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The unit-cell dimensions and space groups of nickel(II), copper(II), and palladium(II) salicylimines.

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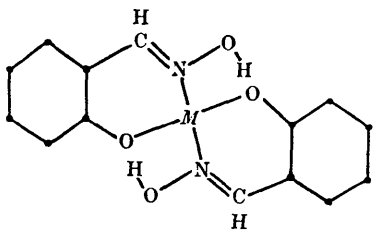
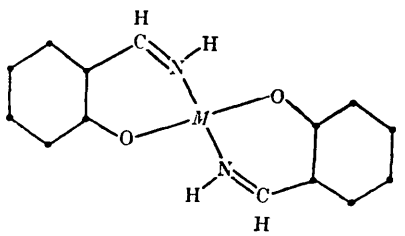
Comparison of the structures of nickel(II) salicylaldoxime (Merritt, Guare & Lessor, 1956) and palladium(II) salicylaldoxime (currently being investigated in this laboratory) indicates slight differences in some of the bond lengths which are probably significant. Also, infrared spectra of these two compounds, obtained by the potassium bromide pellet technique, show a greater shift in the –OH– stretching frequency in nickel salicylaldoxime than in palladium salicylaldoxime, indicating a difference in hydrogen bonding. Copper(II) salicylaldoxime, although belonging to the same space group ($P2_1/c$, Cox & Webster, 1935) as the above compounds, contains four molecules per unit cell, whereas the nickel and palladium compounds contain two molecules per unit cell.

In order to investigate further the differences in nickel(II), copper(II), and palladium(II) complexes, a series of compounds similar to the salicylaldoximes, nickel(II), copper(II), and palladium(II) salicylimines, has been prepared.



M(II) Salicylaldoxime



M(II) Salicylimine

Single crystals were grown by saturating butyrolactone at 80° C. and allowing the solution to cool slowly to room temperature. The unit-cell dimensions were measured from rotation, Weissenberg, and precession photographs, using Cu $K\alpha$ radiation; and the space groups were determined uniquely by the systematic extinctions. Densities were measured by flotation. The results are given in Table 1.

Table 1. *Unit-cell dimensions and space groups of nickel*(II), *copper*(II), *and palladium*(II) *salicylimines*

	Nickel(II) salicylimine	Copper(II) salicylimine*	Palladium(II) salicylimine
a_0 (Å)	8·12	8·09	8·08
b_0 (Å)	5·85	5·87	5·82
c_0 (Å)	14·60	14·75	14·69
β	118° 00′	118° 27′	117° 30′
Space group	$P2_1/a$	$P2_1/a$	$P2_1/a$
$D_{meas.}$ (g.cm.$^{-3}$)	1·616	1·630	1·882
$D_{X\text{-}ray}$ (g.cm.$^{-3}$)	1·622	1·639	1·880
N (molecules/unit cell):	2	2	2

* Stackelberg (1947) reported copper salicylimine to be monoclinic, space group $P2_1/c$, with:

$$a_0 = 12{\cdot}94 \pm 0{\cdot}10,\ b_0 = 5{\cdot}84 \pm 0{\cdot}05,\ c_0 = 8{\cdot}05 \pm 0{\cdot}05\ \text{Å};$$
$$\beta = 85{\cdot}7°;\ D = 1{\cdot}65 \pm 0{\cdot}04\ \text{g.cm.}^{-3}.$$

We have chosen a different unit cell as preferable for projection of the molecule in the *ac* plane, and for comparison with nickel, copper, and palladium salicylaldoximes. Stackelberg's *c* is the new *a*, and his *a* is the new [$\bar{1}$01]. Calculating from the new cell, [$\bar{1}$01] = 13·01 Å, and the angle between *a* and [$\bar{1}$01] is 85° 19′, agreeing well with Stackelberg's values.

The symmetry and two molecules per unit cell require the metal atoms to occupy centers of symmetry.

Integrated intensity data of the (*h*0*l*) zone have been collected for nickel salicylimine, and an *ac* projection has been calculated. The agreement index, *R*, at this stage is 18%. However, the tilt of the molecule is so great that all atoms in the benzene ring are not clearly resolved, and other projections must be made. Intensity data are being collected for other zones of nickel salicylimine, and for the copper and palladium salicylimines. The nickel(II) salicylimine structure is being refined by least squares, and full details will be reported at a later date.

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References

COX, E. G. & WEBSTER, K. C. (1935). *J. Chem. Soc.* p. 731.

MERRITT, L. L., JR., GUARE, C. & LESSOR, A. E. (1956). *Acta Cryst.* **9**, 253.

STACKELBERG, M. (1947). *Z. anorg. Chem.* **253**, 136.