The titrimetric assay of solasodine, a spirosolane of commercial importance from *Solanum laciniatum* Ait.

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Solasodine is a starting material for the steroid industry. It occurs in *Solanum laciniatum* as its 3-β-*O*-glycosides. These are most abundant in the green, unripe fruits. Existing assays for solasodine involve solvent extraction of the glycosides from dried plant material, after which, because the glycosides are not readily estimated, they are hydrolysed and the aglycone is separated and determined titrimetrically or colorimetrically (Birner, 1968). It has been shown that aqueous incubation of sapogenin affording plant material alone and with additives increases sapogenin yield (Hardman & Brain, 1971; Hardman & Wood, 1971). Therefore our assay has been designed to process powdered plant material in water. Extraction of glycosides from aqueous media presents problems; their hydrolysis *in situ* by the addition of HCl to the media was hence the first stage in the assay. Preliminary results are now reported.

Dried unripe fruit, 2·5 g, which had been partially defatted by 24 h continuous extraction with light-petroleum (40–60°), was refluxed for 3 h with 50 ml 2N HCl and cooled to 80°. Solution of ammonia (2 × 20 ml, s.g. 0·880) was added. After further cooling the mixture was filtered and the residue washed and then dried overnight at 60°. The residue with filter paper was extracted with $CHCl_3$ for 24 h in a soxhlet and the extract adjusted to 100 ml with solvent. Aliquots of this solution were titrated automatically with 0·01N $HClO_4$ in dioxan using a recording potentiometric titrator.

Preliminary experiments have shown that 65·5% of the solasodine liberated during the hydrolysis is dehydrated to solasodiene. The end point potentials of this and of solasodine are very similar and the titration does not distinguish between them. The results are therefore expressed as solasodine.

Using this method we have made preliminary investigations of the effects of fine powdering and of partial defatting on the assay and its reproducibility (Table 1).

Table 1. *Replicate assays of dried, unripe fruits of* S. laciniatum.

Undefatted powder; 60% retained by a No. 30 sieve	Undefatted fine powder; 98% passed through a No. 30 sieve	Partially defatted fine powder	Light-petroleum extractive*
2·34	3·60	3·00	0·32
2·61	3·47	3·01	
2·55	3·31	2·98	
		3·00	
		2·98	

Results expressed as % base calculated as solasodine with reference to the dried fat-containing fruits (moisture, 7·5% fat, 6·4%*).

REFERENCES

BIRNER, J. (1968). *J. pharm. Sci.*, **58**, 258–259.
HARDMAN, R. & BRAIN, K. R. (1971). *Phytochem.*, **10**, 519–523.
HARDMAN, R. & WOOD, C. N. (1971). *Ibid.*, **10**, 757–763.

The determination of diosgenin and yamogenin in fenugreek seed by combined column chromatography and infrared spectrometry

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Acid hydrolysis of Moroccan seed of *Trigonella foenumgraecum*, L. (fenugreek), followed by extraction with light-petroleum 40–60° affords a mixture of the epimers diosgenin and yamogenin, 1%, with fixed oil, 6% and free sterol, sterol esters, spirostadienes, and gitogenin. We have separated diosgenin from yamogenin by preparative-t.l.c. and have described how they