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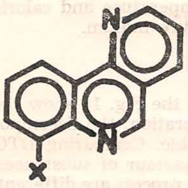
TG AND DTA STUDIES OF 1,5- AND 1,6-BENZO(h)NAPHTHYRIDINES AND THEIR DERIVATIVES

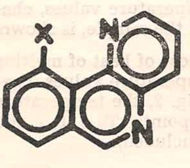
Benzo[h]naphthyridines are angular heteroaromatics belonging to diazaphenanthrenes. Two nitrogen atoms present in the vicinal rings of the molecule show a perturbing influence on its electronic properties. These compunds seem to be of interest, some derivatives of this class of heterocycles exhibiting biological activities [1—4].

Benzo[h]naphthyridines are not yet so much studied, only their ^{1}H and ^{13}C NMR, as well as UV spectra investigations were reported [1, 5], however, so far, their thermal properties were not examined. The present study of thermal behaviour of benzo[h]naphthyridines using thermogravimetric (TG) method and differential thermal analysis (DTA), is a continuation of our research on these heterocycles.

For the thermal analysis the following compounds were chosen:



X=H 1,6-benzo[h]naphthyridine
X=NO_2 7-nitro-1,6-benzo[h]naphthyridine
X=NH_2 7-amino-1,6-benzo[h]naphthyridine



X=H 1,5-benzo[h]naphthyridine
X=NO_2 10-nitro-1,5-benzo[h]naphthyridine

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The examined benzo[h]naphthyridines were synthesized from the appropriate aminoazanaphthalenes in the Skraup procedure, and their nitro- and aminoderivatives were obtained by the nitration and the next reduction of the resulting nitrocompounds.

The preparative methods, as well as the structure elucidation of the above compounds are given in [1, 6].

Among analyzed compounds, II and III contain trace amounts of impurities, probably dinitro- and diaminoderivatives. As they are very difficult to eliminate, and presumably do not seem to influence at a significant degree the measurement results, the compounds II and III were for comparative purposes included to our investigations. For this reason all analyzed samples are referred to as substances I—V.

EXPERIMENTAL

The simultaneous thermogravimetric and differential thermal analyses were made using the Mettler TA 2 apparatus.

The samples of 7 to 10 mg were placed in platinous crucibles with perforated covers. The air under the atmospheric pressure was passed over the furnace, and the heating rate was 10°/min.

At the temperature range corresponding to the melting process of substances, the differential thermal analysis was made, using the Mettler TA 2000 A thermograph.

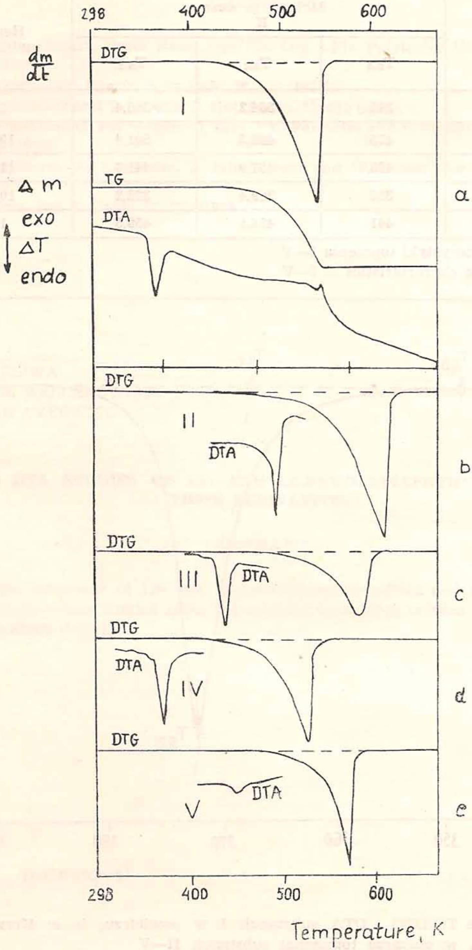
The samples of a few mg were placed in standard aluminium pans with perforated covers and heated in the air atmosphere at the rate 10°/min. As the reference the empty pan was used. The temperature and caloric calibration of the apparatus was made using the pure indium.

RESULTS

The TG, DTG and DTA curves of I presented in the Fig. 1a show the melting process of the substance, followed by evaporation; the decomposition or combustion in vapour phase is also possible. Comparing DTG curves of I—V (Figs 1a—e) one can see similar behaviour of substances, although the temperature ranges of their evaporation process are different, this fact being due to the different boiling points of examined samples.

In the Fig. 2 the way of determination of the temperature values, characteristic for the DTA curve in the melting region of the sample, is shown.

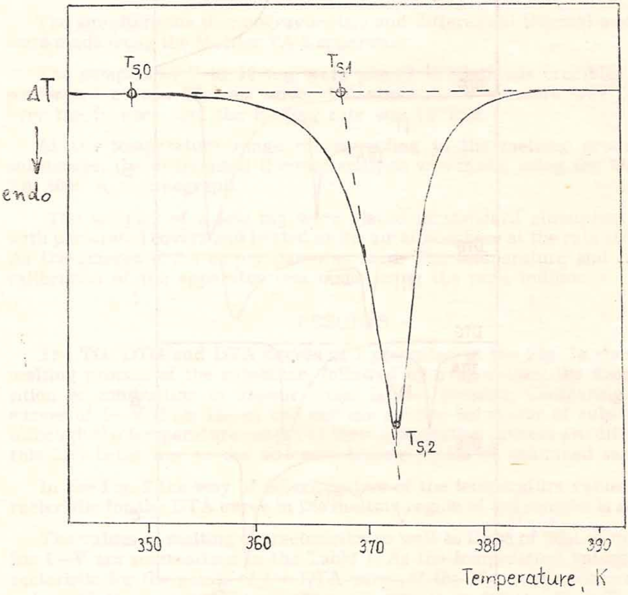
The values of melting characteristics as well as those of heat of melting for I—V are summarized in the Table 1. As the temperature values, characteristic for the points of the DTA curve of the Fig. 2, the temperature values of the sample (T_s), corresponding to these points, $T_{S,0}$, $T_{S,1}$ and $T_{S,2}$, are given (the temperature lagging has been included).



Rys. 2. Krzywa DTA substancji I w powietrzu (pasmo topnienia)
Fig. 2 DTA curve of substance I in the air (the melting peak)

Substance	Melting process K			Heat of melting J/g
	$T_{S,0}$	$T_{S,1}$	$T_{S,2}$	
I	345	364,2	366,4	92,4
II	475	499,3	501,4	135,1
III	428	437,9	441,7	119,6
IV	357	369,4	372,2	105,8
V	441	454,1	459,5	16,7

Tabl. 1. Charakterystyki topnienia I—V
Table 1. Melting characteristics of I—V



Rys. 1a: Krzywe TG/DTG i DTA substancji I w powietrzu; b—e: Krzywe DTG (i DTA w obszarze topnienia) substancji II—V
Fig. 1a: TG/DTG and DTA curves of substance I in the air; b—e: DTG (and DTA in the melting region) curves of substances II—V

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SUMMARY

Thermal behaviour of 1,5- and 1,6-benzo[h]naphthyridines and their nitro- and aminoderivatives was studied using thermogravimetric (TG) method and differential thermal analysis (DTA).