

## The Preparation of *tert*-Butylsuccinic Acid

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In a recent publication<sup>1</sup> the conjugate addition of Grignard reagents to maleic esters to give alkylsuccinic esters was described. The directions for the preparation of *tert*-butylsuccinic esters seem, however, to be insufficient to secure the yields reported. Some additional details of the procedure used should therefore be given.

For the preparation of the Grignard reagent from *tert*-butyl chloride directions are given in Ref. <sup>2</sup>. The amount of Grignard reagent required is 4.0 equiv. (In Ref. <sup>1</sup>, Table 1, foot-note <sup>g</sup> the amount is erroneously reported to be 0.4 equiv.). Thus for 0.2 mole of ester the Grignard reagent from 0.8 mole of *tert*-butyl chloride should be used. The copper(I) chloride catalyzed addition reaction to give *tert*-butylsuccinic ester was otherwise carried out essentially as described in previous communications<sup>1-4</sup>.

The Grignard reagent (0.8 mole in 500 ml of ether) was cooled in an ice-salt bath (-10—-18°) for 15 min. The ester, dissolved in 200 ml of ether, was added during 2 h. Concurrently with the ester, 1.4 g of copper(I) chloride (1.75 mole % with respect to the Grignard reagent) was added in seven 0.2 g portions. After further stirring and cooling of the reaction mixture for 15 min, the ice-salt bath was removed, and the stirring was continued at room temperature. After 1 h, another 0.2 g of copper(I) chloride was added, and after another 1 h of stirring the reaction mixture was worked up the conventional way<sup>4</sup>.

The *tert*-butylsuccinic acid was isolated as reported in Ref. <sup>1</sup>.

1. Andersen, V. K. and Munch-Petersen, J. *Acta Chem. Scand.* **16** (1962) 947.
2. Munch-Petersen, J. and Andersen, V. K. *Acta Chem. Scand.* **15** (1961) 293.
3. Munch-Petersen, J. and Andersen, V. K. *Acta Chem. Scand.* **15** (1961) 271.
4. Munch-Petersen, J. *Org. Syn.* **41** (1961) 60.

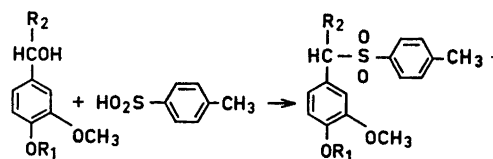
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## Sulphones of Some Lignin Models

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In the reaction between lignin and sulphite, the benzyl alcohol or ether groupings in the lignin are transformed into sulphonic acids. The reaction between lignin and a sulphinic acid would likewise be expected to give sulphones. Hinsberg<sup>1</sup> and Kenyon and coworkers<sup>2,3</sup> have also prepared sulphones from benzyl alcohols and benzhydrols by this method. The introduction of the strongly electron attracting sulphone group into lignin presents a possible approach to the controlled degradation of this product. To study this possibility, the initial step was to study the reaction between some simple model substances (I—IV) and *p*-toluene sulphinic acid.



- I  $R_1 = R_2 = H$   
 II  $R_1 = CH_3; R_2 = H$   
 III  $R_1 = H; R_2 = CH_3$   
 IV  $R_1 = R_2 = CH_3$

Vanillyl alcohol (I) and 1-(4-hydroxy-3-methoxyphenyl)-ethanol (III), in which the reactivity of the alcoholic group is enhanced by the *p*-hydroxyl substituent, reacted readily at pH 4.5 and 100°. Veratryl alcohol (II) and 1-(3,4-dimethoxyphenyl)-ethanol (IV) reacted more readily under acid conditions. Aqueous acetic acid, to which a small amount of sulphuric acid had been added, and a reaction temperature of 100° proved to be satisfactory conditions for the preparation of all the four sulphones. These were obtained in good yields as crystalline, easily purified substances. The sulphone from vanillyl alcohol was also formed at room temperature and a longer reaction time.

*Experimental.* Melting points are corrected. (4-Hydroxy-3-methoxyphenyl)-methyl *p*-tolylsulphone. (a) Vanillyl alcohol (1.00 g) and