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Bulletin of the

CLRI Madras

Nov 1957

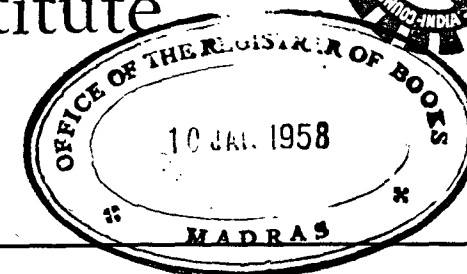


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Bulletin of
The Central Leather
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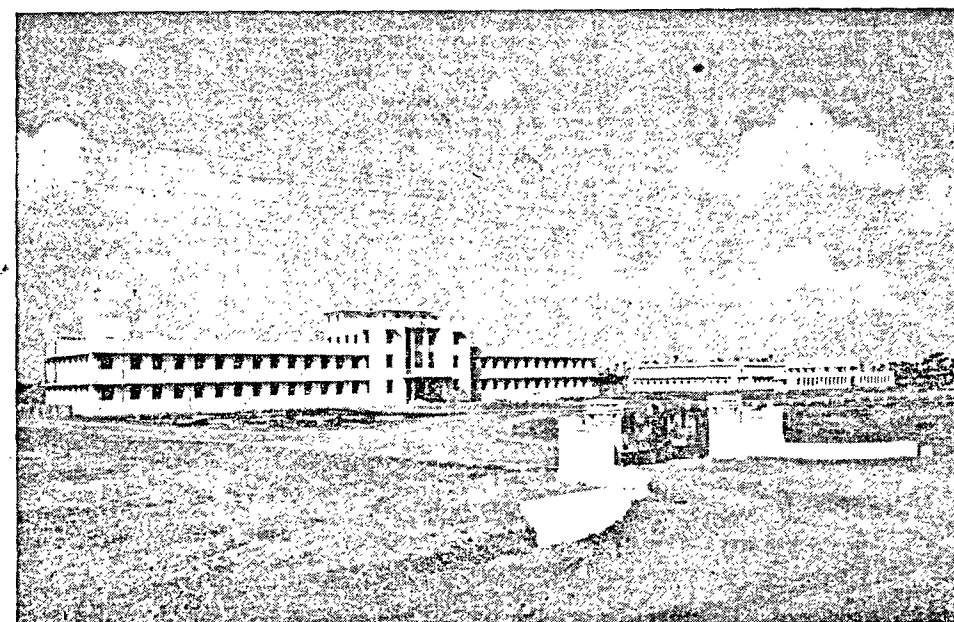


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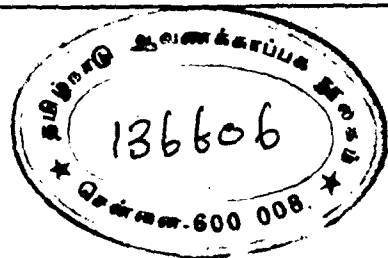
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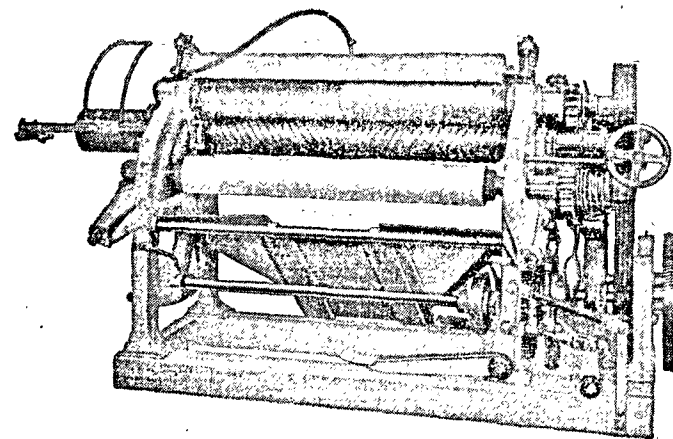
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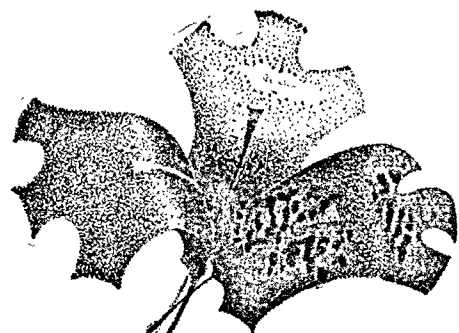
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**STUDIES ON INSECT DAMAGE TO HIDES
AND SKINS—II**

By the beetle *Attagenus gloriosae* (Fabricius)

R. BHASKARAN & S. N. SEN

SUMMARY

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The nature of damage caused by the larval forms of *Attagenus gloriosae* to hides and skins is described.

Different kinds of beetles have been recorded on hides, skins and on leather from time to time, causing some kind of damage. Of these, the damage caused to the leather industry by the beetles of the *Attagenus* species appears interesting. Members of this species have been recorded on furs, hides, skins, leather, as well as on some vegetable tanning materials, like Myrobalans and Divi-Divi pods. *Attagenus piceus* has been recorded on kid leather,¹ hides, skins and furs,² while *Attagenus byturoides* has been recorded on leather.³ *Attagenus pello*, the 'furriers' beetle was said to occur on pelts and skins.⁴ Hinton stated that a few *Attagenus alferi*⁵ were found on myrobalans loaded in India. The same author noted that the beetle *Attagenus gloriosae* was imported into England with cargos of Divi-Divi pods, celery seed-bags, ground nuts and fleshings from India.⁶

Recently, this beetle — *Attagenus gloriosae* — was seen on hides, skins and finished leather with hair on, causing damage to them, and was, therefore, taken up for the study of the nature of such damage.

The larval forms of this beetle were first noticed on flint and drysalted cow hides, and skins, and also on chrome tanned leathers finished with hair on, on which they were found on the hairy side. In the process of feeding which

was mostly restricted to the hairy side, the epidermal tissue was eroded off, involving the grain layer also. This resulted in the production of depressed map-like scars on the hairy surface, exposing the corium below (Fig. 1). In some places, much deeper excavations were also seen. The larvae were particularly seen along the borders and folds of hides, as these appear to give a suitable lodging place.

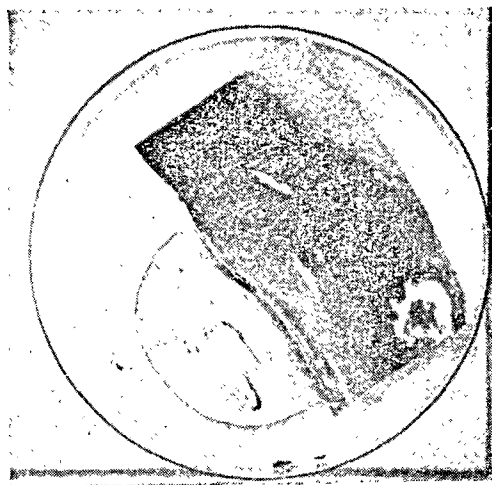


Fig. 1

The larvae are brownish in colour with transverse lighter coloured bands at the posterior part of each segment, giving a beaded appearance. The last tail segment bears a tuft of backwardly directed hairs.

These larvi, when maintained on hides at room temperature, developed into pupae. The pupae were whitish in colour with brown bands on the dorsal aspect. The pupae period was about 9 days, after which the adults emerged. Immediately after emergence, the adults were of a light brown colour, which darkened on exposure to light. This beetle can be easily identified with the help of the whitish band running across the upper surface of its elytra or upper wings. The head and thorax are covered by testaceous

hairs, so also the white band running across the wing. The rest of the body is covered with blackish hairs. (Fig. 2).

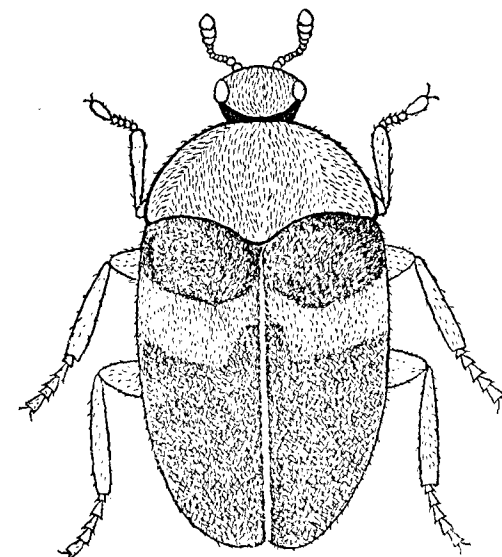


Fig. 2. *Attagenus gloriosae* (diagrammatic)

The adults, when left in contact with the hides, did not cause any damage, similar to those produced by the larvi.

Acknowledgement: Thanks are due to Mr. V. S. Krishnamurthy for help in drawing the picture.

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DRY SALTING OF SKINS AND HIDES

J. B. INGRAM & K. V. VAIDYALINGAM

(*The Skins & Leathers (Private) Ltd., Coimbatore*)

The aim of the Indian Tanning Industry should be to process all available raw hides and skins into finished leathers either for local consumption or for export abroad. The livestock is distributed all over India and the hide and skin production centres are not always near the tanning centres. The raw material has to move over long distances before it reaches the tannery and has to be preserved till then. The hides that are received from Malabar are very badly cured and in most cases are unfit for chrome tanning. As a major portion of the hides and skins emanate from the villages, it is necessary to educate the villagers the proper methods of curing and preservation so that the valuable raw materials do not putrefy even if there is delay in transport. The various Government agencies have already taken up the programme of training village chamars in the proper methods of flaying and it will be in the interest of the industry if the proper methods of curing are also taught to the villagers.

Experiments were conducted to work out a simple but effective method of dry salting goat skins in our tannery and the results have been found to be very encouraging. The details of the process of curing are given below.

Raw materials: 50 green goat skins of average weight of 2.42 lbs. and prime quality of Coimbatore origin were taken for the experiment.

Process: The skins were opened and worked over the beam once to remove the thick layers and fat on the flesh

side and also to open the fibres a little. In the case of cow-hides it is better to do the beaming after washing them thoroughly.

The skins were soaked in 2% salt solution for about one hour and then washed well in three changes of plain water. They were then soaked in a liquor made up as follows:

Water	..	200 per cent on green wt.
Salt	..	40 per cent „
Sulphuric acid (commercial)	..	4 oz. per cent „

The next day, they were hauled up, dried and then salted on the flesh side with the following mixture of salt.

Powdered salt	..	10 lbs.
Corilene DG or Teepol or any other wetting agent	..	1 oz.
Water	..	1 oz.

After salting the skins were folded and left overnight on a sloping floor to enable the excess water to drain off. Next day, they were salted a second time with the same mixture of salt and folded and left overnight. The skins were then dried in the shade and when nearly dry, were piled flat. The next day, they were dried completely in the morning sun, trimmed and brushed on both sides.

The percentage yield by weight by the above process was 41.2% which is less than the weight usually specified in the trade at present. But the yield of area per pound was correspondingly higher. The approximate cost of dry salting skins by the above method is given below:

1. Wages per 100 skins	..	Rs. 3.06
2. Materials	..	Rs. 7.00
3. Supervision & overheads	..	Rs. 9.94
.	..	Rs. 20.00

Half a dozen of the above skins representing the average lot were kept stored in a shed for exactly two years. It was found after two years that the skins had not developed any putrefaction or red-heat and they were still pliable unlike the usual dry-salted goat skins which, when folded, fold sharply along straight lines. The hair was tight, clean and of good lustre.

It was easy to soak back these skins in plain water. These skins were processed for Chamois leather and also chrome tanned leather along with normal wet salted skins. There was no visible difference in the character of grain or the feel and no loss of substance either.

The above process may easily be adopted for calf skins and cow hides also. This method can be followed with slight modifications, if necessary, while carrying out curing on a large scale.

A NOTE ON THE TANNING PROPERTIES OF HEXAMETHYLENE TETRAMINE-PHENOL COMPLEXES

Hexamethylenetetramine is known to form a variety of molecular complexes due to its weak tertiary basic nature. Such complexes are believed to be involved in Duff reaction,¹ Sommelet reaction,² Delepine reaction³ and the conversion of indoles to indole-3-aldehydes.⁴ The complexes of phenols with hexamethylenetetramine are crystalline solids which decompose on heating to give a resin and ammonia. Hexamethylenetetramine is a stable compound under ordinary pH conditions. However, the compound is rapidly hydrolyzed by treatment with hot mineral acids and more slowly with organic acids.⁵ In view of the various functional groups present in collagen it was considered desirable to study its reaction with hexamethylenetetramine to find out whether the formation of complexes from hexamethylenetetramine and the various functional groups of collagen will effect tanning.

It was found that, while hexamethylenetetramine has got little or no tanning properties, the molecular complex formed by mixing equal amounts of saturated solutions of resorcinol and hexamethylenetetramine was found to be a good tanning agent. The tanning was found almost independent of pH from 7 downwards and the quality of leather produced was found good with a shrinkage temperature of 98°C. Above pH 7 the complex decomposes rather rapidly to give a red resin and ammonia and consequently no tanning is observed above pH 7. While similar complexes prepared from hexamethylenetetramine with catechol and pyrogallol was found to possess good tanning properties the

complex from hexamethylenetetramine with phenol was found to have no tanning action.

In this connection, it was also deemed of interest to study the mechanism of this type of tannage and to draw comparison between phenol-hexamethylenetetramine and resorcinol-hexamethylenetetramine complexes.

The tanning by resorcinol-hexamethylenetetramine may be either due to the formation of complex salts with certain groups of collagen or it could be due to the formation of chemical compounds involving the breaking down of the complex.

Nitrogen estimations of the complexes showed that they are equimolecular compounds of the phenols and hexamethylenetetramine. A method has been developed to analyse the complexes taking advantage of their decomposition to the phenols, formaldehyde and ammonia with hot mineral acids.

In the case of resorcinol-hexamethylenetetramine tannage by analysing the leather produced and the waste liquors it was found that some amount of the complex is broken down to affect tanning. The fact that the complex breaks down in presence of the pelt indicated that there is some strong tendency to form a chemical compound between resorcinol, formaldehyde and some groups of collagen (hexamethylenetetramine is known to behave as a donor of formaldehyde under compelling conditions.⁶) Ammonia was excluded since it was recovered unchanged from the waste liquors.

It has been suggested by Marvel⁷ that resorcinol and formaldehyde combine with the primary or secondary amino groups of collagen by a Mannich type of reaction. In the light of this observation, it was considered probable that such a type of reaction taking place in this tannage. Indeed, it was soon found that while hexamethylenetetramine and

phenol-hexamethylenetetramine complex does not suffer any change when treated with glycine, the resorcinol-hexamethylenetetramine complex is broken down to resorcinol, formaldehyde and ammonia. This explains also why only the latter has got good tanning properties. The fact that there was no fixation when resorcinol-hexamethylenetetramine complex was treated with acetylated collagen seems to support the view that amino groups of collagen are involved in this type of tannage.

There is a wide disparity in the properties of these two complexes. While the phenol hexamethylenetetramine complex is very stable both in boiling water and in boiling 50% acetic acid, the corresponding resorcinol complex undergoes rapid polymerization on keeping it in moist condition, boiling with water and boiling with 50% acetic acid for 30 seconds. The decomposition is invariably accompanied by liberation of ammonia and the resins formed are coloured yellow, brown or red depending upon the conditions of decomposition. The reactivity of the resorcinol-hexamethylenetetramine complex is probably due to the tendency of resorcinol to combine with formaldehyde alone to form polymers or with formaldehyde and primary or secondary amines to effect tanning. This tendency is undoubtedly responsible for breaking down of the complex.

In conclusion two competing reactions are possible when an aqueous solution of the resorcinol-hexamethylenetetramine complex is treated with pelt. One is the reaction of the primary and secondary amino groups of collagen with resorcinol and formaldehyde and the other is the breaking down of the complex by water with the formation of resins. The former reaction seems to be faster in view of the fact that no resin is formed when the tanning is carried out. As it was thought the primary and secondary amino groups of collagen are incapable of breaking down the complex, and tanning is effected by reaction with resorcinol and formaldehyde formed by decomposition of the complex with water,

dehydrated pelt was subjected to treatment with resorcinol hexamethylenetetramine complex in alcoholic medium under similar conditions. Since no tanning was effected, it seems probable that water is essential for this type of tannage.

Further work is in progress for utilization of hexamethylenetetramine in tanning operations and it is hoped the full details could be published soon.

Biochemistry Laboratory,
2nd Sept. 1957.

S. RANGANATHAN,
S. M. BOSE &
Y. NAYUDAMMA,

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RESEARCH BEARS FRUIT

Practical Demonstration No. 7

Process No. 1

Manufacture of East India Tanned Kips using a blend of Karada and Konnam barks in place of Mimosa bark and extract.*

K. RAJABATHER

Raw Material: Wet salted cow hides.

Soaking: The hides are weighed after removing the excess of salt from them. They are soaked in plain water for a period of three hours and then washed in four to five changes of water.

Liming and Unhairing: 20 to 25 per cent of slaked lime on the wet salted weight of the hides is added to once used straight lime liquor, i.e., the liquor from which the hides of the previous lots have been taken for fleshing. Sometimes this quantity of lime may be added in two equal instalments during the period of first liming, i.e., before unhairing the hides. They are then piled in the pit keeping the flesh side up except the outermost one and left for a period of one hour. Then they are hauled and put back in the pit after stirring the lime liquor and left overnight. The hides are hauled and put back in the pit after stirring the lime liquor once every day for a period of 7 days. On the 8th day the hides are not handled. On the 9th day the hides are unhaired and put back in the same liquor until all the hides are unhaired.

Reliming: The unhaired hides are relimed in a fresh bath with the addition of 7 to 10 per cent of slaked lime on

* Process demonstrated during the months of August and September 1957.

the weight of the wet-salted hides. The hides are left in the new lime liquor for three days or so during which period they are handled daily and put back after stirring the lime liquor. But this time all the hides are merely thrown into the pit keeping the flesh side up. On the fourth day, the hides are taken out of the lime liquor and put in plain water overnight. Next day they are fleshed by hand over beam with fleshing knife and weighed. Then they are fleshed a second time with a scudding knife. The hides are then washed by trampling and scudded. They are washed again by trampling and left overnight in plain water.

Deliming : Next day the hides are fleshed a third time with a scudding knife. They are then trampled in a pit containing fresh water and scudded. Then they are washed by trampling and scudded two more times. Finally the hides are treated with $\frac{1}{4}$ to $\frac{1}{2}$ per cent Sulphuric acid, 3 to 6 per cent common salt and sufficient quantity of fresh water for a period of 15 to 30 minutes. The hides are sometimes washed and sometimes they are taken to liquors without washing.

TANNING

First Bark : Always some old bark liquor is to be used initially with the addition of fresh barks or a mixture of barks and extracts. The old bark is first removed from the pit and then the clear liquor is decanted to another pit to which fresh water is added. Calculated quantity of bark or a mixture of barks equivalent to about 9 per cent tannins is soaked in the above liquor on the day the hides are taken for fleshing. This 9 per cent tannins is based on the fleshed weight of the hides and represents about 50 per cent of the total quantity of the tannin required for the entire tanning which is about 18 per cent on the fleshed weight of the hide. If a mixture of Karada bark and Konnam or Babul bark

are used, they are blended in the following proportions and first and second barks are used on the fleshed weight:

Babul or Konnam bark equivalent to 3 per cent tannin on the fleshed weight.

Karada bark equivalent to 6 per cent tannin.

(*Note* : Both Konnam and Babul bark contain 12 per cent tannin and Karada bark contains 24 per cent tannin).

When Konnam and babul barks are used, it is advisable to cut them into small pieces if they are very big and soak them in water. Sometimes, the barks, if they are uncrushed, are immersed in water and taken out giving 2 to 3 changes of water for cleaning them from adhering dirt etc. The cleaned barks are then soaked in liquor as mentioned above and the liquor is stirred twice or thrice during the period of two days.

The delimed hides are dipped in the liquor one by one moving to and fro for a few minutes and then immersed by pressing the hides to the bottom of the pit. Immediately after 5 to 10 minutes, they are hauled up and put back. Again after 15-30 minutes, they are hauled up and put back in the pit keeping half the bark at the bottom and remaining half on the top of the hides. Next day, at about 8 A.M., the hides are hauled and put back in the liquor. If the hides appear to be pulled down to an undesirable extent when hauled, they should be paired grain to grain and piled in the liquor sprinkling the bark on the flesh side of each pair. On the third day, the hides are beamed on the flesh side and put back in the same liquor. After beaming, they are hauled up and put back in the liquor by keeping bark in between each hide and left for 5 to 6 days. The goods are hauled and put back once daily during these days as above. On the last day, the hides are taken to the second bark.

Second Bark : The quantity of bark used is equivalent to about 9 per cent tannin on the fleshed pelt weight. The

blends are taken in the same proportion as in the first bark. The mixture of tanning materials should be soaked in old liquor a day or two before use. As in the case of first bark, it is advisable to cut the bark into small pieces if they are big pieces, cleanse and wash before it is soaked to prepare the tan liquor. The hides from the first bark are beamed and then wrung by twisting to remove water and piled in the second bark liquor. After 10 to 15 minutes, they are again hauled and put back one by one sprinkling the bark over each hide. Next morning, they are hauled up and put back in the liquor sprinkling bark over each hide. The goods are left in this liquor for 7 to 8 days during which period the hides are daily hauled and put back sprinkling bark over each hide. If the hides are flacid during this period, they should be beamed and left in the liquor with the addition of little bark. Otherwise, no extra bark need be added. On the last day, the hides are kept in piled condition in the liquor without keeping any bark in between each hide. But half of the bark should be kept at the bottom of the pit and remaining on the top. Next day the piled hides are left undisturbed.

Bleaching : Next day the hides are rinsed in water, bleached with Perfectan O. Again, they are rinsed in water and beamed.

MYROBING

First Myrobing : On the previous day, crushed myrobalans of good quality, calculating the quantity at $\frac{1}{4}$ lb. of myrobalan for every pound of expected leather yield which may be taken as an average yield of 40 per cent on the fleshed weight of the pelt, are soaked in hot water overnight. The myrobalan liquor is now diluted to the required barkometer strength with cold water. The beamed hides are dipped in the myrobalan liquor one by one and placed in another tub or pit which should be near to it. When all the hides are so dipped and piled, after five to ten minutes, they are hauled up and put back in another tub or in the same

tub pouring the myrobalan liquor on the top of the pile of hides. Next day, the hides are hauled up and put back as was done on the previous day. Now the same quantity of crushed myrobalans is soaked in water in a separate tub as before and left overnight.

Second Myrobing : Next day, the hides are wrung and beamed and put in the myrobalan liquor just as in the case of first myrobing. Next day, they are hauled and put back as before. The following day, the colour of the hides is to be examined. If the hides are found to be having too much of myrobalan colour, they should be rinsed either with water or with the same myrobalan liquor diluted with cold water. The hides are then beamed and taken for oiling.

Oiling and Finishing : The hides are piled with flesh side up and applied a solution containing 3 to 4 per cent of epsom salt (calculated on 100 lbs. of expected leather weight.)

The hides are left for a period of half an hour and then oiled on the grain side with pungam oil and piled. Now a syrup solution is prepared by mixing $1\frac{1}{2}$ to 2 per cent of corn flour and $1\frac{1}{2}$ to 2 per cent of glucose for every 100 lbs. of expected leather. This solution is applied on the flesh side of each hide and then each hide is hung up to sam. When the hung up hides are in semidry condition, they are piled and left overnight. Next day, they are set on the flesh side on wooden plank or table with a stainless slicker or with a brass slicker. If white flesh side is required, a solution of corn flour is applied using $1-1\frac{1}{2}$ lbs. of cornflour for every 100 lbs. of expected leather. The pasted hides are set on the grain side and hung up to semi-dry condition. The semi-dried hides are slicked on the flesh side, and again dried a little. Then they are slicked on the grain side and dried completely. The dried hides are lightly slicked on the grain to produce gloss. The hides are then trimmed and sorted into grades.

TECHNICAL SEMINAR

The Manufacture of Glace kid Leather *

Shri J. C. Deb was deputed by the Institute to the United States under Point Four Programme for advanced training in the manufacture of glace kid leather and leather finishes.

During his visits to a number of plants, the following observations were made :

Glace kid industry in the U.S.A. is dependent on India to a great extent for the supply of raw material, viz., dry-salted goatskins. The glace kid tanneries are considerably big in size with a soaking capacity of 10,000 to 25,000 skins per day. All the operations for processing are completely mechanised. In addition to it, chemical manufacturing organisations and research institutions supply the necessary advanced knowledge as well as newer type of quality auxiliaries for making rapid progress in producing high quality glace kid. The tendency is to make leathers by one-bath tanning in preference to the old two-bath tanning though two-bath tanning is practised in some places. The leathers are not nailed or toggled after staking as it produces thinness and flatness. Seasoning and finishing are done by automatic machines.

The marketing of the kid leathers is well organised and the "Kid Leather Guild" helps to publicise and popularise the kid leathers and their products, to ascertain the views of the public and to set latest fashions in vogue. Publicity

* Summary of the seminar held at the Institute.

is also obtained through seasonal exhibitions. The progress of this industry may mainly be attributed to the well-coordinated efforts of the tanners, the leather auxiliary firms and the marketing firms.

Mr. Deb is of the sincere opinion that good quality glace kid could be made in India with advantage because of the quality raw material available in India and a joint Indo-American venture may prove fruitful in establishing glace kid industry in India. The processing methods and other technical know how could readily be supplied by the Central Leather Research Institute to the Indian tanners.

CLASSIFIED ABSTRACTS

PROCESSES PRIOR TO TANNING

INVESTIGATIONS ON BATING III: THE MECHANISM OF BATING. G. H. Green, *J.S.L.T.C.*, 41, 7, 222 (1957).—The following series of changes is proposed to account for the known effects of bating on the pelt and on the leather:

	lime	heat	trypsin
Collagen	progelatin	gelatin	
		low molecular weight peptides	

Progelatin is equivalent to the β collagen of Marriott and the filamented collagen of Borasky and Rogers. These transformations are confined to the fibre surface, but as liming splits up the fibres, additional surfaces become available. Unless the progelatin is removed, the fibres restick when the leather is dried thus nullifying the beneficial effect of the liming. Because progelatin resists proteolysis it must first be converted into gelatin by thermal denaturation. This explains why bating is ineffective below 35°C unless the skin is pre-heated to 40-45°C. Degradation of the progelatin is unnecessary provided it can be removed mechanically. This can be achieved to some extent by scudding alone; this gives better results with calf than with goatskin because of the more open structure of the former. The rate of bating is controlled (at normal bating temperatures) by the penetration of the enzyme into the skin, the difference in case of bating of calf and goatskin again being due to differing compactness of structure. Bating affects mainly the grain layer owing to its fine fibres and close weave which facilitate sticking.

Liming hydrolyses the amide groups of progelatin, and lowers the iso-electric point to about pH 5. Thus some swelling is retained on deliming to pH 8 because of mutual repulsion of fibres with a nett negative charge on their surface. The removal of the progelatin, either by bating or scudding, restores the neutrality of the fibre surfaces and the skin "falls". Falling is associated with the development of flacidity and compressibility, and permeability to air; whilst reduced constriction of the hair follicles loosens the

hair residues. Similar effects to bating can be obtained by methods which prevent the leather fibres from sticking on drying, e.g., fat-liquoring, drying under tension, or acetone dehydration, but then the effect is easily reversed. Reliming after bating produces more progelatin and renders rebating necessary. Straight lime does not immediately inactivate the bating enzymes which greatly accelerate the liming action and formation of progelatin. It is suggested that trypsin, like lime, behaves as a hydrogen bond breaker, and lowers the shrinkage temperature of progelatin. Skin un-haired by sweating or proteolytic enzyme appears superficially like bated pelt, but the leather therefrom is more characteristic of that from limed pelt. This is because comparatively little splittings of the fibres has occurred. The optimum pH for bating of a number of proteolytic enzymes coincides with the optimum for gelatinase activity.

INVESTIGATIONS ON BATING—IV: THE IMPROVEMENT IN BATING ACTIVITY OF A BATE LIQUOR WITH CONTINUOUS PARTIAL RE-USE. G. H. Green, *J.S.L.T.C.*, 41, 9, 303 (1957).—The gradual improvement in bating activity which a freshly made bate liquor undergoes with continuous partial re-use seems to be due to the development of particular species of bacteria. A number of bacteria, mainly *Bacillus* and *Micrococcus* spp. have been isolated from used bate liquors and characterized.

INVESTIGATIONS ON BATING—V: THE INFLUENCE OF LIME BLAST ON BATING AND SCUDDING. G. H. Green, 41, 9, 309 (1957).—Lime blast, due to microscopic particles of calcium carbonate in a skin, is the principal cause of roughness of the grain of a delimed skin. Its loss during bating, due to depletion of the skin and possibly partly due to dissolution, is the main reason for the development of silkiness. The presence of lime blast makes scudding of the delimed or bated skin more difficult. It also impedes the bating of goat skin but not of calf skin. Scudding can replace bating with calf skin and wool sheep skin but only partly with goat skin and hair sheep skin. The improvement in bating obtained by continuous partial re-use of a bate liquor, as related previously to the quality of the bated pelt (Part IV) has now been related to the crust leather.

INVESTIGATIONS ON BATING—VI: THE INFLUENCE OF COLLOIDAL ENZYME ADSORBENTS. G. H. Green, *J.S.L.T.C.*,

41, 9, 321 (1957).—Pieces of calf and goat skin have been bated with trypsin in the presence and absence of kaolin, the concentration of trypsin in solution being the same. Any improvement in bating due to the presence of kaolin could be attributed to adsorbed enzyme being released by the kaolin during bating. Doubt is cast on Wood's theory that enzyme may be carried into the skin in higher concentration than normal though being adsorbed on colloidal particles, and so improve the bating action.

VEGETABLE TANNING

A COMPARISON OF THE RESULTS OF ESTIMATING BLACK WATTLE TANNIN BY THE OFFICIAL HIDE POWDER METHOD AND BY A PROPOSED ULTRA-VIOLET SPECTROPHOTOMETRIC METHOD. C. G. Gordon-Gray, *J.S.L.T.C.*, 41, 8, 269 (1957).—In order to compare the Official Hide Powder method of tannin estimation with a proposed ultra-violet spectrophotometric method extracts made from 94 black wattle bark samples, as well as a number of green and silver wattle samples, were analysed by the two methods. Agreement in actual values for tannin was obtained within 1.0% in 60 samples of black and 1 sample each of green and silver wattle, but greater differences were noted in the remaining 54 samples. These differences do not bear a constant relationship to the Official method. Procedures are detailed for ensuring the concordance of results among laboratories using the U. V. method. Results obtained by Forestal Laboratories, Harpenden, and the Wattle Research Institute, Pietermaritzburg, are recorded.

THE ESTIMATION OF TANNINS IN BLACK WATTLE BARKS AND COMMERCIAL "MIMOSA" EXTRACTS, PART II, STATISTICAL COMPARISON OF THE OFFICIAL HIDE-POWDER (SHAKE) METHOD AND PHOTOMETRIC METHODS OF TANNIN ANALYSIS. D. G. Roux, *J.S.L.T.C.*, 41, 8, 275 (1957).—Statistical comparison is made of results obtained from the same extract solutions by the ultra-violet spectrophotometric and official shake hide-powder methods of tannin estimation. Analysis of variance of Gordon-Gray's 94 duplicate bark analyses shows that differences between methods are not significant. Differences obtained on duplication of analyses on the same samples by the same methods are much smaller than differences between methods. Both the U. V. and the hide-powder of tannin estimation and the extraction procedure are shown to be highly reproducible

for bark analyses, but pronounced skewness is evident for the hide powder method ($g_1 = -0.30$) compared with the U. V. method ($g_1 = +0.02$), when using the same solutions for analysis. The observed skewness for the hide-powder method, although influencing differences between methods is shown not to be significant. Comparison of analyses by each method over 132 bark samples (varying from 17.41% and averaging 33% tannin content) shows high overall agreement between methods ($r = 0.961$) but individual differences as high as 3 and 4% in actual value in certain instances ($s = \pm 1.26$). A standard deviation of errors due to different methods ($s = \pm 1.05$) is calculated, confirming that inherent differences between methods are mainly responsible for observed variations, while experimental errors incurred during estimation contribute a minor but appreciable proportion of observed differences ($P = 16\%$ for differences $> 1\%$ in actual value). Regression equations based on bark analyses only, are calculated for each method. Comparative commercial extract analyses show high correlation between methods ($r = 0.977$) and low standard deviation ($s = \pm 0.63$). The high correlations for bark analyses over the range 20-40% tannin content, and also for extract analyses of 60-70% tannin content, show that excellent overall agreement between methods exists over a wide range of tannin values, in spite of relatively wide differences in a low proportion of individual bark analyses. The adoption of the ultra-violet method as useful alternative to the official hide-powder procedure would therefore, appear to have little overall effect on black wattle tannin estimation. PART III. A CRITICAL EXAMINATION OF THE OFFICIAL HIDE-POWDER METHOD. D. G. Roux, *J.S.L.T.C.*, 41, 8, 287 (1957).—Statistical comparison of the official hide-powder method of tannin estimation with the ultra-violet photometric method emphasises the excellent overall agreement between methods, but also shows that in a low percentage of analyses inherent differences between the methods exist. Causes of difference are investigated by studying the affinity of the major fractions of black wattle extract towards hide powder. The commercial extract contains polyphenols (ca. 60%) alcohol-insolubles (5-12%) and sugars (ca. 10%). The hide-powder method allows the escape of certain polyphenols which are absorbed by hides during sole-leather tannage. These constitute 4-6% of the polyphenolic fraction. 55-81% of the variable proportion of the alcohol-insolubles or gums reacts as tannins towards hide powder under the standard conditions. A major percentage (55%) of gums does

not take part in the tanning process due to their large molecular size, and the different physical condition of the hide compared with hide powder. Gums cannot be regarded as "tannins" under these circumstances. Sugars are returned as non-tannins by the hide-powder method. The ultra-violet and ferrous tartrate photometric methods measure phenolic content only and appear to be unaffected by the presence of gums, starches and sugars. A partial balance between the loss of a low proportion of wattle polyphenols and the absorption of a high percentage of gums in the hide-powder method, probably accounts for the high correlation between methods. Occasional wide variations in the 5-12% gums present are at least partly, and probably mainly, responsible for occasional differences between methods. The significance of these findings in relation to tannery practice and the analysis of other vegetable extracts is discussed. For purposes of tannin estimation the photometric methods appear to differentiate more faithfully between "tannins" and "non-tannins" than the hide-powder method, and have the advantage of extreme rapidity.

PLANT POLYPHENOLS. I. POLYPHENOLIC CONSTITUENTS OF THE PELLICLE OF THE WALNUT (*Juglans Regia*). L. Jurd, *J. Amer. Chem. Soc.* 78, 3445 (1956), *via. J.S.L.T.C.*, 41, 9, 328 (1957).—A study of the natural anti-oxidants in walnut pellicles has been undertaken. Ellagic acid, methyl gallate, gallic acid, and a tannin were isolated from extracts of the pellicle. The tannin was composed chiefly of four polyphenolic esters or glucosides, and on hydrolysis gave ellagic acid, gallic acid, methyl gallate, and glucose. Preferential oxidation of the pyrogallol derivatives in the pellicle probably aids in stabilising walnut kernel constituents against decomposition.

MICROSCOPICAL STUDY ON THE CHARACTERISTICS OF FIBRE STRUCTURE OF V. T. SOLE LEATHER MANUFACTURED FROM HE- AND SHE-BUFFALO HIDE OF INDIAN ORIGIN. M. Banerjee and S. S. Guha, *J.L.T.A. (Ind.)* 5, 2, 352 (1957).—A Microscopical study of the Sole Leathers made out of the Buffalo hides revealed that the Sole Leathers made out of She-buffalo hides were found to be better than the He-buffalo hides.

K. S. R.

PROCESSES AFTER TANNING

METHOD OF PREPARING OIL EMULSIONS, AND THEIR PRACTICAL USE. Dr. Kessler, *Rev. tech. Industr. Cuir*, 48, 183 (1956) *via. J.S.L.T.C.*, 41, 9, 327 (1957).—The conditions controlling the formation of a stable emulsion of sperm oil and sulphated sperm oil are discussed and illustrated by photographs. Sperm oil emulsions prepared with a proprietary sulphated product are stable to hard water and chrome tanning salts.

LEATHER DRYING. G. W. Douglas, *Tanner* 12, 4, 127 (1957).—The variability of the quality of leather and rate of drying with seasonal conditions is explained. The various equipments used, the methods of drying and their advantages and disadvantages are discussed. It is suggested that some form of automatic control of temperature and humidity is essential in drying sheds, lofts tunnels, etc., in order to reduce costs of production and to improve the quality of the products.

K. S. R.

COLLAGEN AND THE MORE SOLUBLE CONSTITUENTS OF SKIN. J. H. Bowes, R. G. Elliot and J. A. Moss, *J.S.L.T.C.*, 41, 7, 249 (1957).—The chief protein constituent of skin is collagen, but experiments on extraction with dilute acid and alkaline solutions indicate the presence of a number of different protein fractions differing in solubility. The methods used for the determination of the amino acid composition and terminal amino groups of such protein fractions are briefly described. The composition of ox hide collagen has been redetermined by the method of Moore and Stein, and some slight differences from previous analyses are pointed out. Attention is drawn to the peculiarities of the amino acid composition of collagen. No terminal amino groups have been detected in the original collagen but after relatively mild treatments a number become available for reaction. A collagenous protein has been extracted from calf skin with citrate buffer—the procollagen of Orekhovich—and some of its properties are described. The amino acid composition of the extracted protein is essentially the same as that of the ox-hide collagen, but certain small differences suggest that the ox-hide collagen is associated with a protein relatively rich in tyrosine, histidine, leucine, and amide-nitrogen, and containing little or no hydroxyproline. Further experiments on the extraction of calf skin with citrate buffer, dilute

acetic acid, and alkali indicate the presence of one or possibly two non-collagenous protein constituents closely associated with the collagen. The significance of the various soluble protein fractions in relation to the pretanning processes is discussed.

THE AMINO ACID COMPOSITION OF AMPHIBIAN, REPTILE AND AVIAN GELATINS. A. A. Leach, *Biochem. J.*, **67**, 1, 83 (1957).—The amino acid composition of gelatins from two reptiles, an amphibian and a bird, has been determined by ion-exchange chromatography. The agreement in values for gelatin from chicken-leg tendon by both microbiological and ion-exchange methods is very good except for alanine, serine and isoleucine. Chicken-leg-tendon gelatin is similar in composition to mammalian materials. The composition of toad-skin gelatin resembles certain fish gelatins more closely than mammalian materials, and the reverse is true for the two reptile gelatins. The relationship between shrinkage temperature and hydroxyproline and serine plus threonine contents was studied. A very approximate relationship appears to exist between hydroxyproline content and shrinkage temperature.

TANNAGE OF GELATIN WITH FORMALDEHYDE. J. Pouradier and H. Chateau, *J. Chim. Phys.*, **53**, 728 (1956) *via. J.S.L.T.C.*, **41**, 9, 329 (1957).—The reactions are described of gelatin solutions of different concentrations with formaldehyde. Gels may be formed at gelatin concentrations above 20 g. per l.

FURFURAL AND ITS DERIVATIVES IN THE LEATHER INDUSTRY. P. Gourlay, *Rev. Tech. Industr. Cuir.* **49**, 263 (1956) *via. J.S.L.T.C.*, **41**, 9, 330 (1957).—Furfural, in saturated solution, can be used in tanning, and gives leather like that from formaldehyde but furfural oxidises easily. Tetrahydrofurfuryl alcohol is not very volatile, has an ignition point of 84°C and is not classed as inflammable. It is used in finishing: as a solvent for dyes, it is penetrating without removing the grease from the grain: as a solvent for resins, it assists their penetration and adhesion to the surface of the leather.

TANNING LEATHER WITH ALUMINIUM COMPOUNDS IN COMBINATION WITH CHROMIUM. L. B. Sankin and I. P. Strakov. *Tanner*, **12**, 2, 51, (1957).—The chrome-alum tanning compounds were prepared in three alternate ways. 1. A mixture

of potassium bichromate, alum and sulphuric acids was prepared and glucose was added gradually to reduce. 2. Potassium bichromate plus alum plus $\frac{1}{2}$ mol of sodium formate per atom of aluminium plus H_2SO_4 further reduced by glucose. 3. 33% basic chromium compound is mixed with aluminium compounds of 20% basicity and the mixture warmed for an hour in a water bath at 40 to 42°C. In tanning with these combination salts it is observed that the best combination is achieved by mixing aluminium compounds into a solution of Potassium Bichromate and acid with subsequent reduction of chromium. Better results were obtained when sodium formate is added.

Y. N.

TESTING

PERMEABILITY OF LEATHER. A. Gohogne, *Rev. Tech. Industr. Cuir.* **48**, 210 (1956) *via. J.S.L.T.C.*, **41**, 9, 325 (1957).—The Brado apparatus is described; this is suitable for determining the permeability of leather to air, water, and water vapour.

THE INTERPRETATION AND USE OF BACTERIAL INFRARED SPECTRA. Eric K. Rideal. *Chem. & Ind.* **24**, 762 (1957).—Based on the relatively small differences between spectra of different species and strains which must have their origin in the relative differences in structural composition of the bacterial cells, the bacteria can be identified by means of infrared spectra. The bacteria were grown under carefully controlled conditions and after freezing they were prepared for spectroscopy by the pressed-disc technique. Fats, nucleic acids, polysaccharides etc. were selectively removed by extraction of the cells with chemical reagents and the spectra of the cells before and after treatment compared. The results obtained with spectroscopic method agree with biochemically analysed bacterial cells. Work upon *M. lysodeikticus* using this method is outlined. It is concluded that the cell wall of *M. lysodeikticus* consists of protein-polysaccharide mucilage together with a small quantity of fat. This agrees with the results of Salton (*Biochem. Biophys. Acta*, **10**, 512, 1953) obtained by direct extraction and weighing. Similar results are obtained with *E. Coli*, *A. aeroglues*, *Micrococcus albus* and *Microbacterium phlei*. This approach is a new and rapid method for the study of the gross structural composition of bacterial cells.

This method may be applied to a wide variety of problems relating to micro-organisms.

K. S. R.

SIMPLE ULTRA-VIOLET SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF PROTEIN. W. J. Waddell (*N. Carolina Univ. Med. Sch. Chapel Hill, N.C. U.S.A.*). *J. Lab. Clin. Med.*, 1956, 48 (2), 311-314 *via. Anal. Abst.* 4, 9, 3079 (1957).—The rapid method described is based on the difference between the extinction at 215 m μ and at 225 m μ of a suitable dilution of the sample in 0.9% aq. NaCl. This difference multiplied by 144 gives the protein concn. in the diluted solution in micrograms per ml. Applications to the determination of total protein and albumin in plasma and to the determination of protein in cerebrospinal and pleural fluid are described. The method is not applicable to urine.

USE AND ABUSE OF pH MEASUREMENTS. I. Feldman (*The Univ. Rochester, N.Y., U.S.A.*) *Anal. Chem.*, 1956, 28 (12) 1859-1866 *via. Anal. Abst.* 4, 6, 1743 (1957).—Following a review of the development of the concept of pH, the liquid-junction potential problem arising from the use of the pH meter is discussed, with special reference to the probable extent of the error in pH measurements in aq. soln. In certain relatively simple conditions there is an approx. relation between the pH-meter reading and the $[H^+]$, otherwise the reading is only a reproducible reference point without theoretical significance, or is not even that. Examples of such instances are given, and the practical interpretation of pH measurements in aq. and non-aq. soln. colloids and suspensions is discussed. Various pH scales are described and compared.

SPECTROPHOTOMETRIC MICROTÉCHNIQUE FOR THE DETERMINATION OF SULPHUR IN ORGANIC COMPOUNDS. C. Bergamini and M. Maltagliati (*Anal. Chem. Inst. Florence Univ., Italy*). *Sperimentale*, 1956, 6 (4-6) 65-71. *Via. Anal. Abst.* 4, 8, 2636 (1957).—The conditions of formation of methylene blue in an alkaline sulphide medium are studied, from the point of view of the micro-determination of S in organic bases by colorimetric absorption at 735 to 660 m μ . The sample is decomposed with Na in a sealed Pyrex-glass tube, in a metal bomb. Methylene blue is formed in the presence of Zn acetate in a Treadwell-Mayer

apparatus. The Beer-Lambert law is followed between 0.2 and 1.0 g of S per ml. Substances containing S in different states of oxidation, such as benzylpenicillin, have been examined.

SEPARATION AND IDENTIFICATION OF FATTY ACIDS. XIX PAPER-CHROMATOGRAPHIC ANALYSIS OF FATTY ACIDS IN NATURAL FAT. Manjiro Noda, Osamu Hirayama and Yoshiyuki Inouye (*Biochem. Lab., Coll. Agric., Kyoto Univ., Sakyo-ku, Kyoto*). *J. Agric. Chem. Soc. Japan*, 1956, 30 (2) 106-111. *Via Anal. Abst.* 4, 7, 2397 (1957).—The paper-chromatographic separation of unsaturated fatty acids and higher saturated fatty acids was studied by the use of addition compounds of their methyl esters with Hg acetate, by the hydroxamic acid method (reversed-phase chromatography) and by the 2:4 dinitrophenylhydrazide method. The treated sample is spotted on filter-paper impregnated with tetralin and developed with a mixture of 90% methanol, acetic acid and tetralin (10 + 2 + 1). The results were applied to the analysis of fatty acids in cows' milk fat. The sample is saponified, extracted with ether and fractionated into 10 groups by distillation, for subsequent chromatographic examination. The first method is the best for unsaturated compounds and the last for saturated compounds.

QUANTITATIVE DETERMINATION OF AMINO GROUPS IN UREA AND UREA FORMALDEHYDE REACTION MIXTURES OF THE INITIAL STAGE AND THEIR CHEMICAL STRUCTURE. Eisaku Ninagawa (*Fac. Educ. Toyama Univ., Gofuku, Toyama*). *J. Chem. Soc., Japan. Ind. Chem. Sect.* 1956, 59 (10) 1227-1228. *Via Anal. Abst.* 4, 7, 2281 (1957).—The determination of amino groups in urea and urea-formaldehyde reaction mixtures (containing hydroxymethyl) urea, di(hydroxymethyl) urea, methylenediurea, (hydroxymethyl) methylenediurea and di(hydroxymethyl) methyleneurea was studied by the Van Slyke method. The analysis is rapidly carried out by the use of a mixture of acetic acid H_2SO_4 , in place of acetic acid, as solvent. In the decomposition tube, containing acetic acid (2 ml.) and 12 N H_2SO_4 (0.5 ml.) is placed 30% $NaNO_2$ (12 ml.); the sample soln. (1 to 2 ml.) a small amount of octanol (as foam suppressor) and 12 N H_2SO_4 (1 ml.) are then added in the order given and shaken for 7 min. and the gas evolved is passed to the nitrometer. The number of amino groups in these addition compounds was also determined, and their possible structure derived.

THE POLAROGRAPHIC DETERMINATION OF SMALL QUANTITIES OF ACRALDEHYDE IN THE PRESENCE OF SATURATED ALDEHYDES. V. P. Granenova and V. A. Molodovskii. *Trudy Gor'kovsk. Politekhn. Inst.*, 1955, 11 (13), 13-16, Ref. Zhur. Khim, 1956, Abstr. No. 51, 202. *Via Anal. Abst.* 4, 7, 2242 (1957).—Introduce 25 ml. of 0.1 N NaCl and 25 ml. of 0.1 N HCl into a cell and pass CO₂ for 10 min. Keep at 25° for 20 min. and take a polarogram of the back-ground; then introduce a freshly prepared soln. containing acraldehyde and take another polarogram. The height of the waves on the reduction curves less that of the residual current is measured. The mean relative error is 5.5%, which is decreased with increase of concn. of the acraldehyde. The presence of saturated aldehydes does not cause interference in an acid medium.

PROPOSED METHOD OF TEST FOR CALCIUM AND MAGNESIUM OXIDES IN LIME. *American Society for Testing Materials* (1916 Race Street, Philadelphia, Pa., U.S.A.) *Bull. A.S.T.M.* 1956 (218) 27-28. *Via Anal. Abst.* 4, 7, 2111 (1957).—The Ca in the sample soln. is titrated first with EDTA (murexide-NaCl indicator), and then the Ca plus Mg is titrated on a fresh aliquot (Eriochrome black T indicator) in the presence of NH₄Cl-aq. NH₃ buffer and, if Ba and Sr are present, of 3 ml. of saturated aq. K₂SO₄. The method is applicable even to samples high in R₂O₃ or Mg or both.

DIRECT SPECTROPHOTOMETRIC DETERMINATION OF ZIRCONIUM WITH SPADNS. Gurupada Banerjee (Ass. Cultivation of Science, Calcutta, India). *Anal. Chim. Acta*, 1957, 16 (1)-62-66 (in English). *Via Anal. Abst.* 4, 7, 2154 (1954).—The method depends on the formation of a crimson-pink complex of Zr with SPADNS (Cf. *Anal. Abstr.* 4, 2156, 1957) *Procedure*—To an aliquot (> 4 ml.) of the sample soln. containing up to 56 µg of ZrO₂ add 3 ml. of a 0.1% soln. of SPADNS and make up the volume to 25 ml. with 0.5 N HCl. Measure the extinction at 580 mµ, against a reagent blank, in a Unicam SP 600 spectrophotometer, with a 1-cm cell.

VOLUMETRIC DETERMINATION OF SODIUM CONTENT IN INDUSTRIAL SALT. Fumikazu Kawamura and Hiroshi Namiki (Faculty of Engng. Yokohama Univ., Minamiku, Yokohama). *Japan Analyst.* 1956, 5 (9) 505-508. *via Anal.*

Abst. 4, 7, 2089 (1957).—The sample soln. (~0.5 g in 100 ml.) is treated with a known amount of 2% Ba(OH)₂ to precipitate BaSO₄, which is filtered off, and the filtrate is boiled with a mixture of Ag₂O (2.0 g) and AgCO₃ (1.5 g). Chloride is quantitatively converted into the silver salt, and Ca, Mg and the excess of Ba are pptd. as carbonate or hydroxide and filtered off. The filtrate, containing NaOH (and KOH), is titrated with 0.2 N HNO₃, with bromophenol blue as indicator; the amount of added Ba (OH)₂ is deducted. When the amount of Ag₂CO₃ is much more than that of Ag₂O, the pptn. of Mg is not quantitative, whilst that of Cl⁻ is incomplete in a deficiency of CO₃²⁻. The error is < 0.3%. Potassium must be determined by some other method and its amount deducted from the titration value.

AN AUTOMATIC APPARATUS FOR THE QUANTITATIVE MICRO-DETERMINATION OF NITROGEN. F. E. Charlton, *Anal.* 82, 9, 78, 643 (1957).—A detailed description is given of an automatic apparatus for the quantitative micro-determination of nitrogen. The method is similar to the micro-Dumas method, and gives equally good results, but has the advantages that time is saved per determination and that the apparatus does not require the individual attention of the operator for the whole time during which a determination is in progress.

FORMOL TITRATION: AN EVALUATION OF ITS VARIOUS MODIFICATIONS. W. H. Taylor, *Anal.* 82, 976, 488 (1957).—The confusion of procedure and interpretation surrounding the formol-titration method of determining amino-acid nitrogen are reviewed, its origins are traced and the principles underlying the application of the method in various experimental circumstances are examined. Under conditions that simulate protein hydrolysis and with a final formaldehyde concentration of 6 to 9 per cent, evidence from indicator and potentiometric titrations supports the conclusions that (a) in determining the degree of proteolytic hydrolysis a direct titration from the pH of hydrolysis to pH 8.5 in formaldehyde gives reasonably quantitative and reproducible results, (b) indirect procedures do not yield reproducible results (c) with preliminary neutralisation to pH 9 amino acids are under-estimated slightly and peptides severely, and (d) with preliminary neutralisation to pH 7 peptides are under-estimated slightly, but amino acids are not.

THE DETERMINATION OF SMALL AMOUNTS OF O-PHENYLPHENOL. D. Harvey and G. E. Penketh, *Anal.* 82, 976, 498 (1957).—Methods are described for the extraction and determination of the o-phenylphenol added as a fungicide to various materials. The determinations are made by ultra-violet spectrophotometry, colorimetry or fluorescence. The ultra-violet and colorimetric methods are rapid, sensitive and reasonably specific. In the colorimetric method use is made of a commercially available stabilised diazo compound, Brentamine fast red GG; it has advantages over the more usual colorimetric methods for phenols in rapidity and specificity. Both methods give satisfactory results with a variety of treated materials. The fluorescence method, although specific and sensitive, is subject to serious interference and is of use mainly as a qualitative test.

SPECTROPHOTOMETRIC DETERMINATION OF IRON CHELATES. PART II. *Anal.* 82, 976, 524 (1957).—In a previous Note (*ibid* 80, 906, 1957) details were given of an ultra-violet spectrophotometric method for the determination of three iron chelates of the polyaminepolycarboxylic acid type, namely Fe-EDTA, Fe-HEEDTA and Fe-DTPA. The method has now been successfully applied to three other chelates that have since become available: these are the ferric chelates of NN'-dihydroxyethyl-ethylenediaminediacetic acid (Fe-HEEDDA) cyclohexane-1:2-diaminotetra-acetic acid (Fe-CDTA) and ethylenediamine-NN'-bis-(o-hydroxyphenylacetic acid) (Fe-EDHPA).

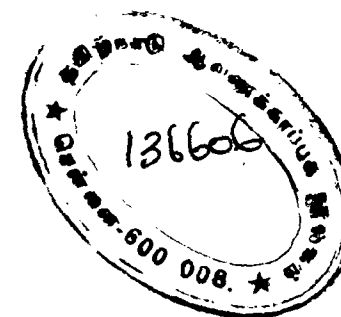
BYE-PRODUCTS

ACTION OF HEAT ON WOOL. J. Marten and J. B. Speakman, *Chem & Ind.* No. 27 955 (1957).—Damage through discolouration and degradation of wool at temperatures above 150°C., can be stopped by introducing unsaturated compounds into the fibres. Wool flannel, when treated with maleic anhydride and subsequently heated in an atmosphere of Nitrogen for 24 hours at 150°C is found to be more resistant to dissolution in NaOH and Urea bisulphite than the heated, untreated flannel. Comparative data obtained by treatment with maleic and succinic anhydride suggest that the protective effect of the former is due to the formation of addition compounds under the influence of heat. Maleic anhydride also minimizes the yellowing of wool during 24 hours of heating at 150°C in either dry Nitrogen or moist air.

K. S. R.

GENERAL

ISOTOPES IN LEATHER MANUFACTURE. E. P. Hugentobler, *Schweiz, Leder -u-Schuh-Ztg.*, 67, 38, 20 (1956) *Rev. tech. Ind. cuir*, 48, 223 (1956) *via J.S.L.T.C.* 41, 9, 326 (1957).—The fate of decylsulphate (DDS) and alkylbenzenesulphonate (ABS) were followed by the incorporation of isotopic S. After using these substances as soaking agents. 3% of the former and 0.3% of the latter were found in the skin. There is a maximum of DDS just below the grain at the level of the hair roots. When added to the pickle, 38% of the material is found in the leather. In a chrome tannage employing DDS about 80% of the substance is found in the leather.



NOTES & NEWS

LEATHER PUMP BUCKETS SPECIFICATIONS. The Indian Standards Institution has published an Indian standard specification for leather pump buckets made from vegetable tanned leather (IS:1015.1956). This specification prescribes the requirements such as material for manufacture, shrinkage in water, cupping test, breaking test, etc. It is hoped that this specification will assure supply of suitable quality of leather pump buckets for use in water systems at normal temperature.

—*Free Press Journal, Bombay, 17th August 1957.*

SYMPOSIUM ON "INDUSTRIAL AND MECHANICAL LEATHERS". A symposium on "Industrial and Mechanical Leathers" will be held in Calcutta in February 1958 under the auspices of the Indian Leather Technologists Association. Along with symposium an All India Leather Exhibition will also be organised. The symposium is being organised to discuss not only the technical aspects but also the economic commercial and statistical aspects as well. Those who intended to participate in the symposium can send the papers to the Office of Association, Mercantile Buildings, Lall Bazaar St., Calcutta-1, before the end of November 1957.

J.L.T.A. (Ind.)—1957.

JAPAN IMPORTS 3.5 MILLION DOLLARS WORTH OF HIDES AND SKINS. Japanese Ministry of Trade has announced that foreign exchange worth 3,500,000 dollars will be allocated for imports of 10,000 tons of cattle hide and calf and kip skins mainly from the U.S.

L.T.R.— 124, 3713, 389 (1957)

WATTLE BARK FOR TANNING INDUSTRY. Clarifying the position regarding the import of wattle bark and wattle extract,

essential to the indigenous tanning industry, the Joint Chief Controller of Imports and Exports Madras, confirmed that there was no reason for tanners to be apprehensive of any shortage of supply. These two items were previously under O.G.L. Since July 1, 1957, however they had come under the licensing procedure based on quota. Licences have been and are being issued by the Joint Chief Controller of Imports and Exports to ensure that the tannin industry does not meet with any difficulty in getting their normal requirements. The trade need not stop their purchases of raw hides and skins, fearing shortage of processing Bark or Extract. The Government and the Leather Export Promotion Council are fully alive to the needs of the tanning industry and it is not likely to suffer from shortage of stocks. The Joint Chief Controller has received assurance from the Chief importers of the raw material that it will continue to be sold at prices at which they have been sold hitherto and there will be no profiteering. With a view to ensure the necessary supplies are kept up, importers and who have imports to their credit outside the basic period will also be considered for the issue of licences on an ad hoc basis on their forwarding applications together with an affidavit certifying their best year imports.

TANNING INDUSTRY IN ANDHRA: CENTRAL LOAN AND GRANT SANCTIONED. Mr. N. Sanjeeva Reddi, Chief Minister announced in the Andhra Pradesh Assembly that the Union Government has sanctioned a loan of Rs. 3,15,963 and a grant of Rs. 1,14,632 for the tanning industry in this State. The State Government he said, proposed to have a model tannery at Warrangal, a production-cum-training centre at Eluru, West Godavari another model tannery at Anantapur, a model leather goods-manufacturing unit at Vijayawada, and tanning industry centres in Cuddapah, Guntur, and West Godavari districts. Rs. 3,00,000 had been ear-marked to meet the non-recurring grants.

—*The Mail, Madras, 3rd August 1957*

GWALIOR LEATHER FACTORY'S PROGRESS. It is learnt that the Gwalior Leather Factory, a Government owned industrial concern, had recently received orders from the Rajasthan Government of leather products worth Rs. 25,000. This work is to be completed by the end of December next. The leather factory had produced 1500 pairs of shoes recently to export to Russia

with a cost of Rs. 20,000. It is also learnt that the factory is making arrangements to start two shifts in place of one in the near future.

—Nagpur Times, Nagpur, 18th July, 1957

LEATHER TOYS PRODUCTION AT JABALPUR: PLAN UNDER WAY. Takhatmal Jain, Minister for Commerce and Industries, told the M.P. Assembly, during question-hour that a plan to establish a centre for manufacturing leather toys at Jabalpur during the Second Five Year Plan was under the consideration of the State Government. He was aware he said, of the increasing demands for leather toys manufactured in foreign countries.

—Nagpur Times, Nagpur, 18th July, 1957

IMPORT OF SKINS OF REPTILES & LIZARDS: In view of representations made, it has been decided to grant licences for import of raw skins of reptiles, lizards etc. from Pakistan under the export promotion scheme, says a trade notice issued in Calcutta by the Joint Chief Controller of Imports and Exports.

—Hindustan Standard, Calcutta, 19th August, 1957

C. L. R. I. NEWS

Dr. Y. Nayudamma, Assistant Director-in-charge returned on the 22nd Oct., 1957 after attending the International Conference of Leather Chemists Societies held in Rome, and both Anniversary meetings of the Society of Leather Trades Chemists at Leeds, England. He also visited the Leather Research Institutes, Tanneries and firms manufacturing Leather Auxiliaries in Italy, England and West Germany.

Dr. Y. Nayudamma, Assistant Director-in-Charge, Central Leather Research Institute attended the Conference of the Directors of the National Laboratories, held during 13th and 17th November 1957.

The Institute is participating in the Congress Exhibition to be held in Madras during December 1957 to January 1958.

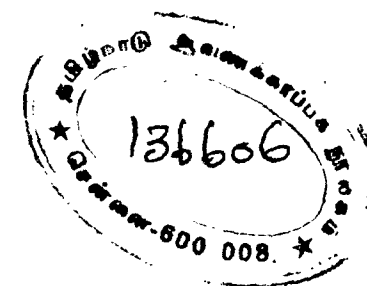
Our Symposium Circular on Leather Auxiliaries dated 2-9-'57.

It has since been decided to hold the Symposium on Leather Auxiliaries by the end of February, 1958 and the last date for receipt of papers for presentation at the Symposium has been extended to 31st Dec. 1957.

In our above cited circular, please read, "Rs. 20 million annually", instead of "Rs. 200 million annually", in page 1, para 2, line 5. "At present, a large amount of leather auxiliaries are imported into India at a great cost valued at about Rs. 20 million annually".

VISITORS

Name	Date
Mr. N. P. Salvi and a party of 19 students from M. S. University of Baroda	15—10—1957
Mr. G. F. Denning	28—10—1957
Mr. N. S. Gnanaprakasam	2 —11—1957
Mr. M. R. Karnik and a party of Textile technology students from University of Bombay	4 —11—1957
Mr. A. H. Ennor, Department of Biochemistry Australian National University, Canberra	4 —11—1957



Editor Y. Nayudamma. Published by N. Subramanian, on behalf of the Central Leather Research Institute, Adyar, Madras-20. Printed by G. Srinivasachari, B.A., at The G. S. Press, 21, Narasingapuram Street Mount Road, Madras-2.

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Bulletin of the

CLRI Madras

Nov 1957



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Bulletin of
The Central Leather
Research Institute
Madras

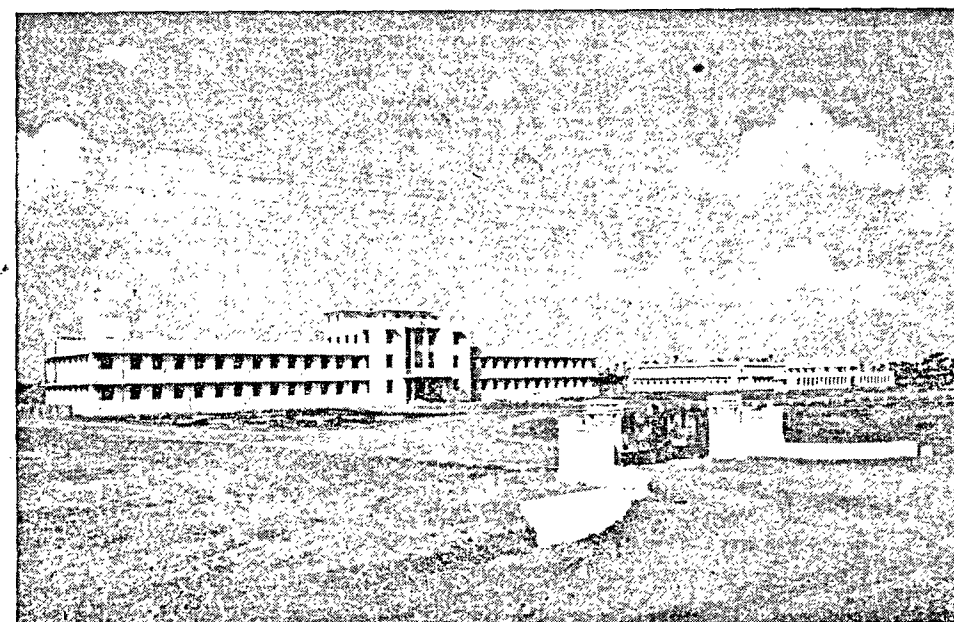


VOL. IV

NOVEMBER, 1957

No. 4

324



COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH
INDIA

Bulletin of The Central Leather Research Institute

Vol. IV	November, 1957	No. 4
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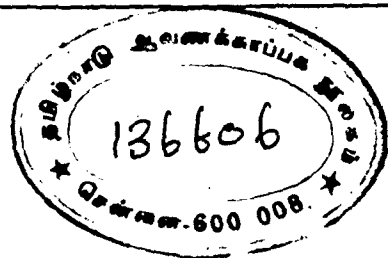
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ADYAR, MADRAS-20.**



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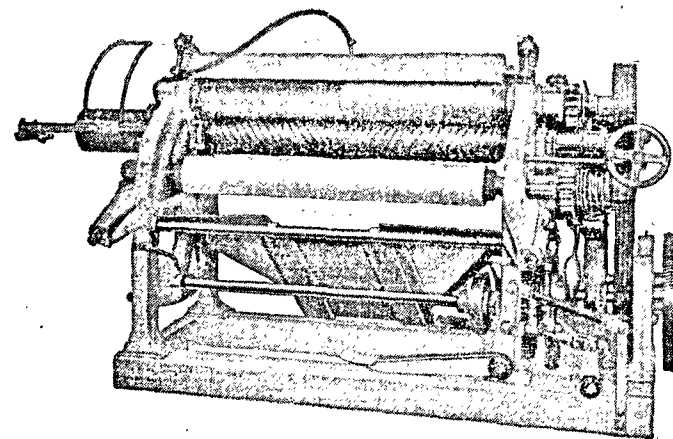
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SYMPOSIUM ON EAST INDIA TANNING INDUSTRY AND TANNING AGENTS (1955): Proceedings, discussions and papers presented. Re. 1/- per copy. Postage extra.

*For further information please contact
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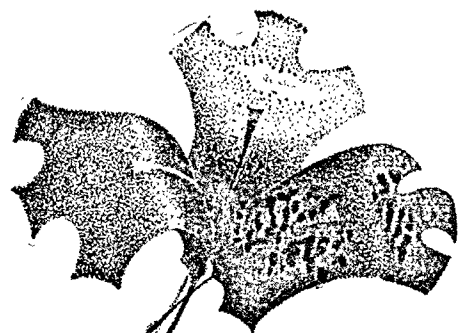
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**STUDIES ON INSECT DAMAGE TO HIDES
AND SKINS—II**

By the beetle *Attagenus gloriosae* (Fabricius)

R. BHASKARAN & S. N. SEN

SUMMARY

324

The nature of damage caused by the larval forms of *Attagenus gloriosae* to hides and skins is described.

Different kinds of beetles have been recorded on hides, skins and on leather from time to time, causing some kind of damage. Of these, the damage caused to the leather industry by the beetles of the *Attagenus* species appears interesting. Members of this species have been recorded on furs, hides, skins, leather, as well as on some vegetable tanning materials, like Myrobalans and Divi-Divi pods. *Attagenus piceus* has been recorded on kid leather,¹ hides, skins and furs,² while *Attagenus byturoides* has been recorded on leather.³ *Attagenus pello*, the 'furriers' beetle was said to occur on pelts and skins.⁴ Hinton stated that a few *Attagenus alferi*⁵ were found on myrobalans loaded in India. The same author noted that the beetle *Attagenus gloriosae* was imported into England with cargos of Divi-Divi pods, celery seed-bags, ground nuts and fleshings from India.⁶

Recently, this beetle — *Attagenus gloriosae* — was seen on hides, skins and finished leather with hair on, causing damage to them, and was, therefore, taken up for the study of the nature of such damage.

The larval forms of this beetle were first noticed on flint and drysalted cow hides, and skins, and also on chrome tanned leathers finished with hair on, on which they were found on the hairy side. In the process of feeding which

was mostly restricted to the hairy side, the epidermal tissue was eroded off, involving the grain layer also. This resulted in the production of depressed map-like scars on the hairy surface, exposing the corium below (Fig. 1). In some places, much deeper excavations were also seen. The larvae were particularly seen along the borders and folds of hides, as these appear to give a suitable lodging place.

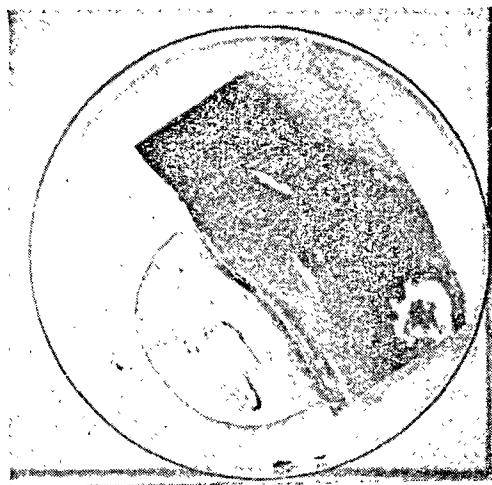


Fig. 1

The larvae are brownish in colour with transverse lighter coloured bands at the posterior part of each segment, giving a beaded appearance. The last tail segment bears a tuft of backwardly directed hairs.

These larvi, when maintained on hides at room temperature, developed into pupae. The pupae were whitish in colour with brown bands on the dorsal aspect. The pupae period was about 9 days, after which the adults emerged. Immediately after emergence, the adults were of a light brown colour, which darkened on exposure to light. This beetle can be easily identified with the help of the whitish band running across the upper surface of its elytra or upper wings. The head and thorax are covered by testaceous

hairs, so also the white band running across the wing. The rest of the body is covered with blackish hairs. (Fig. 2).

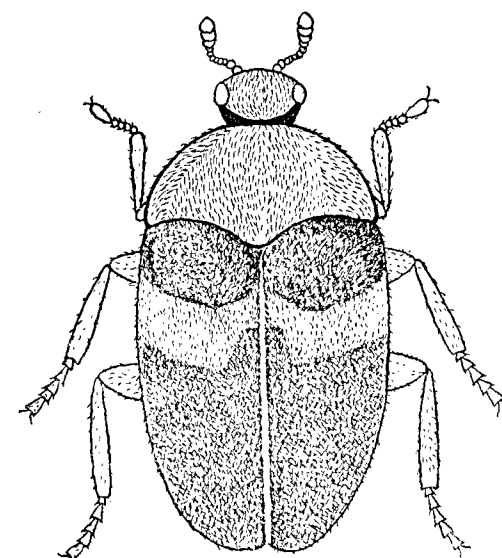


Fig. 2. *Attagenus gloriosae* (diagrammatic)

The adults, when left in contact with the hides, did not cause any damage, similar to those produced by the larvi.

Acknowledgement: Thanks are due to Mr. V. S. Krishnamurthy for help in drawing the picture.

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6. Idem. Ibid. p. 321.

DRY SALTING OF SKINS AND HIDES

J. B. INGRAM & K. V. VAIDYALINGAM

(The Skins & Leathers (Private) Ltd., Coimbatore)

The aim of the Indian Tanning Industry should be to process all available raw hides and skins into finished leathers either for local consumption or for export abroad. The livestock is distributed all over India and the hide and skin production centres are not always near the tanning centres. The raw material has to move over long distances before it reaches the tannery and has to be preserved till then. The hides that are received from Malabar are very badly cured and in most cases are unfit for chrome tanning. As a major portion of the hides and skins emanate from the villages, it is necessary to educate the villagers the proper methods of curing and preservation so that the valuable raw materials do not putrefy even if there is delay in transport. The various Government agencies have already taken up the programme of training village chamars in the proper methods of flaying and it will be in the interest of the industry if the proper methods of curing are also taught to the villagers.

Experiments were conducted to work out a simple but effective method of dry salting goat skins in our tannery and the results have been found to be very encouraging. The details of the process of curing are given below.

Raw materials: 50 green goat skins of average weight of 2.42 lbs. and prime quality of Coimbatore origin were taken for the experiment.

Process: The skins were opened and worked over the beam once to remove the thick layers and fat on the flesh

side and also to open the fibres a little. In the case of cow-hides it is better to do the beaming after washing them thoroughly.

The skins were soaked in 2% salt solution for about one hour and then washed well in three changes of plain water. They were then soaked in a liquor made up as follows:

Water	..	200 per cent on green wt.
Salt	..	40 per cent „
Sulphuric acid		
(commercial)	..	4 oz. per cent „

The next day, they were hauled up, dried and then salted on the flesh side with the following mixture of salt.

Powdered salt	..	10 lbs.
Corilene DG or Teepol or any other		
wetting agent	..	1 oz.
Water	..	1 oz.

After salting the skins were folded and left overnight on a sloping floor to enable the excess water to drain off. Next day, they were salted a second time with the same mixture of salt and folded and left overnight. The skins were then dried in the shade and when nearly dry, were piled flat. The next day, they were dried completely in the morning sun, trimmed and brushed on both sides.

The percentage yield by weight by the above process was 41.2% which is less than the weight usually specified in the trade at present. But the yield of area per pound was correspondingly higher. The approximate cost of dry salting skins by the above method is given below:

1. Wages per 100 skins	..	Rs. 3.06
2. Materials	..	Rs. 7.00
3. Supervision & overheads	..	Rs. 9.94
.	..	Rs. 20.00

Half a dozen of the above skins representing the average lot were kept stored in a shed for exactly two years. It was found after two years that the skins had not developed any putrefaction or red-heat and they were still pliable unlike the usual dry-salted goat skins which, when folded, fold sharply along straight lines. The hair was tight, clean and of good lustre.

It was easy to soak back these skins in plain water. These skins were processed for Chamois leather and also chrome tanned leather along with normal wet salted skins. There was no visible difference in the character of grain or the feel and no loss of substance either.

The above process may easily be adopted for calf skins and cow hides also. This method can be followed with slight modifications, if necessary, while carrying out curing on a large scale.

A NOTE ON THE TANNING PROPERTIES OF HEXAMETHYLENE TETRAMINE-PHENOL COMPLEXES

Hexamethylenetetramine is known to form a variety of molecular complexes due to its weak tertiary basic nature. Such complexes are believed to be involved in Duff reaction,¹ Sommelet reaction,² Delepine reaction³ and the conversion of indoles to indole-3-aldehydes.⁴ The complexes of phenols with hexamethylenetetramine are crystalline solids which decompose on heating to give a resin and ammonia. Hexamethylenetetramine is a stable compound under ordinary pH conditions. However, the compound is rapidly hydrolyzed by treatment with hot mineral acids and more slowly with organic acids.⁵ In view of the various functional groups present in collagen it was considered desirable to study its reaction with hexamethylenetetramine to find out whether the formation of complexes from hexamethylenetetramine and the various functional groups of collagen will effect tanning.

It was found that, while hexamethylenetetramine has got little or no tanning properties, the molecular complex formed by mixing equal amounts of saturated solutions of resorcinol and hexamethylenetetramine was found to be a good tanning agent. The tanning was found almost independent of pH from 7 downwards and the quality of leather produced was found good with a shrinkage temperature of 98°C. Above pH 7 the complex decomposes rather rapidly to give a red resin and ammonia and consequently no tanning is observed above pH 7. While similar complexes prepared from hexamethylenetetramine with catechol and pyrogallol was found to possess good tanning properties the

complex from hexamethylenetetramine with phenol was found to have no tanning action.

In this connection, it was also deemed of interest to study the mechanism of this type of tannage and to draw comparison between phenol-hexamethylenetetramine and resorcinol-hexamethylenetetramine complexes.

The tanning by resorcinol-hexamethylenetetramine may be either due to the formation of complex salts with certain groups of collagen or it could be due to the formation of chemical compounds involving the breaking down of the complex.

Nitrogen estimations of the complexes showed that they are equimolecular compounds of the phenols and hexamethylenetetramine. A method has been developed to analyse the complexes taking advantage of their decomposition to the phenols, formaldehyde and ammonia with hot mineral acids.

In the case of resorcinol-hexamethylenetetramine tannage by analysing the leather produced and the waste liquors it was found that some amount of the complex is broken down to affect tanning. The fact that the complex breaks down in presence of the pelt indicated that there is some strong tendency to form a chemical compound between resorcinol, formaldehyde and some groups of collagen (hexamethylenetetramine is known to behave as a donor of formaldehyde under compelling conditions.⁶) Ammonia was excluded since it was recovered unchanged from the waste liquors.

It has been suggested by Marvel⁷ that resorcinol and formaldehyde combine with the primary or secondary amino groups of collagen by a Mannich type of reaction. In the light of this observation, it was considered probable that such a type of reaction taking place in this tannage. Indeed, it was soon found that while hexamethylenetetramine and

phenol-hexamethylenetetramine complex does not suffer any change when treated with glycine, the resorcinol-hexamethylenetetramine complex is broken down to resorcinol, formaldehyde and ammonia. This explains also why only the latter has got good tanning properties. The fact that there was no fixation when resorcinol-hexamethylenetetramine complex was treated with acetylated collagen seems to support the view that amino groups of collagen are involved in this type of tannage.

There is a wide disparity in the properties of these two complexes. While the phenol hexamethylenetetramine complex is very stable both in boiling water and in boiling 50% acetic acid, the corresponding resorcinol complex undergoes rapid polymerization on keeping it in moist condition, boiling with water and boiling with 50% acetic acid for 30 seconds. The decomposition is invariably accompanied by liberation of ammonia and the resins formed are coloured yellow, brown or red depending upon the conditions of decomposition. The reactivity of the resorcinol-hexamethylenetetramine complex is probably due to the tendency of resorcinol to combine with formaldehyde alone to form polymers or with formaldehyde and primary or secondary amines to effect tanning. This tendency is undoubtedly responsible for breaking down of the complex.

In conclusion two competing reactions are possible when an aqueous solution of the resorcinol-hexamethylenetetramine complex is treated with pelt. One is the reaction of the primary and secondary amino groups of collagen with resorcinol and formaldehyde and the other is the breaking down of the complex by water with the formation of resins. The former reaction seems to be faster in view of the fact that no resin is formed when the tanning is carried out. As it was thought the primary and secondary amino groups of collagen are incapable of breaking down the complex, and tanning is effected by reaction with resorcinol and formaldehyde formed by decomposition of the complex with water,

dehydrated pelt was subjected to treatment with resorcinol hexamethylenetetramine complex in alcoholic medium under similar conditions. Since no tanning was effected, it seems probable that water is essential for this type of tannage.

Further work is in progress for utilization of hexamethylenetetramine in tanning operations and it is hoped the full details could be published soon.

Biochemistry Laboratory,
2nd Sept. 1957.

S. RANGANATHAN,
S. M. BOSE &
Y. NAYUDAMMA,

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RESEARCH BEARS FRUIT

Practical Demonstration No. 7

Process No. 1

Manufacture of East India Tanned Kips using a blend of Karada and Konnam barks in place of Mimosa bark and extract.*

K. RAJABATHER

Raw Material: Wet salted cow hides.

Soaking: The hides are weighed after removing the excess of salt from them. They are soaked in plain water for a period of three hours and then washed in four to five changes of water.

Liming and Unhairing: 20 to 25 per cent of slaked lime on the wet salted weight of the hides is added to once used straight lime liquor, i.e., the liquor from which the hides of the previous lots have been taken for fleshing. Sometimes this quantity of lime may be added in two equal instalments during the period of first liming, i.e., before unhairing the hides. They are then piled in the pit keeping the flesh side up except the outermost one and left for a period of one hour. Then they are hauled and put back in the pit after stirring the lime liquor and left overnight. The hides are hauled and put back in the pit after stirring the lime liquor once every day for a period of 7 days. On the 8th day the hides are not handled. On the 9th day the hides are unhaired and put back in the same liquor until all the hides are unhaired.

Reliming: The unhaired hides are relimed in a fresh bath with the addition of 7 to 10 per cent of slaked lime on

* Process demonstrated during the months of August and September 1957.

the weight of the wet-salted hides. The hides are left in the new lime liquor for three days or so during which period they are handled daily and put back after stirring the lime liquor. But this time all the hides are merely thrown into the pit keeping the flesh side up. On the fourth day, the hides are taken out of the lime liquor and put in plain water overnight. Next day they are fleshed by hand over beam with fleshing knife and weighed. Then they are fleshed a second time with a scudding knife. The hides are then washed by trampling and scudded. They are washed again by trampling and left overnight in plain water.

Deliming : Next day the hides are fleshed a third time with a scudding knife. They are then trampled in a pit containing fresh water and scudded. Then they are washed by trampling and scudded two more times. Finally the hides are treated with $\frac{1}{4}$ to $\frac{1}{2}$ per cent Sulphuric acid, 3 to 6 per cent common salt and sufficient quantity of fresh water for a period of 15 to 30 minutes. The hides are sometimes washed and sometimes they are taken to liquors without washing.

TANNING

First Bark : Always some old bark liquor is to be used initially with the addition of fresh barks or a mixture of barks and extracts. The old bark is first removed from the pit and then the clear liquor is decanted to another pit to which fresh water is added. Calculated quantity of bark or a mixture of barks equivalent to about 9 per cent tannins is soaked in the above liquor on the day the hides are taken for fleshing. This 9 per cent tannins is based on the fleshed weight of the hides and represents about 50 per cent of the total quantity of the tannin required for the entire tanning which is about 18 per cent on the fleshed weight of the hide. If a mixture of Karada bark and Konnam or Babul bark

are used, they are blended in the following proportions and first and second barks are used on the fleshed weight:

Babul or Konnam bark equivalent to 3 per cent tannin on the fleshed weight.

Karada bark equivalent to 6 per cent tannin.

(*Note* : Both Konnam and Babul bark contain 12 per cent tannin and Karada bark contains 24 per cent tannin).

When Konnam and babul barks are used, it is advisable to cut them into small pieces if they are very big and soak them in water. Sometimes, the barks, if they are uncrushed, are immersed in water and taken out giving 2 to 3 changes of water for cleaning them from adhering dirt etc. The cleaned barks are then soaked in liquor as mentioned above and the liquor is stirred twice or thrice during the period of two days.

The delimed hides are dipped in the liquor one by one moving to and fro for a few minutes and then immersed by pressing the hides to the bottom of the pit. Immediately after 5 to 10 minutes, they are hauled up and put back. Again after 15-30 minutes, they are hauled up and put back in the pit keeping half the bark at the bottom and remaining half on the top of the hides. Next day, at about 8 A.M., the hides are hauled and put back in the liquor. If the hides appear to be pulled down to an undesirable extent when hauled, they should be paired grain to grain and piled in the liquor sprinkling the bark on the flesh side of each pair. On the third day, the hides are beamed on the flesh side and put back in the same liquor. After beaming, they are hauled up and put back in the liquor by keeping bark in between each hide and left for 5 to 6 days. The goods are hauled and put back once daily during these days as above. On the last day, the hides are taken to the second bark.

Second Bark : The quantity of bark used is equivalent to about 9 per cent tannin on the fleshed pelt weight. The

blends are taken in the same proportion as in the first bark. The mixture of tanning materials should be soaked in old liquor a day or two before use. As in the case of first bark, it is advisable to cut the bark into small pieces if they are big pieces, cleanse and wash before it is soaked to prepare the tan liquor. The hides from the first bark are beamed and then wrung by twisting to remove water and piled in the second bark liquor. After 10 to 15 minutes, they are again hauled and put back one by one sprinkling the bark over each hide. Next morning, they are hauled up and put back in the liquor sprinkling bark over each hide. The goods are left in this liquor for 7 to 8 days during which period the hides are daily hauled and put back sprinkling bark over each hide. If the hides are flacid during this period, they should be beamed and left in the liquor with the addition of little bark. Otherwise, no extra bark need be added. On the last day, the hides are kept in piled condition in the liquor without keeping any bark in between each hide. But half of the bark should be kept at the bottom of the pit and remaining on the top. Next day the piled hides are left undisturbed.

Bleaching : Next day the hides are rinsed in water, bleached with Perfectan O. Again, they are rinsed in water and beamed.

MYROBING

First Myrobing : On the previous day, crushed myrobalans of good quality, calculating the quantity at $\frac{1}{4}$ lb. of myrobalan for every pound of expected leather yield which may be taken as an average yield of 40 per cent on the fleshed weight of the pelt, are soaked in hot water overnight. The myrobalan liquor is now diluted to the required barkometer strength with cold water. The beamed hides are dipped in the myrobalan liquor one by one and placed in another tub or pit which should be near to it. When all the hides are so dipped and piled, after five to ten minutes, they are hauled up and put back in another tub or in the same

tub pouring the myrobalan liquor on the top of the pile of hides. Next day, the hides are hauled up and put back as was done on the previous day. Now the same quantity of crushed myrobalans is soaked in water in a separate tub as before and left overnight.

Second Myrobing : Next day, the hides are wrung and beamed and put in the myrobalan liquor just as in the case of first myrobing. Next day, they are hauled and put back as before. The following day, the colour of the hides is to be examined. If the hides are found to be having too much of myrobalan colour, they should be rinsed either with water or with the same myrobalan liquor diluted with cold water. The hides are then beamed and taken for oiling.

Oiling and Finishing : The hides are piled with flesh side up and applied a solution containing 3 to 4 per cent of epsom salt (calculated on 100 lbs. of expected leather weight.)

The hides are left for a period of half an hour and then oiled on the grain side with pungam oil and piled. Now a syrup solution is prepared by mixing $1\frac{1}{2}$ to 2 per cent of corn flour and $1\frac{1}{2}$ to 2 per cent of glucose for every 100 lbs. of expected leather. This solution is applied on the flesh side of each hide and then each hide is hung up to sam. When the hung up hides are in semidry condition, they are piled and left overnight. Next day, they are set on the flesh side on wooden plank or table with a stainless slicker or with a brass slicker. If white flesh side is required, a solution of corn flour is applied using $1-1\frac{1}{2}$ lbs. of cornflour for every 100 lbs. of expected leather. The pasted hides are set on the grain side and hung up to semi-dry condition. The semi-dried hides are slicked on the flesh side, and again dried a little. Then they are slicked on the grain side and dried completely. The dried hides are lightly slicked on the grain to produce gloss. The hides are then trimmed and sorted into grades.

TECHNICAL SEMINAR

The Manufacture of Glace kid Leather *

Shri J. C. Deb was deputed by the Institute to the United States under Point Four Programme for advanced training in the manufacture of glace kid leather and leather finishes.

During his visits to a number of plants, the following observations were made :

Glace kid industry in the U.S.A. is dependent on India to a great extent for the supply of raw material, viz., dry-salted goatskins. The glace kid tanneries are considerably big in size with a soaking capacity of 10,000 to 25,000 skins per day. All the operations for processing are completely mechanised. In addition to it, chemical manufacturing organisations and research institutions supply the necessary advanced knowledge as well as newer type of quality auxiliaries for making rapid progress in producing high quality glace kid. The tendency is to make leathers by one-bath tanning in preference to the old two-bath tanning though two-bath tanning is practised in some places. The leathers are not nailed or toggled after staking as it produces thinness and flatness. Seasoning and finishing are done by automatic machines.

The marketing of the kid leathers is well organised and the "Kid Leather Guild" helps to publicise and popularise the kid leathers and their products, to ascertain the views of the public and to set latest fashions in vogue. Publicity

* Summary of the seminar held at the Institute.

is also obtained through seasonal exhibitions. The progress of this industry may mainly be attributed to the well-coordinated efforts of the tanners, the leather auxiliary firms and the marketing firms.

Mr. Deb is of the sincere opinion that good quality glace kid could be made in India with advantage because of the quality raw material available in India and a joint Indo-American venture may prove fruitful in establishing glace kid industry in India. The processing methods and other technical know how could readily be supplied by the Central Leather Research Institute to the Indian tanners.

CLASSIFIED ABSTRACTS

PROCESSES PRIOR TO TANNING

INVESTIGATIONS ON BATING III: THE MECHANISM OF BATING. G. H. Green, *J.S.L.T.C.*, 41, 7, 222 (1957).—The following series of changes is proposed to account for the known effects of bating on the pelt and on the leather:

	lime	heat	trypsin
Collagen		progelatin	
		gelatin	
		low molecular weight peptides	

Progelatin is equivalent to the β collagen of Marriott and the filamented collagen of Borasky and Rogers. These transformations are confined to the fibre surface, but as liming splits up the fibres, additional surfaces become available. Unless the progelatin is removed, the fibres restick when the leather is dried thus nullifying the beneficial effect of the liming. Because progelatin resists proteolysis it must first be converted into gelatin by thermal denaturation. This explains why bating is ineffective below 35°C unless the skin is pre-heated to 40-45°C. Degradation of the progelatin is unnecessary provided it can be removed mechanically. This can be achieved to some extent by scudding alone; this gives better results with calf than with goatskin because of the more open structure of the former. The rate of bating is controlled (at normal bating temperatures) by the penetration of the enzyme into the skin, the difference in case of bating of calf and goatskin again being due to differing compactness of structure. Bating affects mainly the grain layer owing to its fine fibres and close weave which facilitate sticking.

Liming hydrolyses the amide groups of progelatin, and lowers the iso-electric point to about pH 5. Thus some swelling is retained on deliming to pH 8 because of mutual repulsion of fibres with a nett negative charge on their surface. The removal of the progelatin, either by bating or scudding, restores the neutrality of the fibre surfaces and the skin "falls". Falling is associated with the development of flacidity and compressibility, and permeability to air; whilst reduced constriction of the hair follicles loosens the

hair residues. Similar effects to bating can be obtained by methods which prevent the leather fibres from sticking on drying, e.g., fat-liquoring, drying under tension, or acetone dehydration, but then the effect is easily reversed. Reliming after bating produces more progelatin and renders rebating necessary. Straight lime does not immediately inactivate the bating enzymes which greatly accelerate the liming action and formation of progelatin. It is suggested that trypsin, like lime, behaves as a hydrogen bond breaker, and lowers the shrinkage temperature of progelatin. Skin un-haired by sweating or proteolytic enzyme appears superficially like bated pelt, but the leather therefrom is more characteristic of that from limed pelt. This is because comparatively little splittings of the fibres has occurred. The optimum pH for bating of a number of proteolytic enzymes coincides with the optimum for gelatinase activity.

INVESTIGATIONS ON BATING—IV: THE IMPROVEMENT IN BATING ACTIVITY OF A BATE LIQUOR WITH CONTINUOUS PARTIAL RE-USE. G. H. Green, *J.S.L.T.C.*, 41, 9, 303 (1957).—The gradual improvement in bating activity which a freshly made bate liquor undergoes with continuous partial re-use seems to be due to the development of particular species of bacteria. A number of bacteria, mainly *Bacillus* and *Micrococcus* spp. have been isolated from used bate liquors and characterized.

INVESTIGATIONS ON BATING—V: THE INFLUENCE OF LIME BLAST ON BATING AND SCUDDING. G. H. Green, 41, 9, 309 (1957).—Lime blast, due to microscopic particles of calcium carbonate in a skin, is the principal cause of roughness of the grain of a delimed skin. Its loss during bating, due to depletion of the skin and possibly partly due to dissolution, is the main reason for the development of silkiness. The presence of lime blast makes scudding of the delimed or bated skin more difficult. It also impedes the bating of goat skin but not of calf skin. Scudding can replace bating with calf skin and wool sheep skin but only partly with goat skin and hair sheep skin. The improvement in bating obtained by continuous partial re-use of a bate liquor, as related previously to the quality of the bated pelt (Part IV) has now been related to the crust leather.

INVESTIGATIONS ON BATING—VI: THE INFLUENCE OF COLLOIDAL ENZYME ADSORBENTS. G. H. Green, *J.S.L.T.C.*,

41, 9, 321 (1957).—Pieces of calf and goat skin have been bated with trypsin in the presence and absence of kaolin, the concentration of trypsin in solution being the same. Any improvement in bating due to the presence of kaolin could be attributed to adsorbed enzyme being released by the kaolin during bating. Doubt is cast on Wood's theory that enzyme may be carried into the skin in higher concentration than normal though being adsorbed on colloidal particles, and so improve the bating action.

VEGETABLE TANNING

A COMPARISON OF THE RESULTS OF ESTIMATING BLACK WATTLE TANNIN BY THE OFFICIAL HIDE POWDER METHOD AND BY A PROPOSED ULTRA-VIOLET SPECTROPHOTOMETRIC METHOD. C. G. Gordon-Gray, *J.S.L.T.C.*, 41, 8, 269 (1957).—In order to compare the Official Hide Powder method of tannin estimation with a proposed ultra-violet spectrophotometric method extracts made from 94 black wattle bark samples, as well as a number of green and silver wattle samples, were analysed by the two methods. Agreement in actual values for tannin was obtained within 1.0% in 60 samples of black and 1 sample each of green and silver wattle, but greater differences were noted in the remaining 54 samples. These differences do not bear a constant relationship to the Official method. Procedures are detailed for ensuring the concordance of results among laboratories using the U. V. method. Results obtained by Forestal Laboratories, Harpenden, and the Wattle Research Institute, Pietermaritzburg, are recorded.

THE ESTIMATION OF TANNINS IN BLACK WATTLE BARKS AND COMMERCIAL "MIMOSA" EXTRACTS, PART II, STATISTICAL COMPARISON OF THE OFFICIAL HIDE-POWDER (SHAKE) METHOD AND PHOTOMETRIC METHODS OF TANNIN ANALYSIS. D. G. Roux, *J.S.L.T.C.*, 41, 8, 275 (1957).—Statistical comparison is made of results obtained from the same extract solutions by the ultra-violet spectrophotometric and official shake hide-powder methods of tannin estimation. Analysis of variance of Gordon-Gray's 94 duplicate bark analyses shows that differences between methods are not significant. Differences obtained on duplication of analyses on the same samples by the same methods are much smaller than differences between methods. Both the U. V. and the hide-powder of tannin estimation and the extraction procedure are shown to be highly reproducible

for bark analyses, but pronounced skewness is evident for the hide powder method ($g_1 = -0.30$) compared with the U. V. method ($g_1 = +0.02$), when using the same solutions for analysis. The observed skewness for the hide-powder method, although influencing differences between methods is shown not to be significant. Comparison of analyses by each method over 132 bark samples (varying from 17.41% and averaging 33% tannin content) shows high overall agreement between methods ($r = 0.961$) but individual differences as high as 3 and 4% in actual value in certain instances ($s = \pm 1.26$). A standard deviation of errors due to different methods ($s = \pm 1.05$) is calculated, confirming that inherent differences between methods are mainly responsible for observed variations, while experimental errors incurred during estimation contribute a minor but appreciable proportion of observed differences ($P = 16\%$ for differences $> 1\%$ in actual value). Regression equations based on bark analyses only, are calculated for each method. Comparative commercial extract analyses show high correlation between methods ($r = 0.977$) and low standard deviation ($s = \pm 0.63$). The high correlations for bark analyses over the range 20-40% tannin content, and also for extract analyses of 60-70% tannin content, show that excellent overall agreement between methods exists over a wide range of tannin values, in spite of relatively wide differences in a low proportion of individual bark analyses. The adoption of the ultra-violet method as useful alternative to the official hide-powder procedure would therefore, appear to have little overall effect on black wattle tannin estimation. PART III. A CRITICAL EXAMINATION OF THE OFFICIAL HIDE-POWDER METHOD. D. G. Roux, *J.S.L.T.C.*, 41, 8, 287 (1957).—Statistical comparison of the official hide-powder method of tannin estimation with the ultra-violet photometric method emphasises the excellent overall agreement between methods, but also shows that in a low percentage of analyses inherent differences between the methods exist. Causes of difference are investigated by studying the affinity of the major fractions of black wattle extract towards hide powder. The commercial extract contains polyphenols (ca. 60%) alcohol-insolubles (5-12%) and sugars (ca. 10%). The hide-powder method allows the escape of certain polyphenols which are absorbed by hides during sole-leather tannage. These constitute 4-6% of the polyphenolic fraction. 55-81% of the variable proportion of the alcohol-insolubles or gums reacts as tannins towards hide powder under the standard conditions. A major percentage (55%) of gums does

not take part in the tanning process due to their large molecular size, and the different physical condition of the hide compared with hide powder. Gums cannot be regarded as "tannins" under these circumstances. Sugars are returned as non-tannins by the hide-powder method. The ultra-violet and ferrous tartrate photometric methods measure phenolic content only and appear to be unaffected by the presence of gums, starches and sugars. A partial balance between the loss of a low proportion of wattle polyphenols and the absorption of a high percentage of gums in the hide-powder method, probably accounts for the high correlation between methods. Occasional wide variations in the 5-12% gums present are at least partly, and probably mainly, responsible for occasional differences between methods. The significance of these findings in relation to tannery practice and the analysis of other vegetable extracts is discussed. For purposes of tannin estimation the photometric methods appear to differentiate more faithfully between "tannins" and "non-tannins" than the hide-powder method, and have the advantage of extreme rapidity.

PLANT POLYPHENOLS, I. POLYPHENOLIC CONSTITUENTS OF THE PELLICLE OF THE WALNUT (*Juglans Regia*). L. Jurd, *J. Amer. Chem. Soc.* 78, 3445 (1956), *via. J.S.L.T.C.*, 41, 9, 328 (1957).—A study of the natural anti-oxidants in walnut pellicles has been undertaken. Ellagic acid, methyl gallate, gallic acid, and a tannin were isolated from extracts of the pellicle. The tannin was composed chiefly of four polyphenolic esters or glucosides, and on hydrolysis gave ellagic acid, gallic acid, methyl gallate, and glucose. Preferential oxidation of the pyrogallol derivatives in the pellicle probably aids in stabilising walnut kernel constituents against decomposition.

MICROSCOPICAL STUDY ON THE CHARACTERISTICS OF FIBRE STRUCTURE OF V. T. SOLE LEATHER MANUFACTURED FROM HE- AND SHE-BUFFALO HIDE OF INDIAN ORIGIN. M. Banerjee and S. S. Guha, *J.L.T.A. (Ind.)* 5, 2, 352 (1957).—A Microscopical study of the Sole Leathers made out of the Buffalo hides revealed that the Sole Leathers made out of She-buffalo hides were found to be better than the He-buffalo hides.

K. S. R.

PROCESSES AFTER TANNING

METHOD OF PREPARING OIL EMULSIONS, AND THEIR PRACTICAL USE. Dr. Kessler, *Rev. tech. Industr. Cuir*, 48, 183 (1956) *via. J.S.L.T.C.*, 41, 9, 327 (1957).—The conditions controlling the formation of a stable emulsion of sperm oil and sulphated sperm oil are discussed and illustrated by photographs. Sperm oil emulsions prepared with a proprietary sulphated product are stable to hard water and chrome tanning salts.

LEATHER DRYING. G. W. Douglas, *Tanner* 12, 4, 127 (1957).—The variability of the quality of leather and rate of drying with seasonal conditions is explained. The various equipments used, the methods of drying and their advantages and disadvantages are discussed. It is suggested that some form of automatic control of temperature and humidity is essential in drying sheds, lofts tunnels, etc., in order to reduce costs of production and to improve the quality of the products.

K. S. R.

COLLAGEN AND THE MORE SOLUBLE CONSTITUENTS OF SKIN. J. H. Bowes, R. G. Elliot and J. A. Moss, *J.S.L.T.C.*, 41, 7, 249 (1957).—The chief protein constituent of skin is collagen, but experiments on extraction with dilute acid and alkaline solutions indicate the presence of a number of different protein fractions differing in solubility. The methods used for the determination of the amino acid composition and terminal amino groups of such protein fractions are briefly described. The composition of ox hide collagen has been redetermined by the method of Moore and Stein, and some slight differences from previous analyses are pointed out. Attention is drawn to the peculiarities of the amino acid composition of collagen. No terminal amino groups have been detected in the original collagen but after relatively mild treatments a number become available for reaction. A collagenous protein has been extracted from calf skin with citrate buffer—the procollagen of Orekhovich—and some of its properties are described. The amino acid composition of the extracted protein is essentially the same as that of the ox-hide collagen, but certain small differences suggest that the ox-hide collagen is associated with a protein relatively rich in tyrosine, histidine, leucine, and amide-nitrogen, and containing little or no hydroxyproline. Further experiments on the extraction of calf skin with citrate buffer, dilute

acetic acid, and alkali indicate the presence of one or possibly two non-collagenous protein constituents closely associated with the collagen. The significance of the various soluble protein fractions in relation to the pretanning processes is discussed.

THE AMINO ACID COMPOSITION OF AMPHIBIAN, REPTILE AND AVIAN GELATINS. A. A. Leach, *Biochem. J.*, **67**, 1, 83 (1957).—The amino acid composition of gelatins from two reptiles, an amphibian and a bird, has been determined by ion-exchange chromatography. The agreement in values for gelatin from chicken-leg tendon by both microbiological and ion-exchange methods is very good except for alanine, serine and isoleucine. Chicken-leg-tendon gelatin is similar in composition to mammalian materials. The composition of toad-skin gelatin resembles certain fish gelatins more closely than mammalian materials, and the reverse is true for the two reptile gelatins. The relationship between shrinkage temperature and hydroxyproline and serine plus threonine contents was studied. A very approximate relationship appears to exist between hydroxyproline content and shrinkage temperature.

TANNAGE OF GELATIN WITH FORMALDEHYDE. J. Pouradier and H. Chateau, *J. Chim. Phys.*, **53**, 728 (1956) via. *J.S.L.T.C.*, **41**, 9, 329 (1957).—The reactions are described of gelatin solutions of different concentrations with formaldehyde. Gels may be formed at gelatin concentrations above 20 g. per l.

FURFURAL AND ITS DERIVATIVES IN THE LEATHER INDUSTRY. P. Gourlay, *Rev. Tech. Industr. Cuir.* **49**, 263 (1956) via. *J.S.L.T.C.*, **41**, 9, 330 (1957).—Furfural, in saturated solution, can be used in tanning, and gives leather like that from formaldehyde but furfural oxidises easily. Tetrahydrofurfuryl alcohol is not very volatile, has an ignition point of 84°C and is not classed as inflammable. It is used in finishing: as a solvent for dyes, it is penetrating without removing the grease from the grain: as a solvent for resins, it assists their penetration and adhesion to the surface of the leather.

TANNING LEATHER WITH ALUMINIUM COMPOUNDS IN COMBINATION WITH CHROMIUM. L. B. Sankin and I. P. Strakov. *Tanner*, **12**, 2, 51, (1957).—The chrome-alum tanning compounds were prepared in three alternate ways. 1. A mixture

of potassium bichromate, alum and sulphuric acids was prepared and glucose was added gradually to reduce. 2. Potassium bichromate plus alum plus $\frac{1}{2}$ mol of sodium formate per atom of aluminium plus H_2SO_4 further reduced by glucose. 3. 33% basic chromium compound is mixed with aluminium compounds of 20% basicity and the mixture warmed for an hour in a water bath at 40 to 42°C. In tanning with these combination salts it is observed that the best combination is achieved by mixing aluminium compounds into a solution of Potassium Bichromate and acid with subsequent reduction of chromium. Better results were obtained when sodium formate is added.

Y. N.

TESTING

PERMEABILITY OF LEATHER. A. Gohogne, *Rev. Tech. Industr. Cuir.* **48**, 210 (1956) via. *J.S.L.T.C.*, **41**, 9, 325 (1957).—The Brado apparatus is described; this is suitable for determining the permeability of leather to air, water, and water vapour.

THE INTERPRETATION AND USE OF BACTERIAL INFRARED SPECTRA. Eric K. Rideal. *Chem. & Ind.* **24**, 762 (1957).—Based on the relatively small differences between spectra of different species and strains which must have their origin in the relative differences in structural composition of the bacterial cells, the bacteria can be identified by means of infrared spectra. The bacteria were grown under carefully controlled conditions and after freezing they were prepared for spectroscopy by the pressed-disc technique. Fats, nucleic acids, polysaccharides etc. were selectively removed by extraction of the cells with chemical reagents and the spectra of the cells before and after treatment compared. The results obtained with spectroscopic method agree with biochemically analysed bacterial cells. Work upon *M. lysodeikticus* using this method is outlined. It is concluded that the cell wall of *M. lysodeikticus* consists of protein-polysaccharide mucilage together with a small quantity of fat. This agrees with the results of Salton (*Biochem. Biophys. Acta*, **10**, 512, 1953) obtained by direct extraction and weighing. Similar results are obtained with *E. Coli*, *A. aeroglues*, *Micrococcus albus* and *Microbacterium phlei*. This approach is a new and rapid method for the study of the gross structural composition of bacterial cells.

This method may be applied to a wide variety of problems relating to micro-organisms.

K. S. R.

SIMPLE ULTRA-VIOLET SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF PROTEIN. W. J. Waddell (*N. Carolina Univ. Med. Sch. Chapel Hill, N.C. U.S.A.*). *J. Lab. Clin. Med.*, 1956, 48 (2), 311-314 *via. Anal. Abst.* 4, 9, 3079 (1957).—The rapid method described is based on the difference between the extinction at 215 m μ and at 225 m μ of a suitable dilution of the sample in 0.9% aq. NaCl. This difference multiplied by 144 gives the protein concn. in the diluted solution in micrograms per ml. Applications to the determination of total protein and albumin in plasma and to the determination of protein in cerebrospinal and pleural fluid are described. The method is not applicable to urine.

USE AND ABUSE OF pH MEASUREMENTS. I. Feldman (*The Univ. Rochester, N.Y., U.S.A.*) *Anal. Chem.*, 1956, 28 (12) 1859-1866 *via. Anal. Abst.* 4, 6, 1743 (1957).—Following a review of the development of the concept of pH, the liquid-junction potential problem arising from the use of the pH meter is discussed, with special reference to the probable extent of the error in pH measurements in aq. soln. In certain relatively simple conditions there is an approx. relation between the pH-meter reading and the $[H^+]$, otherwise the reading is only a reproducible reference point without theoretical significance, or is not even that. Examples of such instances are given, and the practical interpretation of pH measurements in aq. and non-aq. soln. colloids and suspensions is discussed. Various pH scales are described and compared.

SPECTROPHOTOMETRIC MICROTÉCHNIQUE FOR THE DETERMINATION OF SULPHUR IN ORGANIC COMPOUNDS. C. Bergamini and M. Maltagliati (*Anal. Chem. Inst. Florence Univ., Italy*). *Sperimentale*, 1956, 6 (4-6) 65-71. *Via. Anal. Abst.* 4, 8, 2636 (1957).—The conditions of formation of methylene blue in an alkaline sulphide medium are studied, from the point of view of the micro-determination of S in organic bases by colorimetric absorption at 735 to 660 m μ . The sample is decomposed with Na in a sealed Pyrex-glass tube, in a metal bomb. Methylene blue is formed in the presence of Zn acetate in a Treadwell-Mayer

apparatus. The Beer-Lambert law is followed between 0.2 and 1.0 g of S per ml. Substances containing S in different states of oxidation, such as benzylpenicillin, have been examined.

SEPARATION AND IDENTIFICATION OF FATTY ACIDS. XIX PAPER-CHROMATOGRAPHIC ANALYSIS OF FATTY ACIDS IN NATURAL FAT. Manjiro Noda, Osamu Hirayama and Yoshiyuki Inouye (*Biochem. Lab., Coll. Agric., Kyoto Univ., Sakyo-ku, Kyoto*). *J. Agric. Chem. Soc. Japan*, 1956, 30 (2) 106-111. *Via Anal. Abst.* 4, 7, 2397 (1957).—The paper-chromatographic separation of unsaturated fatty acids and higher saturated fatty acids was studied by the use of addition compounds of their methyl esters with Hg acetate, by the hydroxamic acid method (reversed-phase chromatography) and by the 2:4 dinitrophenylhydrazide method. The treated sample is spotted on filter-paper impregnated with tetralin and developed with a mixture of 90% methanol, acetic acid and tetralin (10 + 2 + 1). The results were applied to the analysis of fatty acids in cows' milk fat. The sample is saponified, extracted with ether and fractionated into 10 groups by distillation, for subsequent chromatographic examination. The first method is the best for unsaturated compounds and the last for saturated compounds.

QUANTITATIVE DETERMINATION OF AMINO GROUPS IN UREA AND UREA FORMALDEHYDE REACTION MIXTURES OF THE INITIAL STAGE AND THEIR CHEMICAL STRUCTURE. Eisaku Ninagawa (*Fac. Educ. Toyama Univ., Gofuku, Toyama*). *J. Chem. Soc., Japan. Ind. Chem. Sect.* 1956, 59 (10) 1227-1228. *Via Anal. Abst.* 4, 7, 2281 (1957).—The determination of amino groups in urea and urea-formaldehyde reaction mixtures (containing hydroxymethyl) urea, di(hydroxymethyl) urea, methylenediurea, (hydroxymethyl) methylenediurea and di(hydroxymethyl) methyleneurea was studied by the Van Slyke method. The analysis is rapidly carried out by the use of a mixture of acetic acid H_2SO_4 , in place of acetic acid, as solvent. In the decomposition tube, containing acetic acid (2 ml.) and 12 N H_2SO_4 (0.5 ml.) is placed 30% $NaNO_2$ (12 ml.); the sample soln. (1 to 2 ml.) a small amount of octanol (as foam suppressor) and 12 N H_2SO_4 (1 ml.) are then added in the order given and shaken for 7 min. and the gas evolved is passed to the nitrometer. The number of amino groups in these addition compounds was also determined, and their possible structure derived.

THE POLAROGRAPHIC DETERMINATION OF SMALL QUANTITIES OF ACRALDEHYDE IN THE PRESENCE OF SATURATED ALDEHYDES. V. P. Granenova and V. A. Molodovskii. *Trudy Gor'kovsk. Politekhn. Inst.*, 1955, 11 (13), 13-16, Ref. Zhur. Khim, 1956, Abstr. No. 51, 202. *Via Anal. Abst.* 4, 7, 2242 (1957).—Introduce 25 ml. of 0.1 N NaCl and 25 ml. of 0.1 N HCl into a cell and pass CO₂ for 10 min. Keep at 25° for 20 min. and take a polarogram of the back-ground; then introduce a freshly prepared soln. containing acraldehyde and take another polarogram. The height of the waves on the reduction curves less that of the residual current is measured. The mean relative error is 5.5%, which is decreased with increase of concn. of the acraldehyde. The presence of saturated aldehydes does not cause interference in an acid medium.

PROPOSED METHOD OF TEST FOR CALCIUM AND MAGNESIUM OXIDES IN LIME. *American Society for Testing Materials* (1916 Race Street, Philadelphia, Pa., U.S.A.) *Bull. A.S.T.M.* 1956 (218) 27-28. *Via Anal. Abst.* 4, 7, 2111 (1957).—The Ca in the sample soln. is titrated first with EDTA (murexide-NaCl indicator), and then the Ca plus Mg is titrated on a fresh aliquot (Eriochrome black T indicator) in the presence of NH₄Cl-aq. NH₃ buffer and, if Ba and Sr are present, of 3 ml. of saturated aq. K₂SO₄. The method is applicable even to samples high in R₂O₃ or Mg or both.

DIRECT SPECTROPHOTOMETRIC DETERMINATION OF ZIRCONIUM WITH SPADNS. Gurupada Banerjee (Ass. Cultivation of Science, Calcutta, India). *Anal. Chim. Acta*, 1957, 16 (1)-62-66 (in English). *Via Anal. Abst.* 4, 7, 2154 (1954).—The method depends on the formation of a crimson-pink complex of Zr with SPADNS (Cf. *Anal. Abstr.* 4, 2156, 1957) *Procedure*—To an aliquot (> 4 ml.) of the sample soln. containing up to 56 µg of ZrO₂ add 3 ml. of a 0.1% soln. of SPADNS and make up the volume to 25 ml. with 0.5 N HCl. Measure the extinction at 580 mµ, against a reagent blank, in a Unicam SP 600 spectrophotometer, with a 1-cm cell.

VOLUMETRIC DETERMINATION OF SODIUM CONTENT IN INDUSTRIAL SALT. Fumikazu Kawamura and Hiroshi Namiki (Faculty of Engng. Yokohama Univ., Minamiku, Yokohama). *Japan Analyst.* 1956, 5 (9) 505-508. *via Anal.*

Abst. 4, 7, 2089 (1957).—The sample soln. (~0.5 g in 100 ml.) is treated with a known amount of 2% Ba(OH)₂ to precipitate BaSO₄, which is filtered off, and the filtrate is boiled with a mixture of Ag₂O (2.0 g) and AgCO₃ (1.5 g). Chloride is quantitatively converted into the silver salt, and Ca, Mg and the excess of Ba are pptd. as carbonate or hydroxide and filtered off. The filtrate, containing NaOH (and KOH), is titrated with 0.2 N HNO₃, with bromophenol blue as indicator; the amount of added Ba (OH)₂ is deducted. When the amount of Ag₂CO₃ is much more than that of Ag₂O, the pptn. of Mg is not quantitative, whilst that of Cl⁻ is incomplete in a deficiency of CO₃²⁻. The error is < 0.3%. Potassium must be determined by some other method and its amount deducted from the titration value.

AN AUTOMATIC APPARATUS FOR THE QUANTITATIVE MICRO-DETERMINATION OF NITROGEN. F. E. Charlton, *Anal.* 82, 9, 78, 643 (1957).—A detailed description is given of an automatic apparatus for the quantitative micro-determination of nitrogen. The method is similar to the micro-Dumas method, and gives equally good results, but has the advantages that time is saved per determination and that the apparatus does not require the individual attention of the operator for the whole time during which a determination is in progress.

FORMOL TITRATION: AN EVALUATION OF ITS VARIOUS MODIFICATIONS. W. H. Taylor, *Anal.* 82, 976, 488 (1957).—The confusion of procedure and interpretation surrounding the formol-titration method of determining amino-acid nitrogen are reviewed, its origins are traced and the principles underlying the application of the method in various experimental circumstances are examined. Under conditions that simulate protein hydrolysis and with a final formaldehyde concentration of 6 to 9 per cent, evidence from indicator and potentiometric titrations supports the conclusions that (a) in determining the degree of proteolytic hydrolysis a direct titration from the pH of hydrolysis to pH 8.5 in formaldehyde gives reasonably quantitative and reproducible results, (b) indirect procedures do not yield reproducible results (c) with preliminary neutralisation to pH 9 amino acids are under-estimated slightly and peptides severely, and (d) with preliminary neutralisation to pH 7 peptides are under-estimated slightly, but amino acids are not.

THE DETERMINATION OF SMALL AMOUNTS OF O-PHENYLPHENOL. D. Harvey and G. E. Penketh, *Anal.* 82, 976, 498 (1957).—Methods are described for the extraction and determination of the o-phenylphenol added as a fungicide to various materials. The determinations are made by ultra-violet spectrophotometry, colorimetry or fluorescence. The ultra-violet and colorimetric methods are rapid, sensitive and reasonably specific. In the colorimetric method use is made of a commercially available stabilised diazo compound, Brentamine fast red GG; it has advantages over the more usual colorimetric methods for phenols in rapidity and specificity. Both methods give satisfactory results with a variety of treated materials. The fluorescence method, although specific and sensitive, is subject to serious interference and is of use mainly as a qualitative test.

SPECTROPHOTOMETRIC DETERMINATION OF IRON CHELATES. PART II. *Anal.* 82, 976, 524 (1957).—In a previous Note (*ibid* 80, 906, 1957) details were given of an ultra-violet spectrophotometric method for the determination of three iron chelates of the polyaminepolycarboxylic acid type, namely Fe-EDTA, Fe-HEEDTA and Fe-DTPA. The method has now been successfully applied to three other chelates that have since become available: these are the ferric chelates of NN'-dihydroxyethylthylenediaminediacetic acid (Fe-HEEDDA) cyclohexane-1:2-diaminotetra-acetic acid (Fe-CDTA) and ethylenediamine-NN'-bis-(o-hydroxyphenylacetic acid) (Fe-EDHPA).

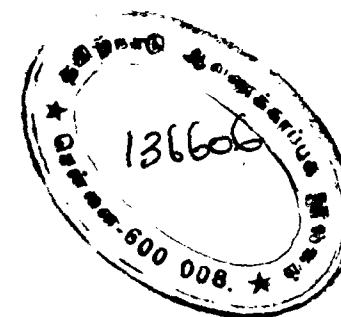
BYE-PRODUCTS

ACTION OF HEAT ON WOOL. J. Marten and J. B. Speakman, *Chem & Ind.* No. 27 955 (1957).—Damage through discolouration and degradation of wool at temperatures above 150°C., can be stopped by introducing unsaturated compounds into the fibres. Wool flannel, when treated with maleic anhydride and subsequently heated in an atmosphere of Nitrogen for 24 hours at 150°C is found to be more resistant to dissolution in NaOH and Urea bisulphite than the heated, untreated flannel. Comparative data obtained by treatment with maleic and succinnic anhydride suggest that the protective effect of the former is due to the formation of addition compounds under the influence of heat. Maleic anhydride also minimizes the yellowing of wool during 24 hours of heating at 150°C in either dry Nitrogen or moist air.

K. S. R.

GENERAL

ISOTOPES IN LEATHER MANUFACTURE. E. P. Hugentobler, *Schweiz, Leder -u-Schuh-Ztg.*, 67, 38, 20 (1956) *Rev. tech. Ind. cuir*, 48, 223 (1956) *via J.S.L.T.C.* 41, 9, 326 (1957).—The fate of decylsulphate (DDS) and alkylbenzenesulphonate (ABS) were followed by the incorporation of isotopic S. After using these substances as soaking agents. 3% of the former and 0.3% of the latter were found in the skin. There is a maximum of DDS just below the grain at the level of the hair roots. When added to the pickle, 38% of the material is found in the leather. In a chrome tannage employing DDS about 80% of the substance is found in the leather.



NOTES & NEWS

LEATHER PUMP BUCKETS SPECIFICATIONS. The Indian Standards Institution has published an Indian standard specification for leather pump buckets made from vegetable tanned leather (IS:1015.1956). This specification prescribes the requirements such as material for manufacture, shrinkage in water, cupping test, breaking test, etc. It is hoped that this specification will assure supply of suitable quality of leather pump buckets for use in water systems at normal temperature.

—*Free Press Journal, Bombay, 17th August 1957.*

SYMPOSIUM ON "INDUSTRIAL AND MECHANICAL LEATHERS". A symposium on "Industrial and Mechanical Leathers" will be held in Calcutta in February 1958 under the auspices of the Indian Leather Technologists Association. Along with symposium an All India Leather Exhibition will also be organised. The symposium is being organised to discuss not only the technical aspects but also the economic commercial and statistical aspects as well. Those who intended to participate in the symposium can send the papers to the Office of Association, Mercantile Buildings, Lall Bazaar St., Calcutta-1, before the end of November 1957.

J.L.T.A. (Ind.)—1957.

JAPAN IMPORTS 3.5 MILLION DOLLARS WORTH OF HIDES AND SKINS. Japanese Ministry of Trade has announced that foreign exchange worth 3,500,000 dollars will be allocated for imports of 10,000 tons of cattle hide and calf and kip skins mainly from the U.S.

L.T.R.— 124, 3713, 389 (1957)

WATTLE BARK FOR TANNING INDUSTRY. Clarifying the position regarding the import of wattle bark and wattle extract,

essential to the indigenous tanning industry, the Joint Chief Controller of Imports and Exports Madras, confirmed that there was no reason for tanners to be apprehensive of any shortage of supply. These two items were previously under O.G.L. Since July 1, 1957, however they had come under the licensing procedure based on quota. Licences have been and are being issued by the Joint Chief Controller of Imports and Exports to ensure that the tannin industry does not meet with any difficulty in getting their normal requirements. The trade need not stop their purchases of raw hides and skins, fearing shortage of processing Bark or Extract. The Government and the Leather Export Promotion Council are fully alive to the needs of the tanning industry and it is not likely to suffer from shortage of stocks. The Joint Chief Controller has received assurance from the Chief importers of the raw material that it will continue to be sold at prices at which they have been sold hitherto and there will be no profiteering. With a view to ensure the necessary supplies are kept up, importers and who have imports to their credit outside the basic period will also be considered for the issue of licences on an ad hoc basis on their forwarding applications together with an affidavit certifying their best year imports.

TANNING INDUSTRY IN ANDHRA: CENTRAL LOAN AND GRANT SANCTIONED. Mr. N. Sanjeeva Reddi, Chief Minister announced in the Andhra Pradesh Assembly that the Union Government has sanctioned a loan of Rs. 3,15,963 and a grant of Rs. 1,14,632 for the tanning industry in this State. The State Government he said, proposed to have a model tannery at Warrangal, a production-cum-training centre at Eluru, West Godavari another model tannery at Anantapur, a model leather goods-manufacturing unit at Vijayawada, and tanning industry centres in Cuddapah, Guntur, and West Godavari districts. Rs. 3,00,000 had been ear-marked to meet the non-recurring grants.

—*The Mail, Madras, 3rd August 1957*

GWALIOR LEATHER FACTORY'S PROGRESS. It is learnt that the Gwalior Leather Factory, a Government owned industrial concern, had recently received orders from the Rajasthan Government of leather products worth Rs. 25,000. This work is to be completed by the end of December next. The leather factory had produced 1500 pairs of shoes recently to export to Russia

with a cost of Rs. 20,000. It is also learnt that the factory is making arrangements to start two shifts in place of one in the near future.

—Nagpur Times, Nagpur, 18th July, 1957

LEATHER TOYS PRODUCTION AT JABALPUR: PLAN UNDER WAY. Takhatmal Jain, Minister for Commerce and Industries, told the M.P. Assembly, during question-hour that a plan to establish a centre for manufacturing leather toys at Jabalpur during the Second Five Year Plan was under the consideration of the State Government. He was aware he said, of the increasing demands for leather toys manufactured in foreign countries.

—Nagpur Times, Nagpur, 18th July, 1957

IMPORT OF SKINS OF REPTILES & LIZARDS: In view of representations made, it has been decided to grant licences for import of raw skins of reptiles, lizards etc. from Pakistan under the export promotion scheme, says a trade notice issued in Calcutta by the Joint Chief Controller of Imports and Exports.

—Hindustan Standard, Calcutta, 19th August, 1957

C. L. R. I. NEWS

Dr. Y. Nayudamma, Assistant Director-in-charge returned on the 22nd Oct., 1957 after attending the International Conference of Leather Chemists Societies held in Rome, and both Anniversary meetings of the Society of Leather Trades Chemists at Leeds, England. He also visited the Leather Research Institutes, Tanneries and firms manufacturing Leather Auxiliaries in Italy, England and West Germany.

Dr. Y. Nayudamma, Assistant Director-in-Charge, Central Leather Research Institute attended the Conference of the Directors of the National Laboratories, held during 13th and 17th November 1957.

The Institute is participating in the Congress Exhibition to be held in Madras during December 1957 to January 1958.

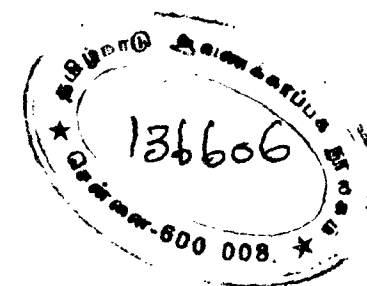
Our Symposium Circular on Leather Auxiliaries dated 2-9-'57.

It has since been decided to hold the Symposium on Leather Auxiliaries by the end of February, 1958 and the last date for receipt of papers for presentation at the Symposium has been extended to 31st Dec. 1957.

In our above cited circular, please read, "Rs. 20 million annually", instead of "Rs. 200 million annually", in page 1, para 2, line 5. "At present, a large amount of leather auxiliaries are imported into India at a great cost valued at about Rs. 20 million annually".

VISITORS

Name	Date
Mr. N. P. Salvi and a party of 19 students from M. S. University of Baroda	15—10—1957
Mr. G. F. Denning	28—10—1957
Mr. N. S. Gnanaprakasam	2 —11—1957
Mr. M. R. Karnik and a party of Textile technology students from University of Bombay	4 —11—1957
Mr. A. H. Ennor, Department of Biochemistry Australian National University, Canberra	4 —11—1957



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