

Decomposition solutions from runs 7 and 8 were mixed with 3 ml 30 % hydrogen peroxide and evaporated to dryness on a water-bath. The residues consisted of white, somewhat sticky crystals which were identified as maleic acid. In the runs 0.935 g and 1.153 g *p*-benzoquinone reacted with ozone and gave 199 mg and 228 mg maleic acid, respectively. Controls by precipitating the dissolved residues with barium chloride were satisfactory.

Formic acid. Aliquots of the decomposition solutions were distilled *in vacuo* to dryness. The distillate was taken up in standard sodium hydroxide and backtitrated with hydrochloric acid. The capillary was connected to a sodium hydroxide-asbestos tube to prevent the entrance of carbon dioxide. Substituting the air in the distilling apparatus with nitrogen did not alter the titration values significantly.

Ion-exchange chromatography. The decomposition solution was passed through a column of De-acidite G and washed with water. The neutral washings which reduced ammoniacal silver solution were combined and precipitated with *p*-nitrophenylhydrazine. This precipitate was extracted with glacial acetic acid and recrystallised from nitrobenzene m. p. ca. 270° (decomp.). Through its infra-red spectrum it was identified as glyoxal *p*-nitrophenylosazone. Further elution was performed with 5 % formic acid when more aldehydic material appeared. The acidic eluate was precipitated with 2,4-dinitrophenylhydrazine and the material extracted with boiling water. In one case there separated yellow needles from the aqueous extracts, m. p. 190°, identified as glyoxylic acid dinitrophenylhydrazone. The residue from the extraction was partially soluble in hot glacial acetic acid. From the acetic acid separated red crystals, m. p. 226° (decomp.). (Found: C 43.9; H 3.2; N 20.6.) The insoluble part was recrystallised from nitrobenzene. Yellowish crystals, m. p. 255° (decomp.). (Found: C 41.6; H 2.7; N 22.4. Calc. for C₁₇H₁₂N₈O₁₀: C 41.8; H 2.5; N 22.9.)

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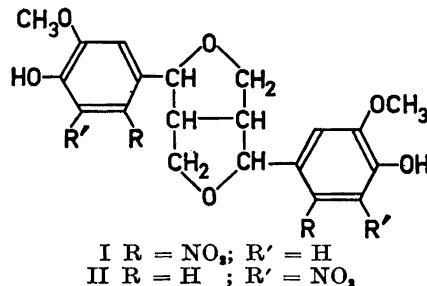
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The Structure of Dinitropinoresinol

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Some years ago the preparation of dinitropinoresinol was reported¹. Its structure was not determined, but it was assumed to be I in analogy with dinitropinoresinol dimethylether. Dinitropinoresinol gives, however, upon treatment with nitric acid 4,6-dinitroguaiacol, m. p. 122°, and must therefore have structure II



The aim of the earlier work¹ was the degradation of the aromatic rings of pinoresinol to carboxylic groups. We have now attempted to achieve this by ozonolysis (*cf.* Ref.²), but the desired acid could not be obtained. The only products that could be identified were oxalic acid and in some experiments where the reaction time was kept very short, maleic acid.

In connection with this work we had to prepare relatively large amounts of pinoresinol and we found that when the crude pinoresinol from the potassium salt³ was treated with dioxan it immediately crystallised. The crystals contain 1 molecule of dioxan, which can readily be removed by heating under vacuum on a boiling water bath. This method of purification is more convenient than the customary one *via* the acetate³.

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