

LETTERS
TO THE EDITOR

Synthesis of Pyridinecarboxylic Acid N-Oxides and Their Amides under Microwave Irradiation Conditions

D. P. Khrustalev

Institute of Organic Synthesis and Coal Chemistry of the Kazakhstan Republic,
ul. Alikhanova 1, Karaganda, 100000 Kazakhstan
e-mail: khrustalev@mail.kz

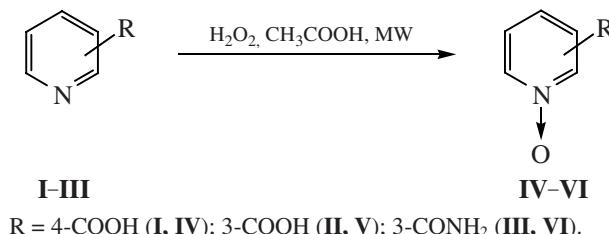
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The steadily growing scientific interest in *N*-oxides is conditioned not only by their essential role in plant and animal metabolism processes [1, 2], but also by their valuable synthetic properties [3].

A standard procedure of synthesis of pyridinecarboxylic acid *N*-oxides and their derivatives

involves prolonged (4–6 h) refluxing of **I–III** with hydrogen peroxide in glacial acetic acid [4]. Using isonicotinic (**I**) and nicotinic (**II**) acids and nicotinamide (**III**) as examples, we studied the possibility of obtaining *N*-oxides **IV–VI** under microwave irradiation, aimed at shortening reaction time. The physicochemical characteristics of the products are listed in the table.



R = 4-COOH (**I**, **IV**); 3-COOH (**II**, **V**); 3-CONH₂ (**III**, **VI**).

Physicochemical characteristics of compounds **IV–VI** prepared under usual and MW irradiation conditions

Comp. no.	mp, °C		Yield, %		Formula	Calculated, %		Found, %	
	Usual conditions [4]	MW conditions	Usual conditions [4]	MW conditions		C	H	C	H
IV	260–262	258–259	89–92	85.5	C ₆ H ₅ NO ₃	51.80	3.62	51.92	3.68
V	269–273	265–267	75–79	70.3	C ₆ H ₅ NO ₃	51.80	3.62	51.87	3.72
VI	289–293	291–292	73–82	70.5	C ₆ H ₆ N ₂ O ₂	52.17	4.38	52.21	4.42

Experiments with varied irradiation power and time showed that the yields of compounds **IV–VI** are higher under the following conditions. Compound **I–III**, 5 g, was dissolved under heating in 30 ml of glacial acetic acid in a conical flask of heat-resistant glass, and, after addition of 15 ml of 30% hydrogen peroxide, the

mixture was subjected to a 70W microwave irradiation for 20 min (4×5 min). The solution was diluted with 15 ml of water which was then distilled off under a vacuum. After cooling to room temperature, the product precipitated and was recrystallized from water. The identity of compounds **IV–VI** were proved by

independent synthesis under convective heating by the procedure in [4].

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