# Derivatives of Trichlorolactic Acid

# II. The Triethyl Ester of $\beta,\beta,\beta$ -Trichlorolactic Acid Dihydrogen Phosphate

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In a publication of Anschütz and Haslam <sup>1</sup> it has been mentioned that  $\alpha,\beta,\beta,\beta$ -tetrachloropropionyl chloride is formed when  $\beta,\beta,\beta$ -trichlorolactic acid reacts with phosphorus pentachloride. Leimu and Ronkainen <sup>2</sup>, however, have shown that the product of the reaction is the dichlorophosphate of  $\beta,\beta,\beta$ -trichlorolactyl chloride. According to Anschütz and Haslam, the reaction product reacts with ethyl alcohol with the formation of the ethyl ester of  $\alpha$ -ethoxy- $\beta,\beta,\beta$ -trichloropropionic acid,  $CCl_3CH(OC_2H_5)COOC_2H_5$ . The formation of this latter compound is possible, although less probable, from the parent compound with the composition proposed by Leimu and Ronkainen. The relatively high boiling point,  $128-130^\circ/12$  mm reported by Anschütz and Haslam for the product of the reaction with alcohol is not in accordance with the structure suggested by them. For this reason it was considered necessary to reinvestigate the reaction. It was established that the reaction product is the triethyl ester of  $\beta,\beta,\beta$ -trichlorolactic acid dihydrogen phosphate:

OPOCI<sub>2</sub>

$$CCl_3CH + 3 C_2H_5OH \longrightarrow CCl_3CH + 3 HCl$$

$$COOC_3H_5$$

It thus appears that also in this case the analytical data of Anschütz and Haslam are considerably in error, as shown by the comparison of the data in Table 1.

| Element      | Calculated                  |  | Found                  |               |
|--------------|-----------------------------|--|------------------------|---------------|
|              | $\mathrm{C_7H_{11}O_3Cl_3}$ | C <sub>9</sub> H <sub>16</sub> O <sub>6</sub> PCl <sub>3</sub> | А&Н                    | L & K         |
| C<br>H<br>Cl | 33.68<br>4.41<br>42.66      | 30.23<br>4.51<br>29.75<br>8.66                                 | 33.29<br>4.73<br>42.86 | 29.63<br>8.48 |

Table 1. Comparison of analytical data.

## **EXPERIMENTAL**

 $\beta,\beta,\beta$ -Trichlorolactyl chloride dichlorophosphate was prepared as described earlier 1,2. This compound was reacted with ethanol under conditions identical with those in the experiments of Anschütz and Haslam, but an excess of ethanol was found advantageous. The product of the reaction was distilled twice under reduced pressure from a Claisen flask. Twelve grams of trichlorolactyl chloride dichlorophosphate yielded 4 g of the ester which boiled at 140-142°/1-2 mm. The ester does not immediately form hydrogen chloride when added to water.

Calc. for C9H16O6PCl3 (358): Cl 29.75, P 8.66 29.35, Found » » 8.48

29.87 29.64

29.43

29.85

#### SUMMARY

When the reaction product of  $\beta,\beta,\beta$ -trichlorolactic acid and phosphorus pentachloride is reacted with ethyl alcohol, the reaction product is not the ethyl ester of  $\alpha$ -ethoxy- $\beta$ , $\beta$ , $\beta$ -trichloropropionic acid, as assumed previously. but the triethyl ester of  $\beta, \beta, \beta$ -trichlorolactic acid dihydrogen phosphate.

### REFERENCES

- 1. Anschütz, R. and Haslam, A. Ann. 253 (1889) 132.
- 2. Leimu, R. and Ronkainen, P. Acta Chem. Scand. 8 (1954) 591.

Received January 18, 1954.