from alkali-treated precursors, having an isoelectric point at p_H 4.7, requires a p_H of approximately 1.

IV. The advantages of gelatin for use in emulsions are enumerated.

V. The efficiency of several colloid mills as compared with that of a homogenizer in preparing liquid emulsions is reported.

VI. Practical formulas and directions for the use of gelatin in emulsions are presented.

REFERENCES.

(1) Briggs and Schmidt, *J. Phys. Chem.*, 19, 484 (1915).
(2) Clayton, *Kolloid-Zeitschrift*, 28, 202 (1921).
(3) Holmes and Child, *J. A. C. S.*, 42, 885 (1926).
(4) Kernot and Knaggs, *J. Soc. Chem. Ind.*, 47, 96T (1928).
(5) Limburg, *Rec. trav. chim.*, 45, 885 (1926).
(6) Briefer and Cohen, *Ind. Eng. Chem.*, 20, 408 (1928). Briefer, *Ibid.*, 21, 266 (1929).
(7) This author, *Am. J. Pharm.*, 107, 158 (1935).

A NOTE ON THE ACTION OF ALKALIES AND ALKALI SALTS ON ANTIPYRINE.*[1,2]

BY LOYD E. HARRIS AND ERCELL D. TEBOW.

A prescription that called for a solution of antipyrine in a concentrated solution of potassium citrate was brought to the laboratory for an explanation. A colorless liquid separated which disappeared on dilution of the mixture with water. Or, on standing, it gradually changed to a crystalline substance. A search of the literature gave no information as to what was happening, so the problem was investigated in the laboratory.

EXPERIMENTAL.

A few grams of the liquid precipitate were obtained by adding antipyrine to a saturated aqueous solution of potassium citrate. This was separated by using a separatory funnel. Crystals began forming after standing a few hours and in about 48 hours the mass was completely crystallized. The crystals resembled antipyrine in physical appearance and the melting point was found to be 111° C. which is the same as the U. S. P. compound. A mixed melting point with antipyrine did not cause any change. The U. S. P. X color tests were positive, thus, further indicating that the crystals were antipyrine.

A saturated alcoholic solution of picric acid was added to a boiling aqueous solution of the immiscible liquid. Almost immediately, long yellow crystals began to appear. After washing with water and drying, their melting point was determined to be 188° C.; antipyrine picrate was prepared in a similar manner and its melting point was the same.

A second portion of the aqueous solution of the immiscible liquid was made acid with hydrochloric acid (no change was apparent in the dilution used) and then sodium nitrite was added. The green precipitate, which formed, was separated by filtration, washed with water and then dried. The melting point was 200° C. and corresponds to the melting point of nitroso antipyrine, prepared in the laboratory.

---

* Scientific Section, A. PH. A., Portland meeting, 1935.

[1] An abstract from a Thesis submitted by Ercell Dale Tebow to the Graduate Faculty, University of Oklahoma, in partial fulfilment of the requirements for the degree of M.S. in Pharmacy, 1933.

[2] From the Pharmacy Laboratory, School of Pharmacy, University of Oklahoma.

Portions of the immiscible fluid obtained from each of the alkalies and alkali salts used were burned, in separate evaporating dishes, at temperatures less than red heat. There was no ash or residue, except in one instance (this was probably due to incomplete separation of the liquids).

The liquid separated and came to the top when antipyrine was mixed with alkali salts, but with sodium hydroxide and potassium hydroxide, in a few instances, it settled to the bottom. This was apparently due to the different specific gravities; the specific gravity of the separated immiscible liquid was found to be 1.0965 at 25° C.

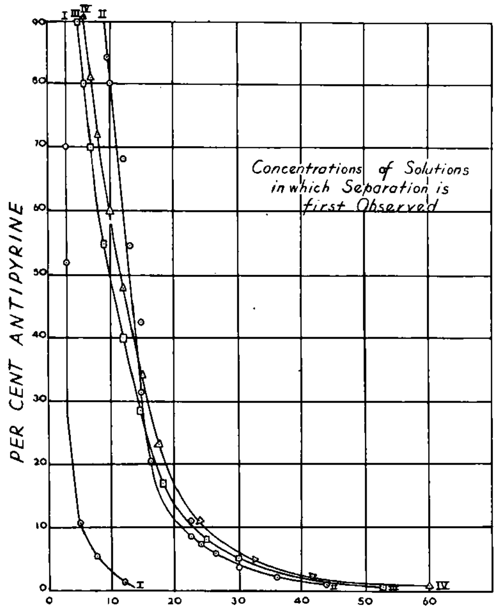

Fig. 1.—Per cent: I, sodium hydroxide; II, potassium hydroxide; III, sodium citrate; IV, potassium citrate.

Crystals formed almost immediately when potassium hydroxide and sodium hydroxide were used, but with potassium citrate and sodium citrate the fluid would frequently remain liquid for several days.

The percentages of alkali and antipyrine were determined at which the immiscible liquid started to separate. In determining the sodium citrate, potassium citrate and antipyrine, the percentages were calculated from the amount of water used and not the total volume of the solution. Due to the hygroscopic nature of potassium hydroxide and sodium hydroxide, they were weighed and dissolved in enough water to make a definite volume and the percentages calculated. Equal amounts of antipyrine were placed in test-tubes, a measured amount of water added and, after it had dissolved, the alkali or alkali salt solution was added from a second burette, shaking after each 0.2 cc., until separation was noticed. The percentage of each was then calculated from the total amount of liquid added. The results are presented in graph form.

A number of other alkali salts were tested qualitatively to see if they would produce similar results on solutions of antipyrine. Each caused the separation into two immiscible layers. The salts used were sodium bicarbonate, sodium acetate, sodium carbonate, sodium thiosulphate, potassium bicarbonate, potassium carbonate, potassium acetate, ammonium acetate and ammonium carbonate.

CONCLUSIONS.

From the foregoing facts, it is believed that the immiscible liquid obtained by the addition of antipyrine to concentrated solutions of alkalies or alkali salts is an isomeric form, which will change to the usual crystalline form on standing.

QUANTITATIVE ANALYSIS OF BARBITURIC ACID DERIVATIVES.

BY KAZIMIERS KALINOWAKI, UNIV. OF POZNAN.

To a solution of 0.2–0.3 Gm. of the substance to be analyzed in 20–25 mils of acetone, or 30–36 mils of ethyl alcohol, 15–20 mils of a normal solution of NaOH and 20–30 mils of water are added. This clear solution is titrated with a $0.1N$ solution of $AgNO_2$ until a turbidity is obtained. As this method is simple and accurate to 0.1 per cent, its use is adapted to pharmaceutical laboratories.—*The Pharmaceutical Journal*, August 31, 1935.