(after 2–4 min), the mixture was stirred vigorously with a glass rod until the reaction had subsided. A second portion was added and rapid stirring was resumed. After the last portion was added the reaction mixture was stirred for 10 min. Heating with periodic stirring was continued until the evolution of HCl gas was no longer evident (about 30 min). The reaction mixture was then cooled and ground in a mortar and then added, with stirring, to a mixture of ice (2 g) and conc. HCl (1 ml). The resulting slurry was stirred for 0·5 hr, the solid was filtered and washed with cold water. After thorough drying *in vacuo* this yellow material (2 g) was examined by TLC (silica gel, $CHCl_3$) and was found to consist of 2,4-dimethoxy-3-acetoxy-6-hydroxyacetophenone with traces of the corresponding deacetylated compound.

A small portion of this mixture (25 mg) was separated by preparative TLC. The major product (20 mg, VI) was recrystallized from MeOH to yield colourless crystals, m.p. 109–110°, λ_{max}: 278, 317 nm; ν. 3520, 1760, 1620 cm^{-1}; NMR signals: τ 3·45 (1H, singlet, Ar–OH), 3 72 (1H, singlet, Ar–H), 6·10 (3H, singlet, Ar–OCH_3), 6·15 (3H, singlet, Ar-OCH_3), 7 32 (3H, singlet, Ar–$COCH_3$), 7·66 (3H, singlet, Ar–$OCOCH_3$); mass spectrum: 254 (5), 212 (100), 197 (80), 194 (15), 151 (17). Found: C, 56 61; H, 5·60. Calc for $C_{12}H_{14}O_6$: C, 56·69; H, 5·55.

2,5-*Dihydroxy*-4,6-*dimethoxyacetophenone*. The crude Fries rearrangement mixture was thoroughly dried, and then refluxed with HCl gas (5%) in MeOH for 1 hr after which time the solvent was removed. Examination of this product by TLC revealed the presence of one major component. Preparative TLC yielded yellow crystalline material, m.p. 162–163° (lit. m.p. 162–163°),[14] λ_{max}: 242, 283 nm, ν_{max} ($CHCl_3$): 3600, 1640 cm^{-1}; NMR signals: τ 3 16 (1H, singlet, Ar–OH), 3·70 (1H, singlet, Ar–H), 4·4 (1H, multiplet, Ar–OH), 6·00 (3H, singlet, Ar–OCH_3), 6·02 (3H, singlet, Ar–OCH_3), 7·30 (3H, singlet, Ar–$COCH_3$); mass spectrum: 212 (90), 197 (100), 182 (38), 179 (20), 169 (25), 151 (50). Found: C, 56·53; H, 5 68. Calc. for $C_{10}H_{12}O_5$: C, 56 60; H, 5·70.

2,3,4-*Trimethoxy*-6-*hydroxyacetophenone*. This compound was obtained via the mild methylation procedure reported by Sastri and Seshadri.[10] The resulting brown amorphous product was found to contain several components by TLC. Preparative TLC (25 mg) yielded a small amount of yellow oil (9 mg) as the least polar component. This oil was found to have spectroscopic properties identical with those of the hydrolysis product of compound D.

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CERCIDIPHYLLACEAE

ISOLATION OF MALTOL FROM *CERCIDIPHYLLUM JAPONICUM*

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Plant. Cercidiphyllum japonicum Seib. et Zucc. *Uses.* Flavour enhancer. *Isolation.* The Et_2O extract from the fresh leaves was steam distilled. The steam distillate was extracted with Et_2O and the extract was evaporated. From the residue maltol[1] (3-hydroxy-2-methyl-4-pyrone), $C_6H_6O_3$, was obtained (1·6% of dried leaves) and recrystallized from EtOAc, m.p. 162°. It was identified by m.p., mixed m.p., UV, IR and NMR.

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[1] T. KARIYONE and T. SAWADA, *Yakugaku Zasshi* **79**, 265 (1959).