Monatshefte für Chemie 133, 241–248 (2002)

Monatshefte für Chemie
Chemical Monthly

Preparation of Hydroxy-Substituted Hexahydrophthalazinones from Cyclohexane- and Norbornanelactones or Ketallactones

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Summary. Cyclohexane- and norbornanelactones (**1**, **5**) as well as ketallactones (**3**, **7**) and their hydrolytic products (**2**, **4a**,**b**, **8**) react with hydrazine hydrate to yield the hydroxy-substituted hexahydrophthalazinones **9**–**12**. In the cyclizations, the configurations of the bridgehead atoms remain unaltered, and the hydroxy group retains its original position in the hydrazinolysis of **5** and **7**. The compounds were characterized by IR, ^{1}H, and ^{13}C NMR spectroscopy.

Keywords. Amino acids; Heterocycles; Ketones; Phthalazinones; Hydrazinolysis.

Introduction

No simple method for the preparation of 2-substituted 4- or 5-hydroxycyclohexane- or -norbornanecarboxylic acids has been described in the literature. One of the known procedures applies electrophilic addition to substituted *cis*- or *trans*-4-cyclohexenecarboxylic acids followed by lactonization and elimination of the undesired group [1–4]. Another route is the *Baeyer-Villiger* oxidation of 2-*endo*-substituted norbornane-7-ones with *m*-chloroperbenzoic acid which results in two isomeric lactones; however, separation of these has not been satisfactorily achieved on a preparative scale [5]. 2-Aroylcyclohexane derivatives yield suitable products upon reaction with aryllithium; the oxo or hydroxy functions have to be incorporated in subsequent steps in this case [6, 7].

By employing *Friedel-Crafts* reactions, we recently have devised a new and simple method for the transannular hydroxylation of aroylcyclohexane- and norbornanecarboxylic acids. The aroyllactones and ketallactones prepared this way [8] seemed to be suitable starting compounds for the above functionalized cyclohexane- and norbornanehydroxy derivatives. As the lactones can be viewed as precursors of the γ-oxo-cycloalkanecarboxylic acids, they were also used for the preparation of cycloalkane-condensed dihydropyridazinones or their methylene-bridged derivatives.

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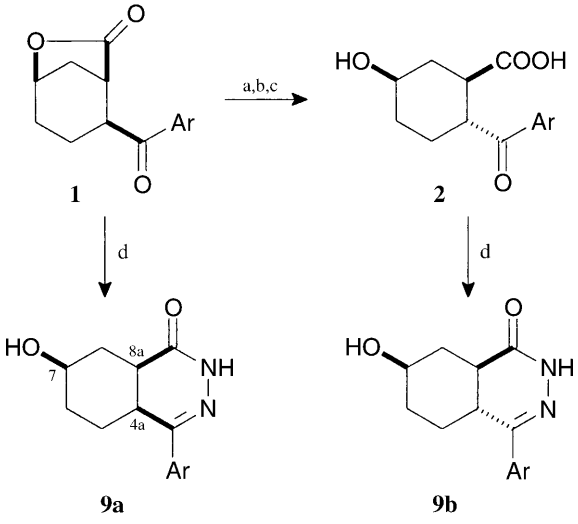
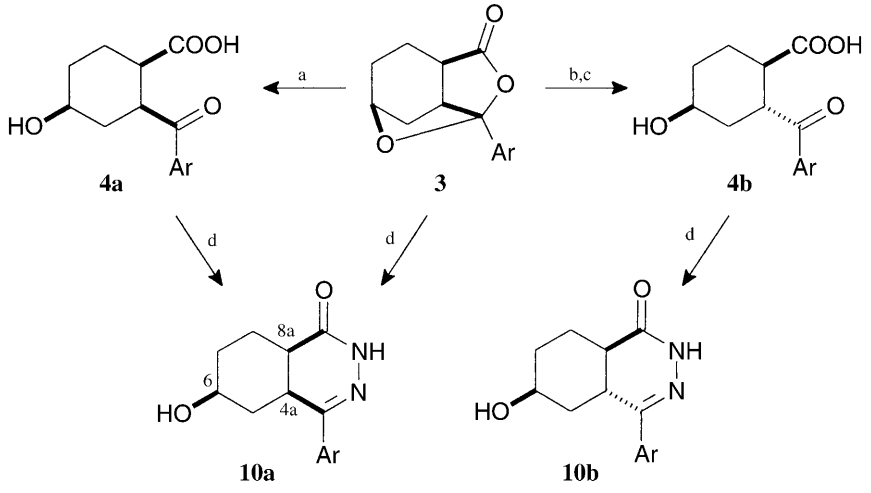
Results and Discussion

In neutral or acidic ethanolic solution, lactone **1** does not undergo hydrolysis. Treatment of **1** in ethanol with aqueous NaOH followed by acidification affords the 2*t*-aroyl-5*c*-hydroxy-1*r*-cyclohexanecarboxylic acid **2** (Scheme 1); in deuteriochloroform, **2** exists predominantly as the more stable conformer containing equatorial carboxyl, aroyl, and hydroxy groups.



Scheme 1. a: $EtOH/H_2O$, b: $NaOH/H_2O$, c: HCl/H_2O, d: $H_2NNH_2 \cdot H_2O$; Ar = *p*-tolyl

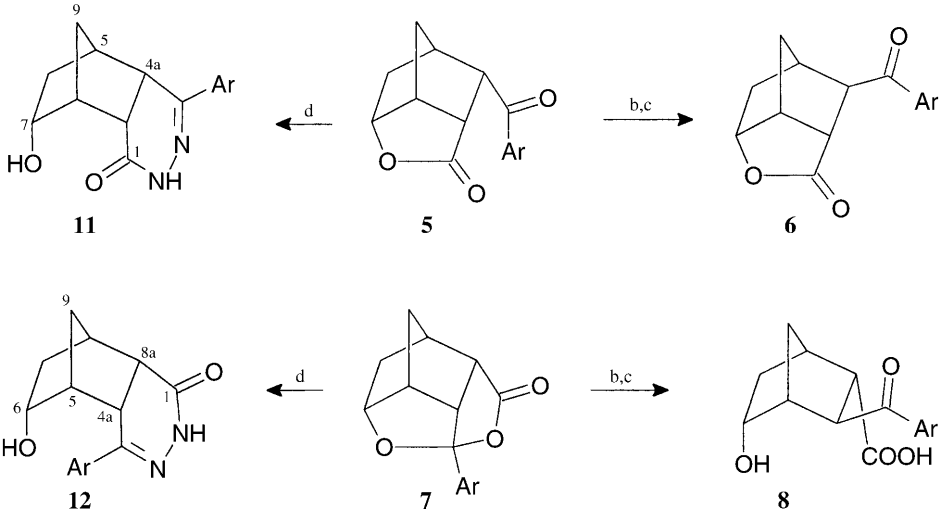
On warming to 80°C in aqueous ethanol, **3** readily hydrolyses to 2*c*-aroyl-4*c*-hydroxy-1*r*-cyclohexanecarboxylic acid (**4a**), all three substituted carbons retaining their original configuration (Scheme 2). In $CDCl_3$ at room temperature, NMR measurements show a predominant conformation of **4a** with axial carboxyl and equatorial aroyl and hydroxy groups. Upon standing at room temperature or



Scheme 2. a: $EtOH/H_2O$, b: $NaOH/H_2O$, c: HCl/H_2O, d: $H_2NNH_2 \cdot H_2O$; Ar = *p*-tolyl

warming in EtOAc, **4a** quickly recycles to ketallactone **3**. However, hydrolysis with base results in 2*t*-aroyl-4*c*-hydroxy-1*r*-cyclohexanecarboxylic acid **4b** with axial hydroxy and equatorial aroyl and carboxyl groups.

Similar to **1**, lactone **5** does not undergo hydrolysis in aqueous ethanol (Scheme 3). In the presence of acid, **5** isomerizes to **6** which contains an *exo* aroyl group. In basic solution, the lactone opens to the aroylhydroxycarboxylate (in which both the hydroxy and the carboxylate groups are *endo* and the aroyl group is presumably *exo*); acidification leads to the *exo* aroyl compound **6** [8]. These examples illustrate the facile *endo* → *exo* isomerization of the aroyl group at the norbornane skeleton.



Scheme 3. a: $EtOH/H_2O$, b: $NaOH/H_2O$, c: HCl/H_2O, d: $H_2NNH_2 \cdot H_2O$; Ar = *p*-tolyl

Unlike **3**, ketallactone **7** does not undergo hydrolysis in neutral aqueous ethanolic solution, even on warming (80°C) for a prolonged time (10 h). Under basic conditions, **7** furnishes **8** in which both the hydroxy and carboxyl groups retain their original *endo* positions, but the aroyl group has been isomerized from *endo* to *exo*.

With hydrazine, lactones **1** and **5** and ketallactones **3** and **7** react analogously to γ-oxo-carboxylic acids to furnish pyridazinones [9–13]. As the position of the hydroxy group in the products may be questionable, both lactones and hydrolytic products were applied in the reactions (the latter only when the steric positions of the carboxyl and oxo groups allows cyclization).

With hydrazine hydrate in ethanol, both lactone **1** and 2*t*-aroyl-5*c*-hydroxy-1*r*-cyclohexanecarboxylic acid (**2**) react. The reaction of **1** yields phthalazinone **9a** containing a *cis*-condensed hetero ring and a 7-*cis*-hydroxy group in an equatorial position. The reaction of **2** results in **9b** with a diequatorial *trans*-condensed hetero ring and the hydroxy group in the *cis*-equatorial position to the lactam carbon bond. With hydrazine, ketallactone **3** and its hydrolysis product **4a** give the same phthalazinone **10a** in which the rings are *cis*-annelated with axial carbonyl and equatorial C=N groups. The hydroxy group is *cis*-oriented with respect to the carbonyl group and hence occupies an equatorial position. However, **4b** gives the diequatorial *trans*-fused compound **10b** with an axial hydroxy group.

On hydrazinolysis, both norbornane lactone **5** and ketallactone **7** lead to methylene-bridged di-*endo*-fused phthalazinones **11** and **12** in which the hydroxy group occupies an *endo*-orientation.

Structure determination

The spectroscopic data[1] (Tables 1–3) shed light on the spatial structures of the new compounds. Whereas all three substituents on the cyclohexane ring are equatorial in **2**, one of them (the carboxyl group in **4a** and the hydroxy group in **4b**) becomes axial in **4a**,**b**. Due to the more strained structures in **4a**,**b**, the sums of the carbon chemical shifts of the cyclohexane ring in these compounds is significantly smaller (245.9 and 242.9 ppm, respectively) than that for **2** (261.3 ppm) [14a].

For **4b**, the equatorial orientation of H-6 follows from the higher chemical shift (3.87 ppm) as compared with that of H-6 in **4a** (3.69 ppm) and that of H-7 in **2** (∼3.4 ppm). The 1,3-diaxial interactions with the hydroxy group are revealed in the upfield shifts of the signals of C-4a and C-8 relative to the corresponding positions in **2** (C-8a,5) and **4a** (C-4a,8) by 2.9 and 4.8 ppm (**2**) and 3.9 and 1.3 (**4a**), respectively, and are caused by the steric compression shift (field effect [14b, 15]). Similarly, the axial position of the carboxyl group in **4a** is obvious from the downfield shift of the signal of the equatorial H-8a atom (2.90 ppm; 2.68 and 2.59 ppm for **2** and **4b**) and from the small splittings (quartet, 4.1 Hz) of this signal, thus excluding diaxial-type couplings [16, 17]. The field effects on the C-7,8a signals are 4.8 and 3.2 ppm as compared to **4b**.

The same principles were used to determine the stereostructures of the further compounds. Additionally, it is to be noted that the sum of the chemical shifts of the cyclohexane carbons of **9a** and **9b** differ significantly (224.3 and 244.5 ppm) because of the strained *cis*-annelation in **9a** leading to upfield shifts of the lines in question. The difference in the annelation in **10a**,**b**, however, is not reflected in a similar way (the sum of the shifts are 227.3 and 226.5 ppm) because the sterically more favourable *trans*-annelation in **10b** is compensated by the 1,3-diaxial interactions of the hydroxy group which is equatorial in **10a**. Consequently, the field effect of different origin in **10b** on the one hand and in **9a** and **10a** on the other is manifested in similar upfield shifts of the cyclohexane carbon lines for both isomers **10a**,**b** as well as for **9a**. The anisotropy of the carbonyl group [14c] in the 1,3-diaxial position leads to a significant downfield shift of the H-7 signal in **9a** (3.92 ppm; **9b**: 3.30 ppm).

The di-*endo* annelation of the hetero ring and the norbornane moiety is shown by the double doublet splittings of the signals of the annelational hydrogens 4a,8a in **11** and **12** in accordance with earlier findings [18, 19] due to the significant H-4a,5 and H-8,8a couplings (in contrast to the di-*exo* analogues where these couplings are insignificant and only the H-4a,8a interaction leads to doublet splittings of the H-4a,8a signals). In the *exo*-*endo*-type **8**, the hydrogen on the *endo*-substituted C-8a gives a ddd signal, whereas the signal of H-4a attached to the *exo*-substituted carbon appears as a doublet.

[1] For comparison of the analogous data, the numbering of **9a** and **10a** (Scheme 1 and 2) was applied

Table 1. ^{1}H NMR data[a] of compounds **2**, **4a**,**b**, **8**, **9a**,**b**, **10a**,**b**, **11**, and **12**[b]

	CH_3	CH_2(9)[c]	CH_2 or CH, alicyclic ring(s)[d]						H-4a	H-8a	C*H*OH	OH	OH/NH	H-2′,6′	H-3′,5′
	s (3H)	2 × *d* (2 × 1H)	Pos. 5		Pos. 6 or 7		Pos. 8		(1H)[e]	(1H)[f]	m(1H)[g]	d(1H)[h]	(1H)[i]	Tolyl group[j]	
2	2.29	–	1.05[k]	~1.78[l]	1.29[k]	~1.78[l]	1.21[k]	2.11[m]	~3.42[n]	2.68	~3.4[n]	4.77	12.1	7.81	7.25
4a	2.43	–	1.86[k]	2.05[m]	1.45[k]	1.75	1.82	2.12	3.61	2.90	3.69	4.70	12.0	7.78	7.36
4b	2.29	–	1.41[o]	1.67[m]	1.21[o]	1.82[m]	1.73[l]		~3.87[n]	2.59	~3.87[n]	4.74	12.0	7.78	7.25
8	2.29	1.19	2.20[m]		0.92[p]	1.73[o]		2.40[q]	4.29	3.38	4.11	5.17	12.15	7.87	7.25
9a	2.30	–	1.38[k]	1.64[m]	~1.75[l]	2.12[m]	~1.73[l]	2.71[m]	3.28	2.91	3.92	~3.4[r]	11.0	7.67	7.21
9b	2.25[l]	–	0.92[k]	1.84[m]	1.09[k]	1.73[m]	0.98[k]	~2.27[l]	2.10	2.52	3.3[r]	4.68	10.8	7.13	7.18
10a	2.24[l]	–	1.06[k,n] 1.63[m,s]		1.06[k,n]	1.63[m,s]	1.35[o]	~2.22[l]	3.22	2.53	3.42	4.63	10.83	7.61	7.15
10b	2.24	–	1.09[o]	~1.87[l]	1.28[o]	1.63[m]	1.45[k]	~1.87[l]	2.89	2.03	3.81	4.46	10.78	7.14	7.11
11	2.30	1.36 1.44	2.71[q]		0.73[m]	1.54[o]		2.46[q]	3.57	2.82	4.04	4.70	10.49	7.54	7.18
12	2.23	1.27 1.38	2.54[q]		0.92[m]	1.69[o]		2.51[q]	3.44	2.88	3.92	4.32	10.58	7.48	7.09

[a] In *DMSO*-d_6 solution at 500 MHz, chemical shifts in ppm ($\delta_{TMS} = 0$ ppm), coupling constants in Hz; [b] assignments were supported by HMQC and COSY for **4a**, by NOE measurements for **12**; [c] AB-type spectrum, $J = 9.8$ (**11**, **12**), singlet-like signal (2H) for **8**; [d] total intensity: 6H, pos. 5, 6, 8 (**2**, **9**), 6H, pos. 5, 7, 8 (**4**, **10**), 4H, pos. 5, 7, 8 (**8**, **12**), 4H, pos 5, 6, 8 (**11**); [e] triple doublet, $J = 10.4$, 4.0 and 4.0 (**4a**), *d*, $J = 6.3$ (**8**), *ddd*/*dt*, $J = 13.1$, 5.9, and 4.0 (**9a**, **10a**), 15.0, 15.0, and 2.8 (**9b**), 15.5, 12.6, and 3.3 (**10b**), *dd*, $J = 14.0$ and 2.6 (**11**), 14.1 and 4.0 (**12**); [f] *ddd*, $J = 13.4$, 10.7, and 3.2 (**2**), 6.0, 4.0 and 2.0 (**8**), 15.2, 12.0, and 3.3 (**10b**), *qu*, $J = 4.1$ (**4a**), *dt*, $J = 10.8$, 10.8, and 5.2 (**4b**), 15.1, 15.1, and 3.2 (**9b**), broad singlet (~*t*) for **9a** and **10a**, *dd*, $J = 14.0$ and 4.2 (**11**), 14.0 and 3.0 (**12**); [g] *td*, $J = 10.0$, 3.8, and 3.8 (**8**), 6.8, 3.3, and 3.3 (**11**), *tt*, $J = 9.5$ and 4.0 (**4a**), 12.0 and 4.0 (**9a**), coalesced *ddd* (**10a**), *qud* (**12**), broad singlet-like signal (**10b**); [h] doublet, $J = 4.2$ (**2**, **9b**), 5.0 (**10a**), 2.7 (**10b**, **11**), 5.6 (**12**), broadened singlet-like signal (**4a**,**b**, **8**); [i] broadened signal of the carboxyl (**2**, **4a**,**b**, **8**) or amide group (**9a**,**b**, **10a**,**b**, **11**, **12**); [j] AA′BB′-type spectrum, 2 × ~*d* (2 × 2H), $J = 8.2 \pm 0.4$; [k,m,o] ~*qu*/*d*/*t* ($J = \sim 13$ Hz, for 5-H in **8**: 3.4 Hz); [l,n,s] overlapping signals; [p] *dd* ($J = 13.0$, 3.5); [q] singlet-like signal; [r] hidden by the H_2O signal of the solvent

Table 2. ^{13}C NMR chemical shifts[a] of compounds **2**, **4a**,**b**, **8**, **9a**,**b**, **10a**,**b**, **11**, and **12**[b]

	C-1[c]	C-4[d]	C-4a	C-5	C-6	C-7	C-8	C-8a	C-9	CH_3	Carbons in tolyl ring			
											C-1	C-2′,6′	C-3′,5′	C-4′
2	176.3	202.7	46.1	28.6	35.1	68.9	38.5	44.1	–	22.0	134.1	129.2	130.2	144.4
4a	174.9	201.3	45.1	34.3	68.0	31.9	25.1	41.5	–	21.5	134.9	128.5	129.5	142.7
4b	177.1	203.2	41.2	32.4	64.1	36.7	23.8	44.7	–	22.0	134.0	129.1	130.2	144.3
8	175.1	200.5	42.8	49.9	71.0	34.3	40.4	48.1	36.2	22.0	134.1	129.6	130.1	144.4
9a	168.7	152.9	33.7	25.0	36.6	57.2	34.2	37.6	–	21.7	132.0	126.5	130.1	140.0
9b	169.9	156.9	38.6	28.5	35.1	68.8	35.6	37.9	–	21.7	134.7	128.5	129.3	138.6
10a	169.5	152.4	34.2	34.0	68.6	32.5	22.6	35.4	–	21.7	132.7	126.4	130.1	139.9
10b	169.9	157.4	33.0	36.9	64.2	31.8	20.6	~40.0[e]	–	21.7	134.5	128.4	129.3	138.6
11	167.7	147.8	40.3	49.2	34.1	71.9	43.2	38.8	37.7	21.7	135.4	126.9	129.8	138.9
12	167.5	150.6	37.9[f]	49.2	72.0	35.0	42.7	41.4	37.9[f]	21.7	135.8	127.1	129.4	138.5

[a] In ppm ($\delta_{TMS} = 0$ ppm) at 126.7 MHz, solvent: *DMSO*-d_6; [b] assignments were supported by DEPT, HMQC, and, except for **4a**,**b**, also by HMBC measurements; [c] C=O-carbons of carboxylic (**2**, **4**, **8**) or amide groups (**9**–**12**); [d] ketone C=O (**2**, **4**, **8**) or C=N carbons (**9**–**12**); [e] hidden by the signal of the solvent; [f] overlapping lines

Table 3. Characteristic IR frequencies (cm^{-1}) of compounds **2**, **4a**,**b**, **8**, **9a**,**b**, **10a**,**b**, **11**, and **12** (KBr pellets)

	ν(OH) band (sharp)[a]	ν(OH) or ν(NH) band (broad or diffuse)[b]	ν(C=X) band[c]	ν(C=O) or amide-I band	$\gamma(C_{Ar}H)$ band[d]
2	3450	3400–2500	1702	1669	820
4a	3420	3400–2500	1701	1678	812
4b	3365	3400–2500	1733	1655	822
8	3376	3400–2500	1697	1670	824
9a	3500	3225	1613	1672	835, 819
9b	3478	3237	1614	1683	816
10a	3460	3245		1659	822
10b	3320	3195		1674	823
11	3471	3203	1619	1669	831
12	3404	3250	1622	1645	825

[a] ν(OH) band of the alcohol group; [b] carboxylic OH group for **2**, **4a**,**b**, and **8**, ν(NH) band for **9a**,**b**, **10a**,**b**, **11**, and **12**; [c] ν(C=O) band (*X*=O) for **2**, **4a**,**b**, and **8**, ν(C=N) band (*X*=N) for **9a**,**b**, **10a**,**b**, **11**, and **12**; [d] split band for **9a**

NOE measurements [14d, 20] on **12** demonstrated the *endo*-position of the hydroxy group. Irradiation of H-6 caused an enhancement of the intensity of H-9-*exo* at 1.27 ppm. For the *endo*-counterpart, the doublet of the latter hydrogen responded in another NOE experiment when the H-4a signal was saturated. This is a direct proof of the di-*endo*-annelation of the hetero ring and the norbornane skeleton. The anisotropy of the close-lying benzene ring [14e] is seen in an upfield shift of the hydroxy signal for **12** (4.32 ppm; **11**: 4.70 ppm).

Experimental

IR spectra were determined in KBr discs on a Bruker IFS-55 FT-spectrometer controlled by Opus 2.0 software. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ solution in 5 mm tubes at room temperature on a Bruker DRX-500 FT spectrometer at 500 (^1H) and 126 (^{13}C) MHz, respectively, using the deuterium signal of the solvent as the lock and *TMS* as internal standard. DEPT spectra were run in a standard way, using only the $\theta = 135°$ pulse to separate the CH/CH_3 and CH_2 lines phased up and down, respectively. For NOE difference, COSY, HMQC, and HMBC measurements, the standard Bruker pulse programs were used. The results of elemental analyses agreed satisfactorily with the calculated values.

5c-Hydroxy-2t-p-toluoyl-1r-cyclohexanecarboxylic acid (**2**; $C_{15}H_{18}O_4$)

A solution of 3.0 g lactone **1** [8] in a mixture of 3.0 g KOH and 30 cm^3 MeOH was refluxed for 1 h and then evaporated to dryness. The residue was dissolved in 50 cm^3 H_2O and acidified with 36% HCl to *pH* 3. The separated solid was filtered off by suction, washed with 20 cm^3 H_2O, and dried to give 2.5 g product (78%) which was crystallized from *n*-hexane–acetone. M.p.: 115–118°C.

4c-Hydroxy-2c-p-toluoyl-1r-cyclohexanecarboxylic acid (**4a**; $C_{15}H_{18}O_4$)

A solution of 1.5 g **3** [8] in a mixture of 50 cm^3 EtOH and 20 cm^3 H_2O was refluxed for 4 h. After evaporation to dryness, the residue was crystallized from EtOAc to give 1.04 g (65%) **4a**. M.p.: 159–162°C.

4c-Hydroxy-2t-p-toluoyl-1r-cyclohexanecarboxylic acid (**4b**; $C_{15}H_{18}O_4$)

A mixture of 2.0 g **3** and 1.0 g KOH in 10 cm^3 MeOH was warmed at 80°C for 1 h, cooled, acidified to *pH* 4 by dropwise addition of 36% HCl, and evaporated to dryness. The residue was suspended in 10 cm^3 H_2O, and the solid (1.92 g, 89%) was filtered off by suction, washed with 5 cm^3 H_2O, dried, and crystallized from EtOAc. M.p.: 172–173°C.

5-endo-Hydroxy-3-exo-p-toluoylbicyclo[2.2.1]heptane-2-endo-carboxylic acid (**8**; $C_{16}H_{18}O_4$)

A solution of 0.1 g **7** [8] in a mixture of 0.1 g KOH and 5 cm^3 MeOH was refluxed for 1 h and evaporated to dryness. The residue was dissolved in 2 cm^3 H_2O and acidified to *pH* 3 by dropwise addition of 36% HCl. After filtration by suction, the solid (0.08 g, 75%) was washed with 3 cm^3 H_2O, dried, and crystallized from EtOAc. M.p.: 193–195°C.

General procedure for the preparation of **9a**,**b**, **10a**,**b**, **11**, *and* **12**

A mixture of lactone, ketallactone, or their hydrolysis products (g) in MeOH (cm^3) was refluxed for 2 h with 90% $N_2H_4 \cdot H_2O$ (g) (**1**: 1.2 g, 20 cm^3, 1.3 g; **2**: 0.65 g, 10 cm^3, 0.5 g; **3**: 1.2 g, 20 cm^3, 1.2 g; **4a**: 0.9 g, 15 cm^3, 0.4 g; **4b**: 0.41 g, 5 cm^3, 0.4 g; **5**: 0.61 g, 10 cm^3, 0.6 g; **7**: 0.55 g, 10 cm^3, 0.8 g). After evaporation, 15 cm^3 H_2O was added to the residue, and the solid was filtered off by suction, washed with water, dried, and crystallized.

7c-Hydroxy-4-p-tolyl-4ar,5,6,7,8,8ac-hexahydrophthalazin-1(2H)-one (**9a**; $C_{15}H_{18}N_2O_2$)

Yield: 0.9 g (71%); m.p.: 221–223°C (MeOH).

7c-Hydroxy-4-p-tolyl-4ar,5,6,7,8,8at-hexahydrophthalazin-1(2H)-one (**9b**; $C_{15}H_{18}N_2O_2$)

Yield: 0.5 g (78%); m.p.: 218–220°C (MeOH).

6c-Hydroxy-4-p-tolyl-4ar,5,6,7,8,8at-hexahydrophthalazin-1(2H)-one (**10a**; $C_{15}H_{18}N_2O_2$)

From **3**: yield: 1 g (79%); from **4a**: yield: 0.77 g (87%), m.p.: 233–235°C (MeOH).

6c-Hydroxy-4-p-tolyl-4ar,5,6,7,8,8at-hexahydrophthalazin-1(2H)-one (**10b**; $C_{15}H_{18}N_2O_2$)

Yield: 0.35 g (88%); m.p.: 207–208°C (EtOH).

7-endo-Hydroxy-5t,8t-methano-4-p-tolyl-4ar,5,6,7,8,8ac-hexahydrophthalazin-1(2H)-one (**11**; $C_{16}H_{18}N_2O_2$)

Yield: 0.42 g (66%); m.p.: 288–291°C (EtOH).

6-endo-Hydroxy-5t,8t-methano-4-p-tolyl-4ar,5,6,7,8,8ac-hexahydrophthalazin-1(2H)-one (**12**; $C_{16}H_{18}N_2O_2$)

Yield: 0.40 g (69%); m.p.: 258–260°C (EtOH).

Acknowledgements

The authors are indebted to Mrs. *Csiszár-Makra* for the typing of the manuscript. Grants: OTKA T 25415, OTKA T 29651, and FKFP-0200/2000.

References

[1] McQuillin FJ, Ord WO, Simpson PL (1964) J Chem Soc 5526
[2] Kutscherow VF, Shabanov AL, Onishchenko AS (1963) Bull Acad Sci USSR; (1963) Div Chem Sci (Eng Transl) 763; (1963) Izv Akad Nauk SSSR Ser Khim 844
[3] Factor A, Traylor TG (1968) J Org Chem **33**: 2607
[4] Wu HJ, Tsai SH, Chern JH, Liu HC (1997) J Org Chem **62**: 6367
[5] Mehta G, Mohal N (1998) J Chem Soc Perkin Trans 1, 505
[6] Angelucci F, Arcamone F, Barchielli G, Suarato A, Vanotti E, Penco S (1984) J Chem Soc Chem Commun 530
[7] Yadav J, Corey P, Hsu Chi-Tung, Perlman K, Sih CJ (1981) Tetrahedron Lett **22**: 811
[8] Szabó JA, Sohár P, Böcskei Zs, Stájer G, Bernáth G (1999) Synthesis 1564
[9] Jucker E, Süess R (1959) Helv Chim Acta **42**: 2506
[10] Stájer G, Szabó AE, Bernáth G, Sohár P (1994) Heterocycles **38**: 1061
[11] Sohár P, Stájer G, Szabó AE, Bernáth G (1996) J Mol Struct **382**: 187
[12] Stájer G, Csende F, Bernáth G, Sohár P, Szúnyog J (1994) Monatsh Chem **125**: 933
[13] Reddy RS, Saravanan K, Kumar P (1998) Tetrahedron **54**: 6553
[14] Sohár P (1983) Nuclear Magnetic Resonance Spectroscopy. CRC Press, Boca Raton, Florida: a) vol 2, p 165; b) vol 2, pp 154–155; c) vol 1, p 32 and vol 2, p 61; d) vol 1, pp 194–197; e) vol 1, pp 35–38 and vol 2, p 73
[15] Grant GM, Cheney BV (1967) J Am Chem Soc **89**: 5315
[16] Karplus M (1959) J Chem Phys **30**: 11
[17] Karplus M (1960) J Chem Phys **33**: 1842
[18] Sohár P, Stájer G, Bernáth G (1983) Org Magn Reson **21**: 512
[19] Sohár P, Pelczer I, Stájer G, Bernáth G (1987) Magn Reson Chem **25**: 584
[20] Sanders JKM, Mersch DJ (1982) Prog Nucl Magn Reson **15**: 353, and references cited therein

Received September 3, 2001. Accepted October 8, 2001