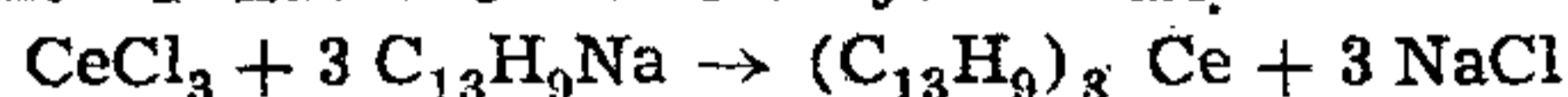


SHORT SCIENTIFIC NOTES

Preparation and Properties of Trifluorenyl Cerium (III).

Fluorenyl derivatives of titanium, zirconium¹, silicon², germanium³, tin⁴ and lead⁵ have been reported by various workers. This note deals with the preparation of trifluorenyl cerium by the reaction of cerium trichloride with sodium salt of fluorene in tetrahydrofuran.



Small pieces of sodium (0.37 g; 0.016 mole) were added to fluorene (2.66 g; 0.016 mole) in tetrahydrofuran (75 ml). The mixture was stirred vigorously for 12–13 hr. at room temperature under nitrogen. The mixture was gradually added with constant stirring to a suspension of cerium trichloride (1.23 g; 0.005 mole) in tetrahydrofuran (100 ml). Stirring was continued for 3–4 hr. at room temperature (31.5° C). The solution was filtered and the residue was washed with tetrahydrofuran. The filtrate along with the washings was evaporated under reduced pressure and the bright yellow crystalline compound thus obtained was found to have the composition $(\text{C}_{13}\text{H}_9)_3\text{Ce}$. The yield was nearly 72% (Found: C, 73.4; H, 4.1; Ce, 21.8; $\text{C}_{39}\text{H}_{27}\text{Ce}$ Calcd.: C, 73.7; H, 4.3; Ce, 22.0%).

The compound is bright yellow in colour, stable in dry and inert atmosphere and melts with decomposition at 98° C. It is immiscible with water and is fairly soluble in common organic solvents like carbon tetrachloride, benzene, cyclohexane, tetrahydrofuran, acetone, dioxane, dimethyl formamide and alcohol.

The infrared spectrum in KBr shows the following absorption peaks: 3000 s, 1710 m, 1650 m, 1550–1530 vw, 1450 vs, 1395 m, 1295 m, 1220 vw, 1180 m, 1150 m, 1085 m, 1020 m, 1000 m, 950 m, 915 w, 860 m, 735 vs, 692 m cm^{-1} .

The frequencies at 3000, 1450, 1150, 1020 and 860 cm^{-1} belong to the C_5H_5^- group⁶, those at 1395, 1650 and 735 cm^{-1} to the phenyl groups and the one at 692 cm^{-1} to methylene.

The authors are thankful to Prof. T. R. Seshadri, F.R.S., for helpful discussions and to C.S.I.R. (India) for the award of a Fellowship to B. L. K.

Chemistry Department,
Delhi University,
Delhi-7, January 8, 1972.

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Spectrophotometric Study of Uranium (VI) Complex with 1-(2-Pyridylazo)-2 Phenanthrol (PAP).

1-(2-Pyridylazo)-2 phenanthrol (PAP)¹ was prepared and its solution was made in methanol. Solution of uranium (VI) was prepared by dissolving uranyl nitrate (E. Merck, G.R.) crystals in double distilled water. All other chemicals used were of reagent grade.

A Unicam, SP-600, spectrophotometer was used for absorbance measurements and a Metrohm pH meter, E 350, for measuring pH's.

The pinkish-red water-insoluble complex formed as a result of interaction of U(VI) with PAP is soluble in 70% methanol and shows maximum absorbance at 550 nm. The complex was found to be stable for 24 hours, after which measurements were discontinued. The absorbance of the complex, measured at 550 nm, remains constant in the pH range 6.5–8.5. Below and above this pH range, the absorbance falls. The system obeys Beer's law up to 8.4 ppm of uranium. The optimum concentration range for the determination of uranium, as found from Ringbom plot is 1.6 to 7.5 ppm and the sensitivity of the reaction is 0.0082 $\mu\text{g U(VI) cm}^{-2}$ for 0.001 absorbance. The molar absorptivity is 30,000. The mole ratio and Job's method of continuous variations suggest that 1 : 2 U : PAP) complex is formed in the system.

The amounts of foreign ions (in ppm) tolerated in estimation of 5.95 ppm of uranium at pH 7.0 are: Cl^- (500), Br^- (500), I^- (500), NO_3^- (500), CNS^- (40), thiourea (1000), SO_4^{2-} (39), NO_2^- (20), Th(IV) (50), Mn^{2+} (100), Ca^{2+} (400), Cr(III) (100). However oxalate, EDTA, F^- , PO_4^{3-} , CH_3COO^-

and other cations were found to interfere seriously. Mercury (II) (10 ppm) could be masked with iodide.

Procedure.—To a suitable aliquot of solution containing between 16–75 μg of uranium (VI) and ten times molar excess of the reagent in methanol is added. The pH is adjusted between 6.5–8.5 using borax-boric acid buffer. The total volume is made to 10 ml, keeping 70% methanol concentration. The absorbance is measured at 550 nm against the corresponding reagent blank and amount of uranium is deduced from the standard calibration curve.

The authors are thankful to the Council of Scientific and Industrial Research, India, for the award of a Research Fellowship to one of them (A. K. R.).

Chemistry Department,
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A Note on the Occurrence of a Spontaneous Tetraploid from a Hexaploid in Cotton

In a programme to establish a number of seed progenies from one of the hexaploids of cotton synthesised for breeding purposes, selfed seeds were sown periodically during the year 1968. The hexaploid ($2n = 78$) containing the genomes of a *Gossypium hirsutum* cultivar Co₂ (AD₁) and that of the wild American cotton *G. raimondii* (D₅) has a genomic constitution of $2[(AD)_1D_5]$. It was synthesized by colchicine-doubling a sterile triploid between *G. hirsutum* ($2n = 52$) and *G. raimondii* ($2n = 26$ —male parent). The hexaploid plant is tall (93 cm), vigorous, and flowers profusely. It is only partly fertile (37.2% pollen fertility) probably due to the formation of two to four multivalents per cell between the D-sub-genome chromosomes of the *hirsutum* parent and the D₅ *raimondii* chromosomes. On selfing, it produces usually about 4–10 seeds per boll. The leaves are coarse, palmate and highly pubescent. The flowers are big and there is a full-red petal spot and highly pigmented filaments, as evidence of the presence of the dominant *raimondii* gene for floral pigmentation.

In one instance, six plants were established from the selfed seeds of the hexaploid. Five of them resembled their parent and proved to be hexaploids, while the sixth one was different. This plant was stunted (49 cm), less

vigorous, less hairy with small sized leaves and flowers and resembled more or less the original *hirsutum* parent involved in the cross. However, it differed from the *hirsutum* parent in that its growth was much reduced and by the presence of a faint petal-spot and deeply pigmented filaments in the flowers denoting the presence of the *raimondii* gene. Upon examining the microsporocytes, it was observed to form 26 bivalents (with an occasional $2_1 + 25_{11}$), indicating that the somatic chromosome number was $2n = 52$, i.e., that of a tetraploid, instead of the expected $2n = 78$ of the hexaploid.

The occurrence of a spontaneous tetraploid from a higher polyploid is difficult to explain unless it is assumed that the entire *raimondii* genome has been probably eliminated during gametogenesis. This elimination, if assumed, must have occurred after gene-exchange for floral pigmentation had taken place between D₁ and D₅ chromosomes because the spontaneous tetraploid possesses the petal spot and the pigmented filaments which are completely absent in the original *hirsutum* parent and therefore must have been contributed by the *raimondii* genome alone present in the hexaploid.

The significance of the evolution of such fertile forms from semi-ferile higher ploidy level is not clear at present.

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Polarographic Determination of Metal Ions with Resacetophenoneoxime via Solvent Extraction

Resacetophenoneoxime was extensively used in these laboratories¹⁻⁷ and elsewhere as an analytical reagent for many metal ions. Further non-aqueous inorganic polarography is finding progressive application in the analysis of the organic phase in the solvent extraction.⁸ Hence the authors have undertaken the investigations of the applicability of the oxime for the polarographic determination of metal ions via solvent extraction with *n*-butanol. In the present communication we report the final results obtained in this regard.

An L-P.55 photographic recording polarograph with a dropping mercury cathode and mercury pool anode was used to record the polarograms. AnalaR (BDH) grade chemicals

TABLE I

Metal ion	pH of the aqueous phase	Concentration range $\mu\text{g/ml}$	Ions	
			Not interfered	Interfered
Cu (II)	5.0	15.88- 63.54	Ni, Pd, Mn, Co, Zn, Cr, Fe (in presence of NaF)	Fe (III)
Ni (II)	8.0	14.68-102.76	Cu, Mn, Cr, Fe (in presence of NaF)	Pd, Co, Zn
Pd (II)	0.02 M HCl	13.30- 79.80	Cu, Fe, Ni, Co, Mn, Zn, Cr,	—
Fe (III)	5.8	27.92-195.47	Pd, Co, Mn, Zn.	Cu, Ni
Co (II)*	8.0			
Mn (II)*	8.0			
U (VI)*	6.0			
Zn (II)**	0.02 M HCl-8.0			
Cr (III)**	do.			
Sn (II)**	do.			

* Formation of complex but no reduction wave.

** No complex formation.

were used in the studies. The oxime was prepared by the procedure described by Raju and Neelakantam¹.

The aqueous solution of the metal ion maintained at the specified pH with the suitable buffer solution was shaken in a separating funnel with a dilute solution of the oxime in *n*-butanol. After equilibration, a known volume of the organic phase was pipetted out into the polarographic cell and mixed with methanolic lithium chloride (0.1 M) solution. The solution was deaerated for about 20 minutes and the polarogram recorded.

Linear calibration curves were obtained in the concentration ranges mentioned against each metal ion in Table I.

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Changes in Free Amino Acid Content During Post-embryonic Development of *Leptocorisa varicornis* Fabricius (Hemiptera : Coreidae)

The review of literature by Chen¹ on amino acid metabolism in insect development indicates that the information is lacking for Hemipterous insects. Indira² studied the changes in the amino acid constituents of the eggs of *Sphaerodema molestum* (Duf.) during embryonic development. Since nothing is known about the changes in the amino acid content during post-embryonic development of any Hemipteroid insect, the different developmental stages of *Leptocorisa varicornis* have been analysed from this angle. The ears of paddy, *Oryza sativa* on which the insect feeds, have been also analysed to find out free amino acids available in its food.

Full grown nymphs and adults of *Leptocorisa varicornis*, commonly known as gundhy bug, were collected from paddy crop. The females readily laid eggs on paddy leaves in glass vials. Three days old eggs were used for the analysis. After washing 500 eggs, seven full grown nymphs, six adults and one ear of paddy were separately homogenised with 2 ml distilled water in an electric homogeniser at a speed of 15000 rpm. The homogenate of each material was centrifuged and supernatant liquid was retained. The proteins and fats were removed by the method described by Micks and Ellis.³ The determination of amino acids in the extracts of different developmental stages of the insect was carried

out by two-dimensional paper partition chromatography technique described earlier.*

In the egg of *Leptocorisa varicornis* fourteen amino acids namely, alanine, arginine, aspartic acid, glutamic acid, glycine, histidine, isoleucine, leucine, lysine, proline, serine, threonine, tyrosine, valine, and an unidentified ninhydrin positive compound have been detected. Except threonine all these compounds are present in its full grown nymph which also contains cystine and another undetermined compound. The amino acid contents of the adult and nymph are almost similar except that the former contains threonine in addition. This similarity is due to the fact that both of them feed on the same food.

The changes in the amino acid content of the different developmental stages, viz., egg, nymph and adult of *L. varicornis* are obviously related to the morphogenetic events during ontogeny. Metabolic changes of free amino acid during larval and pupal development have been also observed in several other insects including

Culex pipiens and *Drosophila melanogaster*¹. Cystine has probably appeared in the nymph and adult as a result of degradation of food proteins, since this compound has been detected in their midguts⁵.

The amino acids, alanine, arginine, aspartic acid, glycine, histidine, lysine, proline, serine, tryptophan and valine have been detected in the ears of paddy on which the insect feeds.

Research Entomologist, A. P. SAXENA.
U.P. Institute of Agricultural
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REVIEWS AND NOTICES OF BOOKS

Yield Point Phenomena in Metals and Alloys.

By E. O. Hall. (Plenum Publishing Corporation, 114, Fifth Avenue, New York, N.Y. 10011, U.S.A.), 1970. Pp: viii + 296, Price \$ 16.00.

Although yield point phenomena and the anomalies in the stress-strain curves of mild steel and many other metals and alloys have been known for a long time, and formed the subject of intensive experimental studies leading to a vast amount of observed data on the subject, yet it is only within recent years that meaningful advances on the theoretical side to explain the various aspects of the phenomena are forthcoming. Developments of new techniques like zone refining and electron beam melting for obtaining metals of high purity, as also developments in instrumentation have largely contributed to these rapid advances in this field. Any successful theory of the yield point will have to explain several features, such as the variation of yield stress with temperature, the effects of strain rate and the existence of delayed yielding, and the phenomena of serrated yielding which arises from strain ageing.

Professor E. O. Hall of the Department of Metallurgy, University of Newcastle, N. S. W.

Australia, reviews in this monograph the theories of yield point phenomena against the background of recent experimental results. In the first chapter the author outlines the general theories connected with yield point phenomena and illustrates them with appropriate examples. Naturally, the Cottrell-Bilby theory and its modifications and extensions are discussed in detail. The chapter is complete in itself and deals with the types of yield point effects, the upper and lower yield points, strain ageing, and pseudo yield points; illustrated with graphs, tables, and plates. This chapter could be read by undergraduates, and it provides the necessary background for understanding the contents of the other chapters which deal separately with particular groups of metals and alloys.

Thus, the second chapter is devoted to iron and its alloys, and the third chapter to the refractory metals of Group Va and VIa. The fourth chapter is concerned with the effects of hydrogen as an interstitial element, on the yield point and strain ageing. Aluminium and its alloys are dealt with in the fifth chapter, while in the sixth chapter are discussed other face-centred cubic metals and alloys like