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PREPARATION OF SOME 10 AROYLEPHENOTHIAZINE DERIVATIVES

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In conection with our investigation on the chemical properties of thio-compounds, it was of interest to synthetize some 10-aroylphenothiazine derivatives. Phenothiazine derivatives of this type have previously been prepared by several authors [1,2,3].

In the present work we describe the preparation of 10-(2-furoyl-)-phenothiazine, 10-(4-brombenzoyl) -phenothiazine, and 10-(3-nitrobenzoyl-)-phenothiazine.

The above-mentioned compounds were prepared by acylation of phenothiazine with the corresponding acid chloride. The acylation reaction was carried out in dry benzene by refluxing the reaction mixture for 4 hrs.

The preparation of 10-(2-pyridoyl-)-phenothiazine and 10-(4-pyridoyl-)-phenothiazine by acylation of phenothiazine in toluene, with 2-pyridoylchloride hydrochloride and 4-pyridoylchloride hydrochloride respectively, followed by neutralization of the resulting 10-(2-pyridoyl-)-phenothiazine hydrochloride resp. 10-(4-pyridoyl-)-phenothiazine hydrochloride with ammonia, is also reported.

Experimental

Melting points are uncorrected.

10-(2-Furoyl-)-phenothiazine

To a solution of 9.96 g (0,05 mole) of dry phenothiazine in 100 ml of dry benzene, 10 g of 2-furoylchloride was added. The reaction mixture was refluxed for 4 hrs. The main part of benzene was then distilled off and petroleum ether was added to the residue. The separated precipitate was filtered off, washed with petroleum ether and dried. After recrystallization from acetic acid-water, 13,5 g (91.70%)of pale yellow crystals m. p. 148—150° were obtained.

Anal. $C_{17}H_{11}NO_2S$ (293.35) calc'd: C 69.60% H 3.78% N 4.77%

found.: C 69.45% H 3.95% N 4.98%

10-(4-Brombenzoyl-)-(phenothiazine

This compound was prepared in the same maner as above by acylation of phenothiazine with 4-brombenzoylchloride. An 98,84% yield of colorless crystals, m. p. 162—163° was obtained by recrystallization from ethanol-water.

Anal. $C_{19}H_{12}ONBrS$ (382.30) calc'd.: C 59. 69% H 3.17% N 3.66%

found.: C 59.22% H 3.40% N 3.80%

10-(3-Nitrobenzoyl-)-phenotiazine

10-(3-nitrobenzoyl-)-phenothiazine was prepared as above by acylation of phenothiazine with 3-nitrobenzoylchloride. A quntitative yield of colorless crystals, m. p. 180°, was obtained by recrystallization from acetic acid-water.

Anal.: $C_{19}H_{12}N_2O_3S$ (348.39) calc'd.: C 65.50% H 3.48%

found.: C 65.42% H 3.58%

10-(2-Pyridoyl-)-phenothiazine

To a solution of 9.96 g of dry phenothiazine in 160 ml anhydrous toluene, 12 g of 2-pyridoylchloride-hidrochloride were added. The reaction mixture was refluxed for 4 hrs. The main part of toluene was than distilled and petroleum ether was added to the residue. The separated precipitate was filtered off, washed with petroleum ether and dried. 9 g of hidrochloride of 10-(2-pyridoyl-)- phenothiazine was obtained. The hidrochloride was disolved in ethanol (300 ml) and the solution was poured in diluted ammonia. The separated precipitate was filtered off, washed with water and dried. 6.3 g (34.38%) of crude 10-(2-pyridoyl-)-phenothiazine were obtained. After five recrystallizations from ethanol-water, pale yellow crystals, m.p. 156°, were obtained.

Anal.: $C_{18}H_{12}N_2OS$ (304.36) calc'd.: C 71.05% H 3.94% N 9.21%

found.: C 70.92% H 4.11% N 8.98%

10-(4-Pyridoyl-)-phenothiazine

This compound was prepared as above by acylation of phenothiazine with 4-pyridoylchloride-hydrochloride[4]. A 49.10% yield of yellowish crystals, m. p 190°, was obtained by recrystalization from ethanol-water.

Anal.: $C_{18}H_{12}N_2OS$ (304.36) calcid.: C 71.05% H 3.94% N 9.21%

found.: C 70.95% H 4.12% N 9.48%

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ИЗВОД

Приготвување на некои 10-арил фенотиазински девивати. Б. Д. Подолешев и Л. Г. Камчева

Дадена е синтезата на: 10-(2-фурил-)-фенотиазин, 10-(4-бромбензоил фенотиазин, 10-(3-нитробензол-)-фенотиазин, 10 (2-пиридил-)-фенотиазин и 10-(4-пиридоил-)-фенотиазин. Овие соединенија се добиени со ацилација на фенотиазин со содтветните киселински хлориди.

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