A Spectrophotometric Dewar Flask With Integral Light Shield

WILLARD B. ELLIOTT

TO LIST ALL the 40 different spectrophotometric Dewar flasks that have been developed for making low-temperature, ultraviolet-visible spectra of organic compounds and biological pigments since the 1927 report by Kronenberger and Pringsheim was published (*1*) is unnecessary. I found no design for a vacuum space specimen Dewar flask with an integral light shield.

Originally, I experimented with a vacuum space specimen in a Hersh Dewar flask (*2*), but believed that a flask of different design would solve problems caused by the Hersh, such as bypassing of the specimen by monochromatic light scattered from the front surface of the specimen (*3*) and inconvenience caused by the great working height of approximately 8 feet required above the cuvette compartment.

The new design is suitable for use in the cuvette compartment of Cary models 11, 14, and 15 recording spectrophotometers and other spectrophotometers with cuvette compartments of similar size. Modification would be required to lower the light path in the light shield in order to use the flask in the Beckman DK spectrophotometer.

The overall height of the new flask is 27.5 inches. A clear area of only 58 inches above the bottom of the cuvette compartment is required when changing the cold finger without removing the Dewar flask from the cuvette compartment.

The Dewar flask (fig. 1) includes annular space A for liquid nitrogen, which provides cooling for the dull black light and heat shield B. (The light shield illustrated is constructed of copper, but a second model with a dull-black coated glass light and heat shield has been used satisfactorily for 2 years.)

The cold-finger specimen holder, which is inserted into the vacuum space, is held in position by O-ring ball joint C. Specimen holder D is similar to a cuvette described by Doebbler and Elliott (*4*); it is a copper frame, 1 millimeter thick, soft-soldered to the copper disk that forms the end of the cold finger. The vacuum space is evacuated continuously by a mechanical pump through side tube E. Provision is made at F for thermocouple leads in the vacuum space. Side tube G permits more rapid escape for gas while the cold finger is being filled with liquefied gas. The picene cemented detachable Pyrex optical windows H can be replaced by quartz. Construction details of the flask can be obtained on request (*A*).

Mr. Elliott is professor of biochemistry at the State University of New York at Buffalo, Buffalo, N.Y.
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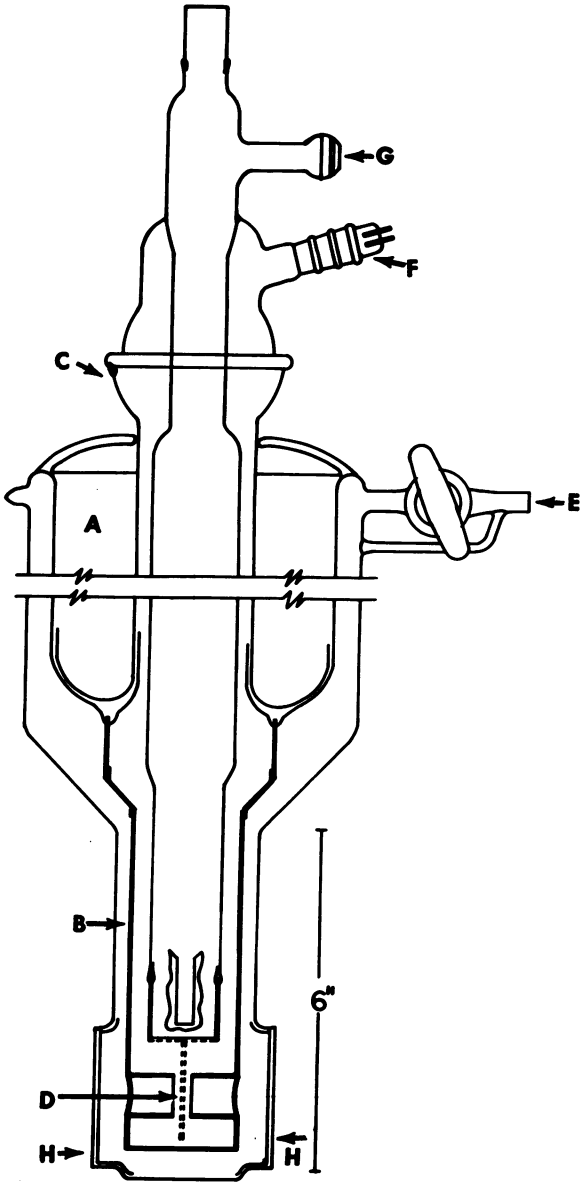
The annular space holds enough liquid nitrogen for 1.5 hours of use; loss of the liquid nitrogen from the cold finger is less than 50 milliliters an hour with 400-micron mercury pressure in the vacuum space. After the cuvette holder and the holder platform are removed, the Dewar flask is centered in the light path by a urethane foam cradle. The upper end of the Dewar flask is supported by a friction tape sling attached to a support frame.

The top of the cuvette compartment is closed with several layers of black velveteen cloth. To minimize reflection of light (through the unsilvered strip) into the vacuum space by liquid nitrogen bubbles in the annular space, the operation must be accomplished in subdued light. The reflected light is most easily detected as excessive noise at the apex of a high absorbance peak (fig. 2, pt. 1.), which shows changes in the noise level occurring at apparent absorbance of 1.43.

Figure 1. Dewar flask

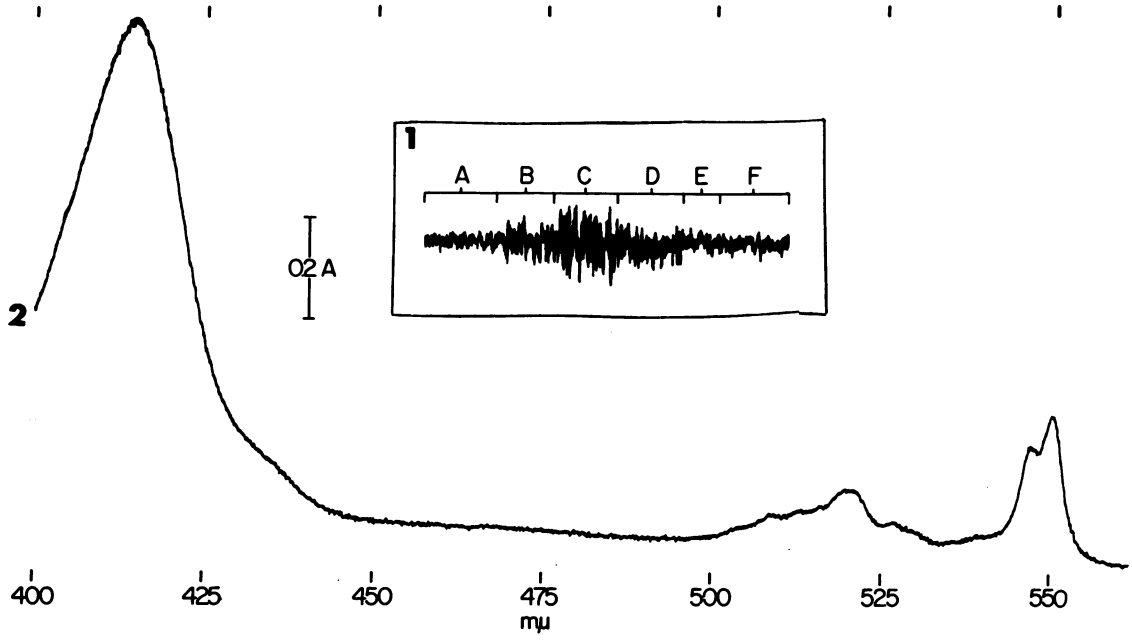


Normally, aluminum plates (1/16-inch thick) are fastened against the lightly greased (4½ parts lanolin, 4½ parts vaseline, and 1 part beeswax) copper frame with giant paper (Gem, *B*) clips to provide a form for casting the specimen block. After the cuvette is coated with 50 percent (volume per volume) methanol-glycerol and dipped in Santocel (*C*) to improve heat transfer (*4*, *5*), it is filled and frozen by immersion in liquid nitrogen, contained in a plastic foam bucket. Often, 20 to 30 milliliters of liquid nitrogen are added to the cold finger immediately before freezing it to precool the metal glass seal.

The aluminum plates are removed after the freezing period, and about 100 milliliters of liquid nitrogen are added to the cold finger. When the cold finger is inserted into the Dewar flask, the vacuum space is evacuated by pumping and the outer space is filled with liquid nitrogen. The outer space must be dry before the liquid nitrogen is added. The portion of the Dewar flask in the cuvette compartment did not frost in 5 hours with adequate pumping of the vacuum space.

Although simple systems that have been described (*4*, *6*) permit the making of useful low-temperature spectra, frosting of specimens held above liquid nitrogen occurs, which changes the baseline. The formation of ice and solid carbon dioxide crystals, which preferentially adhere to the face of the specimen block, along with the noise produced by nitrogen bubbles passing through the light path when the specimens are immersed in liquid nitrogen, also limit the application of these systems in many situations. Dewar flasks in which the vacuum space specimen is stable for several hours permit the use of on-line computers to enhance minor absorption peaks (*7*) as well as vary the temperature of the specimen block over a wide range of temperature by using cold nitrogen gas instead of liquid nitrogen to cool the cold finger.

Figure 2. Spectrophotometer records



Part 1. Changes in noise level at an apparent absorbance of 1.43.

A. Room lights off and door closed to give very subdued light.

B. Normal laboratory lights.

C. High-intensity lamp directly over Dewar flask.

D. Normal laboratory lights.

E. Door open to permit entry of indirect light from hall.

F. Same as **A.**

Part 2. Reduced cytochrome *c* in 50 percent (volume per volume) glycerol: water glass, 2 millimeters thick. Recorded by Cary model 11 spectrophotometer (dynode 2, slit control 50, slit width 0.012 millimeter at 650 millimicrons. Absorbance, compared to air, at the Soret absorption peak was 1.06.

The winged bar indicates 0.2 absorbance and applies to both parts of figure 2.

The light path shielding is sufficient to permit the use of devitrified glycerol: water blocks containing reduced cytochrome *c*. A typical specimen had an apparent absorbance of 1.8 at the Soret absorption peak when measured against a reference beam containing neutral density filters plus disposable wipers, called Kimwipes (*D*), having an absorbance of 2.32 at the same wavelength. The gamma-alpha ratio for the specimen was 3, indicating that stray light and light bypassing the specimen were not limiting absorbance at the Soret peak.

Absorption spectra of specimens of reduced cytochrome *c* (50 percent in a glycerol: water glass) taken before and after 5 hours in the vacuum space were superimposable. A typical reduced cytochrome *c* absorption spectrum is shown in figure 2, part 2. The gamma-alpha ratio of 3 for the reduced absorption peaks is comparable to that obtained in other Dewar systems (*3*) with similar specimens.

REFERENCES

(*1*) Kronenberger, A., and Pringsheim, P.: Uber das absorptions-spektrum des festen benzols bei-180°. Z fur Physik 40: 75–91 (1927).

(*2*) Hersh, H. M.: Color centers in X-rayed potassium iodide, Phys Rev 105: 1158 (1957).

(*3*) Elliott, W. B., and Doebbler, G. F.: Problems in low-temperature spectrophotometry of turbid materials. Anal Biochem 15: 463–469, June 1966.

(4) Doebbler, G. F., and Elliott, W. B.: A simple method for absorption spectra of biological materials at low temperature. Biochim Biophys Acta 94: 317–323, Mar. 29, 1965.
(5) Cowley, C. W., Timson, W. J., and Sawdye, J. A.: Ultrarapid cooling techniques in the freezing of biological materials. Biodynamica 8: 317–329, December 1961.
(6) Elliott, W. B., and Tanski, W.: Attachment for making low temperature spectra in a recording spectrophotometer. Anal Chem 34: 1672–1673, November 1962.
(7) Elliott, W. B., Hayford, R. E., and Tanski, W.: Improvement of low temperature absorption spectra by use of an on-line computer. Biochim Biophys Acta 88: 219–222, July 29, 1964.

EQUIPMENT REFERENCES

(A) H. S. Martin Co., Evanston, Ill.
(B) Scoville Mfg. Co., Oakville, Conn.
(C) Monsanto Co., St. Louis, Mo.
(D) Kimberly-Clark, New York, N.Y.

Tearsheet Requests

Prof. W. B. Elliott, Department of Biochemistry, State University of New York at Buffalo, Buffalo, N.Y. 14214

Public Health Service Staff Appointment

Dr. Emery A. Johnson has been appointed director of the Indian Health Service, Health Services and Mental Health Administration. The position carries the rank of Assistant Surgeon General.

Dr. Johnson will be responsible for administering the comprehensive health services provided by the Government for about 410,000 American Indians, Eskimos, and Aleuts living in 23 Federal Reservation States and Alaska.

A career officer in the Public Health Service, Dr. Johnson has 14 years' experience in the Indian health program. He joined the Service in 1955 as a medical officer at the Public Health Service Indian Hospital in White Earth, Minn. He has served as medical officer in charge at White Earth, at the Indian Hospital in Winnebago, Nebr., and at the Indian Medical Center, Phoenix, Ariz., and as deputy Indian Health Area director in Phoenix.

Dr. Johnson was Indian Health Area director in Billings, Mont., at the time of his appointment to headquarters in June 1966 as assistant director and chief, Office of Program Services. He was made deputy director of the service in September 1967 and has been acting director since July 1, 1969, when Dr. E. S. Rabeau, former director, transferred to Tucson, Ariz., to head the service's research and training complex.

Born in Sioux Falls, S. Dak., April 16, 1929, Dr. Johnson took his B.S. degree at Hamline University, St. Paul, Minn., in 1951 and his M.D. degree at the University of Minnesota Medical School, Minneapolis, in 1954. He interned at Asbury Methodist Hospital, Minneapolis. In 1964 he earned an M.P.H. degree at the University of California School of Public Health, Berkeley.

Dr. Johnson's professional affiliations include the American Medical Association, American Public Health Association, American Academy of General Practice, and the Association of Military Surgeons. In 1966 he was awarded the Public Health Service Commendation Medal.

phr synopses

PEARMAN, J. R. (School of Social Welfare, Florida State University, Tallahassee): *Survey of unmet medical needs of children in six counties in Florida. Public Health Reports, Vol. 85, March 1970, pp. 189–196.*

A study undertaken in 1967 to ascertain the amount of unmet medical needs of children under age 16 in six counties of Florida—Leon, Franklin, Gadsden, Jefferson, Liberty, and Wakulla—revealed that low family incomes, compared to averages in the State and the nation, and relatively high dependency ratios severely limited family capacity to purchase medical services.

Two avenues of investigation were used. Head Start medical records were reviewed in the three counties sponsoring Head Start programs, and 389 families in the six counties, having a total of 1,177 children, were interviewed regarding immunizations and health conditions of the children. Additional information was obtained from reports of the Florida State Board of Health and other public agencies.

Although infant mortality rates had declined from 1960 to 1967, many serious gaps in child health care were apparent: (*a*) 70 percent of the Head Start children were in need of dental care, (*b*) hypochromic anemia was common, (*c*) more than half of the Head Start children were not immunized against smallpox, diphtheria, tetanus, pertussis, and poliomyelitis, (*d*) a third of the children in the interview survey had health conditions which indicated need for examination by a physician or dentist, (*e*) a third of the interview children had not been vaccinated against smallpox and a fourth were not protected against diphtheria, tetanus, pertussis, or poliomyelitis, (*f*) nonwhite children had appreciably less medical care than white children, in terms of services available and per patient expenditures. Subsequent reports by the State board of health supported findings of inadequate medical care in the area studied.

TAMBLYN, PETER B. (National Communicable Disease Center, Public Health Service), and JACOBSON, JOANNE: *Continuance of contraception post partum by patients of Cook County Hospital. Public Health Reports, Vol. 85, March 1970, pp. 220–224.*

High rates of acceptance of contraceptive services in the immediate post partum period indicate that this period offers an effective means of initially reaching the women in need of family planning services. The effectiveness of this strategy needs to be evaluated, however, not only in terms of initial acceptance but in the continuance of contraceptive practices as well.

An unbiased sample of 100 women who had accepted a method of family planning before leaving the delivery wards of Cook County (Ill.) Hospital was selected for study by the Planned Parenthood Association, Chicago Area. Followup 2 to 3 months post partum revealed that 69 of the 77 women who were reached (89.6 percent) continued to practice a method of birth control. Women lost to followup are a highly mobile group who presumably would have lower rates of continuance of contraception than those who could be contacted. Intensive efforts to reach women normally lost to followup should, therefore, reveal more dropouts and thus lower the overall continuance rate. Yet, even though the proportion in the study lost to followup was only half that found in previous PPACA evaluations at Cook County Hospital, the women who could be followed showed as high a rate of continuance as those in previous evaluations.

Rates of continuance of contraception by method were higher for oral contraceptives than for foam; referrals for intrauterine devices were least often completed.

BIGLER, W. J. (Florida Division of Health), COLLINS, T. E., NICHOLS, J. B., GALTON, M. M., and PRATHER, E. C.: *Trends of sporadic leptospirosis in Florida, Public Health Reports, Vol. 85, March 1970, pp. 225–232.*

Since 1957, the staff of the Florida Division of Health's bureau of laboratories have considered all cases of aseptic meningitis and other diseases related to the central nervous system as potential cases of leptospirosis. Between 1958 and 1967, an examination of 6,066 serum specimens revealed 86 cases of leptospirosis. In the microscopic agglutination test, 78 of these specimens showed significant agglutinins to the leptospiral organisms of 10 serogroups.

The Florida experience differs from the national one both in the predominant infecting serotype and the age group of patients. In Florida, 46 percent of the leptospirosis cases were attributed to *Leptospira canicola*, while only 28.4 percent of the cases in the United States between 1947 and 1967 were attributed to this serotype. Among the Florida cases, 9.5 percent occurred in persons in the age groups 30–39 years and 52.7 percent in the age group under 20. National data showed 21.4 percent to be in the 30–39 age group and only 22.4 percent in the group under 20. Between 1964 and 1967, 11 of 15 *L. canicola* cases in Florida occurred in children 6 to 12 years old.

Intensive epidemiologic investigations were conducted of two cases in 12-year-old boys from different families who had cared for the family hunting dogs. *L. canicola* infections related to contact with dogs represent the predominant kind of human leptospirosis in Florida.

phs synopses

FREDERIKSEN, HARALD (Agency for International Development), and RAVENHOLT, R. T.: ***Thromboembolism, oral contraceptives, and cigarettes. Public Health Reports, Vol. 85, March 1970, pp. 197–205.***

The upper limits of the excess mortality from thromboembolism in 1966 that was possibly attributable to use of oral contraceptives by American women 20–44 years of age is estimated to be in the order of three or four deaths per 100,000 users. Less than 171 of the deaths from thromboembolism in the United States in that year could be attributed to use of oral contraceptives by women 20–44 years of age.

These results, obtained by an entirely different method, confirm previous estimates of the levels of mortality due to thromboembolism resulting from use of oral contraceptives in the United Kingdom and the United States.

The annual risk of mortality attributed to thromboembolism from use of oral contraceptives is comparable to the mortality risks due to all causes of death that result from complications of pregnancy, child birth, and the puerperium—namely, 3.1 deaths per 100,000 women 20–44 years of age and 3.6 deaths per 100,000 women 20–44 years of age not using oral contraceptives. The risk of death from thromboembolism resulting from oral contraception increases sharply with age.

Users of oral contraceptives who were heavy smokers had an incidence of thromboembolism 23 times that of women who neither used oral contraceptives nor smoked cigarettes. When the proportion of heavy smokers among users of oral contraceptives having thromboembolism was compared with the proportion of heavy smokers among users of oral contraceptives having no thromboembolism, an association was found between heavy smoking and thromboembolism that would occur by chance about 1 in 15 times.

Firm conclusions concerning the significance of any interactions between cigarette smoking and oral contraception must be based on larger samples. Nevertheless, the available data can hardly be used to rule out the possibility that cigarettes may potentiate the etiological role of oral contraceptives in the pathogenesis of thromboembolism. This possible potentiating effect suggests that oral contraception be considered as another contraindiction to cigarette smoking.

HILL, CHARLES A., Jr. (Public Health Service): ***Measures of longevity of American Indians, Public Health Reports, Vol. 85, March 1970, pp. 233–239.***

Measures of comparative longevity are often misused by persons describing the differences between the health status of American Indians and that of other Americans. In 1967, the expectation of life at birth for Indians was 64 years and the average age at death was 46 years. For the total population in the United States, the life expectancy was 70 years and the average age at death was 65 years. A common mistake is to compare the Indian average age at death with the life expectancy in the general population; another error is to use the terms as if they meant essentially the same thing.

A life expectancy is based on a life-table calculation and represents the average number of years a member of a hypothetical cohort of persons could expect to live at the time of birth. This cohort is assumed to experience throughout its lifetime the set of age-specific death rates observed in the underlying population during a short, fixed observation period. As used by the Indian Health Service, the average age at death simply means the arithmetic mean of the ages of all persons dying during the year in an actual population. The average age at death is of questionable value as a measure of longevity because it ignores the size and age distribution of the population; also, it may not adequately represent the bimodal and skewed age distribution typical of deaths. Nor is a life expectancy meant to forecast; it is based on current death rates rather than on projections of future mortality levels.

VERMA, MAHADEO P. (Continental Research Institute, New York City), BECKER, WILLIAM H., CHRISTAKIS, GEORGE, and SCHILLING, FRED J.: ***Hyperlipidemia as a prognosticator of abnormal glucose tolerance. An exploratory study. Public Health Reports, Vol. 85, March 1970, pp. 241–245.***

In an exploratory study to determine whether an association exists between lipid levels and response to the glucose tolerance test, 134 men and women, aged 40–65 years, were classified as either "apparently healthy" or "predisposed to diabetes," based on results of medical examinations. They were then categorized further according to serum lipid levels. All the subjects were given a glucose tolerance test.

Seventeen of 63 apparently healthy and 25 of 71 predisposed-to-diabetes subjects had an abnormal glucose tolerance response. Within the lipid categories (defined as combinations of high and low serum triglyceride and serum cholesterol) in the predisposed-to-diabetes group, the glucose positive subjects ranged from 26 to 48 percent. Among the apparently healthy group this range was 5 percent (in the low serum triglyceride and low serum cholesterol category) to 62 percent (in the high serum triglyceride and high serum cholesterol category).

The results indicate that increased levels of serum triglyceride ($\geq$ 160 mg. per 100 ml.) in conjunction with increased levels of serum cholesterol ($\geq$ 260 mg. per 100 ml.) in apparently healthy mature people have a prognostic potential for a positive glucose tolerance response.