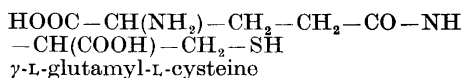


In the seeds it can partly be present in reduced form which is oxidized during the treatment of the plant material and isolation procedure



Peptide R X, γ -L-glutamyl-S-propyl-L-cysteine. This peptide was eluted from the Dowex 1 \times 8 column partly in the same fractions as R IX (Fig. 1). They are poorly separated from each other also on the cellulose powder column and on the paper chromatogram. Peptide X was, however, isolated in pure form and its structure could be elucidated. It proved to be γ -L-glutamyl-S-propyl-L-cysteine. Because this peptide was earlier isolated in this laboratory² from garlic (*Allium sativum*) it is not treated in this preliminary communication.

This research has been financed in part by a grant from the *United States Department of Agriculture, Agricultural Research Service*.

Our thanks are due to Mr. T. Moisio, M. A., for his mass spectrometric determinations.

1. Virtanen, A. I. and Matikkala, E. J. *Suomen Kemistilehti B* 35 (1962) 2461; Matikkala, E. J. and Virtanen, A. I. *Acta Chem. Scand.* 16 (1962) 2461.
2. Virtanen, A. I., Hatanaka, M. and Berlin, M. *Suomen Kemistilehti B* 35 (1962) 52.

Received June 27, 1963.

Facile Preparation of 2-Acetylcyclopentane-1,3-dione and 2-Acetylcyclohexane-1,3-dione

FERENC MERÉNYI and MARTIN NILSSON

Division of Organic Chemistry, Royal Institute of Technology, Stockholm 70, Sweden

2-Acetylcyclopentane-1,3-dione was needed for further spectroscopic investigations of hydrogen bonding in enolised β -tricarboxyl compounds¹⁻⁵. A solid believed to be this compound was obtained by Sieglitz and Horn⁶ in 0.2 %

yield from succinyl chloride, vinyl acetate and aluminium chloride in an unsuccessful attempt to make cyclopentane-1,3-dione. The 2-acetylcyclopentane-1,3-dione was presumably formed in a secondary reaction. For the present purpose a corresponding diacylation of isopropenyl acetate seemed more promising.

We found that 2-acetylcyclopentane-1,3-dione can be obtained in ca. 45 % yield from succinyl chloride and isopropenyl acetate in 1,2-dichloroethane or 1,1,2,2-tetrachloroethane in the presence of aluminium chloride. Although preparatively useful, this reaction seemed to involve an excess of "acylating power". Stoichiometrically, the reaction between succinic anhydride and isopropenyl acetate should give the desired product and we have indeed found that this reaction gives up to 55 % 2-acetylcyclopentane-1,3-dione. The corresponding reaction with glutaric anhydride gives a 40 % yield of 2-acetylcyclohexane-1,3-dione, previously obtained in 25 % yield by *C*-acetylation of cyclohexane-1,3-dione⁷.

In these reactions varying amounts of cyclopentane-1,3-dione and cyclohexane-1,3-dione, respectively, are formed. These compounds may also be obtained by hydrolysis of the corresponding triketones. This opens a new route to the otherwise rather inaccessible cyclopentane-1,3-diones (cf. Refs. 8-10).

The present method for diacylation of isopropenyl acetate seems to be fairly general¹¹. Some further applications will be described shortly.

Experimental. 2-Acetylcyclopentane-1,3-dione. Succinic anhydride (0.1 mole) and anhydrous aluminium chloride (0.2 mole) are suspended in 1,2-dichloroethane (100 ml). Isopropenyl acetate (0.1 mole) is added with stirring. The reaction brings the temperature to ca. 70°. The mixture is refluxed for 15 min., left to cool and is then poured into a mixture of dilute hydrochloric acid (250 ml, 2 M) and crushed ice (250 g). The organic phase is separated and shaken with dilute hydrochloric acid. The combined aqueous phases are extracted continuously with chloroform overnight. The combined dichloroethane and chloroform solutions are dried (sodium sulphate) and the solvents removed. The solid residue contains acetylcyclopentanedione, some cyclopentanedione, and succinic acid. The acetyl compound is isolated in ca. 50 % yield by repeated extractions with boiling light petroleum or by sublimation at 60° and 0.1 mm.

Recrystallisation from light petroleum (b.p. 40–60°) and sublimation gives the pure product, m.p. 73–74°. Lit.⁶ m.p. 69–71°. (Found: C 59.5; H 5.9. Calc. for C₇H₈O₃: C 60.0; H 5.8.) The infrared spectrum (carbon tetrachloride) contains strong bands at 1710, 1635, and 1595 cm⁻¹. The ultraviolet spectrum (cyclohexane) shows maxima at 2650 Å ($\epsilon = 7040$) and 2200 Å ($\epsilon = 11\,800$). 2-Acetylcyclopentane-1,3-dione dissolves readily in chloroform, water and alcohols but is less soluble in light petroleum or in ether. With copper(II) acetate it gives a greenish blue copper salt, which loses solvent at about 110° and melts with decomposition at 325°.

When heated with water or dilute acids acetylcyclopentanedione gives cyclopentane-1,3-dione in moderate to good yields. The sublimed product melts at 150–152° (lit.⁸ m.p. 151.5–152.5°) and its infrared spectrum (KBr) is identical with that reported⁸.

2-Acetylcyclohexane-1,3-dione. The corresponding reaction with glutaric anhydride in dichloromethane or 1,2-dichloroethane without the final refluxing gives a mixture from which 2-acetylcyclohexane-1,3-dione is conveniently isolated by distillation in 40% yield, b.p. 63–68°, 0.1 mm. The melting point, 29–32°, and the infrared spectrum agree with those given by Smith⁷. Extraction of the distillation residue with boiling ethyl acetate and subsequent sublimation gives ca 10% crude cyclohexane-1,3-dione, m.p. 98–102°.

Acknowledgements. The work has been supported by the *Swedish Technical Research Council*, the spectra have been recorded by Miss Gurli Hammarberg and the English has been checked by Dr. R. E. Carter.

1. Forsén, S. and Nilsson, M. *Acta Chem. Scand.* **13** (1959) 1383.
2. Forsén, S. and Nilsson, M. *Acta Chem. Scand.* **14** (1960) 1333.
3. Forsén, S. and Nilsson, M. *Arkiv Kemi* **17** (1961) 523.
4. Forsén, S., Nilsson, M. and Wachtmeister, C. A. *Acta Chem. Scand.* **16** (1962) 583.
5. Forsén, S. and Nilsson, M. *Arkiv Kemi* **19** (1963) 569.
6. Sieglitz, A. and Horn, O. *Chem. Ber.* **84** (1951) 607.
7. Smith, H. J. *J. Chem. Soc.* **1953** 803.
8. Boothe, J. H., Wilkinson, R. G., Kusher, S. and Williams, J. H. *J. Am. Chem. Soc.* **75** (1953) 1732.
9. DePuy, C. H. and Zaweski, E. F. *J. Am. Chem. Soc.* **81** (1959) 4920.

10. Roedig, A. and Ziegler, H. *Chem. Ber.* **94** (1961) 1800.

11. Nilsson, M. and Merényi, F. *Swedish Patent Application* 5285 (1963).

Received July 5, 1963.

Correction to "Activation of Mitochondrial Propionyl-CoA Carboxylase" *

HALINA Y. NEUJAHN and
S. P. MISTRY

Division of Animal Nutrition, University of Illinois, Urbana, Illinois, U.S.A.

In Table 11, last column, first line

for 932 read 632

Received August 9, 1963.

* *Acta Chem. Scand.* **17** (1963) 1140.

Corrections to "Inhibition of Photophosphorylation in Isolated Spinach Chloroplasts by Lower Aliphatic Straight-Chain Alcohols" *

HERRICK BALTSCHOFFSKY**

Department of Zoology, University of California, Los Angeles, USA

On p. S 309, fourth line from above
for 0.6 μ moles PMS read 0.06 μ moles PMS

On p. S 310, legend to Fig. 1
for Preincubation with 1% (v/v) butanol
read Preincubation with 0, 1, 2 and 5% (v/v) butanol.

Received August 22, 1963.

* *Acta Chem. Scand.* **17** (1963) *Suppl.* **1**, S 308.

** Present address: Wenner-Gren Institute, University of Stockholm, Stockholm, Sweden.

Acta Chem. Scand. **17** (1963) No. 6