

04-Crystallography of Biological Small Molecules

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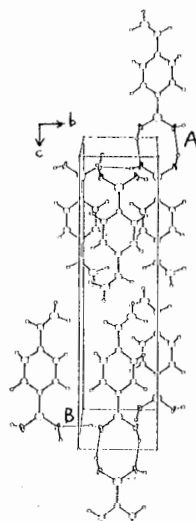
PS-04.05.03 THE CRYSTAL AND MOLECULAR STRUCTURE OF STYRYL BORONIC ACID: A POTENTIAL WOOD PRESERVATIVE. By G.J. Gainsford* and R.H. Meinhold, New Zealand Institute for Industrial Research and Development (Industrial Research Limited), PO Box 31-310, Lower Hutt, New Zealand

A range of organo-boron compounds have been synthesized. ^{11}B and ^{13}C solid state NMR have been used to study the compounds in their natural state and after impregnation into pinus radiata timber. The structure of styryl boronic acid has been determined to provide base information for its action and polymerization.

The title compound $\text{C}_9\text{H}_9\text{O}_2\text{B}$ crystallizes as transparent, extremely thin plates, ($\sim 0.01\text{-}0.03\text{mm}$) which are difficult to handle and form intergrowths. Use of polarized light and a microscope on a mounting table with television pickup assisted crystal selection, but even then, over eight crystals were mounted before a satisfactory diffraction profile was obtained. The crystals are monoclinic with a 19.36(2), b 5.128(6), c 8.159(6) Å and β 100.11(6)°, and space group $\text{P2}_1/\text{c}$. A total of 317 reflections were observed ($I(\text{net}) > 2.5\sigma(I(\text{net}))$) out of the 1322 reflections measured at low temperature to 2θ of 48° on a Nicolet R3m diffractometer. Final R_r , R_w values are both 0.055 for the 20 atoms and 99 parameter restricted refinement.

The structure (see Figure) consists of strongly hydrogen bonded (B-O-H...O) molecules with a 26 degree angle between the styryl and boronic acid (B(OH) $_2$) planes. Two types of hydrogen bonds are noted: end-on pairs across the crystallographic centres of symmetry (A), and between adjacent molecules up the short b axis (B). The unexpected nonplanarity of the molecule can be rationalised by the hydrogen bond spatial requirements and may play an important part in any solid state reactions, e.g. polymerisation, which is under investigation.

At the meeting, we will present the structural parameters, reports of continuing studies and any conclusions about viability for wood preservation and solid state reaction mechanisms. The assistance of the Forest Research Institute of New Zealand for the field trials, and the support of the Foundation for Research Science and Technology of New Zealand is gratefully acknowledged.



Figure

Selected molecules of styryl boronic acid in the crystal showing hydrogen bonding (see text)

PS-04.05.04 X-RAY STUDY OF A SATURATED METHYLENE-BRIDGED, IMIDAZO [2,1-a]ISQINDOLONE By A. Kapor^a, B. Ribár^a, G. Stájer^b, S. Frimpong-Manso^b, G. Bernáth^b and P. Engel^c, ^aInstitute of Physics, Faculty of Sciences, University of Novi Sad, Novi Sad, Yugoslavia, ^bInstitute of Pharmaceutical Chemistry, Albert Szent-Györgyi Medical University, Szeged, POB 121, Hungary, ^cLaboratory for Chemical and Mineralogical Crystallography, University of Berne, Switzerland.

In a continuation of studies on saturated condensed isoindolones, phenyl-substituted derivatives containing a norbornane moiety instead of a cyclohexane ring have now been synthesized with a view to preparing compounds with anorexic activity. The reaction of 3-endo-benzoyl-6-exo-phenylbicyclo [2.2.1] heptane-2-carboxylic acid and ethylenediamine in toluene yields 6,9-methano-7,9b-diphenyl-1,2,3,5a,6,7,8,9,9a,9b-5H-decahydro-imidazo [2,1-a] isoindol-5-one $\text{C}_{23}\text{H}_{24}\text{N}_2\text{O}$. The crystals belong to the monoclinic space group $\text{P2}_1/\text{n}$, $a=7.657(4)$ Å, $b=23.005(9)$ Å, $c=10.267(5)$ Å, $\beta=94.52(2)^\circ$, $V=1802.9(15)$ Å 3 , $Z=4$, $D_x=1.27$ Mg m $^{-3}$, $\lambda(\text{MoK}\alpha)$, $\mu=0.07$ mm $^{-1}$, $F(000)=736$, $T=293$ K. Structure was solved by direct methods using SHELXS-86. Atom parameters were refined by the least-square fit (SHELX-76) up to a final R-factor of $R=0.061$ ($N_{\text{ref}}=2747$, $n_p=292$, $w=1$, $S=0.89$). Hydrogen atoms were isotropically refined except the phenyl hydrogens which were generated from assumed geometry. Fig. 1 shows a perspective view of the compound. Analysis of the geometry indicates diendo annellation of the norbornane moiety and the pyrrolidine ring ($\text{H5a-C5a-C9a-H9a}=5.2(23)^\circ$). The phenyl group on the carbobicyclic ring is 7-exo ($\text{C10-C6-C7-C18-97(2)^\circ}$, $\text{C9-C8-C7-C18}=-130.6(2)^\circ$), while the 9b-phenyl group and the exo H-5a,9a at the annellation of the norbornane and pyrrolidine are trans relative to the five-membered ring ($\text{H9-C9a-C9b-C12}=102.4(14)^\circ$). Conformational analysis of the five-membered N1-C2-C3-N4-C10 and pyrrolidine ring N4-C5-C6-C9A-C9B from the values of asymmetry factors [$f_{\text{C5}}=0.022(6)$ Å, (C9B), $f_{\text{C2}}=0.015(9)$ Å (C5A)] (Kálman, A., Czugler, M. & Simon, K., (1982) Molecular Structure and

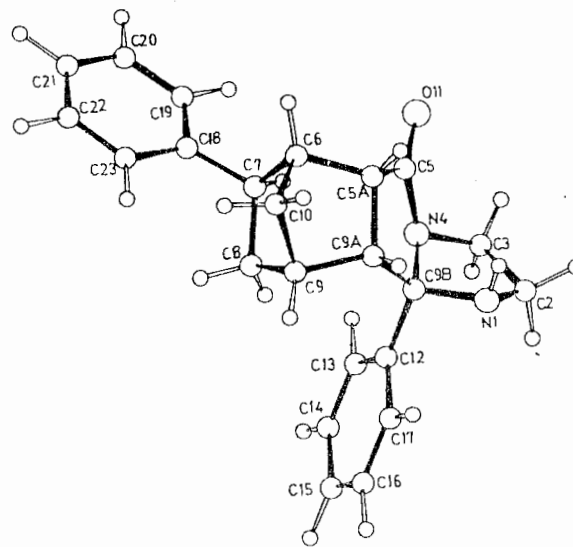


Fig. 1.

Biological Activity, ed. J.F. Griffin & W.L. Duax, pp. 367-376, New York, Elsevier Biomedical) shows that it adopts envelope and half-chair conformations, respectively. The location of the 9b-phenyl group is stabilized by two short intramolecular contacts $\text{C13-H13}\dots\text{N4}$ ($2.847(3)$ Å, $98.7(1)^\circ$, and $\text{C17-H17}\dots\text{N1}$ ($2.927(4)$ Å, $81.8(18)^\circ$).