<h1 style="text-align:center">STRUCTURES
OF ORGANIC COMPOUNDS</h1>

X-ray Mapping in Heterocyclic Design: VIII. Synthesis and X-ray Diffraction Study of Dimethyl 3-(*p*-Chlorobenzoyl)-5-Chloroindolizine-1,2-Dicarboxylate and the Product of Its Cyclization 1,2-Bis(carbomethoxy)-6-Chloro-3*H*-Isoquinolino[1,2,3-*d*,*c*]indolizine-3-one

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Abstract—Dimethyl 3-(*p*-chlorobenzoyl)-5-chloroindolizine-1,2-dicarboxylate, $C_{19}H_{13}Cl_2NO_5$, (**2**) and the product of its cyclization 1,2-bis(carbomethoxy)-6-chloro-3*H*-isoquinolino[1,2,3-*d*,*c*]indolizine-3-one, $C_{19}H_{12}ClNO_5$, (**3**) are synthesized, and their molecular and crystal structures are determined by the single-crystal X-ray diffraction technique. Crystals **2** are monoclinic, $a = 9.627(3)$ Å, $b = 6.646(2)$ Å, $c = 28.500(9)$ Å, $\beta = 98.72(2)°$, $Z = 4$, and space group $P2_1/c$. Crystals **3** are monoclinic, $a = 7.048(4)$ Å, $b = 10.582(4)$ Å, $c = 21.760(7)$ Å, $\beta = 97.23(4)°$, $Z = 4$, and space group $P2_1/c$. The structures are solved by the direct method and refined in the anisotropic approximation by the full-matrix least-squares procedure to $R = 0.0504$ and 0.0510 for **2** and **3**, respectively. In both structures, the intramolecular and intermolecular contacts involving the C, H, and O atoms are observed. © 2002 MAIK "Nauka/Interperiodica".

INTRODUCTION

This paper continues our structural investigations of heterocyclic compounds that are able to enter readily into various rearrangements and reactions of ring transformations [1–14]. As in the previous studies, we have performed the step-by-step structure determination of all the intermediates and the final products of the multistage cyclization and recyclization reactions. Note that data on the molecular structures discussed in this paper are not available in the Cambridge Structural Database [15].

EXPERIMENTAL

Synthesis. Indolizine **2** was synthesized by the reaction between the pyridinium salt **1** and dimethyl acetylenedicarboxylate according to the following scheme:

Scheme I
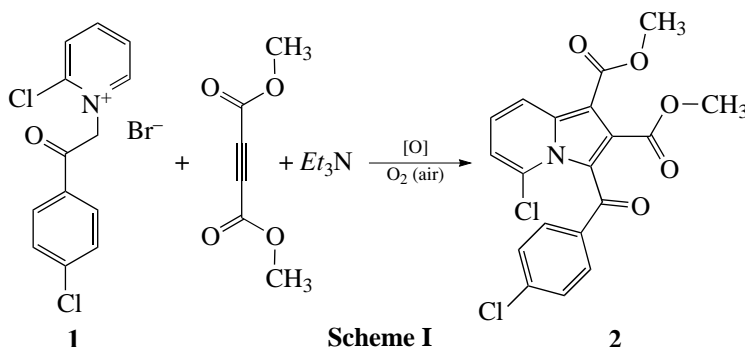
Salt **1** (1.5 g, 4.32 mmol) and dimethyl acetylenedicarboxylate (0.642 g, 4.52 mmol) were dissolved in dry dimethylformamide (*DMF*) (17 ml) on heating to 40°C. After the solution was cooled to room temperature, absolute *Et*₃N (0.496 g, 4.91 mmol) was added to it.

The resultant reddish brown solution was allowed to stand for a day at room temperature and was then poured into water (400 ml) on stirring. The precipitate was filtered off, washed with water (5 × 30 ml), and dried. The resultant greenish amorphous powder was

dissolved in a minimum amount of *Me*OH, the solution was passed through a column filled with SiO_2 (Silpearl, $l_{col} = 5$ cm, $d_{col} = 2$ cm, *Me*OH as an eluent) in order to remove impurities of resins and *DMF*, the eluate was evaporated to dryness, and the residue (1.70 g, 96.8%) was purified by column chromatography on SiO_2 (Silpearl, $l_{col} = 8$ cm, $d_{col} = 1.5$ cm, benzene : acetone = 10 : 1 as an eluent). The yellow substance obtained was indolizine **2**, $T_{mp} = 175$–177°C (*Me*OH). The yield was 0.71 g (40%). The ^1H NMR spectrum agrees with the formula assigned to this substance. Indolizine **2** is unstable: on long standing or under the effect of aluminum oxide, this compound undergoes an intramolecular cyclization. We found that the product of this cyclization has a tetracyclic structure of **3**:

2   **Scheme II**   **3**
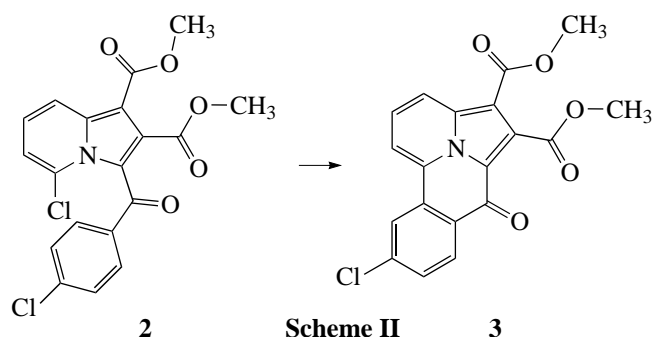
A weighed portion of **2** (20 mg) was dissolved in $CHCl_3$ (20 ml), Al_2O_3 [0.5 g; for chromatography, activity grade III (Brockman)] was added to the solution, and the solvent was evaporated to dryness. Aluminum oxide with the substance absorbed was allowed to stand at room temperature for two days and was then treated twice with $CHCl_3$ (20 ml). The extract was evaporated. The yield of **3** was 15 mg (83%); $T_{mp} = 193$–194°C.

X-ray diffraction analysis. The data sets for X-ray analysis were collected at room temperature on an Enraf–Nonius CAD4 four-circle automated diffractometer (MoK_α radiation, graphite monochromator, ω-2θ scan mode) [16]. The unit cell parameters were determined and refined using 25 reflections in the θ range 14°–16°. The crystal data for compounds **2** and **3** are summarized in Table 1. The crystals of the compounds studied are small in size and are characterized by small linear absorption coefficients; therefore, the empirical absorption correction was not applied.

The primary processing of the sets of diffraction data was performed with the WinGX98 program [17]. The structures were solved by the direct method. The coordinates and thermal parameters for all the non-hydrogen atoms were refined in the anisotropic approximation. All the calculations on the solution and refinement of the crystal structures were performed with the SHELX97 program package [18]. All the hydrogen atoms in both structures were located from the difference Fourier syntheses and refined in the isotropic approximation of thermal parameters. The atomic coordinates and equivalent thermal parameters for compounds **2** and **3** are listed in Tables 2 and 3, respectively. The spatial arrangement of atoms in molecules **2** and **3** and the atomic numbering, which were obtained with the PLUTON96 program [19], are shown in Figs. 1 and 2, respectively. For convenient comparison of the geometric parameters in the two molecules, we used a unified atomic numbering. The interatomic distances in structures **2** and **3** were calculated with the PARST95 program [20]. They are listed in Tables 4–6.

Table 1. Crystal data and details of the X-ray diffraction experiment and refinement for structures **2** and **3**

Compound	$C_{19}H_{13}Cl_2NO_5$ (**2**)	$C_{19}H_{12}ClNO_5$ (**3**)
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a, Å	9.627(3)	7.048(4)
b, Å	6.646(2)	10.582(4)
c, Å	28.500(9)	21.760(7)
β, deg	98.72(2)	97.23(4)
V, Å^3	1802.4(9)	1610(1)
Z	4	4
ρ_{calcd}, g/cm^3	1.497	1.525
μ(Mo, K_α), cm^{-1}	3.92	2.70
Crystal size, mm	$0.24 \times 0.12 \times 0.06$	$0.50 \times 0.20 \times 0.10$
θ_{max}, deg	25	26
Number of reflections with $I \geq 2\sigma(I)$/Number of parameters	1679/297	2538/284
R_1/wR_2	0.0504/0.0575	0.0510/0.0426
$\Delta\rho_{max}/\Delta\rho_{min}$, e/Å^3	0.173/–0.196	0.159/–0.166

Table 2. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters U_{eq} ($\text{Å}^2 \times 10^3$) for molecule **2**

Atom	x	y	z	U_{eq}/U_{iso}	Atom	x	y	z	U_{eq}/U_{iso}
Cl(1)	2392(1)	1572(2)	1586(1)	51(1)	C(14)	−2082(3)	4406(6)	−18(1)	61(2)
Cl(2)	−2706(1)	6002(2)	−483(1)	125(1)	C(15)	−1769(4)	2404(6)	−103(1)	59(2)
N(1)	133(2)	1089(4)	2015(1)	22(1)	C(16)	−1273(3)	1200(7)	261(1)	58(1)
O(1)	−4394(2)	1008(4)	2310(1)	61(1)	C(17)	−3213(3)	1239(6)	2494(1)	35(1)
O(2)	−2823(2)	1475(4)	2960(1)	45(1)	C(18)	−3903(3)	1447(6)	3252(1)	59(1)
O(3)	−4238(2)	2646(4)	1320(1)	61(1)	C(19)	−3452(3)	1222(7)	1384(1)	50(1)
O(4)	−3624(2)	−507(4)	1143(1)	58(1)	C(20)	−4803(4)	−559(7)	775(1)	91(2)
C(2)	−842(3)	1044(5)	1596(1)	35(1)	H(6)	−250(20)	1260(50)	3138(8)	56(9)
C(3)	−2147(3)	1194(5)	1736(1)	27(1)	H(7)	2200(20)	1300(40)	3237(7)	55(9)
C(4)	−1988(3)	1273(5)	2234(1)	24(1)	H(8)	3380(20)	1270(50)	2556(8)	49(9)
C(5)	−574(3)	1252(5)	2407(1)	26(1)	H(12)	−1170(20)	4480(40)	1115(9)	67(10)
C(6)	239(3)	1250(5)	2864(1)	30(1)	H(13)	−2090(20)	6600(40)	492(8)	62(10)
C(7)	1644(3)	1267(6)	2918(1)	39(1)	H(15)	−1920(20)	1840(40)	−431(8)	64(10)
C(8)	2358(3)	1258(5)	2519(1)	35(1)	H(16)	−1020(20)	−230(40)	202(9)	68(10)
C(9)	1585(3)	1246(5)	2073(1)	35(1)	H(18A)	−3460(20)	1650(40)	3612(8)	59(10)
C(10)	−523(3)	564(5)	1115(1)	48(1)	H(18B)	−4410(30)	70(50)	3218(9)	83(12)
O(10)	94(2)	−1033(4)	1057(1)	63(1)	H(18C)	−4620(20)	2620(40)	3150(7)	36(9)
C(11)	−1099(3)	1927(6)	731(1)	42(1)	H(20A)	−4860(20)	−1960(40)	609(8)	60(10)
C(12)	−1355(3)	3934(5)	800(1)	45(1)	H(20B)	−4700(20)	570(40)	533(8)	59(10)
C(13)	−1876(4)	5207(6)	433(1)	67(2)	H(20C)	−5720(20)	−300(50)	917(8)	69(11)

Table 3. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters U_{eq} ($\text{Å}^2 \times 10^3$) for molecule **3**

Atom	x	y	z	U_{eq}/U_{iso}	Atom	x	y	z	U_{eq}/U_{iso}
Cl(2)	8183(1)	7124(1)	2456(1)	71(1)	C(19)	7616(5)	1835(3)	−851(1)	45(1)
N(1)	7310(3)	5100(2)	−316(1)	33(1)	C(20)	9794(6)	143(3)	−930(2)	78(1)
C(2)	7676(4)	3821(2)	−208(1)	35(1)	O(1)	6997(3)	2793(2)	−2105(1)	71(1)
C(3)	7495(4)	3233(2)	−776(1)	35(1)	O(2)	6423(3)	4857(2)	−2251(1)	58(1)
C(4)	7071(4)	4123(3)	−1240(1)	36(1)	O(3)	6278(3)	1119(2)	−874(1)	59(1)
C(5)	6936(4)	5315(3)	−952(1)	36(1)	O(4)	9431(3)	1482(2)	−876(1)	53(1)
C(6)	6504(4)	6541(3)	−1150(1)	43(1)	O(10)	8355(3)	2242(2)	524(1)	56(1)
C(7)	6432(4)	7477(3)	−716(1)	44(1)	H(6)	6180(30)	6660(20)	−1640(9)	46(8)
C(8)	6822(4)	7211(3)	−87(1)	44(1)	H(7)	6030(40)	8350(20)	−887(11)	68(10)
C(9)	7261(4)	6015(3)	128(1)	34(1)	H(8)	6680(30)	7740(20)	187(9)	27(8)
C(10)	8063(4)	3369(3)	416(1)	40(1)	H(13)	7460(30)	7400(20)	1150(9)	41(8)
C(11)	8064(4)	4332(3)	905(1)	37(1)	H(15)	8870(30)	4560(20)	2426(8)	32(7)
C(12)	7691(4)	5598(3)	767(1)	33(1)	H(16)	8810(30)	2960(20)	1579(9)	32(7)
C(13)	7717(4)	6461(3)	1256(1)	40(1)	H(18A)	5660(40)	5490(30)	−3072(13)	116(14)
C(14)	8107(4)	6042(3)	1854(1)	44(1)	H(18B)	4990(40)	3920(30)	−3024(13)	102(12)
C(15)	8473(4)	4778(3)	1995(1)	50(1)	H(18C)	7460(40)	4280(30)	−3004(12)	83(12)
C(16)	8453(4)	3918(3)	1515(1)	47(1)	H(20A)	10940(50)	70(40)	−859(17)	190(20)
C(17)	6844(4)	3835(3)	−1899(1)	46(1)	H(20B)	9350(40)	−330(20)	−1336(11)	70(10)
C(18)	6145(6)	4660(4)	−2921(1)	78(1)	H(20C)	9200(40)	−400(30)	−606(12)	95(12)

Table 4. Interatomic distances d (Å) in structures **2** and **3**

| Bond	d (**2**)	d (**3**)	Bond	d (**2**)	d (**3**)
Cl(1)–C(9)	1.706(3)		C(10)–O(10)	1.239(4)	1.229(3)
Cl(2)–C(14)	1.732(4)	1.736(3)	C(10)–C(11)	1.464(4)	1.473(4)
N(1)–C(9)	1.386(3)	1.370(3)	C(11)–C(16)	1.412(4)	1.392(4)
N(1)–C(2)	1.402(3)	1.392(3)	C(11)–C(12)	1.376(5)	1.391(4)
N(1)–C(5)	1.399(3)	1.396(3)	C(12)–C(13)	1.380(5)	1.401(4)
C(2)–C(3)	1.378(4)	1.376(3)	C(13)–C(14)	1.378(5)	1.368(3)
C(2)–C(10)	1.484(4)	1.432(3)	C(14)–C(15)	1.393(5)	1.389(4)
C(3)–C(4)	1.406(4)	1.385(3)	C(15)–C(16)	1.339(5)	1.385(4)
C(3)–C(19)	1.484(4)	1.492(4)	C(17)–O(1)	1.187(3)	1.200(3)
C(4)–C(5)	1.376(3)	1.416(4)	C(17)–O(2)	1.336(3)	1.337(3)
C(4)–C(17)	1.483(4)	1.456(3)	O(2)–C(18)	1.425(4)	1.460(3)
C(5)–C(6)	1.413(3)	1.389(4)	C(19)–O(3)	1.208(5)	1.206(3)
C(6)–C(7)	1.339(4)	1.374(4)	C(19)–O(4)	1.336(5)	1.341(3)
C(7)–C(8)	1.416(4)	1.392(4)	O(4)–C(20)	1.425(4)	1.447(4)
C(8)–C(9)	1.370(4)	1.370(4)	C(9)–C(12)		1.456(3)

Table 5. Parameters of intramolecular and intermolecular contacts in structure **2**

D–H	$d(D–H)$, Å	$d(D \cdots A)$, Å	$d(H \cdots A)$, Å	$\omega(D–H \cdots A)$, deg	A	Symmetry operation
C(6)–H(6)	0.97(3)	3.005(4)	2.45(2)	116(2)	O(2)	[x; y; z]
C(16)–H(16)	1.00(3)	2.860(4)	2.57(3)	97(2)	O(10)	[x; y; z]
C(7)–H(7)	0.98(2)	3.721(4)	2.96(3)	135(2)	O(4)	[$-x$; $y + 1/2$; $1/2 - z$]
C(6)–H(6)	0.97(3)	3.622(4)	2.90(3)	132(2)	O(10)	[$-x$; $y + 1/2$; $1/2 - z$]
C(8)–H(8)	0.98(2)	3.285(4)	2.36(2)	156(2)	O(1)	[$x + 1$; y; z]
C(13)–H(13)	0.97(3)	3.461(4)	2.91(2)	117(2)	O(10)	[x; $y + 1$; z]
C(15)–H(15)	1.00(2)	3.486(5)	2.74(3)	132(2)	O(10)	[$-x$; $-y$; $-z$]
C(18)–H(18B)	1.04(3)	3.426(4)	2.56(3)	141(2)	O(3)	[$-x - 1$; $y - 1/2$; $1/2 - z$]
C(18)–H(18C)	1.05(2)	3.694(4)	2.71(2)	157(2)	O(1)	[$-x - 1$; $y + 1/2$; $1/2 - z$]

RESULTS AND DISCUSSION

The transformation of pyridinium salt **1** into indolizine **2** provides an example of 1,3-dipolar cycloaddition, which is characteristic of *N*-phenacylpyridinium ylides of the **4a** type. The specific feature of the reaction described is the regioselective cycloaddition of acetylene dienophile through the formation of adduct **4b** (followed by oxidation to indolizine **2**):

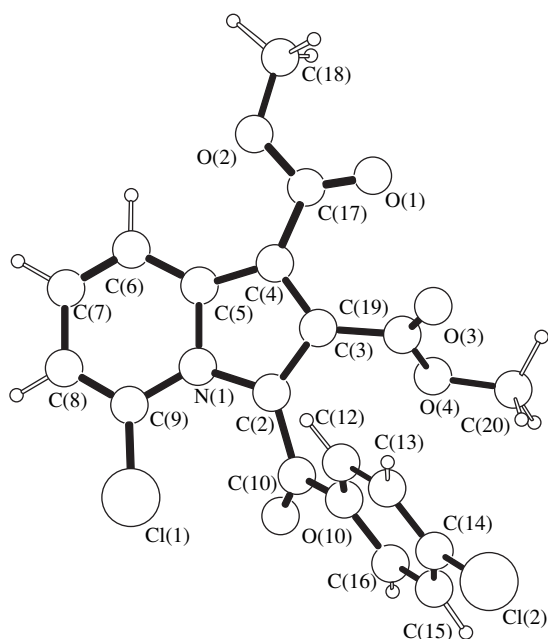E = COO*Me*, Ar = *p*-Cl*Ph*
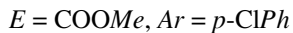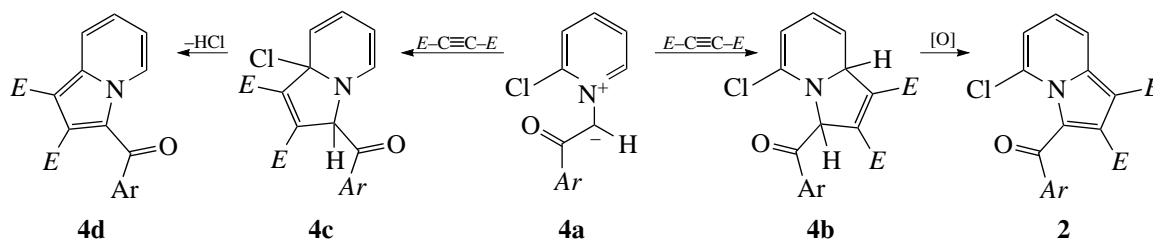
Scheme III

Fig. 1. Structure of molecule **2** and the atomic numbering.
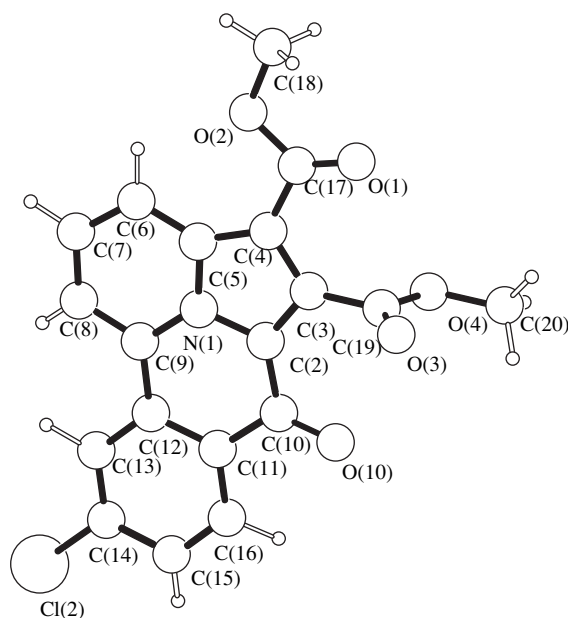

Fig. 2. Structure of molecule **3** and the atomic numbering.

An alternative cycloaddition resulting in the formation of cycloadduct **4c** would necessarily be followed by the aromatization of this structure to indolizine **4d**. Our special experiments (independent synthesis of indolizine **4d** and comparison of its chromatographic behavior with that of trace contaminants that are formed in the reaction mixture) revealed that indolizine **4d** is not formed even in trace amounts.

In the structure of heterocycle **2**, the indolizine bicycle is planar (the atomic deviations from the rms plane are within 0.06 Å). The dihedral angle between the planes of the indolizine nucleus of the molecule and the phenyl ring of the benzoyl group is 75.1(1)°. The O(10) atom deviates from the plane of the phenyl ring and the plane of the indolizine bicycle by 0.366(6) and 1.372(5) Å, respectively. The dihedral angles formed by the planar C(17)O(1)O(2)C(18) and C(19)O(3)O(4)C(20) ester radicals (the atomic deviations from each of the rms planes are within 0.1 Å) with

the nine-membered bicycle are equal to 7.0(1)° and 66.8(1)°, respectively. Earlier [21], we studied the crystal structure of dimethyl 3-(*p*-nitrobenzoyl)-5-chloroindolizine-1,2-dicarboxylate, $C_{19}H_{13}Cl_1N_2O_7$. The similarity of the structural fragments of these two compounds allows us to compare their geometric parameters. In these molecules, the O(1)C(17)C(4)C(3) torsion angles of the C(17)O(1)O(2)C(18) ester groups are +5.3(6)° (**2**) and –176.2(2)° [21] and the O(3)C(19)C(3)C(4) torsion angles of the C(19)O(3)O(4)C(20) ester groups are +66.0(6)° (**2**) and –98.3(3)° (*p*-nitro derivative [21]). This reorientation of the ester fragments is not accompanied by changes in their geometric charecteristics; that is, their bond lengths and angles are equal within standard deviations. The same tendency is observed in the molecule as a whole.

The tetracyclic product **3** is formed from indolizine **2** through the formation of the C–C bond between the

Table 6. Parameters of intramolecular and intermolecular contacts in structure **3**

D–H	$d(D$–H), Å	$d(D \cdots A)$, Å	$d(H \cdots A)$, Å	$\omega(D$–H$\cdots A)$, deg	A	Symmetry operation
C(6)–H(6)	1.07(2)	2.981(3)	2.35(2)	116(1)	O(2)	[x; y; z]
C(16)–H(16)	1.05(2)	2.786(3)	2.40(2)	100(1)	O(10)	[x; y; z]
C(7)–H(7)	1.02(2)	3.870(4)	2.94(2)	152(2)	O(3)	[x; $y + 1$; z]
C(8)–H(8)	0.83(2)	3.663(4)	2.97(2)	142(2)	O(3)	[$1 - x$; $1 - y$; $-z$]
C(13)–H(13)	1.03(2)	3.143(4)	2.62(2)	111(1)	O(4)	[$2 - x$; $1 - y$; $-z$]
C(18)–H(18*A*)	0.99(3)	3.324(4)	2.60(3)	130(2)	O(3)	[$1 - x$; $1/2 + y$; $-z - 1/2$]
C(20)–H(20*C*)	1.04(3)	2.927(4)	2.59(3)	98(2)	O(10)	[$2 - x$; $-y$; $-z$]

benzoyl group and the pyridine fragment of indolizine followed by dehydrohalogenation. The mechanism of this transformation is somewhat unusual. Although the chlorine atom in the α position with respect to the nitrogen atom of the pyridine fragment is probably rather mobile, it is difficult to assume that the chlorine atom is replaced by the benzoyl fragment according to the mechanism of aromatic nucleophilic substitution (it is apparent that the benzoyl group in the *ortho* position is not nucleophilic). Therefore, it is highly improbable that the reaction mechanism includes the formation of intermediate **5a**:
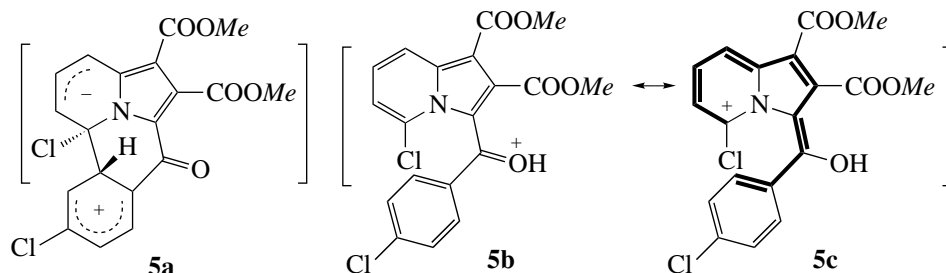
Scheme IV

The only reasonable explanation for the mechanism of the cyclization observed is provided by the following hypothesis. Indolizine **2** undergoes cyclization under the effect of either Al_2O_3 or acids (as was observed in our special experiments). Acids (as well as the acid OH groups, which are always present in aluminum oxide) protonate the benzoyl group of indolizine **2** to form cation **5b** (scheme IV). This direction of protonation of 3-acylindolizines is well known [22]. In this case, the indolizine skeleton and the adjacent benzoyl fragment form a common system (shown by heavy lines in intermediate **5c**) that consists of eleven atoms and contains a total of ten π electrons. Evidently, the ten-electron system can undergo a pericyclic cyclization reaction, which is allowed by the Woodward–Hoffmann rules. This unusual 1,11-cyclization would result (upon the detachment of HCl) in the formation of tetracycle **3**.

The tetracyclic system **3** is planar and consists of 19 atoms, including C(17) and C(19) (Fig. 2). The rms atomic deviations from this plane are within 0.036(3) Å. The ester radicals are also planar (the rms atomic deviations are within 0.003 Å in each of them), and their arrangement is quite different from that in **2**: the C(17)O(1)O(2)C(18) group is almost coplanar with the heterocyclic system [the dihedral angle is 0.68(7)°], whereas the C(19)O(3)O(4)C(20) group is situated almost perpendicularly to the latter system [the dihedral angle is 88.13(9)°]. This position of the ester groups with respect to the heterocyclic nucleus of the molecule is due to the minimum repulsion of the O(10) and O(1) atoms from the O(3) and O(4) atoms, respectively.

In molecules **2** and **3**, the intramolecular C(6)–H(6)···O(2) and C(16)–H(16)···O(10) hydrogen bonds have almost identical geometric parameters (Tables 5, 6). The parameters of the intermolecular contacts involving the C, H, and O atoms are also included in these tables.

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