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Carnotite – the discovery & naming of the uranium ore on which the American radium industry was based

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It is usually assumed, for example by the Muséum National d'Histoire Naturelle, Galerie de Minèralogie et de Géologie, Jardin des Plantes in Paris [1], that the French mineralogists Charles Friedel & Eduard Cumenge discovered the uranium ore carnotite in the USA and that in their 1899 paper [2] in Comptes rendus de l'Académie des sciences they were responsible for naming this mineral after Adolphe Carnot. To date this 1899 paper has never been translated into English and we considered that this was an important omission because carnotite formed the basis of the commercial production in the USA of radium sources until pitchblende was discovered in the Belgian Congo and exploited by the Union Minière du Haut Katanga in the early 1920s. The Katangan pitchblende was far richer in uranium than carnotite and hence ended the USA's role as the principal supplier of radium. As with reconstructing other 100-year old events in the history of radium, such as who & when was the first to have the idea of using radium to treat skin conditions, to use of radium sources in clinical practice, the first use of the principle of afterloading, etc., [3] this did not prove to be simple. The translation indicated that Friedel & Cumenge were not on a geological expedition in USA but were only sent ore samples to analyse in Paris by a French chemist living in Denver: Charles Poulot. Another unexpected piece of information was that the Curies were involved in the analysis. This paper unravels these events.

Key words: carnotite, radium, Charles Poulot, Charles Friedel, Eduard Cumenge, Pierre & Marie Curie, American radium industry

Introduction

In the 1996 publications [1] to mark Henri Becquerel's discovery of radioactivity, the Muséum National d'Histoire Naturelle, Galerie de Minèralogie et de Géologie, Jardin des Plantes, (where Henri Becquerel was Professor of Physics as Applied to the Natural Sciences), it was stated that 'Carnotite has been described by Friedel & Cumenge in 1899, from samples brought back by Cumenge from Montrose county in Colorado. It has been dedicated to the French chemist Adolphe-Marie Carnot. In the 1920s the important sedimentary layers of uranium in Colorado were the main worldwide producers of radium. Their ores were in great part made of carnotite'. Carnotite has the chemical formula $K_2(UO_2)(VO_4)_2.3H_20$ [3] and is canary yellow in colour (**Figure 1**).

The discovery of the uranium ore carnotite marked the commercial start of the American radium industry which became the principal supplier of radium sources until the uranium-richer pitchblende ore deposits in the Belgian Congo were discovered before World War I although they were not exploited by the Union

Figure 1. Carnotite sample. Photograph courtesy of Hermann Eisenbeiss from a specimen provided by Walter Schumann: see *Collins Photo Guide to Rocks, Minerals & Gemstones*. English translation by R. Bradshaw & KAG Mills. Harper Collins, 1992.

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Minière du Haut Katanga (UMHK) until the 1920s. This effectively dated from 1922 when the first gram of radium was produced by the UMHK at their Oolen factory in Belgium. The Standard Chemical Company of Pittsburgh, with its subsidiary the Radium Chemical Company, was the major radium company in the USA. It was dissolved in December 1933. The cost of radium peaked in 1912-1914 at US$180,000 per gram but by 1933-1937 this had fallen to less than US$20,000 per gram in 1938 because of competition between Canadian and Belgian radium refining companies.

Friedel & Cumenge, 1899, *Comptes rendus de l'Académie des sciences*

The English translation of the paper [1] by Friedel & Cumenge now follows and for which we are most grateful to Mr Michael Barnes.

MINEROLOGY. – On a new uranium ore, carnotite; by MM. C. Friedel and E. Cumenge

One of us has received from America several samples of a natural, uranium-bearing substance designated as uraconise. It appears as a powder or in loosely-agglomerated yellow masses which can be easily broken up with the fingers. It was found in Montrose County, Colorado by Mr. Charles Poulot, a French chemist who is currently living in Denver, Colorado. The mineral can be found in cavities, or in the sorts of pools to be found on the surface of sandstone, and is accompanied by chessylite and malachite. About 10 tons have been extracted from this particular deposit.

First examination shows that, in some of the samples at least, the mineral consists mostly of silica in the form of quartz sand. The sand is intimately mixed with a yellow pulverized material. It is crystalline, since examination at high magnification confirms its action on polarized light, without, however, being able to determine any crystalline form. The material is fine enough to stain the fingers badly. The silica is easily separated because the yellow material is soluble in nitric acid, and in dilute hydrochloric acid, making a yellow solution with the former and, with the second, a solution of a lovely green colour. The silica, once it has been separated by filtration and washing, is white after burning.

The mineral contains a certain proportion of water, but the amount cannot be determined by burning to red heat, because it becomes very difficult to attack with acids after burning it, and the silica remains the brown colour that the mineral sample acquires when it becomes red hot.

After the material was dissolved in nitric acid it was found to contain, in addition to a significant proportion of uranium, vanadium acid, potassium, and small and variable proportions of iron and aluminium. Assays on about 450 g of the material also detected the presence of traces of copper, lead, barium and, as will be shown later, the radioactive metals of M. and Mme. P. Curie which, as is known, are often found to accompany uranium ores.

The most practical method of analysing the new mineral is to dissolve it in dilute nitric acid so as to separate out the silicate sand, and then to evaporate the nitric solution in a double boiler, with the addition of further nitric acid as required. Under these conditions, the vanadium separates out as a red precipitate with very low solubility in water, especially when it contains ammonium nitrate. To be on the safe side, it is best to evaporate a second and even a third time. In addition to uranium nitrate, the liquid filtrate contains potassium nitrate, which does not decompose at the temperatures reached. The iron and aluminium are carried with the vanadium and may be separated from it by filtering with ammonia. This can be done easily by using a funnel with a tap, or simply with a rubber end-piece closed with a clamp. The iron and aluminium stay in the filter. Vanadium acid is obtained by evaporating and heating the ammoniac liquid.

The uranium is precipitated from the aqueous solution by ammonia at boiling point. The potassium is obtained by evaporating the filtrate, and heating with an excess of sulphuric acid.

After evaporating the uranium solution to dryness, it is also possible to reconstitute it with 95° alcohol, which easily dissolves the uranium nitrate and leaves the potassium nitrate. However, the amount extracted is much smaller than using the previous method. These results suggest the formula $2U_2O_3.V_2O_5.K_2O.3H_2O$.

U_2O_3	63.54	U_2O_3	64.70	62.46
V_2O_5	20.12	V_2O_5	20.31	19.95
K_2O	10.37	K_2O	10.97	11.15
H_2O	5.95	Fe_2O_3	0.96	0.65
	99.98	H_2O	5.19	»

The proportions of silica are highly variable, from 60%, which is the most common value, to 7.2 and even 2.6 in extremely pure samples.

The iron is also quite variable and is easily distinguishable in the yellow mass as clear iron-coloured veins that stand out in particular areas.

There remains a little uncertainty over the proportion of water. The process of analysis was quite simple, being air-dried, and some hygroscopic water may have remained.

Regarding the radioactive metals, M. and Mme. P Curie have helpfully been able to examine the material, as it stood, using their methods, with the following results.

Radioactivity	of the mineral (containing 54% silica)	1.25
"	of the pure mineral	2.6
"	of insoluble sulphates (Ba and radium)	35
"	of untreated sulphides	11
"	of sulphides of Bi and of polonium	50 to 60

For the last value, the activity was so weak that it was not possible to obtain a good clear result. We thank M. and Mme Curie for being so good as to determine these values. The substance we have described here constitutes a new kind of mineral. We propose to dedicate it to M. Adolphe Carnot, Member of the Institute, and Inspector-General of Mines, whose work on mineral analysis is well known. In his "Methodes nouvelles d'analyse" (p 55), he suggested the quantification of vanadium by precipitation using uranium, giving a composition that seems to be related to the new species "carnotite".

Charles Friedel

Charles Friedel (1832-1899) was a French chemist and mineralogist who was educated at the University of Strasbourg and at the Sorbonne. He was then appointed to the Sorbonne by Charles-Adolphe Wurtz as an organic chemist and later on the death of Wurtz in **1884**, became Professor of Mineralogy. He is best known for the development in **1877** with James Crafts of the Friedel-Crafts reactions [4]. Friedel has also been described as 'Pierre Curie's staunch friend and supporter', that one of his students was Charles Poulot who graduated from the Paris School of Mines, and that Pierre's brother Jacques Curie (1856-1941) was once Friedel's laboratory assistant [5].

Eduard Cumenge

Eduard Cumenge (1828-1902) was the chief of several Sorbonne-sponsored mining expeditions in the late 1880s and amassed a collection of mineral samples for the Sorbonne, some of which were advertised for sale on the internet in 2009. Cumenge in **1898** published a textbook on the mineralogy and geology of gold deposits [6].

Pierre & Marie Curie

That the Curies knew Charles Friedel is recorded above [5] and also that they examined the carnotite mineral sent by Charles Poulot from the USA [1]. Some of Marie Curie's results on the radioactivity of minerals is given in **Table I** and is taken from Thomson's **1903** textbook *Conduction of Electricity through Gases* [7].

Table I. Radioactivity of various minerals of the same bulk, measured by the saturation current i ampères. For the same bulk of metallic uranium i x 10^{11} ampères = 2.3.

Mineral	i x 10^{11} ampères
Pitchblende from Johanngeorgenstadt	8.3
Pitchblende from Joachimsthal	7.0
Pitchblende from Priban	6.5
Carnotite	6.2
Chalcolite	5.2
Autinite	2.7
Orangeite	2.0
Pitchblende from Cornwall, UK	1.6

It cannot be determined if the carnotite sample referred to in **Table I** is the Friedel & Cumenge specimen because the source is not recorded and in any event the Marie Curie collection of minerals housed several samples of carnotite. Nevertheless **Table I** indicates that the two highest grade uranium ores are pitchblende and carnotite.

In practical refining terms, Stephen Lockwood (1874-1971) described in **1904** the process involving carnotite ore as 'much the same as used by Marie Curie but on a much larger scale. To produce 1 gram of radium from 500-600 tons of carnotite required 10,000 tons of distilled water, 1000 tons of coal and 500 tons of chemicals' [8]. A decade later, in **1913**, the Standard Chemical Company of Pittsburgh had developed a process of manufacture which was producing 1 gram of radium per month. However, carnotite was not so rich a uranium ore as pitchblende, and to produce 1 gram of radium in **1914** necessitated 500 tons of ore, the handling of 2000-2500 tons of sandstone rock, 500 tons of chemicals and 10,000 tons of sulphate-free distilled water, as well as six months of time, because of the time consuming fractional crystallisation method [9].

For a **1904** comparison with France, the refining process used by Armet de Lisle (1853-1926) to obtain 2-5 centigram of pure radium bromide from 1 ton of pitchblende ore used 5 tons of chemical products and 50 tons of water [10]. This French data was provided by M. de Razet engineer of the Ecole de Physique et de Chimie, and curator of the laboratory.

Although the difficulties experienced by the Curies in obtaining pitchblende from St. Joachimsthal are well known in general terms it is worth repeating their story and including numerical data which is not well known. It is taken from a 1916 publication by Dr Maximilian Kraus and a 1930 history of the Vienna Radium Institute by Stefan Meyer (1872-1949) [5, 11, 12]

'Pierre wrote to Dr Edward Suess, then President of the Austrian Academy of Sciences, explaining their difficulties (in obtaining pitchblende), asking that he intercede with the Austrian Government to the end that they might buy some of this residue. At that particular time the St. Joachimsthal mines were pretty thoroughly worked out, and the uranium, always of minor importance since it had so little commercial use, could not offset the silver and lead depletion. The Superintendent of Mines, G. Krompa, had loaded several waggonfuls of residue, probably with the thought that some of the value might be recovered. Fate was with the Curies!

Dr Suess had pleaded the Curies' case so eloquently that the Austrian Government made the French scientists an out-and-out gift of 100 kilograms of residue. This was only the beginning. Between 1898 and 1902 the Curies had 11,000 kilograms of residue from this same source. On 26 December 1899 they had 1000 kilograms, on 18 February 1900 & on 17 August 1902, shipment of 5000 kilogram each making a total of approximately 11 metric tons. The Austrian Government charged them transportation only for the 1899 and 1900 lots and sold them the 1902 lot at a special low price. Stefan Meyer estimated that the Curies must have had at their disposal during the final stages of this work the equivalent of more than 5 gram of radium – 5.6 gram to be precise.'

That in these early years before World War I that the St. Joachimsthal mines were the premier source of high grade pitchblende for radium refining is attested to in several publications. For example, Charles Parsons the Chief of the Division of Mineral Technology, US Bureau of Mines, wrote in 1913 [13] 'Pitchblende at Joachimsthal is the richest and most eagerly sought uranium radium ore. Outside of Austria, the only pitchblende deposits of any size are those in Gilpin County, Colorado, from which some 30 tons more or less have been procured since the mineral became valuable as a source of radium.'

Carnotite mining in Colorado 1881-1904

Carnotite deposits in San Miguel & Montrose Counties, Colorado were known several years before the discovery in December **1898** of polonium & radium by the Curies. For example, a US Bureau of Mines report of 1913 [14] stated that as far back as **1881** Andrew J. Talbert mined some (carnotite) ore and sent it to Leadville, where it was tested for gold, silver and copper. The report said it carried $5 gold.

In **1896** Gordon Kimball & Thomas Logan sent specimens to the Smithsonian Institution at Washington DC, and were informed that the mineral contained uranium. Shortly afterwards Kimball & Logan mined 10 tons of ore and shipped it to Denver where it was sold for $2600 to Poulot. (Earlier Kimball had sent Poulot a sample of the 'yellow stuff' from the Roc Creek claim: so perhaps the *real credit* for the discovery of carnotite should go first to Talbert, followed by Kimball and only then by Poulot.)

In **1899** (or possibly as early as **1897**) Poulot & Voilleque, two Frenchmen, visited Paradox Valley, received specimens collected by Thomas McKee and then sent them to Friedel & Cumenge in France, who announced in the French journal the existence of a new mineral which they named carnotite and described as potassium urano-vanadate [14].

In **1900** Poulot & Voilleque began operating at a copper mine at Cashin in Paradox Valley, Montrose County, where they used leaching vats to extract the uranium. Shortly afterwards they built a small mill in the McIntyre District, south of Paradox Valley, which ran until **1902**, and during this time produced about 15,000 pounds of uranium oxide. The mill was started again in **1903** by the Western Refining Company but ran only until **1904**. Shortly afterwards the Dolores Mining Company built a new mill a short distance from the old one, but after running some year it, too, shut down [14]. Later, Paradox Valley became the source for most of the carnotite mined for the Standard Chemical Company of Pittsburgh.

Pitchblende mining in Colorado 1871

Pitchblende was discovered in Colorado a decade earlier than the discovery of carnotite. In **1871** a London firm, the Rochdale Mining Company hired Richard Pearce (1836-1927) an English metallurgist who in 1859 & 1863 had identified veins of pitchblende in Cornwall, to inspect some gold properties they owned in Russell District, Gilpin County, Colorado [15]

He presented his August **1871** discovery to a meeting of the Colorado Scientific Society in **1895** [16]. He described how in the course of his investigations he found on the dump of one claim a heavy black material which proved to be uraninite 'coated with a beautiful canary yellow material'. The mine had been worked for some years previously for gold but the results were unsatisfactory. The heavy residue after sampling was found to contain no gold, was pronounced worthless and thrown into the creek. However, Pearce recognised the material from his work in Cornwall where he was used to finding it in only small quantities and not in the enormous mass in Colorado. Pearce obtained a quick test result at a mill in Russell Gulch and proved the existence of uraninite, or pitchblende as it was commonly called.

In **1871** he returned to the UK and presented his results to the Royal Geological Society of Cornwall in **1875** [17] about finding some 200 pounds of pitchblende which he reported to astounded American mining engineers that it was worth about £400 in the UK (equivalent to $2000 in the US). In **1872** Pearce returned to Colorado to take charge of a Rochdale Mining smelter, working mines under lease and sorting the ore to produce uranium oxide to be shipped to London [15]. Pearce returned to Cornwall in **1902**, carrying with him crystals taken from a radioactive spring, which he called 'radium crystals' and believing in their therapeutic value he added them to everything he drank until his death in **1927** [18].

Charles Poulot

The two chemists Charles Poulot (1859-1929) & Charles Voilleque (this is the usual spelling of this surname, except for Otto Brill's [24] version *Verilleque*) were partners in obtaining the mining samples sent to Friedel & Cumenge but thereafter appear to vanish from the literature on uranium mining & geology. Poulot arrived in Denver around **1897** [15, 19] with an eye to the purchase of rare metal ores, but later focussed on uranium. In December **1897** newspaper headlines reported 'A French chemist buying uranium in Colorado' [19, 20]. A process for extracting uranium from the ore had been patented by Poulot but it required at least 20% uranium in the ore and most of what was obtainable contained only 1%. Charles Poulot returned to France in **1902**, the year his carnotite processing plant closed, taking a large quantity of concentrates as a partial settlement with his partners for his share in the venture [15].

Thomas McKee

Thomas M. McKee was a pioneer Montrose County photographer & prospector, and Kathleen Bruyn the author of *Uranium Country* [5] is 'on the basis of credibility, convinced that Tom McKee started the chain reaction' which led to the analysis of carnotite by Friedel & Cumenge [2]. Such stories are hard to prove or disprove after all the years that have elapsed: but they make interesting reading!

McKee had since ~**1880** been dissolving the mineral in nitric acid and applying it to photographic plates to turn the plates a purple colour and he personally stated that he gave Poulot several pounds of the yellow mineral in Roc Creek in **1897** [21, 22].

The claim in Roc Creek was owned in the **1890s** by an Irishman Thomas Dullan and following the disclosure that the Dullan mysterious yellow ore contained uranium and might therefore be a source of radium, prospectors swarmed into the area. Many later lost their claims by default because of their failure to work them. Dullan sold his claim for $10,000 cash and it was then known as the *Copper Prince* mine. Subsequently it passed through several ownerships and was eventually renamed the *Rajah* mine [5].

Carnotite, Montroseite or Coloradoite?

The first name for the mineral appears to have been *chrome-copper*, because of its colour, although it turned out that there was no copper contained. Poulot called the mineral *autonite* or *uranochre* according to Bruyn [5] or *uraconise* according to Friedel & Cumenge [2]. McKee frankly admitted to being an American chauvinist 'and remembered raging because his pet ore had been named for a foreigner. Why he demanded could they not have called it *Montroseite* or *Coloradoite*? [5].*

Afterword in 1913 from Otto Brill of the Standard Chemical Company

It is appropriate for more than one reason why we give Otto Brill the final words in this paper. He was an Austrian chemist with a DSc from the University of Vienna who worked at St. Joachimsthal and was then employed in **1912** by the Standard Chemical Company of Pittsburgh (which owned the Radium Chemical Company) and wrote in the first issue of the Company's house journal, *Radium*, on *Uranium in Colorado* [24]. He was recalled to Austria in **1913** by the Austro-Hungarian government when it was decided that pitchblende/radium was too important to export and that the knowledge of scientists like Brill should remain in Austria [25].

Brill notes the very earliest discovery of carnotite, when it was valued only for its colour, quoting Tom McKee [21] as his source. 'The characteristic bright yellow color of this mineral attracted the attention of the early settlers in San Miguel and Montrose Counties, and the Ute and Navajo Indians are said to have used it for the production of yellow pigments for body and buckskin adornment'.

Brill concludes with an estimate of the amount of radium which could be expected to be obtained from

* When discoveries in one country are initially reported in another country there are often mistakes which occur. The most famous example relates to the discovery of X-rays by Wilhelm Conrad Röntgen. The first public notice had been on 5 January 1896 in Vienna's *Die Presse* newspaper but by the time this was reported in London's *Daily Chronicle* on 6 January it was said that the discovery was made in Vienna (not Würzburg) by Professor Routgen (not Röntgen). For carnotite there were reporting mistakes in a 1904 textbook *Radium & Other Radio-active Elements* [23]. 'Radium has been discovered in the mineral *carnallite*, in Utah, USA. In Texas there appears to be a large deposit of radiferous material, although whether radium is present in sufficient quantity for profitable extraction is as yet unknown. Radium also found in various places in Saxony.'

carnotite in Colorado [24]. 'Conservative experts estimate the amount of uranium in this *carnotite belt* of Colorado to be about 8,000,000 pounds U_3O_8. According to our experience, this would correspond to an amount of about 900 grams of radium, or about 4 pounds of pure radium bromide.'

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