546. *The Synthesis of Alkoxy-1,2,3,4-tetrahydronaphthalene Derivatives. Part II.*[1] *2-Carboxy Hydrazides*

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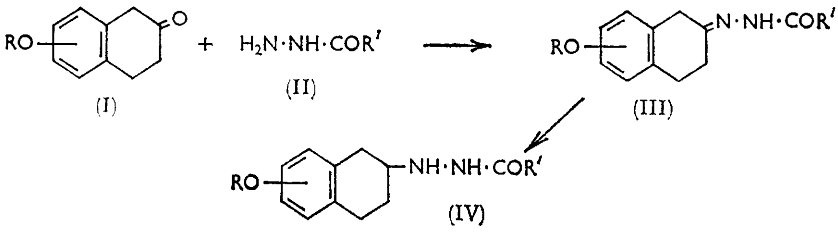
Condensation of the previously-reported[1] 5-, 7-, and 8-alkoxy-2-tetralones (I; R = Me or Et) with known carboxyhydrazides (II) gave 2-naphthylidene hydrazides (III) which were then reduced with sodium borohydride to the saturated naphthyl hydrazides (IV).



The hydrolysis of 2-(1,2,3,4-tetrahydro-7-methoxy-2-naphthyl)benzohydrazide (IV; R = 7-Me, R′ = Ph) with 6N-hydrochloric acid gave 1,2,3,4-tetrahydro-7-hydroxy-2-naphthylhydrazine hydrochloride.

Experimental.—*Carboxyhydrazides* (II). Commercial benzohydrazide was used. Anthranilichydrazide,[2] isonicotinichydrazide,[3] and 2-thiophenecarboxyhydrazide[4] were prepared by refluxing ethanolic solutions of the corresponding esters with hydrazine hydrate.

2-(*Alkoxy*-1,2,3,4-*tetrahydro*-2-*naphthylidene*)*carboxyhydrazides* (III). A mixture of the alkoxy-2-tetralone (0·1 mol.), carboxyhydrazide (0·1 mol.), and ethanol (250 ml.) was refluxed for 5 hr.; the solution was concentrated to half its volume and cooled. The solid that separated was recrystallised from ethanol. Details of the crystalline *hydrazides* thus obtained are given in Table 1.

Table 1

2-(Alkoxy-1,2,3,4-tetrahydro-2-naphthylidene)carboxyhydrazides (III)

R	R′	M. p.	Yield (%)	Found (%) C	H	N	Formula	Required (%) C	H	N
5-Me	4-Pyridyl	173—175°	48	69·4	5·8	14·6	$C_{17}H_{17}N_3O_2$	69·1	5·8	14·2
5-Me	2-Thienyl	159—161	53	64·2	5·4	9·4	$C_{16}H_{16}N_2O_2S$	64·0	5·4	9·3
7-Me	o-C_6H_4·NH_2	143—145	57	70·1	6·1	13·6	$C_{18}H_{19}N_3O_2$	69·9	6·2	13·6
7-Me	Ph	Not purified								
7-Me	4-Pyridyl	127—130	65	69·3	6·3	13·8	$C_{17}H_{17}N_3O_2$	69·1	5·8	14·2
7-Me	2-Thienyl	148—150	90	64·5	5·4	9·1	$C_{16}H_{16}N_2O_2S$	64·0	5·4	9·3
8-Me	o-C_6H_4·NH_2	170—173	75	70·4	6·3	13·2	$C_{18}H_{19}N_3O_2$	69·9	6·2	13·6
8-Me	4-Pyridyl	176—177	89	69·5	5·5	14·1	$C_{17}H_{17}N_3O_2$	69·1	5·8	14·2
8-Me	2-Thienyl	162—165	82	63·8	5·6	9·2	$C_{16}H_{16}N_2O_2S$	64·0	5·4	9·3
8-Et	4-Pyridyl	164—166	52	70·3	6·1	13·2	$C_{18}H_{19}N_3O_2$	69·9	6·2	13·6

2-(*Alkoxy*-1,2,3,4-*tetrahydro*-2-*naphthyl*)*carboxyhydrazides* (IV). The unsaturated hydrazide (0·033 mol.), in absolute ethanol (600 ml.), was added dropwise to a stirred suspension of sodium borohydride (0·045 mol.) in ethanol (150 ml.) at 0°. The mixture was kept at 0° for 2 hr., and then allowed to reach room temperature overnight. A solution of acetic acid (14 ml.) in water (50 ml.) was added and the whole poured into cold water (1 l.). The reduced *hydrazide* that separated was either purified by recrystallisation from aqueous ethanol or converted into its *hydrochloride*. Detazils of these products are given in Table 2.

1,2,3,4-*Tetrahydro*-7-*hydroxy*-2-*naphthylhydrazine hydrochloride*. 2-(1,2,3,4-Tetrahydro-7-methoxy-2-naphthyl)benzohydrazide (13·0 g.) and 6N-hydrochloric acid (100 ml.) were refluxed for 10 hr., cooled, and filtered. The filtrate was concentrated to 10 ml., and the solid that separated was recrystallised twice from 8N-hydrochloric acid (charcoal). This *hydrochloride*

[1] Part I, *J.*, 1965, 2636.
[2] H. H. Fox and J. T. Gibas, *J. Org. Chem.*, 1952, **17**, 1653.
[3] S. Takizawa, Japanese Patent 7,472/1954.
[4] T. Curtius and H. Thyssen, *J. prakt. Chem.*, 1902, **65**, [2], 1.

(2·8 g.) had m. p. 201—203° (Found: C, 55·7; H, 7·2; Cl, 16·9; N, 12·9. $C_{10}H_{14}N_2O,HCl$ requires C, 55·9; H, 7·0; Cl, 16·5; N, 13·0%).

TABLE 2

2-(Alkoxy-1,2,3,4-tetrahydro-2-naphthyl)carboxyhydrazides (IV) and hydrochlorides

R	R′	M. p.	Yield (%)	Found (%) C	H	N	Formula	Required (%) C	H	N
5-Me	4-Pyridyl	227—229°	59	53·9	6·0	10·9	$C_{17}H_{19}N_3O_2$·2HCl,½H_2O	53·8	5·8	11·1
7-Me	*o*-C_6H_4·NH_2	219—220	66	56·6	6·5	10·9	$C_{18}H_{21}N_3O_2$·2HCl	56·2	6·0	10·9
7-Me	Ph	138—139	52	73·0	7·2	9·2	$C_{18}H_{20}N_2O_2$	73·0	6·8	9·5
7-Me	4-Pyridyl	224—225	57	54·8	5·8	11·2	$C_{17}H_{19}N_3O_2$·2HCl	55·1	5·7	11·4
7-Me	2-Thienyl	127—129	72	63·6	5·9	9·3	$C_{16}H_{18}N_2O_2S$	63·6	6·0	9·3
8-Me	*o*-C_6H_4·NH_2	184—185	90	69·3	6·7	13·7	$C_{18}H_{21}N_3O_2$	69·4	6·8	13·5
8-Me	4-Pyridyl	160—161	91	68·6	6·0	14·0	$C_{17}H_{19}N_3O_2$	68·7	6·4	14·1
8-Me	2-Thienyl	142—144	87	63·6	5·9	8·9	$C_{16}H_{18}N_2O_2S$	63·6	6·0	9·3
8-Et	4-Pyridyl	223—224	60	55·9	6·0	10·8	$C_{18}H_{21}N_3O_2$·2HCl	56·3	6·0	10·9

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