approximately 1:1.66:6.23:0.42, when 2,3,4,6-tetra-*O*-methyl-D-glucose was taken as unity.

Thus it appears that the chemical structure of *Ishige* laminaran is composed of (1→3)- and (1→6)-linked D-glucose residues, and thus it is similar to that of *Eisenia* laminaran. The linkages are, however, in a different ratio, namely, in the former polysaccharide 4:1, in the latter one 3:1. Moreover, *Ishige* laminaran differs in having a single branch, but its position is not known. These results show that the structure of laminaran seems to depend on the source. In addition, it is probable that the variations in solubility of laminaran may be due not only to the degree of branching, as pointed out by Annan *et al.*[7], but also to the content of (1→6)-linkage.

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Bis(1,2:5,6-di-*O*-isopropylidene-3-*O*-thiocarbonyl-α-D-glucofuranose) disulfide

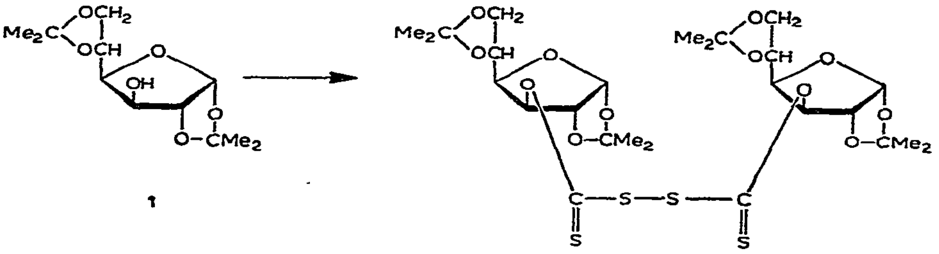
A rapid method is reported for the quantitative preparation of bis(1,2:5,6-di-*O*-isopropylidene-3-*O*-thiocarbonyl-α-D-glucofuranose) disulfide (**2**) from 1,2:5,6-di-*O*-isopropylidene-α-D-glucofuranose (**1**). Yields of **2** approaching 100% are obtained within 1 h. By other methods[1], maximum yields were only 66% in 16 h.

Compound **2** is a useful intermediate for the following syntheses: preparation of 1,2-*O*-isopropylidene-α-D-glucofuranose 5,6-thionocarbonate[1] (by removal of the 5,6-isopropylidene group from **2**, followed by treatment with pyridine); preparation of *O*-(alkoxythiocarbonyl) derivatives[2] of **2** (by treating **2** with alcohols under specific conditions); and the indirect preparation of some *S*-alkylxanthates[3].

Carbohyd. Res., 7 (1968) 99–100

EXPERIMENTAL

Compound **1** (15 g) was dissolved in methyl sulfoxide (15 ml), and aqueous sodium hydroxide solution (5M, 15 ml) was added, along with carbon disulfide (10 ml).



1

2

The resultant yellow solution, which turned red almost immediately, was stirred for 7 min at 25°. The solution was cooled to 5° and acetic acid (5M) was added dropwise until the mixture became yellow (pH about 5). The xanthate present in the mixture was oxidatively crosslinked immediately with a cold, aqueous solution of iodine. The iodine was added as fast as was convenient, to minimize decomposition of the xanthate to other products. A dark-brown syrup resulted, which was separated and dissolved in ether. The extract was washed, first with aqueous sodium thiosulfate until the color was a bright yellow, and then with water. The ether layer was dried (anhydrous magnesium sulfate), and evaporated to yield **2** (19.6 g, 100%) as a heavy syrup which crystallized as needles on standing. T.l.c. (ethyl acetate–carbon disulfide, 9:1 v/v, as solvent, and Silica Gel G* as adsorbent) revealed a single component that had an R_F value the same as that of authentic **2**. After the product had been recrystallized from alcohol–water, its m.p. was undepressed in admixture with an authentic sample of **2**. Although yields for several preparations of **2** were almost 100% by this procedure, yields of only 60–70% of **2** were obtained when cooling to 5° was omitted.

This facile procedure should be applicable for preparation of xanthates and of oxidatively coupled xanthates of various hydroxy compounds.

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