

or three millimicrons toward longer wave lengths with respect to that of 2-(*p*-aminobenzenesulfonamido)-thiazole. This shift is compatible with the substitution of a hydrogen by an alkyl radical.

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A Continuous Source for the Spekker Photometer

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The principles of the hydrogen discharge tube are so well known that the description of another model would be superfluous, were it not that the tube to be described here has certain practical advantages over other types. Its form is such that it can be used satisfactorily as a source in connection with a Hilger Spekker photometer, where a continuous spectrum is highly desirable. It also requires very little attention. One of these tubes has been in use for three years without having to be refilled.

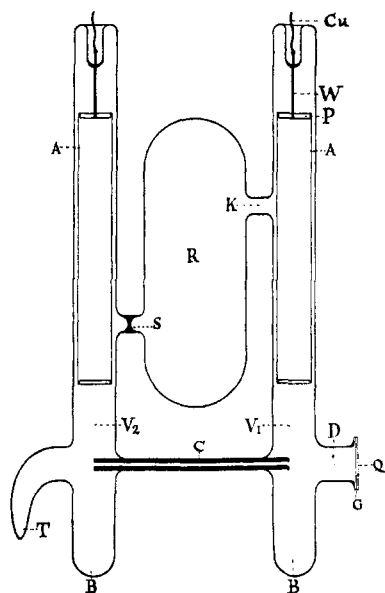


Fig. 1.—Hydrogen discharge tube for ultraviolet spectrograph.

A scale diagram of the tube¹ is shown in Fig. 1. With the exception of the quartz window Q, it is made of Pyrex glass which, besides cheapness, has the advantage of absorbing ultraviolet light so that goggles are unnecessary. The reservoir R is open to the rest of the tube on one side K, but is sealed off on the other S to prevent any short circuit through it. The horizontal tube C is a thick-walled capillary of 1-mm. bore, which connects the two vertical

(1) This is the tube referred to by R. N. Jones, in THIS JOURNAL, 62, 148 (1940).

cylinders V, and in which the light is generated. This capillary extends almost, but not quite, to the centers of the cylinders on both sides, as shown in the diagram. It is essential that the capillary be straight and horizontal. Its protrusion to the center of the vertical cylinder V₁ causes a cone of light to come to a point at its end and thus to form the point source necessary for a Spekker. If the capillary tube terminates at the wall, a diffuse source results.

Opposite the end of the capillary, the cylinder V₁ is provided with a collar D with a flange to which the quartz window is affixed. For use with the Spekker photometer, the distance from the end of the capillary to the outside surface of the window Q must not exceed 4 cm., to permit correct focusing of the point of light onto the prism of the Spekker. To cylinder V₂, opposite the other end of the capillary, is attached a side tube bent down in the form of a cornucopia T. This serves to trap reflections, and is also used for outgassing the tube.

Below the ends of the capillary tube, the cylinders V are prolonged for 8 cm. As the tube is set up in a vertical plane, with the electrodes pointing down, these prolongations B act as traps for any sputtered aluminum or impurities from the electrodes. After a year or so of operation, the traps become coated with material, some of which would otherwise find its way to the inner surface of the quartz window. If the traps are made shallower, after some time the inside of the window becomes coated with enough material to weaken markedly the output of the tube in the short ultraviolet. The over-all length of the cylinders V is 42 cm., and their outside diameter is 3.2 cm. The hydrogen reservoir is 21.5 cm. high by 7.5 cm. in diameter.

The quartz window, which must be exactly perpendicular to the axis of the capillary tube C, is 3.8 cm. in diameter and 1 mm. thick. It is sealed to the flange of collar D with glyptal G, which should be allowed to dry for forty-eight hours before evacuating the tube.

The electrodes A are of rolled aluminum, and are long enough (20 cm.) to permit considerable cooling. Their external diameter is 2.2 cm., and the aluminum is 0.5 mm. thick. Care must be taken in the rolling of the aluminum that no impurities such as mercury are on the bench, as it is impossible to get them out of the tube afterward. The plug P connecting each electrode with a tungsten rod W which supports it through the glass must be carefully fastened to the electrode, preferably by a set of aluminum rivets. This contact must be perfect, lest it offer enough resistance to become a source of heat. The tungsten rods are fused into depressions in the tops of the cylinders, and outside of the tube are welded to heavy flexible copper wires Cu.

The apparatus is evacuated by means of a mercury pump, and after twenty-four hours is washed out with hydrogen. This process is repeated three or four times, and the tube is finally filled with hydrogen at a pressure of 1.0 to 1.5 mm.

The tube can be operated for periods up to ten to fifteen minutes on 15,000 volts, 100 milliamperes, without special air cooling. For longer periods it has been found satisfactory to blow a current of air over the capillary with an ordinary fan. It can be used with much higher amperages,

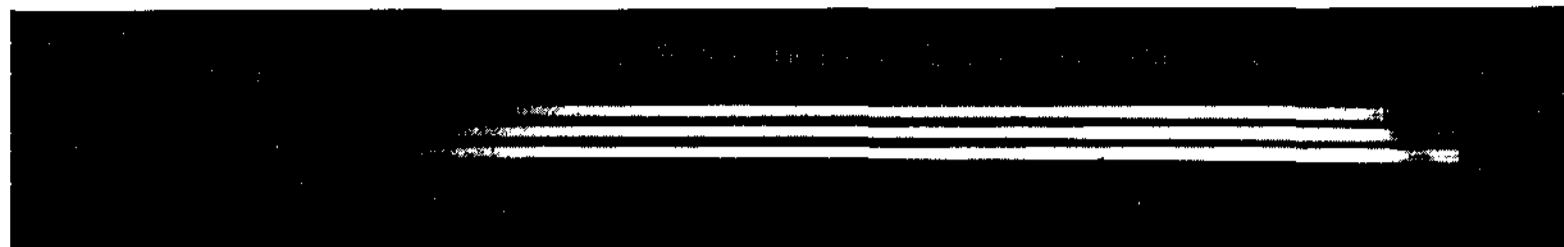


Fig. 2.

however, by cooling with water. This can be done by placing the lower end of the tube in a can with a hole in it to accommodate the window. The window is cemented into the hole with glyptal, and running water is passed through the can. This system has not been found necessary in this Laboratory. The intensity of our arc is such that even now, at the end of three years, our longest exposures for ordinary unsensitized ortho plates are of the order of three minutes, and our shortest five seconds. For these periods no cooling is needed.

Figure 2 shows a series of exposures (taken with a medium quartz spectrograph (Hilger E 3) through a Spekker, for five, ten, and twenty seconds, using a non-sensitized DC ortho plate, and D 72 developer.

For use with the Spekker, this tube of course needs just as careful a system of adjustment as any spark source, because the point of light coming from the end of the capillary has to be exactly opposite the edge of the prism in the Spekker, and the axis of the capillary has also to be in exact alignment with the path of the light into the Spekker. A rigid but adjustable mounting is afforded by a device consisting of a solid base with two rigid iron uprights which hold the vertical cylinders of the tube. A shelf is built in between the two supports for the hydrogen reservoir to rest upon. The position of the base is adjusted by three thumb screws with lock nuts, which should be provided with a vernier. The spectrograph, the Spekker photometer, and the mounting for the hydrogen discharge tube are all carried on a steel I-beam, which makes an ideal optical bench.

The design and construction of this hydrogen discharge tube would have been impossible without the advice and assistance of Dr. Gordon L. Locher.

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On Brassicasterol, the Characteristic Sterol of Rapeseed Oil¹

BY ERHARD FERNHOLZ AND HOMER E. STAVELY

In the year 1906 Windaus and Hauth² discovered that phytosterol obtained from calabar beans contained a doubly unsaturated component characterized by the formation of a sparingly soluble acetate tetrabromide. The new sterol was named stigmasterol, after the botanical name of the calabar bean, *Physostigma venenosum*.

(1) Given at the Cincinnati meeting of the American Chemical Society, Food and Agricultural Division.

(2) Windaus and Hauth, *Ber.*, **39**, 4378 (1906).

Various workers have since investigated phyto-sterols from different sources by this bromination method. The formation of the characteristic insoluble tetrabromide of the acetate has often been and sometimes still is considered sufficient evidence for the presence of stigmasterol. An early paper by Windaus and Welsch³ describes a sterol occurring in rapeseed oil which forms an acetate tetrabromide difficult to distinguish from the stigmasterol derivative. This sterol was named brassicasterol from its origin (*brassica rapa*). The following table gives a comparison of the properties of stigmasterol and brassicasterol and some derivatives.

TABLE I

M. p. of	Stigmasterol	Brassicasterol
Sterol	170	148
Acetate	142	158
Propionate	122	132
Benzoate	160	167
Acetate (tetrabromide)	205-212	205-213

It is obvious that the mere isolation of an acetate tetrabromide of the proper melting points is not enough to establish the existence of stigmasterol in a mixture of plant sterols, since it could equally well be brassicasterol. However, debromination would enable an investigator to distinguish between stigmasteryl acetate and brassicasteryl acetate. Brassicasterol has received little attention since its first isolation in 1909. Some years ago Schmid and Waschka⁴ investigated the tetrabromide by crystallographic methods and found it indistinguishable from stigmasteryl acetate tetrabromide. We are unaware of other publications concerning brassicasterol. In the last few years degradation methods for sterols have been greatly improved, and we thought that in spite of the difficulty of preparing large amounts of brassicasterol the few grams we had on hand would be sufficient for the investigation of its structure. The close similarity of stigmasterol and brassicasterol suggested application

(3) Windaus and Welsch, *ibid.*, **42**, 1912 (1909).

(4) Schmid and Waschka, *Monatsh.*, **48**, 139 (1917).