SMALL SOURCES, BIG IMPACT: WHAT BRACHYTHERAPY SOURCES ARE REALLY MADE OF

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Abstract— **Following the discovery of radium in 1898, the scientific community set out to procure as much of the radioactive material as possible in Europe, the US, and the rest of the world. Radium, in the early 20th century, was considered the most valuable substance on earth. The mining, processing, standardization, and encapsulation of the world's first brachytherapy source is a journey through geology, chemistry, and physics.**

Keywords— **brachytherapy, brachytherapy sources, brachytherapy source construction, radium, radon**

I. Introduction

Most radiation physicists are well acquainted with the discovery of radium-226 by Marie and Pierre Curie in 1898 and the subsequent award to Marie Curie of the Nobel Prize in chemistry for her work isolating radium. What is less often shared is the complex process of mining and procuring radium and radium byproducts for medical use. Radium and its daughter product, radon-222, were the preferred radioactive materials used during the early 1900s because they were isolatable from rocks and ore containing uranium, had comparatively long half-lives, and had detectable radiation emissions. In Europe, most uranium deposits were found in pitchblende, also known as uraninite. It has a dark, heavy appearance and resembles black tar or pitch. In the US, it was primarily found in mineral carnotite, which is famous for its canary-yellow coloring. This paper will describe how radium and radon were extracted in Europe and the US and how they were used for medical purposes in the early 20th century.

II. The hunt for radium in europe

In the decade following its discovery, radium's potential as a medical and imaging device caused great excitement. Marie Curie and her colleagues desperately needed more radium to advance scientific study and medical experimentation. Uranium minerals had been mined since the mid-1500s in the Ore mountains along the modern Czech-German border, long before the formal 'discovery' of uranium in 1789. [1] Pitchblende was initially applied in the glass-making industry for the creation of yellowish-green glass, known as 'uranium glass' or 'Vaseline glass'. After the discovery of radium, large uranium mining areas, such as those in Bohemia (Austria), switched their operations to focus on radium ore extraction. [2]

A. The process of extracting radium from pitchblende

Compared to carnotite found in North America, pitchblende contains more uranium (in the form UO_2) and is not contaminated with vanadium. Rather, it contains lead and rare earth elements. The radium is not part of the surrounding rock but is embedded in the ore structure itself due to natural radioactive uranium decay. Pitchblende also contains large amounts of barium. The radium must be chemically released before it can be separated. First, the pitchblende is crushed and treated with boiling sulfuric acid. Acid dissolution removes the uranium as UO_2SO_4, leaving radium behind in the solid residue or sludge, which can then be converted to carbonates - and ultimately salts. The chemical process of extracting radium from pitchblende is similar to the extraction from carnotite (described in more detail in Section III). However, unlike radium separation which happens immediately with carnotite, radium extraction is the last stage when processing pitchblende - after the removal of the uranium, lead, and other metals. As described in her PhD thesis, it took Marie Curie 3 years to isolate just 0.1 gram of pure radium chloride from several tons of pitchblende. [3]

B. Isolation of metallic radium

After the mining and production of radium salt became routine, it was important to isolate radium to confirm its properties and place it properly on the periodic table. Isolated radium was also required to characterize the emission spectra. Marie Curie used electrolysis to isolate metallic radium from a radium chloride solution in 1910. When passing current through a radium chloride solution, the radium ions migrated to the cathode, and a radium-mercury amalgam was created. As mercury has a much lower melting point (357°C) than radium (700°C), the mercury could be distilled in a heated, hydrogen environment or a vacuum, leaving behind pure radium metal. Radium was kept in salt form for storage and stability purposes as metallic radium is too chemically reactive to handle safely in air or water.

C. Radium production in Europe and WWI

In 1911, Austrian mines of St. Joachimsthal (now Jáchymov in the Czech Republic) produced 2.65 grams of radium chloride and processed the ore in their own radium factories. [4] The New York Times stated the world's supply of radium in 1913 amounted to "not more than 6 or 7 grams" as reported by a French scientist. At that time, Marie Curie was identified as the chief holder with approximately 2.6 to 3 grams of radium. [5] Unlike the US, where radium production was commercialized by private corporations, Europe reserved its supplies through state monopoly to benefit research and medicine. Austria nationalized their mines and stopped the export of uranium ore in 1904. Radium production dropped after this time to less than one gram annually. This was unfortunate as 1 gram of radium could be obtained from 5-6 tons of pitchblende as opposed to 500-600 tons of carnotite. [2] Production decreases were due to export limitations and the start of World War I. In parallel to the escalation of hostilities in Europe, the demand for radium shifted from medicinal to practical. When mixed with zinc sulfide, radium would glow and was thus widely incorporated for military purposes. By 1918, military use accounted for 95% of the radium produced in the US. [6]

Unknown at the time and not revealed until after the completion of the war, the Shinkolobwe mine in the Belgian Congo (now known as the Democratic Republic of Congo) contained exceptionally rich ore containing up to 65% uranium oxide. European deposits averaged between 0.5 and 5% uranium, and North American ores were approximately 1%. Use of the Shinkolobwe mine caused the price of radium to drop by over 50% by the early 1920s. This ore deposit was critical in supplying uranium for the Manhattan Project several decades later.

III. The hunt for radium in the US

After Marie Curie's discovery, US mining operations also pivoted to take advantage of the new market. Carnotite that was already being mined in Colorado for heavy metal extraction was sent to France for analysis and was identified as containing radioactive uranium in addition to the heavy metal vanadium. [7] For a few years following this discovery, carnotite was mined in the US and then shipped to Europe for radium extraction, which was a very inefficient process given the quantity of ore required to produce a small amount of product. Radium mining and extraction in the US started around 1910 and was considered large-scale by 1911 with the founding of the Standard Chemical Company (SCC) by Joseph Flannery. [2] The production of radium was essentially a spin-off of Flannery's carnotite ore business named the American Vanadium Company, which he owned with his brother James. The American Vanadium Company was mining carnotite ore for use in the steel and glass industries, but the development of carnotite as a source of radium resulted in a new business venture. Flannery was motivated beyond financial reasons; the previous year his sister was diagnosed with uterine cancer. Following a trip to Europe, he learned that cancers could be treated with radium, but the supply for the medical industry was critically low. Furthermore, Europe had enacted an embargo on pitchblende exports, so Flannery realized that any radium for medical purposes in the US would have to come from US deposits. The SCC established mines in the Colorado region and transported ore by train to a processing plant in Pennsylvania.

A. The process of extracting radium from carnotite

As mentioned previously, the extraction from carnotite was similar to extraction from pitchblende, mostly due to Europeans inventing the pitchblende process but not patenting it. Carnotite is a naturally occurring potassium uranium vanadate mineral with a chemical formula of $K_2(UO_2)_2(VO_4)_2\ 3H_2O$ and contains around 50% uranium. A bright yellow radioactive material often found in sandstone, carnotite was primarily located in the high plateau region of the US including deposits in western Colorado, eastern Utah, and Wyoming. [8] The target radium, along with trace amounts of barium, exists in the host rock. Carnotite deposits at the time were thought to contain approximately 3.5 – 15 mg of radium per ton. [9] To isolate radium, one method was to dissolve the other materials and precipitate the radium in a sludge or slime. Several different methods to extract radium existed at the time and the precise chemicals used varied from plant to plant. [10] In one example, the carnotite ore was first crushed and then leached (*i.e.*, dissolved and separated by a solvent) with sulfuric or nitric acid. [11] The result was a solution of dissolved uranium, vanadium, and radium called a "pregnant liquor" or "pregnant leach solution":

$$K_2(UO_2)_2(VO_4)_2 * 3H_2O + 4H_2SO_4 \rightarrow 2UO_2^{2+} + 2VO_2^{+} + 2K^{+} + 4SO_4^{2-} + 7H_2O + RaSO_{4(S)} + BaSO_{4(S)} \quad (1)$$

The target $RaSO_4$ and $BaSO_4$ were left behind as an insoluble residue, called "slime," immediately following the acid leaching. If there were not enough naturally occurring barium, barium chloride could be added to the liquid to precipitate radium and barium sulfate. [12]

For completeness and interest, the process of uranium oxide separation will be briefly described, as it was important to extract and use these other metals from Equation (1) for financial reasons because radium extraction was inefficient and complicated. Remaining impurities were removed from the solution through separation chemistry by filtration and the addition of lime or limestone (*i.e.*, calcium carbonate) that solidifies metal byproducts which can then be removed. The remaining solution containing liquid uranium and vanadium compounds is doused with ammonia or other materials to precipitate the uranium as ammonium diuranate. Carefully controlling the pH of the solution allows separation of uranium and vanadium. The precipitate is centrifuged before

being calcined (*i.e.*, thermally treated to remove oxygen) to a stable uranium oxide commonly known as "yellowcake."

$$(NH_4)_2U_2O_7 \rightarrow U_3O_8 + NH_3 + H_2O \qquad (2)$$

A graphic of this complicated process is shown in Figure 1. The mildly radioactive yellowcake could then be packaged and shipped to a refinery. Modern use of yellowcake includes producing nuclear reactor fuel. Further processing of the original solution would eventually precipitate the vanadium for commercial use in steel and iron production.

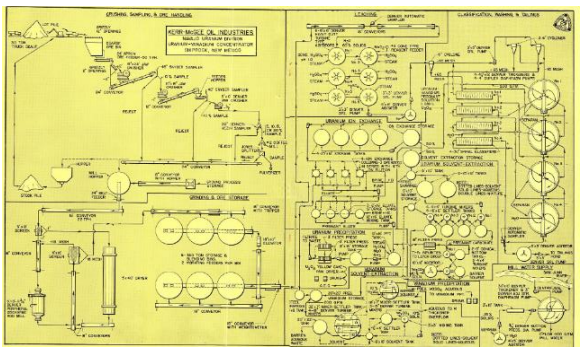

Figure 1: Flowsheet of the "Raw Leach" extraction method [13]

Continuing from the radium rich slime - it was difficult to separate the barium sulfate (99% by weight) from the radium sulfate (1%) as they are both Group 2 alkaline earth metals and react similarly. [14] The next step was turning the precipitates into soluble forms through the addition of sodium carbonate or soda ash. Once the residues were boiled with soda ash, the insoluble radium sulfate converted into soluble radium carbonate. Carbonates were desirable because they are generally more reactive to acid-base reactions than sulfates.

$$RaSO_4 + Na_2CO_3 \rightarrow RaCO_3 + Na_2SO_4 \qquad (3)$$

It was then dissolved with hydrochloric acid:

$$RaCO_3 + 2HCl \rightarrow RaCl_2 + CO_2 + H_2O \qquad (4)$$

Using a fractional crystallization technique, the solution would slowly crystalize. The radium carbonate was slightly less soluble than the barium carbonate, allowing separation of the two compounds over thousands of iterations. The radium would finally be isolated as $RaCl_2$, also known as radium salt.

B. Radium production in the US

By 1913, the shortage of radium for use as a medical product in the US and Europe was indisputable. Political discourse arose over the privatization of mining operations and the US Secretary of the Interior, Franklin Lane, got involved. James Douglas, a mining engineer and industrialist, and Dr. Howard Kelly joined together to incorporate the National Radium Institute (NRI), which was a private institute that partnered closely with the US Bureau of Mines (with Lane as the cabinet official overseeing it). Kelly, a physician at Johns Hopkins University, wanted to use radium products for the treatment of gynecological diseases. Douglas had his own, personal interests in procuring radium – his daughter was treated for cancer in England during 1911. Douglas made many financial donations to Memorial Hospital (later known as Memorial Sloan Kettering) with the stipulation that the center should focus on treating cancer with radiation. In conjunction with the Bureau of Mines, the NRI leased land in Colorado and built their main production plant in Denver. None of the radium produced through this plant would be available for shipping and selling abroad. Lane continued his attempts to develop a radium supply from both public and private lands, which affected the success of the SCC. Lane publicized their extraction methods through Bureau of Mines publications (conveniently used in this document) rather than keeping them a secret.

The NRI/Bureau of Mines method utilized a similar procedure to that described above in their extraction operations. In terms of efficiency, a report from the US Department of the Interior described a total US production of 28.8 tons of uranium oxide containing approximately 9.77 grams of radium chloride in 1912. [4] The 1910 market price of radium was $120,000 per gram. [8]

Figure 2: Carnotite ready for shipment to the SCC [2]

By late 1915, Lane announced that 5 grams of radium had been extracted in Denver. Half went to Kelly's clinic in Baltimore and the Memorial Hospital in New York. After sustaining political pressure from Flannery and the SCC, the NRI plant closed in 1917 after producing a total of 8.5 grams of radium and processing 1500 tons of uranium ore. Also in 1917, the United States Radium Corporation was founded in New Jersey to process carnotite ore and later became infamous for the radiation sickness of dial workers. [15] By 1920, 90% of the world's radium supply came from the US, which was remarkable given that far superior radium yield from pitchblende. [2] In 1921, Marie Curie visited the US and received a gift of one gram of radium (mined by the

Flannerys), presented to her by President Harding. By 1921 SCC had produced half of the world's radium. [16] Following the breakup of their monopoly and the publicization of the high-grade deposits in Europe, the SCC ceased radium production in 1922 and focused instead on uranium reclamation.

IV. DEVELOPMENT OF CALIBRATION STANDARDS

It soon became clear that there was a compelling need to establish a radiation standard; for example, one report noted that no other material on the open market offered a better chance for fraud due to the inability of the average chemist to tell whether the radium salt was pure or not. [4] In 1912, the price of radium salt was $70 per mg, corresponding to $2,000,000 per ounce (approximately $70 million dollars in 2026 US dollars). The fraud went beyond financial repercussions as physicians noted treatment outcomes being influenced by misrepresented quantities of radium as well. [17] Initial methods of standardization involved comparing darkening of photographic plates. Another method was to measure the radioactivity via electroscope and ensure that the radium was 2,000,000 times as radioactive as a similar weight of uranium oxide. The Curie method involved measuring the ionization of air. [18] Using a Quartz-Piezo electrometer, Curie could measure currents induced as small as 10^{-12} amps detecting radium in ore in the one part per million range.

In 1910, the International Radium Standards Committee was formed in Brussels to establish more robust standards for radium. Key members included Marie Curie, Ernest Rutherford, Otto Hahn, and American radiochemist Bertram Boltwood. The first order of business was to establish a unit for radioactivity. All agreed on the use of the "Curie" in memory of Pierre Curie that was equivalent to the quantity of ^{222}Rn in equilibrium with one gram of its parent ^{226}Ra. [19] Of note, the elemental name of radon was not accepted until 1923, and it was known mostly as an "emanation" or "radium exhalation" during this time period.

The second goal was to establish an international standard for radium that could be used across the globe. This was highly important for medical uses and intercomparison of treatment methodologies. The physical "standard" as prepared in Paris by Marie Curie was 21.99 mg of radium chloride sealed in a glass tube and stored at the International Bureau of Weights and Measures (BIPM) in France. [20] In 1912, the original standard was compared to three similarly constructed devices manufactured in Vienna by evaluating the gamma-ray emission rates and thus created several secondary standards. In 1913, the US National Bureau of Standards (NIST) received a secondary standard certified to contain 20.28 mg RaCl, equivalent to 15.40 mg of Ra as shown in Fig 3.

COMMISSION INTERNATIONALE DES ÉTALONS DE RADIUM.

CERTIFICAT.

Figure 3: Certification of Curie standard received by NIST [19]

Following the calibration standardization of sources came early attempts at treatment standardization, at least within a particular institution. The Radium Institute of London attempted to standardize treatments with their radium applicators. A "full strength" applicator represented 0.5 cg of radium bromide covering 1 cm^2 of surface area, "half strength" was 0.25 cg of radium, and "quarter strength" was 0.125 cg. These were eventually modified to match the colloquial naming convention of full being =1 cg, half = 0.5 cg, and quarter =0.25 cg. [21] Other less rigorous methods involved prescribing a qualitative skin erythema dose or watching for moist desquamation and necrosis. The Roentgen, or R, was not defined until 1928.

V. ENCAPSULATION AND APPLICATION

As soon as it was discovered, radium was used to treat various maladies. At the time, it was understood that radium possessed α, β, and γ emissions and each had different penetrating power. In 1901, Parisian Danlos and colleagues started applying radium sulfates (gifted by Pierre Curie) directly to skin lesions. However, to prevent radium salt loss, the material had to be stored in some sort of container. He used small glass tubes containing radium salt or radium sulfites covered in rubber sheathing. [22] Alexander Graham Bell suggested in 1903 to seal the radium in glass tubing and implant within a tumor. [23] Radium could also be mixed with a varnish and painted over a surface for topical application.

In Figure 4, radium was enclosed in a brass case with a 0.01 in. thickness of aluminum for all radiation emanations to pass through, and a 0.1 in. lead backing, to act as a "screen".

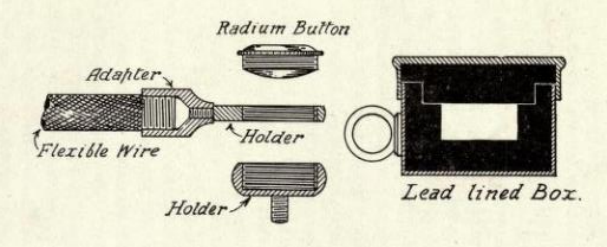

Figure 4: Example of an early radium applicator [24]

The outside of the radium button had a screw threading where it could be placed on a holding device and inserted into small holes of the body, such as the nostril or larynx. The holder was for use in external applications, carrying the radium at the end of a brass rod with a lead shield to protect the hand of the treating physician.

Other methods of "screening" or filtration were noted in skin applications where a variety of materials were used, including shoe leather and asbestos. Applying such materials to absorb the alpha and lower energy particles was associated with better cosmetic results. [25]

The radium salts such as those shown in Figure 5 were often put into platinum tubes with a wall thickness of 0.5 mm. Attempts were made for a 0.3 mm thickness, but it was challenging to embed a screw thread at such a small thickness. It was known that the use of this thickness of platinum would absorb 75% of the β emissions and 4% of the γ rays. The tube was then filled with the powder, and a screw plug was gold-soldered into position. Care had to be taken to "tin" the thread with gold before screwing in the plug or the emanation could escape. [26] The efficiency of the seal could be tested by leaving the source in a box for a few days and then testing to see if the box itself became contaminated. Radium tubes were most often used in external applications (surface/skin), intracavitary (nose, vagina), or implanted surgically. The use of radium tubes and needles were short lived for practical reasons, as will be described below.

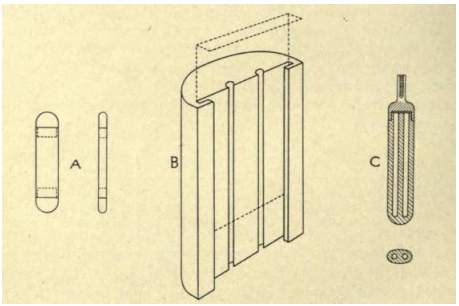

Figure 5: A) flat applicator for use with radium salt in the dashed lines. B) silver applicator bored out for use with two tubes of radium salts and ten platinum shutters that could be slid down. C) oval section filter, also for use with two tubes. [26]

VI. FROM RADIUM TO RADON

Radon, in its unnamed state, was discovered almost in conjunction with radium as scientists knew that "something" was being emitted. Its discovery is attributed to both German scientist Friedrich Dorn and the team of Rutherford and Owens at McGill university. [27] It was further understood in London during 1903-1904 that radium was "giving off" helium (See Fig. 6). [24]

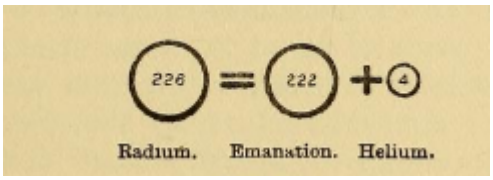

Figure 6: An emanation and an α particle [28]

Radon was finally isolated by Ramsay and Whytlaw-Gray in 1911. They also subsequently determined the density and atomic weight of the gas. Radon was given its official name in 1923.

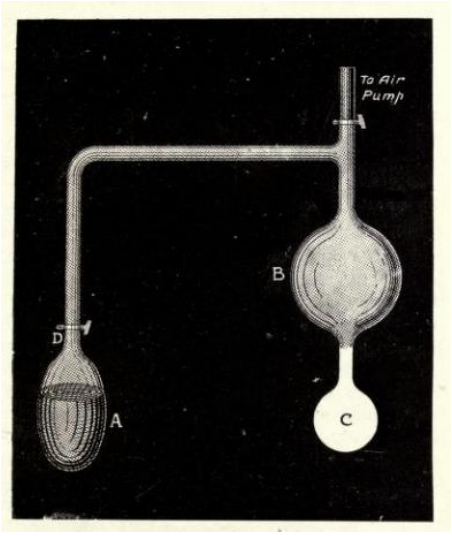

Figure 7: Method for radon gas isolation [24]

Curie's method to isolate radon gas via condenser is shown in Figure 7. The source (bulb A) contained radium bromide in solution that undergoes α decay, constantly producing radon gas. Air was removed from bulbs B and C, which were coated with zinc sulfide. While the stop cock (D) was closed, a vacuum was created. When the stop cock was opened, the particles struck the coating and acted as a scintillator. By immersing bulb C in liquid air (~200°C), the radon gas would condense (radon's boiling point is -61.7°C). The rest of the gases were then pumped away, leaving isolated pure radon. This method was nicknamed the radium "cow" because it could be milked every few days.

Radon was instantly used in a therapeutic manner, as being a gas, it could be sealed in capillary glass tubes that were

smaller than radium tubes (Fig 8). One Ci of radon occupies less than 1 mm^3 in volume at standard temperature and pressure, thus a radon needle could be much thinner than a radium one. [29] Additionally, once radium was procured, the amount of radon that could be acquired and used was virtually endless. The glass tubes were 3-4 mm in length, 0.3-0.4 mm in diameter, and the glass thickness was on the order of 0.1 mm. With a higher specific activity than radium, radon emanations were able to treat malignancies faster. It was found that these tubes could also be permanently implanted in tumors and left to decay. [30] Interestingly, these sources were assumed to be point sources and the inverse square law was applied to calculate radiation intensity.

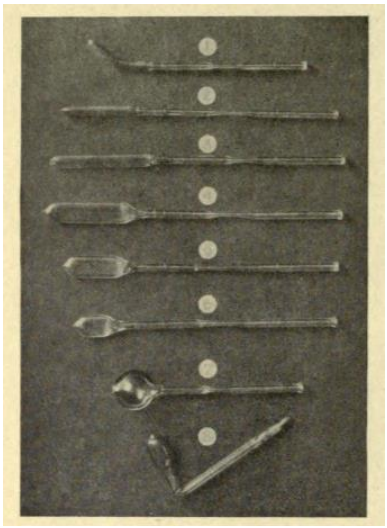

Figure 8: Small glass and platinum tubes used for radon treatment. [26]

The shift away from radium tubes and needles continued as they were prone to leaking; micro-leaks allowed radon gas to escape, which would leave radioactive dust everywhere (even in this early period, it was identified as a health hazard). It was also due in part to the need to off-gas the helium build-up inside tubes and needles. Radium tubes exploding due to internal pressures up to 20 atmospheres were not only a financial loss to the institution of the radium source, but also a radiation safety incident (though little was known about all of the related risks at the time). [31] Marie Curie was aware of the build-up of helium in 1912 when developing the radium standard, and she recommended source venting every 15 years. [20]

In 1915, the first radon plant in the US was built at Massachusetts General Hospital (MGH) in Boston under Dr. William Duane. He had studied with Marie Curie in Paris and became interested in radium byproducts, namely radium milk or radon gas. Memorial Hospital soon followed and began using radon-filled needles for the treatment of prostate cancer as early as 1915, though they didn't have their own radon plant at the time. In 1917, Duane helped establish the Memorial radon plant after a radium gift from James Douglas and the NRI. [32] The first director of medical physics at Memorial, Dr. Failla, trained with Marie Curie and developed his own radon collection system. He hired the soon-to-be famous Edith Quimby in 1919, who would be instrumental in establishing systems of dose calculations. [33]

VII. INITIAL DOSING SYSTEMS AND FILTRATION

Initially, the methodology for a radium dosing system was extremely rudimentary:

$$\text{Dose (mg} - \text{hr)} = Q(mg) * T(hr) \tag{5}$$

Where Q was the amount of radium bromide in milligrams (not "radium element") and T was the exposure length in hours. [34] Of course, this did not consider the effects of filtration or of the encapsulation of the source. It also did not specify the type of application or malignancy to which the radium would be applied.

Failla's team modified the above equation for use with radon glass tubes, incorporating the absorption by tissue and decay of the radon in the tubes. Equation 5 was thus modified to:

$$\mathrm{Q}_x = kI_x * T * R_{av} \tag{6}$$

Where Q_x was the quantity of radiation absorbed by an elemental volume of tissue at distance x from the center of the source; I_x was the radiation intensity at distance x; T was the duration in hours; and R_{av} was the average value of radon in mCi during time T in hr. The factor k was a constant associated with scattering properties of the source and medium. [30] With these radon seeds, the critical time-activity relationship was defined in mCi-hr.

While glass tubes were cheap and easy to work with, they resulted in overdoses and necrotic tissue. Failla discovered that replacing the glass with 0.3 mm gold tubing filtered out most of the beta rays (that were unneeded therapeutically) and thus gold seeds began to replace glass ones. They were also more durable than glass. Failla's team was instrumental in laying the foundation for modern brachytherapy dosimetry. In his landmark paper "Dosage in radium therapy" he proposed that radiation doses should be expressed in terms of energy absorption and not in mg-hr. [35] In this 1921 paper, he also proposed specifying conditions of the treatment, which look amazingly similar to the components of the modern day written directive. He wrote that the following must be carefully specified:

1. The strength of the radioactive source
2. Its distribution
3. The total filtration used
4. The duration of the irradiation
5. The relative positions and distances of the source of radiation, pathological tissue, and normal tissue.

Platinum was often used for needles as it was inert and performed well as a filter. However, its softness led to the need to add an iridium alloy for rigidity. Silver was more cost-effective than platinum and used at 0.5 mm thicknesses with rubber casings to absorb secondary x-rays. Quimby realized in 1922 that adding layers of filtration modified the delivered dose to the oblique radiation passing through it. She published the first clinical along and away tables for tubes, circles, squares, and rectangles. [36] In this same time frame, the Sievert integral was defined to describe the distribution of radiation accounting for filtration. [37]

VIII. CONCLUSIONS

Early brachytherapy sources emerged from a demanding pipeline of mining, chemical separation, transportation, isolation, and encapsulation. Almost immediately, radium and radon were used in therapeutic methods. Treatments had a steep learning curve with no standardization of activity or dose. These driving factors led to initial dosimetry concepts that resulted in modern practices performed today.

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