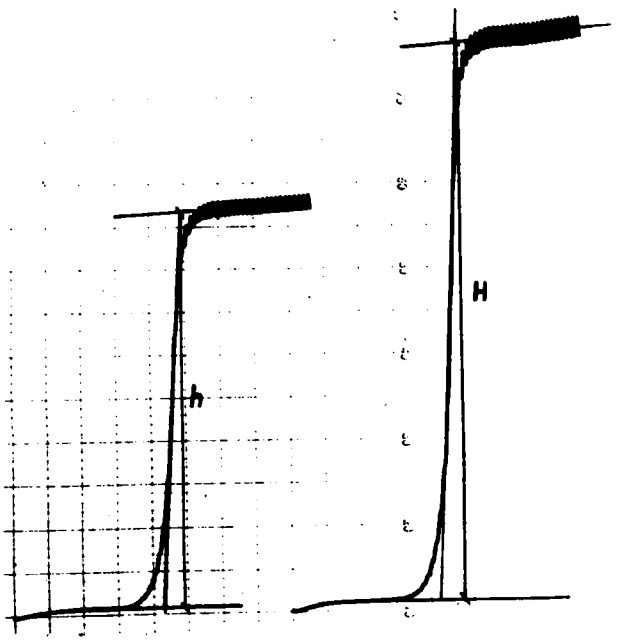

Figure 1—*Polarographic waves of tinidazole before and after standard addition.*

determination, h = wave height of tinidazole before standard addition, H = wave height of tinidazole after standard addition, and b = average weight of a tablet (grams).

RESULTS AND DISCUSSION

The polarographic wave of tinidazole obtained under the described conditions is well developed, and the precision of the proposed method is evident from Table I. The polarographic current was diffusion controlled and increased linearly with concentration in the range used for the determination.

The method of standard addition was preferred because it is more rapid than the concentration–diffusion current plot method.

Table I—Results of 10 Polarographic Analyses of Tinidazole in Tablets[a]

Analysis	Tinidazole per Tablet, mg
1	150
2	148
3	156
4	153
5	147
6	151
7	151
8	146
9	149
10	154
Average	150.5
SD	±2%

[a] Fasigyn tablets, Pfizer; analyzed by Pfizer Specification No. 3-FPS-255. This sample contains 152 mg of tinidazole/tablet.

Since the currents are diffusion controlled and possess the normal temperature coefficient of less than 2%/degree, no thermostating of the cell is necessary and the curves for the solution to be analyzed and for the solution after standard addition are recorded at room temperature.

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Isolation of Pinastric Acid and Ergosterol from *Parmelia caperata* (L.) Arch.

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Abstract □ Among other common compounds, pinastric acid and ergosterol were isolated for the first time from *Parmelia caperata*. The isolation of these compounds is described; identification was made from the melting point and UV, IR, and mass spectral data.

Keyphrases □ Pinastric acid—isolated from *Parmelia caperata*, UV, IR, and mass spectral data □ Ergosterol—isolated from *Parmelia caperata*, UV, IR, and mass spectral data □ *Parmelia caperata*—isolation of pinastric acid and ergosterol, UV, IR, and mass spectral data □ Lichens—*Parmelia caperata*, isolation of pinastric acid and ergosterol, UV, IR, and mass spectral data

Parmelia caperata (L.) Arch. is a widely studied lichen (1), but there are no references to the identification of pulvinic acid derivatives and sterols among the several compounds isolated from this species. The present paper reports the extraction and identification of pinastric acid and ergosterol from a Portuguese specimen of *P. caperata*, in which several common compounds, such as (+)-usnic, caperatic, and protocetraric acids, also were found.

Pinastric acid is a methoxy derivative of vulpinic acid, the latter having shown antibiotic activity (2). Because of its chemical constitution, pinastric acid cannot be considered an artifact formed during extraction but may be a term in a probable biogenetic chain: pulvinic acid → vulpinic acid → pinastric acid → leparinic acid → leparinic acid methyl ether.

Ergosterol is widely distributed in plants. Although it has been found in several lichens (1), its isolation from *P. caperata* has not been reported. This compound also was identified by GC–mass spectrometry in the unsaponifiable matter of the same lichen species, along with several other sterols (3) presenting a common biogenetic relationship.

EXPERIMENTAL[1]

The air-dried lichen[2] (330 g) was extracted in a soxhlet apparatus with petroleum ether (bp 40–60°) until the solvent was colorless. By concentration of this extract under reduced pressure, an amorphous product was formed (2.5 g after washing with petroleum ether and drying). It showed, on TLC [Kieselgel G[3] plates activated at 110° for 30 min, thickness ~0.25 mm, developed with chloroform–acetone (80:20)], a spot corresponding to (+)-usnic acid, which was isolated and identified by melting point, $[\alpha]_D$, and UV, IR, and mass spectral data (1, 4, 5).

Further concentration of the petroleum ether extract to 50 ml gave another amorphous substance (675 mg), 105 mg of which was extracted by methanol. The resulting orange solution was submitted to TLC as described previously and showed spots corresponding to usnic acid, atranorin, and chloratranorin (?). It also showed a blue spot (R_f ~0.5), after spraying with anisic aldehyde (6), and a yellow spot (R_f ~0.4) in visible light, became red under UV (350 nm). On concentration and cooling of the methanolic solution, a small amount of orange rectangular plates was formed.

Recrystallization from methanol gave 5 mg of pinastric acid (corresponding to the yellow-red spot), mp 204–206°; UV: λ_{max} (methanol) 293 and 388 nm; λ_{min} 238 and 338 nm; IR: ν_{max} (potassium bromide) 3020, 2960, 2840, 2540, 1775, 1765, 1680, 1600, 1575, 1520, 1465, 1450, 1440, 1420, 1370, 1335, 1310, 1280, 1260, 1195, 1160, 1110, 1090, 1070, 1030, 965, 910, 850, 840, 820, 790, 775, 740, 710, and 700 cm^{-1}; mass spectrum: M^+ = 352 (24%), *m/e* (%) 353 (5), 321 (20), 320 (100), 308 (6), 294 (7), 293 (3), 292 (11), 278 (2), 265 (10), 264 (50), 237 (10), 234 (2), 209 (14), 208 (80), 175 (14), 165 (10), 164 (2), 149 (6), 148 (20), 147 (50), 146 (10), 145 (40), 135 (10), 120 (11), 119 (56), 118 (8), 117 (28), 105 (18), 91 (19), 90 (14), 89 (71), 77 (19), 76 (18), 65 (18), and 63 (18); m* 164 (264 → 208); m* 207.5 (264 → 234); m* 264 (293 → 278); m* 291 (352 → 320).

By concentration of mother liquors from which pinastric acid was separated, a small amount of colorless plates was obtained (7 mg). Recrystallization from methanol gave 5 mg of ergosterol[4], mp 159–161° (Liebermann positive). Only one spot (the blue one referred to previously) was obtained on TLC [chloroform–acetone (80:20) and benzene–dioxane–acetic acid (90:25:4)]; UV: λ_{max} (methanol) 271, 282, and 293 nm; IR: ν_{max} (potassium bromide) 3390, 2900, 2840, 1667, 1471, 1370, 1070, 1042, 990, 971, 835, and 800 cm^{-1}; mass spectrum: M^+ = 396 (100%), *m/e* (%) 397 (30), 337 (50), 271 (45), 253 (56), 251 (23), 211 (49), 199 (28), 197 (30), 185 (28), 183 (28), 158 (65), 157 (70), 145 (78), 143 (85), 119 (58), 109 (55), 107 (60), and 105 (75).

[1] Melting points were determined on a Kofler microscope and are uncorrected. UV spectra were determined on a Bausch & Lomb Spectronic 505 spectrophotometer. IR spectra were determined on a Perkin-Elmer 257 instrument, and only the major bands are quoted. Mass spectra were recorded on a Hitachi RMU-6M with an ionizing potential of 70 ev.

[2] The plant material was collected from *Pinus Pinaster* Sol. trees at Cortegaça, Portugal, in May 1972 and identified as *P. caperata* by Prof. C. Tavares. A voucher specimen is deposited in the Laboratório de Quimica Orgânica, Faculdade de Farmácia, Universidade do Porto, Portugal.

[3] Merck.

[4] All analytical determinations were compared with an authentic sample.

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Quantitative NMR Analysis of a Four-Component Mixture of Phenylglycine Derivatives

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Abstract □ A rapid, accurate, and precise NMR analytical method for the analysis of phenylglycine, dihydrophenylglycine, tetrahydrophenylglycine, and cyclohexylglycine in combination with each other was developed. The method is based on the integration of the NMR signal characteristic of each component relative to the signal from tetramethylammonium bromide, which is added as an internal standard. No prior separation of the four components is required.

Keyphrases □ Phenylglycine and derivatives—NMR analysis in four-component mixture □ NMR spectroscopy—analysis, phenylglycine and derivatives in four-component mixture

NMR spectroscopy is being used increasingly for quantitative analysis of pharmaceuticals in dosage forms (1, 2) and chemical (3, 4) and isomeric (5) mixtures. The method offers advantages of speed, relatively good precision and accuracy, and ease of execution, and four components do not need to be sepa-