

The filtrate from the first precipitation was chromatographed on filter paper using 77% ethanol as solvent to show the presence of DL- $\beta$ -phenylalanine in the hydrolysate.

**DL- $\beta$ -Phenylalanine-N-acetic Acid Hydrochloride.**—Sixty mg. of the DL- $\beta$ -phenylalanine-N-acetic acid was converted to the hydrochloride<sup>2</sup> to give 50 mg. of material, m.p. 203–206° dec. (lit.<sup>2</sup> 200–201°).

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(7) Federal Security Agency, Public Health Service.

### A Note on the Occurrence of Panstroside<sup>1</sup>

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RECEIVED MARCH 19, 1952

v. Euw and Reichstein<sup>2</sup> have reported on the glycoside content of the seeds of *Strophanthus petersianus*, Klotzsch, from which they isolated sarmentocymarin, sarmentogenin and three new substances identified as No. 792, 793 and 794. We find that in addition the seeds of this species contain panstroside which we isolated in a crude yield of 0.035%.

The seeds were received from Mr. P. Topham, Nyasaland, Africa, through the courtesy of the New York Botanical Garden. A flowering specimen and a mericarp sent as botanical vouchers for the seeds were identified as *S. petersianus* at the New York Botanical Gardens. We wish to thank Mr. Topham for the collection and Mr. Joseph Monachino for the botanical examination of this sample.

Panstroside frequently crystallizes slowly or not at all<sup>3</sup> and may be easily overlooked. When the presence of this glycoside is suspected we regularly crystallize the chloroform extract from acetone from which panstroside crystallizes more readily than from methanol and ether.

#### Experimental

The seeds (1111 g.) were extracted as previously described.<sup>4</sup> The chloroform extract on concentration gave 2.58 g. (0.23%) of crude total glycoside. This was dissolved in about 10 cc. of acetone, and allowed to crystallize in the refrigerator. We obtained 366 mg. of crude panstroside,  $[\alpha]_D +25^\circ$ ; m.p. 210–219°. An additional 19 mg. was isolated by chromatography of the mother liquors.

Recrystallization of this panstroside gave preparations that agreed in rotation and melting point with our former preparations but the absorption spectrum showed that a small amount of impurity was retained tenaciously. It was accordingly chromatographed on alumina, and from the eighth and ninth fractions eluted with chloroform and chloroform-methanol (99:1), 193 mg. of panstroside was obtained after crystallization from methanol;  $[\alpha]_D +27^\circ$ . Panstroside was recrystallized three times and yielded 154 mg.;  $[\alpha]_D +31.0^\circ$ ; m.p. 222–228°; Keller-Kiliani test negative; legal test positive. Color test with 84% sulfuric acid pink becoming red in 2 min., developing a blue edge in 3 min., and becoming blue in 20 min. The mixed melting point with panstroside from *S. intermedius* showed no depression. Ultraviolet absorption spectrum maximum at 218 m $\mu$ , log  $\epsilon$  4.23; plateau at 265–280 m $\mu$ , log  $\epsilon$  1.93.

*Anal.* Calcd. for C<sub>30</sub>H<sub>44</sub>O<sub>11</sub>: C, 62.06; H, 7.64. Found: C, 61.84, 61.63; H, 7.43, 7.61.

(1) J. v. Euw, H. Hess, P. Speiser and T. Reichstein, *Helv. Chim. Acta*, **34**, 1821 (1951).

(2) J. v. Euw and T. Reichstein, *ibid.*, **33**, 1551 (1950).

(3) See for example J. v. Euw and T. Reichstein, *ibid.*, **33**, 2153 (1950).

(4) M. R. Salmon, Eric Smith and W. G. Bywater, *THIS JOURNAL*, **78**, 3624 (1951).

We wish to thank Dr. W. G. Bywater for advice and suggestions and the Upjohn Company for encouragement and support.

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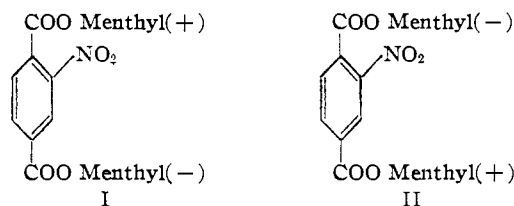
### Active (+) Menthyl (–) Menthyl Nitroterephthalates

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RECEIVED APRIL 25, 1952

Compounds containing two like asymmetric groups of opposite configuration, A(+) and A(–), and no other asymmetric groups, but which nevertheless have molecular asymmetry, are of interest in that they have some bearing on the entrenched idea of internal compensation. It might be argued that the rotation of A(+) should cancel that of A(–) or that, if it did not, the rotation of the compound would be small. An example of such compounds would be those of the type (+)A-Z-X-Y-Z-A(–) and (–)A-Z-X-Y-Z-A(+), where X, Y and Z are not asymmetric. It is difficult to be certain that this type of compound has not been discussed or prepared previously, but an examination of several available books on stereochemistry and a search of the literature for likely specific compounds have not revealed a reference to this aspect of the problem.

We have prepared one pair of enantiomorphs of this type, namely, the 1-(+)menthyl 4-(–)menthyl 2-nitroterephthalate (I) and the 1-(–)menthyl 4-(+)menthyl 2-nitroterephthalate (II). These com-



pounds have specific rotations in benzene solution of +59.1 and –59.7°, respectively. Thus the molecular asymmetry not only confers activity on these compounds, but the rotation is appreciable.

#### Experimental

**Nitroterephthalic acid** was prepared from terephthalic acid in 78% yield by a procedure essentially the same as that of Wegscheider<sup>1</sup> except that 70% nitric acid and 30% fuming sulfuric acid were used instead of fuming nitric acid and pyrosulfuric acid. It was converted to the (–)menthyl ester by the procedure of Cohen and de Pennington<sup>2</sup> except that, in the preparation of the acyl chloride, twice the calculated amount of phosphorus pentachloride was used and a small amount of phosphorus oxychloride was added to start the reaction. The purified ester, obtained in over-all yield of 59%, melted at 86–88°;  $[\alpha]_D^{25} -159^\circ$  (c 1.996 in benzene).

1-(–)Menthyl 2-nitroterephthalate was obtained in 42% yield by the partial saponification of the (–)menthyl ester.<sup>2</sup> After crystallization from aqueous ethyl alcohol, it melted at 73.5–75°, compared to the 75° previously reported. Recrystallization from hexane, however, raised the melting point to 128.5–129.5°. A determination of neutralization equivalents showed that the product melting at 75° contains one molecule of water of crystallization, whereas that melt-

(1) R. Wegscheider, *Monatsh.*, **21**, 621 (1900).

(2) J. B. Cohen and H. S. de Pennington, *J. Chem. Soc.*, **113**, 57 (1918).