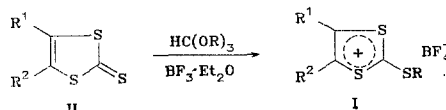


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2-Alkylthio-1,3-dithiolium salts (I) are valuable intermediates for a number of syntheses [1]; however, not all of the derivatives are readily accessible. Thus, whereas IIA is alkylated quantitatively by methyl iodide, methyl fluorosulfonate was used for the alkylation of 1,3-dithiole-2-thiones II by the simultaneous action of ortho esters of formic acid and boron trifluoride etherate.



a $R^1=R^2=H$, $R=CH_3$; b $R^1=R^2=CH_3$, $R=C_2H_5$; c $R^1=C_{17}H_{35}$, $R^2=H$, $R=CH_3$; d $R^1=R^2=COOCH_3$, $R=CH_3$; e $R^1=CH_2COOC_2H_5$, $R^2=H$, $R=CH_3$; f $R^1=Ph$, $R^2=H$, $R=C_2H_5$; g $R^1=R^2=Ph$, $R=CH_3$

A 10-mmole sample of thione II was dissolved in chloroform, and 11 mmole of the ortho ester and 11 mmole of boron trifluoride etherate were added simultaneously. The reaction product precipitated in the form of a yellowish oil, which crystallized when ether was added and the mixture was cooled. In the alkylation of thione IIId, the reagents were added dropwise in the course of 30 min from two dropping funnels to a refluxing solution of the thione in chloroform. The following salts were obtained [yields and PMR data (δ , CF_3COOH) given]: Ia, 100%, 2.93 (3H, s, CH_3) and 8.30 ppm (2H, s, CH); Ib, 100%, 1.38 (3H, t, CH_3), 2.34 (6H, s, CH_3), and 3.31 ppm (2H, q, CH_2); Ic, 100%, 1.0 (35H, broad, CH_3 , CH_2 , broad), 2.75 (3H, s, CH_3), and 7.75 ppm (1H, s, CH); Id, 92%, 2.90 (3H, s, CH_3) and 2.98 ppm (6H, s, CH_3); Ie 100%, 1.09 (3H, t, CH_3C), 2.90 (3H, s, CH_3), 4.05-4.12 (4H, m, $CH_2OC(O)CH_2$), and 8.09 ppm (1H, s, CH); If, 100%, 1.43 (3H, t, CH_3), 3.45 (2H, q, CH_2), 7.35 (5H, m, C_6H_5), and 8.15 ppm (1H, s, CH); Ig, 98%, 2.93 (3H, s, CH_3) and 7.18 ppm (10H, m, C_6H_5). The structures of salts Ia-g were also confirmed by their IR spectra and chemical transformations; the results of elementary analysis were in agreement with the calculated values.

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