Chem. Ber. 119, 3316–3325 (1986)

The Condensation Product of 2-Aminophenol and Glyoxal. Structure and Photochemistry

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Received June 25, 1986

It has been shown by *X*-ray analysis that the condensation product of 2-aminophenol and glyoxal is 5a,6,11a,12-tetrahydro[1,4]benzoxazino[3,2-*b*][1,4]benzoxazine (**5**) and not, as generally assumed[1], 2,2'-bibenzoxazoline (**4**). The structure of **5** is preserved if it is dissolved in neutral solvents. Thermal oxidation of **5** leads preferentially to the formation of bibenzoxazole **7** and only with small yields to benzoxazinobenzoxazine **8**. The photooxidation reaction of **5** is wavelength dependent. Long wavelength excitation yields exclusively **7**, short wavelength excitation yields also **7**, but in addition **8**. The photooxidation **5**→**7** requires two photons: The first product is the metastable dihydrobibenzoxazole **10** which is converted by a second photon into **7**. The photooxidation **5**→**8** is presumably a one-photon process.

Struktur und Photochemie eines Kondensationsproduktes aus 2-Aminophenol und Glyoxal

Mit Hilfe der Röntgen-Strukturanalyse konnte gezeigt werden, daß bei der Kondensation von 2-Aminophenol und Glyoxal 5a,6,11a,12-Tetrahydro[1,4]benzoxazino[3,2-*b*][1,4]-benzoxazin (**5**) und nicht, wie allgemein angenommen[1], 2,2'-Bibenzoxazolin (**4**) entsteht. Beim Auflösen von **5** in neutralen Lösungsmitteln ändert sich dessen Struktur nicht. Bei der thermischen Oxidation von **5** entsteht vorzugsweise Bibenzoxazol **7** und nur mit kleiner Ausbeute Benzoxazinobenzoxazin **8**. Der Verlauf der Photooxidation von **5** hängt von der Wellenlänge des eingestrahlten Lichtes ab. Bei langwelliger Anregung entsteht ausschließlich **7**. Bei kurzwelliger Anregung entsteht ebenfalls **7**, daneben jedoch auch **8**. Zur Photooxidation **5**→**7** sind zwei Photonen erforderlich. Zunächst bildet sich das metastabile Dihydrobibenzoxazol **10**, aus dem nach der Absorption eines zweiten Photons **7** entsteht. Die Photooxidation **5**→**8** ist wahrscheinlich ein Ein-Quanten-Prozeß.

The Structure of the Condensation Product

For the condensation product (CP) of 2-aminophenol (**1**) and glyoxal (**2**) the structures **3** to **6** are conceivable (Scheme 1). Since CP forms chelates with a number of divalent cations (e.g. $Cu^{2\oplus}$, $Mg^{2\oplus}$, $Ca^{2\oplus}$), *Bayer*[1] first assumed that the Schiff base **3** is the chelating reagent. CP is commercially available (Fluka)

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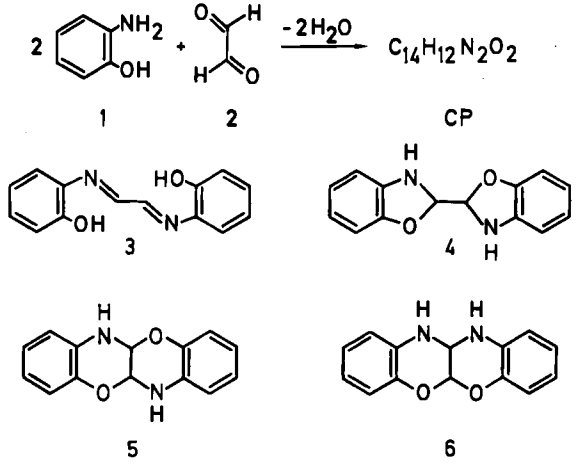
0009–2940/86/1111–3316 $ 02.50/0

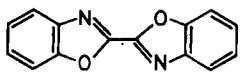
still under the name „glyoxal-bis-2-hydroxyanil" („Calciumred") although it soon became clear from IR and UV spectroscopic studies that **3** cannot be the correct formula[2]. For instance, one would expect by analogy with other aromatic Schiff bases, that **3** has an absorption peak around 360–400 nm, which a neutral solution of CP, however, does not exhibit (cf. Fig. 1). Oxidation of CP yields bibenzoxazole **7** as the main product. Therefore, it is now generally assumed that a neutral solution of CP consists of 2,2′-bibenzoxazoline (**4**). In alkaline solution the dianion of **3** is formed from CP and can react with divalent cations to yield very stable and poorly soluble chelates, whereby the solution is gradually depleted of CP. The 270-MHz ^{1}H NMR spectrum of CP (see Exp. Part) is in agreement with structure **4**, but also with **5**. The spectra of both compounds will be similar with a slight (and scarcely predictable) difference in the δ values. However, because of this expected difference, one can at least state that within the NMR detection limit (3–5%) only one compound, either **4** *or* **5**, is present in solution. **6** can be excluded from the list of possible CP structures because in this isomer the two central C–H protons are not equivalent and would, therefore, yield a more complicated NMR signal than observed.

Scheme 1

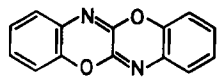


Since the results of our photochemical experiments with CP made structures **4** and **5** equally plausible, the *X*-ray structure of CP was determined. In the crystalline state the condensation product is 5a,6,11a,12-tetrahydro[1,4]benzoxazino[3,2-*b*][1,4]benzoxazine, i.e. **5** (cf. Fig. 2). This is surprising because oxidation of this compound even under mild conditions yields, as already mentioned, **7** as main product with only ca. 2% [1,4]benzoxazino[3,2-*b*][1,4]benzoxazine (**8**). Therefore, the possibility has to be considered that a rearrangement **5**→**4** takes place if **5** is dissolved. Such a reaction would be similar to the rearrangement of the benzoxazoline **9a**, which is stable in the crystalline state[3]. If **9a** is dissolved

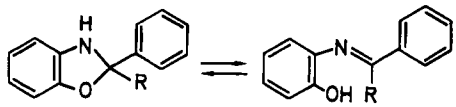
in, e.g., methylcyclohexane (MCH), it slowly rearranges (with a "halflife" of about 3 hours at room temperature) into the Schiff base **9b** until an equilibrium **9a** ⇄ **9b** is established[3].



7



8



9a 9b R = $C(CH_3)_3$

The NMR spectrum of CP shows signals of one compound only. No indication of any rearrangement **5**→**4** could be detected. A quickly recorded UV absorption spectrum of a freshly prepared solution of **5** in cyclohexane (CH) is shown in Fig. 1. This spectrum does not change if the solution is stored in the dark. Furthermore, the colourless solution of **5** in a 90% methanol/10% water mixture turns red very slowly (within about 3 minutes) if 0.1 M NaOH is added. This change in colour is due to the formation of the ionized Schiff base **3**. On neutralization, the red colour disappears and **5** is reformed. The mixed melting point of a sample of the starting material and of the compound recovered from the neutralized solution shows no depression. We conclude from these findings that **5** does not rearrange into **4** if it is dissolved and that the previously assumed structure **4** is neither in the crystalline state nor in solution the correct one.

Photochemical Reactions

All reactions described below were carried out in MCH or CH. In these nonpolar solvents **5** is only poorly soluble, but in polar solvents (methanol, 2-propanol, dichloromethane, and diethyl ether) photodecomposition into (unidentified) products with uncharacteristic absorption spectra takes place. Saturated solutions of **5** at room temperature contain 31 mg/l (1.3×10^{-4} M) of the solute in MCH or CH and 12 mg/l (0.5×10^{-4} M) in n-hexane.

The photochemistry of **5** is wavelength dependent in the singlet manifold. Illumination of an air-equilibrated, saturated solution of **5** with light of wavelength ≥ 260 nm, i.e., excitation into the first absorption band (cf. Fig. 1) of **5** yields bibenzoxazole **7** as final product with a chemical yield of 12%. **7** can easily be detected in the reaction mixture because of its characteristic absorption bands between 300 and 335 nm. By monitoring its emergence at, e.g., 335 nm as a function of illumination time one observes an induction period, which indicates that the formation of **7** from **5** is not a single-step reaction. The first photoproduct which is detectable in the absorption spectrum of the reaction mixture is the dihydrobibenzoxazole **10**. It is formed from **5** by photodehydrogenation and has at room temperature a lifetime of τ_{10} (298 K) = 98 min. A second photon is

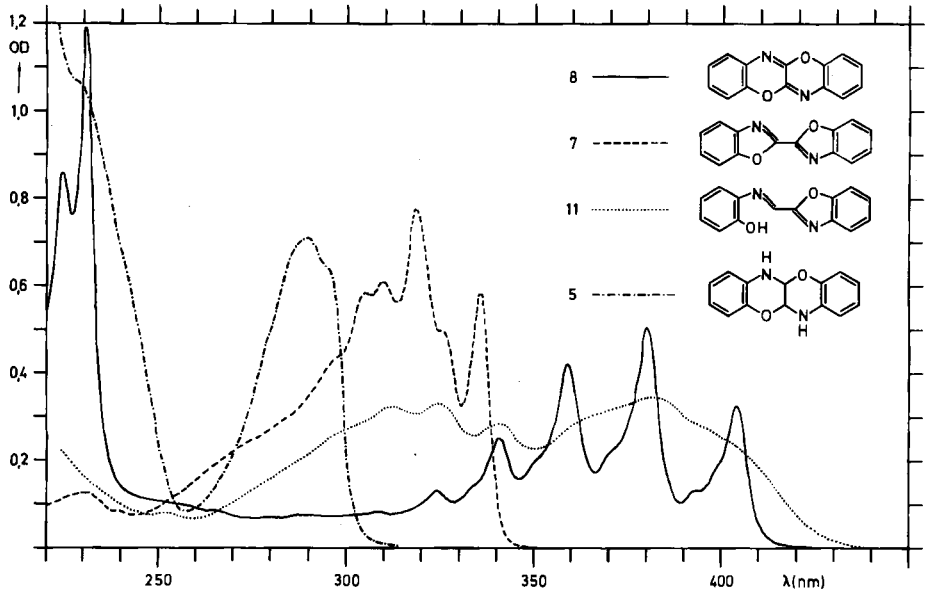
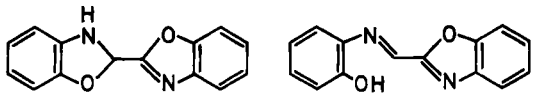


Figure 1. Absorption spectra of a 1.00×10^{-4} M solution of **5** and of 2.00×10^{-5} M solutions of **7**, **8**, and **11** in air-equilibrated cyclohexane at 296 K. Optical pathlength d = 1.00 cm

required to convert this transient by photodehydrogenation into **7**. Thermally, **10** rearranges by ring opening into the Schiff base 2-[[(2-benzoxazolyl)methylene]amino]phenol (**11**). **11** is a stable compound and has, like many similar aromatic Schiff bases, an absorption maximum at 380 nm. By light it is also, like **5**, converted into **10**. It is therefore possible to study the formation of **7** in more detail by investigating the photochemical properties of **11**. Since **10** and **7** absorb below 350 nm one can excite **11** exclusively and thus avoid photoreactions of **10**, once it has been formed from **11**. In this way one can show that in the dark **10** reverts completely to **11**. The rate of this thermal back reaction **10**→**11** (rate constant k_{10}) was determined in MCH at different temperatures between 290 and 350 K, and from an Arrhenius plot of the rate constants $k_{10}(T)$ we obtained $k_{10} = 11 \exp(-E_{10}/RT)\ s^{-1}$, $E_{10} = 27$ kJ/mol. Now, if one illuminates a solution of **5** for a *short* period of time and monitors the absorption spectrum after turning off the actinic light, an absorption with $\lambda_{max} = 380$ nm appears with exactly the same rate constant (k_{10}) with which **10** reacts thermally, yielding **11**, as just described. Therefore, it is safe to assume that the first step in the photochemical formation of **7** from **5** is the reaction **5**→**10**.



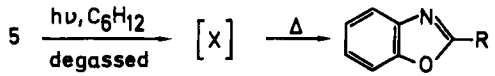
10 **11**

In addition to the reaction **11**→**10** a *trans*→*cis* photoisomerization and the thermal back-reaction **11** (*cis*)→**11** (*trans*) about the C=N double bond takes place. At the long wavelength edge of the absorption spectrum, above 435 nm, the extinction coefficients of **11** (*cis*) are larger than those of **11** (*trans*). Therefore, the exponential decay of the unstable *cis*-isomer **11** (*cis*) into **11** (*trans*) (rate constant k_c) can be measured in flash experiments at different temperatures. From an Arrhenius plot of the rate constants $k_c(T)$ determined between 275 and 347 K we obtained $k_c = 3.5 \times 10^{11} \exp(-E_c/RT)\ s^{-1}$, $E_c = 57$ kJ/mol.

Short wavelength illumination of **5** with a mercury resonance lamp (λ_{exc} = 254 nm) leads also to the formation of **7** via **10**, but in addition, [1,4]benzoxazino-[3,2-*b*][1,4]benzoxazine (**8**) is formed with a chemical yield of 4%. To our knowledge, this compound has not yet been described in the literature. (The condensation of **1** with oxalic acid yields **7** and not, as reported[4] **8**.) **8** has characteristic absorption bands between 325 and 405 nm (Fig. 1). It fluoresces with the high quantum yield of $\Phi_f = 0.98$ and is therefore photochemically rather stable. The fluorescence spectrum is the mirror image of the long wavelength absorption bands with a very small Stokes shift of 180 cm^{-1}. Because of its characteristic long wavelength absorption and fluorescence spectrum it is very easy to detect this photoproduct in the reaction mixture.

Obviously, excitation into the first absorption band of **5** (λ_{max} = 290 nm, cf. Fig. 1) does not lead to the formation of **8**. At 254 nm the onset of the second absorption band system of **5** is located, presumably still overlapped by the short wavelength tail of the first absorption band. One could, therefore, expect that excitation at shorter wavelengths would increase the chemical yield of **8**, eventually at the expense of the formation of **7**. Excitation with 245, 240, and 230 nm light (bandwidth ≈5 nm) did, however, not improve the yield.

In the absence of oxygen a surprising reaction takes place. On illumination of a degassed solution of **5** in cyclohexane one first observes the formation of a transient (**X**) that has an absorption maximum at 310 nm. At room temperature **X** has a lifetime of about 3 hours and decays thermally into a mixture of stable products. From this mixture 2-cyclohexylbenzoxazole (**12**) and 2-(cyclohexylmethyl)benzoxazole (**13**) were gas-chromatographically isolated and the GC retention times and the UV and mass spectra compared with authentic samples.

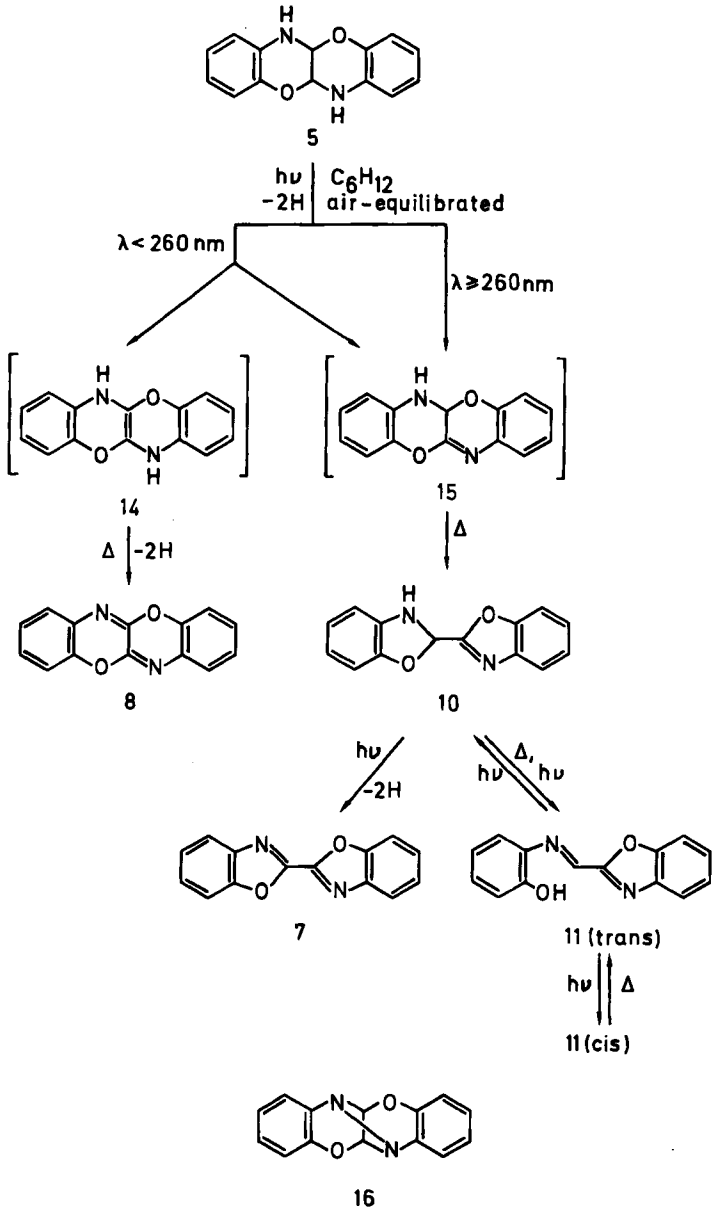


12: R = C_6H_{11}

13: R = CH_2-C_6H_{11}

The reaction has not yet been studied in detail. So far, we know that it takes place in the triplet manifold because the formation of **X** is quenched by oxygen and piperylene, and it can be sensitized with triphenylene. Formation of **X** does not depend on the wavelength of the exciting light. The formation of **12** and **13** from **X** is not influenced by oxygen.

Scheme 2



Discussion

In Scheme 2 the photochemical reactions of the condensation product CP in air-equilibrated cyclohexane (oxygen content ca. 10^{-3} M) are summarized. **7**, **8**, and **11** are isolated compounds that have been compared with authentic samples.

The oxazoline **10** is, without doubt, the precursor of **7** as well as of **11**. It rearranges like other oxazolines[3,5] thermally into the stable Schiff base **11** and we have shown that a second photon is required to convert it by photooxidation into **7**.

Included in Scheme 2 are two hypothetical oxidation products, **14** and **15**, whose existence in the reaction mixture could not be proven experimentally. Their postulation is based on the assumption that the tetrahydrobenzoxazinobenzoxazine **5** is indeed the investigated compound. We believe that this assumption is well justified because in the crystalline state the assignment of structure **5** to the condensation product is unambiguous and the spectral properties of solutions of **5** show that it does not rearrange into **4** when it is dissolved.

The mechanism of the formation of the final product **7** is known in more detail than that of **8** because **10** has been observed in the reaction mixture. The structure of the latter follows from the fact that it can also be prepared by excitation of the Schiff base **11**. Circumventing the formation of **10** from **5** by starting the investigation with **11**, instead of **5**, also makes it possible to demonstrate, that **8** is neither formed from **10** nor from **11**. It can also be shown that in cyclohexane or methylcyclohexane **7** does not photochemically rearrange into **8**, and **8** not into **7**. Thus, it appears logical to assume that the first step in the reaction sequence, which leads ultimately to the formation of **7** and **8**, is a photodehydrogenation reaction which yields two products, **14** and **15**, on short wavelength ($\lambda < 260$ nm) and only one (either **14** or **15**) on long wavelength ($\lambda > 260$ nm) excitation. If this assumption is correct, it is very likely that **14** is the precursor of **8**, and **15** the precursor of **10**, as indicated in Scheme 2. Intuitively, we presume that the formation of the third conceivable oxidation product, **16**, is less probable.

Apparently, no second photon is required to oxidize **14** to **8**. If the intensity of the exciting light ($\lambda < 260$ nm) was reduced 100 fold and the illumination time prolonged by the same factor, the amount of **8** formed was the same, i.e. the quantum yield does not depend on light intensity. Furthermore, no induction period was observed when the time dependence of **8** formation was examined. We conclude from these findings that the formation of **8** from **14** is a thermal reaction.

If the mechanism proposed in Scheme 2 is correct, the remarkable wavelength dependence of the photoreaction of **5** arises from the fact that two different photodehydrogenation reactions take place. Short wavelength excitation into the second absorption band converts a small fraction of the excited molecules into **14**. The larger fraction relaxes from higher excited singlet states, S_n, $n \geq 2$, into the first excited singlet state, S_1, and yields, like long wavelength excitation, the intermediate **15**. Once **15** is formed, the rearrangement **15**→**10** is apparently so rapid that a photodehydrogenation **15**→**8** does not take place.

We thank Mrs. *A. Heinrich* for technical assistance.

Experimental

Spectra: UV: Perkin Elmer Model 320. – Fluorescence: Spex Fluorolog. – ^{1}H NMR: Bruker WH 270. – MS: Varian MAT CH7.

Melting points were determined in open capillaries and are uncorrected. Illumination and flash experiments were performed as described recently[6].

Solvents: Methylcyclohexane (purum, Fluka) was purified on a SiO_2 (Silica-Woelm, 100–200, active) – Al_2O_3 (Alumina Woelm B-Super I) column. Cyclohexane (Uvasol Merck-Schuchardt) was used without further purification.

5a,6,11a,12-Tetrahydro[1,4]benzoxazino[3,2-b][1,4]benzoxazine (**5**) was prepared similarly to a method described by *Murase*[2a]. 10.9 g (0.1 mol) of 2-aminophenol was dissolved under a nitrogen atmosphere in 100 ml of ethanol at 60°C and 9.7 g (0.05 mol) of glyoxal, dissolved in 20 ml of water, was added. The solution was allowed to cool and to stand about 12 h. The precipitate formed was filtered, washed with ethanol and recrystallized from ethanol under nitrogen. Yield 6.8 g (57%). Colourless crystals, m.p. 231°C (lit.[2a] 212°C). – UV (in MCH): λ_{max} = 290 nm (lg ε = 3.85). – ^{1}H NMR ([D$_6$]DMSO): δ = 5.2 (d, *J* = 5 Hz, 2H, C*H*NH), 6.5–6.8 (m, 8H, arom.), 7.3 (d, *J* = 5 Hz, 2H, CHN*H*). Upon addition of CH_3OD to **5** the signal at δ = 7.3 disappears, while the doublet at δ = 5.2 becomes a singlet. ^{1}H NMR (80% vol. CCl_4, 20% C_6D_{12}): δ = 4.58 (bs, 2H, CHN*H*), 5.24 (d, *J* = 5 Hz, 2H, C*H*NH), 6.52–6.73 (m, 8H, arom.).

X-ray Analysis of **5**

Crystal data: Monoclinic, colourless plates, *a* = 1205.6(5), *b* = 888.2(2), *c* = 1157.8(2) pm, β = 112.79(2)°, *V* = 1.143 nm^3, space group $P2_1/c$ (no. 14); *Z* = 4, *M* = 240.26 g · mol^{-1}, d_{calc} = 1.396 g · cm^{-3}, μ = 0.9 cm^{-1}.

Intensities: STOE-Siemens AED four-circle diffractometer with Mo-K_α radiation, crystal size 0.4 · 0.3 · 0.1 mm, 2 Θ ≤ 45°; 3140 reflections. Direct methods and refinement with SHELXTL[7]; 1331 reflections with $F > 3\sigma(F)$ for 170 parameters: C,N,O ansisotropic and U(H) = 1.2 · U_{iso}(C), H geometrically positioned at C–H = 96 pm, position of N–H freely refined. R = 0.039, R_w = 0.047, $w^{-1} = \sigma^2(F) + 0.002\,F^2$.

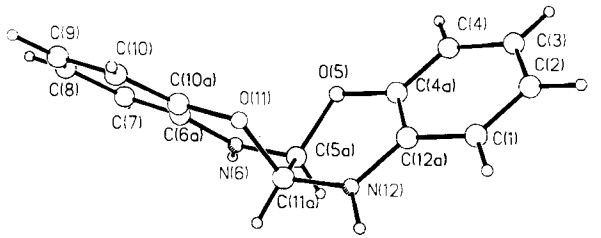


Figure 2. Molecular structure of **5**
Some selected bond lengths (pm): C(4a)–O(5) = 138.4(3), C(5a)–O(5) = 145.5(3), C(5a)–N(6) = 140.6(3), C(5a)–C(11a) = 151.7(3), C(6a)–N(6) = 138.9(3), C(10a)–O(11) = 137.9(3), C(11a)–O(11) = 144.9(3), C(11a)–N(12) = 140.0(3), C(12a)–N(12) = 139.6(2).

Atom coordinates and equivalent U_{iso} are given in Tab. 1*). The molecule has no crystallographic symmetry because the heterocyclic rings adopt opposite twist conformations so that an overall V-shaped twist results. The torsion angle N(6)–C(5a)–C(11a)–N(12) is

*) Additional crystallographic material (complete bond lengths and angles, H atom coordinates, structure factors, temperature factors) can be ordered from the Fachinformationszentrum Energie Physik Mathematik, D-7514 Eggenstein-Leopoldshafen 2. Please quote reference no. CSD-52089 and the names of the authors and the title of the article.

nearly antiplanar −177°, while the O(5)−C(5a)−C(11a)−O(11) torsion angle is 64.4° (+ synclinal) and H(5a)−C(5a)−C(11a)−H(11a) is −55.5° (− synclinal).

During the preparation of this article the *X*-ray structures of derivatives of **5** have been published[8]. The *X*-ray analysis reported here confirms and supplements these results.

Table 1. Atomic coordinates (× 10^4) and isotropic thermal parameters (pm^2 × 10^{-1}) of **5**

	x	y	z	U
C(1)	4089(2)	6207(2)	961(2)	47(1)*
C(2)	5020(2)	5523(2)	1910(2)	56(1)*
C(3)	5081(2)	5567(2)	3124(2)	57(1)*
C(4)	4204(2)	6325(2)	3378(2)	50(1)*
C(4a)	3274(2)	7029(2)	2429(2)	39(1)*
O(5)	2411(1)	7777(2)	2723(1)	49(1)*
C(5a)	1729(2)	8914(2)	1824(2)	44(1)*
N(6)	755(1)	9389(2)	2116(2)	51(1)*
C(6a)	−313(2)	8577(2)	1684(2)	41(1)*
C(7)	−1240(2)	8901(2)	2068(2)	52(1)*
C(8)	−2312(2)	8120(3)	1572(2)	63(1)*
C(9)	−2472(2)	7023(3)	687(2)	62(1)*
C(10)	−1551(2)	6674(2)	301(2)	52(1)*
C(10a)	−473(2)	7439(2)	799(2)	40(1)*
O(11)	445(1)	7038(1)	430(1)	47(1)*
C(11a)	1282(2)	8246(2)	517(2)	43(1)*
N(12)	2255(1)	7706(2)	265(1)	50(1)*
C(12a)	3199(2)	6964(2)	1203(2)	37(1)*

* Equivalent isotropic *U* calculated as one third of the trace of the orthogonal U_{ij} tensor.

2-[[(2-Benzoxazolyl)methylene]amino]phenol (**11**): 545 mg (3.3 mmol) of 2-benzoxazolecarbaldehyde hydrate (synthesized by the procedure of *Gauss* et al.[9]) and 330 mg (3.0 mmol) of 2-aminophenol were dissolved in 30 ml of ethanol (absol.)/MCH (1:1 by vol.), the solution was warmed to 50°C for 5 min, the solvent quickly evaporated and the residue recrystallized from MCH. The yellow crystals were twice purified on a silica gel column with diisopropyl ether as eluant. Yield 558 mg (78%). Yellow crystals, m.p. 144°C. − ^{1}H NMR ($CDCl_3$): δ = 6.9−7.9 (m, 9H, arom. H + OH), 8.7 (s, 1H, CH=N). After shaking with CH_3OD the multiplet at 6.9−7.9 consists of 8H. − UV (C_6H_{12}): λ_{max} (lg ε) = 381 (4.23), 341 (4.15), 325 (4.22), 312 (4.21), 254 nm (3.61). − MS: *m/z* = 238 ($M^{\oplus}$).

$C_{14}H_{10}N_2O_2$ (238.2) Calc. C 70.45 H 4.36 N 11.66 O 13.58
Found C 70.59 H 4.20 N 11.76 O 13.44

[1,4]Benzoxazino[3,2-b][1,4]benzoxazine (**8**): **8** was prepared analogously to the method of *Stephens* et al.[10]. 12.0 g (0.05 mol) of **5** was dissolved in 500 ml of dried benzene. Within 30 min 50 g (0.1 mol) of lead tetraacetate (95%) was added in small fractions under stirring. The stirring was continued for 2 h, the precipitated lead diacetate removed by filtration and the solvent evaporated. The reaction product (≈12 g) was chromatographed on silica gel (Silica-Woelm, 100−200, 70−150 mesh, activity 1) with toluene as eluant. The first fraction consists of starting material, the second fraction is **8**, easily detectable because of its brilliant light-blue fluorescence, followed by **7** (which is the main fraction). After recrystallization from toluene 248 mg (2.1%) of **8** was obtained. Light yellow needles, m.p. 290−291°C. −

^{1}H NMR ($CDCl_3$): δ = 7.45–7.95 (m, arom.). – UV (isooctane): λ_{max} (lg ε) = 404 (4.22), 380 (4.42), 359 (4.34), 341 (4.11), 324 (3.82), 308 (3.59), 265 (3.62), 230 (4.71), 224 nm (4.63). – Fluorescence (MCH): λ_{max} (relative intensity) = 408 (0.62), 434 (1.0), 463 (0.6), 495 (0.21), 530 nm (0.06). Fluorescence quantum yield (MCH) $\phi = 0.98 \pm 0.05$ (standard: 2,5-diphenyloxazole (POP), $\phi = 1.00$[11]). – MS (70 eV): m/z = 236 ($M^{\oplus}$).

$C_{14}H_8N_2O_2$ (236.2)	Calc.	C 71.19	H 3.39	N 11.86	O 13.56
	Found	C 71.23	H 3.39	N 11.81	O 13.70

Our attempts to improve the yield failed. Using water-free acetic acid as solvent, instead of benzene, lowered the yield to 0.8%. Oxidation of **5** with Ag_2O[12] in CH_2Cl_2 yields 25% **7** and only 1.5% **8**.

If 3.26 g (0.02 mol) of 2*H*-1,4-benzoxazine-2,3-(4*H*)dione, 2.2 g (0.02 mol) of 2-aminophenol and 200 g of polyphosphoric acid (85% P_2O_5) are heated for 4 h under nitrogen and the reaction product is poured onto 1 kg of ice and immediately extracted with $CHCl_3$, **8** is also obtained. After purification as described above the yield is 56 mg (1.2%).

Kehrmann[4] reported the synthesis of **8** in 1925. We repeated the experiments and it turned out that **7** instead of **8** was synthesized by this author. It was also impossible to obtain **8** from the 2-hydroxyanilide of oxalic acid by heating this compound with polyphosphoric acid.

2-Cyclohexylbenzoxazole (**12**) was prepared from 2-aminophenol and cyclohexylformic acid according to *Skraup*[13].

2-(Cyclohexylmethyl)benzoxazole (**13**) was prepared analogously to **12** from 2-aminophenol and cyclohexylacetic acid. Colourless oil, b.p. 133–135°C (1 Torr). – ^{1}H NMR ($CDCl_3$): δ = 7.78–7.1 (m, 4H), 2.8 (d, 2H), 2.2–0.8 (m, 11H). – MS: m/z = 215 ($M^{\oplus}$).

$C_{14}H_{17}NO$ (215.3)	Calc.	C 78.14	H 7.91	N 6.51	O 7.44
	Found	C 78.27	H 7.93	N 6.57	O 7.49

CAS Registry Numbers

5: 104462-81-9 / **7**: 7210-07-3 / **8**: 258-19-5 / **10**: 104462-84-2 / **11**: 70299-33-1 / **12**: 104462-82-0 / **13**: 104462-83-1 / cyclohexylformic acid: 98-89-5 / 2-benzoxazolcarbaldehyde: 62667-25-8 / 2-aminophenol: 95-55-6 / glyoxal: 107-22-2 / cyclohexylacetic acid: 5292-21-7

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