VACUUM BEHAVIOUR OF VARIOUS MATERIALS WITH RADIATION AND HEAT*

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The vacuum chamber of the Alternating Gradient Synchrotron contains, of necessity, many materials which are not, at first, objectionable in vacuum, but with radiation and heat become inimical. Merely providing metal gaskets is not the entire answer because other seals, feed throughs, insulators and casting resins are a necessary part of the designs. These are all subject to a radiation field of about $1x10^9$ rads in a year of operation at the average AGS intensity of $1x10^{12}$ protons per pulse. It has been decided to investigate a number of materials as to their suitability before and after radiation to $5x10^9$ rads and also at temperatures of 120°C. This report is by no means complete but is the first in a continuing series.

The test apparatus is shown in Fig. 1. It consists of a 400ℓ/sec. sputter-ion pump, a valve and a chamber divided by an orifice plate of known conductance of 67ℓ/sec. The gas flow Q is obtained from the formula

$$Q = C(P_1 - P_2).$$

A calibration run is made on the chamber when empty and this Q is subtracted from the flow measured with a sample present. The only organics in the system are some Viton "O" rings associated with the valves. The mass spectrometer is a Veeco GA-3 and the ion gages are nude Bayard-Alpert with Cook controls.

The procedure in vacuum testing is as follows:

1. Clean sample in alcohol and air dry.
2. Install sample in chamber.
3. Leak check.
4. Pump on sample for 24-hours.
5. Vent chamber and sample to air for (1) one hour.
6. Repump and record pressures every four (4) hours for 100 hours.
7. Record mass spectrometer after one hundred (100) hours.
8. Vent chamber and remove sample.

The radiation tests were done in the Brookhaven graphite reactor and gamma facility. The samples were in vacuo, at a few microns during irradiation.

The first series of tests was conducted with the plastics, Lexan and Vespel. Lexan is a GE product, a polycarbonate resin with an excellent resistance to stability changes under U-V radiation. It could be useful as a casting resin for magnet insulation and as a potting resin. The outgassing rate after 100 hours is shown in Fig. 2 as $9x10^{-8}$ Torr ℓ/sec.cm^2. This is better than most epoxies. The mass spectrometer analysis (not shown) gives a typical spectrum for the hydro-carbons and water vapor. Mechanical tests after radiation show only a slight change in hardness or brittleness at $5x10^9$ rads. Its Rockwell H reading was 60-70 before and after radiation. Outgassing rate curves after radiation are not yet available.

Vespel, a Du Pont product, is a polyimide resin very similar to "H" film in chemical formulation. Its main use in the AGS would be for bearings, gasketing, insulators, and valve seats. Fig. 2 shows the outgassing rates before and after radiation. Before, it is better than any epoxies, but after $5x10^9$ rads it is down in the range of most of the metals and still improving after 100 hours. The mass spectrometer analyses shown in Figs. 5 and 6 illustrate the decrease in outgassing, particularly in the water vapor content. This is significant and makes this a very promising material for high intensity machines. The mechanical properties of Vespel are particularly attractive in that it has a Rockwell hardness of 85-90 which does not change after $5x10^9$ rads. This material has been used as a gasket with a sealing pressure of 150#/linear in., not much greater than the elastomers. DuPont reports that after 10^{11} rads in the Brookhaven reactor, Vespel was brittle but still form stable. Varian Associates reports that an improvement by a factor of 10 in the outgassing rate of Vespel can be achieved by baking at 250°C.

The next class of materials investigated was the elastomers. Two rubber compounds developed for their radiation resistant properties were received from Precision Rubber Co. It is not known just what "antirad" additives were used. These had been tested by Precision at 10^8 rads with no significant changes in hardness or elongation properties. The outgassing rates of these rubbers PRC-446-1387 and 446-4387 are shown in Fig. 3. These rates are twenty times worse than the Viton now used in the AGS as gaskets. However the tests with radiation at Brookhaven show that a significant change in hardness occurs at $5x10^8$ rads, the values of durometer going from 69 to 82. At $5x10^9$ rads the durometer readings are over 150 and the materials are useless as a conventional gasket.

Viton was tested next and its outgassing rate is shown in Fig. 3. Again, the durometer hardness increased upon radiation to 150 level and to the extent that the material is worthless above $1x10^9$ rads.

A Urethane formulation 3000/AH-18 has been tested. The reason for this was that it seems to be the best of the elastomers for resistance to radiation damage. (See Chart Page 282 of Bolt and Carroll, "Radiation Effects on Organic Materials" Academic Press, New York 1963).

*Work done under the auspices of the U.S. Atomic Energy Commission.

The outgassing rate of this Urethane is very good, about like Viton as shown in Fig. 3. It is now being exposed to $5x10^9$ rads and further tests on outgassing and mechanical properties will be forthcoming.

The third class of materials tested were the ceramics. We were most interested in the machineable ceramics which can be used in the unfired condition. Mykroy 750 and 1100 are glass-filled mica compounds which are now all made with synthetic mica. Fig. 4 shows the outgassing rates of these two materials. Mykroy 1100 is very superior to the 750 series at 100 hours and is still improving whereas the 750 has just about flattened out. Inspection of Figs. 7 and 8 shows the significant decrease in the hydrogen and water vapor and hydrocarbon peaks in the 1100 series Mykroy. While it is true that most of the literature gives a very high radiation resistance to all ceramics, some changes were found in the mica compounds at $5x10^8$ rads. (See "Effects of Radiation on Materials and Components" Kircher and Bowman, Reinhold, New York, 1964). We are experimenting to see if any water of hydration is liberated at $5x10^9$ rads. If so, this might make this material less attractive than it now seems.

Boron nitride is the latest of our sample materials. The grade "M" has the best outgassing rate of all the materials tested so far. Fig. 4 shows a rate of $2.5x10^{-10}$ Torr ℓ/sec/cm^2 which is as good as, or better than, most metals. The mass spectrometer analysis of Fig. 9 shows a typical spectrum except for some gases up around mass 50.

Boron nitride is easily machinable, has excellent electrical properties and acceptable mechanical properties. To date the radiated samples have not been completed.

Our conclusions are that there is a satisfactory polymer, Vespel, which has excellent vacuum characteristics before radiation and that these improve after radiation. The sealing forces required are such that it would serve as a valve seat and in many other areas where a metal gasket is difficult to utilize. The elastomers such as Viton and PRC-446-1387 and 4387 are not useable where radiation above $1x10^8$ rads is to be expected. Urethane is still to be tested. There are two machinable ceramics with excellent outgassing rates. Mykroy 1100 is to be preferred over the 750 series. Boron nitride is superior to either and should find wide applications in accelerator design .

Further information will be available shortly on ferrites, high alumina porcelain, Mylar, "H"-film, nitrile rubbers and some new Vitons and other materials.

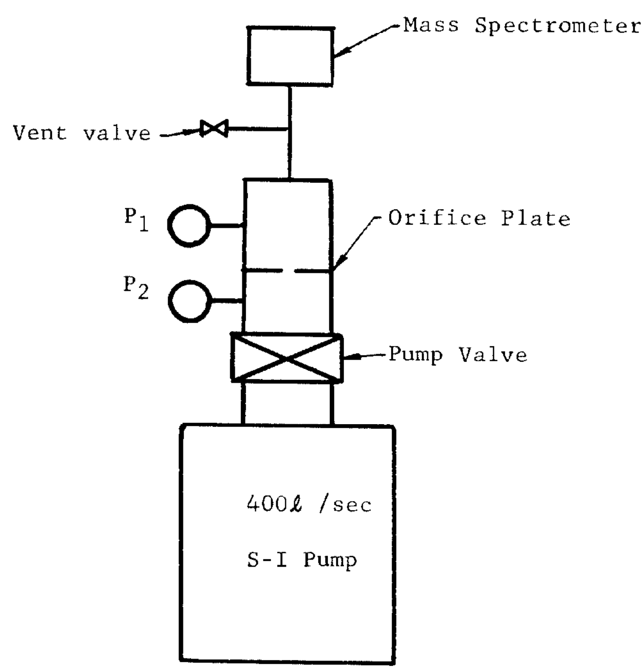

Fig. 1. Test Apparatus.

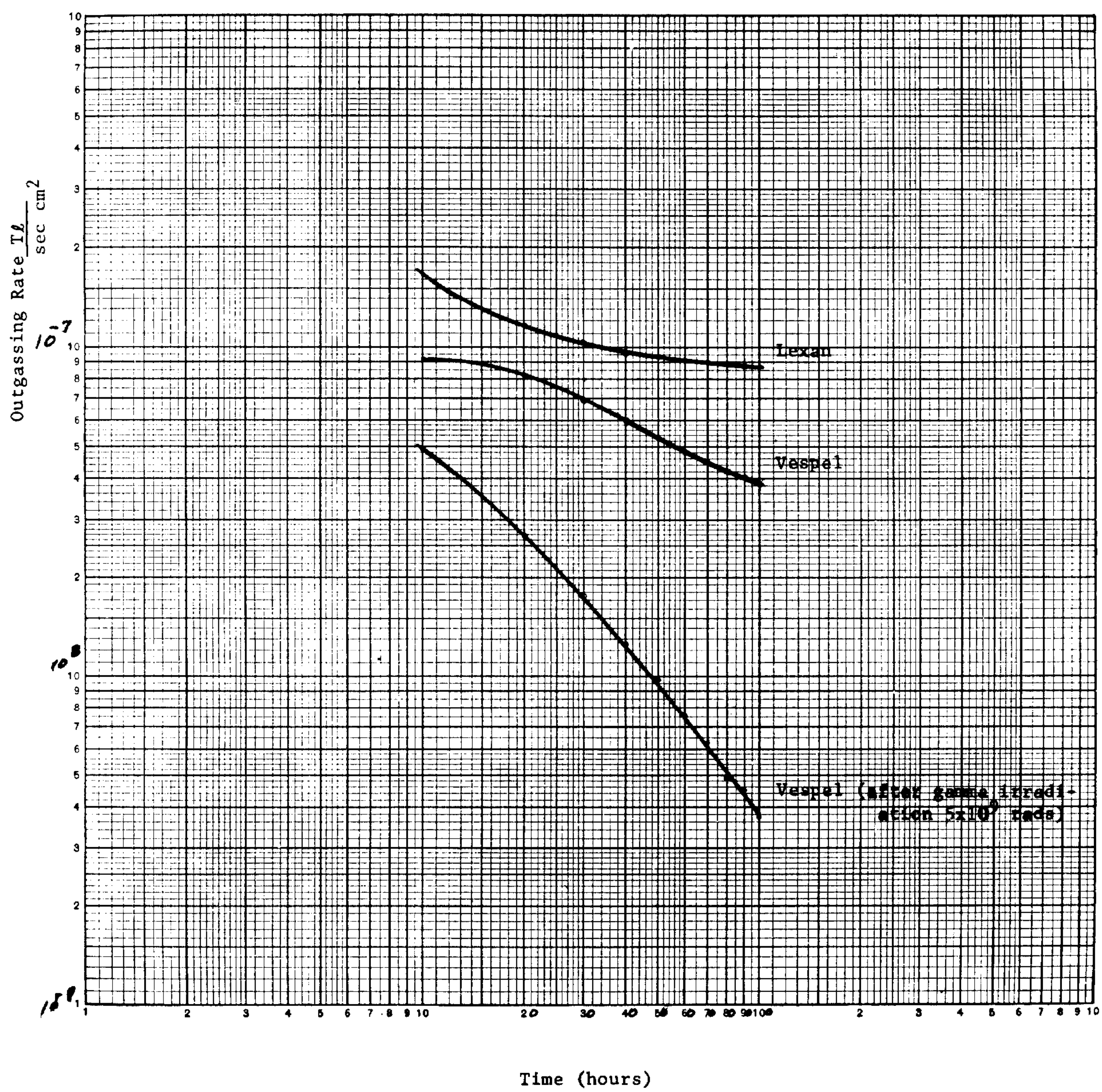

Fig. 2. Outgassing Rates of Plastics.

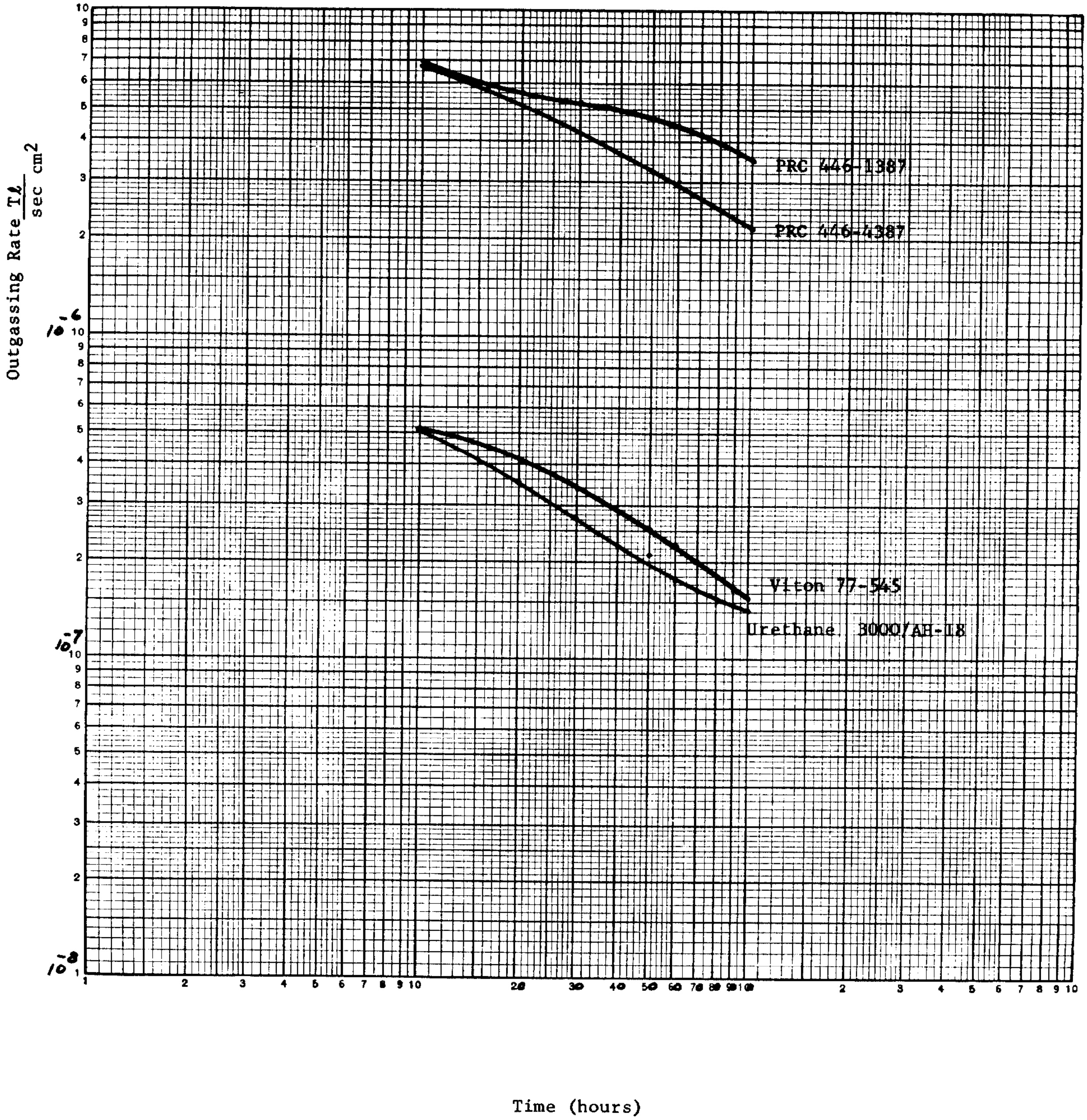

Fig. 3. Outgassing Rates of Rubbers.

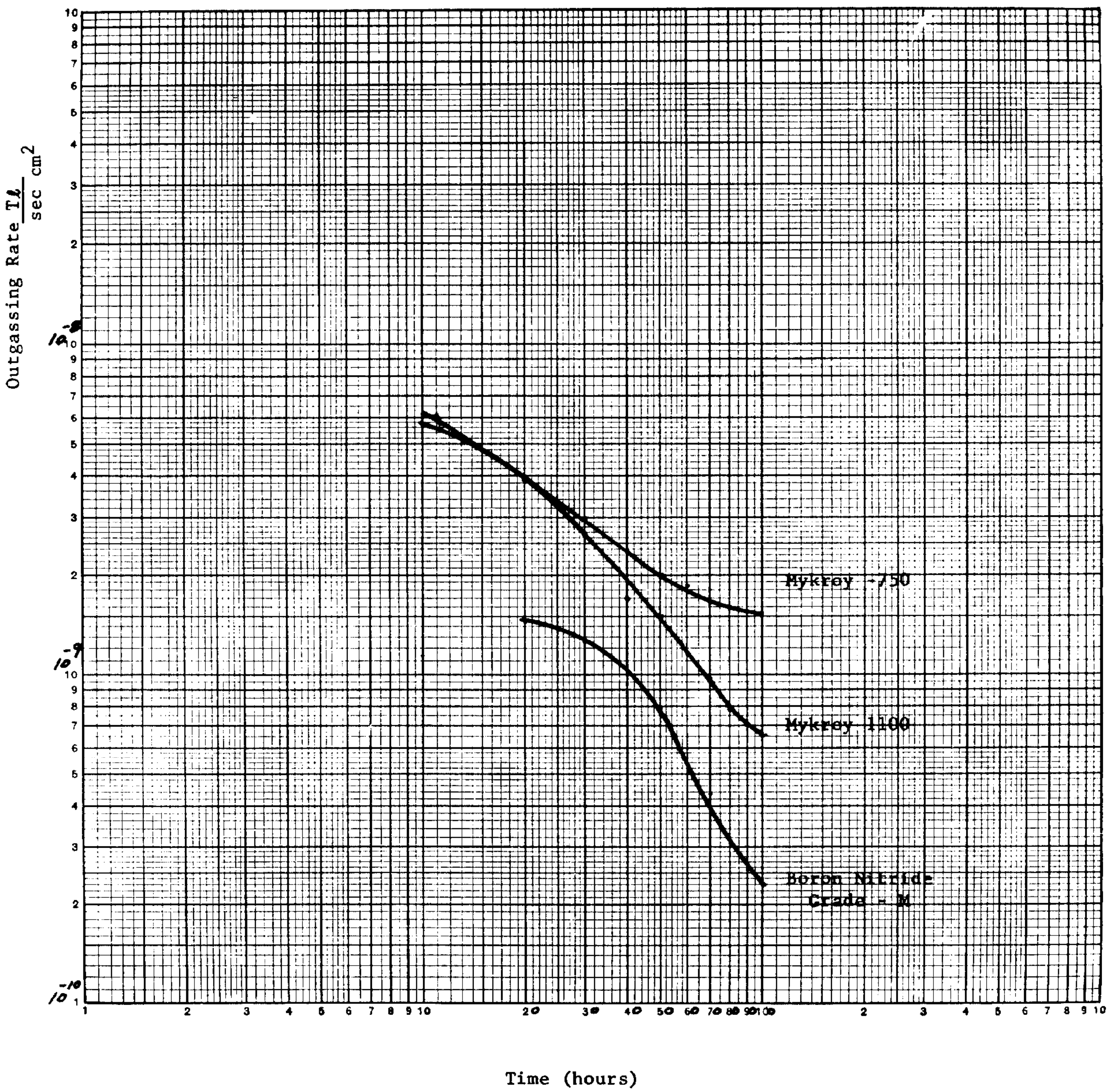

Fig. 4. Outgassing Rates of Ceramics.

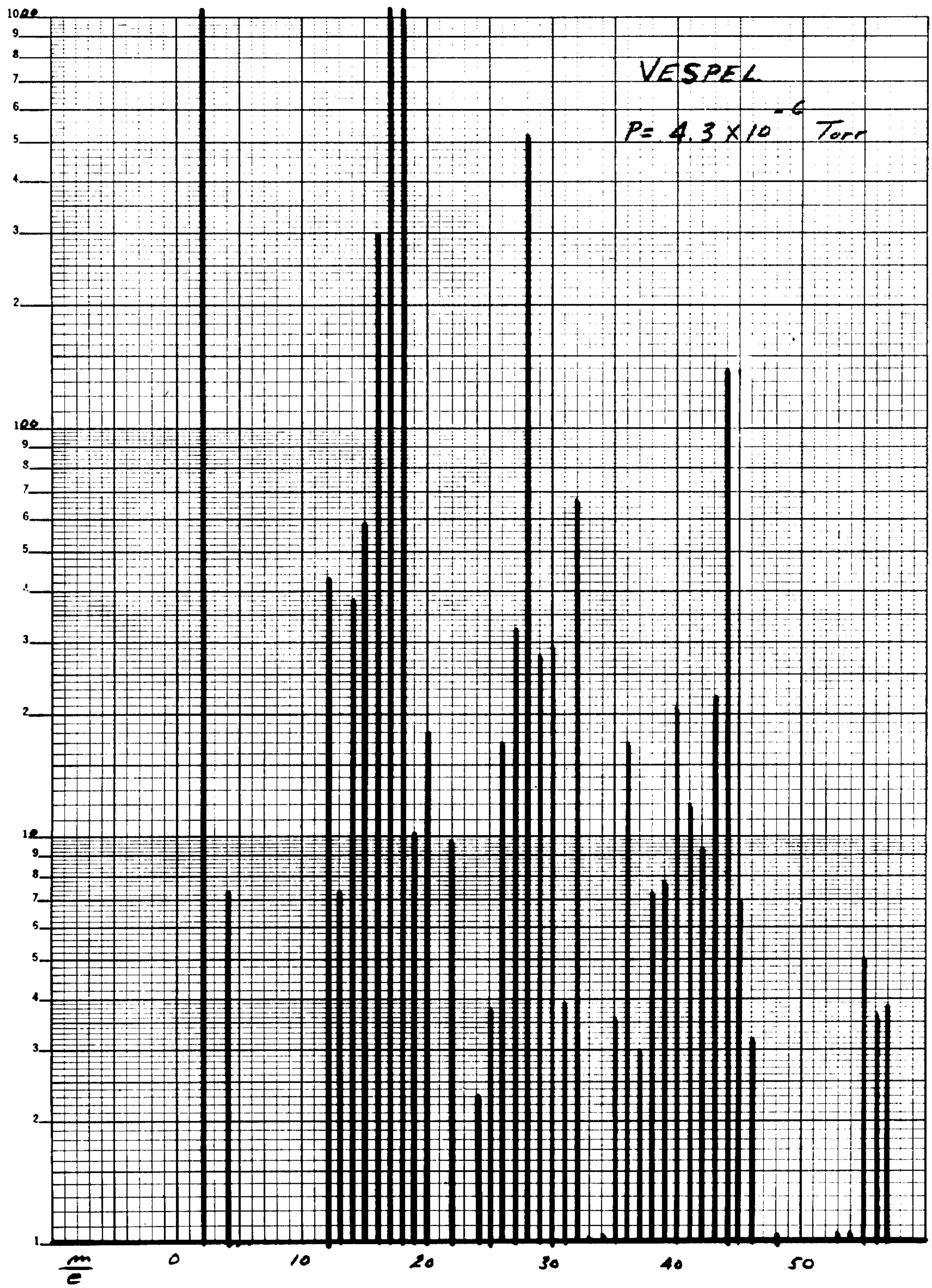

Fig. 5. Mass Spectrum of Vespel before Radiation.

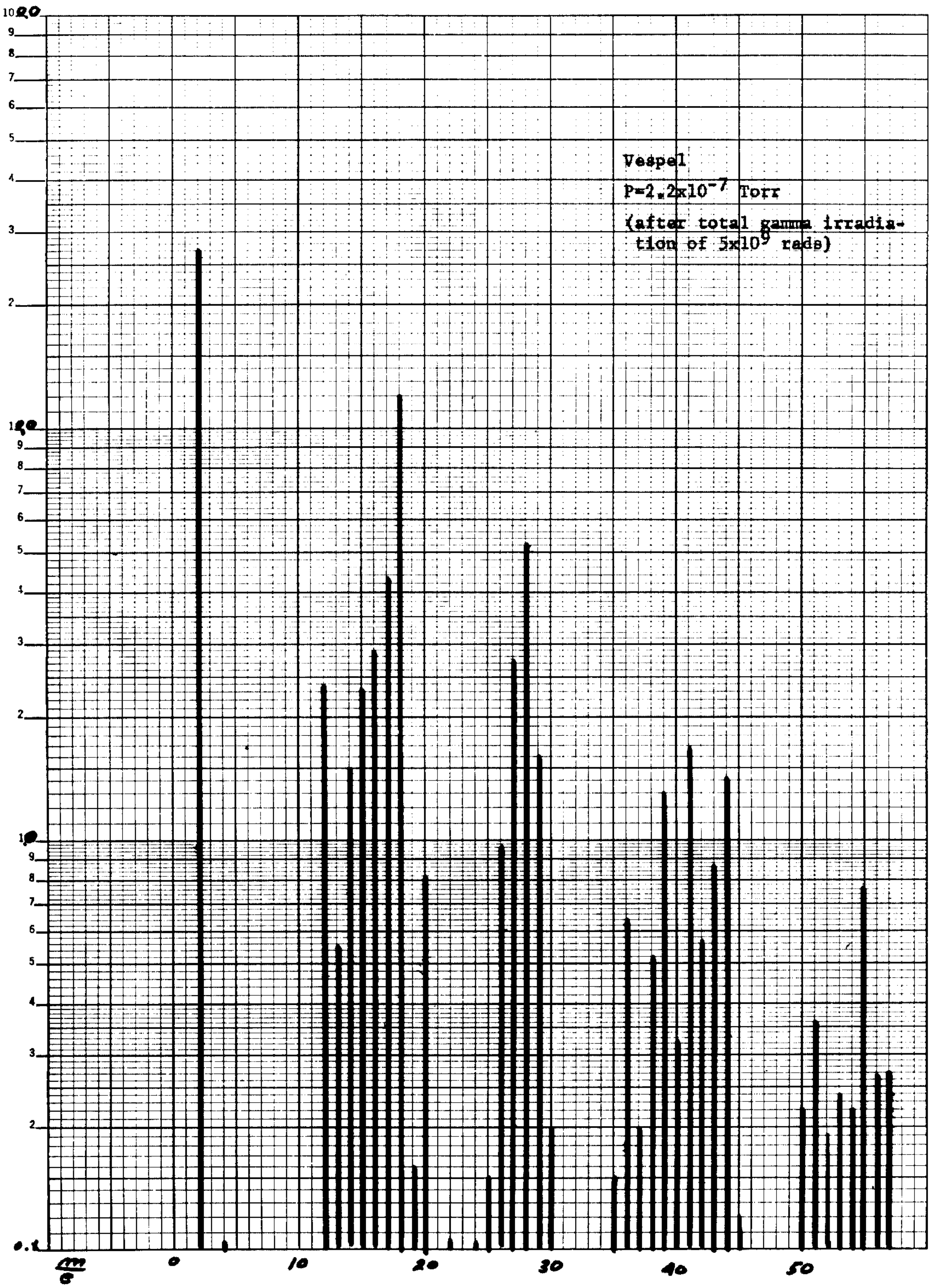

Fig. 6. Mass Spectrum of Vespel after Radiation.

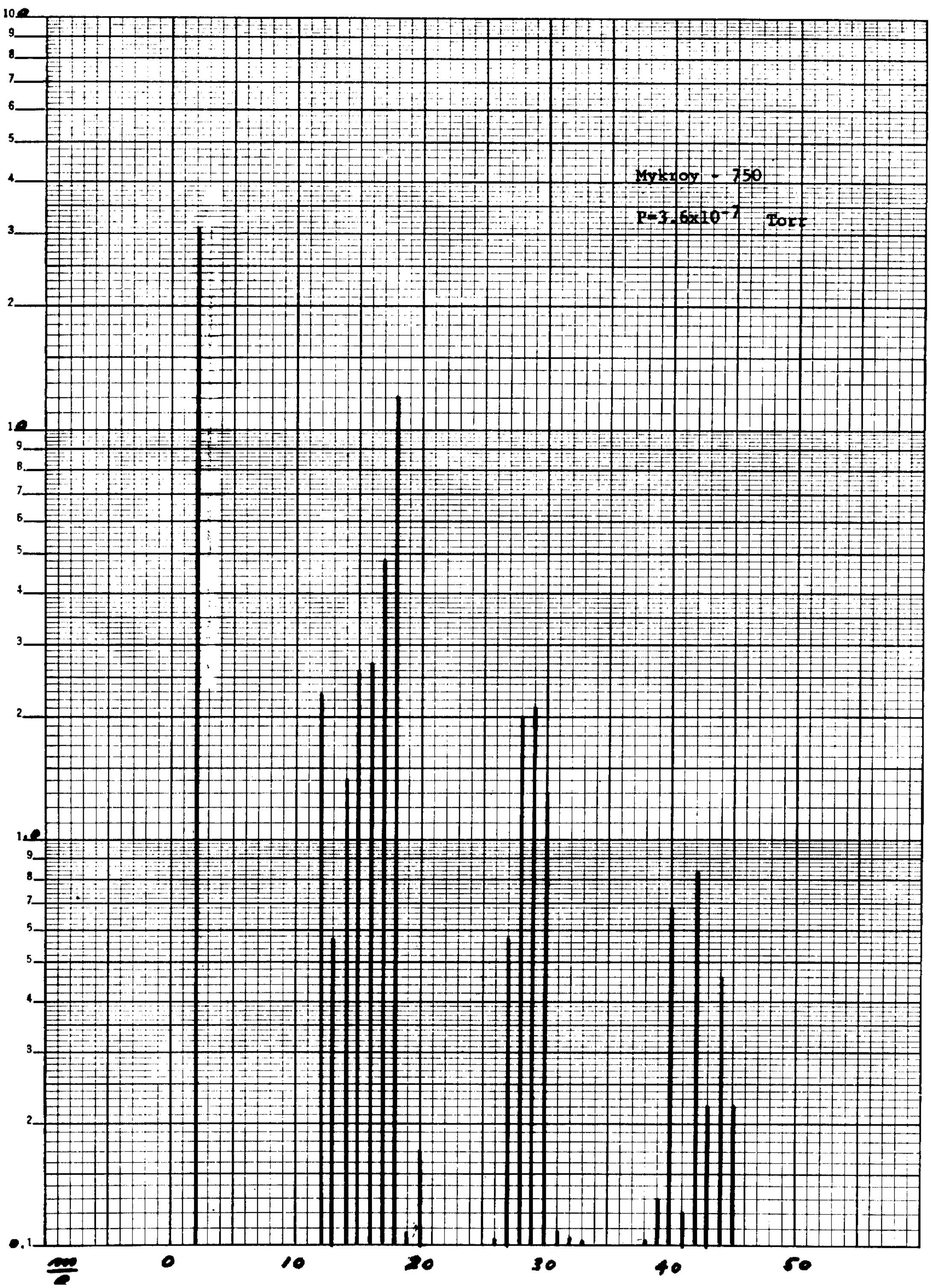

Fig. 7. Mass Spectrum of Mykroy 750.

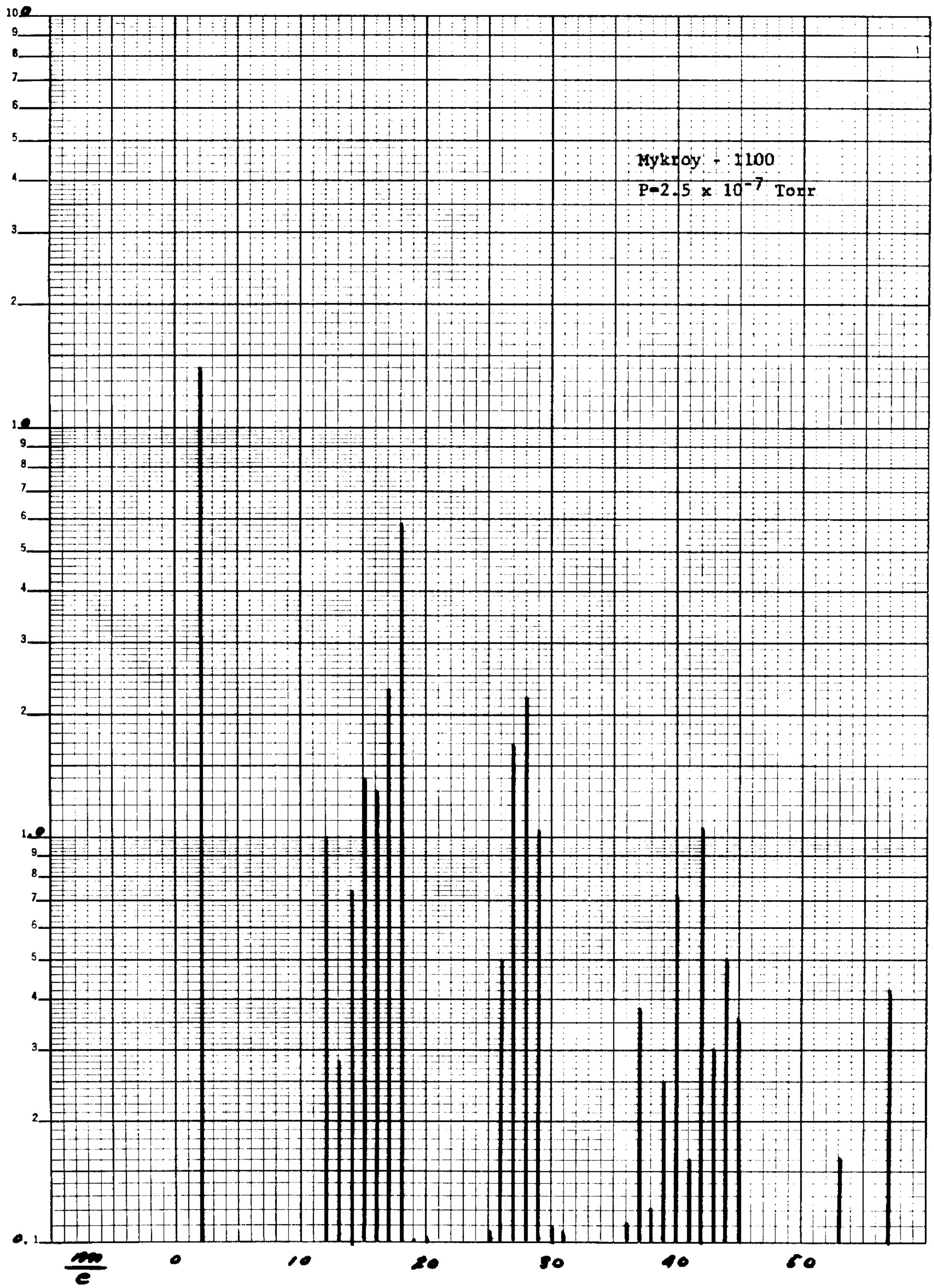

Fig. 8. Mass Spectrum of Mykroy 1100.

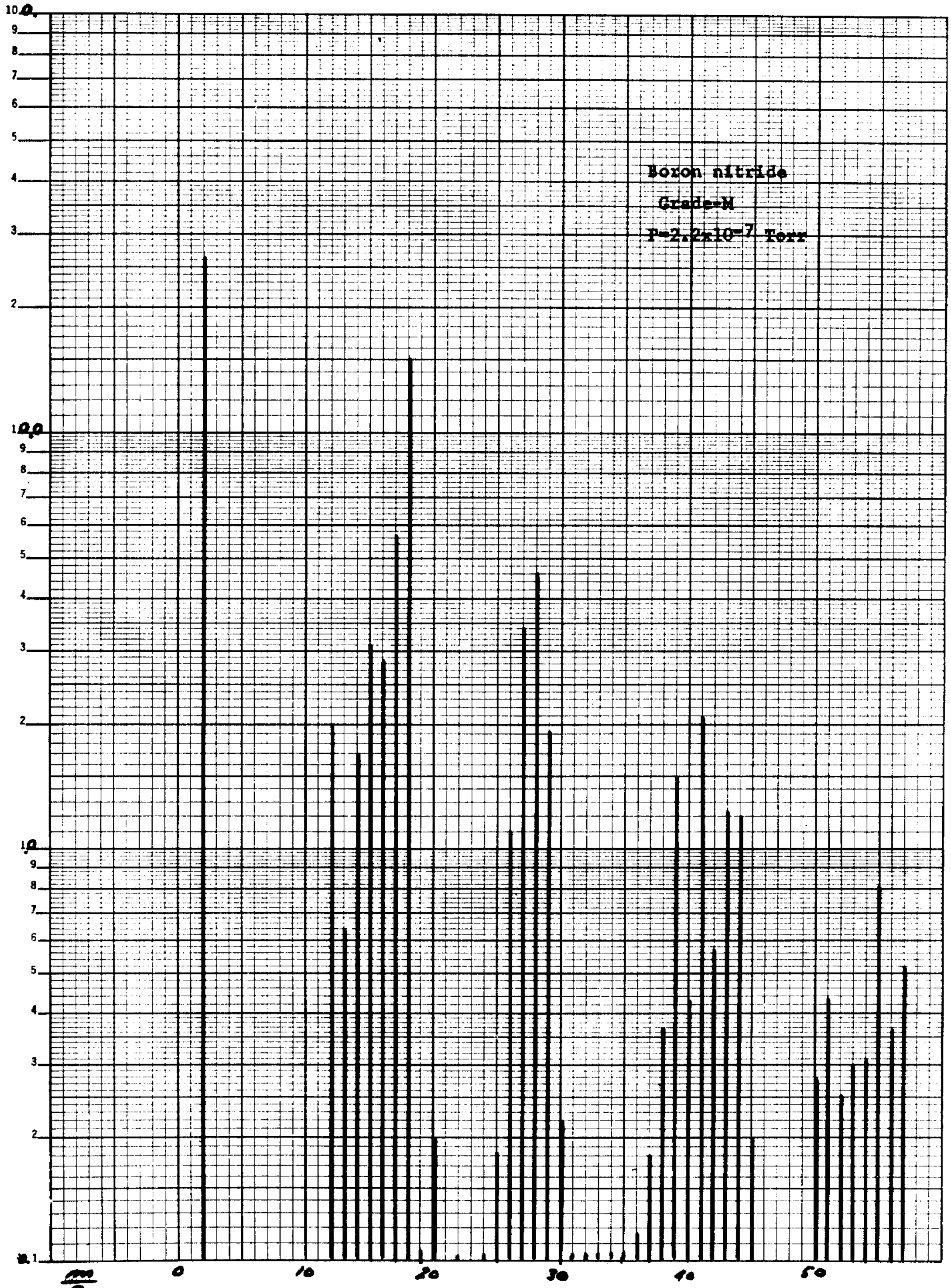

Fig. 9. Mass Spectrum of Boron Nitride.