

## Nottingham Section.

Meeting held at Nottingham on Oct. 8, 1903.

MR. O. QUIBELL IN THE CHAIR.

### THE USE OF ALUMINIUM IN THE ESTIMATION OF IRON.

BY F. J. R. CARULLA.

In this paper, the author recommends the use of aluminium in place of zinc for the reduction of ferric solutions before titration with potassium permanganate.

## Journal and Patent Literature.

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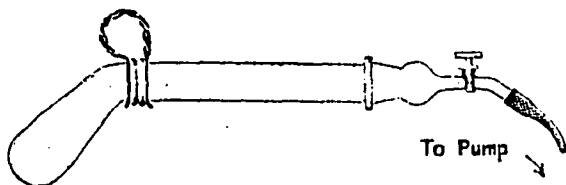
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### I.—PLANT, APPARATUS, AND MACHINERY.

*Vacuum sublimation; Practical studies on* — R. Kempf. *J. prakt. Chem.*, 1908, 78, 201—259.



The author points out that purification by means of sublimation is worthy of more extensive application than it receives at present. Compared with crystallisation, there is little loss of material, and no foreign substances are introduced. Sublimation is also frequently preferable to distillation, as it can be effected at a lower temperature, and hence with less risk of decomposition. In the separation of mixtures by sublimation, the pressure should be so adjusted that the operation is effected at a temperature at which the vapour pressures of the constituents are most widely apart, but as the nature of the impurities is frequently unknown, it is recommended in such cases to work at the lowest practicable temperature. The author has devised a simple vacuum sublimation apparatus of refractory Jena glass (see figure). The junction between the pear-shaped receptacle and the horizontal tube is formed by ground glass flanges as in an exsiccator. The two parts of the apparatus may be held together, when the vacuum is broken, by means of a brass clip as shown. The ground surfaces of the joints are rubbed over with graphite before being brought in contact. With an ordinary water pump a vacuum of 10—15 mm. of mercury can be produced in the apparatus, and with a Geryk vacuum pump (Fleuss patent, type C), one of about 0.4 mm. For heating the apparatus, an air-bath constructed of aluminium and asbestos-board is used, having in the bottom a central hole, 1—2 cm. above which is a thick iron plate. For high temperatures several sheets of asbestos gauze may be laid on the bottom of the air-bath. The material to be sublimed should be first ground as finely as possible, and may also be mixed with powdered glass in order to increase its surface and thus accelerate sublimation. Fractional sublimation is frequently observed, part of the sublimate collecting in the pear-shaped vessel, and part in the horizontal tube.

In other cases fractionation can be effected by placing a part of the horizontal tube in the air-bath with the pear-shaped vessel, at first, and withdrawing it after the most volatile portion of the substance has sublimed. The author points out the uselessness of the so-called sublimation-temperature of a substance as frequently given in chemical literature. What is needed is a statement of the rate of sublimation at a given temperature and pressure. The article concludes with a detailed account of sublimation experiments with 53 different substances, including elements and inorganic compounds, hydrocarbons, phenols and quinones and their derivatives, dyestuffs, aldehydes, organic acids and anhydrides, amino-acids, diketopiperazines, purine derivatives, alkaloids, odoriferous compounds such as vanillin, coumarin, coniferin, and camphor, and pharmaceutical compounds such as aspirin (acetylsalicylic acid), saccharin, sulphonal (diethylsulphodimethylmethane), and veronal (C-diethylmalonylurea).

Zinc dust was readily freed from arsenic and iron, and was obtained much purer than by distillation. Sublimed, crystallised phosphorus pentoxide is much more chemically reactive than the amorphous form. Mercuric sulphide on sublimation in a vacuum, yields a black powder, which is converted into cinnabar on rubbing in an agate mortar. To obtain cinnabar directly, the sublimation must be carried on slowly and under pressure. It is possible to effect a quantitative separation of catechol and its two mononitro-derivatives, and also the detection and determination of alizarin in admixture with flavopurpurin and anthrapurpurin by fractional sublimation. By sublimation of a mixture of malic acid or succinic acid and phosphorus pentoxide, malic anhydride or succinic anhydride is obtained.—A. S.

### PATENTS.

**ERRATUM:**—This J., 1908, 1010, col. 2, title, l. 27 from top, for "Medberger and Medberger" read "Medberg and Medberger."

*Lubricating compounds [Graphite]*. B. L. Philips-Smith, London. Eng. Pat. 23,022, Oct. 25, 1907.

THE lubricating compound consists of a mixture of finely-divided graphite and either formalin and glycerin, or tannic or gallotannic acid and glycerin. The graphite is thoroughly mixed into a paste with a 40 per. cent. solution of formalin, and about 2 oz. of this paste are mixed with glycerin. The graphite may be mixed with the tannic acid by the Acheson process (U.S. Pat. 844,980 of 1907; this J., 1907, 417).—W. C. H.