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AAEC/E26

AUSTRALIAN ATOMIC ENERGY COMMISSION

DILUENTS FOR THE SOLVENT EXTRACTION OF THORIUM USING TRIBUTYL PHOSPHATE GENERAL CONSIDERATION AND THE THIRD PHASE PROBLEM

by

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Summary

An examination of three possible diluents available from Australian sources of supply, has shown that 'odourless mineral spirits' from Vacuum Oil Co. Pty. Ltd., is the purest in alkane content, and is the most suitable of the three for use with TBP for the chemical processing of irradiated fuel, for example by the Thorex process. A small quantity of sec-octyl alcohol will suppress third phase formation, but further work is required to ensure that its use is otherwise compatible with the process.

September, 1958

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1. INTRODUCTION

The properties which affect the choice of a diluent for the solvent extraction of irradiated fuels are as follows:–

(i) Density: The density must be sufficiently higher or lower than the water phase to give clean separations at all times.

(ii) Viscosity: The viscosity should be low. The density difference – viscosity relationship (Stokes factor) is important (2).

(iii) Flash–point: The flash–point should be high enough to ensure low fire risk (1, 3).

(iv) Chemical Stability: The diluent must be chemically stable in the various process streams (3, 4).

(v) Toxicity: The toxicity of the diluent should be as low as possible.

(vi) Radiation Stability: The diluent must be sufficiently stable to the radiation expected in the process (5) and under irradiation must not form any compound which may be deleterious (6).

(vii) Effect on Partition Coefficient: The diluent or any additive may adversely affect the partition coefficients of the cations to be separated. U.S. specifications for diluents are given in the appendix.

(viii) Phase Stability: Only two phases should be present at any stage of the process (1). The formation of a third phase will cause difficulties in the operation of solvent extraction equipment.

Aromatic and unsaturated hydrocarbon diluents are considered to be unsatisfactory because of low flash–point (1,3) chemical instability, (3,4,7) and radiation instability (5). Carbon tetrachloride has too high a density if the organic material is to be the disperse phase but is otherwise suitable. However there is some radiolysis of CCl_4 (10) giving amongst other compounds free chlorine. This could cause corrosion problems in plant constructed of the usual 18/8/1 welding–quality stainless steel.

For reasons of cost commercial paraffinic diluents are used and these generally contain various kinds and quantities of impurities such as aromatic and unsaturated compounds. The impurities may cause trouble in the form of lower decontamination factors, crud formation, and by acting as surface–active agents. Thus commercial paraffinic diluents normally require pretreatment (7,8,9). From the point of view of the above requirements pure paraffin type diluents (alkanes) are satisfactory (3,5) with the exception that tetravalent metals will produce third–phase formation in this type of diluent.

2. THE THIRD–PHASE PROBLEM

2.1 General Survey. A large amount of work has been done in the U.S.A. on the choice of a suitable diluent for solvent extraction of irradiated fuels (3,11,12,14). Third–phase formation during the solvent extraction of tetravalent metals has been reported on by J.E.Savolainen (13), Schulz and Voiland (14) and by workers at ORNL (1). Both Savolainen and Schulz report that third phase formation depends on thorium concentration, acidity, and temperature; Schulz (14) reports that there was no third phase formation with CCl_4. Savolainen (13) and Gresky (1) report the complete solubility of the third phase in benzene. Chesne and Regnaut (15) showed that the addition of aromatic hydrocarbons to an alkane diluent prevented third–phase formation when processing irradiated thorium.

In the U.K., McKay (16) and Healy and McKay (17) obtained a third phase when extracting thorium with T.B.P. in an odourless kerosene diluent. McKay (16) states that third-phase formation is a function of aqueous phase acidity, thorium concentration, and temperature. McKay also points out that third-phase formation reaches a maximum with 20–25% odourless kerosene diluent. He gives as a possible explanation, the slight solubility of the thorium-TBP solvate in the diluent in the case of the low % TBP solvent, and in the TBP in the case of the high % TBP solvent. McKay (16, 17) infers that the third phase is a TBP solvate which is insoluble in the kerosene diluent, and shows that this is a common phenomenon to all nitrates of the 4^{+} elements investigated, viz:– Th^{4+}, Zr^{4+}, Ce^{4+}, Np^{4+}, and Pu^{4+}. Hudswell (18) reports that zirconium nitrate forms a third phase upon extraction into a TBP/decalin solvent. No third phase was formed when extracting $Zr(NO_3)_4$ with TBP when using as a diluent benzene, xylene, chloroform, or carbon tetrachloride (18).

The use of additives to the diluent to suppress third-phase formation was also studied at ORNL (1). Such additives as methyl cyclohexane, hexane, and benzene were used.

2.2 <u>Third-phase Formation:</u> Savolainen's Theory.

Savolainen (13) states that Th^{4+} has a co-ordination number of 8, and that the primary complex $Th(NO_3)_4.4TBP$ can exist as a cubic structure. He then states that dimerization occurs when two primary complexes share two nitrate ions at adjoining corners, liberating two TBP molecules. This process of polymerization can continue producing the infinite polymer $Th(NO_3)_4.2TBP$. These polymers have strong van der Waals forces in a paraffin hydrocarbon. These forces lead to condensation of the polymer thus giving a dense organic phase. Savolainen further explains the effect of aromatics in the solvent as being due to a dipole induced in the aromatic ring by the electrostatic nature of the Van der Waals force of the thorium nitrate TBP polymer. With this induced dipole, aromatics will solvate the polymer chain, thus making them more compatible with the paraffin hydrocarbon solvent.

2.3 <u>The choice of a diluent with respect to third phase formation:</u>

It is apparent that the formation of a condensed TBP – solvate insoluble in a paraffinic hydrocarbon diluent is a normal phenomenon in the extraction of tetravalent elements (16,13,14,1). This third phase would cause operating difficulties in the operation of solvent extraction equipment(1). This problem may be overcome in various ways:–

(i) decreasing the concentration of the thorium (or other tetravalent ion) in the aqueous phase, or by increasing the solvent-to-feed ratio. This action will decrease the volumetric efficiency of the plant;

(ii) the use of a pure aromatic diluent or of an aromatic containing diluent. This raises other problems of chemical and radiation stability and is further discussed below;

(iii) the use of a pure paraffinic hydrocarbon (alkane) diluent containing small quantities of a saturated additive to suppress third-phase formation;

(iv) the use of other suitable diluents such as CCl_4 (14) that do not form a third phase;

(v) operation of the solvent extraction plant at a sufficiently high temperature to prevent third-phase formation.

From the available information it appears that non-alkane diluents can cause serious operating difficulty, due in the first place to radiolysis (5,6). Even with alkane diluents some trouble is caused by radiolysis. At Savannah River a test (Zirconium Index Test) was devised to check that degradation products of the radiolysis of the kerosene diluent had been removed in the solvent recovery cycle prior to reuse (19).

This report deals with the problem of the "third–phase formation" during the TBP extraction of thorium when using three paraffinic diluents (kerosenes) which are available in Australia. These are:–

(i) Avtur (Aviation turbine) fuel from Caltex Pty. Ltd.

(ii) Odourless Kerosene – From Caltex Pty. Ltd.

(iii) Odourless Mineral Spirits from Vacuum Oil Co. Pty. Ltd.

3. EXPERIMENTAL

3.1 Reagents

Thorium Nitrate: Two sources of supply were used

(i) "Analar" $Th(NO_3)_4 . 4H_2O$ and

(ii) "Riedel–De–Haen A.G."

The salt analyzed as 97.5% $Th(NO_3)_4 . 4H_2O$

Nitric Acid: "Ajax" A.R. S.G. 1.42

Tributyl Phosphate: "Albright and Wilson" Technical grade.
The whole solvent was washed with $M/2$ Na_2CO_3, 7M HNO_3, M/2 Na_2CO_3 and demineralized water.

Diluents:

(i) Aviation Turbine Fuel: This may contain up to 20% aromatic and unsaturated compounds but normally contains about 12%. This diluent was fractionated and the bromine number of each fraction was determined. The results are shown on Fig. 7 of the appendix.

(ii) Deodorized Kerosene:

(iii) Odourless mineral spirits (R100). The suppliers (21) claim that this solvent has not greater than 2% aromatics and less than 0.5% unsaturated compounds.

3.2 General Procedure

A range of $Th(NO_3)_4$ solutions was made up from 0.5M to 2.0M in demineralized water. A similar range of $Th(NO_3)_4$ solutions was made up in 3.0M HNO_3. One ml. of the aqueous solution and one ml. of the selected solvent were added to a glass ampoule. The ampoule was sealed off in a gas flame.

A series of acid deficient solutions of $Th(NO_3)_4$ were also used with 20% and 40% TBP/Avtur solvents, but gels formed in the organic phase and at the interface. The cause of this gel formation is not known. As the formation of this gel interfered with the observation of the normal third phase formation the use of acid–deficient thorium nitrate solutions was therefore discontinued.

A series of samples of each diluent was made up and agitated in a water bath at various known constant temperatures. The temperature at which the third phase disappeared was noted.

3.3 The effect of addition of an aromatic substance on third–phase formation

Using a mixture of 40% TBP/odourless mineral spirits as solvent, and aqueous phases of 0.5M, 1.0M, 1.5M and 2.0M $Th(NO_3)_4$ (no added nitric acid) the effect of aromatics was determined by adding from 5–60% v/v of xylene to the organic phase, in steps of 5%. The ampoules were sealed off and the temperature of disappearance of the third–phase determined as above.

3.4 The effect of sec–octyl alcohol addition on third–phase formation (20)

The procedure in this case was the same as for the xylene addition above, with the exception that various volume percentages of sec–octyl alcohol were added to the organic phase, (viz: 1, 3, 6, 9, 12 and 15%).

3.5 Organic phase analyses

At a temperature just before the appearance of the third phase, a sample of the organic phase was removed by breaking the neck of the ampoule and withdrawing a sample of known mass. Knowing the density of the solvent used at room temperature, the volume at that temperature was calculated, correcting for the thorium content of the sample.

A colorimetric method using APANS was used to determine the thorium concentration.

3.6 Third phase formation with Thorex feed

To check third–phase formation with the aqueous feed to the Thorex process, a synthetic Thorex feed based on the ORNL practice (7) was made up from acid deficient $Al(NO_3)_3$, with $Th(NO_3)_4$, $UO_2(NO_3)_2$ and $Hg(NO_3)_2$ added.

SYNTHETIC THOREX FEED	
Density	1.642g/ml at 16°C
Acidity	* pH 1.38 (Acid deficient)
Th^{4+}	1.5M
Al^{+++}	0.6M
Hg^{++}	0.004M

* Using a potassium tetroxalate buffer 1.68pH at 20°C

Using 40%TBP/odourless mineral spirits as the solvent and the synthetic Thorex feed, three ampoules were loaded with varying solvent and aqueous volume ratios viz: 1:5, 1:3, and 1:1 and equilibrated at various temperatures. Using the two Caltex diluents a further two ampoules were loaded with a 40% TBP solvent and the synthetic Thorex feed in the volume ratio of 1:5. The Thorex Process uses a 1:5 volume flow ratio between the aqueous feed and the solvent feed (7).

4. RESULTS

Plots of the original aqueous thorium concentration (molar) versus temperature of disappearance (°C) of the third phase are given in the appendix for the three commercial diluents at varying % TBP. in the solvent and at varying acidity. (Two levels, nominally O.ON and 3.0N HNO_3, in the aqueous phase).

Figure 1. Avtur

Figure 2. Deodourised kerosene

Figure 3. Odourless Mineral Spirits

The effect of aromatic content in the solvent is shown in fig. 4 by a plot of % by volume of xylene in the solvent, versus temperature of disappearance of the third phase. Figure 5 shows a plot of % sec–octyl alcohol in the solvent versus temperature of disappearance of the third phase. Figure 6 shows the relationship between each of the commercial diluents with each other, with respect to their capability of forming a third phase. The commercial diluents are also compared with odourless mineral spirits containing various amounts of xylene, and finally with odourless mineral spirits containing sec–octyl alcohol. Table III in the appendix tabulates the few results obtained upon analysis of the organic phase at the consolute temperature for the Avtur series only.

The experiments with the synthetic thorex feed are given in Table I below.

TABLE I

Third Phase Formation with Thorex Feed

Solvent	Diluent	Volume Flow Ratio A:O	Consolute Temp. °C (Third phase)
40% TBP	Odourless mineral spirits	1:5	below 3°C
,,	,,	1:3	8°C
,,	,,	1:1	above 45° C
,,	Odourless kerosene	1:5	below 3°C
,,	Avtur	1:5	below 3° C

5. DISCUSSION OF THE RESULTS

5.1 The commercial diluents

A third phase formed with each of the diluents used. For the same conditions the temperature of the disappearance of the third phase was different. This difference is probably related to the quantity of non–paraffinic compounds in the diluents. This is shown by adding xylene, which changes the consolute temperature.

Third–phase formation is also dependent on thorium concentration, acidity and TBP concentration and temperature. (See figs. 1–3). The higher the thorium or nitric acid concentration, and the lower the temperature the more likely the third phase is to form, as previously reported (16, 13, 14, 1). Figure 3 also shows that a third phase is more likely to form with a 20% TBP than with 5% or 40% TBP.

5.2 The effect of aromatic compounds on third–phase formation

Sufficient xylene to the solvent will prevent third–phase formation (Fig. 4). For example, the consolute temperature is lowered 15°C by the addition of 15% v/v xylene to 40% TBP/odourless mineral spirits with a 1.5M $Th(NO_3)_4$ concentration in the aqueous phase.

5.3 The effect of sec–octyl alcohol on third–phase formation

The addition of small quantities of sec–octyl alcohol to the solvent will prevent third–phase formation, (fig. 5), 3% v/v of sec–octyl alcohol having the same effect on the consolute temperature as 15% v/v of xylene.

5.4 Grading of the commercial diluents tested

Figure 6 shows the consolute temperature for various thorium nitrate concentrations with three commercial diluents and a paraffinic hydrocarbon containing various amounts of xylene. From this it is possible to grade the three commercial diluents on a basis of alkane purity, the diluent which forms the third–phase at the lowest thorium concentration at a given temperature being the purest. (This technique might be developed into a suitable test for commercial diluent purity.)

TABLE II

Comparison of Diluent Quality and Cost

Diluent	Cost/gallon for 44 gal. units	Test Value from Fig.6	Alkane Purity
Odourless Mineral Spirits	6/10	2	BEST
Deodourized Kerosene	3/8	6	
Aviation Turbine Fuel	2/4	26	WORST

5.5 Diluent suitability for the Thorex process

The results (see Table I) show that with odourless mineral spirits (the purest alkane diluent), no third phase will form with the Thorex feed if the volume flow ratio is 1:5 (A:O), but that a third phase forms at 8°C when the volume flow ratio is lowered to 1:3. With a volume flow ratio of 1:1, the third phase persists at 45°C. Thus with the Thorex feed it is essential, when using a fairly pure alkane diluent, to operate with volume flow ratio well above 1:3. It may also be necessary if the solvent feed fails, for an interlock to operate to cut the aqueous feed to prevent a third–phase forming in the extractor.

6. CONCLUSIONS

6.1 The choice of a diluent

For the less highly radioactive fuels, such as irradiated thorium, where large concentrations of tetravalent metals are to be extracted, an aromatic or partially aromatic diluent will eliminate third-phase formation, although a sufficiently high volume flow ratio of the solvent to the feed will also prevent it. However for the more highly radioactive fuels, an alkane diluent free from aromatics and other unsaturated compounds appears to be necessary perhaps with a small amount of suitable additive to suppress the third phase. The products of radiolysis of these compounds cause trouble in the solvent extraction process (3, 5, 6) although there is some doubt about the role of aromatics in this effect (22).

The purest of the commercial Australian alkanes examined is the odourless mineral spirits while Avtur is the most impure. Thus the former would be expected to be a more satisfactory diluent except from the point of view of third-phase formation, and this can be prevented by using a high enough solvent-to-feed flow ratio. On the basis of American experience (1, 5, 7, 8, 21), Avtur is unsuitable for use in the chemical processing of irradiated fuel. The Caltex deodourized kerosene would probably be satisfactory after pretreatment. The odourless mineral spirits could probably be used in the Thorex process without pretreatment, but is the most expensive.

Although costs may be important the cheaper and less pure diluents will need pretreatment, so that any saving from a lower purchase price may be offset by the pretreatment cost.

6.2 The choice of an additive to suppress third phase formation

On the basis of the available information it would be unwise to choose an aromatic additive if radiolysis of the diluent is likely to occur. Sec-octyl alcohol may be a suitable additive. It has the advantage that only a small quantity of it is required to suppress third phase formation. However sec-octyl alcohol may also suffer radiation damage possibly by oxidation to a carboxylic acid. This could be serious, as one of the main constituents of crud is considered to be heavy-metal soaps resulting from the degradation of hydrocarbon diluents (6). The sec-octyl alcohol might also affect the partition coefficients of the cations to be separated. Its chemical stability towards the other constituents of the process streams would also have to be demonstrated.

7. ACKNOWLEDGMENTS

The assistance of the Analytical Chemistry Services Group in carrying out the thorium analyses and determining the bromine numbers of the Avtur fractions is acknowledged. Acknowledgments are also to D. F. Sangster for many helpful discussions.

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TABLE III

Organic Phase Analyses

Original nominal concentrations			Consolute Temperature °C	Analyses of organic phase (sampled at consolute temp.)	
TBP in organic % v/v	Th in aqueous, molar	HNO_3 in aqueous, molar		Th in organic, molar	HNO_3 in organic, molar
40	0.8	3.0	8	0.29	0.41
40	1.0	3.0	17	0.52	0.35
40	1.5	3.0	36	0.22	0.43
40	1.0	0	1	0.40	–
40	1.5	0	11	0.33	–
40	2.0	0	25	0.25	–
20	0.8	3.0	10	0.11	0.24
20	1.0	3.0	18	0.18	ND
20	1.5	3.0	38	0.18	0.14
20	1.5	0	18	0.17	–
20	2.0	0	30	0.29	–

APPENDIX

U.S. DILUENT SPECIFICATIONS.

A typical analysis of untreated Amsco 125–82 (7)

Color Saybolt	+30
Doctor	Sweet
Corrosion	Passes
Odor	None

Distillation range (760mm Hg)

IBP	178°C
10%	181°C
50%	184°C
90%	194°C
E.P.	202°C

Flash point, tag closed cup	53.3°C
API gravity 15.6/15.6°C	55.5
Specific gravity 15.6/15.6°C	0.7567
Density, lb/gal. 15.6/15.6°C	6.3
Kauri butanol value	26.2
Straight aniline point	85°C
Amount of unsaturated compounds present	~ 0.6vol%

Treated AMSCO 125–82 is used in the ORNL Thorex pilot plant.
Diluent treatment increases Iodine D.F. from 10 to 200+.

Properties of Amsco 125–90W (8) are similar to the above

S.g 25/4°C	= 0.7570
Kauri Butanol No	= 26.0
Aniline cloud point (°F)	= 186.5
Refractive Index 25° C	= 1.4226
Viscosity (millipoises)	= 14.1
Boiling Range (°C)	= 186–199
Midpoint °C	= 190

Flash point, tag closed cup (°F) = 133

Composition (%)

Aromatics	1
Napthenes	–
Paraffins	–
Iodine No	0.49
Odour	Slight
Colour	Water white

L. L. Burger (3) stated that AMSCO 125–90W and SUPER SOL were the purest alkane type diluents examin
Burger (3) also gives table of properties for ten other commercial diluents.

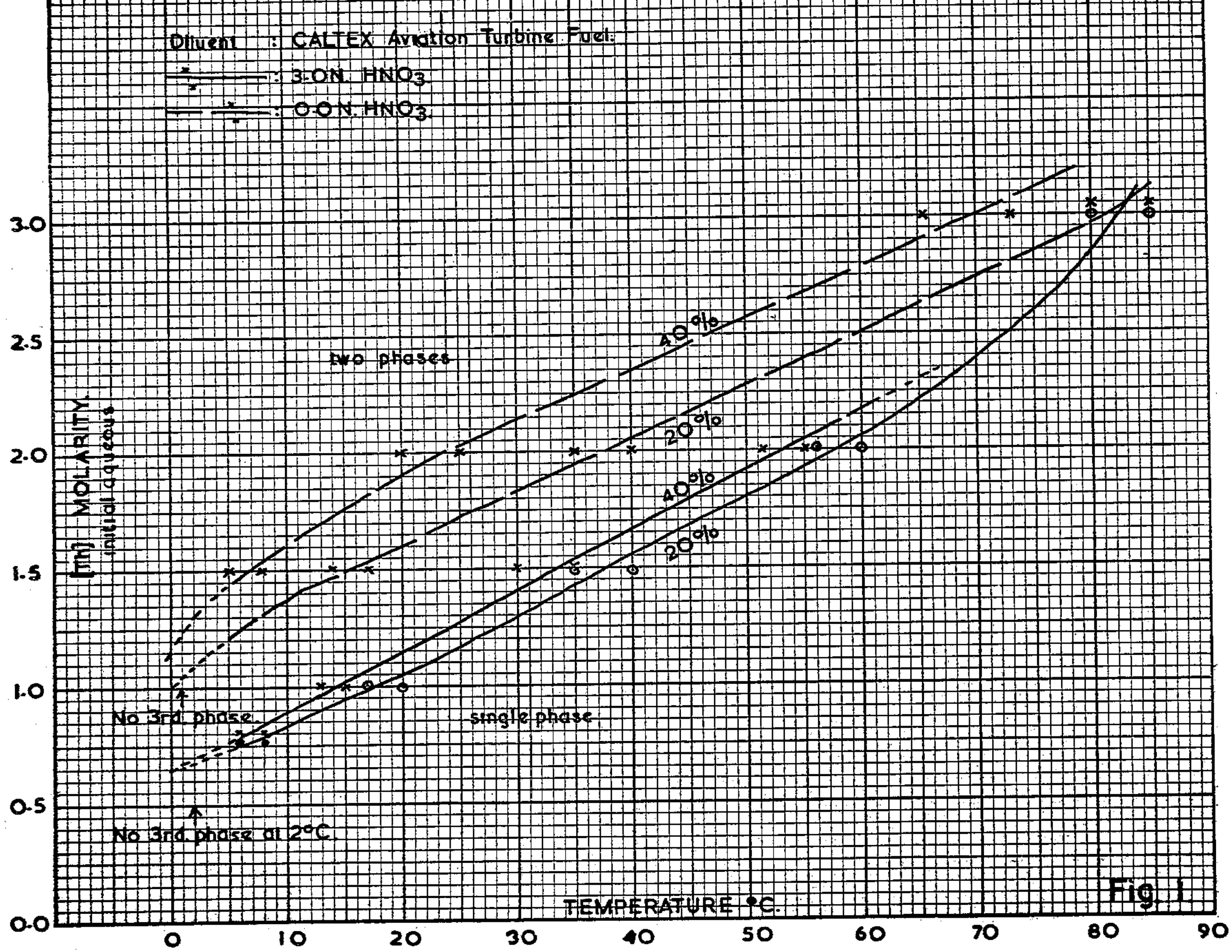
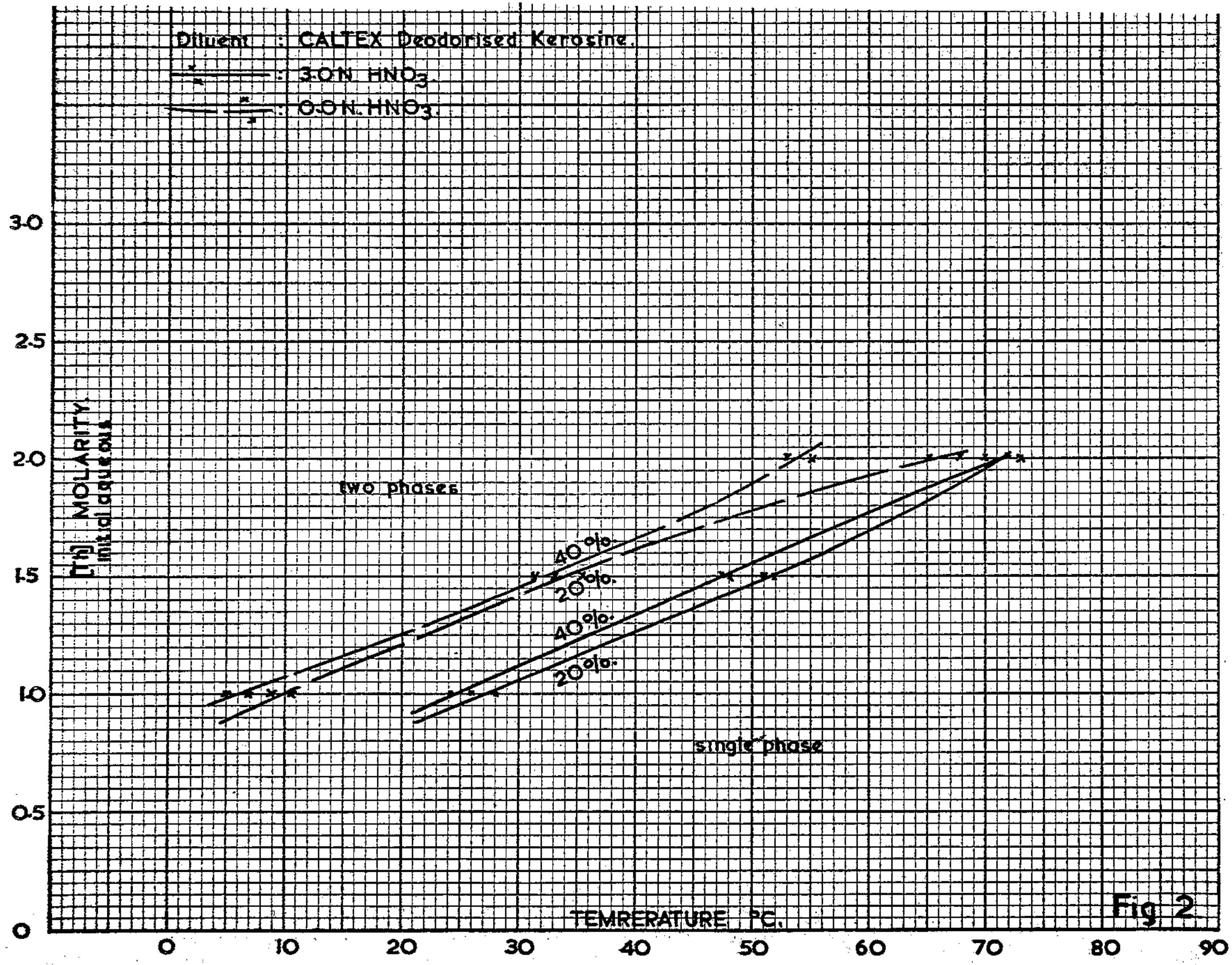

Diluent : CALTEX Aviation Turbine Fuel.
: 3·0N. HNO3
: 0·0N. HNO3
two phases
single phase
No 3rd. phase
No 3rd. phase at 2°C.
40%
20%
40%
20%
[Th] MOLARITY.
initial aqueous
TEMPERATURE °C.
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0
10
20
30
40
50
60
70
80
90

Fig. 1



Diluent : CALTEX Deodorised Kerosine.
: 3·0N. HNO3.
: 0·0N. HNO3.
[Th] MOLARITY.
initial aqueous
two phases
40%.
20%.
40%.
20%.
single phase
TEMRERATURE °C.
0
0·5
1·0
1·5
2·0
2·5
3·0
0
10
20
30
40
50
60
70
80
90

Fig 2

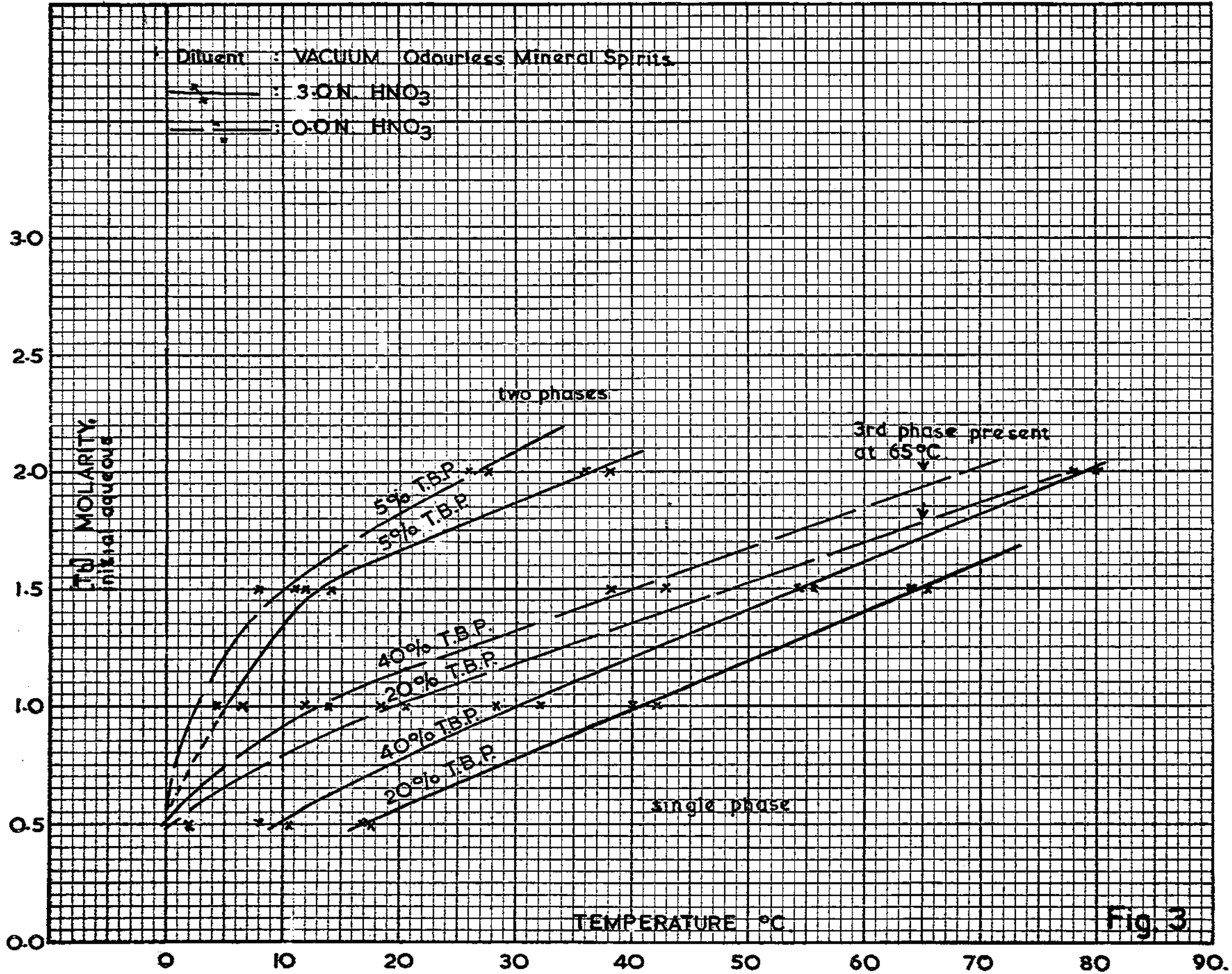
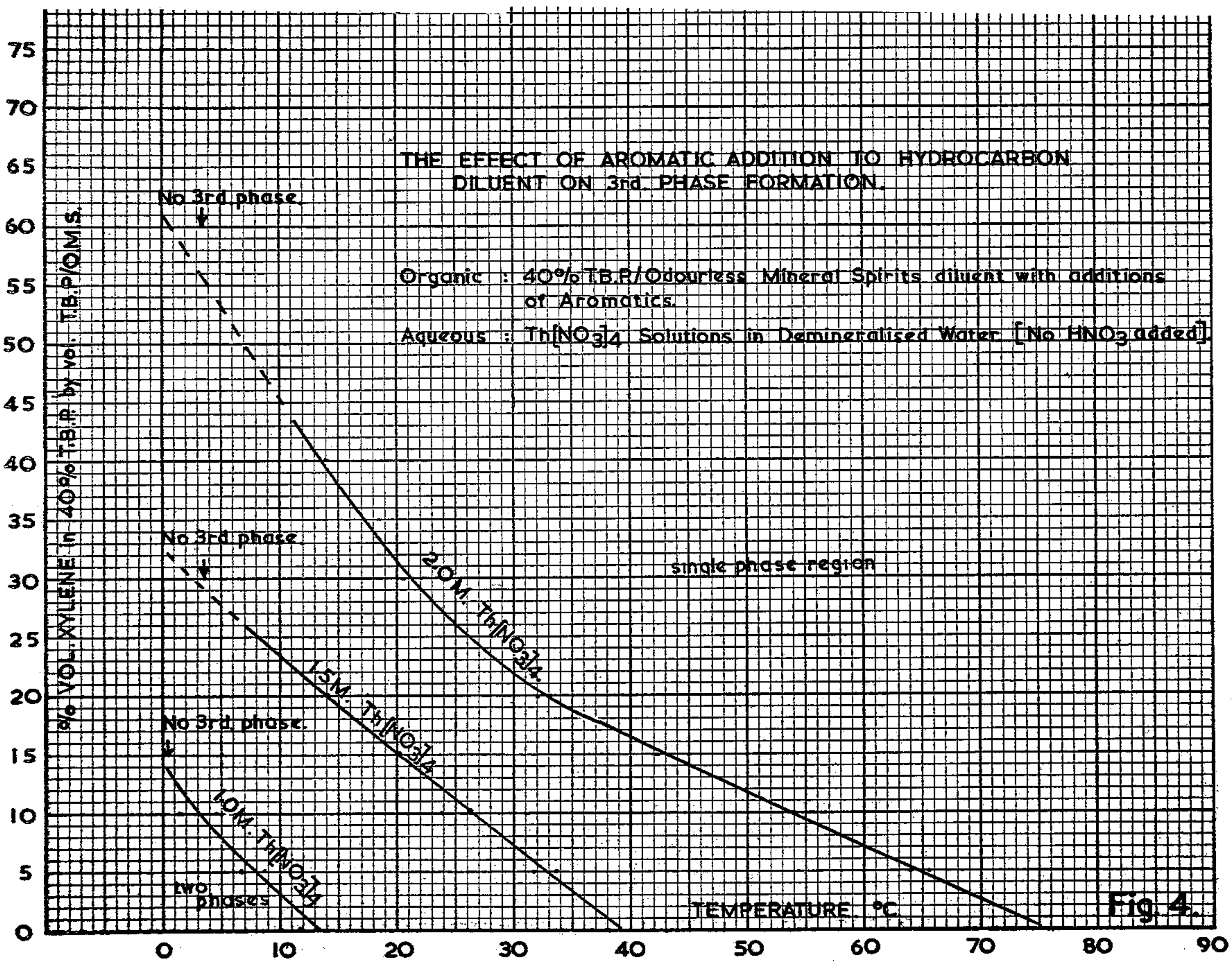
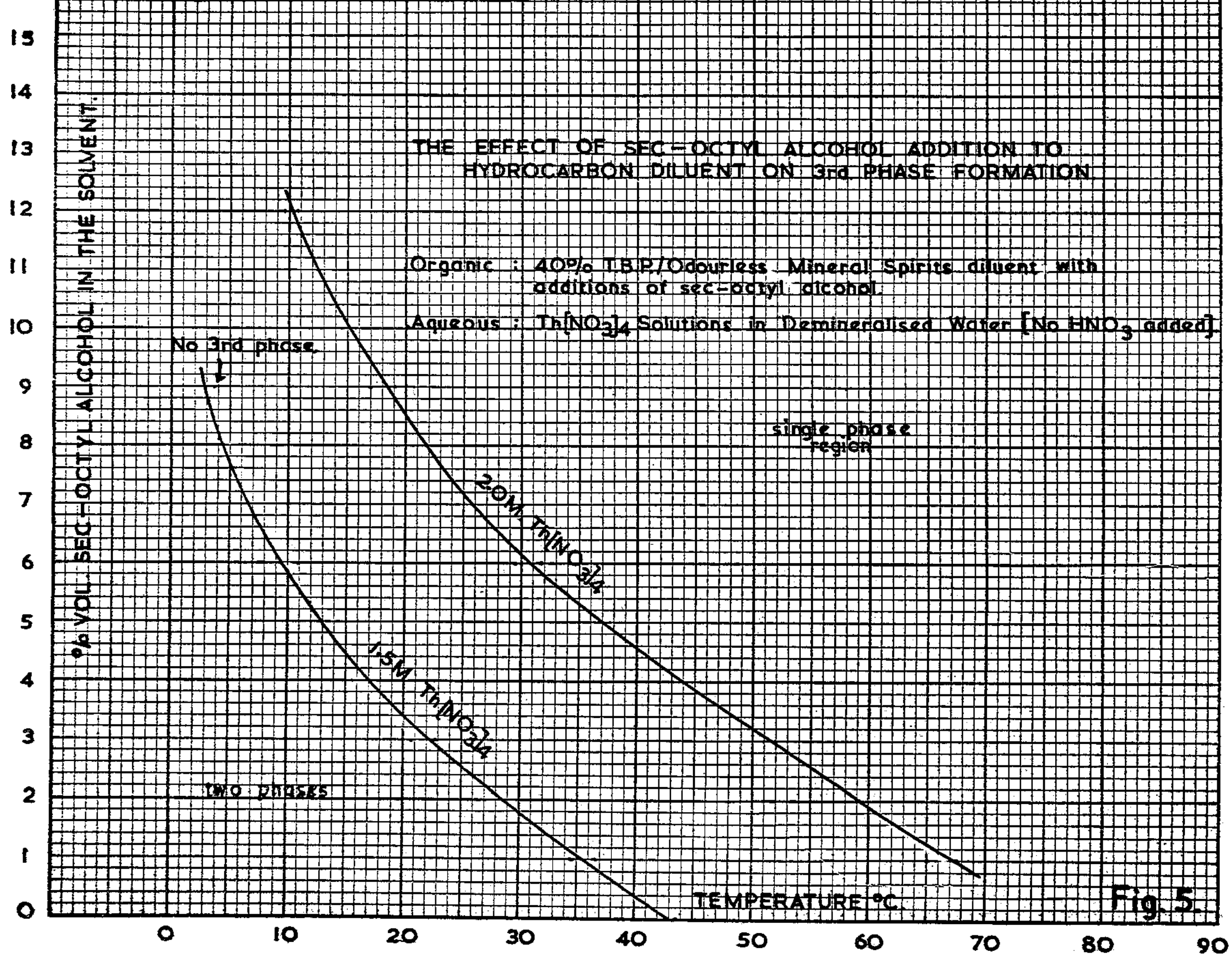

Diluent : VACUUM Odourless Mineral Spirits
: 3.0 N. HNO3
: 0.0 N. HNO3
[Th] MOLARITY, initial aqueous
TEMPERATURE °C
two phases
single phase
3rd phase present at 65°C
5% T.B.P.
5% T.B.P.
40% T.B.P.
20% T.B.P.
40% T.B.P.
20% T.B.P.
0.0
0.5
1.0
1.5
2.0
2.5
3.0
0
10
20
30
40
50
60
70
80
90.

Fig. 3



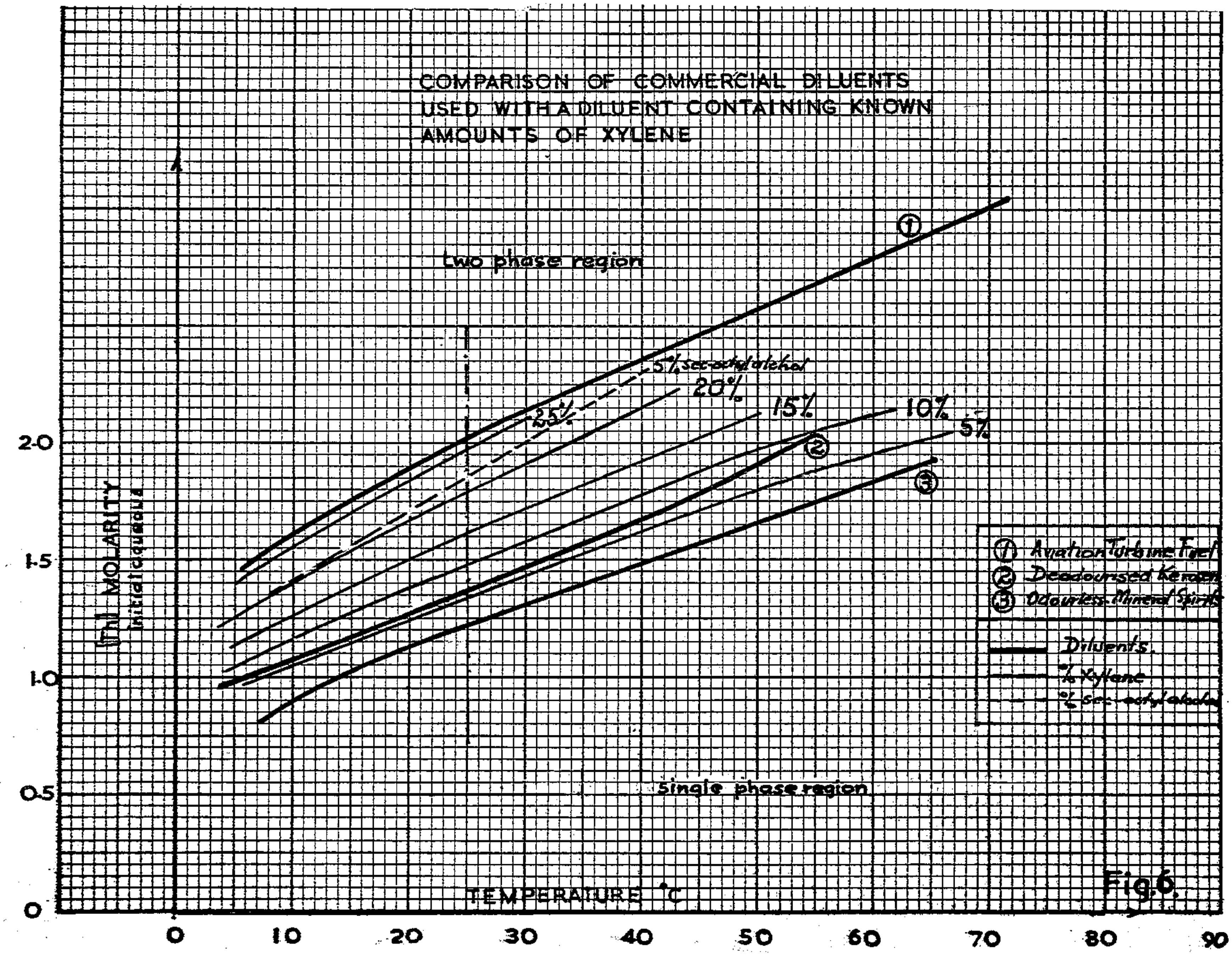
THE EFFECT OF AROMATIC ADDITION TO HYDROCARBON DILUENT ON 3rd. PHASE FORMATION.
Organic : 40% T.B.P./Odourless Mineral Spirits diluent with additions of Aromatics.
Aqueous : Th[NO3]4 Solutions in Demineralised Water [No HNO3 added].
% VOL. XYLENE in 40% T.B.P. by vol. T.B.P./O.M.S.
TEMPERATURE °C
No 3rd phase.
No 3rd phase.
No 3rd phase.
2.0M. Th[NO3]4
1.5M. Th[NO3]4
1.0M. Th[NO3]4
single phase region
two phases
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
0
10
20
30
40
50
60
70
80
90

Fig. 4.



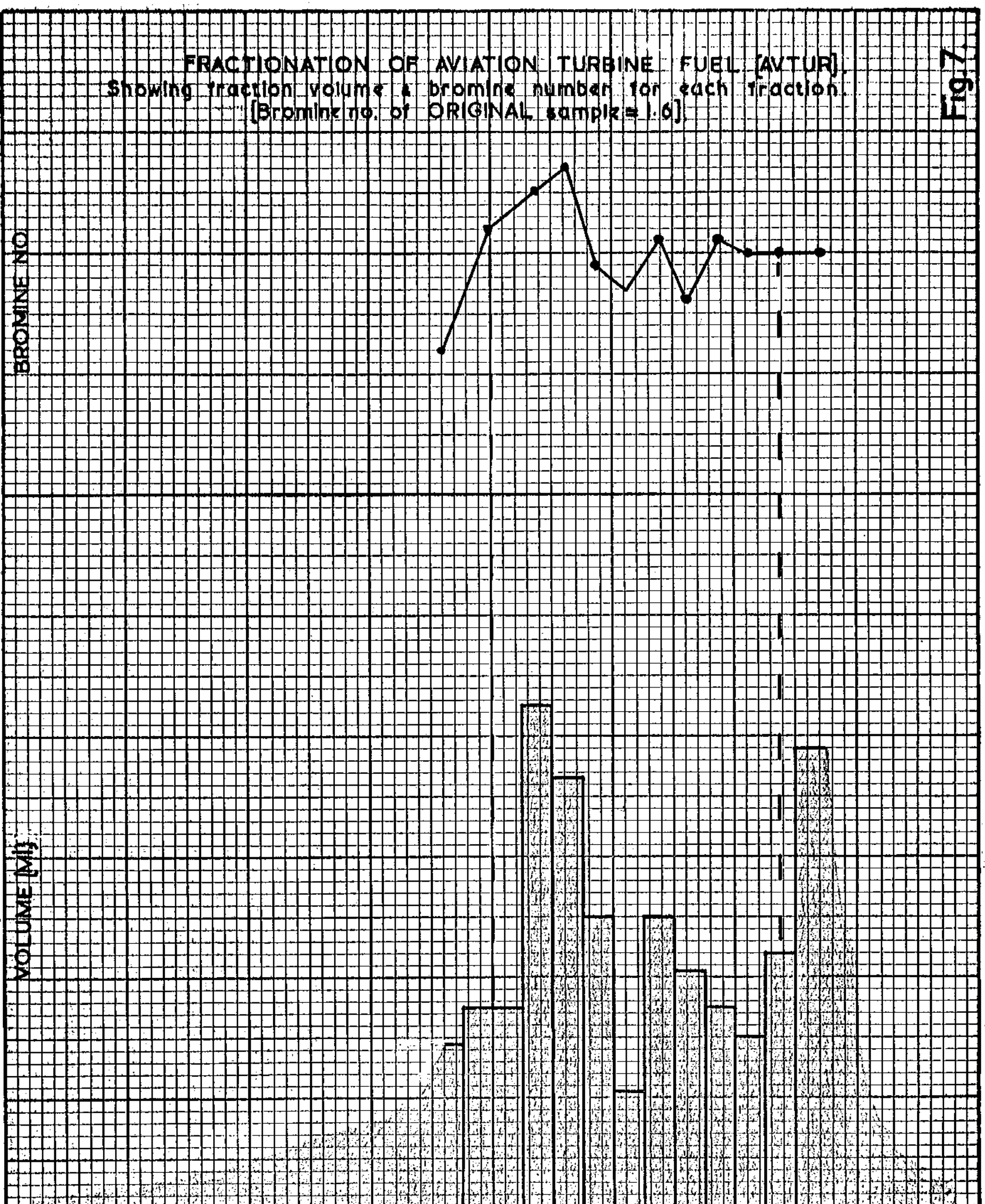
THE EFFECT OF SEC-OCTYL ALCOHOL ADDITION TO
HYDROCARBON DILUENT ON 3rd PHASE FORMATION.
Organic : 40% T.B.P./Odourless Mineral Spirits diluent with additions of sec-octyl alcohol.
Aqueous : Th[NO3]4 Solutions in Demineralised Water [No HNO3 added]
No 3rd phase.
single phase region
2·0M. Th[NO3]4
1·5M Th[NO3]4
two phases
% VOL. SEC-OCTYL ALCOHOL IN THE SOLVENT.
TEMPERATURE °C.
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
0 10 20 30 40 50 60 70 80 90

Fig. 5.



COMPARISON OF COMMERCIAL DILUENTS
USED WITH A DILUENT CONTAINING KNOWN
AMOUNTS OF XYLENE
two phase region
single phase region
[Th] MOLARITY
initial aqueous
TEMPERATURE °C
0
0.5
1.0
1.5
2.0
10
20
30
40
50
60
70
80
90
5% sec-octyl alcohol
25%
20%
15%
10%
5%
①
②
③
① Aviation Turbine Fuel
② Deodourised Kerosen
③ Odourless Mineral Spirits
Diluents.
% Xylene
% sec-octyl alcohol

Fig.6.



FRACTIONATION OF AVIATION TURBINE FUEL [AVTUR].
Showing fraction volume & bromine number for each fraction.
[Bromine no. of ORIGINAL sample = 1.6]
Fig. 7.
BROMINE NO.
VOLUME [Ml]
80
100
120
140
160
180
200
220
TEMPERATURE. °C.