AMINOFURAZAN SALTS

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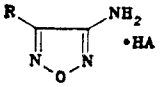
It is thought that aminofurazans do not form stable salts because of their low basicity [1, 2]. We now report conditions in which they can be prepared.

When freshly prepared solutions of 3,4-diaminofurazan (I) or 3-amino-4-methylfurazan (II) in trifluoroacetic acid at 0°C are treated with inorganic acids the corresponding salts precipitate as colorless crystals and prove to be stable to storage in the absence of moisture (see Table 1).



I-II

I $R=NH_2$, II $R=CH_3$; I a $A^-=ClO_4$, b $A^-=Br$, c $A^-=Cl$, d $A^-=NO_3$, e $A^-=BF_4$; II a $A^-=ClO_4$, б $A^-=Cl$

The salts were characterized by elemental analytical data and by separation of the corresponding aminofurazan by recrystallization of the salts from water or by their sublimation in vacuo. The molecular weights of salts Ia-e were determined in water by alkali titration and were close to those calculated.

The IR spectrum of Ia showed bands for NH_2, NH_3^+, the furazan ring, and the perchlorate ion but the absence of bands which might be expected for protonation of the ring nitrogen. Our inability to prepare stable salts of 3,4-dimethylfurazan also supports protonation at the attached amino group.

IR spectrum of Ia (paraffin and fluorinated paraffin mulls): 3080 ($\nu_{HN_3}+$), 1638 ($\delta_{aNH_3}+$), 1504 ($\delta_{sNH_3}+$), 3383 (ν_{aNH_2}), 3301 (ν_{sNH_2}); 1627 (δ_{NH_2}); 1611, 1543, 1457, 1130, 1011, 946, 894 (furazan ring); 1089, 927, 635, 458 cm^{-1} (perchlorate ion).

TABLE 1. Properties of the Aminofurazan Salts I and II

Compound	Empirical formula	Mp, °C	Yield %	Compound	Empirical formula	Mp, °C	Yield, %
I a	$C_2H_5ClN_4O_5$	153...155	90	Ie	$C_2H_5BF_4N_4O$	152...154	71
I b	$C_2H_5BrN_4O$	158...160	63	IIa	$C_3H_6ClN_3O_5$	120...122	25
I c	$C_2H_5ClN_4O$	180...181	74	IIb	$C_3H_6ClN_3O$	103...105	52
I d	$C_2H_5N_5O_4$	110...112	45				

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