168. 2′-Deoxyisoguanosine and Base-Modified Analogues: Chemical and Photochemical Synthesis

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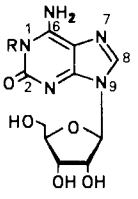
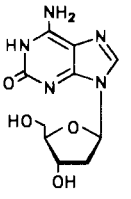
The synthesis of 2′-deoxyisoguanosine (**2**), and the pyrrolo[2,3-*d*]pyrimidine and pyrazolo[3,4-*d*]pyrimidine 2′-deoxyribonucleosides **3** and **4** is described. Condensation of the imidazole precursor **5** with benzoyl isocyanate followed by reaction with ammonia gave **2**. Its N(7) regioisomer was obtained from **6**. Compound **2** was also prepared by the photochemically induced conversion of 2-chloro- and 2-bromopurine 2′-deoxyribofuranosides **9a** and **10**, respectively, in aqueous solution. The photo reaction was further used for the synthesis of the compounds **3** and **4** starting with the amino-chloro-2′-deoxynucleosides **9b** and **9c**, respectively.

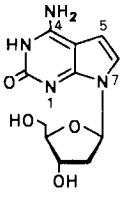
Introduction. – Isoguanine ribofuranoside (**1a**) and 2′-deoxyribofuranoside (**2**) are not constituents of nucleic acids. However, their incorporation into DNA and RNA [1–3] or their polymerization to oligoribonucleotides [4] was described. Isoguanosine (crotonoside; **1a**) was isolated from *Croton tiglium* L. [5]. More recently, isoguanine was obtained from butterfly wings of *Prioneris thestylis* [6]. The 1-methylisoguanosine (doridosine; **1b**), a natural product isolated from the marine sponge *Tedania digitata,* possesses potent pharmacological activity [7].



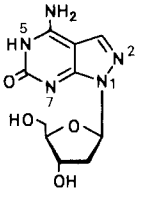
1a R = H[a])
b R = CH_3



2



3[b])



4[b])

a) Purine numbering. b) Systematic numbering.

In contrary to several syntheses of isoguanosine (**1a**) reported in the literature [8–12], 2′-deoxyisoguanosine (**2**) is unknown. Only 5′-mono- and 5′-triphosphates of **2** were prepared by photochemical transformations [13] [14]. In this paper, a chemical and photochemical synthesis of 2′-deoxyisoguanosine (**2**) is presented. The photochemical method is also applied to other heterocyclic congeners, such as 7-deaza-2′-deoxyisoguanosine [15] (**3**) and 8-aza-7-deaza-2′-deoxyisoguanosine (**4**).

Results and Discussion. – We have synthesized the regioisomeric 2′-deoxyribofuranosides **5** and **6** of 5-aminoimidazole-4-carbonitrile during photochemical studies on 2-aza-2′-deoxyadenosine [16]. These compounds are now used as synthetic precursors of 2′-deoxyisoguanosine (**2**) and its isomer **7** (*Scheme 1*). Reaction of **5** or **6** with benzoyl isocyanate followed by treatment with ammonia resulted in annelation of the pyrimidine ring under simultaneous deprotection of the sugar moiety, giving **2** and **7** in 43 and 63% yield, respectively, after chromatographic purification. The differences in the yields can be attributed to changes in substituent reactivity and/or steric effects occurring during the attack of benzoyl isocyanate on the imidazole amino group.

The structure of **2** and **7** was established by ^{1}H- and ^{13}C-NMR spectroscopy (*Table*) as well as by elemental analyses. Compound **2** showed a bathochromic shift of its UV maximum (292 nm) compared to that of regioisomer **7** (282 nm). The HPLC retention time of **2** was longer than that of regioisomer **7** but shorter than those of 2′-deoxyguanosine and 2′-deoxyadenosine (*Fig. 1*). The proton-catalyzed hydrolyses of **2** and **7** provided isoguanine, which was identified by HPLC. The 7-isomer **7** is more labile than

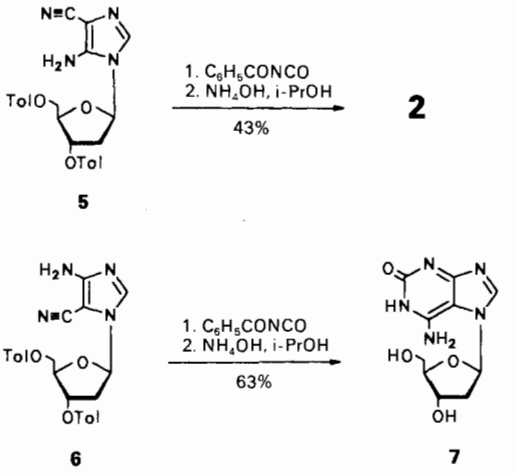
Table. *^{13}C-NMR Chemical Shifts of 2′-Deoxyribonucleosides in (D_6)DMSO at 23°*

	C(2)[a]) C(2)[b]) C(6)[c])	C(4)[a]) C(7a)[b]) C(7a)[c])	C(5)[a]) C(4a)[b]) C(3a)[c])	C(6)[a]) C(4)[b]) C(4)[c])	– C(5)[b]) C(3)[c])	C(8)[a]) C(6)[b]) –
1[a])	152.5[d])	[e])	109.8	155.9[d])	–	138.4
2[a])	152.6[d])	[e])	109.8[f])	156.5[d])	–	137.5[f])
2-Anion[a])	168.6[d])	153.0	112.2[f])	157.4[d])	–	137.0[f])
3[b]) [15]	152.6[d])	153.9[d])	92.9	156.0[d])	100.8[f])	119.0
3-Anion[b])[g])	168.2[d])	155.5[d])	96.1[f])	160.0[d])	100.7[f])	118.4[d])
4[c])	155.2[d])	[e])	92.6[f])	157.0[d])	134.7[f])	–
4-Anion[c])	169.4[d])	158.6[f])	95.3[f])	160.1[d])	134.8[f])	–
7[a])	154.0[d])	[e])	102.8[f])	156.6[d])	–	141.6[d])
9a[a])	153.0[d])	150.0[f])	116.1[f])	156.8[d])	–	139.8[f])
9b[b]) [24]	150.3[d])	152.6[d])	101.3	158.2[d])	99.9	121.6
9c[c])	155.7[d])	153.6[d])	99.6	157.3[d])	133.6	–
10[a])	144.1[d])	149.9[f])	118.5[f])	156.6[d])	–	139.6
12[a])[f])	152.5	148.9	119.4	156.2[f])	–	139.7

	C(1′)	C(2′)	C(3′)	C(4′)	C(5′)
1	87.8	73.1	70.9	86.1	61.9
2	83.8	[h])	71.0	87.9	61.8
2-Anion	83.8	40.6	71.9	89.1	63.1
3	83.3	39.5	71.2	87.3	62.3
3-Anion	83.7	39.5	72.3	87.1	63.1
4	84.3	[h])	71.2	87.6	62.5
4-Anion	84.0	38.8	72.0	88.2	63.3
7	85.5	38.6	69.3	87.9	60.5
9a	83.5	[h])	70.7	87.9	61.6
9b	82.7	38.5	70.8	87.1	61.8
9c	83.9	37.9	71.1	87.8	62.5
10	83.5	39.3	70.7	87.7	61.6
12	84.1	[h])	71.1	88.1	62.0

[a]) Purine numbering, see **1**. [b]) Pyrrolo[2,3-*d*]pyrimidine numbering, see **3**. [c]) Pyrazolo[3,4-*d*]pyrimidine, see **4**. [d]) Tentative. [e]) Not detected. [f]) From gated-decoupled spectra. [g]) In 1N NaOD. [h]) Superimposed by DMSO.

Scheme 1



the 9-glycosylated compound **2** (**7**: $\tau_{1/2} = 5.3$ min; **2**: $\tau_{1/2} = 11.6$ min; 40°, 0.5N HCl). Compound **2** exhibits two pK_a values (4.0 for protonation and 9.9 for deprotonation) which are similar to those of 9-methylisoguanine (3.85 and 9.9) [17] and different from 2′-deoxyguanosine (2.5 and 9.5). pK_a Values of 4.6 and 10.6 were determined for the regioisomer **7**.

The formation of isoguanosine (**1a**) by irradiation of adenosine *N*-oxide was reported earlier [18]. However, this reaction did not provide a single product. More recently, **1a** was prepared from 2-iodoadenosine photochemically [19]. We observed that the very potent antileucemic drug [20] 2-chloro-2′-deoxyadenosine (**9a**) prepared from dichloro compound **8a** [21[[22] as well as its bromo analogue **10** [23] also underwent photochemical transformation. As we considered the syntheses of the base-modified 2′-deoxyisoguanosine analogues **3** and **4**, we tested the applicability of this reaction on compounds **9b**

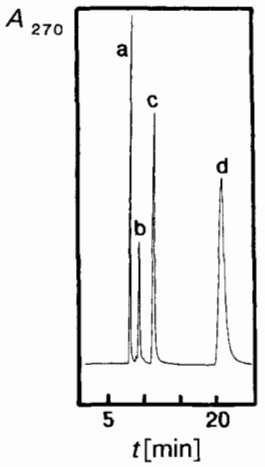
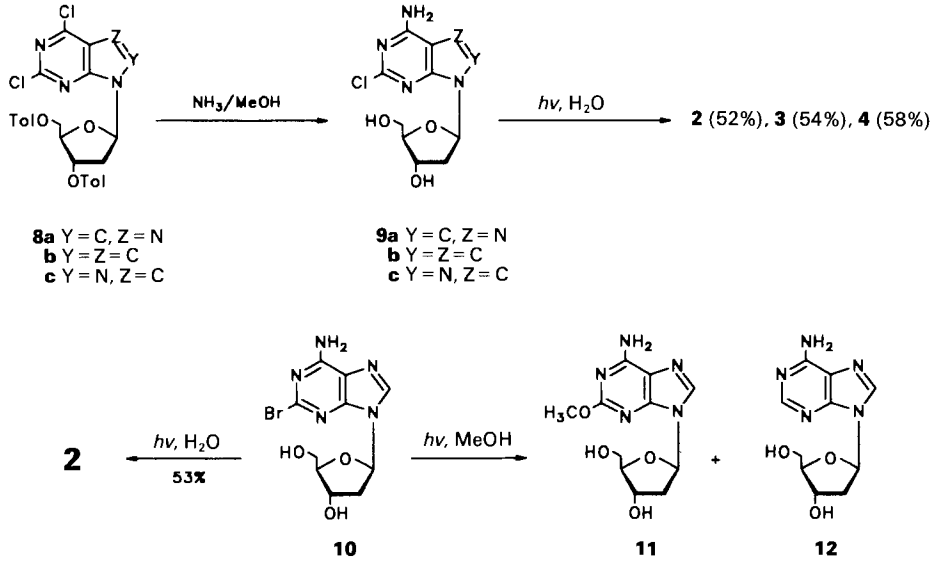


Fig. 1. *HPLC profile of 2′-deoxyribonucleosides:* a) 7, b) **2**, c) *dG, and* d) *dA. RP-18-LiChrosorb* column 4 × 250 mm (*Merck*, Germany), 0.1M $(Et_3NH)OAc$ (pH 7.0)/MeCN 95:5.

Scheme 2



and **9c**. Pyrrolo[2,3-*d*]pyrimidine derivative **9b** was prepared earlier from **8b** [24], and pyrazolo[3,4-*d*]pyrimidine derivative **9c** was obtained from **8c** [25] by selective displacement of the 4-chloro substituent with methanolic ammonia under simultaneous deprotection of the toluoyl groups (*Scheme 2*). The ^{13}C-NMR spectra of **9a–c** are summarized in the *Table*. Chemical-shift assignments of the 5-membered-ring C-atoms as well as those of the sugar moiety was accomplished by gated-decoupled spectra; the assignment of the C-atoms carrying the substituents is ambiguous.

The time-dependent UV pattern of irradiated 10^{-4} M solutions of **9a–c** and **10** in H_2O (*Fig. 2*) show two isosbestic points. This finding demonstrates conversion into one reaction product only. TLC analyses and comparison of the extinction coefficients confirmed this observation. Preparative-scale experiments were carried out in H_2O in the presence of a trace NH_3 in a quartz reactor. Compound **2** was obtained from either **9a** or **10** after *XAD* chromatography and crystallization in yields of 52 and 53%, respectively. The UV irradiation of **9b** and **9c** furnished the 2′-deoxyisoguanosine analogues **3** and **4** in yields of 54 and 58%, respectively. The reaction was carried out in a buffered medium or under slightly alkaline conditions to avoid hydrolysis of the N-glycosylic bond. The ^{13}C-NMR spectra (DMSO) of **2** and **4** showed only 4 signals for the base moiety. This was already observed in the case of compound **1a** [10]. However, all signals appeared, when the measurement was carried out in 1N NaOD (anion formation).

The irradiation of **10** in MeOH instead of H_2O provided two reaction products: 2′-deoxy-2-methoxyadenosine (**11**; 58%) and 2′-deoxyadenosine (**12**; 15%). Formation of **2** from **10** (in H_2O) is independent from the pH value. The quantum yield of the photoreaction depends more strongly on the particular halogen substituent than on the heterocyclic moiety. The values are 0.023, 0.028, and 0.021 for **9a–c**, whereas the quantum yield is 0.09 in the case of **10**, all determined in H_2O.

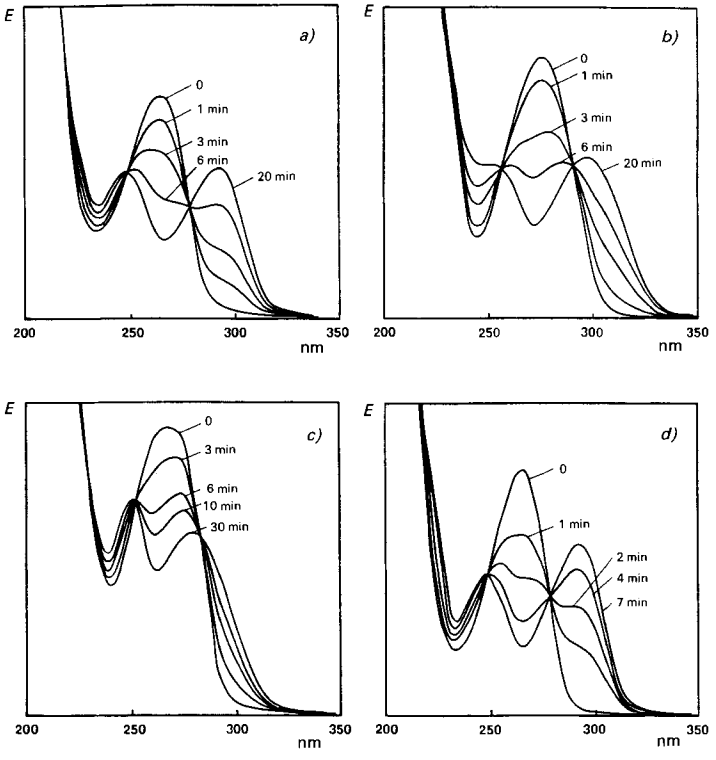


Fig. 2. *Time-dependent UV spectra of* a)–c) **9a–c** *and* d) **10** *in water irradiated with light source* A. The reactions were carried out in a 10-mm quartz cell fitted with a filter of AcOH and followed in time intervals as shown.

It is noteworthy that displacement reactions at C(2) of purine nucleosides including those with OH^- as nucleophile are difficult and extremely hard to accomplish in the case of pyrrolo[2,3-*d*]pyrimidine nucleosides. Furthermore, the imidazole ring of purine nucleosides is sensitive against strong alkaline conditions. Photochemical displacement reactions like those reported above can circumvent such unfavourable reaction conditions and can provide deoxyisoguanosine nucleosides more easily.

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Experimental Part

General. See [26]. Anal.-scale irradiations (*A*): 4-W resonance mercury lamp (*Heräus Instruments*, Germany), light intensity $1.8 \cdot 10^{17}$ quanta min^{-1} cm^{-2}. Prep.-scale experiments (*B*): quartz reactor fitted with a 30-W germicidal lamp (*Philips*, Netherlands). In all irradiations, the light was passed through a 2-mm layer of 20% AcOH to avoid transmission below 230 nm.

*6-Amino-9-(2′-deoxy-β-*D-erythro-*pentofuranosyl)-1,9-dihydro-2*H*-purin-2-one* (= *2′-Deoxyisoguanosine;* **2**). *Method A.* To a soln. of 5-amino-1-[2′-deoxy-3′,5′-di-*O*-(4-toluoyl)-β-D-*erythro*-pentofuranosyl]-1*H*-imidazole-4-carbonitrile [16] (**5**; 400 mg, 0.87 mmol) in dry MeCN (15 ml), benzoyl isocyanate (0.6 ml, 90% purity, 4.3 mmol) was added under stirring at r.t. A precipitate was formed within 15 min. The mixture was stirred overnight and

then refluxed for 30 min. The solvent was evaporated, the residue dissolved in i-PrOH/25% aq. NH_3 soln. 1:1 (100 ml), and the soln. stirred for 2 days at r.t. Upon evaporation, the residue was extracted with Et_2O (2 × 50 ml) under stirring. The precipitate was dissolved in H_2O (100 ml) containing a few drops of 25% aq. NH_3 soln. and filtered. The volume of the filtrate was reduced to 30 ml and applied to an *XAD-4* column (2 × 20 cm). H_2O (200 ml) removed inorg. material, **2** was eluted with H_2O/i-PrOH 9:1. Evaporation yielded a residue which gave colourless crystals from EtOH (100 mg, 43%) decomposing > 230°. TLC (cellulose, BuOH saturated with H_2O): R_f 0.25. UV (pH 1): 235 (5200), 284 (11 500). UV (pH 7): 247 (9100), 292 (10 100). UV (pH 13): 249 (6600), 284 (9800). ^{1}H-NMR ((D_6)DMSO): 2.18, 2.30 (*2m*, 2 H–C(2′)); 3.55 (*m*, 2 H–C(5′)); 3.84 (*m*, H–C(4′)); 4.35 (*m*, H–C(3′)); 5.30 (br. *s*, OH–C(3′), OH–C(5′)); 6.11 (*'t'*, *J* = 6.4, H–C(1′)); 7.95 (*s*, H–C(8)); 8.1 (NH_2). Anal. calc. for $C_{10}H_{13}N_5O_4$ (267.2): C 44.94, H 4.90, N 26.20; found: C 44.70, H 5.03, N 25.99.

Method B. A soln. of 2 bromo-2′-deoxyadenosine [23] (**10**; 200 mg, 0.61 mmol) in H_2O (250 ml) containing conc. aq. NH_3 soln. (1 ml) was irradiated in a quartz reactor (*B*) for 30 min. Then the soln. was concentrated to *ca.* 20 ml and applied to an *XAD-4* column (3 × 21 cm, 200–400 mesh). Elution with a H_2O to 50% MeOH gradient (1200 ml) afforded **2** at 15–20% MeOH. After evaporation the residue was crystallized as described above: 85 mg (53%) of **2**, identical to that obtained by *Method A*.

Analogously, **2** (52%) was obtained from 2-chloro-2′-deoxyadenosine (**9a**) [21] [22] after 1 h irradiation.

*6-Amino-7-(2′-deoxy-β-*D*-*erythro*-pentofuranosyl)-1,7-didehydro-2*H*-purin-2-one* (**7**). As described for **2**, from 4-amino-1-[2′-deoxy-3′,5′-di-*O*-(4-toluoyl)-β-D-*erythro*-pentofuranosyl]-1*H*-imidazole-5-carbonitrile [16] (**6**; 300 mg, 0.65 mmol) and benzoyl isocyanate (0.272 ml, 90%, 1.95 mmol). Within 1 h, a precipitate was formed. The mixture was stirred overnight and then evaporated. The residue was stirred with i-PrOH/conc. aq. NH_3 soln. 1:1 (75 ml) for 2 days at r.t. After evaporation, the residue was triturated with AcOEt/Et_2O 1:1 (75 ml). The precipitate was filtered, dissolved in H_2O (40 ml), and applied to an *XAD-4* column (2 × 20 cm). The resin was washed with H_2O (200 ml) and then with H_2O/i-PrOH 9:1 (1 l). The residue of the nucleoside-containing fractions was crystallized from EtOH: colourless powder (110 mg, 63%) which decomposed > 230°. TLC (cellulose, BuOH saturated with H_2O): R_f 0.15. UV (pH 1): 290 (8600). UV (pH 7): 242 (7000), 282 (7100). UV (pH 13): 285 (5700). ^{1}H-NMR ((D_6)DMSO): 2.25, 2.40 (*2m*, 2 H–C(2′)); 3.55 (*m*, 2 H–C(5′)); 3.84 (*m*, H–C(4′)); 4.34 (*m*, H–C(3′)); 5.35 (br. *s*, OH–C(3′), OH–C(5′)); 6.12 (*'t'*, *J* = 6.6, H–C(1′)); 7.27 (br. *s*, NH_2); 8.19 (*s*, H–C(8)). Anal. calc. for $C_{10}H_{13}N_5O_5$ (267.2): C 44.94, H 4.90, N 26.20; found: C 44.78, H 5.05, N 25.82.

*4-Amino-7-(2′-deoxy-β-*D*-erythro-pentofuranosyl)-3,7-dihydro-2*H*-pyrrolo[2,3-*d*]pyrimidin-2-one* (**3**). The soln. of 4-amino-2-chloro-7-(2′-deoxy-β-D-*erythro*-pentofuranosyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine [24] (**9b**; 85 mg, 0.3 mmol) in H_2O (200 ml) containing conc. NH_3 soln. (1 ml) was irradiated in a quartz reactor for 1 h. The soln. was concentrated to 30 ml and applied to the *XAD* column (2 × 20 cm). The resin was washed with H_2O (150 ml) followed by H_2O/i-PrOH 9:1 (1000 ml). The residue of the nucleoside-containing fractions was crystallized from EtOH: **3** (43 mg, 54%), identical to that synthesized earlier [15].

*4-Amino-6-chloro-1-(2′-deoxy-β-*D*-erythro-pentofuranosyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine* (**9c**). The suspension of 4,6-dichloro-1-[2′-deoxy-3′,5′-di-*O*-(4-toluoyl)-β-D-*erythro*-pentofuranosyl]-1*H*-pyrazolo[3,4-*d*]pyrimidine [25] (**8c**; 540 mg, 1 mmol) was stirred for 2 days at 60° with MeOH saturated with NH_3 at 0° (40 ml). The mixture was evaporated and the residue chromatographed on silica gel *60H* (column 4 × 15 cm) with CH_2Cl_2/MeOH 9:1 yielding **9c** (200 mg, 70%). Crystallization from H_2O afforded colourless needles. M.p. 183–185°. TLC (silica gel, CH_2Cl_2/MeOH 9:1): R_f 0.45. UV (pH 1): 265 (10 200). UV (pH 7): 272 (10 400). ^{1}H-NMR ((D_6)DMSO): 2.45, 2.75 (*2m*, 2 H–C(2′)); 3.50 (*m*, 2 H–C(5′)); 3.81 (*q*, H–C(4′)); 4.41 (br. *s*, H–C(3′)); 4,71 (*t*, OH–C(5′)); 5.27 (*d*, OH–C(3′)); 6.44 (*'t'*, *J* = 6.2, H–C(1′)); 8.16 (NH_2); 8.34 (*s*, H–C(3)). Anal. calc. for $C_{10}H_{12}ClN_5O_3$ (285.7): C 42.04, H 4.23, N 24.51; found: C 42.29, H 4.27, N 24.48.

*4-Amino-1-(2′-deoxy-β-*D*-erythro-pentofuranosyl)-1*H*-pyrazolo[3,4-*d*]pyrimidin-6(5*H*)-one* (**4**). As described for **3**, from **9c** (105 mg, 0.37 mmol). A colourless powder (57 mg, 58%) was obtained from EtOH. M.p. 235° (dec.). TLC (cellulose, BuOH saturated with H_2O): R_f 0.30. UV (pH 1): 268 (8500). UV (pH 7): 221 (21 200), 251 (7900), 283 (6700).UV (pH 13): 221 (26 600), 255 (6600), 278 (9000). ^{1}H-NMR ((D_6)DMSO): 2.20, 2.52 (*2m*, 2 H–C(2′)); 3.30 (*m*, 2 H–C(5′)); 3.64 (*q*, H–C(4′)); 4.21 (br. *s*, H–C(3′)); 5.1 (hr. *s*, OH–C(3′), OH–C(5′)); 6.12 (*'t'*, *J* = 6.4, H–C(1′)); 7.79 (*s*, H–C(3)); 8.1 (NH_2). Anal. calc. for $C_{10}H_{13}N_5O_4$ (267.2): C 44.94, H 4.90, N 26.20; found: C 44.89, H 5.05, N 25.89.

Photolysis of **10** *in MeOH.* A soln. of **10** (200 mg, 0.61 mmol) in anh. MeOH (250 ml) was irradiated in an quartz reactor for 30 min. Then conc. aq. NH_3 soln. (1 ml) was added and the solvent evaporated. The residue was dissolved in H_2O (30 ml) and applied to an *XAD-4* column (3 × 21 cm, 200–400 mesh). Elution with a gradient of H_2O → 60% MeOH gave *2′-deoxyadenosine* (**12**; 23 mg, 15%; with 20–25% MeOH) and *2′-deoxy-2-methoxyadenosine* (**11**; 98 mg, 58%; with 40–50% MeOH). Compound **11** was spectrophotometrically identical with an authentic sample [21].

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