LETTERS TO THE EDITOR

NEW APPROACH TO THE SYNTHESIS OF A TRIFURYLCARBONIUM PERCHLORATE

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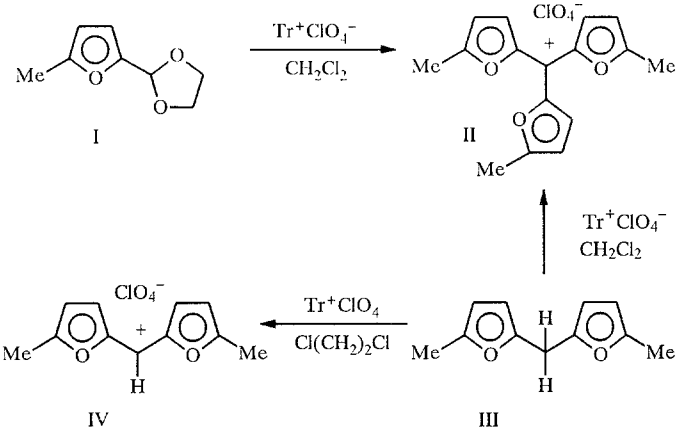
The corresponding dioxolanium salts are formed in the action of trityl perchlorate on 2-substituted dioxolanes in methylene chloride [1].

We have established that this reaction proceeds differently with 2-(5-methyl-2-furyl)dioxolane (I) and leads to tris(5-methyl-2-furyl)carbonium perchlorate (II), which was previously obtained by the method in [2], in virtually quantitative yield.



This transformation can be explained by the lability of the C—Fur bond under the given conditions and gradual accumulation of the number of furan rings around the positively charged carbon atom. The reaction of difurylmethane III with trityl perchlorate in methylene chloride leads to a similar result. According to the PMR data, replacement of methylene chloride by acetonitrile and rapid precipitation of the reaction products by the addition of ether leads to a mixture of II and IV in a ratio of 1:10. The use of finely dispersed trityl perchlorate and carrying out the reaction in dichloroethane made it possible to isolate difurylmethyl salt IV in 80% yield; this is explained by the low solubility of salt IV in this solvent and its removal from the reaction sphere.

Tris(5-methyl-2-furyl)carbonium Perchlorate (II, $C_{16}H_{15}ClO_7$). This compound had mp 234-235°C (dec.). PMR spectrum (CF_3COOH): 2.23 (9H, s, CH_3); 6.43 (3H, d, 4-H); 7.75 ppm (3H, d, 3-H); spin-spin coupling constant (SSCC): $J_{3,4}$ = 4.0 Hz.

Bis(5-methyl-2-furyl)carbonium Perchlorate (IV, $C_{11}H_{11}ClO_6$). This compound had mp 152-153°C (dec.). PMR spectrum (CF_3COOH, 25°C): 2.35 (s, CH_3); 2.37 (s, CH_3' + CH_3''); 6.53 (d, 4-H'); 6.57 (d, 4-H''); 6.68 (d, 4-H); 7.33 (s, CH); 7.48 (s, CH' + CH''); 7.70 (d, 3-H'); 7.75 (d, 3-H''; 8.22 ppm (d, 3-H); SSCC: $J_{3,4}$ = 4.0 Hz. The triple set of signals in the PMR spectrum of the II cation probably indicates its existence in solution in the form of three rotamers, the signals of which merge when the temperature at which the spectrum is recorded is raised.

REFERENCES

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