NOTES

Isolation and Identification of the Organic Nitrogenous and Non-Nitrogenous Compounds Occurring in the Alaska Pea. II. The Isolation of Pyrrolidone-carboxylic Acid, Tyrosine, and Diamino Acids from the Non-Proteins of the Alaska Pea

By S. L. Jodidi

Previously the presence of citric acid[1] in Alaska pea was reported. Work on phenylalanine and cystine was undertaken because they were never reported to occur among the non-proteins of the pea. Phenylalanine is known to be hard to isolate. However, when heated with potassium dichromate plus sulfuric acid it develops the odor of an aldehyde which oxidizes to benzoic acid. This reaction which was used for elaborating a quantitative method for the estimation of phenylalanine as benzoic acid[2] was applied to the work on that amino acid.

Four hundred grams of meal prepared from Alaska peas of sizes 2, 3 and 4 was treated twice with ether to remove fat, lecithin and pigments. The defatted meal was extracted with 92% alcohol, and the filtered extract evaporated *in vacuo* to dryness. The residue was oxidized by boiling with Beckmann's mixture (20% of potassium dichromate + 17% of sulfuric acid) for five to six hours. The oxidation liquid was extracted with ether in a continuous extractor[3] and the ethereal layer filtered into anhydrous sodium sulfate. The dehydrated filtered ether solution left on evaporation crystals whose aqueous solution showed acid reaction. Recrystallized from alcohol they melted at 182°. Benzoic acid melts at 120–121°, pyrrolidonecarboxylic acid at 182–183°.

Anal. Silver salt. Calcd. for $C_5H_6NO_3Ag$: Ag, 45.76. Found: Ag, 45.67.

The acid reaction, melting point and silver salt agree with the properties of pyrrolidonecarboxylic acid. Its origin is undoubtedly due to glutamine[4] present in peas.

For isolation of cystine, 500 g. of pea meal was extracted with water, the extract evaporated in a vacuum to dryness, and the residue treated with absolute alcohol to remove citric acid, fats, lecithin, etc. The undissolved portion was dissolved in water, purified with basic lead acetate, the filtrate treated with hydrogen sulfide, and the filtrate from lead sulfide concentrated *in vacuo*. The concentrate was treated repeatedly with mercuric sulfate in a 5% sulfuric acid solution. The mercuric sulfate precipitates obtained were suspended in water, decomposed with hydrogen sulfide, the filtrate from mercuric sulfide neutralized with potassium hydroxide solution, treated with bone black and the filtrate concentrated in a vacuum. The concentrated solution yielded in a vacuum desiccator crystals which failed to yield with alkali and lead acetate the cystine reaction, but they gave the Millon and Mörner reactions characteristic of tyrosine, and the pyrrole and diazo reactions, showing the presence of histidine.

To obtain arginine, the filtrate from the mercuric sulfate precipitates was treated with hydrogen sulfide, the filtrate from the mercuric sulfide acidified with sulfuric acid and treated with flavianic acid. There was thus obtained 2.25 g. of arginine flavianate.

Anal. Calcd. for $C_{16}H_{20}N_6SO_{10}$: N, 17.22; S, 6.57. Found: N, 17.31; S, 6.75.

The filtrate from arginine flavianate which probably contained lysine was lost by accident.

Summary.—Pyrrolidone-carboxylic acid, the isolation of which from peas is reported here for the first time, appears to owe its origin to glutamine occurring in that legume. The presence of histidine was demonstrated by the diazo and pyrrole reactions. The quantity of arginine obtained was equal to 0.7% of the oven-dried peas, while tyrosine was identified by Millon and Mörner reactions.

Division of Fruit and Vegetable
Crops and Diseases
U. S. Department of Agriculture
Washington, D. C.
Received March 6, 1935

---

(1) Jodidi, This Journal, **55**, 4663 (1933).

(2) Kollmann, *Biochem. Z.*, **194**, 1 (1928).

(3) Palkin, Murray and Watkins, *Ind. Eng. Chem.*, **17**, 612 (1925).

(4) Schulze und Winterstein, *Z. physiol. Chem.*, **65**, 431 (1910).

The Preparation of Mercury Diisobutyl

By Adolf Müller

As has been long known, ethyl iodide[1] and ethyl bromide[2] react readily with sodium amalgam to form the mercury diethyl. H. F. Lewis and E. Chamberlin[3] found, however, that isobutyl iodide yielded 50–55% of the mercury compound, whereas isobutyl bromide yielded only traces under the same conditions.

I have succeeded in preparing in several experiments mercury diisobutyl from the bromide under conditions very similar to the modified procedure of A. Müller and A. Sauerwald for preparing mercury diethyl.[4] However, the yield of the ethyl compound was about 44% and that of the isobutyl compound 27%.

The proportions used were one mole of diisobutyl bromide, 0.15 mole of ethyl acetate, 1.5 to 2 moles of sodium contained in 0.5 to 1% amalgam.

---

(1) Frankland and Duppa, *J. Chem. Soc.*, **16**, 418 (1863); *Ann.*, **130**, 109 (1864).

(2) Chapman, *J. Chem. Soc.*, **19**, 150 (1866); *Ann.*, **139**, 128 (1866).

(3) Lewis and Chamberlin, This Journal, **51**, 291 (1929).

(4) A. Müller and A. Sauerwald, *Monatsh.*, **48**, 738 (1927).