Values of Chemical Shifts of Signals Due to Protons, Derived from PMR Spectra of Morpholine and Two of its Derivatives (I and II)

| Name of proton-containing group | Value of the chemical shift, ppm | | |
|---|---|---|---|
| | I | II | III |
| $CH_2$ α to N | 2.4 | 2.12 | 2.22 |
| $CH_2$ β to N | 1.29 | 1.26 | 1.45 |
| NH | — | — | 2.6 |
| Oxocyclobutane $CH_2$ | 0.57 | 0.53 | — |
| $CH_3$ | 3.5 | — | — |
| $CH_2$ connected to oxacyclobutane and the N atom of morpholine | 2.5 | 2.38 | — |

It would be interesting to explain the cause of the multiplicity of the signal from the oxacyclobutane $CH_2$ groups in compound I. Possibly it is due to the dissymmetry of the groups joined to them. However, only further research will answer this question.

## REFERENCES

1. N. A. Zakharova, N. V. Khromov-Borisov, S. Z. Kaplan, and A. S. Zvontsova, ZhOrKh, 1, 68, 1489, 1965.

2. S. Z. Kaplan and A. S. Zvontsova, ZhOKh, 3412, 1963.

3. J. A. Pople, W. G. Schneider, and H. J. Bernstein, High-Resolution Nuclear Magnetic Resonance [Russian translation], IL, Moscow, 327, 1962.

4. L. Meyer, A. Saika, and H. Gutowsky, J. Am. Chem. Soc., 75, 4567, 1953.

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UDC 547.712 + 547.722 + 542.951.4

# BISTETRAHYDROFURFURYL ETHER

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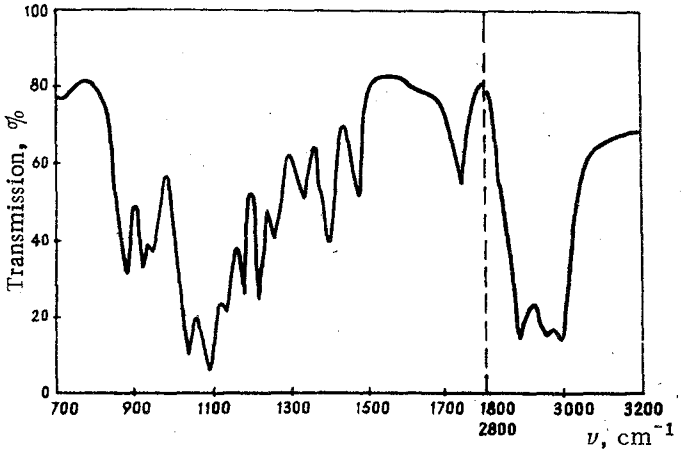
Bistetrahydrofurfuryl ether I, hitherto not described in the literature, has been isolated in the course of a study of the thermal condensation of tetrahydrofurfurylhydroxysilanes.



IR spectrum of bistetrahydrofurfuryl ether.

At the same time, we synthesized I in 48% yield by reacting tetrahydrofurfuryl bromide with sodium tetrahydrofurfurylate at 180° C for 6 hr.

Bp 74°C (1.5 mm); $d_4^{20}$ 1.0347; $n_D^{20}$ 1.4550. Found: C 64.30, 64.65; H 9.68, 9.75%; M (cryoscopic in benzene) 170; 175; $MR_D$ 48.46. Calculated for $C_{10}H_{18}O_2$: C 64.50; H 9.73%; M 186; $MR_D$ 48.91.

The correctness of the identification of the compound synthesized as bistetrahydrofurfuryl ether is confirmed by its IR spectrum (Fig. ) showing absorption bands characteristic of vibrations of C–H bonds in the heterocyclic ring (880, 925, and 945 $cm^{-1}$), the heterocyclic group C–O–C (1030 $cm^{-1}$), an ether link (1090, 1140 and 1180 $cm^{-1}$), and $CH_2$ groups (1340 and 1460 $cm^{-1}$).

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# SYNTHESIS OF VINYL MONOMERS IN THE DIARYLOXAZOLE SERIES

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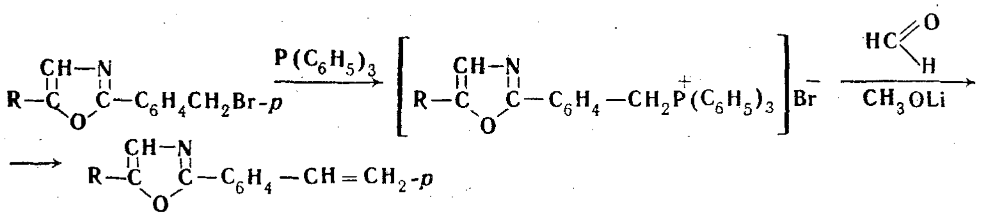
In preparing scintillating plastics, interest attaches to formation of polymer chains containing structural groupings which scintillate [1, 2]. With this aim in mind, we have synthesized monomers, vinyl derivatives of diaryloxazoles of general formula I, by reacting halogenomethylaromatic compounds with triphenylphosphine, paraform, and lithium methoxide (Wittig method):



Ia—c with $R=C_6H_5$; b $R=o\text{-}C_6H_5C_6H_4$; c $R=\alpha\text{-}C_{10}H_7$

The 2-(p-bromomethylphenyl)-5-aryloxazoles required for this synthesis are easily prepared by cyclizing (p-bromomethylbenzoyl)-ω-amino derivatives of aliphatic-aromatic ketones with sulfuric acid or phosphorus oxychloride.

2-(p-Vinylphenyl)-5-phenyloxazole (Ia). 2.62 g (0.01 mole) $Ph_3P$ was introduced, with heating, into a solution of 3.14 g (0.01 mole) 2-(p-bromomethylphenyl)-5-phenyloxazole in 5 ml dimethylformamide, the mixture refluxed for 1 hr, the precipitate separated off, washed with petrol ether, and dried, yield 5.7 g (100%) phosphonium salt. It was dissolved in 100 ml MeOH, 0.3 g (0.01 mole) paraform and a solution of 0.07 g (0.01 g at) Li in 50 ml MeOH added, and the whole stirred for 3 hr. 150 ml water was added, the precipitate separated off, and after drying, purified on an aluminum oxide column. Yield 1.2 g (50%), white crystals, mp 65–67° C, crystals luminescence $\lambda_{max}$ 415 mμ, in MeOH it was 390 mμ. Found: N 5.66%. Calculated for $C_{17}H_{13}NO$: N 5.66%.

2-(p-Vinylphenyl)-5-biphenyloxazole (Ib). Prepared similarly, 40% yield, mp 158–161°, luminescence $\lambda_{max}$ for crystals 445 mμ, for MeOH solution 405 mμ. Found: N 4.29%. Calculated for $C_{23}H_{17}NO$: N 4.33%.

2-(p-Vinylphenyl)-5-α-naphthyloxazole (Ic). This was prepared similarly. Mp 80–82°, luminescence $\lambda_{max}$, for crystals 465 mμ, in MeOH 410 mμ. Found: N 4.96%. Calculated for $C_{21}H_{15}NO$: N 4.71%.

## REFERENCES

1. J. Drefahl and K. Winnefeld, J. Pr. Chem., 28, 236, 1965.
2. K. G. Volodaikin and A. S. Cherkasov, ZhOrKh, 1, 927, 1965.
3. L. A. Yanovskaya, Usp. khim., 30, 813, 1961.

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