EUR 4228 e

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

SOLUBILITY OF TERPHENYLS AND OM_2 MIXTURES IN PURE AND TECHNICAL SOLVENTS

by

G. MOSSELMANS and J. NIENHAUS

1969

Joint Nuclear Research Center
Ispra Establishment - Italy

Protection

LEGAL NOTICE

This report is on sale at the addresses listed on cover page 4

at the price of FF 7.—	FB 70.—	DM 5.60	Lit. 870	Fl. 5.10

When ordering, please quote the EUR number and the title, which are indicated on the cover of each report.

Printed by SMEETS
Brussels, April 1969

UR 4228 e

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

EUR 4228 e

SOLUBILITY OF TERPHENYLS AND OM_2 MIXTURES IN PURE AND TECHNICAL SOLVENTS by G. MOSSELMANS and J. NIENHAUS

European Atomic Energy Community - EURATOM
Joint Nuclear Research Center - Ispra Establishment (Italy)
Protection
Luxembourg, April 1969 - 56 Pages - 70 FB

The solubility of terphenyls and OM_2 mixture in pure and technical solvents is studied.

Results are presented on curves where the weight percent terphenyl or OM_2 versus temperature is plotted.

EUR 4228 e

SOLUBILITY OF TERPHENYLS AND OM_2 MIXTURES IN PURE AND TECHNICAL SOLVENTS by G. MOSSELMANS and J. NIENHAUS

European Atomic Energy Community - EURATOM
Joint Nuclear Research Center - Ispra Establishment (Italy)
Protection
Luxembourg, April 1969 - 56 Pages - 70 FB

The solubility of terphenyls and OM_2 mixture in pure and technical solvents is studied.

Results are presented on curves where the weight percent terphenyl or OM_2 versus temperature is plotted

Joint Nuclear Research Center
Ispra Establishment - Italy

Protection

2 MIXTURES

ENTS

ABSTRACT

The solubility of terphenyls and OM_2 mixture in pure and technical solvents is studied.

Results are presented on curves where the weight percent terphenyl or OM_2 versus temperature is plotted.

The results allow some considerations about the influence of the structure and the nature of functional groups on the solvent ability.

Finally a selection of solvent is made for decontamination purposes.

KEYWORDS

SOLUBILITY
TERPHENYLS
SOLVENTS
DIAGRAMS
DECONTAMINATION

C O N T E N T S

Solubility of Terphenyls and OM_2 Mixtures in Pure and Technical Solvents. (*)

Scope of investigation.

The organic compounds thought of until now as coolants in nuclear reactors are polyphenyls, usually a mixture of terphenyls.

These materials present serious difficulties for decontamination as they are little soluble, chemically stable, and melt only at elevated temperatures. Literature data (1) (2) on terphenyl solubility are rather scarce; the results given (3) do not furnish more than a base for the development of decontamination procedures. Consequently and in order to find a reliable base for the decontamination methods to be prepared, the solubility of terphenyls had to be examined systematically.

The investigation has intentionally been made as broad as could reasonnably be justified even if some of the solvents might probably find no use in later decontamination techniques.

This method allowed not only to get information but also helped to solve the following question : how do the solvent properties change within homologue series or when introducing new functional groups into the solvent molecule ?

As a result of a demand from our medical service, some oils which might be useful for human skin decontamination have been included in the study.

After having chosen some solvents among those tested on terphenyls, a study is made on the behaviour of the "OM_2" mixture used in reactors.

The possibility to produce an emulsion of the polyphenyls either by treating them with a solvent emulsion or an appropriate solvent shall also be examinated as well as the solubility behaviour of the high boilers, built in the OM_2 under reactor operation.

Results on these investigations will be reported later.

(*) Manuscript received on 4 November 1968.

1. Solubility of pure terphenyls.

1.1. Solvents and organic products used.

The terphenyls used were the isomers pure from Merck A.G. with the following purity qualifications :

O. Ø 3	99,9 %	O. - Ø 3	
m. Ø 3	97 - 98 %	m. - Ø 3	(3-2 % - Ø 3)
p. Ø 3	99,9 %	p. - Ø 3	(for scintillation use)

m. Ø 3 was not purified any further, as the solubility of the mixture as indicated above does not differ from that of pure m Ø 3 ; indeed the two components cannot be separated by fractional cristalliastion from any solvent in the concentration region (5). The choice of solvents has to be determined technologically, after their solvent ability has been tested, from their conventional behaviour (boiling point, flash point, explosivity, toxicity) and their price.

The eventual corroding properties of a solvent have not been taken into account, as the items to be decontamined will be too different chemically, so that the choice of an appropriate solvent will have to be made on the spot.

As the fouling process is certainly influenced by chlorine (4) the use of chlorinated solvents is not possible for materials which have to be used in reactor loops or experiments.

Characterisation of tested solvents is given in the following tables.

TABLE 1

- General characteristics of all solvents tested

- Pure solvents
  We give only the quality that we have used

- Technical solvents
  The semi-quantitative composition is given.

Table 1

Solvent	Manufacturated by	Type — composition
GROUP ALIPHATICS		
n- HEXANE	MERCK	FOR CHROMATOGRAPHY
OCTANE	ESSO CHIMICA	PETROL FRACTION C.SATURATED. 95 % PARAFFINES
ISOPAR G	" "	PETROL FRACTION C.SATURATED. 12 % C_9, 56 % C_{10}, 32 % C_{11}
ISOPAR K	" "	PETROL FRACTION C.SATURATED. 26 % C_{10}, 60 % C_{11}, 13 % C_{12}.
ISOPAR M	" "	PETROL FRACTION C.SATURATED: all C_{12}
VASELINE OIL	CARLO ERBA	D A B 6
GROUP ALICYCLICS		
CYCLO-HEXANE	MERCK	FOR CHROMATOGRAPHY
DECALIN	"	FOR SYNTHESIS
GROUP AROMATICS		
BENZENE	MERCK	CRISTALLIZABLE
TOLUENE	"	P.A.
XYLENE	"	LAB. REAGENT
SOLVESSO 100	ESSO CHIMICA	AROMATIC PETROL FRACTION = 11% C_8, 85 % C_9, 3 % C_{10}
SOLVESSO 150	ESSO CHIMICA	AROMATIC PETROL FRACTION : 1 % C_9, 73 % C_{10}, 23 % C_{11}
H A N	" "	AROMATIC PETROL FRACTION = 5 % C_9, 14 % C_{10}, 30 % C_{11}, 29 % C_{12} 13 % PARAFFINES
H B 40	MONSANTO	HYDROGENATED TERPHENYLS MIXTURE 18 % O - Ø 3, 82 % Ø 3 - H
THERMIP P2	ESSO CHIMICA	AROMATIC PETROL FRACTION FROM REFORMING= 69 % , - METHYLNAPHTHALENE, 8% NAPHTHALENE, 23 % ALKYLNAPHTHALENE + $Ø_2$

Table 1

Solvent	Manufacturated by	Type — composition
THERMIP LOURDE	ESSO CHIMICA	SIMILAR TO THERMIP P2 AROMATIC PETROL FRACTION FROM REFORMING PHENANTHRENE, FLUORENE, PYRENE + ALKYLATED PH, F., P.,
GROUP : HETEROCYCLICS		
TETRAHYDROFURAN	MERCK	PURE
PYRIDINE	"	FOR CHROMATOGRAPHY
GROUP : CHLORINATED HYDROCARBONS		
CARBONTETRACHLORIDE	MERCK	p. A.
TRICHLOROETHYLENE	CARLO ERBA	PURE
1.2-DICHLOROBENZENE	MERCK	FOR EXTRACTION ANALYSIS
GROUP : KETONES		
ACETONE	MERCK	p.A.
METHYLETHYLKETONE	CARLO ERBA	PURE
METHYL-i-BUTYLKETONE	" "	PURE
ACETYLACETONE	MERCK	PURE
CYCLOHEXANONE	MERCK	PURE
ACETOPHENONE	CARLO ERBA	PURE
I- PHORONE	B D H	LAB. REAGENT

Table 1

Solvent	*Manufacturated by*	*Type — composition*
GROUP: ALCOHOLS		
ETHYL-ALCOHOL	MERCK	ABSOLUTE
I-PROPYL-ALCOHOL	MERCK	p.A.
TERT.BUTYL ALCOHOL	MERCK	FOR CHROMATOGRAPHY
n- BUTYL-ALCOHOL	MERCK	FOR CHROMATOGRAPHY
BENZYL-ALCOHOL	MERCK	FOR CHROMATOGRAPHY
GROUP : ACIDS		
ACETIC-ACID	MERCK	p.A.
GROUP : ETHERS		
DI-I-PROPYL ETHER	MERCK	p.A.
DIOXANE	MERCK	p.A.
ANISOLE	CARLO ERBA	R.P.
GROUP : ESTERS		
ETHYL-ACETATE	MERCK	p.A.
I-BUTYL-ACETATE	CARLO ERBA	R.P.
n- BUTYL-ACETATE	MERCK	FOR CHROMATOGRAPHY
GROUP : AMINES		
ANILINE	SCHUCHARDT	p.A.

Table 1

Solvent	Manufacturated by	Type — composition
GROUP DIVERSE		
ACETO NITRILE	MERCK	FOR SYNTHESIS
DIMETHYL FORMAMIDE	MERCK	PURE
DIMETHYL SULFOXIDE	B D H	LAB. REAGENT
CASTOR OIL	CARLO ERBA	D A B 6
OLIVE OIL	CARLO ERBA	VICTUALS

TABLE 2

Pure Solvents

Conventional properties and cost

Remarks

1. Flash range :
   According ASTM Standard the values are given for "open cup" (OC) and for "closen cup" (CC)

2. Explosive range :
   The values are given in volume per cent of vapour in air at 20 °C.

3. Toxicity :
   It is given according to SAX toxic hazard rating code :

   0 = None
   1 = Slight
   2 = Moderate
   3 = High
   U = Unknown

4. Cost :
   Also for pure solvent the cost is given for the technical quality
   1 U.C. is equivalent to 1 U.S. $

5. Specific weight is given in g/ ml at 20 °C

Table 2 (PURE SOLVENTS)

Solvent	Specific Weight	Boiling Range °C	Flash Range °C	Explosive Range	Toxicity		Cost uc/100 Kg.
					Skin	Inhalation	
GROUP : ALIPHATICS							
n- HEXANE	0.66	69	-28 O.C -22 C.C	1.2-6.9	1	1	35
GROUP: ALICYCLICS							
CYCLO-HEXANE	0.778	79-81	+17 C.C.	1.3-8.4	U	2	21
DECALIN	0.88	186-195	57 C.C.		1	2	80
GROUP: AROMATICS							
BENZENE	0.88	80	-11C.C.	1.4-8.0	3	3	15
TOLUENE	0.87	111	+ 4C.C.	1.3-7.0	2	2	15
XYLENE	0.86	138-144	25C.C.	1.1-7.0	1	1	15
GROUP: METERO-CYCLICS							
TETRAHYDRO-FURAN	0.89	66	-17C.C.	2.3-11.8	3	3	160
PYRIDINE	0.99	114-116	20 C.C.	1.8-12.4	2	2	290
GROUP CHLORINATED HYDROCARBONS							
CARBONTETRA CHLORIDE	1.59	76-77			3	3	37
TRICHLORO-ETHYLENE	1.46	87			2	2	30
TETRACHLORO ETHYLENE	1.63	120-22			2	2	30
O- DICHLORO BENZENE	1.30	180-83	77 O.C.		3	2	190
GROUP: KETONES							
ACETONE	0.79	56	-17 CC. - 9 O.C	2.1-13.0	1	2	19
METHYL-ETHYLKETONE	0.80	79-80	-6 O.C. -7 C.C.	1.8-11.5	1	2	76

Table 2

Solvent	Specific Weight	Boiling Range °C	Flash Range °C	Explosive Range	Toxicity		Cost uc/100 Kg.
					Skin	Inhalation	
METHYL-i-BUTYLKETONE	0.80	114-116	24 O.C. 16 C.C.	1.2-8.0	2-3	2-3	96
ACETYL-ACETON	0.97	139	41 O.C.		2	1	1100
CYCLO-HEXANONE	0.95	156	42 C.C.		2	1	80
ACETO-PHENONE	1.03	199-203	105		1	1	128
i-PHORONE	0.92	215	96 O.C.		3	3	500
GROUP : ALCOHOLS							
ETHYL ALCOHOL	0.79	78	16 O.C. 14 C.C.	3.3-19	1	2	48
i- PROPYL ALCOHOL	0.79	82	22 O.C. 12 C.C.	2.0-5.2	1	2	410
tert.BUTYL ALCOHOL	0.78	83	9.C.C.	2.3-8	2	2	64
n-BUTANOL	0.81	118	35 C.C. 40 O.C.	1.4-18	2	2	72
BENZYL ALCOHOL	1.04	206	101 C.C.		1	1	108
GROUP ACIDS							
ACETIC ACID	1.05	118	40 C.C		3	3	48
GROUP ETHERS							
DI-i-PRO-PYL-ETHER	0.72	68	-28CC	1.4-21	3	2	120
DIOXAN	1.04	101	11 C.C.	1.9-22	2	3	160
ANISOL	0.996	154	52 O.C.		2	3	320

Table 2

Solvent	Specific Weight	Boiling Range °C	Flash Range °C	Explosive Range	Toxicity		Cost uc/100 Kg.
					Skin	Inhalation	
GROUP: ESTERS							
ETHYL-ACETATE	0.90	77	-5 C.C. +7 O.C.	7.2-11.5	1-2	2	42
n- BUTYL ACETATE	0.88	126	23 C.C. 34 O.C.	1,7-15	1	1- 2	80
i- BUTYL ACETATE	0.87	117	17 C.C.		2	2	160
GROUP: AMINES							
ANILINE	1.02	184	76 C.C.		3	3	140
GROUP: DIVERSE							
ACETO-NITRILE	0.79	82	6.0.C.		3	3	270
DIMETHYL FORMAMIDE	0.95	152-155	67 O.C	2.2-15.2	3	2	140
DIMETHYL SULFOXYDE	1.10	DECOMP. 100 °	90 O.C		U	U	400

TABLE 3

TECHNICAL SOLVENTS

Conventional properties and cost

Remarks.

1. Molecular weight :

It is given as the average value

For the explanations of the other columns see table 2

Table 3 (TECHNICAL SOLVENTS)

Solvent	Molecular Weight	Specific Weight	Boiling Range °C	Flash Range °C	Explosive Range	Toxicity		Cost
						Skin	Inhalation	uc/100 Kg
GROUP: ALIPHATICS								
OCTANE	114	0.73	116-121	< 1 OC.	1-6	1	2	35
ISOPAR G	138	0.75	159-179	40 C.C.		1	1	40
ISOPAR K	156	0.76	176-95	52 C.C.		1	1	40
ISOPAR M	177	0.78	206-47	80 C.C.		1	1	39
VASELINE OIL	430	0.89	> 360			1	0	60
GROUP: AROMATICS								
SOLVESSO 100	138	0.87	160-75	48 O.C.	1-6	1-2	1-2	34
SOLVESSO 150	138	0.89	187-212	66 O.C.	1-6	1-2	1-2	34
HAN		0.93	181-281	58 O.C.		1-2	1-2	31
H B 40	~245	1.005	345	174 C.C 180 O.C		U	U	71
THERMIP P2	153	0.99	230-60	>100C.C 1280.C		U	U	32
THERMIP LOURDE		1.006	> 260	> 100		U	U	32
AKP - M	206	1.07	327-72	178 C.C. 208 O.C		U	U	32
GROUP: DIVERSE								
CASTOR OIL	860	0.97	313	230C.C.		1	1	96
OLIVE OIL	790	0.91		225 O.C		0	0	80

1.2. Determination of the solubility.

A weighted (or measured) amount of solvent is added to a weighted quantity of terphenyl; both are heated together until producing a clear solution which then is cooled.

A preliminary test gives an indication on the temperature where crystallisation begins.

After reheating until a clear solution is obtained (control of weight), cooling is restarted slowly (cooling rate $\approx$ 1 min. $^{\circ}C^{-1}$) while stirring with the thermometer and eventual seeding of single large crystal easily distinguishable.

The temperature where the first crystal appears thus exactly can be determined. This point is the saturation point of the solution at the determined temperature.

The known concentration together with the temperature which has been measured gives one point of the solubility curve.

By addition of further quantities of solvent the concentration changes and a new point of the curve is determined as described above.

One point of the solubility curve is the fusion point of pure terphenyl (c= 100 % Ø 3). The curve then down to room temperature (20 °C) is determined experimentally as described above.

The weight per cent versus temperature °C is plotted on the solubility curves. This method is described (7) as the synthetic method to determine solubility : for a known concentration is checked the temperature, where a 2- phase system becomes in 1-phase system etc..

The term weight per cent means g terphenyl in 100 g solution. If the reader wants to get information on the solubility per g terphenyl in 100 g of solvent,-an other usual unit,-he can calculate it using the following formula :

$$S = \frac{g \ \emptyset \ 3}{100 \ g \ solvent} = \frac{\% \times 100}{100 - \%} \quad (1)$$

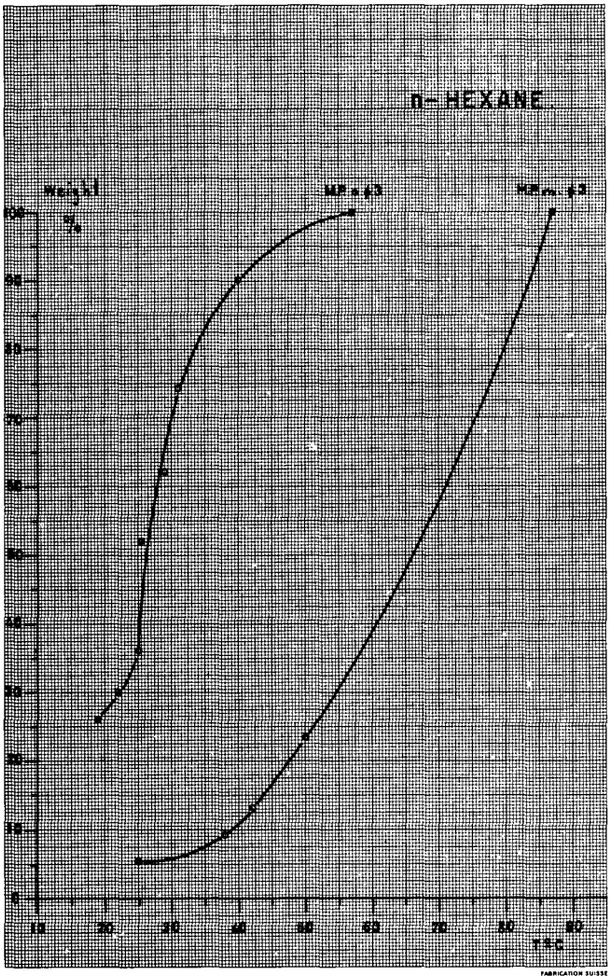
The amounts used depend on solubility.
The mass of terphenyl and solvent together has been chosen large with respect to the thermometer mass; the combined mass further must not be too small in order to get constant cooling velocities.
All results have been taken from experiments with at least 100 g total mass.
Weights have been determined with a Mettler balance (maximum 800 g), division of scale 0,1 g). The smallest quantity of terphenyl used in case of bad solubility was 3 g.
The maximum error of weighing (0,05 g difference are discernible) is thus 1,5 %...
The thermometers used (1/1° scale graduation) were calibrated.

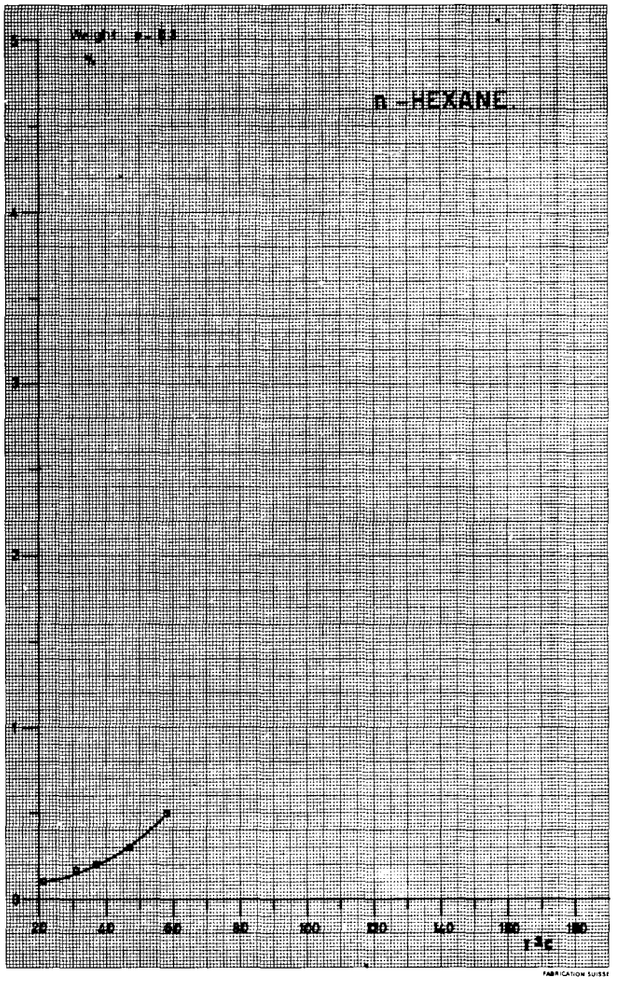
1.3. Results.

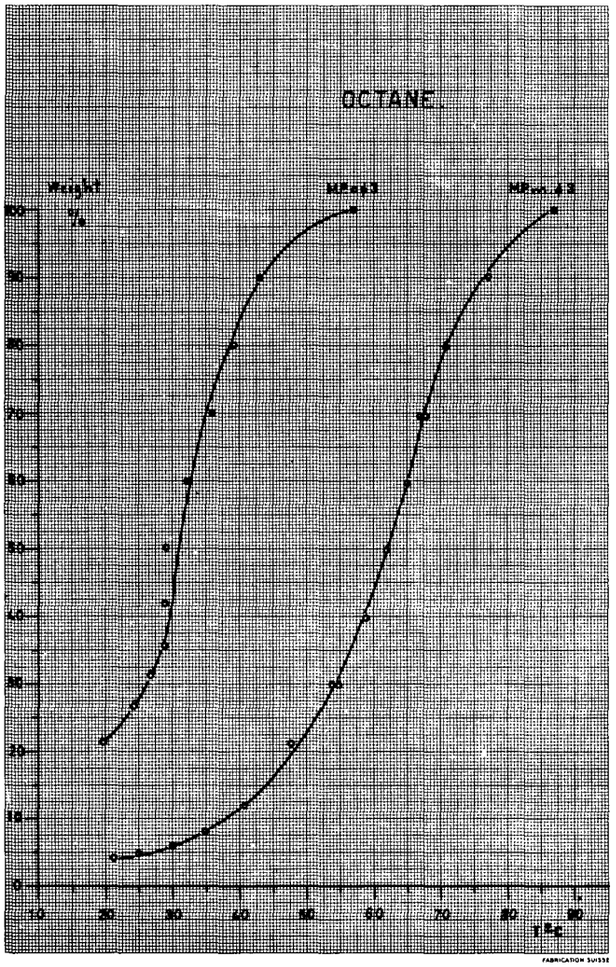
The curves are given according to the solvent list of table 1.

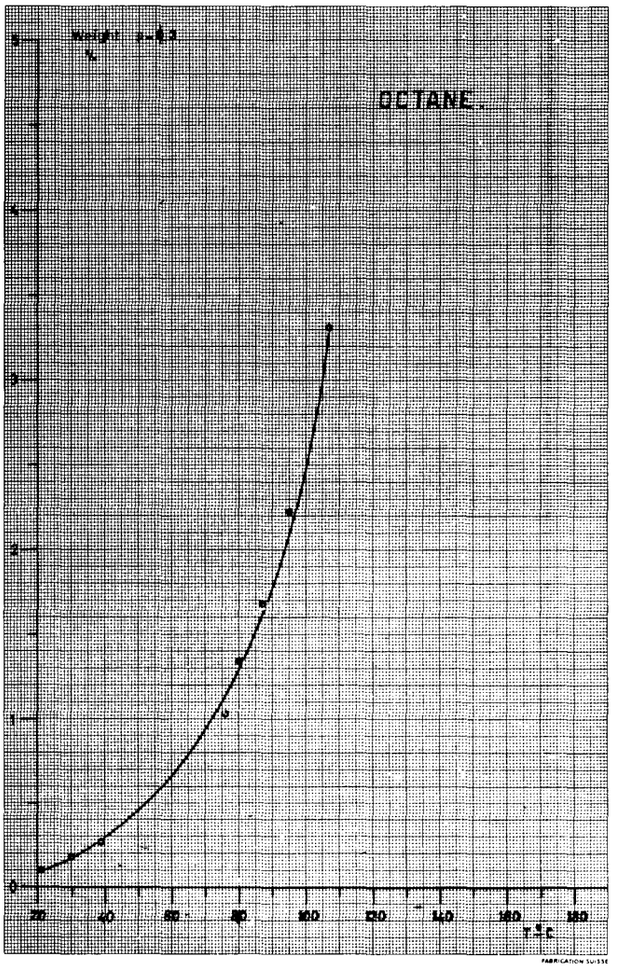
Solubility curves.

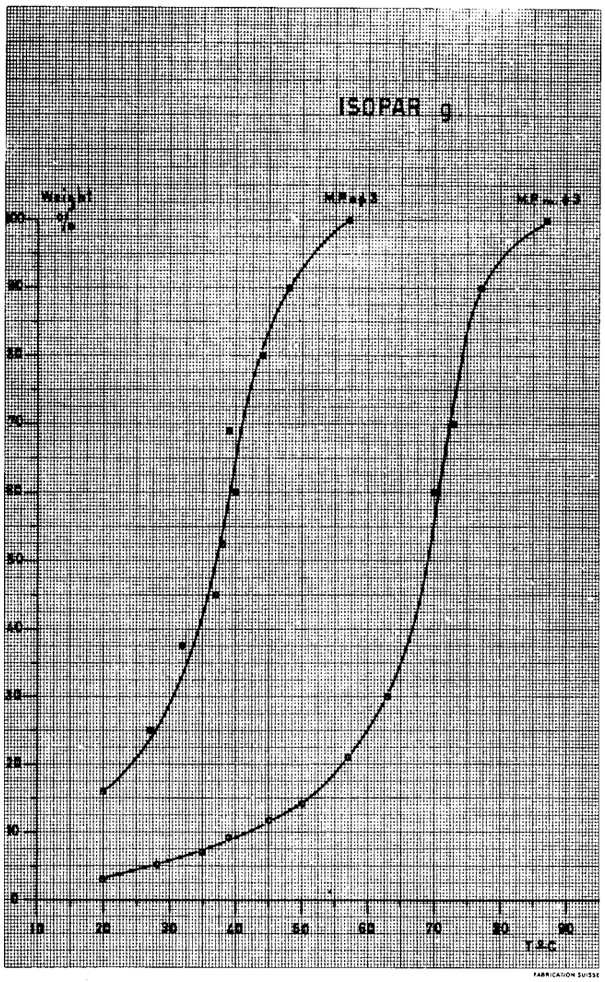
On the solubility curves are platted weight -% Ø 3 versus temperature for every solvent tested. Two different scales are used : one for the solubility of o - and m - Ø 3 up to a "solution" of 100 % Ø 3 = melting point of pure Ø 3 : another up to 5 or 10 % p- Ø 3 respectively, due to the very different solubility of the p - Ø 3.

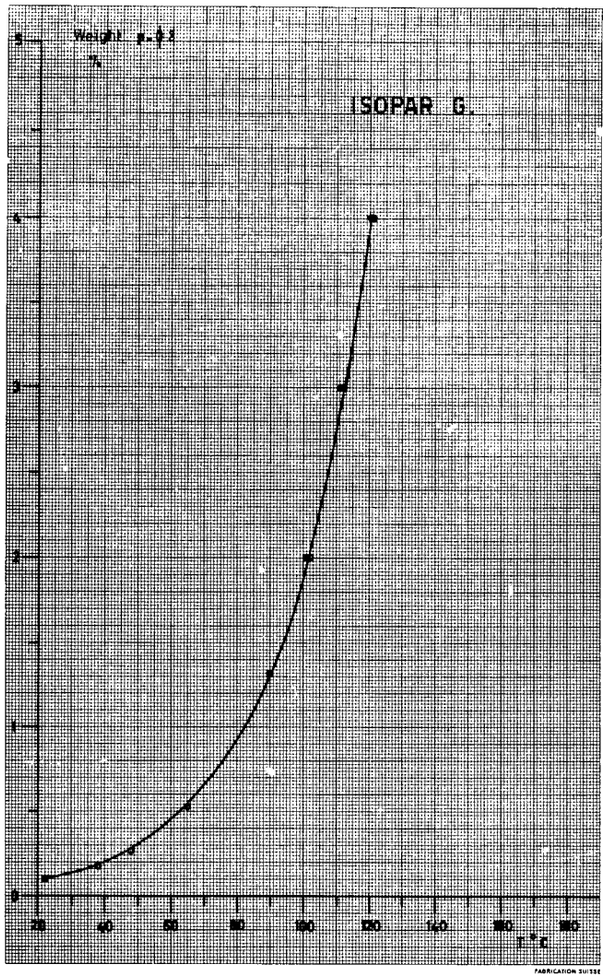

n-HEXANE
Weight %
MP = 63
MP = 63
T °C

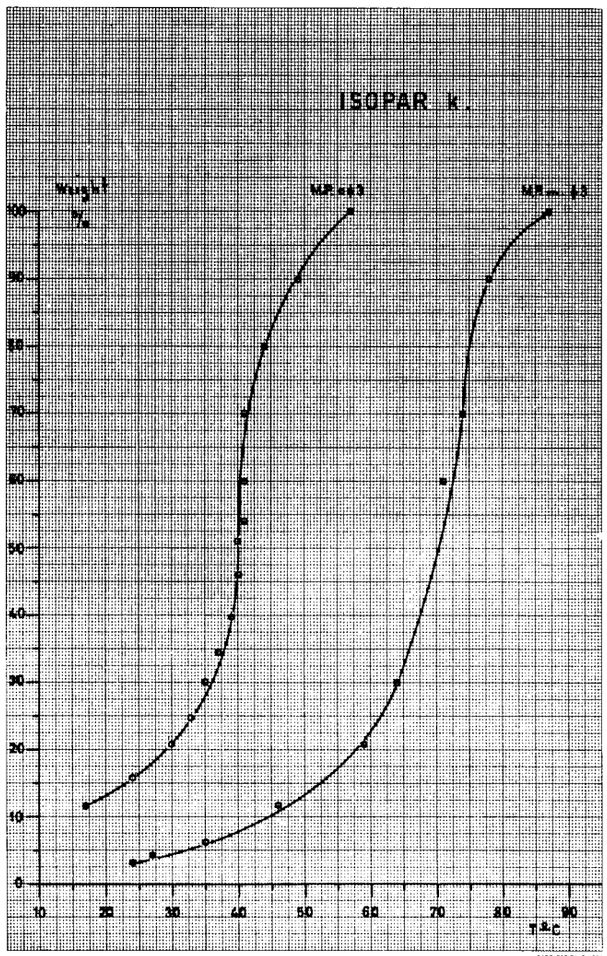

n-HEXANE
T °C

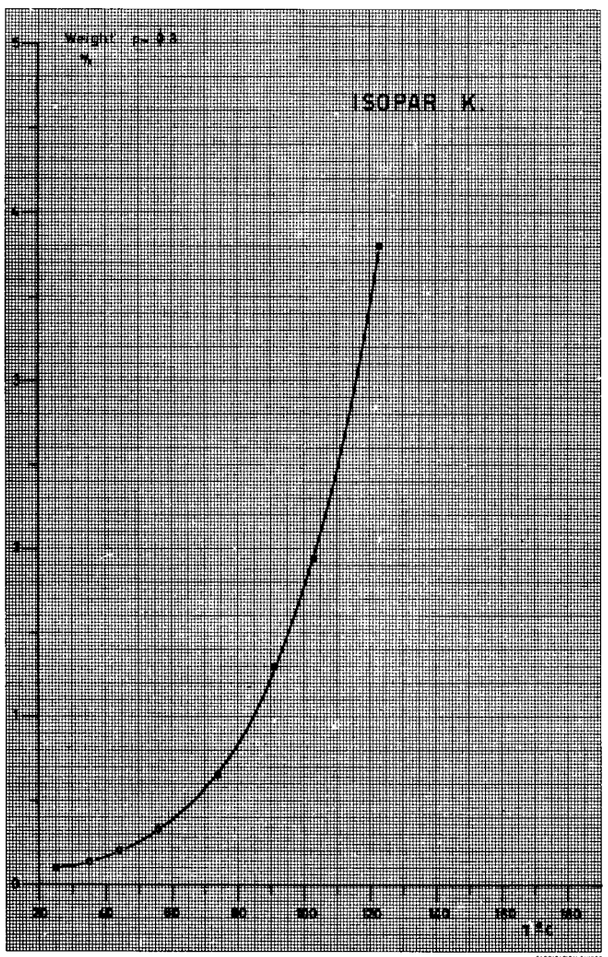

OCTANE
Weight %
MP = 63
MP = 63
T °C

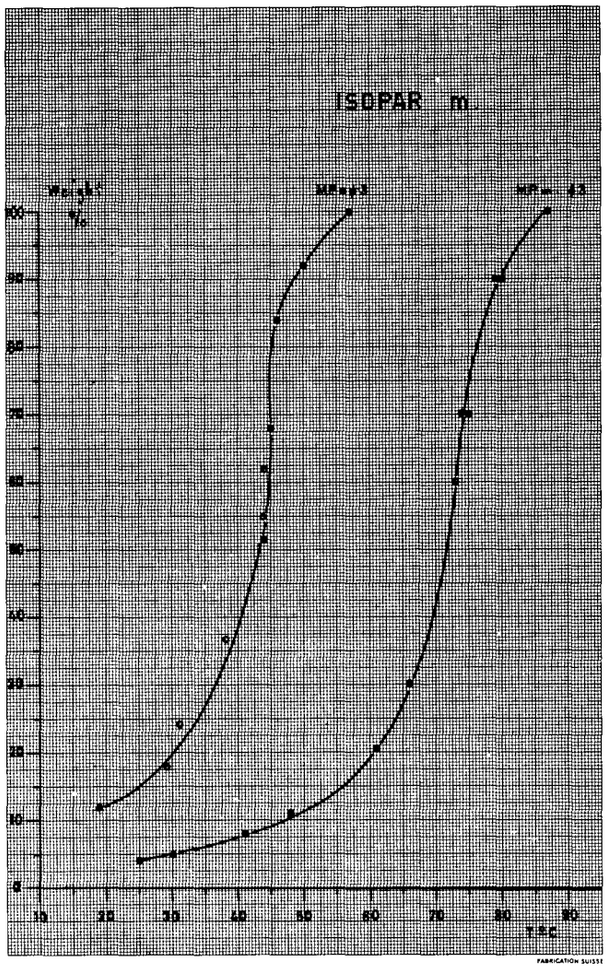

OCTANE
T °C

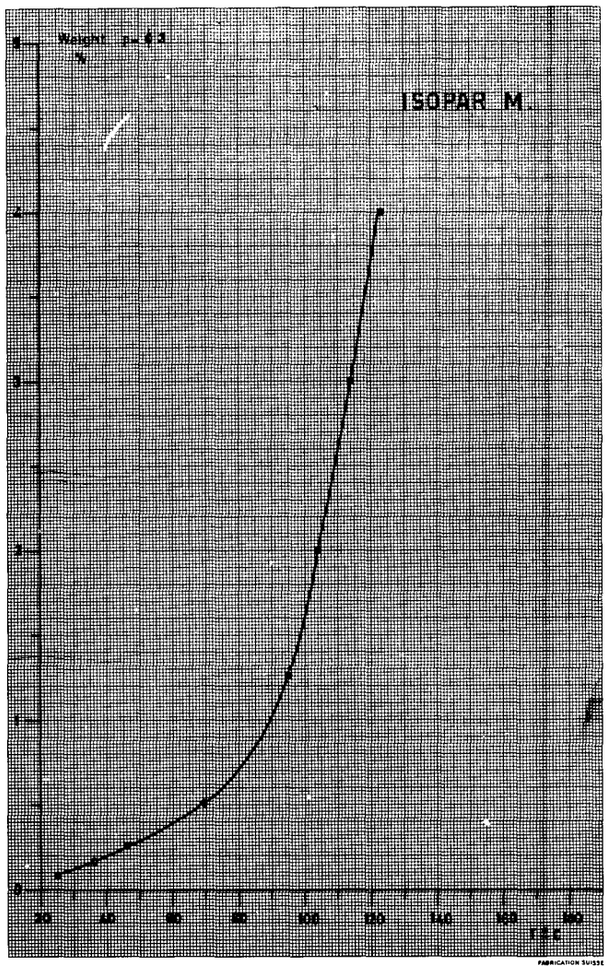

ISOPAR g.
Weight %
MP = 0.5
MP = 4.5
T °C
FABRICATION SUISSE

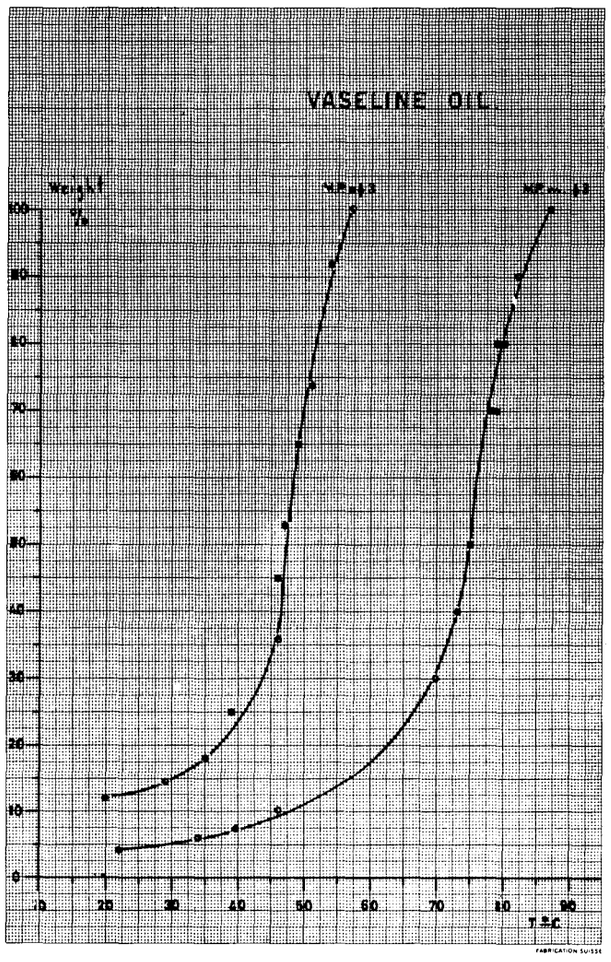

Weight %
ISOPAR G.
T °C
FABRICATION SUISSE

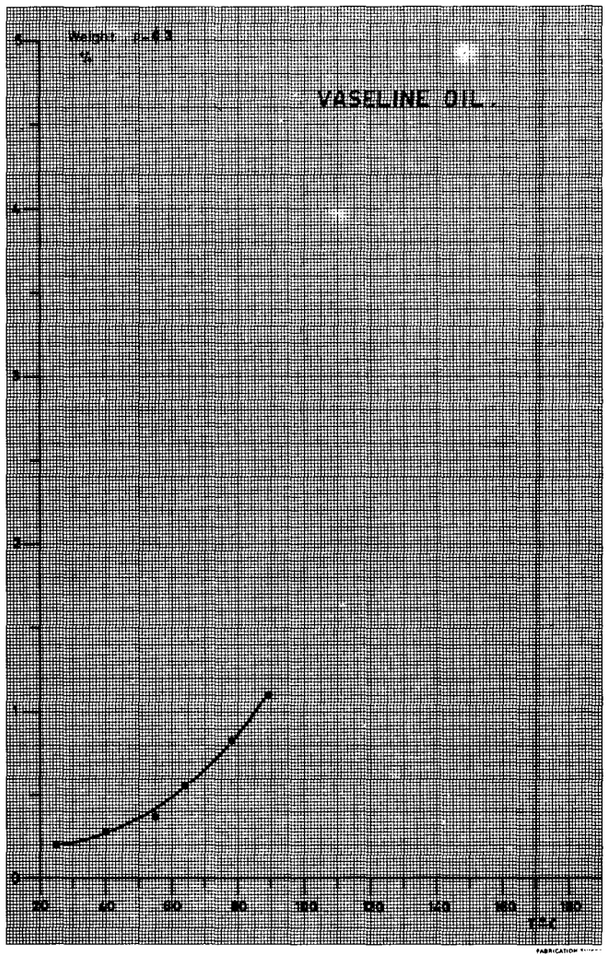

ISOPAR k.
Weight %
MP = 0.5
MP = 4.5
T °C
FABRICATION SUISSE

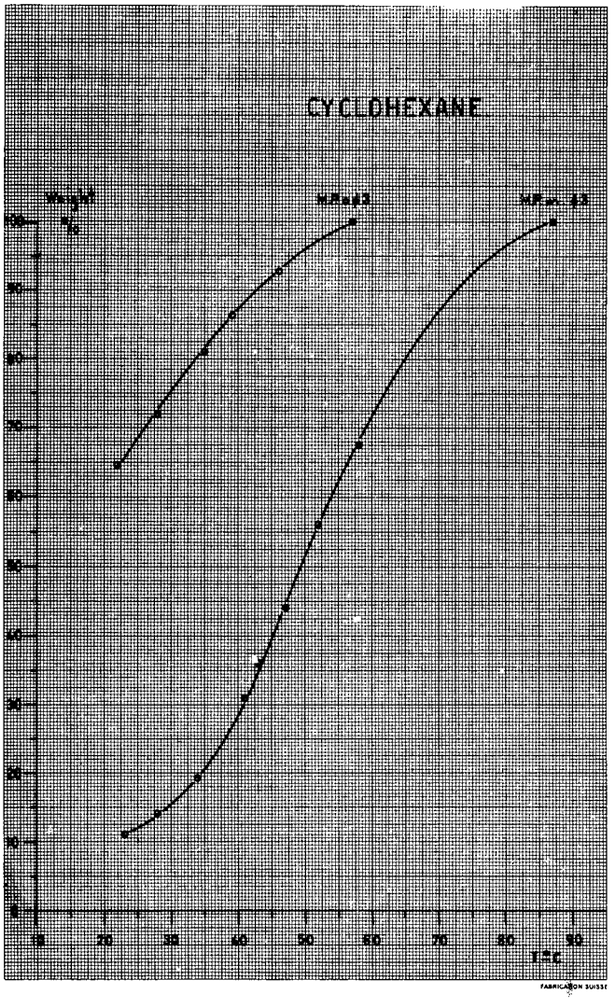

Weight %
ISOPAR K.
T °C
FABRICATION SUISSE

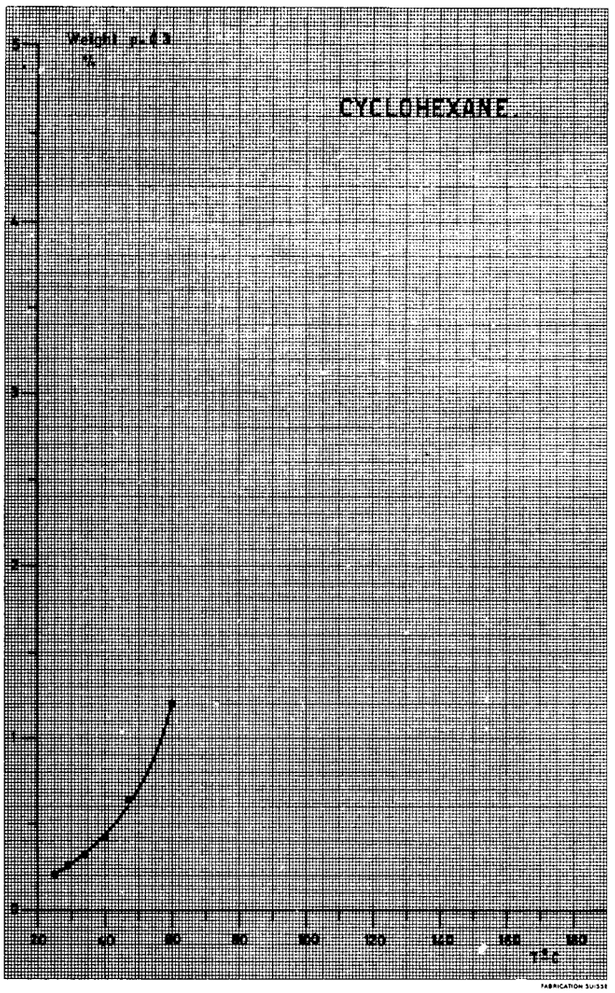

ISOPAR M.
Weight %
MP = 93
MP = 43
T °C
FABRICATION SUISSE

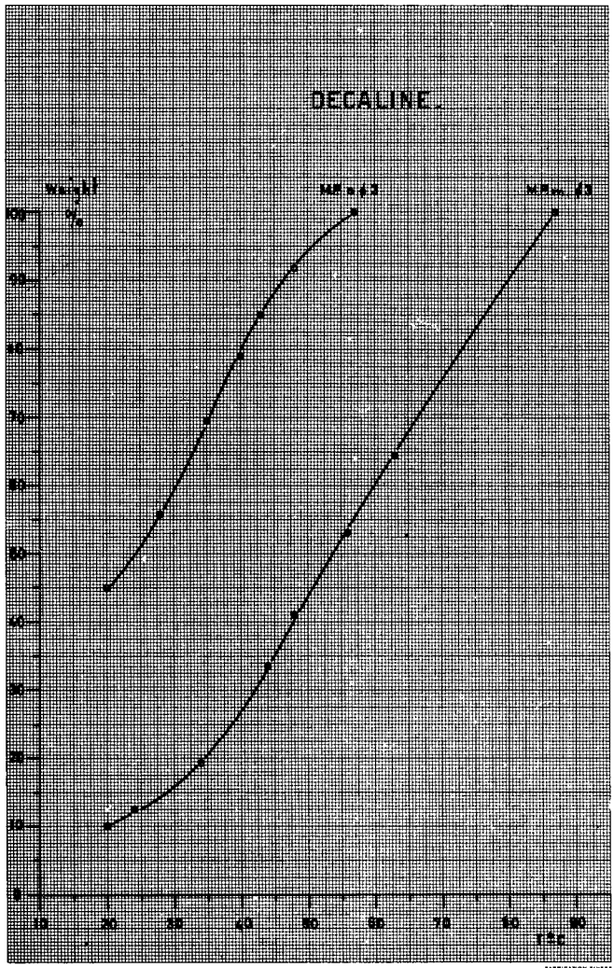

ISOPAR M.
Weight %
T °C
FABRICATION SUISSE

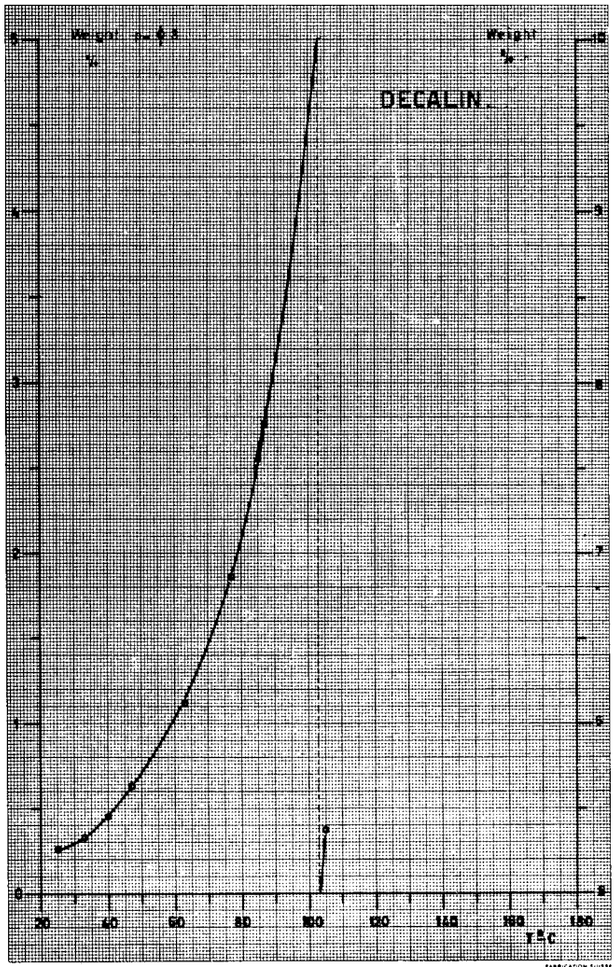

VASELINE OIL
Weight %
MP = 93
MP = 43
T °C
FABRICATION SUISSE

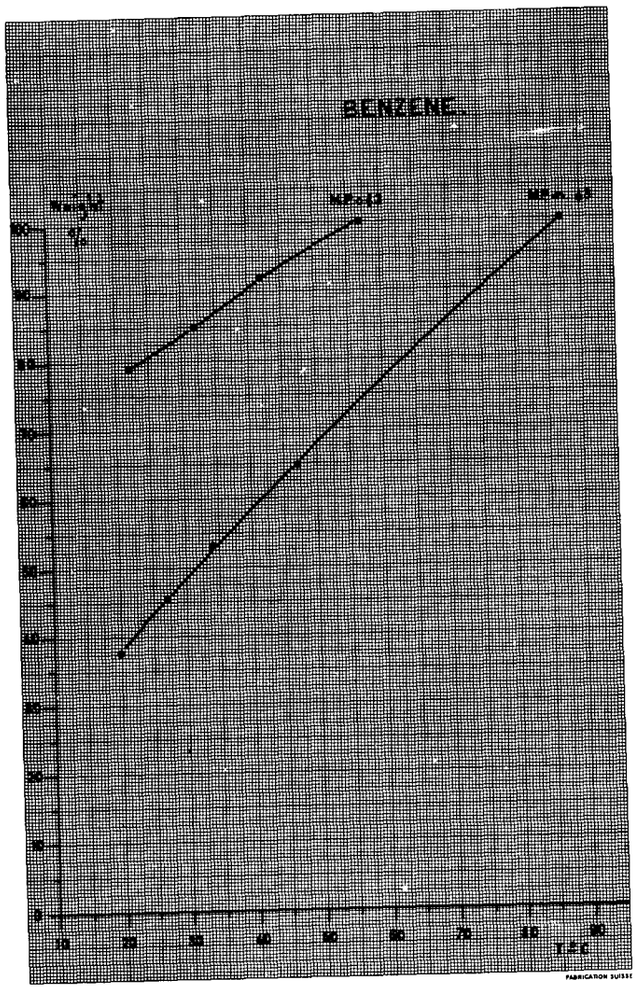

VASELINE OIL
Weight %
T °C
FABRICATION SUISSE

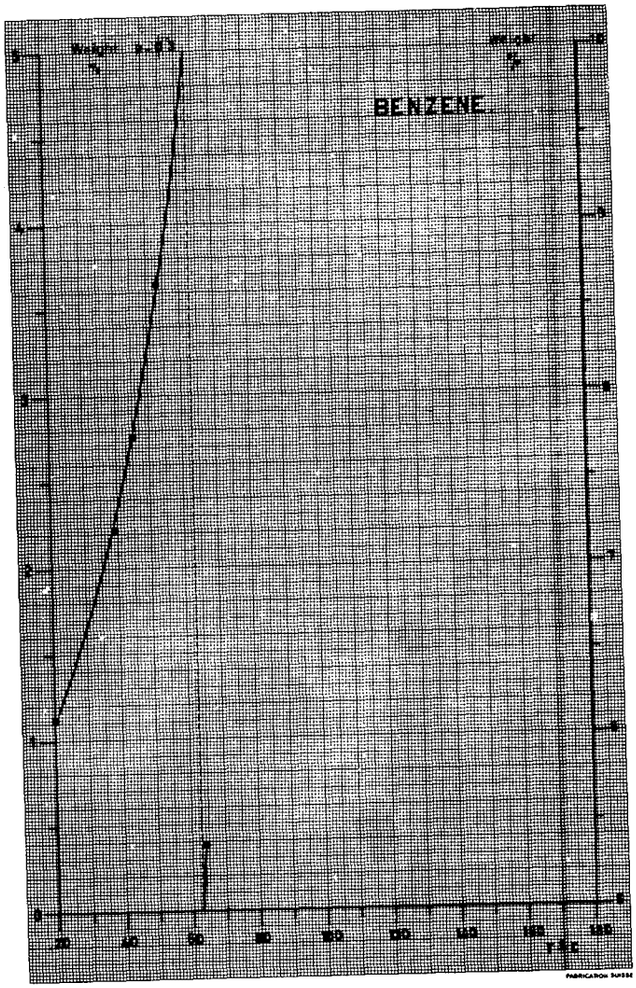

CYCLOHEXANE
Weight %
T.°C

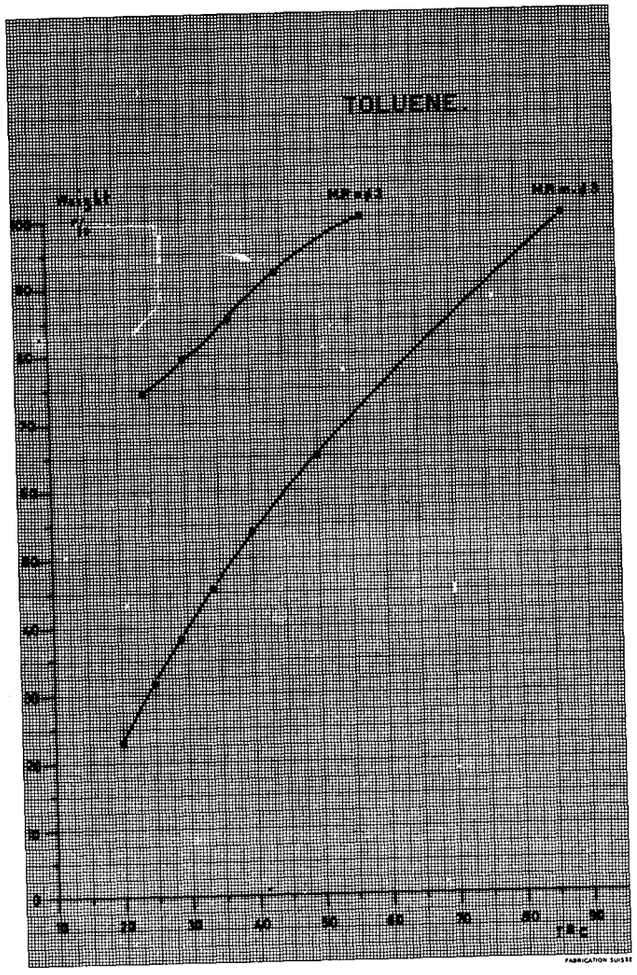

CYCLOHEXANE
T.°C

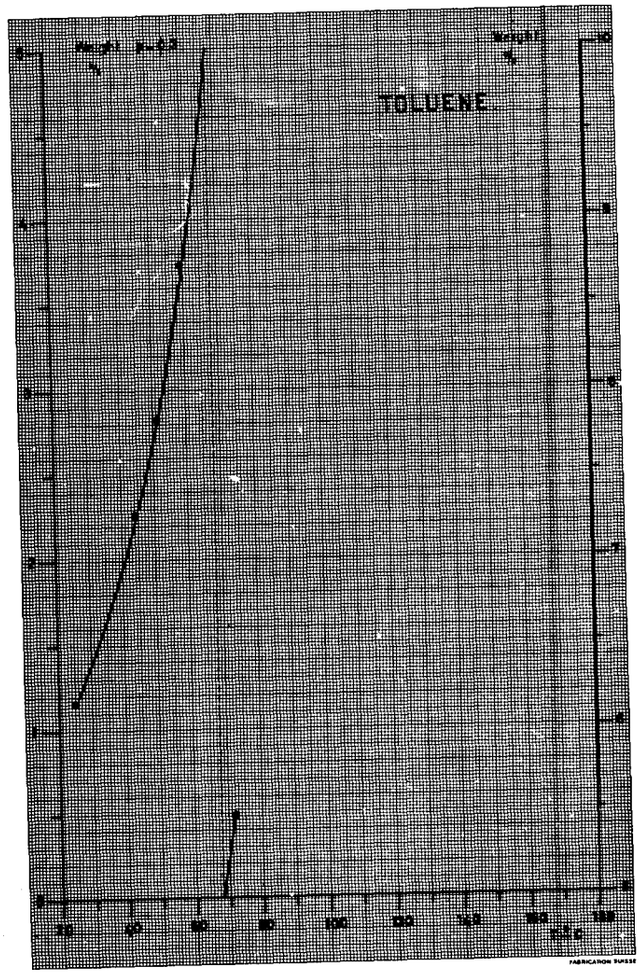

DECALINE
Weight %
T.°C

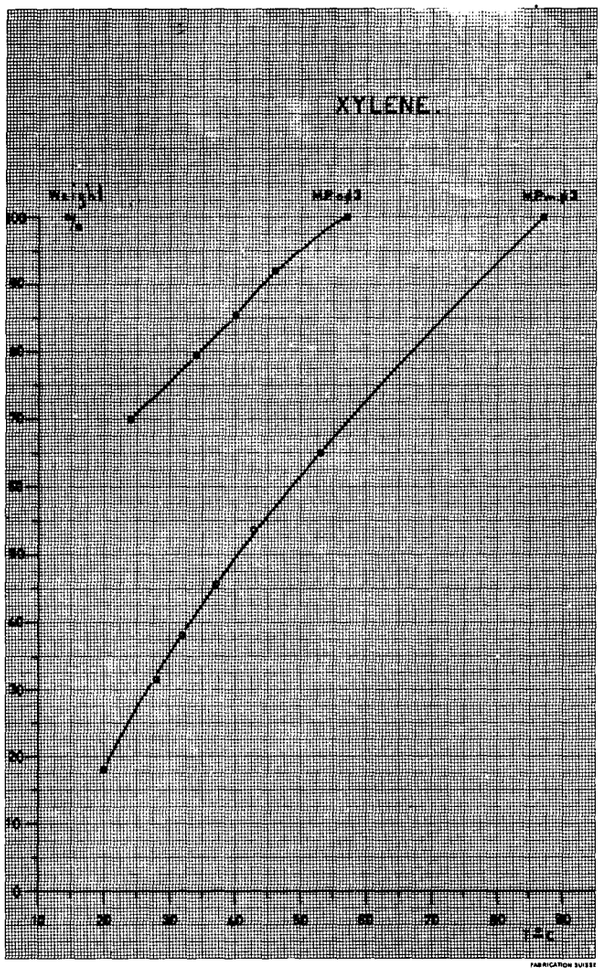

DECALIN
T.°C

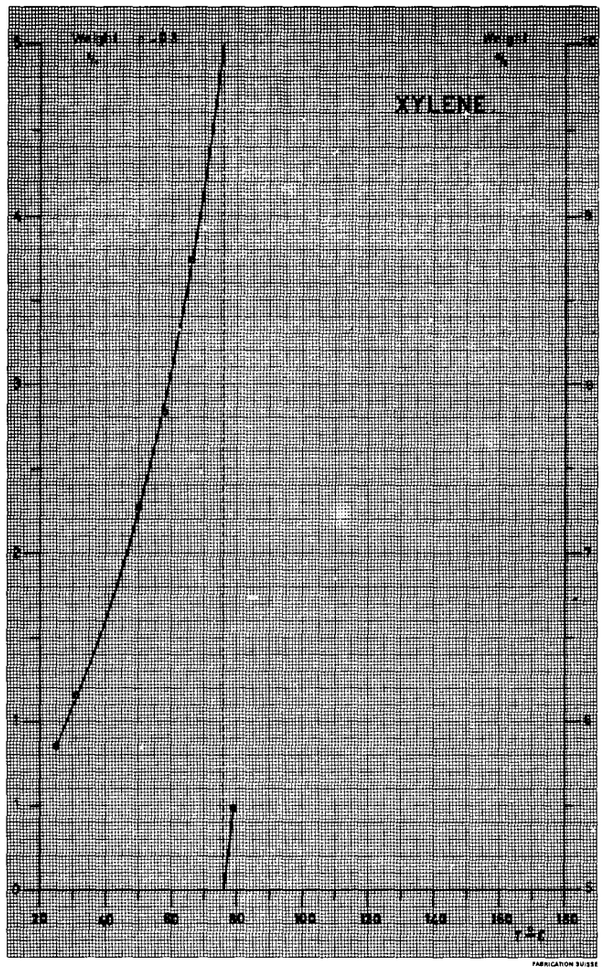

BENZENE.
FABRICATION SUISSE

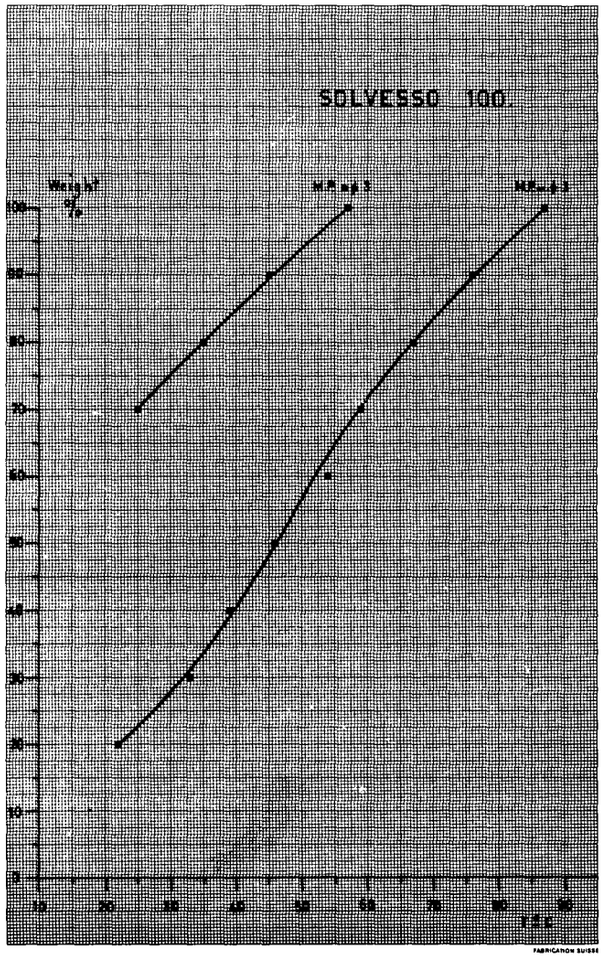

BENZENE.
FABRICATION SUISSE

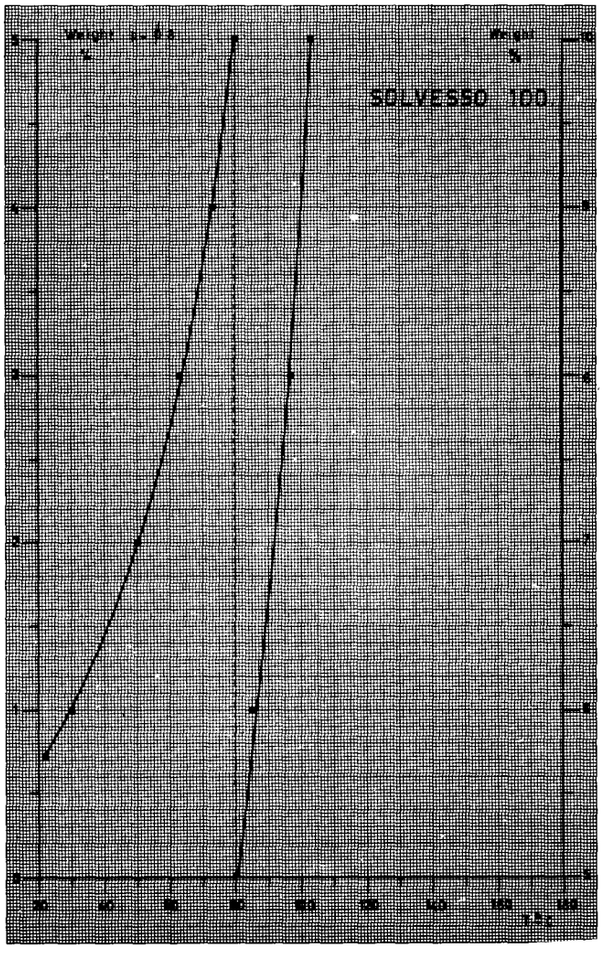

TOLUENE.
FABRICATION SUISSE

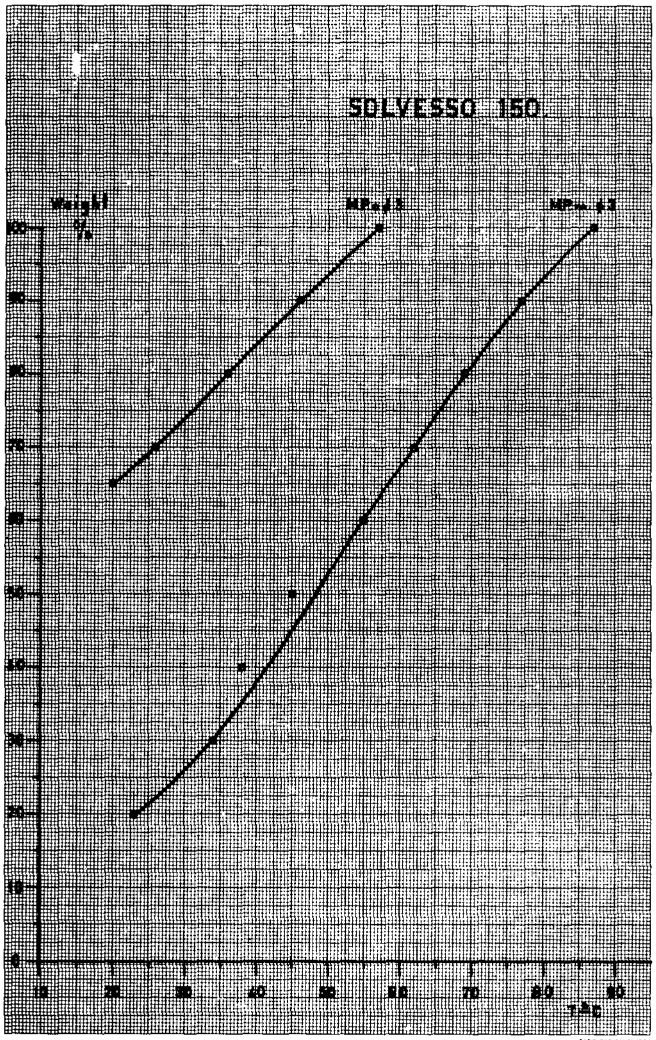

TOLUENE.
FABRICATION SUISSE

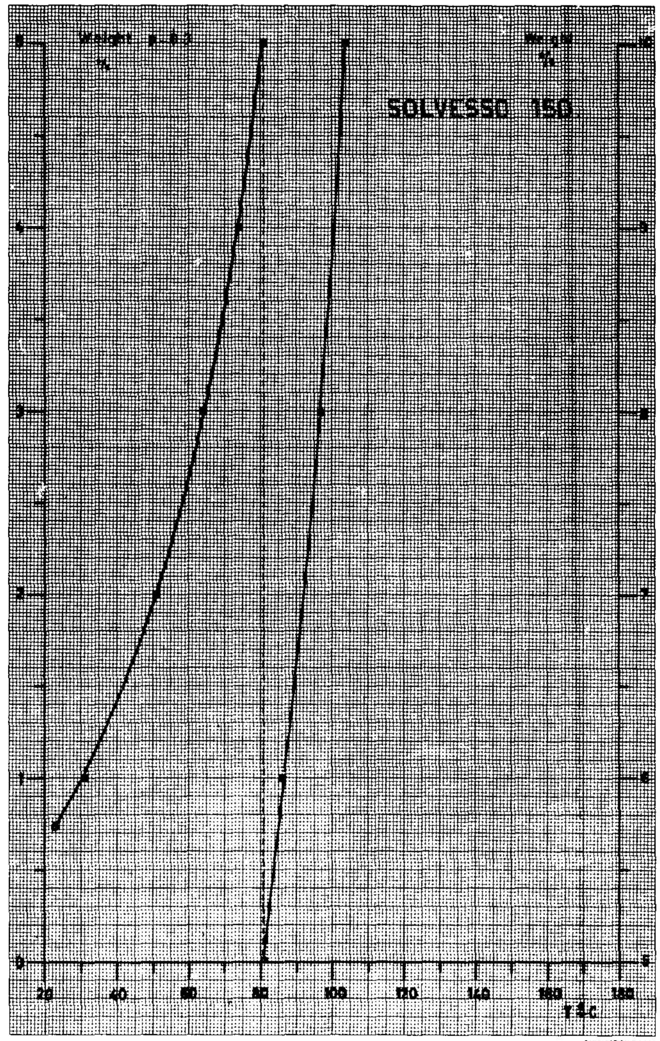

XYLENE.
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T°C
FABRICATION SUISSE

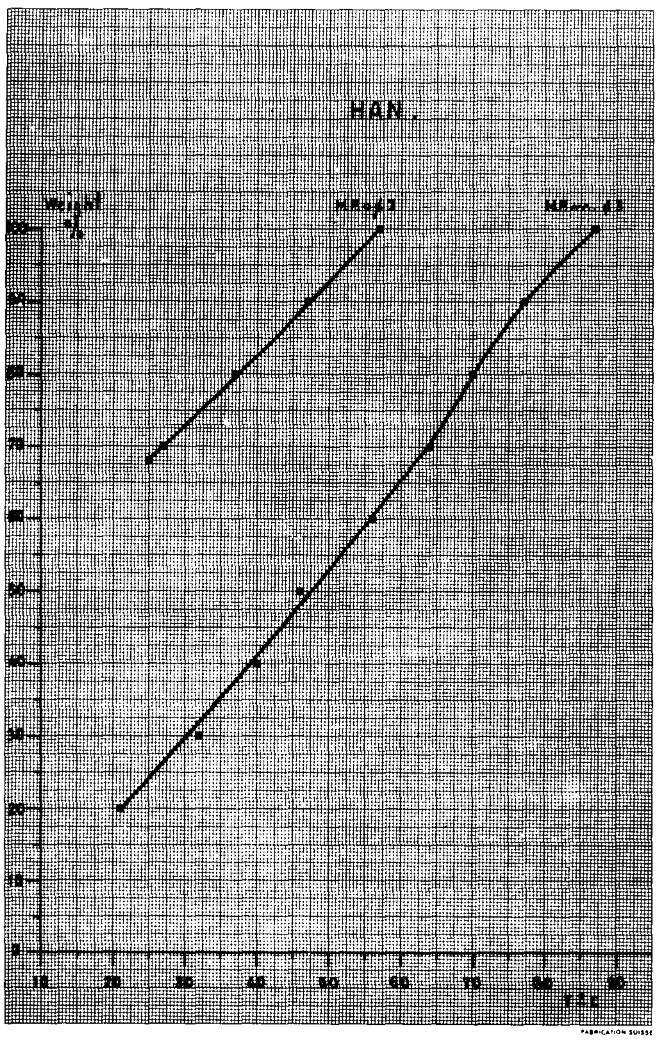

XYLENE.
Weight %
Weight %
5
4
3
2
1
0
20
40
60
80
100
120
140
160
180
T°C
FABRICATION SUISSE

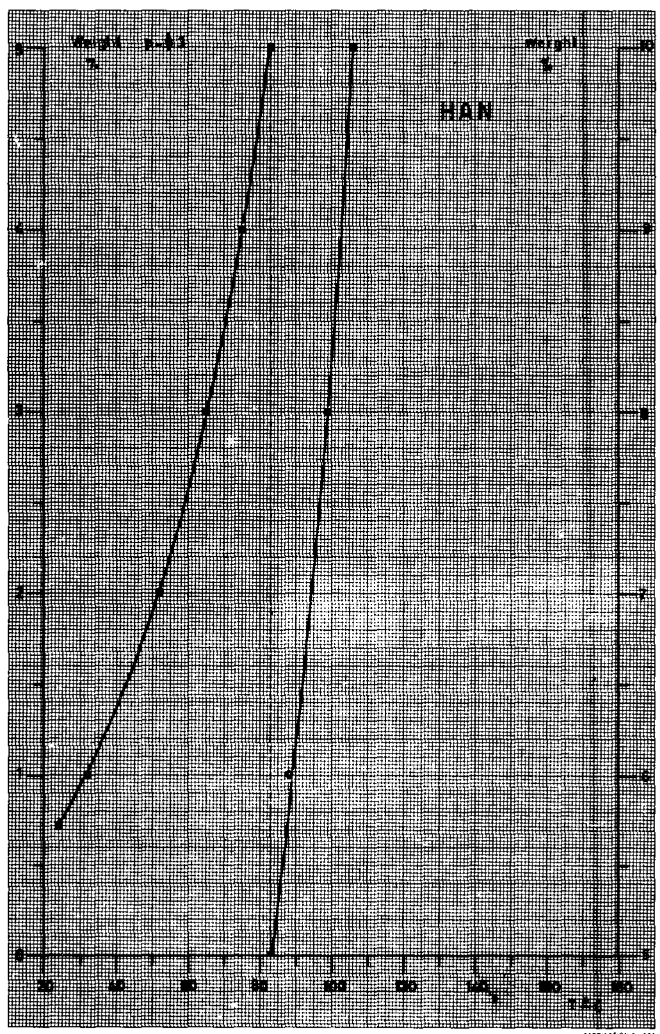

SOLVESSO 100.
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T°C
FABRICATION SUISSE

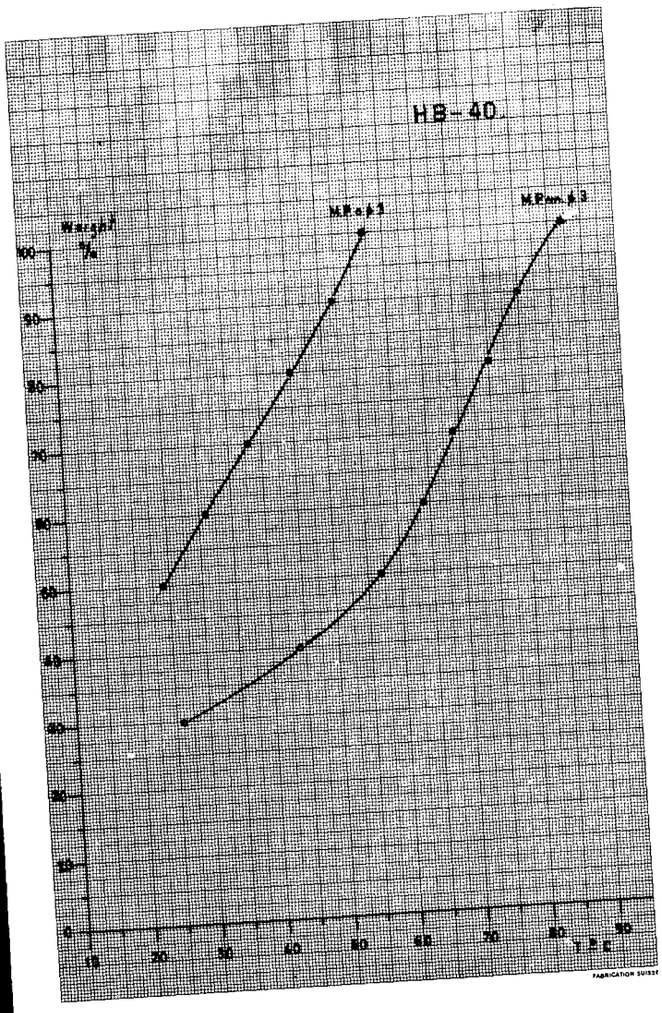

SOLVESSO 100.
Weight %
Weight %
5
4
3
2
1
0
20
40
60
80
100
120
140
160
180
T°C

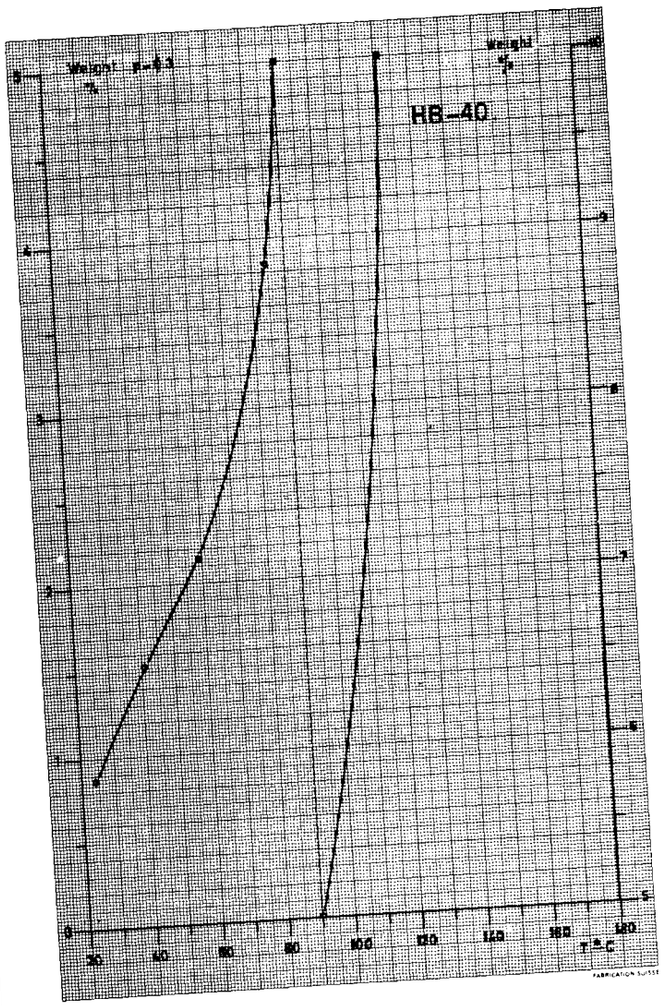

SOLVESSO 150.
Weight
%
T °C

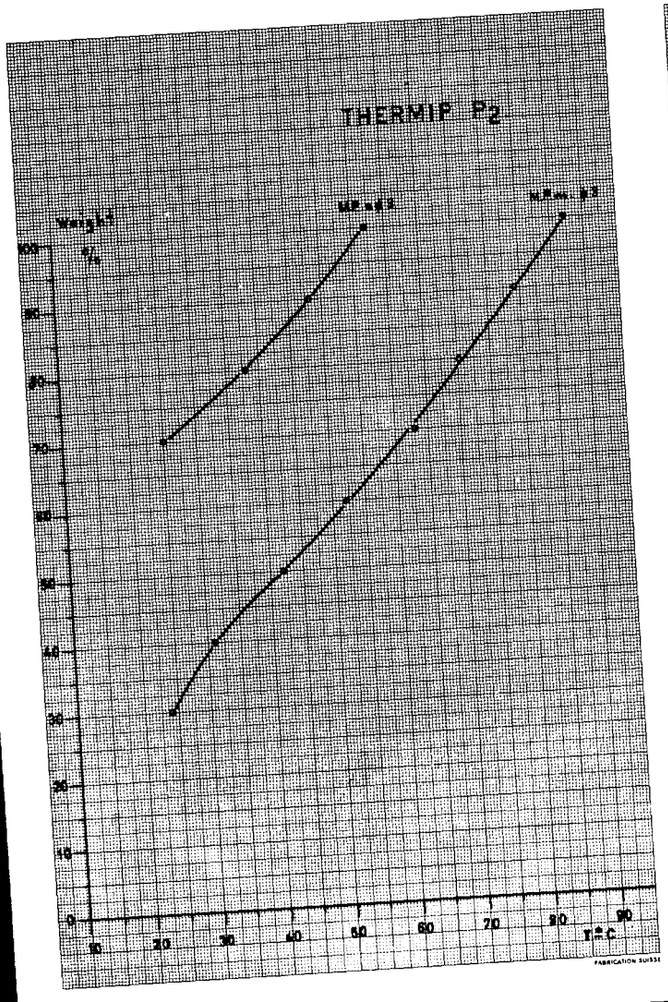

SOLVESSO 150.
Weight
%
T °C

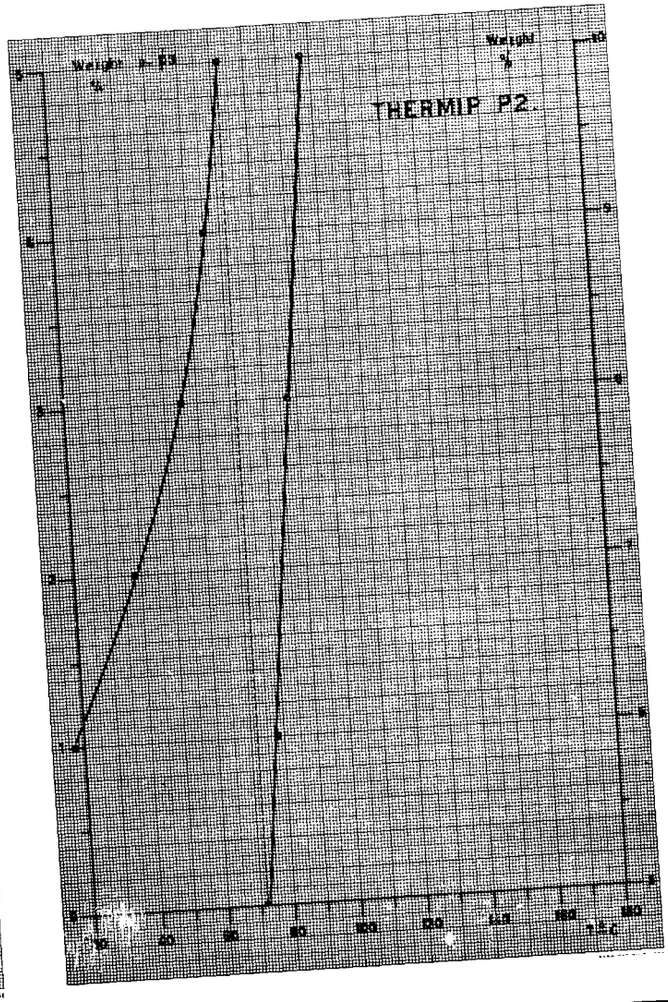

HAN.
Weight
%
T °C

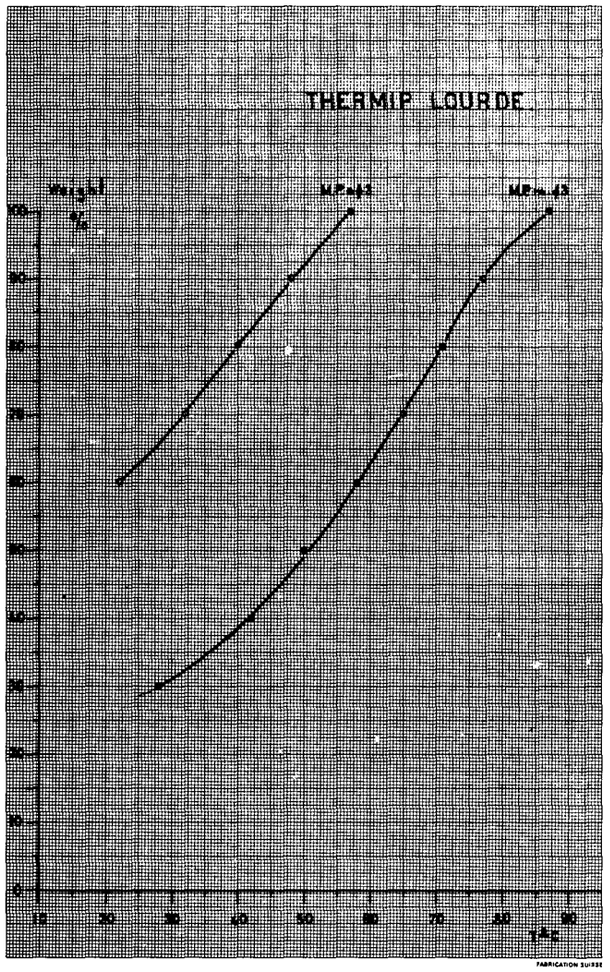

HAN
Weight
%
T °C

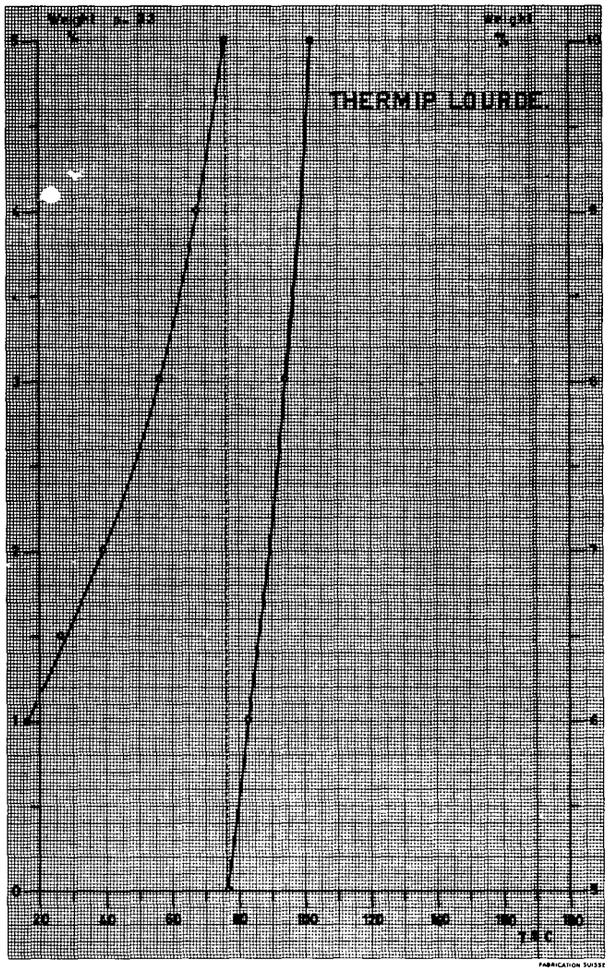

HB-40
Weight %
M.P. o.f.3
M.P.m.f.3
T°C

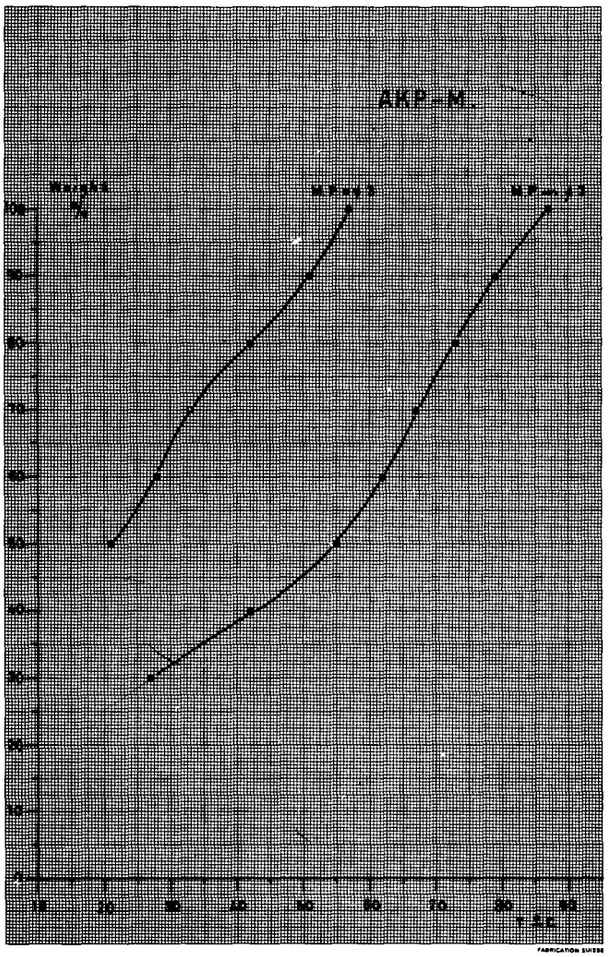

HB-40.
Weight %
T°C

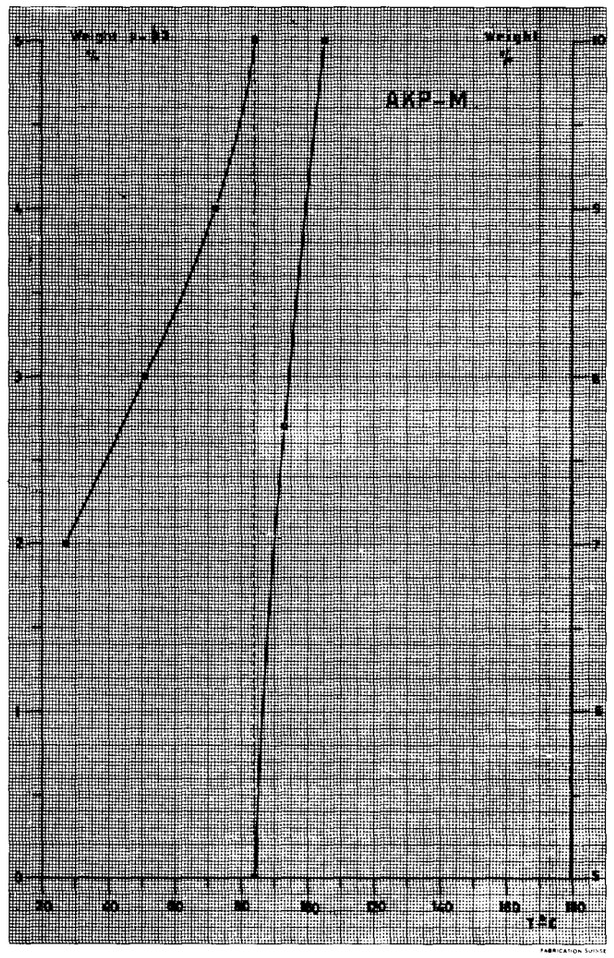

THERMIP P2
Weight %
M.P. o.f.3
M.P.m.f.3
T°C

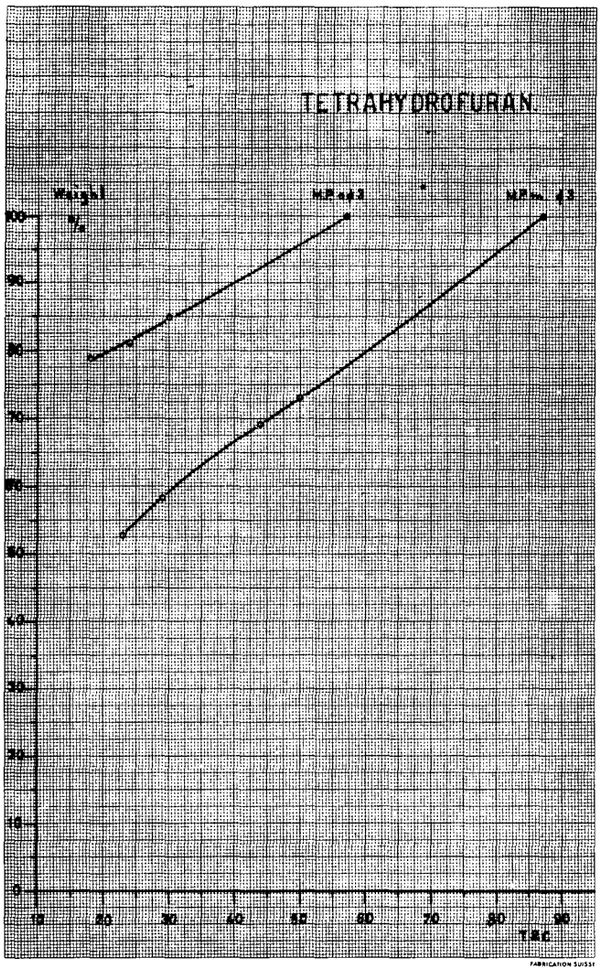

THERMIP P2.
Weight %
T°C

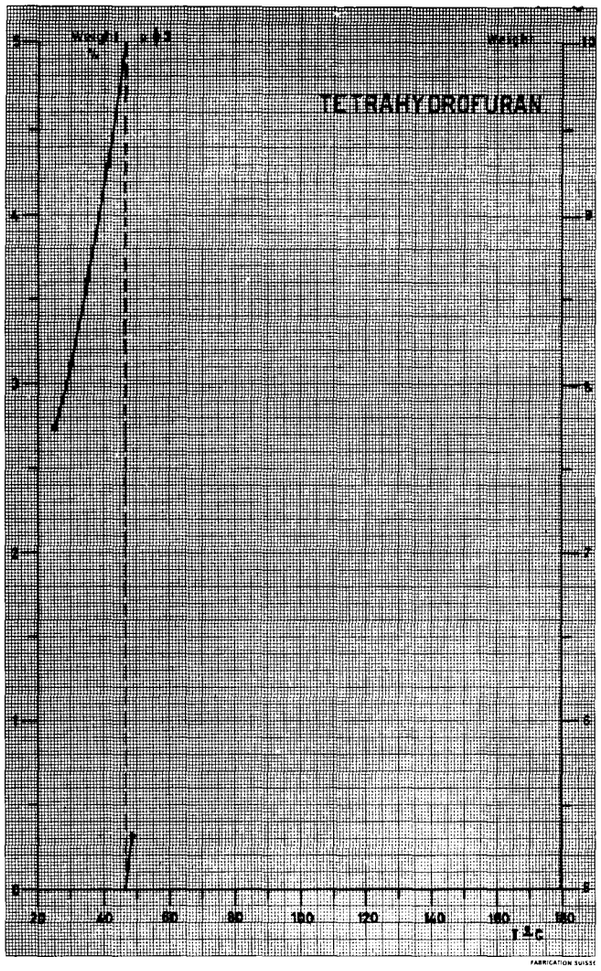

THERMIP LOURDE
Weight %
MP-43
MP-43
T.G.C
FABRICATION SUISSE

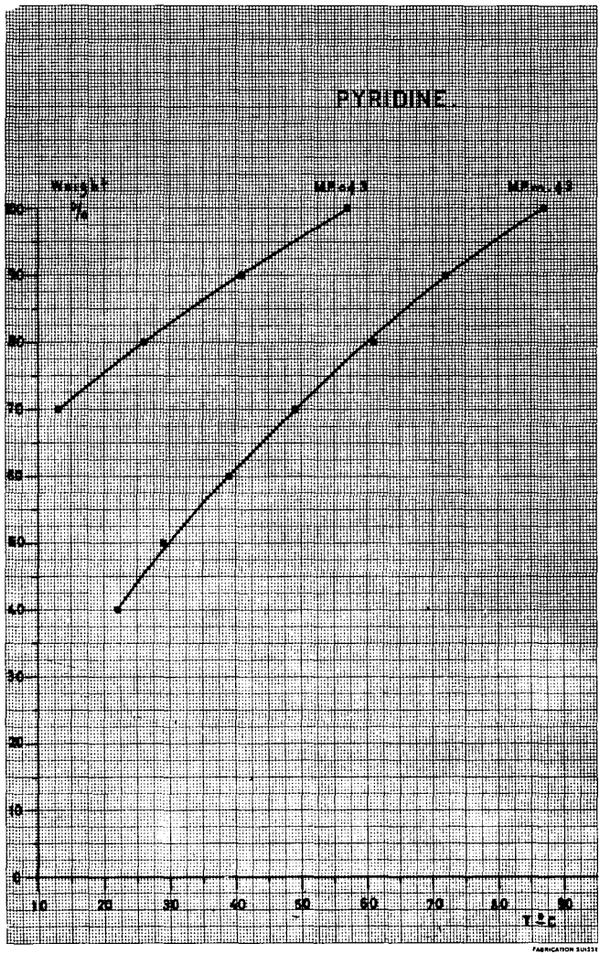

THERMIP LOURDE
T.G.C
FABRICATION SUISSE

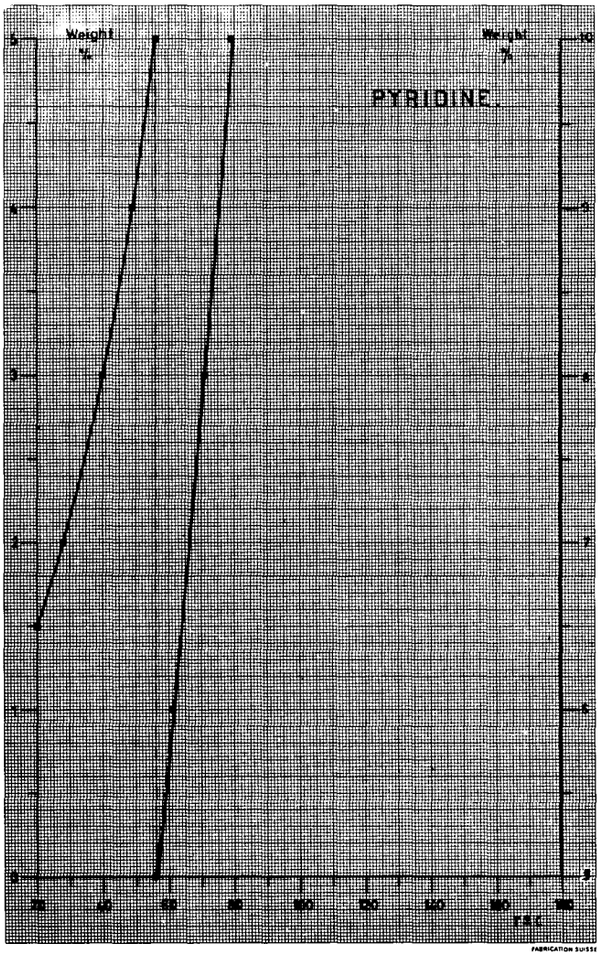

AKP-M.
Weight %
T.G.C
FABRICATION SUISSE

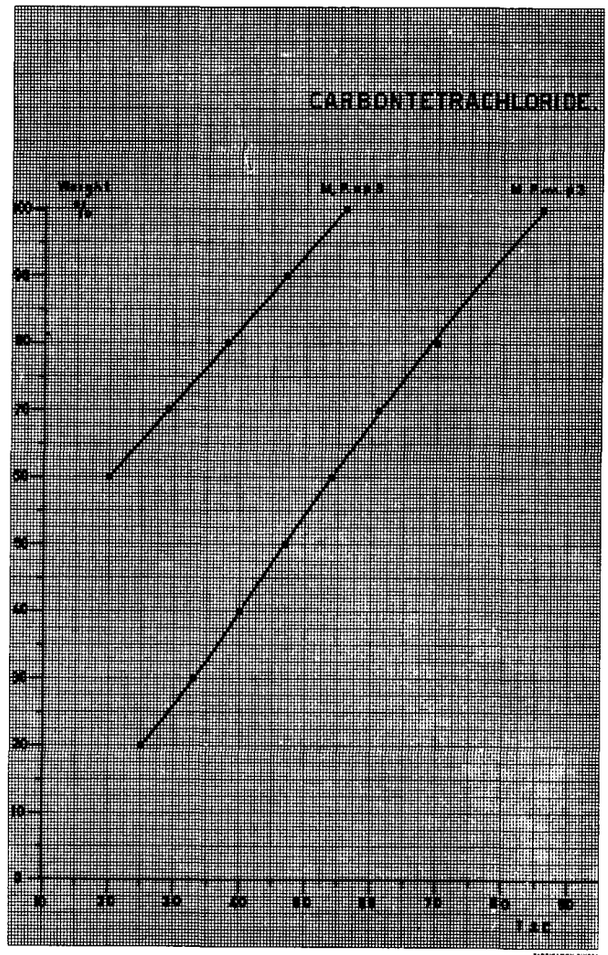

AKP-M.
T.G.C
FABRICATION SUISSE

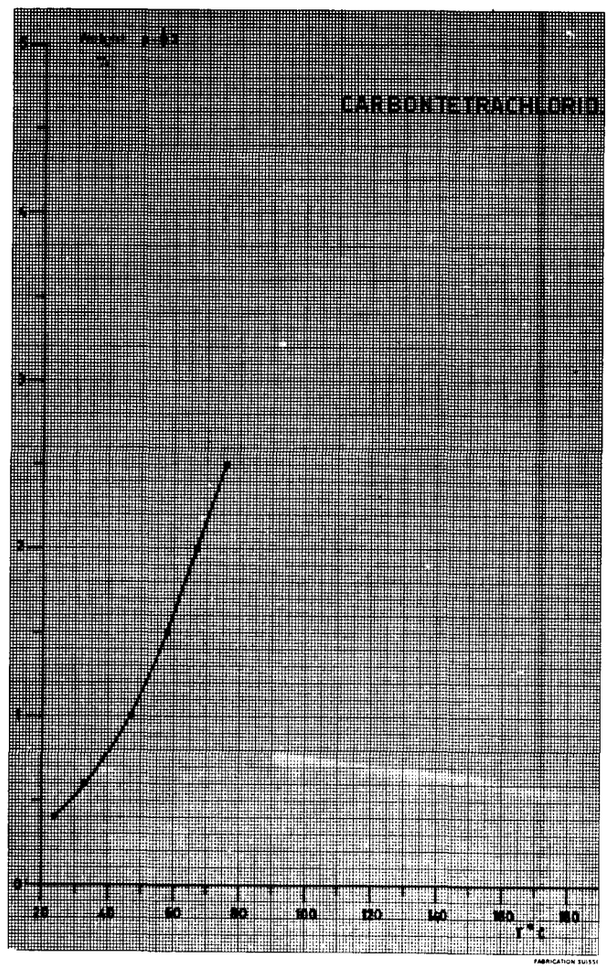

TETRAHYDROFURAN
Weight %
MP=43
MP=43
T°C
FABRICATION SUISSE

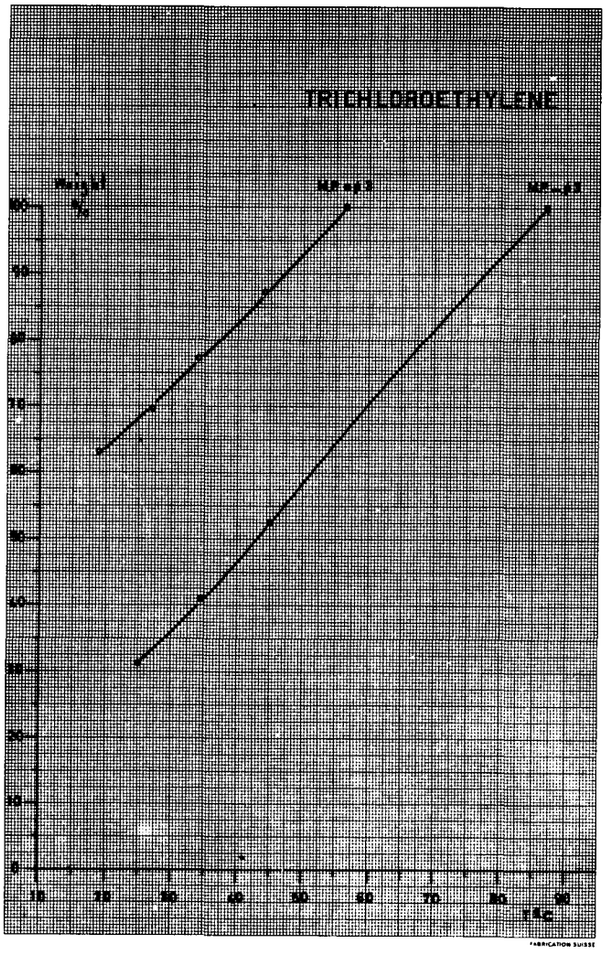

TETRAHYDROFURAN
Weight %
Weight %
T°C
FABRICATION SUISSE

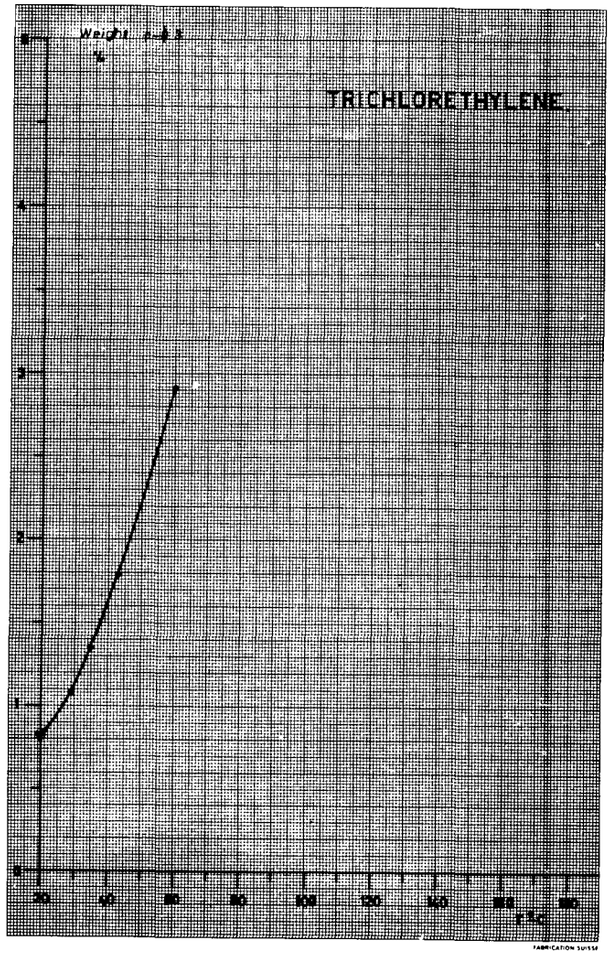

PYRIDINE
Weight %
MP=43
MP=43
T°C
FABRICATION SUISSE

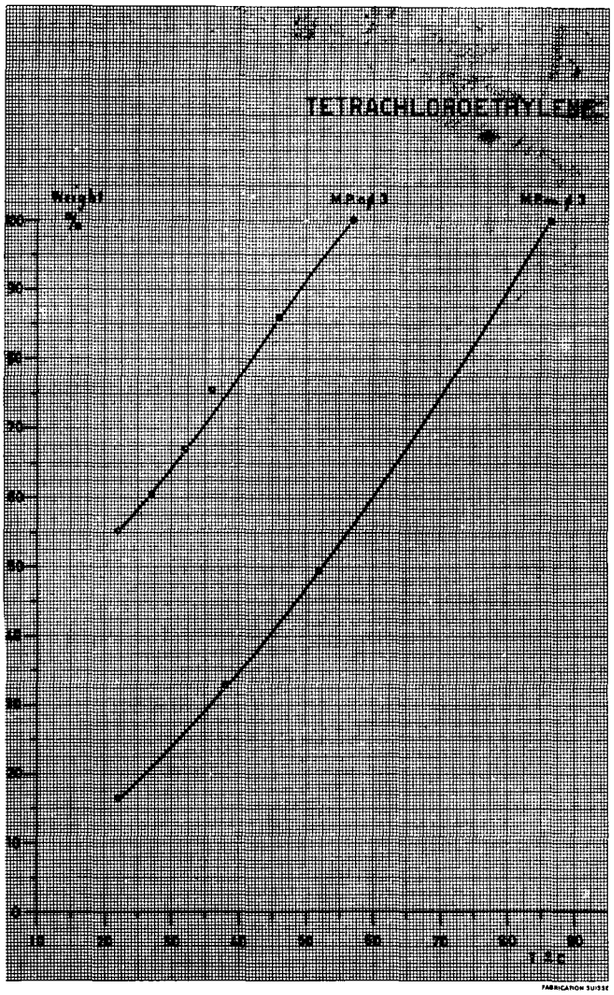

PYRIDINE
Weight %
Weight %
T°C
FABRICATION SUISSE

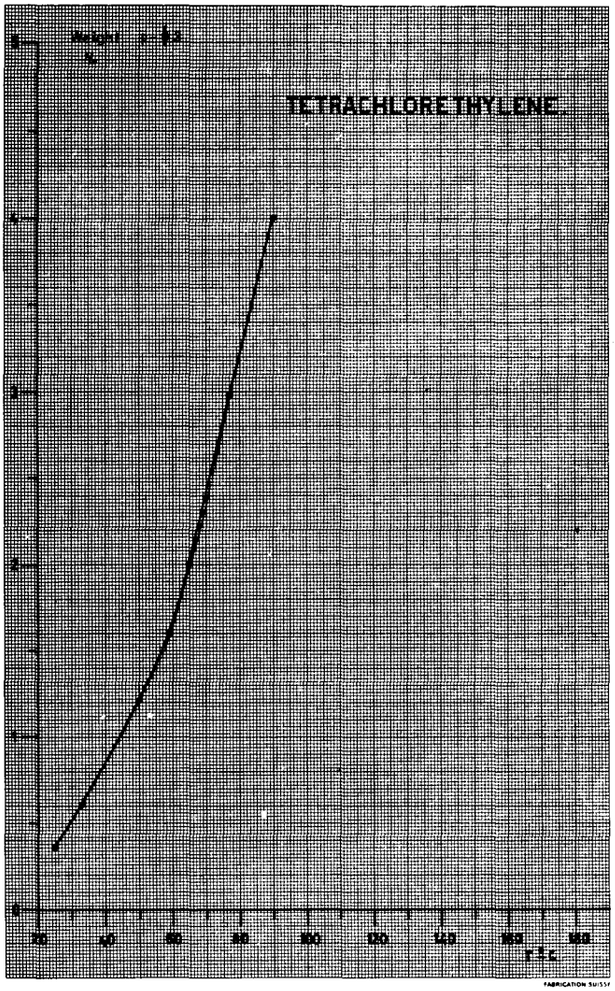

CARBONTETRACHLORIDE
Weight
%
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
°C
FABRICATION SUISSE

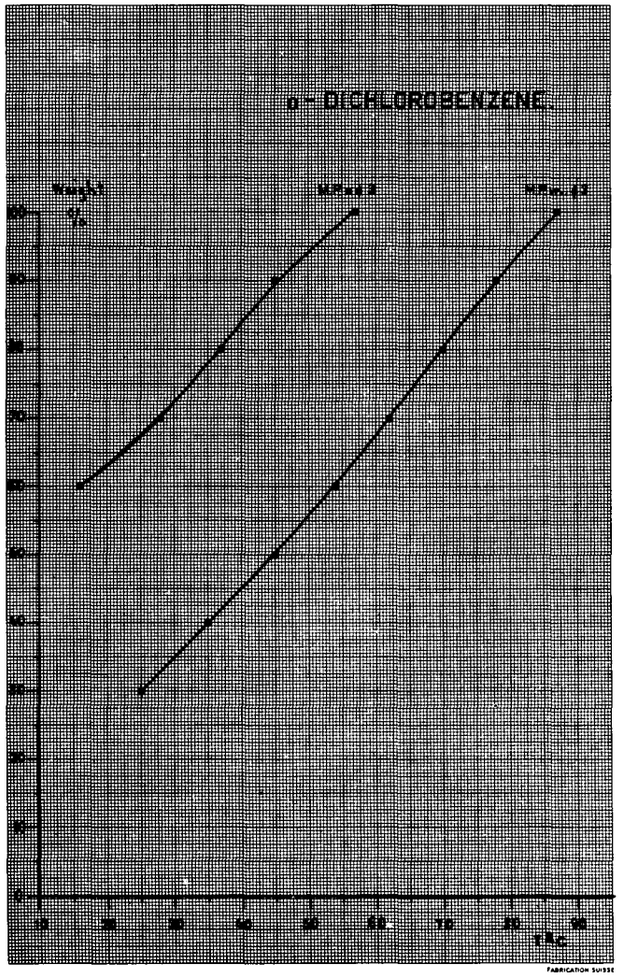

CARBONTETRACHLORID
4
3
2
1
0
20
40
60
80
100
120
140
160
180
°C
FABRICATION SUISSE

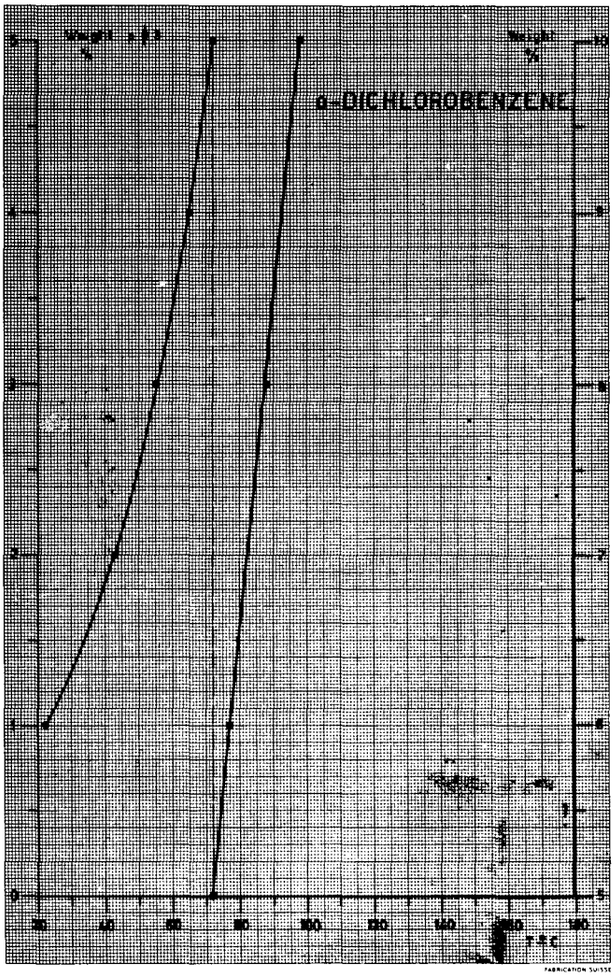

TRICHLORAETHYLENE
Weight
%
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
°C
FABRICATION SUISSE

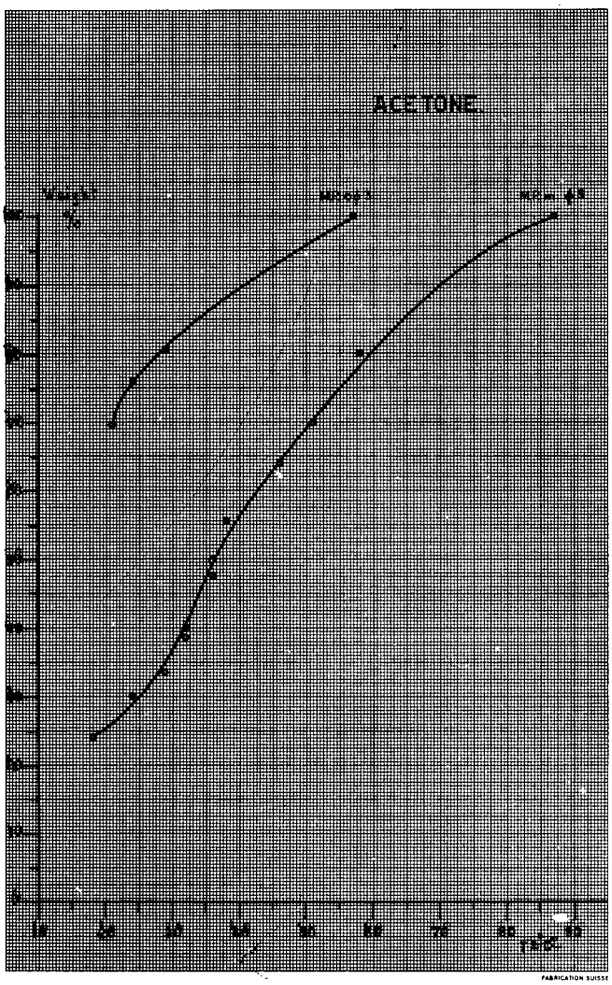

TRICHLORETHYLENE
4
3
2
1
0
20
40
60
80
100
120
140
160
180
°C
FABRICATION SUISSE

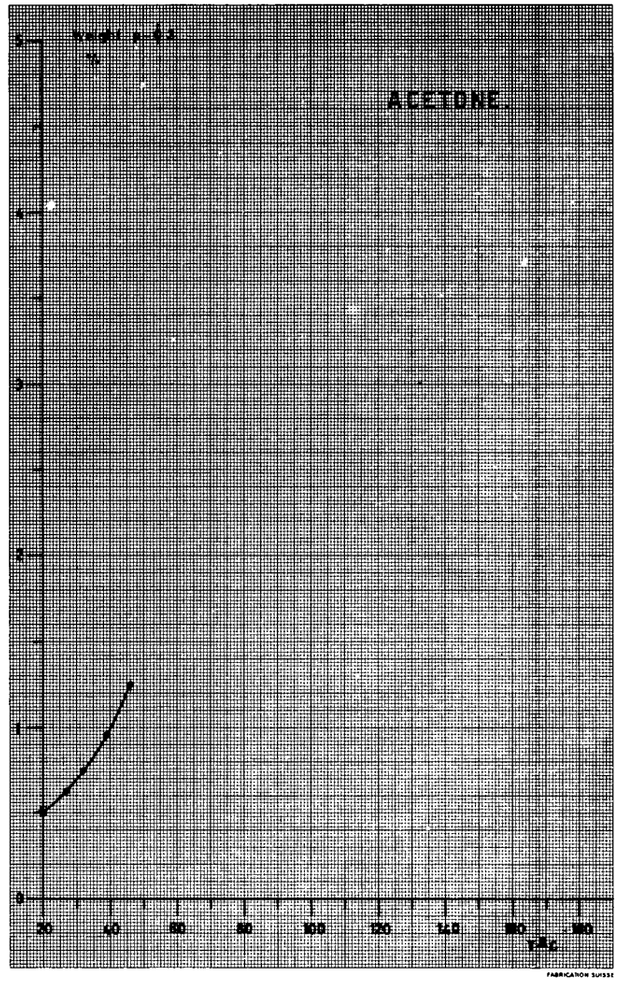

TETRACHLORDETHYLENE
Weight %
FABRICATION SUISSE

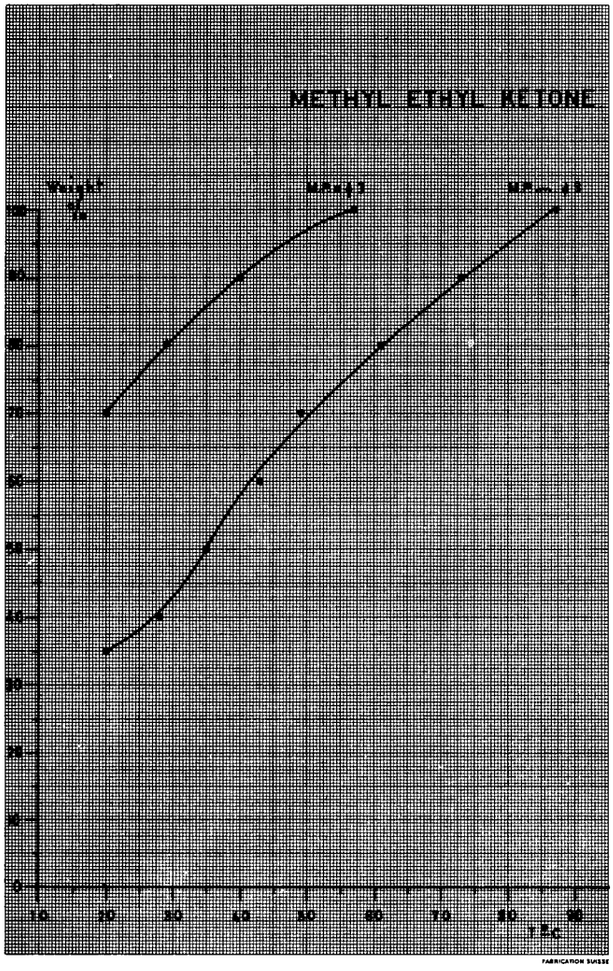

TETRACHLORETHYLENE
FABRICATION SUISSE

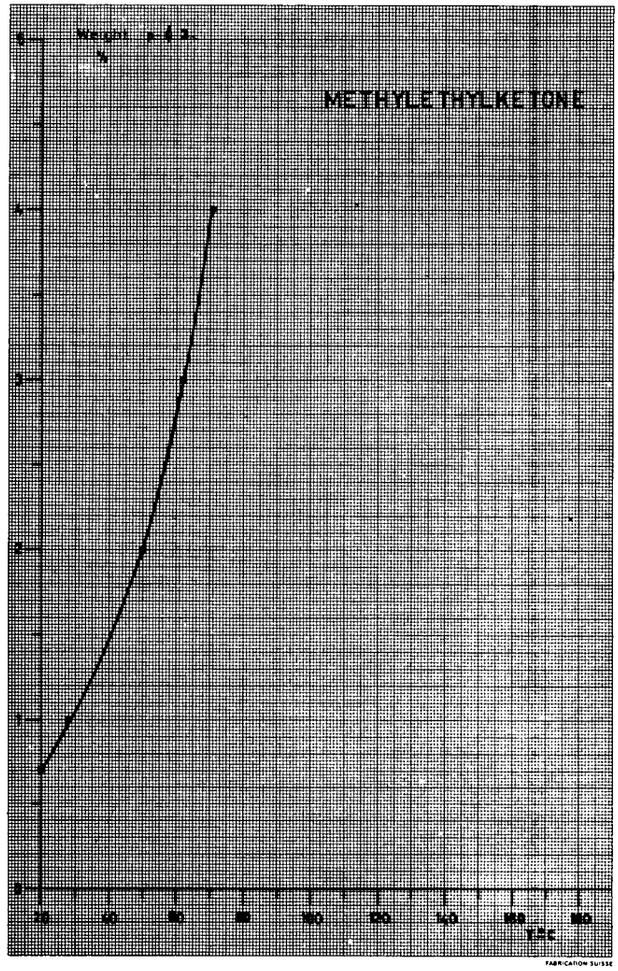

o-DICHLOROBENZENE
FABRICATION SUISSE

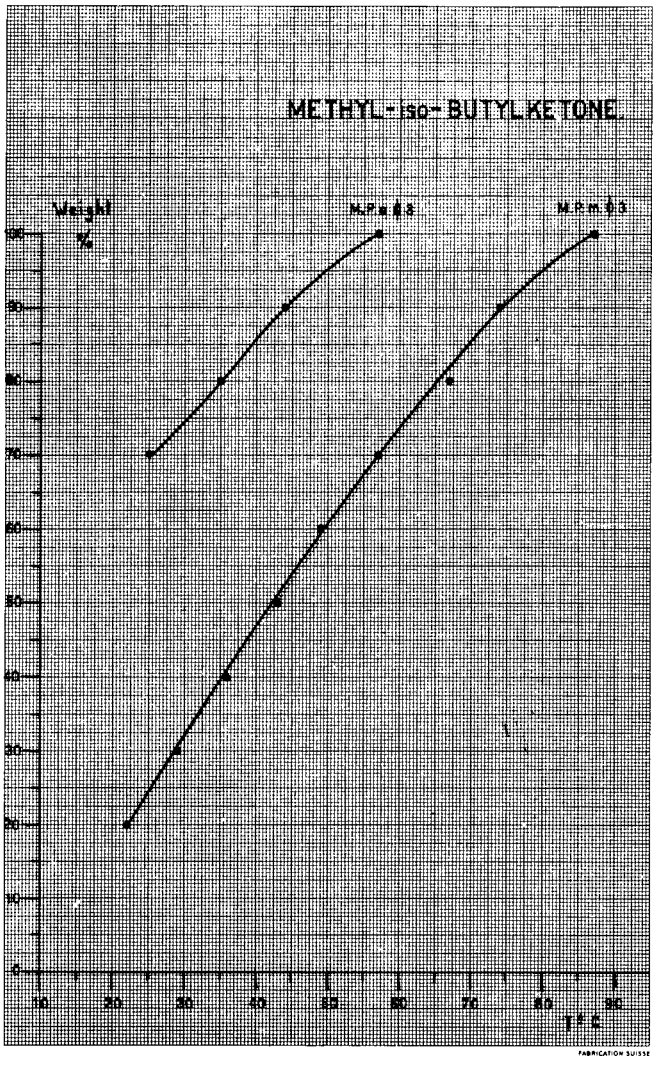

o-DICHLOROBENZENE
FABRICATION SUISSE

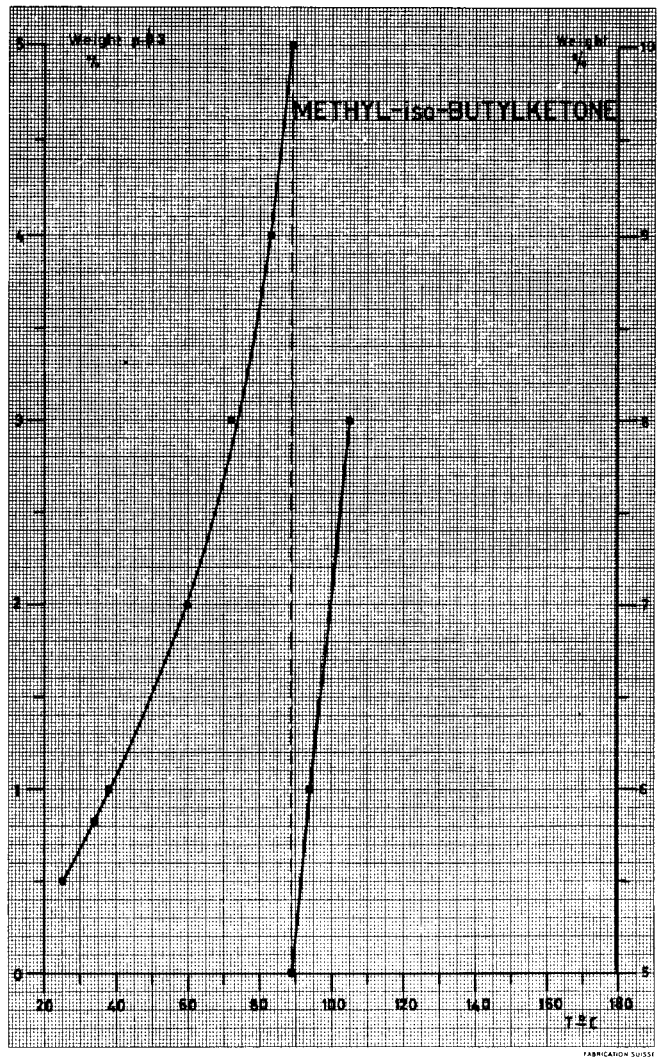

ACETONE
Weight %
FABRICATION SUISSE

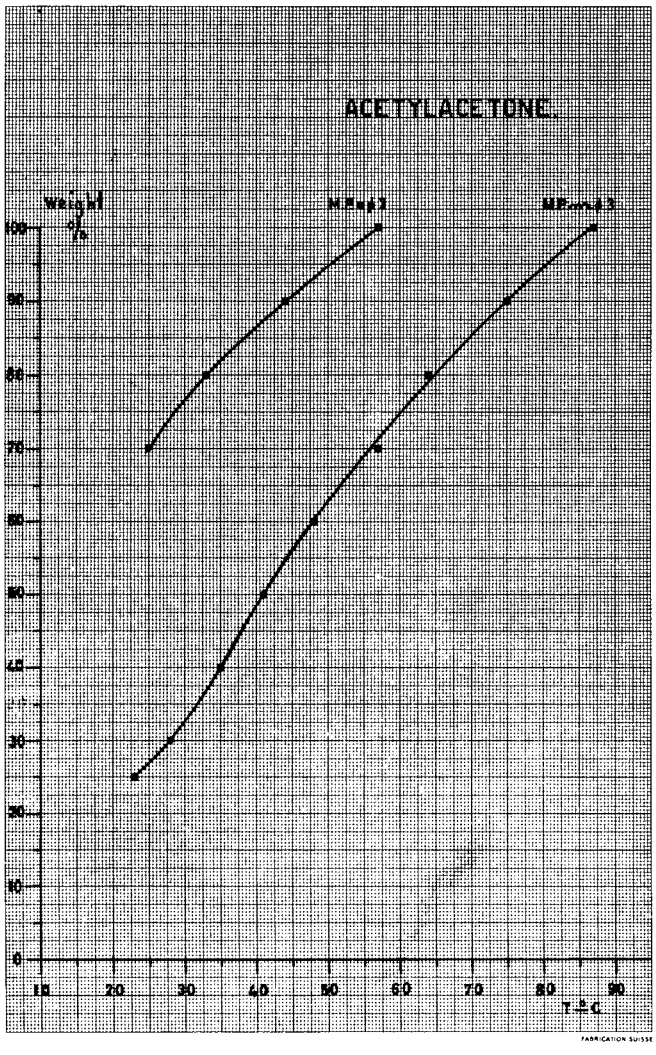

ACETONE
FABRICATION SUISSE

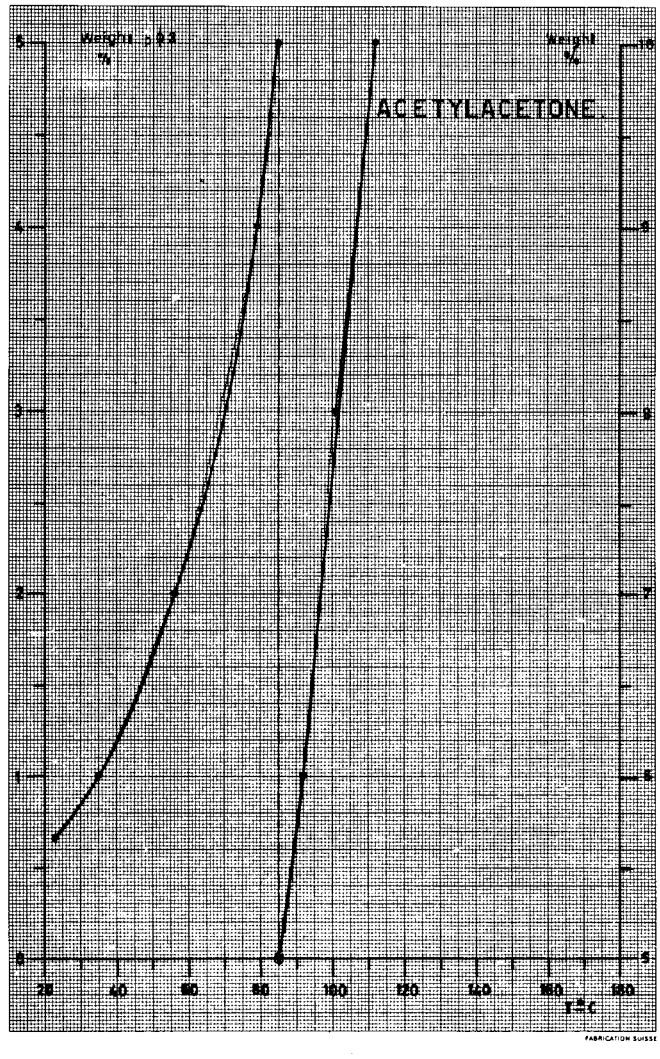

METHYL ETHYL KETONE
Weight %
FABRICATION SUISSE

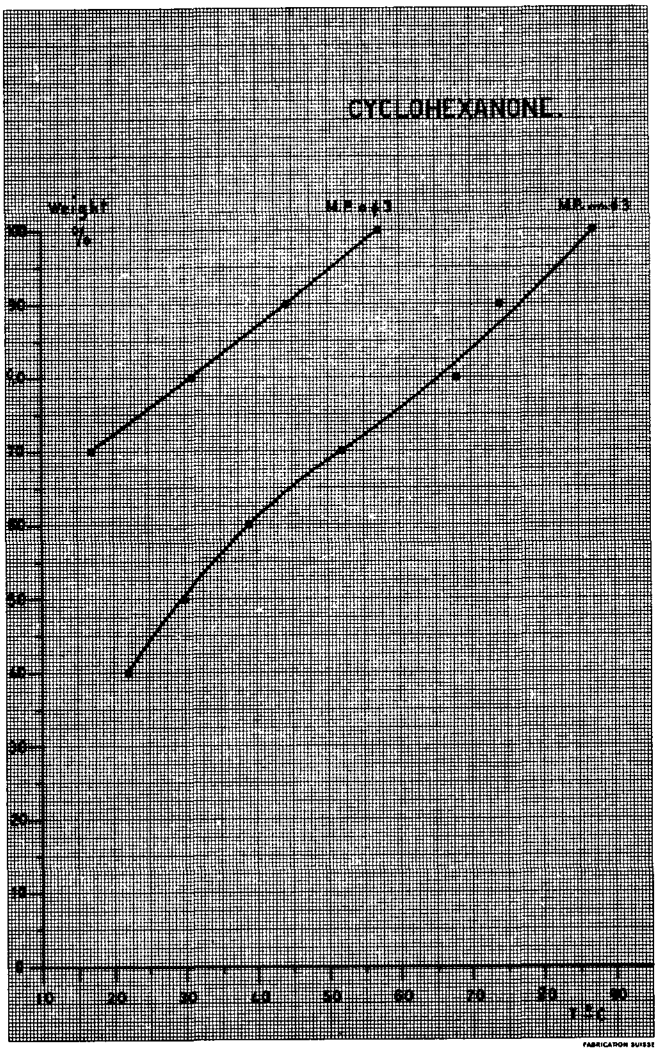

METHYLETHYLKETONE
FABRICATION SUISSE

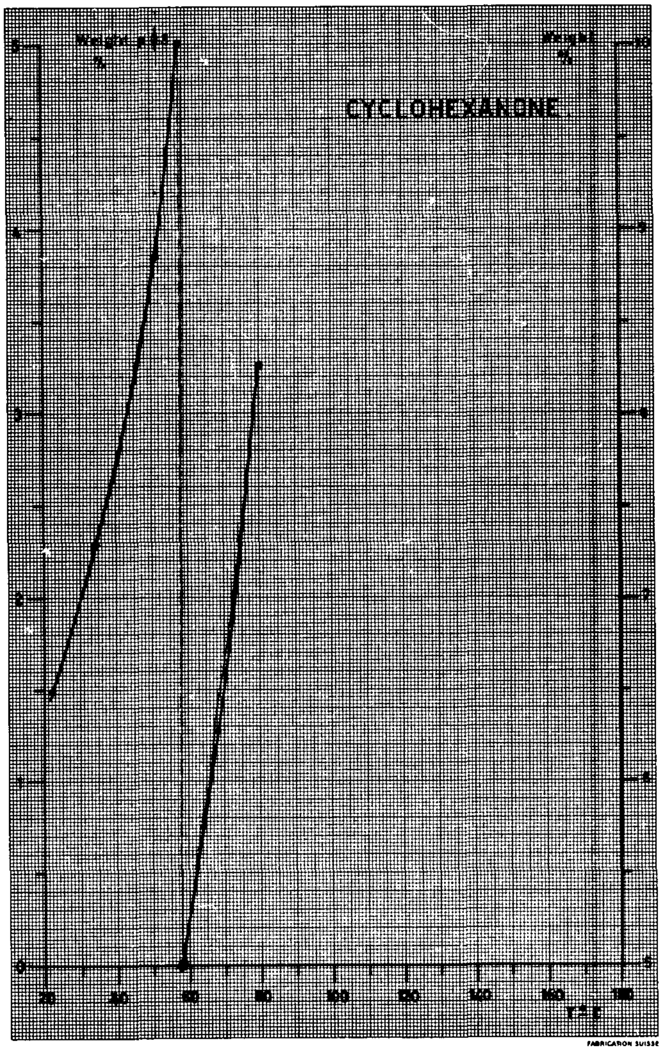

METHYL-iso-BUTYLKETONE
Weight %
T° C
FABRICATION SUISSE

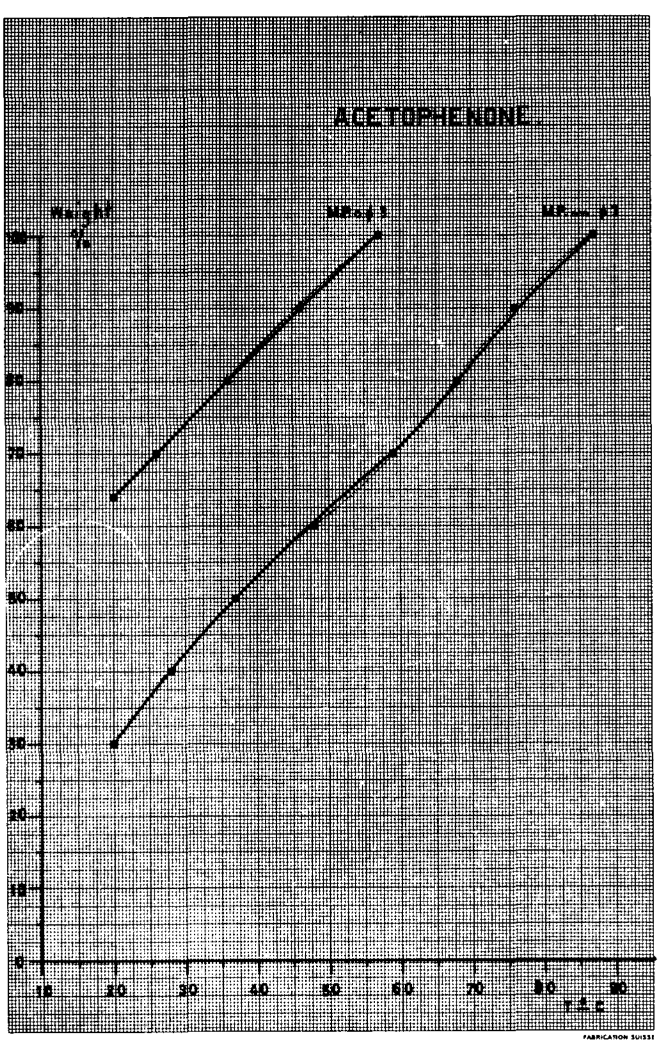

METHYL-iso-BUTYLKETONE
T°C
FABRICATION SUISSE

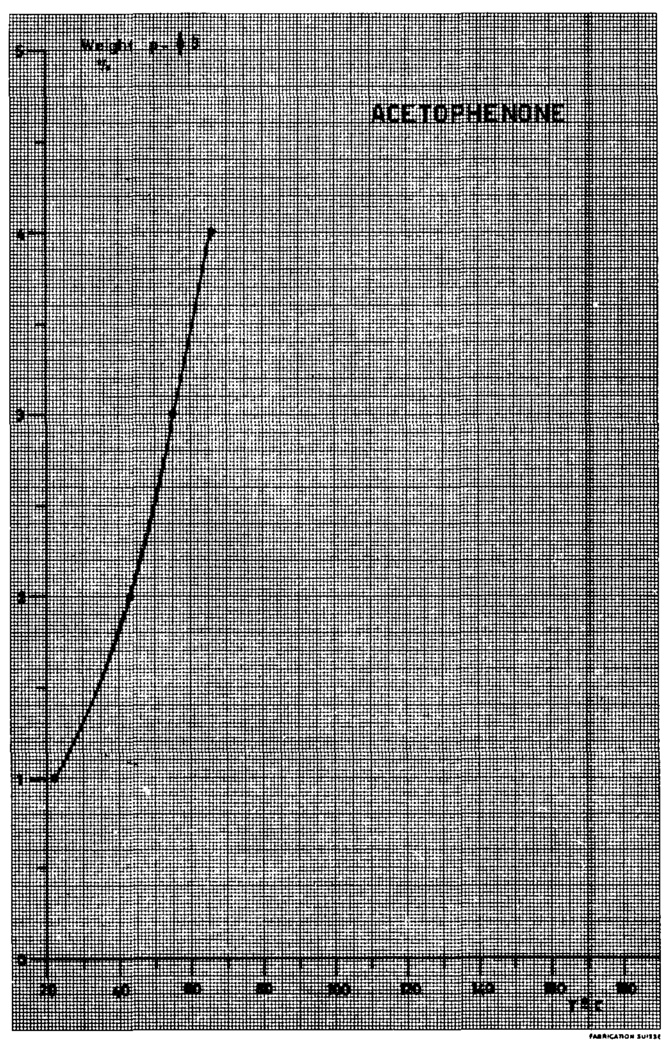

ACETYLACETONE
Weight %
T °C
FABRICATION SUISSE

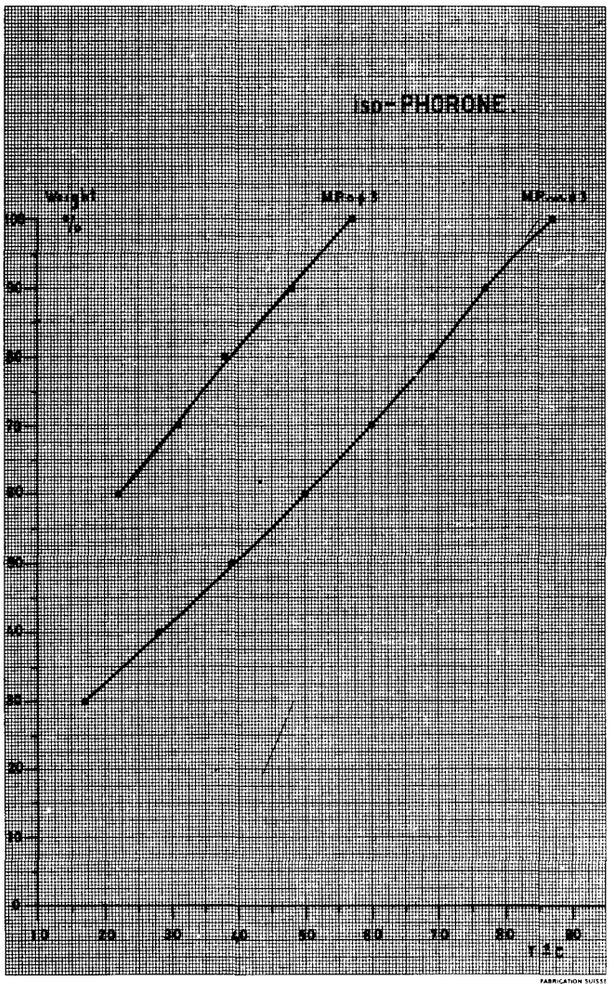

ACETYLACETONE
T°C
FABRICATION SUISSE

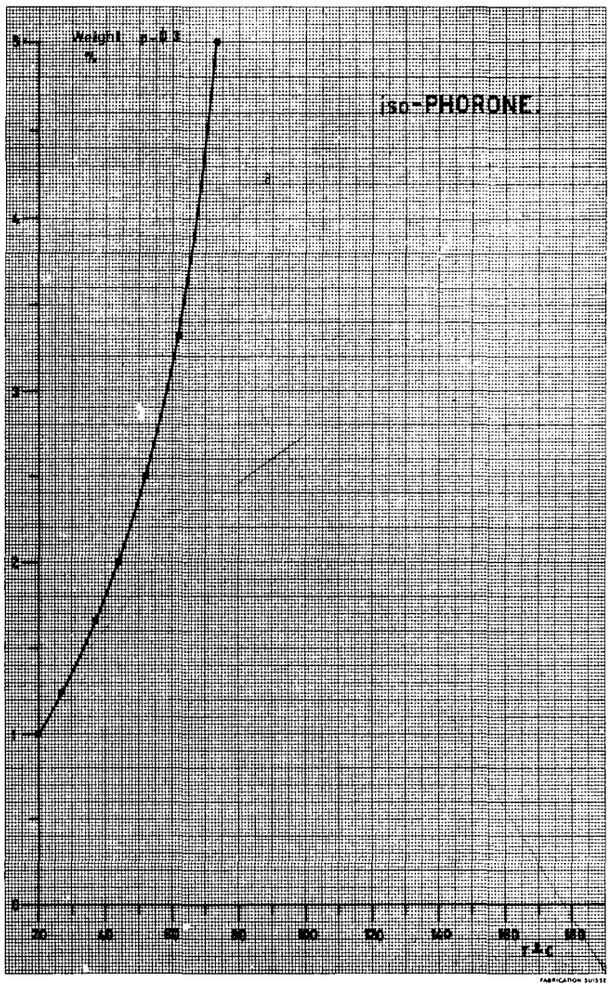

CYCLOHEXANONE.
Weight %
T °C
FABRICATION SUISSE

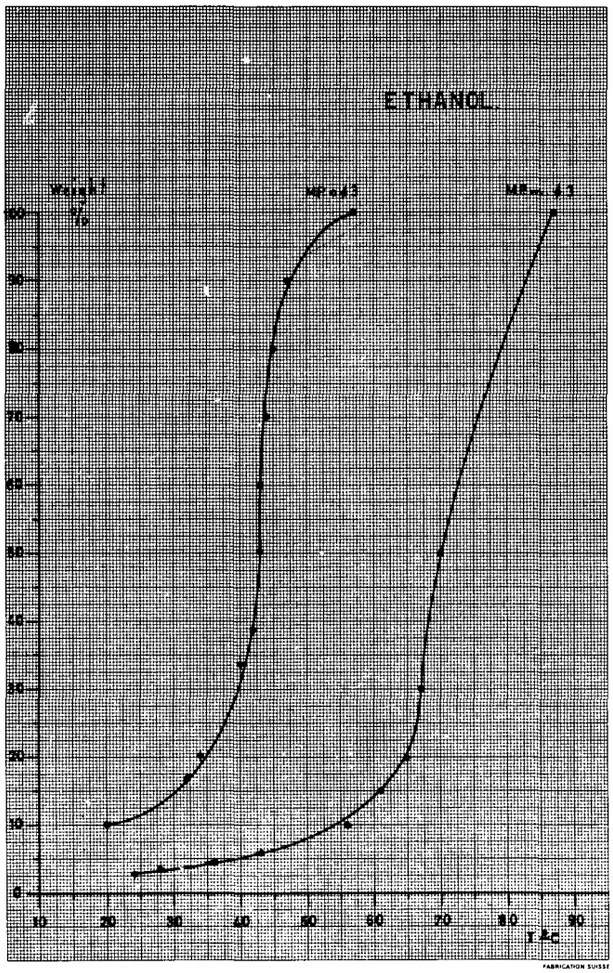

CYCLOHEXANONE.
Weight %
Weight %
T °C
FABRICATION SUISSE

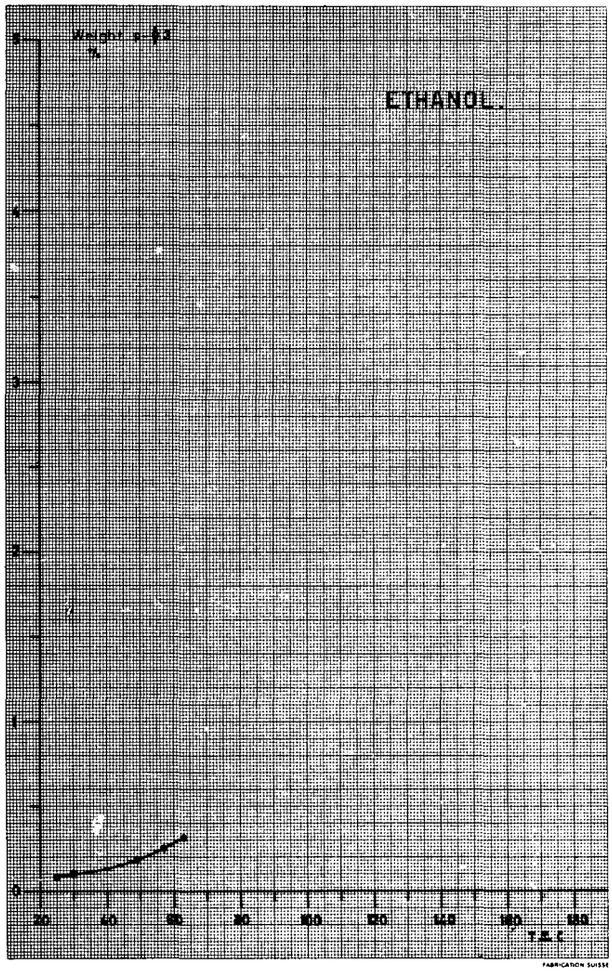

ACETOPHENONE.
Weight %
T °C
FABRICATION SUISSE

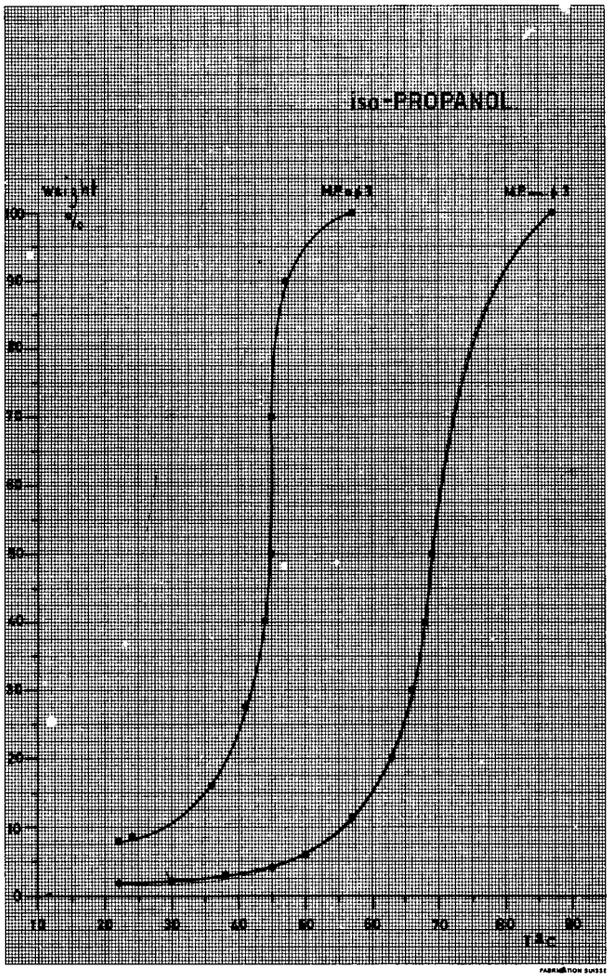

ACETOPHENONE
Weight %
T °C
FABRICATION SUISSE

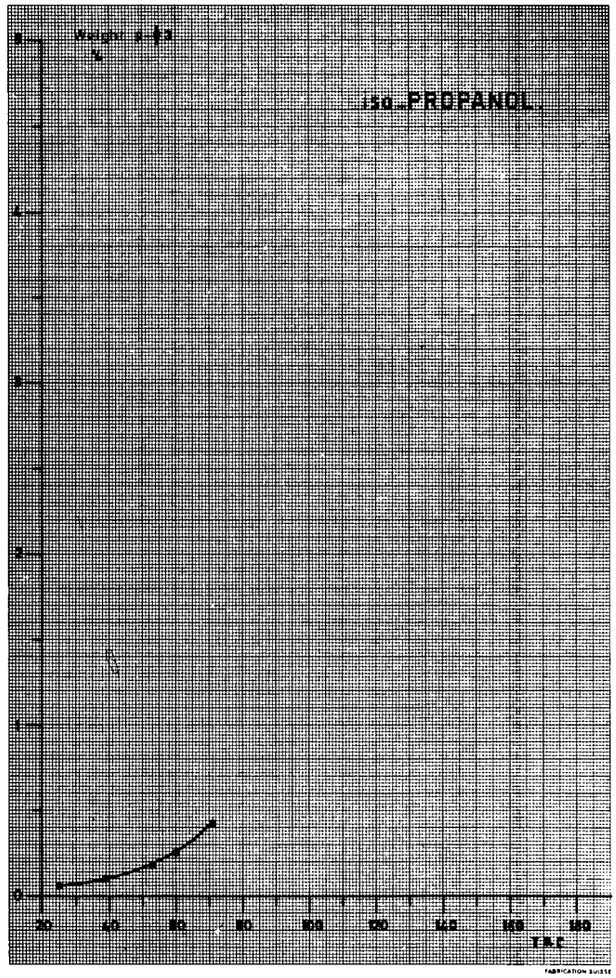

iso-PHORONE.
Weight %
MP=4.9
MP=4.3
T ± C
FABRICATION SUISSE

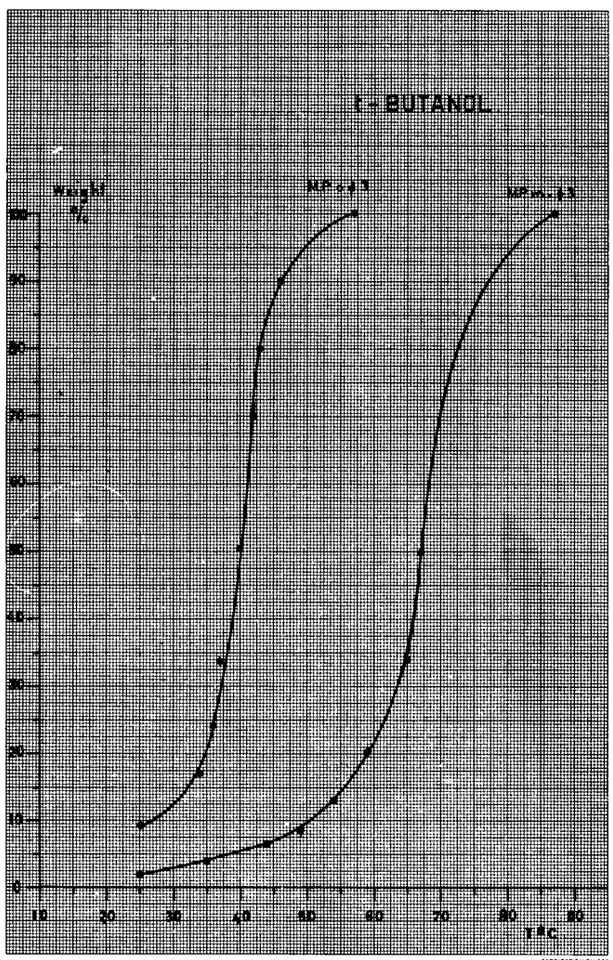

Weight %
iso-PHORONE.
T ± C
FABRICATION SUISSE

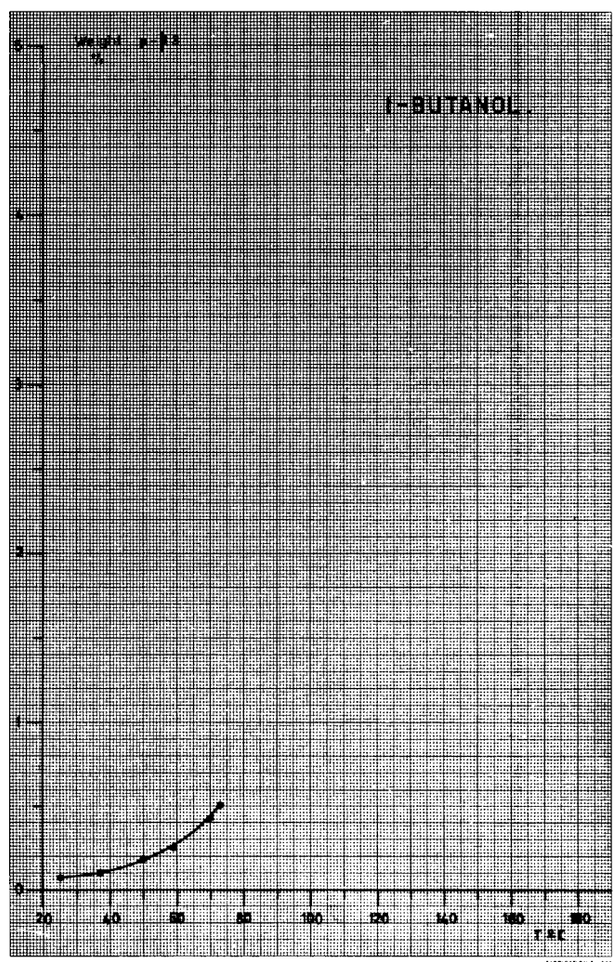

ETHANOL.
Weight %
MP=4.3
MP=4.9
T ± C
FABRICATION SUISSE

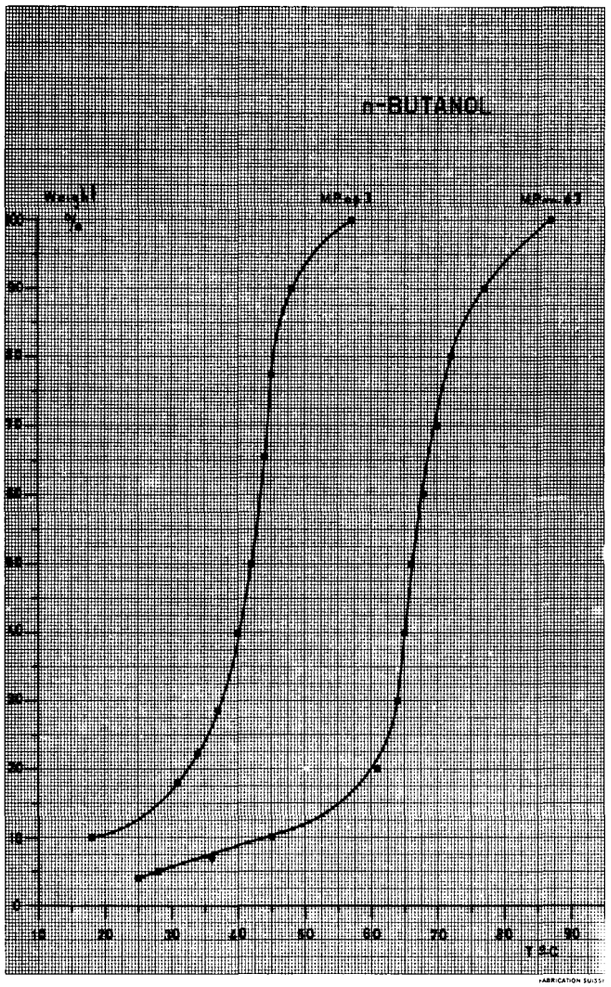

Weight %
ETHANOL.
T ± C
FABRICATION SUISSE

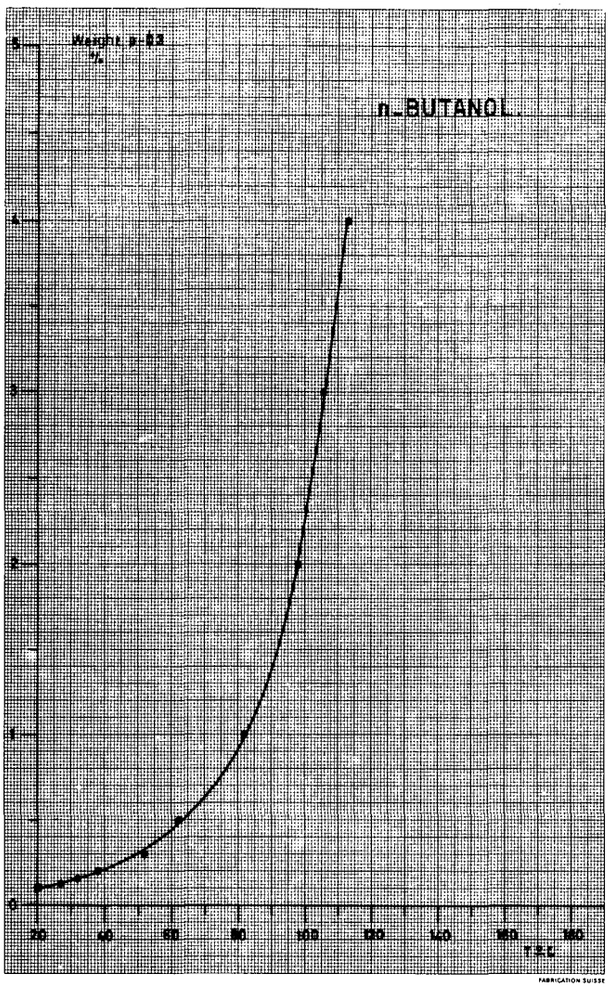

iso-PROPANOL
MP.=+3
MP.=-3
T°C

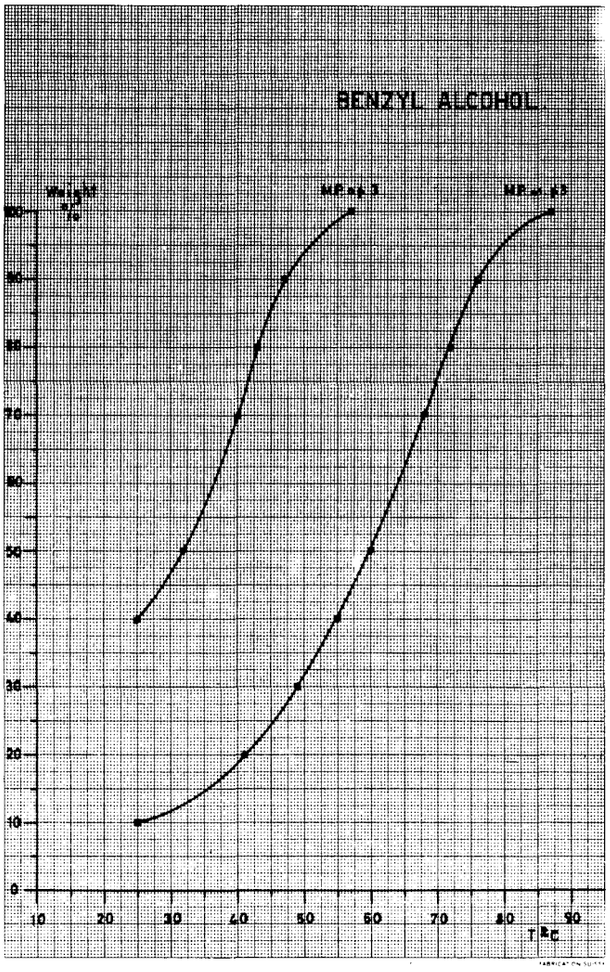

iso-PROPANOL
T°C

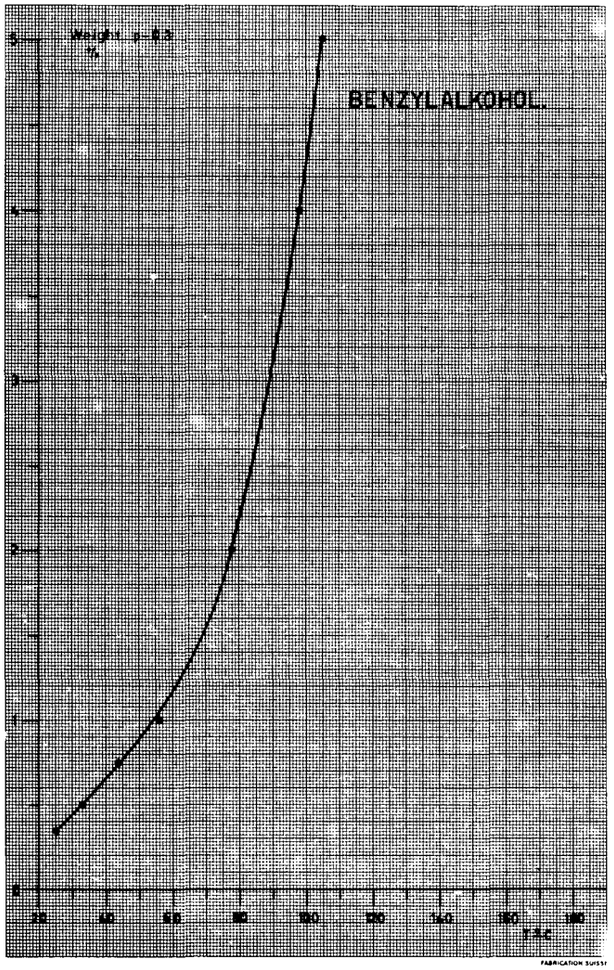

t-BUTANOL
MP.=+3
MP.=-3
T°C

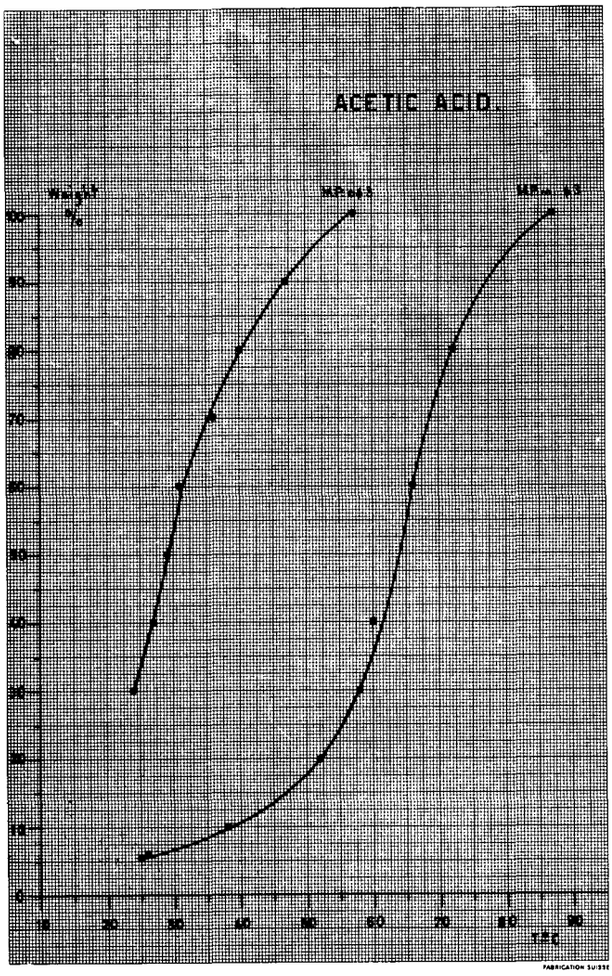

t-BUTANOL
T°C

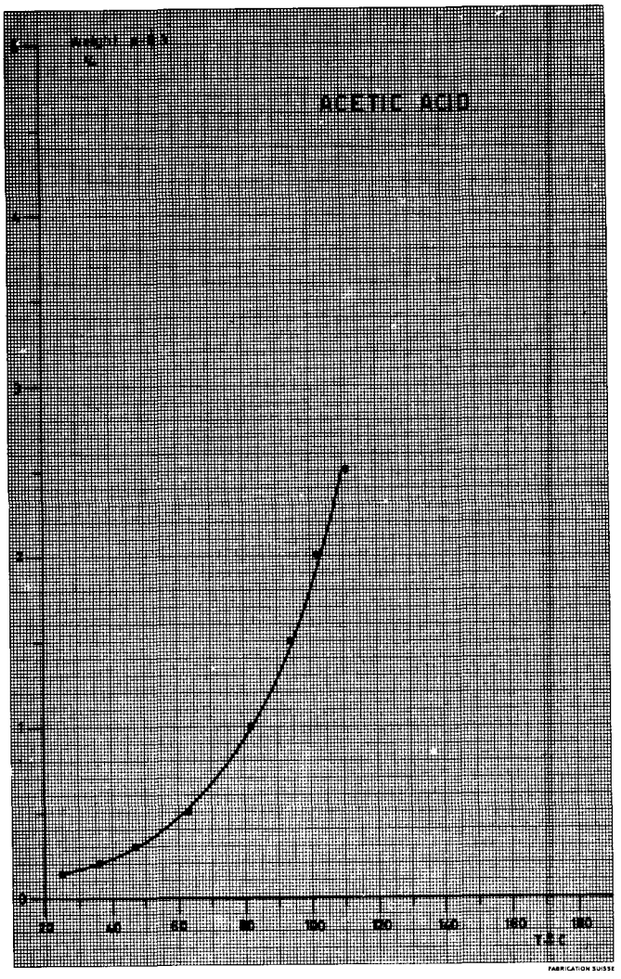

n-BUTANOL
Weight %
MP = 1
MP = 43
T °C
FABRICATION SUISSE

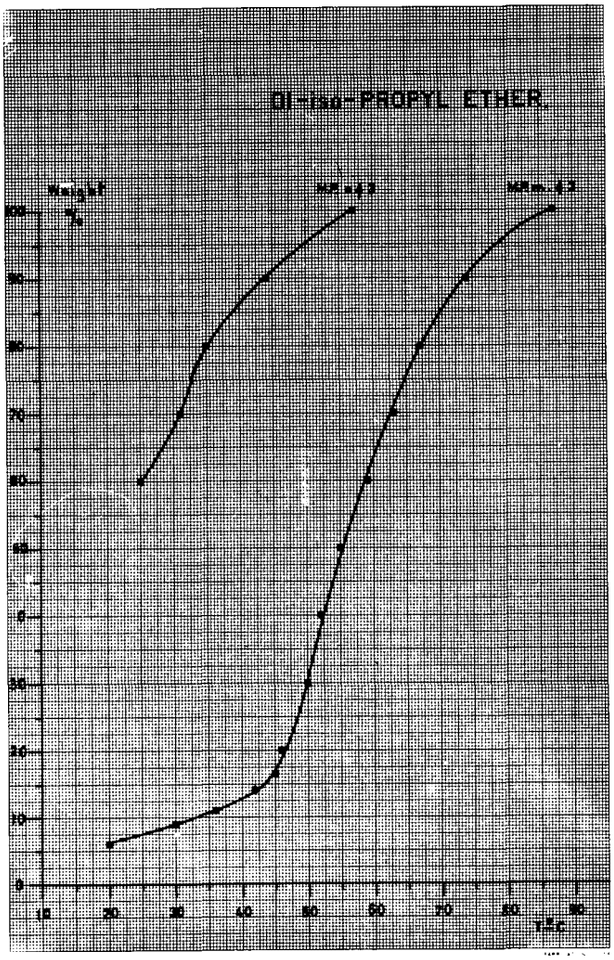

Weight % - 82
n-BUTANOL
T °C
FABRICATION SUISSE

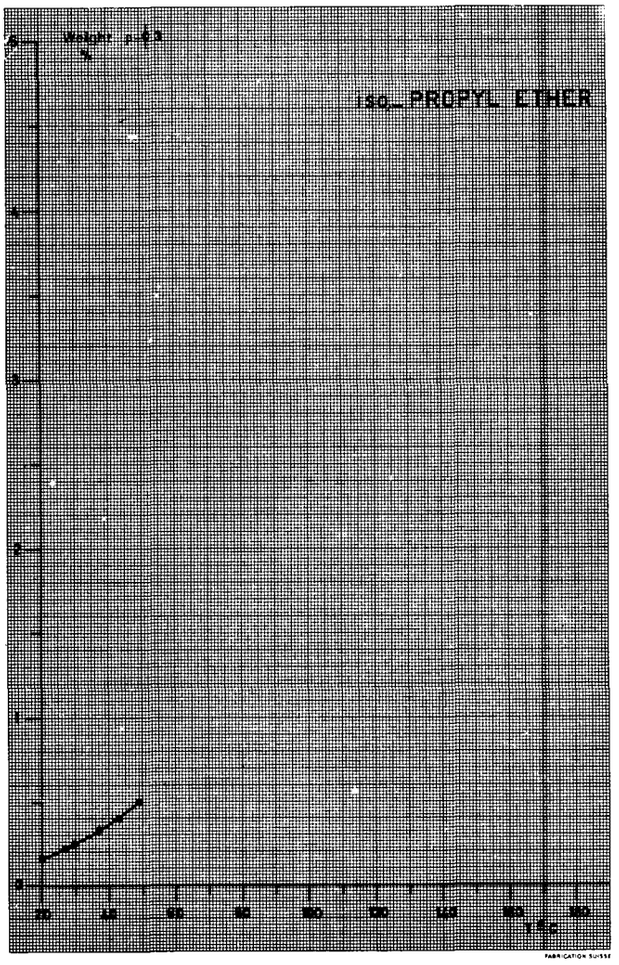

BENZYL ALCOHOL
Weight %
MP = 1
MP = 43
T °C

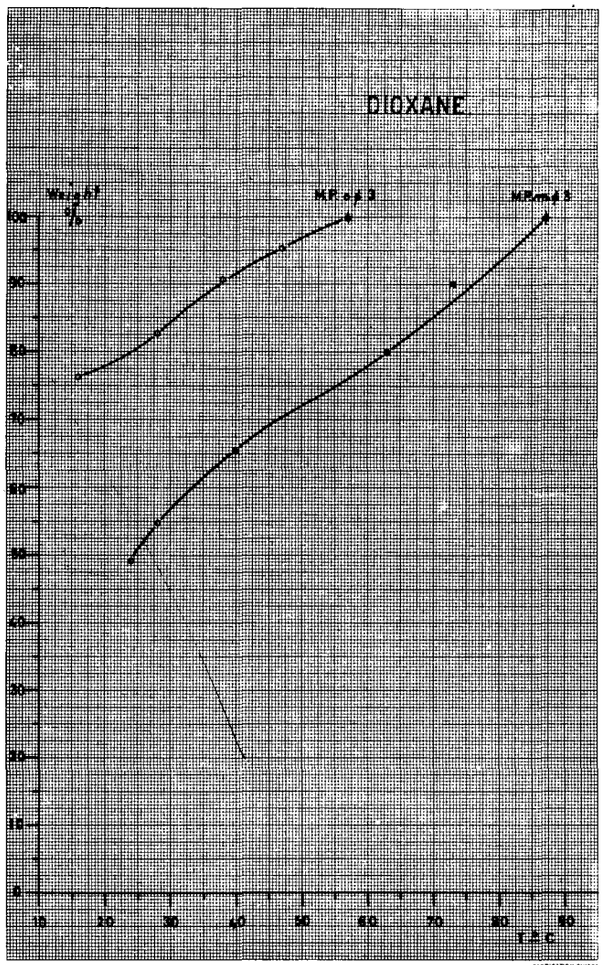

Weight % - 83
BENZYLALKOHOL
T °C
FABRICATION SUISSE

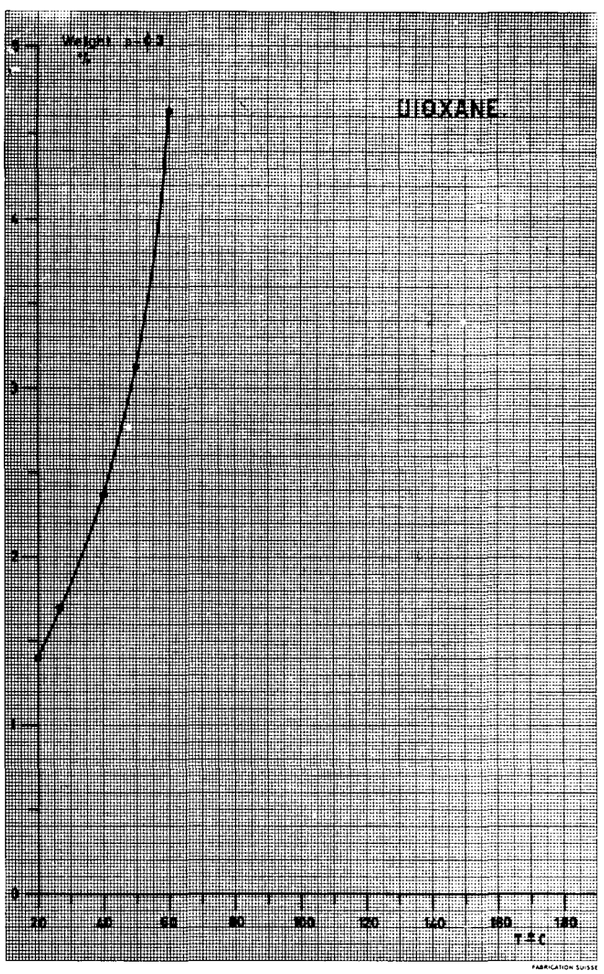

ACETIC ACID.
Weight %
T°C
FABRICATION SUISSE

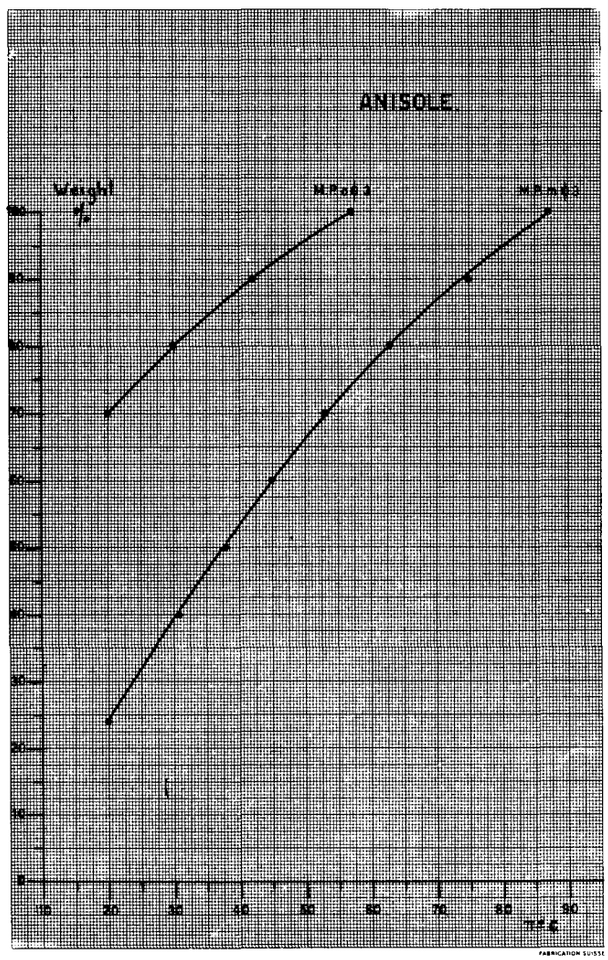

ACETIC ACID
T°C
FABRICATION SUISSE

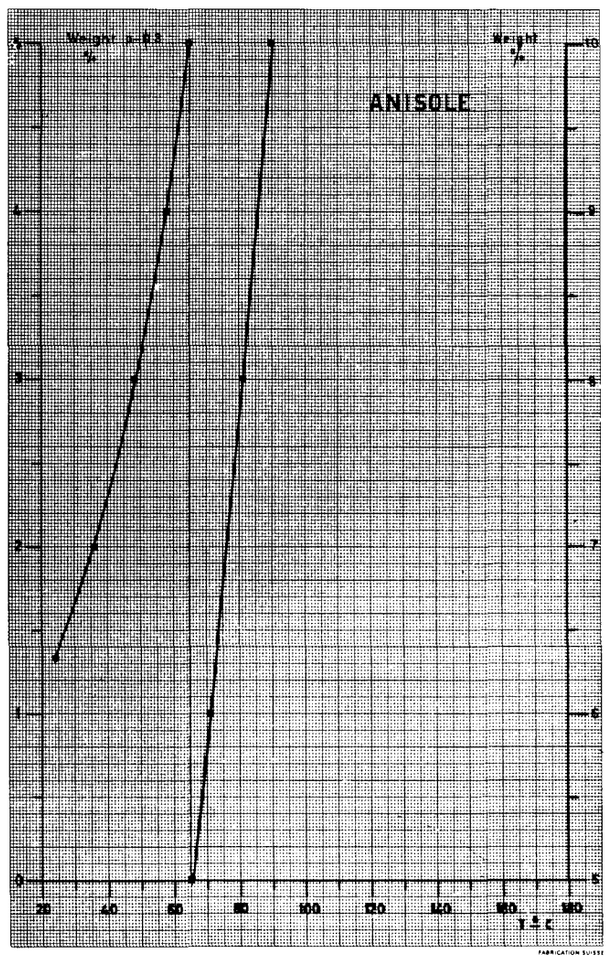

DI-iso-PROPYL ETHER.
Weight %
T°C

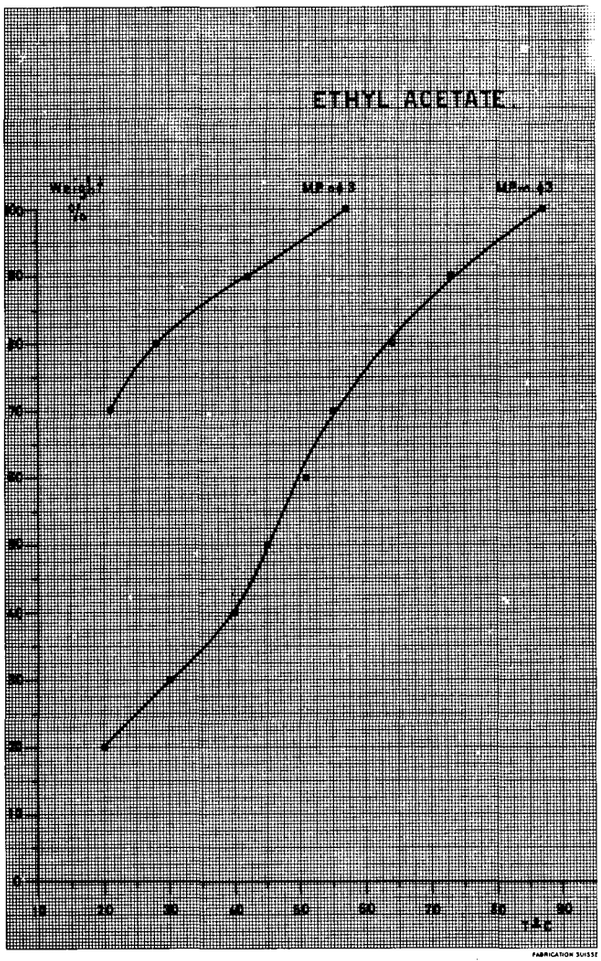

iso-PROPYL ETHER
T°C
FABRICATION SUISSE

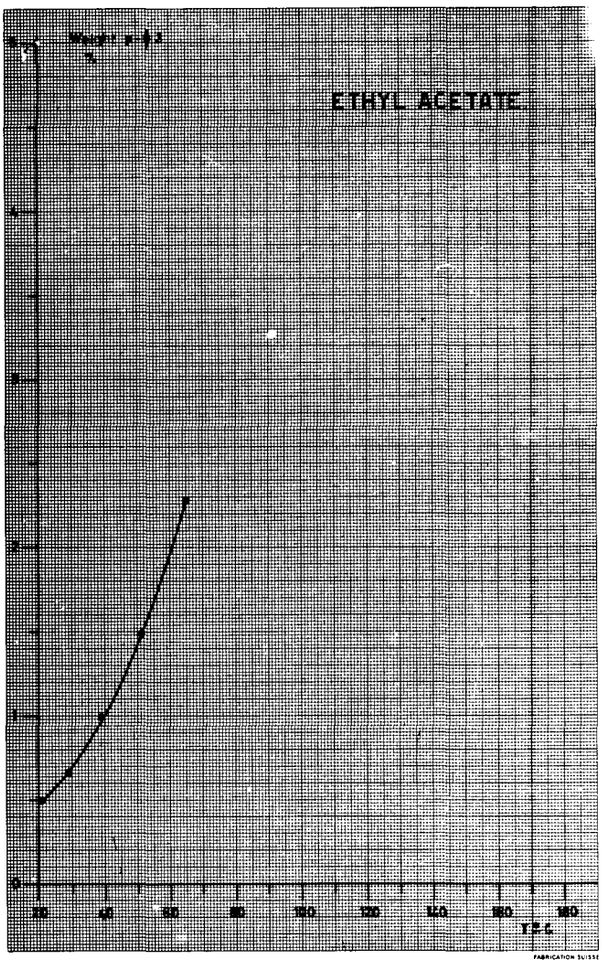

DIOXANE
Weight %
M.P. +4.3
M.P.-4.3
T ± C
FABRICATION SUISSE

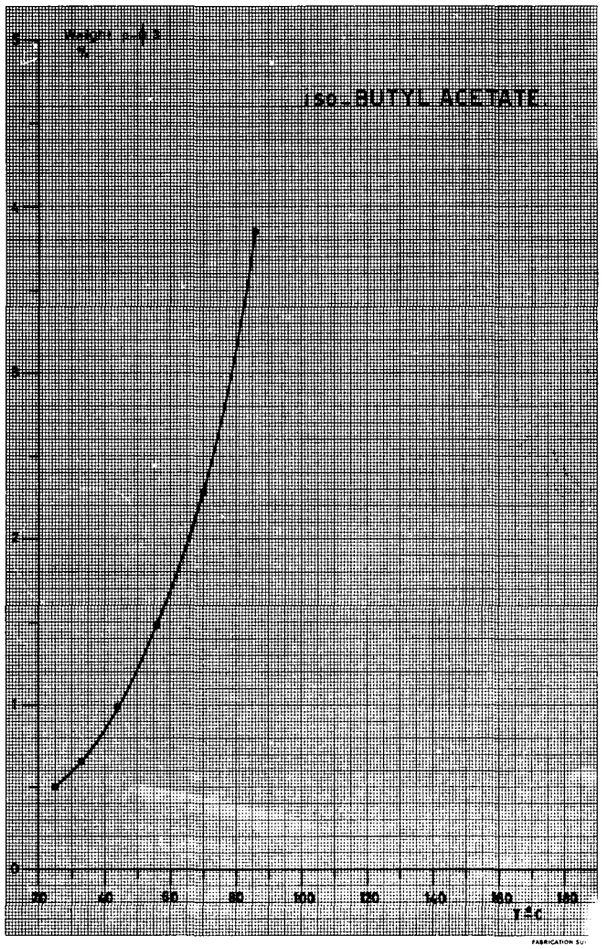

DIOXANE
Weight +-0.3 %
T ± C
FABRICATION SUISSE

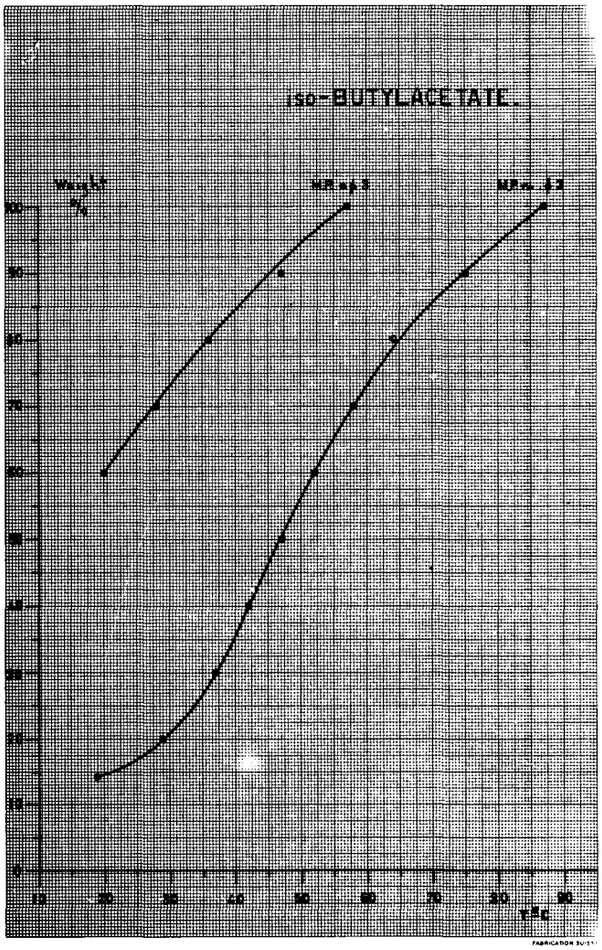

ANISOLE
Weight %
M.P.+0.4
M.P.-0.4
T ± C
FABRICATION SUISSE

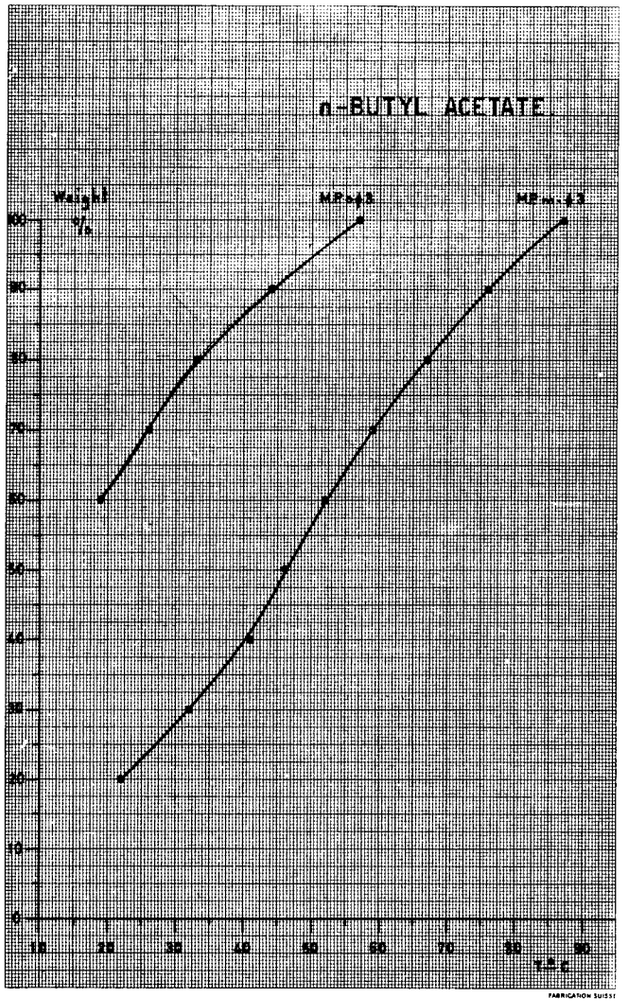

ANISOLE
Weight +-0.3 %
Weight %
T ± C
FABRICATION SUISSE

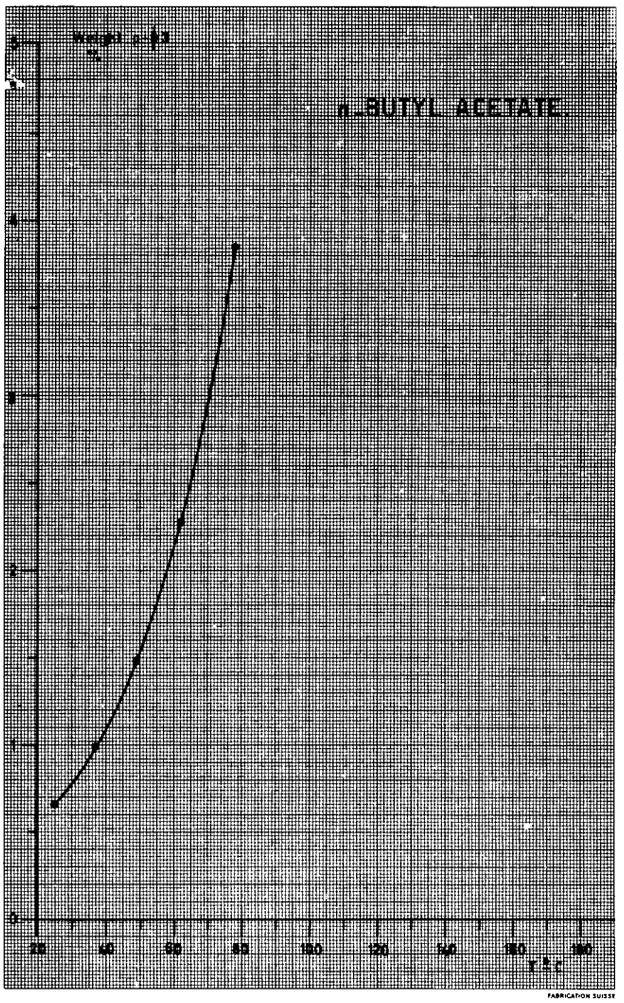

ETHYL ACETATE.
MR=4.3
MR=4.3
T°C

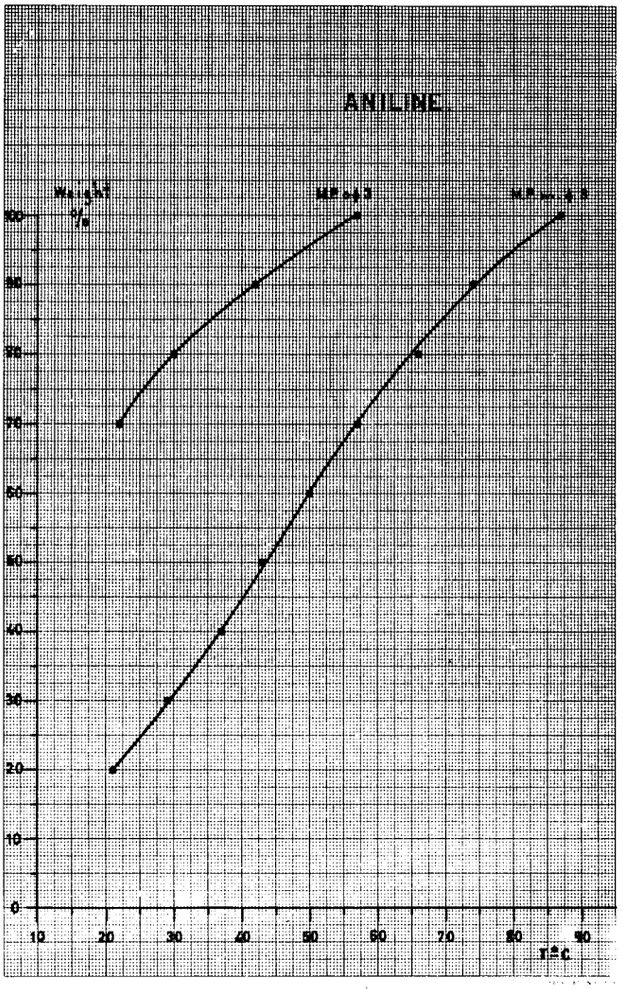

ETHYL ACETATE.
T°C

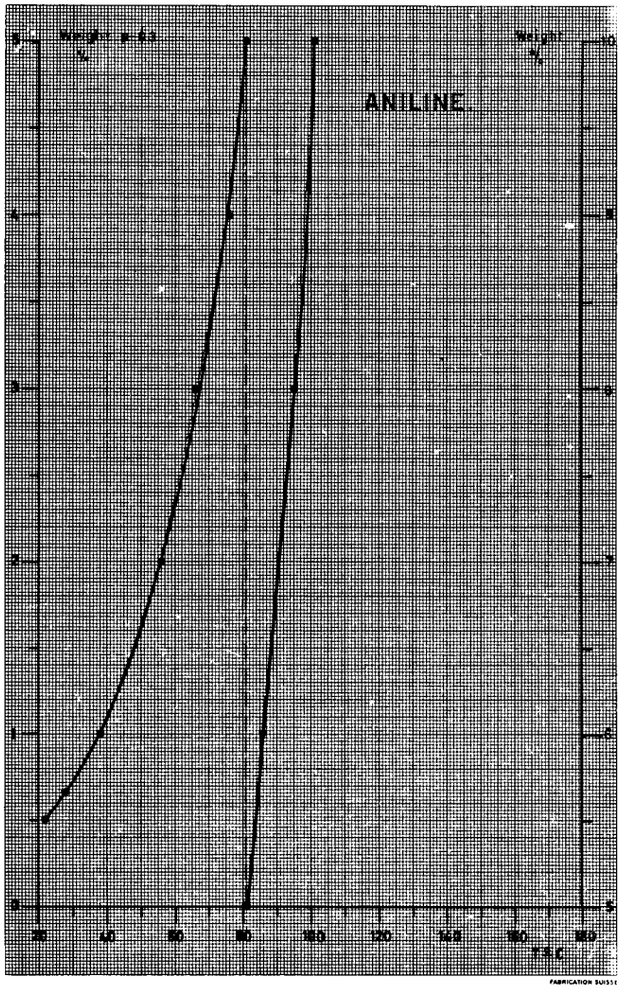

ISO-BUTYL ACETATE.
T°C

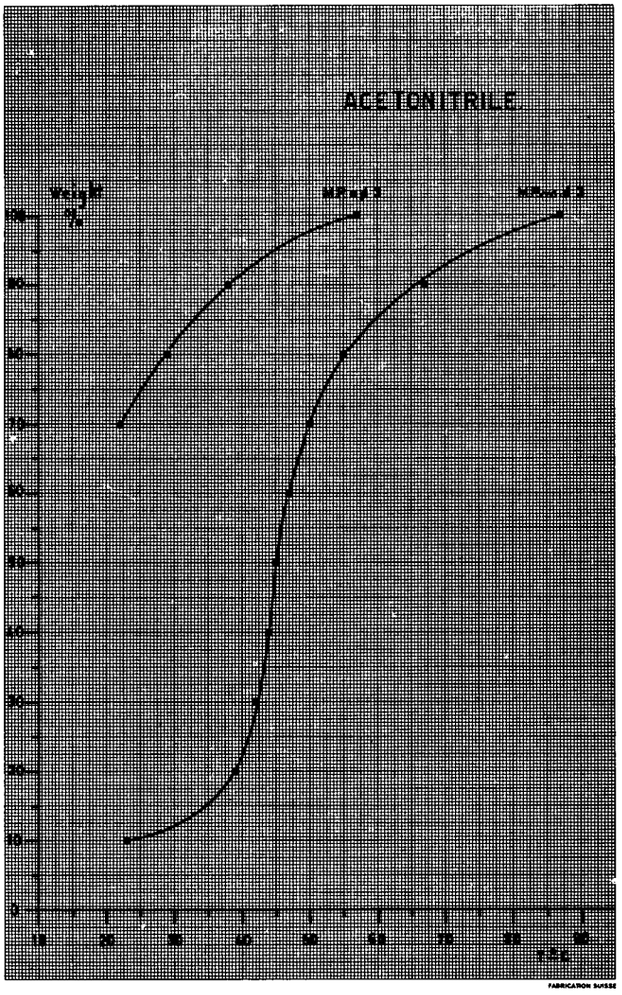

ISO-BUTYLACETATE.
MR=4.3
MR=4.3
T°C

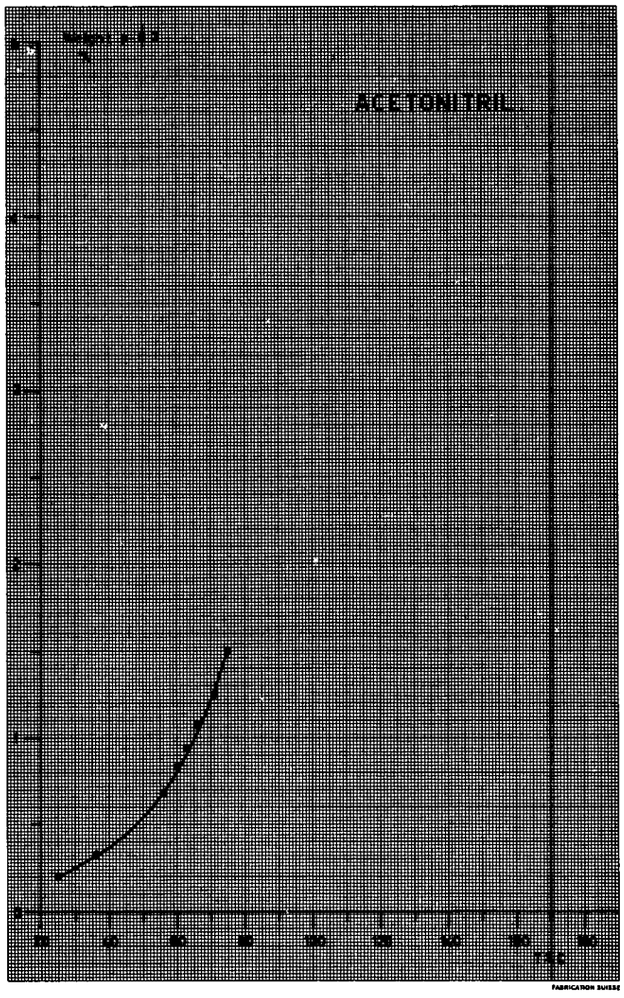

n-BUTYL ACETATE
Weight %
M.P. +0.5
M.P. -0.5
T°C
FABRICATION SUISSE

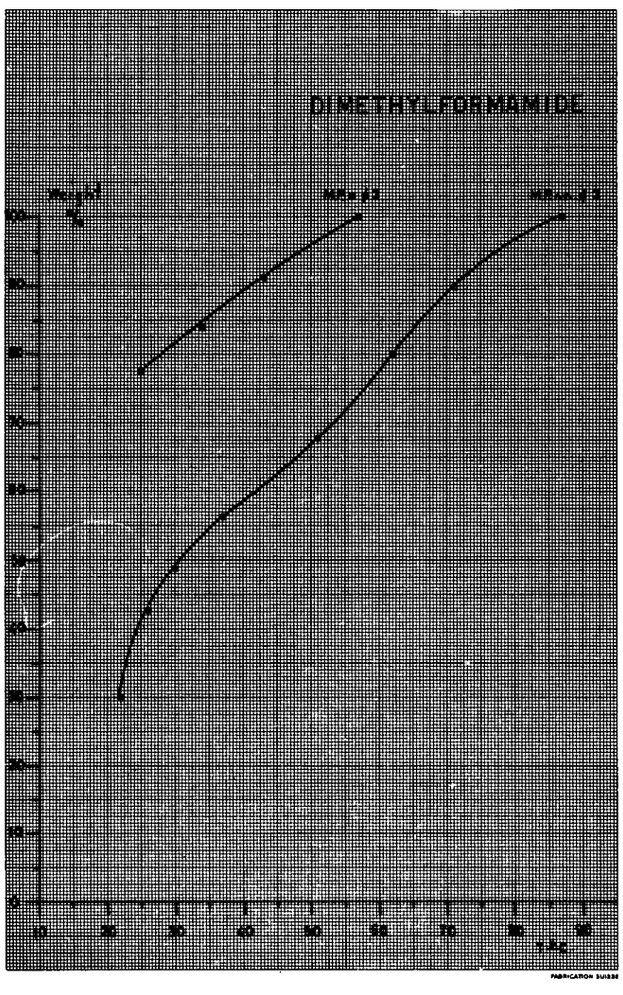

n-BUTYL ACETATE
T°C
FABRICATION SUISSE

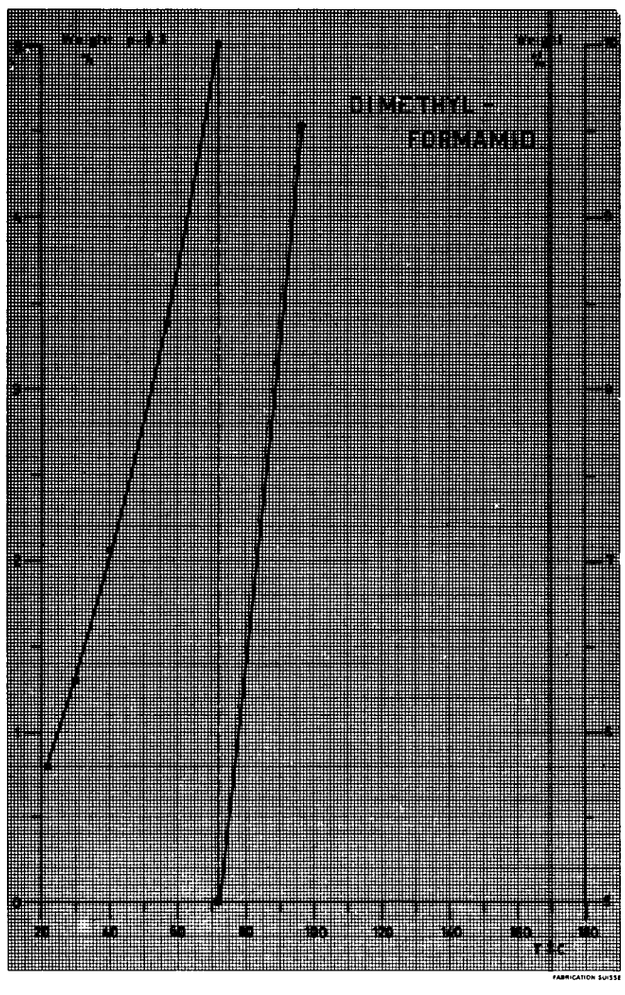

ANILINE
Weight %
M.P. +0.5
M.P. -0.5
T°C

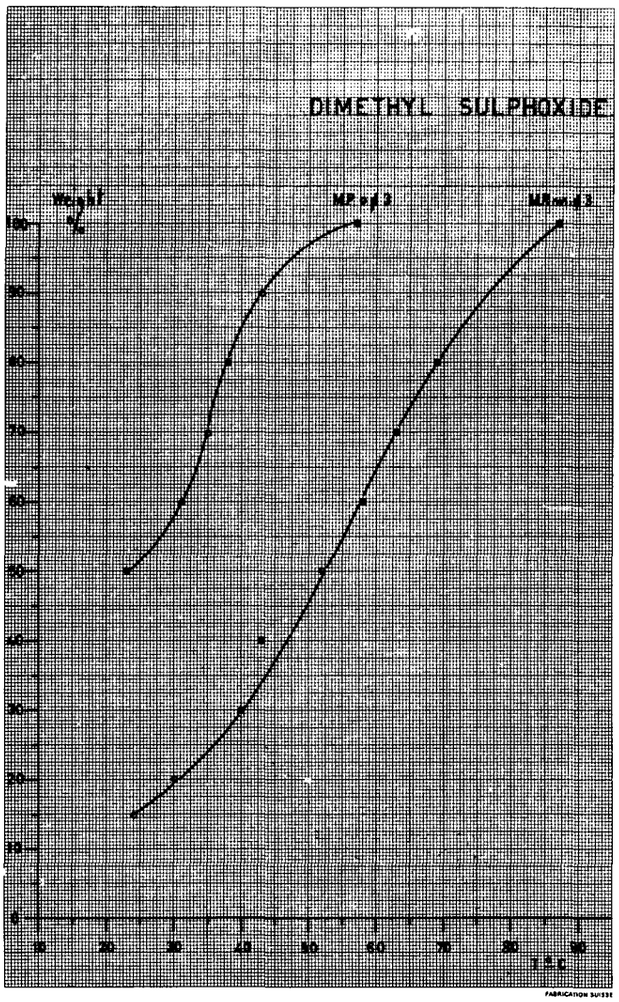

ANILINE
Weight %
T°C
FABRICATION SUISSE

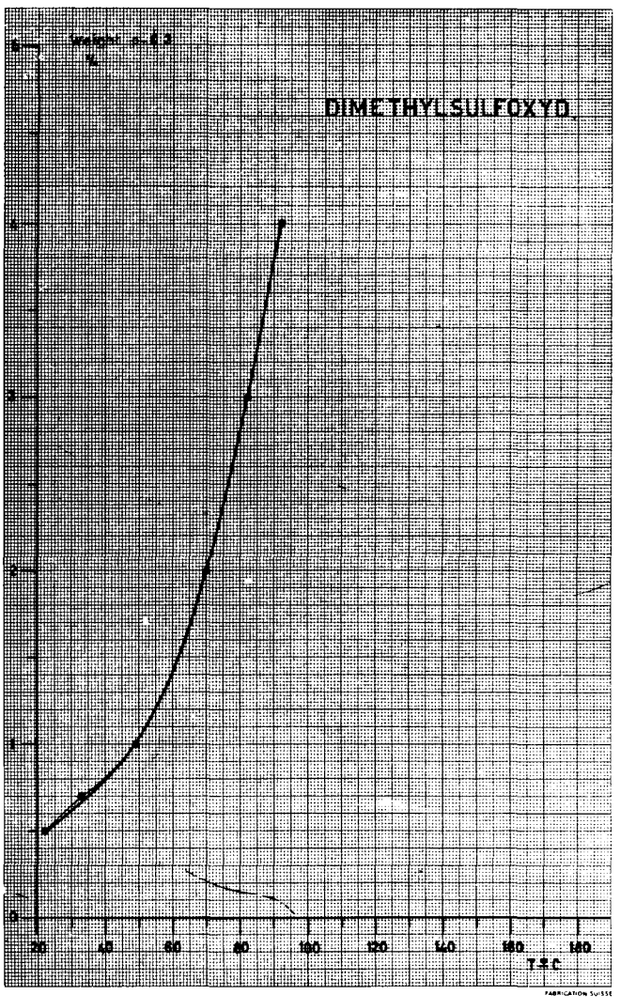

ACETONITRILE
T °C

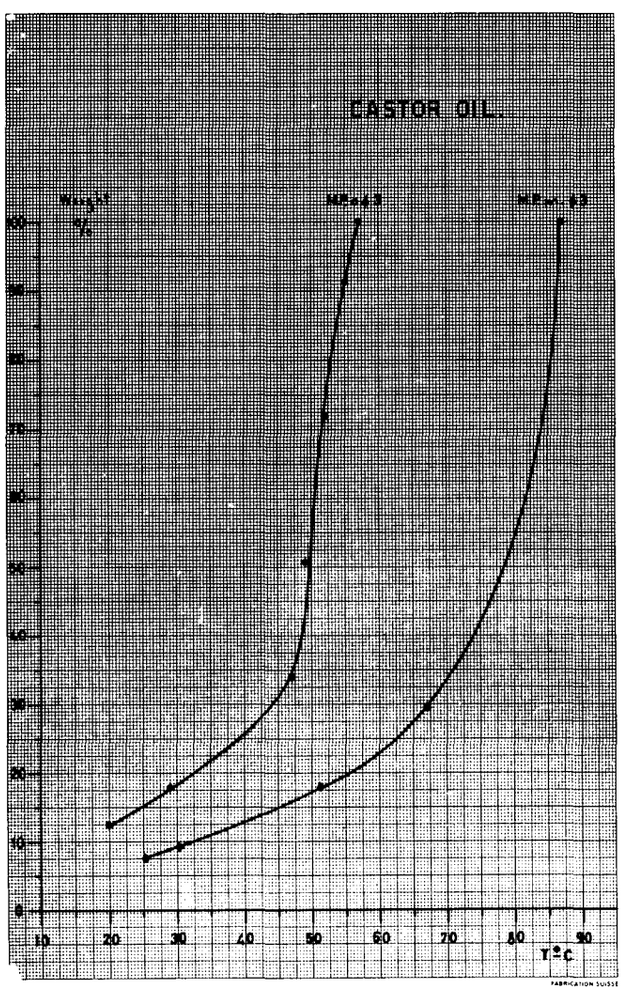

ACETONITRIL
T °C

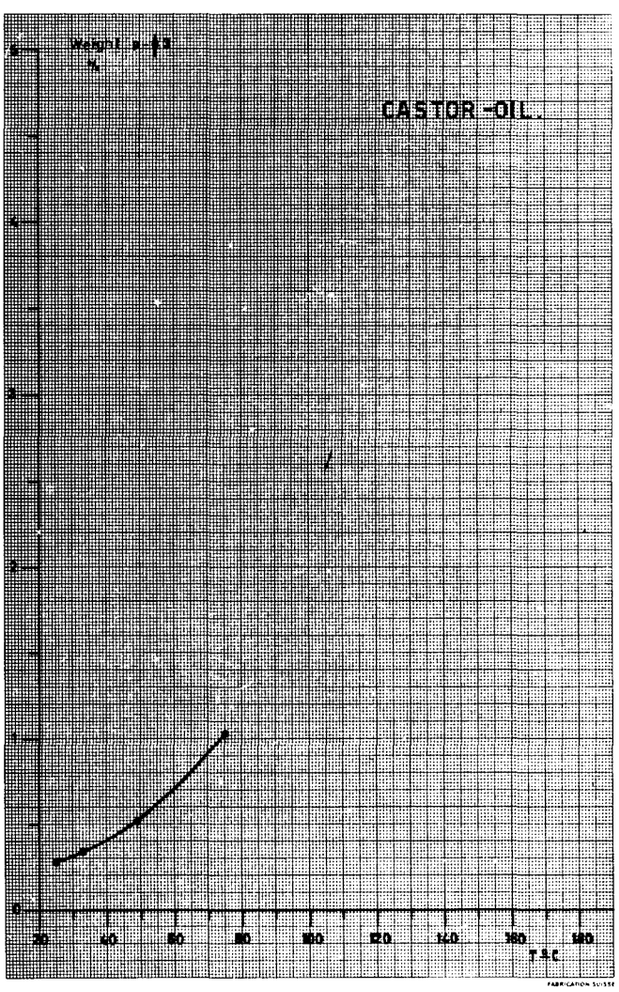

DIMETHYLFORMAMIDE
T °C

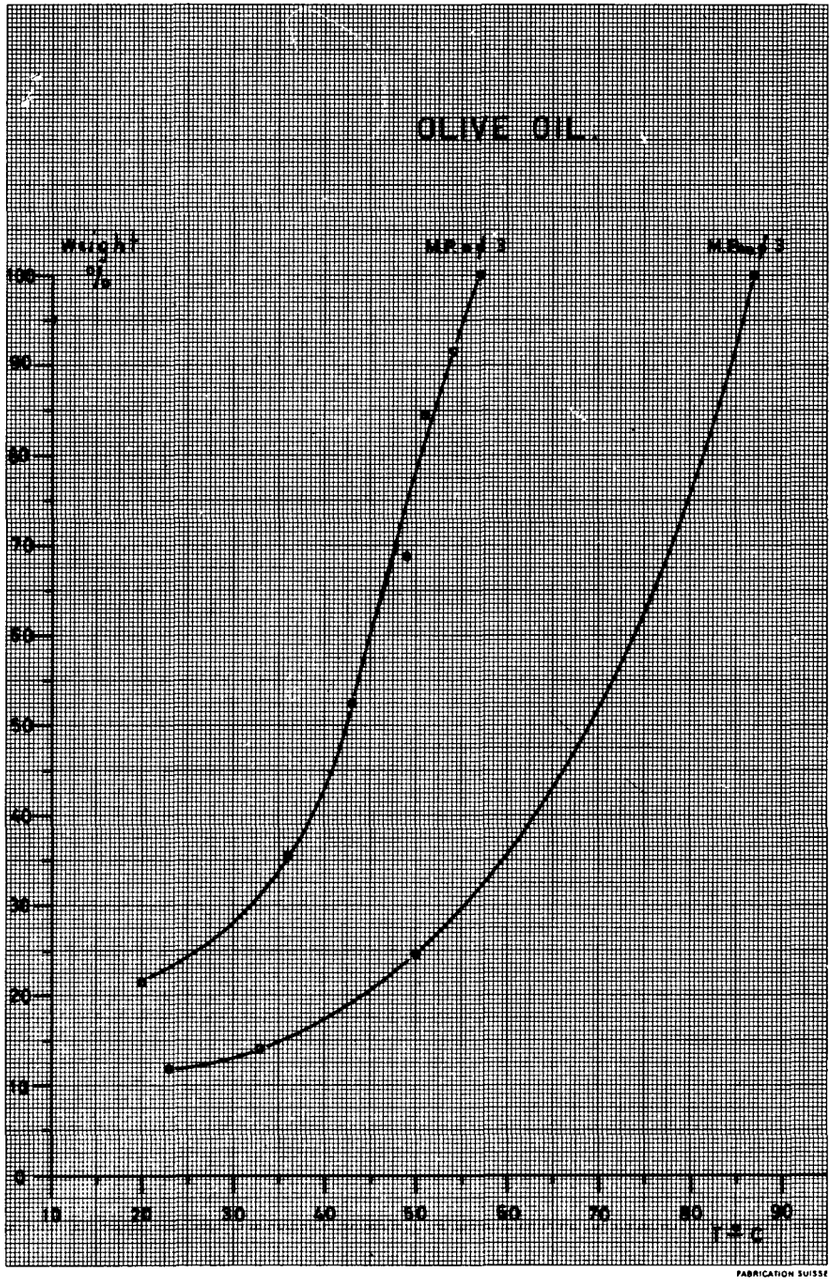

DIMETHYL-
FORMAMID
T °C

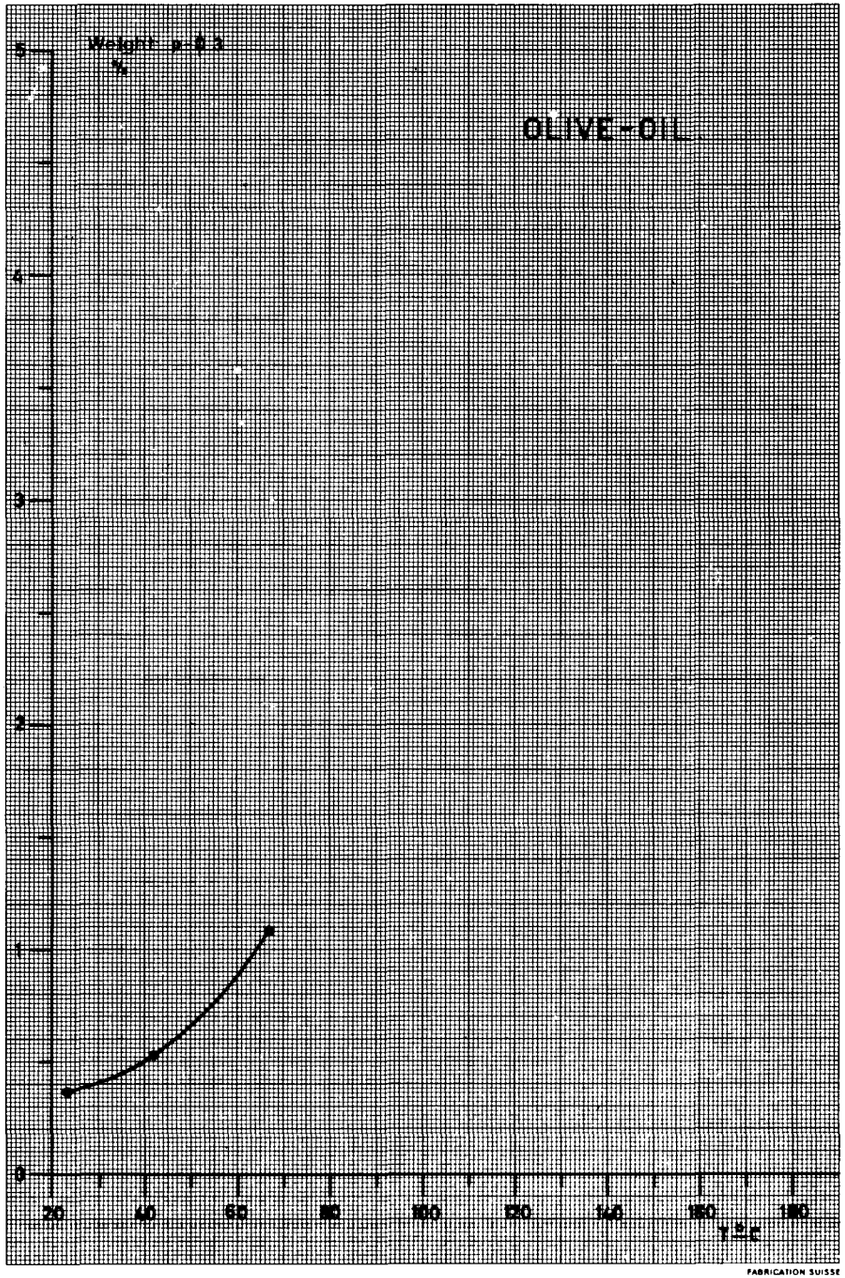

DIMETHYL SULPHOXIDE
Weight %
MP n°1
MP n°2
T°C
FABRICATION SUISSE


DIMETHYLSULFOXYD
T°C
FABRICATION SUISSE


CASTOR OIL
Weight %
MP n°1
MP n°2
T°C
FABRICATION SUISSE


CASTOR-OIL
T°C
FABRICATION SUISSE


OLIVE OIL.
Weight %
MP = f t
MP = f t
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
t ° C
FABRICATION SUISSE


Weight p = f t
%
OLIVE-OIL.
5
4
3
2
1
0
20
40
60
80
100
120
140
160
180
t °C
FABRICATION SUISSE

T A B L E 4

SOLVENTS ARRANGED ACCORDING TO THEIR SOLVENT ABILITY.

Remarks :

An artificial % - solvent ability given in the table (first column) was introduced for the following reason :
In several cases the difference of the solubility between near related solvents considered only at one temperature are in the range of experimental error. Such values cannot be taken as the basis of discussion for solubility rules. To overcome this difficulty and to correct the wrong impression one can have, looking only to one point of the curve and without consideration for the form and the slope of the curves, we tried to classify the solvents by an artificial solubility- number. We took from the curves the solubility-values of o-, m-, and p- Ø 3 for every solvent at 25 °C and 50 °C. The sum of this six values for one solvent is our solubility- number with the dimension Σ of g o-, m-, p- Ø 3 at 25 °C and 50 °C in 600 g solution-. We assumed it to be 100 for benzene as reference solvent and received in this manner our %- solvent ability. Practically with this operation we made a first step of integration of the 3 curves and in fact we found it to be sufficient to arrange the solvents in a logical way with respect to their solvent power. With the addition of the values of the 3 terphenyls we can expect a better elimination of the experimental error because it is unlikely that the experimental error is in the same direction for all 3 terphenyls. The addition of the values at two different temperatures takes into account in first approximation also the form and the slope of the curves.

Table 4

% Solvent ability	Solvent	WEIGHT-%					
		o-ϕ_3		m-ϕ_3		p-ϕ_3	
		25° C	50° C	25° C	50° C	25° C	50° C
107.5	TETRAHYDROFURAN	82.0	96.0	55.0	73.0	2.75	5.50
104.3	DIOXANE	80.5	96.0	51.0	72.0	1.60	3.12
101.0	PYRIDINE	79.0	96.0	44.0	71.0	1.80	4.10
100	BENZENE	80.5	96.5	43.5	67.5	1.35	3.25
98.8	CYCLOHEXANONE	76.0	94.5	44.5	69.0	1.65	3.70
97.5	DIMETHYLFORMAMIDE	77.5	96.0	41.0	67.0	1.00	2.80
96.6	METHYLETHYLKETONE	76.0	97.0	38.0	69.0	0.88	2.00
93.7	ACETONE	77.0	96.0	30.0	69.0	0.60	1.50
93.4	ANISOLE	75.0	95.5	32.0	66.5	1.40	3.15
92.8	TOLUENE	75.0	96.5	31.5	65.0	1.16	2.68
90.6	ACETOPHENONE	69.0	93.5	36.5	62.5	1.10	2.50
88.6	ETHYLACETATE	76.5	95.0	25.0	61.0	0.58	1.40
88.3	XYLENE	71.0	95.0	27.5	61.5	0.85	2.27
87.9	ACETYLACETONE	70.0	95.0	27.0	63.0	0.70	1.65
87.7	ANILINE	74.5	95.5	24.5	60.0	0.58	1.60
87.5	i-PHORONE	63.0	92.5	36.0	60.0	1.15	2.35
87.1	THERMIP P 2	70.0	92.0	31.5	57.5	1.35	2.65
86.6	TRICHLOROETHYLENE	68.0	92.5	31.5	58.5	0.94	2.22
86.5	ACETONITRIL	74.5	97.5	10.5	70.0	0.20	0.54
86.1	METHYL-i-BUTYL-KETONE	70.0	95.0	24.5	60.5	0.50	1.52
85.8	o-DICHLOROBENZENE	67.5	94.0	30.0	56.0	1.10	2.50
84.0	SOLVESSO 100	70.0	94.0	22.5	56.0	0.84	2.00
83.5	n-BUTYLACETATE	68.5	95.0	22.5	56.0	0.66	1.50
82.1	H A N	68.0	92.5	24.0	53.0	0.75	1.90
81.8	SOLVESSO 150	69.0	93.5	21.5	52.5	0.80	1.95
80.8	THERMIP LOURDE	62.5	92.0	28.0	50.0	1.35	2.60
80.5	i-BUTYLACETATE	67.0	94.0	17.0	56.0	0.50	1.20
80.0	CARBONTETRACHLORIDE	65.0	93.0	20.0	54.5	0.42	1.14
78.0	CYCLOHEXANE	68.0	96.0	12.0	51.5	0.12	0.70
75.8	AKP-M	55.0	88.5	28.5	45.0	1.90	2.95
74.2	TETRACHLOROETHYLENE	58.5	91.0	19.0	47.0	0.36	1.20
73.4	HB - 40	51.0	87.5	29.5	44.0	0.90	1.83
72.4	DECALIN	61.0	94.0	12.5	43.5	0.26	0.70
72.0	DIMETHYLSULPHOXIDE	51.5	97.0	15.5	45.0	0.54	1.04

Table 4

% Solvent ability	Solvent	WEIGHT-%					
		o-ϕ_3		m-ϕ_3		p-ϕ_3	
		25° C	50° C	25° C	50° C	25° C	50° C
66.1	DI-i-PROPYL ETHER	60.0	95.0	7.5	30.0	0.20	0.52
60.4	BENZYL ALCOHOL	40.0	94.0	10.0	31.5	0.35	0.90
55.7	n-HEXANE	36.0	97.5	5.5	23.5	0.11	0.56
52.1	OCTANE	27.5	97.0	5.0	22.5	0.12	0.45
49.4	ACETIC ACID	32.5	93.5	5.5	17.5	0.14	0.33
46.3	OLIVE OIL	24.5	77.5	12.0	20.5	0.38	0.65
45.8	ISOPAR G.	21.0	93.0	4.5	14.0	0.11	0.30
42.8	ISOPAR K	16.5	92.0	3.5	13.0	0.10	0.25
41.9	ISOPAR M	15.0	92.0	4.0	11.5	0.08	0.28
41.6	n- BUTANOL	12.5	93.0	4.0	12.0	0.12	0.30
40.1	ETHANOL	11.5	94.5	3.0	8.0	0.08	0.20
39.7	t- BUTANOL	9.5	95.0	2.0	9.5	0.08	0.18
38.0	i- PROPANOL	8.0	95.0	2.0	6.0	0.06	0.16
33.9	VASELIN OIL	13.0	70.0	4.5	11.0	0.20	0.36
31.9	CASTOR OIL	15.0	52.5	7.5	17.5	0.28	0.52

1.4. Discussion of experimental results.

In table 4, the compounds are classified according to their solvent ability for terphenyls. These data indicate the following relations between molecular structure and solvent ability.

a- Benzene is the fundamental aromatic compound. We propose thus to adopt his solvent ability to be 100 %

b- Only heterocyclic compounds solve better than benzene.
example : Tetrahydrofurane - Dioxane - Pyridine.

c- The substitution of hydrogen in the benzene nucleus decrease the solvent ability from little changing to large one according to the following substitutions.

H substituted by an ether group : Benzene → Anisole
H substituted by (an)alkyl groups: Benzene → Xylene
H substituted by an chlorine " : Benzene → Dichlobenzene
H substituted by an amino group : Benzene → Aniline
H substituted by an hydroxyl " : Toluene → Benzyl alcohol

d- The higher the degree of substitution the more decreases the solvent ability
example : Toluene, Xylene, Solvesso.

e- The hydrogenation of the nucleus results in a decrease of the solvent ability similar to that which occurs by the substitution of H by amino and hydroxyl groups.
example : Benzene - Cyclohexane
Thermip - Decalin

f- For aliphatic and alycyclic hydrocarbons the solvent ability generally decreases simultaneously with the molecular weight
example : Hexane - Vaseline Oil
Cyclohexan - Decalin
For compounds with a similar number of C. atoms, the alicyclic hydrocarbons solves better than

the aliphatic one

example : Hexane - Cyclohexane

g- In comparison to the hydrocarbons of the aliphatic and alicyclic series, the introduction of oxygen increase the solvent ability in the following line :

i) ether bound
ii) ester bound
iii) Keto bound

The introduction of oxygen as OH group decreases, at least for the low-molecular weight compounds, the solvent ability of the hydrocarbons.

1.5. Choice.

We have selected to test and use for decontamination purposes following solvents

ALIPHATICS	:	ISOPAR G
AROMATICS	:	BENZENE
		THERMIP
		SOLVESSO 150
KETONES	:	ACYCLOHEXANONE
		METHYETHYKETONE
		ACETONE
HETEROCYCLICS	:	TETRAHYDROFURAN
ETHERS	:	DIOXANE
ESTERS	:	ETHYLACETATE

This selection is made on the base of the experimental results in comparison with the conventional properties and the price (table 1 to 3).

If Benzene, Tetrahydrofuran and Dioxane remain in this selection it is only due to their excellent ability to solve terphenyls.

2. Solubility of OM_2 mixtures.

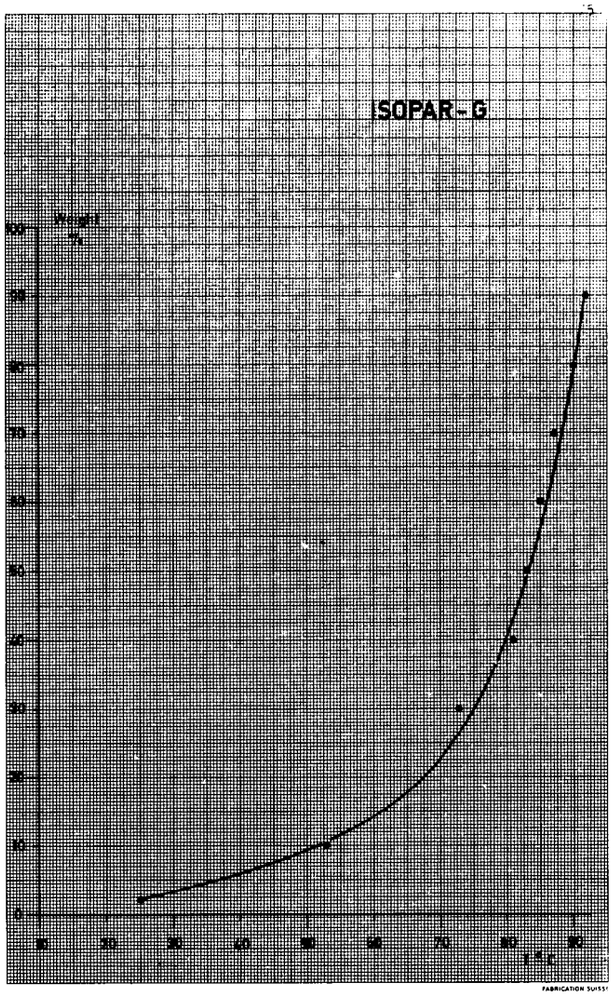
2.1. To complete our investigations for practical decontamination purposes we have determined the solubility of OM_2 mixtures in the solvents selected in part. 1.
The OM_2 mixture we have used were prepared by us from the three pure terphenyls following the average composition of OM_2 :

p. Ø 3 : 5 %
m. Ø 3 : 75 %
o. Ø 3 : 20 %

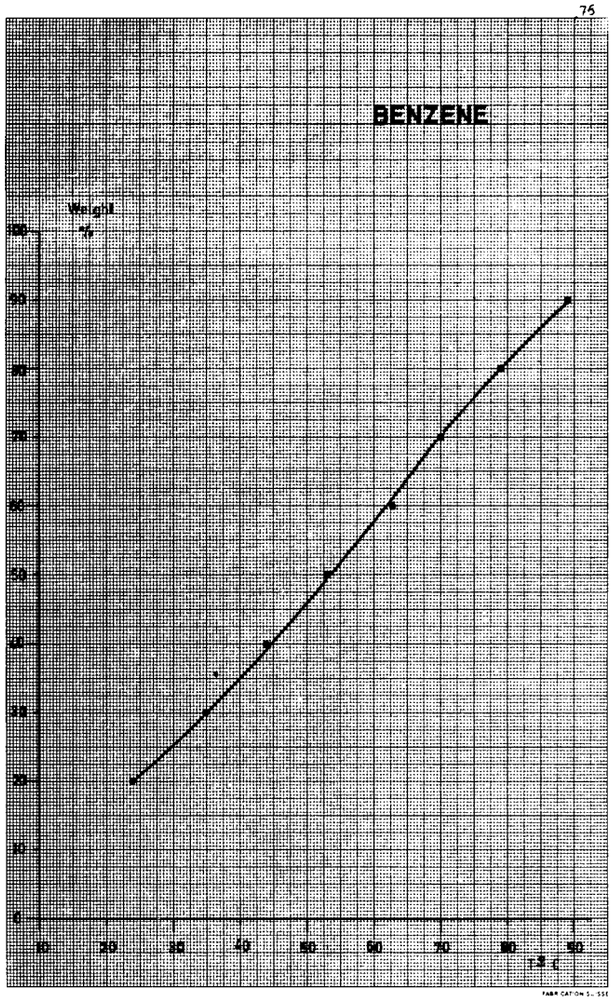
This procedure we used in order to avoid sampling errors.
The method used to measure the solubility is the same as descripted in part. 1., this means determination of the temperature where crystallization begins.
The measured temperature indicates the point where the mixture solvent-OM_2 is liquid.
After the crystallization-point is reached, we have practically a fractional crystallization of the components of OM_2 mixture.
This phenomenon 15 of no interest for decontamination work, because we have to work in the total liquid range.

2.2. Results.
Results are presented in the following curves.
The values obtained for the "synthetic OM_2 mixture" were controlled using OM_2 technical grade manufactured by PROGIL.
The relative difference in the solubility of two mixtures is in the range of $\pm$ 5 % for temperatures from 25 °C to 80 °C.

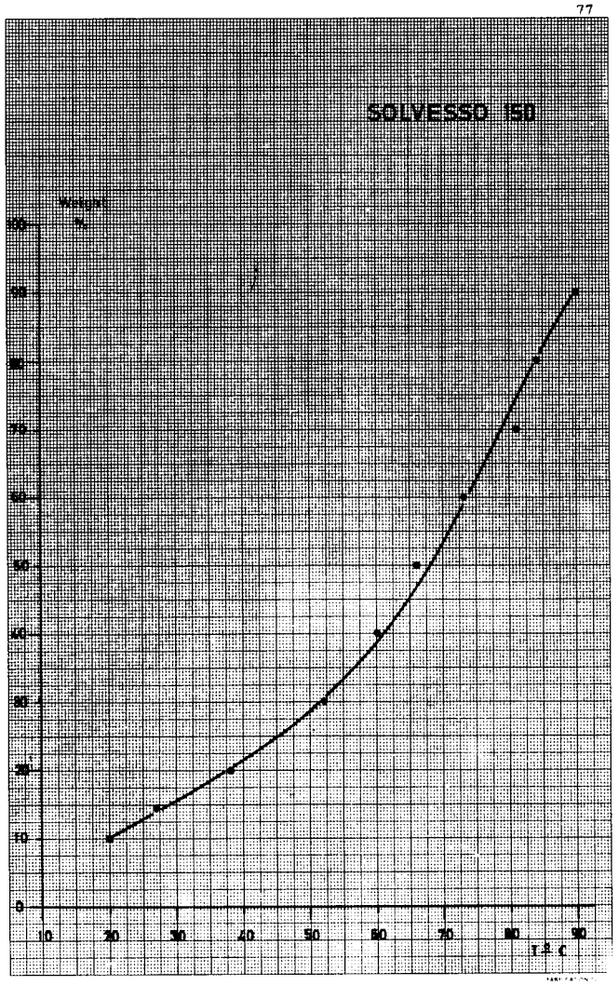
75


ISOPAR-G
Weight %
T ± C

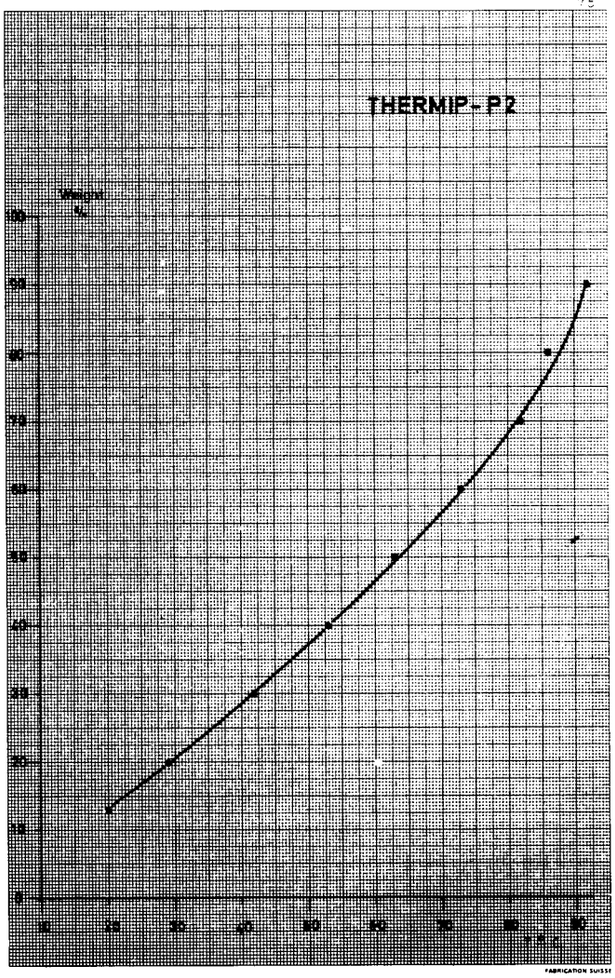
76

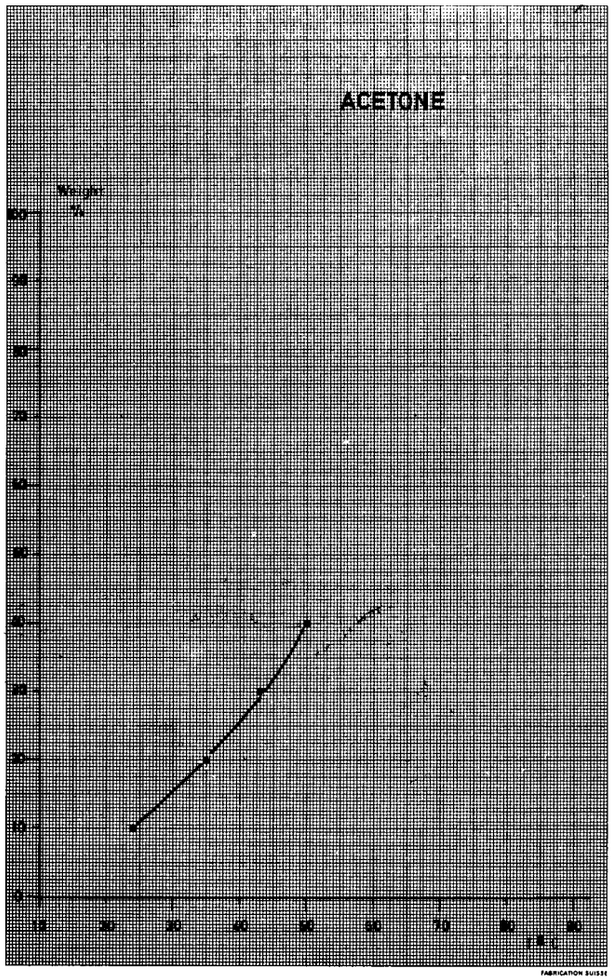

BENZENE
Weight %
T ± C

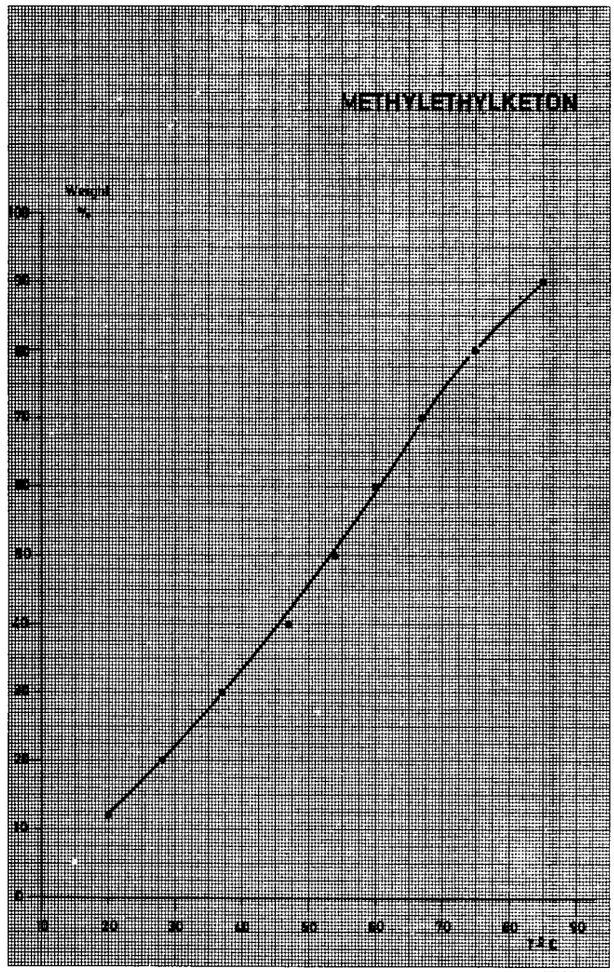
77

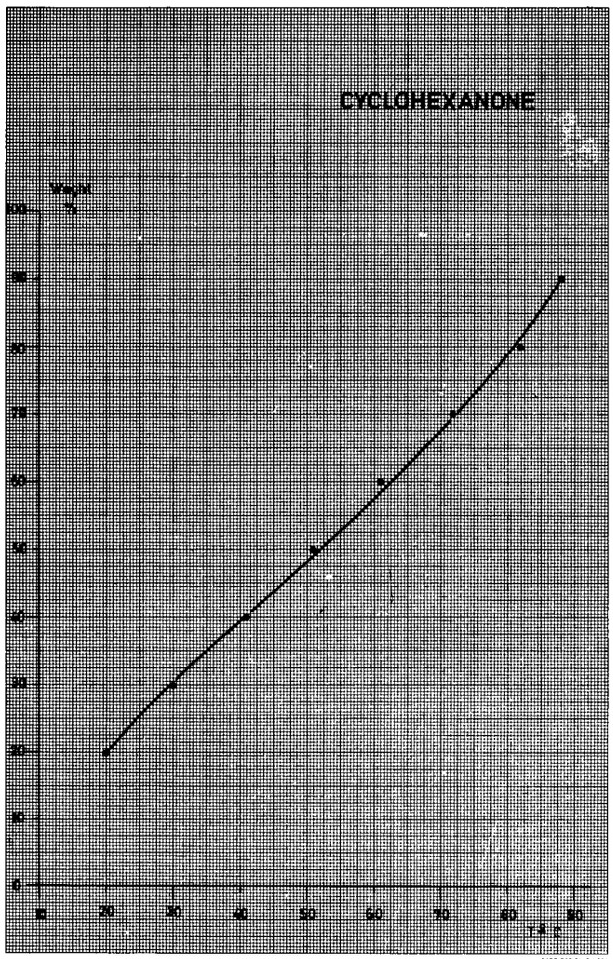

SOLVESSO 150
Weight %
T ± C

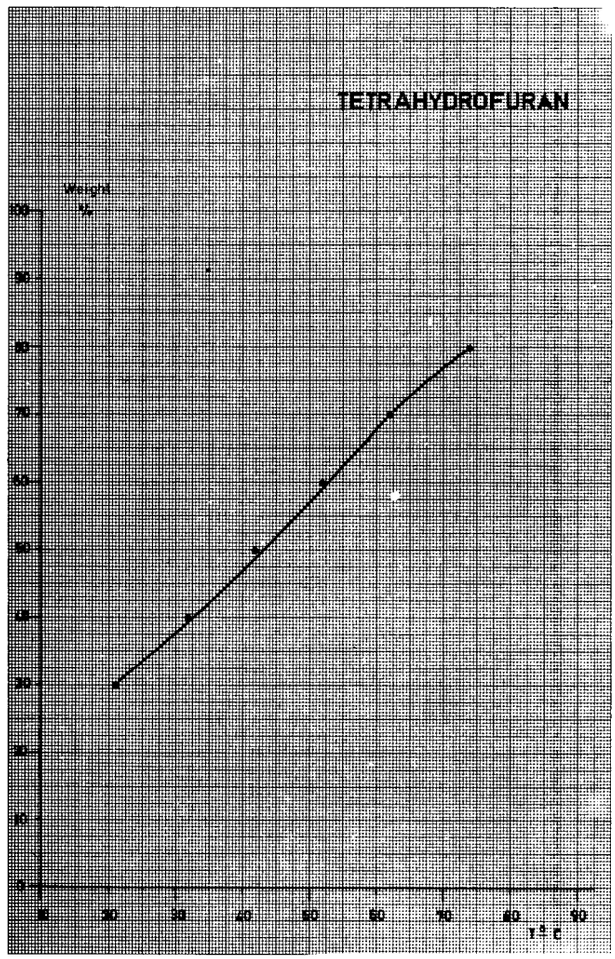
78

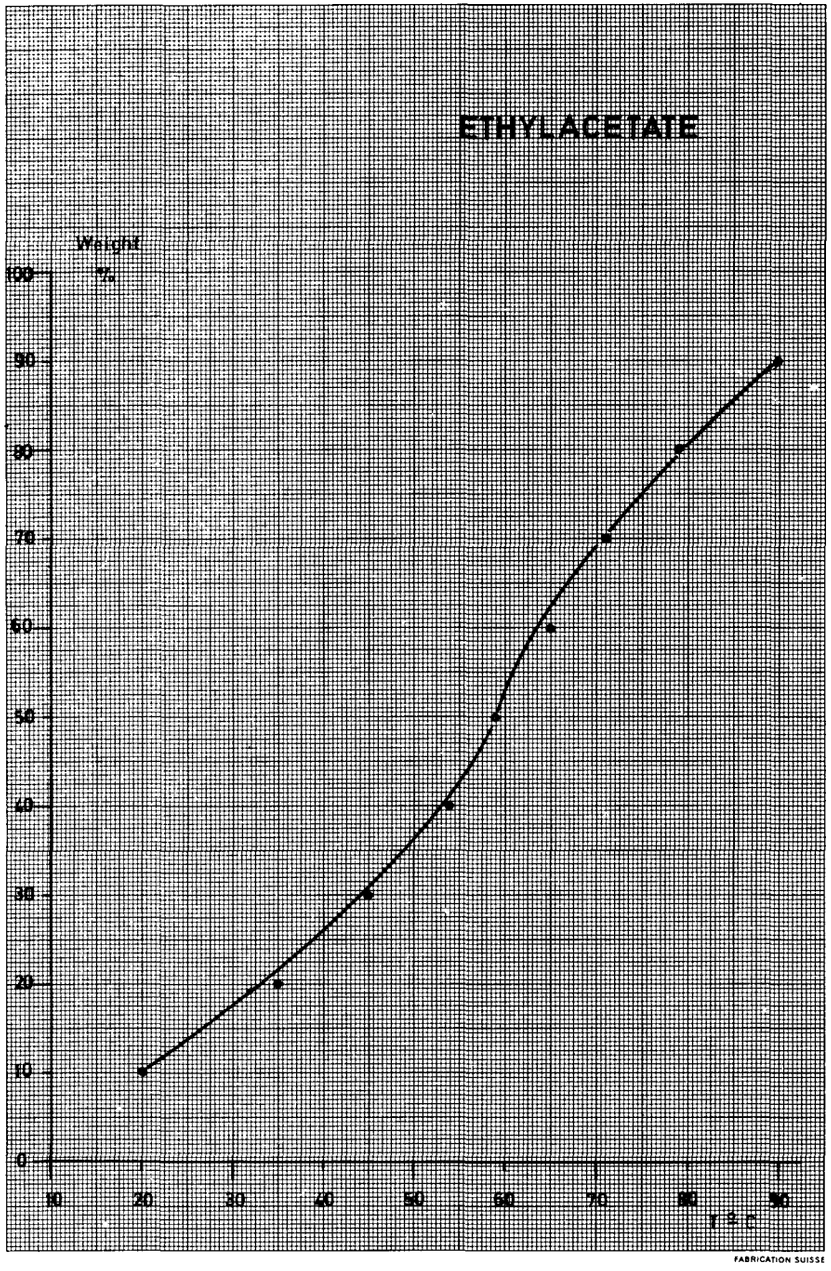

THERMIP-P2
Weight %
T ± C

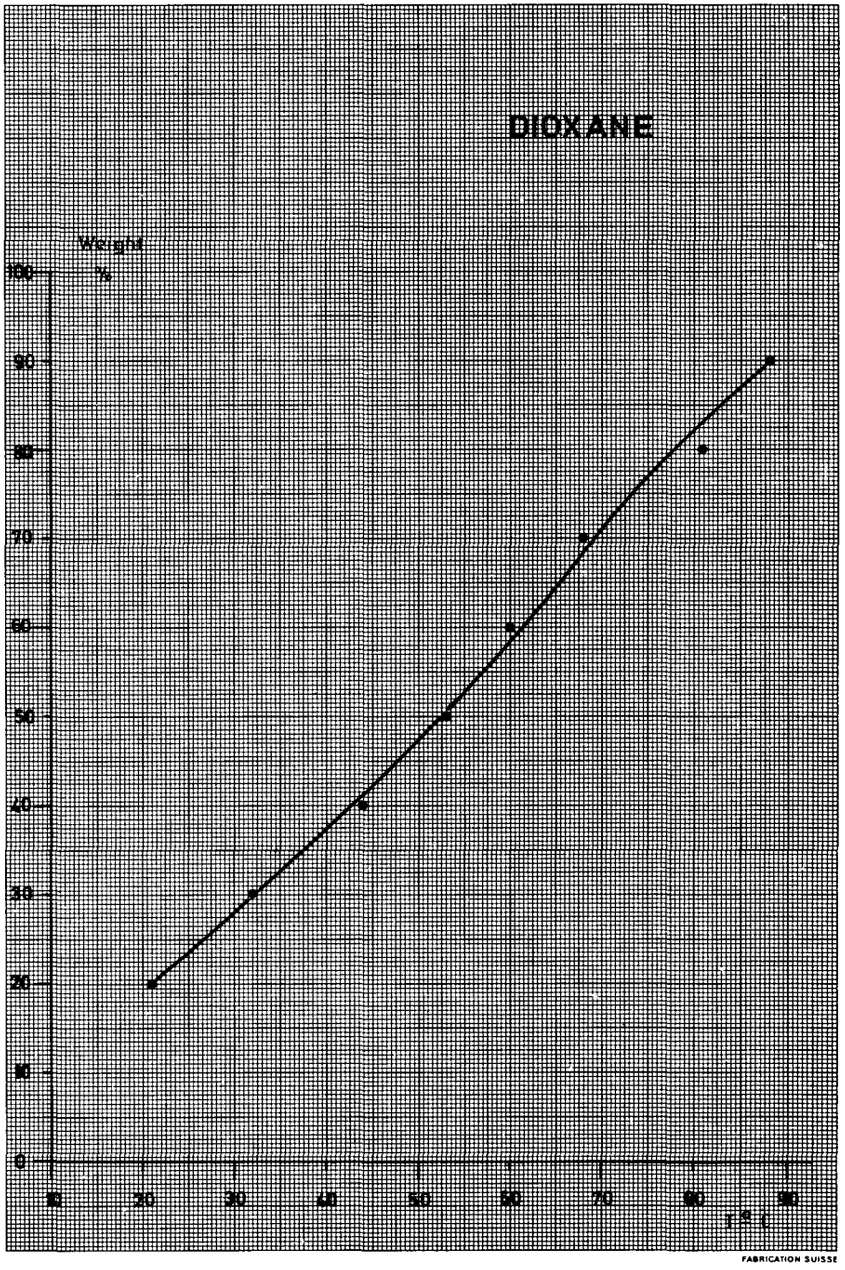

ACETONE
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T° C
FABRICATION SUISSE


METHYLETHYLKETON
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T° C
FABRICATION SUISSE


CYCLOHEXANONE
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T° C
FABRICATION SUISSE


TETRAHYDROFURAN
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T° C


ETHYLACETATE
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T °C
FABRICATION SUISSE


DIOXANE
Weight %
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
T °C
FABRICATION SUISSE

2.3. Discussion of experimental results.

According to the curves the solubility of O M_2 mixtures is about the half of the solubility of the pure m. terphenyls.
For O M_2 we find the same classification of solvent in respect of their solvent ability as given table 4.

3. Conclusion.

From experimental results and in comparison with the conventional behaviours and the price we had finally choosen the following solvents for our decontamination purposes:

ISOPAR G
SOLVESSO 150
THERMIP
ACETONE
METHYLETHYLKETONE

Isopar G is take in account only for his inerty in front of the isolated electrical components.
This choice has been confirmed by preliminary decontamination tests with success.
Decontamination techniques based on the use of this solvents are under study.
For skin decontamination, our medical service recommand the following solvent mixtures =

80 % METHYETHYLKETONE + 20 % OLIVE OIL
+ eventual local anesthetic agent.

4. Acknowledgements.

The authors acknowledge with thanks the discussions with and advice from Mr F.Geiss (Chemistry Department).

Thanks are also due to Mr. Hannaert, Chief of Organic Chemistry Service.

Bibliography.

(1) Organic coolant Data book
Tech. Publ. Nº AT - 1 July 1958
Mosanto Chemical comp, St Louis.

(2) E. E. Mc. Langlin, H.A. Zainal;
- Solubility Behaviour of Aromatic Hydrocarbons in benzene.
J. Chem. Soc. 1959 p- 863 - 867
- Solubilitues in Carbon Tetrachloride.
J. Chem. Soc. 1960 p- 2485- 488

(3) R.T. Keen
Solubility of terphenyls at room temperature
NAA - SR - Memo 2412 (1958)

(4) B. VERSINO ed al.
Analisi delle miscelle organiche sostitute dei terfenili
(in press) Ispra 1967.

(5) H. KIENITZ
Bestimmung der Löslichkeit
in HOUBEN-WEYL - Band III/ lp. 219 - 254

General literature.

- C. MARSDEN
Solvents guide
CLEVER - HUNE PRESS 1962 London
- N. Irving Sax
Dangerous properties of indutrial materials
Reinhold Publishing Coprporation 1963 New York.
- Technical Information about solvents.
ESSO CHIMICA - Milano -
- HOUBEN WEYL - Methoden der Organischen Chemie
THIEME VERLAG 1959 - Stuttgart.

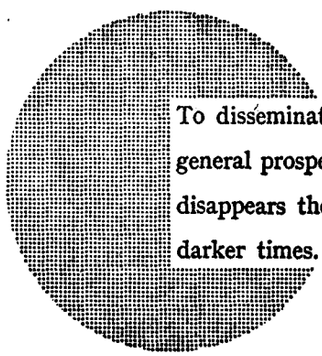
NOTICE TO THE READER

All Euratom reports are announced, as and when they are issued, in the monthly periodical **EURATOM INFORMATION**, edited by the Centre for Information and Documentation (CID). For subscription (1 year: US$ 15, £ 6.5) or free specimen copies please write to:

Handelsblatt GmbH
"Euratom Information"
Postfach 1102
D-4 Düsseldorf (Germany)

or

Office central de vente des publications
des Communautés européennes
2, Place de Metz
Luxembourg



To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

SALES OFFICES

All Euratom reports are on sale at the offices listed below, at the prices given on the back of the front cover (when ordering, specify clearly the EUR number and the title of the report, which are shown on the front cover).

OFFICE CENTRAL DE VENTE DES PUBLICATIONS DES COMMUNAUTES EUROPEENNES
2, place de Metz, Luxembourg (Compte chèque postal N° 191-90)

BELGIQUE — BELGIË
MONITEUR BELGE
40-42, rue de Louvain - Bruxelles
BELGISCH STAATSBLAD
Leuvenseweg 40-42, - Brussel

DEUTSCHLAND
BUNDESANZEIGER
Postfach - Köln 1

FRANCE
SERVICE DE VENTE EN FRANCE
DES PUBLICATIONS DES
COMMUNAUTES EUROPEENNES
26, rue Desaix - Paris 15e

ITALIA
LIBRERIA DELLO STATO
Piazza G. Verdi, 10 - Roma

LUXEMBOURG
OFFICE CENTRAL DE VENTE
DES PUBLICATIONS DES
COMMUNAUTES EUROPEENNES
9, rue Goethe - Luxembourg

NEDERLAND
STAATSDRUKKERIJ
Christoffel Plantijnstraat - Den Haag

UNITED KINGDOM
H. M. STATIONERY OFFICE
P. O. Box 569 - London S.E.1

EURATOM — C.I.D.
51-53, rue Belliard
Bruxelles (Belgique)