

SHORT SCIENTIFIC NOTES

1-(2-Pyridylazo)-2-Phenanthrol (PAP) as a Visual Indicator for the Determination of Copper (II) with EDTA

1-(2-Pyridylazo)-2-phenanthrol (PAP), first prepared by Chiswell *et al.*¹ has been used as an analytical reagent for transition and platinum metals². This reagent has now been found to be a suitable indicator for the titration of copper with EDTA.

Copper forms a reddish-pink complex with PAP. The pH and temperature ranges found suitable for the titration are 3.0 to 7.2 and 20 to 60°C respectively. Below 20°C, the colour change takes place slowly and above 60°C, the colour changes before the end point is reached. Two drops of indicator solution (0.01 M) in dioxan are sufficient to cause a sharp change of colour almost instantaneously. Too large an amount of indicator results in dark colour and the visual observation of colour change at the end point becomes difficult.

The following foreign ions did not cause any interference, in the titration of 0.127 mg of copper present in 25 ml, when they are present in amounts given in mg in parentheses: Cl⁻ (500), PO₄³⁻ (100), oxalate (20), SO₃²⁻ (500), NO₃⁻ (500), Br⁻ (500), I⁻ (500), thiocyanate (500), Ca²⁺ (8), Mg²⁺ (10), Mn²⁺ (4), Ba²⁺ (10), Sr²⁺ (12). However Cd²⁺, Hg(II), Al(III), Co²⁺, Ni²⁺, Pd(II) and iron(II and III), citrate, tartrate, thiourea, thiosulphate cause interference.

The proposed reagent is better than 1-(2-pyridylazo)-2-naphthol³ where heating is necessary and is more sensitive. (Sensitivity = 0.0021 µg and 0.0034 µg for log I₀/I = 0.001.)

Recommended Procedure.—Pipette out 5 ml of 0.01 M EDTA solution into an Erlenmeyer flask, buffer to pH 4.5 with sodium acetate-acetic acid buffer and add two drops of indicator solution (0.01 M in dioxan). Titrate with copper solution taken in a microburette. Shaking is necessary after each addition of copper solution. A sharp colour change from yellow to violet indicates the end point.

Department of
Chemistry,
University of Delhi,
Delhi-7, January 3, 1972.

A. K. RISHI.
B. S. GARG.
R. P. SINGH.

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Metal Complexes of α -Nitroso- β -Naphthol

A survey of literature shows that not much work has been done on the metal complexes of α -nitroso- β -naphthol^{1,2}. Here we report the preparation and spectral study of Zn(II), Cd(II), Hg(II), Pb(II), Mn(II), and Pb(II) complexes.

Complexes were prepared in aqueous medium by reacting the respective metal salt with the ligand in a 1:2 ratio. There was an almost immediate formation of the complex on adjusting the pH between 2.0 and 6.0. After removing excess ligand by soxhlet extraction with petroleum ether, the product was dried over calcium chloride and analysed. The analytical data are given in Table I.

TABLE I

Complex	Per cent Metal		Per cent Nitrogen	
	Found	Calc.	Found	Calc.
(C ₁₀ H ₆ NC ₂) ₂ Zn ..	16.7	16.3	5.9	6.2
(C ₁₀ H ₆ NO ₂) ₂ Cd ..	24.1	24.6	6.0	6.2
(C ₁₀ H ₆ NO ₂) ₂ Hg ..	46.9	47.2	5.2	5.2
(C ₁₀ H ₆ NO ₂) ₂ Pb ..	47.5	47.3	5.0	5.1
(C ₁₀ H ₆ NO ₂) ₂ Mn ..	13.6	13.8	6.8	7.0
(C ₁₀ H ₆ NO ₂) ₂ Pd ..	23.3	23.6	6.1	6.3

Infrared spectra were recorded on Nujol mull. The main features of the spectra are as follows:

- (i) the spectra of the complexes do not show the characteristic band attributable to co-ordinated water in the region 3400 cm⁻¹;
- (ii) the band characteristic of intra-molecular hydrogen bonded OH which occurs at 3090 cm⁻¹ in the ligand is not observed in the spectra of complexes;
- (iii) it has been reported that the N=O stretching vibration occurs around 1596 cm⁻¹ in the case of β -nitroso- α -naphthol in alcohol³. We have found N=O band at 1626 cm⁻¹ in our ligand; but in each of our complexes there is a negative shift of the band indicating co-ordination of the ligand to the metal through oxygen.

* Masked with iodide (100 mg).

Department of Chemistry,
Karnatak University,
Dharwar, December 28, 1971.

S. V. PATIL,
J. R. RAJU.

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IR Study of Thiophene Adsorbed on Ultramarine

A comprehensive study of the structural and catalytic properties of ultramarines containing different amounts of sulphur has recently been made¹ using the techniques of X-ray diffraction and fluorescence analysis, radial electron density (r.e.d.) distribution and surface area measurements.

In the present note, an IR study of the adsorption of thiophene on an ultramarine sample of composition $(\text{SiO}_2)_{0.406} (\text{Al}_2\text{O}_3)_{0.233} (\text{Na}_2\text{O})_{0.218} \text{S}_{0.125}$ is reported. Thiophene at a partial pressure of 80 mm was adsorbed on a film of ultramarine (got by pressing the powder at 500 kg/cm²) at 25° C for 30 minutes. It was then desorbed for 30 minutes at 25, 100, 200 and 300° C, respectively and the IR spectra of the film was recorded (Beckman IR 12) in the region 400–4000 cm⁻¹. The IR cell designed by Uytterhoeven *et al.*² was used. The setting of the instrument was: slit 2.2 x standard slit; gain 7.7%; period 2; scanning speed 80 cm⁻¹/sec. The spectral resolution was about 3 cm⁻¹.

No C–H stretching frequencies (2800–3000 cm⁻¹) nor O–H frequencies were observed. On desorption at 100° C, a new peak at 2630 cm⁻¹ is observed which splits into two bands (2500 and 2650 cm⁻¹) at 200° C. A peak at 2690 cm⁻¹, characteristic of the S–H stretching vibrations in H₂S (2688 cm⁻¹) is also observed at 200° C. Adsorbed H₂S is thus present at 200° C. All the three peaks disappear at 300° C. S–H stretching vibrations on the surfaces of reduced GeS_x (x = 1.4) and MoS₂ have already been observed³ in the IR domain of 2500–2700 cm⁻¹. GeS_x exhibits a single S–H band at 2575 cm⁻¹ which progressively shifts to 2660 cm⁻¹ on heating to 200° C. In MoS₂ two bands are observed at 2640 and 2500 cm⁻¹ which decrease in intensity at increasing temperatures. Based on this analogy, the bands at 2630, 2650 and 2500 cm⁻¹ are assigned to S–H stretching vibrations. A striking parallel between the present study and the previous work³ is the behaviour of

the S–H band at 2630 cm⁻¹. On adsorbing an electron donor such as H₂O on reduced GeS_x at 100° C, the S–H band splits into a higher frequency component above 2700 cm⁻¹ and a low frequency component around 2500 cm⁻¹. The same observation was also made by adsorbing thiophene below 200° C. Hydrogen transfer between the surface and the adsorbed species was inferred to be at the origin of these vibrations. A similar phenomenon is probably the cause of the splitting observed at 200° C for the ultramarine sample also.

Laboratoire de Physico-
Chimie Minerale,
Institut des Sciences
de la Terre,
42, de Croylaan,
3030, Heverlee, Belgium,
November 1, 1971.

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Further Observations on *Eimeria intricata* Spiegl 1925

The oocysts of *Eimeria intricata* Spiegl, 1925, described from 7 hosts including domestic sheep and goat, have been studied by different workers and the various size-ranges have been compiled recently by Levine and Ivens⁵. A brief description of the oocysts from Indian sheep has been given by Bhatia and Pande². Information on its endogenous stages is meagre^{3,6,7}. Prepatent period is reported to be 20–27 days³, 23 days⁴ and 20–23 days⁹. A preliminary study was, therefore, made to know the typical shape and size of the oocysts and the prepatent period in Indian sheep.

During regular faecal examinations, a lamb showed once a large number of oocysts of *E. intricata* mixed with *E. arloingi*, *E. faurei* and *E. ninakohlyakimovi*. On morphological study of *E. intricata* oocysts, these exhibited a greater size-range with some difference in shape and structure including a few in abnormal form. After sporulation, the oocysts of *E. intricata* were isolated from the mixed culture following the technique adopted in *E. maxima* in poultry¹. Nearly 100 sporulated oocysts were fed to a clean lamb (4 months age) to study the time taken for the first and last discharge of the oocysts in the faeces,

The characteristic capped oocysts, with the outer thick scabrous brown and inner yellowish-brown layers, were spherical, sub-spherical, ovoid, ellipsoid and a few abnormal (4%) in shape. These measured (100 measured) $35-56 \mu \times 27-42 \mu$ in size. The polar cap was present as crescent to dome-shaped structure. It was absent in 14% of the oocysts examined. The length-width ratio (L/W) varied from 1.00-2.01 (i.e., 1.00-1.2 in 16% ; 1.21-1.3 in 19% ; 1.31-1.4 in 41% ; and 1.41-2.01 in 24% of the total number studied). These observations indicate that the typical shape is ellipsoidal and falls between $39-50 \mu$ (L) \times $29-37 \mu$ (W) size-range.

The lamb, infected with about 100 sporulated oocysts of *E. intricata*, voided the first oocyst on the 27th day post-infection. The number gradually increased and a peak reached on the 5th day after first discharge. Subsequently, there was gradual reduction in their number in the next 2 days and was abrupt in the following 2 days. Thus, the oocystic discharge continued for 9 days. The lamb, autopsied immediately when it became negative, was thoroughly searched for any endogenous stage in the intestinal lining. There was no stage of *E. intricata* except for a few small epithelial schizonts, gametocytes and oocysts in mucosa of caecum and colon which could be identified, on histological examination, as belonging to *E. faurei*. Possibly, the lamb picked up a light infection of the latter in the later stage of our observations.

The present study revealed some much smaller oocysts ($35 \mu \times 27 \mu$) which do not come within size-range given as smallest ($37-54 \mu \times 29-41 \mu$)². Davis and Bowman³ recorded the prepatent period in one case as 27 days with peak on the next day when the oocystic discharge also ended. In 4 other cases, they recorded the prepatent period as 20-23 days and the patent period as 6-11 days with peak reaching the same day in 3 cases and 3 days after in one case. Except for the peak of oocystic discharge reaching on the 5th day after their first appearance in faeces, the prepatent period (27 days) recorded in Indian sheep tallied with one of the 5 cases studied by Davis and Bowman³.

U.P. College of Veterinary Science
and Animal Husbandry,
Parasitological Unit,
Mathura, November 15, 1971,

B. B. BHATIA.
S. S. AHLUWALIA.
P. P. S. CHAUHAN.

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Frequency of PTC Taste Blindness Among Punjabis

The ability to taste an organic compound Phenyl-thiocarbamide (PTC) is inherited and is controlled by a pair of alleles. Tasters are dominant over non-tasters (tt). This taste polymorphism in human population has been studied by many workers and interracial variability has been observed. The frequency of tasters ranges from 63% in Arabs to about 98% among American Indians¹.

A survey was conducted to test the frequency of tasters and non-tasters and 1,440 persons were studied including 218 females. The frequency of tasters was found to be 43% while 57% persons were non-tasters. The observations are summarised below :

Sect.	No. of individuals studied		Percentage of		Allele frequency	
			Tasters	Non-tasters	T (p)	t (q)
Hindus	334	Obs.	41.01	58.99	0.23	0.77
		Exp.	40.71	59.29		
Non-Jat Sikhs	285	Obs.	44.92	55.08	0.25	0.75
		Exp.	43.75	56.25		
Jat Sikhs	536	Obs.	41.04	58.96	0.23	0.77
		Exp.	40.71	59.29		
Others	285	Obs.	47.37	52.63	0.27	0.72
		Exp.	46.71	53.29		
Pooled	1,440		43.06	56.94	0.24	0.76

The population comprised of three major caste groups in the State and the percentage of tasters and non-tasters was not significantly different in these groups. The gene frequencies for T and t alleles also did not differ significantly. Slightly higher percentage of tasters was obtained in the group classed 'others' but this was not significantly different from the three main groups. The observed number of

persons in tasters and non-tasters class was compared with the expected in each group. No significant differences, however, were observed indicating that for this character, the population is at equilibrium.

Basu and Ghosh² have studied the distribution of taste sensitivity genes in Indian population. They have reported that the frequency of *t* allele varied from 0.1 to 0.8, the largest number of samples falling in the classes 0.6-0.7 and 0.7-0.8. In the present study the frequency of this allele varied from 0.73 to 0.77 in the different groups studied. It seems, therefore, the caste groups in Punjab do not differ from each other in this respect, though they are expected to be genetically isolated from each other due to restricted inter-caste marriages. Such studies can be of use in tracing the ethnic origin of different sections of society.

Human Genetics Unit, J. S. DHESI.
Dept. of Genetics, A. K. GUPTA.
Punjab Agricultural University, R. G. SAINT.
Ludhiana, December 23, 1971.

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Post-Emergent Control of Dicot Weeds with Bromacil

Bromacil (5-bromo-3-sec-butyl-6-methyluracil) has been reported to be a non-selective herbicide, particularly in the control of broad-leaved annual and perennial weeds, but selective in crops such as pineapple and citrus⁴. It has been reported to be effective in the control of weeds such as *Cyperus rotundus*¹, *Panicum repens*³, *Ipomoea* spp., *Mimordica charantia*, *Leonotis nepetaefolia*, *Croton lobatus*, *Euphorbia* spp., and *Caperonia palustris*.²

An investigation was undertaken to eradicate certain dicot weeds growing in the gardens of the Department of Botany, Central College, Bangalore, by using Bromacil. Sufficiently replicated relevés of one metre diameter were marked to cover the serious dicot weeds of the area—*Lagasca mollis* Cav., *Galinsoga parviflora* Cav., *Euphorbia geniculata* Ort., *Cyanotis axillaris* Roem. and Sch., *Oxalis corniculata* Linn., *Tridax procumbens* Linn., *Ageratum conyzoides* Linn., *Bidens pilosa* Linn. and *Desmodium tortuosum* DC. The relevés were sprayed with the weedicide at 1.12 and 3.36 Kg/ha. They were watered regularly. The response of the weeds to the weedicide was

watched for about a month and then a second spray was given to eradicate those which resisted the first spray.

Lagasca mollis was the most susceptible one to the first spray even at 1.12 Kg/ha. Within 8-10 days of the treatment the weed showed signs of desiccation and was completely killed in 15-20 days. *Galinsoga parviflora*, *Euphorbia geniculata*, *Cyanotis axillaris* and *Oxalis corniculata* were the others to follow. Desiccation and gradual death were noticed within 15-20 days. *Tridax procumbens*, *Ageratum conyzoides*, and *Bidens pilosa* were little affected at 1.12 Kg/ha and showed only slight desiccation. At 3.36 Kg/ha, though there was greater desiccation, the weeds were not killed. In *Desmodium tortuosum* which proved to be the hardiest of the weeds studied, although there was considerable defoliation after 20 days of the spray at both concentrations used, resprouting was observed in the plants treated with 1.12 Kg/ha; such resprouting was less at the higher concentration.

With the second spray at 1.12 Kg/ha, *Tridax procumbens* and *Ageratum conyzoides* were killed within 8 days after the treatment, whereas *Bidens pilosa* and *Desmodium tortuosum* showed only heavy defoliation. However the latter were also killed in 10-20 days after the spray at 3.36 Kg/ha.

Use of Bromacil, as a post-emergent weedicide at 1.12 Kg/ha resulted in the control of *Lagasca mollis*, *Galinsoga parviflora*, *Euphorbia geniculata*, *Cyanotis axillaris* and *Oxalis corniculata*, whereas a second spray at 1.12 Kg/ha for *Tridax procumbens* and *Ageratum conyzoides* and at 3.36 Kg/ha for *Bidens pilosa* and *Desmodium tortuosum* was necessary for complete control.

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Department of Botany, JAYACHANDRA.
Central College, VIDYA MENON.
Bangalore, October 8, 1971.

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