ciding with the plane of symmetry (except for atoms $C_{(14)}$, $C_{(14')}$, $C_{(15)}$, and $C_{(15')}$ of the N-phenyl ring, which due to steric interactions with the o-hydroxyphenyl ring rotates by 90° around the $N_{(1)}-C_{(13)}$ bond). However, this rotation does not completely destroy the spatial difficulties arising from formation of a planar tricyclic system as reflected in the distorted exocyclic angles around atoms $N_{(1)}$ [$N_{(2)}N_{(1)}C_{(13)}$, 117.4(2)° and $C_{(5)}N_{(1)}C_{(13)}$, 132.7(2)°], $C_{(5)}$ [$N_{(4)}C_{(5)}C_{(6)}$, 122.4(2)° and $N_{(1)}C_{(5)}C_{(6)}$, 128.7(2)°], and $C_{(6)}$ [$C_{(5)}C_{(6)}-C_{(7)}$, 118.4(2)° and $C_{(5)}C_{(6)}C_{(11)}$, 123.9(2)°].

EXPERIMENTAL

Crystals of IIA ($C_{15}H_{13}N_3O$) are rhombic, at 20°C: a = 17.606(1), b = 10.252(1), c = 7.143(5) Å, V = 1289.3(2) Å^3, Z = 4, d_{calc} = 1.30 g/cm^3, space group Pnam. Cell constants and intensities of 1098 independent reflections were measured on a Hilger–Watts automatic 4-circle diffractometer (λ_{MoK_α}, θ/2θ scanning, $2° \leq \theta \leq 30°$).

The structure was solved by direct methods using MULTAN and 758 independent reflections with $I \geq 2\sigma$ and was refined by block diagonal anisotropic least squares of 750 reflections with $|F| > 4\sigma$ using the weighting scheme $1/w = \sigma(F)^2 + (0.01F_{mea})^2$. Hydrogen atoms were located by temperature factors. Final agreement factors were R = 0.050 (R_W = 0.051). All calculations were carried out on an IBM EKLIPS S/200 using programs INEXTL [6]. Atomic coordinates are given in Table 2.

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SYNTHESIS OF 5-AMINO-1,2,3-THIADIAZOLE

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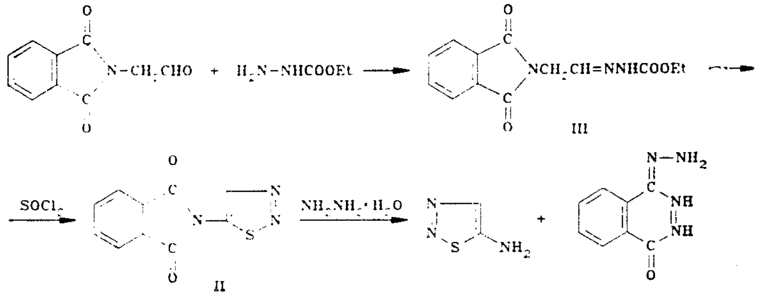
A convenient method is proposed for the synthesis of ethyl phthalimidoethylideneaminocarbamate, an intermediate in the synthesis of 5-amino-1,2,3-thiadiazole.

5-Amino-1,2,3-thiadiazole (I) is used as an intermediate in the synthesis of 1,2,3-thiadiazol-5-yl ureas having pesticidal action. One of the methods for the preparation of thiadiazole I is the reaction of ethoxycarbonylhydrazine with phthalimidoacetaldehyde, sub-



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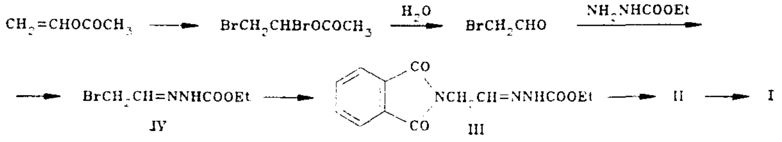
sequent treatment of the resulting phthalimidoacetaldehyde ethoxycarbonylhydrazone (III) by thionyl chloride and reaction of the obtained phthalimido-1,2,3-thiadiazole (II) with hydrazine hydrate for removal of the shield [1, 2].

Phthalimidoacetaldehyde is obtained from potassium phthalimide and bromoacetaldehyde diacetal [3].

To develop a more convenient and practicable method for the synthesis of compound I, we carried out the condensation of ethyl β-bromoethylideneaminocarbamate (IV) with potassium phthalimide, which afforded hydrazone III in up to 88% yield according to the scheme



Thus, we used vinyl acetate for the synthesis of hydrazone III in order to expand the starting-material base. During the study, we found experimentally the conditions for conversion of 1,2-dibromoethyl acetate to bromoacetaldehyde and its subsequent condensation with ethyl carbazate, without recovery, with formation of ester IV, the constants of which agreed with those described in the literature.

EXPERIMENTAL

Ethyl hydrazinoformate (ethyl carbazate) was prepared by the method of [4] in 87.4% yield with mp 45°C, and 1,2-dibromoethyl acetate was prepared by the method of [5] in 60% yield with bp 80°C (20 torr).

Ethyl β-Bromoethylideneaminocarbamate (IV). A solution of 31.2 g (0.3 mole) of ethyl carbazate in 200 ml of water was added dropwise with stirring at 30°C to an emulsion of 76.6 g (0.31 mole) of 1,2-dibromoethyl acetate in 170 ml of water, kept beforehand at 70-80°C for 30-40 min. The material was kept at room temperature for 40-50 min, and the precipitate that formed was filtered off and dried. Obtained: 53 g (84%) of compound IV, mp 125-127°C. According to the data of [6], mp 126-127°C.

Ethyl Phthalimidoethylideneaminocarbamate (III). To a solution of 44 g (0.21 mole) of compound IV in 190 ml of DMFA was added 39 g (0.21 mole) of potassium phthalimide, and the whole was kept at 80-100°C for 30 h. Then the material was cooled and diluted with water, and the resulting precipitate was filtered off. Obtained: 18.5 g (88%) of compound III with mp 165-167°C. According to the data of [1], mp 165-167°C. Infrared spectrum: 1730, 1720 (CO), 1607 cm^{-1} (CH=N).

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