UDC 548.3

SYNTHESIS, CHARACTERIZATION, AND STUDY OF THE CRYSTALLINE PROPERTIES OF *ASTERISM* COMPOUNDS

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Received 15.05.2024
Accepted 04.07.2024

Abstract: *This study involves the synthesis of asterism compounds (D1-D6) through the reaction of 1 mole of benzene 1,3,5-triol with 3 moles of prepared pentacyclic ring ester derivatives, dissolved in absolute ethanol. The validity of the compound structures was confirmed using physical and spectroscopic methods such as infrared spectroscopy, and proton nuclear magnetic resonance spectroscopy. Additionally, melting points and purity were determined, and reaction progress was monitored by Thin-Layer Chromatography (TLC). The liquid crystal phases of certain prepared compounds were examined using a polarizing optical microscope (POM).*

Keywords: *Asterism compounds, Esters, Liquid crystals.*
DOI: 10.32737/2221-8688-2024-4-478-488

1. Introduction

Liquid crystals represent a transitional state between the irregular liquid state and the ordered crystalline solid state [1, 2]. Liquid crystals were described as the fourth state of matter and were called fluid crystals or floating crystals [3]. Liquid crystals exhibit a state intermediate between the solid phase, where molecular motion is constrained and molecular organization is complete in terms of position and direction [4], and the isotropic or liquid phase [5, 6], where molecular motion is free and this phase exhibits random organization, despite displaying properties attributable to both liquid and solid states, they possess unique properties not present in either liquid or solid states [7]. Liquid crystals are a type of fluid containing a specific system in which molecules are arranged [8]. This arrangement makes the substance anisotropic, meaning its physical properties are not present in all directions [9]. Classification of different phases of thermotropic liquid Crystals Nematic-thread like molecules, parallel arrangement and Discotic -disk like molecules and Smectic-soap like smectic A molecules with transitional/ rotational motion and smectic C molecules arrangement in layers parallel tonormal and Chiral-also known as cholesteric [10].

The most distinguishing feature of these molecules is their possession of flat shapes resembling sheets or elongated shapes resembling rods, and with the assistance of these shapes, the molecules arrange themselves parallel to each other [11, 12]. Liquid crystals have numerous applications in medicine and industry, including common applications such as their use in watches, computers, television screens, and electronic games [13, 14].

This study tries to synthesize asterism compounds (D1-D6) via a meticulous organic reaction. The significance of this process lies in the strategic choice of benzene 1,3,5-triol as the central core due to its symmetrical structure and reactivity, which facilitates optimal interaction with the pentacyclic ester moieties. Dissolving the reactants in absolute ethanol ensures a high-purity solvent environment that minimizes potential side reactions and maximizes product yield. This methodological approach not only underscores the precision required in forming complex organic compounds but also highlights ethanol role as an efficient solvent in promoting effective esterification reactions. The resulting asterism compounds may exhibit unique chemical properties and potential applications across various fields such as pharmaceuticals, materials science, and supramolecular chemistry. By carefully controlling reaction conditions and leveraging the inherent reactivity of benzene triol and pentacyclic esters, this study will advances our ability to design and synthesize novel compound architectures with tailored functionalities.

2. Experimental part

2.1. Material and Devices used.

The chemical materials are collected from Fluka, Aldrich, and BDH and used without further purification. The melting points were measured using Electrothermal Melting Apparatus 9300. Bruker FT-IR 8400S spectrophotometer with a scale of (400-4000) cm^{-1} by KBr disc. ^{1}H-NMR spectra on Bruker instruments running at 400 MH_Z. Thin Layer Chromatography (TLC) was performed using Fluka silica gel plates with 0.2 mm thickness, activated with fluorescent silica gel G, and visualization was achieved using UV light.

2.2. Synthesis of Asterism Compounds (discotic) (D1-D6)

In a 50 mL round-bottom flask containing 20 mL of absolute ethanol, 0.001 mol (0.5 g) of benzene 1,3,5-triol is dissolved to this 0.003 mol of prepared thiazolidinone ring esters derivatives dissolved in absolute ethanol is added. After the addition is complete, the reaction mixture is refluxed for 7 hours. The progress of the reactions was monitored using thin-layer chromatography (TLC) and iodine staining. Upon completion of the reflux period, the mixture was cooled and stirred for an additional hour. Precipitate formation was noticed, collected, dried, and recrystallized using ethanol [18, 19]. Table (1) show some physical properties of the synthesized compounds.

Table 1. Yield ratios and some physical properties of compounds (D1-D6).

Comp.	Molecular Formula	X	Yields %	R*f*	M.P. ^oC	Colour
D1	$C_{54}H_{39}N_3O_9S_3$	H	82	0.58	205-208	Yellow
D_2	$C_{54}H_{36}Cl_3N_3O_9S_3$	Cl	70	0.54	188-190	Orange
D_3	$C_{54}H_{36}Br_3N_3O_9S_3$	Br	68	0.54	118-120	Orange
D_4	$C_{54}H_{36}N_6O_{15}S_3$	NO_2	67	0.54	199-202	Orange
D_5	$C_{57}H_{45}N_3O_{12}S_3$	OCH_3	59	0.64	173-175	Orange
D_6	$C_{57}H_{45}N_3O_9S_3$	CH_3	72	0.61	194-196	Orange
X represent replacement functional group (Figure 1) Compounds (D1-D6) are newly synthesized for the first time M.P. indicates melting points						

2.3. Study of Liquid Crystal Phases

The liquid crystal phases of certain prepared compounds were examined using a polarizing optical microscope (POM) carried with an electric heater. The microscope was also carried with a high-resolution camera and a heat-resistant lens to obtain high-precision images. The compound under examination, whose liquid crystal phases were to be investigated, was placed in the liquid crystal phase measurement device, and the compound was heated to its melting point. Subsequently, the compound was cooled, and this process was repeated several times to obtain liquid crystals with clearer geometric shapes [20, 21].

3. Results and Discussion

Asterism Compounds (discotic) (D1-D6) react by 3 moles at 1,3,5-triyl with 1 mole benzene to produce thiazolidinone (Fig. 1).

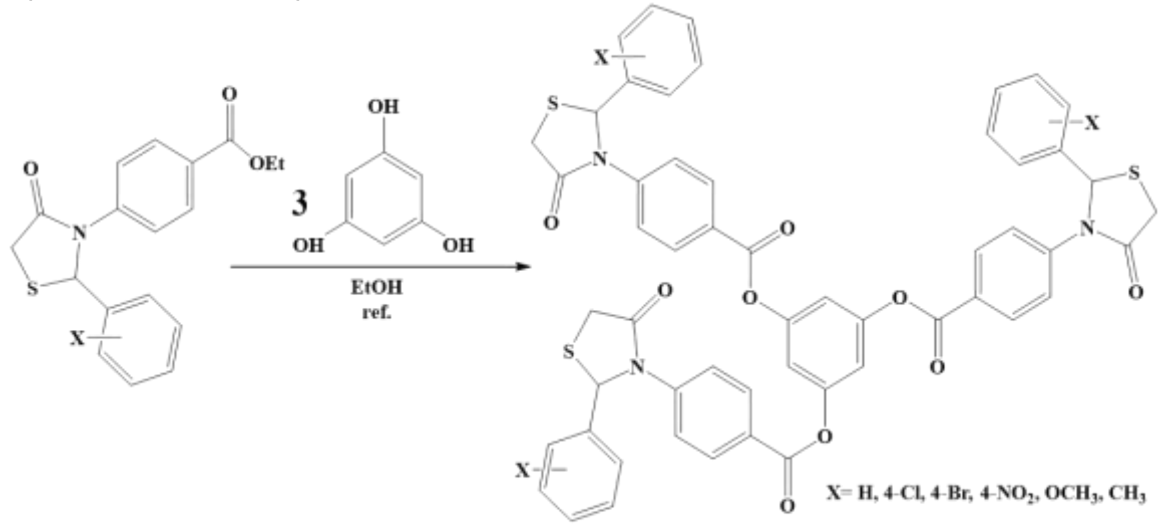

Fig. 1. The structural formula of the prepared compounds (D1-D6). Reactant (discotic, left side of the equation), productant (thiazolidinone, right side of the equation).

3.1. Properties of Compounds (D1-D6) by FT-IR

The infrared spectrum of compounds (D1-D6) noted that an absorption band appeared in the range (3010-3065) cm^{-1}, which belongs to the stretching of the aromatic (CH) bond. Also, an absorption band appeared in the range (1681-1696) cm^{-1} due to the stretching of the carbonyl (C=O) ester bond and an absorption band appeared in the range (1520-1600, 1412-1459) cm^{-1} due to the stretching of the (C=C) ring bond, and an absorption band appeared in the range (659-696) cm^{-1} is due to the stretching of the (C-S) bond, [22, 23] (Table 2).

Table 2. FT-IR absorption results for oxazepine derivatives (D1-D6) (cm^{-1})

Comp.	-X	νC-H Arom.	νC=O ester	ν C=C ring		νC-S	Others
D1	H	3021	1684	1520	1413	696	……….
D2	Cl	3065	1686	1561	1412	674	C-Cl 782
D3	Br	3030	1696	1550	1459	681	C-Br 654
D4	NO_2	3025	1684	1573	1454	668	NO_2 1538, 1293
D5	OCH_3	3048	1686	1600	1444	659	C-H alip. 2993
D6	CH_3	3010	1681	1593	1425	694	C-H alip.2953

3.2. Characterization of compounds (D1-D6) by ^{1}H-NMR

The solvent (DMSO-d^6) was used for ^{1}H-NMR study of all six compounds (D1-D6).

When studying the ^{1}H-NMR spectrum of the compound [D1], it was observed that a multiple signal in the range (7.45-9.06) ppm is attributed to the protons of the aromatic rings, a single signal appears at the chemical shift (6.57) ppm due to the proton of three groups (CH) in the thiazolidinone ring, and a single signal appears at the chemical shift (4.17) ppm due to the protons of tree groups (CH_2) in the thiazolidinone ring, and the appearance of a single signal upon chemical shift (3.34) ppm is attributed to the protons of water (HDO), and a signal appears at the chemical shift (2.48-2.50) ppm is attributed to the protons of the solvent (DMSO-d^6) [24,25] (Fig. 2).

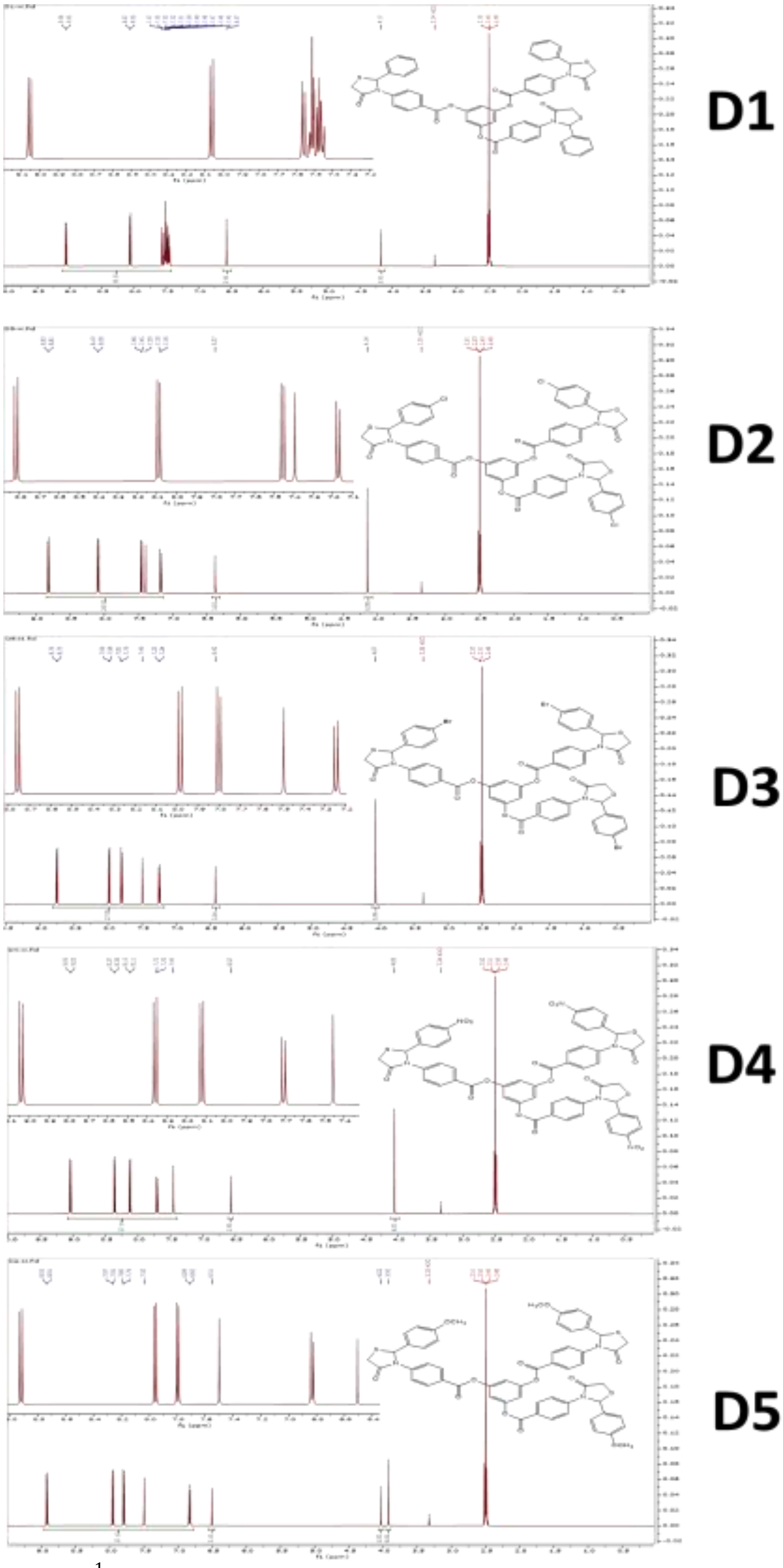

Fig. 2. ^{1}H-NMR spectrum of the compound (D1 to D5).

The ^{1}H-NMR study of the compound [D2] shows that a multiple signal in the range (7.16-8.83) ppm is attributed to the protons of the aromatic rings, a single signal appears at the chemical shift (6.37) ppm due to the proton of three groups (CH) in the thiazolidinone ring, and a single signal appears at the chemical shift (4.14) ppm due to the protons of tree groups (CH_2) in the thiazolidinone ring, and the appearance of a single signal upon chemical displacement (3.35) ppm is attributed to the protons of water (HDO), and a signal appears at the chemical shift (2.48-2.51) ppm is attributed to the protons of the solvent (DMSO-d^6), [26,27] (Fig. 2).

The ^{1}H-NMR spectrum of the compound [D3] shows that multiple signals in the range (7.24-8.76) ppm is attributed to the protons of the aromatic rings, a single signal appears at the chemical shift (6.42) ppm due to the proton of three groups (CH) in the thiazolidine ring, and a single signal appears at the chemical shift (4.07) ppm due to the protons of tree groups (CH_2) in the thiazolidine ring, and the appearance of a single signal upon chemical displacement (3.36) ppm is attributed to the protons of water (HDO), and a signal appears at the chemical shift (2.48-2.53) ppm is attributed to the protons of the solvent (DMSO-d^6) [28, 29] (Fig. 2).

The ^{1}H-NMR spectrum of the compound [D4] reveals that multiple signals in the range (7.46-9.05) ppm is attributed to the protons of the aromatic rings, a single signal appears at the chemical shift (6.57) ppm due to the proton of three groups (CH) in the thiazolidine ring, and a single signal appears at the chemical shift (4.05) ppm due to the protons of tree groups (CH_2) in the thiazolidine ring, and the appearance of a single signal upon chemical displacement (3.34) ppm is attributed to the protons of water (HDO), and a signal appears at the chemical shift (2.49-2.52) parts per million is attributed to the protons of the solvent (DMSO-d^6) [30, 31] (Fig. 2).

When studying the ^{1}H-NMR spectrum of the compound [D5], it was observed that a multiple signal in the range (6.82-9.38) ppm is attributed to the protons of the aromatic rings, a single signal appears at the chemical shift (6.51) ppm due to the proton of three groups (CH) in the thiazolidinone ring, and a single signal appears at the chemical shift (4.03) ppm due to the protons of tree groups (CH_2) in the thiazolidinone ring, and a single signal appears at the chemical shift (3.92) ppm due to the proton of tree groups (CH_3), and the appearance of a single signal upon chemical displacement (3.33) ppm is attributed to the protons of water (HDO), and a signal appears at the chemical shift (2.48-2.51) parts per million is attributed to the protons of the solvent (DMSO-d^6), [32,33] (Fig. 2).

The protons of the aromatic rings are responsible for a multiple signal in the range of 7.02-8.85 ppm, according to the chemical [D6's] 1H-NMR spectra, and , a single signal appears at the chemical shift (6.51) ppm due to the proton of three groups (CH) in the thiazolidinone ring, and a single signal appears at the chemical shift (4.04) ppm due to the protons of tree groups (CH_2) in the thiazolidinone ring, and the appearance of a single signal upon chemical displacement (3.34) ppm is attributed to the protons of water (HDO), and a signal appears at the chemical shift (2.49-2.51) ppm is attributed to the protons of the solvent (DMSO-d^6), and a single signal appears at the chemical shift (2.22-2.25) ppm due to the proton of tree groups (CH_3) [34, 35].

3.3. Diagnosis and Discussion of Liquid Crystal Behavior

One of the pivotal factors driving the study of liquid crystal behaviour in synthesized compounds is the unique physical and chemical characteristics of these liquid crystalline compounds. Therefore, the liquid crystal behaviour of prepared compounds was investigated using polarizing optical microscopy (POM) equipped with an electric heater. This involved taking a quantity of the material to be studied and placing it on a glass slide, then observing the thermal transitions of liquid crystalline phases during heating. Subsequently, the nature of these transitions was identified, and the thermal stability of each transition was studied. To understand the nature of these transitions and their thermal stability, it is essential to grasp some general concepts about the nature of the prepared compounds and the probability of liquid crystalline phases appearing in them [36, 37].

Organic compounds were prepared, one with a rod-like shape and the other discotic, both being morphologies indicative of liquid crystalline phases. Mesogenic units were prepared from a molecular core consisting of more than two aromatic rings connected by groups enhancing electronic delocalization along the molecular axis, represented by groups (-C=N, -OC=O). The liquid crystal behaviour of prepared compounds was monitored using POM to determine the liquid crystalline transitions and to study the nature of these transitions and their thermal stability. It was observed that most discotic compounds exhibited liquid crystalline transitions with high thermal stability, depending on the nature of the mesogenic units for each compound [38, 39]. When studying the liquid crystal behaviour of compounds under investigation, it was noticed that compounds with rod-like mesogenic units did not exhibit liquid crystalline properties, possibly due to the ratio of molecular length to width not falling within the range (L/d ≥ 4.0-6.4), where d is the average molecular diameter and L is the molecular length [40,41]. The appearance of discotic liquid crystal phases in compounds might be attributed to the mesogenic unit's disc-like structure possessing a disparate radial axis, with the molecular diameter significantly larger than the longitudinal axis represented by the disc thickness [42]. Therefore, the appearance of liquid crystalline properties in discotic compounds is attributed to the presence of a proper structure of mesogenic units composed of the molecular core containing more than two aromatic rings linked by bonds enhancing electronic delocalization along the molecular axis.

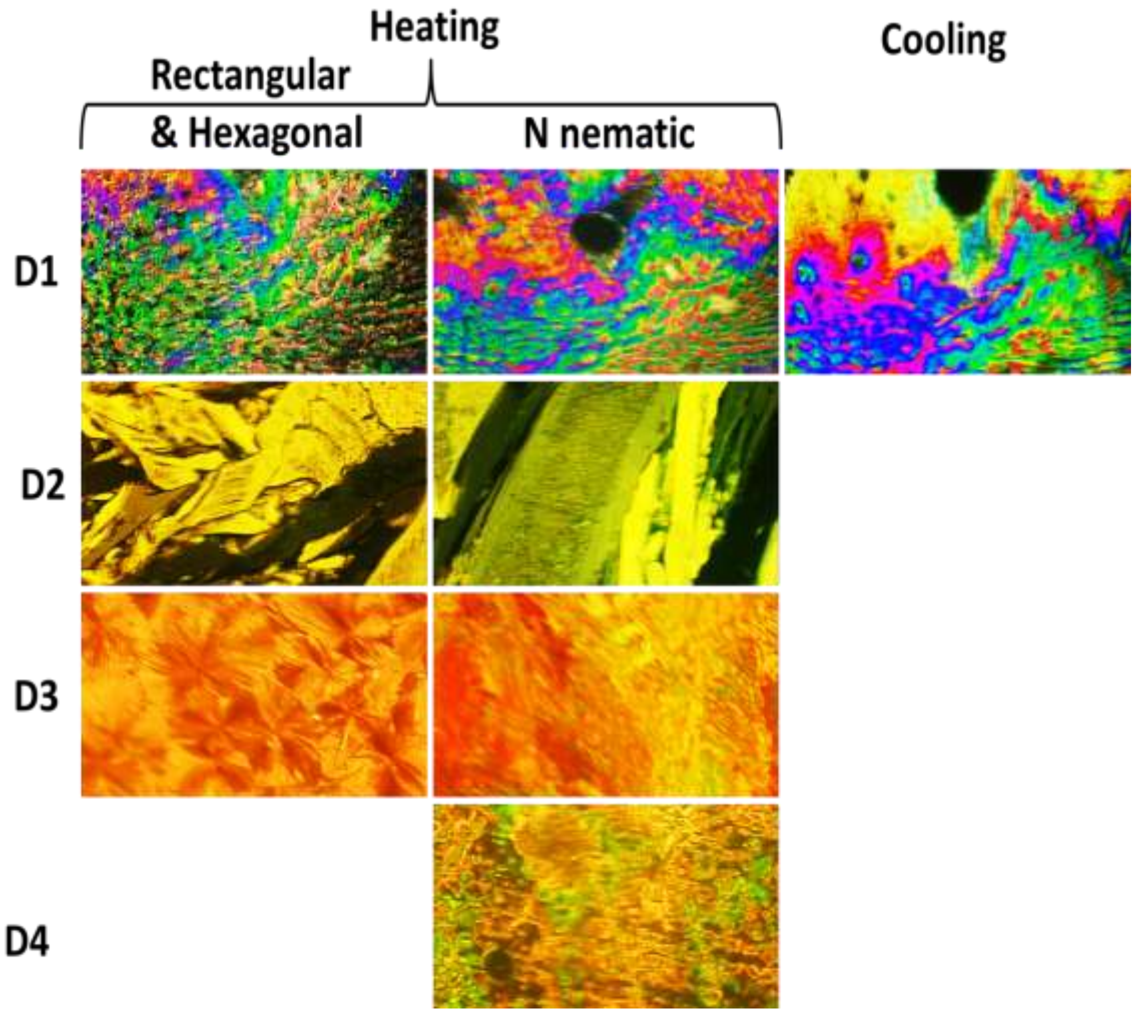

Fig. 3. Heating cooling phases of liquid crystal of the generated compound D1 to D4 showing N nematic, rectangular, and hexagonal shapes.

Furthermore, the presence of polar terminal groups increasing terminal bonding forces and side attraction forces between mesogenic unit molecules favour the appearance of rectangular and homogeneous liquid crystal phases. Additionally, the presence of polar terminal groups increases terminal bonding forces favoring the appearance of nematic liquid crystal phases in the compound. It was observed that the thermal stability of the nematic liquid crystal phase (ΔN) increases with the presence of polar terminal groups, as the presence of groups like (NO_2, OCH_3) at the molecule's end provides greater thermal stability to the nematic phase compared to groups like (H, CH_3) [43, 44]. Studying the produced compounds' liquid crystal characteristics revealed that the transitions were monotropic and only happened when the compounds heated, except in compound (D1), where they appeared during both heating and cooling, indicating an enantiotropic property [45, 46]. Table (3) illustrates the transition temperatures for the compounds under study, and (Figure 3) depicts images of the liquid crystalline compounds obtained.

Studying the produced compounds' liquid crystal characteristics revealed that the transitions were monotropic and only happened when the compounds heated.

Table 3. Transition temperatures and liquid crystal phases of prepared compounds.

No.	Cr	S_A	S_C	N	$ΔS_A$	$ΔS_C$	ΔN
	Cr	Col.h	Col.r	N	Δ Col.h	Δ Col.r	ΔN
D1	203		241	258		38	17
	208			252			44
D2	180		219	241		39	22
D3	119	134		159	15		25
D4	193			226			33
Cr=Crystal phase S_A, S_C , = Smectic phase N= Nematic phase							

4. Conclusions

The prepared compounds were of high purity and this was confirmed by various spectroscopic examinations. The increase in the number of aromatic rings led to an increase in melting points. The absence of crystal phases in some compounds may be due to the effect of increasing the number of aromatic rings that increased the hardness of the molecule, which prevented the appearance of crystal phases as well as compensated aggregates on the rings, which increase the width of the molecule and thus not align.

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ASTERİZM BİRLƏŞMƏLƏRİNİN SİNTEZİ, XARAKTERİSTİKASI VƏ KRİSTAL XÜSUSİYYƏTLƏRİNİN ÖYRƏNİLMƏSİ

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Xülasə: Bu tədqiqat asterizm quruluşlu birləşmələrinin (D1-D6) mütləq etanolda həll edilmiş 1 mol benzol 1,3,5-triol və 3 mol hazırlanmış pentasiklik halqalı efir törəmələri ilə reaksiyası vasitəsilə sintezinə həsr olunmuşdur. Birləşmələrin strukturları infraqırmızı spektroskopiya və proton nüvə maqnit rezonans spektroskopiyası kimi fiziki və spektroskopik üsullardan istifadə etməklə təsdiq edilmişdir. Bundan əlavə, alınmış birləşmələrin ərimə nöqtələri və təmizlik dərəcələri müəyyən edilmişdir. Reaksiyanın gedişi Nazik Örtük Xromatoqrafiyası (TLC) ilə müşahidə edilmişdir. Bir sıra birləşmələrin maye kristal fazaları polyar optik mikroskop (POM) vasitəsilə tədqiq edilmişdir.
Açar sözlər: asterizm birləşmələri, efirlər, maye kristallar.

СИНТЕЗ, ХАРАКТЕРИСТИКА И ИЗУЧЕНИЕ КРИСТАЛЛИЧЕСКИХ СВОЙСТВ СОЕДИНЕНИЙ АСТЕРИЗМА

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Резюме: Это исследование включает синтез соединений со звездоподобной структурой (D1-D6) посредством реакции 1 моля бензол-1,3,5-триола с 3 молями приготовленных производных пентациклического кольца эфира, растворенных в абсолютном этаноле. Достоверность структур соединений была подтверждена с использованием физических и спектроскопических методов, таких как инфракрасная спектроскопия и протонная ядерная магнитно-резонансная спектроскопия. Кроме того, были определены точки плавления и чистота, а ход реакции контролировался с помощью тонкослойной хроматографии (ТСХ). Жидкокристаллические фазы некоторых приготовленных соединений были исследованы с помощью поляризационного оптического микроскопа (ПОМ).
Ключевые слова: соединение астеризма, эфиры, жидкие кристаллы.