# **URANIUM NUMBERS ALERT**

Author: Colin C. Brooks Submitted for Australia's Uranium Conference Proceedings

> Contact person: Colin C. Brooks 10 Winifred Ave., South Plympton, S.A. 5038 Tel/fax 61-(0)8-829 74 928 e-mail: colbrx@attglobal.net

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Colin C. Brooks, Independent Exploration Consultant Fellow, AusIMM Fellow, AIG Fellow, AAG Member, SME Member, CIM Member Geol. Soc. Aus.

Member, MICA Chartered Professional (Geology), AusIMM Professional Geoscientist, AIG

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#### ABSTRACT

Uranium property exploration and evaluation pose many of the challenges common to base and precious metal deposit sampling, sub-sampling, sample preparation and analysis. As always, geological interpretation plays a major role in planning sampling, selection of techniques and changing the planning as results come to hand as well as in the mechanics of obtaining numbers and their interpretation.

Abnormal mineral hardnesses of both ore minerals and gangue minerals, specific gravity differentials, recovery problems in both sampling and sample preparation as well as in primary metallurgical evaluation need consideration.

Brief summaries of tricks and traps of sampling of uranium deposits in Australia in the past are given, with potential and real impacts of misunderstanding the numbers.

Sample preparation techniques, past and present, with recent advances to minimise problems are discussed.

Analytical techniques, and parts of their evolution over the last fifty years are mentioned.

Simple outlines of uranium decay chains are given as essential to understanding modern analytical technology.

While all of these factors warrant constant attention to detail, several aspects specific to uranium mineralisation are emphasised. Included are recovery of fine-grained minerals in sample preparation, analysis and mining and metallurgy. Radiometric disequilibrium of sufficient proportion to invalidate simple interpretation of radiation as being directly proportional to uranium content is shown by direct comparison with PFN (Prompt Fission Neutron) downhole logging.

This paper is intended to be an alert to those producing and/or using uranium numbers in property evaluation.

#### Introduction

Uranium, base and precious metal exploration and evaluation, all need evolving soundly based geological interpretation to guide and control sampling. From well designed programs, well-taken samples need appropriate sample preparation, including sub-sampling and appropriate analysis, to provide reliable data from which tonnages and grades can be interpreted at appropriate levels of confidence. Bore hole-logging techniques and control of both observations and interpretation of data are included here. Indeed in some properties they dominate the effort.

A serious worry, which has received much publicity over the years, is non-technical. This is the decision-making embarked on by people with insufficient knowledge and experience even to seek advice when they are out of their depths in such exercises. Despite the plethora of papers and texts alerting such people to critical errors of omission and commission by those in control, financial disasters caused by shortcomings in generation and usage of uranium numbers still present high financial risks.

Many executives, journalists, economists and even scientists have had restricted access to some of these realities, leading them to assume any resource estimate is publication ready. Understanding enough specific details is essential for them to determine the level of quality assurance achieved and thereby avoid errors of judgment. This particularly applies where estimates are prepared, often in good faith, by those who were supplied with inadequate data or have insufficient technical knowledge and experience to understand the derivation of the numbers and chose an appropriate calculation methodology. This problem was highlighted in Roadblocks to the Evaluation of Ore Reserves, by Journel, (2005, p19), in which he stated:

### "Better an inaccurate geology than an automatic interpolation algorithm ..." and "The major source of uncertainty is the geological uncertainty."

This brief review highlights some areas of uranium exploration and evaluation that have and are providing grounds for concern. It is intended to amplify warnings deemed necessary by recent observations such as those aired at the Australia's Uranium Conference held in Adelaide, South Australia, in July 2006, by authors including L. Pretorius, B.L. Dickson & A.M. Giblin and R.A. Bowden & R. P. Shaw. (Abstracts only of those papers were available at the time of writing).

Extremely careful review of uranium resource and reserve numbers by experienced people is essential before they are released to the public domain.

#### Sampling

In 1982, King et al., stressed that the key deficiency in ore reserve estimation was the geological factor. Two uranium examples among the many available should be enough to support that contention.

Early in the development history of Radium Hill, a highly competent senior geologist was asked for his best estimate of the resource based on limited drilling and underground exposure. As this work had exposed a significant bulge in width of one of the ore veins and only about a quarter of the known longitudinal projections of the known veins had been tested, his reasonable forecast included three more postulated pods. A few years later, when exploration and development demonstrated there were no additional pods, the "resource" was halved, albeit with no appreciable grade change. This caused consternation in London and Washington because no one further up the line knew enough to understand the true import of question and answer. Many had assumed that they were seeing a firm reserve figure.

Nabarlek, the subject of an open public enquiry, had an original resource estimate in excess of 50,000 tons of contained  $U_30_8$  published. A lawyer ran the controlling company. The highly experienced Chief Geologist had a doctorate from an excellent university and many years of experience - but that was in other countries, dominantly related to oil search. Neither man knew they had insufficient experience and knowledge to calculate resources or reserves of narrow high-grade uranium mineralisation. Consequently neither sought expert advice before going public. When the number was cut to 10,000 tons there was an uproar.

The same company did not understand the geology of the ore zone, nor its surface expression, despite cutting several trenches, before such an announcement. A refolded fold nose is shown in Figure 1, a photograph taken in one of those trenches.



Fig 1: Refolded fold nose in Nabarlek trench

Subsequently a geologist placed by the company in charge of field project work to generate a revised resource was allocated the work based on seven years of uranium experience. Investigation by this author determined that four years of those were prior to his tertiaryeducation and the three years after that were mainly in uranium fieldwork using a sensing technique which involved no detailed geological knowledge relevant to the task. None of his company superiors seemed aware that these credentials were quite inappropriate with respect to "*competent person*" status in this situation.

One result was the drilling shown in Figure 2. The diamond drill hole pointed at was planned to come out of the ground less than 30 metres down slope from its collar. The driller refused to drill it. So the geologist steepened the design by five degrees. When the driller had a problem he stopped the machine, walked several metres down-slope and dug a small hole with a pick and shovel to sort out the drill bit and send it on its way.



Fig 2: Drill collars at Caramal prospect

Another part of this problem is the lack of understanding at senior levels that large disseminated deposits (of uranium and base metals) require a drilling intensity of at least one metre per 6,000 tonnes of resource, perhaps one per 4,000 or otherwise sufficient to demonstrate reliable continuity of grade. Recently there have been unpleasant surprises after mine development construction predicated on reserves of several 100 million tonnes based on one drilled metre per 12,000 tonnes or even 16,000 tonnes . There is never a "right" answer in this endeavour but often a wrong one.

#### **Sub-sampling**

Many traps exist in the processes from sub-sampling to assaying. A few are mentioned to illustrate potential impacts to be avoided if the results are not to be misleading or even disastrous. Figure 3 (the white horizontal line is the mark of the halving blade!) shows gross incompetence reflected in many of the so-called half-cores from this high-grade uranium deposit remaining in the trays after the other parts had been taken for crushing-grinding-pulverising prior to sub-sampling for assay. The percentage of volume taken for assay varied from 15% shown here, to 70% in other trays. The errors were random, but commonly large.

As those who have halved core (often by diamond sawing) and submitted both halves for assay have found variation between the two is commonly 10% to 15% of the lower assay.



Fig 3: Poorly halved core Nabarlek

Sample grain size reduction and determination of appropriate sub-sampling volumes have been the subject of countless publications for many years. The best known used to be the Mining Engineers' Handbook (Peele, Ed.) published first in 1918, while others such as Pitard's "Pierre Gy's Sampling Theory and Sampling Practice", first published in 1989, are on many bookshelves. Hoffman and Dunn (1992) stress the problems that can arise when ore minerals have much higher SG than host rock minerals. Despite this wealth of knowledge, disregard of basic principles and practice is still very common. Peele's section 29, Ore Sampling, is still a good reference to many of the forms of sub-sampling although modern rotary splitters, which have been developed to solve some of the uncertainties, are gradually becoming more common.

Figure 4 is a generalised sample mass nomogram (available in relevant textbooks in a more complete form with instructions as to usage). This form of guide is commonly ignored or unknown. Crusher and splitter operations in the field and in laboratories are often sources of significant errors today as a result.



Fig 4: Generalised sample mass nomogram

Worse are cavalier methods of using often poorly constructed sub-sampling equipment and techniques, despite much exposure in recent conferences, symposia and the technical press - c.f., Brooks 1999 and 2004 and the references therein.

While riffle splitters are slowly being replaced by rotary splitters, their designs, manufacture and operations remain sources of unacceptable errors in resultant assay numbers.

As examples, consider Figure 5 adapted from Pitard, 1989. Obviously "W", the slot width should be constant and the number of slots even. In this century, on a major mine, both edicts were seen denied. As recently as June 2006, on a significant Australian drilling project, a splitter was in use with alternate slots being 20mm and 22mm wide.

Usage in the laboratories of major assaying companies and on mines often leaves a lot be be desired- c.f. Brooks 2004, Figures 6 & 7 in for examples.



Fig 5: Jones riffle splitter longitudinal section diagram

Outlined on Figure 6 are some of the principles are which were being denied on a high grade uranium property where the catch bins, which were too long for this application, were



Fig 6: Jones riffle splitter cross section showing potential problems

used to feed a well-made Jones type splitter Figure 7. As the feed indicated on Figure 6 was being thrown onto the sloping feed surface in the near corner of the splitter, an obvious gravity segregation and upgrading was taking place. The top 20 high grade samples' reject



Fig 7: Misuse of Jones riffle splitter - Nabarlek – both gravity separation and unequal sub-samples

portions from this operation were reduced to minus 400 mesh and rotary split into three by re-combining products from sets of four non-adjacent bins of a twelve-way splitter. One of those thirds was sent to Canada, one to U.S.A. and the last re-analysed in Australia. Results by radiometric and chemical analyses were virtually identical but showed that the original work had created a minimum of 15% error above the revised average value of one sample, a maximum of 28% error above another, and a mean error of 22% above the accepted re-assay values. That is the errors were systematically in the one sense - all too high, as expected from the field examination. How anyone could use three or four significant figures for a tons/grade ore reserve from numbers derived in such a manner is astounding

While relevant technology is improving rapidly it seems that too little of it is permeating the operating levels of our exploration and mining companies. Too often inappropriate sample preparation protocols, dictated by mining companies to their staff and to commercial assay laboratories, are followed blindly, resulting in large cash flows being foregone by misallocation of feed to mill or waste, and/or incorrect designation of ore boundaries.

Pitchblende grains having about four times the SG of its host rock is prone to gravity settling within a sample. This leads to common sources of sampling error which on occasion may occur sequentially in combinations between drill hole and analysis with potentially disastrous consequences.

Two sequential examples illustrate the problem. In the scooping of pulverized sub-samples off a mat, after roll mixing, using a scoop blade too thick to collect any of the fine pitchblende grains coating the mat surface where they have been concentrated, results in reduced uranium content. If 250g of the depleted product were then placed in a Kraft packet, agitated by transport, to where an analyst scoops out 30 or 50g from the packet, the sample analysed would be further depleted due to the many fine, heavy grains that settled toward the base of the packet. Even emptying the packet may still leave a pitchblende concentrate in the base corners of the packet.

#### Analysis

Eight techniques were outlined for uranium and thorium analysis in a symposium held in 1970 by the Australian Atomic Energy Commission (Florence (ed) 1970). Many others have been published in textbooks and IAEA publications over the years.

Dominant are radiometric, neutron activation, ICP-MS and X-ray fluorescence used in uranium exploration, evaluation and grade control. Obviously deficiencies in digestions or fusions and failures to calibrate data can grossly affect the levels of precision desired and obtained. These points require constant vigilance.

Another is that technically trained exploration staff are frequently unaware of the specifics of techniques they employ to determine grades. This was borne out starkly at AusIMM's 2006 Australia's Uranium conference in Adelaide, particularly by Pretorius (op. cit.) who detailed inappropriate sample collection of carnotite samples, resulting in significant loss of

sample before analysis. Holes were radiometrically logged down hole and checks made by hand-held scintillometry but the results did not correlate at all well with over 2,000 checks run for uranium by XRF. No correlation was found between radioactivity and carnotite distribution in 2006 due to secular radionuclear disequilibrium. This effect, while well-detailed in appropriate literature over many years, was not on someone's attention list earlier in exploration he detailed.

A good summary of the effect and a solution to the disequilibrium problem was published in 1989 by Givens and Stromswold, and the tool, PFN (or prompt fission neutrons) has been in use in Australia for several years. The problem was well known in the early 1970's, as Givens and Stromswold's references attest, and various methods devised to deal with it. Hallenburg (1979) gave a very clear introduction to this and to the delayed fission neutron technique at an AMF Course in 1982 in Adelaide, S.A.

A brief outline only is presented here before giving some examples of the significance of disequilibrium on exploration by considering results from two drill holes from an operating property. The ore mineralogy is very fine grained "coffinite intimately associated with kaolinite which forms coatings on the surfaces of quartz grains and party fills the interstices between them" (Marsland-Smith, 2005).

Figure 8 is a simplified plot of the radiometric decay series of each of U238, U235 and thorium (Th232). Points to note are that there is about 140 times as much U238 as U235 in natural uranium, that the major gamma emitter in the U238 chain is bismuth (Bi214) and that due to solution/mobility effects there are two areas of disconnect in both uranium decay chains marked by wavy lines on the figure. By solubility of radium (Ra) and mobility of radon gas (Rn) it is possible for Bi214 to be generated in locations remote from its parent U238 - hence the gamma signature of uranium in non-uranium bearing locations. Details of these decay series, their half-lives, radiation, gamma ray energies etc. were checked against table 10.2 on p. 743 of Telford et al, 1976 - one of many such sources of these data.

Calculations indicate that equilibrium of uranium will be restored by natural decay processes in just over a million years, provided that there is no chemical or physical disturbance. In other words, if separation of uranium from its highly radioactive daughter products is taking place or has taken place less than a million years ago gamma determination cannot give an accurate measure of uranium content at the point of measurement.



Fig 8: Radiometric Decay Series, U235, U238 Th232 with two disconnects between parents & daughters

The PFN, down-hole logging tool was invented as one of a few ways round the problem. Basically fast neutrons are generated by impacting tritium with deuterium resulting in rapidly pulsed bursts of high-energy neutrons. These very quickly lose energy such that when they have become of thermal energy they can be absorbed by U235 which then emits other neutrons which can be counted directly, exactly as in INAA measurement of uranium. For example see Hoffman, (1992). Bivens et al (1980) describe a variant of the tool that uses the gamma count from a spectral window in lieu of the thermal neutron flux.

The generator has a 14 MeV neutron output burst of  $5x10^7$  n/s which lasts about 20 microseconds. The pulsing frequency is about 1 kHz.

The tool has a tritium epithermal neutron-detecting core surrounded by a cadmium shield and eight beryllium trifluoride thermal neutron detectors. Both forms of detector are gas filled. Signals are sent uphole in analogue form. Neutron pulses are counted uphole during a single broad time-gate that begins 50-100 microseconds after the end of each neutron burst and stops with the trigger for the next burst.

On Figure 9 geophysical logs of point resistance, self-potential and neutrons are shown on the right. Interpreted geology from these is shown in the centre and interpreted uranium grade from gamma and PFN logs on the left. The red uranium cut-off grade line shows clearly that while the gamma signature is present, it is below that indicating economic uranium grade while the PFN grade is clearly above cut-off. Assuming excellent calibration, appropriate logging speed and positioning, the identified coffinite locked in clay at top and/or bottom of any such ore section will not be amenable to recovery by ISL technology, stressing the need for interpretation of the geophysical logs.



Fig 9: Disequilibrium - U3O8 with low gamma count.

In an in-situ leach operation the detailed nature of the host geology needs careful consideration, as do depth correlations of the logging tools in addition to very careful calibration considerations. Formation density and borehole diameter are two of the correction requirements relative to a standard be it gamma or PFN data.

A false ore grade zone is indicated on Figure 10, just below 116 metres, while above and below in this intercept both gamma and PFN results indicate the presence of ore grade uranium. This shows the danger of relying solely on gamma logging in such geology.



Fig 10: Low gamma count with and without U3O8

#### Summary

One might quote Thompson's edict from 1992:

"Non-representative samples will not yield a valid interpretation, no matter how good the subsequent analysis."

Good geological understanding and good communication are mandatory precursors to all other work. Even with excellent understanding of geology, well taken, prepared and analysed samples and first class interpretation of those results, it is highly unlikely that more than two significant figures can be used for uranium resources or reserves, particularly for podiform or ISL type deposits. It is therefore seen as essential that those producing uranium numbers and those using them each have excellent understanding of the whole process of how and why the numbers have been produced from geology through sampling, sub-sampling and analysis to mathematical treatment of the analyses in their three dimensional space, and how and why they have been interpreted.

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