

	R _f in systems*				UV spectrum			
					0.1 N HCl		0.5 N NaOH	
	1	2	3	4	λ _{max} , nm	ε	λ _{max} , nm	ε
II	0.70	0.54	0.24	0.12	264	16 800	260	13 200
III	0.80	0.70	0.72	0.45	252	18 400	279	14 000

*Systems: 1) isopropanol-water-concentrated hydrochloric acid (65.0:18.4:16.4); 2) ethyl acetate-98% formic acid-water (70:20:10); 3) tert-butanol-methyl ethyl ketone-water-98% formic acid (44:44:11:0.26); 4) n-butanol-2 N aqueous ammonia-ethanol (20:5:2).

no inhibiting activity on the growth of the tumors in either males or females. Compound II inhibited the growth of transplanted Walker's carcinosarcoma in the rat to the extent of 37% after ten doses of 60 mg/kg of the substance and to the extent of 57% after a similar administration of 120 mg/kg. The inhibiting effect was shown similarly in males and females. Compound II had no clear inhibiting effect on the growth of Pleace's lymphosarcoma. The LD₅₀ in white mice with intraperitoneal administration was 600 mg/kg for compound II and 300 mg/kg for compound III.

REFERENCES

1. W. Wilson, J. Chem. Soc., 1157, 1948.
2. W. J. Haggerty, R. H. Springer, and C. C. Cheng, J. Med. Chem., **8**, 797, 1965.

1 April 1967

Institute of Organic Synthesis
AS LatvSSR, Riga

FORMATION OF 5-METHYL-4-PHENYL-1, 2-DITHIOLEN-3-THIONE BY THE REACTION OF 2-PHENYLBUTANE WITH ELEMENTARY SULFUR

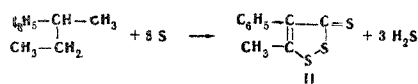
M. G. Voronkov and A. N. Pereferkovich

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 3, No. 6, p. 1133, 1967

UDC 547.738+546.22-121+547.734

Previously, one of us has shown [1, 2] that the reaction of sulfur with 1- and 2-phenylbutanes leads to the formation of 2- and 3-phenylthiophenes, respectively, with yields of about 5%. The action of sulfur on 2-methyl-4-phenylbutane has given 4-methyl-2-phenylthiophene (yield about 12%) [3]. On the other hand, the catalytic reaction of sulfur with 1- and 2-phenylpropanes leads to the formation of 5- and 4-phenyl-1, 2-dithiolen-3-thiones, respectively [4, 5].

Continuing our investigation of the reaction of sulfur with aryl-substituted butanes and higher arylalkanes, we have shown that when it is carried out catalytically the corresponding aryl-1, 2-dithiolen-3-thiones are formed in many cases in addition to arylthiophenes. Thus, for example, when 185.0 g (1.375 mole) of 2-phenylbutane was heated with 88.0 g (2.75 g-atom) of sulfur in the presence of 0.7 g (0.016 mole-%) of mercuriacetamide at 180°-200° C for 30 hr, 5.5 g (5.6% of theoretical, calculated on the 2-phenyl-butane that had reacted) of 3-phenylthiophene (I) and 1.5 g (1.1%) of 5-methyl-4-phenyl-1, 2-dithiolen-3-thione (II) were isolated, the latter having been formed in the following way.



To isolate the reaction products, the unchanged 2-phenylbutane was distilled off (102 g or 0.76 mole), the free sulfur was eliminated from the residue by means of dimethylformamide, and it was distilled with superheated steam. The resulting reddish crystals were separated chromatographically on a column of silica gel. The eluant was a mixture of petroleum ether and benzene (5:1). From the appropriate fraction of the eluate were obtained red plate-like crystals with mp 91°-92° C (from acetic acid), which corresponds to literature

data [6] for II (mp 92° C). Found, %: C 53.40; H 4.00; S 42.64. Calculated for C₁₀H₈S₃, %: C 53.53; H 3.59; S 42.88.

Similarly, I was isolated with mp 91°-92° C (literature data [2], mp 91.5°-92° C). This substance was shown to be 3-phenylthiophene by a mixed melting point.

By modifying the reaction conditions (for example, by the dropwise addition of 2-phenylbutane containing 5 mole-% of morpholine into an excess of sulfur heated to 200°-210° C), the yield of I can be raised to 10-15%.

REFERENCES

1. M. G. Voronkov and A. S. Broun, ZhOKh, **18**, 70, 1948.
2. M. G. Voronkov, A. S. Broun, G. B. Karpenko, and B. L. Gol'shtein, ZhOKh, **19**, 1356, 1949.
3. M. G. Voronkov and B. L. Gol'shtein, ZhOKh, **20**, 1219, 1950.
4. M. G. Voronkov and T. V. Lapina, KhGS [Chemistry of Heterocyclic Compounds], 342, 1965.
5. M. G. Voronkov and T. V. Lapina, KhGS [Chemistry of Heterocyclic Compounds], 522, 1966.
6. L. Legrand, Y. Mollier, and N. Lozach, Bull. Soc. chim. France, 327, 1953.

26 June 1967

Institute of Organic Synthesis,
AS LatvSSR, Riga