

Isloxazolinium Chloride-Ferric Chloride Salts

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(II) 2-methyl-3-phenyl-5-*p*-bromophenylisoxazolinium chloride-ferric chloride salt:

| Color | M.P. | Analysis |
|--------|------|---|
| Yellow | 138° | Calcd. for $C_{16}H_{15}BrCl_4FeNO$: C, 37.3; H, 2.9; Fe, 10.8 Found: C, 37.2; H, 3.0; Fe, 10.8 |

LITERATURE CITED

- (1) Blatt, A.H., *J. Am. Chem. Soc.*, **71**, 1861 (1949).
- (2) Blatt, A.H., Gross, Norma, *Ibid.*, **77**, 5424n(1955).

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COMPOUNDS (I) and (II) are oxidized with red chromic acid in glacial acetic acid to their corresponding isoxazol chloride-ferric chloride salts (1, 2).

(I) 2-methyl-3-*p*-bromophenylisoxazolinium chloride-ferric chloride salt:

| Color | M.P. | Analysis |
|--------|------|---|
| yellow | 115° | Calcd. for $C_{15}H_{13}BrCl_4FeNO$: C, 37.3; H, 2.9; Fe, 10.8 Found: C, 37.2; H, 3.0; Fe, 10.7 |

Mixed Ketals and Acetals of 2-Methoxy-2-Phenylethanol and Methanol

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IN CONNECTION with another problem it was necessary to prepare 2-(2-methoxy-2-phenylethoxy)-2-methoxypropane and 1-(2-methoxy-2-phenylethoxy)-1-methoxypropane. The transketalization procedure reported by Lorette and Howard [*J. Org. Chem.* **25**, 521 (1960)] provided a convenient method for preparation of these new compounds.

EXPERIMENTAL

Preparation of 2-(2-Methoxy-2-phenylethoxy)-2-methoxypropane. A mixture of 450 grams (2.96 moles) of 2-methoxy-2-phenylethanol, 1800 grams (17.3 moles) of 2,2-dimethoxypropane, and 180 grams of dry Dowex 50 acid ion exchange resin was stirred and heated under reflux for 3 hours. The Dowex resin was then removed by filtration and the excess 2,2-dimethoxypropane and methanol removed under vacuum. Distillation of the high boiling

material yielded 168.5 grams of unreacted 2-methoxy-2-phenylethanol and 345 grams of 2-(2-methoxy-2-phenylethoxy)-2-methoxypropane. The yield based on converted 2-methoxy-2-phenylethanol was 83.3%.

Preparation of 1-(2-Methoxy-2-phenylethoxy)-1-methoxypropane. A mixture of 200 grams (1.92 moles) of 1,1-dimethoxypropane, 50.0 grams (0.329 mole) of 2-methoxy-2-phenylethanol, and 20 grams of dry Dowex 50 acid ion exchange resin was treated in exactly the same manner as described above. On distillation of the product there was obtained 24.6 grams of unreacted 2-methoxy-2-phenylethanol and 37.2 grams (0.166 mole) of 1-(2-methoxy-2-phenylethoxy)-1-methoxypropane. The yield based on converted 2-methoxy-2-phenylethanol was 98.7%.

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| | B.P. at 2.5 Mm. of Hg, ° C. | n_D^{20} | d_{20} | Refraction, <i>M</i> | | % Acetone on Acid Hydrolysis, % ($C_{13}H_{20}O_3$) | |
|---|--------------------------------|------------|----------|----------------------|--------|---|--------|
| | | | | Found | Calcd. | Found | Calcd. |
| 2-(2-Methoxy-2-phenylethoxy)- 2-methoxypropane | 110 | 1.4830 | 1.002 | 63.92 | 63.56 | 24.7 | 25.9 |
| | B.P. at 2 Mm. Hg, ° C. | | | | | % Propionaldehyde on Acid Hydrolysis, % ($C_{13}H_{20}O_3$) | |
| 1-(2-Methoxy-2-phenylethoxy)- 1-methoxypropane | 98-99 | 1.4827 | 1.004 | 63.68 | 63.56 | 24.4 | 25.9 |