

**AN IMPROVED THIN-LAYER
CHROMATOGRAPHIC METHOD FOR
THE DETECTION OF *ABRUS*
PRECATORIUS SEEDS**

As the seeds of *Abrus precatorius* (jequirity, rosary bean) are highly poisonous¹ and are often encountered in cases of criminal poisoning, there is need for a simple analytical procedure for its detection in powdered or ingested seed material involved in such cases. Ghosal and Dutta² have shown that the major alkaloidal constituents of jequirity seed are N-methyltryptophan (abrine), precatorine and hypaphorine. Since precatorine and N-methyltryptophan are characteristic for *A. precatorius*²⁻³ their detection in the seed is of value in its identification.

The thin-layer chromatographic (TLC) procedure suggested recently by Genest⁴ provides only for the detection of the indole derivatives N-methyltrypto-

chromatography, the plates are air-dried and viewed in short wave UV light (254 nm). The separated alkaloids show up as dark spots against a greenish fluorescent background and are readily identified with the help of the reference substances. The R_f values of the three alkaloids and their detection limits in UV₂₅₄ light are given in Table I.

TABLE I

Alkaloid	R_f	Detection limit (μg) in UV ₂₅₄
N-methyltryptophan	0.53	5
Hypaphorine	0.36	10
Precatorine	0.10	4

Further detection is made possible by the use of the spray reagents indicated in Table II which

TABLE II

Colour reactions of the alkaloids of A. precatorius

Spray Reagent ^b	N-methyltryptophan	Hypaphorine	Precatorine
Dragendorff	..	Orange (10)	Orange Red (5)
van Urk	violet changing to blue (0.5)	violet changing to blue-violet (0.5)	..
Procházka	yellowish-brown ^a (5) greenish-yellow ^b (0.1)	brownish-violet ^a (5) golden brown ^b (0.1)	..
Salkowski	brown ^a (5) greenish-yellow ^b (0.1)	brownish-violet ^a (5) rusty brown ^b (0.1)	..

^a=visible colour; ^b=fluorescence colour in UV₃₆₆; ()=detection limit (μg).

phan and hypaphorine. In this communication, we report an improved TLC procedure in which the use of Silica Gel GF₂₅₄ as adsorbent affords a simple means of detecting all the three major alkaloids of jequirity seed by their quenching effect on the fluorescence of the UV indicator incorporated into the adsorbent. Further detection is possible by the use of spray reagents in the usual way.

A methanolic extract of powdered jequirity seed kernels and about 5 to 10 μg each of the reference compounds (authentic samples of the three alkaloids from methanolic solutions) are spotted in a row on Silica Gel GF₂₅₄ (Merck) layers (0.25 mm) previously activated at 110°C for half an hour. The plates are then developed in the solvent system butanol-acetic acid-water (65:13:22) by the ascending technique with chamber saturation. After

shows the colour reactions of the alkaloids with the chromogenic reagents together with their detection limits on thin-layer chromatograms.

In comparison with the previously reported method⁴, the procedure described above has the following advantages: (a) permits detection of three alkaloids, two of which are characteristic for *A. precatorius*; (b) provides improved resolution of the alkaloids giving compact and round spots; (c) enables the separated alkaloids to be detected without being chemically modified in any reaction.

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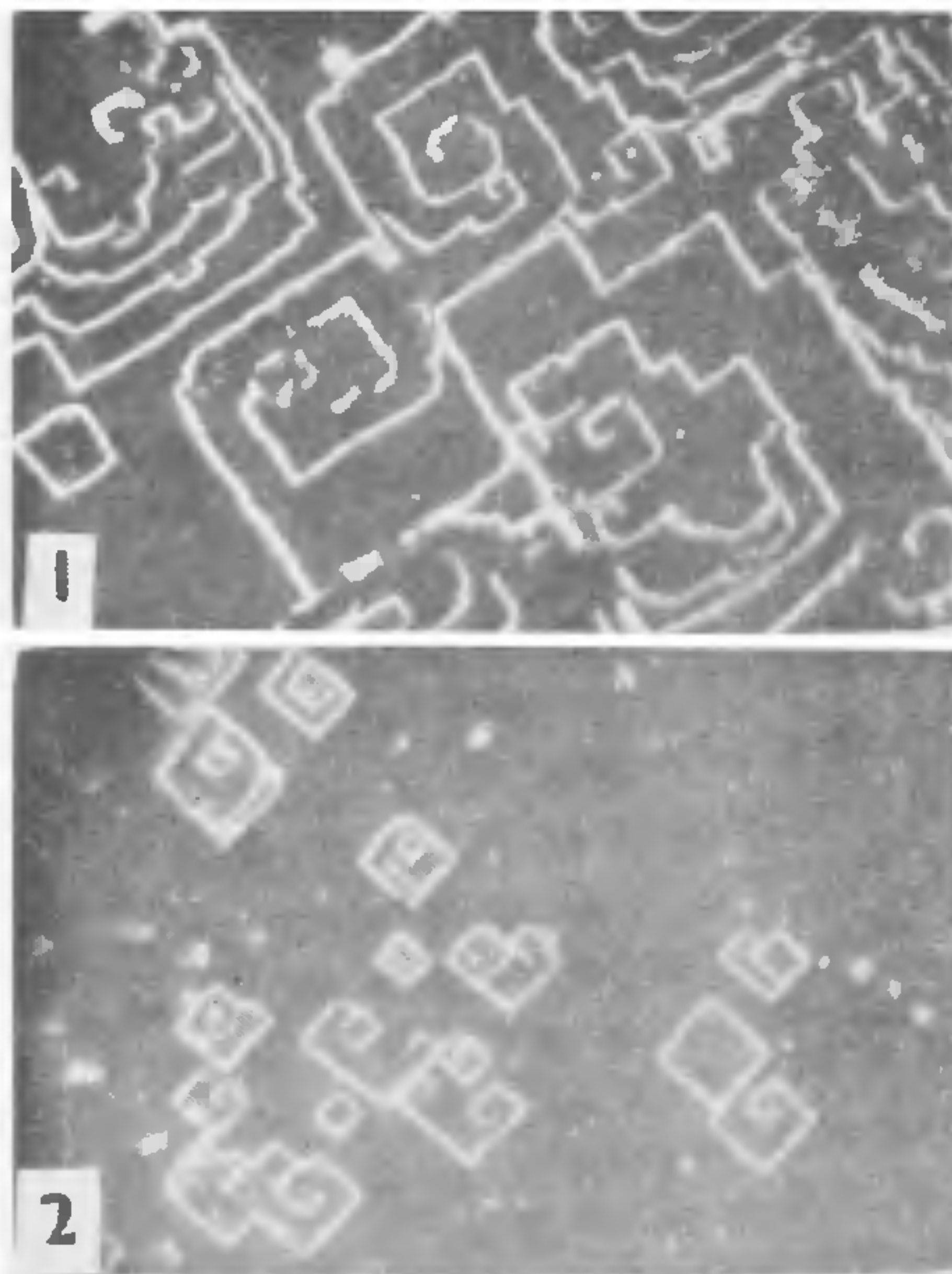
FORMATION OF SPIRALS DURING ANODIC DISSOLUTION OF COPPER (100) PLANE

GROWTH spirals during electrodeposition of copper on copper single crystal substrate¹⁻², lead on copper (111) plane³, titanium from molten salt⁴ and on silver-indium alloy⁵ have been reported. It is known that the dissolution of closed packed plane of a crystal is reciprocal of its growth⁶ at least near undersaturation conditions. So far formation of spirals on single crystal planes of metals are not noticed when they are anodically dissolved. Therefore it is felt that it would be interesting to study the anodic dissolution of copper single crystal (100) plane.

The crystal was fixed in tygon tubing so that only (100) plane was exposed. The crystal was mechanically polished on 4/0 emery paper and was electropolished in 1:1 orthophosphoric acid at a cell potential of 1.2 V for 30 minutes as described earlier⁷. After electropolishing the crystal was anodically dissolved at various current densities (cds) in a three compartment cell containing deaerated, stirred 0.1 N sulphuric acid. After dissolution to a thickness equivalent to 5 C/cm², the crystal was taken out and washed with triple distilled water and dried with alcohol. The crystal surface was observed under a metallurgical microscope and microphotographs were taken.

When copper (100) plane was dissolved between cds 0.1-0.5 mA/cm², occasional pyramidal pits with layers in the background were observed. At 0.6 mA/cm², only striated square layers were seen. Spirals along with striated square layers were observed when cd was increased to 0.7 mA/cm²

and they completely covered the surface (Fig. 1). The distance between the steps of spiral decreased as the cd was gradually increased (Fig. 2), as noticed in the case of silver⁸.



FIGS. 1-2. Fig. 1. Dissolution of copper (100) plane in deaerated stirred 0.1 N H₂SO₄ at 0.7 mA/cm² upto a thickness of 5 C/cm², × 600. Dark field illumination. Fig. 2. Dissolution of copper (100) plane in deaerated 0.1 N H₂SO₄ at 4 mA/cm² upto a thickness of 5 C/cm², × 600. Dark field illumination.

The above results indicate that, during anodic dissolution screw dislocations are operative on the surface. It is in agreement with the observation of the earlier workers⁹ that screw dislocations become active when cd at which spirals are observed must be greater than the exchange current density. The decrease in the distance between steps of spiral with increase in cd can be explained in the light of the theory of Burton, Cabrera and Frank¹⁰. Since the dissolution is an exact reciprocal of growth, it is expected that spiral unwinds during dissolution as pointed out by Bockris *et al.*¹¹.

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