Unexpected Chlorination Products as a Result of Self-Diazotization of Three Amino-dinitro-2,3,4,5-tetrahydrobenzo[*b*][1,4]dioxocin Derivatives

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Diazotization of the dinitroaryl amines **1** with nitrous acid (generated from hydrochloric acid and sodium nitrite) furnished the unexpected chloronitro azides **3** as a result of self-diazotization. Subsequent heating of **3** in ethylene glycol afforded the corresponding chloro-nitro amines **5** and/or the deaminated products **4**. Some mechanistic aspects of these transformations are discussed.

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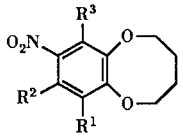
We have recently reported on some dioxocino-annelated unsubstituted [1], nitro-, and acetamidobenzofuroxans and benzofurazans [2]. During preparation of the nitrobenzofuroxans in particular, our initial efforts to oxidize the appropriate dinitro amines **1** with hypochlorite ion [3]



	1a	1b	1c	2a	2b	2c	8	14
R^1 =	H	NH_2	NH_2	H	N_3	N_3	NHAc	NO_2
R^2 =	NH_2	NO_2	H	N_3	NO_2	H	H	H
R^3 =	NO_2	H	NO_2	NO_2	H	NO_2	NO_2	NO_2

were thwarted by unwanted chlorinated side products, thus very low yields of the desired furoxans were obtained. Similarly, attempts to prepare the corresponding azides **2** (which give furoxans on thermolysis) [1,2,4] *via* diazotization of the amines **1** and subsequent treatment with azide ion [5], afforded the chloro derivatives as the major products. We have investigated these reactions further, and report the results herein.

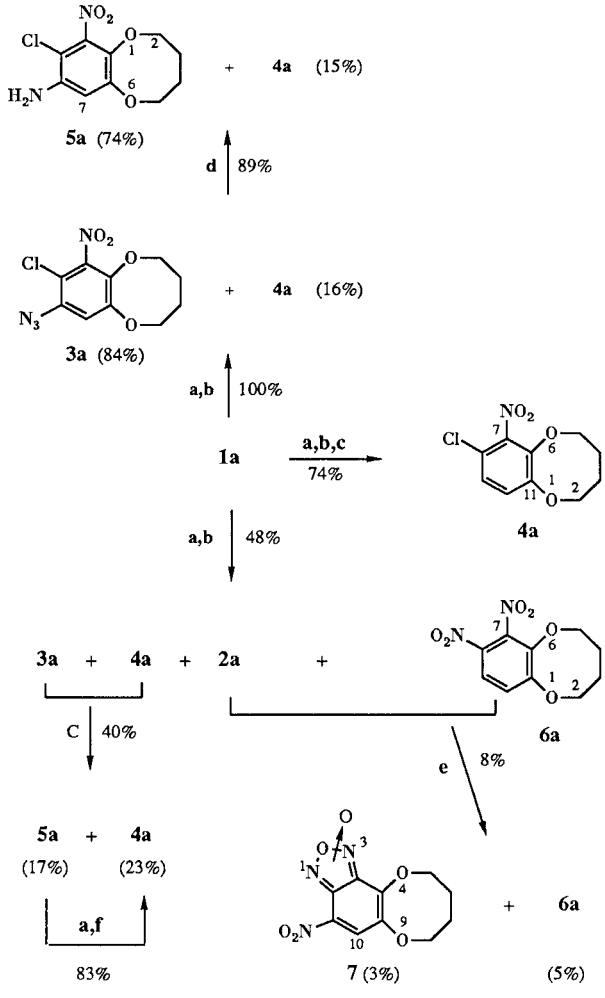
Diazotization of dinitro amine **1a** in tetrahydrofuran with hydrochloric acid and sodium nitrite followed by *in situ* addition of sodium azide and subsequent heating in ethylene glycol, afforded the chloronitro derivative **4a** shown in Scheme 1.

Repetition of the reaction, but without further heating in ethylene glycol furnished **3a** mainly, along with **4a** as determined by ^{1}H nmr. The inseparable mixture **3a** + **4a** was reduced to **5a**, **4a** and separated by column chromatography. In a different run, column chromatography furnished two fractions, each consisting of two components. The first fraction was converted to **5a** and **4a** after heating in ethylene glycol, whereas the second fraction was thermolyzed in toluene to give the nitrobenzofuroxan **7** [2] and **6a** [1]. The chloroamine **5a** was deaminated to **4a**.

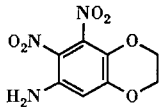
Analogous (yet not entirely) results have been obtained with the dinitro amines **1b** and **1c**, as shown in Scheme 2.

Noteworthy is that the acetamido-dinitro derivative **8**, which under milder conditions was hydrolyzed to the corresponding dinitro amine [2], afforded, under more vigorous conditions, the chloronitro compound **4c** and two or three unidentified products. Similarly, Heertjes and coworkers [6], in an attempt to deaminate compound **9** to the dinitro compound **10**, isolated the chloronitro derivative **11** instead.

Scheme 1



Reagents. a: HCl, $NaNO_2$, THF, 0°; b; NaN_3, 0°; c: $HOCH_2CH_2OH$, Δ; d: $NaBH_4$, EtOH, Δ; e: C_6H_5Me, Δ; f: 50-60°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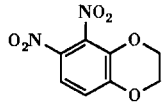
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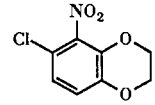
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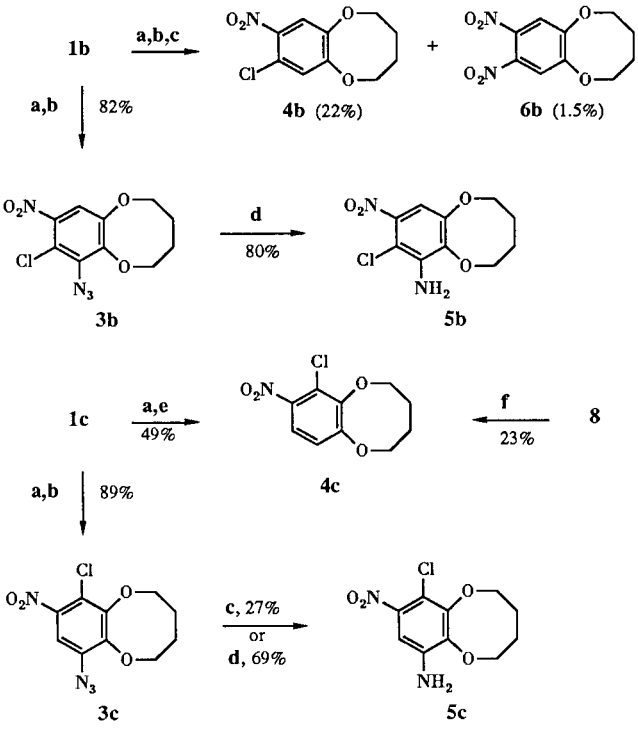
Based on the above results, these reactions seem to take place according to the sequence shown in Scheme 3, using one of the amines, *i.e.*, **1a** as an example. Step C is the major process and is called self-diazotization [7]; it has been discovered by Meldola and Eyre [8] and exemplified by Sihlbom [9]. Mono-, di and trinitroanilines and naphthtalenes lead to mono- and dichlorinated derivatives when the reactions are carried out in acetic acid at 40-100° [9,10]. We have shown that chlorination of the dinitroaryl amines **1** is also facile in aqueous tetrahydrofuran at 0-5°. It is known that nitro groups *ortho* and *para* to the amino group are substituted by chlorine (or bromine) and, of the two, the *ortho* position is more reactive [7,9-12]. Moreover, N_2^+ activates the departure of a leaving group [7,12,13], and quite often it is the halogen counterion from $ArN_2^+ X^-$ that is the attacking nucleophile [12,13]. Even though an S_N2 type mechanism is invoked rarely in nucleophilic aromatic substitutions [13], it is likely to occur in these systems [7,12].

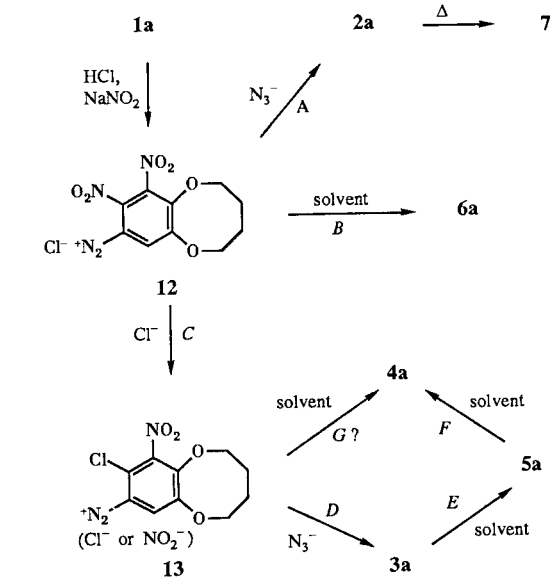
Scheme 2



Reagents. a: HCl, $NaNO_2$, 0°; b: NaN_3, 0°; c: $HOCH_2CH_2OH$, Δ; d: $NaBH_4$, EtOH, Δ; e: 50-60°; f: HCl, EtOH, Δ.

In order to determine whether nucleophilic attack by chloride ion on diazonium salt **12** to furnish **13** (Scheme 3) is intermolecular or intramolecular, we carried out a series of control experiments. Thus, trinitrobenzodioxocin **14** [1] (in which the nitro group at C-7 is substituted easily by N_3^- at room temperature) [2] remained stable after refluxing in ethanol:water = 4:1 in the presence of a large excess of sodium chloride. Addition of concentrated hydrochloric acid and further heating at reflux gave starting material only. The attempted reaction of **14** with hydrochloric acid and sodium nitrite in tetrahydrofuran followed by heating at 50-60° furnished **14** unreacted, as did the attempted reaction with benzenediazonium chloride at 50-60°. These experiments provide some evidence that the conversion of **12** to **13** is not likely to occur intermolecularly. It is postulated then that the attack by chloride ion to furnish **13** (step *C* in Scheme 3) is an intramolecular S_Ni process, particularly for the amines **1a** and **1b** where the $N_2^+ Cl^-$ group is *ortho* to the nitro group displaced by chloride.

Scheme 3



In addition to step *D*, there are several other competitive processes in Scheme 3 worth mentioning. The sequence *CDEF* is the major reaction pathway. Depending on experimental conditions and the structural requirements, **3a,b,c**, or **4a,b**, or **5c** can be the major products, whilst **4** may also come directly from **13** at low temperatures, although this seems to be a minor process, as are *A* and *B*. Steps *A* and *D* are well known nucleophilic displacements of the N_2^+ moiety by azide ion [5,13], whereas in *B* and *G*, the diazonium group is replaced by hydrogen. The mechanisms of the latter two processes and, in addition, that of *F* have not been studied. However, there is cogent evidence that diazonium salts, in general, decompose *via* an S_N1 type mechanism [13]. Interestingly, the hydrogen which replaces the diazonium (or an NH_3^+) group may come from the solvent as hydride ion according to one example at least in an analogous system [14].

EXPERIMENTAL

General.

The general experimental has been described previously [2]. On column chromatography, the columns were eluted with petroleum ether (bp 65-71°): ethyl acetate = 4:1 (v:v). All solids were recrystallized from boiling ethanol (95%). The ir and ^{1}H nmr (80 MHz) spectra were obtained in carbon tetrachloride and deuteriochloroform containing 2% tetramethylsilane, respectively. A ^{1}H nmr and a ^{13}C nmr spectrum were obtained on a Bruker 250-MHz instrument. Mass spectra were obtained at 70 eV on a double focusing VG Tritech VGTS-250 instrument. Exceptions are noted.

7-Nitro-8-chloro-, 8-Amino-9-chloro-10-nitro-, 7,8-Dinitro-2,3,4,5-tetrahydrobenzo[*b*][1,4]dioxocins and 11-Nitro[1,4]dioxocino[2,3-*e*]-5,6,7,8-tetrahydro-2,1,3-benzoxadiazole 1-Oxide (**4a**, **5a**, **6a** and **7**).

A. Compound **4a**.

Into a mixture of dinitroamine **1a** (579 mg, 2.15 mmoles) [2] in tetrahydrofuran (20 ml), water (2 ml) and concentrated hydrochloric acid (4 ml) kept at *ca.* 0°, was added a solution of sodium nitrite (1 g, 14 mmoles) in water (2 ml) and the mixture was stirred at this temperature for 0.5 hour. Sodium azide (1 g, 15 mmoles) in water (2 ml) was added slowly (foaming) and stirring continued at *ca.* 0° for an additional 0.5 hour. Extraction, washing with 5% sodium carbonate solution, drying and concentration *in vacuo* furnished a reddish semisolid which was thermolyzed in ethylene glycol (5 ml) at 140-150° for 0.5 hour. The mixture was decanted into water (50 ml), extracted, dried and concentrated. Column chromatography afforded 388 mg (74%) of **4a** as a pale-yellow semisolid; ir (neat): ν 1596 (w), 1542 (s), 1480 (s), 1370 (s), 1300 (s), 1255 (m), 1083 (m), 1053 (m), 996 (s), 958 (m), 815 (m), 794 (m) cm^{-1}; ^{1}H nmr (250 MHz): δ 1.94 (m, 4H), 4.40 (m, 4H), 7.01 (s, 2H); ^{13}C nmr (63 MHz): δ 25.99, 26.95 (C_3, C_4), 72.40, 74.55 (C_2, C_5), 117.86 (C_8), 123.74, 123.78 (C_9, C_{10}), 130.81 (C_7), 141.79 (C_{12}), 149.48 (C_{11}); ms: m/z (% relative intensity) 243/245 ($M^{+\cdot}$, 69), 209 (6), 200/202 (8), 189/191 (33), 167 (7), 155/157 (9), 154/156 (8), 149 (20), 143/145 (38), 141 (56), 127 (16), 125 (11), 115 (28), 113 (44), 99/101 (24), 97 (15), 87 (13), 85 (18), 79 (14), 78 (12), 77 (15), 73 (14), 71 (13), 63 (13), 62 (13), 55 (100), 41 (43).

Anal. Calcd. for $C_{10}H_{10}ClNO_4$: C, 49.30; H, 4.14; N, 5.75. Found: C, 49.08; H, 4.31; N, 5.61.

B. Compounds **4a** and **5a** (*via* **3a**).

The amine **1a** (327 mg, 1.21 mmoles) [2] was diazotized and subsequently treated with sodium azide as described under procedure A above, except that one-half of the quantities of the reagents were used. The dark-red solid obtained (*ca.* 340 mg) was shown by ^{1}H nmr to be an 84:16 mixture of 8-azido-9-chloro-10-nitro-2,3,4,5-tetrahydrobenzo[*b*][1,4]dioxocin (**3a**) (δ 6.88, aromatic H) and **4a** (δ 7.01, aromatic H), respectively; ir: ν 2115 (s, N_3). Neither fractional crystallization, nor column chromatography separated the mixture into its components; it was finally reduced with sodium borohydride (110 mg, 2.91 mmoles) in refluxing ethanol (15 ml), 15 minutes. Column chromatography gave **4a** as the first fraction (45 mg, 15% overall) and the amine **5a** as the second fraction (232 mg, 74% overall), mp (yellow needles) 98-100°; ir: ν 3490 (w), 3400 (w), 1626 (m), 1547 (s), 1494 (s), 1369 (m), 1341 (m), 1298 (m), 1228 (m), 1219 (m), 1187 (m), 1099 (w), 991 (m), 978 (m) cm^{-1}; ^{1}H nmr: δ 1.89 (m, 4H), 4.04 (br s, 2H, exchangeable), 4.17 (t, J = 5 Hz, 2H), 4.45 (t, J = 5 Hz, 2H), 6.42 (s, 1H); ms: m/z (% relative intensity) 258/260 ($M^{+\cdot}$, 80), 216/218 (7), 212/214 (5), 204/206 (53), 203/205 (17), 187/189 (40), 170/172 (22), 160 (7), 159 (17), 158 (29), 157 (37), 156 (39), 149 (9), 148 (12), 142 (13), 132 (7), 131 (16), 130 (20), 129 (34), 120 (11), 114 (21), 113 (11), 112 (14), 68 (38), 65 (27), 55 (100), 41 (44).

Anal. Calcd. for $C_{10}H_{11}ClN_2O_4$: C, 46.44; H, 4.29; N, 10.83. Found: C, 46.31; H, 4.11; N, 10.68.

C. Compounds **4a**, **5a** (*via* **3a**), **6a** and **7** (*via* **2a**).

The amine **1a** (376 mg, 1.40 mmoles) [2] was diazotized and subsequently treated with sodium azide as described under procedure B. Column chromatography furnished two fractions: the first (264 mg) was a mixture of **3a** + **4a** (see procedure B above); the second fraction was also a mixture of **2a** [2] + **6a** [1] (35 mg) as revealed by ir and ^{1}H nmr spectroscopy.

The first fraction was heated in ethylene glycol (3 ml) at 140-150° for one hour. Work-up as in procedure A followed by column chromatography afforded **4a** (78 mg, 23% overall) and **5a**. The latter was purified further by column chromatography (benzene) to yield 63 mg (17% overall).

The second fraction was thermolyzed in refluxing toluene (6 ml) for one hour. Column chromatography (benzene) furnished 17 mg (5%) of **6a** [1] and 10 mg (3%) of **7** [2].

7-Azido-8-chloro-9-nitro-, 8-Nitro-9-chloro-, 7-Amino-8-chloro-9-nitro- and 8,9-Dinitro-2,3,4,5-tetrahydrobenzo[*b*][1,4]dioxocins (**3b**, **4b**, **5b** and **6b**).

A. Compounds **4b** and **6b**.

The amine **1b** (345 mg, 1.28 mmoles) [2] was diazotized and then treated with sodium azide as described in procedure B. Subsequent thermolysis was carried out according to procedure A above. Column chromatography (benzene) furnished 70 mg (22%) of **4b**, 5 mg (1.5%) of **6b** [1] and 60 mg of an unidentified oil. Compound **4b** had mp (ethanol at 50°, off-white needles) 56-58°; ir: ν 1601 (w), 1562 (w), 1528 (s), 1483 (s), 1336 (m), 1306 (s), 1263 (m), 1171 (m), 1083 (w), 1058 (w), 984 (s), 925 (w), 893 (w), 861 (w) cm^{-1}; ^{1}H nmr: δ 1.94 (m, 4H), 4.26 (t, J = 5 Hz, 2H), 4.55 (t, J = 5 Hz, 2H), 7.05 (s, 1H), 7.68 (s, 1H); ms: m/z (% relative intensity) 243/245 ($M^{+\cdot}$, 53), 200/202 (18), 189/191 (19), 171/173 (9), 159/161 (8), 155/157 (16), 154/156 (12), 143/145 (9), 141 (6), 131/133 (5), 125/127 (14), 115 (10), 113 (19), 99/101 (18), 97 (20), 87 (7), 85 (12), 79 (7), 77 (10), 63 (18), 62 (18), 55 (100), 53 (24), 50 (25), 41 (32).

Anal. Calcd. for $C_{10}H_{10}ClNO_4$: C, 49.30; H, 4.14; N, 5.75. Found: C, 49.26; H, 4.03; N, 5.90.

B. Compound **3b**.

The amine **1b** (202 mg, 0.750 mmole) [2] in tetrahydrofuran (5 ml), concentrated hydrochloric acid (1.5 ml) and water (1 ml) was treated with sodium nitrite (300 mg, 4.3 mmoles) dissolved in water (1 ml), followed by addition of sodium azide (300 mg, 4.6 mmoles) according to procedure A (compound **4a**). Column chromatography afforded 175 mg (82%) of the azide **3b**, mp (ethanol:water = 4:1, v:v at 60°, pale-yellow needles) 73-74°; ir: ν 2130 (s), 2110 (s), 1530 (s), 1469 (m), 1452 (m), 1422 (m), 1358 (m), 1346 (m), 1324 (m), 1232 (w), 1184 (w), 1087 (w), 1081 (w), 1028 (w), 1006 (m), 992 (m) cm^{-1}; ^{1}H nmr: δ 1.99 (m, 4H), 4.33 (t, J = 5 Hz, 2H), 4.54 (t, J = 5 Hz, 2H), 7.33 (s, 1H); ms: m/z (% relative inten-

sity) 284/286 ($M^{+\cdot}$, 10), 256/258 (12), 210/212 (2), 168/170 (3), 156/158 (3), 126/128 (11), 100 (15), 98 (35), 91 (3), 75 (5), 72 (5), 63 (4), 55 (100), 41 (26).

Anal. Calcd. for $C_{10}H_9ClN_4O_4$: C, 42.19; H, 3.19; N, 19.68. Found: C, 41.91; H, 3.08; N, 19.48.

C. Compound **5b**.

The azide **3b** (83 mg, 0.29 mmole) was reduced with sodium borohydride (44 mg, 1.2 mmoles) in refluxing ethanol (5 ml), 0.5 hour, and purified by column chromatography to yield 60 mg (80%) of **5b**, mp (ethanol at −20°, yellow needles) 46-47°; ir: ν 3500 (w), 3400 (m), 1604 (s), 1529 (s), 1479 (s), 1342 (s), 1291 (m), 1220 (s), 1114 (s), 1043 (m), 974 (s) cm^{-1}; ^1H nmr: δ 1.94 (m, 4H), 4.29 (t, J = 5 Hz, 2H), 4.51 (t, J = 5 Hz, and br s, 4H, 2 hydrogens are exchangeable), 7.06 (s, 1H); ms: m/z (% relative intensity) 258/260 ($M^{+\cdot}$, 52), 216/218 (16), 215/217 (9), 204/206 (24), 186/188 (7), 174/176 (6), 170/172 (11), 158/160 (14), 142 (10), 140 (21), 114 (10), 112 (15), 102 (9), 100 (20), 77 (12), 76 (14), 65 (27), 55 (100), 41 (22).

Anal. Calcd. for $C_{10}H_{11}ClN_2O_4$: C, 46.44; H, 4.29; N, 10.83. Found: C, 46.23; H, 4.09; N, 10.75.

7-Azido-9-nitro-10-chloro-, 7-Chloro-8-nitro- and 7-Amino-9-nitro-10-chloro-2,3,4,5-tetrahydrobenzo[*b*][1,4]dioxocins (**3c**, **4c** and **5c**).

A. Compound **4c**.

The amine **1c** (70 mg, 0.26 mmole) [2] in tetrahydrofuran (3 ml) was diazotized with excess reagent according to the procedure B (compound **3b**) described above (subsequent addition of sodium azide was omitted). After 0.5 hour at 0°, the mixture was heated at 50-60° for an additional 0.5 hour and worked-up. Column chromatography furnished 31 mg (49%) of **4c**, mp (ethanol at 50°, pale-yellow needles) 58-59°; ir (potassium bromide): ν 1580 (m), 1507 (m), 1339 (m), 1303 (m), 1262 (s), 1050 (w), 981 (m), 968 (m), 844 (m), 814 (w), 798 (w), 763 (w), 733 (w) cm^{-1}; ^1H nmr: δ 1.95 (m, 4H), 4.32 (t, J = 5 Hz, 2H), 4.59 (t, J = 5 Hz, 2H), 6.90 (d, J = 9 Hz, 1H), 7.60 (d, J = 9 Hz, 1H); ms: m/z (% relative intensity) 243/245 ($M^{+\cdot}$, 43), 200/202 (17), 189/191 (19), 171/173 (10), 159/161 (7), 155/157 (6), 154/156 (10), 143/145 (5), 141 (8), 125 (8), 113/115 (15), 99/101 (16), 97 (11), 87 (10), 85 (17), 62 (12), 55 (100), 41 (27).

Anal. Calcd. for $C_{10}H_{10}ClNO_4$: C, 49.30; H, 4.14; N, 5.75. Found: C, 49.40; H, 4.20; N, 5.62.

Hydrolysis of the acetamido derivative **8** (780 mg, 2.51 mmoles) [2] with concentrated hydrochloric acid (4.0 ml) in refluxing ethanol (10 ml) for 1.5 hours, followed by purification of the mixture by column chromatography (chloroform), gave 141 mg (23%) of **4c** along with 270 mg of two or three unidentified products.

B. Compound **3c**.

The amine **1c** (305 mg, 1.13 mmoles) [2] was diazotized and subsequently treated with sodium azide according to procedure A (compound **4a**) above, with the exception that one-half of the quantities specified therein were used, to give 288 mg (89%) of **3c**, mp (ethanol at 60° and then −20°, orange needles) 85-87°; ir (chloroform): ν 2115 (s), 1529 (s), 1478 (m), 1453 (m), 1346 (s), 1298 (w), 1247 (w), 1088 (w), 1052 (w), 1023 (m), 1004 (w), 989 (w), 945 (w) cm^{-1}; ^1H nmr: δ 2.00 (m, 4H), 4.37 (t, J = 5 Hz, 2H), 4.58 (t, J = 5 Hz, 2H), 7.28 (s, 1H); partial ^1H nmr (carbon tetrachloride): δ 7.14 (s, 1H); ms: m/z (% relative intensity) 284/286 ($M^{+\cdot}$, 26), 256/258 (21), 210/212 (10), 184/186 (4), 168/170 (10), 156/158 (9), 140 (9), 138 (19), 128 (11), 126 (23), 112/114 (11), 110 (12), 102 (12), 100 (37), 98 (59), 91 (6), 77 (9), 76 (10), 75 (19), 73 (13), 72 (13), 63 (16), 55 (100), 41 (58).

Anal. Calcd. for $C_{10}H_9ClN_4O_4$: C, 42.19; H, 3.19; N, 19.68. Found: C, 42.04; H, 3.11; N, 19.48.

C. Compound **5c**.

The azide **3c** (49 mg, 0.17 mmole) in ethylene glycol (2 ml) was heated at 140-150° for one hour according to the procedure A (compound **4a**) described above. Column chromatography furnished 12 mg (27%) of **5c**.

The azide **3c** (119 mg, 0.418 mmole) in ethanol (5 ml) was reduced with sodium borohydride (71 mg, 1.9 mmoles) at 50-60°, 0.5 hour. Purification by column chromatography (petroleum ether:ethyl acetate = 2:1, v:v) afforded 74 mg (68%) of **5c**, mp (ethanol at 60° and then −20°, yellow needles) 92-93°; ir: ν 3495 (w), 3400 (m), 1613 (m), 1527 (s), 1482 (s), 1458 (m), 1346 (s), 1287 (m), 1259 (m), 1221 (m), 1168 (m), 1122 (m), 1089 (m), 1052 (m), 963 (m) cm^{-1}; ^1H nmr: δ 1.96 (m, 4H), 4.05 (br s, 2H, exchangeable), 4.32 (t, J = 5 Hz, 2H), 4.58 (t, J = 5 Hz, 2H), 7.04 (s, 1H); ms: m/z (% relative intensity) 258/260 ($M^{+\cdot}$, 39), 223 (2), 216/218 (6), 215/217 (4), 204/206 (15), 170/172 (7), 158/160 (8), 140/142 (11), 112/114 (9), 102 (5), 101 (6), 100 (10), 87 (4), 85 (8), 65 (12), 55 (100), 41 (19).

Anal. Calcd. for $C_{10}H_{11}ClN_2O_4$: C, 46.44; H, 4.29; N, 10.83. Found: C, 46.61; H, 4.37; N, 10.77.

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