

pyridine-water (4:1) as solvent) as the sulfur-containing fragment in LBF hydrolysates.

Methyl pantothenate (1.5 moles), β -mercaptoethylamine (1.5 moles) and acetamide (0.0025 mole) were refluxed in two liters of methanol for eleven hours. Assay^{2,4} showed 2400 LBF units per mg. of product. Excess methyl pantothenate was hydrolyzed by treatment for one hour at room temperature with *N* methanolic KOH. After neutralization with methanolic HCl and removal of the solvent, the residue was partitioned between *n*-butanol and water. The butanol contained 18,000 LBF units per mg. of solids. After drying, the butanol solution was poured onto a Superfiltrol column (20 g. adsorbent per g. of solids), the latter washed thoroughly with butanol, then developed with water-saturated butanol. The most active fractions contained 29,000 to 30,000 LBF units per mg. of solids.

Though non-crystalline, this product appears essentially pure, and is indistinguishable from LBF prepared from natural sources in (a) R_F values on paper from water-saturated butanol (0.93) or *n*-amyl alcohol (0.76) and (b) activity for *Lactobacillus helveticus*, *Saccharomyces carlsbergensis*, and *Lactobacillus arabinosus*.² Both products give an immediate nitroprusside test only after reduction with NaCN; activity for *L. helveticus* was unchanged by such reduction. Both products are destroyed by digestion with a liver enzyme with release of 0.03 μ g. of calcium pantothenate per LBF unit. Thus LBF may exist as N-(pantothenyl)- β -aminoethanethiol or as the corresponding disulfide.

Anal. Calcd. for $C_{11}H_{22}O_4N_2S$: N, 10.07; S, 11.52; Found: N, 10.2; S, 12.0. *Pantetheine* and *pantethine* are suggested as names for the thiol and disulfide forms, respectively, of the growth factor. Its relation to coenzyme A was indicated previously.²

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AN EFFICIENT SEPARATION OF DYSPROSIUM AND YTTRIUM¹

Sir:

A rapid and efficient separation of the difficultly separable rare earth pair, dysprosium and yttrium, has been accomplished by elution with 5% ammonium citrate solution at room temperatures on Nalcite H.C.R. resin. This is achieved because of a shift in the order of elution of yttrium relative to the other rare earths. With 0.1% citrate

(1) This work was performed in the Ames Laboratory of the Atomic Energy Commission.

solution, the order of elution is Dy-Y-Tb,² while the order observed with 5% citrate is Dy-Tb-Y. Previously, it had been reported³ that elution at 100° of tracer amounts of rare earths with 5% citrate showed yttrium eluting ahead of dysprosium.

When eluting with 5% citrate at pH values greater than 2.8, the rare earth band immediately precedes the hydrogen band on the column, and results in a square-type band front. At lower pH values, the ammonium ions pass the rare earth ions and consequently the rare earths are on an ammonium bed. The resultant band front at these lower pH values is sloping and the elution curves resemble those previously obtained with other rare earths using similar conditions.⁴ At all pH values when 5% ammonium citrate is used as eluant, the observed order of elution is Dy-Tb-Y.

A load of 7.6 g. of oxides was placed on a column containing 450 g. of resin and eluted with 5% citrate. The resin bed length was 60 cm. and the diameter was 4 cm. These oxides, obtained by column fractionation of R_2O_3 from gadolinite ore, had the composition 10% Dy_2O_3 , 89% Y_2O_3 , 0.5% Ho_2O_3 , 0.5% Tb_4O_7 . Analyses were made by spectrophotometric and spectrographic methods. At a pH value of 2.80, 85% of the available Y_2O_3 was obtained spectrophotometrically pure (>99%). Previous work using 0.1% citrate at pH values of 5.5-6.0, eluted less than 10% of the available Y_2O_3 with comparable purity. The total time of a run using 5% citrate at a pH of 2.80 is only one-third that required with 0.1% citrate. The concentration of rare earths in the eluate using 5% citrate is correspondingly higher.

It appears that the most efficient way to obtain pure Y_2O_3 and pure Dy_2O_3 from an ore concentrate involves two ion exchange procedures: (1) first a preliminary elution using 0.1% citrate with pH values between 5.8 and 6.1, which separates dysprosium and yttrium from the other rare earths; (2) elute the yttrium-dysprosium fractions so obtained with 5% citrate using a pH value of 2.8, to obtain the pure salts.

(2) F. H. Spedding, E. I. Fulmer, J. E. Powell, T. A. Butler, and I. S. Yaffe, paper presented before the Sept. 1950 meeting of the American Chemical Society in Chicago.

(3) B. H. Kettle and G. E. Boyd, *THIS JOURNAL*, **69**, 2800 (1947).

(4) F. H. Spedding, A. F. Voigt, E. M. Gladrow and N. R. Sleight, *ibid.*, **69**, 2777 (1947).

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THE STRUCTURE OF THE CARYOPHYLLENES

Sir:

An optically inactive α -caryophyllene (humulene), n_D^{20} 1.5035, was prepared from hop oil by fractionation in an efficient column with sub-