CHEMICAL EXAMINATION OF INDIAN ERGOT OF THE NILGIRIS

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Some time back Thomas and Ramakrishnan[1] described their very successful experiments on the production of ergot of rye in the Nilgiris. Mr. Thomas sent us a sample as early as October 1942. We intended to make a detailed study of it as the first sample grown under Indian conditions and obtain information regarding the influence, if any, of habitat on the chemical composition. Owing to serious difficulties which we then had of laboratory and library we could not undertake the complete examination immediately; but after a proximate analysis the material was defatted and preserved. Further examination was carried out in March 1943 and the results were not without interest. Meanwhile Mukherji and Dey[2] published their note on the assay of Indian ergot and expressed their opinion that the Nilgiris ergot was at least of the British Pharmacopœial quality, if not better. Our results indicate that our sample is really of very high quality comparing favourably with the richest ever produced in different parts of the world and hence they are presented here.

Proximate analysis of the entire drug by standard methods gave the following values; the values reported by previous workers and taken from Barger's book on 'Ergot and Ergotism'[3a] are given within brackets for comparison.

Moisture	7·9% (4·4 – 10·0)
Ash	3·0% (2·2 – 7·0 ; average 4·0)
Total Nitrogen	3·57%
Fat (Petroleum ether extract)	27·3% (21·0%)

The fat which was a liquid had the following characteristics ; the figures within brackets have the same significance as above.

Refractive index at 32° C. ..	–1·466 (1·4685 – 1·4739 at 20° C.)
Saponification value ..	196·2 (178·4 – 196·9)
Iodine value (1 – hour Wij's)	–72·9 (69·55–73·8)
Unsaponifiable matter ..	1·7% (0·35–1·04; average 1·0)
Hehner number	96·5 (96·0–96·3)

The rest of the sample of ergot was powdered, defatted thoroughly with petroleum ether (boiling range 40–50°) by percolation, dried in the air and preserved in an air-tight container. The alkaloidal assay was carried out by the method of Hampshire and Page[4] as described in Garratt's book *Drugs and Galenicals*.[5] 7·0 gm. of the defatted sample were taken for the assay. The rest of the procedure was in general according to the description given in the above-mentioned reference except that a slightly larger volume of 1% tartaric acid was used for extracting the alkaloids from ether solution and the final tartaric acid solution had to be rediluted to give an easily measurable intensity of colour with the special alkaloidal reagent prepared according to the 1936 Addendum to the 1932 B.P. The concentration of alkaloids was obtained by using the Lovibond Tintometer of the B.D.H. pattern for measuring the intensity of colour. When the colour was developed using 1 ml. of the above solution and 2 ml. of the reagent, 8 blue units on the Lovibond Tintometer were taken to represent a concentration of 0·0001 gm. of anhydrous ergotoxine per ml. (*vide* Garratt, p. 112). The following values were obtained: Total alkaloids as anhydrous ergotoxine 0·585%; water-insoluble alkaloids as anhydrous ergotoxine 0·417%; water-soluble alkaloids as ergometrine 0·090%. The last figure was calculated by using the relation obtained by Hampshire and Page (*loc. cit.*) between ergotoxine and ergometrine, *viz.*, that the colour equivalent of ergometrine is 1·86 times that of ergotoxine. All the above figures are with reference to the defatted ergot sample. The corresponding values calculated on the original ergot sample will be 0·425, 0·303 and 0·0654% respectively.

In the meanwhile the note of Mukherji and Dey (*loc. cit.*) appeared in *Current Science* reporting values as low as 0·1213% for total alkaloids and 0·0237% for water-soluble alkaloids, based on the results of analysis by the same chemical method of Hampshire and Page and confirmed by pharmacological assays. With a view to see if our high value was due to any errors in the technique or in the assumed relation between ergotoxine content and blue value the sample was reassayed according to the standard method of 1932 B.P. modified by the 1936 Addendum. However, in view of our previous high results only 2·0 gm. of defatted ergot powder was taken. The rest of the procedure was according to the B.P. The total volume of the tartaric acid solution used in the extractions of the alkaloid from ether solution finally amounted to 52 ml. and this solution was further diluted with an equal volume of 1% tartaric acid for convenient colour matching. The comparison was done against a freshly prepared solution of ergotoxine ethanesulphonate taken from a freshly opened sealed tube of the compound procured from Messrs. B.D.H. and a Duboscq type of colorimeter was employed. The

value for total alkaloids obtained by this method was 0·465% as anhyd ergotoxine calculated with reference to the defatted ergot and 0·338% on entire ergot. It is well known that the B.P. method of assay includes a portion of the water-soluble alkaloids.

The standard solution of ergotoxine ethanesulphonate was also uti for checking the accuracy of the tintometric colour relation with the alka content. Careful readings showed that the tintometer used in the prev assay entirely satisfied the colorimetric relation. 0·0001 gm. of anhyd ergotoxine contained in 1 ml. of solution gave with the standard rea under standard conditions a colour whose blue component was 8 units that the previous assay using only the tintometer for the evaluation of the and water-soluble alkaloids was entirely correct.

Thus it is obvious that the sample of ergot examined by us is very in alkaloid content. There is nothing improbable in this high va In his well-known book Barger[3b] gives numerous data for the alkal content of various samples as obtained by different workers. Therein va as high as 0·38 and 0·414% are found for certain samples. More rece Bekes[6] has reported a value as high as 0·74% obtained in the course of l scale experiments on the intensive production of rye-ergot. The same au has also studied the alkaloidal content of individual sclerotia[7] and repo values as high as over 1% of total alkaloids with several sam It should however be noted that variations are possible in the qualities of samples depending on differences in the conditions of collection and pre vation and this may account for the differences between our results and t of others.

In any project for the production of ergot adequate precaution ha be taken to prevent infected rye grains finding their way into those colle for food, since ergot is a poison. A reliable chemical test for the pres of ergot makes use of the colour reaction for sclererythrin which is a crim violet colouring matter present in the walls of the cortical hyphæ, prob as the calcium salt. The test was carried out as below with the present sam of ergot: About 0·1 gm. of the powdered ergot was skaken with 5 ml ether and a few drops of dilute sulphuric acid for 5 minutes. The ether s tion was then decanted, diluted to 10 ml. and half the volume treated with 2 of a saturated solution of sodium bicarbonate. A deep violet colour formed in the aqueous layer. This colour test shows that the Nilgiris e easily conforms to the ordinary requirements of ergot for purposes testing food grains.

Summary

A sample of Indian ergot of the Nilgiris has been studied in detail. It contains remarkably high percentage of total and water-soluble alkaloids. With respect to other components it is normal.

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