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4-HYDROXY-2-QUINOLONES
170*. SYNTHESIS AND BROMINATION OF N-ALLYLISATIN

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Independently of the reaction conditions N-allylisatin forms only the 2,3-dibromo derivative upon treatment with molecular bromine in contrast to N-allyl-substituted quinolin- or pyrid-2-ones which readily undergo halocyclization to the corresponding 2-bromomethyl oxazoles.

Keywords: N-allylisatin, alkylation, bromination, halocyclization, X-ray structural analysis.

Decolorization of bromine is one of the widest known analytical reactions for alkenes but does not always prove to be due to the conventional addition of halogen to an unsaturated bond. With the existence of certain structural features such reactions can be accompanied by the formation of novel 5-, 6- and even 7-membered rings thanks to which they are often referred to as bromo- or halocyclizations [2-7].

This type of chemical reaction occurs in the bromination of N-allyl-substituted 4-hydroxy-2-quinolones which is, in principle, a simple method for preparing 4R-2-bromomethyl-1,2-dihydrooxazolo[3,2-*a*]quinolines [8-10]. For evaluating the synthetic potential of this interesting reaction and to work out a fuller and more proven idea of the actual mechanism occurring in these chemical processes there is much value in the accumulation and subsequent analysis of experimental material including the maximum use of a broad range of model compounds.

This report is a part of such an investigation, the aim of which was a study of the behavior of N-allylisatin **1** (with a structural similarity to 4-hydroxy-2-quinolones) under conditions of bromination using molecular bromine. According to a known outline [11], the alkylation of isatin **2** principally needs its conversion to a sodium salt and used sodium ethylate in absolute ethanol or, indeed, NaH in anhydrous DMF. Preference is usually given to the latter variant since, in this case, the initial separation of the salt is not essential. However, from our data, the use of this method is far from always justified. At least with allyl bromide this reaction is much more conveniently carried out using the system DMSO/K_2CO_3. The N-alkylation occurs very rapidly at room temperature and in virtually quantitative yield.

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* For Communication 169 see [1].

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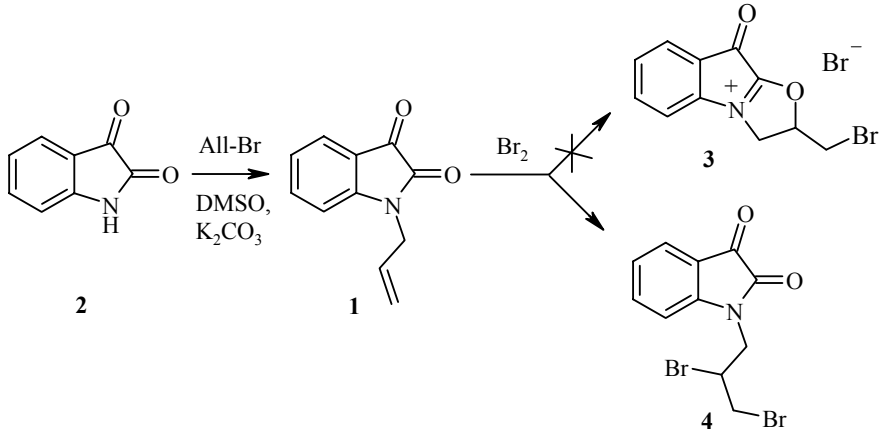
As had been expected, the N-allylisatin **1** was brominated very readily by molecular bromine in glacial acetic acid, the brown color of the bromine disappearing immediately after mixing the reagents. In principle this reaction can occur by two alternative routes via halocyclization to 2-bromomethyl-9-oxo-3,9-dihydro-2H-oxazolo[3,2-*a*]indol-4-ylium bromide (**3**) or by the simple addition of the bromine to the allyl double bond giving N-(2,3-dibromopropyl)isatin (**4**). A parallel formation of both of these compounds cannot be ruled out.



A positive Beilstein test and TLC monitoring showed that the bromination of the N-allylisatin **1** occurs selectively since a single product was obtained. Both possible bromo-substituted products **3** and **4** contain the same number of protons with quite similar chemical environments. For this reason it was not possible to establish the true structure of the compound formed on the basis of just a simple ^{1}H NMR spectrum.

An unambiguous solution to this structural problem was achieved through X-ray structural analysis (see Figure 1 and Tables 1 and 2). It was found that the product obtained is N-(2,3-dibromopropyl)isatin (**4**). The independent part of the unit cell for this compound consist of two molecules (**A** and **B**) differing in several geometric parameters. For both molecules the bicyclic fragment and atoms O(1), O(2), and C(9) lie in a single plane to within an accuracy of 0.02 Å. The C(7)–C(8) (1.558(6) in molecule **A** and 1.538(6) Å in **B** is somewhat lengthened when compared with the mean value [12] for a C_{sp2}–C_{sp2} bond of 1.455 Å and this has also been observed previously in studied isatins [13-16].

The repulsion between the substituent on the N(1) atom and the benzene ring with a shortened intramolecular contact H(10)···C(2) of 2.85 Å in **A** and 2.82 Å in **B** (sum of van der Waal radio [17] = 2.87 Å) leads to the 2,3-dibromopropyl fragment being positioned virtually perpendicularly to the bicyclic ring plane (torsional angle C(1)–N(1)–C(9)–C(10) 72.9(5)º in **A** and 71.6(5)º in **B**) and occurs in an *ap*-conformation (torsional angle N(1)–C(9)–C(10)–C(11) = -171.6(4)º in **A** and -168.5(3)º in **B**). The bromine atoms occupy a mutual +*sc*-conformation to one another (torsional angle Br(1)–C(10)–C(11)–Br(2) 66.2(3)º in **A** and 68.7(3)º in **B**). A shortened intramolecular contact for H(9a)···Br(2) of 2.95 in molecule **A** and 2.94 Å in **B**.

A series of shortened intermolecular contacts is seen for the **A** and **B** molecules in the crystal of compound **4**: H(3a)···Br(2a)' (0.5-*x*, -0.5+*y*, 0.5-*z*) 3.12 (3.23); H(4a)···Br(1b)' (-0.5+*x*, 0.5-*y*, 0.5+*z*) 3.03 (3.23); H(11b)···Br(2b)' (0.5-*x*, -0.5+*y*, -0.5-z) 3.11 (3.23); H(3b)···Br(1)' (0.5-*x*, -0.5+*y*, -0.5-*z*) 3.09 (3.23); H(3b)···Br(2b)' (0.5-*x*, -0.5+*y*, -0.5-*z*) 3.10 (3.23); H(4b)···Br(1a)' (0.5+*x*, 0.5-*y*, -0.5+*z*) 3.17 (3.23); H(11c)···Br(1a)' 3.05 (3.23); H(11a)···Br(2a)' (-0.5+*x*, 0.5-*y*, -0.5+*z*) 2.99 (3.23); Br(1a)···Br(2b)' 3.73 (3.94); Br(1a)···Br(2b)' (-*x*, 1-*y*, -*z*) 3.57 (3.94) and Br(2a)···Br(1b)' (1-*x*, 1-*y*, -*z*) 3.74 Å (3.94 Å).

Hence the bromination of the N-allylisatin **1** at room temperature in glacial acetic acid occurs exclusively *via* a classical addition of halogen to the double bond. Bearing in mind that the course of a similar reaction is sometimes significantly affected by the solvent and temperature used [18, 19] we additionally carried out this synthesis in carbon tetrachloride at -20ºC. However, in this case only compound **4** was formed.

One obstacle to bromocyclization of N-allylisatin **1** to the oxazoloindole **3** can firstly be identified as relating to the size of the ring (already having several stresses in its pyrrole fragment) when attaching a further five-membered ring to it and so making it energetically less favored than the process of forming the dibromo derivative **4**. However, a more detailed analysis shows that this quite logical explanation, at first glance, does not agree with numerous experimental results. In particular, it is well known that successful halocyclizations can accompany the formation of heterocyclic systems of this type, i.e. those containing five-membered rings mutually annelated [2, 18-24]. On the other hand, it turns out that not each six-membered 1-N-allyl-substituted

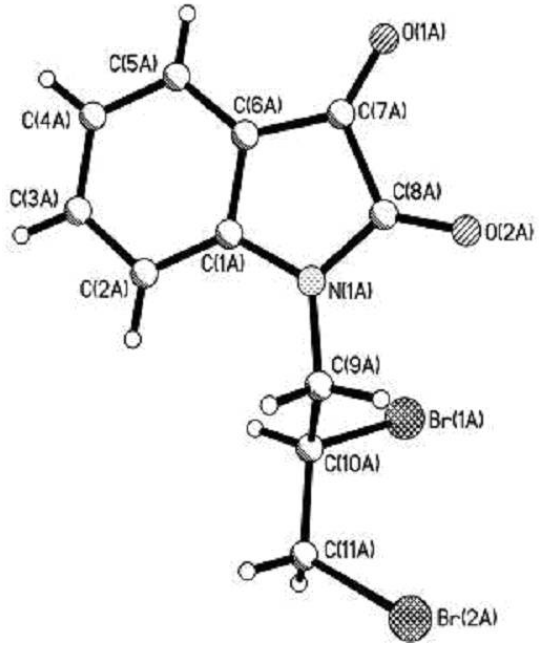


Fig. 1. Structure of the N-2,3-dibromopropylisatin (**4**) structure with atomic numbering.

TABLE 1. Bond Lengths (*l*) in the N-(2,3-Dibromopropyl)isatin (**4**) Structure

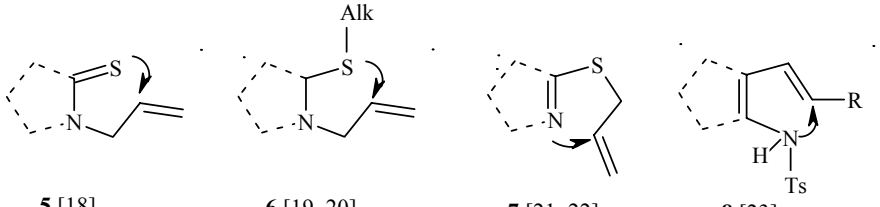
Bond	*l*, Å	Bond	*l*, Å
Br(1A)–C(10A)	1.952(4)	Br(2A)–C(11A)	1.937(5)
N(1A)–C(8A)	1.366(5)	N(1A)–C(1A)	1.411(5)
N(1A)–C(9A)	1.443(5)	O(1A)–C(7A)	1.201(5)
O(2A)–C(8A)	1.210(5)	C(1A)–C(2A)	1.370(6)
C(1A)–C(6A)	1.406(6)	C(2A)–C(3A)	1.390(7)
C(3A)–C(4A)	1.373(7)	C(4A)–C(5A)	1.382(6)
C(5A)–C(6A)	1.385(6)	C(6A)–C(7A)	1.450(6)
C(7A)–C(8A)	1.558(6)	C(9A)–C(10A)	1.522(5)
C(10A)–C(11A)	1.497(6)	Br(1B)–C(10B)	1.961(4)
Br(2B)–C(11B)	1.945(4)	N(1B)–C(8B)	1.369(5)
N(1B)–C(1B)	1.427(5)	N(1B)–C(9B)	1.450(5)
O(1B)–C(7B)	1.196(5)	O(2B)–C(8B)	1.202(5)
C(1B)–C(2B)	1.372(5)	C(1B)–C(6B)	1.382(6)
C(2B)–C(3B)	1.376(6)	C(3B)–C(4B)	1.382(6)
C(4B)–C(5B)	1.376(6)	C(5B)–C(6B)	1.381(6)
C(6B)–C(7B)	1.468(5)	C(7B)–C(8B)	1.538(6)
C(9B)–C(10B)	1.522(5)	C(10B)–C(11B)	1.474(6)

TABLE 2. Valence Angles (ω) in the Compound **4** Structure

Angle	ω, deg	Angle	ω, deg
C(8A)–N(1A)–C(1A)	110.8(4)	C(8A)–N(1A)–C(9A)	124.4(4)
C(1A)–N(1A)–C(9A)	124.7(4)	C(2A)–C(1A)–C(6A)	121.2(4)
C(2A)–C(1A)–N(1A)	128.4(5)	C(6A)–C(1A)–N(1A)	110.4(4)
C(1A)–C(2A)–C(3A)	116.5(5)	C(4A)–C(3A)–C(2A)	123.3(5)
C(3A)–C(4A)–C(5A)	120.1(5)	C(4A)–C(5A)–C(6A)	117.9(5)
C(5A)–C(6A)–C(1A)	121.0(4)	C(5A)–C(6A)–C(7A)	131.3(5)
C(1A)–C(6A)–C(7A)	107.7(4)	O(1A)–C(7A)–C(6A)	130.8(5)
O(1A)–C(7A)–C(8A)	124.2(4)	C(6A)–C(7A)–C(8A)	105.0(4)
O(2A)–C(8A)–N(1A)	127.5(4)	O(2A)–C(8A)–C(7A)	126.4(4)
N(1A)–C(8A)–C(7A)	106.1(4)	N(1A)–C(9A)–C(10A)	113.5(3)
C(11A)–C(10A)–C(9A)	114.3(4)	C(11A)–C(10A)–Br(1A)	111.0(3)
C(9A)–C(10A)–Br(1A)	109.4(3)	C(10A)–C(11A)–Br(2A)	114.7(3)
C(8B)–N(1B)–C(1B)	110.7(4)	C(8B)–N(1B)–C(9B)	124.4(4)
C(1B)–N(1B)–C(9B)	124.9(3)	C(2B)–C(1B)–C(6B)	122.2(4)
C(2B)–C(1B)–N(1B)	127.5(4)	C(6B)–C(1B)–N(1B)	110.3(4)
C(1B)–C(2B)–C(3B)	116.9(4)	C(2B)–C(3B)–C(4B)	122.6(4)
C(5B)–C(4B)–C(3B)	119.2(5)	C(4B)–C(5B)–C(6B)	119.6(4)
C(5B)–C(6B)–C(1B)	119.5(4)	C(5B)–C(6B)–C(7B)	132.8(4)
C(1B)–C(6B)–C(7B)	107.7(4)	O(1B)–C(7B)–C(6B)	130.1(5)
O(1B)–C(7B)–C(8B)	124.5(4)	C(6B)–C(7B)–C(8B)	105.4(4)
O(2B)–C(8B)–N(1B)	127.5(4)	O(2B)–C(8B)–C(7B)	126.6(4)
N(1B)–C(8B)–C(7B)	105.9(4)	N(1B)–C(9B)–C(10B)	113.0(3)
C(11B)–C(10B)–C(9B)	114.4(3)	C(11B)–C(10B)–Br(1B)	111.8(3)
C(9B)–C(10B)–Br(1B)	109.4(3)	C(10B)–C(11B)–Br(2B)	114.5(3)

2-oxoheterocycle can undergo halocyclization during bromination*. Hence the failure we have experienced when trying to transform the N-allyl derivative **1** to oxazoloindole **3** called for totally different reasoning.

Attention was turned to the fact that bicyclics of the type discussed are only readily formed in those conditions where a sulfur or nitrogen atom takes part in the halocyclization (see structures **5-8**). Closing of the second ring at an oxygen atom is encountered very rarely and then as the result of the iodination of carboxylic



5 [18] **6** [19, 20] **7** [21, 22] **8** [23]

9 [24] **10** [25] **11**

*We have shown that the reaction of 1-allyl-3-(2-hydroxyethyl)-1H-quinazoline-2,4-dione with molecular bromine in glacial acetic acid also occurs with addition of the halogen to the allyl double bond, i.e. the only product is the corresponding 1-N-(2,3-dibromompropyl) derivative.

acids containing an olefine bond (structure **9**). A feature of such reactions is the participation of the oxygen atom of a hydroxyl group in the cyclization to form lactones in the final step, hence being referred to as iodolactonization. But here examples of the formation of similar bicycles involving the carbonyl oxygen atom are generally unsuccessful.

In the halocyclization reactions the carbonyl oxygen atom is thought to become a nucleophilic center *via* the formation of stable α-deprotonation products, i.e. the hydroxy form [2]. For N-allylisatin **1** a similar reaction is impossible in principle and this might be taken as a quite convincing reason for the failure of bromocyclization. However, in the case of 1-allyl-4-methyl-2-oxo-1,2-dihydroquinoline-3 carboxylic acid (**10**), which is also unable to form a 2-hydroxy derivative, its brominative cyclization to 2-bromomethyl-4-carboxy-5-methyl-1,2-dihydrooxazolo[3,2-*a*]quinolinium bromide is in no way hindered. This was explained by X-ray analysis which showed a marked contribution of the bipolar aromatic form **11** in the resonance hybrid [25].

These facts allow us to propose that the route of the reaction studied by us depends to a marked extent not so much on the sizes of the rings created or the possibility of enol forms as on a dependence of the structure of the molecule as a whole on the polarizability of potential nucleophilic centers, i.e. the sulfur, nitrogen, or oxygen atoms. For isatins the contribution of a polar form of type **11** is evidently not large, hence bromination of its N-allyl-substituted derivative **1** occurs only *via* the usual addition of bromine to an allyl double bond. It is likely that, for this reason, the potassium or sodium salts of isatin (even though formally ambident nucleophiles) are none the less alkylated exclusively at the nitrogen atom. For comparison, the pyrid- and quinolin-2-ones (the 1-N-allyl-substituted derivatives of which are brominated very readily) form a mixture of the N- and O-alkyl-substituted isomers under the same conditions [26, 27]. In other words, the polarizability of the 2-carbonyl oxygen atoms in the pyrid- and quinolin-2-ones is markedly greater than in isatins and this particular feature is reflected in their chemical properties.

EXPERIMENTAL

^{1}H NMR spectra for the compounds synthesized were recorded on a Varian Mercury-VX-200 (200 MHz) instrument using DMSO-d_6 solvent and TMS as internal standard.

N-Allylisatin 1. Finely divided K_2CO_3 (2 g) was added to a solution of isatin **2** (1.47 g, 0.01 mol) in DMSO (15 ml), stirred, and treated with allyl bromide (0.93 ml, 0.011 mol). The reaction product changed color from violet to red virtually instantaneously, after which it was diluted with cold water. The bright red precipitate of N-allylisatin **1** was filtered off, washed with cold water, and dried. Yield 1.83 g (98%); mp 86-88ºC (ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.62 (1H, t, *J* = 7.8, H-6); 7.54 (1H, d, *J* = 7.3, H-4); 7.11 (1H, t, *J* = 7.6, H-5); 7.04 (1H, d, *J* = 8.0, H-7); 5.84 (1H, m, C<u>H</u>=CH_2); 5.31 (1H, d, *J* = 17.3, NCH_2CH=C<u>H</u> *trans*); 5.16 (1H, d, *J* = 10.4, NCH_2CH=C<u>H</u> *cis*); 4.28 (2H, d, *J* = 4.8, NCH_2).

N-(2,3-Dibromopropyl)isatin (4). A. Bromine (0.52 ml, 0.01 mol) was added with stirring to a solution of N-allylisatin **1** (1.87 g, 0.01 mol) in acetic acid (20 ml) and the color disappeared immediately. The reaction mixture was diluted with water. The red precipitate was filtered off, washed with cold water, and dried. R_f 0.48 (Silufol UV-254, hexane–CH_2Cl_2–acetone, 5:5:1). Yield 3.26 g (90%); mp 129-131ºC (ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.67 (1H, tt, *J* = 7.8 and 1.4, H-6); 7.56 (1H, dt, *J* = 7.3 and 1.4, H-4); 7.25 (1H, dd, *J* = 7.8 and 2.0, H-7); 7.13 (1H, tt, *J* = 7.5 and 1.3, H-5); 4.67 (1H, q, *J* = 6.4, NCH_2C<u>H</u>Br); 4.15 (2H, d, *J* = 7.1, NCH_2); 4.02 (2H, dd, *J* = 5.9 and 2.1, CH_2Br). Found, %: C 37.93; H 2.50; N 4.13. $C_{11}H_9Br_2NO_2$. Calculated, %: C 38.07; H 2.61; N 4.04.

B. A solution of bromine (0.52 ml, 0.01 mol) in CCl_4 (20 ml) was added with vigorous stirring and cooling to -20ºC to a solution of N-allylisatin **1** (1.87 g, 0.01 mol) in CCl_4 (150 ml) also cooled to -20ºC. After addition of all of the bromine the cooling was removed. Solvent was removed under reduced pressure to a volume of ~ 30 ml then cooled. The red precipitate was filtered and dried. Yield 3.40 g (94%). A mixed sample

with compound **4** made by method A did not give a depression of melting point. The ^{1}H NMR spectra and R_f values of these compounds were identical.

X-ray Crystallographic Investigation. Crystals of N-(2,3-dibromopropyl)isatin (**4**) were monoclinic (acetone). At 20ºC: a = 12.988(2), b = 13.013(3), c = 15.092(3) Å, β = 113.41(2)º, V = 2340.7(7) Å^3, M_r = 347.01, Z = 8, space group $P2_1/n$, d_{calc} = 1.969 g/cm^3, μ(MoKα) = 6.910 mm^{-1}, F(000) = 1344. The unit cell parameters and intensities of 12819 reflections (4071 independent with R_{int} = 0.033) were measured on an Xcalibur-3 diffractometer (MoKα radiation, CCD detector, graphite monochromator, ω-scanning to $2\theta_{max}$ = 50º). The absorption was calculated by a semiempirical method from the results of multiscanning (T_{min} = 0.208, T_{max} = 0.501).

The structure was solved by a direct method using the SHELXTL program package [28]. The positions of the hydrogen atoms were revealed from electron density difference synthesis and refined using the "riding" model with U_{iso} = 1.2 U_{eq} for a non-hydrogen atom bound to the given hydrogen. The structure was refined *via* F^2 full-matrix least-squares analysis in the anisotropic approximation for non-hydrogen atoms to wR_2 = 0.071 for 4054 reflections (R_1 = 0.034 for 2430 reflections with $F>4\sigma(F)$ and S = 0.838). The full crystallographic information was placed in the Cambridge Structural Data Base (reference CCDC 717535). Interatomic distances and valence angles are given in Tables 1 and 2.

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