

Physical Sciences Section

Pakistan J. Sci. Ind. Res., Vol. 14, No. 6, December 1971

STUDIES ON COORDINATION COMPOUNDS

Part IV.—Adipyl Dihydrazide Complexes of Copper, Zinc and Cadmium

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(Received November 3, 1971)

Adipyl dihydrazide (ADH) complexes of the type ADH.MX_2 where $\text{X} = \text{Cl, Br, NO}_3$ or $\frac{1}{2}\text{SO}_4$ and $\text{M} = \text{Cu, Zn}$ or Cd , $\text{ADH}(\text{MX}_2)_n$ where $n = 3$ or 4 and $\text{X} = \text{Cl}$ or $\frac{1}{2}\text{SO}_4$ for $\text{M} = \text{Cu}$ and $n = 2$ and $\text{X} = \text{Cl}$ or Br for $\text{M} = \text{Cd}$ and $\text{ADH}_3(\text{MX}_2)_4$ where $\text{M} = \text{Cu}$ and $\text{X} = \text{Cl}$ or Br have been prepared. Unstable compounds of the type $(\text{Cu}_2\text{AdipatoX}_2)$ where $\text{X} = \text{Br}$ or NO_3 have also been obtained which indicate the reducing properties of the ligand. This property is suggested to be responsible for stabilizing the bridged polynuclear clusters. The bridged structure gives lower magnetic moments whereas the monochelates of copper have normal values. The stability constant for the former class of compounds is higher than for the latter. The IR spectra have been discussed with respect to the NH_2 rocking frequencies and it is suggested that the amido nitrogen is linked to the copper atom, whereas the amino nitrogen is bonded to the other metals in their respective complexes.

STUDIES ON COORDINATION COMPOUNDS

Part V.—Metallic Chloranilates

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(Received October 9, 1971)

Metallic chloranilates of the first transition series from vanadium to copper and of palladium and platinum have been prepared. They are shown to be polymers with varying degree of polymerization. The palladium, platinum, iron(III) and chromium(III) compounds are soluble in hydroxylic solvents enabling an electronic spectral study. Certain assignment of the bands have been made and it is found that chloranilic acid has the same position in the spectrochemical series as the amines. The band positions suggest a planar structure for palladium and platinum complexes and octahedral for those of iron(III) and chromium(III). The IR spectra of the chloranilates shows the M—O frequency at 530—80 cm^{-1} which increases in the same order as the Irving-Williams series.

STUDIES ON COORDINATION COMPOUNDS

Part VI.—Amide Complexes of Copper

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(Received October 9, 1971)

Copper complexes of the type $[\text{Cu}(\text{Amide})_n]\text{X}_2$ where the amide=acetamide, *N*-methylacetamide or *N,N*-dimethylacetamide, $n=1, 2$ or 4 and $\text{X}=\text{Cl}, \text{Br}$ or NO_3 . The variation in stoichiometry and the structure of the complexes have been discussed in view of their magnetic and spectral data. They have been suggested to have tetragonally distorted planar or pseudooctahedral structure having anion bridging. From the IR spectra the nitrate complex of at least *N,N*-dimethylacetamide is seen to have a bridged arrangement and the $\text{M}-\text{O}$ frequency is placed at $430-50 \text{ cm}^{-1}$.

THE ENERGY BARRIER FOR THE RACEMISATION OF OPTICALLY ACTIVE 8,8'-BISBROMOMETHYL-1,1'-BINAPHTHYL

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(Received November 19, 1970; revised April 20, 1971)

Optically active 8,8'-(bisbromomethyl)-1,1'-binaphthyl has been synthesised¹ and its rates of racemisation studied. From experimental results the Arrhenius parameters E and $\log_{10}A$ and transition state theory functions ΔH^\ddagger , ΔF^\ddagger and ΔS^\ddagger for the racemisation have been determined and a comparison has been made with other optically active 8,8'- and 2,2'-disubstituted-1,1'-binaphthyls investigated previously.^{1,2}

Pakistan J. Sci. Ind. Res., Vol. 14, No. 6, December 1971

COPOLYMERIZATION OF ACRYLONITRILE AND CROTONIC ACID AND DETERMINATION OF REACTIVITY RATIOS

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(Received September 29, 1970; revised March 22, 1971)

Copolymerization of acrylonitrile and crotonic acid has been carried out at 60°C using benzoyl peroxide as initiator. Monomer reactivity ratios, r_A and r_B were determined using different methods. These values of r_A and r_B were then compared with the calculated values using Hammett equation.

THE BASIC STRENGTH OF FURAN

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(Received July 30, 1970; revised February 27, 1971)

The best possible pK_a value of furan was found spectrophotometrically using Hammett H_0 indicator method. The value came out to be -0.11 . The problem of polymerization of furan in high concentration of acid solution has been overcome by extrapolation method.

IR SPECTRAL CHARACTERISTICS OF NONCYCLIC DIACYLIMIDES*

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(Received June 23, 1971)

Open chain diacylimides have N—H frequencies higher than cyclic diacylimides. The occurrence of several weak bands in the region $3300\text{--}3100\text{ cm}^{-1}$ alongwith a regular N—H band may be considered as characteristic of noncyclic diacylimides. By contrast the two carbonyl bands of cyclic diacylimides, appear as one strong band at $1735\text{--}1720\text{ cm}^{-1}$ in the noncyclic compounds with a weak shoulder at $1690\text{--}1680\text{ cm}^{-1}$.

MAGNESIUM PHOSPHATE**Part VI.—Study of X-ray Powder Diffraction, IR Absorption and Thermal Decomposition of the Compounds $\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$, MgHPO_4 and $\text{Mg}_2\text{P}_2\text{O}_7$**

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(Received April 21, 1971)

X-ray powder diffraction data of $\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$ have been recorded at room temperature (26°C). Its IR spectrum has been recorded and studied in detail. Differential thermal analysis of the compound has been made. The compound MgHPO_4 is amorphous to X-rays; its IR spectrum has been studied. IR spectrum of the compound $\text{Mg}_2\text{P}_2\text{O}_7$ has also been studied.

Pakistan J. Sci. Ind. Res., Vol. 14, No. 6, December 1971

CHEMICAL INVESTIGATION ON THE LENZITES TRABEA

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(Received October 20, 1970; revised February 1, 1971)

Two compounds have been isolated from *Lenzites trabea* and their structures elucidated by IR, UV and NMR spectroscopy and mass measurements.

STUDIES ON TINOSPORA CORDIFOLIA MIERS (BENG: GULANCHA)**Part IV.—Isolation of Heptacosanol, β -sitosterol and Three Other Compounds Tinosporidine, Cordifol and Cordifolone**

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(Received August 7, 1970; revised March 3, 1971)

Petroleum-ether extraction of the dry stems of *Tinospora cordifolia* yielded heptacosanol and a compound, m.p. 233–235°C, which has been tentatively named cordifol. From the alcoholic extract of the fresh stems, β -sitosterol and a compound $C_{32}H_{64}O_3$, m.p. 94–97°C, have been isolated. The name *tinosporidine* has been assigned to this compound. Dry leaves of *Tinospora cordifolia*, on extraction with petroleum ether, yielded heptacosanol and a carbonyl compound $C_{33}H_{64}O$, m.p. 62–62.5°C, tentatively named cordifolone.

INFLUENCE OF DIELECTRIC CONSTANT OF THE MEDIUM ON THE SPECIFIC RATE CONSTANT OF IODIDE-PERSULPHATE REACTION

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(Received November 4, 1971)

Rate of reaction (k) between the persulphate and iodide ions were studied at various concentrations of the reacting ions and at various dielectric constants (ϵ) of aqueous ethanol mixtures at 25° C. From the plot of $\log k_0$ against ϵ the values of r^\ddagger and r_{AB} , the radius of the transient species for single and double sphere models were calculated. Comparison of the experimental and theoretical values of r^\ddagger and r_{AB} showed that the description of the transient species could best be explained by the single sphere model

Biological Sciences Section

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EFFECT OF HEAT ON THE EXTRACTABILITY OF LIPID FROM LEAF PROTEIN MEAL

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(Received April 2, 1970; revised January 15, 1971)

Drying of leaf proteins at elevated temperature results in a decrease in the extractability of the lipids. This seems to be due to oxidation of free lipids, and formation of insoluble complexes by phospholipids and oxidation products of the lipids with proteins. Liberation of bound lipids by 0.2N HCl or 90% phenol suggests that the lipids are not bound to proteins by covalent linkages and the decrease was due to a physical change in the protein.

EFFECT OF SODIUM CHLORIDE AND FLUORIDE ON THE SELECTIVE PRODUCTION OF THE TETRACYCLINE ANTIBIOTICS BY STREPTOMYCES AUREOFACIENS

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(Received September 15, 1970; revised January 11, 1971)

The tetracycline antibiotics are closely related antimicrobial metabolites. The constituents of the fermentation medium are very important for the selective production of chlortetracycline or tetracycline. The basal fermentation medium was able to produce about 64.3% CTC and 35.7% TC. On increasing the concentration of NaCl, the production of CTC increased. When the NaCl level was 80 mg/l in the basal medium, the percentage of CTC and CT was about 90.6 and 9.4% respectively. On the contrary, increasing the concentration of NaF in the fermentation medium increased the production of TC. Therefore, at 80 mg NaF/l the percentage of TC and CTC was 99.4 and 0.6 respectively. To nullify the effect of the chloride level present in the fermentation medium, different concentrations of NaF must be added. The basal medium and 80 mg NaF/l were able to produce TC (100%).

STREPTOMYCES SPECIES PRODUCING ACTINOMYCIN C

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(Received November 9, 1970)

Taxonomic studies on *S. sp.* NRC-152 which was isolated from Egyptian soil were carried out. Its morphological and physiological characteristic features were identical with *S. chrysomallus*. The antibiotic produced was extracted and purified from the fermentation cultures and the mycelial mats of the organism. On the basis of physicochemical and biological properties, the antibiotic was identified as actinomycin C.

COPROPHILOUS FUNGI OF WEST PAKISTAN. PART IV

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(Received July 14, 1970; revised February 16, 1971)

Sixteen species have been recorded for the first time from Karachi on dung except *Podospora ostlingospora* and *Stachybotrys atra*, *Coprinus velox*, *Panaeolus fimicola*, *Penicillium ehrlichii*, *Perichaena corticalis*, *Peziza vesiculosa*, *Physarum pusillum*, *Psilocybe coprophila* and *P. merdaria* which have never been reported from West Pakistan.

STUDIES ON STORED FOOD GRAIN FUNGI

Part IV.—Fungi from Pulses

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(Received July 1, 1970)

The present investigation relates to the microorganisms infesting certain pulses like *Cyamopsis psoralioides* DC (Guara) *Phaseolus mungo* L. (Moong), *Phaseolus radiatus* L. (Mash or Urad), *Cajanus indicus* Spreng (Arhar), *Lens esculenta* Moench (Masoor), *Cicer arietinum* L. (Desi chana), and *Cicer arietinum* L. (Kabuli chana). Thirty-six species belonging to sixteen genera were isolated from the above-mentioned pulses. Fungi Imperfecti was the most dominant group with an occurrence of 86%. The genus *Aspergillus* was most frequent in contributing 12 species with a total prevalence of 60%. *A. flavus*, among the species of *Aspergillus* accounted for maximum damage 24%. *Cicer arietinum* (Desi chana) was the most heavily infested pulse with an infestation of 98% of the samples investigated while *Phaseolus radiatus* was the least infested pulse with an infestation of 45% of the samples studied.

MARINE FISH TREMATODES OF WEST PAKISTAN**Part VII.—*Ectenurus crenidensis* n. sp. from *Crenidens indicus* Day, from the Karachi coast***

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(Received February 24, 1971; revised May 22, 1971)

Ectenurus crenidensis sp. n. is described from the gills of *Crenidens indicus* (Day), from the Karachi coast, West Pakistan. *Ectenurus crenidensis* is characterised by possessing (1) a transversely elongate, ovate or distinctly bilobed ovary, (2) a well developed shell gland, (3) small ovate eggs, (4) nearly symmetrical testes, (5) an elongate seminal vesicle, (6) an elongate pyriform sinus sac with a well developed sinus organ, (7) a compact mass of prostatic glands at the base of sinus sac, (8) a genital pore ventral to pharynx, and (9) vitelline tubes frequently extending into the ecsoma. *Ectenurus crenidensis* is the first species of the genus recorded from Pakistan and from the gills of a fish.

**CRICONEMOIDES KAMALIEI n. sp. (CRICONEMATIDAE: NEMATODA) FROM
SIND REGION**

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(Received August 24, 1971; revised February 1, 1971)

Criconemoides kamaliei n. sp. is new to science having peculiar shape of the body, poorly marked anatomiasis and double rows of oöcytes. A revised key of its species is also given for further investigation of the new species.

**DIGENEA OF PAKISTAN FRESHWATER FISHES I. HAPLORCHOIDES SINDICUS
N. SP. (FAMILY: CRYPTOAGONIMIDAE) FROM THE INTESTINE OF MYSTUS AOR
(HAM.), SUKKUR, WEST PAKISTAN**

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(Received December 30, 1971)

A new species *Haplorchoides indicus* n.sp. (Family: Cryptogonimidae) has been recorded from the intestine of *Mystus aor* (Ham.) of the Indus river, Sukkur, West Pakistan. The new species resembles closely *H. attenuatum* (Srivastava, 1935) obtained from the same host.

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EFFECT OF POST-TREATMENT TEMPERATURE ON THE TOXICITY OF PETKOLIN TO SUSCEPTIBLE HOUSEFLIES, *MUSCA DOMESTICA* L

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(Received March 4, 1971)

Laboratory-reared (PCSIR strain) houseflies, *Musca domestica* L., were treated topically with 1.2 μ l acetone solutions of Petkolin, DDT and Heptachlor and held at the post-treatment temperatures of 10, 25 and 40°C for 48 hr. With Petkolin and Heptachlor there were significantly higher mortalities at 40°C whereas in the case of DDT the highest mortality was recorded at 10°C.

THE PERSISTANCE OF PETKOLIN RESIDUES MEASURED BY BIOLOGICAL METHOD

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(Received January 14, 1970; revised January 15, 1971)

The recovery of Petkolin residue on cotton plant leaves was determined by bioassay method. Emulsions of 1, 1.5, 2 and 3% Petkolin were sprayed on cotton leaves at the rate of 1 ml per leaf. Leaves from each concentration were picked 24 hr, 72 hr and 1 week after the treatment. Extractions were done in acetone and the extracts were tested against larvae of *Aedes aegypti* (L) according to WHO method. From 1, 1.5, 2 and 3% Petkolin emulsion sprays, respectively 7, 8, 10 and 11% of the sprayed Petkolin after 24 hr; 4, 4.3, 4.8 and 6% of the sprayed Petkolin after 72 hr and 2, 2.5, 2.9 and 3.3% of the sprayed Petkolin after 1 week was recovered. It was found that Petkolin is a short-lived insecticide and rapidly loses its residual effect.

Technology Section

Pakistan J. Sci. Ind. Res., Vol. 14, No. 6, December 1971

PRESERVATION OF CITRUS FRUITS WITH FUNGICIDAL WAX EMULSION

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(Received October 5, 1971)

A fungicidal wax emulsion (Furitex) developed in these Laboratories was examined in detail with regard to its suitability for applying to oranges for extending their storage life. Changes in moisture, total sugar and reducing sugar contents, brix, acidity, vitamin C and respiration rate were determined after treating the fruits with Fruitex, alongwith the control (untreated) fruits. These were found significantly different from those of the treated lots and demonstrated the delaying effect of the ripening process in Fruitex-treated lots. Spoilage of fruits due to mould growth was found to be less in treated lots as compared with untreated lots. Organoleptic evaluation of fruits showed no significant difference between appearance, taste and differently treated fruits.

PECTIN EXTRACTION FROM ROSELLE SEPALS

Effect on the Quality of Pectin During Storage of Dehydrated Sepals

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(Received September 30, 1970)

Roselle (*Hibiscus sabdariffa* L.) thrives well in our conditions. In this investigation the effects on recovery and quality of pectin extracted from dehydrated sepals of Roselle during six month's storage at ambient temperature are studied.

CYCLIC HYDROCARBONS IN A LOW TEMPERATURE COAL-TAR

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(Received January 23, 1971)

Stepwise extractions with various strengths of H_2SO_4 of an aromatic hydrocarbon fraction from a low temperature coal-tar, and examination of the subfractions, confirmed the presence of alkylated monocyclics and bicyclics, and of olefinic structures, and pointed to the presence of alkylated cycloaromatic hydrocarbons of higher average molecular weight than the rest, and of benzenes having long alkyl side-chains.

MAGNESITE OCCURRENCE IN HAZARA DISTRICT

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(Received July 1, 1970)

Magnesite occurs as three lens-like bodies near Sherwan (Hazara) in the Abbottabad Formation of Upper Paleozoic system. Total length of two outcrops is around 900 ft and width about 150 ft. The third outcrop is 150 ft. long and 20 ft wide. The deposit is probably hydrothermally metasomatized type. The properties of the magnesites were determined by utilizing microscopic, chemical, X-ray diffraction and differential thermal techniques. The conclusion drawn from the results obtained is that ores are fairly rich in magnesite with some minor impurities such as calcite, hematite, clay, siderite, quartz and limonite.

BRUCITE DEPOSITS OF HINDUBAGH (WEST PAKISTAN)

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(Received October 22, 1970; revised February 24, 1971)

Brucite bearing serpentine rocks are located at an aerial distance of 5 miles north of Hindubagh and extend from the village Khazina to the west of the village Telri Muhammed Jan. Brucite is of fairly good quality and suitable for the production of the magnesium chemicals as well as for use in building materials.

CERAMIC COLOURS**Part IV.—Green Stains**

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(Received January 7, 1971)

A comprehensive study is made on the preparation and use of the various green colours, having different shades. Optimum conditions and composition have been established for stains as well as glazes. The stains have been studied for use as under-glaze, over-glaze and also as in-glaze stains. The colours can be applied at various temperature ranging from 900-1200°C.

INCIDENCE OF MEDULLATION IN CARPET WOOLS

Part I.—Relationships of Medullation Characteristics with Diameter

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(Received March 11, 1971)

Percentage proportions of true, heterotypical, medullated and kempy fibres were evaluated in ten samples of Pakistani carpet wool. The diameters of the samples as well as of the assorted subsamples were determined and correlation coefficients for the various relationships estimated. It appears that, with increase in diameter, it is not only the number of medullated fibres that increases but that their diameter also increases significantly in comparison to that of true as well as heterotypical fibres.

AREA SHRINKAGE DUE TO RELAXATION AND FELTING OF PLAIN-WOVEN SILK-WOOL FABRICS

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(Received August 22, 1970; revised January 23, 1971)

Observations on a variety of silk-wool fabrics reveal that the rate of relaxation shrinkage falls off significantly with higher yarn tex, ply number and fabric stiffness. The fabric felting rate, however, shows highly significant negative correlation with the stiffness parameter and yarn tenacity. The effects of yarn tex and twist factor on felting rate seem to be significant in case of the 1-ply fabrics but they are nonsignificant in the 2-ply fabrics. Although the 2-ply coarse fabric shows lower felting rate than that of the 1-ply counterpart, the ply effect may reverse its sense in fine cloths due, perhaps, to the influence of higher plying and weaving strain on the finer wefts. Finally, the rank correlation of loose wool felting rate with yarn and fabric felting rates appear to be highly significant if the relevant singles are slightly untwisted by any means.

SHORT COMMUNICATION
TECHNOLOGIES SECTION

Pakistan J. Sci. Ind. Res., Vol. **14**, No.6, December 1971

PARTIAL SYNTHESIS OF Δ 15-20- ANHYDRO VINBLASTINE*

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(Received October 27, 1971)

SHORT COMMUNICATION**TECHNOLOGIES SECTION**

Pakistan J. Sci. Ind. Res., Vol. **14**, No.6, December 1971

**CONSTITUTENTS OF LAVANDULA STOECHAS LINN.
PART III*.- SPECTRAL STUDIES OF LAVANOL**

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(Received June 14, 1971)

SHORT COMMUNICATION**TECHNOLOGIES SECTION**

Pakistan J. Sci. Ind. Res., Vol. **14**, No.6, December 1971

PRELIMINARY CHEMICAL INVESTICATIONS ON EMINIUM SPICULATUM

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(Received October 7, 1971)

SHORT COMMUNICATION**TECHNOLOGIES SECTION**

Pakistan J. Sci. Ind. Res., Vol. **14**, No.6, December 1971

THE CRYSTAL STRUCTURE OF HARMIDINE (HARMALINE)

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(Received December 3, 1971)