09.2-42  THE CRYSTAL STRUCTURES AND ABSOLUTE CONFIGURATIONS OF FOUR 1,3,2-OXAZAPHOSPHOLIDINES DERIVED FROM 1-PHPHEDRINE. By T.J. Barkczech and Z. Galdecki, Institute of General Chemistry Technical University, 90-924 Łódź and (in part) M. Rutkowska, Regional Laboratory of Physicochemical Analysis and Structural Research, Jagiellonian University, 30-46 Kraków, Poland and (in part) Yu.T. Struchkov and N.Y. Antipin, Institute of Organocement Compounds, USRR Academy of Sciences, Moscow 117312, USSR.

The diasterocisomers (2S,4S,5R)- and (2R,4S,5R)-2-chloro-3,4-dimethyl-5-phenyl-1,3,2-oxazaphospholidine-2-thiones (A and B) were synthesized from (α)-ephedrine i.e. 1R,2S-2-methylthiomethyl-1-phenylpropan-1-ol (Inch and al., J. Chem. Soc., Perkin 1 1977, 1969-1980). The 1H NMR spectra for A and B were not easy to interpret and the configuration at the P atom was assigned as Ps for A and Po for B on the basis of chemical analysis. The title compound is a condensation product of 1,3-diaminopropane and the methylester of 2-amino-1-cyclopentene-1-dithiocarboxylic acid (Nordas et al., J. Org. Chem. (1972) 37, 1727). The compound crystallized in the space group P2₁/n, with the following cell dimensions: a = 6.802(3), b = 24.242(14), c = 11.953(4) Å, α = 100.90(3)°, β = 90°. Altogether, 39% reflections were collected on the single crystal diffractometer (CAD-4) of which 1607 with F > 4σ(F) were considered for the final refinement which gave R = 0.08. Using MULTAN-78, the structure was solved for all the non-hydrogen and 24 hydrogen atoms.

09.2-43  N,N'-DI(2-METHYLDITHIOCARBONYL-1-CYCLOPENTENYL)-1,3-DIAMINOPROPANE, C₈H₁₄N₂S₄. By P.B. Sarkar and S.P. Sen Gupta, Department of General Physics and X-rays, Indian Association for the Cultivation of Science, Calcutta - 700032, India.

The title compound is a condensation product of 1,3-diaminopropane and the methylester of 2-amino-1-cyclopentene-1-dithiocarboxylic acid (Bordas et al., J. Org. Chem. (1972) 37, 1727). The compound crystallized in the space group P2₁/n, with the following cell dimensions: a = 6.802(3), b = 24.242(14), c = 11.953(4) Å, α = 100.90(3)°, β = 90°. Altogether, 39% reflections were collected on the single crystal diffractometer (CAD-4) of which 1607 with F > 4σ(F) were considered for the final refinement which gave R = 0.08. Using MULTAN-78, the structure was solved for all the non-hydrogen and 24 hydrogen atoms.
These thiazetidine rings are planar and more symmetrical than other published thiazetidine rings because both carbon atoms of the ring have external double bonds (C-S: 1.81-1.88 Å and C-N: 1.37-1.40 Å).

The chemistry of diterpenoids occurring in the genus Teucrium, is described in a recent general review (Piozzi, Heterocycles. (1981) in press). The structures of ten of these compounds shown below have been determined by X-ray diffraction. The purpose of this study is to classify these compounds according to the conformation of their rings. Special attention has been devoted to the decaline group. Parameters definition: for this study Cremer's parameters have been used (Cremer and Pople, J.Am.Chem.Soc. (1975) 97(6), 1354-58), $\theta(%)$, $\gamma(%)$ and $\Delta(\AA)$. Chairs occur for $\theta=0,180^\circ$, and boat or twist for $\theta=90^\circ$. $\theta$ describes the boat form ($\theta=0,60,120...$) and twist form ($\theta=30,90,150...$), and $\Delta$ the puckering. The table shows the experimental X-ray values for the decaline moieties. $\gamma$ is the internal torsion C1-C10-C5-C4 and $\gamma$ is C6-C5-C10-C9. It is observed that $\gamma$ is nearly constant: $108^\circ$ for sp3-sp3 bonds and $53^\circ$ for sp2-sp3 bonds. The $108^\circ$ value is diminished by the CS-C9 axial interaction and the oxo substituted C6 in compounds I, IV and VI. For saturated cyclohexane rings $\gamma$ increases with $\theta$ to reach a maximum at $\theta=90^\circ$ and decreases again towards $\theta=180^\circ$. Other interesting fact is the lower stability of these K-salts versus the Na-salts (C.Esteban-Calderón, M. Martínez-Ripoll, to be published elsewhere). The free compounds behave as sequestering agents for Na ions.