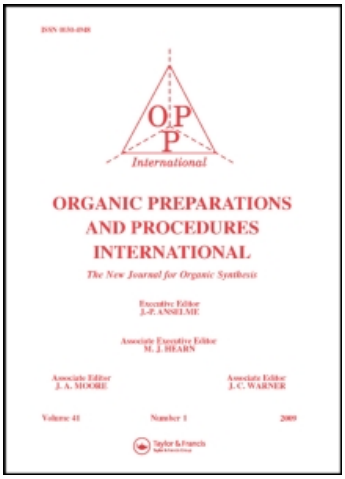


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# IMPROVED PREPARATIONS OF CYCLONONEN-3-OL, CYCLONONEN-3-ONE, CYCLONONANONE AND CYCLONONANOL

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# IMPROVED PREPARATIONS OF CYCLONONEN-3-OL, CYCLONONEN-3-ONE, CYCLONONANONE AND CYCLONONANOL

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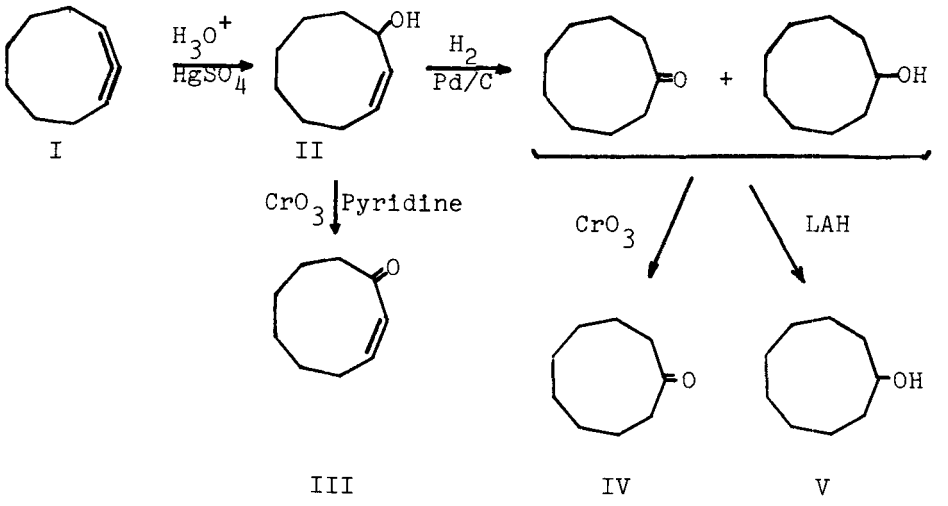
An improved method for the preparation of the title compound is described. 1,2-Cyclononadiene(I) obtained via 9,9-dibromobicyclo (6.1.0) nonane[2,3] was hydrated by a modification of the procedure of Sharma, Shoulders and Gardner[4] to give II in 74% yield. Oxidation of II with Sarett's reagent[5] yielded III. Catalytic reduction of II over 5% palladium on carbon produced a mixture of IV and V,



which could be reduced by LAH to give pure V or oxidized by Jones reagent[6] to yield IV.

## EXPERIMENTAL

Cyclonon-2-en-1-ol(II). Cyclonona-1,2-diene (30 g., 0.24 mole) dissolved in tetrahydrofuran (200 ml.) was added to a solution prepared from dilute sulfuric acid (20 g. in 30 ml. of water), mercuric sulfate (3 g.) and tetrahydrofuran (300 ml.). The mixture was refluxed for 4 hr. under nitrogen and filtered to remove the precipitated inorganic salts. The organic phase was separated by dilution with water (200 ml.) and extraction with three 100-ml. portions of ether. The combined ethereal extract was washed with 50-ml. portions of brine, dried and freed of solvent to give 28.3 g. of a pale yellow oil. Distillation yielded 25.1 g. (74%) of a colorless liquid, bp. 88-90°/1 mm., $n_D^{26}$ 1.4961; lit.[4] reports 17% yield. The vpc analysis of the product indicated < 99% homogeneity. The above liquid on trituration with pentane and chilling readily crystallized to give a white, waxy solid, mp. 31°. Its IR spectrum displayed signals due to two olefinic protons between $\tau$ 3.67 and 4.52 (multiplet) and a one proton absorption at $\tau$ 5.34 (broad).

Catalytic Reduction of Cyclonon-2-en-1-ol. The unsaturated alcohol (6 g.) in ethyl acetate (40 ml.) was hydrogenated at 25°C and atmospheric pressure over 5% Pd-C catalyst (600 mg.). After an uptake of approx. 0.7 mole further consumption of hydrogen ceased. The catalyst was removed by filtration and the residue was distilled to give 4.8 g. of colorless oil (96%).

Vpc examination of the above material in conjunction with IR and NMR spectroscopy indicated it to be a 70:30 mixture of cyclononanol and cyclononanone.

Cyclononanol. The above material (2.1 g.) in dry ether was reduced with an excess of lithium aluminum hydride. Usual work-up followed by distillation gave 2.0 g. (94%) of cyclononanol as a viscous colorless liquid, bp. 85-6°/2 mm., $n_D^{26}$ 1.4914.

Cyclononanone. The mixture from hydrogenation of cyclonon-2-en-1-ol (2 g.) in acetone (10 ml.) was oxidized by Jones' reagent (0.7 M) and worked up routinely to furnish on distillation 1.68 g. (84%) of cyclononanone, bp. 78-9°/3 mm., $n_D^{26}$ 1.4773. A semicarbazone was prepared by standard procedure, mp. 177-79°, lit.[7] mp. 179.5-180.5°.

Cyclonon-2-en-1-one. Cyclonon-2-en-1-ol (1 g., 0.007 mole) in pyridine (3 ml.) was oxidized by Sarett's reagent[5] (prepared by adding chromium trioxide (430 mg.) to 3 ml. of pyridine) and left aside at room temperature for 24 hrs. under a nitrogen atmosphere. Dilution with 25 ml. of water and extraction with two 25 ml. portions of ether, followed by washing with 10% sodium bicarbonate and two 15 ml. portions of brine and drying, gave a pale yellow oil (690 mg.). Distillation yielded 617 mg. (61%) of colorless liquid, bp. 81-2°/3mm., $n_D^{26}$ 1.5029. Its IR spectrum had bands characteristic of α,β-unsaturated ketones at 1670 $cm^{-1}$. Its NMR spectrum showed two low field olefinic protons between τ 3.5 and 4.17 (multiplet).

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