of the compounds prepared are stated in Table I. Previously reported compounds are noted therein.

Experimental

α-(*p*-Ethylbenzenesulfonamido)-isocaproic Acid.—A solution of 7.72 g. (0.038 mole) of *p*-ethylbenzenesulfonyl chloride, prepared as described elsewhere,[3] in 25 ml. of acetone was added slowly from a separatory funnel to a mechanically stirred solution of 5.0 g. (0.038 mole) of L-leucine in 55 ml. of 2 *N* sodium hydroxide and 25 ml. of acetone. During the addition the mixture was cooled in an ice-bath. Water was added to dissolve the precipitated sodium chloride. The acetone was removed under vacuum at 60° and the cooled solution was acidified to thymol blue with concd. hydrochloric acid. The precipitated crystals were collected and dried. The yield of crude material was 5.48 g. or 48% of the theoretical amount. Recrystallization from hot water gave crystals, m.p. 117–118°, used in the analyses.

α-(*p*-Ethylbenzenesulfonamido)-isocapramide.—Three grams (0.01 mole) of the acid as prepared above was dissolved in 10 ml. of purified thionyl chloride and heated on a steam-bath to solution. The gaseous products and excess thionyl chloride were removed under vacuum leaving a viscous liquid. Excess concd. ammonium hydroxide was added to this liquid. The crude reaction mixture was treated with 200 ml. of water. The crystals were collected and dried. The yield was 1.96 g. or 65% of the theoretical amount. Recrystallization from 95% ethanol gave crystals, m.p. 202–203°, used in the analysis.

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[3] R. H. Wiley and R. P. Davis, THIS JOURNAL, **74**, 6142 (1952).

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Improved Preparation of Stachyose

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The tetrasaccharide stachyose was first isolated by von Planta and Schulze[2] from the root sap of *Stachys tuberifera*. Although stachyose has been found in a number of other plants, the tuber of *Stachys tuberifera* is still one of the better sources of this sugar. The method of isolation has been modified by Tanret.[3]

We wish to report herein a simple procedure for obtaining stachyose from the tuber of *Stachys tuberifera* wherein the organic solids in the extract of the tuber were adsorbed on a column of carbon and removed selectively by washing with increasing strengths of ethanol, according to the general adaptation of Whistler and Durso.[4] From the eluates the sugar could readily be crystallized.

Experimental

Fresh tubers of *Stachys tuberifera* (1 kg.) were ground in a food chopper and were refluxed on a water-bath with 1750 ml. of 85% ethanol for 80 min. in the presence of 2.5 g. of precipitated calcium carbonate. The liquid was removed by filtration and the extraction was repeated with 500 ml. of 75% ethanol for 60 min. The extract was filtered and combined with the first filtrate to produce a clear brownish solution which was concentrated under reduced pressure to a sirup; yield 80 g. The sirup was dissolved in 1000 ml. of water and placed on a 290 × 105 mm. (i.d.) column of Darco G-60[5]:Celite[6] (1:1 by wt.) and washed with 10 l. of water. This solution was evaporated under reduced pressure to a sirup which failed to crystallize; yield 17 g. The column was then washed with 10 l. of 5% ethanol. Upon evaporation under reduced pressure, this solution yielded a sirup (13 g.) which was crystallized by dissolving in the minimum amount of warm water, cooling to room temperature, adding ethanol (95%) to incipient crystallization and allowing to stand at room temperature. The column was again washed with 10 l. of 10% ethanol. Upon evaporation this solution yielded 35 g. of sirup which crystallized from water–ethanol as described above. A further yield of sirupy material was obtained by washing the column with 10 l. of 20% ethanol. The sirup from this fraction showed only a slight tendency to crystallize. This residue was combined with the mother liquors from the 5 and 10% ethanol washings and evaporated to a sirup which was extracted with 500 ml. of boiling 85% ethanol to yield a further amount of 6 g. of crystals. The combined crystalline material was recrystallized from water by the addition of ethanol; yield of recrystallized material 39 g., m.p. 101–105° (sealed tube), $[\alpha]^{25}\text{D}$ +131.3° (*c* 4.5, water). These constants are in good agreement with those given in the literature for stachyose tetrahydrate.[3]

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[1] Corn Industries Research Foundation Associate.

[2] A. von Planta and E. Schulze, *Ber.*, **23**, 1692 (1890).

[3] G. Tanret, *Bull. soc. chim.*, [4] **13**, 176 (1913).

[4] R. L. Whistler and D. F. Durso, THIS JOURNAL, **72**, 677 (1950).

[5] Decolorizing carbon; a product of Darco Department, Atlas Powder Co., New York, N. Y.

[6] No. 535, a siliceous filter-aid produced by Johns-Manville Co. New York, N. Y.

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The Reaction of Phosphorus Pentachloride with Epimeric 2-Aminocycloalkanol Hydrochlorides

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During the course of work being carried out in this Laboratory, it became desirable to investigate the synthesis of diastereoisomeric 2-chlorocycloalkylamines through the reaction of phosphorus pentachloride with *cis*- and *trans*-2-aminocycloalkanol hydrochlorides. The results of these studies, together with evidence for the stereochemistry of the products, are presented below.

The synthesis of a 2-chlorocyclohexylamine hydrochloride (IIa) from *trans*-2-aminocyclohexanol hydrochloride (Ia) by this route was first reported by Osterberg and Kendall.[2] Direct evidence for the *cis* configuration (and therefore displacement by phosphorus pentachloride with inversion) has been presented by Carter, *et al.*,[3] and, more recently, by Paris and Fanta.[4] The isomeric 2-chlorocyclohexylamine and derivatives have been obtained by several methods.[4–7] Attempts to obtain this *trans* isomer through the action of phosphorus pentachloride on the hydrochloride of *cis*-2-aminocyclohexa-

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[1] Abstracted from a research report submitted by Richard S. Wilson in partial fulfillment of the requirements for the Master of Science degree, University of Wisconsin, February, 1952.

[2] A. E. Osterberg and E. C. Kendall, THIS JOURNAL, **42**, 2616 (1920).

[3] G. E. McCasland, R. K. Clark, Jr., and H. E. Carter, *ibid.*, **71**, 637 (1949).

[4] O. E. Paris and P. E. Fanta, *ibid.*, **74**, 3007 (1952).

[5] G. E. McCasland and D. A. Smith, *ibid.*, **72**, 2190 (1950); **71**, 637 (1949).

[6] W. S. Johnson and E. N. Schubert, *ibid.*, **72**, 2187 (1950).

[7] T. L. Cairns, P. J. Graham, P. L. Barrick and R. S. Schreiber, *J. Org. Chem.*, **17**, 751 (1952).