Journal of Thermal Analysis, Vol. 26 (1983) 145–150

Short Communications

STUDY OF THE THERMAL PROPERTIES OF DERIVATIVES OF SULFONAMIDES

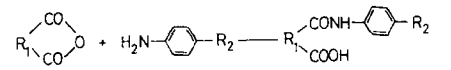
*J. Ciba, J. Sycz and J. Trzcionka**

DEPARTMENT OF ANALYTICAL AND GENERAL CHEMISTRY, SILESIAN TECHNICAL UNIVERSITY, PL 44-101 GLIWICE, *INSTITUTE OF DRUGS, SILESIAN MEDICAL ACADEMY, PL 41-201 SOSNOWIEC, POLAND

Thermal analysis of acid derivatives of sulfonamide was performed between room temperature and 600°. The tested substances were obtained by fusing and also by synthesis in boiling acetone. Information was obtained on the thermostabilities of these compounds and on the thermal decomposition processes they undergo.

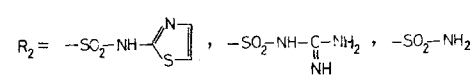
Acyl derivatives of sulfathiazole have found application in medicine for curing certain diseases of the alimentary canal [2–4] and as inhibitors on the oxidation of ammonium salts to nitrates in soil [1].

The reaction of dicarboxylic acid anhydrides with aromatic amines gives N-acyl derivatives. Low-temperature synthesis is usually described by reaction (I).



(I)

$R_1 = -Ph- \;,\; -CH_2-CH_2- \;,\; -CH=CH-$



Synthesis at high temperature leads to intermolecular reactions (cyclization) and further uncontrolled reactions. In this work, method chosen according to scheme (I) was tested.

The following acids were obtained:

1. Phthalylsulfathiazole (PST)
2. Phthalylsulfaguanidine (PSG)
3. Phthalylsulfanilamide (PSA)
4. Succinylsulfathiazole (SST)
5. Succinylsulfaguanidine (SSG)
6. Succinylsulfanilamide (SSA)
7. Maleylsulfathiazole (MST)
8. Maleylsulfaguanidine (MSG)
9. Maleylsulfanilamide (MSA)

The above acids were heated and thermally tested. The results can be useful for identification purposes. TG curves for some sulfonamide derivatives were obtained in earlier work [5].

Experimental

Reagents

Sulfathiazole, sulfaguanidine, sulfanilamide (Polfa–Poland); phthalic anhydride (POCh–Poland); succinic anhydride (APOLDA–DDR); maleic anhydride (Reachim).

Synthesis

The conditions for the preparation of the sulfonamide derivatives and the subsequent thermal testing are given in Table 1. All substances obtained were purified from starting materials by washing with water and methanol. To shorten the duration of the synthesis by the fusion method, higher reaction temperatures than those described in the literature were used. The substances obtained were compared with products synthesized in organic solution.

Purity testing of substances

The purities of substances were checked by paper chromatography; the eluents described in the literature for determination of sulfonamides were used [16–20]. The substances were dissolved in NaOH, HCl, methanol or formamide solution and detected with the Erlich reagent. No other substances were found by chromatography.

Thermal analysis

The thermal analysis of the synthesized substances was performed on a MOM (Budapest) derivatograph. Samples of 100–150 mg were heated in corundum crucibles, from ambient temperature to 600° within 50 minutes; the reference substance was Al_2O_3. DTA, DTG and TG curves were obtained. The thermal changes and the decomposition points observed in the DTA and TG curves are summarized in Table 2.

The calculated mass decreases for the investigated substances and the presumed water and acetone contents of the samples are presented in Table 3.

The melting points determined in the classical way in a capillary tube were compared with the temperature found from the DTA curve.

Results and discussion

Thermal investigations of nine acid derivatives of sulfonamides, as well as the sulfonamides used for their synthesis, were performed. The sulfonamide derivatives were obtained by fusion of sulfonamides with dicarboxylic acid anhydrides; four acids were synthesized in boiling acetone solution (Table 1, Nos 1, 2, 4, 9).