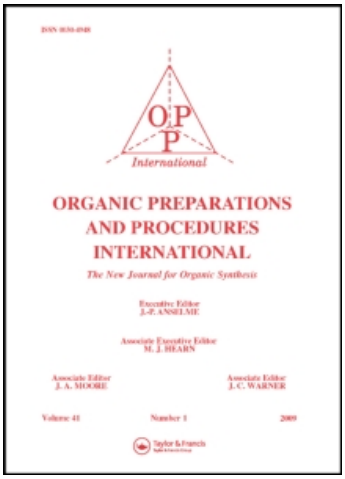

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AN IMPROVED PREPARATION OF CORONENE

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AN IMPROVED PREPARATION OF CORONENE

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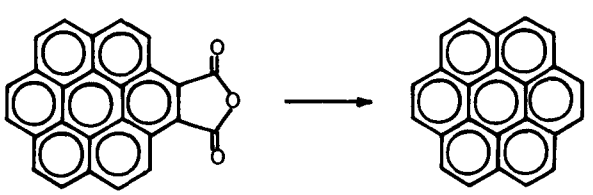
Synthesis of coronene is generally on a mg scale. Our attempts to increase the scale were frustated until we developed a heater and improved glassware[1] which enabled successfully carrying out the reaction at the required temperature. Previously reported yields of coronene were 66% (mp. 425-428°)[2] and 73% crude with 18% purified.[3] We consistently obtained pure coronene in 80% yield, mp. 434-438° (uncorr.) on a gram scale.



I II

EXPERIMENTAL

Melting points were determined on Mel-temp capillary melting point apparatus with a 90-510° thermometer from H-B instru-

ment Co. and are uncorrected.

Fractional Sublimation of Coronene-1,2-dicarboxylic Anhydride (I).- The anhydride (I) (20.8 g, 0.056 mol) was placed in a 250 ml flask. The flask was attached to a sublimation system[1] and alternately evacuated and flushed with argon. Upon heating with a sand bath, 1,12-benzoperylene and other volatile impurities were sublimed away from the anhydride at 250-400° (0.2 mm Hg) during 4 hrs. After cooling, the receiver was replaced and the system evacuated and flushed with argon (4x). The flask was again heated using the high temperature sand bath[1] to 470-490° (0.2 mm Hg). The bulk of the anhydride sublimed suddenly at 490° giving 20.2 g (97%) of bright red-orange crystals of I, mp. > 505°, [lit.[2] mp. 490-503°]. TLC[5] studies showed absence of benzoperylene. However, traces of coronene were observed.

Preparation of Coronene (II) from Anhydride (I).- A 4.0 g (0.01 mol) sample of anhydride I and 10 g soda lime (Malkinckrodt) were thoroughly mixed with a mortar and pestle. This mixture was placed in the bottom of the reaction flask[1] and covered with a 6 g layer of powdered soda lime and then 35 g of 4-8 mesh Soda-Lime particles. Glass wool was then placed above the reactants. The system was alternately evacuated and flushed with argon (4x). Upon heating to 350-400° (0.05 mm Hg), coronene sublimed above the glass wool. This sublimate was placed in a Soxhlet apparatus[4] charged with Merck, activity I, basic alumina. The alumina was contained as a 5 cm diameter x 6 cm volume. The hydrocarbon was eluted with refluxing toluene to give a suspension of coronene (II). Cooling and filtering

gave 2.4 g (80%) of II as bright yellow fluffy needles, mp. 434-438° (uncorr.).

Purification of Coronene (II).- The combined product of 8 reactions (16.2 g) was placed in a Soxhlet column charged with basic alumina and eluted with refluxing toluene. The resulting solution was filtered to give 15.0 g. Vacuum-sublimation gave 14.4 g, mp. 437-440° (uncorr.) [lit.[3] mp. 440°]; mass spectrum m/e 300 (M^+). HPLC analysis with Waters liquid chromatograph using a 254 nm UV detector, and a (3.9 nm x 30 cm) C_{18}-μ-Bondapak column with acetonitrile solvent (1 ml/min) showed the purity to be greater than 99%.

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