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ENHANCED COMPRESSIBLE FLUIDS INVESTIGATION

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20. ABSTRACT (cont)

fluid was examined. Results indicate that perfluorinated ether provides improved compressibility but has drawbacks such as a lower viscosity and a lower boiling point.

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INTRODUCTION

The purpose of this program was to provide the additional data necessary for one or more fluids to be used in compressible fluid recoil mechanisms. To be a new candidate, a fluid must have a balance of properties superior to those of Dow Corning's DC-200 (10 centistoke), particularly with respect to compressibility $(\frac{\Delta V}{V_0})_p$ over a temperature range of -50 to 300°F. The new candidate, which could be a silicone modified to improve its operational properties, would be subsequently tested in the Compressible Fluid Test Fixture (CFTF) at ARRADCOM.

A number of fluids with different molecular structures were investigated in a previous program (Reference 1). Two general families of compounds (fluorinated polyethers and perfluorocarbons) exhibited higher compressibilities than DC-200 (10) when compared on a molecular weight basis. When compared on the basis of reduced temperature or equal molecular volume, the silicone structure appeared superior.

The current program consisted of two principal tasks; characterization and modification of fluids (by synthesis or formulation if necessary) and trade-off studies. Desirable properties of a candidate fluid are listed in the appendix for easy reference. These were used as guidelines for both tasks.

The general approaches of the current program were (1) to identify operational deficiencies of commercially available fluids, (2) explore methods of improvement, (3) synthesize fluids with potentially higher compressibility, and (4) continue to canvass industrial and academic institutions for new compounds having potential as compressible fluids or fluid additives.

TECHNICAL DISCUSSION

This section discusses the experimental results and their implications regarding future chemical structures and fluid compositions. Details of new compounds, trade-off studies, theoretical considerations, and approaches toward composite liquids are included.

The PVT apparatus for testing compressibilities of small volumes of liquids (25-50 cc) is shown schematically in Figure 1 and has been described in Reference 1. Statistically derived compressibility equations and compressibilities at 5000 psig and room temperature are given in Table 1 for a number of fluids. The list supplements data obtained on other fluids during the previous program. Because the tests were conducted for a variety of reasons, the results in Table 1 and those in Reference 1 will be referred to frequently in the technical discussion.

Fluid Synthesis

The previous study indicated that the dimethylsilicones and perfluorinated carbon compounds have higher compressibilities than all other compounds tested. When specific compounds are compared on an approximately equal molecular volume basis (195-275 cc), the silicone structure appears to impart the highest compressibility. As shown in Figure 2, the compressibility values of hexamethyldisiloxane (HMDS) are higher than those of other structures when compared at reduced temperatures (test temperature divided by critical temperature of the liquid).

In the current program, it was hypothesized that compounds containing both disiloxane (SiOSi) and fluorocarbon moieties on the same molecule might yield relatively high compressibilities. This was supported by data obtained on a compound (S-1174 Mod) synthesized and tested in the previous program (Reference 1). The compressibility of this compound, which consisted of "blocks" of perfluorocarbon/hydrocarbon/disiloxane structures, was slightly higher than DC-200 (10) (4.1 versus 4.0% at 5000 psig and 23°C). It may be noted, as a side issue, that this compound is soluble in both silicone and perfluorinated polyether fluids and may, therefore, be applicable as a fluid additive.

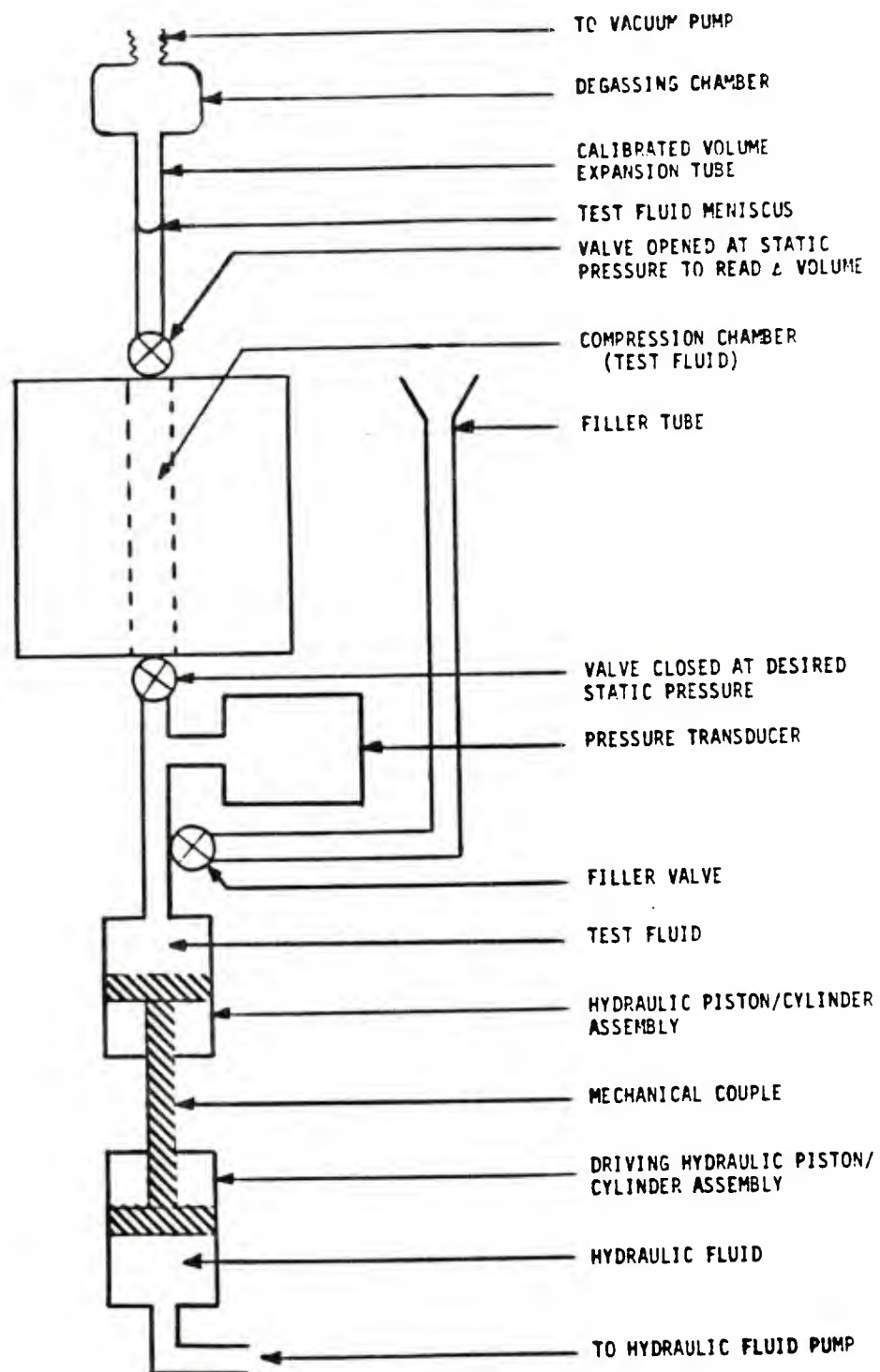


Figure 1. Schematic of the PVT Test Apparatus

Table 1. Compressibility Equations From 0 to 5000 Psig^a

Fluid	Test Temp, °C	a	$b_1 (\times 10^3)$	$b_2 (\times 10^8)$	Compressibility _b @ 5000 psig, %
FC-70	23	0.198	0.830	-3.00	3.40
FC-77 ^c	25	0.068	1.086	-2.357	4.84
FC-84	25	0.128	1.353	-4.714	5.59
L-4412	-44	-0.034	0.706	-2.429	2.92
	25	0.088	0.951	-2.286	4.18
	66	0.27	1.26	-4.00	5.32
Perfluoroheptane	25	-0.16	1.547	-7.857	5.77
TFA	-46	0.09	0.483	0.714	2.59
	25	0.056	0.882	-3.00	3.66
	66	0.006	1.166	-5.00	4.58
TFB	-21	0.406	0.351	1.071	2.02
	25	0.288	0.475	0.714	2.55
	62	-0.096	0.890	-4.357	3.36
n-Nonane	23	-0.032	0.755	-1.857	3.31
Diethylphthalate (DOP)	25	0.094	0.351	0.071	1.77
Polybutene #8	25	0.048	0.621	-3.143	2.32
Z03, Z10	23	-0.031	0.920	-3.271	3.78
FE-2/814-Z (50/50)	22	0.31	0.863	-0.50	4.19
HMDS	-46	0.096	0.759	-1.143	3.51
	25	0.030	1.211	-2.929	5.32

Table 1. (cont)

Fluid	Test Temp, °C	a	$b_1 (\times 10^3)$	$b_2 (\times 10^8)$	Compressibility, @ 5000 psig, % ^b
DC-200 (1 cs.)	23	0.106	1.037	-3.143	4.40
DC-200 (5 cs.)	-46	0.088	0.556	-0.429	2.67
	22	-0.118	1.037	-4.714	4.01
	66	0.188	1.108	-4.643	4.38
2,4-Dimethylpentane	23	0.038	0.958	-2.786	4.09

^a $y = a + b_1 x + b_2 x^2$ where y = % compressibility, x = pressure in psig.

^b Corrected to $a = 0$.

^c FC-77 with and without SF_6 saturation.

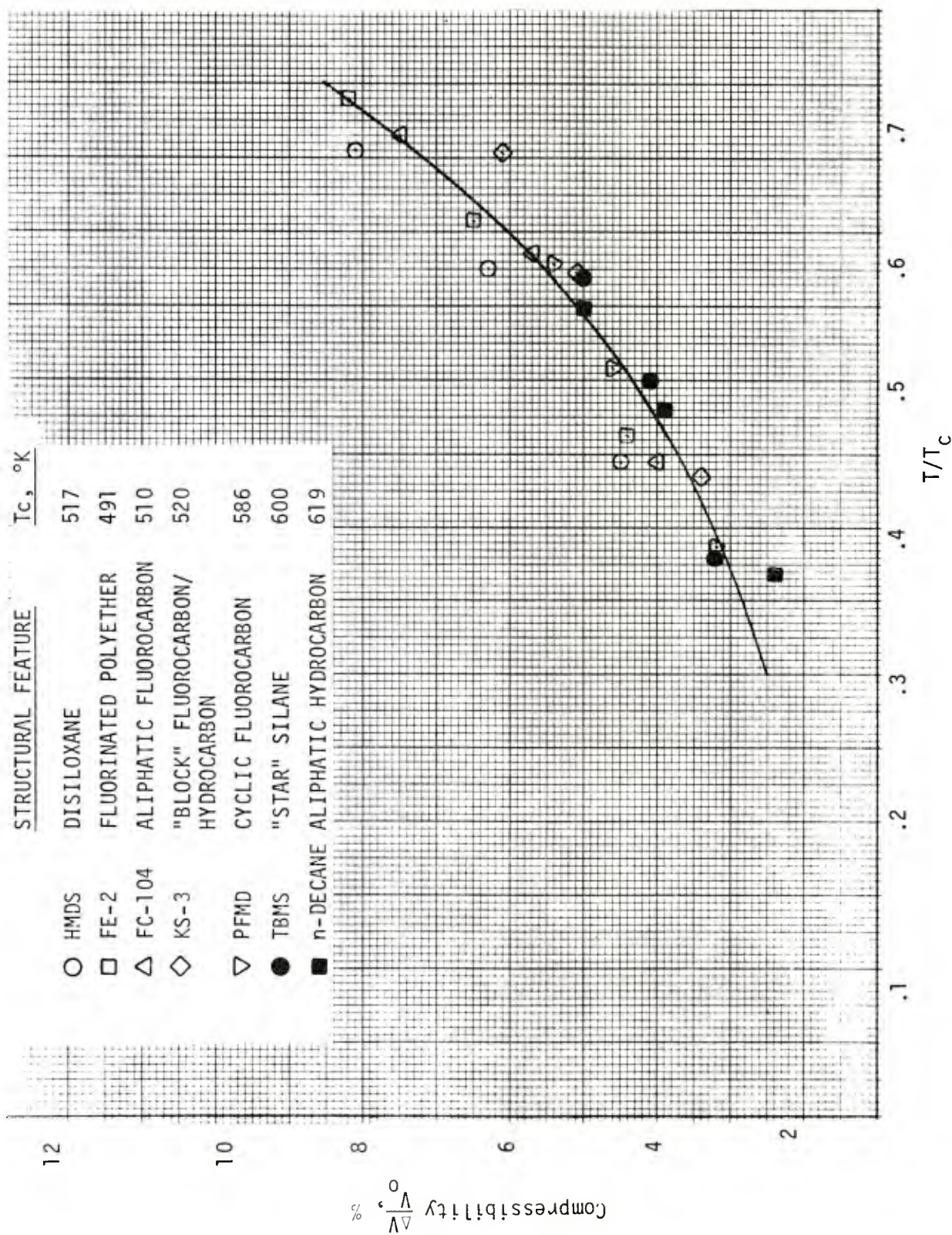


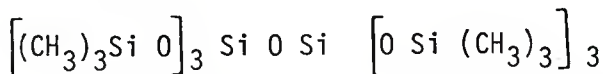
Figure 2. Compressibility at 5000 psig of compounds with molecular volumes of 195 - 275 cc at reduced temperatures

A search of the open literature revealed that only a very limited number of such compounds had been synthesized. One compound, $F_3CF_2SiOSiF_2CF_3$ (Reference 2), most closely met the criteria; however, the compound decomposes at 100°C. Unless the fluorine atoms are separated from the silicon by two carbon atoms, such general structures are unstable and SiF is evolved at elevated temperatures.

It was therefore suggested, as an alternative, that compounds of the general structure, $(R)_3SiO(R)_3$, where $R = CF_3CH_2CH_2-$, would provide both thermal stability and relatively high compressibility. Two such compounds, 1,3-Bis(3,3,3-trifluoropropyl) tetramethyldisiloxane and 1,1,3,3-Tetrakis(3,3,3-trifluoropropyl) dimethyldisiloxane, were synthesized by Silar Laboratories (Scotia, New York). If these showed a favorable compressibility trend, synthesis of a third compound, where all the R groups are $CF_3CH_2CH_2-$, would be attempted.

The structures, chemical analysis, and properties of the two synthesized compounds are listed in Table 2. For convenience the compounds are designated as Test Fluid A and B (TFA, TFB). The marked increase in boiling point and viscosities show that, in spite of the symmetry of the molecules, the $CF_3CH_2CH_2-$ group imparts high polarity and therefore high cohesive energy density. The decrease in compressibility is consistent with the viscosity and density data, since compressibility is inversely proportional to cohesive energy density. As indicated by molecular models of the compounds, the lower values are not attributed to rotational impediment of the SiOSi bond by the $CF_3CH_2CH_2-$ groups. Based on these results, the third compound was not synthesized.

Compounds with a preponderance of trimethylsiloxy groups were also considered as a means of attaining higher compressibility. To test this hypothesis, hexakis(trimethylsiloxy)disiloxane was synthesized by Silar Laboratories. The structure of the compound as inferred by its method of preparation is as follows:



Unexpectedly, the compound turned out to be a solid at room temperature with no definite melting or boiling point. Although it appears to

Table 2. Characterization of Synthesized Fluids

Molecular Structure	$[\text{CF}_3\text{CH}_2\text{CH}_2(\text{CH}_3)_2\text{Si}]_2\text{O}$	$[(\text{CF}_3\text{CH}_2\text{CH}_2)_2\text{CH}_3\text{Si}]_2\text{O}$
Elemental Analysis (%)		
Carbon, Theory	36.79	34.28
Carbon, Found	37.78	35.05
Hydrogen, Theory	6.17	4.52
Hydrogen, Found	6.29	4.54
Silicon, Theory	17.21	11.45
Silicon, Found	17.44	11.51
Fluorine, Theory	34.92	46.48
Fluorine, Found	35.39	46.91
Molecular Weight, g.	326.4	490.5
Boiling point, °C	182	160/1 mm.
Kinematic Viscosity, cs.		
-49°C	14.5	—
24°C	1.8	13.2
66°C	1.0	4.0
Density, g/cc		
-46°C	1.102	—
24°C	1.0625	1.2985
66°C	0.961	1.2095
Compressibility % (at 5000 psig)		
-46	2.59	—
25	3.66	2.55
66	4.58	3.36

sublime on heating, it is not known whether or not its sublimation is accompanied by partial decomposition. The effect of its structure on compressibility was tested as a 50/50 solution by weight in HMDS. The solution had a lower compressibility than HMDS itself (4.4 versus 5.3% at 5000 psig and 23°C).

Compressibility Screening of Other Compounds

During the course of the current investigation, a consistent effort was made to obtain and test new compounds. This effort had two objectives: (1) to evaluate the fluids per se as candidate fluids, and (2) to gain further insight into the effect of molecular structure on compressibility.

Samples of three compounds not previously tested were provided by the Commercial Chemicals Division of 3M. Two of these, FC-70 and FC-84, are commercially available as "Fluorinert" fluids. FC-70 is believed to be a perfluorinated tertiary amine and FC-84 a perfluorocarbon. L-4412, an experimental fluid, is a perfluorocarbon substituted hexafluorosulfide, $C_6F_{13}SF_5$. The compressibilities at 5000 psig/23°C and related properties of these materials are as follows:

	Boiling Point, °C	Pour Point, °C	Density, g/cc, at 25°C	Kinematic Viscosity, cs, at 25°C	Compressibility, % 5000 psig/23°C
FC-70	215	-25	1.94	13.4	3.4
FC-84	80	-95	1.73	0.55	5.6
L-4412	118	-31 (m.p.)	1.89	—	4.2

As in previous experience, relatively high compressibilities are obtained with perfluorinated compounds but only at a prohibitive expense of lower viscosities and boiling points. In general, none of the tested structures show any potential as a "breakthrough."

Based on previous data in Reference 1, it was believed that a lower molecular weight "Fomblin Z" fluid would provide relatively high compressibility. These "Z" fluids are products of Montedison and are available only in limited quantities. They are of particular interest because of their low viscosity to molecular weight ratios, low viscosity/temperature slopes, and their stabilities.

According to the vendor, the "Z" fluids have a general structure $\text{RF}-(\text{OCF}_2\text{CF}_2)_m(\text{O}-\text{CF}_2)_n-\text{RF}$ where RF is an inert fluorinated group. Their molecular weights are in the range of ten to twenty thousand. Hypothetically the properties vary with the ratio of m to n in the above formula.

Two samples of "Z" fluids, Z03 and Z10, were recently supplied by Montedison for evaluation. The compressibility of both materials at 23°C was 3.8% at 5000 psig. The results were lower than those obtained on previous samples supplied by Bray Oil Company. One distilled fraction from Bray had a viscosity of 10 cs at room temperature and exhibited a compressibility of 4.4% at the same conditions (Reference 1). The nominal viscosities at room temperature of Z03 and Z10 were 30 and 100 cs, respectively. Thus, the latter samples are most probably of higher molecular weight than the Bray sample. It is believed that a low molecular weight "Z Fluid" may be difficult to achieve synthetically. Further research is required in order to assess this type of perfluorinated polyether structure as a compressible fluid.

One 50/50 volumetric solution of fluids, Fomblin Z and FE-2, was tested for a possible synergistic effect. The data show that the compressibility was not improved even though the density of the solution was lower than predicted (1.734 g/cc).

<u>Fluid</u>	<u>Density g/cc at 25°C</u>	<u>Viscosity, cs at 25°C</u>	<u>Compressibility, % at 22°C, 5000 psig</u>
FE-2	1.660	0.8	5.5
814-Z	1.814	30.0	4.1
50/50	1.700	5.0	4.2

Gas Solubility Experiments

The effect of solubilized gas on the compressibility of a fluid was investigated. Potentially this effort had both practical and theoretical implications.

In most hydraulic systems it is necessary to degas the fluid and keep air out of the system for efficient operation. Degassing is desirable to avoid desolubilizing air during shear and dynamic compression cycling; if air is released and can agglomerate, the hydraulic system becomes "soft"

and relatively unresponsive. The same effect would be expected in the compressible fluid recoil mechanism.

Assuming that a recoil mechanism chamber is filled with degassed liquid, it is doubtful that air could be kept out of the system over an extended period or with temperature fluctuations during operation. Under these conditions an assured hermetic seal is difficult to achieve. Whether air diffuses into the liquid or is forced out of solution during dynamic conditions, it seemed desirable to determine the solubility of air and the rate of its dissolution in the liquid.

For air solubility measurements an instrument with the trade name "Air-ometer" was leased from Seaton-Wilson Control Technology Division of Systron Donner. This instrument has an internal hand-operated mercury pump to lower the pressure on a liquid sample introduced and sealed from the atmosphere. The air displaced from the liquid (in accordance with Henry's Law) is measured above the liquid column in a calibrated tube when the internal system is returned to atmospheric pressure. Reproducibility is good (within $\pm 5\%$), and the instrument is very convenient to operate. The values obtained on the "Fluorinert" fluids FC-72, FC-77, FC-80, and FC-104 were within $\pm 10\%$ of those reported by 3M (Reference 3).

Results of air solubility and compressibility tests conducted on a wide variety of materials are presented in Table 3. The data are plotted in Figure 3 as compressibility at 5000 psig and 23°C as a function of air solubility (as volume of gas per volume of liquid). The correlation is statistically significant. It should be noted, however, that the compressibility/gas solubility correlation could probably be improved if a pure gas were used instead of air since the partition coefficient for N_2 and O_2 could vary significantly between liquids. Also there are slight errors in the solubility and compressibility measurements. Nevertheless the correlation is potentially useful as a tool for estimating compressibility (or bulk modulus) of a liquid.

It may be noted that the compressibility of the dimethylsiloxanes appear somewhat higher than predicted by the mean regression line. (These values were not used in the regression analysis.) The deviation of these

Table 3. Air Solubility and Compressibility of Liquids With Various Structures

Acronym or Chemical Name	Chemical Name or Basic Structure	Air Solubility, Room Temp., cc/cc liquid	Compressibility, % at 5000 psig, 23°C
HMDS	Hexamethyldisiloxane	0.30	5.30
DC-200 (1 cs)	Polydimethylsiloxane	0.26	4.40
DC-200 (10 cs)	Polydimethylsiloxane	0.19	4.01
n-Nonane	Aliphatic hydrocarbon	0.20	3.30
2,4-Dimethylpentane	Branched aliphatic hydrocarbon	0.26	4.10
Tetraethylsilane	Aliphatic organosilane	0.22	2.84
Tributylmethylsilane	Aliphatic organosilane	0.18	3.07
Dibutyldimethylsilane	Aliphatic organosilane	0.16	3.06
S-1174-Mod ^a	3-(Heptafluoroisopropoxy)propyl-di-(trimethylsiloxy)methylsilane	0.28	4.13
D-1 ^b	Polyperfluoroisopropyl ether	0.32	4.3
Y-25 ^b	Polyperfluoroisopropyl ether	0.29	4.15
Y0-4 ^b	Polyperfluoroisopropyl ether	0.31	3.94
FE-2	Polyfluoroisopropyl ether	0.44	5.47
FE-4	Polyfluoroisopropyl ether	0.36	4.54
3X-823-1 ^c	RF-(CF ₂ CF ₂ O) _m -(CF ₂ O) _n -RF ₃	0.31	4.20
3X-823-2 ^c	RF-(CF ₂ CF ₂ O) _m -(CF ₂ O) _n -RF ₃	0.31	4.41
814-Z ^c	RF-(CF ₂ CF ₂ O) _m -(CF ₂ O) _n -RF ₃	0.30	4.12
TFA ^a	1,3-Bis(3,3,3-trifluoropropyl)tetramethyldisiloxane	0.32	3.66

Table 3. (cont)

Acronym or Chemical Name	Chemical Name or Basic Structure	Air Solubility, Room Temp., cc/cc liquid	Compressibility, % at 5000 psig, 23°C
TFB ^a	1,1,3,3-Tetrakis(3,3,3-trifluoro propyl)dimethyldisiloxane	0.21	2.55
PS-181 ^d	Poly(methyl-3,3,3-trifluoro- propyl)siloxane	0.20	2.89
FC-72 ^e	Perfluorohexane	0.44	5.97
FC-77 ^e	Mixture of FC-80 and FC-104	0.42	4.73
FC-80 ^e	Perfluoro-2-butyltetrahydrofuran	0.42	4.62
FC-104 ^e	Perfluorooctane	0.42	4.69
L-4412 ^f	Perfluoro-n-hexyl sulfur penta- fluoride	0.36	4.18
Perfluoroheptane	Perfluorinated aliphatic carbon	0.48	5.77
Perfluoromethyldecalin	Bicyclic perfluorinated carbon	0.26	3.59
DOP	Di(2-ethylhexyl)phthalate	0.13	1.77
H ₂ O	Water	0.017	1.4

^a Compounds synthesized by Silar Laboratories, Scotia, NY

^b Montedison "Fomblin Y" fluids

^c "Fomblin Z" fluids and fractionated cuts obtained from Bray Oil Co., Irvine, CA.

^d Product of Petrach Systems Inc., Bristol, PA.

^e 3M "Fluorinert" fluids

^f 3M research sample

materials from others is consistent with their singular behavior in compressibility versus reduced temperature as shown in Figure 2.

An attempt was made to estimate the rate of air dissolution into various liquids. Most of the fluids have relatively low viscosities as compared with more common hydraulic fluids. Therefore their rates of air dissolution are much more rapid. It was found that even a brief exposure to air, such as a hypodermic sampling of fluid and transfer into the Aireometer, saturated the fluids. In compression cycling tests of FE-2 and FE-4 fluids (fluorinated polyethers), no evidence was seen of formation of air pockets in the system. These results do not mean that the lower viscosity fluids will not lose air during high shear but they imply that the problem becomes less severe. In view of the necessity for special equipment and the above results, determination of accurate rates was not pursued.

Highly Soluble Gases

The compressibility of any of the fluids was the same whether the fluid was deaerated or not, at least within experimental error. This was repeatedly demonstrated by measuring the air saturated fluid in the PVT apparatus, degassing the fluid while in the apparatus, and then re-measuring compressibility.

The relatively high solubility of sulfur hexafluoride in perfluorocarbons presented an opportunity to check these results more clearly. A fluid, FC-77, was saturated with SF_6 at room temperature. Weight pickup at room temperature was 1.38%, or 3.82 cc of gas per cc of liquid. In contrast, FC-77 dissolves only 0.42 cc of air per cc of liquid. Densities of the liquids were 1.7890 (23.2°C) and 1.7909 (23.6°C), without and with SF_6 saturation respectively. Based on a density of 1.91 g/cc for SF_6 as a liquid, the predicted density of the SF_6 solution was 1.791. The SF_6 solution was tested in the PVT apparatus before and after degassing. The compressibility measurements were identical.

Trade-off Studies

The objective of this study was to provide the basis for selection of the best fluid or fluids to be tested further in the ARRADCOM Compressible Fluid Test Fixture (CFTF). Because no one fluid would be expected to have

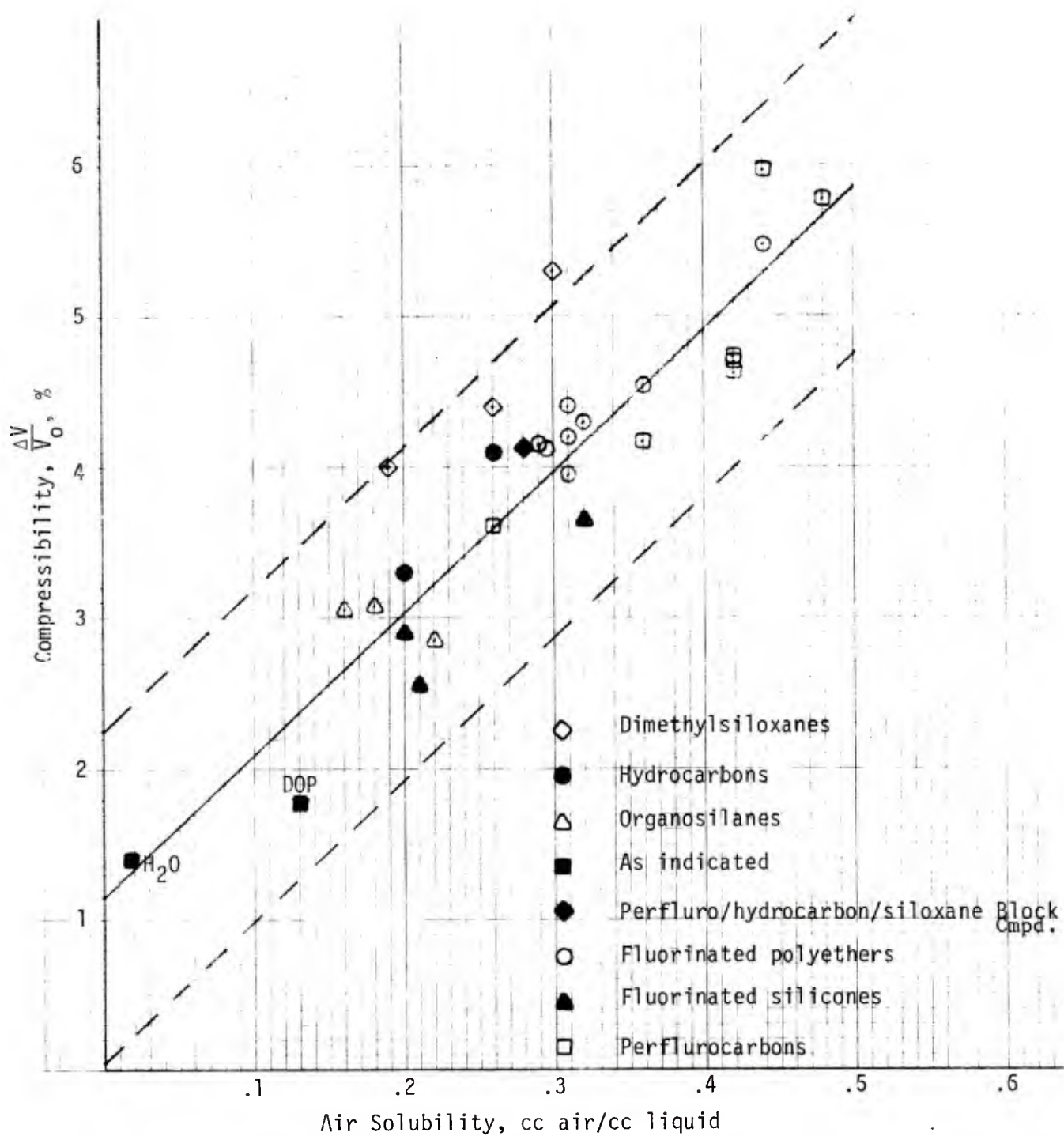


Figure 3. Correlation of Compressibility at 5000 psig and Air Solubility

superior properties in all categories, compromises would be necessary.

The approach was (1) to gather as much information as possible from the vendors and open literature on those fluid structures showing the higher compressibilities, and (2) to supplement these data with tests as necessary. From these results methods of improving a fluid (or fluids) would be explored.

Fluids selected for comparative purposes were DC-200 (10 cs), two fluorinated polyethers (FE-2, FE-4), two perfluorinated polyethers (814-Z, D-1), and a perfluorocarbon (FC-104). The fluorine-containing fluids all have higher compressibilities than DC-200 (10) and represent different molecular structures. It should be noted that FE-2 and FE-4 are not fully fluorinated and contain a hydrogen atom in the terminal group of the polymer. Although D-1 and the FE polyethers have similar backbone structures, D-1 is fully fluorinated. Also the molecular weight of FE-4 is approximately 70% greater than FE-2 and equal to D-1. Therefore, the effect of molecular weight and the presence of the hydrogen atom could be assessed.

Comparative values on properties, for which data are available on most of the materials, are given in Table 4. The properties or parameters are discussed below either individually or in combination as their interaction appears applicable.

Compressibility/Viscosity Indices

A combination of high viscosity index (low viscosity/temperature slope) and high compressibility over the temperature range is desirable in order to simplify the design of the recoil mechanism. Increased stiffness of the mechanism with decreasing temperature is expected to be a function of both these parameters. Because viscosity is an exponential function of temperature, it is difficult to compare the fluids on this basis without some simple means of indexing.

In Table 5 the fluids are compared on a basis of compressibility/temperature (C_T) and viscosity/temperature (V_T) coefficients. The reference temperatures are -46 and 25°C. The ratios of C_T/V_T , which may be viewed as a compliance ratio, are also listed as a comparative aid. To emphasize the effect of molecular weight, two lower molecular weight

Table 4. Comparison of compressible fluid candidates

Property	Requirement (R) or Desired Values	DC 200 (10)	FE-2	FE-4	Fomblin "Z"	Fomblin "Y" (D-1)	Fluorinert FC-104
(R) Compressibility, %							
-50°F	>DC-200 (10)	2.6	3.4	2.8	2.9	2.9	3.0
77	"	4.0	5.5	4.5	4.1	4.3	4.7
150	"	4.8	7.2	5.8	5.7	5.9	6.5
Viscosity, cs.							
-65°F	3500	100	5	130	1100	—	13
-40	80	60	3	40	40	150	4.4
77	—	12	0.6	2.3	24	4.5	0.4
100	14	9	0.53	1.8	18	3	0.3
150	—	6	—	1.1	10	1.6	—
300	—	2.6	—	—	3	—	—
Thermal stability							
	Able to with- stand 300°F	Upper useful T = 400°F	Decompo- sition Rate = .03%/24 hrs at 670°F (FE-3)	>1000°F	Negligible under 500°F (in air)	Small am't decomposition >392°F	
Flash point							
	500°F (min)	325°F	>1000°F	>1000°F	None	None	N.A. ^b
Volatility: boiling pt., °F							
	TBD ^a —	>320 at 0.5 TORR	219	381	>480 at 8 mili TORR	419	214
Shear stability							
	Equal or better than MIL-H-6083	No decomposition indicated by GC spectra after 12 minutes of ultrasonic exposure at 50-60 watts. ^b					

Table 4. (cont)

Property	Requirement or Desired Values	DC 200 (10)	FE-2	FE-4	Fomblin "Z"	Fomblin "Y" (D-1)	Fluorinert FC-104
Surface tension, (dynes/cm)	Less or equal to MIL-H-6083	20.1 (72°F)	12.9 (72°F)	15.2 (77°F)	20 (68°F)	18 (68°F)	14 (77°F)
Thermal conduc- tivity, Btu/ft hr °F	TBD	0.077	0.0372	0.0383	0.05	0.041	0.037
Thermal expansion ft ³ /ft ³ °F	TBD	0.00062	0.00084	0.00070	0.0006	0.00069	0.0008
Specific heat Btu/lb °F	TBD	0.43 (104°F)	0.24	0.24	0.24 (100°F)	0.24 (77°F)	0.25 (77°F)
Water solubility (ppm)	TBD	>200	103	58	—	—	11
Density, lbs/gal	TBD	8	14	15	15	15	14
Approximate cost per lb., \$	—	9	43	43	400	60	23

^a TBD = to be determined.^b N.A. = not available.

Table 5. Viscosity and Compressibility Temperature Coefficients of Selected Fluids

Fluid	Viscosity, cs		Compressibility, %		Viscosity ^a		Compressibility ^b	Coefficient
	-46°C	25°C	-46°C	25°C	Coefficient, V_T	Coefficient, C_T		
HMDS	1.9	0.65	3.5	5.3	0.018	0.025	1.40	
DC-200 (5 cs)	43	5	2.7	4.0	0.54	0.018	0.034	
DC-200 (10 cs)	70	12	2.6	4.0	0.82	0.020	0.024	
FE-2	5	0.8	3.4	5.5	0.059	0.026	0.44	
FE-4	73	2.5	2.8	4.5	0.99	0.024	0.024	
D-1	276	4.8	2.9	4.3	3.82	0.02	0.0052	
814-Z	500	30	2.9	4.3	6.62	0.017	0.0026	
FC-104	4.6	0.75	3.0	4.7	0.054	0.024	0.44	

^a Change in viscosity per degree of temperature

^b Change in compressibility at 5000 psig per degree of temperature

dimethylsiloxanes have been added to the list [DC-200 (5) and HMDS].

In the dimethylsiloxane fluids, markedly higher C_T/V_T ratios are only obtained below a viscosity of 5 cs. The difference between FE-2 and FE-4 is also highlighted by this criterion; the ratios are 0.441 and 0.024, respectively. FC-104 compares favorably with FE-2. The C_T/V_T ratios of all other listed materials are either equal to DC-200 (10) or lower.

On this basis the low molecular weight materials, FE-2, HMDS [or perhaps DC-200 (1 cs)], or FC-104, could be expected to function much better than the other materials between ambient and low temperatures.

The possibility of using "viscosity improvers," such as those used in hydrocarbon lubricating fluids, was considered. These materials lower the viscosity/temperature slopes by increasing the fluids' viscosities at the higher temperatures without affecting the viscosities at the lower temperatures. Hypothetically, a lower slope would aid in the performance of the silicones or fluorinated fluids over the required temperature range. A canvass of technical people in the applicable disciplines failed to indicate either availability or any research being conducted on viscosity improvers for these materials. Therefore, the concept could not be tested adequately. However, a two-phase system consisting of one percent by weight of a finely divided Teflon powder (DLX-6000 from du Pont) in a Fomblin Y fluid, Y0-25 (Montedison), was examined. The mixture was sheared with a high speed blender until the particles became gel-like in appearance. The resulting suspension, which qualitatively appeared to have increased markedly in viscosity, could not be tested in the PVT apparatus. Apparently the main problem was entrapment of relatively large quantities of air in the mixture. The suspension could not be degassed in the PVT apparatus to permit compressibility tests. This concept of using insoluble particles as a means of attaining a lower viscosity/temperature coefficient may have some merit but would require considerably more effort.

It should be noted that the incorporation of soluble viscosity improvers in hydrocarbons probably lowers the compressibility of the resulting fluid. This effect is indicated by a single experiment with n-nonane containing 20% by weight of Acryloid 702 (Rohm and Haas Company).

The compressibility at 23°C and 5000 psig of the solution was 3.1% as compared to 3.3% for n-nonane alone.

Thermal Properties

Based on the data in Table 4 the fluorinated polyethers are superior to DC-200 in thermal stabilities and have higher flash points. The low molecular weight fluids (FE-2, FC-104, and the low viscosity dimethylsiloxanes) all have the disadvantage of low boiling points and relatively high volatility. None offer a trade-off advantage with respect to a balance of volatility and compressibility.

As a heat transfer fluid, DC-200 (10) has a clear advantage; its thermal conductivity is significantly higher than the other fluids. As a heat sink, the fluids are approximately equal if heat capacity is calculated on a volumetric basis. Thermal expansion coefficients are approximately the same for all fluids if molecular weight is taken into consideration. At higher temperatures the lower boiling fluids would increase the internal pressure of a closed recoil system. However, if no leaks occurred, the higher compressibility would be expected to compensate for a higher residual pressure.

Stability Under Dynamic Conditions

The PVT apparatus was used to simulate compression/decompression cycling in order to assess the stabilities of FE-2 and FE-4 under dynamic conditions. The fluids were subjected to a maximum of 150 cycles between 0 and 5000 psig at room temperature. The valve orifice at the bottom of the compression chamber provided the principal site of mechanical stress. At intervals of 50 cycles, the fluid compressibility was measured. The results are shown in Figures 4 and 5. The observed displacements of the curves are considered to be within the experimental variability of all other compressibility measurements. No evidence of breakdown was observed; no air or gas bubbles were evolved when the system was opened to atmospheric pressure.

The fluids were further checked for evidence of decomposition by gas chromatography. Although minor differences in the spectra were discernible, lower molecular weight fractions, which would indicate major molecular decomposition, were not evident.

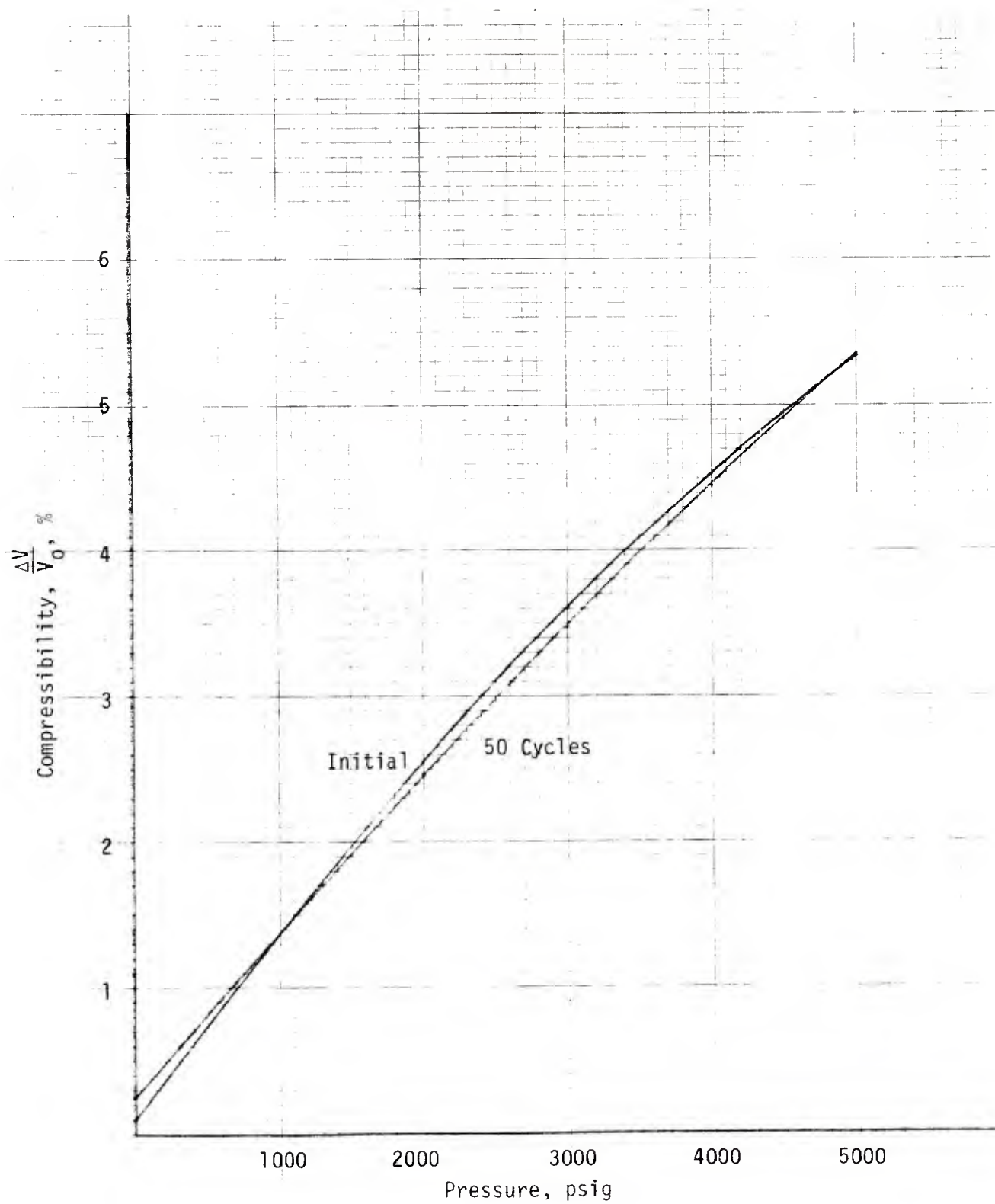


Figure 4. FE-2 Compression/Decompression Cyclic Tests

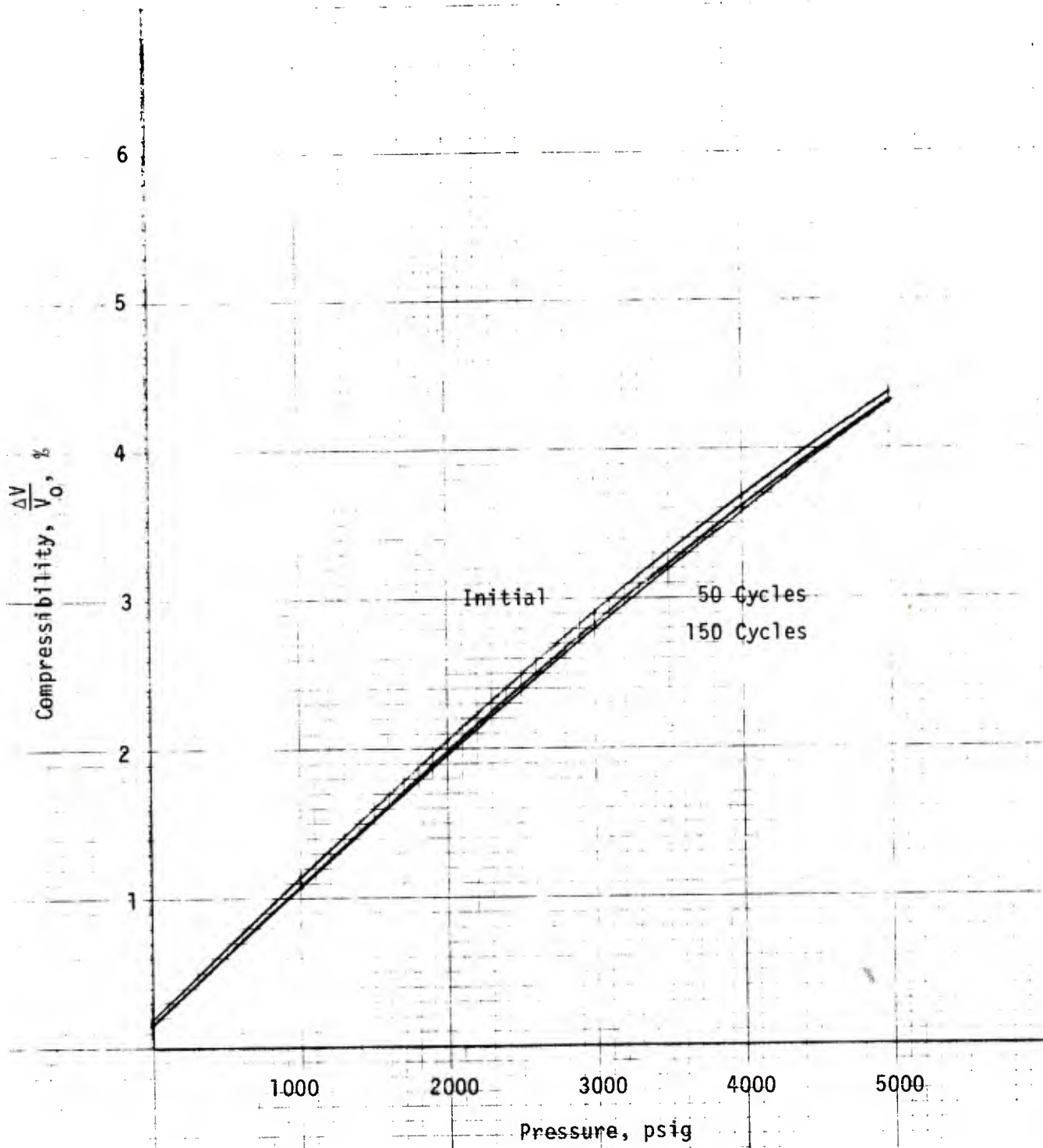


Figure 5. FE-4, Compression/Decompression Cyclic Tests

As indicated in Table 4, the dynamic shear stabilities of the fluorinated polyethers and DC-200 (10) fluids were compared through ultrasound exposure and subsequent chemical analysis. Shear stability measurements as described in MIL-H-6083 could not be duplicated since the recommended apparatus is no longer available. The tests were conducted in open containers thermostated in an ice bath. The samples were sonicated by a metal probe (Sonifier Cell Disrupter, Model W-135) for 3, 6, and 12 minutes. During sonification, the "Z" fluid (and the D-1 and FE-4 fluids to a lesser degree) developed noticeable acrid odors, indicating evolution of low molecular weight volatile materials and/or decomposition products. By this criteria it might be concluded that DC-200 (10) is more stable under high shear at room temperature than the fluorinated compounds. However, the decomposition products of the silicone fluid are markedly less irritating and would not be detected by odor. Comparison of the gas chromatographic spectra before and after sonification showed no major change in any of the fluid compositions. Therefore they are judged approximately equal in shear stabilities.

Compatibility With Seals

Detailed data on the compatibility of the silicones and fluorinated carbon compounds with different elastomers and plastics are available from the vendors or manufacturers of these materials. The fluids are chemically inert and are sparingly soluble in most materials used in making seals. Notable exceptions are silicone oils with silicone rubbers, fluorocarbon fluids in fluorinated elastomers, and other systems wherein the seal matrices and the fluids have similar chemical structure.

No data were available on the fluids' compatibility with seals prepared with phosphorous-nitrogen-fluorine polymers (e.g., PNF by Firestone Tire and Rubber Company). Samples of three seal compositions were obtained from Greene Tweed and Company. The percent swell of these materials were determined with DC-200 (10), FE-2, FE-4, and a Fomblin Z fluid fraction. Tests were conducted according to ASTM D1460. The data are presented in Table 6. Only slight differences between the PNF compositions are apparent in the overall data. Of the four fluids, swelling is least in DC-200 (10). There is a progressive increase in swelling as the molecular weight

Table 6. Swell of PNF Seal Compositions in Selected Fluids

Sample ^a	Fluid		T = 25°C		T = 100°F	
	Trade Designation	Type	ΔL%	ΔV%	ΔL%	ΔV%
732	FE-4 ^b	Fluorinated polyester based on perfluoropropylene oxide.	0.8	2.4	5.6	17.7
733			1.2	3.6	5.6	17.7
734			1.6	4.8	5.6	17.7
734	FE-2 ^b	Same as above but of lower molecular weight.	6.8	21.8	(N.A. - boiling pt. = 104°C)	
732	Fomblin Z ^c	Perfluorinated polyether	1.2	3.6	1.6	4.8
733			1.2	3.6	1.2	3.6
734			0.8	2.4	1.6	4.8
732	DC-200 (10) ^d	Polydimethylsiloxane	0	0	0.8	2.4
733			0	0	1.2	3.6
734		(Silicone with viscosity of 10 cs)	0	0	0.8	2.4

Sources: ^a Greene Tweed & Co., North Wales, PA.
^b Helix Associates, Inc., Newark, Del.
^c Montedison/Bray Oil Co., Irvine, CA.
^d Dow Corning Corp., Midland, Mich.

decreases among the fluorinated polyethers, as expected. The color of the DC-200 (10) fluid was unaffected; a slight yellowing of the fluorinated fluids occurred at the higher temperature. This indicates an extraction of a chemically unbound material from the PNF seals, perhaps a polymerization catalyst. Qualitatively, there was no apparent physical deterioration of any of the PNF samples even after an additional month's immersion in the fluids.

Prolonged exposure of the PNF seal materials to any of the tested fluids at room temperature is not expected to affect their performance. Although the FE-2 fluid will swell the PNF materials to a much greater degree at elevated temperature, intermittent short-time exposure should pose no serious problem. Nevertheless, two areas need to be addressed: (1) the performance of the seals under actual use conditions (dynamic compression and decompression cycling), and (2) the nature of the chemical(s) extracted by the fluorinated fluids and their effects on other parameters such as fluid stabilities or compatibilities with other materials.

PNF seals have been tested in a current seal program (Reference 4). The seals (Firestone 200-009) failed after 43,945 short piston strokes at 275°F in chlorotrifluoroethylene. Failure was due to frictional wear. No comparative data with other fluids are available.

Compatibility With Metals

As in the case of compatibility with organic polymers, a great deal of specific information is available from the vendors or manufacturers of the fluids. In general the fluids are chemically inert to most metals; no change in the fluorinated fluids or metals occur up to 500°F, even in the presence of oxygen. Again there are exceptions; the fluids will react with fresh aluminum or magnesium surfaces. Montedison, in their product manual on the Fomblin Y fluids, points out that "pristine surfaces of magnesium and aluminum can catalyze the decomposition of perfluorinated polyether fluids." They do not recommend their use where friction can rub off the protective oxide film from these metals.

Because the polyperfluoroalkyl ethers are of interest in high performance aerospace systems, anti-corrosion and oxidation inhibitors (or metal

deactivators) are being researched for high temperature applications. One class of compounds, substituted perfluorophenylphosphines, was effective in extending the upper temperature use of the fluids with ferrous and titanium alloys (Reference 5). Phospha-s-triazines have been synthesized and found to be effective anti-corrosion additives for these fluids (References 6, 7). These materials, however, are not currently available commercially.

Lubricity

The subject of lubricity encompasses a number of interrelated factors, some of which have been discussed briefly above with respect to compatibility with seals and metals. Lubricity is defined as the non-viscosity part of lubrication (Reference 8) and, for any given fluid, depends on the metallurgy and topography of a surface. Also, a minute concentration of an additive or contaminant may impart excellent or poor lubricity to a fluid (Reference 9).

A number of different test methods have been devised for measurement of lubricity; among these are the Shell Extreme Pressure Four-Ball and Falex Wear Tests. Shell test data are available from product manuals on some of the fluids. However, the data cannot be compared unambiguously because the test conditions are dissimilar. Qualitatively the dimethyl silicones appear to be inferior to the fluorinated polyethers; both are inferior to a hydrocarbon oil with an extreme pressure additive. (According to Dow Corning's data sheet, the fluorosilicones, such as FS-1265, are comparable to the hydrocarbon lubricants; this is based on results of Falex Wear Tests.)

The ability of different fluids to prevent wear between dissimilar metals varies markedly with the metals or alloys involved. DC-200 fluids are classified as "O.K." between aluminum and copper, magnesium, brass, and bronze, and between bronze and steel; they are classified as "bad" for steel on steel and aluminum on aluminum (Reference 10). Similar classifications were not found for the other test fluids in question.

Special "use tests" are necessary to determine the adequacy of lubrication in any system. It is reasonable to assume that the low viscosity

fluids, such as FE-2, will not be adequate at higher temperatures without viscosity improvers. As discussed previously, this requires further R&D.

Toxicity and Safety

All of the compared liquids are non-toxic. Oral ingestion or skin contact have shown no ill effects. Above 500°F the perfluorinated compounds decompose, evolving toxic products. Within the required temperature range (up to 300°F), no toxicity problem is anticipated with any of the fluids.

Cost

The costs per pound in Table 4 are approximate and reflect, for the most part, the current state of development and demand for these materials. The Fomblin Z fluids are just now emerging from the research stage and are now available commercially, although in limited quantities. The Freons, Fomblin Y, and Fluorinert fluids represent older and more developed technology. Most developed and in largest supply are the DC-200 fluids.

It is difficult, if not impossible, to project future costs of these materials without detailed knowledge of the chemistry, engineering requirements, product use, etc. However, in general, there is some justification for expecting lower costs of the Fomblin Z fluids. Both the Y and Z fluids involve a single step synthesis, although the latter requires a gas phase reaction. Also, in general, it is doubtful that any of the fluorinated materials will ever compete pricewise with the silicone fluids.

Results of Discussion

Based on the above trade-off study, the polydimethylsiloxanes have an overall edge as a family of fluids for the compressible fluid recoil mechanism. Because they are more hydrocarbon-like than fluorinated carbons, it is probable that existing knowledge regarding viscosity improvers and extreme pressure additives for conventional lubricants might be adapted more readily to improve the silicones. However, additives for the polyperfluoroalkyl ethers are under study and progress in this field looks promising.

Increased compressibility is attainable in any family of fluids through the use of lower molecular weight homologs. This adversely affects lubrication, thermal stability, and compatibility with other materials. The extent and seriousness of such effects can best be determined by use tests more representative of dynamic conditions. Further R&D will be necessary to correct adverse effects.

Use of lower molecular weight dimethylsiloxanes may be prohibited because of their lower flash points, although development of additives to improve this property is possible. For safety reasons, fluorinated fluids such as FC 77 or FE-2 are currently preferred in testing low molecular weight/low viscosity materials with relatively high compressibilities.

Composite Fluids

Although the principal approach toward high compressibility has been through molecular structures of single fluids or combinations of fluids, the possibility of using biphasic or multiphase systems has been considered. For instance, colloids offer some promise of relatively high compressibility because their components would repel each other. Problems with this approach include homogenization of two incompatible liquids (preferably both having high individual compressibilities) and the stability of such mixtures, particularly with pressure and temperature fluctuations.

The use of microballoons in a compressible fluid appears more feasible. The concept has been explored by ARRADCOM, and a study of materials and wall thicknesses have been conducted (Reference 11).

Availability of the PVT apparatus and commercial quantities of polyvinylidene chloride microballoons presented an opportunity to assess the concept experimentally on this program. The microballoons were obtained as a sample from Pierce and Stevens, Buffalo, New York. The particular sample was "Mirulite 176" having an apparent bulk density of 0.020 g/cc, and a true density of 0.040 g/cc. The average particle size was 40 microns and the estimated wall thickness was 1 micron.

The first compressibility test was made with 1% by weight microballoons in a mixture of DC-200 (10) and 0.5% Cabosil 17 (Cabot Corporation) as a thixotropic agent. The latter aided to keep the microballoon in

suspension. This mixture exceeded the volume capacity of the calibrated expansion tube; the compressibility at 1000 psig was greater than 15%.

A second mixture consisted of 0.1% by weight of microballoons in DC-200 (10). No thixotropic agent was used. The calculated percent volume of microballoons to fluid was 3% or 6%, based on the two densities listed above. At 1000 psig the measured compressibility of this mixture was 5.2% as contrasted to 0.9% for DC-200 (10) fluid above. This is a 5.8-fold increase in compressibility. Further readings at higher pressures were meaningless because some of the microballoons had escaped into the expansion tube or some of the microballoons had collapsed, or both.

The immediate problem with the concept is keeping the microballoons in suspension. An obvious approach is to match the effective density of the microballoons with that of the liquid carrier. This appears feasible with the polyvinylidene chloride (PVDC) polymers in a silicone fluid. At room temperature the density of the polymer (as a solid) ranges from 1.65 to 1.75 g/cc; the density of DC-200 (10) is nominally 0.935 g/cc. As an approximation, it can be shown that

$$\frac{(r_i)^3}{(r_s)^3} = 1 - \frac{\rho_l}{\rho_p}$$

where r_i = radius of the inner (hollow) sphere

r_s = radius of the outer sphere

ρ_l = density of the liquid

ρ_p = density of the polymer

Thus, if 1.65 is assumed as the density of the polymer, then

$$\frac{(r_i)^3}{(r_s)^3} = 1 - \frac{.935}{1.65} = 0.433$$

if the radius of the sphere = 1 micron

$$(r_i)^3 = 0.433 \text{ and}$$

$$r_i = \sqrt[3]{0.433} = .757$$

and the wall thickness is $1 - .757 = 0.243$ micron and the ratio of wall thickness to the diameter of the sphere would be $\frac{0.243}{2} = 0.122$

It is possible that this ratio can be approximated with current technology, but some development would be required. Currently microballoons can be prepared with a bulk density of 0.08 g/cc, which is a four-fold increase over those tested to date.

Other problems inherent in the concept have been discussed in Reference 11. The relative difficulties with these and other more subtle problems can only be assessed experimentally.

CONCLUSIONS

Although substantial progress has been made in understanding the effect of molecular structure on compressibility, tailoring compounds and/or predicting fluid properties remain somewhat speculative. Two synthesized compounds, wherein 3,3,3-trifluoropropyl groups were substituted for CH_3 groups in hexamethyldisiloxane, exhibited decreased rather than increased compressibility as had been anticipated. Tests of other new structures, including a perfluorinated tertiary amine and a perfluorinated hexyl sulfur pentafluoride, add to the list of structures on which future predictability can be improved.

Possible effects of solubilized gases on fluid compressibility were investigated. Most compounds of interest as compressible fluids absorb 0.2 to 0.5 cc of air per cc of liquid, but tests indicated that separation and subsequent agglomeration of air in a closed system is not a major problem. Resaturation of air at ambient pressure and temperature is a rapid process. Solubilized gases had no effect on compressibility as shown by a fluorocarbon containing 3.4 cc SF_6 per cc of liquid.

Compressibility correlates well with the solubility of air in all liquids tested. The developed correlation provides a convenient method of estimating the compressibility of other compounds.

Trade-off studies were conducted on commercially available fluids which had been tested on this program. Comparisons between DC-200 and fluorinated compounds showed a slight superiority of the former, particularly in a better balance of thermal properties and in cost. Compatibility with PNF seals and stability under dynamic conditions were judged nearly

equal, although slight decomposition was exhibited by the fluorinated polyethers during shear stability tests. Their relative lubricities and compatibilities with metals appear similar. However, a detailed assessment could not be made because test conditions were not comparable. The fluorinated compounds have very high flash points compared to DC-200 (10) but have the disadvantage of evolving noxious gases on decomposition at relatively high temperatures.

Indexing of compressibility and viscosity with respect to temperature indicates that low molecular weight homologs of fluorinated polyethers or silicones will provide less stiffness and less differential in gun barrel travel with temperature. If this approach is taken, viscosity improvers for the fluid are probably necessary to provide lubrication at the higher temperatures.

The most promising approach toward a highly compressible fluid is through the incorporation of elastomeric microballoons. A five to six-fold increase in compressibility is indicated by an exploratory experiment using polyvinylidene chloride balloons in DC-200 (10) fluid. The immediate problem is maintaining suspension of the buoyant particles in the fluid. Potentially this can be solved through control of wall thickness of the microballoon.

RECOMMENDATIONS

The concept of "composite fluids," particularly the incorporation of microballoons in a carrier fluid, should be pursued. This will require technological advancements in microballoon manufacture and a coordinated test program to evaluate fluid compositions over the required temperature range. Silicone fluids are recommended as carrier fluids because of their low densities, low viscosity/temperature coefficients, availabilities, and costs.

Testing and further development of fluids by themselves should not be abandoned. A low viscosity fluid, such as DC-200 (0.65 cs) or FE-2 (a low molecular weight fluorinated polyether), should be tested in the CFTF. Data on the effect of viscosity indices on the dynamic response of the recoil mechanisms would be valuable in the development of "composite fluids."

Further research and development of additives to improve operational properties such as lubricity and viscosity of fluorinated carbons and silicones should be pursued. A compound (S-1174 Mod), which was synthesized on the previous program, may have use as an additive because of its mutual solubility in both fluorinated carbons and silicones. Additives to improve the flash point of silicones are also desirable.

Testing of seals, particularly those prepared with PNF compounds, can and should be tested under dynamic conditions. Compatibility tests are considered inadequate because they do not address friction and wear.

Use tests are recommended to obtain more specific data on the effect of fluid compositions and contaminants with respect to metal compatibility and lubricity. General tests available at present do not represent actual metallurgical or environmental conditions.

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APPENDIX
REQUIREMENTS FOR COMPRESSIBLE FLUID

I. Essential Requirements:

1. Operating Pressures: 5000 psi (3.45×10^7 Pa) Max; 4000 psi (2.76×10^7 PA).
2. Compressibility: ARRADCOM has, as an in-house effort, experimentally evaluated the compressibility of various commonly available fluids. They include Dow Corning DC 200, Fluorocarbon FC75, Halocarbon Polymer Oil 411E and 11-14E, Fomblin Y04 and Freon E3. The maximum compressibility achieved with any of these fluids was 3.4%. Based on this finding, it is anticipated that the fluids formulated under this contract possess a significant increase in compressibility. A compressibility of 8 to 10% at maximum pressure and minimum temperature would be considered ideal.
3. Operating Temperature Range: -50°F to +300°F (-45.6°C to 148.9°C).
4. Toxicity: Should be non-toxic, non-hazardous and non-allergenic to operating personnel; specific requirements to be determined or approved by Surgeon General.

II. Desirable Properties

1. Viscosity: Similar to MIL-H-6083; should be sufficient to lubricate bearings at 300°F (148.9°C).
2. Thermal Stability: Able to withstand 300°F (148.9°C) exposure.
3. Corrosiveness and Oxidation Stability: Similar to MIL-H-6083; expected exposure to 4130 and 4140 steels, manganese and aluminum bronzes, and lead.
4. Effect on Seals: 10-15% swell in Greene Tweed Type seals (Nitrile Compound).
5. Flash Point and Autoignition Temperature: 550°F (288°C) minimum.
6. Surface Tension: Less than or equal to MIL-H-6083.
7. Volatility: Requirement to be established.
8. Shear Stability: Mechanical shear stability equal to or better than MIL-H-6083.
9. Corrosivity: Pass galvanic corrosivity test (See MIL-H-6083 specification).

10. Copper Strip Corrosion: Similar to MIL-H-6083.
11. Lubricity:
 - a. Establish best fluid-metal combination by LFW-1 (Falex Model 1 Ring and Block Test Machine) or LFW-3 (concentric ring) friction and wear tests.
 - b. Using results obtained in a., establish specification test based on 4-ball method.
12. Particulate Contamination: Same as MIL-H-6083.
13. Foaming Characteristics: Same as MIL-H-6083.
14. Abrasiveness: To be established.
15. Hydrolytic Stability: To be established, depending on type of fluid.
16. Corrosion Protection: Humidity cabinet corrosion inhibition test same as MIL-H-6083.
17. Compatibility with Other Materials: Should be compatible with other hydraulic fluids, seals, and seal lubricants.
18. Thermal Conductivity: To be established.
19. Specific Gravity: To be established; can be designed for various densities.
20. Storage Stability: Same as MIL-H-6083.
21. Compatibility with Water: Long periods of contact with water which may condense in the recoil system should not cause deleterious effects such as corrosion or sticking valves.

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