mixture was also dependent on the temperature. At about 5°C both complexes are insoluble, at 20°–30°C only the CS-4 complexes are soluble and at 65°C both complexes are soluble. On cooling the CS-6 complex first precipitates and the CS-4 complex does not precipitate until the temperature is lowered below 10°C. Another factor of importance for the solubility of the CP-complexes is the presence of salt in the solution. Concentrations of salts (Na_2SO_4 or $MgCl_2$) necessary to dissolve the CP-hyaluronic acid complex in water are sufficient to dissolve the CP-CS-6 complex in the mixture of organic solvents used in this investigation.

A detailed description of the technique for separations on the macro- as well as on the micro-scale is to be published elsewhere.

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The Identification of Organic Compounds
III. Preparation of *p*-Phenylphenacyl Oxalate

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Drake and Bronitsky[1] introduced *p*-phenylphenacyl bromide as a reagent for the identification of carboxylic acids. The ester was prepared by refluxing the sodium salt of the acid with *p*-phenylphenacyl bromide in alcoholic solution. The ester of oxalic acid could not be prepared in this way due to the insolubility of sodium oxalate. By using the methylammonium salt, however, Drake and Bronitsky obtained the oxalate, m.p. 165.5° (decomp.).

In a previous paper[2] an improved method for the preparation of *p*-bromophenacyl oxalate was given. This procedure has now been used for the preparation of *p*-phenylphenacyl oxalate. Sodium oxalate and *p*-phenylphenacyl bromide were refluxed in methyl cellosolve. A derivative with m.p. 246° (decomp.) was obtained and the analytical data confirmed that it was the diester of oxalic acid. Obviously a side reaction has taken place by using the methylammonium salt. We have not been able to obtain a compound with a constant melting point of about 165° so the structure of the compound prepared by Drake and Bronitsky still remains to be elucidated.

Experimental. Our procedure for the preparation of *p*-bromophenacyl oxalate[2,p.641] was followed. 0.25 g of oxalic acid dihydrate and 1 g of *p*-phenylphenacyl bromide were used. Yield about 45 %. Usually a pure derivative was obtained; it can be recrystallised from glacial acetic acid (sparingly soluble). M.p. 246° (decomp.) (corr.). Capillary tube introduced at 225–230°, rate of heating 4°/min. (Found: C 75.08; H 4.55. Calc. for $C_{30}H_{22}O_6$: C 75.30; H 4.63).

Microanalysis by Mr. A. Bernhardt, Max-Planck-Institut, Mülheim, Germany.

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