[Third International Electronic Conference on Synthetic Organic Chemistry (ECSOC-3), www.mdpi.org/ecsoc-3.htm, September 1-30, 1999]

[A0021]

2-METHYLBENZOTHIAZOLE SYNTHESIS FROM 2-MERCAPTOANILINE AND ACETIC ACID

Vladimir N. Bulavka*, Tatiana N. Boiko and Igor I. Boiko

Technologist Co., Ltd., Mendeleev sq.2, Pereslavl-Zalesskii, Yaroslavl region,152140, Russian Federation
*Present address: Scientific-Research Phototechnical Institute on Slavich Company (NIFTI-Slavich)
Mendeleev sq. 2, Pereslavl-Zalesskii, Yaroslavl region, 152140, Russian Federation
E-mail: nifti@slavich.botik.ru

Received: 30 July 1999 / Uploaded: 13 August 1999

Keywords: thiocyanines synthesis half-product, 2-methylbenzothiazole, 2-methylbenzothiazole synthesis, 2-mercaptoaniline.

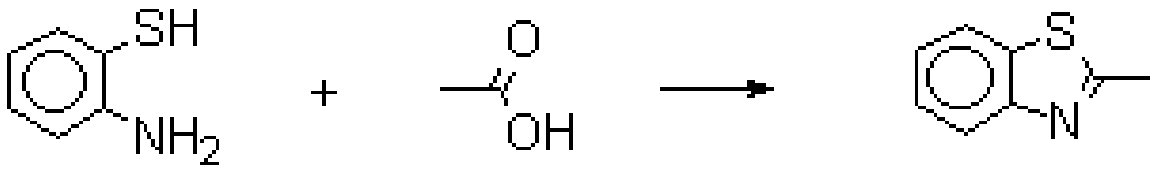
2-Methylbenzothiazole is widely used as the half-product for the thiocyanine dyes photosensitisers synthesis. It usually is synthesized by action of an acetic anhydride in excess on 2-mercaptoaniline.

High-temperature reaction of 2-mercaptoaniline derivatives with the organic acids at temperatures above 200^{o}C was extensively studied, but the reaction with acetic acid was not described [1-5].

The method of benzothiazole and its lower 2-alkylsubstituted derivatives synthesis from zinc 2-aminomercaptide and lower aliphatic acids was earlier described, but even in this paper in the examples of 2-methylbenzothiazole synthesis acetic anhydride alone or its mixture with benzene was used [6].

We have elaborated a new efficient method of 2-methylbenzothiazole synthesis from 2-mercaptoaniline and acetic acid shown at the scheme 1.

Scheme 1.



2-Mercaptoaniline and acetic acid (120% excess) were refluxed 3h. Then the fraction boiling at 103-108^{o}C was slowly distilled with a short Vigreux column. After distillation of all formed water, the column was removed and the excess of acetic acid with b. p. 108-144^{o}C was distilled. Fractioning of the remained substance gave the first fraction with b. p. 144-240^{o}C (10%) and the crude product, b.p. 240-250^{o}C (78%), n_D^{15} 1.1619. Further distillation at reduced pressure gave the pure 2-methylbenzothiazole, b.p. 93-97^{o}C/6 mm, n_D^{20} 1.1617.

References

[1] A. W. Hofmann, *Ber. Deutsch. Chem. Ges.* 1879, *12*, 2359-2365.

[2] A. W. Hofmann, *Ber. Deutsch. Chem. Ges.* 1880, *13*, 8-22.

[3] A. W. Hofmann, *Ber. Deutsch. Chem. Ges.* 1880, *13*, 1223-1238.

[4] A. W. Hofmann, *Ber. Deutsch. Chem. Ges.* 1887, *20*, 1798-1805.

[5] P. Petitcolas, R. Sureau, J. Frenkiel, R. Goupil, *Bull. Soc. Chim. Franc*. 1949, 103-112.

[6] A. I. Kiprianov, Z. P. Sytnik, N. E. Grigorjeva, *Zh. Obshch. Khim.* 1936, *6*(2), 232-235.

All comments on this poster should be sent by e-mail to (mailto:ecsoc@listserv.ariz ona.edu) ecsoc@listserv.arizona.edu with **A0021** as the message subject of your e-mail.
