PROPELLANES. PART LXXXIII*,[1]. SOLID STATE CONFORMATIONS OF SEVERAL SUBSTITUTED TRIAZA-[3.3.3]PROPELLANES AND OF AN OXADIAZA[3.3.3]PROPELLANE

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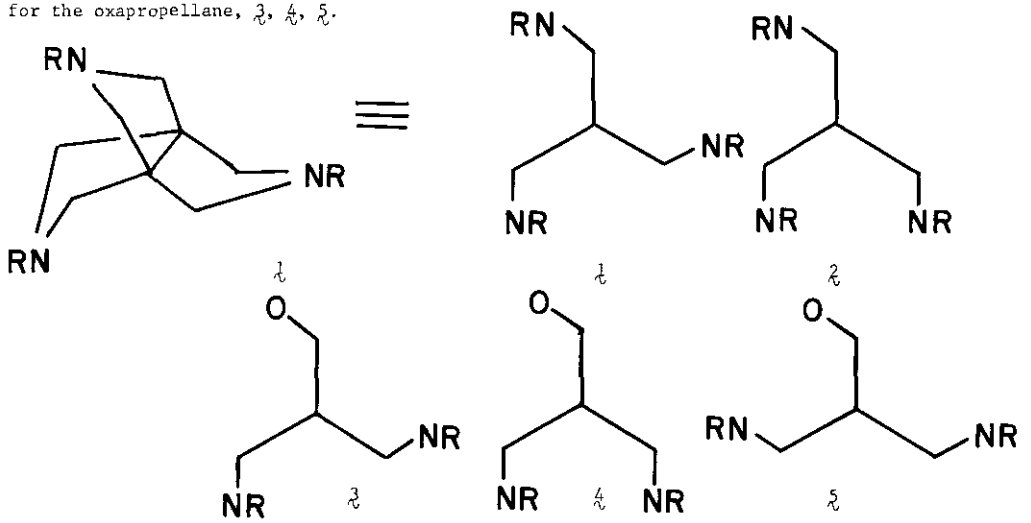
Abstract - All of the title compounds have their five-membered rings in envelope conformations with the flaps all tilted in the same direction.

The title compounds were prepared by reduction of compounds having higher oxidation states, either imide dilactams or dilactams, respectively[2]. We are studying their quaternary salts by NMR spectroscopy, after reaction with methyl iodide[3]. We wished to observe the solid state conformations of these amines and compare them to other propellanes which contain five-membered tetrahydrofuran[4] or thiophan rings[5]. On this basis we would expect envelope conformations for all of the rings in the title propellanes.

There are two structures possible for such envelope-containing triazapropellanes, 1 and 2 and three for the oxapropellane, 3, 4, 5.



When both R and R' are present in such a triazapropellane, one conformation must be added to 1 and 2.

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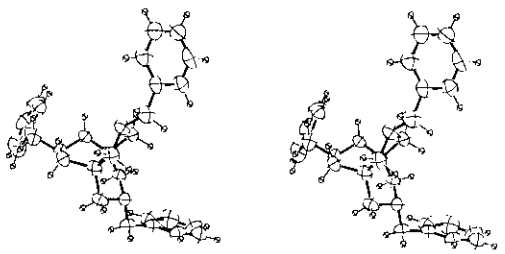
* Dedicated to Maître Professor Gilbert Stork on the occasion of his 65th birthday.

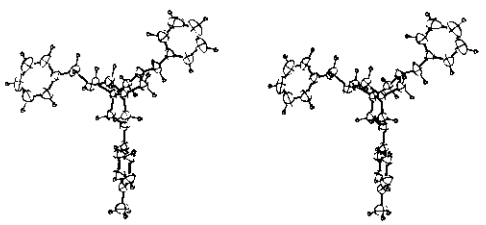
The cost in energy to flip any of these envelope flaps, e.g. $\underset{\sim}{1} \rightleftharpoons \underset{\sim}{2}$, $\underset{\sim}{3} \rightleftharpoons \underset{\sim}{4} \rightleftharpoons \underset{\sim}{5}$, is expected to be very low in solution but this does not mean that unique conformations may not be maintained in the crystal.

The ORTEP projections show that in tribenzyl-3,7,10-triaza[3.3.3]propellane and dibenzyl, p-tolyl-3,7,10-triaza[3.3.3]propellane each of the five-membered rings indeed has the envelope conformation with all flaps tilted in the same direction. Similarly the ether-diamine prefers conformation $\underset{\sim}{3}$ in the crystal. Thus, in all cases the steric interactions are minimized[6].

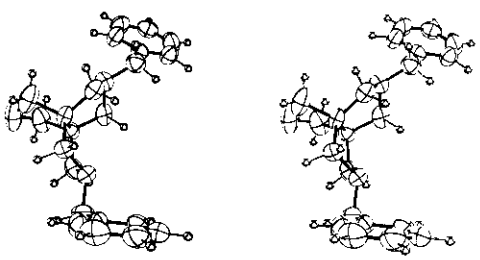
The parent 1, R=H, did not give suitable reflections at either room temperature or at low temperature.



ORTEP - Tribenzyl



ORTEP - Dibenzyl, p-tolyl



ORTEP - Oxa-dibenzyl

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