

CONCLUSIONS

From the analysis of data of different temperatures and concentrations one can choose the etching conditions such that the:

1. Etched tracks with well defined contour are formed.
2. Etching time is within the range of convenience of measurement.

The linear relationships between diameter and etching time give the following best etching conditions:

Detector	Etchant	Concentration	Temperature	Time
1. Lexan	NaOH	6N	65°C	15 min.
2. Glass	HF	20%	35°C	15 sec.

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February 14, 1976.

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* The work was financially supported by PL-480 NBS (G)-182 research grant.

† The source was purchased from Oak Ridge National Laboratory, U.S.A. and consisted of 1/2" diameter electrodeposit of 0.1 μ gm Californium oxalate on 1" platinum disc.

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IRON (III) THIOCYANATE COMPLEXES WITH SOME SCHIFF BASES

THE chloro and the bromo complexes of iron (III) with ethylenediamine-salicylaldehyde are reported to be dimeric¹. A few cyanide complexes of iron (III) with Schiff bases showing interesting magnetic behaviour have been reported recently². We present here the results of our studies on iron (III) thiocyanate complexes of the Schiff bases derived from salicylaldehyde and aniline (salanil), ethylenediamine and 2-hydroxy acetophenone (aphen) or 2-hydroxy 5-methyl acetophenone

(maphen), and glyoxal and *o*-aminophenol (glyoxanil).

2-OH-acetophenone and 2-OH, 5-CH₃-acetophenone were prepared by known methods³. The Schiff bases were prepared by condensing the respective aldehyde or ketone with the amines in ethanol. The ligands separated were filtered, washed with ethanol and recrystallised from benzene. An aqueous solution of iron (III) thiocyanate was prepared by mixing 30 ml of FeCl₃·6H₂O solution (10%) with 70 ml KCNS solution (10%). The deep red iron (III) thiocyanate was extracted into an ether layer (75 ml). The ether layer separated was then mixed with aphen or maphen in benzene (1:1). The salanil and glyoxal bis (2-hydroxy anil) complexes were prepared by refluxing with iron (III) thiocyanate solution in ethanol and methanol respectively (~4 hrs.). The pH was adjusted to 5 using HAc-NaAc buffer. The dark brown complexes separated were filtered, washed with water and benzene, then with ether and dried in vacuum over P₂O₅.

The analytical data, conductance values, magnetic moments and important infrared frequencies are summarised in Table I. The electrical conductance data of the complexes indicate that they behave as non-electrolytes in nitrobenzene. The decrease in the magnetic moment values from the spin-only value found in the thiocyanate complexes are quite comparable to that of the chloro complexes of ensal⁴. This is due to considerable interaction between iron atoms.

The complexes have been further characterised on the basis of their infrared spectra (KBr and Nujol mull). The broad, medium intensity band observed around 3000 cm⁻¹ in the spectra of the free ligands is assignable to the hydrogen bonded-OH group. In the spectra of the aphen and maphen complexes, a broad band appears at 3450 cm⁻¹ showing the presence of free hydroxyl group. Thus, the infrared spectra of the complexes Fe (aphen) (SCN)₂, Fe (maphen) (SCN)₂, along with the analytical data indicate that one of the hydrogens of the hydroxyl groups of the free ligand is replaced by the metal while the other remains free. This type of behaviour is not uncommon in the case of Schiff base complexes⁵. In the case of the other two complexes, Fe (salanil) SCN and Fe (glyoxalanil) SCN, the -OH group of the free ligand disappears on complexation showing that both the hydrogens are replaced by the metal. The free ligand show medium intensity band around 1500 cm⁻¹ which is characteristic of the C=N group. This band is shifted to around 1540 cm⁻¹ indicating the participation of C=N group in complexes. A strong, sharp characteristic absorption at 2090 and 2120 cm⁻¹ observed in the thiocyanate complexes, but absent in the free ligands can be assigned to the thiocyanate group coordinated through nitrogen⁶.

TABLE I
Analytical and physical data

Complex	Colour	Fe %		S %		μ_{eff} B.M	Electrical conduc- tance ohm ⁻¹ cm ² mole ⁻¹ (nitro- benzene)	IR frequen- cies and their assign- ments Cm ⁻¹	Assign- ments
		Found	Calcd.	Found	Calcd.				
Aphen	Yellow	2900 bm 1510 m	νOH $\nu\text{C}=\text{N}$
Fe (aphen) (SCN) ₂	Dark brown	11.87	11.96	13.41	13.70	5.37	3.7	3450 m 1540 m 880 m	νOH $\nu\text{C}=\text{N}$ $\nu\text{C}-\text{S}$
Maphen	Yellow	2950 bm 1500 m	νOH $\nu\text{C}=\text{N}$
Fe (maphen) (SCN) ₂	Dark brown	10.95	11.28	12.28	12.93	5.42	9.2	3450 m 1520 m 830 m	νOH $\nu\text{C}=\text{N}$ $\nu\text{C}-\text{S}$
Salanil	Yellow	3000 bm 1460 m	νOH $\nu\text{C}=\text{N}$
Fe (salanil) ₂ SCN	Dark brown	11.18	11.04	6.96	6.32	5.20	3.7	1480 m 860 m	$\nu\text{C}=\text{N}$ $\nu\text{C}-\text{S}$
Gly. anil	Colourless	3450 m 1500 m	νOH $\nu\text{C}=\text{N}$
Fe (glyanil) SCN	Dark brown	16.14	15.87	10.30	9.09	3.93	0.6	1520 m 860 m	$\nu\text{C}=\text{N}$ $\nu\text{C}-\text{S}$

The authors wish to thank the Propellant Engineering Division of the Space Science and Technology Centre, Thumba, Trivandrum, for the IR spectra. One of the authors (ANS) thanks UGC for financial assistance. Department of Chemistry, University of Kerala, Trivandrum 695001, November 22, 1975.

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ANALYTICAL APPLICATIONS OF 6-AMINO-5-NITROSO-2, 4-PYRIMIDINEDIOL

2-AMINO-5-nitroso-4, 6-pyrimidinediol has been used earlier¹ as reagent for Fe²⁺, Co²⁺ and Ru³⁺. To investigate the effect of substituents in the molecule, the complexation reactions of the ligand with groups interchanged at C₂ and C₆ compared to its precursor are now reported. Moreover, the present compound, viz., 6-amino-5-nitroso-2, 4-pyrimidinediol (monosodium salt) is more water-soluble.

The aqueous solution of the ligand is light red in colour absorbing maximum at 225 and 315 nm. The characteristics of its metal complexes are summarised in Table I.

Procedure.—To a suitable aliquot of metal solution (Fe²⁺, Co²⁺ or Ru³⁺), sufficient excess of the diol (0.008 M) is added followed by 5.0 ml of acetate buffer (pH 5.0). In the case of Ru³⁺, 0.1 M NaClO₄ strength is also maintained and the solution heated for 45 minutes on a steam bath. After making up the volume, the absorbance is measured at λ_{max} of the complex. The amount of the metal is calculated by comparing the absorbance with a calibrated curve.

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