# NOTES.

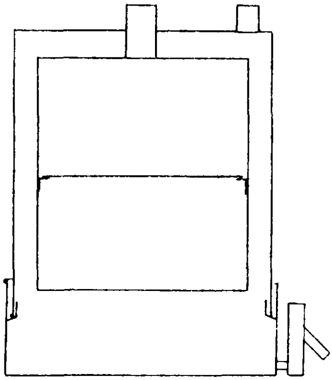
## *The Formation of Liesegang Rings as a Periodic Coagulation Phenomenon.* By JACK REGINALD IRONS HEPBURN.

IN a paper with the above title (J., 1928, 2714) Hedges and Henley have provided experimental evidence in support of an hypothesis advanced originally by Freundlich (" Colloid and Capillary Chemistry," p. 735), although they make no reference to his views.

Previous to the publication of the above paper, the author had carried out experiments with the object of testing the coagulation theory of the production of periodic structures. For instance, in connexion with his study of the formation of malachite (J., 1925, **127**, 1008), basic cupric carbonate was formed by admixture of a solution of 0·1*N*-copper sulphate and 10% gelatin, with an equal volume of 0·1*N*-sodium carbonate and 10% gelatin. The product was a green, transparent, slightly opalescent gel, instead of the ordinary blue flocculent precipitate. The set gel was covered with *N*-sodium carbonate solution, which was allowed to diffuse into it. After a week, three intensely violet rings had appeared, and the number had increased to thirteen after three weeks. When, however, *N*-copper sulphate solution was used as coagulant, only one light green band was produced, after which diffusion ceased.

The production of stratified formations by the coagulation of sols has been studied by Quincke (*Ann. Physik*, 1902, **9**, 987), who describes the coagulation of an arsenic trisulphide hydrosol—unprotected by gelatin—by diffusion of solutions of ferric chloride and of aluminium sulphate. He mentions the production of layers after flocculation, richer and poorer in flocks, with horizontal or convex surfaces.—NORTHERN POLYTECHNIC, LONDON, N. 7. [*Received, November 17th,* 1928.]

*A Steam Drying Oven with Detachable Base.* By HENRY J. S. SAND. THE base of this oven is constructed as a separate unit distinct from the rest, the junction between the two parts being made by a hydraulic seal (see fig.). This is effected by fitting the top of the base with a gutter, which is maintained full of water by condensation from the walls of the oven, the excess overflowing into the base. The general construction will be apparent from the section shown. The device is useful for small laboratory steam-ovens, since the base can be readily freed from scale, and renewed if necessary. It is, however, particularly valuable for larger combined ovens and stills. For these, the top of the oven is fixed rigidly to a stand, the base being supported on two cranked rods, to which handles are attached, by the turning of which the base may be instantly lowered and removed for cleaning. In ovens which are fitted with a reflux condenser and water gauge and are charged with distilled water, all loss of water by evaporation should be prevented: this may be effected by floating a small amount of paraffin wax on the outer portion of the water forming the seal.—SIR JOHN CASS TECHNICAL INSTITUTE, E.C. 3. [*Received, November 20th*, 1928.]



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*Preparation of Quinaldinyl Chloride and Ethyl Quinaldinylacetoacetate.* By DALZIEL LLEWELLYN HAMMICK and WILLIAM P. DICKINSON.

QUINALDINYL chloride was first prepared by Meyer (*Monatsh.*, 1904, **25**, 1199; *Ber.*, 1905, **38**, 2488) by the action of thionyl chloride on the acid and described as a very sparingly soluble, crystalline substance of m. p. 175—176°. Besthorn and Ibele subsequently (*Ber.*, 1905, **38**, 2127) claimed to have obtained, also by the action of thionyl chloride on the acid, an acid chloride of m. p. 96° and readily soluble in organic solvents. Meyer's inability to obtain Besthorn's product was ascribed by Besthorn (*Ber.*, 1908, **41**, 2003) to the presence of anticatalysts (notably tin) in the thionyl chloride used. With Meyer's demonstration (*loc. cit.*) that Besthorn's criticisms were unfounded, the problem of the preparation of the soluble form of the acid chloride appears to have been left unsolved, the position being that the compound has only been prepared in one laboratory.

We have attempted to prepare the low-melting acid chloride according to the directions of Besthorn and Ibele (*loc. cit.*; *Ber.*, 1906, **39**, 2329) and confirm the observations of Meyer that the high-melting form is invariably obtained (Found : Cl, 18·0. Calc. : Cl, 18·5%), even when the purest thionyl chloride is used after redistillation from several successive quantities of quinaldinic acid. We have, however, obtained an acid chloride of m. p. 97° in two ways : (*a*) 12 G. of quinaldinic acid, freshly crystallised from ligroin of b. p. 100—120° (this crystallisation is essential for success), were suspended in 300 c.c. of ligroin (b. p. 100—120°), and 12·4 g. of phosphorus pentachloride added. The mixture was boiled under reflux until evolution of hydrogen chloride ceased and the liquid went clear. Large needle-shaped crystals of the acid chloride, m. p. 97°, separated on cooling. Yield, 7·5 g. or 60% (Found : Cl, 18·7%).

(*b*) Ethyl quinaldinate (fine needles, m. p. 36°, prepared by boiling the acid in ethyl alcohol containing sulphuric acid) (0·5 g.) was dissolved in 40 c.c. of ligroin (b. p. 100—120°), and 0·5 g. of phosphorus pentachloride added. Crystals of the acid chloride began to separate at once and, after stirring for an hour at room temperature, 0·45 g. of the low-melting form was obtained (yield, nearly theoretical).

The high- and the low-melting acid chloride, on treatment with water, each gave only hydrochloric and quinaldinic acids.

*Ethyl Quinaldinylacetoacetate*, $CH_3\cdot CO\cdot CH(CO\cdot C_9H_6N)\cdot CO_2Et$.—To the suspension produced by cooling a solution of 2·4 g. of sodium and 13·6 g. of ethyl acetoacetate in 200 c.c. of dry warm benzene, 10 g. of quinaldinyl chloride (m. p. 97°), dissolved in 300 c.c. of dry benzene, were added and the mixture was shaken for 24 hours. The solid product was then collected, washed with ether, dissolved in water, and acidified with dilute acetic acid. The oil that separated, after being dried in ether with anhydrous sodium sulphate, solidified on standing and was recrystallised twice from ligroin (b. p. 60—80°); m. p. 61° (Found : N, 4·8. $C_{16}H_{15}O_4N$ requires N, 4·9%). Yield, 30%. On treatment with copper acetate in aqueous alcohol it gave a copper derivative, m. p. 178° (decomp.), that was readily soluble in alcohol, benzene, and chloroform.

All attempts to prepare ethyl quinaldinylacetate from the acetoacetic ester failed. Dry ammonia in ether (Bouveault and Bougert, *Bull. Soc. chim.*, 1902, **27**, 1088) gave a quantitative yield of quinaldinamide. Cold ammonia and ammonium chloride (Perkin and Needham, J., 1904, **85**, 148) had no effect; at 50° the amide was again obtained.—THE DYSON PERRINS LABORATORY, OXFORD.

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