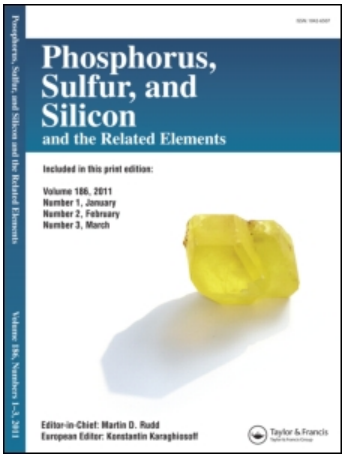

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

THE SYNTHESIS OF Se-(3-AMINOPROPYL)-SELENOSULFURIC ACID

Enrico Sanjust[a]; Maria Benedetta Fadda[a]; Giovanni Floris[a]; Augusto Rinaldi[a]
[a] Istituto di Chimica Biologica, Università di Cagliari, Cagliari

To cite this Article Sanjust, Enrico , Fadda, Maria Benedetta , Floris, Giovanni and Rinaldi, Augusto(1982) 'THE SYNTHESIS OF Se-(3-AMINOPROPYL)-SELENOSULFURIC ACID', Phosphorus, Sulfur, and Silicon and the Related Elements, 13: 3, 357 — 358
To link to this Article: DOI: 10.1080/03086648208081196
URL: http://dx.doi.org/10.1080/03086648208081196

Phosphorus and Sulfur, 1982, Vol. 13, pp. 357–358
0308-664X/82/1303-0357/$06.50/0

THE SYNTHESIS OF Se-(3-AMINOPROPYL)-SELENOSULFURIC ACID

ENRICO SANJUST, MARIA BENEDETTA FADDA, GIOVANNI FLORIS and AUGUSTO RINALDI*

Istituto di Chimica Biologica, Università di Cagliari, via della Pineta 77, 09100 Cagliari

(*Received April 4, 1982*)

Recently, it has been shown that Se-(2-aminoethyl)selenosulfuric acid ($H_2NCH_2CH_2$—$SeSO_3H$) or Se-sulfoselenocysteamine (SeSC) may undergo transamination with α-ketoglutaric acid in the presence of sonicated rat liver mitochondria giving acetaldehyde and selenosulfate as final products, in addition to glutamate.[1] In the same conditions selenohypotaurine may also transaminate[2] whereas other analogues, e.g. hypotaurine, taurine, selenotaurine and S-sulfocysteamine do not react. This is a very interesting example of an enzymic reaction in which the substitution of a selenium atom by a sulfur atom in the substrate molecule significantly affects the substrate specificity of the enzyme.

In order to investigate further the specificity of this enzyme, we have prepared the higher homologue of SeSC. In the present note we report details for the synthesis of Se-(3-aminopropyl)selenosulfuric acid ($H_2NCH_2CH_2CH_2$—$SeSO_3H$) or Se-sulfoselenohomocysteamine (SeSHC) carried out by reaction between 3-bromopropylamine and selenosulfate.[3,4]

1.58 g (0.01 mole) of freshly prepared potassium sulfite was dissolved in 40 ml of distilled water. 0.87 g (0.011 mole) of powdered selenium was added and the suspension, under continuous stirring, was heated at reflux temperature for 1 hr. In these conditions the bulk of selenium was dissolved. A solution of 3-bromopropylamine hydrobromide (10% in 80% ethanol) was added dropwise to the stirred selenosulfate solution maintained at reflux temperature. The addition was stopped when a drop of the reaction mixture added to 1 ml of 2N HCl did not separate red selenium within 30 sec. 15 ml of 96% ethanol was then added and the warm solution filtered by suction to remove salts and unreacted selenium. The precipitate was washed with a few ml of ethanol which was combined with the filtrate, then washed with water, dried and weighed. The clear filtrate was taken to dryness in a rotary evaporator at 50°C and the white residue was extracted with four 50 ml portions of boiling 96% ethanol. The combined extracts, after cooling at room temperature, were left at −20°C for 12 hrs. The finely powdered precipitate was recrystallized from water at 50°C. The crystals were collected by centrifugation, washed with absolute ethanol and dried.

1.79 g (88% yield based on reacted selenium) of colorless, birefringent crystals was obtained, which darkened at 145°C (Kofler uncorrected) and decomposed at

* Author to whom all correspondence should be addressed.

300°C. Analysis: found % (calcd for $C_3H_9NO_3SeS$): C, 16.47 (16.51); H, 4.26 (4.12); N, 6.42 (6.42).

The ^{1}H-NMR spectrum (Varian FT-80 A spectrometer, DSS as internal standard) shows a multiplet (2H) centered at δ2.27 attributed to the β-protons and a multiplet (4H) centered at δ3.29 due to the overlapping of the signals of α- and γ-protons.

IR: $-NH_3^+$, 3100, 1600, 1530 cm^{-1}; CH_2, 2900 cm^{-1}; $-SO_3^-$, 1150 cm^{-1}.

Thin layer chromatography (DC-Alufolien-Kieselgel from Merck) of SeSHC gives a single well-defined spot reactive to ninhydrin. Rf-values: pyridine-methanol-water (4:80:20) 0.65; collidine-lutidine (1:1, water saturated) 0.68; water-saturated phenol 0.31; butanol-formic acid-water (75:15:10) 0.09.

REFERENCES

1. G. Floris, A. Rinaldi, C. De Marco, *Bull. Mol. Biol. Med.*, **4**, 286 (1979).
2. A. Rinaldi, G. Floris, P. Cossu, C. De Marco, *Bull. Mol. Biol. Med.*, **3**, 234 (1978).
3. D. L. Klayman, *J. Org. Chem.*, **30**, 2454 (1965).
4. D. L. Klayman, W. H. H. Gunther, in "Organic Selenium Compounds" Wiley Interscience, N.Y., 1973, p. 49.