Carbocyclic and nitrogen-containing fused CH-acids with an annelated indenyl fragment. Thermogravimetric and X-ray structural analysis of 6*H*-indeno[1,2-*b*]quinoline

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Based on thermogravimetric characteristics first obtained for the model 6*H*-indeno[1,2-*b*]quinoline, the scheme of thermal conversions of this compound in the temperature range 20—700 °C has been proposed, and the limit of its thermal stability (~300 °C) has been determined. This temperature is recommended as the optimum for synthesizing fused benzoaza(diaza)fluorenes. Based on the results of X-ray structural analysis, the molecules of the studied indenoquinoline form centrosymmetric pairs, which are arranged in (110) layers. The molecules are orientationally disordered. The observed self-association of these molecules is similar to the π—π association of fused heterocyclic systems with π-excessive and π-deficient fragments. It has been suggested that interferon-inducing and antitumor compounds with an annelated indenyl fragment have a common mechanism of action according to the intercalation model of stacking structures.

Key words: CH-acids; 6*H*-indeno[1,2-*b*]quinoline, heterogeneous catalytic dehydrogenation, thermogravimetric and X-ray structural analysis; interferon-inducing and antitumor intercalators; topological factor; stacking structures.

Fused indenoazanaphthalenes and azaindenonaphthalenes with linear and angular structures form a large group of heterocyclic CH-acids, which includes 6*H*-indeno[1,2-*b*]quinoline (2,3-benzo-4-azafluorene). Analysis of the data reported in the literature and the results that we obtained previously[1,2] indicates that the above-mentioned group of CH-acids is of substantial interest in the search for biologically active compounds exhibiting hormonal, antibacterial, and antitumor action as well as in the synthesis of alkaloid-like systems, particularly benzannelated analogs of new and poorly studied onychine alkaloids from the tropics, structurally based on the keto-form of 4-azafluorenone.[3] Benzannelated azaindenes can be used for finding synthetic inducers of interferon and for studying the mechanism of interferon genesis. The high interferon-inducing ability of the compounds of the fluorene series topologically related both to azafluorenes (indenopyridines) and to fused benzoaza(diaza)fluorenes [indenoquinolines (-isoquinolines, -quinoxalines)] has been demonstrated.[4]

Analysis of information on compounds exhibiting antitumor and interferon-inducing action requires consideration of a complex of factors that are regarded as necessary indications of specific features of the biological action of compounds and that are suitable for revealing and predicting active compounds by suitable rapid methods prior to the performance of total screening at the *in vivo* stage. The molecular nature of the object, the type of its functionality, the geometric parameters of the molecule, and its topology should be considered the most important features in the prediction of antitumor and interferon-inducing compounds. As is evident from the analysis of the specific features and the efficiency of the physiological action of many natural and synthetic medicines of organic origin, the geometric parameters and topology often exert a crucial influence on the total effect of their biological action. However, these factors, particularly, the topological factor, have received little

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attention. We performed the heterogeneous catalytic synthesis of tri- and tetranuclear carbocyclic and nitrogen-containing CH-acids with an annelated indene fragment by high-temperature dehydrocyclization of *o*-methyl-substituted diaryls and arylazahetaryls in the presence of the industrial oxide catalyst K-16 (see Refs. 5 and 6). Pentacyclic CH-acids of the fused indenofluorene (fluoreneacetone) type were also obtained by this method.[7,8] In particular, dehydrocyclization of 2-*o*-tolylquinoline (**1**) at 540—550 °C on K-16 affords 6*H*-indeno[1,2-*b*]quinoline (**2**) in a yield of 32—34 %.[1,6] Under similar conditions, unsubstituted and methyl-containing fluorenes, azafluorenes, benzoaza(diaza)-fluorenes, and fused indenofluorenes were prepared. The carbon framework of the obtained CH-acids is the basis or a component of the molecular structures of many of the most important biologically active compounds of natural and synthetic origin; compounds of this kind are also used in chemistry of organometallic π-complexes.[9,10]

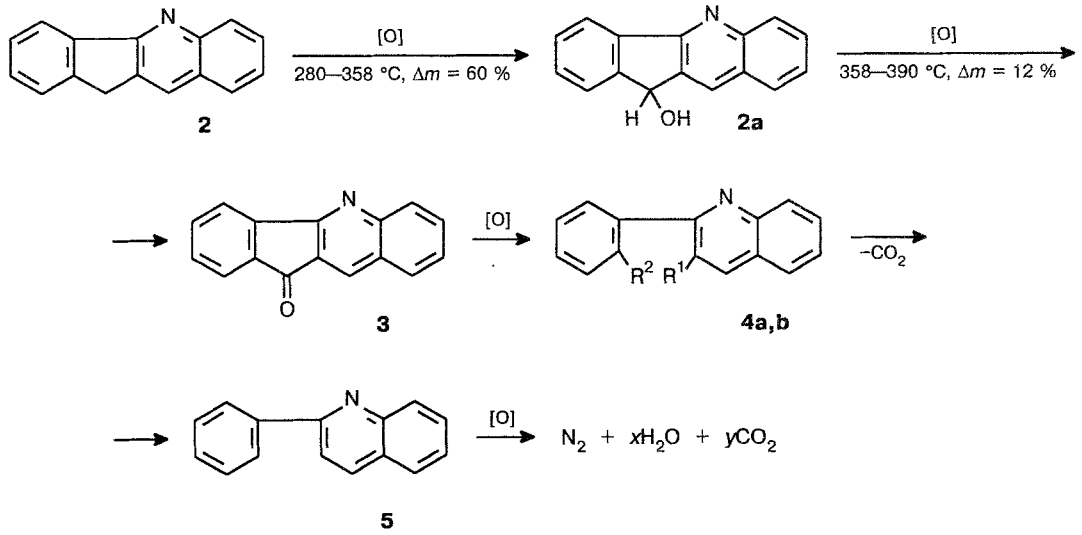
This work is devoted to the thermogravimetric analysis of indenoquinoline **2**, the study of its molecular and crystal structure by X-ray structural analysis, and the use of the obtained geometric parameters of model compound **2** for interpreting its thermogravimetric characteristics and assessing its properties as an intercalator of antitumor and interferon-inducing action that undergoes π–interaction with DNA.

Results and Discussion

To determine the optimum temperature conditions for heterogeneous catalytic synthesis of fused nitrogen-containing indenonaphthalenes, we performed thermogravimetric analysis of three genetically related compounds: 2-*o*-tolylquinoline **1**, indenoquinoline **2**, and 6*H*-indeno[1,2-*b*]quinolin-6-one (**3**), a ketone that forms rather readily[1,6] when the methylene group of CH-acid **2** is oxidized. Thermogravimetric analysis of these compounds was carried out in the temperature range 20—700 °C with an accuracy of measurements of 2—4 °C. The derivatogram of tolylquinoline **1** shows that this compound is rather thermally stable and undergoes sublimation in the temperature range 275—350 °C. Taking into account the character of the DTG, DTA, TA, and TG curves for indenoquinoline **2** (Fig. 1), we believe that under the conditions in which the derivatogram was recorded, this CH-acid undergoes the following series of successive conversions. At ~160 °C melting occurs. Two small endothermal and exothermal effects in the DTA and DTG curves corresponding to the temperature ranges 280—358 °C and 358—390 °C characterize the conversion of **2** to alcohol (**2a**) and subsequent oxidation of the latter to ketone **3**, respectively. As the derivatogram shows, a horizontal region is present in the TG curve, where weight loss occurs in the range 390—540 °C (the DTA and TA curves) and, apparently, in this temperature range, ketone **3** is oxidized to acids (**4a,b**) (Scheme 1).

The appearance of several bends in the TG curve in the temperature range 540—640 °C may be caused by decarboxylation of acids **4a,b** to form 2-phenylquinoline (**5**) and by oxidation of the latter with the release of the final products of decomposition: nitrogen, water, and carbon dioxide. However, judging from the data of the derivatogram of the pure sample of ketone **3**, the latter melts in the temperature range 174—175 °C; the second broad peak of the endothermal effect observed in the

Scheme 1



4a: R^1 = H, R^2 = COOH; **4b:** R^1 = COOH, R^2 = H

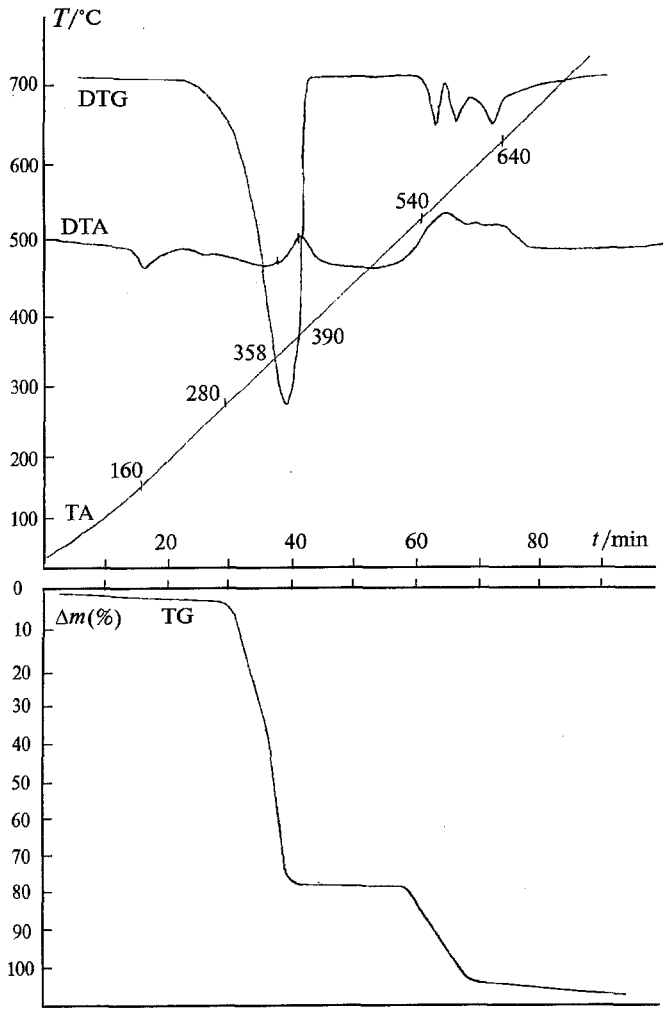


Fig. 1. The derivatogram of indenoquinoline **2**.

DTA and DTG curves of this ketone corresponds to the temperature range 315—390 °C, in which ketone **3** sublimates; hence, we failed to detect the expected transition (oxidation) of ketoindoquinoline **3** to acids of the type **4a,b** from the derivatogram. However, regardless of the validity of the proposed scheme of thermal conversions of indenoquinoline **2**, the analysis of its derivatogram taking into account the characteristic features and interpretation of the TG curve clearly shows that the major portion of the weight loss (Δm) of the compound under study in these thermal processes is observed at 280—390 °C and is ~72 %, of which ~60 % falls in the temperature range 280—358 °C, and the remaining ~12 % falls in the range 358—390 °C. Hence, even at temperatures 100—250 °C lower than the experimentally determined optimum temperature range for obtaining indenoquinoline **2** by heterogeneous catalytic dehydrocyclization in the presence of the K-16 catalyst, more than half of this compound undergoes thermal decomposition. In light of these data, the reason for the rather low (9—34 %) yields of benzoaza(diaza)fluorenes including indenoquinoline **2** also becomes clear; when these compounds are prepared by dehydrocyclization of *o*-methyl-containing arylquinoline on the above-mentioned catalyst,[1,5,6] dehydrocyclization of the initial compounds is accompanied by vigorous thermal decomposition of the formed benzoaza(diaza)fluorenes.

Clearly, the foregoing scheme of thermal conversions of indenoquinoline **2** is hypothetical and does not exclude alternative schemes. However, we proposed this scheme taking into account experimentally performed of successive transitions from indenoquinoline **2** to arylquinoline **5** by chemical methods.[1] The quantum-chemical characteristics of ketoindenoquinoline **3** and its methyl analogs also indicate that thermal decomposition of this ketone at single bonds involving its carbonyl carbon atom is favorable, *i.e.*, the oxidation of ketone **3** to acids of the type **4a,b** is possible under the conditions of recording of the derivatogram.[1,6] Actually, according to the results of the Pariser—Parr—Pople method, a substantial difference in the lengths of the single bonds* in the five-membered cycles of these ketones compared to the parameters of the remaining bonds is observed.[1,6]

In an effort to establish the accurate molecular geometry of indenoquinoline **2** and to determine the characteristic features of its crystal structure, we performed X-ray structural analysis of this compound. Unfortunately, we failed to solve the problems in hand in full measure owing to the observed partial disorder of molecule **2** in the crystal; this molecule is distributed over two sites (I and II) with substantially different occupancies (3/4 and 1/4, respectively). Molecules **2** (Fig. 2) overlap in the mirror-symmetry** fashion so that their mean planes coincide, while the outer phenylene cycles change places with each other, superimposing almost exactly. The central five- and six-membered cycles also change places; the methine C(5) atom of one molecule occupies the position of the methylene C(6) atom of the other molecule and *vise versa*; the positions of the central C(5a) atoms of both molecules coincide. Only the positions of the N (N(1) and N(1')) atom, one of the C (C(10b) and C(10')) atoms bonded to the N atom, and the methine and methylene H atoms of the central rings do not coincide (see Fig. 2).

Undoubtedly, due to the observed disorder, the determined geometric parameters should be considered critically because most of these parameters are virtually weighted mean values of two different bonds and angles of the superimposed molecules. However, inevitable errors decrease slightly because of a substantial differ-

* The C—CO bond lengths in the molecule of ketoindenoquinoline **3**[1,6] are in the range 1.461—1.463 Å; the remaining bond lengths are in the range 1.320—1.424 Å.

** The local symmetry plane is perpendicular to the mean plane of the molecules in positions I and II and passes through the C(5a) atom approximately through the middle of the N(1)—C(10b) bond.

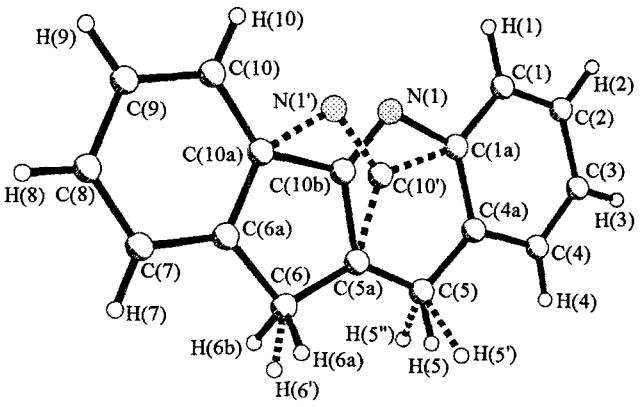
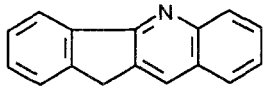


Fig. 2. The structure of disordered molecule **2**. Atomic numbering scheme is given for the molecules occupying the major position I. The portions of the molecule (located in the less occupied position II) that do not coincide with the corresponding portions of the molecule in the major position are shown by dashed lines; the corresponding atomic numbers are primed.

ence in the contributions of two positions (3 : 1). Therefore, the observed geometric parameters (Table 1) are determined mainly by the contribution of only one of molecules **2** located in the site I which is higher occupied.

An analysis of the bond lengths in molecule **2** provides evidence (despite the disorder) that the following form makes the largest contribution to the observed structure among the several possible resonance forms.



Actually, the structure of molecule **2** is characterized by a substantially localized N(1)=C(10b) bond (1.309(4) Å, see Table 1), the length of which is close to the length of a pure double bond (1.28 Å[11]), unlike that of the formally equivalent N(1)—C(1a) bond (1.413(4) Å), which is virtually identical to the tabulated value[11] for an unambiguously non-conjugated $C(sp^2)$—$N(sp^3)$ bond (1.416 Å). The C(5)=C(5a) bond in the heterocycle is also substantially localized (1.381(4) Å) compared to the formally equivalent C(5)—C(4a) bond (1.432(4) Å). In the five-membered cycle, of the three $C(sp^2)$—$C(sp^2)$ bonds, only one bond shared with the phenylene cycle (C(6a)—C(10a), 1.390(3) Å) exhibits a conjugated character, while the two remaining bonds are essentially elongated, the C(10a)—C(10b) bond being even longer (1.489(4) Å) than the $C(sp^2)$—$C(sp^3)$ bonds in the same cycle (1.459 and 1.487(4) Å). Apparently, this is evidence of disruption of the π conjugation in the tetracyclic framework of molecule **2** in the region of the partially saturated five-membered ring. It is reasonable to suggest that, when molecule **2** is deprotonated, the obtained completely aromatic carbanion should exhibit a more uniform π conjugated structure and the gain in energy due to electron delocalization should substantially assist dissociation of the methylene group of **2** thus causing the observed CH-acidic properties of the compound.

Although each cycle of molecule **2** is essentially planar [the maximum of the mean deviations of atoms from the planes of the corresponding cycles is observed in the benzene nucleus fused with the five-membered cycle (0.007 Å) and the minimum value is observed in the heterocycle (0.001 Å)], the molecule as a whole exhibits slight transverse folding: the dihedral angle between the mean planes of the outer phenylene cycles is 4.2°. The essentially planar structure of molecule **2** favors close packing in the crystal (the packing index for **2** calculated according to Kitaigorodskii[12] is 72.4 %);

Table 1. Geometric parameters of molecules of **2**

Bond	*d*/Å	Angle	ω/deg
C(1)—C(1a)	1.393(4)	C(1a)—C(1)—C(2)	119.9(3)
C(1)—C(2)	1.366(4)	C(1)—C(1a)—C(4a)	119.5(2)
C(1a)—C(4a)	1.416(4)	C(1)—C(1a)—C(10')	151.1(5)
C(1a)—N(1)	1.413(4)	C(4a)—C(1a)—C(10')	89.3(4)
C(1a)—C(10')	1.56(1)	C(1)—C(1a)—N(1)	111.7(3)
C(2)—C(3)	1.388(4)	C(4a)—C(1a)—N(1)	128.8(2)
C(3)—C(4)	1.365(4)	C(1)—C(2)—C(3)	121.2(3)
C(4)—C(4a)	1.411(4)	C(2)—C(3)—C(4)	120.6(3)
C(4a)—C(5)	1.432(4)	C(3)—C(4)—C(4a)	119.8(3)
C(5)—C(5a)	1.381(4)	C(1a)—C(4a)—C(4)	119.1(2)
C(5a)—C(6)	1.459(4)	C(1a)—C(4a)—C(5)	116.4(2)
C(5a)—C(10b)	1.442(4)	C(4)—C(4a)—C(5)	124.5(3)
C(5a)—C(10')	1.43(1)	C(4a)—C(5)—C(5a)	115.4(3)
C(6)—C(6a)	1.487(4)	C(5)—C(5a)—C(6)	132.0(2)
C(6a)—C(7)	1.386(3)	C(5)—C(5a)—C(10b)	123.2(2)
C(6a)—C(10a)	1.390(3)	C(6)—C(5a)—C(10b)	104.9(2)
C(7)—C(8)	1.371(4)	C(5)—C(5a)—C(10')	93.9(4)
C(8)—C(9)	1.385(4)	C(6)—C(5a)—C(10')	134.1(4)
C(9)—C(10)	1.378(4)	C(5a)—C(6)—C(6a)	106.8(2)
C(10)—C(10a)	1.395(4)	C(6)—C(6a)—C(7)	127.9(2)
C(10a)—C(10b)	1.489(4)	C(6)—C(6a)—C(10a)	112.4(2)
C(10a)—N(1')	1.466(8)	C(7)—C(6a)—C(10a)	119.7(2)
C(10b)—N(1)	1.309(4)	C(6a)—C(7)—C(8)	119.8(3)
C(10')—N(1')	1.35(1)	C(7)—C(8)—C(9)	120.9(3)
		C(8)—C(9)—C(10)	119.9(3)
		C(9)—C(10)—C(10a)	119.5(3)
		C(6a)—C(10a)—C(10)	120.1(2)
		C(6a)—C(10a)—C(10b)	103.3(2)
		C(10)—C(10a)—C(10b)	136.6(2)
		C(6a)—C(10a)—N(1')	142.0(4)
		C(10)—C(10a)—N(1')	97.8(4)
		C(5a)—C(10b)—C(10a)	112.7(2)
		C(5a)—C(10b)—N(1)	124.7(3)
		C(10a)—C(10b)—N(1)	122.6(3)
		C(1a)—C(10')—C(5a)	125.0(7)
		C(1a)—C(10')—N(1')	113.6(8)
		C(5a)—C(10')—N(1')	121.4(8)
		C(1a)—N(1)—C(10b)	111.6(3)
		C(10a)—N(1')—C(10')	103.3(7)

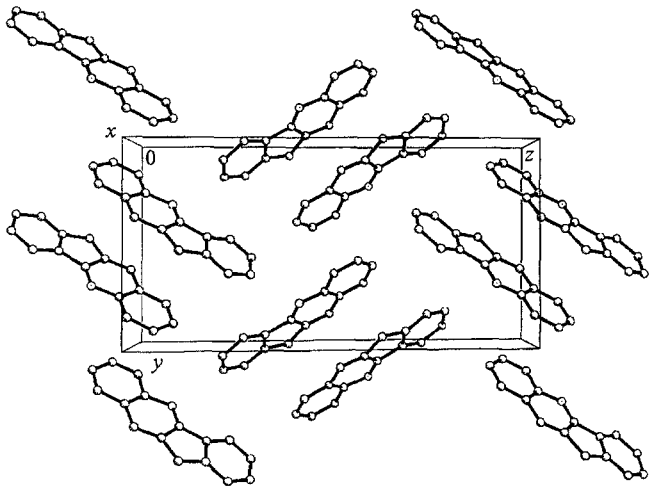


Fig. 3. The crystal structure of **2**. For simplicity, only molecules occupying positions I are shown.

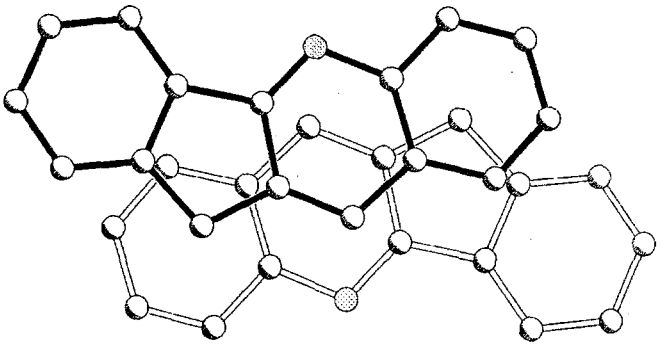


Fig. 4. The fashion of overlapping of molecules **2** in the dimeric pairs in the crystal (positions I related to each other by a type A center of symmetry).

parallel molecules are arranged in nonuniform stacks aligned along the *y* axis (Fig. 3). The molecules in the stacks are related to each other by centers of symmetry [[0 0 1/2]] (A) and [[0 1/2 1/2]] (B) (and by translationally equivalent centers). In the stacks, the dimers are separated because in the molecules related by type B centers of symmetry only the outer phenylene cycles of the π systems overlap; the planes of these cycles are separated by 3.62 Å. The π systems of the molecules related by type A centers of symmetry, on the contrary, overlap rather substantially (Fig. 4); the corresponding interplanar distance is 3.49 Å.

The disorder observed in the crystal suggests the possibility of alternative, though less favorable, ways in which the molecules of **2** in the dimer can overlap.

Analysis of the bond lengths observed in the molecule of indenoquinoline **2** also allows the single bonds at the methylene carbon atom to be considered weaker than the remaining bonds; this fact is in complete agreement with the qualitative energetic estimation of single bonds at the carbonyl carbon atoms of ketoindoquinolines, which we made based on the results of the quantum-chemical prediction. Therefore, high-temperature chemical conversions of indenoquinoline **2** and of the corresponding ketone **3** under the conditions in which the derivatograms were recorded should proceed with the participation of the single bonds at the C(6) atoms of these compounds. Taking into account the radical mechanism of the thermal oxidation of organic compounds[13] (the chemical conversions of compounds **2** and **3** in the course of thermal analysis should also be considered as processes of this type), it is believed that the formation of alcohol **2a** and acids **4a,b** during oxidative thermolysis of indenoquinoline **2** and ketone **3** should be preceded by the production of peroxide and hydroperoxide type radical intermediates formed primarily when the bonds at the C(6) atoms in indenoquinoline **2** and in ketone **3** are cleaved.

The observed pattern of molecular packing in indenoquinoline **2** is somewhat similar to the intercalation model for π interacting molecules in stacked structures, which has gained wide acceptance in studies of the mechanisms of binding of antitumor compounds, including compounds containing condensed nitrogen-containing heterocyclic systems with purine and pyrimidine bases of DNA.[14] The intercalation of some synthetic inducers of interferon in cellular nucleic acids is also considered to be one of the major stages in the mechanism of the genesis of interferon. In particular, the intercalation mechanism of action is assumed for 2,7-bis(2-diethylaminoethoxy)fluoren-9-one hydrochloride, known as tilorone, which is a highly active inducer of interferon in rodents.[15] A very high titer of interferon (*IU_{50}/mL = 640) is induced also by 2,7-bis(anabasinoacetylamino)fluoren-9-one.[4] However, according to the data in Ref. 4, the latter, like tilorone, does not intercalate in the DNA double helix but is located in the minor groove of the helix, forming complexes predominantly with AT base pairs. Evidently, the mentioned difference between tilorone and its anabasine analog in the titer of induced interferon (for tilorone, IU_{50}/mL = 320) and in the character of their binding with DNA is attributable only to the effect of the nature and volume of the substituents in their molecular topological base, the role of which is played in this case by the fluorenone framework.

If stacking structures are considered to be responsible for the probable mechanism of antitumor and interferon-inducing action (we hold this viewpoint), the selection of characteristics that would ensure the manifestation of these biological effects in the series of derivatives of CH-acids with an annelated indenyl fragment should be directed primarily to the occurrence of π—π

type bonding between these compounds and DNA. In our opinion, whatever the set of required features causing the antitumor and interferon-inducing action of compounds, the total overall effect of the biological action of these compounds amounts to the following: both types of biologically active compounds act (or should act according to predictions) as a wedge, preventing cross-linking of the helical turns of DNA along its axis and maintaining the size determined by nature (3.4 Å) of the minor groove of DNA. Therefore, antitumor compounds as well as inducers of interferon favor normal functioning of this survival process, DNA replication, thus enhancing the immunity of an organism to cancer and viral diseases.

Therefore, the results obtained in this work on estimation of thermal stability of indenoquinoline **2** and the characteristic features of its molecular and crystal structure lead to the following conclusions: (1) to attain optimum yields of fused benzoaza(diaza)fluorenes obtained by heterogeneous catalytic dehydrocyclization of *o*-methyl-substituted arylquinolines (-isoquinolines and -quinoxalines), contact masses exhibiting dehydrocyclizating ability at temperatures no higher than ~300 °C are required; (2) the revealed character of self-association of indenoquinoline **2** is similar to the association of π—π type of fused heterocyclic systems with π-excessive and π-deficient fragments, which allows fused nitrogen-containing CH-acids with an annelated indenyl cycle to be potential intercalators in the search for and study of the mechanism of the action of synthetic inducers of interferon and antitumor compounds. This conclusion is partially confirmed by the high antitumor activity of 2,2,8,9-tetramethoxy-11*H*-indeno[1,2-*c*]isoquinoline-5,11-dione determined recently by screening *in vivo*[14] as well as by the above-mentioned results.

Experimental

Compounds **1** (m.p. 74—75 °C, from hexane), **2** (m.p. 160—162 °C, from benzene), and **3** (m.p. 173—174 °C, from acetone) were synthesized according to the procedures reported previously.[1,6]

Thermogravimetric analysis of compounds **1—3** was performed on a MOM derivatograph (Hungary) in the temperature range 20—700 °C with an accuracy of 2—4 °C.

X-ray structural analysis was carried out on an automated four-circle Siemens P3/PC diffractometer (Mo-Kα radiation, a graphite monochromator, θ/2θ scanning technique, $(\sin\theta/\lambda)_{max} = 0.64$). Crystals of **2** are monoclinic. $a = 6.058(2)$, $b = 9.650(3)$, $c = 19.185(4)$ Å, $\beta = 94.17(2)°$, $V = 1119(1)$ Å^3, the space group is $P2_1/n$, $Z = 4$ ($C_{16}H_{11}N$), mol. weight is 217.26, $d_{calc} = 1.290$ g cm^{-3}. The structure was solved by the direct method and refined by the least-squares method with anisotropic temperature factors for non-hydrogen atoms. The H atoms and partially occupied (with $g = 25$ %) positions of non-hydrogen atoms of the disordered portion of the molecule were located from the difference Fourier synthesis and refined isotropically. Final values of the R factors are as follows: $R = 0.033$, $R_w = 0.035$, $S = 0.64$ using 924 reflections with $I > 3.5\sigma(I)$. The following weighting scheme was used: $w = 1/(\sigma^2(F) + 0.0044F^2)$. All calculations were performed on an IBM-PC/AT computer using the SHELXTL Plus program package.[16] Atomic coordinates are given in Table 2.

Table 2. Atomic coordinate (×10^4; ×10^3 for H atoms) and equivalent isotropic[a] (isotropic for H atoms) temperature parameters (×10^3) for the structure of **2**

Atom	*x*	*y*	*z*	*U*/Å^2
C(1)	−2573(6)	3484(3)	5019(2)	78(1)
C(1a)	−956(5)	2545(3)	5269(1)	68(1)
C(2)	−2397(5)	4119(3)	4390(1)	79(1)
C(3)	−625(5)	3850(3)	3991(2)	76(1)
C(4)	1000(5)	2946(3)	4221(1)	71(1)
C(4a)	869(5)	2269(2)	4869(1)	65(1)
C(5)	2454(5)	1284(3)	5156(2)	70(1)
C(5a)	2000(3)	724(2)	5792(1)	50(1)
C(6)	3171(5)	−295(4)	6243(1)	71(1)
C(6a)	1837(4)	−497(2)	6856(1)	61(1)
C(7)	2243(5)	−1403(3)	7411(1)	71(1)
C(8)	759(5)	−1501(3)	7915(1)	74(1)
C(9)	−1123(5)	−683(3)	7888(1)	76(1)
C(10)	−1528(5)	241(3)	7346(1)	73(1)
C(10a)	−55(4)	322(2)	6821(1)	62(1)
C(10b)[b]	74(5)	1090(3)	6152(2)	48(1)
C(10')[c]	2(18)	1495(10)	5834(5)	49(3)[d]
N(1)[b]	−1440(5)	1978(3)	5920(1)	56(1)
N(1')[c]	−1260(14)	1353(7)	6380(4)	54(2)[d]
H(1)	−386(5)	367(3)	533(2)	11(1)
H(2)	−357(5)	482(3)	424(2)	10(1)
H(3)	−52(4)	431(3)	355(1)	8(1)
H(4)	217(4)	271(3)	395(1)	8(1)
H(5)[b]	361(5)	108(3)	493(1)	2(1)
H(5')[c]	354(25)	−108(20)	604(9)	5[e]
H(6a)[b]	472(6)	−3(3)	638(2)	6(1)
H(6b)[b]	336(20)	−120(14)	597(6)	16(3)
H(6')[c]	442(13)	165(8)	508(4)	5[e]
H(6'')[c]	294(13)	44(8)	473(4)	5[e]
H(7)	356(5)	−197(3)	741(1)	7(1)
H(8)	100(5)	−213(3)	830(2)	9(1)
H(9)	−213(4)	−78(3)	825(1)	8(1)
H(10)	−277(4)	82(3)	734(1)	7(1)

[a] Equivalent isotropic parameters U were determined as one-third of the trace of the orthogonalized U_{ij} tensor.
[b] $g = 75$ %. [c] $g = 25$ %. [d] U_{iso}. [e] Fixed values.

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