

Preparation of the Crystalline Reineckate of the Antibiotic.—This salt was precipitated by addition of a saturated aqueous solution of freshly recrystallized ammonium reineckate to a neutral aqueous solution of the antibiotic. After crystallizations from 95% ethanol and 50% aqueous acetone, red needles were obtained which gave no characteristic melting point. Assay: 15,000 units/mg. *vs.* *B. subtilis*.^{4,5} *Anal.*⁹ Calcd. for $C_{25}H_{42}N_8O_7S_4Cr$: C, 43.05; H, 5.24; N, 15.58; S, 15.85; ash (Cr_2O_3), 9.40. Found: C, 43.17, 43.27; H, 5.24, 5.33; N, 15.36; S, 15.62; ash, 10.77.

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The Decarboxylation of Simple Fatty Acids

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Although textbooks almost universally state that fusion of the fatty acid salts with sodium hydroxide yields hydrocarbons according to the equation: $RCO_2Na + NaOH \rightarrow RH + Na_2CO_3$, it is difficult to support this statement with evidence. Berthelot¹ reported in 1866 that this reaction was not general.

We have heated the sodium salts of acetic, propionic, butyric and caproic acids with equimolar quantities of sodium hydroxide, and separated the gaseous products. An examination of the data in Table I shows that the decomposition of sodium acetate alone gives products in accord with the above equation.

TABLE I

Sodium salt (0.05 mole) Reaction temp., °C. Gas liberated, mole	Acetate	Propionate	Butyrate	Caproate
	371-376 0.045	370-380 0.044	360-365 0.07	355-360 0.056
	Composition of gas, %			
H ₂	0.5	33	31	38
CH ₄	98.9	20	39	37.6
C ₂ H ₆	..	44	7	1.4
C ₂ H ₈	17	1.3
C ₄ H ₁₀	3.8
C ₅ H ₁₂	12.1
Unsaturates	..	0.3	5.7	2.5

It is noted that these decompositions all occur in the same temperature range. The reaction is exothermic and the major portion of the gas is liberated in a few minutes. Analysis of the water-soluble residues showed that in all cases the amount of sodium carbonate formed was above 90% of the theoretical.

(1) Berthelot, *Ann. chim. phys.*, [4] 9, 444 (1866).

Berthelot considered that the formation of methane, hydrogen, etc., from sodium propionate was caused by the thermal decomposition of the ethane first formed. Although this explanation may, in part, account for these products, the thermal stability of ethane and propane at such temperatures² and the rapidity of the reaction suggest that the primary pyrolytic products are complex. In any case the method is not suitable for the preparation of the simple paraffin hydrocarbons.

Experimental

The sodium salts, excepting the acetate, were prepared from aqueous sodium hydroxide and an excess of the acid, followed by evaporation to dryness. After washing with ether, the salts were recrystallized from water and dried *in vacuo* to constant weight over phosphorus pentoxide.

By calculation from the analyses for sodium, all the salts were better than 98% pure.

The apparatus for the fusion consisted of a side-arm test-tube (35 mm. o.d.) placed inside a jacket wound with a heating element, and insulated. The test-tube was closed with a rubber stopper carrying a thermocouple well and connected through a condenser to a gas collecting bottle.

A mixture of the dried sodium salt (0.05 mole) and sodium hydroxide (0.05 mole), powdered in a "dry box," was placed in the test-tube and the system flushed with dry nitrogen. The mixture was heated to the decomposition temperature, when the internal temperature increased and gas was rapidly evolved; most of the gas was evolved in about five minutes.

The gases were separated by a cryostat, using isothermal distillations at successive temperatures.^{3,4} The gas fractions were identified by combustion analyses. The data are shown in Table I.

The carbonate in the residues was determined by standard procedures.

(2) Egloff, "Reactions of Pure Hydrocarbons," Reinhold Publishing Corp., New York, N. Y., 1937, p. 99 *et seq.*, p. 119 *et seq.*

(3) Ailman, Ph.D. Thesis, The Pennsylvania State College, 1938.

(4) We are indebted to Dr. H. D. Zook and Mr. W. J. McAleer for these analyses.

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Mass Spectrometric Evidence for a New Boron Hydride

BY FRANCIS J. NORTON

In examining with the mass spectrometer the residues from pure B_5H_{11} which had been stored at -78° for a long time, there was detected a small amount of $B_{10}H_{14}$, B_6H_{10} and B_5H_9 with the B_5H_{11} . In addition, a group of hitherto unobserved peaks, dominant peak mass 105, was found. It is believed they represent a new boron hydride, B_9H_{13} .

The spectrum of pure $B_{10}H_{14}$ is given in Fig. 1 (A), from mass 85 to 124, the parent peak. This was obtained with mass spectrometer operating conditions already described.¹ Peaks of double ionization for $B_{10}H_{14}$ were observed in the region 55-59.

(1) F. J. Norton, *THIS JOURNAL*, 71, 3488 (1949)