45	0.292470099	92	1.222452801	139	2.7905258233
0.8	9.2435E-05	46	0.3056132	93	1.249172292	140	2.8308217037
0.9	0.00011699	47	0.31904516	94	1.276180642	141	2.8714064434
1	0.00014443	48	0.33276598	95	1.303477851	142	2.9122800425
2	0.00057772	49	0.346775659	96	1.331063919	143	2.953442501
3	0.00129987	50	0.361074197	97	1.358938847	144	2.9948938188
4	0.00231087	51	0.375661594	98	1.387102635	145	3.0366339959
5	0.00361074	52	0.390537851	99	1.415555282	146	3.0786630325
6	0.00519947	53	0.405702968	100	1.444296788	147	3.1209809283
7	0.00707705	54	0.421156943	101	1.473327153	148	3.1635876836
8	0.0092435	55	0.436899778	102	1.502646378	149	3.2064832982
9	0.0116988	56	0.452931473	103	1.532254462	150	3.2496677721
10	0.01444297	57	0.469252026	104	1.562151405	151	3.2931411054
11	0.01747599	58	0.485861439	105	1.592337208	152	3.3369032981
12	0.02079787	59	0.502759712	106	1.622811871	153	3.3809543501
13	0.02440862	60	0.519946844	107	1.653575392	154	3.4252942615
14	0.02830822	61	0.537422835	108	1.684627773	155	3.4699230322
15	0.03249668	62	0.555187685	109	1.715969013	156	3.5148406623
16	0.036974	63	0.573241395	110	1.747599113	157	3.5600471518
17	0.04174018	64	0.591583964	111	1.779518072	158	3.6055425006
18	0.04679522	65	0.610215393	112	1.81172589	159	3.6513267087
19	0.05213911	66	0.629135681	113	1.844222568	160	3.6973997763
20	0.05777187	67	0.648344828	114	1.877008105	161	3.7437617032
21	0.06369349	68	0.667842835	115	1.910082502	162	3.7904124894
22	0.06990396	69	0.687629701	116	1.943445757	163	3.837352135
23	0.0764033	70	0.707705426	117	1.977097873	164	3.8845806399
24	0.08319149	71	0.728070011	118	2.011038847	165	3.9320980043
25	0.09026855	72	0.748723455	119	2.045268681	166	3.9799042279
26	0.09763446	73	0.769665758	120	2.079787374	167	4.027999311
27	0.10528924	74	0.790896921	121	2.114594927	168	4.0763832533
28	0.11323287	75	0.812416943	122	2.149691339	169	4.1250560551
29	0.12146536	76	0.834225825	123	2.18507661	170	4.1740177162

Table 1

Permeability

The permeability of "Kel-F 81" Plastic films with respect to air, water vapor and other gases is extremely low. The data in the following table apply to the amorphous form of the polymer.

TABLE IV
VAPOR PERMEABILITY CONSTANTS*
Values of P = 1 x 10^{10} cm^3/cm^2/mm/sec/cm Hg P

Temperature °C	°F	N_2	He	O_2	CO_2	H_2	H_2S	H_2O
0	32	-	-	0.07	0.35	3.2	-	-
25	77	0.05	21.7	0.40	1.4	9.8	-	1
50	122	0.30	-	1.40	2.4	24	0.35	10
75	167	0.91	-	5.70	15	-	2.0	28
100	212	-	-	-	-	-	-	100

*The true permeability constant (P) of "Kel-F 81" Plastic is expressed in cubic centimeters of gas under normal conditions traversing 1 cm^2 of surface, 1 mm thick, per sec., per cm of mercury pressure.

MOISTURE RESISTANCE

"Kel-F 81" Plastic is an unusual thermoplastic material because of its near zero moisture absorption. Because of its non-wettability and near zero water absorption, "Kel-F 81" Plastic polymer retains its dimensional stability and excellent electrical properties in high humidity or tropic conditions.

Water Absorption (per ASTM D570)

	Weight Change	Volume Change
21 days/25°C (77°F)	0%	0%

RADIATION RESISTANCE

"Kel-F 81" Plastic has the ability to perform satisfactorily while absorbing large doses of gamma radiation. "Kel-F 81" Plastic will suffer only minimal physical property loss at a dosage level of 18 megarads. This behavior in amorphous material is shown below.

TABLE V

	16 M. Rads*	24 M. Rads*
Tensile strength, % retained	70	60
Elongation, % retained	80	80
Yield strength, % retained	70	110
Modulus of elasticity, % retained	115	110

*Dose rate of 0.07 megarad/hr in air at 25°C.

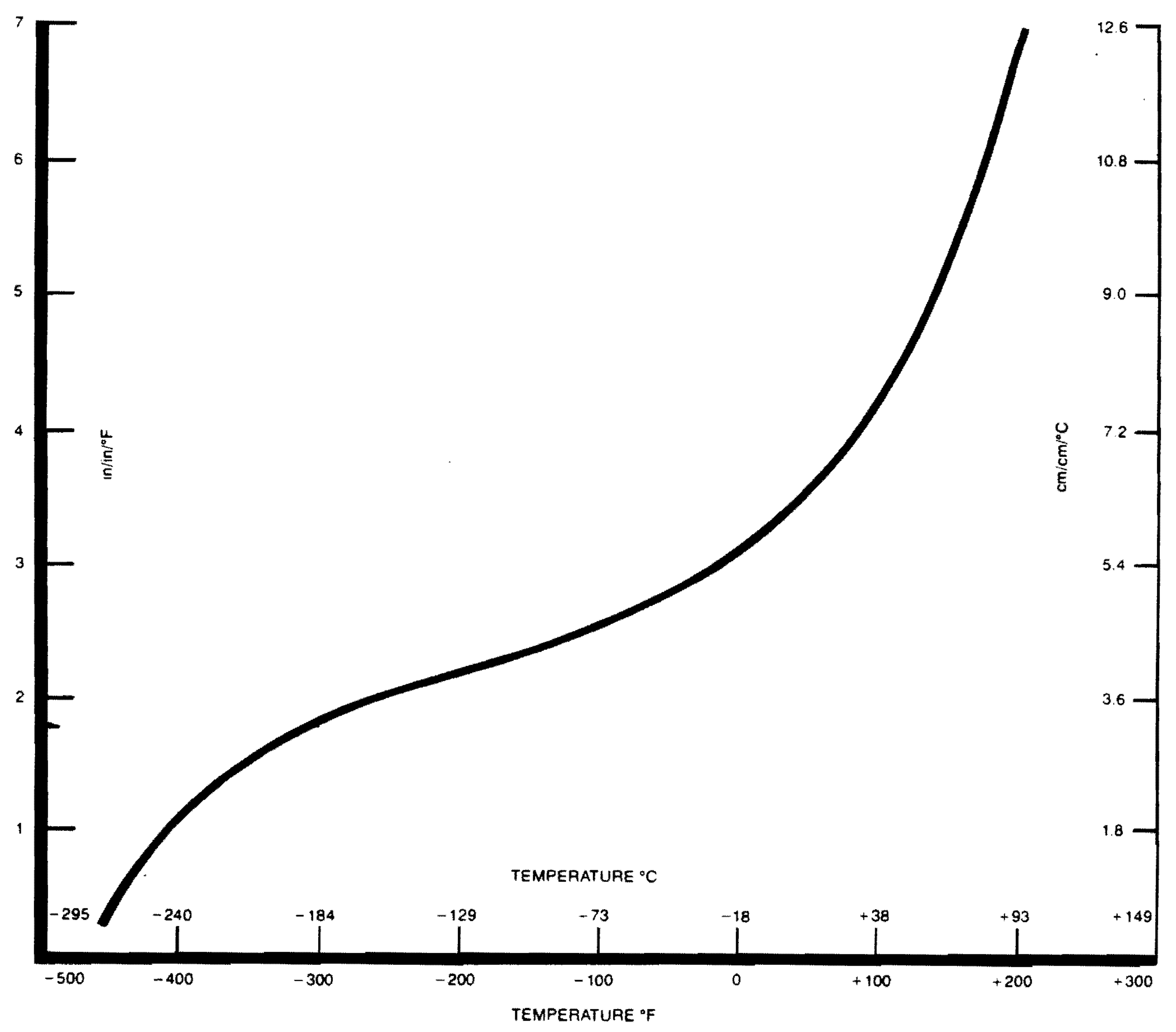
Coefficient of Linear Thermal Expansion

Because there is an increase in molecular vibration with an increase in temperature, most solids expand when heated and contract when cooled. This volume change—expansion or contraction—affects all dimensions of the solid. The Coefficient of Linear Thermal Expansion is the increase in length per unit length per degree temperature change as shown in Figure II.

The extent to which the solid will expand depends not only on the temperature change, but also on the attracting forces between the network of molecules and their spatial arrangement. Thus, the coefficient differs from material to material, such as the change from plastic to glass to metals.

For "Kel-F" Plastic the coefficient of linear thermal expansion, shown in Figure II, has three areas of change as a function of temperature. The information

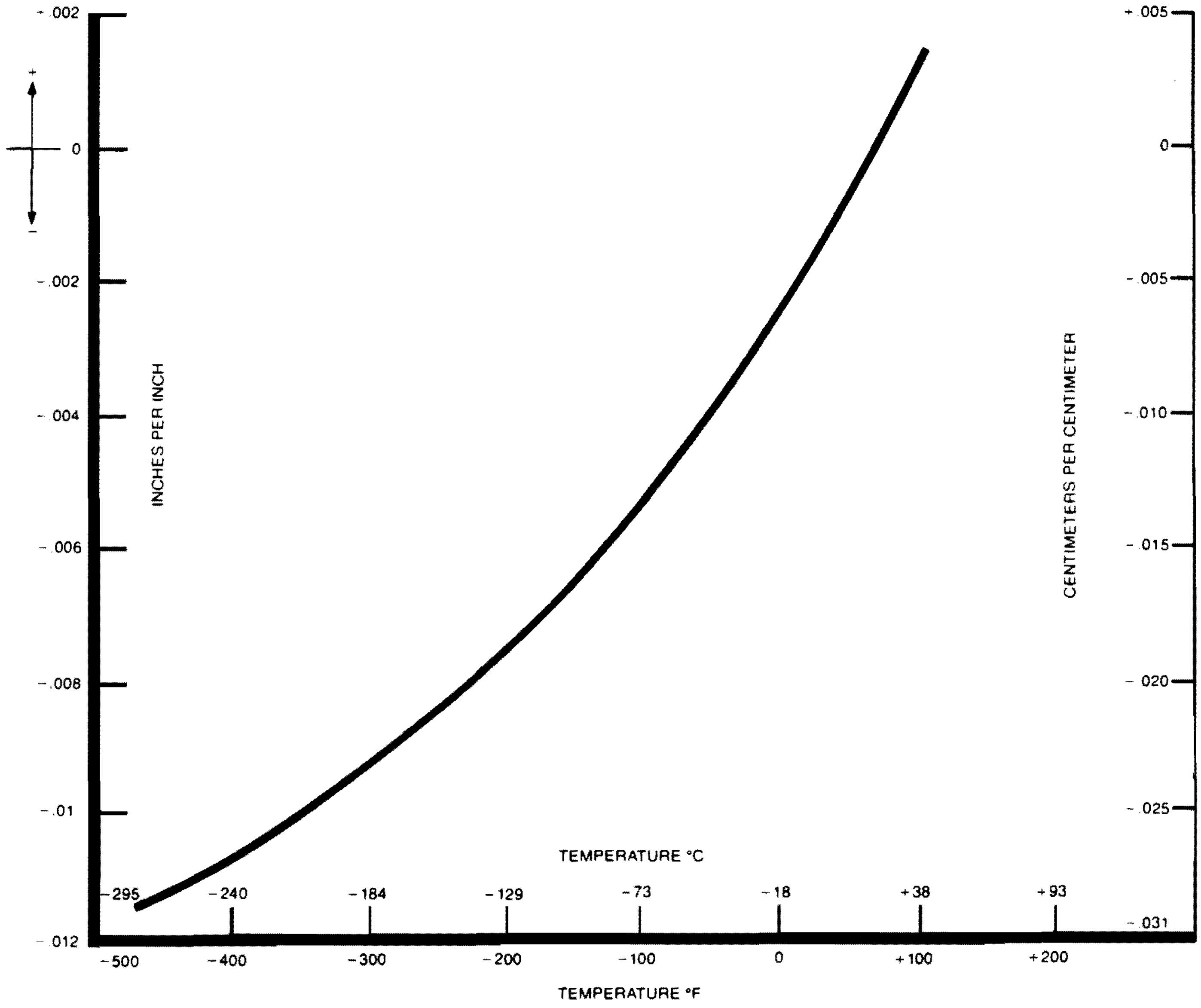
FIGURE II
COEFFICIENT OF LINEAR THERMAL EXPANSION VS TEMPERATURE



presented in this graph permits the selection of the correct metal-to-plastic combination to perform the desired end use function over the use temperature range suitable for "Kel-F" Plastic. In general, the coefficient of linear thermal expansion decreases as the temperature decreases. The rise in the region from 21 to 60°C (125 to 140°F) is due to the glass transition of this polymer, while the drop to zero at absolute zero is due to the cessation of molecular vibration.

The Coefficient of Linear Thermal Expansion, Figure III, depicts a "rate of expansion" change. The Thermal Linear Expansion graph represents the actual accumulative change in length. The vertical coordinate presents this length change in inches and represents a contraction as the specimen was cooled from 21°C (70°F) to the temperature of liquid hydrogen. Thus the numbers are preceded with a minus sign.

FIGURE III
THERMAL LINEAR EXPANSION
OF "KEL-F 81" PLASTIC VS TEMPERATURE



Chemical Resistance

"Kel-F 81" Plastic possesses exceptional chemical resistance due to its molecular structure. The high energy of the carbon-fluorine bond (110 kcal/mol) gives "Kel-F 81" Plastic excellent resistance to all inorganic corrosive liquids, including oxidizing acids. "Kel-F 81" Plastic is also resistant to most organic solvents excepting some highly halogenated and aromatic materials. While such materials will cause "Kel-F 81" Plastic to swell somewhat, in many cases this does not preclude its use with these materials.

Examples of the chemical resistance of "Kel-F 81" Plastic are shown in Table VI. All specimens were prepared for immersion from sheet molded into a highly amorphous form. More crystalline forms of the plastic will have substantially more chemical resistance than the amorphous form. Therefore, the swell values in Table VI may be considered *maximum* values for "Kel-F 81" Plastic. "Kel-F 81" Plastic in a crystalline form will show improved chemical resistance.

TABLE VI

Material	**Temp. °C.**	**Immersion Duration Days**	**Weight % Change**
Acetic acid, 5%	25	7	0.0
Acetic acid, 50%	175	7	0.1
Acetic acid	25	7	0.0
Acetic acid	118 B.P.	11	2.7
Acetic acid, glacial	70	7	0.2
Acetic acid, glacial	80	7	1.5
Acetic acid, glacial	175	7	2.5
Acetic anhydride	25	7	0.0
Acetic anhydride	70	7	0.1
Acetic anhydride	139 B.P.	1	3.6
Acetone	25	7	0 1
Acetone	56	1	1.0
Acetophenone	25	7	0.0
Acetyl chloride	25	7	0.1
Aluminum chloride*	175	7	0.0
Allyl chloride	25	7	0.2
Ammonia anhydrous	25	7	0.0
Ammonium chloride*	175	7	0.1
Ammonium hydroxide, 10%	25	7	0.0
Ammonium hydroxide, 28%	90	7	0.3
Ammonium hydroxide, 28%	175	7	0.6
Ammonium persulfate*	175	7	0.0
Ammonium sulfate*	175	7	0.1
Amyl acetate	25	7	0.0
Amyl acetate	70	7	0.9
Amyl acid phosphate	25	7	0.0
Aniline	25	7	0.0
Aniline	70	7	0.0
Antimony pentachloride	25	7	0.0
Aqua Regia	25	7	0.0
Aqua Regia	B.P.	7	0.3
Aroclor 1242	25	7	0.0
Aroclor 1248	25	7	0.0
Aroclor 1254	25	7	0.0
Arsenic acid	175	7	−0.1
Benzaldehyde	25	7	0.0
Benzene	25	7	0.2
Benzene	81 B.P.	1	6.6
Benzene	90	7	7.0
Benzene	135	7	107.0
Benzoic acid*	90	7	0.1
Benzonitrile	25	7	0.0
Benzyl alcohol	25	7	0.0
Benzoyl chloride	25	7	0.0
Bleaching Lye	25	30	0.0
Bromine	25	7	0.0
Bromobenzene	25	7	0.0
Bromobenzene	70	7	1.9
Butyl acetate	25	7	0.3
Butyl acetate	80	7	5.1
Butyl acetate	90	7	5.8
Butyl acetate	125 B.P.	1	6.7
Butyl acetate	135	7	6.5
Butyl alcohol	25	7	0.0
Butyl alcohol	70	7	0.0
Butyl alcohol	117 B.P.	1	0.6
N-Butyl ether	25	7	0 0
N-Butyl sebacate	25	7	0.0
Calcium chloride*	25	7	0.0
Calcium chloride*	80	7	0.0
Calcium chloride*	175	7	3.9
Carbitol acetate	25	7	0.0
Carbon disulfide	25	7	0.1
Carbon disulfide	25	30	0.5
Carbon tetrachloride	25	7	0.4
Carbon tetrachloride	25	60	0.9
Carbon tetrachloride	70	7	9.7
Carbon tetrachloride	90	7	18.0
Carbon tetrachloride	135	7	600.0
Cellosolve acetate	25	7	0.0
Chlorine gas	25	60	0.0
Chlorine liquid	−40	2 hours	0.0
Chlorine liquid	25	6	12.3
Chlorine liquid	50	6	9.0
Chloroacetic acid	140	7	1.7
Chlorobenzene	132 B.P.	1	21.8
Chloroform	90	7	8.5
1-Chloro-1-nitropropane	25	7	0.0
2-Chloropropane	25	7	0.3
Chlorosulfonic acid	25	30	0.0
Chlorosulfonic acid	140	7	0.2
0-Chlorotoluene	25	7	0.0
p-Chlorotoluene	25	7	0.0
Chlorotrifluoroethylene	8-25	7	9.1
Chlorotrifluoroethylene oil	25	7	0.0
(low mol. weight polymer)	110	50	19.0
Chromic acid cleaning solution	25	7	0.0
Chromic acid, 50%	B.P.	7	0.0
Chromic acid	80	7	−0.1
Chromic acid	175	7	0.0
Chromosulfuric acid*	140	7	slight swelling
Cresol	25	7	0.0
Cresol	140	7	2.0
Cupric chloride*	175	7	0.0
Cupric sulfate*	175	7	0.0
Cyclohexanone	25	7	0.0
Cyclohexanone	155 B.P.	1	10.5
Dibutyl phthalate	25	7	0.0
Dibutyl sebacate	25	7	0.0
1,2-Dichlorobutane	25	7	0.0
Dichloroethylene	25	7	0.0
Dichloroethylene	70	7	1.2
Dichloroethyl ether	25	7	0.0
1,2-Dichlorohexafluorocyclobutane	25	7	0.1
Dichloropropylene	25	7	0.0
2,4-Dichlorotoluene	25	7	0.0
3,4-Dichlorotoluene	25	7	0.0
Dicyclopentadiene	25	7	0.0
Diethylamine	25	7	1.9
Diethyl carbitol	25	7	0.1
Diethyl cellosolve	25	7	0.8
Diethylene triamine	25	7	0.0
Diisobutyl ketone	90	7	1 2
Dimethylhydrazine unsym.	25	7	0.1
Dimethyl formamide	25	7	0.0
Dioxane	25	7	0.0
Dioxane	90	7	5.7
Ethanol, 50%	25	7	
Ethanol, 95%	25	7	0 0
Ethanol, 95%	135	7	0.4
Ethanol, absolute	78 B.P.	1	0.1
Ethanol, absolute	80	7	0.2
Ethyl acetate	25	7	1.2
Ethyl acetate	25	30	5
Ethyl acetate	70	7	6.
Ethyl acetate	77 B.P.	1	5.9
Ethyl n-butyrate	25	7	0.5
Ethylene bromide	131 B.P.	1	6.6

*saturated solution @ 25°C

Material	Temp. °C.	Immersion Duration Days	Weight % Change
Ethylene chloride	25	7	0.0
Ethylene chloride	70	7	1.2
Ethylene glycol	175	7	0.0
Ethylene oxide	25	reported swelling	
Ethyl ether	25	7	3.8
Ethyl ether	35 B.P.	1	5.2
Ethyl formate	25	7	0.2
Ethyl propionate	25	7	1.0
Ferric chloride*	175	7	0.0
Ferrous chloride*	175	7	0.0
Ferrous sulfate*	175	7	0.0
Fluorine gas	85	14	0.0
Formaldehyde	135	7	0.7
Formic acid	25	7	0.0
Formic acid	25	12	0.0
Formic acid	101 B.P.	12	0.7
Formic acid, 87%	90	7	0.0
Formic acid, 87%	135	5 hours	2.9
Freon 11	25	7	6.4
Freon 12	25	7	3.0
Freon 22	25	7	2.1
Freon 113	25	7	1.2
Freon 113	90	7	22.4
Furan	25	7	2.4
Gallic acid	175	7	0.2
Gasoline (60-95°C.)	B.P.	1	0.5
Glycerol	175	7	−0.1
Halowax 1000	25	7	0.0
Hexane	80	7	4.3
Hexane	90	7	4.5
Heptane	25	7	0.0
Heptane	80	7	2.8
Heptane	90	7	1.8
Hollingshead H-2 (Hydrolube)	25	8	0.0
Hollingshead H-2 (Hydrolube)	80	8	0.0
Hydrobromic acid, 48%	B.P.	7	0.2
Hydrochloric acid, 10%	25	7	0.0
Hydrochloric acid, 20%	B.P.	7	0.0
Hydrochloric acid, 37%	175	7	0.3
Hydrofluoric acid, anhyd.	25	7	0.0
Hydrochloric acid, anhyd.	50	60	0.0
Hydrofluoric acid, 50%	25	7	0.0
Hydrogen peroxide, 3%	25	7	0.0
Hydrogen peroxide, 30%	25	7	0.0
Hydrogen peroxide, 30%	25	30	0.0
Hydrogen sulfide*	175	7	0.1
Isoamyl alcohol	135	7	1.4
Isopropyl ether	25	7	0.2
Machine oil	100	1	0.0
Mercuric chloride*	175	7	−5.6
Methallyl chloride	25	7	0.1
Methanol	25	7	0.0
Methyl acetate	25	7	1.0
Methylal	25	7	1.3
Methyl ether	25	7	0.2
Methyl ethyl ketone	25	7	0.2
Methyl ethyl ketone	90	7	4.6
Methyl formate	25	7	0.1
Methyl n-butyrate	25	7	0.8
Methyl proprionate	25	7	1.4
Mineral oil	25	7	0.0
Naphtha solvent	25	7	0.0
Nickel ammonium sulfate*	175	7	0.3
Nitric acid, 10%	25	7	0.0
Nitric acid, 60%	B.P.	7	0.0
Nitric acid, 30%	175	7	0.1
Nitric acid, 70%	25	7	0.0
Nitric acid, 70%	70	7	0.0
Nitric acid, fuming 85-95%	25	7	0.0
Nitric acid, white fuming	90	7	0.3
Nitrobenzene	25	7	0.0
Nitrobenzene	140	7	1.5
Nitrogen tetroxide	5	7	9.9
Nitromethane	25	7	0.0
Oleic acid	25	7	0.0
Oleum, 100%	25	2	0.1
Oxalic acid	175	7	−0.2
Ozone, 5% in oxygen	150	2	0.0
	(no molecular degradation)		
Pentachloroethane	25	7	0.0
Perchloric acid, 70%	25	30	0.0
Perchloric acid	25	14	−0.2
Perfluorotriethylamine	25	7	0.0
Phenol, 5%	70	7	0.0
Phosphoric acid, 30%	175	7	0.1
Phosphoric acid, 85%	175	7	0.0
Phosphoric acid	140	7	0.0
Piperidine	25	7	0.0
Potassium dichromate*	175	7	0.0
Potassium hydroxide, 10%	25	7	−0.2
Potassium hydroxide, 10%	80	7	0.1
Potassium hydroxide, 50%	B.P.	7	0.1
Potassium permanganate*	25	30	0.0
Potassium persulfate*	25	30	0.0
n-Propyl acetate	25	7	0.6
Propylene chloride	25	7	0.0
n-Propyl ether	25	7	0.3
n-Propyl formate	25	7	0.1
n-Propyl propionate	25	7	0.4
Pyridine	25	7	0.0
Pyridine	115	1	7.4
Pyrogallic acid*	175	7	0.1
Salicylic acid*	175	7	0.2
Santicizer B-16	25	7	0.0
Santicizer E-15	25	7	0.0
Santicizer M-17	25	7	0.0
Santicizer 8	25	7	0.0
Santolube 31	25	7	0.0
Silicone Oil DC-200	175	7	0.1
Silicone Oil DC-200	70	7	0.1
Silicone Oil DC-200	190	7	−0.3
Sodium bisulfite*	175	7	−0.7
Sodium borate*	175	7	0.2
Sodium carbonate, 2%	25	7	0.0
Sodium chloride, 10%	25	7	0.0
Sodium chloride*	175	7	0.0
Sodium hydroxide, 1%	25	7	0.0
Sodium hydroxide, 10%	25	7	0.0
Sodium hydroxide, 30%	175	7	−1.2
Sodium hydroxide, 50%	B.P.	7	0.1
Sodium phosphate*	175	7	0.0
Stannic chloride*	25	7	0.0
Stannic chloride*	175	7	0.1
Sulfur dioxide anhyd.	25	7	0.1
Sulfuric acid, 3%	25	7	0.0
Sulfuric acid, 30%	25	7	0.0
Sulfuric acid, 20% free SO_3	25	7	0.0
Sulfuric acid, 96%	70	7	0.0
Sulfuric acid, 50%	140	30	0.0
Sulfuric acid, 92%	140	30	0.0
Sulfuric acid, 30%	175	7	0.0
Sulfuric acid, 95%	175	7	0.0
Tetrachloroethane sym.	25	7	0.0
Tetrachloroethylene	25	7	0.8
Tetrahydrofurane	25	7	8.5
Tetrahydrofurane	64	1	8.2
Thionyl chloride	90	7	8.5
Titanium tetrachloride	90	7	2.6
Toluene	25	7	0.4
Toluene	110	7	5.0
Trichloroacetic acid	70	7	0.0
1,1,1-Trichloroethane	25	7	0.1
1,1,2-Trichloroethane	25	7	0.0
Trichloroethylene	25	7	2.3
Trichloroethylene	80	7	9.2
1,2,3-Trichloropropane	25	7	0.0
Tricresyl phosphate	25	7	0.0
Tricresyl phosphate	140	7	0.0
Triethylamine	25	7	0.2
Xylene	25	7	0.4
Xylene	90	7	6.5
Xylene	138	7	27.0
Zinc sulfate*	175	7	0.4

*saturated solution @ 25°C

Electrical Properties

Consistently high insulation resistance of 15,000 to 25,000 volts over extended time through thermal cycling and high humidity has qualified "Kel-F 81" Plastic for electrical component applications. "Kel-F 81" Plastic with a non-wettable surface and near zero moisture absorption, exhibits minimal tracking, corona formation and surface flash-over.

The presence of chlorine on the "Kel-F 81" Plastic polymer backbone introduces a dipole moment, which only affects electrical properties in the VHF and UHF range. However, the dielectric constant in this frequency range compares favorably with that of other fluorocarbon polymers (Refer to Figures IV and V). The dissipation factor varies with frequency and temperatures as shown in Figures VI and VII.

TABLE VII

Property	ASTM Method	Values
Dielectric strength short time (volts mil)	D-149	500
Arc resistance, seconds	D-495	360
Volume resistivity, ohm cm^2 cm 50% RHC 25°C (77°F)	D-257	1.2×10^{18}
Surface Resistivity, ohm 50% RHC 25°C (77°F)	D-257	10^{15}

FIGURE IV
DIELECTRIC CONSTANT AT 60 CYCLES PER SEC. VS. TEMPERATURE

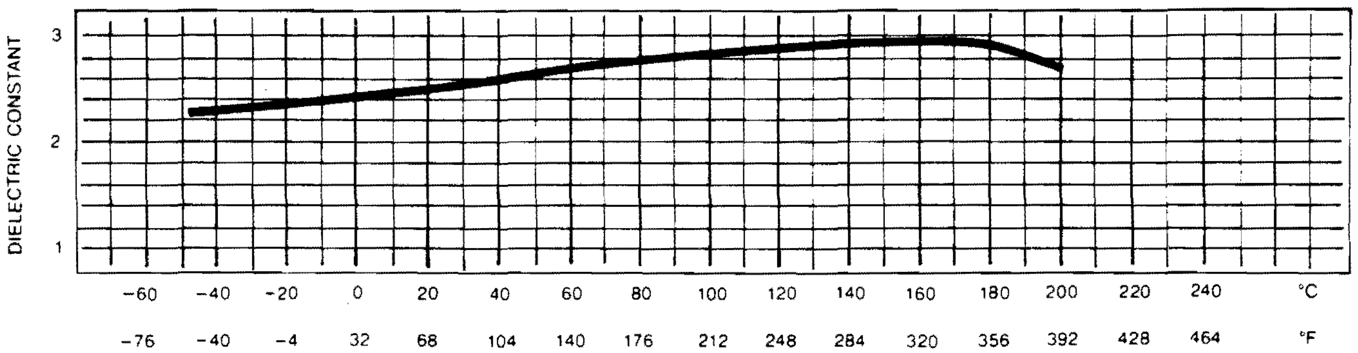


FIGURE V
DIELECTRIC CONSTANT VS. FREQUENCY @ 25°C (77°F)

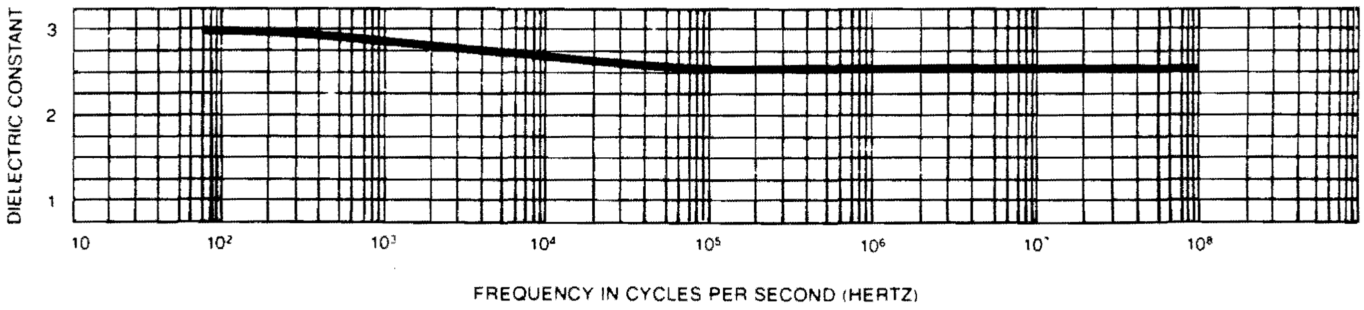


FIGURE VI
DISSIPATION FACTOR AT 60 CYCLES PER SEC.
VS. TEMPERATURE

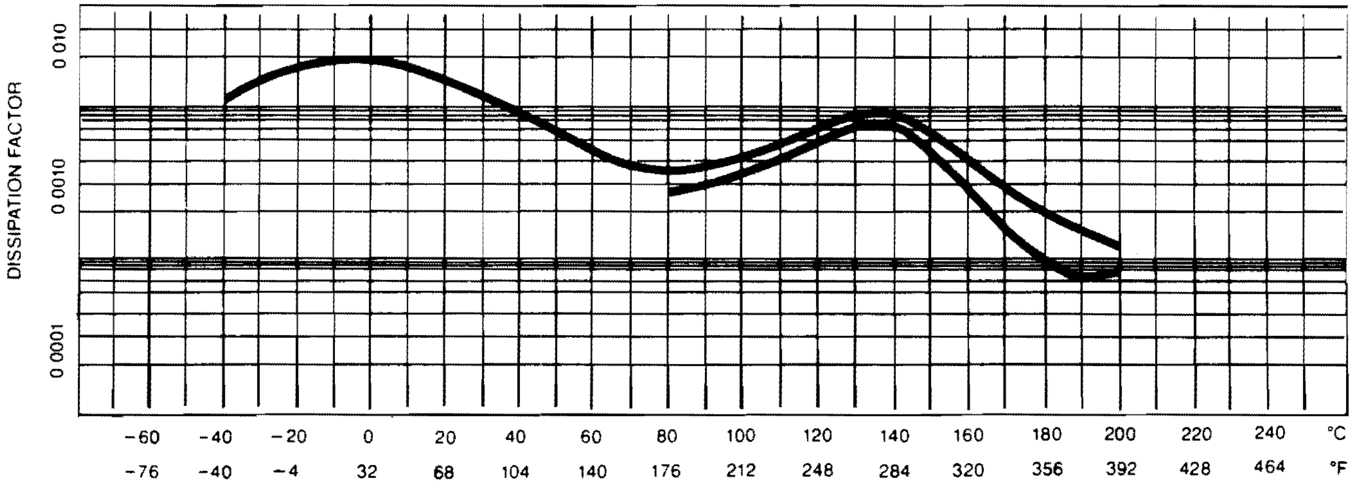
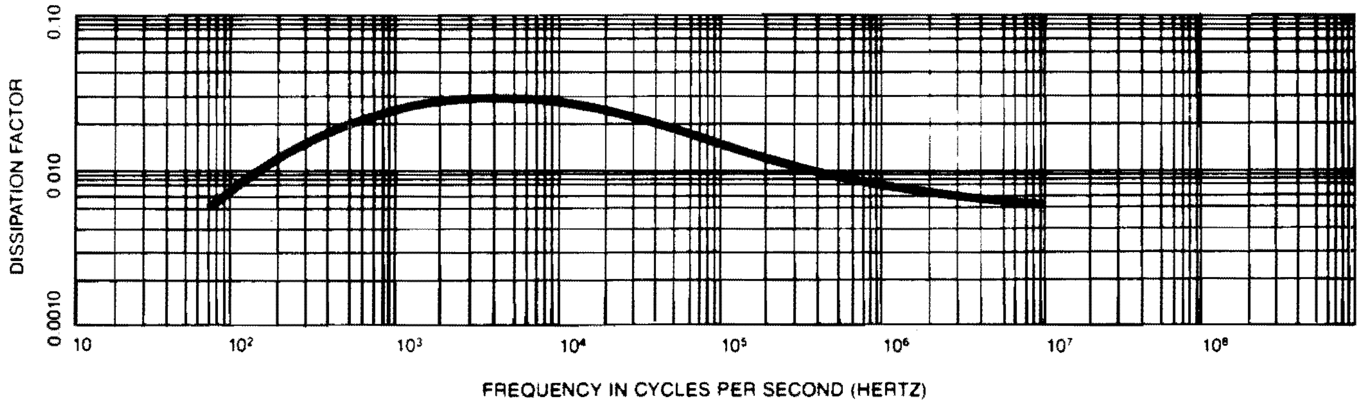


FIGURE VII
DISSIPATION FACTOR VS. FREQUENCY
@ 25°C (77°F)



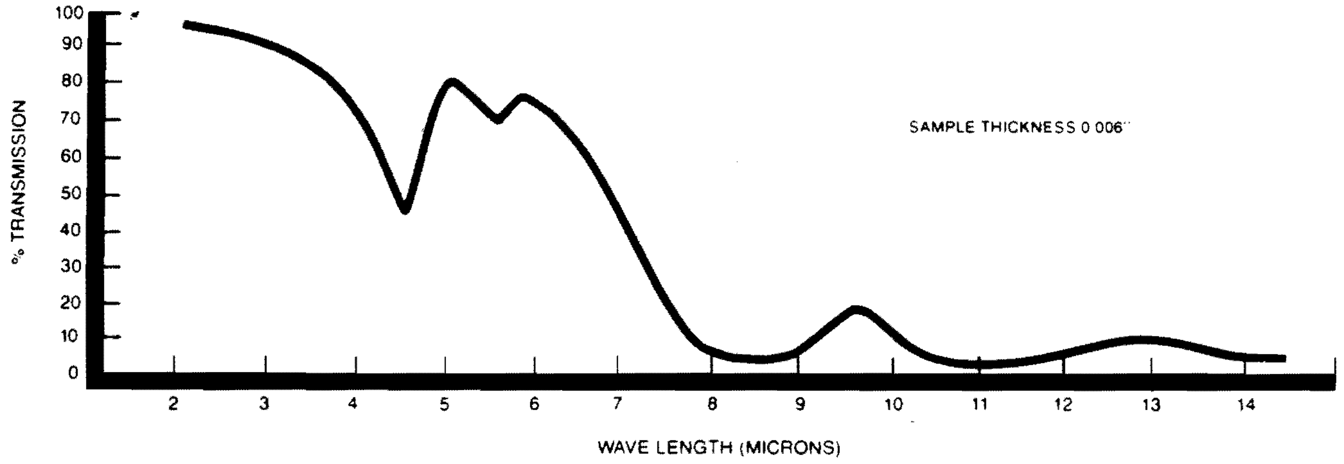
Optical Properties & Spectra

The inclusion of a chlorine atom on the "Kel-F 81" Plastic polymer backbone can be directly translated into benefits in the polymer optical properties. By controlling the thermal history ("quick-quenched") the amorphous plastic is less dense and more elastic, with greater optical clarity and toughness. The properties shown below coupled with chemical resistance, long-term weatherability and mechanical strength suggest the use of "Kel-F 81" Plastic polymer for a variety of optical applications including sight glass retainers, solar energy cell covers, and fiber optics connectors.

TABLE VIII

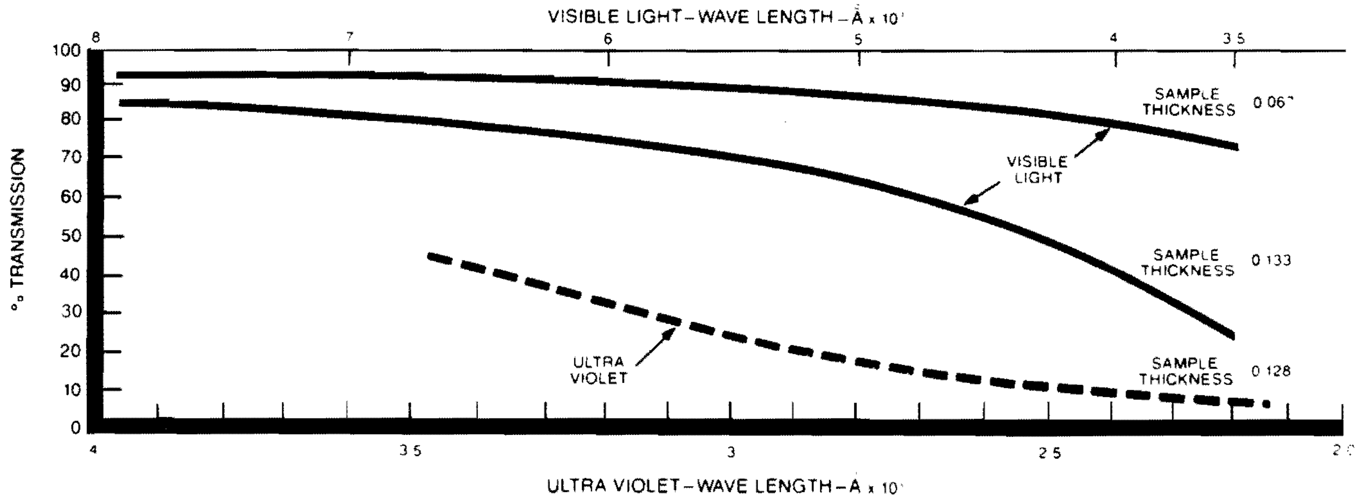
Property	ASTM Method	Value
Refractive Index, Amorphous N_D^{25}	D-542	1.435
Luminous Transmittance, Amorphous	D-1003	90%
Haze, Amorphous	D-1003	4%

FIGURE VIII
INFRARED SPECTRUM OF "KEL-F 81" PLASTIC



The transmission of "KEL-F 81" Plastic in the visible and ultra violet regions is shown below.

FIGURE IX
VISIBLE AND ULTRA VIOLET SPECTRUMS OF "KEL-F 81" PLASTIC



Dr. Gordon M. Kline, Technical Editor

PLASTICS[*]
TECHNICAL SECTION

MATERIALS • PROPERTIES • TESTING METHODS AND INSTRUMENTATION • STANDARDS • CHEMISTRY

Permeability of chlorotrifluoroethylene polymers

Kel-F 300
15 nov
Kel-F 81

By A. W. Myers[†], V. Tammela[†], V. Stannett[†], and M. Szwarc[†]

The permeability of a number of modifications of chlorotrifluoroethylene polymers to gases (N_2, O_2, and CO_2) and vapors (water and methanol) has been investigated. The effects of crystallinity, plasticization, and copolymerization with vinylidene fluoride are considered.

The transmission of a gas or vapor through a polymer takes place primarily by a diffusion controlled mechanism, wherein the gas dissolves in one surface of the film, passes through by an activated diffusion process, and evaporates from the opposite surface. The rate of permeation through the film per unit area, q, may be expressed quantitatively by Fick's first law:

$$q = -D \frac{dc}{dx} \qquad \text{Eq. 1}$$

where the proportionality factor D is the diffusion constant and dc/dx is the concentration gradient across the polymer film. Under steady state conditions, and assuming that D is not concentration dependent, Equation 1 can be integrated and one obtains:

$$q = \frac{D(c_1 - c_2)}{t} \qquad \text{Eq. 2}$$

where t is film thickness and c_1 and c_2 are the concentrations at the high and low pressure surfaces, respectively. Applying Henry's law, one obtains:

$$q = \frac{DS(p_1 - p_2)}{t} \qquad \text{Eq. 3}$$

where S is the solubility of a gas in cc./cc. of polymer at a pressure of 1 cm. Hg with the volume of gas corrected to S.T.P., and p_1 and p_2 are the pressures of the gas at the high pressure and low pressure surfaces, respectively.

P, the permeability constant, is defined as cc. of gas at S.T.P. permeating per second through a film of 1 cm.^{2} area and 1 mm. thickness under a pressure difference of 1 cm. Hg and may be expressed as:

$$P = DS = \frac{tq}{p_1 - p_2} \qquad \text{Eq. 4}$$

P is independent of thickness and pressure for gases. For vapors, it may increase with pressure. The temperature dependence of P may be expressed as an Arrhenius relationship, as follows:

$$P = P_0 \exp. (-E_p/RT) \qquad \text{Eq. 5}$$

where E_p is the activation energy and P_0 is the pre-exponential factor associated with the overall permeation process.

Method

The experimental method used to measure P was similar to that described by Barrer (1).[1] The

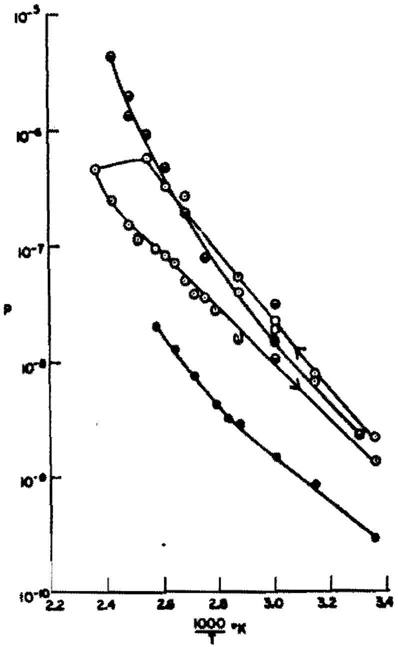


FIG. 1: Permeability of Kel-F films to water vapor (about 20 mm. Hg pressure). Legend: ⊖—Kel-F 300 P25 (plasticized homopolymer); ⊙—Kel-F 500 (copolymer containing 3% vinylidene fluoride); ●—Kel-F 300 (quenched low-crystallinity homopolymer).

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[1] Numbers in parentheses link to references at the end of article, p. 211.

[*] Reg. U. S. Pat. Off.

[†] Chemistry Dept., State University College of Forestry at Syracuse University, Syracuse, N. Y.

Table I: Permeability of amorphous and crystalline polychlorotrifluoroethylene[a]

Gas	Temp., °C.	P, 10^{-10} cc./mm./cm.²/sec./cm. Hg Amorphous	Crystalline
N_2	25	0.05	0.03
	40	0.20	0.09
	50	0.35	0.12
	60	0.63	—
	75	—	0.51
O_2	0	0.07	—
	25	0.40	—
	40	0.92	0.25
	50	1.44	0.43
	60	—	0.68
	75	5.74	—
	80	—	1.85
CO_2	40	2.11	0.48
	50	—	0.89
	60	6.12	1.37
	80	18.5	3.67

[a] Unplasticized Kel-F 300 films of about 30 and 80% crystallinity, respectively.

Table II: Permeability of extruded films versus crystalline and amorphous films of unplasticized Kel-F 300

Film	P, 10^{-10} cc./mm./cm.²/sec./cm. Hg N_2 (50° C.)	N_2 (75° C.)	CO_2 (75° C.)
Extruded 2 mil	0.18	0.86	5.8
Extruded 5 mil	—	0.94	6.15
Molded and annealed (~80% crystalline)	0.12	0.51	3.0
Molded and quenched (30% crystalline)	0.35	1.8	14.5

Table III: Temperature dependence of the permeability constant for chlorotrifluoroethylene polymer and copolymers

Elastomers

Film[a]	Gas	P_o	E_p kcal./mole
Amorphous polymer	N_2	0.20	14.3
" "	O_2	0.0058	11.2
" "	CO_2	0.033	11.8
Crystalline polymer	N_2	0.0014	11.9
" "	O_2	0.00093	10.9
" "	CO_2	0.0027	11.1
Copolymer X-500 (3)	N_2	0.085	13.6
" "	O_2	0.40	13.8
" "	CO_2	0.31	13.1
" X-800 (25)	N_2	6.75	15.5
" "	CO_2	24.1	15.1
" X-3700 (70)	N_2	1.55	13.5
" "	O_2	1.25	12.8
" "	CO_2	1.30	11.9
" X-5500 (50)	O_2	3.7	13.1
" "	CO_2	60	13.9

[a] The figures in parentheses are the percent of vinylidene fluoride in the copolymer.

apparatus consists essentially of three parts: 1) a source of high vacuum, 2) a constant pressure source of gas or vapor under study, and 3) a diffusion cell containing the polymer film.

The film is first placed into the diffusion cell and sealed with a mercury seal. The cell is then attached to the high vacuum apparatus and the whole diffusion cell and adjoining parts are thoroughly degassed. Gas or vapor at a known pressure is then admitted to one side of the film and the gas permeates at essentially a constant pressure through the film into a receiving section where the increase in pressure with time may be recorded by a McLeod gage. When the slope of the pressure-versus-time curve remains a constant, the steady state has been reached and P may be readily calculated. Details of the equipment have been described in a number of recent publications (2,3), which should be referred to for further information.

Effect of crystallinity

Chlorotrifluoroethylene polymers may be modified in various ways, including varying the degree of crystallinity, plasticization, and copolymerization.

The effect of crystallinity was studied by preparing two samples by compression molding. One sample was quenched and was mainly amorphous; the other was carefully annealed to give a more highly crystalline film. The crystallinity of the quenched specimen was estimated to be about 30% and that of the annealed to be 80 percent. The permeability values were measured and are tabulated in Table I, above, where it can be readily seen that a sharp reduction in the permeability constant results from crystallization of the material.

This same behavior has been found with other polymers, such as polyethylene. Undoubtedly, the main contribution to the gas permeation comes from the amorphous regions, with the crystalline regions generally being impermeable. Thus, increasing the crystallinity from about 30 to 80% reduces the permeability constant to almost one-third of the original value, or approxi-

Table IV: Effect of plasticizer on permeability of polychlorotrifluoroethylene

Gas	Temp., °C.	P, 10^{-10} cc./mm./cm.²/sec./cm. Hg Unplasticized	Plasticized[a]
N_2	50	0.18	5.5
	75	0.86	25.7
O_2	0	0.04	1.1
	30	0.52	5.6
	60	2.90	28.0
CO_2	50	3.7	75.0

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[a] Plasticizer was low molecular weight polychlorotrifluoroethylene.

Table V: Permeability of chlorotrifluoroethylene copolymers[a]

		P, 10^{-10} cc./mm./cm.²/sec./cm. Hg			
Gas	Temp., °C.	X-500 (3)	X-800 (25)	X-5500 (50)	X-3700 (70)
N_2	0	—	0.029	—	0.25
	12.5	—	—	—	—
	25	0.11	0.30	—	1.62
	50	0.52	2.34	—	12.7
	75	2.92	13.1	—	56.2
O_2	0	—	—	1.11	0.62
	12.5	—	—	3.74	—
	25	0.20	—	9.34	5.46
	40	1.18	—	—	—
	50	2.05	—	50.5	34.1
	75	7.73	—	—	111
CO_2	0	0.10	0.19	—	2.73
	25	0.80	1.83	39.8	27.2
	50	5.25	15.2	248	137
	75	19.5	70.8	—	440

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[a] The figures in parentheses are the percent of vinylidene fluoride in the copolymer.

Table VI: Permeability of polychlorotrifluoroethylene to water and methanol vapors (amorphous unplasticized Kel-F 300)

Vapor	Temp., °C.	P, 10^{-8} cc./mm./cm.²/sec./cm. Hg
Water (~20.5 mm. pressure)	25	0.29
	45	0.85
	60	1.5
	75	2.8
	80	3.3
	85	4.3
	95	7.6
	105	13.0
	115	20.0
Methanol (~50 mm. pressure)	60	0.81
	67	1.4
	75	2.4
	85	5.1
	95	8.7
	105	9.1
	115	9.4
Permeability recorded after a run at 115° C.	67	0.62

mately in line with the decrease in the amorphous content. A more complete discussion on the effect of crystallinity on gas and vapor permeability is given in References 4 and 5.

The degree of crystallinity and the gas permeability of commercial films will depend on the heat treatment accorded them in processing. Values for two typical extruded films are shown in Table II, p. 140, compared with the corresponding values for the quenched and annealed films. It can be seen that intermediate values are obtained but closer to the values obtained with the more highly crystalline film.

The temperature dependence of the permeability constant for N_2, O_2, and CO_2 transmission through polychlorotrifluoroethylene polymers is shown in Table III, p. 140. There is little change in solubility with "real" gases at the temperature range investigated; the main contribution to the activation energy of permeation is the activation energy of diffusion. The activation energy for diffusion may be thought of as the energy needed to separate the polymer molecules sufficiently to allow the gas molecule to move between and is principally a result of thermal motion of the polymer molecules. Thus, activation energies for diffusion can be classed according to the molecular diameter (6). It can be seen from experimental data that oxygen (molecular diameter 2.98 A.) has a lower energy of permeation than nitrogen (molecular diameter 3.18 A.) for the same material. The activation energy for diffusion also increases with, for example, increasing cohesive energy of the polymer and, therefore, diffusion rates will be dependent on the polymer. The high polarity and cohesive energy of Kel-F films results in high energies of permeation. A further discussion of the temperature dependence is given in the copolymers section.

Effect of plasticization

The effect of plasticizer on the permeability constant of polychlorotrifluoroethylene polymers is shown in Table IV, above, where it can be seen that the plasticized film is many times more permeable than the unplasticized. The permeabilities of plasticized and unplasticized Kel-F[2] films to hydrogen and carbon dioxide at a number of temperatures have been reported by Brubaker and Kammermeyer (7). Similar large increases in the permeability constants with plasticization were found. In an earlier publication (8), the permeability constants of Trithene[3] B for various gases and temperatures were reported. We want to clearly point out that those results were for the plasticized film and thus gave understandably higher results compared to the unplasticized film as shown in Table IV. Undoubtedly, the increase in permeability for the plasticized film is mainly due to an increase in the diffusion constant. The solubility of permanent gases is not believed to be greatly affected by plasticization. The plasticizer reduces the cohesive forces giving rise to easier penetration by the diffusing species. This is reflected in both the larger diffusion constant and the lower energy of activation for the overall permeation process. A further discussion on the effects of plasticizers on gas and vapor permeability may be found in References 2 and 9.

Copolymerization

A series of chlorotrifluoroethylene copolymer films were prepared and their permeabilities measured. These results and the approximate composition of the copolymers are shown in Table V, p. 142.

All the copolymers have higher gas permeability constants than even the amorphous homopolymer. This would be anticipated, since copolymerization provides internal plasticization allowing easier diffusion of the penetrant gas or vapor. Furthermore, the solubility will increase as the crystallinity decreases due to copolymerization. As the percentage of the copolymer is increased beyond about 50%, the permeability constant begins to decrease again. This type of behavior is characteristic of a range of copolymers, although complicated in this case since both monomers alone lead to crystalline polymers.

The temperature dependencies of the various copolymers, together with those found for amorphous and crystalline polychlorotrifluoroethylene, are given for the permanent gases in Table III. No clear pattern in the values can be seen in the activation energies, although somewhat larger values are obtained with the copolymer. It should be pointed out, however, that with highly impermeable films such as these, the experimental error is enough to mask the expected differences in the activation energies. Differences in the pre-exponential factors are more striking and reflect, to some extent, the openness of the polymer structure. Thus, P_0 values for the amorphous homopolymer are more than 100 times those of the crystalline sample. Increasing copolymerization first increases P_0 and then begins to decrease it, presumably towards the value of the homopolymer of the co-monomer.

Water and methanol permeabilities

The permeabilities of a number of Kel-F polymers to water vapor are shown in Table VI, p. 142. The method used for measuring these values has been (To page 211)

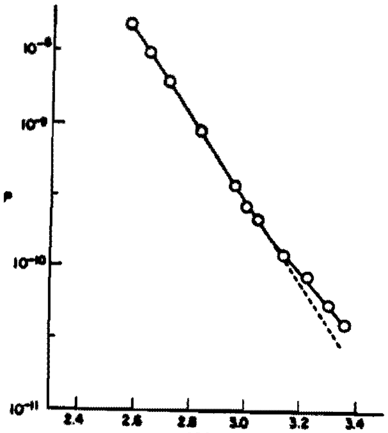

FIG. 2: Permeability of Kel-F 300 (quenched low-crystallinity homopolymer) to carbon dioxide.

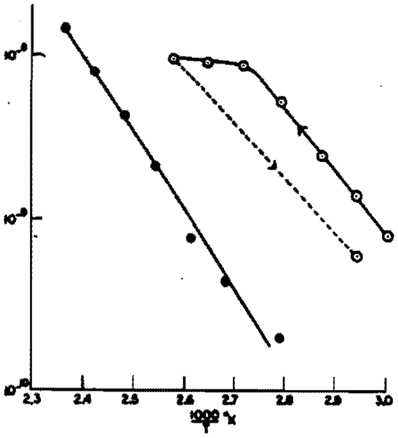

FIG. 3: Permeability of Kel-F films to methanol. Legend: ⊙—Kel-F 300, quenched (50 mm. Hg methanol pressure); ●—Kel-F 500 (100 mm. Hg methanol pressure).

Table VII: Permeability of plastics films to water vapor (25° C.) and to gases (30° C.)

Material	P, 10^{-10} cc./cm.²/sec./mm./cm. Hg H_2O	 N_2	 O_2	 CO_2
Kel-F 300 (unplasticized)	<2.9[a]	0.03	0.10	0.72
Saran	10.0	0.0094	0.053	0.29
Polyethylene terephthalate (Mylar A)	1,300	0.05	0.22	1.53
Rubber hydrochloride, unplasticized (Pliofilm NO)	260	0.08	0.30	1.7
Nylon 6	1,770	0.10	0.38	1.6
Polyvinyl chloride (unplasticized)	1,560	0.4	1.2	10
Polyethylene (0.953 density)	160	3.3	11.0	43
Butyl rubber	——	3.1	13.0	52
Cellulose acetate (plasticized)	68,000	2.8	7.0	68
Polyethylene (0.938 density)	298	6.6	21	74
Polystyrene	12,000	2.9	11	88
Rubber hydrochloride, plasticized (Pliofilm P-4)	——	6.2	24	182
Polyethylene (0.922 density)	1,000	22	69	280

[a] On quenched low-crystallinity sample, lower values are obtained with annealed films.

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[2] Trademark of Minnesota Mining & Mfg. Co.

[3] Trademark of Visking Co., div. of Union Carbide Corp.

Permeability

(From pp. 139–145)

described in a previous paper (3). A constant pressure of vapor was maintained over the film by using a supply of degassed liquid water or methanol at the appropriate temperature. The vapor pressure was measured with a Zimmerli gage. Figure 1, p. 139, shows the effect of temperature on the water vapor permeability of the homopolymer, plasticized homopolymer, and the Kel-F 500 copolymer. It can be seen that after keeping the latter at a high temperature, there was a marked drop in the permeability constant, which was presumably due to further crystallization.

Furthermore, there is an inflection in the Arrhenius plots, similar to those found with other polymers (3, 6), when passing through the glass temperature and the inflection point can be similarly interpreted. This would lead to a glass temperature of about 75° C. for the "amorphous" homopolymer. Attempts were made to determine the glass temperature with other gases.

Figure 2, p. 145, demonstrates a similar plot with carbon dioxide. Here there is only a slight inflection, indicating a glass temperature of about 50° C. This is closer to the value of 52° C. found by Hoffman and Weeks using the specific volume method (10). The higher values found with water vapor could be due to the larger size of diffusing species; this could indicate the movement of water vapor through the hydrophobic film in the form of clusters, as suggested by Rouse (11).

The Kel-F 500 films are internally plasticized by a small degree of copolymerization and the Kel-F 300P25 films are plasticized externally by the addition of low molecular weight Kel-F as plasticizer. Both of these treatments, as would be anticipated from the gas data, lead to a marked increase in the water vapor permeability constants.

Data for the methanol permeabilities of Kel-F 300 homopolymer and Kel-F 500 copolymer are given in Fig. 3, p. 145. The phenomenon of irreversible lowering of the permeability at higher temperatures is again shown and is presumably due to further crystallization of the polymer as it is "annealed" during the measurements at higher temperature.

The methanol permeabilities are much higher for the "amorphous" homopolymer than for the Kel-F 500 copolymer. This may be due to the low crystallinity leading to a greater solubility in the former case. With organic polymers solubility plays a much more important role than with the gases in determining the size of the permeability constant.

Typical permeability values for polychlorofluoroethylene films and other polymer films to the common gases and to water vapor are given in Table VII, p. 145. It can be seen from these data that Kel-F films provide exceptional gas and water vapor resistance.

We would like to thank the M. W. Kellogg Co. for sponsoring this study and providing the samples of the various films and the data concerning them. The Kel-F Div. of the company has since been absorbed by the Minnesota Mining & Mfg. Co.

POLYCHLOROTRIFLUOROETHYLENE

Since disclosure in 1937 (1), polychlorotrifluoroethylene [*9002-83-9*] (PCTFE) has been developed to serve many demanding industrial and military needs. The preparation and characterization of this high molecular weight thermoplastic was studied intensively during the Manhattan Project (2). The unique combination of chemical inertness, radiation resistance, low vapor permeability, and thermal stability of this polymer filled an immediate need for a construction material in the gaseous diffusion process for the separation of uranium isotopes (see Diffusion separation methods).

Properties

PCTFE properties are governed by the molecular weight and percent crystallinity. Since PCTFE has limited solubility in selected solvents, correlations of the number average molecular weight with the zero-strength time (ZST) (3–4) have been made. The high molecular weight thermoplastic has a melt temperature (T_m) of 211–216°C and glass transition temperature (T_g) of 71–99°C, and is thermally stable up to 250°C. Because of the loss of strength above the T_m, the useful range of operation is from −240 to 205°C. An increase of upper temperature service can be realized through use of commercially available filled versions of the plastic.

The specific gravity of PCTFE for the amorphous and crystalline polymers has been calculated to be 2.075 and 2.185, respectively (5–9). In practice, the final PCTFE parts have some degree of crystallinity ranging from about 45% (specific gravity of 2.10) for quick-quenched to 65% (specific gravity of 2.13) for slow-cooled parts. The higher crystalline forms are less transparent, with higher tensile modulus, lower elongation, and have more resistance to the penetration of liquids and vapors. The less crystalline form is optically clear, tough, and ductile.

The combination of mechanical properties that makes PCTFE a unique engineering thermoplastic is shown in Table 1. In addition, PCTFE has a low coefficient of thermal expansion. A general outline of cryogenic mechanical properties is shown in Table 2.

The high fluorine content contributes to resistance to attack by essentially all

Table 1. Mechanical Properties of Polychlorotrifluoroethylene

Property	Value
tensile strength, MPa[a]	32–39
compressive strength, MPa[a]	380
modulus of elasticity, MPa[a]	1423
hardness, Shore D	76
deformation under load, 0.3% at 24 h, MPa[a]	6.86
heat deflection temperature, °C at 1.78 MPa[a]	126

[a] To convert MPa to psi, multiply by 145.

Table 2. Typical Cryogenic Mechanical Properties of Polychlorotrifluoroethylene (ASTM D 1430 Type I, Grade I, Class C)

Property	PCTFE % crystallinity	Temp, °C	Value[a]
tensile: ultimate strength, MPa[b]	40	25	38.6
		−129	150
		−252	200
elongation, %	40	25	140
		−129	9
		−252	5
modulus of elasticity, MPa[b]	40	25	1521
		−129	5494
		−252	8682
impact strength notched Izod, J/m[c]	60	25	13.7
		−196	12.8
		−252	13.7

[a] Ref. 10.
[b] To convert MPa to psi, multiply by 145.
[c] To convert J/m to ft·lbf/in., divide by 53.38 (see ASTM D 256).

chemicals and oxidizing agents; however, PCTFE does swell slightly in halogenated compounds, ethers, esters, and aromatic solvents. PCTFE has the lowest water–vapor transmission rate of any plastic (11), is highly impermeable to gaseous vapors, and does not carbonize or support combustion (see also Barrier polymers).

This plastic is compatible with liquid oxygen, remains flexible at cryogenic temperatures (12–15), and retains its properties when exposed to either uv or gamma radiation. Unlike other fluoroplastics, PCTFE resists cold flow as reflected by its high compressive strength which qualifies this plastic as a construction material in many load-bearing structural parts.

Manufacture and Processing

The preparation of the high molecular weight thermoplastic from chlorotrifluoroethylene [*79-38-9*] has been carried out in bulk (16–20), solution (21–23), suspension (24–29), and emulsion (30–34) systems using free-radical initiators, uv, and gamma radiation. The polymer obtained from emulsion represents a more thermally stable system than bulk or suspension-derived polymers. Polymerizations can be carried out in glass or stainless steel agitated reactors. Pressures (0.34–1.03 MPa or 50–150 psi) and temperatures (21–52°C) normally employed present no unusual equipment design problems.

Following the polymerization step, the polymer is then isolated from the latex or suspension. The suspension polymer, already in powder form, is washed to remove initiator residues and then dried. The emulsion polymer is coagulated from the latex by freezing or by the addition of salts, acids, and solvents (see Latex technology). The isolated powder is then washed and dried. The dried powder from either process additionally can be chemically treated to remove trace impurities that can result in chain degradation during further processing; treatment with carboxylic acids (35), ozone in air (36), or chlorine (37) improves thermal stability, color, and light transmission of the final polymer. The stabilized polymer is then sold to molders as powder or melt-extruded pellets in filled or unfilled form.

The lower molecular weight oils, waxes, and greases of PCTFE can be prepared directly by telomerization of the monomer or by pyrolysis of the higher molecular weight thermoplastic (38–47). PCTFE plastics can be processed by the standard thermoplastic fabrication techniques eg, extrusion, injection, compression, and transfer molding (see Plastics technology). The control of processing temperatures is important to avoid prolonged overheating (above 260°C) which can result in degradation of the polymer. The plastic can be readily machined from molded billets or rod stock on standard equipment to fabricate more exacting part geometries.

Economic Aspects

At present, there are several commercial suppliers of PCTFE and vinylidene fluoride-modified copolymers [*9010-75-7*] as shown in Table 3. PCTFE plastics are sold in the range of $37–66/kg, depending on the molecular weight, grade, product form, and supplier. As a result, PCTFE thermoplastics are used in high-technology, specialty engineering areas where the unique combination of properties and longevity of service demands a high performance thermoplastic.

Table 3. PCTFE Manufacturers and Products

Trademark	Manufacturer	Product forms
Homopolymers		
Daiflon	Daikin Koygo Yodogawa, Osaka, Japan	molding powder, pellets, dispersion oils, and greases
Kel-F 81 Brand Plastic	3M Company, St. Paul, Minn.	molding powders, pellets, and filled pellets
Halocarbon oil	Halocarbon Products Corporation, Hackensack, N.J.	oils, waxes, and greases
Voltalef	Ugine Kuhlmann, Pierre-Benite, France	molding powders and pellets
Copolymers		
Aclon, Aclar	Allied Chemical, Morristown, N.J.	molding powders, pellets, and film
Kel-F 82; Kel-F 800	3M Company, St. Paul, Minn.	molding powders and latex

Specifications

The specifications met by PCTFE plastics are Military Specifications, MIL-P-46036 and MIL-P-21470; Federal Specification LP-385B; and ASTM 1430 Grades II and III. Standards for fabricated forms are available for compression molded heavy sections (AMS-3645), molded sheets (AMS-3646 and -3649), thin-walled tubing (AMS-3648), and rod, sheet, and molded shapes (AMS-3650). PCTFE plastics have been approved for use in contact with food by the FDA (48).

Test Methods

The test methods employed are: determination of molecular weight as measured by ZST (ASTM D 1430); specific gravity (ASTM D 792); tensile strength, elongation and modulus (ASTM D 638); compressive strength and modulus (ASTM D 695); modulus of rigidity (ASTM D 747); deformation under load (ASTM D 621); heat deflection (ASTM D 648); impact strength (ASTM D 256); flammability (ASTM D 2863); hardness (ASTM D 2240 and D 785); and coefficient of linear expansion (ASTM D 696).

Health and Safety Factors

In general, the PCTFE resins have been found to be low in toxicity and irritation potential under normal handling conditions. Specific toxicological information and safe handling procedures are provided by the manufacturer of specific PCTFE products upon request.

Uses

The major uses for PCTFE plastics today have been in the areas of electrical/electronics, cryogenic, chemical, and medical instrumentation industries. Applications include chemically-resistant electrical insulation and components; cryogenic seals, gaskets, valve seats (49–50) and liners; instrument parts for medical and chemical (51) equipment; medical packaging; fiber-optic applications (see Fiber optics); and seals for the petrochemical/oil industry.

The lower molecular weight PCTFE oils, waxes, and greases are used as inert sealants and lubricants for equipment handling oxygen and other oxidative or corrosive media. Other uses include gyroscope flotation fluids and plasticizers for thermoplastics. The greases have specialty lubrication uses in severe environments such as in corrosive or oxidative media.

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