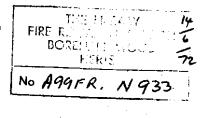


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## Fire Research Note No.933

STORAGE PROPERTIES OF FOUR FOAM LIQUIDS (Interim Report)

by

T.B. Chitty, D.J. Griffiths, D.M. Tucker and J.G. Corrie April 1972

# FIRE Research Station

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#### SUMMARY

At the request of the Defence Materials Standardisation Committee, various foam liquids have been placed in storage for an overall period of 2 years, at temperatures of  $-12^{\circ}$ C, approx.  $+15^{\circ}$ C and  $+38^{\circ}$ C. At periodic intervals, samples are being withdrawn for test on the standard fire test of UK Defence Standard 42-3, Issue 1.

In this interim report, the performances after storage for 1 year are discussed.

KEY WORDS: Extinguishing agent, tests, foam, protein, storage, fluoroprotein, fluorochemical.

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DEPARTMENT OF THE ENVIRONMENT AND FIRE OFFICES' COMMITTEE JOINT FIRE RESEARCH ORGANIZATION

FR Note No.933 April 1972

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#### INTRODUCTION

The development of new types of foam liquids for the control of hydrocarbon fires has resulted in the need for information on their storage characteristics. This note describes the first part of a study of the keeping qualities of various foam liquids when stored in dilute and concentrated form at room temperature  $(10-20^{\circ}C)$ , and at 38°C. Other samples have been subjected to repeated freezing  $(-18^{\circ}C)$  and thawing cycles. This report covers the results for the first year of storage, and six freezing cycles.

FOAM LIQUIDS USED IN THE TEST

<u>Protein</u> - a commercial sample of protein foam liquid conforming to UK Defence Standard 42-3, recommended for use at 4 per cent concentration.

<u>Fluoroprotein A</u> – a commercial sample of a protein foam liquid containing perfluorochemicals – recommended for use at 4 per cent concentration, but not recommended for storage in the diluted form.

<u>Fluoroprotein B</u> – a commercial sample of a protein foam liquid containing perfluorochemicals, from a different manufacturer. Recommended for use at 4 per cent concentration.

<u>Fluorochemical</u> - a commercial sample of a foam liquid based on perfluorochemicals. The batch used was Lot 312 - Type **194**, which has now been superseded by later developments.

#### STORAGE PROCEDURE

The foam liquids were dispersed into new 1 gallon or 5 gallon plythene containers with screw caps. The dilute solutions were prepared using potable water, a single manufacturer's batch being used in each case. One group of containers was placed in a cabinet maintained at  $38^{\circ}$ C. A second group was kept at room temperature in a room which has a heating system which maintains an appropriate temperature for active work, the temperature varying from approximately  $10^{\circ}$ C to  $20^{\circ}$ C at various times of the year. A third group of containers was subjected to freezing in a cabinet at  $-18^{\circ}$ C, followed by thawing at room temperature. Several days were necessary for each freezing and thawing cycle. In several of the initial cycles with the fluorochemical, the freezing temperature was only  $-8^{\circ}$ C, and this is indicated in the test results.

Containers were withdrawn from storage at intervals and allowed to reach room temperature before testing. When a container was withdrawn for test, any foam liquid not used was discarded, only sealed containers being kept in storage.

Discretion was used in selecting the test intervals in order to minimize the work involved; shorter intervals being chosen for the samples at 38°C and the diluted solutions, as compared with the concentrated liquids kept at room temperature.

#### EXPERIMENTAL PROCEDURE

The UK Defence Standard 42-3 fire test was used, this being an appropriate size to permit the required number of tests with the quantities of foam liquid which were stored.

This fire test employs a 0.28  $m^2$  (3 ft<sup>2</sup>) circular fire tray with narrow boiling range petrol (62-68°C) as fuel. The foam is prepared by feeding metered quantities of diluted foam liquid and air through a packed column. The liquid flow is maintained constant at 0.04  $1/m^2/s$  (0.05 gal/ft<sup>2</sup>/min). The expansion is adjusted to the required value by adjusting the air flow, and the shear stress of the foam is adjusted by varying the packing in the column. The foam is fed gently onto the fuel surface at the perimeter of the fuel tray. The base of the fuel tray is conical and is connected to a vertical graduated perspex tube which enables the drainage occurring during the test to be measured. A preburn time of 30 s is normally allowed and foam is then applied for a period of 4 minutes. The heat radiation from the fire is measured by four radiometers arranged symmetrically around the fire tray and connected in series through an amplifier to a recorder. In the Defence Standard procedure, the drainage should be recorded 10 minutes after commencing from application; this was modified to 5 minutes because some of the foams being examined were very fast draining and the measuring tube would have been overfilled in a 10 minute period.

Care was taken to wash out the apparatus thoroughly between tests of different foam liquids and it was occasionally dismantled completely to ensure that no accumulation of scale or other deposits took place.

The air flow rates and the column packings were adjusted to produce foams from each foam liquid with similar expansion and shear stresses to those obtained with a No.2 branchpipe in earlier experiments with other batches of the same foam liquids. Thereafter these expansions and column packings were maintained constant for subsequent retests. Some foam liquids change progressively after they are diluted. For instance, a sample of fluoroprotein A was diluted to 4 per cent concentration and used in the foam-making apparatus. Shear stress readings were taken over a period of 15 minutes as follows - 21.8, 18.6, 25.0, 27.6, 28.2, 29.5  $N/m^2$ . To minimize such effects, tests were progressed at as uniform a rate as possible, avoiding any long delays. If a delay occurred a new dilution was prepared.

Twenty five per cent drainage time and shear stress were determined on all foam samples using the methods described in Defence Standard 42-3.

#### METHOD OF ASSESSING DETERIORATION

If deterioration consists of the destruction of active ingredients in the foam liquid to inert materials, the effect of deterioration should be the same as that of dilution with water. Figure 1 shows the 90 per cent control times obtained with fluorochemical used at various concentrations. It can be seen from the shape of this curve that if a fire test is made with a 6 per cent solution only, 80 per cent deterioration must occur before the control time will change, and that a further small increase in deterioration will result in no control. Similar shaped curves were obtained with all the other foam liquids when tested in this manner. Clearly a test at 6 per cent concentration only, will not serve as an effective measure of deterioration. If tests are made at a range of concentrations the position of the 'heel' of the curve (Fig.1) will be a measure of the deterioration. Three measurements suggest themselves as follows:

- .1. The minimum concentration for control.
  - 2. Concentration to give 75 seconds control.
- 3. Concentration just to give minimum control time.

Number 2 is the easiest to read accurately, but No.3 has been selected as a basis for comparison because it is more meaningful - this reading is described in the report as 'minimum concentration for maximum effectiveness'. It is shown in Figure 1.

This method is based upon the premise that decay will consist of the change of identical proportions of all active ingredients to completely inactive ingredients. As will be apparent from the results presented later, deterioration does not follow such a simple pattern and in some cases the curves obtained do not have such a sharp bend as that shown in Fig.1, and the minimum concentration for maximum effectiveness cannot be estimated with great precision.

Another relevant point is that the laboratory foam generator is a more efficient foam producer than branchpipes in general use and although the minimum

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control time can be achieved with a concentration as low as  $1\frac{1}{2}$  per cent on this apparatus, it is probable that a higher concentration would be required in a branchpipe.

In spite of these limitations, the determination of the minimum concentration for maximum effectiveness is a practical and useful method for the difficult problem of quantifying deterioration. Figure 21 is a good example showing how this method reveals a very substantial degree of deterioration which would not be revealed by a single test at the recommended concentration.

#### EXPERIMENTAL RESULTS

Tables 1-4 record the data obtained at each testing period when each foam liquid was used at the recommended concentration and also the estimated 'minimum concentration for maximum effectiveness' at each testing period. These were obtained by constructing curves similar to Figure 1.

Such curves, including the other observed data, for the one year retests (or 6 freezing cycles), are shown in Figures 2-22. The data obtained at the commencement of the test is included for comparison. In the case of 4 per cent protein solutions (Figs 5 and 6) the 4 month retest data is shown, because by the following retest at 6 months, deterioration had progressed too far for data to be obtained.

Table 5 is a synopsis of the results. In this table the change in the 'minimum concentration for maximum effectiveness' is converted to a percentage deterioration value. The results for several ancilliary experimental observations are included at appropriate points in the following discussion of the results. DISCUSSION

#### Protein

The concentrates, stored at room temperature and at 38°C for one year (Figs 2 and 3), had improved control times at all concentrations. Such improvements in control time are a frequent finding and are due to the more fluid foam spreading more rapidly over the fuel surface. The fall in shear stress is associated with a reduction of the 25 per cent drainage time and an increase in fire drainage. Thus we can see that, although the control time showed an improvement, very significant changes were occurring in the protein concentrate. These changes, if continued, would eventually cause an increase in control time. The progression of changes of this nature is shown in Figure 23 using the test data for 4 per cent protein solution stored at room temperature. An improvement in

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control time, while good in itself, may therefore also be a warning of the onset of deterioration.

Protein stored as 4 per cent solution, both at room temperature and at  $38^{\circ}$ C, showed major deterioration in only 6 months. The sample stored at room temperature would not extinguish the test fire when used at 4 per cent concentration. The sample stored at  $38^{\circ}$ C just extinguished the test fire when used at 4 per cent concentration but failed to extinguish when used at  $3\frac{1}{2}$  per cent concentration. The test data obtained at the 4 months period is shown in Figures 5 and 6, and the marked differences in the drainage values and shear stresses show that changes were occurring although the control times had not greatly changed.

The freezing of protein (Figures 4 and 7), both as concentrate and as 4 per cent solution, resulted in a marked fall in the shear stress and 25 per cent drainage time and a marked rise in the fire drainage, but only in the case of the concentrate was the control time increased.

#### Fluoroprotein A

Fluoroprotein A concentrate at the end of 1 year at room temperature or at 38°C, and also after 6 freezing cycles, showed few significant changes (Figures 8, 9 and 10). One change was the reduction in control time by the concentrate (Figure 9) - but this was not associated with marked changes in the drainage values and shear stress.

The shear stress rose substantially after freezing.

Fluoroprotein A is <u>not</u> recommended by the manufacturer for storage as a premixed solution and all samples developed bacterial growth. When discarded after the first retest at 3 weeks the samples were almost black, showed some precipitation, were evolving gas, and had a pronounced offensive odour of anaerobic sewage, unlikely to be overlooked. Nevertheless, all three samples gave fire control times similar to the initial sample.

Fluoroprotein B

Fluoroprotein B, like fluoroprotein A, showed few changes when stored as concentrate for 1 year, at either temperature or after 6 freezes (Figures 11, 12, 13). In all three cases the control times improved at low concentrations, and the samples at room temperature showed a reduction of shear and 25 per cent drainage time, but this was not accompanied by an increase in the fire drainage.

When stored as 4 per cent solution for one year at room temperature and at  $38^{\circ}$ C, the control times improved at low concentrations, and this was associated with

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decreased 25 per cent drainage time, lower shear stress and increased fire drainage.

Freezing of 4 per cent solution of fluoroprotein B resulted in very little change except that the control time increased at low concentrations. This is shown in the following table.

| Test<br>concentration | 90 per cent control time - s |            |             |  |  |  |  |  |
|-----------------------|------------------------------|------------|-------------|--|--|--|--|--|
| Per cent              | Initial                      | 1 Freezing | 6 Freezings |  |  |  |  |  |
| 0.5                   | æ                            | -          | -           |  |  |  |  |  |
| 075                   | 117                          | $\infty$   | <u>∞</u>    |  |  |  |  |  |
| 1.0                   | 82                           | 83         | ø           |  |  |  |  |  |
| 1.5                   | -                            | 70         | 62          |  |  |  |  |  |
| 2.0                   | . 58                         | 60         | 68          |  |  |  |  |  |

#### Effect of freezing 4 per cent fluoroprotein B on the 90 per cent control time

#### Fluorochemical

As the storage tests proceeded, it became apparent that the fluorochemical samples were giving some anomalous results. This is illustrated in Figure 24 which shows the 90 per cent control time curves for fluorochemical concentrate after six periods of storage at 38°C.

All the similar sets of curves for the other 5 test regimes also show erratic patterns. The explanation is almost certainly that there was already a substantial variation between the manufacturer's containers of Lot 312 when the test was commenced.

An empty container of Lot 312, used in the tests, was cut open. Approximately 2 mm of light brown sludge had formed on the base, as a soft but tenacious layer. The sludge was collected and dried in an air oven to a light brown powder. The weight of dry solid was 1.4 per cent of the full drum contents. Sixty six per cent of the solid was inorganic ash. Some rusting was evident on the interior seam of the drum. Different amounts of similar sludge were observed in other containers of this batch. Because ten containers were used to prepare the storage samples a considerable number of results may be displaced by initial container variations. A careful study of all the results suggests that initial variations of up to 25 per cent in the 'minimum concentration for maximum effectiveness' may originate in container variations and we cannot draw any assured conclusions on the storage tests

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unless deterioration proceeds well beyond this point. Fluorochemical concentrate stored for one year at room temperature and at 38°C (Figures 17 and 18) both showed an increase in control time at low concentrations and a fall in the 25 per cent drainage time. The estimated deteriorations (Table 5) are 40 and 54 per cent, substantially in excess of variations attributable to the differences in decay which had occurred in the manufacturers containers before the test was commenced.

The 6 per cent solutions stored for one year at room temperature at at 38°C show even greater increases of control time at low concentrations, namely 54 and 65 per cent. In these cases the 25 per cent drainage times show a more marked fall at all concentrations and the fire drainages increased substantially.

Freezing fluorochemical concentrate or 6 per cent solution resulted in some increase of control times at the low concentrations but the fire drainages were reduced appreciably.

#### INTERIM CONCLUSIONS

1. All four foam liquids showed differences of properties after storage, indicating that changes are occurring, although in most cases, when used at the recommended concentration, the time to control the test fire did not increase.

2. Protein foam liquid when stored as a 4 per cent solution, at room temperature or at  $38^{\circ}$ C, would extinguish the test fire satisfactorily after 4 months, but would not do so after 6 months. Freezing caused a marked deterioration of the protein concentrate, but did not affect the 4 per cent solution.

3. Both fluoroproteins retained their fire suppression property well for 12 months under all regimes, but some changes of drainage values were found. This statement does not include fluoroprotein A as a premixed solution for which purpose it is not recommended by the manufacturer.

4. Evidence of variations between different drums of the same batch of fluorochemical was obtained.

5. Fluorochemical foam liquid, after storage for 12 months, under each regime, would extinguish the test fire rapidly when used at the recommended concentration of 6 per cent. When used at lower concentrations, appreciable deterioration was revealed, part of which may have occurred in the manufacturers drums before commencement of the controlled storage period. The deterioration was greatest in the sample stored as 6 per cent solution at 38°C.

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6. Decrease of 25 per cent drainage time, and increase of the fire drainage appear to be indications of deterioration which are evident before the control time is affected.

7. Storage at  $38^{\circ}$ C does not consistently accelerate deterioration as compared with storage at room temperature.

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|   | TABLE | 1                     |
|---|-------|-----------------------|
| 1 |       | and the second second |

PROTEIN - TESTS AT 4 PER CENT CONCENTRATION 0.28  $m^2(3 \text{ ft}^2)$  fires; .04 1  $m^{-2}s^{-1}(.05 \text{ gal/ft}^2/\text{min})$ 

| Material                             | Storage<br>conditions | Storage<br>period<br>days    | Test<br>No                      | Expansion            | Shear<br>stress<br>N/m <sup>2</sup>  | 25 per cent<br>drainage<br>time<br>min - s     | 75 per cent<br>control<br>time<br>s    | 90 per cent<br>control<br>time<br>s | Extinction<br>time<br>s         | 5 mín<br>fire<br>drainage<br>per cent | Minimum concentration<br>for maximum<br>effectiveness<br>per cent |
|--------------------------------------|-----------------------|------------------------------|---------------------------------|----------------------|--------------------------------------|--|--|-------------------------------------|---------------------------------|---------------------------------------|---|
| Protein<br>concen-<br>trate          | Room temp             | 0<br>131<br>186<br>369       | 199<br>351<br>409<br>515        | 8.1<br>8.2           | 19•3<br>16•0<br>12•2<br>17•3         | 3 - 24<br>3 - 20<br>2 - 00<br>2 - 55           | 60<br>53<br>55<br>50                   | 75<br>70<br>70<br>61                | 142<br>133<br>98<br>85          | 28<br>26<br>39<br>32                  | 1.3<br>1.25<br>1.25<br>1.25<br>1.25                               |
|                                      | 38 <sup>°</sup> C     | 0<br>27<br>111<br>181<br>364 | 199<br>232<br>329<br>420<br>510 | 8.2<br>- 8.3<br>8.47 | 19.3<br>19.2<br>14.7<br>18.9<br>19.2 | 3 - 24<br>2 - 07<br>2 - 30<br>2 - 52<br>2 - 40 | 60<br>54 -<br>52<br>58 -<br><u>4</u> 8 | 75<br>71<br>70<br>72<br>58          | 142<br>140<br>113<br>132<br>142 | 28<br>29<br>33<br>28<br>33            | 1.3<br>1.3<br>1.0<br>1.25<br>1.0                                  |
|                                      | -18 <sup>0</sup> C    | _0<br>6cycles                | 1 <u>99</u><br>299              |                      | 1.9•3<br>17•3                        | 3 - 24<br>3 - 05                               | 60<br>58                               | 75<br>74                            | 142<br>130                      | 28<br>33                              | 1.3<br>3.5  |
| Protein<br>4 per<br>cent<br>solution | Room temp             | 0<br>32<br>116<br>186        | 199<br>222<br>334<br>417        | 8.15<br>8.10         | 19.3<br>19.8<br>16.0<br>14.1         | 3 - 24<br>3 - 15<br>2 - 30<br>2 - 20           | 60<br>57<br>55                         | 75<br>72<br>67<br>No con            | 142<br>168<br>130<br>trol       | 28<br>33<br>39                        | 1.3<br>1.75<br>1.5<br>≻4  |
|                                      | 38°C                  | 0<br>28<br>111<br>181        | 199<br>227<br>339<br>425        | 8.2<br>7.9           | 19.3<br>19.2<br>12.8<br>16.4         | 3 - 24<br>2 - 54<br>1 - 55<br>2 - 24           | 60<br>67<br>66<br>92•5                 | 75<br>77<br>79<br>110               | 142<br>147<br>90<br>131         | 28<br>37•6<br>45<br>54                | 1.3<br>2.0<br>2.0<br><b>&gt;</b> 4                                |
|                                      | –18 <sup>°</sup> C    | 0<br>6cycles                 | 199<br>366                      |                      | 19.3<br>16.0                         | 3 - 24<br>3 - 20                               | 60<br>52                               | 75<br>69                            | 142<br>86                       | 28<br>33                              | 1.3<br>1.5  |

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FLUOROPROTEIN A - TESTS AT 4 PER CENT CONCENTRATION 0.28 m<sup>2</sup>(3 ft<sup>2</sup>) fires; .04 1 m<sup>-2</sup>s<sup>-1</sup>(.05 gal/ft<sup>2</sup>/min)

| - •  |  |                       | L-                                 |  |  |  |  |                                     | t_i                                 | <i>د</i>                               |   |   |
|------|--|-----------------------|------------------------------------|--|--|--|--|-------------------------------------|-------------------------------------|--|---|---|
|      | Material   | Storage<br>conditions | Storage<br>period<br>days          | Test<br>No.                            | Expansion  | Shear<br>stress<br>N/m <sup>2</sup>          | 25 per cent<br>drainage<br>time<br>min - s               | 75 per cent<br>control<br>time<br>s | 90 per cent<br>control<br>time<br>s | Extinction<br>time<br>s                | 5 min<br>Efire,<br>drainage<br>per cent | Minimum concentration<br>for maximum<br>effectiveness<br>per cent |
|      | Fluoro-<br>protein<br>A.<br>concen-<br>trate               | Room temp             | 0<br>68<br>192<br>369              | 124<br>241<br>371<br>500               | 8.0<br>8.4<br>8.1<br>8.0                             | 21.8<br>25.8<br>29.0<br>22.4                 | 2 - 57<br>3 - 12<br>3 - 50<br>3 - 00                     | 80<br>75<br>75<br>73                | 97<br>97•5<br>93<br>93              | 187<br>181<br>131<br>193               | 28<br>24<br>25<br>28                    | 1.75<br>2.25<br>1.50<br>2.0                                       |
| 1    |  | 38 <sup>0</sup> С     | 0<br>32<br>48<br>116<br>192<br>370 | 124<br>188<br>206<br>280<br>376<br>505 | 8.0<br>9.0<br>8.2<br>8.2<br>8.2<br>8.2<br>8.2<br>8.2 | 21.8<br>23.0<br>25.6<br>15.3<br>25.0<br>21.8 | 2 - 57<br>3 - 57<br>3 - 54<br>2 - 40<br>3 - 40<br>3 - 05 | 80<br>87<br>84<br>60<br>80<br>67    | 97<br>108<br>102<br>80<br>100<br>83 | 187<br>188<br>167<br>147<br>168<br>144 | 28<br>21:<br>26<br>31<br>25<br>28       | 1.75<br>1.9<br>1.75<br>1.75<br>1.50<br>2.0                        |
| -10- |  | –18 <sup>°</sup> C    | 0<br>1cýcle<br>2cycles<br>6cycles  |  | 8.0<br>8.7<br>7.8.<br>8.2                            | 21.8<br>24.2<br>25.2<br>30.4                 | 2 - 57<br>3 - 55<br>3 - 43<br>2 - 30                     | 80<br>84<br>77•5<br>69              | 97<br>97<br>97•5<br>95              | 187<br>162<br>205<br>175               | 28<br>22<br>29<br>28                    | 1.75<br>1.75<br>1.5<br>1.6  |
|      | Fluoro-<br>protein<br>A.<br>6 per<br>cent<br>solut-<br>ion | Room temp             | 0<br>25                            | 124<br>156                             | 8.0<br>8.3   | 21.8<br>23.7                                 | 2 - 57<br>3 - 00   | 80<br>67.5                          | 97<br>84                            | 187<br>150                             | 28<br>                                  | 1.75<br>1.90  |
|      | ··· -  | 38°C                  | 0<br>21                            | 124<br>152                             | 8.0<br>7.8   | 21.8<br>16.6                                 | 2 - 57<br>2 - 40   | 80<br>59                            | 97<br>73                            | 187<br>115                             | 28<br>39                                | 1.75<br>2.0   |
|      |  | -18 <sup>°</sup> C ,  | 0<br>1cycle                        | 124<br>162                             | 8.0<br>8.2   | 21.8<br>22.4                                 | 2 - 57<br>3 - 16   | 80<br>67•5                          | 97<br>91                            | . 187<br>108                           | 28<br>30                                | 1.75<br>2.0   |

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TABLE 3

|                                    | 3 – TESTS AT 4 PER CEN             |                      |
|------------------------------------|------------------------------------|----------------------|
| $0.28 \text{ m}^2(3 \text{ ft}^2)$ | fires; $.04 \ 1 \ m^{-2} s^{-1}$ ( | $(.05 gal/ft^2/min)$ |
|                                    | <u></u>                            |                      |

|  |                       |                                    |  |   |   | 7=====7  |                                     |                                     |                                  |                                       |   |
|--|-----------------------|------------------------------------|--|---|---|--|-------------------------------------|-------------------------------------|----------------------------------|---------------------------------------|---|
| Material   | Storage<br>conditions | Storage<br>period<br>days          | Test<br>No•                            | Expansion                               | Sheår<br>stress<br>N/m <sup>2</sup>       | 25 per cent<br>drainage<br>time<br>min - s               | 75 per cent<br>control<br>time<br>s | 90 per cent<br>control<br>time<br>s | Extinction<br>time<br>s          | 5 min<br>fíre<br>drainage<br>per cent |   |
| Fluoro-<br>protein<br>B<br>concen-<br>trate          | Room temp             | 0<br>55<br>196<br>367              | 216<br>269<br>427<br>530               | 8.1<br>8.2<br>8.5<br>8.2                | 7•7<br>7.05<br>7.05<br>5•5                | 2 - 40<br>2 - 38<br>3 - 00<br>2 - 30                     | 42<br>47<br>36<br>42                | 54<br>. 62<br>45<br>60              | 84<br>95<br>46<br>69             | 35<br>41<br>37<br>34                  | 2.0<br>1.5<br>1.25<br>1.5                   |
| - · · · · · · · · · · · · · · · · · · ·              | 38°c                  | 0<br>14<br>55<br>105<br>203<br>367 | 216<br>257<br>275<br>356<br>434<br>535 | 8.1<br>8.35<br>8.0<br>8.1<br>7.6<br>8.2 | 7•7<br>7•3<br>5•75<br>7•05<br>6•4<br>5•55 | 2 - 40<br>2 - 45<br>2 - 30<br>3 - 15<br>3 - 00<br>2 - 32 | 42<br>45<br>46<br>47<br>45<br>45    | 54<br>61<br>60<br>64<br>59<br>58    | 84<br>92<br>97<br>73<br>77<br>66 | 35<br>35<br>38<br>31<br>32<br>34      | 2.0<br>1.75<br>1.70<br>1.50<br>1.25<br>1.25 |
|  | –18 <sup>°</sup> C    | 0<br>1cycle<br>6cycles             | 216<br>245<br>347                      | 8.1<br>8.6<br>8.4                       | 7.7<br>9.0<br>6.4                         | 2 - 40<br>2 - 50<br>3 - 00                               | 42<br>41<br>49                      | 54<br>57•5<br>60                    | 84<br>102•5<br>82                | 35<br>31<br>31                        | 2.0<br>; 1.5<br>1.2                         |
| Fluoro-<br>protein<br>B<br>4 per<br>cent<br>solution | Room temp             | 0<br>10<br>86<br>204<br>366        | 216<br>245<br>342<br>439<br>520        | 8.1<br>8.1<br>8.4<br>7.9<br>8.1         | 7.7<br>7.7<br>6.4<br>7.0<br>3.35          | 2 - 40<br>2 - 38<br>2 - 35<br>2 - 55<br>2 - 05           | 42<br>41<br>49<br>44<br>49          | 54<br>55<br>61<br>60<br>62          | 84<br>92•5<br>77<br>75<br>92     | 35<br>40.6<br>36<br>36<br>41          | 2.0<br>2.25<br>1.4<br>1.25<br>1.25          |
|  | 38°C                  | 0<br>15<br>54<br>112<br>203<br>367 | 216<br>263<br>290<br>361<br>444<br>525 | 8.1<br>8.15<br>8.1<br>8.7<br>7.8<br>7.5 | 7.7<br>8.5<br>7.05<br>7.7<br>7.0<br>6.1   | 2 - 40<br>2 - 38<br>1 - 45<br>3 - 00<br>2 - 45<br>2 - 34 | 42<br>44<br>48<br>43<br>40<br>46    | 54<br>57•5<br>63<br>62<br>55<br>60  | 84<br>91<br>93<br>69<br>66<br>90 | 35<br>35<br>48<br>29<br>39<br>38      | 2.0<br>1.3<br>1.8<br>1.5<br>1.25<br>1.25    |
|  | –18 <sup>°</sup> C    | 0<br>1cycle<br>6cycles             | 216<br>251<br>295                      | 8.1<br>8.6<br>8.5                       | 7•7<br>8•7<br>7•0                         | 2 - 40<br>2 - 50<br>2 - 40                               | 42<br>45<br>40                      | 54<br>63<br>62                      | 84<br>92•5<br>97                 | 35<br>35<br>30                        | 2.0<br>1.85<br>1.50                         |

TABLE 4

F.R.Note No.933

FLUOROCHEMICAL - TESTS AT 6 per cent CONCENTRATION 0.28  $m^2(3 \text{ ft}^2)$  fires; 0.04 1  $m^{-2}s^{-1}(0.05 \text{ gal/ft}^2/\text{min})$ 

|      | Material   | Storage<br>conditions           | Storage<br>period<br>days                       | Test<br>No                                   | Expansion                                    | Shear<br>stress<br>N/m <sup>2</sup>    | 25 per cent<br>drainage<br>time<br>min - s   | 75 per cent<br>control<br>time<br>s |                                  | Extinction<br>time<br>s            | 5 min<br>fire<br>drainage<br>per cent  |   |
|------|--|---------------------------------|---|--|--|--|--|-------------------------------------|----------------------------------|------------------------------------|--|---|
| ×    | Fluoro-<br>chemical<br>concen-<br>trate<br>312               | Room Temp                       | 197   | 1–3<br>81<br>183<br>317<br>355               | 11.0<br>12.2<br>11.6<br>13.5<br>12.6         | 5.0<br>5.7<br>6.4<br>5.2<br>5.75       | 2 - 37<br>2 - 36<br>2 - 40<br>3 - 00<br>2 - 20   | 31<br>31<br>34<br>30<br>29          | 44<br>41<br>45<br>37<br>41       | 63<br>54<br>150<br>163<br>59       | 35<br>32<br>28<br>27<br>32             | 1.5<br>2.0<br>1.75<br>1.75<br>2.5         |
|      | •<br>•.•<br>•  | 38°C                            | 0<br>11<br>. 36<br>87<br>190<br>361             | 1–3<br>54<br>87<br>172<br>307<br>351         | 11.0<br>12.6<br>12.8<br>12.3<br>12.4<br>11.6 | 5.0<br>5.1<br>5.1<br>5.8<br>5.1<br>4.5 | $\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$   | 31<br>32<br>31<br>35<br>30<br>30    | 44<br>44<br>41<br>47<br>39<br>45 | 63<br>60<br>139<br>120<br>47<br>62 | 35<br>26<br>28<br>28<br>28<br>28<br>45 | 1.5<br>2.0<br>2.0<br>2.5<br>1.9<br>3.25   |
| -12- |  | 8 <sup>°</sup> C<br>8°C<br>18°C | 0<br>1cycle<br>4cycles<br>4 <del>;</del> &ycles |  | 11.0<br>12.3<br>12.1<br>13.75                | 5.0<br>5.1<br>4.2<br>5.1               | 2 - 37<br>2 - 28<br>2 - 06<br>2 - 25   | 31<br>27•5<br>28<br>29              | 44<br>38<br>36<br>38             | 63<br>47<br>44<br>47•5             | 35<br>26<br>36<br>25                   | 1.5<br>2.5<br>3.0<br>2.75                 |
|      | Fluoro-<br>chemical<br>312<br>6 per<br>cent<br>solu-<br>tion | Room temp                       | 0<br>35<br>86<br>197<br>367                     | 1-3<br>75<br>178<br>303<br>360               | 11.0<br>12.4<br>12.7<br>11.7<br>11.8         | 5.0<br>5.0<br>4.5<br>5.1<br>4.5        | 2 - 37<br>1 - 56<br>2 - 40<br>2 - 25<br>2 - 0  | 31<br>30<br>31<br>28<br>28          | 44<br>47•5<br>44<br>37<br>35     | 63<br>58<br>57<br>128<br>130       | 35<br>37<br>28<br>33<br>42             | 1.5<br>2.25<br>2.9<br>2.25<br>3.25        |
|      |  | 38°C                            | 0<br>11<br>35<br>87<br>198<br>365               | 1–3<br>58<br>93<br>167<br>322<br><b>4</b> 44 | 11.0<br>12.4<br>12.4<br>11.5<br>12.1<br>10.8 | 5.0<br>5.1<br>4.1<br>4.5<br>3.2<br>4.5 | 2 - 37 2 - 30 2 - 05 2 - 05 2 - 05 2 - 10 1 - 50   | 31<br>27<br>32<br>31<br>29<br>27    | 44<br>39<br>48<br>44<br>44<br>37 | 63<br>50<br>177<br>76<br>127<br>75 | 35<br>30.5<br>39<br>36<br>37<br>49     | 1.5<br>2.75<br>3.25<br>2.5<br>3.0<br>4.25 |
|      |  | -8°C<br>-8°C<br>-18°C           | 0<br>1cycle<br>3cycle<br>3+6<br>cycles          |  | 11.0<br>12.6<br>12.7<br>14.1                 | 5.0<br>5.7<br>5.1<br>5.1               | $\begin{array}{cccc} 2 & - & 37 \\ 2 & - & 28 \\ 2 & -^{\circ}17 \\ 2 & - & 40 \\ \end{array}$ | 31<br>30<br>-<br>28                 | 44<br>46<br>-<br>37              | 63<br>56<br>-<br>150+              | 35<br>31<br><br>26.5                   | 1.5<br>2.0<br>2.75<br>2.0                 |

#### TABLE 5

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#### SYNOPSIS OF TESTS AFTER 1 YEAR

|                                      | · .                   | ,               | Control time of                                    | observations                  | n an the second s | Estimated deterioration                                   |  |
|--------------------------------------|-----------------------|-----------------|--|-------------------------------|---|---|--|
| Material                             | Storage<br>conditions | Storage<br>time | At recommended<br>concentration<br>4 or 6 per cent | At lower                      | Other observations  | from 90 per cent<br>control time x<br>concentration curve |  |
| · · · · ·                            |                       | Days/cycles     |  |                               |   | ,   |  |
| Protein<br>concen-<br>trate          | Room temp.            | 369             | Fell 14 s  | Marked fall                   | Shear fell, 25 per cent drainage<br>time marked fall, fire drain<br>slight increase   | Nil (improved)  |  |
|                                      | 38°C                  | 364             | Fell 17 s  | Marked fall                   | 25 per cent drainage time marked fall, fire drain increased   | Nil (improved)  |  |
|                                      | -18°C                 | 6 cycles        | No change  | Marked increase               | Shear fell, 25 per cent drainage<br>time fell, fire drain increased   | 60 per cent   |  |
| Protein<br>4 per<br>cent<br>solution | Room temp.            | 186             | No control   | No control                    | Shear fell, 25 per cent drainage<br>time fell   | >62 per cent  |  |
|                                      | 38°C                  | 181             | Increased 35 s                                     | No control at<br>3.5 per cent | Shear fell, 25 per cent drainage<br>time fell, fire drain increased   | ≻62 per cent  |  |
| ·                                    | -18°C                 | 6 cycles        | No change  | No change                     | Shear fell, 25 per cent drainage<br>time fell, fire drain increased   | Nil   |  |
| Fluoro-<br>protein A<br>concen-      | Room temp.            | 369             | No change  | No marked<br>change           | No marked changes   | 12 <del>1</del> per cent                                  |  |
| trate                                | 38°C                  | 370             | Fell 14 s  | Marked fall                   | Higher shear at low concentrations  | 12½ per cent  |  |
|                                      | -18°C                 | 6 cycles        | No change  | Slight fall                   | Shear increased, 25 per cent<br>drainage time fell  | Nil (slightly improved)                                   |  |

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### Table 5 (cont'd)

|      |  |                       |                                | Control time of                                    | observations               |   | Estimated deterioration                                   |  |
|------|--|-----------------------|--------------------------------|--|----------------------------|---|---|--|
|      | Material                                       | Storage<br>conditions | Storage<br>time<br>Days/cycles | At recommended<br>concentration<br>4 or 6 per cent | At lower<br>concentrations | Other observations  | from 90 per cent<br>control time x<br>concentration curve |  |
|      |  | Room temp.            | 25                             | Fell 13 s  | ζ                          |   |   |  |
| ļ    | protein A<br>4 percent                         | 38°C                  | 21                             | Fell 14 s  |                            | nded for predilution-bacterial<br>decomposition occurred  | · · · ·   |  |
|      | solution                                       | -18 <sup>0</sup> C    | 1 cycle                        | No change  | )                          | •   | · · · · · · · · · · · · · · · · · · ·                     |  |
|      | Fluoro-<br>protein B<br>concen-<br>trate       | Room temp.            | 367                            | No change  | Fell .                     | Shear fell, 25 per cent drainage<br>time fell, fire drain fell<br>slightly                                | Nil (improved)  |  |
| -14- |  | 38 <sup>0</sup> C     | 367                            | No change  | Fell at<br>low conc.       | No other changes  | Nil (improved)  |  |
|      |  | -18 <sup>0</sup> C    | 6 cycles                       | No change  | Increased at<br>low conc.  | 25 per cent drainage time<br>increased, fire drain decreased  | Nil (improved)  |  |
|      | Fluoro-<br>protein B<br>4 per cent<br>solution | Room temp.            | 366                            | Increased 8 s                                      | Fell at<br>low conc.       | 25 per cent drainage time fell<br>markedly. Shear showed marked<br>fall and fire drain marked<br>increase | Nil (improved)  |  |
|      |  | -38°C                 | 367                            | Increased<br>slightly                              | • No large<br>change       | 25 per cent drainage time fell,<br>fire drain increased   | Nil (improved)  |  |
|      |  | -18°C                 | .6 cycles                      | Increased 8 s                                      | Increased                  | No other changes  | Nil (improved)  |  |

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F.R.Note No.933

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|   |                       |            | Control time of  | observations              |   | Estimated deterioration                                   |  |
|---|-----------------------|------------|--|---------------------------|---|---|--|
| Material                                | Storage<br>conditions |            | At recommended<br>concentration<br>4 or 6 per cent<br>Concentrations |                           | Other observations  | from 90 per cent<br>control time x<br>concentration curve |  |
| Fluoro-<br>chemical<br>concen-<br>trate | Room Temp.            | 367        | No change  |                           | 25 per cent drainage time fell.<br>Fire drain increased at low<br>conc.         | 40 per cent   |  |
|   | 38°C                  | 361        | No change  | Increased at<br>low conc. | 25 per cent drainage time fell  | 54 per cent   |  |
|   | -8°C/-18°C            | 3+6 cycles | No change  | Increased at<br>low conc. | Fire drain fell   | 45 per cent   |  |
| Fluoro-<br>chemical<br>6 per<br>cent    | Room temp.            | 367        | Fell 9 s   | Increased at<br>low conc. | 25 per cent drainage time fell<br>markedly. Fire drain increased<br>markedly    | 54 per cent   |  |
| solution                                | 38°C                  | 365        | Fell 7 s   | Marked<br>increase        | 25 per cent drainage time fell<br>markedly and fire drain<br>increased markedly | 65 per cent   |  |
|   | -8°C/-18°C            | 3+6 cycles | Fell 7 s   | Increased                 | Fire drain fell   | 25 per cent   |  |

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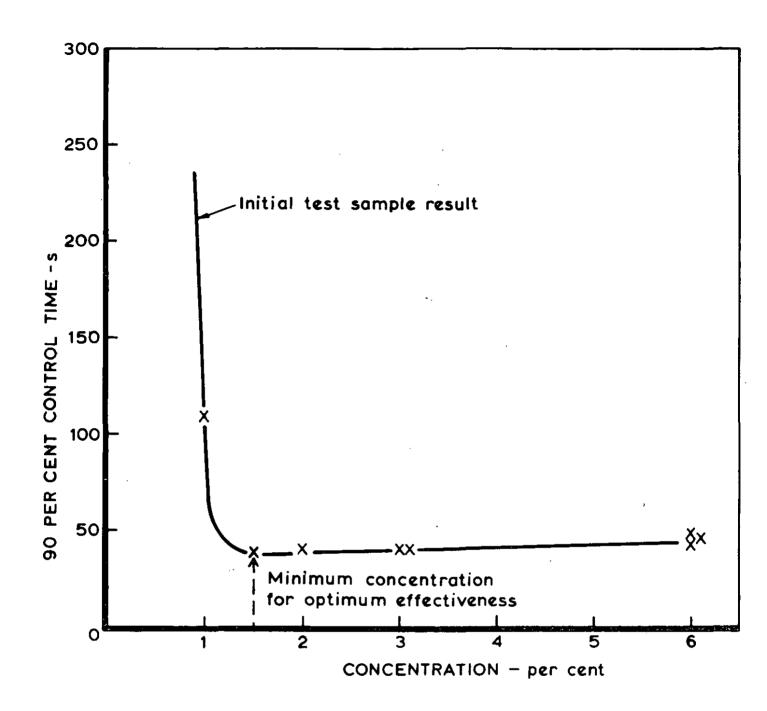


FIG.1 FLUOROCHEMICAL - EFFECT OF CONCENTRATION ON 90 PER CENT CONTROL TIME

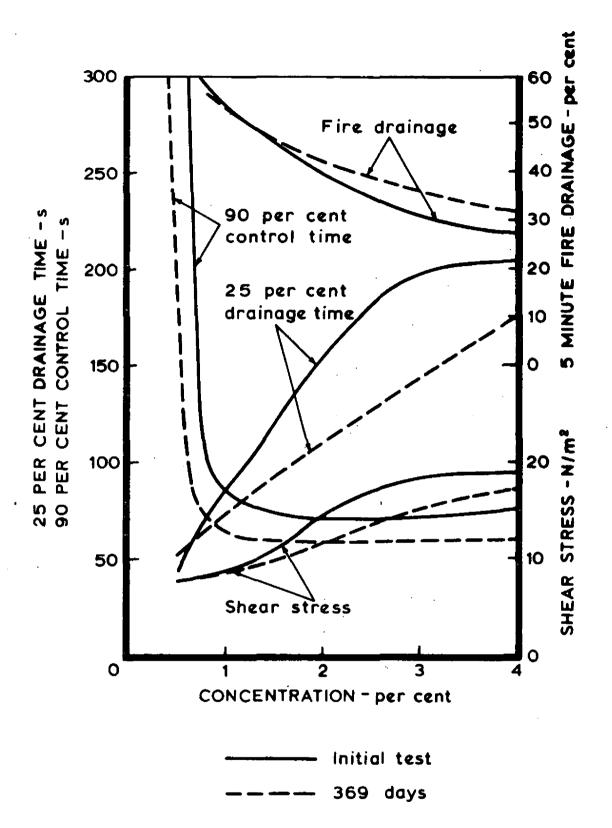
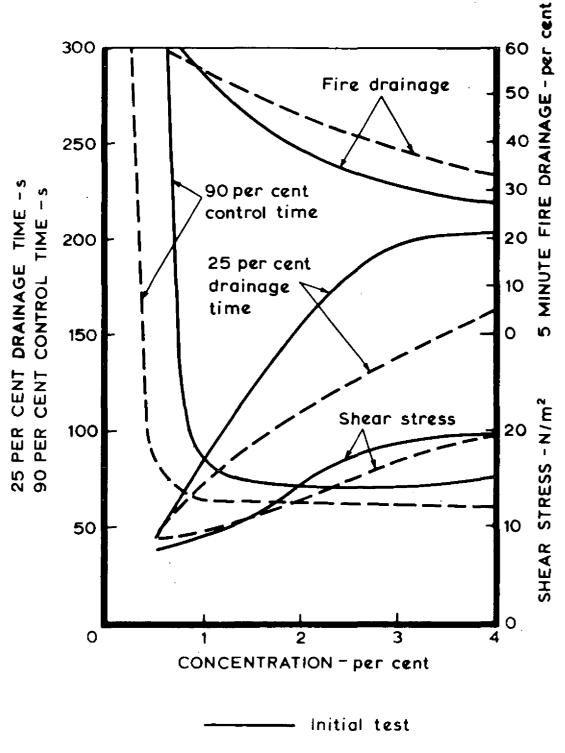


FIG.2 PROTEIN CONCENTRATE STORED FOR 369 DAYS AT ROOM TEMPERATURE



——— 364 days

FIG.3 PROTEIN CONCENTRATE STORED FOR 364 DAYS AT 38°C

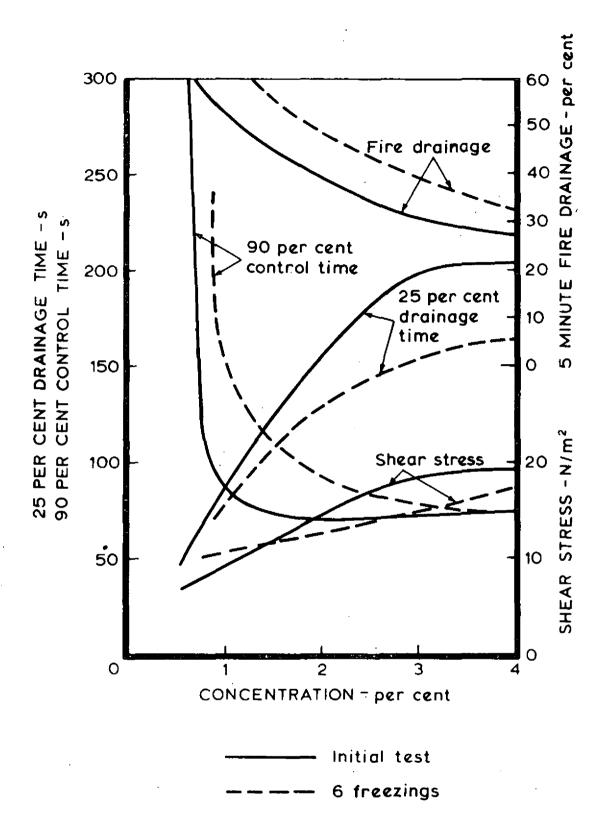


FIG. 4 PROTEIN CONCENTRATE - AFTER 6 FREEZING CYCLES AT -18°C

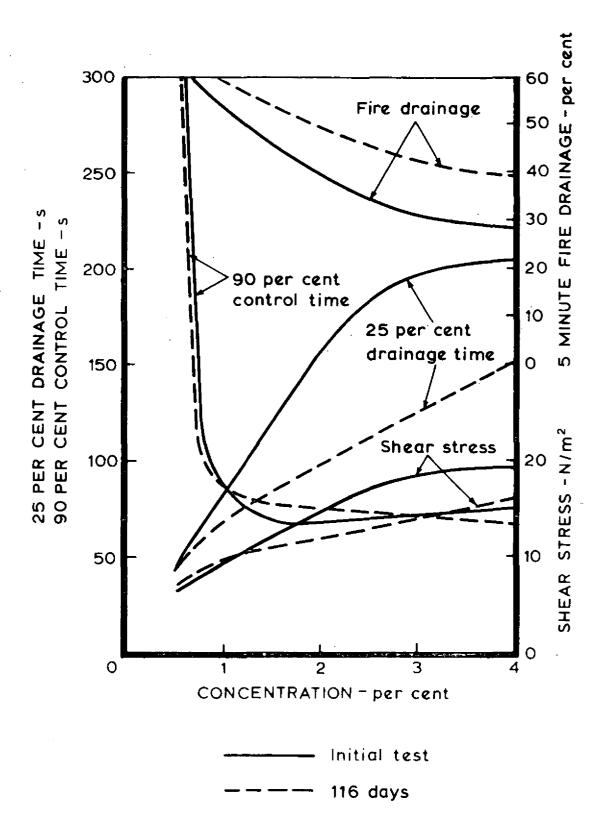


FIG. 5 PROTEIN-4 PER CENT SOLUTION STORED FOR 116 DAYS AT ROOM TEMPERATURE

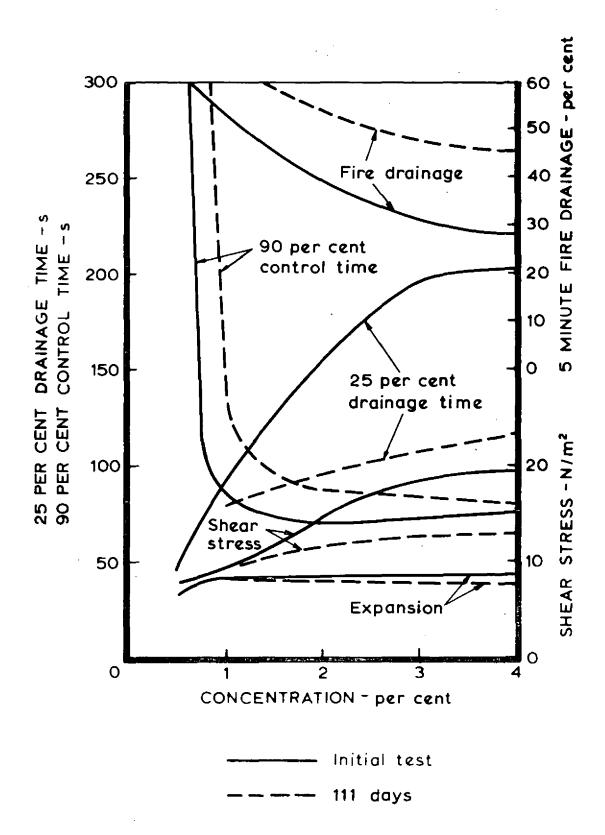


FIG. 6 PROTEIN-4 PER CENT SOLUTION FOR 111 DAYS AT 38°C

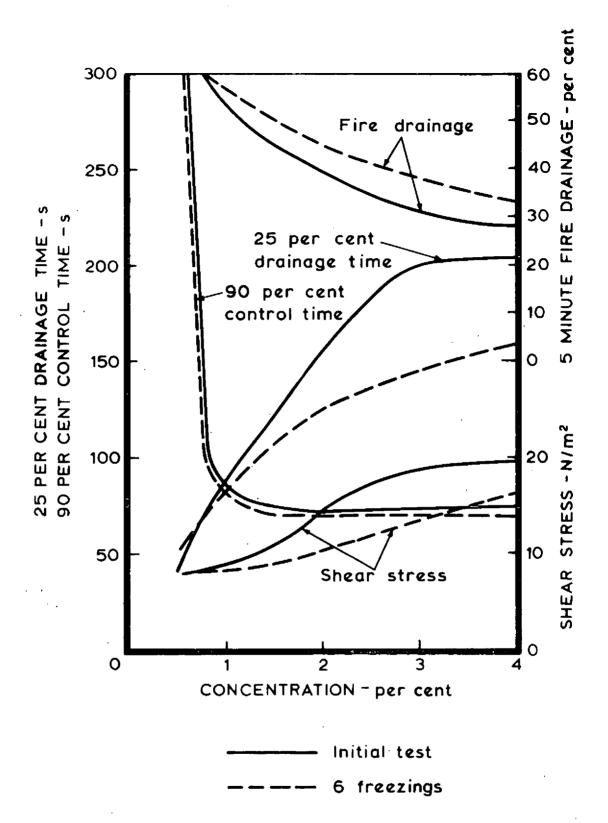


FIG.7 PROTEIN -4 PER CENT SOLUTION AFTER 6 FREEZING CYCLES AT - 18°C

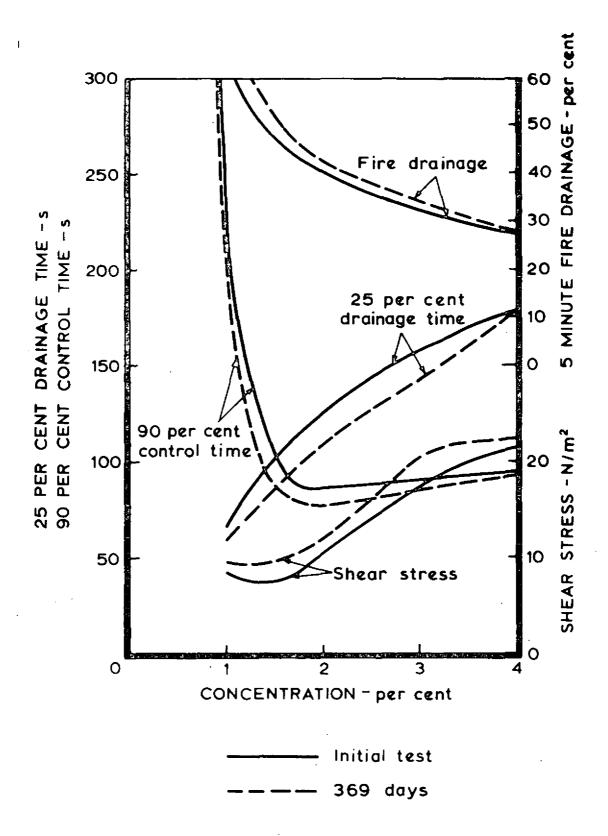


FIG.8 FLUOROPROTEIN 'A' CONCENTRATE STORED FOR 369 DAYS AT ROOM TEMPERATURE

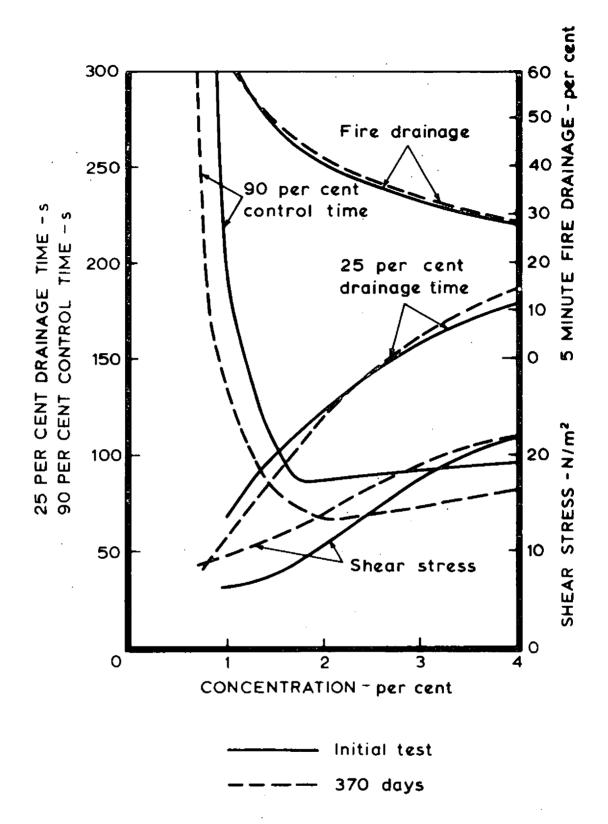


FIG. 9 FLUOROPROTEIN 'A' CONCENTRATE STORED FOR 370 DAYS AT 38°C

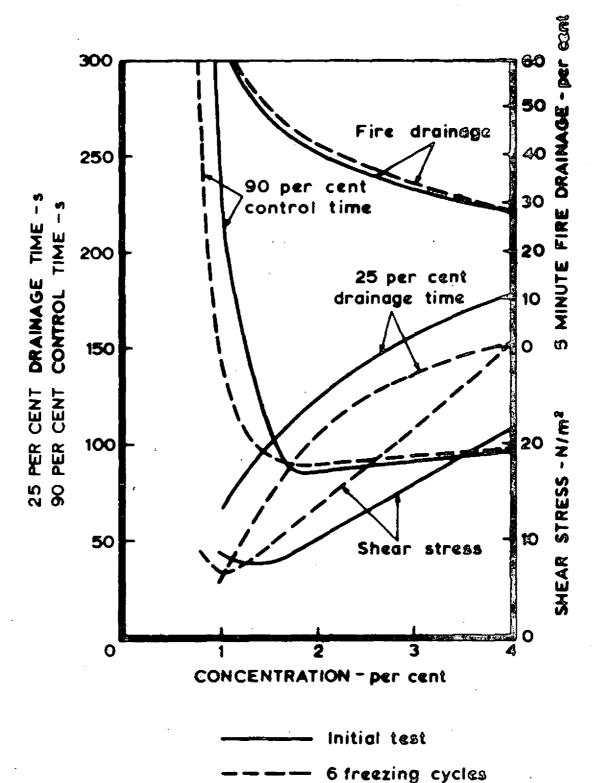


FIG. 10 FLUOROPROTEIN 'A' CONCENTRATE AFTER 6 FREEZING CYCLES AT -18°C

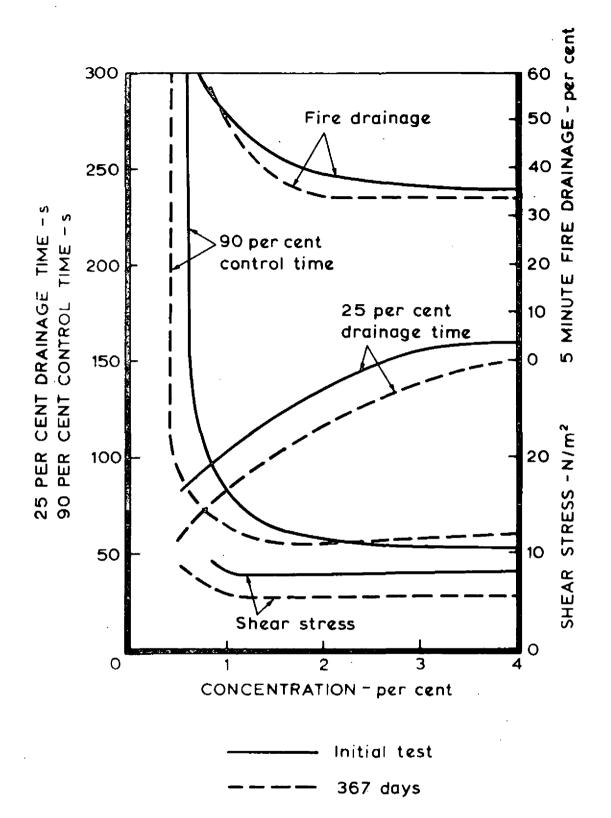
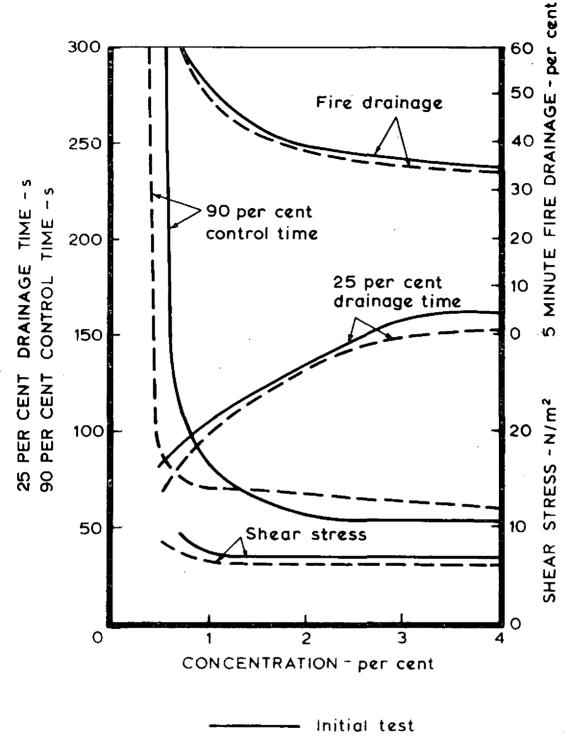


FIG 11 FLUOROPROTEIN 'B' CONCENTRATE STORED FOR 367 DAYS AT ROOM TEMPERATURE



----- 367 days

FIG. 12 FLUOROPROTEIN 'B' CONCENTRATE STORED FOR 367 DAYS AT 38°C

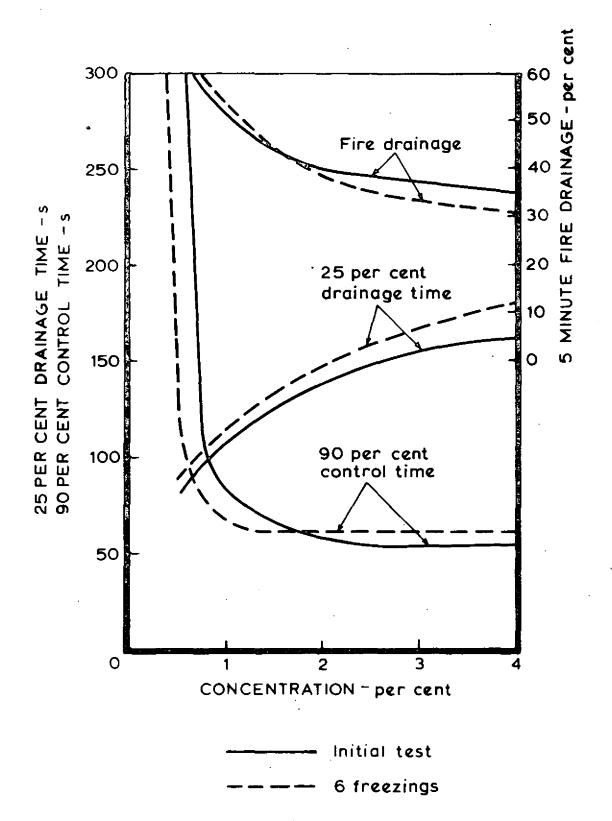


FIG 13 FLUOROPROTEIN 'B' CONCENTRATE AFTER 6 FREEZING CYCLES AT -18°C

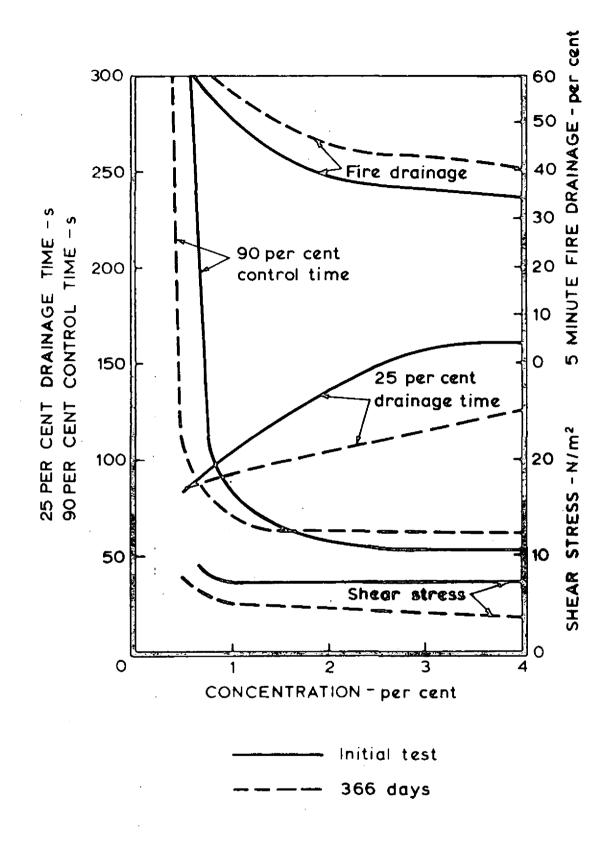


FIG.14 FLUOROPROTEIN 'B' - 4 PER CENT SOLUTION STORED FOR 366 DAYS AT ROOM TEMPERATURE

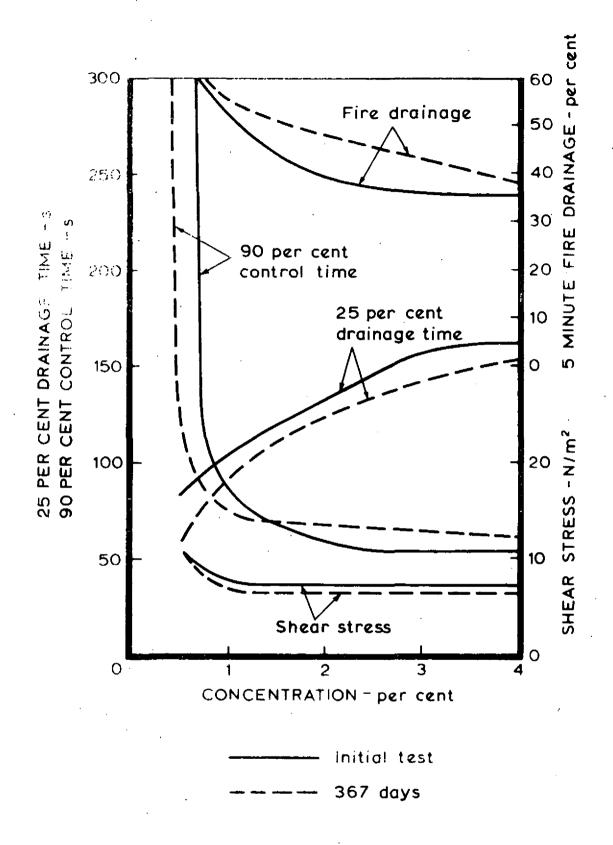


FIG.15 FLUOROPROTEIN B'-4 PER CENT SOLUTION STORED FOR 367 DAYS AT 38°C

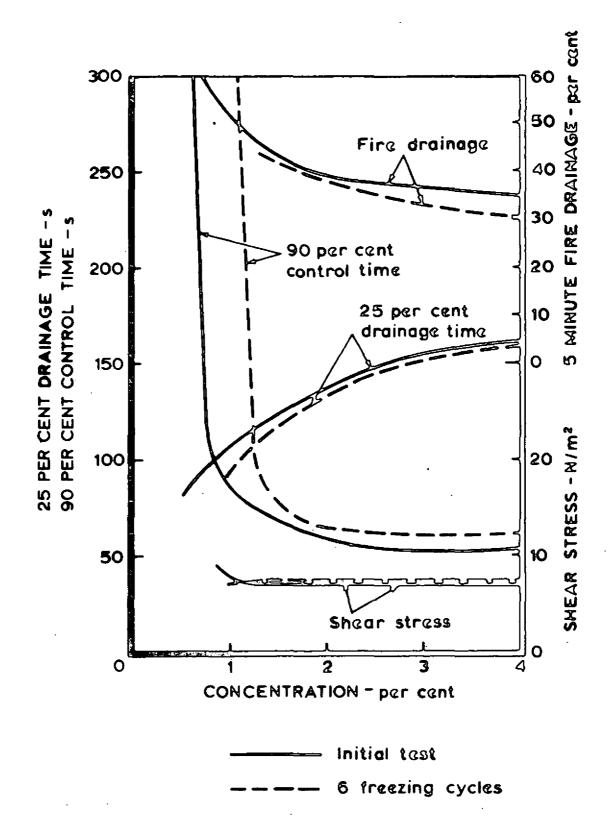


FIG. 16 FLUOROPROTEIN 'B'-4 PER CENT SOLUTION AFTER 6 FREEZING CYCLES AT -18°C

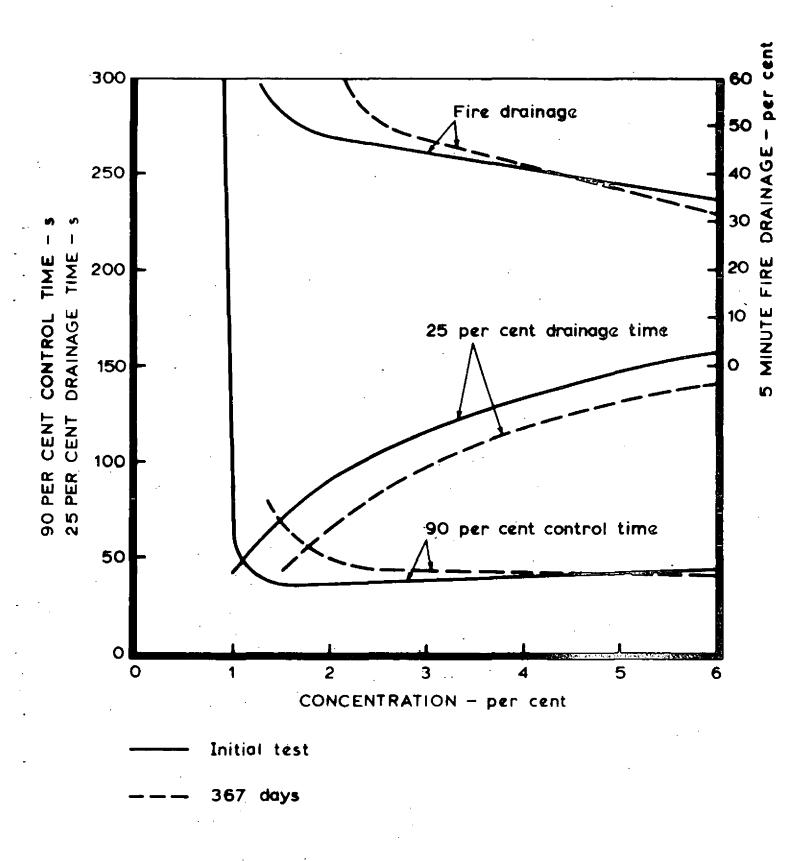
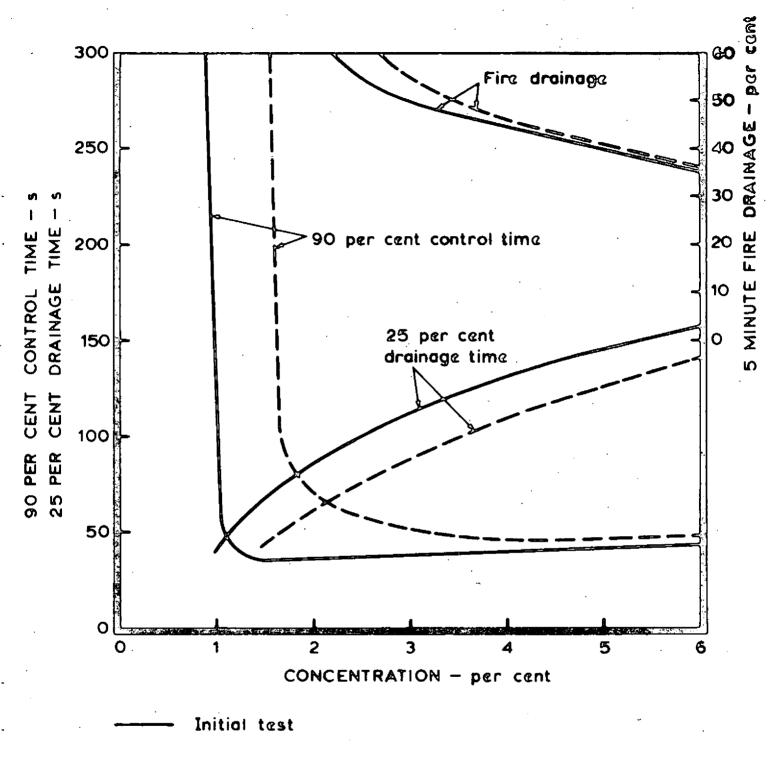


FIG. 17 FLUOROCHEMICAL CONCENTRATE STORED FOR 367 DAYS AT ROOM TEMPERATURE



-- 361 days

FIG. 18 FLUOROCHEMICAL CONCENTRATE STORED FOR 361 DAYS AT 38°C

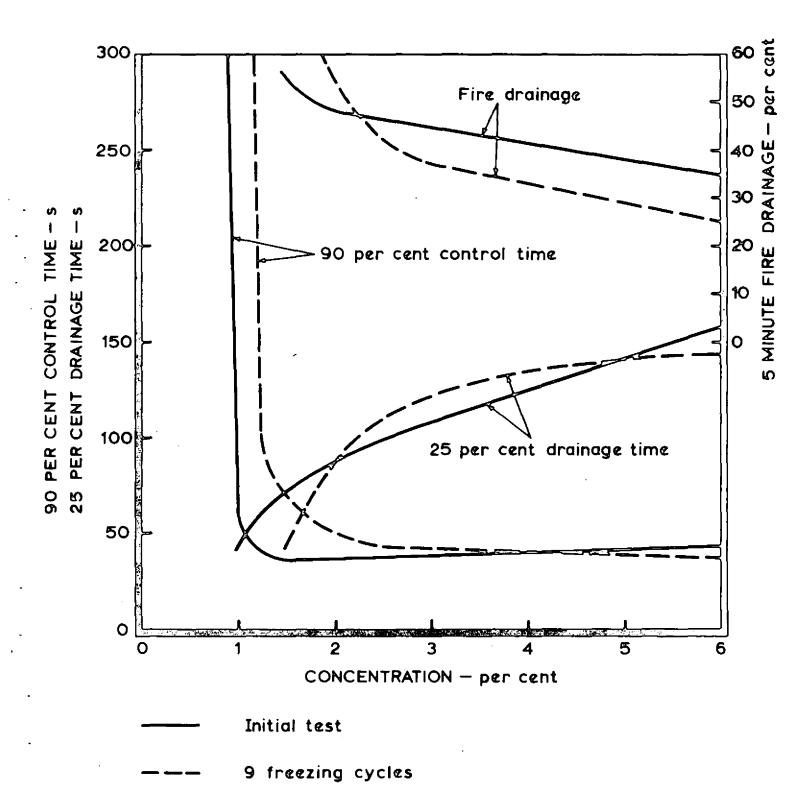


FIG 19 FLUOROCHEMICAL CONCENTRATE AFTER 3 FREEZING CYCLES AT -8°C AND 6 CYCLES AT -18°C

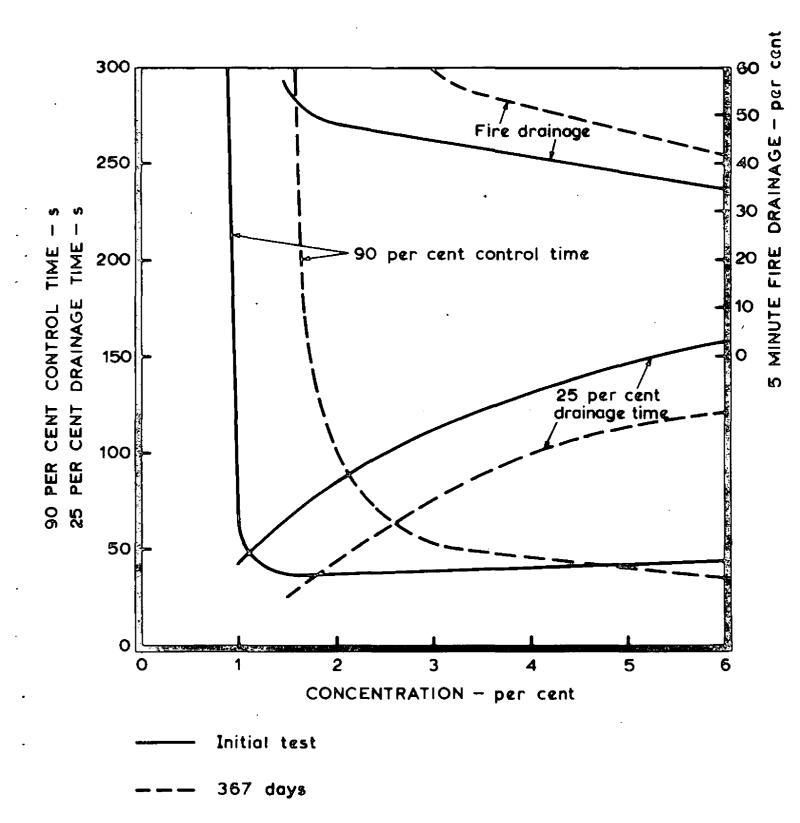


FIG. 20 FLUOROCHEMICAL-6 PER CENT SOLUTION STORED FOR 367 DAYS AT ROOM TEMPERATURE

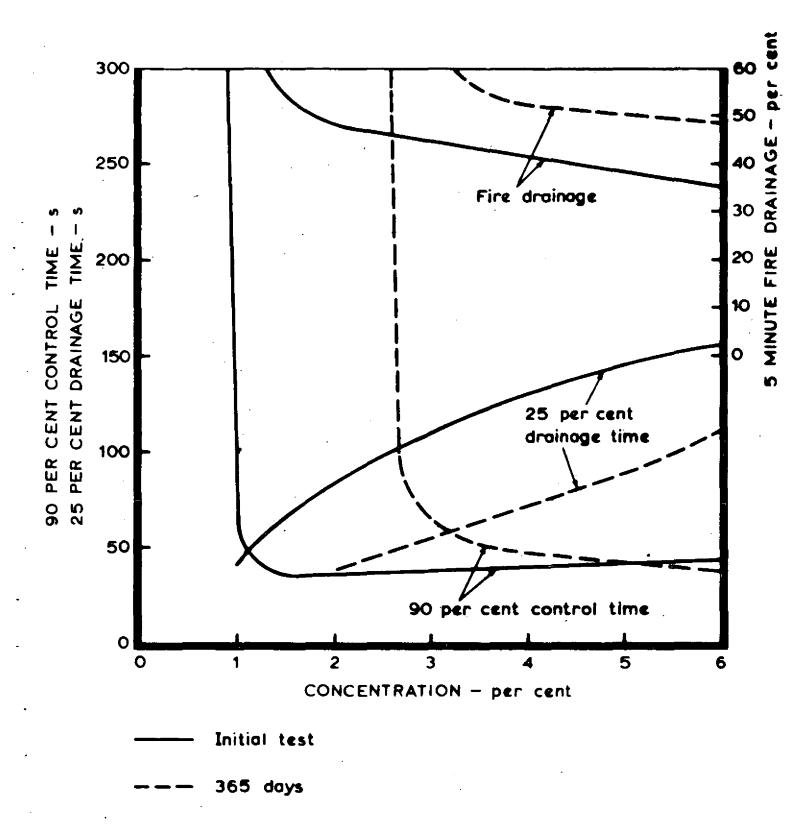


FIG. 21 FLUOROCHEMICAL - 6 PER CENT SOLUTION STORED FOR 365 DAYS AT 38°C

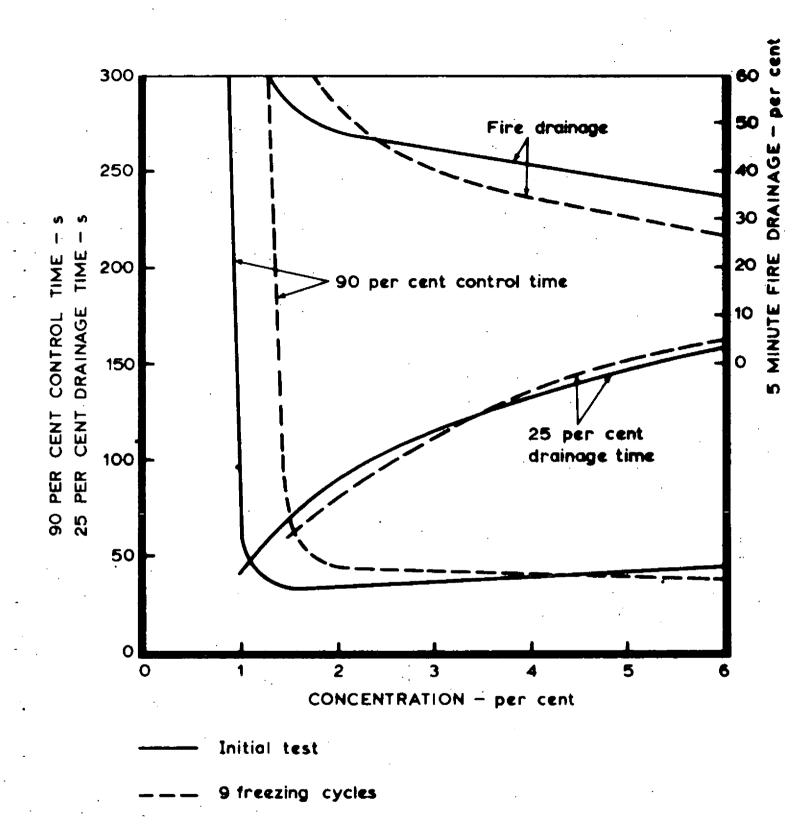


FIG. 22 FLUOROCHEMICAL - 6 PER CENT SOLUTION AFTER 3 FREEZING CYCLES AT -8°C AND 6 CYCLES AT -18°C

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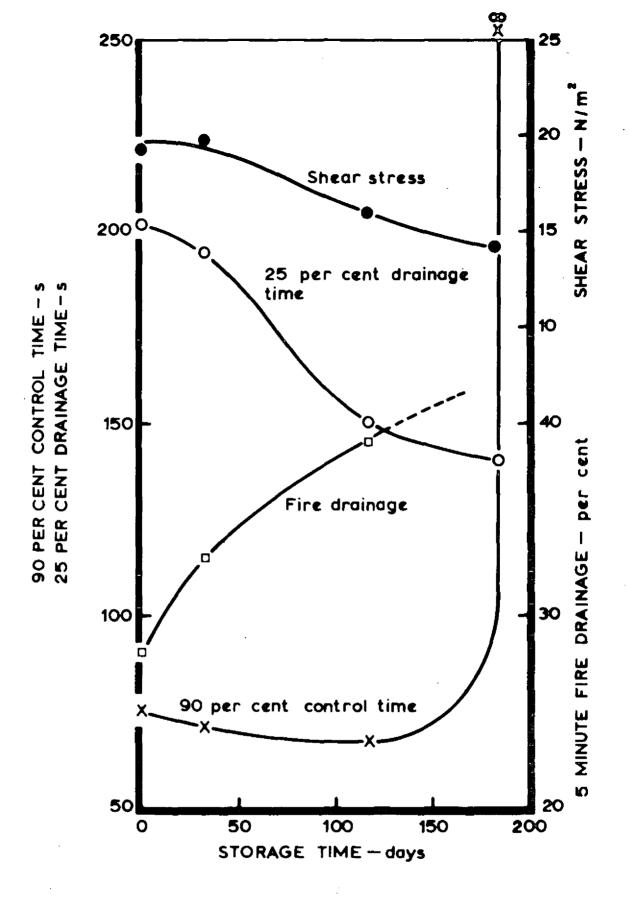
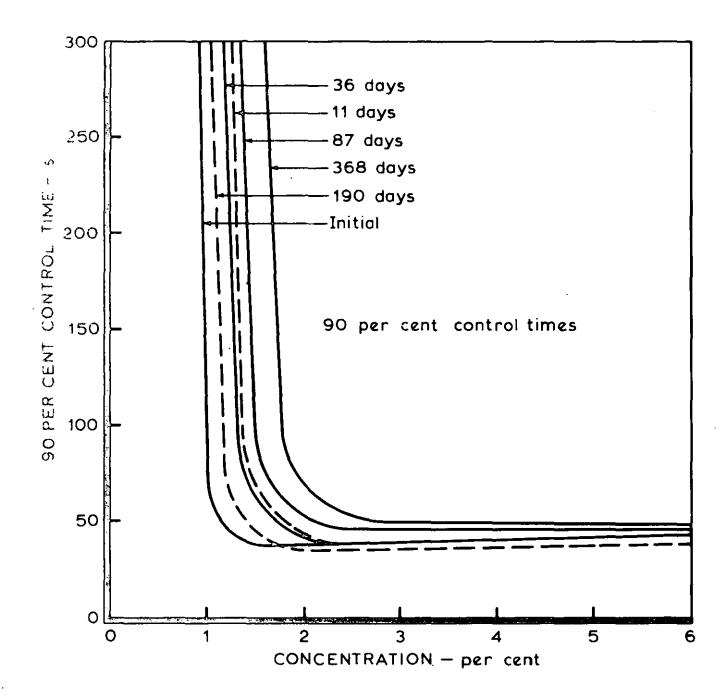


FIG. 23 CHANGES IN PROTEIN STORED AS 4 PER CENT SOLUTION AT ROOM TEMPERATURE AND TESTED AT 4 PER CENT CONCENTRATION



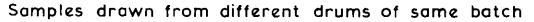


FIG. 24 FLUOROCHEMICAL CONCENTRATE – 90 PER CENT CONTROL CURVES AFTER STORAGE AT 38°C

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