used at once. The calcium chloride must not be alkaline to litmus in aqueous solution, because such calcium chloride causes very rapid polymerization.

Under normal conditions the tar in the generator flask can be readily and quickly washed out with warm water. At other times there is a small amount of non-acid tar, which can be removed easily with the chromic acid cleaning mixture.

CHICAGO, ILL.

THE PHOSPHATES OF 2,3-DISTEARIN.[1]

BY R. R. RENSHAW AND R. R. STEVENS.

Received June 22, 1914.

Grun and Kade[2] have recently described a number of distearyl phosphates obtained by the action of phosphoric anhydride on 2,3-distearin. It would appear that the publication of their researches was delayed and that their results were obtained, in part at least, as early as 1910. We also began the study of the phosphatization of distearin in the summer of 1910, but our object was to obtain a definite substance and not *per se* to study the interaction of phosphorus pentoxide and distearin. Our investigation is not as complete as that of Grun and Kade, and we bring up no question of priority. There are, however, certain similarities and differences in our results which we think desirable to indicate at this time.

Under certain conditions, Grun and Kade obtained the mono-2,3-distearyl glyceryl phosphate and from the decomposition of this, by standing or by recrystallization from alcohol, ether or ligroin, they seem to have obtained primary, secondary, tertiary and quinquenary esters of *o*-phosphoric acid. Suggesting as probable, the intermediate formation of metaphosphates, they propose the following scheme to represent the decomposition, where R represents the distearyl radical:

$$RH_3P_2O_7 \longrightarrow RH_2PO_4 \longrightarrow R_2HPO_4 \longrightarrow R_3PO_4 \longrightarrow R_5PO_5$$

That is, for instance, the primary distearyl glyceryl phosphate breaks down in part into distearin and phosphoric acid and the distearin formed reacts with some of the undecomposed primary phosphate to form the secondary phosphate, and similar reactions yield the tertiary and quinquenary phosphates. The evidence offered seems to be sufficient with the possible exception of the quinquenary phosphate.[3]

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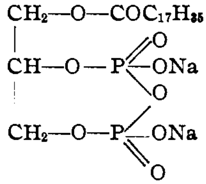
[1] Presented at the 49th General Meeting of the Am. Chem. Soc., Cincinnati, Ohio, April 8, 1914.

[2] *Ber.*, **45**, 3358.

[3] In connection with this, it may be recalled that Grun himself has shown that diglycerides form very stable addition products with the fatty acids, as for instance, dimyristin forms $C_3H_5(OH)(OCOC_{13}H_{27})_2 + 2HOOCC_{13}H_{27}$. The phosphorus content of the quinquenary ester and of such an addition product of the tertiary phosphate with four molecules of stearic acid differ within the allowable analytical error.

Both from experimental data and from analogy with the reversibility of the reaction of concentrated sulfuric acid and stearic acid on mono and distearin, the decomposition proposed by Grun and Kade was viewed with surprise by the present authors. Our results show clearly the tendency of the phosphates to hydrolyze, forming products poorer in fatty acid and richer in phosphorus rather than the reverse as found by Grun and Kade.

We have confirmed the work of Grun and Kade and of Hundeshagen[1] on the preparation of the primary *o*-phosphate of 2,3-distearin. In addition we have isolated salts of a pyrophosphate which appears to be



This was obtained in an attempt to simplify the process for the separation of the primary distearyl glyceryl phosphate. A typical example of the procedure follows: 2.5 g. of distearin and 1.3 g. of phosphorus pentoxide were heated for an hour at 100° with constant stirring. The reaction product was cooled, pulverized and sifted with stirring into a saturated solution of sodium hydrogen carbonate. The brownish, granular material separating in the bottom of the beaker was filtered, washed with water and extracted with acetone to remove stearic acid and distearin. About one-fourth of the residue from the acetone extraction was soluble in boiling benzene. The remainder, insoluble in benzene, was boiled with water, and its concentrated aqueous solution precipitated with acetone. In the boiling stearic acid was split off. The combined soluble products were purified by dissolving in benzene and precipitating with acetone, and finally recrystallizing from petroleum ether. It separated from this solvent in prismatic plates melting at 167–8° (uncor.). The yield was about 0.47 g.

Calc. for $C_{21}H_{40}O_9P_2Na_2$: P, 11.39%; stearic acid, 52.2%. Found: P, 11.51%, 11.43%; stearic acid, 51.86%.

This sodium salt dissolves in hot water, giving a solution resembling that of soap. It is soluble in hot benzene and in petroleum ether and is nearly insoluble in acetone.

A silver salt was obtained by precipitating a concentrated solution of the sodium salt with silver nitrate. It melts at 147.5° (uncor.) and readily darkens.

Calc. for $C_{21}H_{40}O_9P_2Ag_2$: Ag, 30.23%. Found: Ag, 30.19%.

Insoluble barium and calcium salts were also obtained.

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[1] *J. prakt. Chem.*, **28**, 232.

No attempt was made to investigate further the product insoluble in benzene.

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OIL OF OCYMUM PILOSUM ROXB.

BY KSHITIBHUSAN BHADURI.

Received June 8, 1914.

The botanical characteristics of *Ocymum Pilosum, Roxb.* are as follows: Shrubby, branches four-sided, and furrowed. Leaves, ovate oblong, cerrat. Bractes petioled, sub-orbicular, hairy; upper tip of the calyx orbicular and hairy, with corrolla twice its length.

The seeds when steeped in water swell into a jelly, which is used medicinally by the natives of India.

The plant has a very strong odor of the oil, which is found in the whole of it as well as in the seeds; but, when the latter are dried, no oil can be got from them by distillation. This may be due to either of the two causes, *viz.*, the oil is extremely volatile and when the plant dries it volatilizes, or in the course of drying, the oil resinifies and, as such, it cannot be obtained by distillation.

The percentage of oil in the green seeds is higher than in that of the leaves. The oil has been obtained by the author by distillation at the laboratory, the total volume obtained being very small. The season being over, it was not possible to obtain a fresh supply of the plant. It is hoped, however, that a good quantity of oil may be obtained the coming season, when the results of further investigation will be communicated.

To obtain the oil, the whole plant, as cut down, can be at once submitted to steam distillation. The whole of the oil comes over within a very short time, about half an hour being sufficient for this purpose. It is a very thin mobile liquid with a pale yellow color. When left exposed to the atmosphere part of it volatilizes and a resinous mass remains. The odor is almost identical with that of lemon-grass oil.

Experimental.

The specific gravity as determined with a pycnometer, was found to be 0.8872 at 25.5°. The refractive index, as determined in a Pulfrich's refractometer, is 1.4843 or 40° 12′ at 24.5°. The oil is laevo-rotatory, the optical rotation being —3.7° in a tube one decimeter long. The specific rotatory power $[\alpha]_D$ is, therefore, —4°10′14″ at 24.5°.

Generally, Basil oils contain methyl-chavieol, which gives a blue coloration with ferric chloride.[1] When ferric chloride solution (neutral) was

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[1] *Richter's Organic Chemistry*, p. 269.