

Experimental

α -(2-Chloroethyl)- β -chlorocrotonaldehyde (I) was added (molar ratio of I : sulfamide = 1 : 2.1) dropwise, with shaking, to a concentrated solution of sulfanilamide, 2-(p-aminobenzenesulfamido)-thiazole, 2-(p-aminobenzene-sulfamido) pyrimidine, or 1-phenyl-3-ethyl-4-methyl-5-(p-aminobenzenesulfamido) pyrazole in HCl (1:1). After all of I had been added, shaking was continued for 30-40 min longer, and the reaction mixture kept for 1-1.5 days at room temperature. A yellow precipitate formed. Compound II_d separated as a crystalline precipitate when the reaction products were diluted with water after reaction was complete. The precipitate was filtered off, washed with ether, and recrystallized from aqueous ethanol containing a few drops of HCl. For analysis the substance was vacuum-dried over P₂O₅.

REFERENCES

1. M. A. Volodina, A. P. Terent'ev, V. A. Kudryashova, and L. N. Kaboshina, KhGS, 1966 (in press).
2. A. P. Terent'ev, M. A. Volodina, and V. A. Kudryashova, DAN, 164, 115, 1965.

13 May 1965

Lomonosov State University, Moscow

UDC 547.794.3

RESEARCHES ON 2, 1, 3-THIA- AND SELENADIAZOLE

XXXIX. Polymorphism of 4-Hydroxybenzo-2, 1, 3-thiadiazole and its Methyl Derivatives*

V. G. Pesin and I. A. Belen'kaya

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 2, No. 2, pp. 313-314, 1966

4-Hydroxy-, 4-hydroxy-5-methyl-, 4-hydroxy-7-methylbenzo-2, 1, 3-thiadiazoles are polymorphous.

4-Hydroxybenzo-2, 1, 3-thiadiazole (I), 4-hydroxy-5-methyl- and 4-hydroxy-7-methylbenzo-2, 1, 3-thiadiazoles (II and III) melt at 114-115°, 110-112°, 100-102° C, respectively, after recrystallization from water [2-4], but after recrystallization from petrol ether [5] they melt at 128-129°, 124-125°, and 119-120° C [5]. In this connection we recrystallized these phenols repeatedly from petrol ether after recrystallizing them from water, and their melting points rose as expected [5]. On the other hand, the compounds with melting points 128-129°, 124-125°, 119-120° C (ex petrol ether), after repeated crystallization from water melted at 114-115°, 110-112°, 100-102° C, respectively.

IR spectra,** measured in vaseline, with a UR-10 instrument and elementary analyses showed the identities of the compounds, consequently it can be concluded that the phenols in question are polymorphous.

It is of interest that 5-hydroxybenzo-2, 1, 3-thiadiazole (mp 156-157° C) and 5-hydroxy-4-methylbenzo-2, 1, 3-thiadiazole (mp 121-123° C) are not altered in melting point when recrystallized from water or benzene ether.

REFERENCES

1. V. G. Pesin and I. A. Belen'kaya-Lotsmanenko, KhGS [Chemistry of Heterocyclic Compounds], 3, 354, 1965.
2. V. G. Pesin, A. M. Khaletskii, and I. A. Lotsmanenko, Author's Certificate, no. 145 243; Byull. izobr. no. 5, 1962.
3. V. G. Pesin, A. M. Khaletskii, and I. A. Lotsmanenko, ZhOKh, 33, 1746, 1963.
4. L. S. Efros, R. P. Polyakova, and M. G. Argititi, ZhOKh, 32, 516, 1962.
5. D. Monte and E. Sandri, Ann. Chim. (Roma), 54, 486, 1964.

2 August 1965

Leningrad Pharmaceutical Chemistry Institute

* For Part XXXVIII see [1].

** Spectra determined by V. S. Korobkov.