alumina to flow into the center of the column with resultant uniform distribution, wetting, and packing. The bulb's size is dictated by the quantity of adsorbent used; however, the diameter of the capillary tube on the bulb remains constant (2 mm I.D. for 100–200 mesh alumina affords an uninterrupted, fine stream). The capillary length should be such as to avoid contact with any upwardly displaced liquid. The addition requires little, or, usually, no monitoring*. Pressure equilibration between the column and bulb is accomplished by three glass beads, equally spaced and molded on the side of the bulb. After the addition is complete, the adsorbent is allowed to settle and the liquid is drawn off, until just above the alumina surface. The tube is repeatedly refilled with the liquid until the adsorbent no longer shows any tendency to settle. During this process, the alumina must *always* remain covered with liquid. The column is then covered with a layer of sand to insure a level alumina surface. Execution of the chromatography is performed in the usual manner.

In this laboratory, the ratio of the diameter of the alumina column to its height (not the height of the tube) has been kept as close to 1:5 as possible with the weight ratio of material to alumina about 1:30. Approximately 100 column adsorption chromatograms have been conducted in this manner, and zones were generally well defined, where visible. It has also been noted that the eluant passes through this column much more rapidly than through the hand-filled and tapped column.

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* A cone formed from glassine- or weighing-paper, may be substituted for the addition bulb, especially for small quantities (<30 g) of alumina. This modification is not recommended for larger quantities of adsorbent, owing to adsorption of moisture.

Reaction of α- and β-conidendrin with chromatographic solvents*

Two developing solvents widely used in the paper chromatographic separation of phenolic wood extractives, namely butanol–acetic acid–water (BAW) (60:15:25) and 2 % aqueous acetic acid, reacted with both α- and β-conidendrin[1] to form small quantities of reaction products. This was first observed in two-dimensional chromatography using BAW followed by 2 % acetic acid. In these solvent systems α-conidendrin

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and its reaction products formed a single spot R_F 0.83 in BAW which in the 2 % acetic acid direction was resolved into two main spots R_F 0.00 (α-conidendrin) and R_F 0.50 together with a lesser spot R_F 0.70. Under similar conditions β-conidendrin had an R_F 0.86 in the first direction and produced only two spots R_F 0.00 (β-conidendrin) and R_F 0.53 in the second. In one-dimensional chromatography using 2 % acetic acid, α-conidendrin gave rise to three spots R_F 0.00, 0.50 and 0.70 and β-conidendrin to two spots R_F 0.00 and 0.58, although considerable streaking was evident in both cases. The larger amounts of reaction products detected when using two-dimensional BAW–2 % acetic acid is indicative of reaction with the acetic acid in both solvent systems. Because of the low concentration of the reaction products, detection was doubtful with diazotized sulfanilic acid (DSA)[2], but was positive using ferric ferricyanide reagent (FF)[3].

Small quantities of reaction products could be conveniently produced by treatment of either α- or β-conidendrin with acetic acid–water (1:1) at room temperature. No increase in the ratio of reaction products to the parent lactone after 12 h was noted indicating equilibrium had been established. These reaction mixtures were chromatographed in different solvent systems both before and after removal of the acetic acid and compared with chemically related compounds as shown in Table I. Attempts to isolate the R_F 0.50 compound from the α-conidendrin–acetic acid mixtures, either by low temperature vacuum distillation or lyophilization, resulted in only starting material and a small quantity of the R_F 0.70 compound, no R_F 0.50 remaining. Under similar conditions the β-conidendrin–acetic acid mixture produced mainly β-conidendrin and a weak spot R_F 0.58. Sufficient R_F 0.70 material was obtained for an infrared spectrum to be taken. A comparison of this spectrum with that of an authentic sample of α-conidendric acid[1] showed agreement of the major absorption peaks, which together with chromatographic data strongly indicated the R_F 0.70 spot to be α-conidendric acid. Thus, while it is still true that the quantitative conversion of α-conidendrin to its hydroxy acid must be done under strong hydrolyzing conditions[1], it is significant that detectable quantities of this acid can be obtained from the relatively mild conditions encountered with 2 % acetic acid at room temperature.

Further evidence of interconversion during paper chromatography was provided by the behaviour of pure α-conidendric acid in 2 % aqueous acetic acid. In this solvent two main spots were evident, R_F 0.70 strong and R_F 0.50 weak. The reaction forming β-conidendric acid, however, as would be expected from a study of its stereochemistry and lactonization rates of the dimethyl-β-conidendric acid[1], was more complicated. An attempt to produce β-conidendric acid using the same experimental conditions advocated by HOLMBERG[1] for α-conidendric acid resulted in a mixture of products heavily contaminated with the lactone. Paper chromatography of the mixture in 2 % acetic acid produced two main spots, R_F 0.77 medium and R_F 0.00 strong (β-conidendrin) with considerable streaking from the origin to R_F 0.58 as is found in papergrams of β-conidendrin in this solvent.

Because of the small quantities and labile nature of these reaction products of α- and β-conidendrin only α-conidendric acid can be assigned with certainty. The other compounds, however, must be closely related since they appear to be in equilibrium with the lactone and its hydroxy acid, although the possibility of some form of double spot chromatographic behaviour cannot be excluded.

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TABLE I

CHROMATOGRAPHIC DATA OF α- AND β-CONIDENDRIN, CONIDENDRIC ACID AND SIMILAR LIGNANS

Lignan	*Solvent system**									
	2% acetic acid			*Butanol–acetic acid–water (60:15:25, v/v)*			*Methanol–amyl alcohol–benzene–water (2:1:1:1, v/v)*			
	R_F value	*FF*	*DSA*	*R_F value*	*FF*	*DSA*	*R_F value*	*FF*	*DSA*	*Acid*
α-Conidendrin	0.00 S	Blue	Pinkish brown	0.83 S	Blue	Pinkish brown	0.81 S	Blue	Pinkish brown	
	↓ 0.50 M	Blue	N.D.							
	0.70 VW	Pale blue	N.D.							
α-Conidendrin–aqueous acetic acid (1:1) reaction mixture	0.00 S	Blue	Pinkish brown	0.86 S	Blue	Pinkish brown	0.75 W	Blue	Pink	Pale yellow
	↓ 0.50 M	Blue	Pinkish brown				0.81 S	Blue	Pinkish brown	
	0.70 W	Pale blue	Pink							
α-Conidendrin–aqueous acetic acid (1:1) reaction mixture after solvent removal	0.00 S	Blue	Pinkish brown	0.86 S	Blue	Pinkish brown	0.75 M	Blue	Pink	Pale yellow
	↓ 0.70 M	Blue	Pinkish brown				0.81 S	Blue	Pinkish brown	

β-Conidendrin	↓ 0.00 S 0.58 W	Blue Blue	Pinkish brown N.D.	0.86 S	Blue	Pinkish brown	0.82 S	Blue	Pinkish brown	
β-Conidendrin–aqueous acetic acid (1:1) reaction mixture (unchanged after solvent removal)	0.00 S ↓ 0.58 W	Blue Blue	Pinkish brown N.D.	0.87 S	Blue	Pinkish brown	0.82 S	Blue	Pinkish brown	N.D.
α-Conidendric acid	0.50 W 0.70 S	Blue Blue	Pinkish brown Pinkish brown	0.86 S	Blue	Pinkish brown	0.75 S	Blue	Pinkish brown	Pale yellow
β-Conidendric acid heavily contaminated with β-conidendrin	0.00 S ↓ 0.58 W 0.77 M	Blue Blue Blue	Pinkish brown Pinkish brown Pinkish brown	0.69–0.94 single elongated spot	Blue	Pinkish brown	0.64–0.90 single elongated spot	Blue	Pinkish brown	N.D.
Matairesinol	0.60 S	Blue	Red purple	0.94 S	Blue	Red purple	0.92 S	Blue	Red purple	
Hydroxymatairesinol	0.85 S	Blue	Orange	0.90 S	Blue	Orange	0.77 S 0.85 W**	Blue Blue	Orange Pinkish brown	

* Detection: FF = ferric ferricyanide reagent[3]; DSA = diazotized sulfanilic acid reagent[2]; acid = bromocresol green plus bromophenol blue acid detector[4]; ↓ = streaking; S = strong; M = medium; W = weak; VW = very weak; N.D. = not detectable.

** α-Conidendrin[5].

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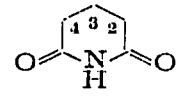
R_F values of some glutarimides

Various derivatives of glutarimide (I) have been shown to possess biological activities (Table I)[1,2] and a few of these are employed in medical practice. As part of an investigation of the metabolic fate of some glutarimides in animals, a study was made of their chromatographic properties.



(I)

All paper chromatograms were carried out with Whatman No. 1 grade. Some papers were impregnated with liquid paraffin (4% in hexane), olive oil (20% in

TABLE I

MAIN PHARMACOLOGICAL ACTION OF THE GLUTARIMIDES USED IN THE STUDY

	Compound	*Pharmacological action*
I	Glutarimide	None
II	3,3-Dimethyl glutarimide	Convulsive
III	3-Ethyl-3-methyl glutarimide (Bemegride)	Convulsive
IV	2,4-Dicyano-3-ethyl-3-methyl glutarimide	None
V	3-Isopropyl-3-methyl glutarimide	Convulsive
VI	3,3-Di-*n*-propyl glutarimide	Hypnotic
VII	2-Phenyl-2-ethyl glutarimide (Glutethimide)	Hypnotic
VIII	2-*p*-Aminophenyl-2-ethyl glutarimide (Aminoglutethimide)	Anticonvulsive
IX	2-Phenyl-2-diethylaminoethyl glutarimide hydrochloride (Phenglutarimide)	Parasympatholytic