EXPERIMENTAL

E X P E R I M E N T A L

PART I - Synthesis And Study of New Mesogens

Synthesis of the following series of compounds was undertaken :-

1. n. Butyl - P (p'-n'-alkoxycinnamoyloxy) benzoate
2. n. Amyl - p-(p'-n'-alkoxycinnamoyloxy benzoate
3. n. Hexyl - p-(p'-n'-alkoxycinnamoyloxy) benzoate
4. n. Heptyl- p-(p'-n'-alkoxycinnamoyloxy) benzoate
5. p. (p'.n-alkoxycinnamoyloxy) benzylidine -p" - nitroaniline
6. p. (p'-n-alkoxycinnamoyloxy) benzylidine -p" - Fluoroaniline.
7. p- (p'-n-alkoxybenzoyloxy) benzoic acid
8. p- (p'-n-alkoxybenzoyloxy) propiophenone
9. p- (p'-n.alkoxybenzoyloxy) butyrophenone

The step wise synthesis of the various series of the new mesogens is given below -

1. P-n. Alkyl bromides

These bromides (C_2 to C_{10}) were synthesized by standard method and the pure products were obtained by repeated distillation. Their boiling points were found to be in accordance as reported in literature (315).

2. P - n - alkoxybenzaldehydes

Various methods have been described for preparing p.n. alkoxybenzaldehyde (316 - 317), but following procedure was adopted with better results in this investigation. Though first member viz. p.methoxy benzaldehyde of the BDH grade was bought and purified. 0.1 mole of p-hydroxybenzaldehyde, 0.15 mole of anhy. potassium carbonate and 0.15 mole of corresponding n.alkyl bromide or iodide were added to dry acetone (60 ml). The mixture is refluxed, using water both, for six to eight hours. The refluxing period was extended to ten to twelve hours in case of higher members. The whole mass was then added to cold water and the aldehydes separated us the form of oily layer were extracted with ether. Ether extract was washed with dilute sodium hydroxide solution to remove unreacted p. hydroxybenzaldehyde followed by water and then dried. Ether was evaporated and p.n. alkoxy benzaldehydes thus obtained were purified by distilling under reduced pressure. Boiling points were found to be in agreement with those reported in literature (320).

3. Trans-p-n. Alkoxycinnamic Acids

These acids were prepared by the method of Gray and Jones (319).

p.n.alkoxybenzaldehyde (0.02 moles), malonic acid (0.04 moles), Pyridine (8 ml) and Piperidine (3 drops) were mixed and heated at 100°C for three hours on a steam bath. The mixture was poured in a beaker containing ice (25 gms) and conc.hydrochloric acid (25 ml). The precipitate were filtered and washed with dilute hydrochloric acid followed by water. The compounds were crystallised from 98% acetic acid. Yield of colourless product was 85 to 90%. The higher homologues were crystallised twice from benzene and then from acetic acid till constant transition temperatures were obtained. These are listed in Table - 2.

4. Trans-p-n-Alkoxycinnamoyl Chlorides

These were prepared by treating the corresponding trans-p-n-alkoxycinnamic acids with excess of thionyl chloride and heating on a water bath till evolution of hydrogen chloride gas ceased. Excess of thionyl chloride was distilled off under reduced pressure using a water pump. The acid chlorides left behind in the form of residue were used for further reaction.

5. n-Butyl-p.hydroxybenzoates

These were prepared by refluxing p.hydroxy benzoic acid (50 gms), absolute n. butanol (200 ml) and concentrated (98%) sulphuric acid (2 ml) on a sand bath

for six to eight hours. The whole mass was then poured on to ice water (100 ml) containing 1 ml concentrated hydro-chloric acid. The product separated from water was washed with aqueous sodium bicarbonate solution (4N) and then with water. It was crystallised from 98% ethanol, and further purified by column chromatography using activated alumina-acetone system. Yield was 85%, m.p. 68°C. Elemental analysis conforms with the calculated values.

6. n-Amyl-p.hydroxybenzoate

This compound was prepared by refluxing dried n-amyl alcohol and p-hydroxy benzoic acid in presence of concentrated sulphuric acid as in A5 above. The product was obtained in liquid form which was extracted with ether. The ether layer was washed with Sodium Bicarbonate solution (4N) and then with water and dried. Yield was 70%. B.P. 60°C.

7. n-Hexyl-p-hydroxybenzoate

This compound was prepared by refluxing dried n-hexanol and p.hydroxy benzoic acid in presence of concentrated sulphuric acid as in A5 above. The liquid

The liquid product was extracted with ether. The ether layer was washed with sodium bicarbonate solution (4N) and then with water. The ether was evaporated and the liquid product was dried over anhydrous sodium carbonate. Yield was 65%. B.P: 112°C.

8. n-Heptyl-p-hydroxy benzoate

This compound was prepared by refluxing absolute n-heptanol and p.hydroxybenzoic acid in presence of concentrated sulphuric acid as in A5 above. The liquid product was extracted with ether. The ether layer was washed with sodium bicarbonate solution (4N) and then with water. The ether was evaporated and the liquid product was dried over anhydrous sodium carbonate. Yield was 65%. B.P. 137°C

9. n-Butyl-p-(p-n-alkoxy cinnamoyloxy) benzoates

These were synthesized by adding dry n - butyl-p-hydroxy benzoate (0.01 mole), dissolved in dry pyridine (A.R. 10 ml), to trans p-n-alkoxy cinnamoyl Chloride (0.015 mole). The mixture was warmed on water bath, while shaking for an hour and was allowed to stand overnight. It was acidified with cold dilute hydrochloric acid. The precipitates were filtered and were washed with cold dilute sodium hydroxide solution followed by

water. The product was crystallised from ethanol to get almost white needles though the higher members were in the form of white or pale yellow powder. The melting points and transition temperatures are listed in Table 5. The analytical data is listed in Table 6. Yield was approximately 50%.

10. n-Amyl-p-(p'-n'.alkoxy cinnamoyloxy) benzoate

These compounds were prepared by reacting n-amyl-p-hydroxy benzoate dissolved in phridine with p-n-alkoxy cinnamoyl chloride as in A9 above. They were crystallised from ethanol in the form of fine white needles or powder. The melting points and transition temperatures are listed in Table - 7. The analytical data is listed in Table 8. Yield was 45%.

11. n-Hexyl-p- (p'-n'-alkoxy cinnamoyloxy)benzoate

These compounds were synthesized by reacting n-hexyl-p.hydroxy benzoate dissolved in dry pyridine with p-n.alkoxy cinnamoyl chlorides as in A 9 above. The products were crystallised in hot alcohol to get the white needles or powder. The melting points and transition temperatures are listed in Table - 9. Analytical data is listed in Table 10. Yield was 40%.

12. n-Heptyl-p- (p'-n'-alkoxy cinnamoyloxy) benzoate

These compounds were prepared by reacting n-heptyl.p-hdroxy benzoates dissolved in dry pyridine with p-n-alkoxy cinnamoyl chlorides as in A 9 above. The products were crystallised in alcohol to get the white needles or powder. The melting points and transition temperatures as listed in Table - 11 and analytical data is compiled in Table 12. Yield was approximately 40%.

13. P-(p'-n-Alkoxy benzoyloxy) benzoic acids

These compounds were prepared by reacting p-n.alkoxybenzoyl chlorides with p.hydroxy benzoic acid. The product were crystallized from acetic acid to get pale yellow colid. The melting points and transition temperatures are given in Table 13. The analytical data are given in Table 14. Yield was 50%.

14. P- (p'-n-alkoxy cinnamoyloxy) benzaldehyde

These compounds were prepared by reacting p-n-alkoxy cinnamoyl chloride with p-hydroxy belzaldehyde as in A 9 above. The precipitates are collected by filtration and are washed with cold dilute sodium hydroxide solution followed by water. The compounds were crystallized from ethanol in the form of pale or dark yellow solid. The melting points and transition temperatures are compiled in Table 4. Yield was approximately 60%.

15. P- (P'-n-alkoxy Cinnamoyloxy) benzylidene-p'-nitroaniline

These compounds are prepared by condensing equimolecular amount (0.01 moles) of P- (p'-n-alkoxy Cinnamoyloxy) benzaldehyde and p.nitroaniline. The mixture is refluxed for one or two hours in 20 to 25 ml ethanol containing a few drops of acetic acid. For higher members the refluxing period was extended to about four hours. The lower members were crystallized from ethanol. The higher members were crystallised from acetic acid. The transition temperatures and analytical data are compiled in Tables 15 and 16 respectively. Yield was approximately 80%.

16. P - (p'-n-alkoxy Cinnamoyloxy) benzylidene-p"-fluoroaniline

These compounds are prepared by condensing equimolecular amount (0.01 moles) of P- (p'-n-Alkoxy Cinnamoyloxy) benzaldehyde and p-fluoro aniline. The mixture is refluxed for two to three hours in 20 to 25 ml. ethanol containing a few drops of acetic acid. The higher members are refluxed for four to five hours. The product were crystallised from alcohol to get fine white or pale yellow powder.

The transition temperatures and analytical data are compiled in Tables 17 and 18 respectively. Yield was approximately 70%.

17. p.n.Alkoxy benzoic acids

These acids were prepared (321) by refluxing 1 mole p.hydroxy benzoic acid dissolved in 2 moles of alcoholic potassium hydroxide with 1.1 mole alkyl iodide or bromide. Under these conditions little or no esterification occurs. To ensure the forward reaction further 1 gm. ofpotassium hydroxide dissolved in very small amount of water, was added to refluxing mixture after seven hours of refluxing. The free acid was liberated by the addition of concentrated hydrochloric acid. The lower members were crystallised from alcohol and higher members were crystallised from acetic acid till constant transitions were obtained. These temperatures are listed in Table 3.

18. P-n. Alkoxybenzoyl chlorides

These were prepared by reacting p.n.- alkoxybenzoic acid and thionyl chloride as in A4above.

19. Phenyl Propionate (322)

This compound was prepared by adding slowly 120 ml of redistilled thionyl chloride to a mixture 150 g. of

pure phenol and 133 ml. of propionic acid. After addition, the mixture was warmed to drive away all the sulphur dioxide and hydrogen chloride, then distilled. Yield was approximately 80%. B.P. 211°C.

20. p-Hydroxy Propiophenone (322)

Dry phenyl propionate (17g) was taken in a round bottom flask and powdered anhydrous aluminium chloride (20g) was added portionwise while keeping the flask cooled in ice cooled water. After half an hour the contents were heated at 55-60°C for about two to three hours andthen left overnight. During this period, hydrogen chloride gas was evolved and the mixture thickened to a brown mass. The cold dilute hydrochloric acid was added to this mass to decompose the aluminium chloride complex. The mixture was then steam distilled to distill off the ortho isomer while the p.hydroxy propiophenone is non-volatile in steam and remained in the flask. It was recrystallised from hot water. Yield was 50%. M.P. 148°C.

21. Phenyl Butyrate

It was prepared by slow addition of 120 ml. of redistilled thionyl chloride to a mixture of 150g of phenol and 160 ml of Butyric acid. The mixture was warned and then left overnight. Then cold dilute hydrochloric acid was added and the mixture was steam distilled. The solid product was

crystallised from hot water. Yield was 60%. B.P. 227°C.

22. p-Hydroxy butyrophenone

Powdered anhydrous aluminium chloride (20.0 g) was slowly added to dry phenyl butyrate (17.0 g). After half an hour the contents were heated at 55 - 60°C for three to four hours and left overnight. The cold dilute hydrochloric acid was added to the mixture and then steam distilled. The p-hydroxy butyrophenone which is non-volatile in steam was crystallised from dilute alcohol. Yield was 40%. M.P. 91°C.

23. p-(p'-n-alkoxybenzoyloxy) propiophenones

Dry p-hydroxypropiophenone (0.01 mole) was dissolved in dry pyridine (A.R. 10 ml.) and was added slowly to the trans-p-n-alkoxybenzoyl chloride (0.015 mole). The mixture was warmed for an hour and then left overnight. It was acidified with cold dilute hydrochloric acid and filtered. The precipitates were washed with cold dilute sodium hydroxide solution followed by water. They were crystallised from ethanol to get fine white needles or powder. Yield was approximately 60%. The melting points and transition temperatures are listed in Table 19, and analytical data are compiled in Table 20.

24. p- (p'-n-alkoxybenzoyloxy) butyrophenones

Dry p-hydroxybutyraphenone (0.01 mole) is dissolved in dry pyridine (A.R. 10 ml) and added slowly to the trans-p-n-alkoxybenzoyl chloride (0.015 mole). The mixture was warmed while shaking for an hour andthen allowed to stand overnight. It was acidified with cold dilute hydrochloric acid and the precipitates were collected by filtration and were washed with cold dilute sodium hydroxide solution followed by water. The esters were crystallised from ethanol to fine white flakes or powder. The melting points and transition temperatures are listed in Table 21 and analytical data is compiled in Table 22. Yield was 50%.

S T U D Y

The study of mesomorphic characteristic was carried by Polarising microscope.

A Polarising microscope, fitted with heating stage arrangement (Kofler) was used to carry out study of mesomorphic properties of newly synthesized compounds(Fig-6).

1. Preparation of Slides

The substance taken on a slide was heated to a little more temperature than its isotropic temperature. A cover slip was placed over it and then made to cool off.

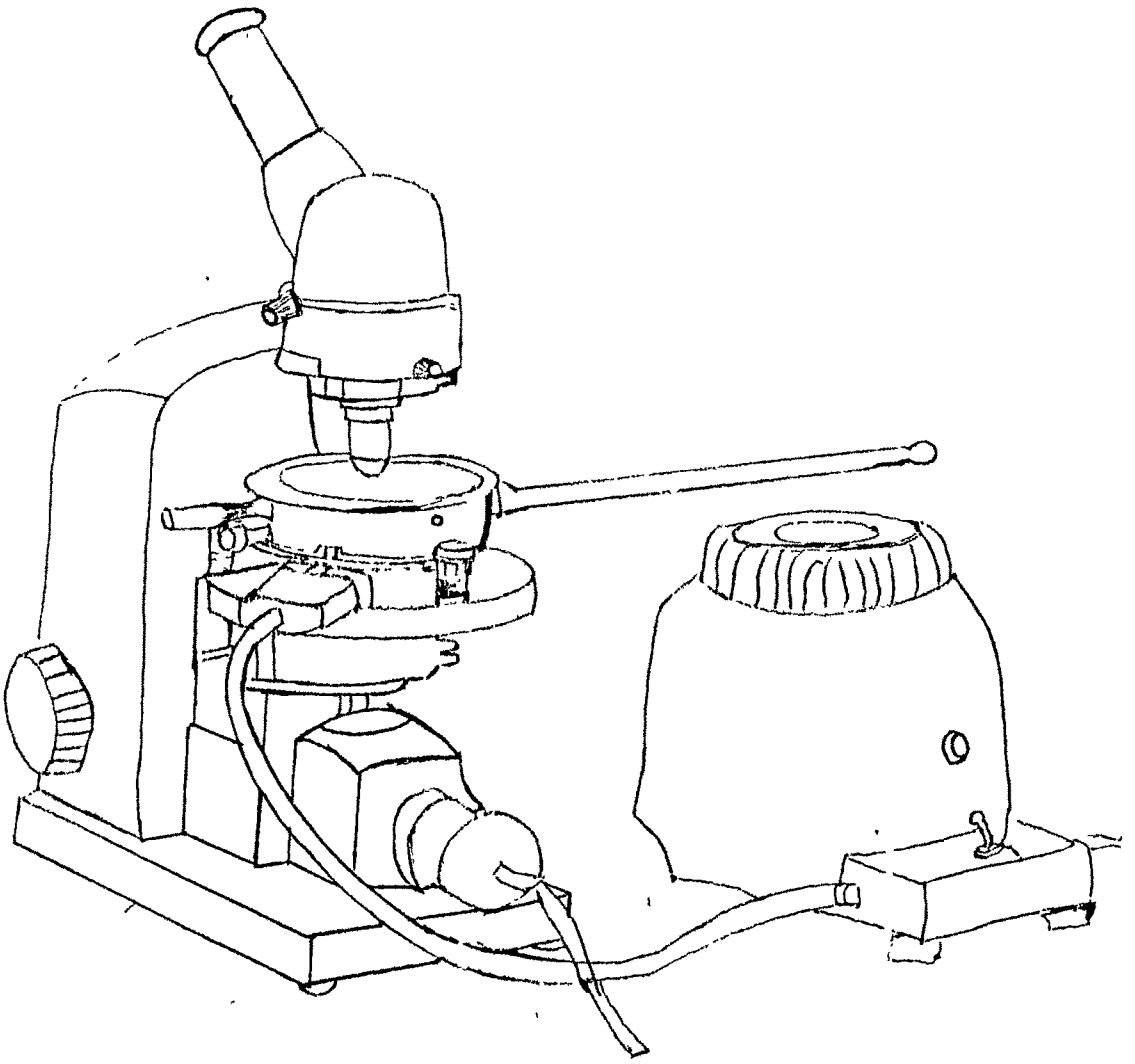


FIG. 6 POLARISING MICROSCOPE WITH KOFLER HEATING STAGE.

2. Heating rate

The prepared slide was then placed over the heating stage of the microscope. A slow rate of about 1° per minute was maintained by means of rheostat attached to the heating stage. The changing textures over the temperature range were carefully observed and characteristics of the phases are noted.

3. Calibration of Thermometer

The thermometer was calibrated by taking melting points and transition points of some known and very pure substances like benzoic acid, p. azoxy-anisole., α.napthol, succinic acid etc.

4. Measurement of transition temperatures of mesogens under study

The prepared slide containing a thin section of the material under study was mounted on the heating stage. Initially the heating rate was kept at 5° per minute to find the transition temperatures approximately. The slide was cooled and, again, temperature was raised, now at a slower rate, the heating rate was regulated to 1°C per minute from about 5°C below the expected transition temperature to measure them accurately. Further, to ensure that phase transition has actually taken place, the cover slip was touched with a spatula. Movement of

cover slip confirms a change from solid to fluid state. The transitions and phases are clearly observed as focal conic, plane, homostropic and threaded textures of smectic and nematic mesophases appear under polarized light. Formation of isotropic liquid is clearly marked as the field of vision becomes extinct in polarized light.

All enantiotropic transitions are clearly detected on cooling the isotropic liquid, as the reverse transitions take place either at the same temperature or within a range of $\pm$ 0.2 to 0.5°C.

Monotropic transitions are determined by carefully observing the isotropic liquid as it cools slowly until batonnets of smectic phase or droplets of nematic phase appeared.

The smectic-nematic transitions are also clearly detected as there is a sharp variation in texture from focal conic to threaded structure while heating and exactly the reversed sequence on cooling under microscope.

All the compounds under study were observed several times under microscope to ensure that no transition was unnoticed. In case of any doubt, the compound was purified again and then observed again under microscope.

DSC and TGA Data

DSC and TGA study of mesomorph is very useful in deciding transformations of structures at the transition. But, due to lack of availability of any facility for conducting these studies, as the apparatus were not available in working conditions, it could not be done. Some samples were sent for DSC but the results of this analysis is still awaited.

T A B L E - 2

Trans p-n-Alkoxycinnamic Acid (319)

n-Alkyl group	Transition Temperatures (in_N°C) S		I
Methyl		174.0	190.0
Ethyl	-	194.0	200.0
Propyl	-	169.0	184.0
Butyl	-	156.0	189.0
Amyl	-	144.0	180.0
Hexyl	-	152.0	180.0
Heptyl	-	148.0	175.0
Octyl	-	145.0	172.0
Decyl	136.0	150.0	169.0
Dodecyl	132.0	157.0	164.0
Tetradecyl	127.0	-	160.0
Hexadecyl	118.0	-	159.0
Octadecyl	120.0	-	157.0

TABLE - 3

Trans-p-n-Alkoxybenzoic Acid (321)

n-alkyl group	Transition Temperatures (in °C)		
	S	N	I
Methyl	-	-	183.4
Etnyl	-	-	197.0
Propyl	-	146.0	156.0
Butyl	-	147.0	160.0
Amyl	-	124.0	151.0
Hexyl	-	105.0	153.0
Heptyl	92.0	98.0	145.0
Octyl	100.0	107.0	146.0
Decyl	97.0	121.0	146.5
Dodecyl	95.0	129.0	137.0
Tetradecyl	93.0	134.5	136.0
Hexadecyl	102.0	-	134.0
Octadecyl	101.5	-	131.0

TABLE - 4

Trans p- (p'-n-Alkoxycinnamoyloxy) benzaldehyde

n-alkyl group	Transition temperatures		
	S	N	I
Methyl	-	(122.5)	125.0
Ethyl	-	(128.0)	144.0
Propyl	-	(106.5)	112.0
Butyl	-	76.5	116.0
Amyl	-	73.0	110.5
Hexyl	-	73.0	113.5
Heptyl	-	71.0	109.5
Octyl	77.0	89.0	111.0
Decyl	83.0	111.5	113.0
Dodecyl	87.0	-	117.0
Tetradecyl	90.0	-	117.5
Octadecyl	94.0	-	118.0

Values in parentheses indicate monotropy.

FIG. 8

p-n-BUTYL (p'-n'-ALKOXYCINNAMOYLOXY) BENZOATE

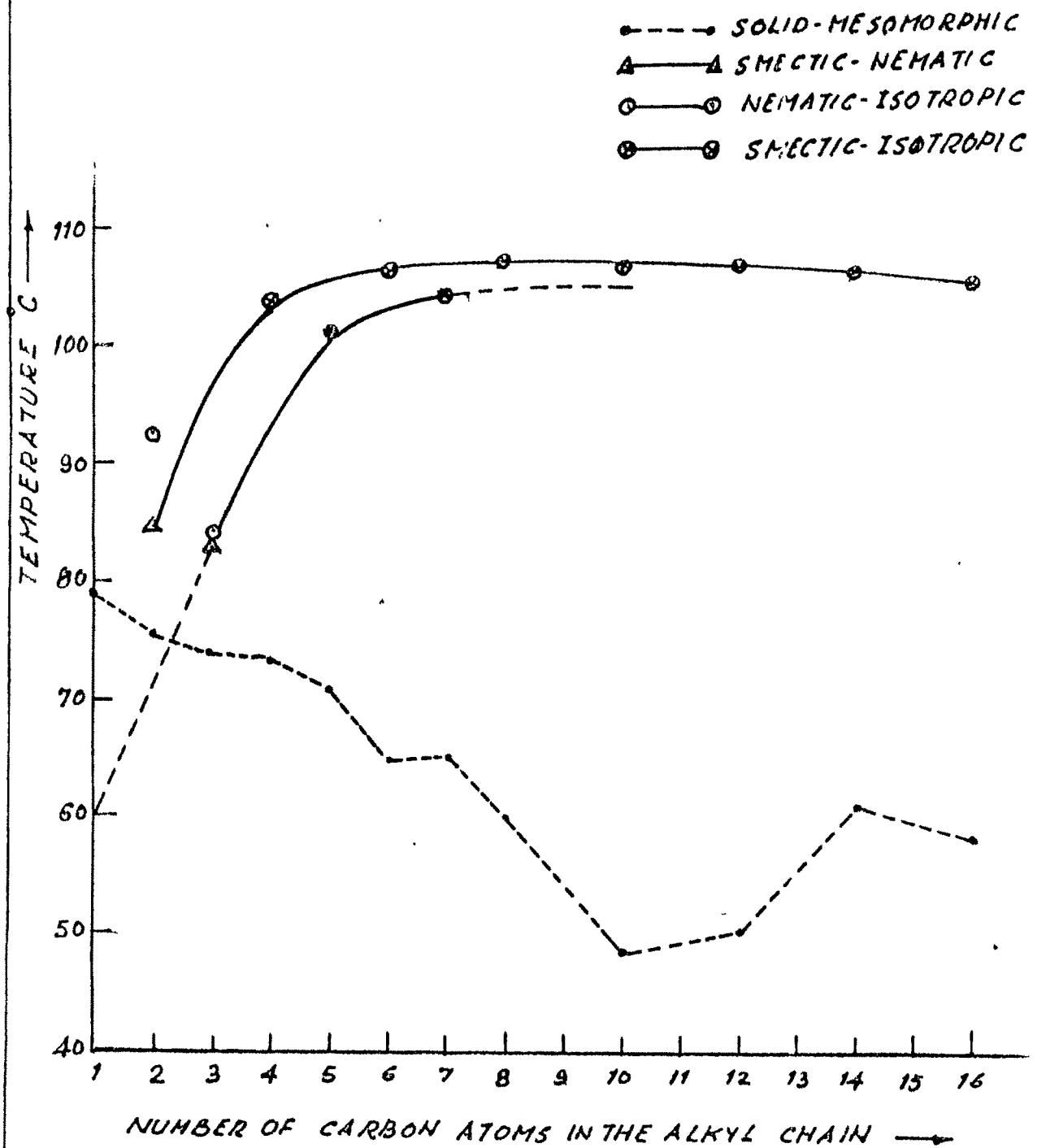


TABLE - 5

P-n-Butyl (p'-n' Alkoxycinnamoyloxy) benzoate

n-Alkyl group	Transition Temperatures (in °C)		
	Smectic	Nematic	Isotropic
Methyl	-	-	79.0
Ethyl	76.0	85.0	92.5
Propyl	74.0	83.0	84.0
Butyl	73.5	-	103.5
Amyl	71.0	-	101.0
Hexyl	65.0	-	106.0
Heptyl	65.5	-	104.0
Octyl	60.0	-	107.0
Decyl	48.5	-	106.5
Dodecyl	50.5	-	106.5
Tetradecyl	61.0	-	106.0
Hexadecyl	59.0	-	107.0

T A B L E - 6

n - Butyl P - (p'-n'-alkoxycinnamoyloxy) benzoate

n-Alkyl group	Molecular formula	% Required		% Found	
		C	H	C	H
Methyl	$C_{21}H_{22}O_5$	71.18	6.21	71.48	6.422
Ethyl	$C_{22}H_{24}O_5$	71.73	6.52	71.83	6.464
Propyl	$C_{23}H_{26}O_5$	72.25	6.80	72.26	6.843
Butyl	$C_{24}H_{28}O_5$	72.72	7.07	72.70	6.840
Amyl	$C_{25}H_{30}O_5$	73.17	7.31	73.18	7.270
Hexyl	$C_{26}H_{32}O_5$	73.58	7.54	73.53	7.313
Heptyl	$C_{27}H_{34}O_5$	73.97	7.76	73.62	7.418
Octyl	$C_{28}H_{36}O_5$	74.33	7.96	74.26	7.909
Decyl	$C_{30}H_{40}O_5$	75.00	8.33	74.28	8.717
Dodecyl	$C_{32}H_{44}O_5$	75.59	8.66	75.37	8.243
Tetradecyl	$C_{34}H_{48}O_5$	76.11	8.95	75.82	8.583
Hexadecyl	$C_{36}H_{52}O_5$	76.59	9.21	76.28	9.113

FIG. 9.

p-n-AMYL (p'-n'-ALKOXYCINNAMOYLOXY) BENZOATE

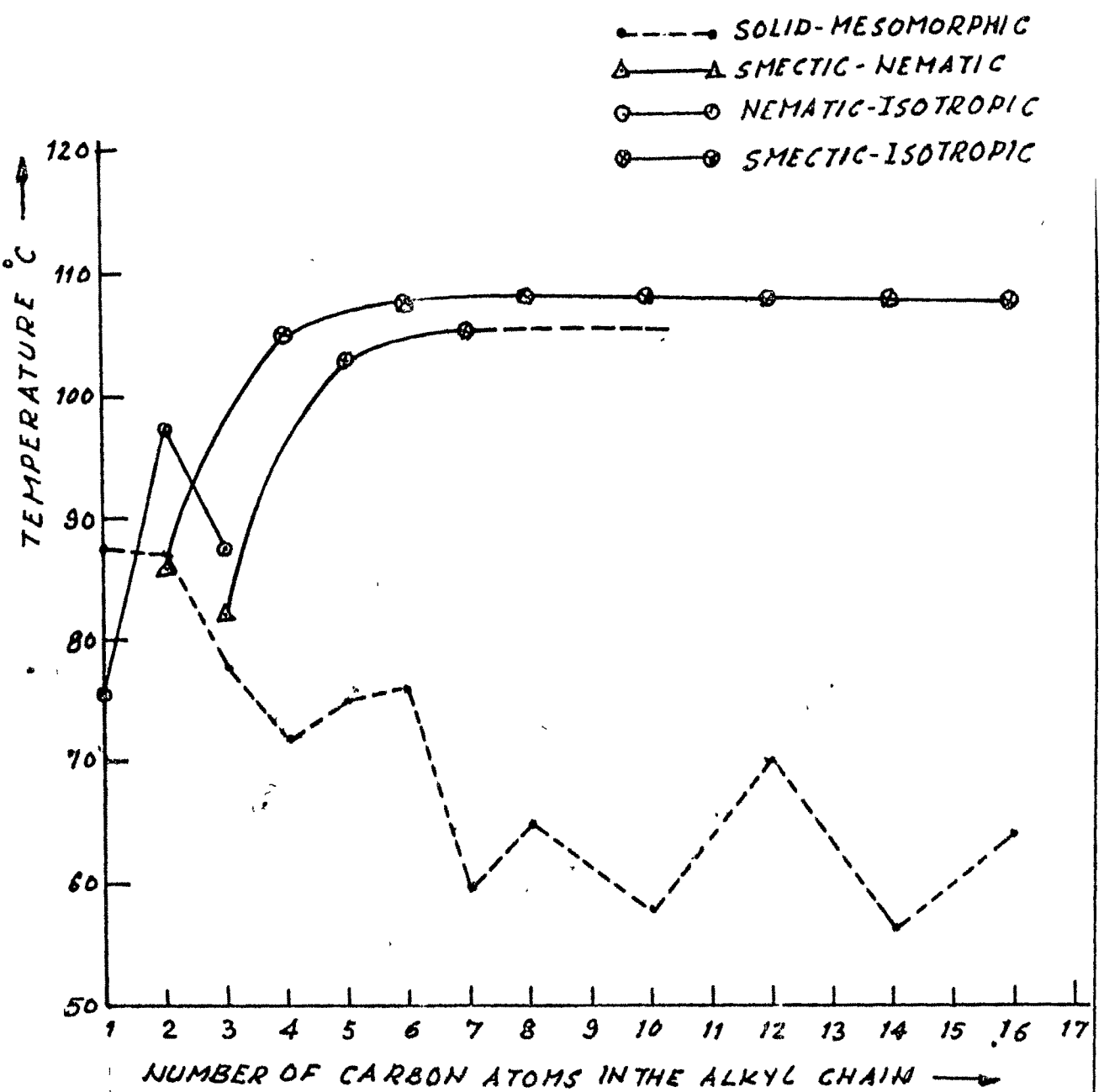


TABLE - 7

n-Amyl o -(p'-n'-Alkoxycinnamoyloxy) benzoate

n.Alkyl group	Transition Temperature (°C)		
	S	N	I
Methyl	-	(76.0)	88.0
Ethyl	(86.0)	87.0	97.5
Propyl	78.0	82.0	87.5
Butyl	72.0	-	105.0
Amyl	75.0	-	103.0
Hexyl	76.0	-	107.5
Heptyl	60.0	-	105.0
Octyl	65.5	-	108.0
Decyl	58.0	-	108.0
Dodecyl	70.0	-	107.5
Tetradecyl	55.0	-	107.5
Hexadecyl	52.0	-	108.0

Values in parentheses indicate monotropy.

TABLE - 8

n - Amyl P - (p'-n'-alkoxycinnamoyloxy) benzoate

n-Alkyl group	Molecular formula	% Required		% Found	
		C	H	C	H
Methyl	$C_{22}H_{24}O_5$	71.73	6.52	71.62	6.232
Ethyl	$C_{23}H_{26}O_5$	72.25	6.80	71.84	6.586
Propyl	$C_{24}H_{28}O_5$	72.72	7.07	72.48	6.834
Butyl	$C_{25}H_{30}O_5$	73.17	7.31	72.70	7.076
Amyl	$C_{26}H_{32}O_5$	73.58	7.54	73.50	7.323
Hexyl	$C_{27}H_{34}O_5$	73.97	7.76	73.54	7.551
Heptyl	$C_{28}H_{36}O_5$	74.33	7.96	74.21	7.910
Octyl	$C_{29}H_{38}O_5$	74.67	8.15	74.53	8.100
Decyl	$C_{31}H_{42}O_5$	75.30	8.50	75.19	8.235
Dodecyl	$C_{33}H_{46}O_5$	75.86	8.81	75.56	8.420
Tetradecyl	$C_{35}H_{50}O_5$	76.36	9.09	76.21	8.728
Hexadecyl	$C_{37}H_{54}O_5$	76.81	9.34	76.80	9.543

FIG. 1090

p-n- HEXYL (p'-n'-ALKOXYCINNAMOYLOXY) BENZOATE

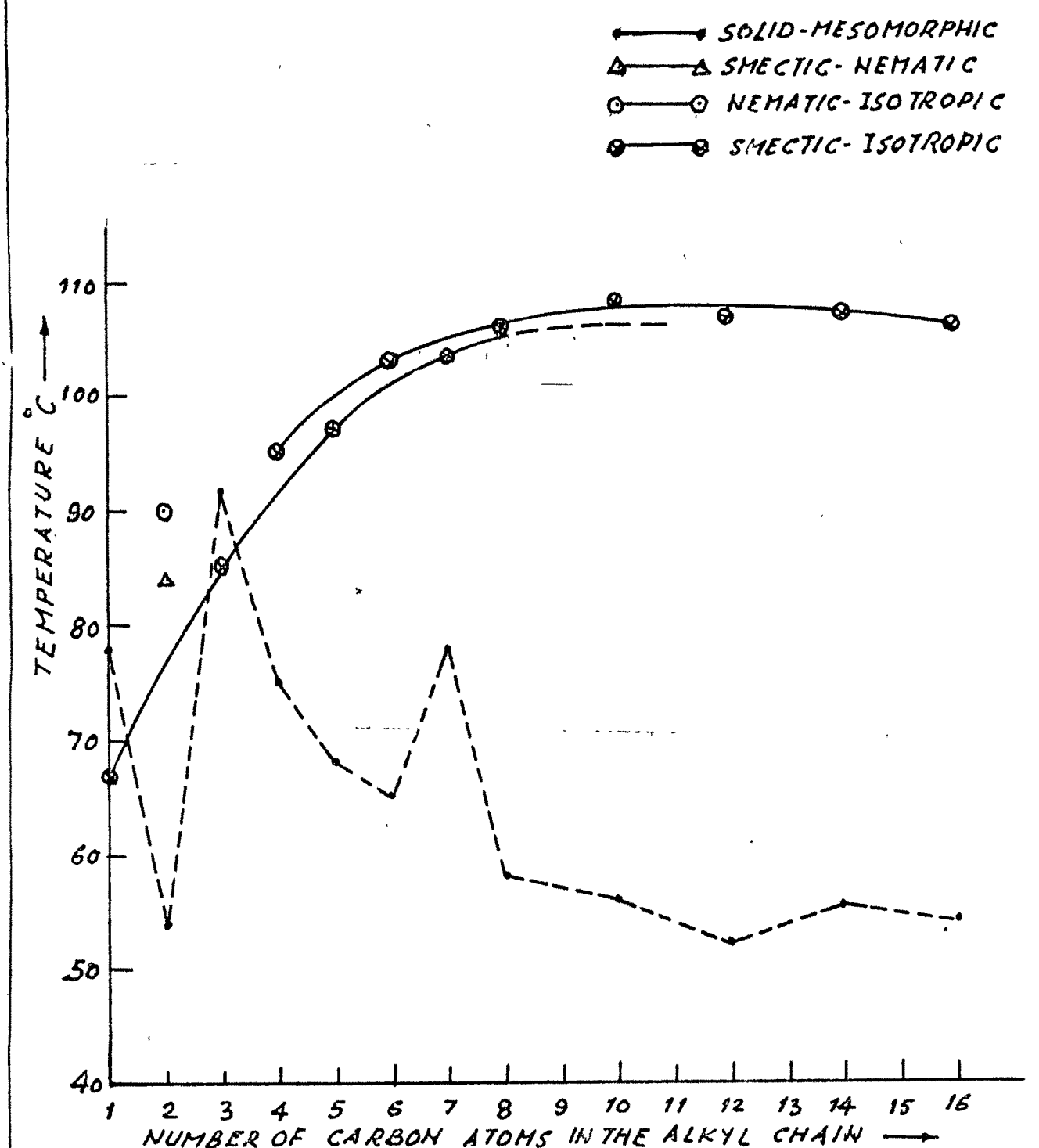


TABLE - 9

n-Hexyl p - (p'-n'-Alkoxycinnamoyloxy) benzoate

n-alkyl Group	Transition Temperature (°C)		
	S	N	T
Methyl	-	(67.0)	78.0
Ethyl	54.0	84.0	90.0
Propyl	(85.0)	-	92.0
Butyl	75.0	-	100.0
Amyl	68.0	-	97.0
Hexyl	65.0	-	103.0
Heptyl	78.0	-	103.5
Octyl	58.0	-	106.0
Decyl	56.0	-	108.0
Dodecyl	52.0	-	106.5
Tetradecyl	55.0	-	107.0
Hexadecyl	56.0	-	107.0

Values in parentheses indicate monotropy.

T A B L E - 10

n- Hexyl P -(p'-n'-alkoxycinnamoyloxy) benzoate

n-Alkyl group	Molecular formula	% required		% Found	
		C	H	C	H
Methyl	$C_{23}H_{24}O_5$	72.25	6.80	72.00	6.761
Ethyl	$C_{24}H_{28}O_5$	72.72	7.07	72.58	6.982
Propyl	$C_{25}H_{30}O_5$	73.17	7.31	72.88	7.041
Butyl	$C_{26}H_{32}O_5$	73.58	7.54	73.52	7.507
Amyl	$C_{27}H_{34}O_5$	73.97	7.76	73.88	7.787
Hexyl	$C_{28}H_{36}O_5$	74.33	7.96	74.42	7.780
Heptyl	$C_{29}H_{38}O_5$	74.68	8.15	74.80	8.224
Octyl	$C_{30}H_{40}O_5$	75.00	8.33	74.94	8.313
Decyl	$C_{32}H_{44}O_5$	75.59	8.66	75.61	8.582
Dodecyl	$C_{34}H_{48}O_5$	76.11	8.95	76.28	6.003
Tetradecyl	$C_{36}H_{52}O_5$	76.59	9.21	76.50	9.112
Hexadecyl	$C_{38}H_{56}O_5$	77.02	9.45	77.11	9.312

FIG. 11.

p-n-HEPTYL (p'-n'-ALKOXYCINNAMOYLOXY) BENZOATE

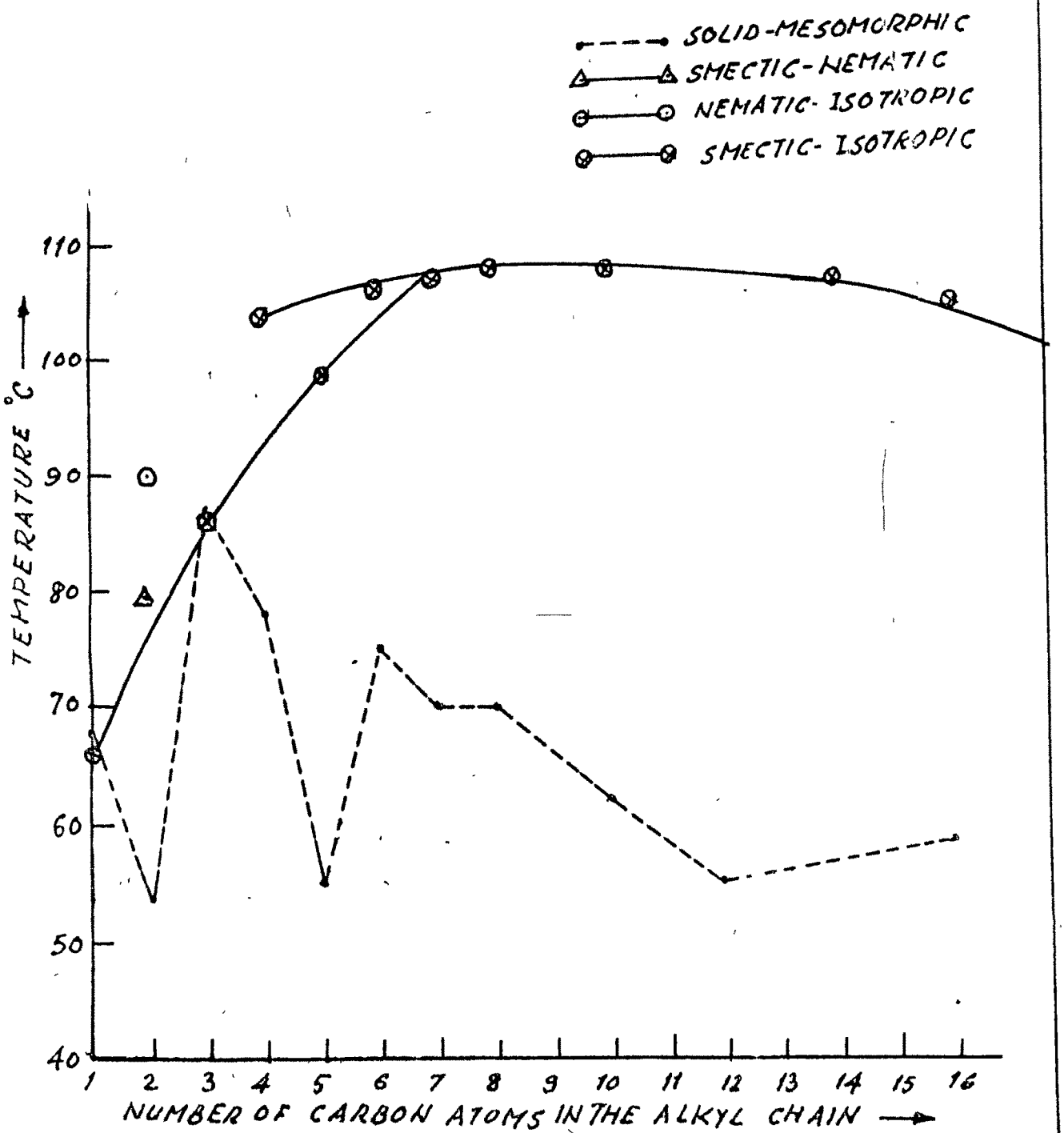


TABLE - 11

n-Heptyl p- (p'-n'- Alkoxycinnamoyloxy) benzoate

n-alkyl Group	Transition Temperatures (°C)		
	S	N	I
Methyl	(66.0)	-	68.0
Ethyl	54.0	80.0	90.0
Butyl	78.0	-	104.0
Amyl	55.0	-	99.5
Hexyl	75.0	-	106.0
Heptyl	70.0	-	107.0
Octyl	70.0	-	107.5
Decyl	62.0	-	107.5
Dodecyl	55.0	-	107.0
Tetradecyl	57.0	-	106.5
Hexadecyl	58.0	-	105.5

Values in parentheses indicate monotropy.

T A B L E - 12

n-Heptyl P- (p'-n'-alkoxycinnamoyloxy) benzoate

n-Alkyl group	Molecular formula	% Required		% Found	
		C	H	C	H
Methyl	$C_{24}H_{28}O_5$	72.72	7.07	72.56	7.121
Ethyl	$C_{25}H_{30}O_5$	73.13	7.31	72.98	7.281
Propyl	$C_{26}H_{32}O_5$	73.58	7.54	73.60	7.554
Butyl	$C_{27}H_{34}O_5$	73.97	7.76	73.85	7.800
Amyl	$C_{28}H_{36}O_5$	74.33	7.96	74.62	7.908
Hexyl	$C_{29}H_{38}O_5$	74.68	8.15	74.67	8.118
Heptyl	$C_{30}H_{40}O_5$	75.00	8.33	75.01	8.321
Octyl	$C_{31}H_{42}O_5$	75.30	8.50	75.25	8.517
Decyl	$C_{33}H_{46}O_5$	75.86	8.81	75.91	8.622
Dodecyl	$C_{35}H_{50}O_5$	76.36	9.09	76.30	9.110
Tetradecyl	$C_{37}H_{54}O_5$	76.81	9.35	76.75	9.307
n-Hexadecyl	$C_{39}H_{58}O_5$	77.21	9.57	77.11	9.452

FIG 12.

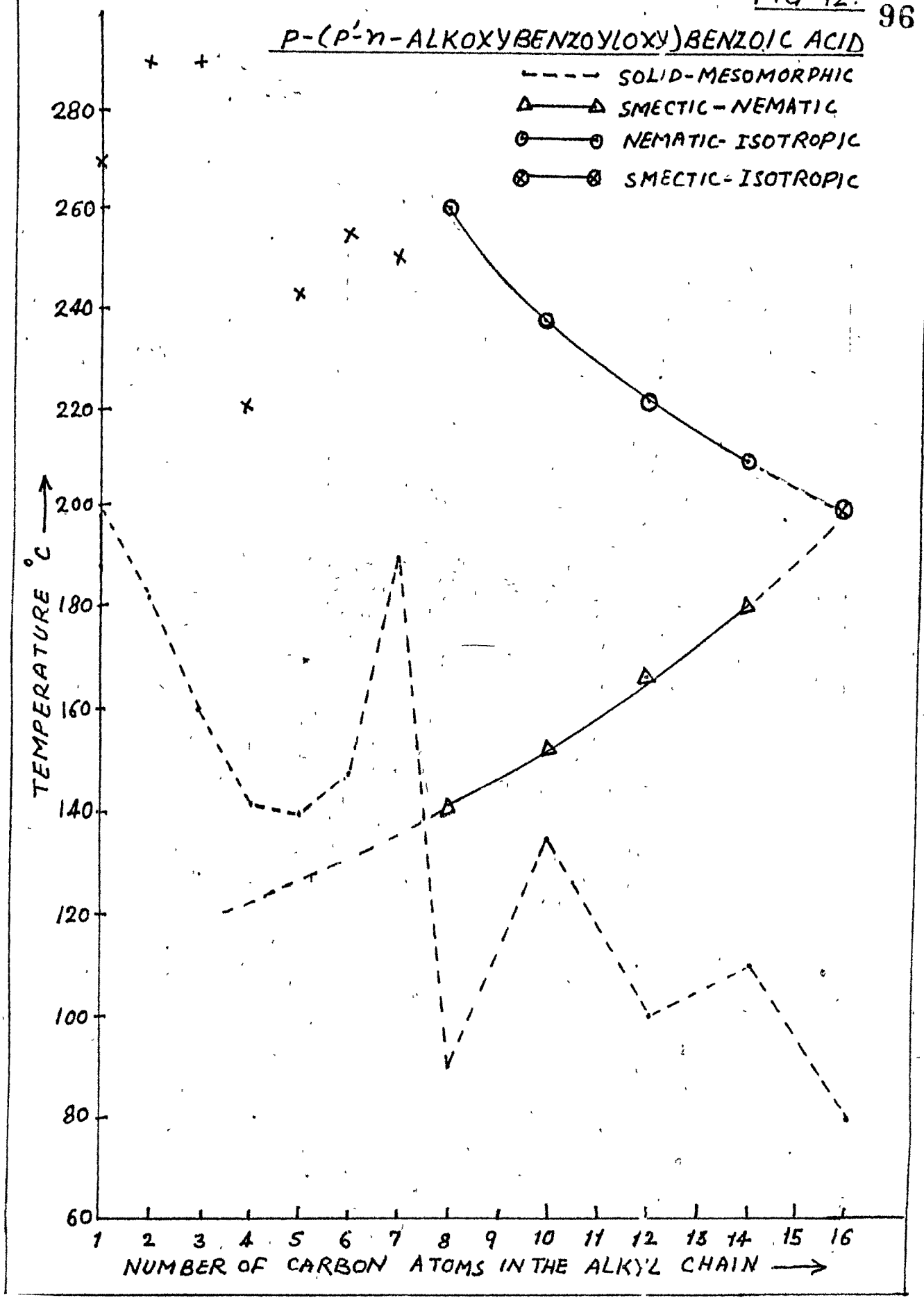

P-(P'-n-ALKOXYBENZOYLOXY)BENZOIC ACID
SOLID-MESOMORPHIC
SMECTIC-NEMATIC
NEMATIC-ISOTROPIC
SMECTIC-ISOTROPIC
TEMPERATURE °C
280
260
240
220
200
180
160
140
120
100
80
60
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
NUMBER OF CARBON ATOMS IN THE ALKYL CHAIN

TABLE - 13

P- (p'-n-Alkoxybenzoyloxy) benzoic acid

n-alkyl group	Transition Temperature (°C)		
	S	N	I
Methyl	-	200.0	270.0 (d)
Ethyl	-	102.0	290.0 (d)
Propyl	-	160.0	290.0 (d)
Butyl	-	142.0	220.0 (d)
Amyl	-	140.0	243.0 (d)
Hexyl	-	147.0	240.0 (d)
Heptyl	-	185.0	250.0 (d)
Octyl	90.0	140.0	260.0
Decyl	135.0	152.0	236.0
Dodecyl	100.0	166.0	220.0
Tetradecyl	110.0	180.0	209.0
Hexadecyl	73.0	-	200.0

T A B L E - 14

P- (p'-n-Alkoxybenzoyloxy) benzoic acid

n-Alkyl Group	Molecular formula	% Required		% Found	
		C	H	C	H
Methyl	$C_{15}H_{12}O_5$	66.17	4.418	66.32	4.60
Ethyl	$C_{16}H_{14}O_5$	67.13	4.891	67.61	4.423
Propyl	$C_{17}H_{16}O_5$	68.00	5.335	67.81	5.728
Butyl	$C_{18}H_{18}O_5$	68.78	5.736	69.13	5.303
Amyl	$C_{19}H_{20}O_5$	69.51	6.092	69.79	5.678
Hexyl	$C_{20}H_{22}O_5$	70.17	6.423	70.47	6.098
Heptyl	$C_{21}H_{24}O_5$	70.78	6.741	70.92	6.68
Octyl	$C_{22}H_{26}O_5$	71.35	7.024	71.11	6.646
Decyl	$C_{24}H_{30}O_5$	72.36	7.538	71.93	7.850
Dodecyl	$C_{26}H_{34}O_5$	73.23	7.981	72.94	7.567
Tetradecyl	$C_{28}H_{38}O_5$	74.00	8.372	73.89	8.013
Hexadecyl	$C_{30}H_{42}O_5$	74.68	8.713	74.52	8.611

FIG. 13.

p(p'-n-ALKOXY CINNAMOYLOXY) BENZILIDINE -p''-NITROANILINE

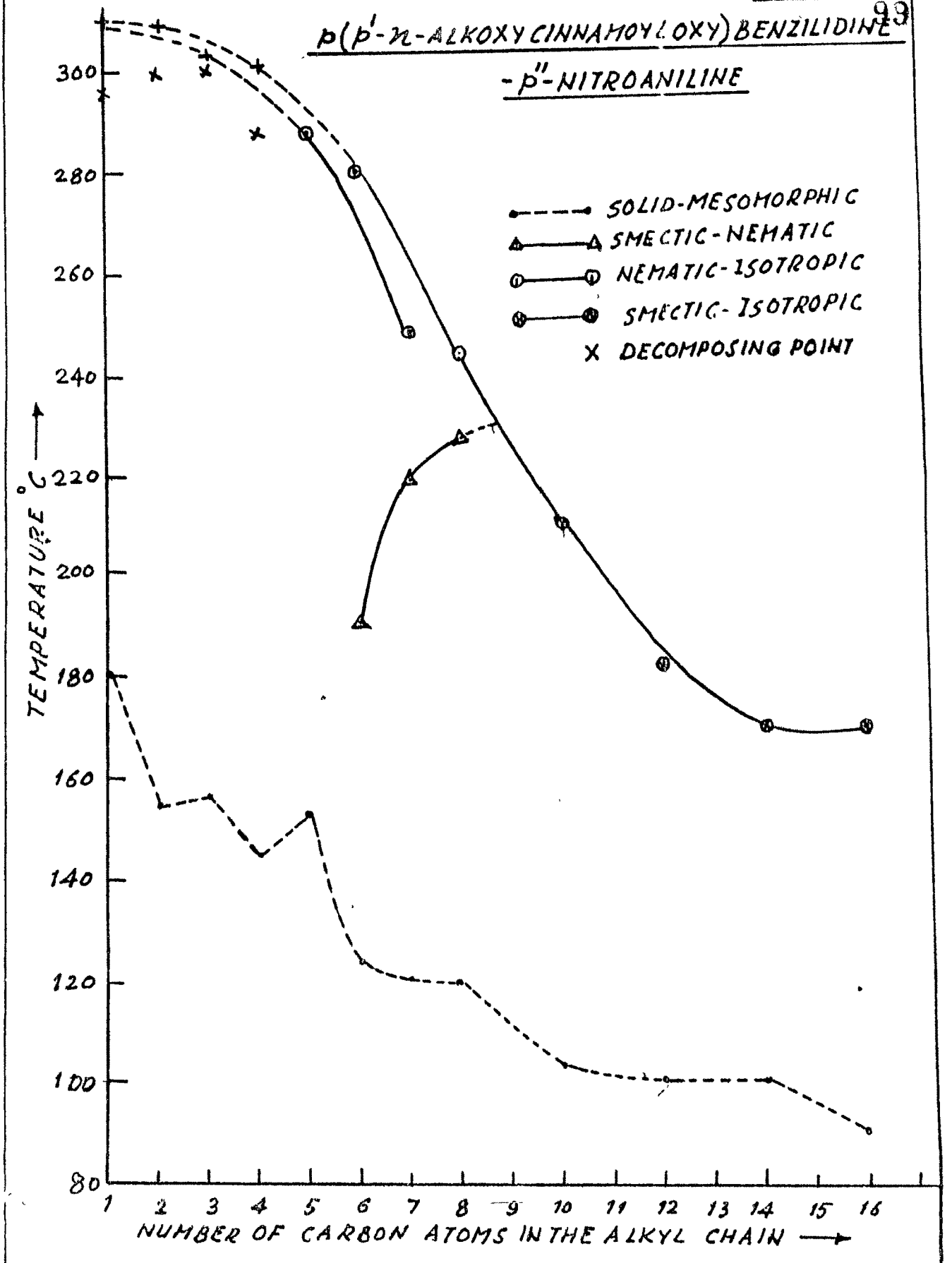


TABLE - 15

P- (p'-n.Alkoxycinnamoyloxy) benzilidine -p"-nitroaniline

n-Alkyl Group	Transition Temperature (°C)		
	S	N	I
Methyl	-	180.0	300.0 (d)
Ethyl	-	157.0	300.0 (d)
Propyl	-	158.0	300.5 (d)
Butyl	-	145.0	288.0 (d)
Amyl	-	152.0	288.0 (d)
Hexyl	124.0	190.0	280.0
Heptyl	121.0	220.0	249.0
Octyl	120.0	228.0	245.0
Decyl	103.0	-	210.0
Dodecyl	100.0	-	183.0
Tetradecyl	100.0	-	170.0
Texadecyl	90.0	-	170.0

d - decomposition temperature.

TABLE - 16

P - (p'-n-alkoxycinnamoyloxy) benzylidene-p"-nitroaniline

n-Alkyl Group	Molecular formula	% Required			% Found		
		C	H	N	C	H	N
Methyl	$C_{23}H_{18}N_2O_4$	74.59	4.86	7.56	74.82	5.018	6.112
Ethyl	$C_{24}H_{20}N_2O_4$	75.00	5.20	7.28	75.25	5.381	6.392
Propyl	$C_{25}H_{22}N_2O_4$	75.37	5.52	7.03	75.66	5.822	6.211
Butyl	$C_{26}H_{24}N_2O_4$	75.72	5.82	6.78	75.93	5.801	6.287
Amyl	$C_{27}H_{26}N_2O_4$	76.05	6.10	6.57	76.32	6.511	6.018
Hexyl	$C_{28}H_{28}N_2O_4$	76.36	6.36	6.36	76.78	6.015	6.127
Heptyl	$C_{29}H_{30}N_2O_4$	76.65	6.60	6.16	76.97	6.412	5.853
Octyl	$C_{30}H_{32}N_2O_4$	76.92	6.83	5.98	77.05	6.405	5.622
Decyl	$C_{32}H_{36}N_2O_4$	77.40	7.25	5.64	77.62	6.927	5.172
Dodecyl	$C_{34}H_{40}N_2O_4$	77.86	7.63	5.34	77.68	7.321	4.032
Tetradecyl	$C_{36}H_{44}N_2O_4$	78.26	7.96	5.06	77.93	7.803	4.381
Hexadecyl	$C_{38}H_{48}N_2O_4$	78.62	8.27	4.82	78.41	8.412	4.286

FIG. 14.

p-(p'-n-ALKOXYCINNAMOYLOXY) BENZYLIDENE-p''-FLUORANILINE

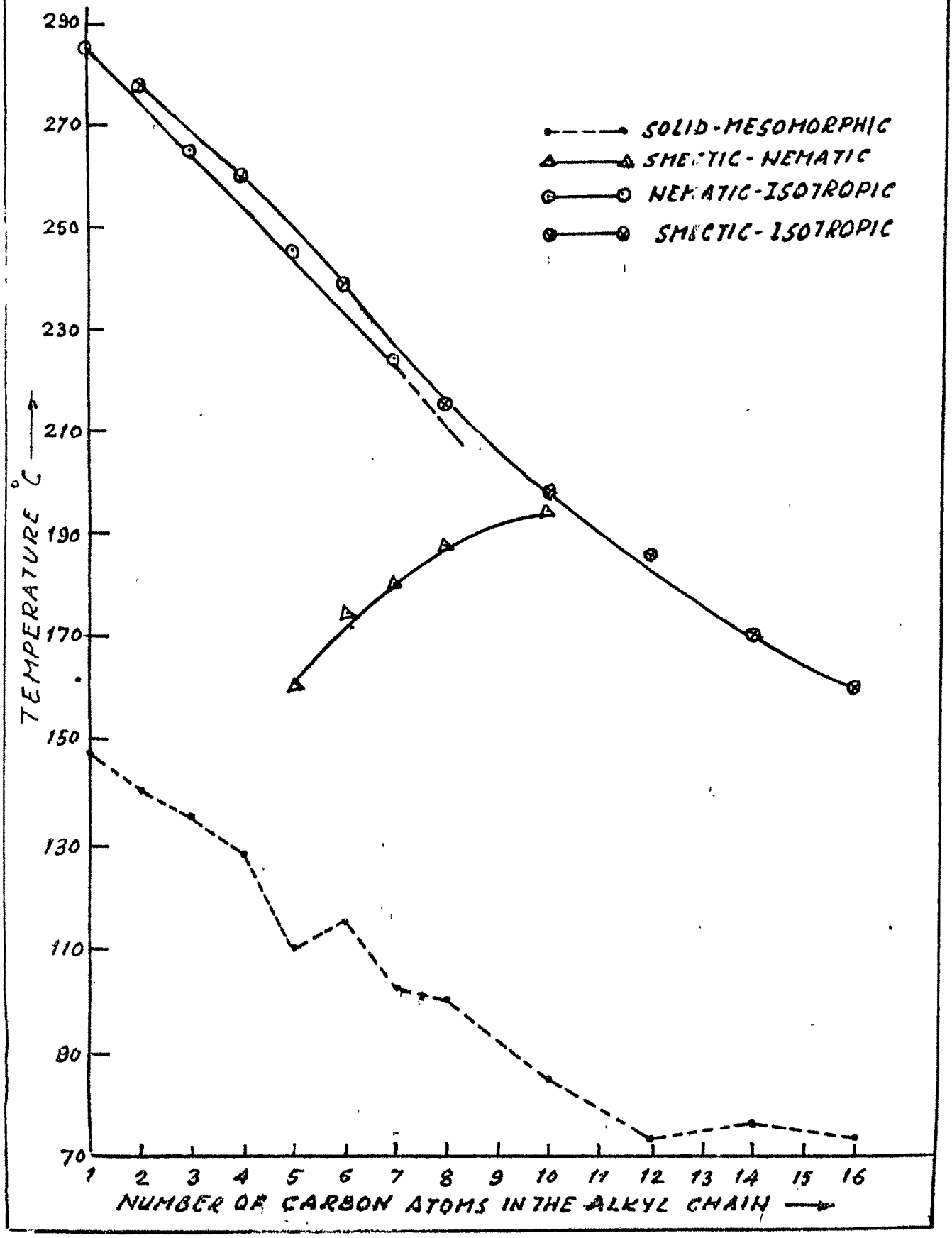


TABLE - 17

P- (p'-n-Alkoxycinnamoyloxy) benzilidine -p' - fluoroaniline

n-Alkyl Group	Transition Temperature (°C)		
	S	N	I.
Methyl	-	147.0	285.0
Ethyl	-	140.0	272.0
Propyl	-	135.0	265.0
Butyl	-	128.0	250.0
Amyl	110.0	152.0	227.0
Hexyl	111.0	168.0	222.0
Heptyl	105.0	180.0	215.0
Octyl	100.0	187.0	215.0
Decyl	85.0	194.0	198.0
Dodecyl	73.0	-	185.0
Tetradecyl	76.0	-	170.0
Hexadecyl	73.0	-	160.0

T A B L E - 18

P - (p'-n-Alkoxycinnamoyloxy) benzylidene - P" - Fluoroaniline

n-Alkyl group	Molecular formula	% Required			% Required		
		C	H	N	C	H	N
Methyl	$C_{23}H_{18}O_3NF$	73.86	4.80	3.73	73.60	5.072	3.359
Ethyl	$C_{24}H_{20}O_3NF$	74.03	5.14	3.59	73.82	5.181	3.621
Propyl	$C_{25}H_{22}O_3NF$	74.44	5.45	3.47	74.67	5.249	2.994
Butyl	$C_{26}H_{24}O_3NF$	74.82	5.75	3.35	74.91	5.513	3.112
Amyl	$C_{27}H_{26}O_3NF$	75.17	6.03	3.24	74.98	5.791	3.015
Hexyl	$C_{2828}H_{}O_3NF$	75.50	6.29	3.14	75.22	5.837	2.912
Heptyl	$C_{29}H_{30}O_3NF$	75.81	6.53	3.05	75.62	6.022	2.891
Octyl	$C_{30}H_{32}O_3NF$	76.10	6.76	2.96	76.325	6.821	2.440
Decyl	$C_{32}H_{36}O_3NF$	76.64	7.18	2.79	76.71	7.257	3.011
Dodecyl	$C_{34}H_{40}O_3NF$	77.12	7.56	2.64	76.78	7.115	2.025
Tetradecyl	$C_{36}H_{44}O_3NF$	77.55	7.89	2.51	77.02	7.613	2.031
Hexadecyl	$C_{38}H_{48}O_3NF$	77.95	8.20	2.39	77.43	7.981	1.981

FIG. 15.

p-(p'-n'-ALKOXY BENZOYLOXY) PROPIOPHENONE

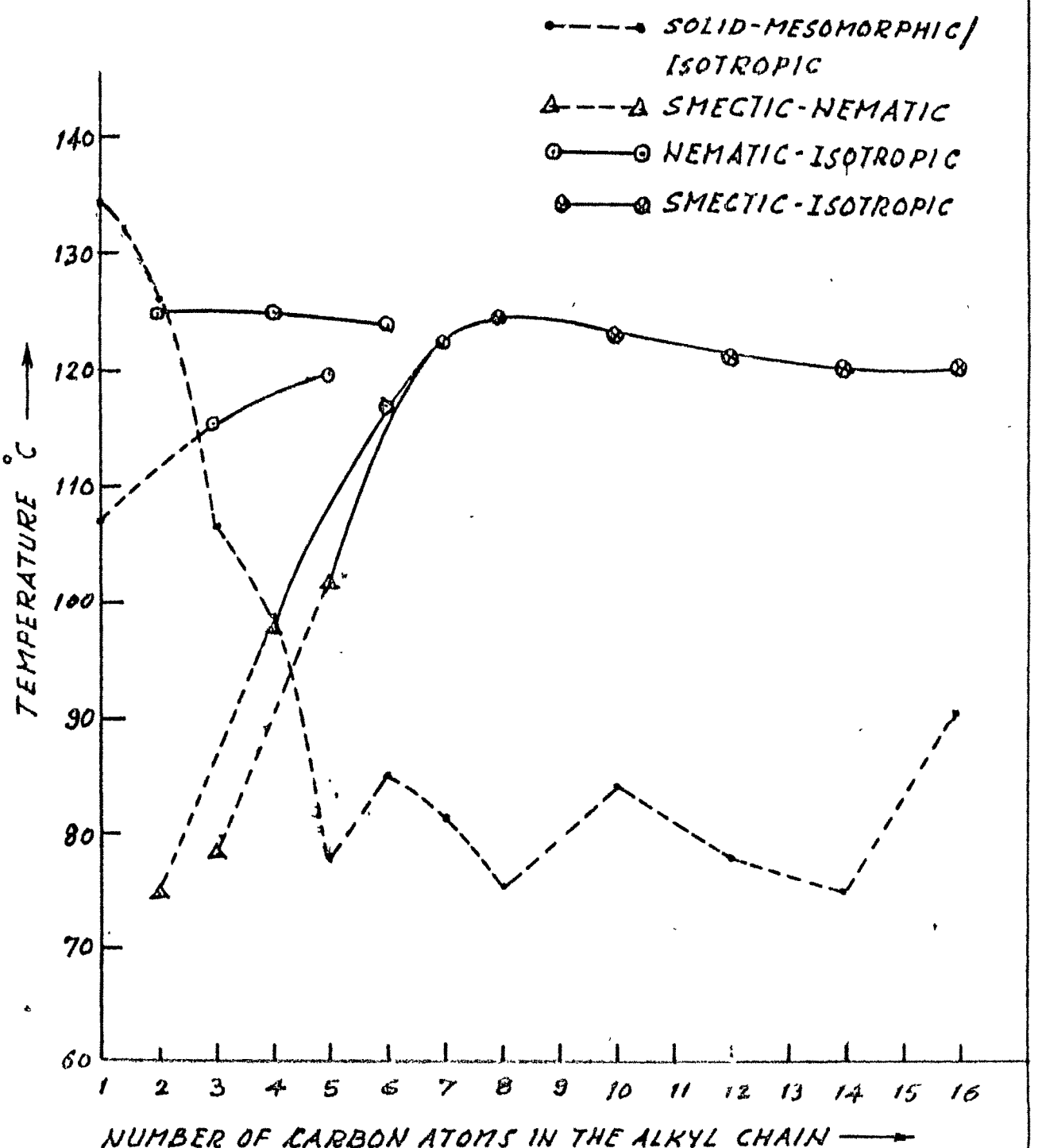


TABLE - 19

P- (p'-n-Alkoxybenzoyloxy) propiophenone

n-Alkyl Group	Transition Temperature (°C)		
	S	N	I
Methyl	-	-	134.5
Ethyl	-	(125.0)	126.0
Propyl	-	106.5	115.5
Butyl	(98.0)	98.5	125.0
Amyl	78.0	102.0	119.5
Hexyl	85.0	117.0	124.0
Heptyl	81.5	-	122.5
Octyl	76.5	-	124.5
Decyl	84.0	-	123.0
Dodecyl	78.0	-	121.0
Tetradecyl	75.0	-	120.0
Hexadecyl	90.0	-	120.0

Values in the paranthesis indicate monotropy.

T A B L E - 20

P - (p'-n- Alkoxybenzoyloxy) propiophenone

n-Alkyl group	Molecular formula	% Required		% Required	
		C	H	C	H
Methyl	$C_{17}H_{16}O_4$	71.83	5.63	71.54	5.625
Ethyl	$C_{18}H_{18}O_4$	72.48	6.04	72.00	5.845
Propyl	$C_{19}H_{20}O_4$	73.07	6.41	73.23	5.960
Butyl	$C_{20}H_{22}O_4$	73.61	6.74	73.60	6.457
Amyl	$C_{21}H_{24}O_4$	74.11	7.05	74.12	6.921
Hexyl	$C_{22}H_{26}O_4$	74.57	7.34	74.35	6.903
Heptyl	$C_{23}H_{28}O_4$	75.00	7.60	74.63	7.223
Octyl	$C_{24}H_{30}O_4$	75.39	7.85	75.78	7.675
Decyl	$C_{26}H_{34}O_4$	76.09	8.29	75.60	7.854
Dodecyl	$C_{28}H_{38}O_4$	76.71	8.67	76.24	8.200
Tetradecyl	$C_{30}H_{42}O_4$	77.25	9.01	77.26	9.034
Hexadecyl	$C_{32}H_{46}O_4$	77.73	9.31	77.61	9.275

FIG. 108

p-(p'-n-ALKOXYBENZOYLOXY) BUTYROPHENONE

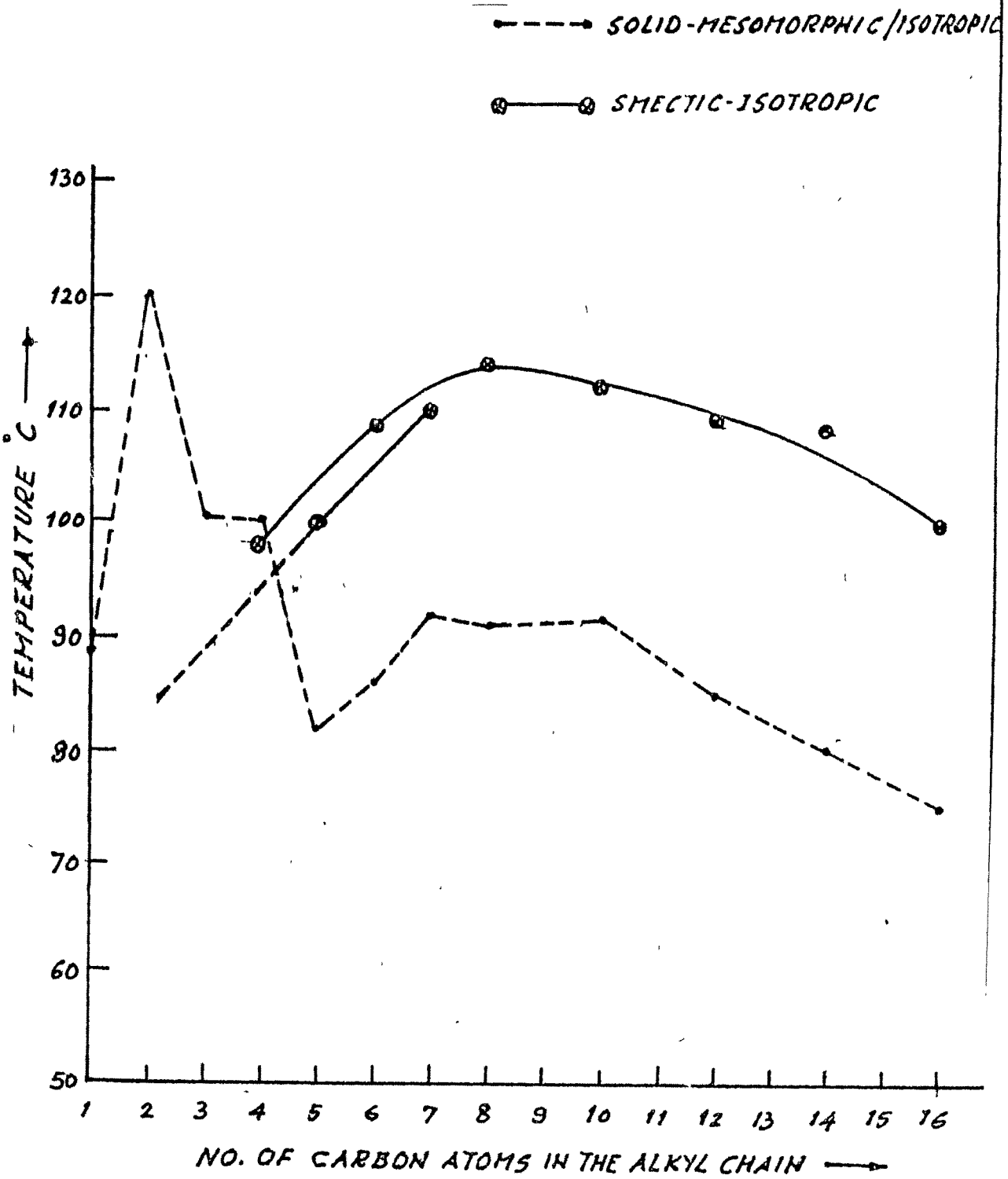


TABLE - 21

P-(p'-n-Alkoxybenzoyloxy) butyrophenone

n-Alkyl Group	Transition Temperature (°C)		
	S	N	I
Methyl	(77)*	-	89.0
Ethyl	(87)*	-	120.0
Propyl	(90)*	-	100.5
Butyl	(98.0)	-	100.0
Amyl	82.0	-	100.5
Hexyl	86.0	-	109.0
Heptyl	92.0	-	110.0
Octyl	91.0	-	114.0
Decyl	91.5	-	112.0
Dodecyl	85.0	-	109.0
Tetradecyl	80.0	-	108.0
Hexadecyl	75.0	-	99.5

* Extrapolated values.

Values in the paranthesis indicate monotropy.

T A B L E - 22

P- (p'-n-Alkoxybenzoyloxy) butyrophenone

n-Alkyl group	Molecular formula	% Required		% Found	
		C	H	C	H
Methyl	$C_{18}H_{18}O_4$	72.48	6.04	72.00	5.758
Ethyl	$C_{19}H_{20}O_4$	73.07	6.41	73.55	6.238
Propyl	$C_{20}H_{22}O_4$	73.61	6.74	73.60	6.575
Butyl	$C_{21}H_{24}O_4$	74.11	7.05	74.32	7.117
Amyl	$C_{22}H_{26}O_4$	74.57	7.34	74.60	7.285
Hexyl	$C_{23}H_{28}O_4$	75.00	7.60	74.85	7.785
Heptyl	$C_{24}H_{30}O_4$	75.39	7.85	75.30	7.911
Octyl	$C_{25}H_{32}O_4$	75.75	8.08	75.50	7.793
Decyl	$C_{27}H_{36}O_4$	76.41	8.49	76.00	8.040
Dodecyl	$C_{29}H_{40}O_4$	76.99	8.84	76.47	8.472
Tetradecyl	$C_{31}H_{44}O_4$	77.50	9.16	77.38	89.272
Hexadecyl	$C_{33}H_{48}O_4$	77.95	9.44	78.00	9.540

PART - 2

MIXED MESOMORPHISM

Study of mixed mesomorphism in binary systems, none one or both components of which are mesogens is very helpful in establishing generalizations concerning factors responsible for exhibition of mesomorphism. Mixed mesomorphism has also contributed recently to applications of liquid crystals by way of widening the scope of utility of certain mesogens.

PREPARATION OF COMPOUNDS

Both components of the binary mixtures are weighed accurately in known proportions in a thoroughly clean sample tube. The total weight of the mixture taken is around 0.1 g. accurately weighed. The mixture in the tube was then heated in an oil bath to a temperature slightly higher than at which the mixture melts. At the melting condition, the mixture is stirred thoroughly with a glass stirrer in order to get a homogenous mixture. The tube is then cooled by quenching it in a beaker containing cold water. The mixture solidifies instantly. The mixture was then taken out of the tube and then ground to a fine powder with the help of a gate mortar and pestle. Thus mixtures of varying proportion were

prepared and studied under polarizing microscope method.

STUDY UNDER POLARIZING MICROSCOPE

Main details of this procedure are outlined in Part 1. More relevant details dealing with the study of binary mixture are given below. The prepared slide containing a little portion of binary mixture was observed under polarizing microscope. The whole of the mixed melt exhibits mesomorphism lending support to its homogeneous nature.

The transition temperatures are studied several times to ensure that none of transitions remain unnoticed.

TABLE - 23

System No.1

Components : (A) -n.Butyl p-(p'-methoxycinnamoyloxy) Benzoate

(B) -n.Butyl p-(p',ethoxycinnamoyloxy) benzoate

Mole % of n-Butyl P (p'-methoxycinnamoyloxy) benzoate	Transition Temperatures (°C) S	N	I.
100.00	-	-	79.0
90.35	-	(66.0)	75.0
80.61	-	(70.0)	73.0
70.80	-	(69.0)	70.0
60.92	-	68.0	69.0
50.96	-	67.0	76.5
40.93	(64.5)	65.0	78.0
32.94	(65.5)	66.0	83.0
20.62	68.0	72.0	85.0
10.35	71.0	75.0	89.0
00.00	76.0	85.0	92.5

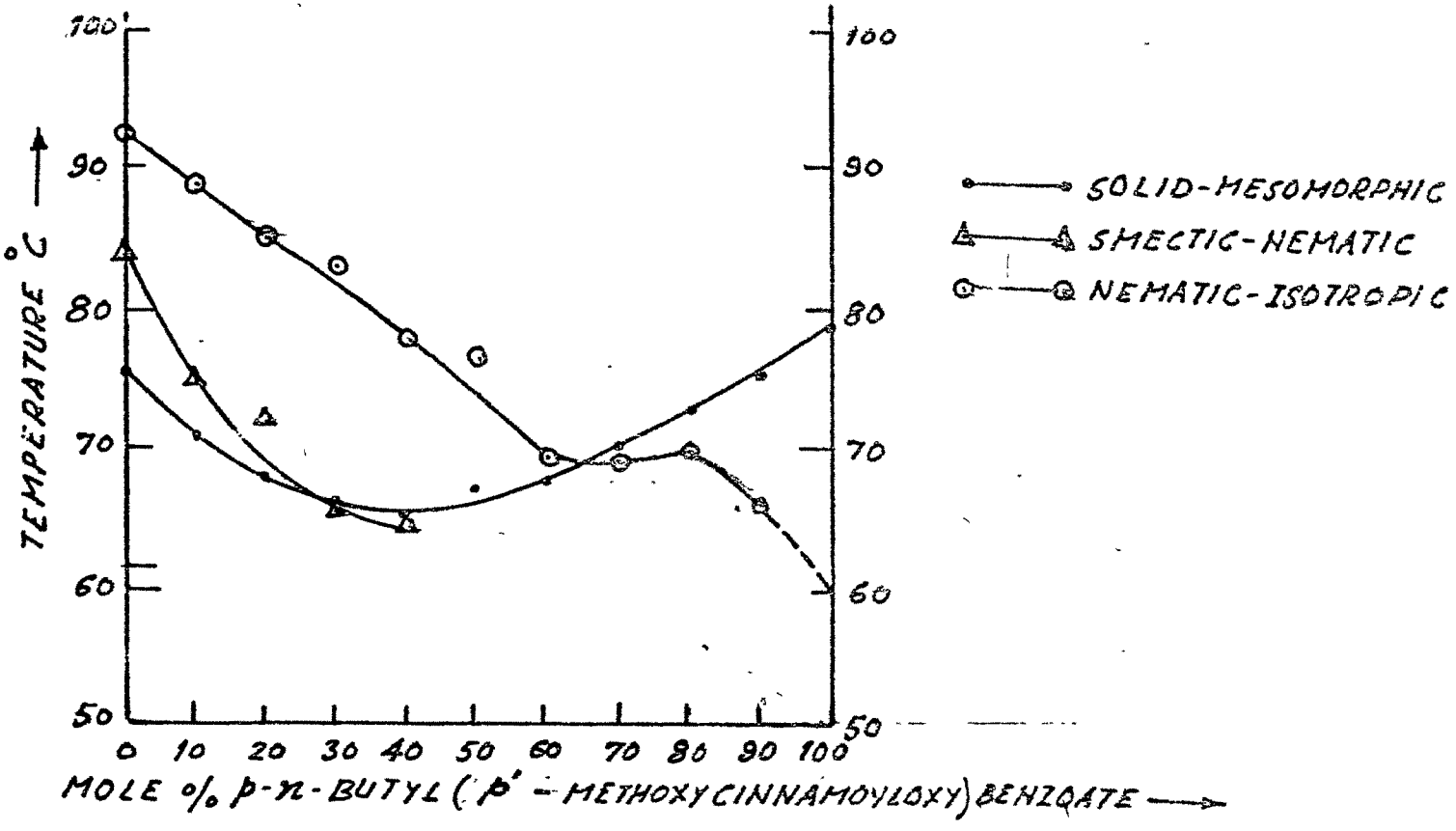
Sintering point = 40°C

Values in parenthesis indicate monotropy.

SYSTEM : 1

FIG. 17

p-n-BUTYL(p'-METHOXYCINNAMOYLOXY)BENZOATE : p-n-BUTYL(p'-ETHOXYCINNAMOYLOXY)BENZOATE



SYSTEM : 2.

FIG. 18

p-n-AMYL (p'-n'-METHOXYCINNAMOYLOXY) BENZOATE : p-n-BUTYL (p'-n'-METHOXYCINNAMOYLOXY) BENZOATE

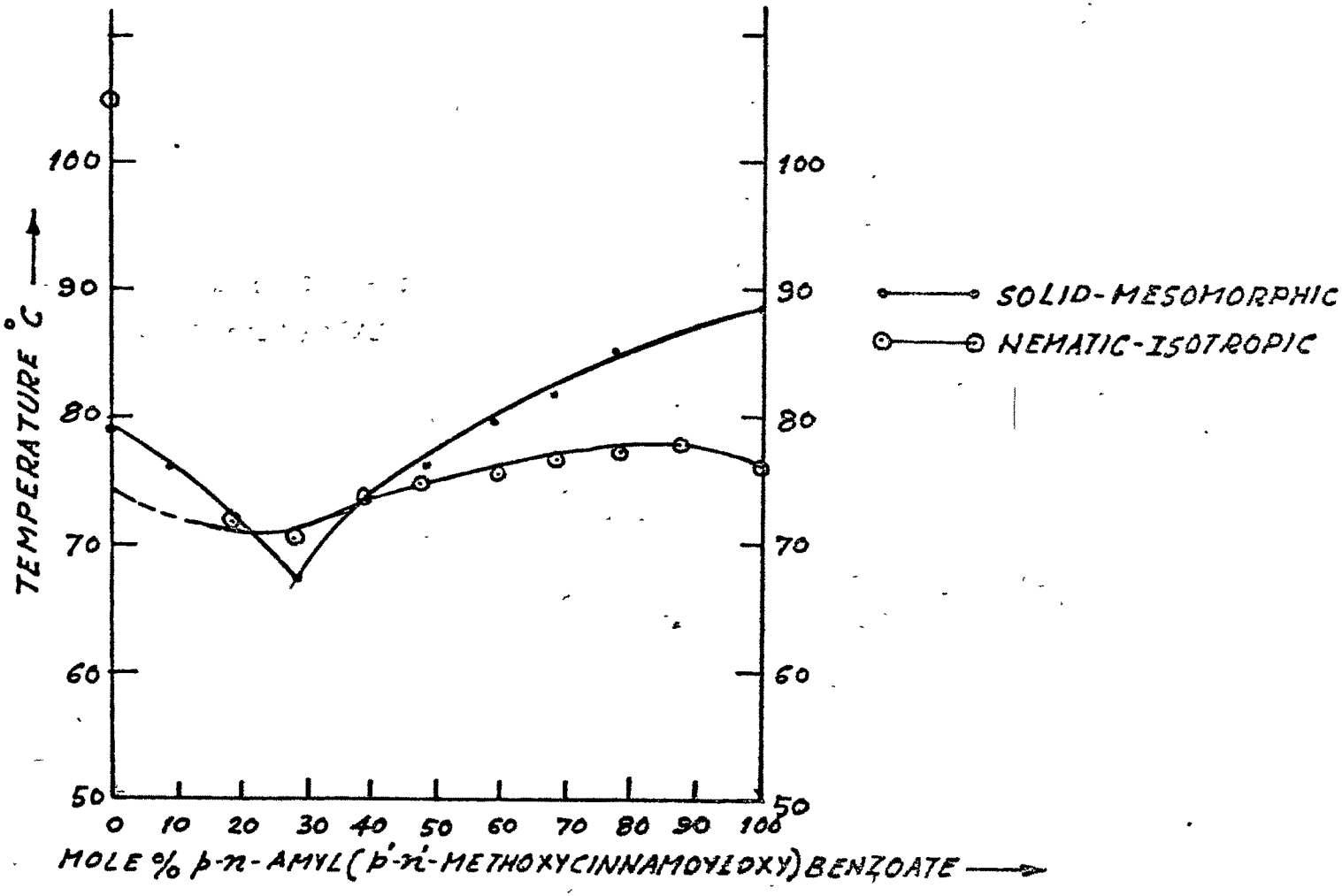


TABLE - 24

System No.2

Components : (A) -n-Amyl ρ-(p'.methoxycinnamoyloxy) benzoate

(B) -n-Butyl ρ-(p'.methoxycinnomoyloxy) benzoate

Mole % of n-Amyl p'-methoxycinna-moyloxy) benzoate	Transition Temperatures (°C) S	N	I
100.00	-	(76.0)	88.0
88.62	-	(77.5)	86.5
79.37	-	(77.0)	85.0
69.17	-	(76.0)	81.5
60.07	-	(75.0)	79.5
49.03	-	(74.5)	76.0
40.06	-	(73.5)	74.0
29.19	-	67.0	70.0
19.38	-	(71.5)	72.0
9.65	-	(72.0)	76.0
00.00	-	-	79.0

Sintering point = 67°C

Values in parenthesis indicate monotropy.

SYSTEM: 3

FIG. 19

p-n-AMYL (p'-n'-ETHOXYCINNAMOYLOXY) BENZOATE: p-n-BUTYL (p'-METHOXYCINNAMOYLOXY) BENZOATE

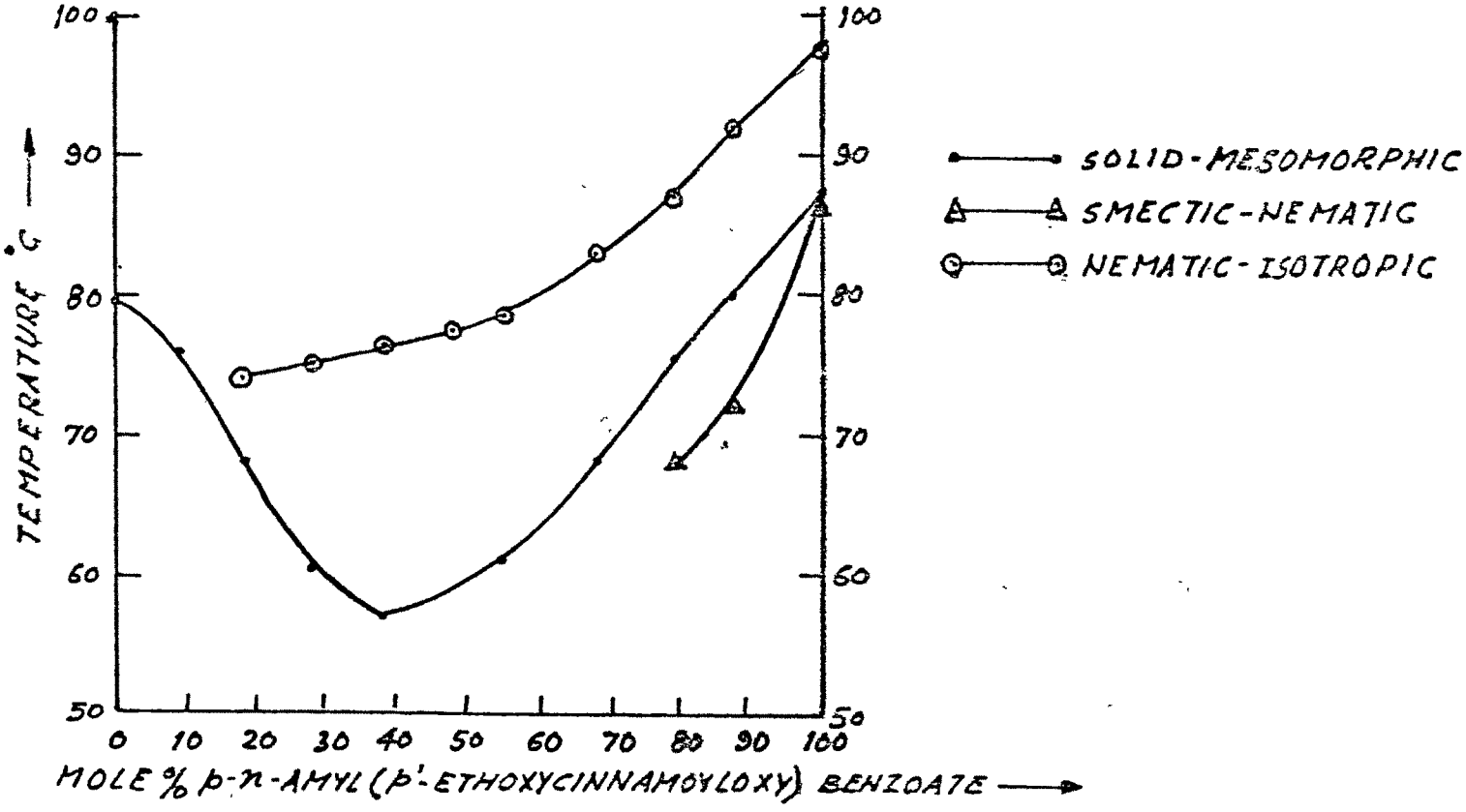


TABLE - 25

System No. 3

Components : (A) n.Amyl P- (p'-ethoxycinnamoyloxy) Benzoate

(B) n.Butyl P-(p'-methoxycinnamoyloxy) Benzoate

Mole % of n-Amyl (p'-ethoxycinnamoyloxy) benzoate	Transition Temperatures (°C) S.	N.	I.
100.00	(86.0)	87.0	97.5
88.89	(72.0)	80.0	92.0
79.19	(68.0)	76.0	87.0
68.37	-	68.0	83.0
55.12	-	61.0	78.5
48.09	-	59.0	77.5
38.18	-	57.0	76.5
28.42	-	60.5	75.0
18.80	-	68.0	74.0
9.33	-	-	76.0
00.00	-	-	79.0

Sintering point = 57°C

Values in parentheses indicate monotropy.

SYSTEM : 4.

FIG. 20

p-n- AMYL (p'-n'-METHOXYCINNAMOYLOXY) BENZOATE: p-n- AMYL (p'-n'-ETHOXYCINNAMOYLOXY) BENZOATE

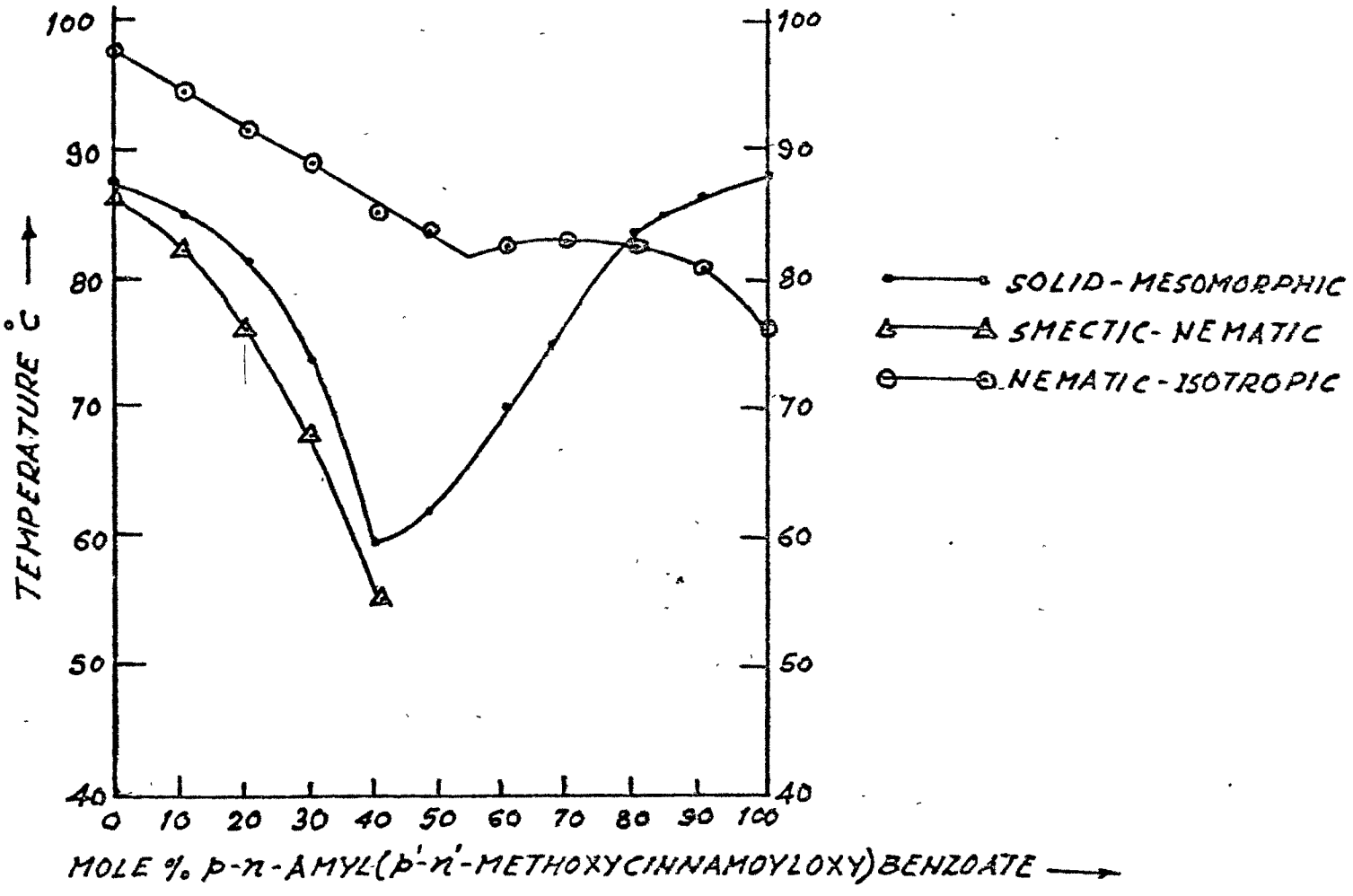


TABLE - 26

System No. 4

Components : (A) n-Amyl -P-(p'Methoxycinnamoyloxy) Benzoate

(B) n-Amyl-p-(p' Ethoxycinnamoyloxy) Benzoate

Mole % of n-Amyl P-(p'-methoxy-cinnamoyloxy)benzoate	Transition Temperatures (°C) S.	N.	I.
100.00	-	(76.0)	88.0
90.33	-	(81.0)	86.0
80.59	-	(82.5)	83.5
68.80	-	75.0	82.5
60.89	-	70.5	81.5
48.93	-	62.0	83.5
40.89	(55.0)	59.5	85.0
30.78	(65.0)	74.0	89.0
20.60	(76.0)	81.5	91.5
10.34	(82.0)	85.0	94.5
00.00	(86.0)	87.0	97.5

Sintering point = 59°C

Values in parentheses indicate monotropy.

SYSTEM: 5.

FIG. 21

p-n-AMYL (p'-n'-ETHOXYCINNAMOYLOXY)BENZOATE: p-n-AMYL(p'-n'-PROPOXYCINNAMOYLOXY) BENZOATE

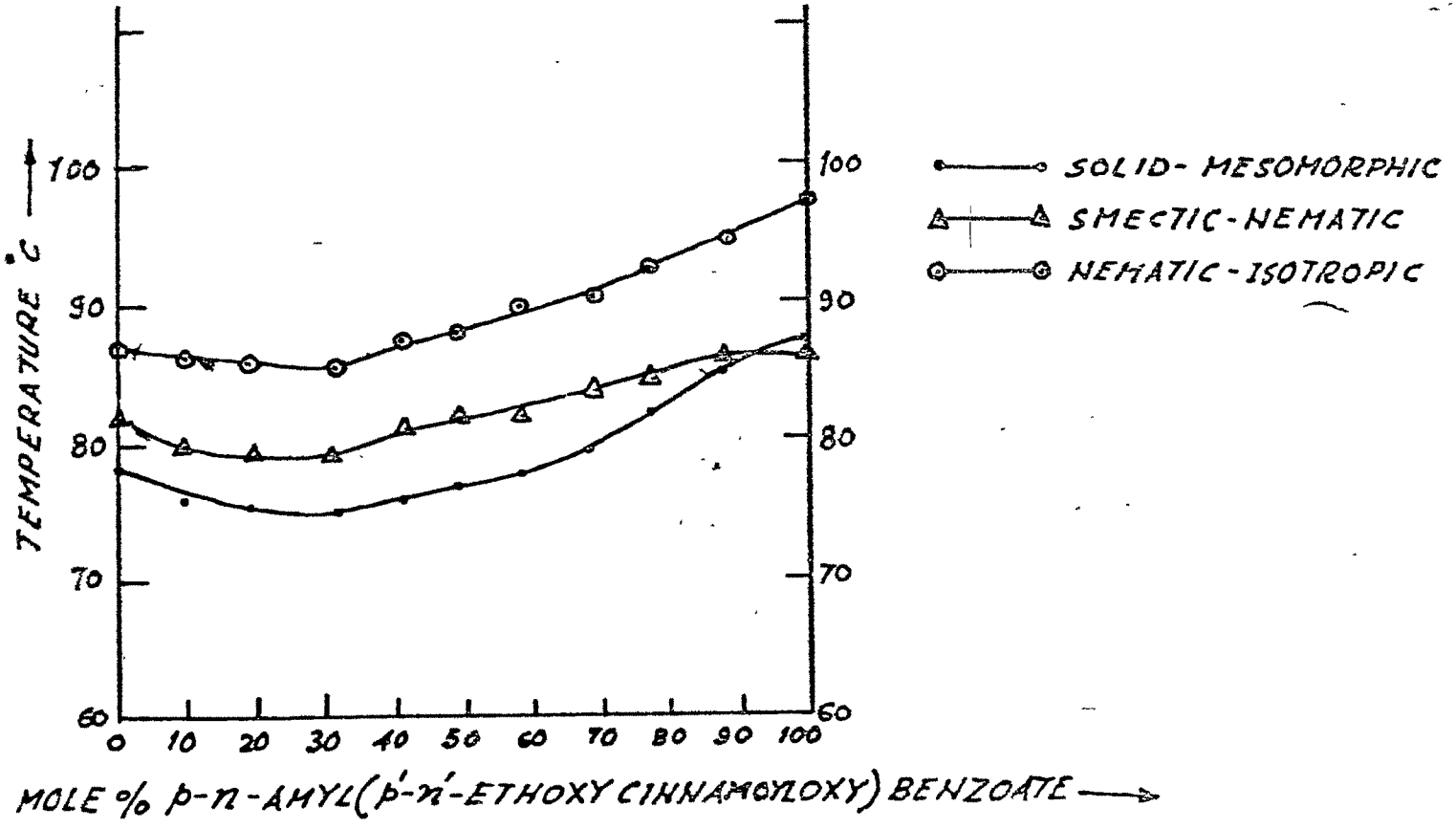


TABLE - 27

System No. 5

Components : (A) n-Amyl P-(p'.Ethoxycinnamoyloxy) benzoate

(B) n-Amyl P-(p'.n. Propoxycinnamoyloxy) benzoate

Mole % of n-Amyl P-(P'-ethoxycinnamoyloxy) benzoate	Transition temperatures (°C) S.	N.	I.
100.00	(86.0)	87.0	97.5
87.61	85.0	86.0	94.5
77.37	82.0	84.5	92.5
69.23	79.5	83.5	90.5
58.13	78.0	82.0	90.0
49.10	77.0	82.0	88.0
41.12	76.0	81.0	87.5
31.22	75.0	79.0	85.5
19.43	75.5	79.5	86.0
9.64	76.0	80.0	86.5
00.00	78.0	82.0	87.0

Sintering point = 75°C

Values in parentheses indicate monotropy.

FIG. 22

SYSTEM: 6.

p-n-AMYL(p'-n'-ETHOXYCINNAMOYLOXY)BENZOATE: p-n-AMYL(p'-n'-BUTOXYCINNAMOYLOXY)BENZOATE

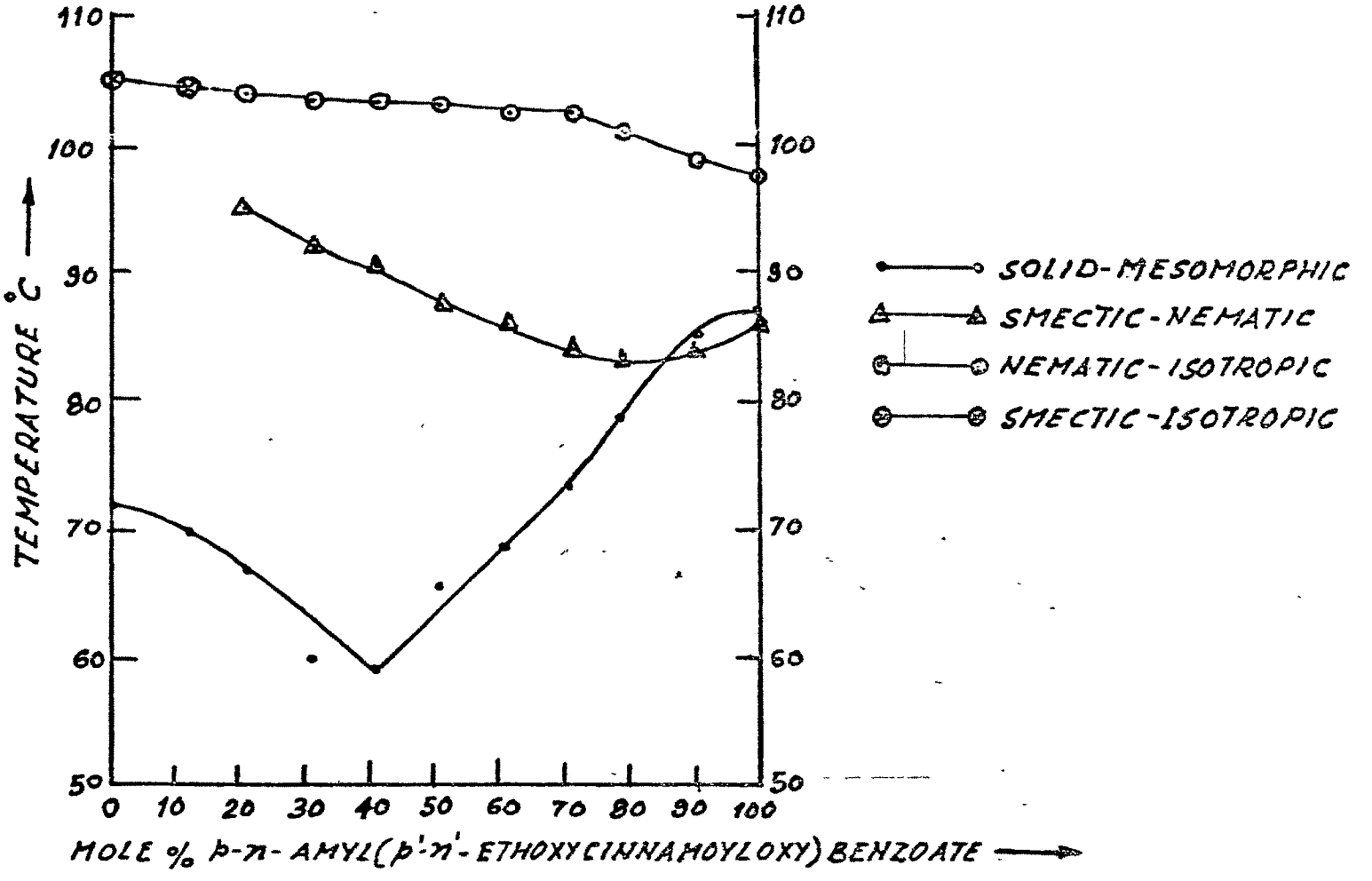


TABLE - 28

System No. 6

Components : (A) n-Amyl-P-(p'-ethoxycinnamoyloxy) benzoate

(B) n-Amyl P-(p'-n'-butoxycinnamoyloxy) benzoate

Mole % of n-Amyl P-(p'-ethoxy-cinnamoyloxy benzoate	Transition Temperatures S.	N.	I.
100.00	(86)	87°0	97.6
90.65	(84)	85°0	98.5
79.17	78.5	83°0	101.0
71.47	73.5	84°0	102.5
61.69	68.5	86°0	102.5
51.76	65.5	87.5	103.0
41.71	59°0	90.5	103.5
31.50	60°0	92°0	103.5
21.14	67°0	95°0	104°0
12.72	70°0	-	104.5
00.00	72°0	-	105°0

Sintering point 58.5°C

Values in parenthesis indicate monotropy.

SYSTEM: 7.

FIG. 23

p-n-AMYL (p'-n'-ETHOXYCINNAMOYLOXY) BENZOATE: p-n-AMYL (p'-n'-AMYLOXYCINNAMOYLOXY) BENZOATE

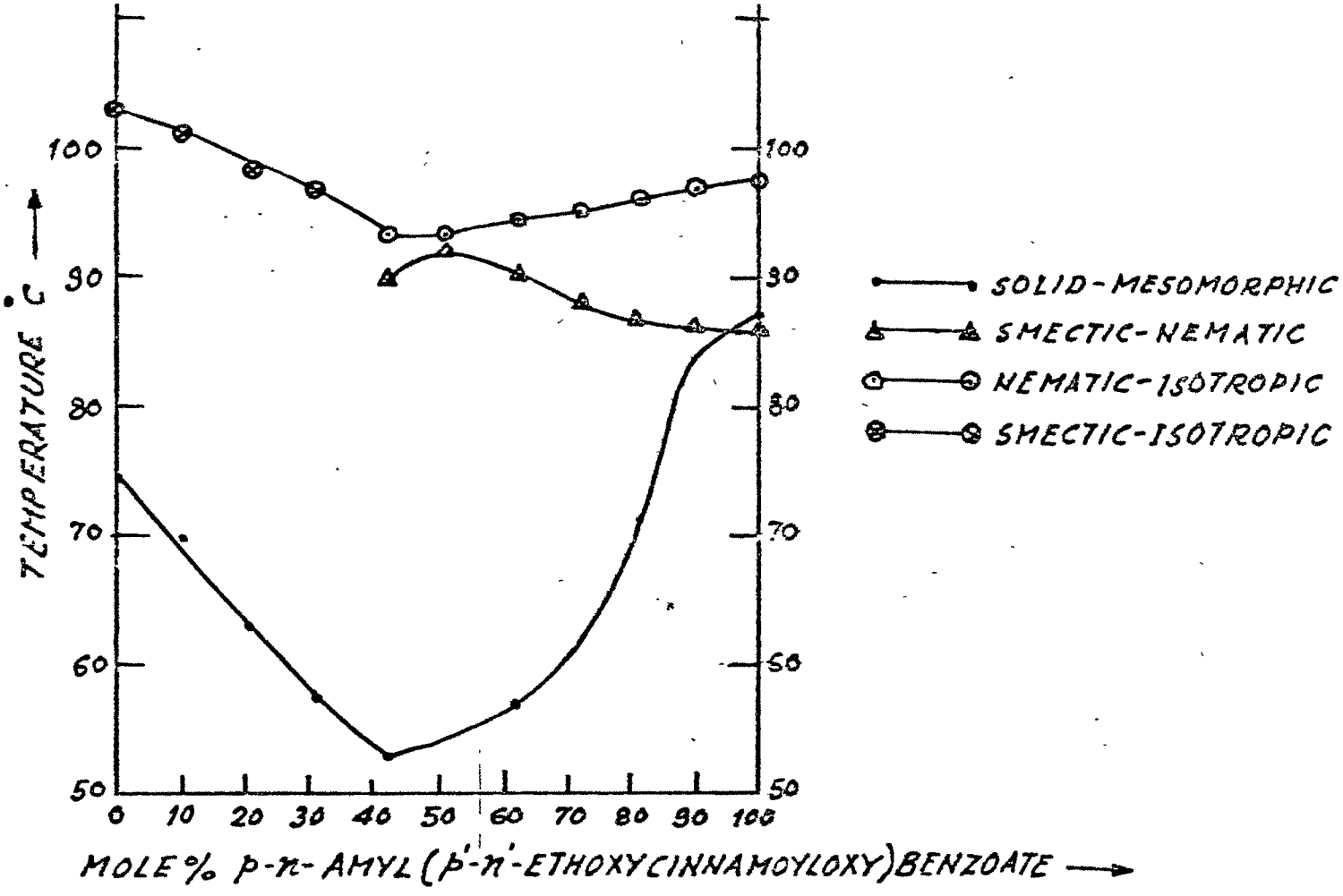


TABLE - 29

System No. 7

Components : (A) n-Amyl P-(p'-ethoxycinnamoyloxy) benzoate

(B) n-Amyl P-(p'-n-amyloxycinnamoyloxy) benzoate

Mole % of n-Amyl P-(p'-ethoxy-cinnamoyloxy) benzoate	Transition Temperatures (°C) S.	N.	I.
100.00	(86.0)	87.0	97.5
90 .90	84.0	86.0	97.0
81.61	70.0	86.5	96.0
72.14	61.5	88.0	95.0
62.47	57.0	90.0	94.0
51.60	56.0	92.0	93.5
42.50	53.0	90.0	93.5
32.23	58.0	-	97.0
21.72	63.5	-	98.5
10.97	70.0	-	101.0
00.00	75.0	-	103.0

Sintering point = 53°C

Value in parenthesis indicates monotropy.

SYSTEM : 8

FIG. 24

p-n-BUTYL(p'-n'-BUTYLOXYCINNAMOYLOXY) BENZOATE : p-n-BUTYL(p'-n'-AMYLOXYCINNAMOYLOXY) BENZOATE

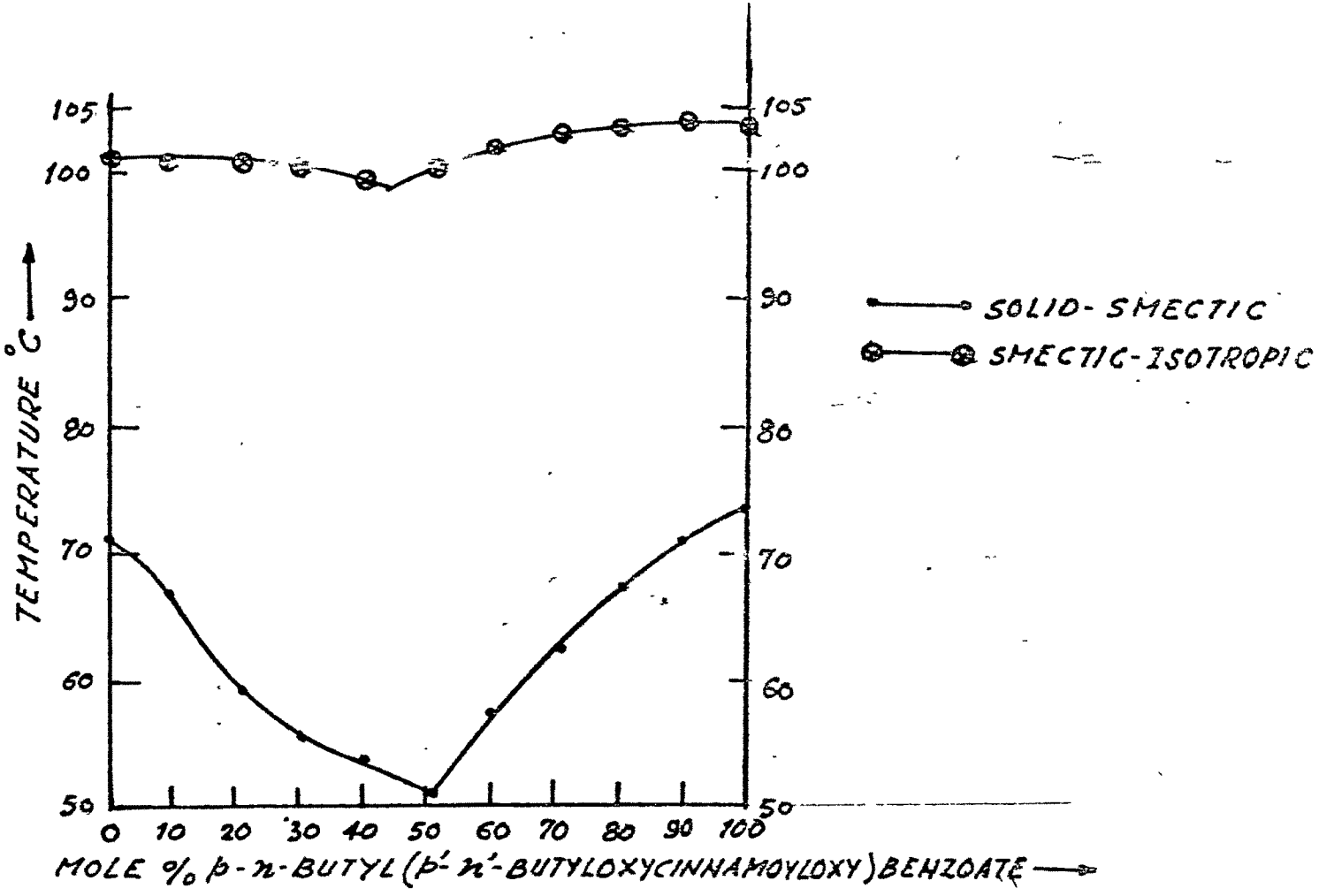


TABLE - 30

System No. 8

Components : (A) n.Butyl-P-(p'-n-'butoxycinnamoyloxy) benzoate

(B) n-Butyl-P-(p'-n'-amyloxycinnamoyloxy) benzoate

Mole % of n-Butyl(p'-n'-butoxycinna-nitkixy) benzoate	Transition Temperatures (°C) S.	N.	I.
100.00	73.5	-	103.5
90.30	71.0	-	103.5
80.55	67.5	-	103.5
70.72	62.5	-	103.0
60.30	57.5	-	102.0
50.80	51.0	-	100.5
40.10	54.0	-	99.5
30.00	56.0	-	100.5
20.80	59.5	-	100.5
9.85	67.0	-	101.0
00.00	71.0	-	101.0

Sintering point = 51.5°C

SYSTEM: 9.

FIG. 25

p-n-BUTYL(p'-n'-BUTOXYCINNAMOYLOXY)BENZOATE: p-n-AMYL(p'-n'-BUTOXYCINNAMOYLOXY)BENZOATE

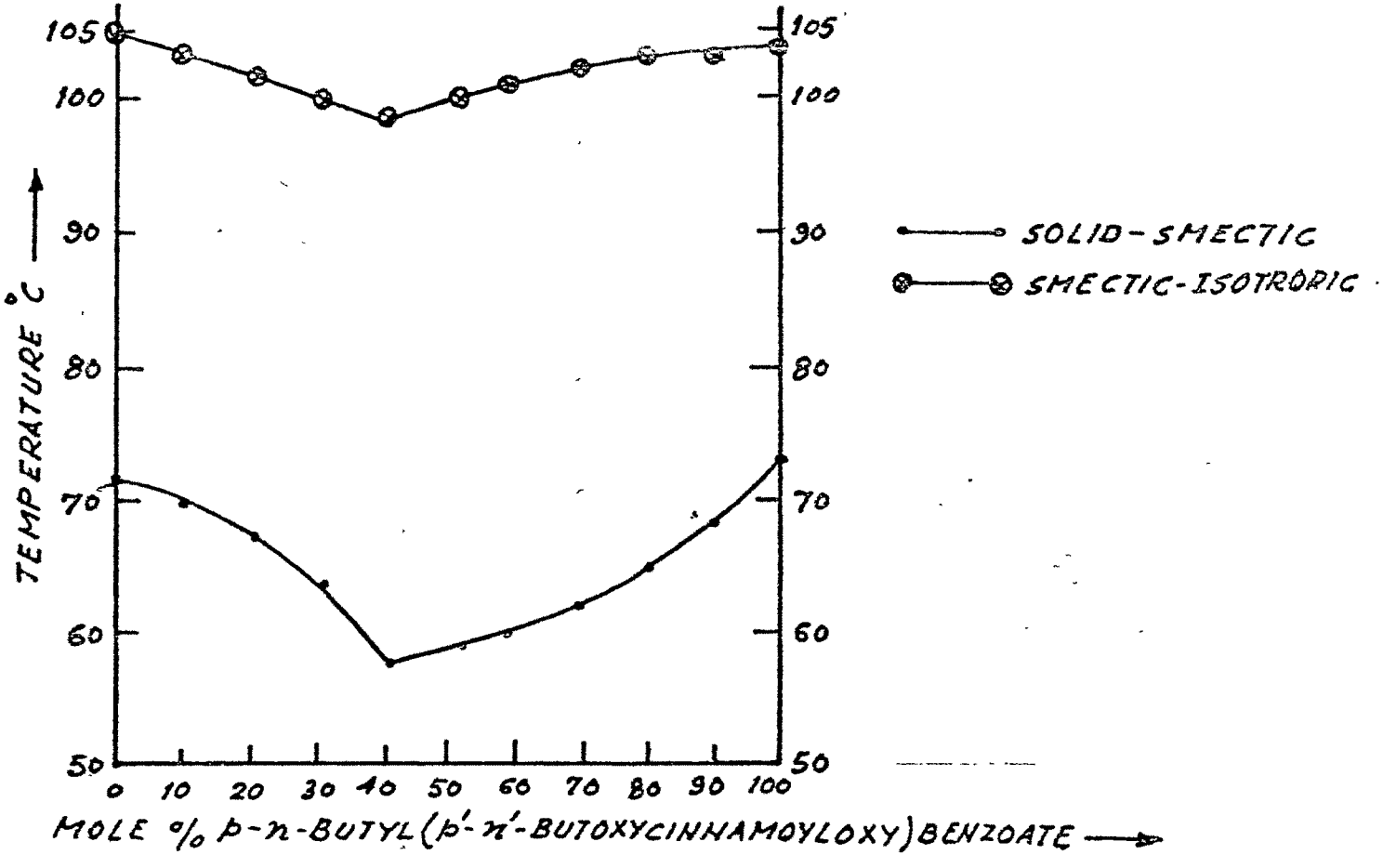


TABLE - 31

System No. 9

Components : (A) n.Butyl P-(p'.n' butoxycinnamoyloxy) benzoate

(B) n.Amyl P-(p'.n' butoxycinnamoyloxy) benzoate

Mole % of n-Butyl-P'-(p'-butoxycinnamoyloxy benzoate	Transition Temperature (°C) S.	N.	I.
100.00	73.5	-	103.5
89.73	68.0	-	103.0
79.80	65.0	-	103.0
69.74	62.0	-	102.0
58.84	60.0	-	101.0
51.86	59.0	-	100.0
40.83	58.0	-	99.0
30.73	64.0	-	100.0
20.56	67.5	-	102.0
10.31	70.0	-	103.5
00.00	72.0	-	105.0

Sintering point = 58°

SYSTEM : 10

FIG.26

p-n-AMYL (p'-n'-BUTOXYCINNAMOYLOXY) BENZOATE : p-n-AMYL (p'-n'-AMYLOXYCINNAMOYLOXY) BENZOATE

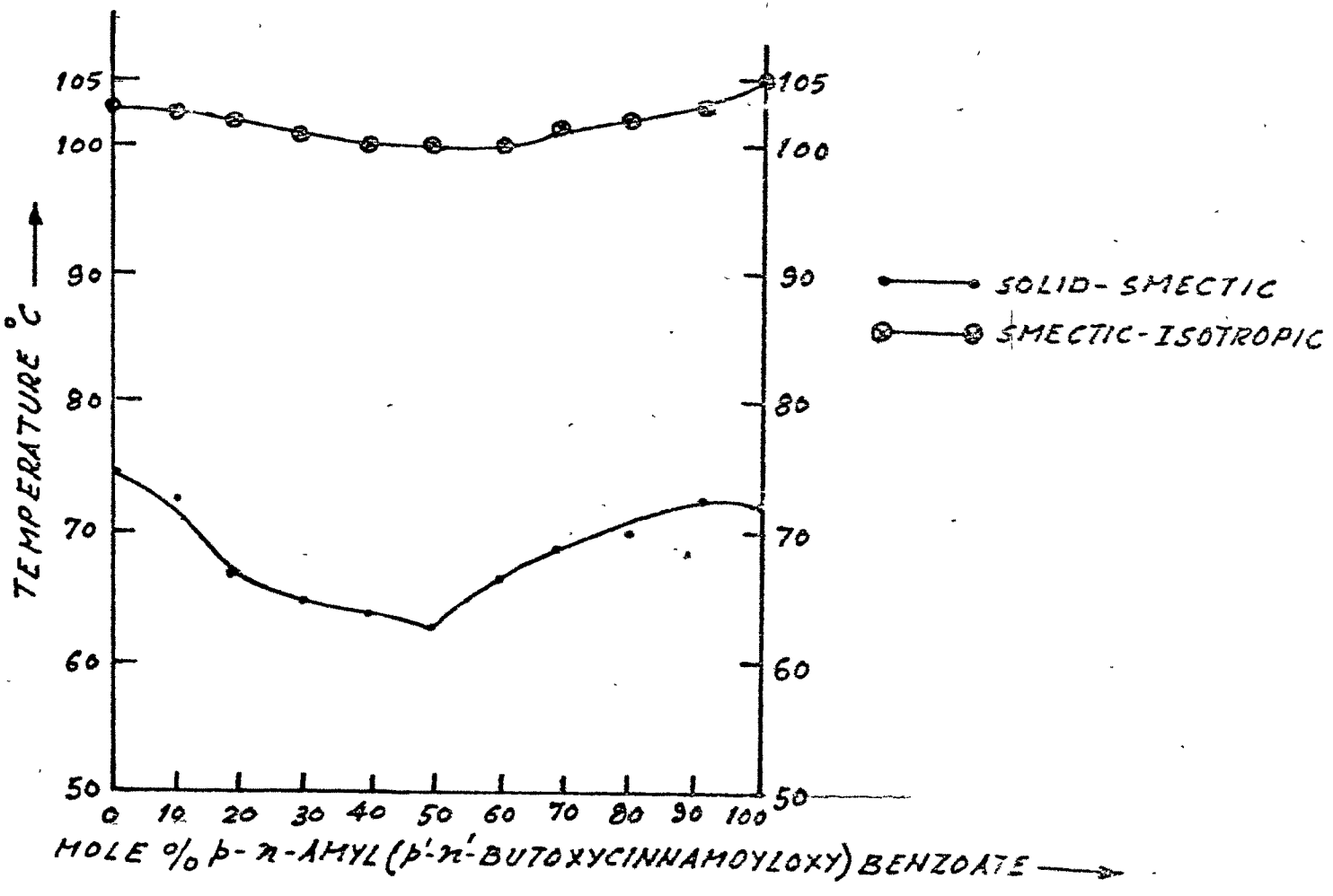


TABLE - 32

System No.10

Components : (A) n-Amyl-P-(p'-n'-butoxycinnamoyloxy) benzoate

(B) n-Amyl-P-(p'-n'-amyloxycinnamoyloxy) benzoate

Mol % of P-n-Amyl(p'-n'-butomycinnamoyloxy) benzoate	Transition Temperature (°C) S.	N.	I.
100.00	72.0	-	105.0
89.25	73.0	-	103.0
80.53	70.0	-	102.0
70.70	69.0	-	101.5
59.80	66.5	-	100.0
50.84	63.5	-	100.0
40.80	64.0	-	100.0
30.70	65.0	-	101.0
20.56	67.0	-	102.0
10.30	73.0	-	103.0
00.00	75.0	-	103.0

Sintering point = 63°C

SYSTEM: 11.

FIG. 27

p-n-BUTYL(p'-n'-AMYLOXYCINNAMOYLOXY)BENZOATE : p-n-AMYL(p'-n'-AMYLOXYCINNAMOYLOXY)BENZOATE.

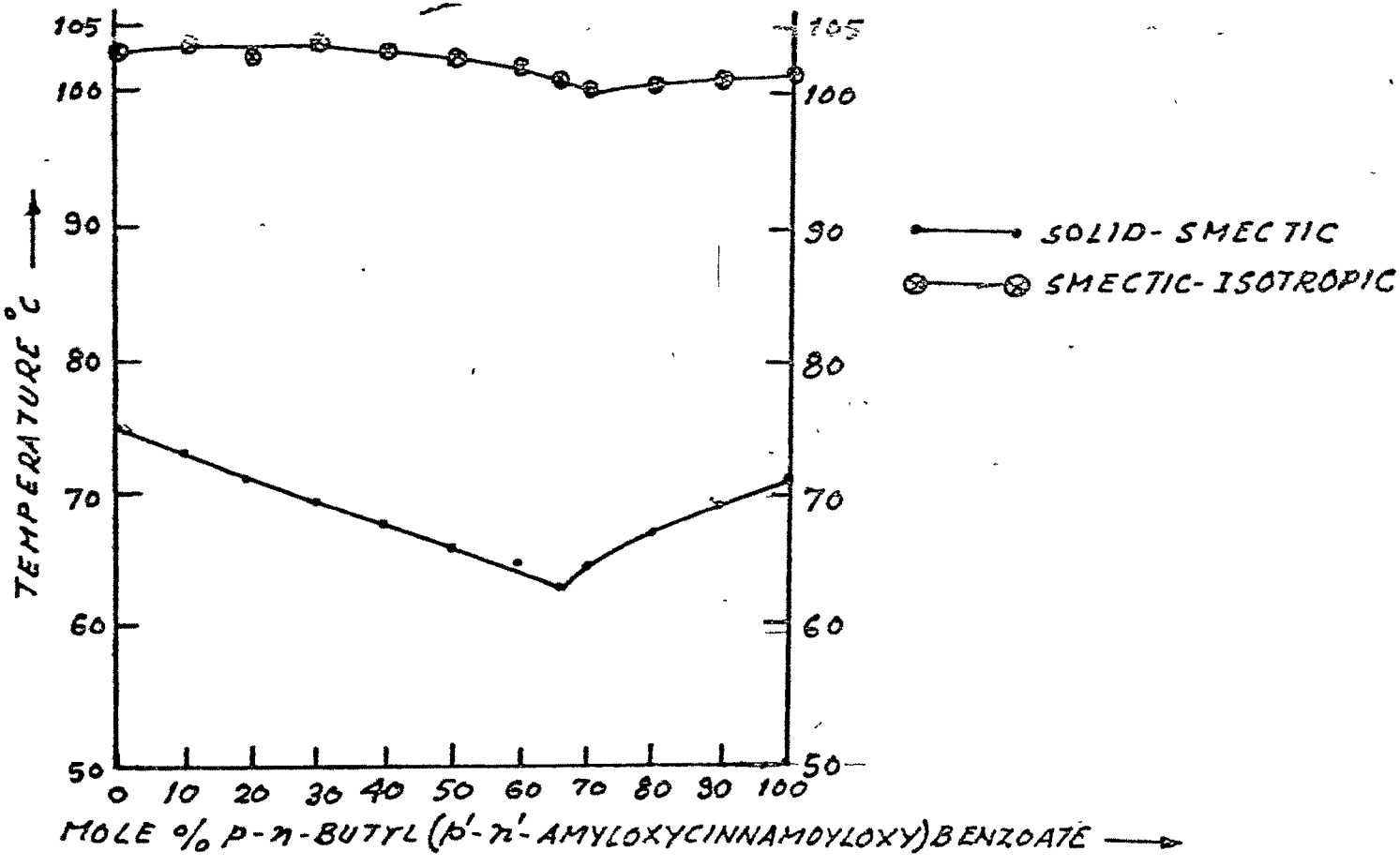


TABLE - 33

System No. 11

Components : (A) n-Butyl P-(p'-n'-amyloxycinnamoyloxy) Benzoate

(B) P-n-Amyl-P-(p'-n'-amyloxycinnamoyloxy) Benzoate

Mole % of n-Butyl-P(p'-n'-amyloxycinnamoyloxy benzoate	Transition Temperature (°C) S.	N.	I.
100.00	71.0	-	101.0
90.25	69.5	-	101.0
80.53	67.5	-	100.5
70.70	65.0	-	100.0
59.80	65.0	-	102.0
50.84	66.0	-	102.5
40.80	68.0	-	103.0
30.70	69.5	-	103.5
21.56	71.0	-	102.5
10.30	73.0	-	103.5
00.00	75.0	-	103.0

Sintering point = 65°C

FIG. 28.

SYSTEM: 12.

p-n-BUTYL(p'-ETHOXYCINNAMOYLOXY) BENZOATE : p-n-BUTYL(p'-n'-PROPOXYCINNAMOYLOXY)BENZOATE

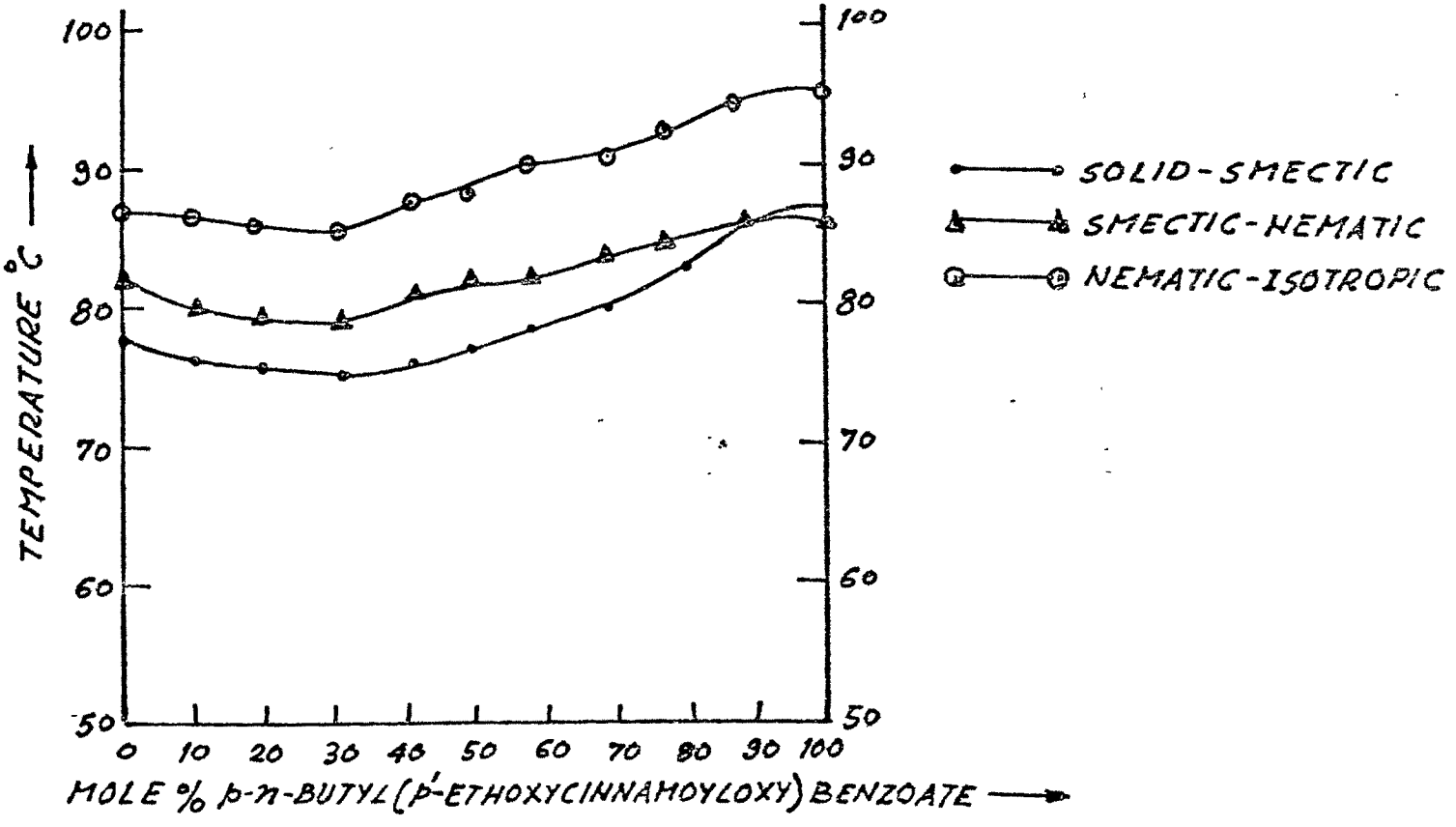


TABLE - 34

System No. 12

Components : (A) n-Butyl-P-(p'-Ethoxycinnamoyloxy) Benzoate

(B) n-Butyl-P-(p'-n'-propoxycinnamoyloxy) Benzoate

Mole % of n-Butyl-P.(p'-ethoxycinnamoyloxy) benzoate	Transition temperatures (°C) S	N	I
100.00	76.0	85.0	92.5
90.33	72.0	83.5	92.0
80.59	70.0	84.0	91.0
70.77	66.0	84.5	90.0
60.89	65.0	85.0	89.5
49.93	60.0	84.5	88.0
40.89	62.0	84.0	86.5
30.78	65.0	83.0	86.0
20.60	67.0	82.5	85.0
10.34	69.5	82.0	83.0
00.00	74.0	83.0	84.0

Sintering point = 60°C

Values in parenthesis indicate monotropy.

SYSTEM: 13

FIG. 29

p-n-BUTYL(p'-n'-ETHOXYCINNAMOYLOXY)BENZOATE: p-n-BUTYL(p'-n'-BUTOXYCINNAMOYLOXY)BENZOATE

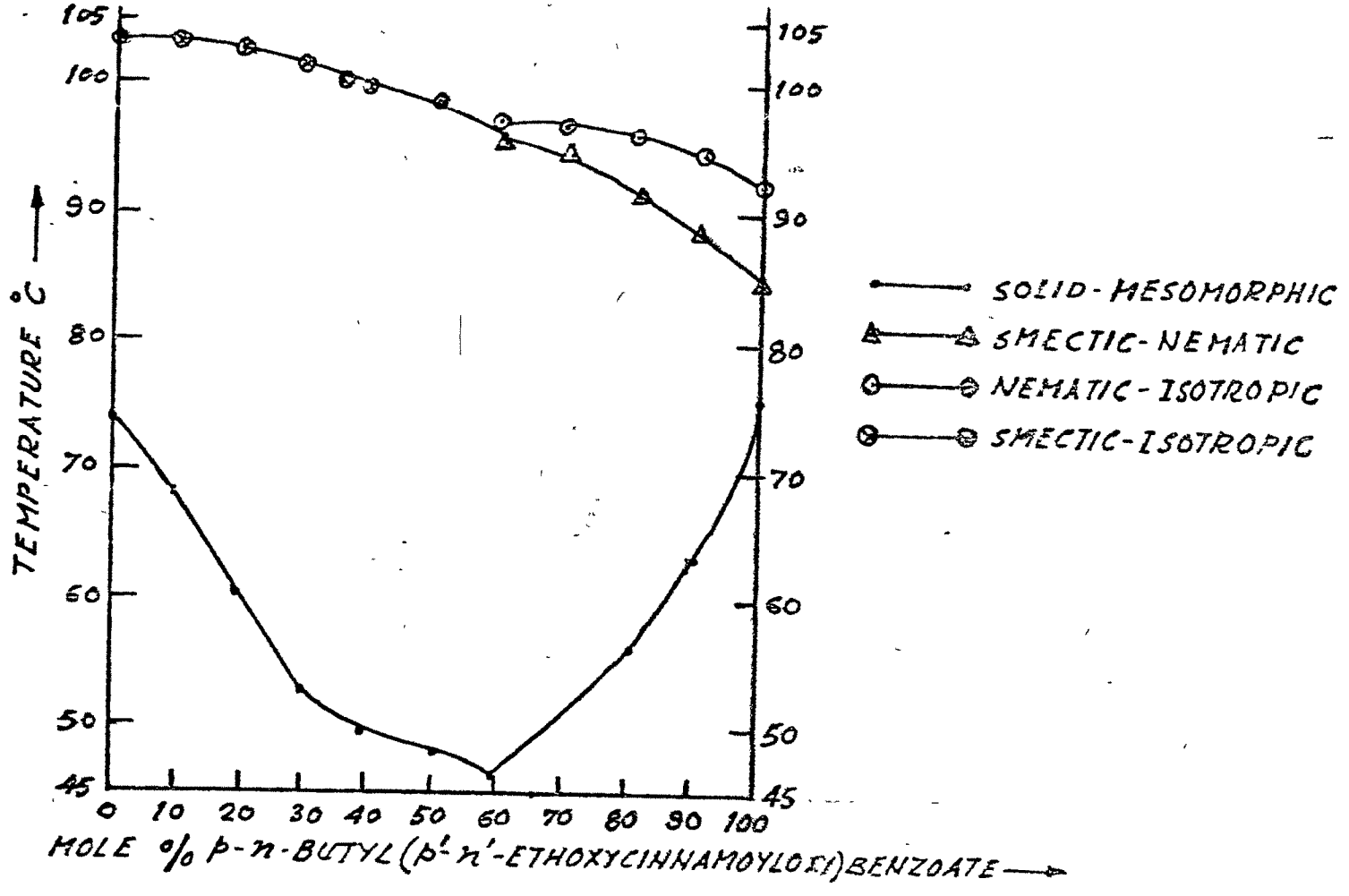


TABLE - 35

System No. 13

Components : (A) n.Butyl-P-(p'-ethoxycinnamoyloxy) benzoate

(B) n-Butyl-P-(p'-n'-butoxycinnamoyloxy) benzoate

Mole % of n-Butyl P(p'-ethoxycinnamoyloxy) benzoate	Transition Temperatures (in °C) S.	N.	I.
100.00	76.0	85.0	92.5
90.62	63.0	89.0	95.0
80.14	56.0	91.5	96.5
71.51	51.0	95.0	97.0
61.72	46.5	96.5	97.5
50.18	48.5	-	99.0
41.77	50.0	-	100.0
31.56	53.0	-	101.5
21.19	60.0	-	103.0
10.97	68.5	-	103.5

Sintering point = 45.5°C

SYSTEM : 14.

FIG-30

p-n-BUTYL(p'-n'-ETHOXYCINNAMOYLOXY) BENZOATE : p-n-BUTYL(p'-n'-AMYLOXYCINNAMOYLOXY) BENZOATE

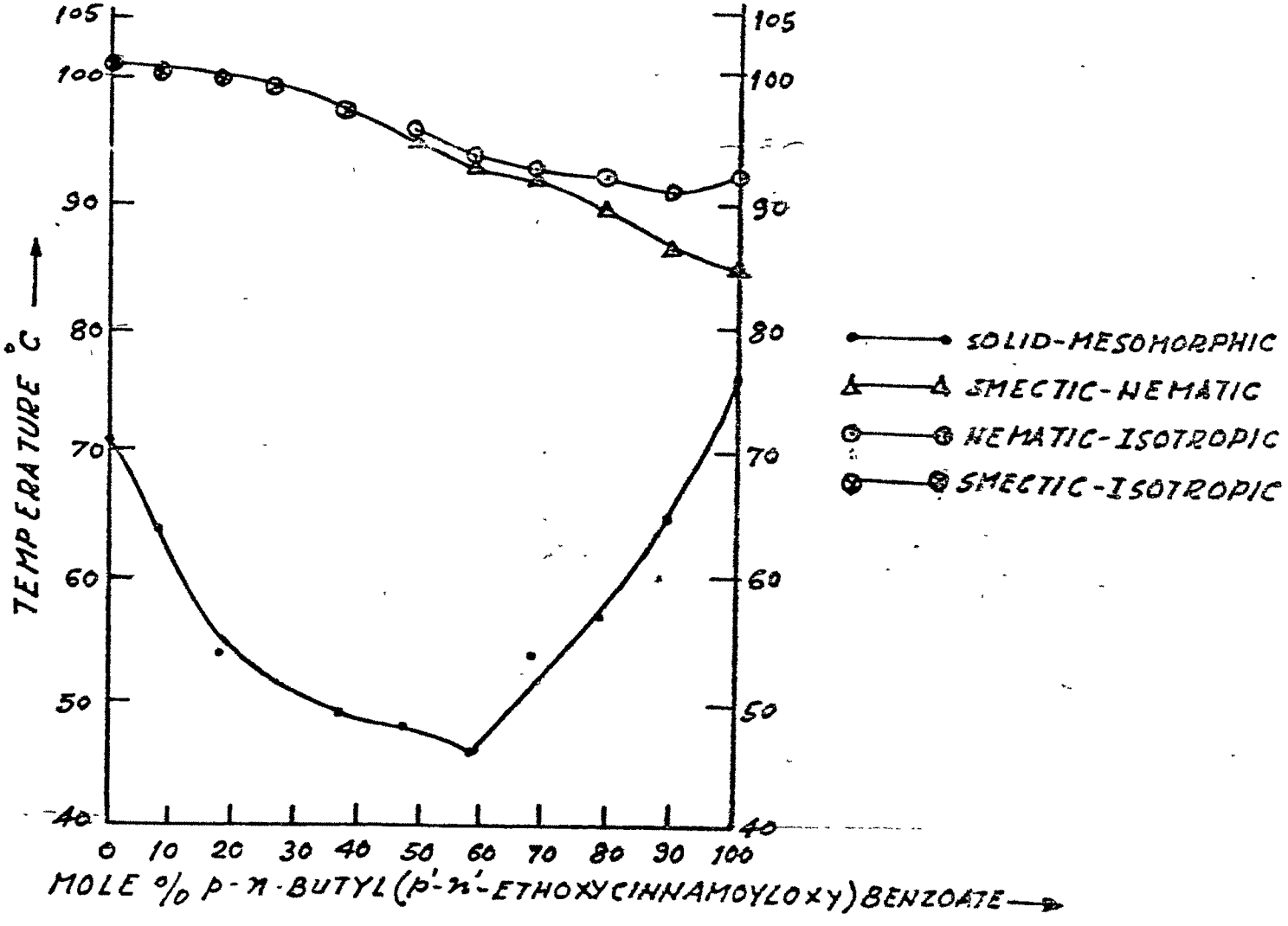


TABLE - 36

System No. 14

Components : (A) n-Butyl-P-(p' ethoxycinnamoyloxy) benzoate

(B) n-Butyl-P-(p'-n'-amyloxycinnamoyloxy) benzoate

Mole % of n-Butyl P-(p'-ethoxycinnamoyloxy) benzoate	Transition Temperature (°C) S.	N.	I.
100.00	76.0	85.0	92.5
90.93	65.0	86.5	91.0
80.67	57.0	90.0	92.5
72.21	54.0	92.0	93.0
62.56	46.0	93.0	94.0
51.69	48.0	95.0	96.0
42.87	49.0	-	97.5
34.31	52.0	-	99.5
21.78	59.0	-	100.0
12.01	64.0	-	100.5
00.00	71.0	-	101.0

Sintering point = 46°C

SYSTEM : 15.

FIG.31

p-n-BUTYL(p'-n'-ETHOXYCINNAMOYLOXY)BENZOATE: p-n-AMYL(p'-n'-ETHOXYCINNAMOYLOXY) BENZOATE

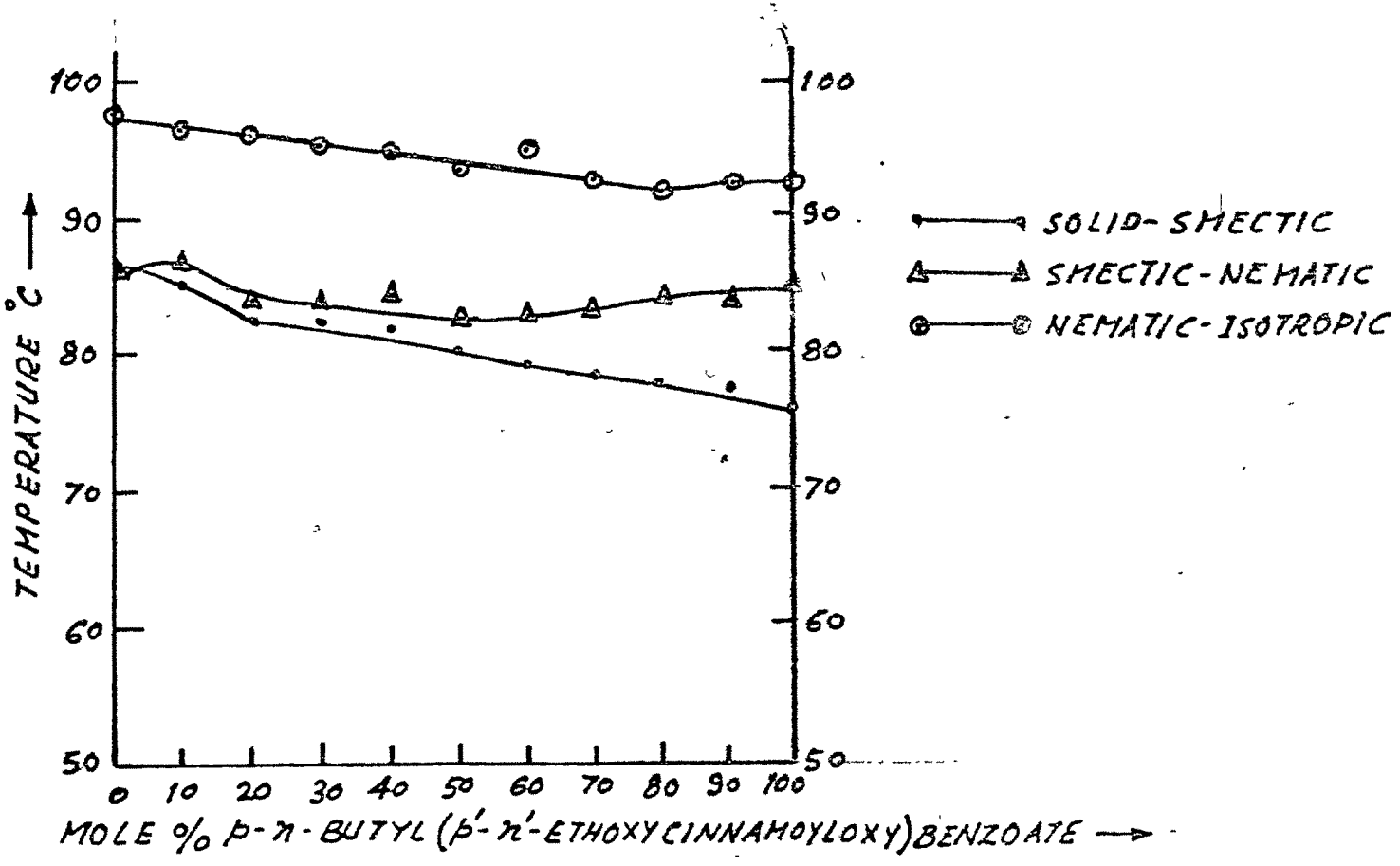


TABLE - 37

System No. 15

Comoonents : (A) n-Butyl-P-(p' ethoxycinnamoyloxy) benzoate

(B) n-Amyl-P-(p' ethoxycinnamoyloxy) benzoate

Mole % of n-Butyl-P-(p'-ethoxycinnamoyloxy) benzoate	Transition Temperatures (in °C) S.	N.	I.
100.00	76.0	85.0	92.5
90.33	77.5	84.0	92.5
80.59	78.0	84.5	92.0
70.77	78.5	83.0	93.0
60.89	79.5	83.0	95.0
50.93	80.0	82.5	93.5
40.89	82.0	84.5	95.0
30.78	82.5	84.0	95.5
20.60	82.5	84.0	96.0
10.34	85.5	86.5	96.5
00.00	(86)	87	97.5

Values in parenthesis indicate monotropy.

SYSTEM : 16.

FIG. 32

p-n-BUTYL(p'-n'-PROPOXYCINNAMOYLOXY)BENZOATE : p-n-AMYL(p'-n'-PROPOXYCINNAMOYLOXY) BENZOATE

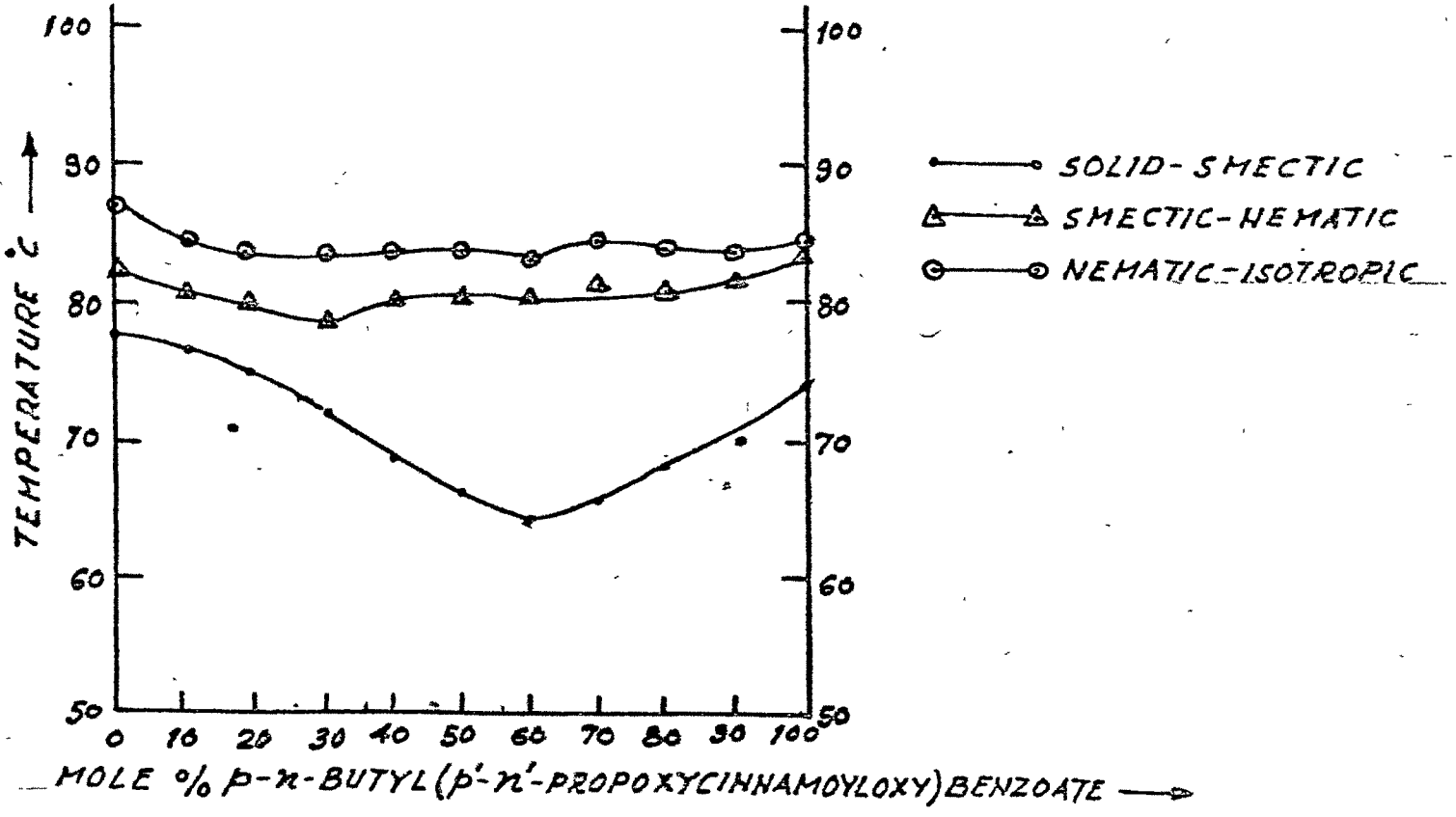


TABLE - 38

System No. 16

Components : (A) n-Butyl-P-(p'-n'-propoxycinnamoyloxy) benzoate

(B) n.Amyl-P-(p'-n'-propoxycinnamoyloxy) benzoate

Mole % of n-Butyl-P(p'-n'-propoxycinnamoyloxy benzoate	Transition Temperatures S.	N.	I.
100.00	74.0	83.0	84.0
90.31	70.0	81.5	83.5
80.56	68.0	80.5	83.5
70.71	65.5	81.0	84.0
60.86	64.0	80.0	83.0
50.89	66.0	80.0	83.5
40.87	68.5	80.0	83.5
30.76	72.0	78.5	83.5
21.65	75.0	80.0	83.5
11.45	76.5	80.5	84.5
00.00	78.0	82.0	87.0

Sintering point = 64°C

SYSTEM: 17.

FIG. 33

p-n-AMYL (p'-n'-PROPOXYCINNAMOYLOXY) BENZOATE : p-n-AMYL (p'-n'-BUTOXYCINNAMOYLOXY) BENZOATE

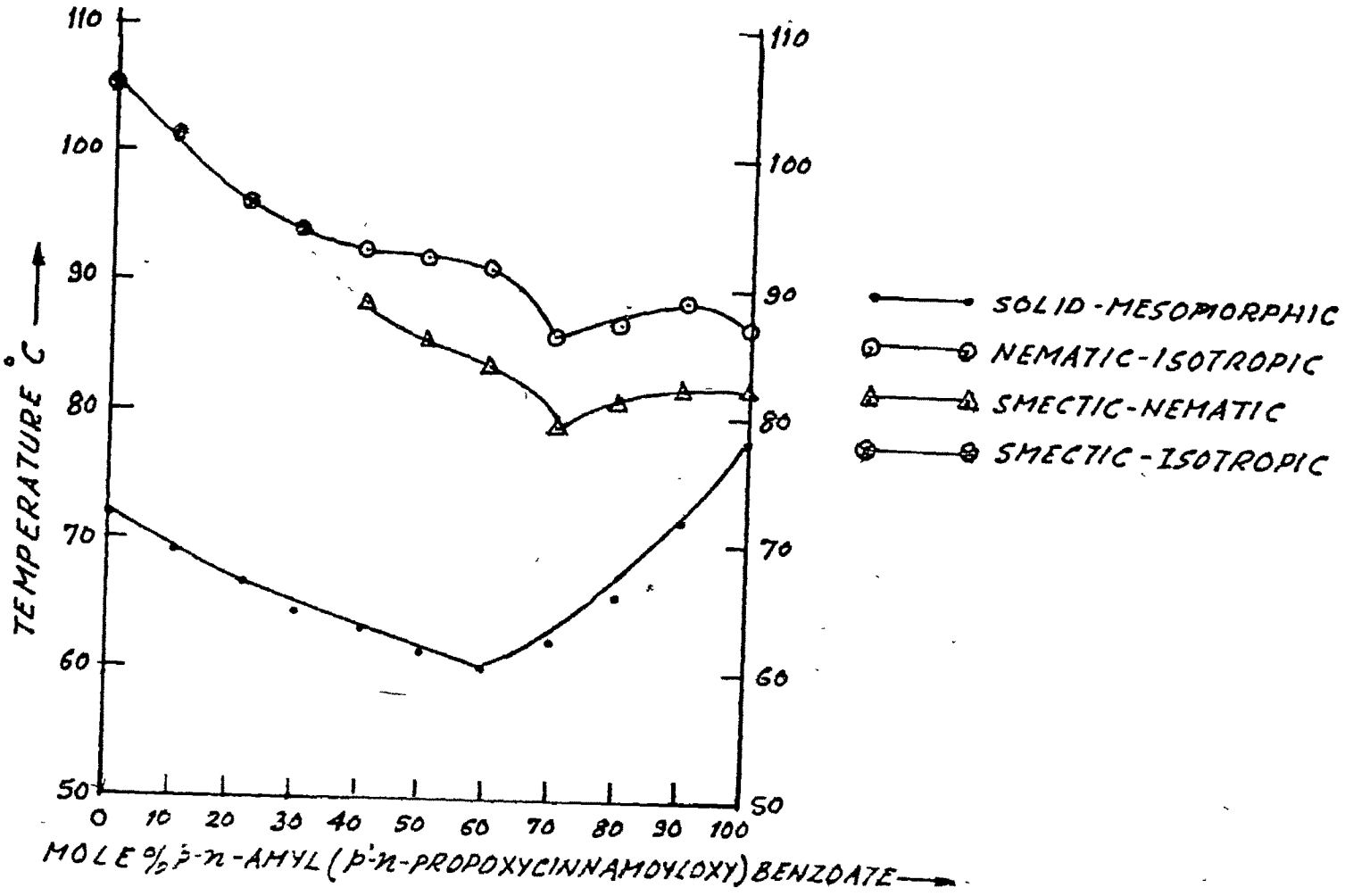


TABLE - 39

System No. 17

Components : (A) n.Amyl-P-(p'-n'-propoxy cinnamoyloxy) benzoate

(B) n.Amyl-P-(p'-n'-butoxycinnamoyloxy) benzoate

Mole % of n-Amyl-P-(p'-n'-propoxycinnamoyloxy) benzoate	Transition Temperatures (°C) S.	N.	I.
100.00	78.0	82.0	87.5
90.30	72.0	82.0	89.0
80.55	66.5	81.0	87.0
70.72	62.5	79.0	86.0
60.83	60.0	84.0	91.0
50.86	61.5	85.5	92.0
40.83	63.5	88.5	92.5
30.73	64.5	-	94.0
22.62	66.5	-	96.0
10.31	69.0	-	101.0
00.00	72.0	-	105.0

Sintering point = 60°C

SYSTEM: 18.

FIG. 34

p-(p'-n-PROPOXYBENZOYLOXY)PROPIOPHENONE : p-(p'-n-METHOXYBENZOYLOXY)BUTYROPHENONE

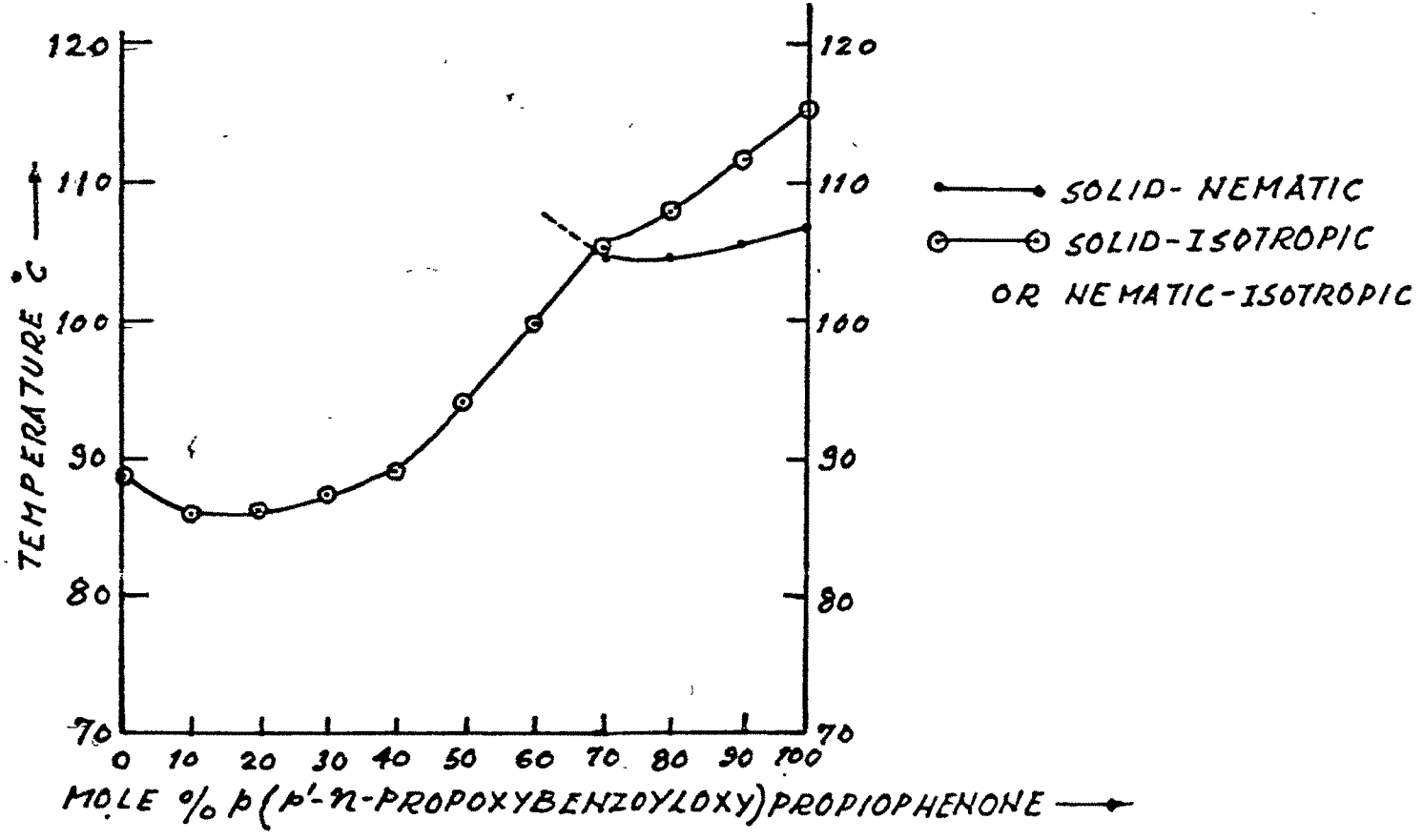


TABLE - 40

System No. 18

Component : (A) p- (p'-n-propoxybenzoyloxy) propiophenone

(B) p- (p'-methoxybenzoyloxy) butyrophenone

Mole % of P-(p'-n-proponybenzoyloxy propiophenone	Transition Temperature (°C)		
	S	N	I
100.00	-	106.5	115.5
90.34	-	105.5	112.0
80.45	-	105.0	108.0
69.89	-	105.0	105.5
59.73	-	-	100.0
50.12	-	-	94.5
40.34	-	-	89.5
30.05	-	-	87.5
20.04	-	-	86.5
9.88	-	-	87.0
00.00	-	-	89.0

FIG. 35

SYSTEM: 19

p-(p'-ETHOXYBENZOYLOXY)PROPIOPHENONE: p-(p'-n-PROPOXYBENZOYLOXY) PROPIOPHENONE

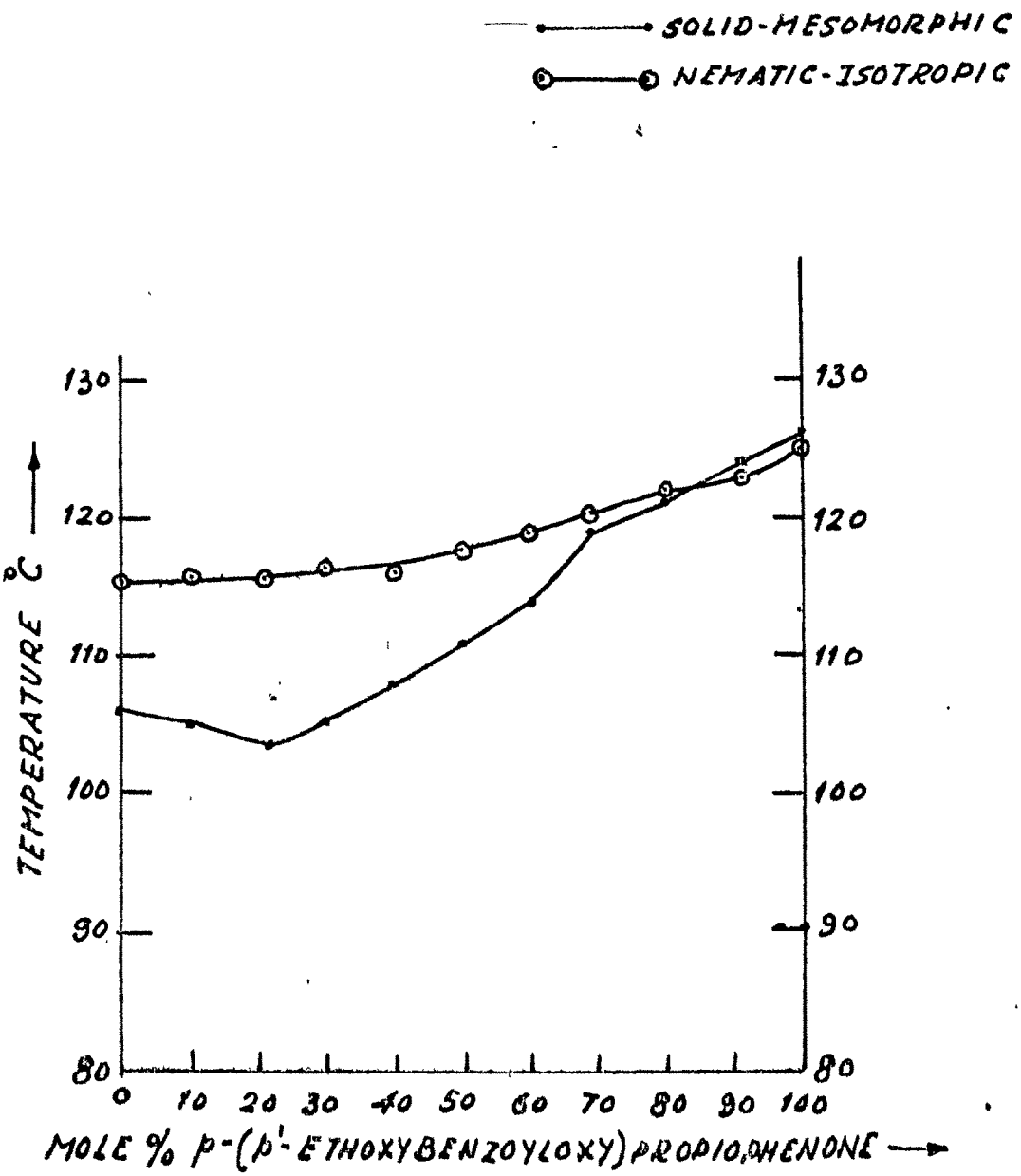


TABLE - 41

System : 19. (A) p- (p'-n-ethoxybenzoyloxy) propiophenone

(B) p- (p'-n-propoxybenzoyloxy) propiophenone

Mole % of p- (p'-n-ethoxybenzoyloxy) propiophenone	Transition Temperatures (°C) S.	N.	I.
100.00	-	(125)	126.0
91.23	-	(123)	124.0
80.12	-	121.5	122.5
69.14	-	119.0	120.5
59.94	-	114.0	118.5
50.02	-	111.0	117.0
40.18	-	108.0	116.0
29.93	-	105.5	116.5
21.33	-	103.5	115.5
10.11	-	105.0	116.0
00.00	-	106.0	115.5

Values in parenthesis indicate monotropy

Sintering point - 103.5

FIG. 36

SYSTEM: 20

p-(p'-ETHOXYBENZOYLOXY)PROPIOPHENONE: p-(p'-n-BUTOXYBENZOYLOXY) PROPIOPHENONE

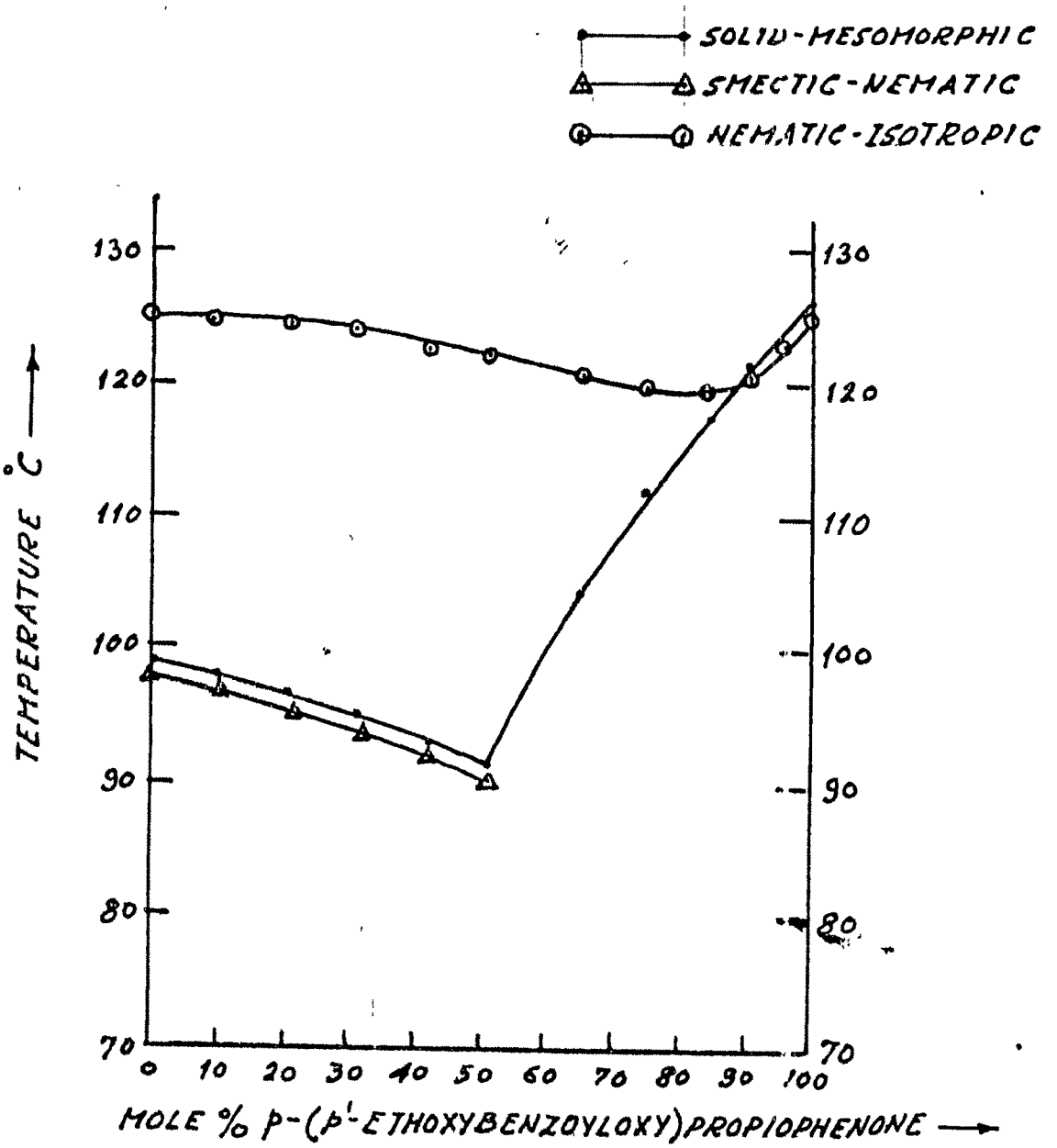


TABLE - 42

System - 20 (A) p- (p'-Ethoxybenzoyloxy) Propiophenone

(B) p- (p'-n-butoxybenzoyloxy) Propiophenone

Mole % of p-(p'-Ethoxybenzoyloxy) propiophenone	Transition Temperatures (°C) S.	N.	I.
100.00	-	(125)	126.0
95.00	-	(122.5)	123.0
90.11	-	(120.0)	121.0
85.03	-	117.5	119.5
75.23	-	112.0	119.5
64.87	-	104.0	121.0
50.92	(90.0)	91.5	122.0
41.89	(92.0)	93.0	122.5
31.08	(93.5)	95.0	124.0
20.78	(95.5)	96.5	125.0
10.33	(97.0)	98.0	125.0
00.00	(97.5)	99.0	125.0

Values in parentheses indicate monotropy.

Sintering point - 91.5

SYSTEM: 21.

FIG. 37

p-(p'-n-BUTOXYBENZOYLOXY) BUTYROPHENONE : p-(p'-n-AMYLOXYBENZOYLOXY) BUTYROPHENONE

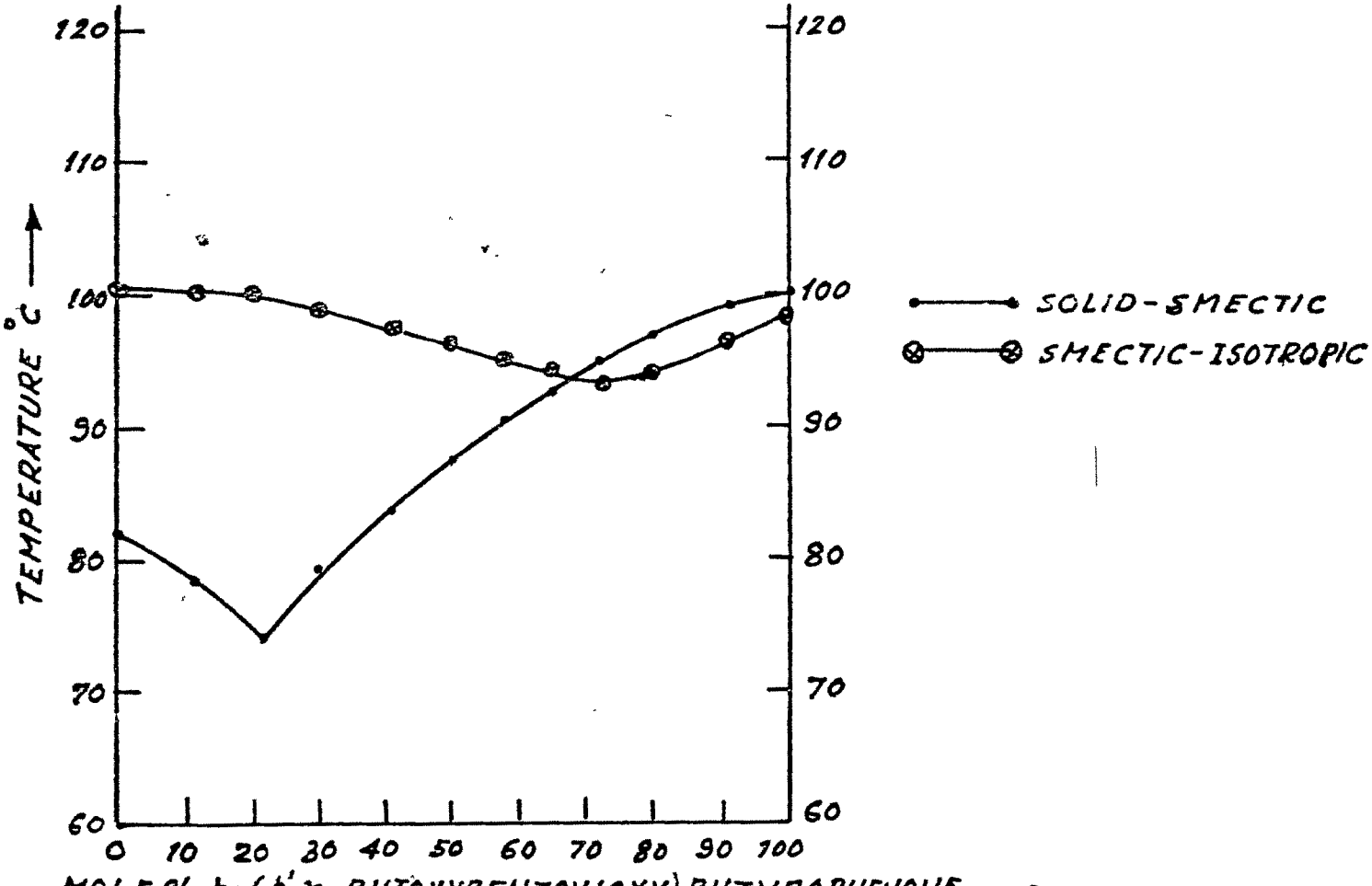


TABLE - 43

System - 21 P-(p'-n-butoxybenzoyloxy) butrophenone

P-(p'-n-amyloxybenzoyloxy) butyrophenone

Mole % of P- (p'-n-butoxy-benzoyloxy) butyro-phenone.	Transition Temperatures (°C) S.	N.	I.
100.00	(98.0)	-	100.0
90.98	(96.0)	-	99.0
80.07	(94.0)	-	97 .0
71.88	(93.0)	-	95.0
65.02	92.5	-	94.0
58.79	90.5	-	95.0
50.11	87.5	-	96.0
40.78	83.5	-	97.5
30.05	79.0	-	99.0
20.11	74.0	-	100.0
10.94	78.0	-	100.0
00.00	82.0	-	100.5

Values in parentheses indicate monotropy.