SHORT COMMUNICATIONS

CATION-EXCHANGE PROPERTIES OF SULPHONATED SHELLAC-FURFURAL CONDENSATE

M.H. KHUNDKAR AND ABDUL MALEK

Department of Chemistry, Dacca University, Dacca

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Shellac is a thermoplastic resin and very little is known about its conversion into a hard thermosetting product through further condensation. It was treated[1] with concentrated H_2SO_4 at 140-145°C, washed and the resulting mass dissolved in NaOH; on treatment of this solution with resorcinol and paraformaldehyde, a resin gel was obtained, which on hardening showed the cation-exchange properties.

Although on direct sulphonation, shellac developed cation-exchange property (capacity of 0.89 m-equiv/g), considerable degradation of the resin itself took place and the product discharged colour even on standing in water. When a 40% formaldehyde solution was refluxed with the varying ratios of shellac (with acid and basic catalysts), only an agglomerated soft cake was formed. Treatment with phenolsulphonic acid also gave a brittle and rather unsatisfactory product. But with furfural, a hard condensation product was obtained, which could be sulphonated or oxidised to give cation exchange resin.

30 g of shellac was dissolved accompanied by stirring along with 50 g of freshly distilled furfural. On standing, a gel was formed within a few minutes. 8 g of HCl (d 1.16) was added when the product changed into dark agglomerates. On heating (to 90°C), the agglomerates softened to a black viscid liquid. On continued heating, a film was first formed and then the condensation started rapidly with the evolution of fumes, resulting ultimately in a hard black mass. The temperature was now slowly raised to 130°C and the product cured for three hours. The yield was 89% of the total original weight. The hard black mass did not soften even at 250°C. It was insoluble in moderately concentrated acids, alkalis and also in organic solvents. It was quite suitable for sulphonation. Some results are presented in Table 1.

TABLE 1.—CONDENSATION OF SHELLAC WITH FURFURAL: EFFECT OF THE SHELLAC/FURFURAL RATIO.

Furfural/shellac ratio (w/w)	Yield (%)	Bulk density (g/cc.)	Nature of the product
0.60	94.2	0.35	Greyish inconsistent mass.
1.00	91.4	0.53	Dark hard mass.
1.66	89.0	0.59	Dark hard mass.
1.66[a]	89.0	0.61	Dark hard mass.
1.66[b]	89.5	0.50	Somewhat softer mass
2.00	88.2	0.49	Dark hard mass.
5.00	79.1	0.63	Somewhat hard mass.
8.00	—	—	Dark soft mass.

(a) Pressure applied at 150-160°C for 5 hr.
(b) Hexamine used for curing.

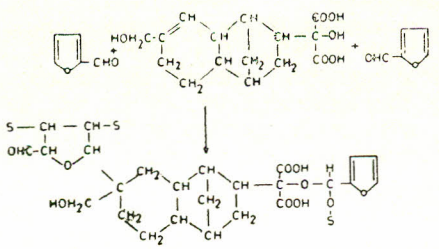
Shellac used = 25 g Temperature=90±2.5°C
HCl used (as catalyst)=10% of total weight of shellac and furfural.

The condensation depends considerably on the ratio of the furfural/shellac. With smaller proportion of furfural (*ca* 0.6 w/w), the product was an inhomogeneous greyish mass and contained some unreacted shellac; with equal ratio of furfural/shellac, results were better, but not quite satisfactory. Best results were obtained with a proportion of furfural of 1.66 to 2.00 parts of shellac. With the larger proportion of shellac >*ca*5 condensation was again unsatisfactory. In other experiments it was noted that application of pressure offered no improvement in the condensation, and the use of hexamine actually arrested the progress of condensation.

The temperature of condensation was also very significant. It took only a minute or so for the condensation to complete at 110-120°C, but the product was soft. At 100°C results were better, but it was best at a temperature of 90°. At this temperature the initiation of polymerization took about twenty minutes, but it took another thirty minutes for the completion of polymerization. The hard product could be carefully broken down into granules having the desired size and subsequently sulphonated.

Sulphonation of the condensation product with concentrated H_2SO_4 introduced both the $-SO_3H$ and -COOH groups[2] with exchange capacities of 1.00 and 1.20 m-equiv/g respectively. The condensation product could also be oxidised to give a monofunctional (carboxylic) cation exchanger. For this, nitric and chromic acids were too drastic, whereas $KMnO_4$ and H_2O_2 were rather mild. But on boiling the product with alkaline copper sulphate (Bertrand's solution containing 62.5 g $CuSO_4$. $5H_2O$/lit. and 20 g. Rochelle salt and 150 g NaOH/lit. of solution) for about fifteen minutes, the polymer developed the -COOH groups (1.25 m-equiv/g) in its structure.

Shellac is a cross-linked, interester resin formed from a mixture of polyhydroxy acids.[3] The structure of shelloic acid,[4,5] the chief constituent of shellac, suggest that condensation with furfural may take place either by addition polymerization or through polyacetal formation. Furan derivatives are known to undergo a host of addition reactions.[6] The polyacetal formation in shellac is possible through some of the -OH groups that still remain after the interesterification has taken place. If we accept Nagel's structure of shelloic acid,[5] both these reactions may take place simultaneously.



The shellac structure may itself accomodate some $-SO_3H$ groups when sulphonated like other naturally occurring substances containing aliphatic or cylic structures.[7] The furan ring can also accomodate $-SO_3H$ groups. The -COOH groups are developed through the rupture of the ester linkages in shellac and also by the oxidation of free aldehyde groups in the furfural skeleton.[8]

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ON THE COLOUR CHANGES EXHIBITED BY CERTAIN LACTAMS

A. M. AHSAN

*School of Pharmacy, 29-39 Brunswick Square, London W.C. 1, U.K.**

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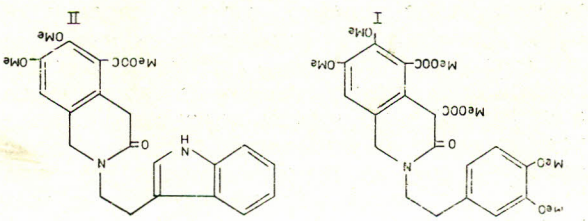
During the course of the synthesis of N-2-(3,4-dimethoxyphenyl)-ethyl-3-keto -5- carbomethoxy - 6,7 - dimethoxy - 1,2,3,4 - tetrahydroisoquinoline[1] the lactam (I) was obtained as a light yellow resin (cf. the colour



of Weisenborn's lactam II),[2] which could not be crystallised, although the quantity of the gum obtained was quite consistent with the yield to be expected assuming complete conversion to the lactam. The failure to obtain the compound in the crystalline form led to the preparation of the lactam 2-(n-propyl)- 3-keto-5-carbomethoxy-6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline.[3]

COLOUR CHANGES EXHIBITED BY THE LACTAMS

The following colour changes were exhibited by the lactams:

(i) The colour of this neutral yellow gum grew more intense on heating.

(ii) The crystalline lactam III was light yellow in colour but on dissolution in carbon tetrachloride lost its colour gradually. The solvent

* Present address: Central Laboratories, P.C.S.I.R., Karachi-32.

on spontaneous evaporation left initially a faintly yellow gum. On heating, this crude solvent-free gum also deepened in colour.

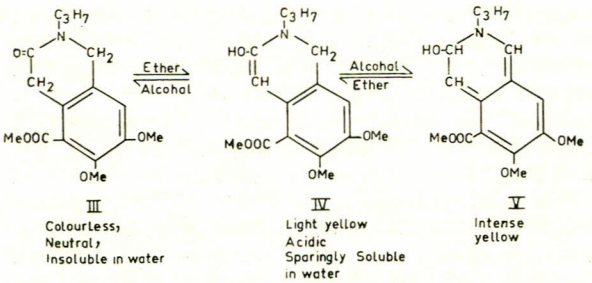
(iii) The original lactam is neutral in reaction but its solution in ether on standing is distinctly acid to litmus. The colour of the solution also deepened on standing.

(iv) In hydroxylic solvents like alcohol the colour deepened.

(v) Further the lactam was sparingly soluble in water giving a yellow solution. Treatment of this aqueous solution with sodium hydroxide produced a deeper yellow colour, which diminished again on treatment with acids.

Discussion

The following explanation is offered for these colour changes.



III
Colourless,
Neutral,
Insoluble in water

IV
Light yellow
Acidic
Sparingly Soluble
in water

V
Intense
yellow

The colour of the crystalline, neutral lactam is explained because of the presence of the intensely yellow quinonoid form V in small amount. Its neutral reaction is understandable as it exists in the main in form III; the minor amounts of IV presumably do not affect litmus.

The diminution of the colour on the dissolution of the crystalline lactam in ether and the acid reaction of the solution to litmus are explained by the shift of the equilibrium between forms III and IV towards the right and that between the forms IV and V towards the left. Therefore, in both cases more of form IV would be formed which explains the acidity of the solution. The diminution of form V would result in the decrease of the colour.

The deepening of the colour in the hydroxylic solvents is explained by the tendency of the form IV to change to forms III and V. The increase in the form V would explain the heightened colour.

The increase in colour on heating and its lessening on cooling is due to the equilibrium between the forms IV and V, the latter being the form presumably increasing at higher energy levels.

Further, form IV explains the solubility in water. The intensification of the colour of this solution with the alkali is explainable by the formation of the alkali salt of IV, which is pre-

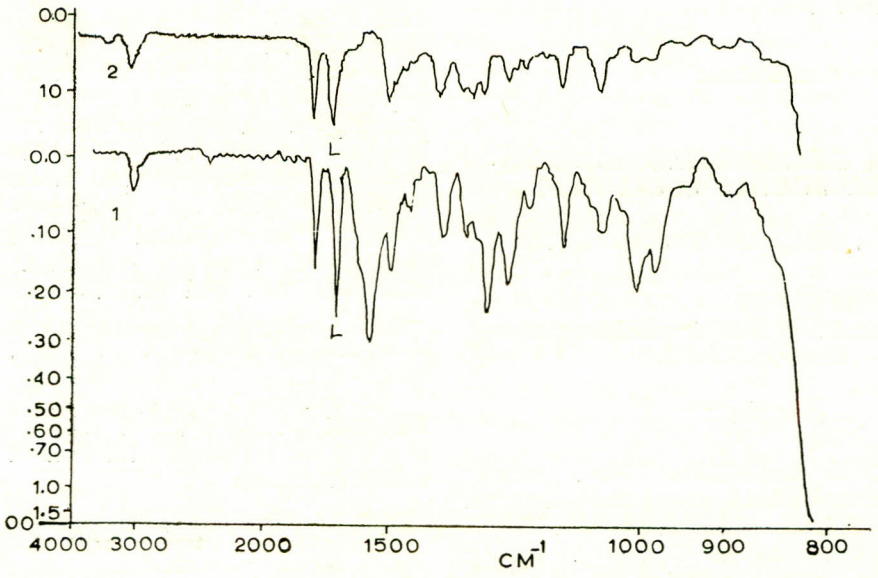


Fig. 1

sumably of a deeper colour like with the phenolic salts. The converse would then be expected on treatment with acid, and thus this change is also understandable.

This change of the lactam on solution is shown by the infrared spectra (recorded with a Perkin-Elmer infrared double beam spectrophotometer) curves in Fig. 1. Spectrum numbered I was taken immediately after dissolving the crystalline lactam in carbon tetrachloride. The second is of the same solution after standing for 51 hours. It would be noticed that the lactam peak (marked L in the diagram) is conspicuously shorter in the latter, and further the other peaks are present, apart from a few but again diminished in length. The one most noteworthy new peak is the broad one at 3,300 to 3,400 cm-[1], which agrees with that expected for an aromatic hydroxyl. The region between 1,500 to 1,650 cm-[1] is doubtful.

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CHEMICAL EXAMINATION OF CROTALARIA BURHIA BUCH.-HAM.

M. AMJAD ALI AND (MISS) ZULEKHA KAPADIA

Drugs and Pharmaceutical Division, Central Laboratories, Pakistan Council of Scientific and Industrial Research, Karachi

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Crotalaria Burhia Buch-Ham. (local name: Drunnu) is an undershrub common in the arid parts of West Pakistan, Rajputana and Gujrat. Its leaves and branches are used as a cooling medicine.[1] No work recorded is available in literature on this plant. The results of a chemical examination of the petroleum-ether extract of the plant are reported in the present communication.

The aerial parts of fresh plants (8 kg) were extracted with petroleum-ether. The extract, on removal of petroleum-ether, gave a residue (83.9 g.) which was saponified by refluxing with 5% alcoholic potassium hydroxide for 4-5 hours. The unsaponifiable matter (69 g) was obtained as a dark brown viscous mass. A small portion (2-3 g) of the unsaponifiable matter was chromatographed on a column of activated alumina using petroleum-ether as the eluant. The first fraction of the eluate, on removal of solvent, gave a semi-solid mass which, after purification by rechromatography in the similar manner, crystallised from ethyl acetate in small shining plates, m.p. 68-69° (Found after drying at 60° *in vacuo*: C, 85.58 and H, 14.54%; mol. wt. 443; Calc. for $C_{31}H_{64}$: C, 85.23 and H, 14.77%; mol. wt. 437). The compound neither absorbed bromine in the chloroformic solution nor produced any colouration with tetranitromethane. It was found to be identical with hentriacontane, a common constituent of plants (lit.[2] m.p., 68°C). The second fraction of the eluate from the alumina column, on being freed from solvent, gave a slightly yellow coloured material but all other fractions of eluates yielded white substances which crystallised on addition of alcohol. The crystalline materials were all combined and purified by chromatographing once again on activated alumina column. The eluate, on removal of solvent, gave a crystalline residue which was recrystallised from methanol of colourless needles, m. p. 137-38°, $[\alpha]_D^{20}$ —32.2° (c = 1.15, $CHCl_3$), $C_{29}H_{50}O$ (Found after drying at room temperature *in vacuo*: C, 83.54 and H, 12.12%; $C_{29}H_{50}O$ requires: C, 83.99 and H, 12.15%). The compound gave positive tests with the Liebermann-Burchard reagent (pink→violet→blue→green) and tetranitromethane. It was identified as βsitosterol (lit.[3] m.p. 140°; $[\alpha]_D^{34}$—36°) by mixed melting point, infra-red spectrum and also by preparation of acetate, colourless needles from methanol, m.p. and mixed m.p. 129-30°, $[\alpha]_D^{34}$ —40.8° (c=0.54, $CHCl_3$), $C_{31}H_{52}O_2$ (Found: C, 81.26, 81.20 and H, 11.20, 11.30%; acetyl value, 9.85%; $C_{29}H_{49}OCOCH_3$ requires: C, 81.52 and H, 11.48%; acetyl value, 9.43%). The infrared spectrum of the acetate was also found to be identical with that of authentic β-sitosterol acetate.

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ALKALOIDS OF SOLANUM INCANUM LINN.

M. AMJAD ALI, SHAHBAZ KHAN AND (MISS) ZULEKHA KAPADIA

Drugs and Pharmaceutical Division, Central Laboratories, Pakistan Council of Scientific and Industrial Research, Karachi

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Introduction

Solanum incanum Linn. is a woody herb which grows wildly in Sind, Punjab and the Western Peninsula. It is reputed in the indigenous medicine as a remedial drug for tooth-ache, sore throat, chest troubles and other ailments.[1] The plant belongs to the Solanaceae family, several species of which have been found to contain glyco-alkaloids, a special class of alkaloids in which the base is chemically linked with sugars, usually glucose, galactose, rhamnose and xylose.[2] Since no work has hitherto been reported on *S. incanum*, it was considered feasible to undertake an examination of this plant for alkaloids. Chemical examination showed the presence of alkaloids in the roots, stems and leaves in small quantities while the fruits were rich in the alkaloidal content. Extraction of the fresh, green berries of *S. incanum* (locally known as *Jangli baigan*) with ethanol and a subsequent examination by paper chromatography of the total base obtained from the extract, revealed the presence of two alkaloids in the fruits. Separation of these alkaloids was accomplished by taking advantage of their solubilities in acetone. One of the alkaloids crystallised from ethanol-water and, after purification by several recrystallisations from the same mixed solvent, showed a constant m.p., 280-81° (decomp.), as well as a single spot on a paper chromatogram. It proved to be a glyco-alkaloid as, on hydrolysis with 5% hydrochloric acid, it yielded an alkamine hydrochloride, m.p., 310°, and a mixture of sugars composing of glactose, glucose and rhamnose. The alkamine hydrochloride, on treatment with ammonia, gave the free base, m.p. 190-91°, which was identified as solasodine (lit.[3] m.p. of solasodine, 198°; hydrochloride, 314.5-315°) by mixed melting point, infrared spectrum and R_f values. The parent glyco-alkaloid was, therefore, solasonine (lit.[4] m.p. 279°). Furthermore, the glyco-alkaloid gave a picrate, m.p. 195-96° (decomp.) (lit.[3] m.p. of solasonine picrate, 197°).

The other alkaloid could not be obtained pure. Paper chromatography, using the solvent mixture n-butanol-acetic acid-water (4:1:1) showed its R_f value as 0.75 while that of solasonine, under identical conditions, was 0.62.

Experimental

Isolation of Solasonine.—Fresh, green berries (155 g) of *S. incanum* were cut into pieces and soaked in ethanol. After about a week, the extract was taken out and completely freed from solvent. The residue left was treated with 5% acetic acid and shaken, at first, with petroleum-ether and then with ether. The acidic aqueous portion was warmed a little and then basified with ammonium hydroxide to pH 8-9 when a dirty-white flocculent precipitate was obtained which was filtered by suction and washed thoroughly with 2% ammonium hydroxide. The precipitated base was then dissolved in 5% acetic acid and again precipitated with ammonium hydroxide. The process was repeated several times until the precipitated base was almost white. The yield of the total base was 1.03 g.

Chromatography of the total base on Whatman No. 1 filter paper with *n*-butanol-acetic acid-water (4:1:1) showed two spots of alkaloids (R_f values 0.75 and 0.62). The alkaloid with R_f 0.75 was found to be much more soluble in acetone in comparison with the other one. Taking advantage of this property, the total base was divided into acetone-soluble and acetone-insoluble fractions. The acetone-insoluble fraction readily crystallised from ethanol-water, while all attempts to crystallise the other fraction did not succeed.

The crystalline alkaloid, solasonine, after several recrystallisations from ethanol-water, gave very

fine, white needles, m.p. 280-81° (decomp.), which showed a single spot at R_f 0.62 when chromatographed on Whatman No. 1 paper with n-butanol-acetic acid-water (4:1:1). Its picrate was obtained as needles, m.p. 195-96° (decom.).

Hydrolysis of Solasonine.—Solasonine was refluxed with 5% hydrochloric acid for three hours. The hydrochloride of the aglycone, solasodine, separated out in crystalline condition from the hot solution. After cooling, the crystals were filtered off, washed thoroughly with water and recrystallised from ethanol-water as colourless needles, m.p. 310° (lit.[3] m.p. of solasodine hydrochloride, 314.5-315).

Solasodine hydrochloride was suspended in hot water, made alkaline with ammonium hydroxide and heated at 100° for one hour. The liberated base, on repeated crystallisation from methanol, gave plates, m.p. 190-91°. It did not depress the m.p. of an authentic sample of solasodine (Light Chemical Co., England), and gave an infra-red spectrum identical with that of solasodine.

The hydrolysis products remaining in concentrated aqueous solution were identified by paper chromatography (descending method) with *n*-butanol-pyridine-water (10:3:3) as galactose, glucose and rhamnose. Authentic samples of galactose, glucose and rhamnose were used for comparison.

Paper Chromatography of Solasodine.—The aglycone obtained on hydrolysis of solasonine, authentic solasodine and a mixture of the two, were chromatographed on Whatman No. 1 filter paper with *n*-butanol-acetic acid-water (4:1:1). The mixture did not resolve and the chromatogram showed only three spots, all having the same R_f 0.91.

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CHEMICAL INVESTIGATION ON THE SEEDS OF HYGROPHILA SPINOSA T. ANDERS

M. AMJAD ALI

Central Laboratories, Pakistan Council of Scientific and Industrial Reserach, Karachi

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Hygrophila spinosa, locally known as *Tal-makhana*, is reputed to be of considerable value in the indigenous medicine.[1] It is used for jaundice, dropsy, rheumatism and other ailments. Its seeds possess diuretic properties due to the presence of large amount of mucilage and potassium salts,[2] and are prescribed in cases of gonorrhoea and spermatorrhoea. Works on the plant, its roots and seeds had been reported earlier. Investigation on the seeds[3,4] previously led to the isolation of an oil which was classified as a semi-drying oil; its fatty acid components were determined. In addition, the seeds were found to contain diastase, lipase and protease.

In the present work, the oil from the seeds has been isolated and its physico-chemical characteristics studied. Besides, two pure crystalline compounds having m.ps., 130-31° and 216-18° have been isolated and characterised by the preparation of their derivatives. The former was found to be a sterol. The ethanolic extract was found to contain a syrupy fraction which, on hydrolysis, yielded glucose, rhamnose and arabinose. The water-soluble mucilage of the seeds was also studied.

Extraction.—The dried seeds were crushed and thoroughly extracted with ethanol. On removal of the solvent, a dark brown viscous liquid was obtained as residue (23% of the wt. of dry seeds). The major portion of this residue was found to dissolve in petroleum-ether (b.p. 40-60°) at the ordinary temperature and the petroleum-ether solution, on being freed from solvent, gave an oil (19% of the wt. of dry seeds). The remaining portion of the residue was a syrupy liquid (3.7% of the wt. of dry seeds) which was completely soluble in water.

The oil was found to have the following physico-chemical characteristics:

Density at 30°	0.9163
Refractive index at 30°	1.46774
Saponification value	187

Iodine value	121
Acid value	113

Isolation of Pure Compounds from the Unsaponifiable Portion of the Oil.—The oil was saponified by refluxing with 5% alcoholic potassium hydroxide. After removal of alcohol, water was added to dissolve the soaps and the unsaponifiable matter was extracted with ether. The total unsaponifiable matter was 1.5% of the oil.

On purification by chromatography on alumina column and fractional crystallisation, two pure, crystalline compounds were obtained from the unsaponifiable portion which melted at 130-31° and 216-18°. The former was a sterol because it responded to the Liebermann-Burchard test and formed an acetate, m.p. 122-24°, with acetic anhydride and pyridine at room temperature. The latter compound also responded to the Liebermann-Burchard test and gave a crystalline acetate, m.p. 215-17°, with acetic anhydride and pyridine at room temperature but it could more probably be a triterpenic alcohol rather than a sterol in view of its high melting point.

The aqueous soap solution was acidified and the precipitated fatty acids were separated into saturated and unsaturated portions by Twitchell's modified lead-salt method. Palmitic and stearic acids were found to be the major constituents of the saturated acids (7% and 13% respectively of the total acid). The two acids were separated by fractional distillation of methyl esters obtained by methylating the total saturated acid with methanol and sulphuric acid.

Sugars.—The water-soluble syrupy liquid, mentioned above, was hydrolysed with 2N H_2SO_4 whereby a mixture of reducing sugars was produced. Paper chromatographic examinations, using the solvent mixture *n*-butanol (40)/ethanol (11)/water (19), revealed the presence of glucose, rhamnose and arabinose in the hydrolysate.

Water-soluble Mucilage.—The seeds were extracted with water at ordinary temperature and the aqueous extract filtered, concentrated to a very small bulk and treated with enthanol. A precipitate of the waters-oluble mucilage was obtained which was filtered off and dried in a vacuum desiccator. The water-soluble mucilage was found to have a very little ash content. It was hydrolysed with 2N H_2SO_4 and paper chromatographic examination revealed the presence of three sugars in the hydrolysate, among which one was identified as xylose and the other two, though found to be uronic acids, could not be fully identified.

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A SYNTHESIS OF PYRIMIDINE

ALI EHSAN AND KARIMULLAH

West Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Lahore

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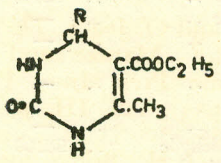
Introduction

Recently, 4-substituted-2-oxo-6-methyl-5-carbethoxy 1,2,3,4-tetrahydropyrimidines have attracted particular interest because of their therapeutic activity against certain group of viruses,[1] their growth inhibiting activities[2] and their mothproofing and germicidal[3] properties. In view of the importance of these compounds, it was considered of interest to devise a workable method for their preparations. As the result of extensive studies, a simple and general procedure for the preparation of these compounds has been described and the formation of the pyridine and pyrimidine rings under mild conditions has been demonstrated.



A practical procedure for preparing 4-substituted 2-oxo-6-methyl-5-carbethoxy 1, 2, 3, 4-tetrahydropyrimidines was first described by Biginelli.[4] This

was based on a reaction of an aldehyde with β-keto ester and urea in alcoholic solution, with hydrochloric acid as a catalyst. Biginelli[4] showed that pyrimidine could also be prepared by the interaction of benzaldehyde and β-carbamide crotonic ester or the interaction of arylidene diuride with ethyl acetoacetate. Hinkel and Hey[5] introduced changes in the experimental technique and reported the formation of dihydropyridine, in the case of benzaldehyde, without any catalyst.

Folkers, Harwood and Johnson[6] showed that the formation of dihydropyridine as obtained by Hinkel and Hey[5] was due to the absence of sufficient amount of acid which prevented the formation of dihydropyridine according to the Hantzsch condensation. They also observed that the reaction between the components in Biginelli condensation was very slow unless it was catalysed by an acid. They carried out their experiments in solvents like ethyl alcohol, acetic acid and dioxan, in the presence of strong hydrochloric acid at the boiling point of different solvents used.

The present work describes the Biginelli condensation in water at different pH values under mild conditions. The authors' findings, after making thorough investigations of the Biginelli condensation, confirm the views of Folkers *et al.*[6] The authors have observed that condensation takes place smoothly in the presence of an acid as a catalyst. At pH 1-2, pure pyrimidine derivatives separate out within 24 hours. In some cases *e.g.*, ortho-nitro-benzaldehyde and heptaldehyde, compounds separate out within fifteen minutes. However, at the higher pH, it takes 5 to 7 days for the compounds to crystallise out. In this case, an interesting feature of the reaction was the formation of dihydropyridine. When formaldehyde was used, the reaction occurred only at pH 5-7 without any catalyst and at lower pH, no condensation took place.

Dihydropyridines were isolated when acetaldehyde and formaldehyde were reacted with urea solution without the use of any catalyst. If the reaction mixture was left for several months at pH 7, in the case of formaldehyde, a mixture of dihydropyridine and pyrimidine derivatives was isolated in measurable amounts but the main product of condensation in all cases was a pyrimidine derivative.

Experimental

The m.ps of all the compounds are uncorrected. The compounds recorded in the Table are insoluble in organic solvents and difficulty soluble in hot alcohol. Experiments were performed in stoppered flasks at room temperature (20-37°C). The proper and suitable concentration of urea was adjusted after a number of trials. The purest available sample of ethyl acetoacetate and aldehydes were taken and all other reagents were of Analar quality. Hydrochloric acid was used as a catalyst. All the experiments were carried out according to the same procedure. Adjustment of pH was carried out with Cambridge bench pH meter.

TABLE.—PERCENTAGE OF YIELD* OF 2-OXO-6-METHYL-5-CARBETHOXY-4-R-1,2,3,4-TETRAHYDROPYRIMIDINES AT DIFFERENT pH VALUES AFTER TWO WEEKS.

No.	Aldehydes(R)	pH	1.0	2.10	3.06	4.00	4.95	6.05	6.64	Melting Point	% of N_2	
											Found	Calculated
1.	Formaldehyde ..	..	—	—	—	—	—	1.00	2.50	255-256° 253-256°(6)	14.92	15.21
2.	Acetaldehyde ..	..	20.00	—	—	—	—	—	—	198-199° 195-196°(4)		
3.	Propionaldehyde	..	15.00	—	—	—	—	—	—	184-185°	13.90	14.13
4.	n-Heptaldehyde ..	..	12.40	—	—	—	—	—	—	—	—	—
5.	Crotonaldehyde ..	..	—	44.10	34.80	32.50	25.70	23.70	traces	268-269°	12.49	12.49
6.	Benzaldehyde ..	..	—	40.00	24.60	18.00	12.00	10.00	7.00	203-204° 202.4° (6)	10.45	10.77
7.	Salicylaldehyde ..	..	—	87.00	86.00	86.00	66.3	60.3	43.3	199-200° 201.202° (6)	9.714	10.14
8.	Anisaldehyde ..	..	—	75.00	70.00	68.00	45.00	23.40	28.7	208-209°	9.86	9.62
9.	o-Nitro-Benzaldehyde	..	50.00	47.00	29.00	—	—	—	—	269-270°		
10.	p-Nitrobenzaldehyde	..	—	—	—	—	—	—	—	—	—	—
11.	p-Dimethylamino benzaldehyde ..	..	10.00	—	—	—	—	—	—	257-258°	14.2	13.90
12.	o-Chloro benzaldehyde	..	20.00	—	—	—	—	—	—	213-215°	9.55	9.33

*All the yields have been recorded on the basis of acetoacetic ester.

Acetoacetic ester and an aldehyde (0.01M of each) were added to 100 ml. of 5% aquous solution of urea. Its pH was adjusted with hydrochloric acid. The flask was stoppered and left at room temperature. The compounds separated out within 24 hours with the exception of those in which formaldehyde was used as the aldehyde.

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INVESTIGATIONS ON DATURA FASTUOSA LINN (SOLANACEAE)

Part II.—Isolation of Daturanolone and Fastusic Acid from the Seeds

A. KHALEQUE, M.A. WAHED MIAH, M. SADRUL AMIN AND NIRMOLENDU ROY

East Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Dacca

(Received June 13, 1966)

In the previous communication,[1] isolation of the alkaloidal constituents of *Datura Fastuosa* was described; the present paper deals with two non-alkaloidal constituents tentatively named daturanolone and fastusic acid which appear to have been isolated for the first time from Datura plant. Daturanolone, $C_{29}H_{48}O_2$, m.p. 273-275°C, $[\alpha]_D^{28} = +50°$ (chloroform), has its characteristic hydroxyl peaks at 3580 cm-[1] (Nujol), 3503 cm-[1] (chloroform) and a ketonic peak at 1700 cm-[1] in the infra-red absorption spectrum.[2] It gives a monoketoderivative of 2,4-dinitrophenylhydrazone $C_{35}H_{52}O_5N_4$, m.p. 232-234°C and a semicarbazone $C_{30}H_{51}O_2N_3$, m. p. 244-245°C. Attempted acetylation of the compound with acetic anhydride in pyridine, even after vigorous refluxing for 7½ hours on an oil bath and its oxidation with chromium trioxide in pyridine could not bring any change in the compound, which indicate that the hydroxyl group is either highly hindered or attached to a tertiary carbon atom. The lithium aluminium hydride reduction of daturanolone in etherial solution yielded a crystalline product, m.p. 233-235°C, which did not give any ketonic peak in the infrared absorption spectrum and the acetylation of this reduced product yielded fine needle-shaped crystals, m.p. 235-236.5°C, having acetate absorption at 1720 cm-[1] (chloroform).

Fastusic acid, $C_{10}H_{10}O_5$, m.p. 202-204°C, dissolves in a dilute sodium bicarbonate solution with the evolution of bubbles, and has its characteristic infrared absorption at 1680, 1592, 1497 and 1474 cm.-[1], as well as U.V. maxima at 291, (ε=2731), 259, (ε=5452) and 205 (ε=3025) mμ. All these indicate that it is an aromatic carboxylic acid. The probability of its being tropic acid (an aromatic carboxylic acid $C_9H_{10}O_3$, m.p. 129°C)[3] may easily be discarded as there are sharp differences in the melting points and the molecular formulae.

Experimental

ISOLATION OF DATURANOLONE

Sun-dried powdered Datura seeds (2.65 kg) were extracted exhaustively in the soxhlet with rectified spirit, and solvent distilled when a viscous, brown coloured, oily material was obtained. This was acidified with 5% sulphuric acid solution and extracted with ether four times. The ether extract was dried, and solvent distilled to yield 215 g of oil (8%) containing other nonbasic materials. It was kept in a refrigerator for over a month when fine needle-shaped, colourless crystals separated at the bottom of the flasks. The crystals (0.530 g; 0.02% of the dry seed) were filtered and purified by four crystallisations (m.p. 273-275°C) from ethanol (Found: C, 81.35; H, 11.09;O,7.58; C-CH_3, 4.55; Act. H^+, O.267%; mol. wt., 399. Daturanolone $C_{29}H_{48}O_2$ requires C, 81.31; H, 11.20; O, 7.71; C-CH_3 (one), 3.50; Act. H^+ (one), 0.233%; mol. wt., 428). It is soluble in ether, chloroform, benzene, ethanol, methanol and insoluble in the low-

boiling petroleum ether. It has characteristic infrared absorption peaks (Nujol) at 3580 (OH), 3030, 1700 (ketone), 1445, 1360, 1250, 1195, 1140, 1050, 970, 918, 815 and 723 cm^{-1}.[2] The compound does not respond to the Liebermann-Burchard test for sterols and it does not appear to be a glycoside as it is not hydrolysed by refluxing with alcoholic hydrochloric acid. When 2,4-dinitrophenylhydrazine was treated with daturanolone in acidic ethanol, brown coloured needles of the 2, 4-dinitrophenylhydrazone derivative separated from the solution. It was filtered and crystallised (m.p. 232-234°C) from ethanol (Found: C, 69.20; H, 8.76; O,12.88; N, 9.05; C-CH_3, 2.40%. $C_{35}H_{52}O_5N_4$ requires C, 69.36; H, 8.65; O, 13.21; N, 9.25; C-CH_3, 2.47%). A semicarbazone derivative of daturanolone was prepared and crystallised (m.p. 244-245°C) from methanol (Found: C, 73.60; N, 8.29%; mol. wt., 510. $C_{30}H_{51}O_2N_3$ requires C, 74.29; N, 8.66% mol. wt., 485).

ISOLATION OF FASTUSIC ACID

Dried Datura seed powder (1.58 kg) was extracted exhaustively with petroleum ether and the defatted drug, in turn, was extracted with rectified spirit at room temperature and alcohol distilled under reduced pressure to yield a thick, dark-coloured mass. It was acidified with 5% sulphuric acid and extracted with ether. The ether-extract, on drying, yielded a brown coloured semi-solid mass (3.10g) which was passed through a column of celitecharcoal mixture. The eighth fraction (eluted with ether) gave an almost colourless solid (0.606 g; 0.022%) which was purified by crystallisation (needles; m.p. 202-204°C) from benzene (Found: C, 56.94; H, 4.72; O, 38.28; Act. H^+, 1.302; no methoxyl group; mol.wt. 192. Fastusic acid $C_{10}H_{10}O_5$ requires C, 57.19; H, 4.80; O, 38.10; Act. H^+ (one), 0.47%; mol. wt. 210). It has infrared absorption peaks (KBr pellet) at 3472 (OH), 2958, 1680 (aryl-COOH), 1592, (aromatic) 1497 (aromatic), 1474 (aromatic), 1449 (aromatic), 1445 (CH_2), 1381 (C-CH_3), 1299, 1234, 1202, 1109, 1026, 915, 883, 818, 805, 764 and 720 cm^{-1}.

Detailed work will be communicated later. The microanalyses were carried out by Dr. Franz Pascher, Microanalytisches Laboratorium, Bonn, West Germany.

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IMPROVEMENT OF PROTEIN VALUE OF COTTONSEED PROTEIN ISOLATE WITH FISH FLOUR AND SKIM MILK POWDER

IFTIKHAR ALI SHAIKH, M. ARSHAD, M. Y. IKRAM-UL HUQ AND S. MAQSOOD ALI

West Regional Laboratories, Pakistan council of Scientific and Industrial Research, Lahore

(Received October 20, 1965)

Introduction

In an earlier communication Ali *et al.*[1] reported the preparation of a protein isolate from the commercial cottonseed cake. It contained 76% protein as compared with 40.60% in the original cake. But since the cottonseed proteins are naturally of low biological value, the protein isolate has to be blended with other proteins to improve its protein value to the level of animal proteins. Previous studies of Ali *et al.*[2,3] have shown that fish flour and skim milk powder greatly enhance the protein value of vegetable proteins. In the present investigation cottonseed protein isolate was mixed with varying proportion of fish flour and skim milk powder with a view to finding the most economical mixture commensurate with maximum efficiency of protein utilization.

Experimental

MATERIALS

The Protein isolate from commercial cottonseed cake was prepared according to the method of Ali *et al.*[1] and the skim milk powder was obtained through the courtesy of Director, Health Services, Lahore.

Fish flour from sun dried fish obtained from Karachi was prepared as follows:

Sun dried fish was soaked in water for about three hours to remove the excess of salt. It was then dried in an air oven at 60° C and then extracted

with light petroleum (b.p. 60-65°C) four times to remove most of the fat. The residue was freed of the solvent by pressing and drying at 60°C for an hour in an oven provided with a motor fan and was finally ground to 60 mesh powder.

DIETS

Cottonseed protein isolate as such or in admixture with fish flour and skim milk powder in varying amounts was incorporated in semi-synthetic diets by the replacement of maize starch in such a manner that the ultimate protein content was about 10%. The protein content (N% × 6.25), of the various concentrates was determined by a semi micro Kjeldhal's method[4] and is given below:

Protein concentrate	Protein percentage
1. Cottonseed protein isolate	76
2. Fish flour	70
3. Skim milk powder	36

The general formula for 1 kg of the diet is given below:

Potato starch	100 g
Glucose	150 ,,
Fat (Star Vanaspati)	150 ,,
Vitamin mixture [5]	50 ,,
Glaxo Salt mixture [5]	50 ,,
Protein concentrate	×
Maize starch	500-×

The quantity (×) of various concentrates and maize starch are shown in Table 1.

METHODS

The nitrogen, calories, protein calories percentage of various diets were determined according to the methods described earlier.[6] Net protein utilization (N.P.U.) value of the various diets was determined by the method of Miller and Bender[7] using male albino rats weighing between 35-40 g. N.P.U. value at 10% protein level were converted to N.P.U. (standardized) using the formula :[8]

$$\text{N.P.U. St.} = \frac{\text{N.P.U.} \times 54}{54\text{-P}} - 8$$

where P = protein cals% in the diet.

Results

The results of the N.P.U. determinations of the various experimental diets are presented in Table 2. The N.P.U. (st) or protein score which is a measure of protein quality independent of protein concentration[7] is shown in the last column.

TABLE 1.—QUANTITIES OF VARIOUS PROTEIN CONCENTRATES AND MAIZE STARCH USED FOR 1 kg OF THE EXPERIMENTAL DIETS.

Mixture Protein source Sl. No.	Protein contributed %	Cotton seed protein isolate g	Fish flour g	Skim milk powder g	Maize starch g
1. Fish flour	10	—	142.86	—	357.14
2. Skim milk powder	10	—	—	289.50	210.50
3. Cotton seed protein isolate	10	131.60	—	—	368.40
4. Cotton seed protein isolate	7.5				
Fish flour	2.5	98.70	35.71	—	365.59
5. Cotton-seed protein isolate	5				
Fish flour	5	65.80	71.43	—	362.77
6. Cotton-seed protein isolate	2.5				
Fish flour	7.5	32.90	107.13	—	359.97
7. Cotton-seed protein isolate	7.5				
Skim milk powder	2.5	98.70	—	69.60	331.7
8. Cotton-seed protein isolate	5				
Skim milk powder	5	65.80	—	139.20	295.00
9. Cotton-seed protein isolate	2.5				
Skim milk powder	7.5	32.90	—	208.80	258.30

It will be observed from the table that N.P.U. st. of cottonseed protein isolate is 41.0% which is shown to be progressively improved by increasing level of fish flour or skim milk powder in the diet. In the case of fish flour supplementation the maximum N.P.U.st. (90.6 %) is given by mixture No. 6 in which the proportion of protein from cottonseed protein isolate and fish flour is 1:3. But in view of higher cost and low platability of the fish protein concentrate as compared with cottonseed protein isolate, mixture No. 5 should be preferred, in which the proportion of proteins from cottonseed protein isolate and fish flour is 1:1. The N.P.U.(st). of this mixture is 82.6 % which is slightly less than that of mixture No. 6 and compares favourably with that of skim-milk powder alone (Mixture No. 2 N.P.U.(st). 84.7%).

In the case of supplementation with skim-milk powder although maximum N.P.U.st. 79.6% is given by mixture No. 9 but it does not differ appreciably from mixture No. 8 (N.P.U. st. 78.6%). Hence the latter will be more economical to produce as it contains much less amount of skim-milk powder as that contained in the former.

TABLE 2.

S. No	Protein %	K. Cals/g	Protein Cals. %	N.P.U. at ca 10 % protein level	N.P.U.(st) %
1.	10.0	4.00	10.0	86.9	98.7
2.	10.0	4.00	10.0	75.5	84.7
3.	10.5	4.29	9.8	40.0	41.0
4.	10.6	4.29	9.9	56.6	51.3
5.	10.0	4.30	9.3	75.2	82.8
6.	10.8	4.28	10.1	80.2	90.6
7.	10.6	4.33	9.8	47.9	50.5
8.	10.0	4.30	9.3	71.7	78.6
9.	10.7	4.29	10.0	71.4	79.6

For the composition of the diets see Table No. 1
Mean value of the duplicates containing four rats each.

Discussion

Protein isolates from oil cakes are receiving increasing attention during recent years due to their high protein content and bland taste. They are also free from insoluble and indigestible carbohydrates which may swell or interfere in the digestion of proteins particularly in children and they are ideally suited as a protein supplement for infants and invalids. In India, a good deal of work has been done in the production and nutritional evaluation of groundnut protein insolate.[9] Attempts have also been made to improve its protein quality by blending it with other protein sources. [10–11] In the present investigation the authors have shown how the protein value of cottonseed protein silate can be improved by adding fish flour or skim milk powder and have indicated the most suitable blends. Production of such concentrates of biological value comparable to that of animal proteins will provide the food industry a cheap source for incorporation into formula diet for infants and invalids.

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OCCURRENCE OF DELPHINIUM KABULIANUM AKHTAR IN WEST PAKISTAN—A NEW RECORD

S.M.A. KAZMI

North Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Peshawar

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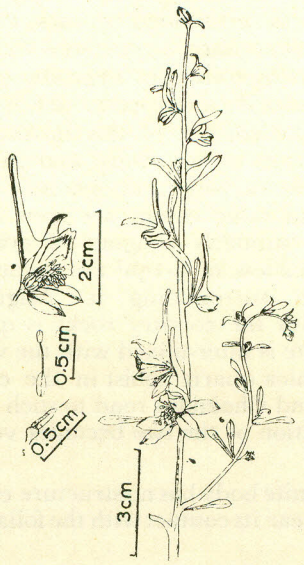
During his tour during the months of April and May, 1965, the author made a collection of about 1,000 plant specimens from Quetta and its suburbs. Among the specimens collected, there were a few specimens belonging to *D. kabulianum Akhtar*, of the family *Ranunculaceae.* The type specimen of the species was collected from Afghanistan, Kabul (altitude: 6,900 feet) near the southern gate of Bagh-e-Baber, where it was growing wild and flowering towards the end of May. The specimens of the species collected by the author on May 8, 1965, were also in flower. These plants were found growing wild at Yaro about twenty two miles north of Quetta (altitude: 5,500 feet) and were thinly scattered over the slopes of low sandy hills.

Delphinium kabulianum has, probably, not yet been reported to occur at any place except that of the type-locality. It is interesting to record the occurrence of this plant near Quetta. Further its occurrence at Yaro shows that the plant has a wider distribution and is not confined to Kabul or its environs.

The author appends a description of the type species in English, adding a diagram (Fig. 1) and a photograph (Fig. 2) which are perhaps, wanting in literature. The specimen figured here is present in the Herbarium of North Regional Laboratories, Peshawar, (West Pakistan) as the Sheet—Kazmi, No. 1485, dated 8th May 1966.



Delphinium Kabulianum Akhtar

Fig. 1.

Fig. 2.

Delphinium kabulianum Akhtar (Fig.) is an annual herb slightly pubescent, erect, about 31 cm high; branches ascendent; leaves alternate, linear, obtuse, approximately 12.5 mm long, 1.5 mm broad; racemes long, terminal, bearing 5-11 flowers; pedicels nearly 6 mm long, curved; flowers bracteate, 1 cm diam. including the horizontal ± reflexed sepals, vertically 7.5 mm diam; sepals 5, yellowish white, longitudinally purple streaked, valvate, at the tips slightly reflexed, pubescent sepals apposite to the axil usually connate at the bases, lateral reflexed, 8 mm long, 2.5 mm broad, spur erect; corolla light violet in the beginning yellowish white later; petals 4, 2 ventral spurred, spur upto 2.6 cm. long; 2 laterals squamiform; stamens, 20-25, included, unequal; filaments white, dilated at the base; anthers, slightly acute, yellow; carpel, solitary, ovary pubescent; style short, pubescent cylindrical; follicles cylindrical, 12.5 mm long, pubescent; seeds 16, biseriate brown, more or less triangular; testa coriaceous, regularly wrinkled.

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A PETROGRAPHIC STUDY OF THE MALAKAND GRANITE

KH. GULZAR AHMAD

5 *E*/9/23, *Nazimabad, Karachi*

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Introduction

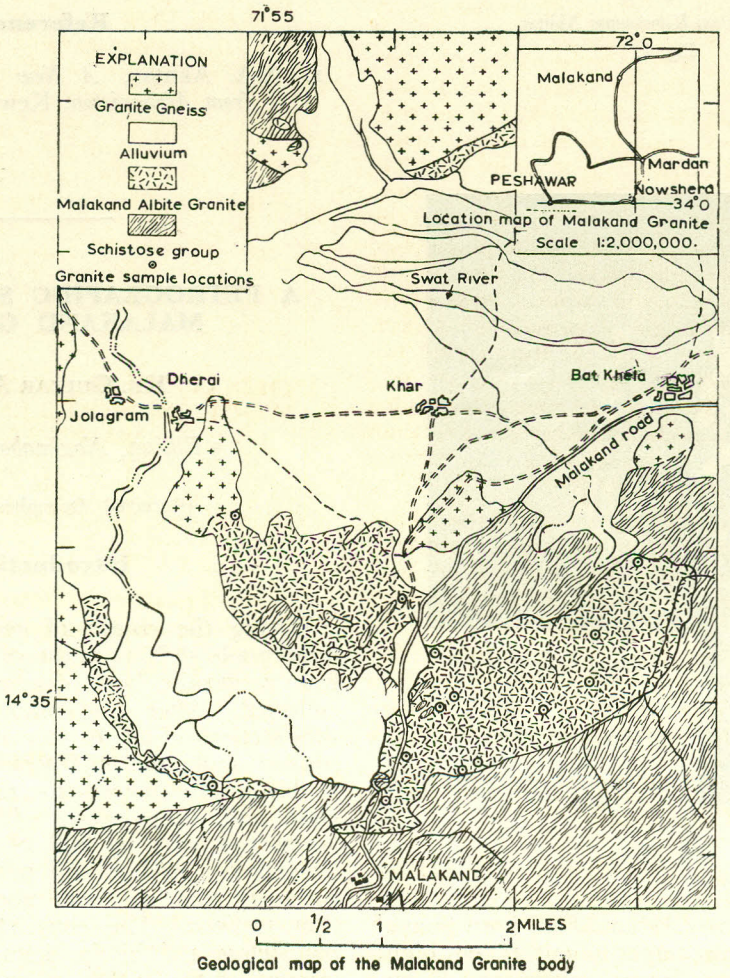
During the course of geological mapping of quadrangle 38 N/14 in the year 1963 some samples from a granite body exposed in the area were collected. After the completion of field work, thin sections of all the important rock types were studied under the microscope. The thin sections of this granite showed that the two felspars orthoclase and albite along with quartz and some accessories make up bulk of the rock. Traces of clinozoisite, epidote and muscovite as dominant accessory minerals were also observed from the thin sections. The complete absence of calcic plagioclase and biotite from the rock raised the interest of the author to study this whole body petrographically.

After finishing the laboratory study of the rock samples of the whole of quadrangle 38 N/14 the author had the opportunity to visit the area for final checking of the contacts of mapped rock units and to collect additional information about the general geology of the area. During his stay in the area, three days were spent on sampling and traversing the Malakand granite. In all fifteen random samples were collected so as to represent all parts of this intrusive body. This granite body is 100 yards north of Malakand (38 N/14) and is exposed on both sides of the Malakand-Chakdara road (Fig. 1). The area is very well connected with Peshawar, Rawalpindi, Nowshera and Mardan.

Geology

The Malakand granite which covers 10 square miles has its northern edge approximately on the northern side of the Swat River where it is seen below the older granite-gneiss body. This granite body which is white, massive, and susceptible to weathering is surrounded by dark, compact, thinly bedded mica-quartz calcareous schist which is richly garnetiferous very near the contact, white, thickly bedded micaquartz schist and granite gneiss. The contact of this intrusive body with the host rocks is very sharp and irregular. Bands of host rocks varying in thickness from 5 to 50 and with considerable extent are seen in the granite mass and similarly tongues of granite material with a thickness of 5-150[1] are encountered in the host rocks surrounding it. Migmatization of granite with the country rocks is quite common. This granite is inter-mixed with the white coloured compact mica quartz schist in the exposures west of Malakand Chakdara road to such an extent that the separation of the two becomes very difficult.

The granite body has no structure except for some foliation near its contact with the foliated host rocks.



Geological map of the Malakand Granite body

Fig. 1.

Petrography of Rock

The thin sections of this granite show that it has 25 to 40% orthoclase, 20 to 40% albite plagioclase, 15 to 30% quartz, 1 to 15% microcline, 2 to 12% muscovite, 1 to 8% biotite (in some samples) 1 to 5% epidote and 1% to 3% clinozoisite with traces of zoisite, chlorite, hornblende, iron oxide, garnet, sphene, and zircon. In two samples single crystal of calcite is also seen. The epidote or clinozoisite is present in all but one of the samples studied under microscope. Four of the thin sections contain small crystals of garnet. Muscovite, zircon, sphene and biotite are the common inclusions in quartz and felspar crystals.

The rock in these samples shows a minor type of cataclastic structure and the quartz is strained. The granite has a generally medium-grained groundmass around phenocrysts of felspars and quartz. The felspar in some thin sections is saussuritized and in one of the samples has a very dirty appearance. The falspar is being replaced by muscovite and quartz. Overgrowths of clear felspar on the older clouded felspar crystals are also seen. The quartz is badly cracked in the thin sections and there is a younger, unstrained quartz which replaces the felspar. The twin lamallae of some felspars have been bent indicating some dynamic metamorphism. The highest average albite twinning normal to (010) of the plagioclase crystals is 14° in most of the thin sections giving the plagioclase an Ab92 An8 Albite Composition. In some samples the angle of twinning has been as high as 18 and as low as 12, thus giving the plagioclase a composition of Ab_{98} An_2 to Ab_{90} An_{10}. In all the samples the refractive index of this mineral has been found to be less than Canada balsam, that is, below 1.54.

The muscovite, chlorite and garnet crystals have a fresher appearance than the other minerals. In a few samples biotite is seen to be altering to muscovite and chlorite. These fresh constituent accessory minerals therefore may be secondary after biotite. The epidote and clinozoisite crystals also have a fresh appearance and are associated with chlorite; they may be the secondary products of anorthite molecules formed by epidotization.

Conclusions

This petrographic study of the granite samples helps to draw the following conclusions:

1. That this granite has only potash and sodium felspar and has no calcium felspar; and therefore it is not a normal granite and can be classified as an "Albite granite".

2. The presence of epidote, clinozoisite and garnet with fresher appearances, the alteration of biotite to muscovite and chlorite, the minor cataclastic structure of the rock, and the replacement of felspar by muscorite and quartz, and strained quartz in all the rock samples go to suggest that this body has been subjected to a sort of autometamorphism which however was not strong enough to greatly alter the rock. The massive, unfoliated structure of this body, with sharp and irregular contact with surrounding well foliated metamorphosed rocks, the effects of contact metamorphism in the contact zone of this mass and the host rocks, the presence of xenoliths of country rocks in the granite body and vice versa, strongly suggest that this granite mass is younger in age than the host rocks. The foliated metamorphosed host rocks in whole of this area were altered to their present form by the regional metamorphism which came with the Himalayan Orogeny. On the basis of the field evidences mentioned above it is concluded that intrusion of Malakand granite took place after the end of the large scale metamorphic activity in Malakand area.

UTILISATION OF MAKRANI (BALUCHI) WOOL IN WOOLLEN AND CARPET MANUFACTURE

TAJ ALI WAZIR, FAIZULLAH KHAN AND A.A. WAKIL

North Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Peshawar

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Introduction

Wakil *et al.*[1–5] first undertook studies on the tensile characteristics of Kaghani and Hashtnagri wool fibres which was followed by Khan, Wazir and Younis.[6] All these authors came to the conclusion that Harnai wool could be utilised in the manufacture of medium to low quality woollen and worsted cloth from a strength point

of view, while Hashtnagri and Bibrik wool are best suited for the manufacture of carpets. The present study also deals with strength characteristics of Makrani or Baluchi wool and its suitability for carpets or woollen fabrics. Forty five samples of wool have been collected from Kharan, parts of Kalat Division and Makran state, which is the home tract of the breed. Fibre samples of 0.06 g. in weight and approximately 2″ in length were sorted into true, heterotypical and medullated wool fibres and tests were carried out on these individually.

MAXIMUM AND MINIMUM VALUES FOR THREE TYPES OF FIBRE.

Type	Fibre strength		Extension (%)		Tensile strength kg/cm²	
	Min.	Max.	Min.	Max.	Min.	Max.
True wool	6.8	16.0	10	43	1188	3565
Heterotypical wool	9.4	25.0	25	46	1069	4158
Medullated wool	4.9	32.0	8	54	237	2732

TABLE 1.—MEAN DIAMETER, SINGLE FIBRE STRENGTH, ELONGATION AND TENSILE STRENGTH OF TRUE WOOL FIBRES.

Mean Diameter 27.2μ.

Diameter (u)	No. of fibre	Breaking strength of single fibre (g)	Elongation (%)	Stress	Tenacity	Tensile strength (kg/cm²)
20.0	298	6.8	28	22.0	2.3	2732
22.0	247	10.1	32	20.8	1.4	1663
24.4	268	13.1	36	28.7	3.2	2801
24.6	325	12.0	28	26.0	3.0	3564
24.8	205	12.0	42	26.0	3.0	3564
26.0	327	13.8	60	24.4	2.6	3088
30.2	193	7.5	30	12.0	1.1	2106
32.2	270	16.0	36	19.5	2.0	2376
32.4	433	9.0	10	11.0	1.1	1188
36.0	312	12.0	43	12.0	1.3	1544

TABLE 2.—MEAN DIAMETER, SINGLE FIBRE STRENGTH, ELONGATION AND TENSILE STRENGTH OF HETEROTYPICAL WOOL FIBRES.

Mean Diameter 33.5μ.

Diameter (μ)	No. of fibre	Breaking strength of single fibre (g)	Enlongation (%)	Stress	Tenacity	Tensile Strength (kg/cm²)
26.4	37	15.0	39	30.1	2.5	2970
28.0	73	9.4	31	50.2	1.6	1900
30.0	98	20.0	39	28.0	3.0	3564
31.2	159	25.0	46	35.0	3.5	4158
32.0	29	10.0	25	13.0	1.2	1425
32.0	272	22.0	46	27.3	2.7	3207
36.0	68	13.0	33.1	13.0	1.5	1797
37.2	47	23.0	27	23.0	2.5	2995
40.0	276	12.0	30	9.0	3.0	1188
42.4	255	11.0	44	8.0	0.9	1069

Experimental

The methods used for the determination of breaking strength and extensions are the same as described by A.A. Wakil [7] in his work on Kaghani wool fibres.

Results and Discussion

Table 1, 2 and 3 give the mean diameter in μ, breaking strength of the single fibres in g, extension at break and tensile strength of true, heterotypical and medullated fibres. These Tables show that there is a marked variation in the characteristics of individual types of fibres.

It is seen that there is no marked difference in the percentage extension of the true and heterotypical wool, but there are variations in the elongation of medullated wool. However, the strength of heterotypical wool is the highest while that of medullated wool is the lowest. True wool takes the intermediate portion.

The relationship between fibre diameter and breaking strength for all three types of wool is shown in the Tables 1, 2 and 3. In the case of true wool, as the diameter increases, the strength also increases and it is at a maximum at a diameter of approximately 36μ. With the heterotypical wool, the tensile strength is at a maximum at 37μ diameter and decreases onward.

The breaking strengths of true and heterotypical wool fibre itself closely resembles each other, while Makrani medullated wool resembles with the Kaghani, true and medullated wool with respect to elongation and breaking strength.

A soft wool having a minimum of 25% of elongation is desirable for weaving apparel cloth. The extension at break of true, heterotypical and medullated wool is approximately 32%, which is above than that prescribed for apparel coth and may be utilised in the manufacture of carpets.

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TABLE 3.—MEAN DIAMETER, SINGLE FIBRE STRENGTH, ELONGATION AND TENSILE STRENGTH OF MEDULLATED WOOL FIBRES.

Mean Diameter 60.5 μ.

Diameter (μ)	No. of Fibre	Breaking Strength of single fibre (g)	Elongation (%)	Stress	Tenacity	Tensile Strength (kg/cm²)
39.4	43	20.0	36	16.8	1.6	1896
39.4	28	11.0	8	8.7	1.0	1188
41.6	158	17.0	17	12.0	1.3	1534
43.4	45	32.0	36	21.0	2.3	2732
50.2	268	6.0	25	2.6	0.3	356
51.2	118	4.9	29	2.3	1.6	1900
54.0	145	20.0	54	9.0	0.9	1086
64.0	180	14.0	11	4.3	0.5	554
92.0	75	20.7	30	3.0	0.3	356
100.0	37	18.5	31	2.3	0.2	237

TABLE 4.—CHARACTERISTICS OF DIFFERENT TYPES OF PAKISTANI WOOL.

Type of Breed	Distribution of fibres		Diameter (μ)	Stretched fibre length	Tensile strength (kg/cm²)
Kaghani	True	70.12	26.13	2.43	2492
	Het.	24.23	41.08	3.22	1518
	Med.	5.64	56.77	2.29	1013
Harnai	True	65.0	24.7	4.1	1285
	Het.	22.1	40.5	6.2	1342
	Med.	12.8	70.9	5.2	984
	Kempy	0.1	—	—	—
Hashtnagri	True	54.0	25.2	4.3	1590
	Het.	25.0	40.2	6.0	1378
	Med.	21.0	51.8	5.7	1057
Bibrik	True	43.2	25.2	2.20	2885
	Het.	35.0	45.6	3.25	1476
	Med.	20.7	68.8	3.00	1188
Makrani	True	59.88	27.2	2.9	2462
	Het.	24.22	33.5	1.9	2627
	Med.	15.33	60.5	4.3	1182

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BOOK NOTICES

The Design of Reinforced Concrete. Henry J. Cowan and Peter R. Smith, 544 pp. Angus & Robertson Ltd., 54, Bartholomew close, London, 1963. Price 105s.

Australian practice in reinforced concrete construction has never fully conformed to either British or American rules. In time a distinctively Australian design procedure has evolved, which leans on American, British and Continental precedent, but differs from all of them.

The book is intended to provide a presentation of the elements in reinforced concrete design in accordance with the new S.A.A. Code for Concrete in Buildings. This code introduces a number of new methods, notably design based on ultimate strength and design based on model analysis. Rules for the design of members, subject to torsion, appear for the first time, a more scientific approach to the determination of the compressive strength of concrete has been adopted, and there are numerous minor changes. This book should be helpful to designers studying the implications of the new code, as well as to students of civil engineering and architecture, for whom it is primarily intended.

Mechanizing Laboratories. E.A. Smith. 205 pp. Iliffe Books Ltd., Dorset House, London, 1965. Price 63s.

It is now generally accepted that if a national industry is not to lag behind its foreign competitors—or if a company is to compete successfully at home or abroad—the utmost possible effort must be put into research and development.

The best use must therefore be made of the trained scientists available, and the present author's theme is that the time of highly qualified men must not be wasted in doing routine jobs which could be done better, much more quickly, and usually more economically, by a machine or instrument. In these days the mechanisation of laboratory tasks is as important as mechanising production lines.

The author stresses that the slogan of every laboratory should be speed with efficiency, and he gives examples of where the time for certain repetitive tests has been cut from hours, or even days, to a matter of minutes. In fact, in certain industrial control laboratories, unless results can be obtained rapidly they are virtually useless.

The purpose this book fulfils is therefore twofold. First, it surveys the general range of mechanical aids and instruments available so as to encourage the scientist or technologist to choose those most suitable for his purpose and, where necessary, to borrow ideas from fields other than his own; secondly, it provides management with information on what is being achieved in science, technology and industry in the way of mechanising laboratory methods and recruiting instrumental aids to increase efficiency.

Economic arguments are put forward in order to show that expenditure on laboratory equipment can be a real investment, not only leading to faster and often more accurate bench work, but also producing, in this age of high labour costs, the desired results more cheaply—particularly where it allows high level work to be carried out by non-graduate staff.

A well equipped laboratory is not, however, an end in itself, and the author therefore discusses what in general terms the aims of the laboratory