ing the U.V. absorption curve of the extract suspected of containing vitamin A from the U.V. absorption curve of the extract after the dehydration reaction. If the "difference curve" is the characteristic triple-peak absorption curve of anhydrovitamin A, the presence of vitamin A in the original extract is proved. The absorption curve of the anhydrovitamin A prepared from the unsaponifiable fraction of lard distillate is shown in Fig. 1. This establishes the presence of vitamin A, per se, in the preparation. The results of these determinations give no indication of more vitamin-A bioactivity than can be determined as vitamin A by conventional chemical analyses. The failure of some investigators to find vitamin A in lard concentrates may have been due to loss during saponification of the small amounts of vitamin A present.

Thus, the postulated "lard factor" of Kaunitz and coworkers has been identified as vitamin A. Vitamin A, per se, has been found in amounts sufficient to account for the vitamin-A bioactivity of lard distillates.

#### **References and Notes**

- Communication No. 201: No. XI of a series entitled "Biochemical Studies on Vitamin A.
- H. Kaunitz, C. A. Slanetz, and R. E. Johnson, Federa-1. tion Proc. 9, 335 (1950). 2. H. Kaunitz and C. A. Slanetz, J. Nutrition 42, 375
- (1950).
- -, Proc. Soc. Exptl. Biol. Med. 75, 322 (1950). 3.
- 5.
- Federation Proc. 10, 360 (1951).
   H. Stoerk, H. Kaunitz, and C. A. Slanetz, Arch. Pathol. 53, 15 (1952).
   J. S. Lowe, R. A. Morton, and R. G. Harrison, Nature 6, 172, 716 (1953)
- J. S. Lowe and R. A. Morton, Biochem. J. (London) 55, 7. 681 (1953).
- 8. C. Engel, Voeding 12, 310 (1951).
- E. H. Herb et al., J. Nutrition 51, 393 (1953).
   E. M. Shantz, J. D. Cawley, and N. D. Embree, J. Am. 10. *Chem. Soc.* **65**, 901 (1943). J. C. Abels *et al.*, *J. Clin. Invest.* **20**, 749 (1941).
- Suggested revision of the USP biological assays for vita-
- mins A and D submitted to the USP by the Animal Nutrition Research Council through C. I. Bliss, 15 Nov. 1948.

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## Graphitization of Diamond

### F. S. Phinney

Crystal Branch, Solid State Division, Naval Research Laboratory, Washington, D.C.

There have been numerous investigations of the graphitization of diamond under a variety of conditions (1–19). From the literature it appears that the diamond-graphite transition rate (in vacuum or inert atmosphere) is zero or immeasurably slow below 1000°C, measurable from 1000° to 1500°C, but at any given temperature has not been reproducibly measured. The temperature at which the transition rate is first measurable has not been established.

An attempt has been made in this laboratory to extend the data on the transition. Each diamond was heated in an atmosphere of dry helium (99.95 percent pure with 0.05 percent  $H_2$ ). The sample was placed in a recrystallized alumina crucible fitted with alumina lid which was encased in a graphite crucible and lids. This crucible assembly was suspended in a gastight mullite tube through which the dry helium was passed. A protected Pt-Pt 10 percent Rh thermocouple adjacent to the crucible was used to follow the temperature.

Clear, industrial-grade diamond maccles that were free from obvious inclusions were selected. These weighed 10 to 20 mg each, were triangular shaped, were approximately 1 mm thick, and had 2- to 3-mm equilateral sides. Individual experiments, summarized in Table 1, were as follows:

Table 1. Graphitization of diamond.

Wt. of dia- mond (mg)	Temp. (°C)	Time (hr)	$\begin{array}{c} \text{Per-}\\ \text{cent-}\\ \text{age}\\ \text{loss}\\ \pm 0.2\% \end{array}$	Microscopic examination after heating
$\begin{array}{c} 16.82\\ 16.82\\ 11.88\\ 11.88\\ 18.70\\ 18.57\\ 17.14\\ 17.18\\ 17.38\\ 15.24\\ 11.76\\ 19.76\\ 9.71 \end{array}$	$\begin{array}{c} 1000\\ 1100\\ 1200\\ 1200\\ 1200\\ 1300\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 1400\\ 100\\ 1$	$16 \\ 17 \\ 24 \\ 50 \\ 24 \\ 50 \\ 50 \\ 6 \\ 24 \\ 49 \\ 49 \\ 48 \\ 48 \\ 48 \\ 48 \\$	$\begin{array}{c} 0.0\\ .0\\ .0\\ .0\\ .2\\ 1.1\\ 0.0\\ .6\\ 1.2\\ 2.1\\ 1.3\\ 2.1\\ \end{array}$	No graphitization         Superficial graphitization         Black coating         Superficial blackening         Black coating

1) A diamond was cleaned with acetone, dried, and weighed to  $\pm 0.01$  mg.

2) It was placed in the alumina crucible encased in the graphite crucible. The crucibles were covered with lids of alumina and graphite, respectively, and the assembly was placed in the furnace.

3) The helium flow was started and the furnace was equilibrated at the desired temperature.

4) At end of the desired heating time, the furnace was shut off and cooled to room temperature with helium flow continued.

5) The diamond was removed, weighed to  $\pm 0.01$  mg, and viewed under a microscope to determine the nature and extent of any change. In some cases x-ray diffraction studies of the surface were made.

6) The diamond was treated with hot perchloric acid and ammonium vanadate to remove any graphite formed.

7) The diamond was cleaned with acetone and reweighed to  $\pm 0.01$  mg. The loss in weight was taken as the percentage of graphitization.

A blank on the original material showed no loss in weight upon treatment with hot perchloric acid and ammonium vanadate. The heating time is taken from the time the furnace reached the specified temperature until the heat was turned off. Approximately  $\frac{1}{2}$  to 1 hr was needed to reach the specified temperature and approximately  $\frac{1}{2}$  hr to cool below 1000°C when the heating current was cut off. The temperatures were maintained within 15°C of those shown in Table 1.

Diamond powders (40-50, 120-140, 230-270, 500, and 4000 mesh) were also heated but with no clear-cut results. The trend of the data indicates that graphitization starts at temperatures of 50° to 100°C lower than for diamond maccles under the same conditions.

Clear, inclusion-free, industrial-grade diamonds when heated under helium for periods of 50 hr (i) do not graphitize at temperatures below 1200°C, (ii) graphitize superficially at 1300°C, and (iii) show approximately 1 to 2 percent graphitization at 1400°C.

Diamond powders appear to start graphitizing 50° to 100°C below the temperature at which maccles graphitize under the same conditions. As the graphitization proceeds, the crystals become progressively darker brown in color. This color develops even at 900°C in crystals that show no graphite by x-ray diffraction examination.

#### References

- J. W. Mellor, A Comprehensive Treatise on Inorganic and Theoretical Chemistry (Longmans, Green, London, 1924), vol. 5, pp. 724-29, 765.
   A. Krause, Ber. deut. chem Ges. 23, 2409 (1890).

- A. Krause, ber. acat. chem Ges. 23, 2409 (1990).
   H. Moissan, Ann. chim. et phys. (7), 9, 133 (1896).
   C. A. Parsons and A. A. C. Swinton, Proc. Roy. Soc. (London) (A) 80, 184 (1907).
   R. Vogel and G. Tammann, Hoppe-Seyler's Z. physiol. 4.
- 5. Chem. 69, 598 (1910).
- 6. C. Doelter, Monatsh. Chem. 32, 280 (1911); Handbuch der Mineralchemie (Steinkopff, Dresden, 1912), vol. 1, p. 39.
- M. Foix, Bull. soc. chim. France 31, 678 (1922) M. Dek. Thompson and P. K. Fröhlich, Trans. Am. Elec-trochem. Soc. preprint (1923). 8.
- M. G. Friedel, Bull. soc. franç. minéral. (1923-24), pp. 46-47, 60-94; M. G. Friedel and G. Riband, *ibid.*, pp. 9. 94-116; Compt. Rend. 178, 1126 (1924).
- 10. P. Lebeau and M. Picon, Compt. Rend. 179, 1059 (1924). B. Lanyl and I. Lzarvasy, Math. u. naturw. Anz. ungar. Akad. Wiss. 48, 137 (1931). 11.
- 12.
- E. Papp, Magyar Chem. Folyóirat 39, 106 (1933).
   N. L. Nagendia Nath, Proc. Indian Acad. Sci. 2A, 143 (1935). 13.
- P. Corriez, Compt. Rend. 202, 59 (1936).
- 15. 16.
- R. M. Burrer, J. Chem. Soc. 1256 (1936).
  R. M. Burrer, J. Chem. Soc. 1256 (1936).
  E. Storfer, Trans. Furaduy Soc. 34, 639 (1938).
  R. S. Krishman, Proc. Indian Acad. Sci. 24A, 33 (1946).
  P. W. Bridgman, J. Chem. Phys. 15, No. 2, 92 (1947). 17. 18.
- H. J. Grenville-Wells, J. Min. Soc. Am. 37, (7 & 8), 700 19. (1952).

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# A Sensitive, Inexpensive Light Meter for Photomicrography

Paul Machowicz and E. W. Powell Biology Department, Western Reserve University, Cleveland, Obio

The light meter described here can be used with 35-mm cameras and is suitable for determining the proper exposure for fixed slides or living preparations.

We found standard light meters to be inconvenient for photomicrography for the following reasons: (i) interference of incident light; (ii) inaccurate readings on light of low intensity; (iii) difficult to adapt readily to ocular tubes. The commercial meters made for photomicrography that cost between \$92 and \$110 seemed expensive. We constructed a meter from the

following materials for between \$20 and \$25: photocell (Klett-Summerson type);  $5 \times$  ocular tube; brass plate for mounting the ocular tube; Simpson 0-50 4in. scale microammeter; contact switch; metal mounting box.

Figure 1 shows the method of assembly. The  $5 \times$ ocular tube could be threaded into the plate, but Fred Lindow, the machinist of the Physics Department who aided us, simply pressed the ocular tube into a snugly fitted hole drilled in the plate. The fitting was sufficiently tight that the ocular tube did not need to be sweated in. Appropriate holes were drilled in the brass plate and metal box. These holes coincided with the holes present in the Klett photocell mounting. The microammeter was mounted to the metal box with metal screws, and the necessary electric connections were made.



Fig. 1. Assembly of light meter.

The calibration was made with Plus-X, 35-mm film in a Leica camera, using a Micro Ibso attachment. Any other camera of similar type may be used. The calibration was made by controlling the light intensity with neutral filters to give the following meter readings: 1, 5, 10, 15, 20, 25, and 30 (Table 1). The exposed Plus-X 35-mm film was developed in a tank with Ansco Finex L developer at 20°C for 15 min and fixed in Edwal Quik fix for the required time at 20°C. The film was washed at 20°C for 20 min.

The correct exposure time was determined by visual inspection of the negatives and the printing quality on glossy, normal contrast, projection paper. The

Table 1. Calibration of meter using Plus-X film.

Meter reading	F	Exposure time (sec)				
1 .	1/10	1/25	1/50	1/125		
5	1/50	1/125	1/200	1/500		
10	1/125	1/200	1/500			
15	1/125	1/200	1/500			
20	1/125	1/200	1/500			
25	1/125	1/200	1/500			
30	1/125	1/200	1/500			