Stereoselective One-Pot Method for the Introduction of an Amino Group to a Cyclohexane Ring. Preparation of the Octahydroisobenzofuro[7a,1-*d*]oxazole Ring System

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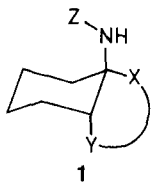
Stereoselective one-pot synthesis of octahydroisobenzofuro[7a,1-*d*]-oxazole-2,5-diones **7–14** via base-catalyzed addition–cyclization reaction of hydroxy lactones **4a–4d** with various isocyanates **5a–5f** is described. The ring system contains diastereoselectively regulated three contiguous asymmetric carbons.

Stereoselective introduction of an amino group to substituted cyclohexane derivatives has drawn the attention of many chemists.[2,3] As a part of our project to develop novel synthetic routes to 1,2-amino alcohol derivatives of biological interest, we needed to prepare 1,2-cis-fused cyclohexylamino moieties **1**. Since 1,2-amino alcohols were often successfully prepared by the base-promoted ring cleavage of oxazolidinones,[4] our interest was focused on building up oxazolidinones having the 1,2-cis-fused cyclohexane framework. For the construction of such heterocyclic compounds, intramolecular cyclization of allylic carbamate is considered to be a very useful methodology. In an allylic system without activating groups on the olefin, the electrophile mediated nucleophilic substitution reaction has been well studied.[5–7] In the case of activated allylic carbamates with an electron-withdrawing group, highly diastereoselective intramolecular conjugate addition has been reported.[8] But the application of this cyclization reaction to polycyclic systems is limited.[9,10] For our synthetic purpose, it is expected that, like **4a–d**, if the electrophilic center is positioned at the bridge head carbon of a bicyclic system, the fused ring system would restrict the direction of the intramolecular Michael addition. With this in mind, we studied the intramolecular Michael addition of allylic carbamates derived from bicyclic cyclohexene derivatives **4a–d** in order to fix the neighboring two carbon functional groups in a cis position on the cyclohexane ring. In this report, we describe a simple, highly stereoselective, one-pot preparation of tricyclic oxazolidinones, in which the 1,2-disubstituents of the cyclohexane ring are cis and the three contiguous asymmetric carbons are diastereoselectively regulated.



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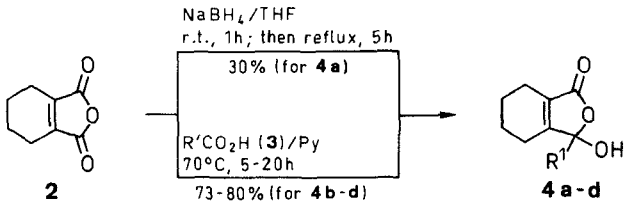
The requisite starting materials, 3-hydroxy-4,5,6,7-tetrahydrophthalides **4a–4d** were easily prepared as follows: Sodium borohydride (0.5 equivalent) reduction of 4,5,6,7-tetrahydrophthalic anhydride **2** gave the hydroxy lactone **4a**.[11] 3-Methyl, ethoxycarbonylmethyl, and cyanomethyl derivatives **4b–4d** were prepared by the reaction of the anhydride **2** with 2 equivalents of malonic acid (**3b**), monoethyl malonate (**3c**) and cyanoacetic acid (**3d**), respectively, in pyridine in high yields[12] (Scheme 1). All the spectral data of these compounds supported the cyclic hydroxy lactone structure, and not the isomeric open chain oxo acid structure (Table 1).



3	R′	4	R[1]
b	CH_2CO_2H	a	H
c	CH_2CO_2Et	b	Me
d	CH_2CN	c	CH_2CO_2Et
		d	CH_2CN

Scheme 1

The reaction of the hydroxy lactones **4a–4d** with various isocyanates **5a–5f** was facilitated in acetonitrile or dichloromethane in the presence of catalytic amount (about 5 mol%) of triethylamine or 1,8-diazabicyclo-[5.4.0]undecene (DBU) (Scheme 2). The formation of the allylic carbamates **6** was completed within 30 minutes as revealed by TLC monitoring, and the corresponding carbamates were easily isolated by quenching the reaction with acetic acid. On prolonged stirring without quencher, however, the subsequent cyclization to tricyclic compounds proceeded in high yield. Summary of these results are shown in Table 2. The stereochemical relation between cyclohexane and the lactone ring of all the compounds listed in Table 2 were determined to be cisoid by measuring NOE between H-5a and *N*-substituents. No evidence for the formation of the transoid compounds (i.e. type **15**) were found throughout these experiments.

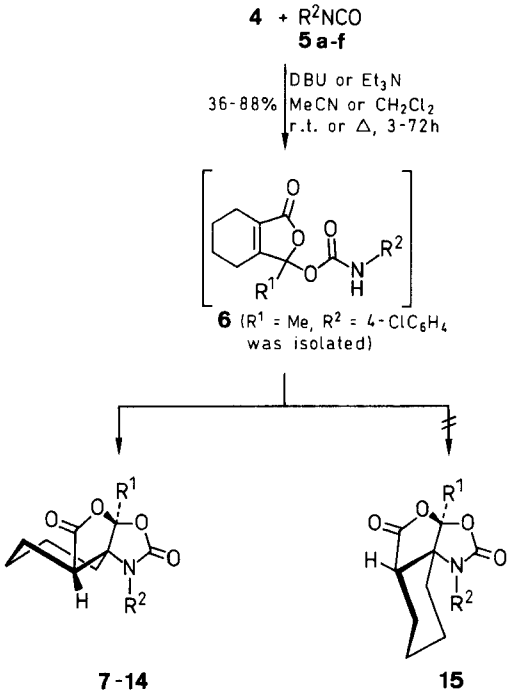
As shown in Table 2, this cyclization reaction is quite general to various substituted isocyanates. Even the sterically hindered aryl isocyanate, for example 2,6-dimethylphenyl isocyanate (**5c**), reacted with **4b** to give **9** in good yield.

The cis-fusion of cyclohexane and the lactone ring was also clearly evidenced by the X-ray crystallographic analysis of **7**. A computer generated conformation of **7** is shown in the Figure.[13] It is obvious from the X-ray analysis that the dihedral angle of the newly formed lactone–oxazolidinone bicyclic system is ca. 120°, and the cyclohexane ring is arranged in chair form cis to the lactone ring, which may ease the strain of the ring system.

Table 1. Hydroxy Lactones **4b–d** Prepared

Product	Reaction Time (h)	Yield (%)	mp (°C) (solvent)	Molecular Formula[a]	^{1}H NMR ($CDCl_3$/TMS) δ, J (Hz)	^{13}C NMR ($CDCl_3$/TMS) δ	MS (CI) m/z (%)
4b	20	73	113–114 (hexane/$CHCl_3$)	$C_9H_{12}O_3$ (168.2)	1.64 (s, 3H, CH_3), 1.70 (m, 4H, H-5, 6), 2.24 (m, 4H, H-4, 7), 4.70 (br s, 1H, OH)	19.52, 21.33, 21.42, 23.37, (C-4 to 7), 106.14 (C-3), 127.31 (C-7a), 163.51 (C-3a), 172.10 (C-1)	169 (M^+ +1, 100), 151 (43)
4c	20	80	81–82 (hexane/Et_2O)	$C_{12}H_{16}O_5$ (240.3)	1.28 (t, 3H, CH_3), 1.75 (m, 4H, H-5, 6), 2.18 (m, 3H, H-4, 7), 2.38 (m, 1H, H-4′ or 7′), 2.65 (d, 1H, CHCO, J = 16), 2.95 (d, 1H, CHCO, J = 16), 4.22 (q, 2H, OCH_2), 6.28 (br, s, 1H, OH)	14.02 (CH_3), 19.77, 21.33, 21.42, 21.57 (C-4 to 7), 40.11 ($\underline{C}H_2CO_2Et$), 51.74 (OCH_2), 104.00 (C-3), 129.29 (C-7a), 160.70 (C-3a), 170.57, 170.69 (C-1, $\underline{C}O_2Et$)	241 (M^+ +1, 7), 223 (100)
4d	5	80	140–141 (hexane/$CHCl_3$)	$C_{10}H_{11}NO_3$ (193.2)	1.72 (m, 4H, H-5, 6), 2.27 (m, 4H, H-4, 7), 3.02 (ABq, 2H, CH_2CN, J = 16), 5.28 (br s, 1H, OH)	19.68, 21.14, 21.33, 21.54 (C-4 to 7), 26.92, 102.84 (C-3), 115.03 (CN), 129.33 (C-7a), 160.40 (C-3a), 169.81 (C-1)	194 (M^+ +1, 100), 176 (35), 153 (13)

[a] Satisfactory microanalyses obtained: C ±0.26, H ±0.28, N ±0.15.



5	R^2	5	R^2
a	4-ClC_6H_4	**d**	Me
b	3-$CF_3C_6H_4$	**e**	CH_2CO_2Et
c	2,6-$Me_2C_6H_3$	**f**	CH_2CH_2Cl

	R^1	R^2		R^1	R^2
7	Me	4-ClC_6H_4	**11**	Me	CH_2CO_2Et
8	Me	3-$CF_3C_6H_4$	**12**	H	CH_2CH_2Cl
9	Me	2,6-$Me_2C_6H_3$	**13**	CH_2CO_2Et	Me
10	Me	Me	**14**	CH_2CN	Me

Scheme 2

On the contrary, if the cyclohexane ring is arranged trans to the lactone ring, the strain of the framework (type **15**) should become intolerably large, and the steric repulsion between the cyclohexane ring and the *N*-substituents might also be significant. Thus, the ring formation reaction controls the relative configuration of three contiguous asymmetric centers in one step.

Table 2. Compounds **7–14** Prepared

Product	Method	Temp. (°C)/ Time (h)	Yield (%)	mp (°C) (solvent)	Molecular Formula[a]
7	A	20/3	86	245–246	$C_{16}H_{16}ClNO_4$
	B	20/24	84	($CHCl_3$)	(321.8)
8	A	20/3	72	140–141	$C_{17}H_{16}F_3NO_4$
				($CHCl_3$/Et_2O)	(355.3)
9	A	20/48	70	223–224	$C_{18}H_{21}NO_4$
				($CHCl_3$/Et_2O)	(315.4)
10	A	20/3	80	160–161	$C_{11}H_{15}NO_4$
				($CHCl_3$/Et_2O)	(225.3)
11	C	reflux/5	88	118–119	$C_{14}H_{19}NO_6$
				($CHCl_3$/Et_2O)	(297.3)
12	A	reflux/72	59	189–190	$C_{11}H_{14}ClNO_4$
				($CHCl_3$/Et_2O)	(259.7)
13	A	20/24	43	94–95	$C_{14}H_{19}NO_6$
				($CHCl_3$/Et_2O)	(297.3)
14	A	20/24	36	163–164	$C_{12}H_{14}N_2O_4$
				($CHCl_3$)	(250.3)

[a] Satisfactory microanalyses obtained: C ±0.23, H ±0.21, N ±0.19.

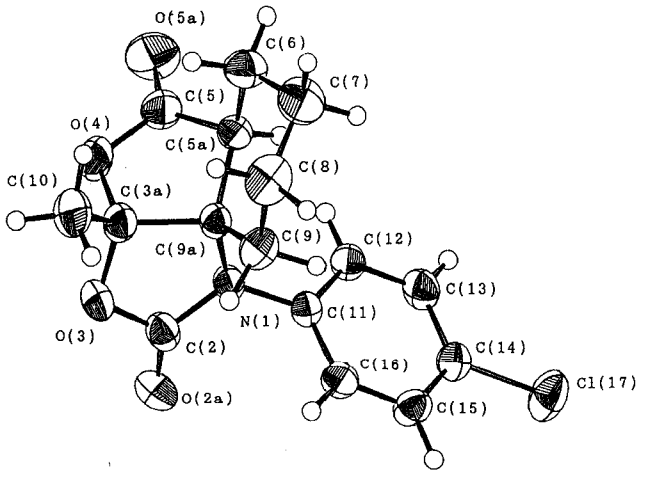


Figure. Crystal structure of compound **7**

Table 3. Spectral Data for Compounds **7–14** Prepared

Product	IR (KBr) $\nu_{C=O}$ (cm^{-1})	^1H NMR ($CDCl_3$/TMS) δ, J (Hz)	^{13}C NMR ($CDCl_3$/TMS) δ, J (Hz)	MS (CI) m/z (%) ($M^+ + 1$)
7	1805, 1765	1.05 (m, 1H, H_{ax}-7), 1.50 (m, 2H, H_{ax}-6, 8), 1.75 (m, 3H, H_{ax}-9, H_{eq}-7, 8), 1.96 (s, 3H, CH_3), 2.12 (m, 1H, H_{eq}-9), 2.21 (m, 1H, H_{eq}-6), 2.77 (q, 1H, J = 6, H-5a), 7.14 (d, 2H_{arom}, J = 6), 7.47 (d, 2H_{arom}, J = 6)	19.94 (CH_3-3a), 20.61 (C-8), 21.07 (C-7), 24.54 (C-6), 25.73 (C-9), 43.71 (C-5a), 68.82 (C-9a), 110.03 (C-3a), 129.75, 131.99, 132.39, 133.89 (C_{arom}), 152.76 (C-2), 173.48 (C-5)	322
8	1800, 1770	1.06 (m, 1H, H_{ax}-7), 1.56 (m, 2H, H_{ax}-6, 8), 1.87 (m, 3H, H_{ax}-9, H_{eq}-7, 8), 1.97 (s, 3H, CH_3), 2.09 (m, 2H, H_{eq}-6, 9), 2.78 (q, 1H, J = 6, H-5a), 7.41 (d, 1H_{arom}, J = 7.5), 7.47 (s, 1H_{arom}), 7.65 (t, 1H_{arom}, J = 7.5), 7.74 (d, 1H_{arom})	20.98 (CH_3), 21.82 (CH_2), 21.87 (CH_2), 26.10 (CH_2), 26.94 (CH_2), 44.71 (C-5a), 69.55 (C-9a), 110.22 (C-3a), 123.24 (CF_3, J_{CF} = 271), 126.46 (J_{CF} = 3.6), 126.99 (J_{CF} = 3.6), 130.81, 132.81 (J_{CF} = 33), 133.65, 133.98 (C_{arom}), 153.33 (C-2), 172.95 (C-5)	356
9	1795, 1765	1.04 (m, 1H, H_{ax}-7), 1.46 (m, 2H, H_{ax}-6, 7), 1.72 (m, 3H, H_{ax}-9, H_{eq}-7, 8), 1.97 (s, 3H, CH_3 at 3a), 2.20 (m, 2H, H_{eq}-6, 9), 2.25 (s, 3H, $ArCH_3$), 2.28 (s, 3H, $ArCH_3$), 2.75 (q, 1H, H-5a), 7.17 (d, 2H_{arom}), 7.23 (m, 1H_{arom})	19.29 (CH_3), 19.86 (CH_3), 21.21 (CH_3-3a), 21.60 (CH_2), 22.07 (CH_2), 26.57 (CH_2), 27.21 (CH_2), 44.35 (C-5a), 72.00 (C-9a), 110.37 (C-3a), 129.17, 129.72, 129.81, 130.61, 138.93, 139.30 (C_{arom}), 152.85 (C-2), 174.03 (C-5)	316
10	1770	1.27 (m, 1H, H_{ax}-7), 1.48 (m, 2H, H_{ax}-6, 8), 1.76 (m, 1H, H_{eq}-7), 1.87 (s, 3H, CH_3-3a), 1.90 (m, 3H, H_{ax}-9, H_{eq}-8, 9), 2.29 (m, 1H, H_{eq}-6), 2.75 (q, 1H, J = 6, H-5a), 2.83 (s, 3H, NCH_3)	20.68 (CH_3-3a), 21.91 (CH_2), 22.22 (CH_2), 24.82 (CH_2), 26.21 (NCH_3), 26.35 (CH_2), 43.27 (C-5a), 67.14 (C-9a), 109.86 (C-3a), 153.93 (C-2), 173.41 (C-5)	226
11	1800, 1765, 1740	1.26 (m, 1H, H_{ax}-7), 1.28 (t, 3H, J = 7, CH_2CH_3), 1.45 (m, 2H, H_{eq}-7, H_{ax}-8), 1.61 (dt, 1H, J = 6, 12, H_{eq}-8), 1.85 (m, 2H, H_{ax}-6, 9), 1.88 (s, 3H, CH_3-3a), 2.03 (m, 1H, H_{eq}-9), 2.23 (m, 1H, H_{eq}-6), 2.69 (q, 1H, J = 6, H-5a), 3.82, 4.03 (ABq, each 1H, NCH_2), 4.21 (m, 2H, OCH_2)	13.99 (CH_2CH_3), 20.67 (CH_3-3a), 21.96 (CH_2), 22.06 (CH_2), 26.32 (CH_2), 26.65 (CH_2), 41.95 (NCH_2), 44.00 (C-5a), 62.12 (OCH_2), 67.48 (C-9a), 110.17 (C-3a), 153.88 (C-2), 168.03 (ester CO), 173.13 (C-5)	298
12	1800–1760 (br)	1.38 (m, 3H, H_{ax}-7, 8, 9), 1.76 (m, 4H, H_{ax}-6, H_{eq}-7, 8, 9), 2.16 (m, 1H, H_{eq}-6), 3.02 (q, 1H, J = 6, H-5a), 3.43, 3.58 (ABm, each 1H, NCH_2), 3.70, 3.80 (ABm, each 1H, CH_2Cl), 6.40 (s, 1H, H-3a)	21.01 (CH_2), 21.09 (CH_2), 25.93 (CH_2), 26.65 (CH_2), 40.56 (NCH_2), 42.45 (CH_2Cl), 42.92 (C-5a), 67.26 (C-9a), 99.41 (C-3a), 154.19 (C-2), 174.32 (C-5)	260, 262 (32)
13	1800, 1775, 1740	1.29 (t, 3H, J = 6, CH_2CH_3), 1.40 (m, 1H, H_{ax}-7), 1.52 (m, 1H, H_{ax}-8), 1.63 (m, 1H), 1.80 (m, 3H, H_{eq}-8, H_{ax}-6, 9), 1.98 (m, 1H, H_{eq}-9), 2.20 (m, 1H, H_{eq}-6), 2.77 (q, 1H, J = 6, H-5a), 2.86 (s, 3H, NCH_3), 3.10, 3.17 (ABq, each 1H, CH_2 at 3a), 4.21 (q, 2H, OCH_2)	14.06 (CH_2CH_3), 21.38 (CH_2), 21.51 (CH_2), 24.89 (CH_2), 25.31 (CH_2), 26.66 (NCH_3), 39.37 (CH_2 at 3a), 43.53 (C-5a), 61.63 (OCH_2), 67.52 (C-9a), 107.57 (C-3a), 153.47 (C-2), 166.20 (ester CO), 173.06 (C-5)	298
14	2270 (CN), 1765	1.38 (m, 2H, H_{ax}-7, 8), 1.75 (m, 4H, H_{ax}-6, 9 H_{eq}-7, 8), 2.04 (m, 2H, H_{eq}-6, 9), 2.83 (m, 3H, CH_3), 3.07 (q, 1H, J = 6, H-5a), 3.70 (ABq, 2H, CH_2 at 3a)	21.01 (CH_2), 21.27 (CH_2), 23.86 (CH_2), 25.00 (CH_2), 26.33 (NCH_3), 38.55 (CH_2 at 3a), 42.15 (C-5a), 67.15 (C-9a), 105.96 (C-3a), 114.74 (CN), 152.25 (C-2), 172.57 (C-5)	251

Finally, the greatest advantage of this reaction is the promising diastereoselectivity giving rise to the 1,2-cis-disubstituted cyclohexylamino moiety. In addition, the product isolation is very easy, because no other reagent except for small amount of catalyst is required. Moreover, the highly functionalized structure of the tricyclic compounds would be amenable to a variety of other heterocyclic systems.

Commercially available chemicals were of reagent grade, and used without further purifications, unless otherwise stated. MeCN and CH_2Cl_2 were dried over molecular sieves 3Å. Melting points were determined on a hot stage apparatus, and are not corrected. IR spectra were recorded on a SHIMADZU IR-440 spectrophotometer. NMR spectra were recorded on Varian Unity 400 and JEOL JNM-GSX 400 spectrometer (400 MHz) in $CDCl_3$ or DMSO-d_6 solutions with TMS as an internal standard. Mass spectra were obtained by HITACHI M-80 spectrometer using chemical ionization method.

To an ice cooled mixture of **2** (20 g, 131 mmol) in THF (100 mL) was added portionwise $NaBH_4$ (90 %, 1.38 g, 32.8 mmol). After stirring for 1 h at r. t. the mixture was refluxed for 5 h. Then 1 N HCl (20 mL) was added slowly to the cold reaction mixture. THF was evaporated under reduced pressure, water (100 mL) was added and the mixture was extracted with $CHCl_3$, dried ($MgSO_4$) and concentrated. The residue was chromatographed on silica gel and recrystallized from Et_2O/hexane to afford **4a** as a colorless solid; yield: 6.0 g (30 %); mp 63–64 °C.

$C_8H_{10}O_3$ (154.2) calc. C 62.33 H 6.54
found 62.22 6.45

MS (CI): m/z (%) = 155 ($M^+ + 1$, 100), 137 (33).

IR (KBr): ν = 3350 (OH), 1720 cm^{-1} (C=O).

^1H NMR ($CDCl_3$/TMS): δ = 1.72 (br m, 4H, H-5,6), 2.20 (m, 3H, H-4, 7), 2.44 (m, 1H, H-4′ or 7′), 5.72 (br s, 1H, OH), 6.01 (s, 1H, H-3).

^{13}C NMR ($CDCl_3$/TMS): δ = 19.68, 21.31, 21.34, 22.46, 98.77 (C-3), 129.28 (C-7a), 161.19 (C-3a), 172.82 (C-1).

3-Substituted 3-Hydroxy-4,5,6,7-tetrahydrophthalides; General Procedure:

To a stirred solution of **2** (25.1 g, 165 mmol) in pyridine (25 mL) was added the appropriate carboxylic acid **3b–d** (329 mmol) at r.t. After stirring for 30 min, the mixture was heated to 70 °C for 5–20 h. Then, the cooled mixture is poured into ice-water (200 mL) containing conc. HCl (32 mL, 360 mmol). In the cases of **4b** and **4d**, the precipitate was filtered, washed with water, dried and recrystallized. In the case of **4c**, the mixture was extracted with $CHCl_3$ washed with water, dried ($MgSO_4$), concentrated and chromatographed on silica gel, using $CHCl_3$ as eluent.

Octahydroisobenzofuro[7a,1-*d*]oxazole-2,5-diones 7–14; General Procedure:

Method A: To a stirred solution of hydroxy lactone **4a–4d** (5 mmol) and the appropriate isocyanate **5a–5f** (5 mmol) in anhydrous MeCN (10 mL) was added DBU (40 mg, 0.25 mmol) at r.t. The reaction was monitored by TLC or ^1H NMR spectroscopy. After the reaction had finished, MeCN was evaporated under reduced pressure. In the cases of **7–12** and **14**, the residue was dissolved small quantity of $CHCl_3$ followed by gradual addition of Et_2O to give the tricyclic compounds. In the case of **13**, the residue was chromatographed on silica gel (eluent: $CHCl_3$).

Method B: TEA (25 mg, 5 mol%) was used as the base instead of DBU. All other conditions were the same as described in method A.

Method C: CH_2Cl_2 (10 mL) was used as the solvent instead of MeCN. All other conditions were the same as described in method A.

Isolation of Carbamate **6** *From* **4b** *and* **5a**: The reaction was carried out according to the general method B. After stirring for 1 h, the reaction was quenched by adding 5 drops of AcOH and the solvent was evaporated under reduced pressure. The residue was chromatographed on silica gel (eluent: hexane/EtOAc, 5:1) to give **6**; yield: 70%; mp 97–98 °C (Et_2O).

$C_{16}H_{16}ClNO_4$	calc.	C 59.73	H 5.01	N 4.35
(321.8)	found	59.83	5.41	4.30

MS (CI): *m/z* (%) = 322 ($M^+ + 1$, 2), 169 (88), 151 (100).

IR (KBr): ν = 3380 (NH), 1765, 1720 cm^{-1} (C=O).

^1H NMR ($CDCl_3$/TMS): δ = 1.73 (s, 3 H, CH_3), 1.80 (m, 4 H, H-5, 6), 2.25 (m, 4 H, H-4, 7), 6.83 (br s, 1 H, NH), 7.25 (m, 4 H_{arom}).

^{13}C NMR ($CDCl_3$/TMS): δ = 19.81, 21.21, 21.40, 21.78 (CH_2), 23.35 (CH_3), 105.61 (C-3), 120.19, 128.51, 129.07 (C_{arom}), 135.66 (C-7a), 149.58 (C-3a), 161.39 (CONH), 170.15 (C-1).

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(13) X-Ray Crystallographic data for **7** has been deposited with Cambridge Crystallographic Data Centre, Lensfield road, Cambridge CB2, 1EW, England.