

EVALUATION OF DEGRADATION INHIBITORS IN POLY(HEXAFLUOROPROPENE OXIDE) FLUIDS

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SUMMARY

The action of various alloys: 440C steel, M-50 steel, Pyrowear 675, Cronidur 30 and Ti(4Al,4Mn); the effect of degradation inhibitors: mono- and diphospho-s-triazines, diphosphatetraazacyclooctatetraene, phosphate esters, phosphate/diester rust inhibiting mixtures, and a phosphine were evaluated in two poly(hexafluoropropene oxide) fluids (143AC and 16256). The degradation promoting action of the ferrous alloys in 16256 fluid were comparable; Ti(4Al,4Mn) alloy was significantly more detrimental. The overall rating of the additives was: phosphates > phosphate/diester mixture > phosphine > phospho-s-triazines. The 16256 fluid was less responsive to additive inhibition than 143AC. Phosphate esters were fully effective over 24 hr exposure in the 16256/440C steel and the 16256/Ti(4Al,4Mn) systems at 330 °C. In general, the phosphine was less effective in the presence of ferrous alloys than the phosphates and phospho-s-triazines.

INTRODUCTION

Perfluoropolyalkylethers as represented by a series of commercial fluids (refs. 1 to 3) and new experimental compositions (ref. 4) are a family of materials of high viscosity index and an exceptional thermal and oxidative stability. A number of investigations have assessed the thermal oxidative stability of these materials in the presence of metals, metal oxides and metal halides (refs. 5 to 11).

These studies have shown that all these materials in the presence of metals undergo degradation and cause metal corrosion, in particular at elevated temperatures and in oxidizing atmospheres. Corrosion and fluid degradation were also observed at ambient temperatures in boundary lubrication situations (refs. 12 to 13).

It is believed that under boundary lubrication conditions, high temperatures are reached at the molecular level leading to thermodynamically favored fluoride formation. These metal fluorides, in turn, promote scissions of the perfluoropoly-alkylether chains, as originally postulated by Gumprecht (ref. 1) and later substantiated by Carré (ref. 12) and others (ref. 5).

Two families of materials, phosphines and phosphorus containing heterocyclics, such as phospho-s-triazines and diphosphatetraazacyclooctatetraenes, have been identified as effective inhibitors of the degradation processes (ref. 5), (refs. 14 to 18). The initially synthesized phospho-s-triazines exhibited hydrolytic instability due to the presence of $-OCF(CF_3)$ linkage adjacent to the ring. Replacement of this group with $-OC(CF_3)_2$ moiety in monophospho-s-triazines, was accomplished (refs. 19 to 20) which alleviated this shortcoming without diminishing the corrosion and degradation arresting action. Corresponding diphospho-s-triazines and diphosphatetraazacyclooctatetraenes were also synthesized (ref. 21). Recently, new phosphate ester additives were developed and shown to represent a very effective family of degradation inhibitors (ref. 22). Thus, the objective of the investigations reported here was to evaluate the action of various alloys: 440C steel, M-50 steel, Pyrowear 675, Cronidur 30 and Ti(4Al,4Mn) and the effect of different phosphorus based degradation inhibitors on two poly(hexafluoropropene oxide) fluids at elevated temperatures in the presence of oxygen.

EXPERIMENTAL

Fluids.—Two commercially available branched poly(hexafluoropropene oxide) fluids ($C_3F_7(CF(CF_3)CF_2O)_x C_2F_5$), $C/O = 3/1$, density $24^\circ C = 1.86-1.91$ g/mL) 143AC ($100^\circ C$ viscosity, 27.3 cSt) and 16256 ($100^\circ C$ viscosity, 47.2 cSt) were studied. These fluids were characterized by 1H and ^{19}F NMR spectroscopy. Both contained very small quantities of hydrogen in the form of CF_3CFHO - groups shown by the typical doublet at 5.6 and 5.8 ppm. Based on the method of synthesis, i.e. telomerization of hexafluoropropene oxide, the only difference between the two fluids should be the number average molecular weight (M_n), which was determined by osmometry (143AC, $M_n = 5500$ and 16256, $M_n = 9900$) and the relative hydrogen content.

Alloys.—Five metal alloys were studied: four ferrous, M-50 steel, 440C steel, Pyrowear 675, Cronidur 30 and one titanium, Ti(4Al,4Mn). The alloys M-50, 440C and Ti(4Al,4Mn) were in the form of coupons with a 1.67 cm² surface area. Pyrowear 675 and Cronidur 30 were in the form of bearing balls of 9.58 and 1.50 cm² surface area, respectively.

Inhibitors.—A series of phosphorus-based inhibitors was evaluated: three hindered phospho-s-triazines:

$[C_3F_7OCF(CF_3)CF_2OC(CF_3)_2CN]_2[(C_6H_5)_2PN]$ (I), $[C_3F_7OCF(CF_3)CF_2OC(CF_3)_2CN][C_6H_5)_2PN]_2$ (II), and $[C_3F_7(OCF(CF_3)CF_2)_2OC(CF_3)_2CN][C_6H_5)_2PN]_2$ (III); a hindered 1,3-diphosphatetraazacyclooctatetraene $[C_3F_7OCF(CF_3)CF_2OC(CF_3)_2CN]_2[(C_6H_5)_2PN]_2$ (IV); a phosphine $[C_3F_7(OCF(CF_3)CF_2)_2C_6H_4]_3P$ (PH-3); four phosphates: $C_3F_7(OCF(CF_3)CF_2)_2C_6H_4OP(O)(OC_6H_5)_2$ (A), $[C_3F_7(OCF(CF_3)CF_2)_4C_6H_4O]_2P(O)OC_6H_5$ (B), $[C_3F_7(OCF(CF_3)CF_2)_3C_6H_4O]_2P(O)OC_6H_5$ (C), $C_3F_7(OCF(CF_3)CF_2)_6C_6H_4OP(O)(OC_6H_5)_2$ (V) and mixtures of C and $C_3F_7(OCF(CF_3)CF_2)_3C_6H_4OP(O)(OC_6H_5)OH$ (D-1,2,4).

Testing.—Degradation studies were performed using Pyrex tubes 420 mm long with the top 345 mm having i.d. of 22 mm and the bottom portion having i.d. of 11 to 12 mm. This permitted a small volume of liquid to cover completely the metal specimen. The tube was equipped with a ground joint which allowed the attachment of a 250 mL glass bulb and stopcock assembly for evacuation, oxygen introduction and volatile products removal at the end of the test. All the tests were carried out in pure oxygen, ~400 mm at room temperature. In a test the fluid and the metal specimen were placed into the tube, after attachment of the stopcock/gas reservoir assembly, evacuation and oxygen introduction the apparatus was closed and heated at the denoted temperature. Following cooling to room temperature the volatiles were removed *in vacuo*. The materials which condensed in liquid nitrogen cooled traps were weighed and quantified. The procedure and apparatus were fully described previously. Tests were carried out on neat fluid (3 to 4 g) at 316, 330, and 343 °C for 24 hr. The concentration of inhibitors was one weight %. The extent of degradation is calculated from the amount of volatile materials condensed by liquid nitrogen cooling and is reported as milligrams of condensable product per gram of original fluid.

RESULTS AND DISCUSSION

The major emphasis in this study was on poly(hexafluoropropene oxide) fluid 16256 and ferrous, alloys in particular 440C steel. The alloy, Ti(4Al,4Mn), was included since titanium alloys are utilized in aircraft. Thus, assessment of their action on perfluoropolyalkylethers is of importance.

The effects of the different alloys on the thermal oxidative degradation of 16256 fluid are presented in figure 1. It is clearly apparent that Ti(4Al,4Mn) is the most detrimental. At 316 °C the quantity of volatiles produced in the presence of Ti(4Al,4Mn) was found to be higher by a factor of 8 compared to 440C steel and by a factor of 4 compared to Cronidur 30 and Pyrowear 675. It is surprising that the last two alloys effected essentially the same degree of degradation since their respective surface areas differ by a factor of 6. At 330 °C the extent of fluid degradation in the presence of 440C steel, as compared to the quantity produced at 316 °C, was in a reasonably good agreement with the general rule of reaction rate doubling for every 10 °C temperature rise. The increase in the degradation extent for Pyrowear 675 and Cronidur 30 with temperature was minimal. With respect to M-50 alloy its action on fluid 16256 at 343 °C was less extensive than that of 440C steel, almost by a factor of 3. Again, the quantity of volatiles produced agreed with the reaction rate/temperature relationship.

The effectiveness of the different additives in arresting the degradation of fluid 16256 in the presence of 440C steel is displayed in figure 2. The most effective additives were found to be the phosphates C, B and V; 0.2 mg/g of volatiles formed at 330 °C. Based on our previous studies (ref. 23), the value of ≤ 0.5 mg/g represents essentially an

absence of degradation. Neither the phospho-s-triazines nor the 1,3-diphosphatetraaza-cyclooctatetraene (IV), with the possible exception of the monophospho-s-triazine (I) (0.7 mg/g), exhibited an acceptable performance even at 316 °C. On the other hand, comparison of the 330 °C results showed I to be definitely more effective than PH-3. It is noteworthy that the rust preventing mixtures, D-1 and D-2, showed degradation inhibition comparable to that of the phosphates A, B, C and V. The phosphate A, $R_fC_6H_4OP(O)(OC_6H_5)_2$, differs from phosphates B and C, $(R_fC_6H_4O)_2P(O)OC_6H_5$, in having only one of the phenoxy groups substituted by a perfluoroalkylether chain. The consequently lower content of perfluoroalkylether units resulted in A being insoluble at the 1 percent concentration at room temperature in 143AC and 16256 fluids. The additive was soluble at ~100 °C. To alleviate this shortcoming, synthesis of an A analogue, having a longer perfluoroalkylether chain, namely Compound V, was carried out. This material was soluble at the 1 percent concentration in 143AC fluid at 25 °C and in the 16256 fluid at 35 °C.

It is evident from the figure 3 data that additives C, I, II and III were fully effective in the presence of Ti(4Al,4Mn) at 316 °C in arresting the degradation of fluid 16256. As mentioned earlier, in the absence of an additive this alloy was much more detrimental than 440C steel. At 330 °C only phosphates A, C and V were fully effective in the presence of Ti(4Al,4Mn) alloy, based on the volatiles production of ≤ 0.5 mg/g. The loss of the Additive I degradation arresting action on raising temperature from 316 to 330 °C was drastic, from 0.4 mg/g to 48 mg/g.

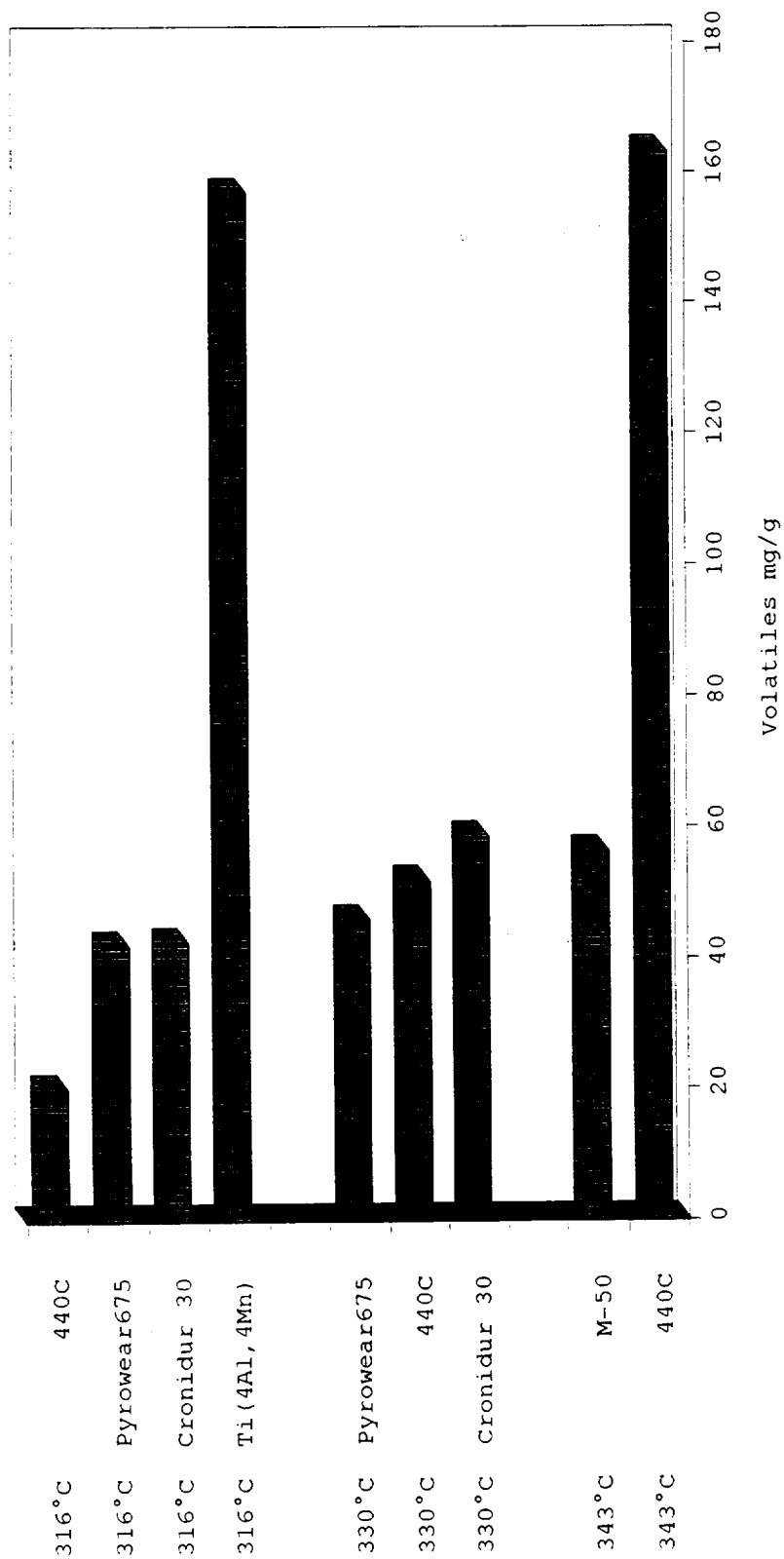
The relative effectiveness of representative additives, namely the rust and degradation inhibiting mixtures D of $[C_3F_7(OCF(CF_3)CF_2)_3C_6H_4O]_2P(O)OC_6H_5$ and $[C_3F_7(OCF(CF_3)CF_2)_3C_6H_4O]P(O)(OC_6H_5)OH$, the hindered monophospho-s-triazine I, the phosphine PH-3 and the phosphate V in arresting the degradation of fluid 16256 in the presence of the three steel alloys (440C, Pyrowear 675 and Cronidur 30) is illustrated in figure 4. It is of interest that in each instance, with the possible exception of Pyrowear 675, the order of effectiveness is essentially the same, $V \geq \text{mixture D} > I > \text{PH-3}$. In the case of 440C steel the differences are sharp; in the case of Cronidur 30 negligible. In Figure 5 are illustrated the differences in thermal oxidative behavior between the 143AC and 16256 fluids in the presence of different metals (M-50, Ti(4Al,4Mn) and 440C) and additives (monophospho-s-triazine and phosphine, PH-3). Invariably, the additives were more effective in 143AC compared to the 16256 fluid based on the volatiles production. Yet, there is virtually no difference between the 143AC and 16256 fluids in the degradation extent at 343 °C promoted by 440C steel in the absence of additives.

To summarize the studies have shown that the action of ferrous alloys on poly(hexafluoropropene oxide) fluids at elevated temperatures in oxidizing atmospheres is comparable insofar as the degradation extent is concerned. However, titanium alloys, such as Ti(4Al,4Mn), are significantly more detrimental. Regardless of the nature of the metal the phosphate esters were found to arrest the fluid degradation completely, over 24 hr in oxidizing atmospheres at 330 °C. Phosphines, phospho-s-triazines and diphosphatetraazacyclooctatetraene were less effective. The response of the two fluids, 143AC and 16256, to additives at high temperatures differed with the 16256 being invariably less responsive.

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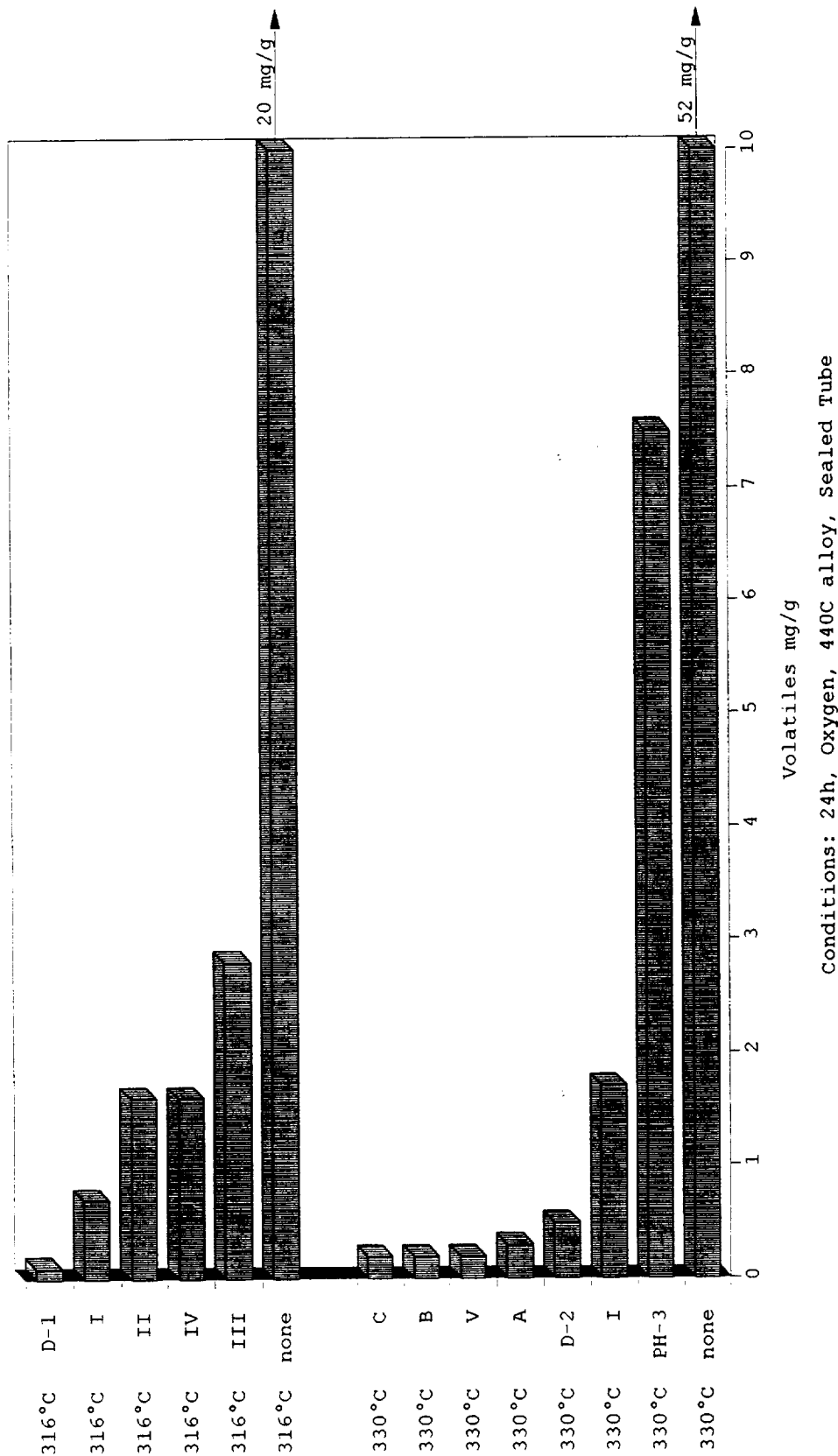
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Conditions: 24h, Oxygen, Sealed Tube

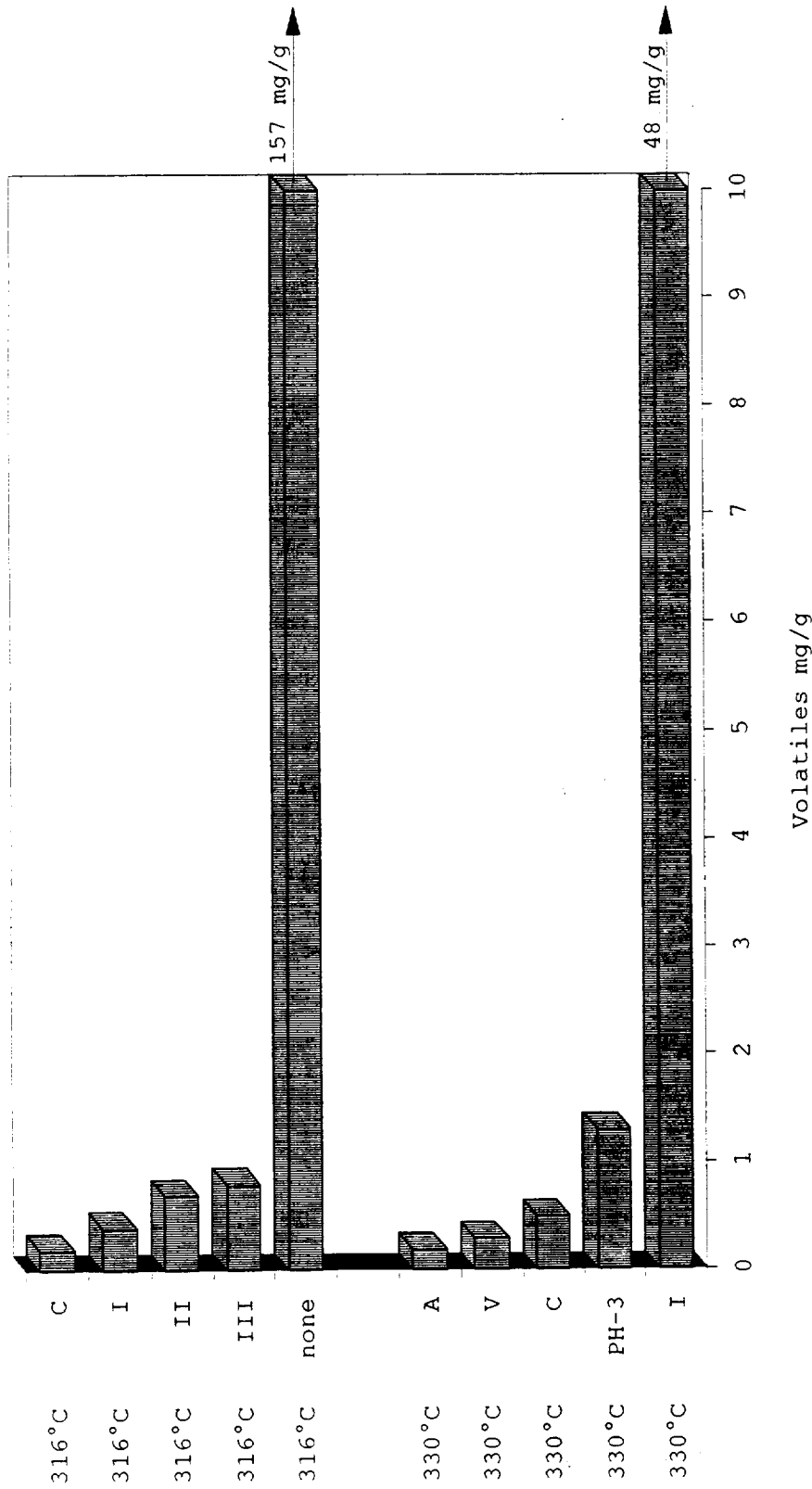
Figure 1. Comparison of the effects of different metals and temperature on the thermal stability of poly(hexafluoropropene oxide) fluid 16256 in an oxygen atmosphere.



Conditions: 24h, Oxygen, 440C alloy, Sealed Tube

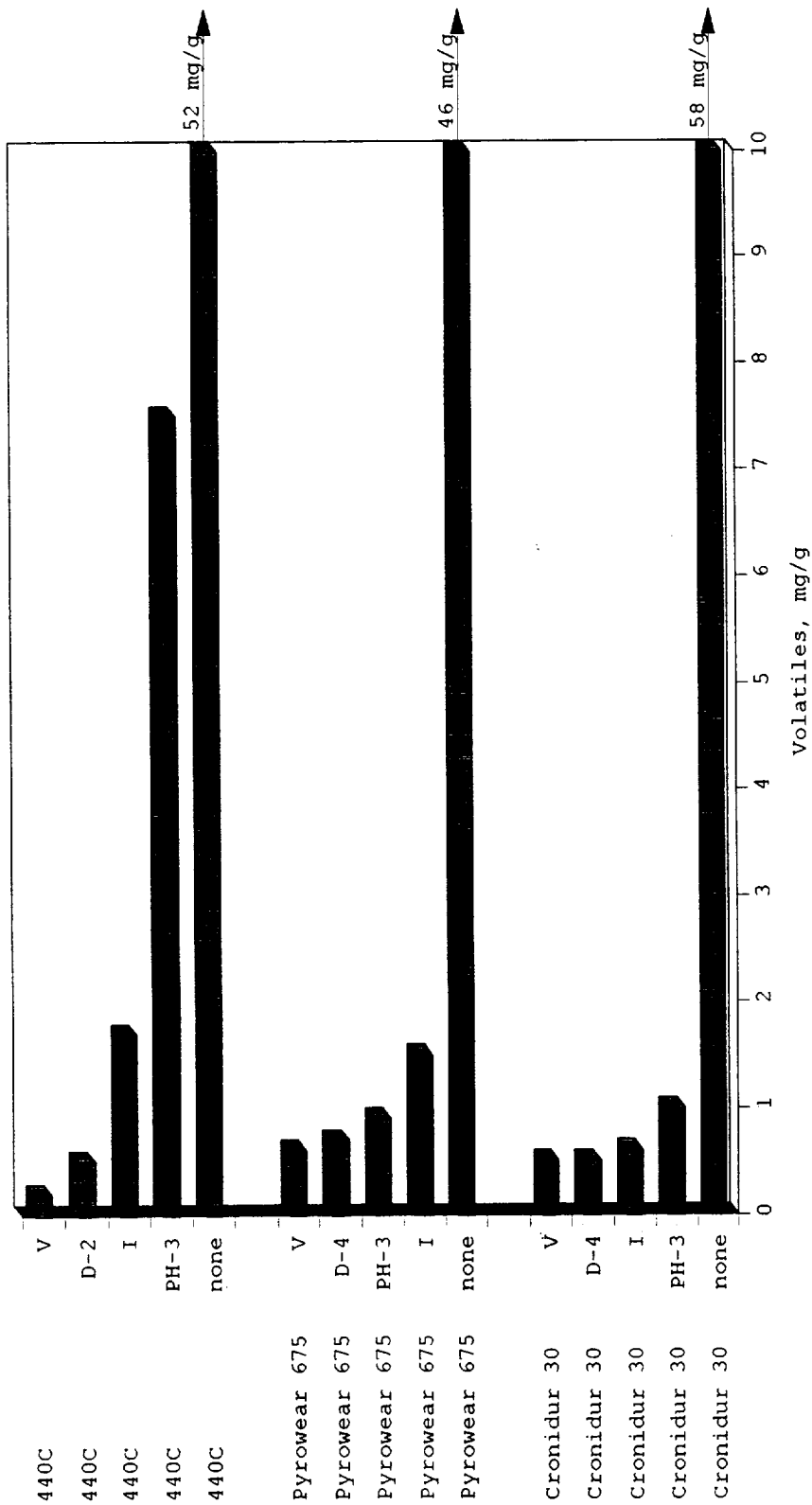
Figure 2. Comparison of the effectiveness of additives

[C₃F₇OCF(CF₃)CF₂OC(CF₃)₂CN]₂[(C₆H₅)₂PN] (I), [C₃F₇OCF(CF₃)CF₂OC(CF₃)₂CN] [(C₆H₅)₂PN]₂ (II), [C₃F₇[OCF(CF₃)CF₂]₂OC(CF₃)₂CN] [(C₆H₅)₂PN]₂ (III), [C₃F₇OCF(CF₃)CF₂OC(CF₃)₂CN]₂ [(C₆H₅)₂PN]₂ (IV), C₃F₇[OCF(CF₃)CF₂]₆C₆H₄OP(O)(OC₆H₅)₂ (V), C₃F₇[OCF(CF₃)CF₂]₂C₆H₄OP(O)(OC₆H₅)₂ (A), (C₃F₇[OCF(CF₃)CF₂]₄C₆H₄O)₂P(O)OC₆H₅ (B), (C₃F₇[OCF(CF₃)CF₂]₃C₆H₄O)₂P(O)OC₆H₅ (C), mixture of (C₃F₇[OCF(CF₃)CF₂]₃C₆H₄O)₂P(O)OC₆H₅ (C) and C₃F₇[OCF(CF₃)CF₂]₃C₆H₄OP(O)(OC₆H₅)OH (D-1, C = 61%; D-2, C = 81%) and [C₃F₇OCF(CF₃)CF₂OCF(CF₃)CF₂C₆H₄]₃P (PH-3), in arresting poly(hexafluoropropene oxide) fluid 16256 thermal oxidative degradation in the presence of 440C alloy.



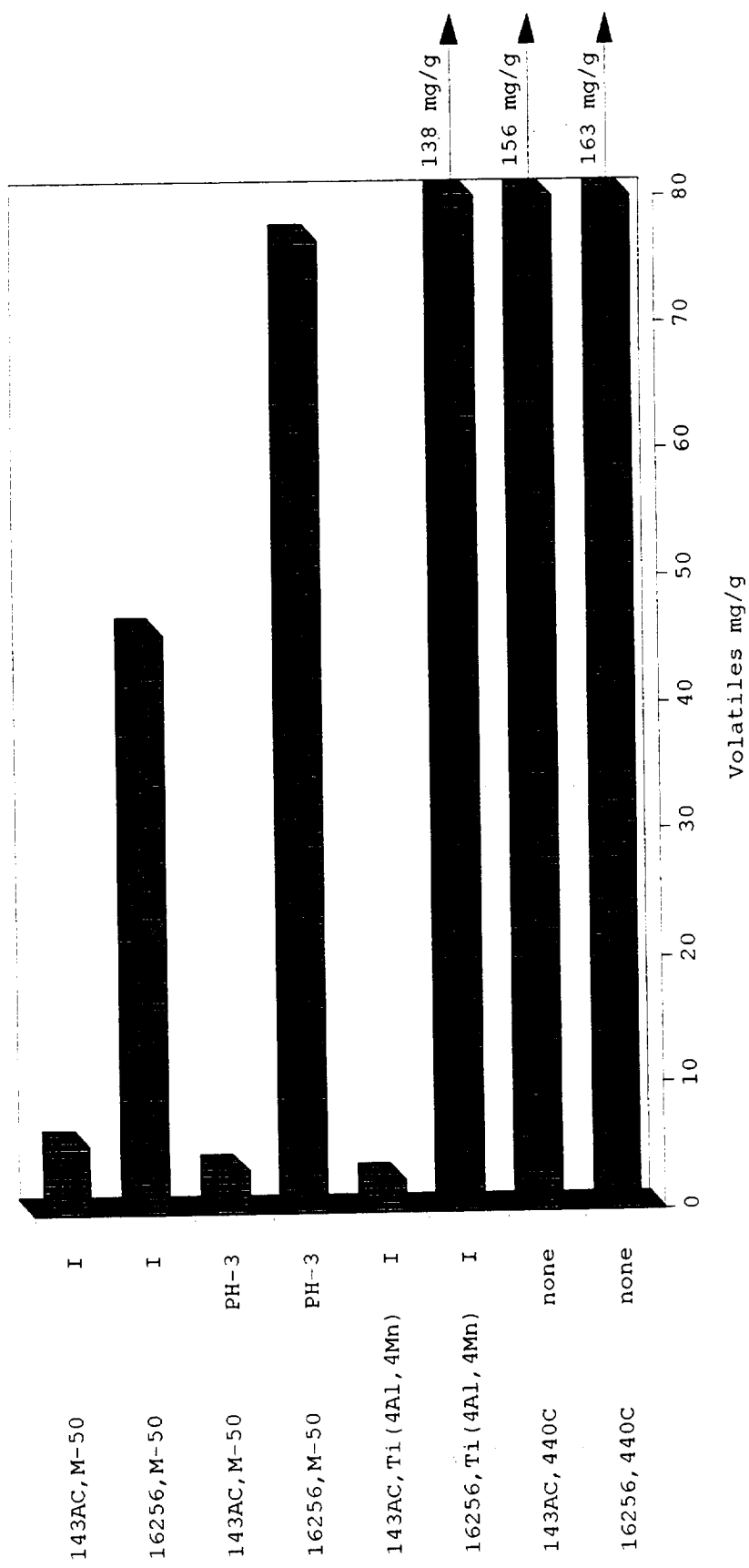
Conditions: 24h, Oxygen, Ti(4Al,4Mn) alloy, Sealed Tube

Figure 3. Comparison of the effectiveness of additives [C₃F₇OCF(CF₃)CF₂OC(CF₃)₂CN] ₂ [(C₆H₅)₂PN] (I), [C₃F₇OCF(CF₃)CF₂OC(CF₃)₂CN] [(C₆H₅)₂PN] ₂ (II), [C₃F₇O[CF(CF₃)CF₂O] ₂ C(CF₃)₂CN] [(C₆H₅)₂PN] ₂ (III), C₃F₇[OCF(CF₃)CF₂] ₆ C₆H₄OP(O)(OC₆H₅)₂ (V), C₃F₇[OCF(CF₃)CF₂] ₂ C₆H₄OP(O)(OC₆H₅)₂ (A), [C₃F₇[OCF(CF₃)CF₂] ₃ C₆H₄O] ₂ P(O)OC₆H₅ (C), and [C₃F₇OCF(CF₃)CF₂OCF(CF₃)CF₂C₆H₄] ₃ P (PH-3) in arresting poly(hexafluoropropene oxide) fluid 16256 thermal oxidative degradation in the presence of Ti(4Al,4Mn) alloy.



Conditions: 330°C, 24 h, Oxygen, Sealed Tube

Figure 4. Comparison of the effectiveness of additives [C₃F₇OCF(CF₃)CF₂OC(CF₃)₂CN]₂[(C₆H₅)₂PN] (I), C₃F₇[OCF(CF₃)CF₂]₆C₆H₄OP(O)(OC₆H₅)₂ (V), a mixture of (C₃F₇[OCF(CF₃)CF₂]₃C₆H₄O)₂P(O)OC₆H₅ (C) and C₃F₇[OCF(CF₃)CF₂]₃C₆H₄OP(O)(OC₆H₅)OH (D-2, C = 81%; D-4, C = 78%), and [C₃F₇OCF(CF₃)CF₂OCF(CF₃)CF₂C₆H₄]₃P (PH-3) in arresting poly(hexafluoropropene oxide) fluid 16256 thermal oxidative degradation in the presence of different ferrous alloys at 330°C.



Conditions: 343°C, 24 h, Oxygen, Sealed Tube

Figure 5. Comparison of the effectiveness of additives [C₃F₇OCF(CF₃)CF₂OC(CF₃)₂CN]₂[(C₆H₅)₂PN] (I) and [C₃F₇OCF(CF₃)CF₂OCF(CF₃)CF₂C₆H₄]₃P (PH-3), in arresting poly(hexafluoropropene oxide) fluids 16256 and 143AC thermal oxidative degradation in the presence of different alloys at 343°C.

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