

purins, results by a spontaneous change. By the use of ethyl alcohol in place of methyl alcohol, or by lowering the temperature, the unstable chlorins became the sole products: quick saponification at an elevated temperature produces chlorin *e* and no unstable chlorins.

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NOTES

Attempted Use of Activated Silica Gel in the Esterification of Salicylic Acid and β -Naphthol.¹—Korolev,² using an especially prepared silica gel suspended in the liquids, obtained 75–80% yields of methyl salicylate, and a 50% yield of methyl naphthyl ether.

Although the use of silica gel in the liquid phase seems to offer a simple method of preparing esters, the preparation of the silica gel as described by Korolev is far from a convenient process. It seemed worth while, therefore, to study the esterification of salicylic acid and β -naphthol using silica gel which had been activated by passing dry air over the gel at a temperature of 150°, according to the commercial process as devised by Patrick (U. S. Patent 1,297,724) and which has been adapted to small-scale production by Holmes.³

Since silica gel activated in this way is known to be a good absorber of water, the presence of the gel should favor production of ester, not only by its catalytic powers, but also by removing the water formed.

The general method used was to place two samples of the mixture to be esterified in flasks fitted with reflux condensers and to heat them in a water-bath. Silica gel was added to one flask and, after refluxing for at least eight hours, the acid was then determined by titration with a standard base.

It was found that a number of different samples of silica gel activated by the method of Patrick do *not* catalyze the esterification of salicylic acid or β -naphthol with methyl alcohol.

CONTRIBUTION FROM THE
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Estimation of Organic Halogen.—In their paper "A General Method for the Determination of Halogens in Organic Compounds" [THIS JOURNAL, 52, 1195 (1930)] the authors, J. J. Thompson and U. O. Oakdale, make no mention in their references to a similar method

¹ The experimental work was done by R. Chelberg, teacher of chemistry at Tracy, Minnesota.

² Korolev, *J. Chem. Ind. (Moscow)*, 4, 547 (1927); *C. A.*, 22, 944 (1928).

³ Holmes, "Laboratory Manual of Colloid Chemistry," 1922, p. 76.

[Robertson, *J. Chem. Soc.*, **107**, 902 (1915); **109**, 218 (1916); *Chem. News*, **120**, 54 (1920)].

In their method fuming sulfuric acid alone or in conjunction with chromic acid or persulfate is used as oxidizing agent, and the liberated halogen absorbed in alkaline arsenite. In the present writer's method ordinary strong sulfuric acid and potassium dichromate are employed with aqueous sodium peroxide as absorbent. The following advantages are claimed. (a) The apparatus is much simpler; it is, in fact, an apparatus for the Bunsen determination of peroxides with slightly modified Peligot tubes for absorption. (b) The use of fuming sulfuric acid, an unpleasant reagent, is avoided. (c) As air is aspirated through the apparatus the time of an estimation is shorter, being fifty to sixty minutes instead of seventy to ninety as stated by Thompson and Oakdale. (d) Sodium peroxide is a reagent which has never been found to be contaminated with halogen.

In the last fifteen years some hundreds of estimations have been carried out by the author and his research students without a single failure. The method is described in various textbooks and is in use in a number of laboratories.

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COMMUNICATIONS TO THE EDITOR

AN ATTEMPT TO DETERMINE NUCLEAR MOMENTS

Sir:

The explanation of the hyperfine structure of line spectra and the alternating intensities of band spectra involves the assumption of the existence of a proton spin in the nucleus of $1/2$ quantum unit. The magnetic moment associated with this spin would stand in the same ratio to the magnetic moment of the electron as e/m for the two charges. This means that the magnetic moment of a hydrogen nucleus would be $1/1840$ of a Bohr magneton, which is too small an amount to detect in the ordinary Stern-Gerlach experiment.

In the case of the heavier atoms the proton spins are usually paired in the nucleus but in some cases, *e. g.*, iodine, several protons remain unpaired and a resultant proton spin of several half units results. Thus a resultant spin of 3 units is to be expected for HI and 5 units for I_2 . The iodine molecule has the larger spin, but the deflection in the Stern-Gerlach experiment is inversely proportional to the absolute temperature of the beam and the beam of iodine must be formed at room temperature while the beam of hydrogen iodide may be formed at a temperature of 130–140° K. Experiments were tried with both molecules.