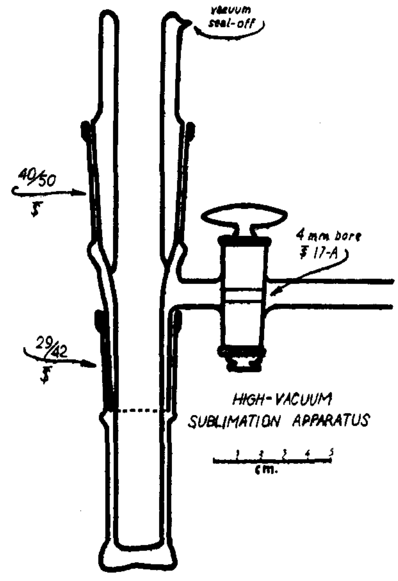

Fig 1.

ground joint, the cup is slipped into place. The condenser is similarly greased and inserted. After evacuation of the apparatus the refrigerant is added. Water may be used, if desired, by inserting a stopper carrying one long and one short tube, and passing the water through these tubes. We have found that any of the low-temperature refrigerants, including liquid nitrogen, has been satisfactory. The lower cup is warmed by insertion into a bath at the desired temperature, and sublimation proceeds. When the process is completed, the sublimate can be washed from the condenser with a few drops of solvent, and worked up in any way desired.

CHICAGO, ILLINOIS RECEIVED MARCH 12, 1938

The Effect of β-Aminopyridine in Experimental Blacktongue

BY Y. SUBBAROW, W. J. DANN AND E. MEILMAN

β-Aminopyridine was isolated from liver extracts by the following method.

Eight liters of solution (Cohn fraction D)[1] representing one ton of fresh liver was diluted to 20 liters with water and treated with 3 kg. of charcoal. The charcoal adsorbate was filtered and was eluted with hot 60% ethyl alcohol. The elute was concentrated to four liters. The concentrate was made strongly alkaline with potassium hydroxide and extracted three times with four liters of normal butyl alcohol each time.

The butyl alcohol was removed from the extract by vacuum distillation, and the residue was taken up with one liter of water. There was some insoluble residue which was discarded.

The clear solution was made strongly alkaline with potassium hydroxide and was extracted eight times with one liter of ether each time. The ether extract was dried with anhydrous potassium carbonate and concentrated to a sirup. The sirup was extracted with 500 cc. of hot benzene. The benzene extract was concentrated to a small volume (30–40 cc.) and dry petroleum ether was added till a precipitate began to appear. After standing at 0° for forty-eight hours, a crystalline precipitate was obtained.

The crystals were filtered, and recrystallized from hot benzene and dried over sulfuric acid; yield 150 mg. The analysis agreed with the formula $C_5H_6N_2$.

The substance was hygroscopic and melted at 65°. Synthetic β-aminopyridine melted at 64°. There was no depression of mixed melting point. It gave an intense diazo reaction and was partially precipitated with cuprous oxide. It reduced phenol reagent (Folin). A gold salt formed readily by the addition of gold chloride in the presence of dilute hydrochloric acid, of composition $C_5H_6N_2 \cdot HAuCl_4$, m. p. 218°. The substance gave a flavianate. The crystals were rectangular plates. They began to char at 212° and melted with decomposition and evolution of gas at 241°. The flavianate of synthetic β-aminopyridine behaved in the same way. The picrate melted at 188–190°. There was no depression of mixed melting point with β-aminopyridine picrate. With concentrated hydrochloric acid, the substance gave transparent plates: β-aminopyridine dihydrochloride, $C_5H_6N_2 \cdot 2HCl$, m. p. 175°.

Six adult dogs weighing, respectively, 9.15, 12.3, 7.75, 15.1, 11.2 and 11.0 kg. were housed in individual cages and fed on Goldberger's blacktongue-producing diet No. 123[2] modified by substituting a more complete salt mixture for the sodium chloride and calcium carbonate used in this diet. The dogs were allowed to develop blacktongue, as judged by inflammation and necrosis of the mucous membranes of the mouth and throat, excessive salivation, listlessness, loss of appetite and loss of weight. After the onset of acute blacktongue each was given daily doses of a solution of β-aminopyridine dihydrochloride in distilled water for five days; administration was by subcutaneous injection. The daily dose for each of the six dogs was 20, 20, 20, 20, 15 and 15 mg., respectively. The dihydrochloride contains 56.5% of the free base.

All the dogs quickly made complete recoveries, as shown by return of the mucous membranes to normal, cessation of the abnormal salivation, and restoration of appetite, body weight and normal activity.

The rapidity and completeness of cure were at least equal to the rapidity and completeness of cure of other dogs to which we have given 20 mg. of nicotinic acid daily for five days, showing that β-aminopyridine is, weight for weight, at least as active in curing blacktongue as nicotinic acid. Further experiments are in progress to determine whether smaller doses of β-aminopyridine will be completely effective in curing an attack of acute blacktongue, as there are indications from a preliminary trial that they may be.

(1) E. J. Cohn, G. R. Minot, G. A. Allen and W. T. Salter, *J. Biol. Chem.*, **77**, 331 (1928).

(2) J. Goldberger, G. A. Wheeler, R. D. Lillie and L. M. Rogers, *U. S. Pub. Health Rep.*, **41**, 297 (1926).

The authors wish to thank Dr. Guy W. Clark and Dr. Merton C. Lockhart of Lederle Laboratory for help in large scale preparation, and the Milton and Proctor funds of Harvard University and the Rockefeller Foundation for financial assistance.

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RECEIVED MARCH 29, 1938

COMMUNICATIONS TO THE EDITOR

COMPARISON OF X-RAY PHOTOGRAPHS TAKEN WITH X AND Y BUILT-UP FILMS

Sir:

Some time ago Mr. Clifford Holley and I reported [*Phys. Rev.*, **52,** 525 (1937)] that X films of calcium stearate have approximately the same grating-space as Y films of barium–copper stearate. In Figs. 1 and 2 are shown some of the photographs taken with films built by Dr. Katharine Blodgett on the basis of which this statement was made. They show $L\alpha$ and $L\beta$ lines of tungsten in the first three observable orders on both sides of the direct beam. Figure 1 was taken with an 1100 layer Y film of barium–copper stearate, Fig. 2 with a 300 layer X film of calcium stearate. In both cases the direction of dipping was parallel to the axis of rotation of the film. It can be seen that the photograph taken with the X film is essentially the same as the one taken with the Y film with respect to both the grating-space and the relative intensities of the various orders. The grating-spaces of these films, corrected for refraction and based upon the ruled grating wave length scale, are: Y film of barium–copper stearate, 50.47 Å.; X film of calcium stearate, 50.12 Å.

Porter and Wyman have shown [THIS JOURNAL, **59,** 2746 (1937)] that the contact potentials of X films increase with the number of layers, whereas those for Y films remain constant. The X-ray photographs, however, seem to be independent of the contact potential. The potentials of an X and a Y film of calcium stearate were measured, after which photographs of the kind shown here were taken. The photographs were much the same as those shown, despite the difference in the contact potentials of the films before exposure. The apparent grating-spaces of the two films in the first order differed by about 1%, the X film having the greater spacing. The two films were built from 10^{-4} molar solutions of calcium chloride. Castor oil was used as piston oil for the X film, and oleic acid for the Y film.

Fig. 1.—Three orders of tungsten L-spectra from Y film.

Fig. 2.—Three orders of tungsten L-spectra from X film.

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RECEIVED APRIL 22, 1938

PRESSURE–AREA RELATIONS OF MONOLAYERS AT THE SOLID–LIQUID INTERFACE

Sir:

While the pressure $F = \gamma - \gamma'$ of a monolayer on water may be determined by a film balance, no method has been known for obtaining F at a solid–liquid interface. In order to determine this value the contact angle θ_1 between the plane surface of the solid and the pure liquid is found by an improved form of the tilting plate method, so arranged that the surface of the liquid can be kept clean by sweeping. Then the pure liquid of surface tension γ_l, *e. g.*, water, is replaced by a solution of the desired solute, of tension $\gamma_{l'}$ and the new contact angle θ_2 is determined. Then

$$\gamma_s = \gamma_{sl} - \gamma_l \cos\theta_1 \qquad (1)$$

$$\gamma_s' = \gamma_{sl}' - \gamma_{l'} \cos\theta_2 \qquad (2)$$

where s indicates a solid.