Analytical data from benzoyl phosphate preparation

	Calculated for $C_6H_5 \cdot COO \cdot PO_3Li_2$ (%)	Found (%)
Hydrogen	2.35	2.64
Carbon	39.29	39.31
Lithium	6.48	6.44
Total P	14.48	15.00
Free P		0.4
Carboxyl phosphate [a]		99.6
Carboxyl phosphate [b]		101.9

[a] Hydroxylamine-ferric chloride method[5]. [b] Enzymatic method using acyl phosphatase.

phosphate has been synthesized previously as follows: 1. reaction between monosilver phosphate and benzoyl chloride; 2. isolation of silver salt of benzoyl phosphoric acid; 3. conversion into lithium salt; 4. purification of lithium benzoyl phosphate. In the present work, a simple and rapid method for benzoyl phosphate synthesis is reported.

Materials and methods. Benzoyl phosphate synthesis. This product was prepared as follows: 120 ml of water, 76 ml of pyridine, and 40 ml of 1 *M* K_2HPO_4 (40 mmol) were mixed. 20 g (90 mmol) of benzoic anhydride was added with stirring to the above solution. The reaction was carried out at room temperature until benzoic anhydride was completely dissolved (for about 10 min). Then 140 ml of 1 *M* LiOH were added and the mixture was poured into 2.4 l of cold ethanol, with stirring. After 30 min at −20°, the precipitate was collected by centrifugation, washed with cold ethanol and then with cold ether. The product was left to dry overnight under vacuum. 5.6 g of product was obtained, containing 73.5% of benzoyl phosphate.

Purification. A 2% solution of the product in cold water was adjusted to pH 6 with acetic acid. The undissolved material was discarded. Then 0.37 volumes of cold ethanol were slowly added with stirring. The suspension was kept at −20° for 30 min, then the precipitate was collected by centrifugation and washed with cold ethanol and ether. The product was about 95% pure.

The analyses described below were carried out on a product which has been further purified by the same procedure. Analytical methods. Hydrogen and carbon. The H and C content was determined by using a mod. Canal-Terzano apparatus. Carboxyl phosphate. The determination of carboxyl phosphate was carried out by 1. the hydroxylamine-ferric chloride method according to LIPMANN and TUTTLE[5]. As a reference standard benzoic anhydride was used. 2. By an enzymatic method. For this purpose we measured the extinction change of a solution of the product which was hydrolyzed completely by using a small amount of horse muscle acyl phosphatase. An extinction change coefficient at 283 nm of 0.630 mM^{-1} cm^{-1} was used[3]. Inorganic phosphate determination. The total phosphate content was determined by the method of FISKE and SUBBAROW[6]. The product was first hydrolyzed with 5 *N* H_2SO_4 at 100° for 30 min. Free phosphate was determined by the method of Baginsky et al.[7].

Lithium content. The determination was carried out by flame emission spectrophotometry using a Beckman DK-1A apparatus, at 670.8 nm. UV-spectra were obtained using a Beckman DK-1A recording spectrophotometer; IR-spectra by a Perkin-Elmer mod. 457 apparatus.

Results and discussion. In the Table the analytical data obtained for our benzoyl phosphate preparation are reported. From these data can be concluded that the preparation is > 98% pure.

As regards UV-spectra (pH 5.3), the following data have been obtained: before hydrolysis : absorption maxima at 232 and 274 nm; shoulder at 283 nm; after hydrolysis: absorption maxima at 224 and 267 nm; shoulder at 262 and 276 nm as expected[2,3]. IR-spectrum of dilithium benzoyl phosphate shows a band at 1025 cm^{-1} that has been attributed to a P-O-C(alkyl) bond[8] (the bond formed by the synthesis). It can also be noted 1. a C=O band at 1700 cm^{-1}, 2. a P=O band at 1340 cm^{-1}, 3. the absence of bands in the range 2500–2700 cm^{-1}, which confirms the absence of free —OH groups as expected for the structure of dilithium benzoyl phosphate. The product obtained after one purification step is pure enough to be used as substrate for acyl phosphatase assay.

[5] F. LIPMANN and L. C. TUTTLE, J. biol. Chem. *153*, 571 (1944).
[6] C. H. FISKE and Y. SUBBAROW, J. biol. Chem. *66*, 375 (1925).
[7] E. S. BAGINSKY, P. P. FOA and B. ZAK, Clin. chim. Acta *15*, 155 (1967).
[8] W. OTTING, in *Spektrale Zuordnungstafel der Infrarot-Absorptionsbanden* (Springer-Verlag, Berlin 1963), p. 15.

CONGRESSUS

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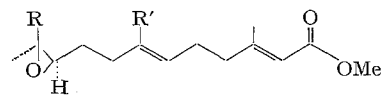
CORRIGENDUM

P. PAGUIA, P. MASNER, K.-H. TRAUTMANN and A. SCHULER: *Juvenile Hormone Active Principle in Attacus atlas* L., Experientia *32*, 122 (1976). The formulae on page 122 should read correctly as follows: