

EXPERIMENTAL

Si-Dimethyl-4-silavalero lactam (I). A 1-liter three-necked flask, fitted with a reflux condenser, stirrer with seal, and bubbler, was charged with a solution of 39.5 g (0.2 mole) of 4-(chlorodimethylsilyl)butyryl chloride in 400 ml of absolute ether. Dry ammonia was passed through the ethereal solution for 3 hr. The precipitate of ammonium chloride was filtered off rapidly and washed with ether. The ether was distilled off from the mother ethereal solution and the residue was distilled in vacuum to give 18.3 g (64%) of I in the form of a colorless viscous oil. Compounds II-V, the properties of which are given in the table, were obtained similarly.

1, 1, 3, 3-Tetramethyl-1, 3-bis(4'-N-methylbutyramide)disiloxane (VI). The addition of 0.5 g of water to 3.15 g (0.02 mole) of III caused a marked evolution of heat. The aqueous layer was separated from the organic layer; the latter was dried over $MgSO_4$ and distilled in vacuum to give 1.7 g of VI in the form of a colorless viscous oil. Compounds VII and VIII were obtained similarly. Compounds IX and X were obtained by the passage of undried ammonia through ethereal

solutions of the corresponding acid chlorides. The properties of the siloxane dicarboxylic amides synthesized are given in the table.

REFERENCES

1. V. F. Mironov and N. S. Fedotov, *KhGS [Chemistry of Heterocyclic Compounds]*, **2**, 453, 1966.
2. V. F. Mironov, N. S. Fedotov, and V. L. Kozlikov *KhGS [Chemistry of Heterocyclic Compounds]*, **4**, 354, 1968.
3. N. S. Fedotov, I. G. Rybalka, and V. F. Mironov, *ZhOKh*, **38**, 896, 1968.

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Moscow

SYNTHESIS OF ARYLETHYLENE DERIVATIVES OF 2, 5-DIPHENYL-1, 3, 4-OXADIAZOLE

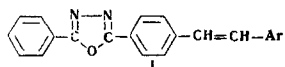
B. M. Krasovitskii and V. I. Grigor'eva

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 4, No. 6, pp. 1127-1129, 1968

UDC 547.793.4.07

The synthesis of arylethylene derivatives of 2, 5-diphenyl-1, 3, 4-oxadiazole by the Wittig reaction from 2-(p-bromomethylphenyl)-5-phenyl-1, 3, 4-oxadiazole and aromatic aldehydes is described. The compounds obtained fluoresce strongly on irradiation with UV light.

We have obtained arylethylene derivatives of 2, 5-diphenyloxazoles which fluoresce strongly on irradiation with UV light [1]. In the present paper, compounds of similar structure containing, instead of the oxazole ring, the 1, 3, 4-oxadiazole nucleus (I) are described.



The starting material for the synthesis of these compounds was the p-bromomethyl derivative of 2, 5-diphenyl-1, 3, 4-oxadiazole, from which compounds I were obtained by the Wittig reaction, using the method that we have described in the production of the vinyl derivatives of 2, 4-diaryl-1, 3, 4-oxadiazoles [2] with the only difference that, instead of paraformaldehyde, the corresponding aldehyde was added. The arylethylene derivatives I fluoresce on irradiation with UV light.

EXPERIMENTAL

A mixture of equimolecular amounts (0.01 mole) of 2-(4'-bromomethylphenyl)-5-phenyl-1, 3, 4-oxadiazole and triphenylphos-

phine was boiled in 15 ml of dimethylformamide for 2 hr. The precipitate that deposited was filtered off, washed with petroleum ether, and dried in the air. Then it was dissolved in 100 ml of methanol and treated with a small excess of the appropriate aldehyde (0.011 mole) and 50 ml of a 0.2 N solution of lithium methoxide in methanol. The mixture was left at room temperature for 5-6 hr. The precipitate that had deposited was filtered off, washed with a small amount of methanol, dried, and isomerized for complete conversion into the trans isomer by being boiled in xylene with a small crystal of iodine for 4-5 hr. Then it was purified on a chromatographic column of alumina in benzene or xylene with subsequent recrystallization from toluene or chlorobenzene.

The analyses, melting points, and yields of the substances obtained are given in the table on p. 126.


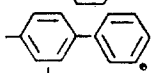
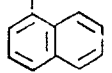
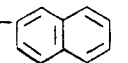
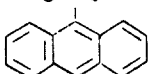
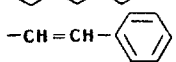

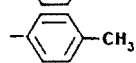
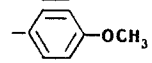
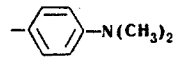
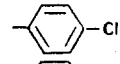
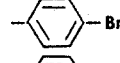
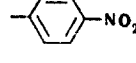
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Chemical Substances, Khar'kov

Characteristics of the Substances Obtained

Ar	Empirical formula	Mp, °C	N, %		Yield, %
			found	calc.	
	C ₂₂ H ₁₆ N ₂ O	170.5—171.5	8.74	8.64	50
	C ₂₈ H ₂₀ N ₂ O	223—224	7.23	7.00	60
	C ₂₆ H ₁₈ N ₂ O	193—194	7.68	7.48	35
	C ₂₆ H ₁₈ N ₂ O	206.5—207.5*	7.64	7.48	70
	C ₃₀ H ₂₀ N ₂ O	272—273	6.79	6.60	70
	C ₂₄ H ₁₈ N ₂ O	210—210.5	8.15	8.00	60
	C ₃₈ H ₂₆ N ₄ O ₂	295.5—296.5	9.54	9.82	50
	C ₂₃ H ₁₈ N ₂ O	193.5—194.5*	8.49	8.28	60
	C ₂₃ H ₁₈ N ₂ O ₂	181—182*	8.17	7.91	50
	C ₂₄ H ₂₁ N ₃ O	227—228	11.60	11.44	50
	C ₂₂ H ₁₅ ClN ₂ O	209.5—210.5*	7.82	7.81	50
	C ₂₂ H ₁₅ BrN ₂ O	210.5—211.5*	7.22	6.94	50
	C ₂₂ H ₁₅ N ₃ O ₃	272—273	11.43	11.38	30

* Turbid melt.