

## NOTES

**Synthesis and Characterisation of Fe(II) and Co(II) Complexes of 1-Azepinyl-3-(p-Chlorophenyl-sulphonyl)-Urea**

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Complexes of glypinamide, 1-azepinyl-3-(p-chlorophenyl sulphonyl) urea, (parinase) with iron and cobalt were isolated with the composition  $(C_{13}H_{17}ClN_3O_3S)_2M \cdot 2H_2O$  ( $M=Fe$  or  $Co$ ). Stability constants are calculated as 13.1010 and 12.0390 litres/mole and free energy changes are 17.6858 and 16.2527 Kcal./mole respectively for Fe and Co complexes.

Sulphonylureas have been intensively used as oral hypoglycemic agents in the treatment of *Diabetismellitus*. Recently, Yoshinaga and Yamamoto<sup>1,2</sup> have been synthesised Zn(II), Cd(II) and Hg(II) complexes with few sulphonyl ureas. Among transition metals iron and cobalt have special chemotherapeutic value, we describe Fe(II) & Co(II) complexes of glypinamide.

Pure glypinamide m.p 470°K (lit 471.5°K) 0.005M, while ferrous sulphate E.P. and cobaltous chloride A.R. both 0.01 M. were prepared in 80% ethanol. Conductivity measurements of (i) Ferrous sulphate versus glypinamide and (ii) cobaltous chloride versus glypinamide were carried out in 80% ethanol, applying monovariation method at 303°K using Toshniwal conductivity bridge and dip type electrodes. 20 ml. of ligand was diluted to 200 ml and titrated against metal solution. After making volume correction and plotting a graph, results show ligand metal ratio as 2 : 1 in the complexes. Ligand metal ratio were further verified by Job's<sup>3,4</sup> method of continuous variation as modified by Turner and Anderson<sup>5</sup>; results are recorded in Table 1.

(a) Glypinamide as its sodium salt, 2 mole (0.8833.g) and ferrous sulphate 1 mole (0.3473 g.) separately dissolved in methanol. One drop of  $H_2SO_4$  was added to ferrous sulphate solution to stabilise the ferrous ion. Ligand solution was added slowly to ferrous sulphate with constant stirring; at first no change was observed but later on solution becomes turbid. On cooling for several hours chinese gold, crystalline complex was separated which was filtered, washed with methanol and dried; yield 54.9%, M.pt. 496°K.

(b) Glypinamide 0.8283 g. and cobaltous chloride 0.2974 g. separately dissolved in minimum quantities of absolute alcohol; ligand solution gradually added to metal salt solution; at once pink precipitate was obtained which on refluxing for several hours and then on cooling gives

TABLE I  
ANALYTICAL DATA, STABILITY CONSTANTS AND COMPOSITION OF  
THE COMPLEXES

Composition	$(C_{13}H_{17}ClN_3O_3S)_2Co \cdot 2H_2O$	$(C_{13}H_{17}ClN_3O_3S)_2Fe \cdot 2H_2O$
Colour/M.P.	Chinese gold (496)	Pink (478)
% Analysis Found/Calcd.		
Fe/Co	7.56 (7.41)	7.50 (7.84)
N	10.75 (11.14)	11.00 (11.10)
S	9.20 (8.49)	8.39 (8.45)
Cl	9.56 (9.42)	9.40 (9.38)
Water	4.80 (4.78)	4.75 (4.75)
Stability constant (lts./mole)	13.1010	12.0396
$-\Delta F$ Free energy change (kcal./mole)	17.6856	16.2527

a pink crystalline precipitate which was filtered washed with ethanol dried and weighed, yield 57.29% M.pt. 478°K.

Iron was estimated in complex by cupferon<sup>6</sup> as oxide while cobalt as tetrapyridine cobalt dithinate<sup>6</sup>, after removing the base. Nitrogen was estimated by modified Kjeldhal's method, sulphur and water were estimated by usual methods. Analytical results are given in Table 1.

The conductivity measurements and Job's method of continuous variation indicate the formation of 2 : 1 complexes between ligand and metal. Analytical data shows that the complexes may be formulated as  $(C_{13}H_{17}ClN_3O_3S)_2M \cdot 2H_2O$  where M is Fe or Co. The complexes are nonhygroscopic, coloured, and insoluble in water and in common organic solvents.

The IR spectra of complexes were recorded on Perkin Elmer Model 237 spectrophotometer, using KBr disc. between 4000–650  $cm^{-1}$ . The IR spectral bands obtained at 675  $cm^{-1}$  and 680  $cm^{-1}$  for Fe–O and Co–O bonds respectively. A frequency at 1660  $\pm$  10  $cm^{-1}$  in ligand which is modified into 1660  $\pm$  20 and 1650  $\pm$  10  $cm^{-1}$  for Fe and Co complexes may be assigned to the C–O linkage. A stretching frequency at 3280  $\pm$  20  $cm^{-1}$  in Fe and 3120  $\pm$  10  $cm^{-1}$  in Co are characteristic of  $\text{>NH}$  frequency while at 2350  $\pm$  10  $cm^{-1}$  and 2340  $\pm$  5  $cm^{-1}$  is an indication of

$\text{>C=NR}$  linkage co-ordination water in these complexes is indicated by the absorption bands obtained at  $3380 \pm 20 \text{ cm}^{-1}$  and  $3900 \pm 5 \text{ cm}^{-1}$ .<sup>7-9</sup>

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