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Preparation of New Quinolinecarbohydroxamic Acids[1]

Short Communication

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A series of new quinolinecarbohydroxamic acids has been prepared by treatment of corresponding methyl quinolinecarboxylates with hydroxylamine.

(Keywords: Hydroxamic acids; Hydroxylamine derivatives; Quinolinecarboxylic acid derivatives)

Darstellung neuer Chinolincarbohydroxamsäuren

(Kurze Mitteilung)

Eine Reihe neuer Chinolincarbohydroxamsäuren wurde durch Behandlung der entsprechenden Methylchinolincarboxylate mit Hydroxylamin hergestellt.

An increased interest in hydroxamic acids chemistry observed lately is mainly due to a wide spectrum of their biological activities. For example, the inhibition of urease has found therapeutic implications for the prevention and treatment of urinary stones, aromatic and heterocyclic hydroxamic acids have been reported to be useful in the therapy of African sleeping sickness, hydroxyurea has been introduced into cancer therapy[2]. The compounds are known for their fungicidal and antitubercular properties[3].

An unusual photochemical phenomenon related to hydroxamic acids has been discovered very recently[4].

Looking for new hydroxamic acids we have paid our attention to quinoline derivatives. Thus, a series of quinolinecarbohydroxamic acids has been prepared in the reaction of hydroxylamine with corresponding methyl quinolinecarboxylates. The synthesis of quinolinecarboxylic acids esters was described in paper[5].

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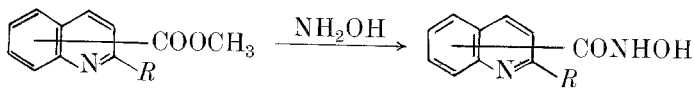

1—15

Synthetic data for the compounds **1**—**15** are presented in Table 1.

Table 1. *Synthetic data for* **1**—**15**

Compd.	Position of CONHOH	*R*	m.p. (°C) (uncorr.)	Yield %	Molecular formula[a] (*M*)
1	2	H	98—99[b]	72	$C_{10}H_8N_2O_2 \cdot H_2O$ (206.7)
2	3	H	250—251	55	$C_{10}H_8N_2O_2 \cdot HCl$ (224.5)
3	4	H	124—124.5	82	$C_{10}H_8N_2O_2$ (188.1)
4	4	H	219—220	60	$C_{10}H_8N_2O_2 \cdot HCl$ (224.5)
5	5	H	166—166.5	90	$C_{10}H_8N_2O_2$ (188.1)
6	5	H	212—212.5	85	$C_{10}H_8N_2O_2 \cdot HCl$ (224.5)
7	6	H	175—176	79	$C_{10}H_8N_2O_2$ (188.1)
8	6	H	241—242	76	$C_{10}H_8N_2O_2 \cdot HCl$ (224.5)
9	7	H	162—163	79	$C_{10}H_8N_2O_2$ (188.1)
10	8	H	183—184[b]	93	$C_{10}H_8N_2O_2$ (188.1)
11	8	H	211—212	95	$C_{10}H_8N_2O_2 \cdot HCl$ (224.5)
12	3	OH	261—262	80	$C_{10}H_8N_2O_2 \cdot HCl$ (224.5)
13	3	Cl	195—196	68	$C_{10}H_8N_2O_2Cl$ (222.5)
14	4	CONHOH	167—167.5	75	$C_{11}H_9N_3O_4$ (247)
15	4	CONHOH	203—204	76	$C_{11}H_9N_3O_4 \cdot HCl$ (284.5)

[a] All the compounds gave satisfactory elemental analyses (C, H, N, Cl). Spectral data (IR, ^1H NMR) were in a good agreement with their structures.

[b] Iron and vanadium complexes of **1** were described in [6], Mo (V) complex of **10** in [7], *Lossen* rearrangement of **1** in [8].

Experimental

General procedure for the preparation of **1**—**15**

0.09 g—atom of Na in 40 ml of methanol was added to a solution of 60 mmol $NH_2OH \cdot HCl$ in 30 ml of methanol at ca. 10 °C. The precipitated NaCl was filtered off and 30 mmol of the corresponding methyl quinolinecarboxylate in ca. 20 ml of methanol was added to the filtrate. The mixture was allowed to stand at room temperature for 48 h. Then the solvent was evaporated *in vacuo* and the obtained sodium hydroxamate was dissolved in ca. 30 ml of water. Impurities were filtered off and the solution neutralized carefully with HCl (1:2). The precipitated hydroxamic acid was filtered off and recrystallized from

water. When the neutralization was not careful enough, mixtures of hydroxamic acids and their hydrochlorides were formed. They were separated by heating with water. Filteration of hot mixtures resulted in solid hydrochlorides. Hydroxamic acids (better soluble in water) precipitated from cooled solutions.

Acidification of the solution of sodium hydroxamates with HCl (1:2) to *pH* ca. 4, gave hydrochlorides of hydroxamic acids (**2**, **4**, **6**, **8**, **11**, **12** and **15**).

References

[1] Chemistry of Hydroxamic Acids. Part 9. For part 8 see: *Eckstein Z., Lipczyńska-Kochany E., Krzemiński J.*, Heterocycles, in press.
[2] Chemistry and Biology of Hydroxamic Acids (*Kehl H.*, ed.). New York: Karger. 1982.
[3] *Eckstein Z., Urbański T.*, Wiadomości Chem., in press.
[4] *Lipczyńska-Kochany E., Iwamura H.*, Chemistry Lett. **1982**, 1825.
[5] *Eckstein Z., Kanik B., Leszczyńska E., Lipczyńska-Kochany E., Pajak J., Zawadzka M.*, Pol. J. Chem. **53**, 2373 (1979).
[6] *Dutta R. L.*, J. Indian Chem. Soc. **36**, 339 (1959).
[7] *Chatterjee B.*, J. Indian Chem. Soc. **45**, 1061 (1968).
[8] *Pająk J., Lipczyńska-Kochany E., Eckstein Z.*, Pol. J. Chem. **56**, 583 (1982).

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