Reports

Lunar Occultation of X-ray Emission from the Crab Nebula

Abstract. *The x-ray flux from the Crab Nebula was observed during a lunar occultation on 7 July 1964. As the moon covered the central region of the nebula, the x-ray flux decreased gradually. The source appears to extend over a volume about 1 light-year in diameter.*

On 7 July 1964, at 2242:30 U.T., an Aerobee rocket was launched from White Sands missile range to observe the lunar occultation of x-ray emission from the Crab Nebula. The purpose of the observation was to determine the angular diameter of the source of x-ray emission. A year ago, exploratory measurements (*1*) had revealed two discrete sources of x-rays in the galaxy. One was located in the Scorpius region; the other was centered on the Crab Nebula. In Scorpius, no visible object or radio source could be identified with the x-ray emission. The Crab Nebula, however, is the remnant of a supernova explosion that occurred in A.D. 1054, and it is one of the strongest radio sources in the galaxy. It was proposed (*1*) that both x-ray sources could be neutron stars, the highly compressed cores that may remain after explosion of the outer portions of supernovae. According to the theory of a neutron star one can predict a strong x-ray source (*2*) with essentially no visible or radio emission, such as required to explain the Scorpius source. A neutron star might also exist, unobservable except for its x-ray emission, in the center of the Crab Nebula.

Lunar eclipses of the Crab Nebula are grouped at intervals of approximately 9 years. The eclipse of 7 July covered the nebula at the rate of one-half minute of arc per minute of time. Since the nebula measures 6 minutes of arc across its greatest dimension, to observe a full eclipse of the nebula would require 12 minutes of x-ray measurements by a rocket above the atmosphere. The Aerobee rocket affords only 5 minutes of time above 100 km. The interval for the experiment was, therefore, chosen so that the eclipse of a central portion of the nebula over a range of about 2 minutes of arc would be observed. The rocket was equipped with a stabilization system to orient two x-ray "telescopes" toward the Crab and to maintain steady pointing during the course of the occultation. If a neutron star x-ray source existed in the center of the Crab, the occultation would be expected to produce an abrupt disappearance of x-rays within a fraction of a second of time. A gradual disappearance of x-ray emission would indicate that the x-ray source was an extended cloud.

Two Geiger counters of nearly identical construction were used to detect the x-rays. Each counter was in the form of a shallow rectangular box strung with five anode wires. Across the face of the box was a plastic window of Mylar, making an x-ray transmitting window of 114 cm^2 area. To provide crude wavelength discrimination, one window was 0.001 inch (0.0025 cm) thick; the other was 0.00025 inch (0.00062 cm) thick. Both counter volumes were connected by tubing so that they shared a common gas filling composed of 89.5 percent Ne, 9.5 percent He, and 1 percent isobutane at atmospheric pressure. A gas-flow system was used to permit flushing both tubes in series, just prior to launch, from a storage tank of gas carried in the rocket. During flight the gas content was static at a regulated pressure. Figure 1 illustrates the calculated spectral efficiencies of the two counters. Up to a wavelength of about 3 Å both tubes had almost identical responses, but at longer wavelengths the thinner window provided increasingly greater efficiency. The two counters were mounted, one above the other, with their windows in the same plane facing outward, in a direction normal to the long axis of the rocket. Each window was covered by a honeycomb collimator, which limited the field of view. The angular response was roughly bell-shaped, with a maximum cutoff angle of 14 deg on either side of the normal direction.

The Aerobee was launched precisely on schedule. It was stabilized by a cold-gas-jet gyro-referenced control system, manufactured by the Space General Corporation under contract to the U.S. National Aeronautics and Space Administration. At launch, the fins of the rocket gave it a spin of 2 rev/sec to stabilize its flight through the atmosphere. After burnout, the rocket was de-spun and returned to the orientation it had before launch. From that moment, an on-board computer guided the rocket through a maneuver that had been programmed to point the detectors toward the Crab. During the roll portion of the maneuver, the detectors scanned the sun, which was only 18 deg from the Crab, and then slightly overshot the Crab. From analysis of the sun signal, magnetometer signals, and the gyroscope data, we conclude that the rocket fixed on its final orientation at 160 seconds after launch and that the detectors pointed within 4 deg of the Crab. From 160 to 400 seconds, the pointing did not deviate by more than ¼ deg.

Figure 2 shows the variation of x-ray counting rate through the course of the flight. At 100 seconds the rocket was above the absorbing atmosphere for x-rays of wavelengths shorter than 10 Å. As the detectors scanned the sun the strong flux of solar x-rays drove the counters rapidly to saturation and then into a choked condition. The degree of choking was far more pronounced in the counter with the thinner window, as would be expected from its longer wavelength sensitivity and higher overall efficiency. The responses of the counters to the entry of the sun into the field of view were almost perfectly mirrored as the sun passed out of the field of view. There was no indication of counting hysteresis; in fact test exposures to intense sources in the laboratory had revealed no tendency of the counters to lag in recovery. Thirteen seconds elapsed between disappearance of the

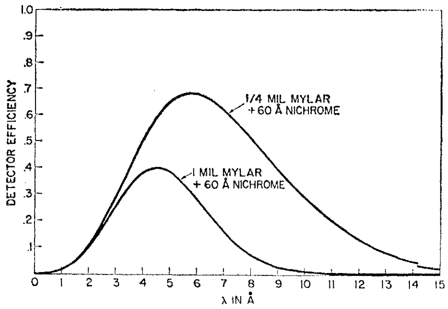

Fig. 1. Computed spectral sensitivity curves for the A and B counters.

sun's signal and acquisition of the final orientation to the Crab.

From 160 to 400 seconds, the variation in x-ray counting rate can be attributed to the progress of the lunar occultation. Figure 3 shows the relation between the time after launch and the disappearance of the Crab behind the moon. The peak of the flight was 221.4 km, which was reached at 250 seconds. For all of the time interval from 160 seconds (184 km) to 320 seconds (199 km) during which the decrease of x-ray flux was observed, the rocket was more than five scale heights above the level of unit optical depth of the atmosphere for wavelengths shorter than 10 Å (the limit of counter B with its 1-mil Mylar window) and more than three scale heights above unit optical depth for 60 Å (the limit of counter A with its ¼-mil Mylar window). The decrease of x-ray flux during the occultation was, therefore, not related to the variation of rocket altitude.

At the beginning of the occultation the counting rate changed very slowly; but as time passed there was a relatively rapid decline until it reached background rate observed in the 100- to 130-second interval. The curve of the sum of the two counter responses was differentiated to give the source distribution of Fig. 2. Three important conclusions can be drawn from these data: (i) the total angular width of the source is about 1 arc minute, which corresponds to about 1 light-year at a distance of 1100 parsec; (ii) the maximum of the x-ray flux distribution was observed at about 230 seconds, when the occultation had not quite reached the center of the visible nebula; and (iii) the spectrum must be confined primarily to the region below 5 Å, since there is no marked difference in the responses of the two counters. The computed ratio of the sensitivity of counter B to counter A was 1.03, 1.14, 1.27, and 1.9 for wavelength bands from 0 to 3 Å, 0 to 4 Å, 0 to 5 Å, and 0 to 8 Å, respectively. If much of the emission spectrum had exceeded 5 Å, counter B would have responded more strongly than A. The slightly weaker response of counter B was consistent with a difference, measured before the flight, in relative sensitivities of the two counters to cosmic ray background (*3*).

Figure 4 illustrates the results of the x-ray observation (*1*) of the Crab Nebula in April 1963. The detector in that flight had an effective area of 65 cm² and was covered by a honeycomb collimator of 10 deg aperture. In three scans, the Crab passed once directly across the center of the field of view and twice slightly off center by 1.5 and 2.5 deg. The average of the three passes showed a signal of 0.8 count/cm² per second above background. In the occultation observation, the intensity measured by the com-

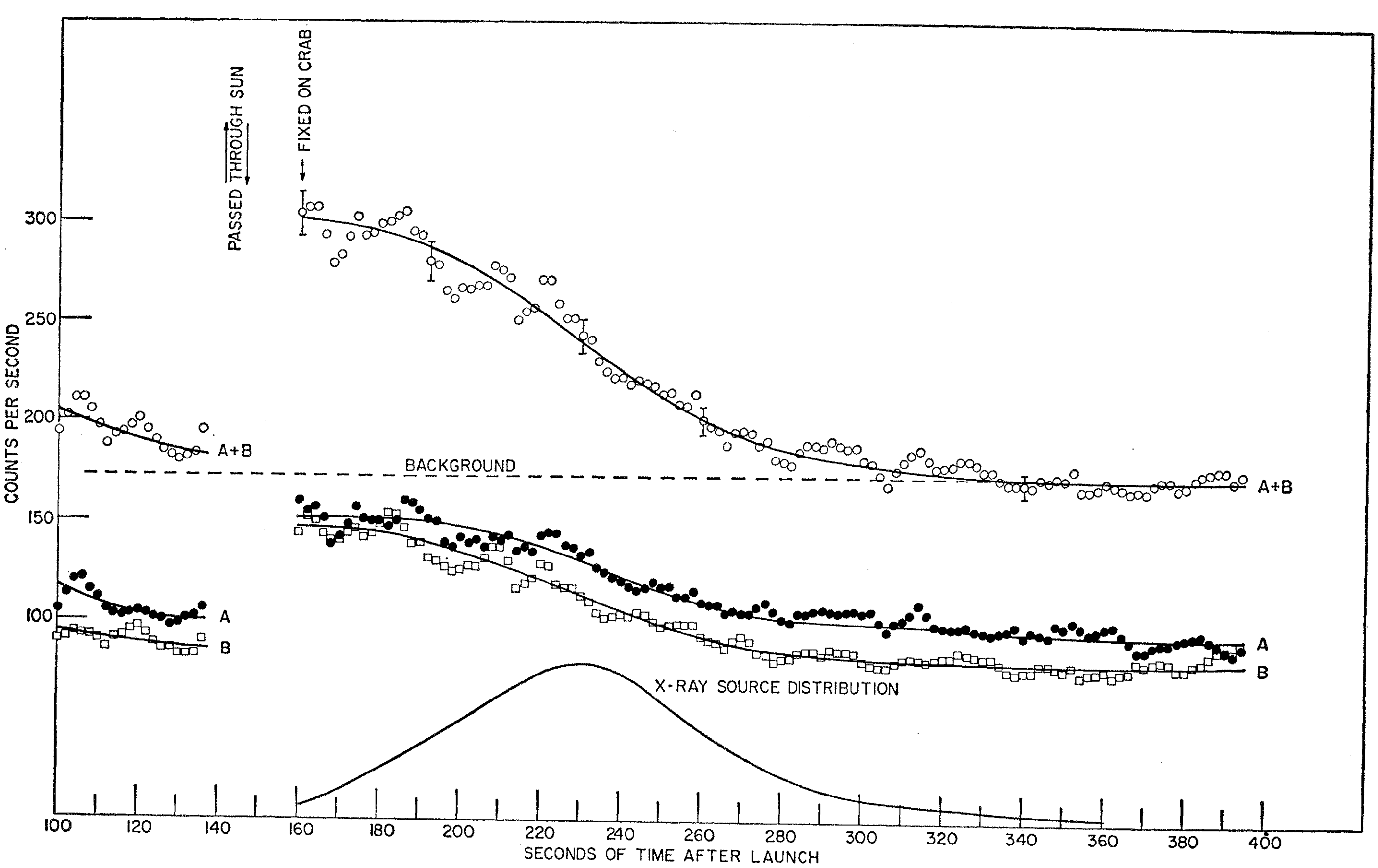

Fig. 2. Variation of the observed x-ray flux during the course of flight of 7 July 1964. The Mylar windows of counters A and B were 1 mil and ¼ mil thick, respectively. Counting rates were computed from the time required for a fixed count of 768 in each counter. A running mean is plotted at 2-second intervals. The x-ray source distribution is the derivative of the *A* and *B* curve.

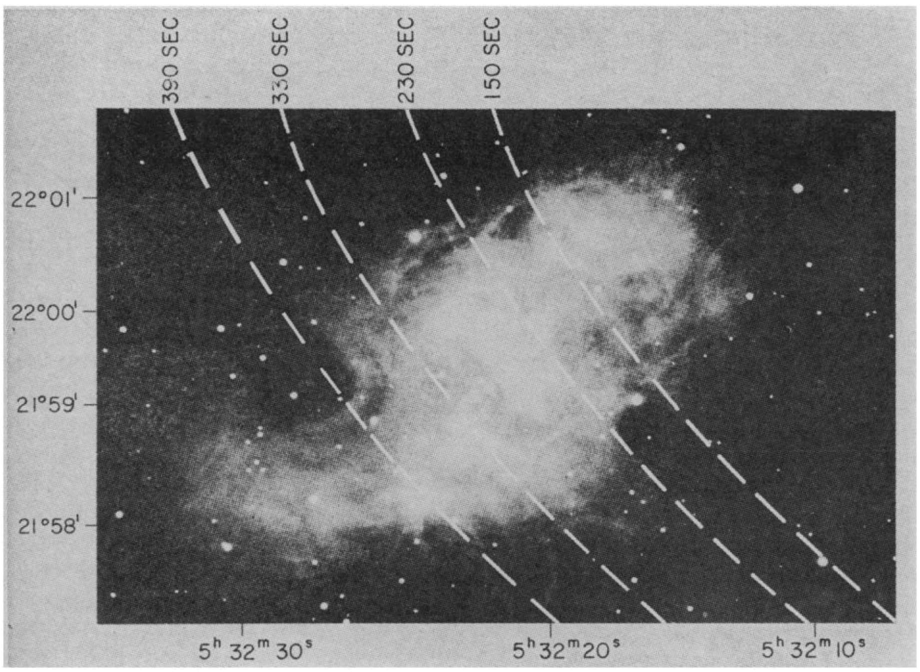

Fig. 3. Progress of the occultation of the Crab Nebula measured in seconds of time after launch of the rocket from White Sands missile range. The dashed curves represent the positions of the edge of the moon. A maximum rate of decrease in x-ray flux was observed at about 230 seconds.

bined A and B counters was 0.45 count/cm^2 per second above background. When these count fluxes are corrected for the efficiencies of the detectors, the agreement between the two sets of observations is quite satisfactory, provided the spectrum of the measured x-rays was concentrated between 3 and 5 Å. This is consistent with the distribution of pulse amplitude observed with the proportional counter in April 1963. Pulses were then sorted into three channels: 1 to 1.5 Å, 1.5 to 2.5 Å, and 2.5 to 8 Å. Nearly all the observed flux was found in the last channel.

Since both the radio spectrum and optical emissions from the amorphous mass of the Crab have been identified as synchrotron radiation, can the observation of x-ray emission from an extended source also be attributed to synchrotron radiation? Woltjer (*4*) has estimated the synchrotron flux in the far ultraviolet by assuming it to be the source of excitation of the filamentary shell. A simple extrapolation of the short-wavelength end of the optical spectrum through the ultraviolet region estimated by Woltjer to the x-ray region falls within an order of magnitude of the observed x-ray flux. Shklovsky (*5*) has also pointed out that with a slight modification in the value of the interstellar absorption of light from the Crab Nebula it is possible to fit the observations of synchrotron emission at 3000 Å to the x-ray flux at about 3 Å with a common spectral index of 1.2. In contradiction to the synchrotron hypothesis, however, is the lack of any indication of a soft-radiation excess in counter B relative to counter A. The synchrotron flux should increase with increasing wavelength, and over the band 0 to 8 Å counter B was approximately twice as sensitive as counter A. The observation, therefore, is incompatible with a synchrotron spectrum unless interstellar opacity sharply attenuates wavelengths longer than about 5 Å.

Table 1. Flux from the Crab Nebula in the region of 1.5 to 8 Å. The data from the Be window counter of the flight on 29 April 1963 and the two Mylar window counters of the flight on 7 July 1964 are best matched by a thermal spectrum. The estimate for the synchrotron spectrum, third column, is based on a spectral coefficient $\gamma = -1.1$, and includes the above absorption figures.

Window (mil)	Date	Flux (10^{-8} erg cm^{-2} sec^{-1})		
		Black body, 2×10^7 °K	Flat spectrum	Synchrotron spectrum
5 Be	4/63	1.5	1.3	1.6
1 Mylar	7/64	1.5	0.84	0.76
1/4 Mylar	7/64	1.1	0.4	0.03

From radio astronomical measurements (*6*), the hydrogen column between the sun and the Crab Nebula has been found to contain 1.5×10^{21} hydrogen atoms per cubic centimeter. Taking Cameron's estimate (*7*) of the relative abundance of the elements and the absorption coefficients of Henke *et al.* (*8*), we find the optical thicknesses at 5, 7, and 10 Å to be 0.04, 0.09, and 0.25. Interstellar opacity, therefore, cannot appreciably modify the source spectrum by preferential absorption of the wavelengths between 5 and 10 Å unless the radio evidence for the hydrogen atom concentration is in error by at least an order of magnitude (see Table 1).

The Crab Nebula appears much larger at meter wavelengths than in the visible range. Most of the optical emission is concentrated within a region measuring approximately 2 minutes of arc in diameter, about two to three times smaller than the radio size. The diameter of the x-ray emitting volume is less than half that of the optical nebula. Shklovsky (*5*) suggests that the small size of the x-ray nebula may be related to the lifetimes of highly relativistic electrons responsible for generation of x-ray synchrotron radiation. The lifetime varies inversely with the 3/2 power of the magnetic field multiplied by the square root of the frequency. Estimates of the field strength range from 10^{-4} to 10^{-3} gauss, and it is most likely that the higher figure is reached near the center of the nebula. An electron radiating in the x-ray spectrum, $\sim10^{18}$ cy/sec, in a field of 10^{-3} gauss would have a half-life of about 0.8 years. Shklovsky proposes that these high energy electrons, $E \sim 10^{13}$ ev, are accelerated in a small central region of the nebula. Traveling at nearly the speed of light, they could get no farther than about 1 light-year from the region of acceleration. This would limit the size of the x-ray emitting region to roughly the observed value.

The x-ray emission may be produced by bremsstrahlung in a nebula of the observed size. If the emission is at peak near 3 Å, the temperature must be about 10^7°K, and the nebula would need to contain about 0.3 $M_\odot$ of hydrogen ($M_\odot$ = 1 solar mass) to account for the observed flux.

To what can we attribute such a high gas temperature? The total thermal content of 1 $M_\odot$ of gas at

10,000,000°K is of the order of 10^{48} erg. The Crab belongs to the Type I class of supernovae which originate in population II stars, of mass comparable to the sun. The total energy output of a Type I supernova is estimated to be of the order of 10^{51} erg. In the first 20 to 30 days of the supernova outburst, the light intensity drops only 2 or 3 magnitudes. During this time the integrated light output is about 4×10^{49} erg. For the Crab Nebula, at present, the following estimates have been made: total kinetic energy of the filaments is between 10^{48} and 10^{49} erg; total relativistic electron energy and total energy in magnetic fields is about 10^{48} erg; and the total radioactive energy is about 10^{48} erg. After 1000 years, the energy left in the Crab Nebula is still of the order of 10^{-3} of the original content of the explosion. Furthermore, it appears that energy is still being generated, as evidenced by the light ripples which are observed near the center of the nebula.

The x-ray nebula may derive its present energy from the original hot gas of the exploding star. At the observed x-ray emission rate the energy lost in the past 900 years would amount to only 5×10^{46} erg. Cooling by x-ray emission is, therefore, very slow relative to the lifetime of the Crab Nebula. At temperatures below several tens of millions of degrees, however, recombination of electrons with heavy ions proceeds rapidly, and cooling by radiation from excited ions is very efficient compared to bremsstrahlung. The x-ray nebula may, therefore, be cooling from outside toward the center with a rather steep temperature gradient near the boundary of the x-ray emission.

After the initial flash of a Type I supernova, the light output follows a nearly exponential decay, with a half-life comparable to that of Californium-254, about 55 days. According to Fowler and Hoyle (*9*), Cf^{254} is formed by rapid neutron capture. Evidence to support this hypothesis comes from observation of the build-up of Cf^{254} within microseconds of a hydrogen bomb explosion. Along with Cf a chain of other radioactive elements must be produced, some of which have much longer half-lives. Burbidge, Burbidge, Fowler, and Hoyle (*10*) calculated that the spontaneous fission of Cf^{254} in the Crab produced energy at the initial rate of 1.45×10^{41} erg/sec. Woltjer (*11*) has extended their calculations to the present time and found that: (i) if the neutron flux was sufficient to transport all the processed material to the range of atomic weights 110 to 260 and cycled in this range, the total energy production after 1000 years would be 5×10^{36} erg/sec; (ii) if steady flow was reached in the neutron capture process leading to abundances such as are found in the solar system, the energy developed after 1000 years would be 1.3×10^{37} erg/sec. In the first case the present activity would be associated with fission of Cm^{250} and in the second case with the beta decay of Si^{32} and Ar^{39}. In comparison to the observed x-ray output of the Crab, about 2×10^{36} erg/sec, the theoretical radioactive energy production appears to be adequate to supply the heat source for x-ray emission.

To explain the observation of x-rays

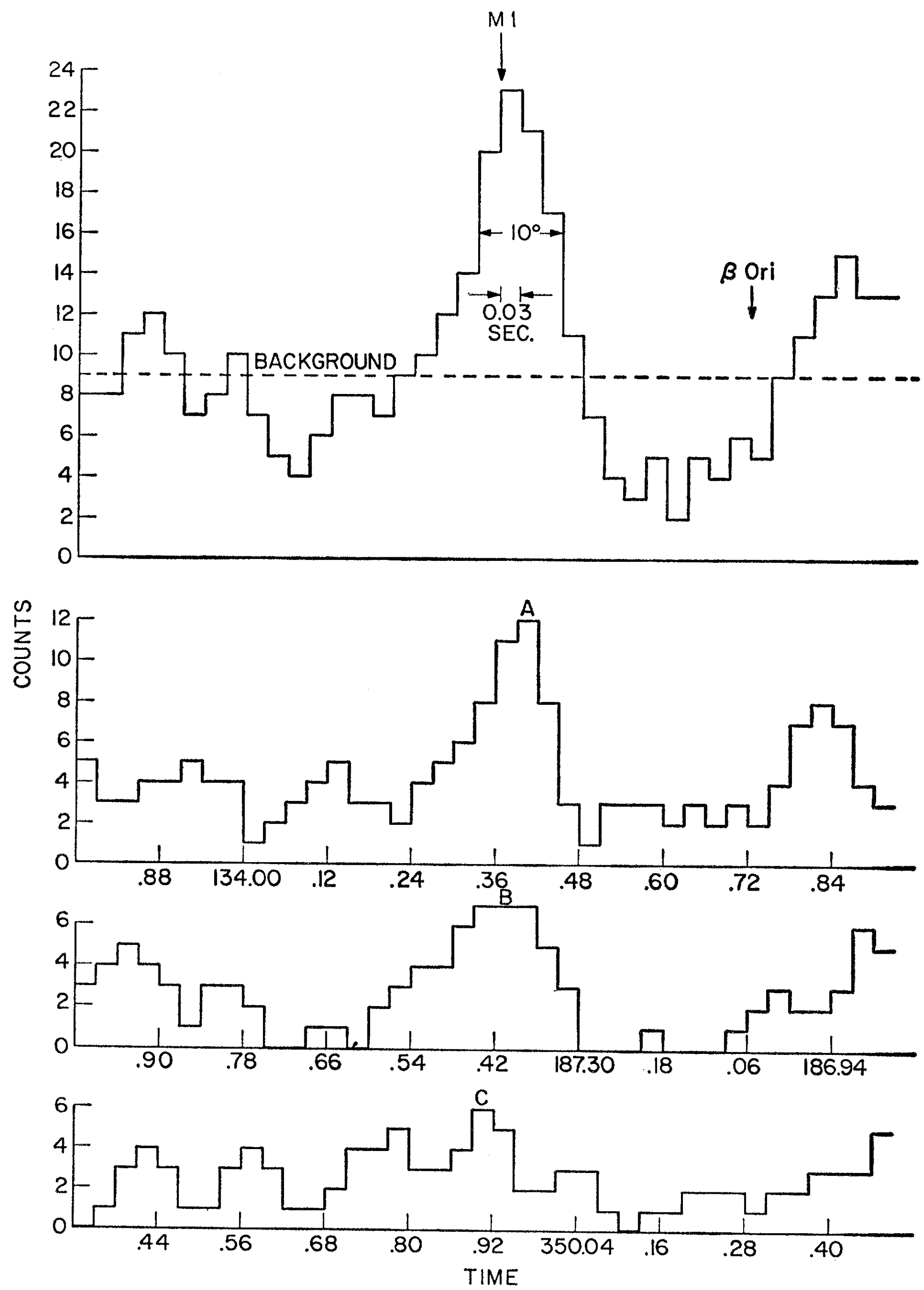

Fig. 4. Observation of the x-ray flux from the Crab Nebula on 29 April 1963. The upper graph is the sum of three separate scans *A*, *B*, and *C*. In scan *A*, the source passed directly across the center of the field of view, which was 10 deg in diameter; scan *B* was 1.5 deg, and scan *C* was 2.5 deg off center. A running mean of counts per 0.09 second is plotted at intervals of 0.03 second. The peak intensity corresponds to 52 count/cm² per second.

from Scorpius, Heiles (*12*) proposed that the shock wave which emanates from a supernova explosion can heat the interstellar gas immediately behind the shock to temperatures as high as 10^7 °K. Applying this hypothesis to the observation of x-rays from the Crab would require that a relatively dense gas cloud existed before the supernova explosion and that, once heated by the shock, it did not cool below 10^7 °K to the present time. This could set a boundary to the high temperature emission region.

According to O'Dell (*13*) the mass deduced from an oblate spheroid model of the Crab is 0.64 $M_\odot$; a prolate model leads to a mass of 3.3 $M_\odot$. These estimates are based on an electron temperature of 17,000 °K for the filaments. The high temperature x-ray nebula would produce a negligible intensity of visible radiation and would not contribute to the mass estimate. It appears that a requirement of a few tenths $M_\odot$ for the x-ray source is not unreasonable.

It has been suggested that the collapse of the pre-supernova might well lead to the formation of several neutron stars and that these might be distributed so as to give the appearance of the observed diffuse source. Consideration of the conservation of angular momentum, however, requires that the stars exist in such close proximity that they would not be distinguishable from a single point source in the occultation. The gravitational energy in such a collection of neutron stars could constitute an important reservoir of energy for continued excitation of the nebula, even if the stars were too cool to produce observable x-rays.

Overbeck (*14*) has considered the possibility that the x-rays from a neutron star at the distance of the Crab would be diffused by small-angle scattering on interstellar dust grains. He found, however, that with the generally accepted values of average grain size and concentration the mean free path for scattering is about eight times the distance to the Crab. It would appear, therefore, that most of the x-ray flux should traverse the interstellar medium unscattered and that only a minor fraction of the flux may be diffused.

Colgate (*15*) has suggested that the supernova explosion could leave a relatively dense cloud of Fe-group elements concentrated near the explosion center, while the envelope of the nebula would be formed of only the lighter hydrogen and helium constituents of the outer shell of the pre-supernova. It would then be possible for x-rays from a point source to be confined by photoelectric absorption in the surrounding nebula, and for fluorescent x-rays to escape. Let us consider a very simplified model in which a neutron star is surrounded by a cloud of iron and the flux from the star is confined to the region below the K absorption edge of Fe (λ = 1.74 Å), but primarily concentrated between that wavelength and about 1 Å. Unit optical depth for the primary radiation would be about 5×10^{19} atoms per square centimeter, or 100 per cubic centimeter in a volume having a diameter of 1 light-year. It would require 2.5 $M_\odot$ to fill this volume to a density of 100 atoms per cm^3. To reduce the escaping radiation to 10 percent or less would require an optical depth of at least 2 units, or $M_\odot$ of nebula. Most of the absorbed radiation would be converted to FeKα (λ = 1.94 Å) which would escape relatively freely from the region within which it was produced. The rocket detectors would then observe this monochromatic fluorescence from an extended source.

The above model, however, is oversimplified. A thermal neutron star spectrum with a peak in the 1.0 to 1.74 Å range would necessarily produce enough radiation to the long wavelength side of the absorption edge so that the stellar source would be seen through the Fe nebula. Shifting the star spectrum to shorter wavelength would require the temperature to be greater than 2×10^7 degrees. According to Morton's calculations (*2*) it is not possible to construct a neutron star model with a photospheric temperature greater than about 1.6×10^7 °K without raising that of the core and, accordingly, increasing the cooling by neutrino emission, to an unacceptably high rate.

The evidence against the existence of a neutron star x-ray source in the Crab raises the question of whether a Type II supernova may be more likely to produce a neutron star and whether the Scorpius source could be such an example. Shklovsky (*16*) has called attention to the close coincidence between the position which we established for the Scorpius x-ray source and the position proposed by Brown, Davies, and Hazard (*17*) for the center of a radio emitting shell associated with the radio object referred to as "The Spur." Radio frequency isophotes at 38 and 158 Mcy/sec have shown a spur of relatively intense emission which emerges from the galactic plane near the sun and curves about an apparent center at right ascension 15 hours, declination −20 deg.

Considering the approximate nature of this position fix (about 10 deg), it agrees quite well with our position of right ascension, 16 hr 15 min, declination −15 deg for the x-ray source in Scorpius. Brown *et al.* proposed that The Spur may be an object similar to the Cygnus loop which is believed to be the remnant of a Type II supernova. The Cygnus loop emission has a distribution consistent with a shell source about 10 parsec thick and 40 parsec in diameter at a distance of 770 parsec. By analogy, The Spur would be at a distance of about 50 parsec, and Shklovsky suggests that the age of this near supernova may be 50,000 to 100,000 years. If the intensity of x-ray emission is simply related to the age, the Scorpius source may be 100 times less intense than that of the Crab, but its close proximity would still make it appear brighter. However, any visible nebula of synchrotron radiation associated with it would have too low a surface brightness to be detectable against the background sky. The same would be true of its radio synchrotron emission. If the Scorpius supernova remnant contains an x-ray emitting region about 1 light year in diameter, its angular width at 50 parsec would be about ⅓ deg. The scan data of the April 1963 flight indicated only that the diameter did not exceed 2 or 3 deg. However, it should be possible in future observations with narrower mechanical collimation to establish the dimensions to a minute of arc or better.

S. Bowyer, E. T. Byram
T. A. Chubb, H. Friedman
E. O. Hulbert Center for Space Research, U.S. Naval Research Laboratory, Washington

References and Notes

1. S. Bowyer, E. T. Byram, T. A. Chubb, H. Friedman, *Nature* **201**, 1307 (1964).
2. D. C. Morton, *ibid.*, p. 1308; H. Y. Chiu and E. E. Salpeter, *Phys. Rev. Letters* **12**, 413 (1964)
3. It is possible that the insensitivity of counter

B relative to counter A may have been dependent on wavelength and, therefore, significantly greater at the longest x-ray wavelengths. Some evidence to this effect is apparent in the data of Table 1.
4. L. Woltjer, "X-rays and type I supernova remnants," preprint (1964).
5. I. S. Shklovsky, "On the energy spectrum of relativistic electrons in the Crab Nebula," private communication.
6. W. L. H. Shuter and G. L. Verschuur, *Monthly Notices Roy. Astron. Soc.* **127**, 387 (1964).
7. A. G. W. Cameron, *Astrophys. J.* **129**, 676 (1959).
8. B. L. Henke, R. White, B. Lundberg, *J. Appl. Phys.* **28**, 98 (1957).
9. F. Hoyle and W. A. Fowler, *Astrophys. J.* **132**, 565 (1960).
10. E. M. Burbidge, G. R. Burbidge, W. A. Fowler, F. Hoyle, *Rev. Mod. Phys.* **29**, 547 (1957).
11. L. Woltjer, *Bull. Astron. Inst. Netherlands* **14**, 39 (1958).
12. C. Heiles, *Astrophys. J.* **140**, 470 (1964).
13. C. O'Dell, *ibid.* **136**, 809 (1962).
14. J. W. Overbeck, "Small-angle scattering by interstellar grains as a source of angular broadening of celestial x-ray sources," preprint (1964).
15. S. Colgate, private communication.
16. I. S. Shklovsky, *Astron. Tzirkulyar* No. 304 U.S.S.R. (1964).
17. R. H. Brown, R. D. Davies, C. Hazard, *Observatory* **80**, 191 (1960).
18. The attitude control system used to stabilize the Aerobee Rocket was furnished by NASA. We thank Dr. W. Nicholson, of the Royal Greenwich Observatory, for supplying the occultation predictions upon which the observational program was based. Sponsored jointly by the Office of Naval Research and the National Science Foundation.

1 October 1964

Graphitization of Organic Material in a Progressively Metamorphosed Precambrian Iron Formation

Abstract. *Organic matter in the sedimentary Biwabik iron formation in northern Minnesota shows a progressive increase in crystallinity where the formation is metamorphosed by the intrusive Duluth gabbro complex. X-ray diffraction of acid-insoluble residues shows that there is a complete range in crystallinity, from amorphous material in the unmetamorphosed sediments to completely crystalline graphite adjacent to the gabbro.*

Examination of carbonaceous material from unmetamorphosed Precambrian sediments (*1*) has shown that such material is not crystalline graphite but is instead an amorphous aggregate of hydrocarbon compounds of high molecular weight which may be related to so-called "kerogen" found in younger sedimentary rocks (*2*).

During a study of progressive metamorphism of the Biwabik iron formation in northern Minnesota (*3*), carbonaceous matter from organic-rich layers in the formation was examined by x-ray diffraction to determine the degree of crystallinity at various metamorphic levels and to detect, if possible, development of graphite during metamorphism.

The Biwabik iron formation, on the Mesabi Range in northern Minnesota, is a chemical sediment of middle Precambrian age. It is composed chiefly of quartz, magnetite, hematite, siderite, ankerite, and several hydrous iron silicates (*3–5*).

On the eastern end of the Mesabi Range, near the town of Babbitt, the sediments were intruded by the Duluth gabbro complex about 10^9 years ago. Progressive changes in the mineralogy of the iron formation occur within a few miles of the contact (*3–6*). The moderately metamorphosed iron formation, 3 to 5 kilometers (2 to 3 miles) from the gabbro, is characterized by the iron-amphibole grunerite; grunerite-ankerite-calcite assemblages are typical. The highly metamorphosed iron formation adjacent to the gabbro is characterized by iron-rich pyroxenes, by reduction of hematite to magnetite, and by the absence of all carbonates except calcite.

The samples studied were collected along the strike of the Biwabik formation from the so-called "Intermediate Slate," a well-known and easily recognized marker bed. The unit is a dark gray to black, fine-grained and finely laminated layer, generally 2 to 15 meters (5 to 40 feet) thick. Analyses of this unit have shown from 1 to 4 percent carbon by weight (*4*, pp. 54–62). Unmetamorphosed "Intermediate Slate" is composed chiefly of siderite and iron-rich chamosite, with minor quartz, stilpnomelane, and magnetite. Grunerite, iron pyroxenes, and fayalite appear where the unit is metamorphosed.

The method of sample preparation was suggested by T. C. Hoering, of the Geophysical Laboratory of the Carnegie Institution of Washington, and has been used by him to separate organic components from sedimentary rocks. About 100 g of the "Intermediate Slate" sample were ground to minus 230 mesh and treated with hot hydrofluoric acid for about 24 hours to remove silicate and carbonate minerals. It was essential that all quartz be removed, because the strong (101) peak of quartz will completely mask the graphite (002) reflection if even a small amount of quartz is present. The insoluble residue was then treated with hot hydrochloric acid for about 24 hours to remove fluorides and fluosilicates. X-ray diffraction patterns were made to monitor the process at each stage.

The carbon concentrate obtained consisted of from 1 to 4 g of a dark gray or black material. Small portions were mounted on a glass slide in an acetone-Duco cement mixture and studied by x-ray diffraction, with Ni-filtered CuKα radiation. Small amounts

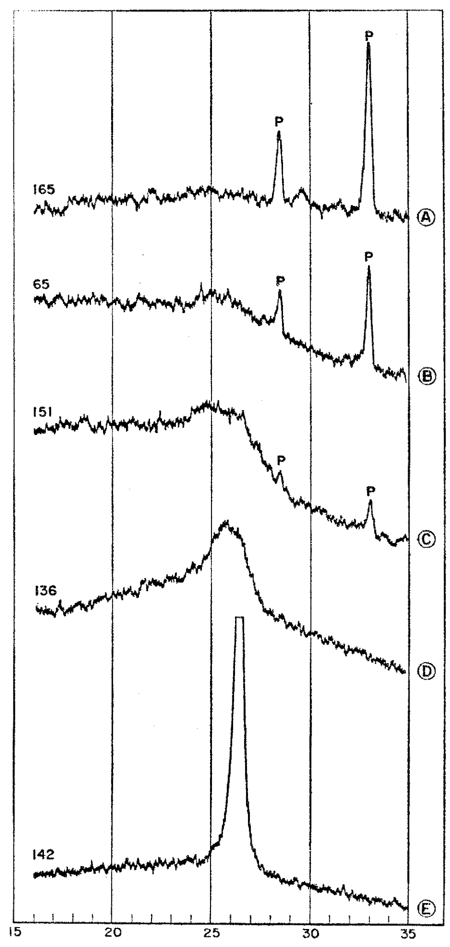

Fig. 1. X-ray diffraction patterns of organic matter extracted from samples of the "Intermediate Slate" member of the Biwabik iron formation. Sample numbers are shown at the left-hand side of the traces; the peaks designated "P" in patterns, *A*, *B*, and *C* are produced by small amounts of pyrite. Increasing graphitization is indicated by the gradual development of the strong (002) graphite reflection at about 26 degrees. Pattern *A* represents amorphous material from unmetamorphosed iron formation. Patterns *B* and *C* show the gradual appearance of a broad peak indicative of asphaltic substances. Pattern *D* is a disordered graphite, and pattern *E* represents graphite. The height of the peak in pattern *E* has been reduced by one-half relative to the other patterns.