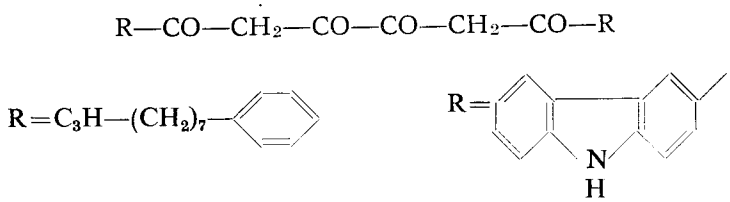
M. Jančevska, L. Fukara-Jovevska, J. Jančulev

SYNTHESIS OF THE 1, 6-DI-(N-OCTANEPHENYLENE) AND 3, 6-DI-(CARBAZYL)-1, 3, 4, 6-HEXANETETRONE

As a continuation of our work in the field of the polyoxo compounds [1, 2, 3] synthesis of 1, 6-Di-(phenylene-n-octane)-1, 3, 4, 6-hexanetetrone and 3, 6-Di-(carbazyl)-1, 3, 4, 6-hexanetetrone have been carried out by the Claisen's and Bromme's [4, 5, 6] methods.

The condensation was carried out of 2 mols of n-octane-p-acetylphenylene [7] as well as of 2 mols 2-acetyl-carbazyl [8] and 1 mol diethyloxalate in the presence of sodium ethylate. The tetracetones were identified by their hinoxaline derivatives.



EXPERIMENTAL

1, 6-Di-(phenylene-n-octane)-hexantetrone

In a pear-shaped flask of 250 ml supplied by reverse fan (cooler) and calcium cloride pipe, sodium ethylate was prepared in apsolute ether of 0,23 g. (0,01 mol) sodium and 0,46 g. (0,01 mol) absolute alcohol.

A mixture of 4,94 g. (0,02 mol) n-octane-p-acetyl phenylene and 1,46 g. (0,01 mol) freshly destilled diethylocalate in absolute ether was added to the obtained sodium ethylate. This reaction mixture which was stirred from time to time stayed so three days. The colour of the condensation product was changing from yellow to dark red, but no sediment occurred. The obtained condensation product had dark brown colour.

The sodium compound of the condensation product was further elaborated by weakened hydrochoric acid (1 : 1), and the nice light yellow cry-

stals were obtained. The product was filtered, washed by water and dried by air. Then a raw produckt with a melting point of 113—115°C was obtained from the ethanole.

Anal. Calc'd for $C_{34}H_{46}$ (518, 708): C, 78,93% H, 9,09%

Found: C, 78,91%, H. 9,18%

Hinoxaline derivatives of 1, 6-Di-(n-octane-phenylene)-1, 3, 4, 6-Hexantetrone

Hinoxaline derivative is obtained by one hour heating of equimolecular amounts of 1, 6-Di-(n-octane-phenylene)-1, 3, 4. 6-Hexanetetrone and o-phenylenediamine in absolute ethanol. After the precrystallisation of ethanol nice red shells with a melting point of 87°C were obtained.

Anal. Calc'd for $C_{40}H_{50}N_2O_2$ (590, 816)

C, 81,52% H, 8,54% N, 4,75%

Found: C, 81,41% H, 8,40% N, 4,41%

3, 6-Di-(carbazyl)-1, 3, 4, 6-Hexanetetrone

The tetraceton was obtained in the way already discribed. The reaction produckt colour was changing from yellow to orange. The sodium salt obtained from the condensation product was elaborated by HCl (1 : 1) and ice. The hard (solid) raw tetraceton has dark yellow colour which is filterred, washed by water and dried by air. After more precrystallisations crystals with light yellow colour and melting point above 250°C are obtained from the alcohol.

Anal. Calc'd for $C_{30}H_{20}N_2O_4$ (472,476)

C, 76,25% H, 4,27% N, 5,93%

Found: C, 76,07% H, 4,29% N, 5,71%

Hinoxaline derivative of 3, 6-Di-(carbazyl)-1, 3, 4, 6-Hexanetetrone

Hinoxaline derivative is obtained by four hours heating of equimolecular amounts of 3, 6-Di-(carbazyl)-1, 3, 4, 6-Hexanetetrone and o-phenylenediamine in medium of absolute ethanol. After many precrystallisation a product of nice red colour and melting point of 250°C was obtained from the alcohol.

Anal. Calc'd for $C_{36}H_{21}N_4O_2$ (544,584)

C, 79,41% H, 4,45% N, 10,29%

Found: C, 79,23% H, 4,12% N, 10,47%

REFERENCES

1. J. Jančulev, M. Jančevska, Bulletin Scientifique Tome, 6, No I (1961)
2. M. Jančevska, B. Podołešov, Łj Fukarova, Annuarie de la Faculté des Sciences de de L' Université de Skopje, Tome (17-18) 127 (1967)
3. J. Jančulev, M. Jančevska, Annuare de la Faculté des Sciences de L'Université de de Skopje Tome 12 (1959) 101
4. C. Beyer, L. Claisen, Ber. 20 (1887) 2078
5. E. Brömme, L. Claisen Ber. 21 (1888) 1134
6. J. Am. Chem. Soc. 75 (1953) 3326
7. A. Lund, J. Bjerrum Ber. 64 (1931) 210
8. Ber. 40 (1907) I 381

ИЗВОД

Направена е синтезата на 1, 6-ди-(н-окта нафтилен)-1, 3, 4, 6-хексан тетракетон (жолти иглици Т.Т. 115—117) и на 3, 6-ди-(карбазил)-1, 3, 4, 6-хексан тетрон (жолти кристали Т.Т. над 250°). Истите со идентификувани преку нивните хиноксалински деривати.

Скопје, 22. I. 1971 год.

Хемиски институт
Природноматематички факултет