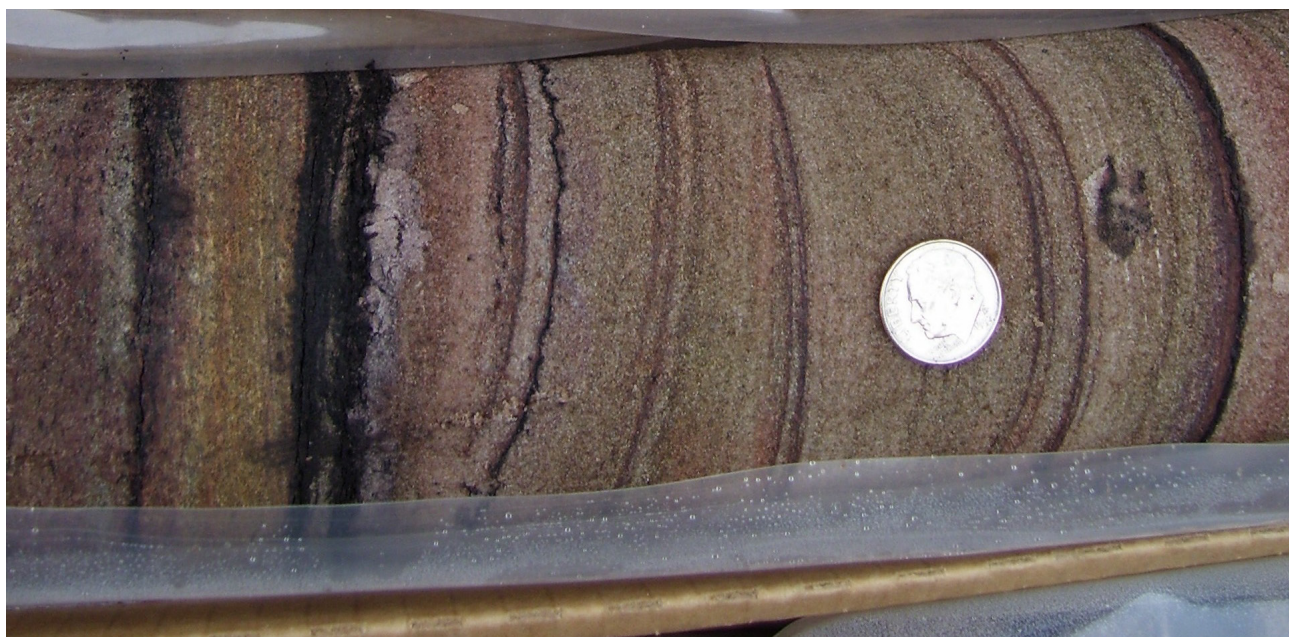




In cooperation with the Environmental Protection Agency

Solid-Phase Data from Cores at the Proposed Dewey Burdock Uranium In-Situ Recovery Mine, near Edgemont, South Dakota

By Raymond H. Johnson, Sharon F. Diehl, and William M. Benzel



Open-File Report 2013–1093

U.S. Department of the Interior
U.S. Geological Survey

U.S. Department of the Interior
SALLY JEWELL, Secretary

U.S. Geological Survey
Suzette M. Kimball, Acting Director

U.S. Geological Survey, Reston, Virginia: 2013

For more information on the USGS—the Federal source for science about the Earth, its natural and living resources, natural hazards, and the environment—visit <http://www.usgs.gov> or call 1-888-ASK-USGS

For an overview of USGS information products, including maps, imagery, and publications, visit <http://www.usgs.gov/pubprod>

To order this and other USGS information products, visit <http://store.usgs.gov>

Suggested citation:

Johnson, R.H., Diehl, S.F., Benzel, W.M., 2013, Solid-phase data from cores at the proposed Dewey Burdock uranium in-situ recovery mine, near Edgemont, South Dakota: U.S. Geological Survey, Open-File Report 2013–1093, 13 p., <http://pubs.usgs.gov/of/2013/1093/>.

Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

Although this information product, for the most part, is in the public domain, it also may contain copyrighted materials as noted in the text. Permission to reproduce copyrighted items must be secured from the copyright owner.

Contents

Abstract.....	1
Introduction.....	1
Methods.....	2
Results.....	3
Observations.....	3
Acknowledgments.....	4
References Cited.....	4

Figures

1. Map showing approximate location of study area	5
2. Map showing satellite imagery overlain with core locations	6
3. Expanded view of Dewey area with core locations.....	7
4. Expanded view of Burdock area with core locations	8
5. Summary of mineralogy from the Fall River Formation in the Dewey area.....	9
6. Summary of mineralogy from the Chilson Formation in the Burdock area	10
7. Scanning electron microscopy and cathodoluminescence images of the same constituent quartz grains from the Fall River Formation in the Dewey area	11
8. Scanning electron microscopy backscatter and cathodoluminescence images of the same constituent quartz grains from the Fall River Formation in the Dewey area.....	12
9. Scanning electron microscopy backscatter and cathodoluminescence images of the same constituent quartz grains from the Chilson Formation in the Burdock area.....	13

Tables

1. Solid-phase geochemistry data	link
2. X-ray diffraction data	link

Appendix Files

1. Carbon/sulfur data file	link
2. Uranium isotopes	link
3. Transmitted light images	link
4. SEM images.....	link
5. SEM elemental maps	link

Solid-Phase Data from Cores at the Proposed Dewey Burdock Uranium In-Situ Recovery Mine, near Edgemont, South Dakota

By Raymond H. Johnson, Sharon F. Diehl, and William M. Benzel

Abstract

This report releases solid-phase data from cores at the proposed Dewey Burdock uranium in-situ recovery site near Edgemont, South Dakota. These cores were collected by Powertech Uranium Corporation, and material not used for their analyses were given to the U.S. Geological Survey for additional sampling and analyses. These additional analyses included total carbon and sulfur, whole rock acid digestion for major and trace elements, $^{234}\text{U}/^{238}\text{U}$ activity ratios, X-ray diffraction, thin sections, scanning electron microscopy analyses, and cathodoluminescence. This report provides the methods and data results from these analyses along with a short summary of observations.

Introduction

Powertech Uranium Corporation (Powertech) has proposed to mine uranium at the Dewey Burdock site using in-situ recovery methods. The Dewey Burdock site is located in the southwestern region of the Black Hills of South Dakota (fig. 1). The uranium-recovery license application submitted by Powertech to the United States Nuclear Regulatory Commission (NRC) is publicly available and contains background information about the site along with technical details and baseline sampling data. The NRC application website is <http://www.nrc.gov/materials/uranium-recovery/license-apps/dewey-burdock.html> (with detailed application documents under the “application documents” link). A brief summary of the site history is provided by Powertech at <http://www.powertechuranium.com/s/DeweyBurdock.asp>.

At the Dewey Burdock site, uranium occurs as roll-front ore bodies in several sandstone units of the Inyan Kara Group of Early Cretaceous Age. The Late Jurassic Morrison Formation (shale) underlies the Inyan Kara Group. In the vicinity of the mine site, the Inyan Kara Group is composed of the Fall River Formation (upper sandstone unit) and the Lakota Formation (lower sandstone unit). The Lakota Formation is composed of the Chilson and the Fuson Members, with the Fuson Member occurring between the Fall River and Lakota Formations. The Fuson Member (shale) is considered a confining unit between the Fall River Formation and the Chilson Member of the Lakota Formation. Uranium roll-front deposits occur in the Fall River Formation and the Chilson Member of the Lakota Formation.

In 2007, Powertech completed several exploration holes that included core collection. Much of the core they collected was not used for their own analyses and was given to the U.S. Geological

Survey (USGS) for additional sample analyses and long-term storage. These analyses were completed with funding from the U.S. Environmental Protection Agency (EPA) Region 8's Regional Applied Research Effort (RARE) program. The EPA is responsible for evaluating the site through its underground injection control program (<http://www.epa.gov/region8/water/uic>) and Powertech has submitted a separate permit application to EPA (see documentation in a link within the above EPA website).

Sampling was completed at 31 selected core locations for total carbon and sulfur, whole rock acid digestion for major and trace elements, $^{234}\text{U}/^{238}\text{U}$ activity ratios (AR), X-ray diffraction (XRD), thin sections, scanning electron microscopy (SEM) analyses, and cathodoluminescence. Locations of these cores are provided in figures 2, 3, and 4, and latitudes and longitudes are shown in table 1. These location maps are similar to maps of well samples provided in Johnson and others (2012). A well from those previous maps is shown in figures 3 and 4, for reference. The following sections discuss all of the analyses methods, data results, and a few general observations.

Methods

Total sulfur and carbon concentrations as weight percent were analyzed by the EPA at their laboratory in Ada, Oklahoma. Replicate analyses were performed for several samples and the values for one standard deviation of error are provided along with the average carbon and sulfur concentration (Appendix 1). Samples were analyzed using a ThermoScientific Flash 2000 CHNS-O analyzer. The samples were combusted at greater than 1,200°C, and the resulting combustion gases were swept into a chromatographic column by a carrier helium gas. Gases were separated in the column and detected by a thermal conductivity column. Five blank analyses were found to be below detection limits (Appendix 1). Total sulfur was analyzed using ASTM method D4239-11 (ASTM, 2011) and total carbon was analyzed using ASTM method D5373-08 (ASTM, 2008).

Uranium concentrations and $^{234}\text{U}/^{238}\text{U}$ activity ratios were measured by Dr. Michael Ketterer at the Northern Arizona University in Flagstaff, Arizona. Details on the analytical procedures can be found in Appendix 2. In addition, three samples were analyzed in duplicate with no differences being greater than 7 percent (Appendix 2).

Whole rock analyses were completed at the USGS Central Mineral and Environmental Resources Science Center (CMERSC) Laboratories in Denver, Colorado. Rock and sediment samples were prepared by air drying and grinding to less than 150 microns (Taylor and Theodorakos, 2002) and then digested using multiple acids. The elemental concentrations in the digestion fluid were analyzed using inductively coupled plasma–mass spectroscopy (ICP–MS) with the procedure described by Briggs and Meier (2002).

Mineral phases were identified using X-ray diffraction analysis with Material Data Inc. (MDI) Jade (version 9.1) search-match software from the International Centre for Diffraction Data's "2009 PDF-4" and the Inorganic Crystal Structure Database developed cooperatively by the National Institute of Standards and Technology and Fachinformationszentrum Karlsruhe. Semi-quantitative mineral estimates were calculated using MDI Whole Pattern Fit software, which simultaneously calculates a whole pattern fit and a Rietveld refinement of the minerals. Reference minerals are selected from the database; some of these reference materials are "structure" references that represent perfect crystals of the mineral, and other entries are more typical mineral specimens. Each reference contains a full crystallographic description of the mineral. A calculated model of the observed pattern is produced by nonlinear, least-squares optimization. The calculations, performed by the software, involve the application of various parameters to improve the fit of the model to the observed data. Modeling parameters include background reduction,

profile fitting, and lattice constants. The software iterates and minimizes a residual error between the calculated X-ray diffraction patterns from the selected references in comparison to the measured scan of the sample. All data were normalized to 100 percent based on the identified minerals, within a 1-percent error. A full description of the Whole Pattern Fit algorithm is available from MDI.

Core samples with elevated uranium concentrations from the Dewey Burdock site were selectively collected to identify uranium and vanadium mineralogy at the Denver Microbeam Lab. Polished thin sections of this core material were examined, using transmitted light microscopy, and a JOEL 5800LV scanning electron microscope (SEM) with an Oxford ISIS energy dispersive X-ray spectrometer (SEM/EDS). Because uranium did not occur as discrete minerals in the Burdock sample(s), element intensity maps, also known as element distribution maps, were gathered on an electron microprobe (EMPA) to determine the spatial distribution of uranium in carbonaceous material. In these element maps, cool blue colors denote lower concentrations of an element, and bright green to yellow and red colors signify higher concentrations. In addition, scanning electron microscope cathodoluminescence (SEM-CL) images were acquired on the SEM with a Centaurus CL detector and photomultiplier, operated at 20 kilovolts (kV) and 10 nanoamps (nA), to identify radiation damage halos in quartz grains.

Results

All of the resulting data from total sulfur and carbon, uranium isotopes, and whole rock analyses are provided in table 1. Table 1 also indicates the depths for all of the samples. The original carbon/sulfur analyses data file from the EPA is provided in appendix 1, which includes quality assurance/quality control (QA/QC) sampling with duplicates, blanks, and an internal standard. The original data file on $^{234}\text{U}/^{238}\text{U}$ activity ratios is provided in appendix 2, which includes QA/QC data from duplicate samples. X-ray diffraction analyses with comments are provided in table 2. The mineralogy from the Dewey Burdock core thin sections and SEM analyses is summarized in figs. 5 and 6. Results of cathodoluminescence in the Fall River Formation in the Dewey area are shown in figs. 7 and 8, and results of cathodoluminescence in the Chilson Formation in the Burdock area are shown in fig. 9. Selected additional core images are provided in appendices 3 through 5 (transmitted light, SEM images, and SEM elemental maps, respectively) with file names matching the sample names in table 1.

Observations

The primary purpose of this report is to release data. However, in looking at the data, major differences in the Dewey and Burdock areas are quite significant. Overall, the samples from the Dewey area (Fall River Formation, DB07-32 series) have higher calcite concentrations, no organic carbon is seen in SEM images, and radiation damage halos are seen in the SEM-CL images (which indicates the past presence of uranium that has later been dissolved away, leaving radiation damage on quartz grains). In addition, vanadium concentrations are quite high in the uranium ore samples (DB07-32-4C-3, 4, and 5). The Burdock area samples (Chilson Formation, DB07-11 series) have lower calcite concentrations, more organic carbon seen in SEM images, and lower vanadium concentrations in the uranium ore samples (DB07-11-14C-5, 6, and 7). In addition, the SEM elemental maps in the Burdock area show uranium that is sorbed to the organic carbon and cathodoluminescence results do not show any radiation damage halos.

Acknowledgments

The authors thank John Horton for his geographic information system (GIS) assistance in preparing the map figures and Heather Lowers in the USGS Denver Microbeam Lab for doing the SEM elemental mapping.

References Cited

- ASTM Standard D4239-11, 2011, Standard test methods for sulfur in the analysis sample of coal and coke using high-temperature tube furnace combustion: ASTM International, West Conshohocken, Penn., 4 p., DOI: 10.1520/D4239-11, accessed February 04, 2013, at <http://www.astm.org/>.
- ASTM Standard D5373-08, 2008, Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal: ASTM International, West Conshohocken, Penn., 9 p., DOI: 10.1520/D5373-08, accessed February 04, 2013, at <http://www.astm.org/>.
- Briggs, P.H., and Meier, A.L., 2002, The determination of forty-two elements in geological materials by inductively coupled plasma–mass spectrometry, chap. I of Taggart, J.E., ed., Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 02–223-I, 14 p. (Also available at http://pubs.usgs.gov/of/2002/ofr-02-0223/I20NAWQAPlus_M.pdf.)
- Johnson, R.H., 2012, Geochemical data from groundwater at the proposed Dewey Burdock uranium in-situ recovery mine, Edgemont, South Dakota: U.S. Geological Survey, Open-File Report 2012–1070, 11 p. (Also available at <http://pubs.usgs.gov/of/2012/1070/>.)
- Taylor, C.D., and Theodorakos, P.M., 2002, Rock sample preparation, chap. A1 of Taggart, J.E., ed., Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 02–223-A1, 7 p. (Also available at http://pubs.usgs.gov/of/2002/ofr-02-0223/A1RxSampPrep_M.pdf.)

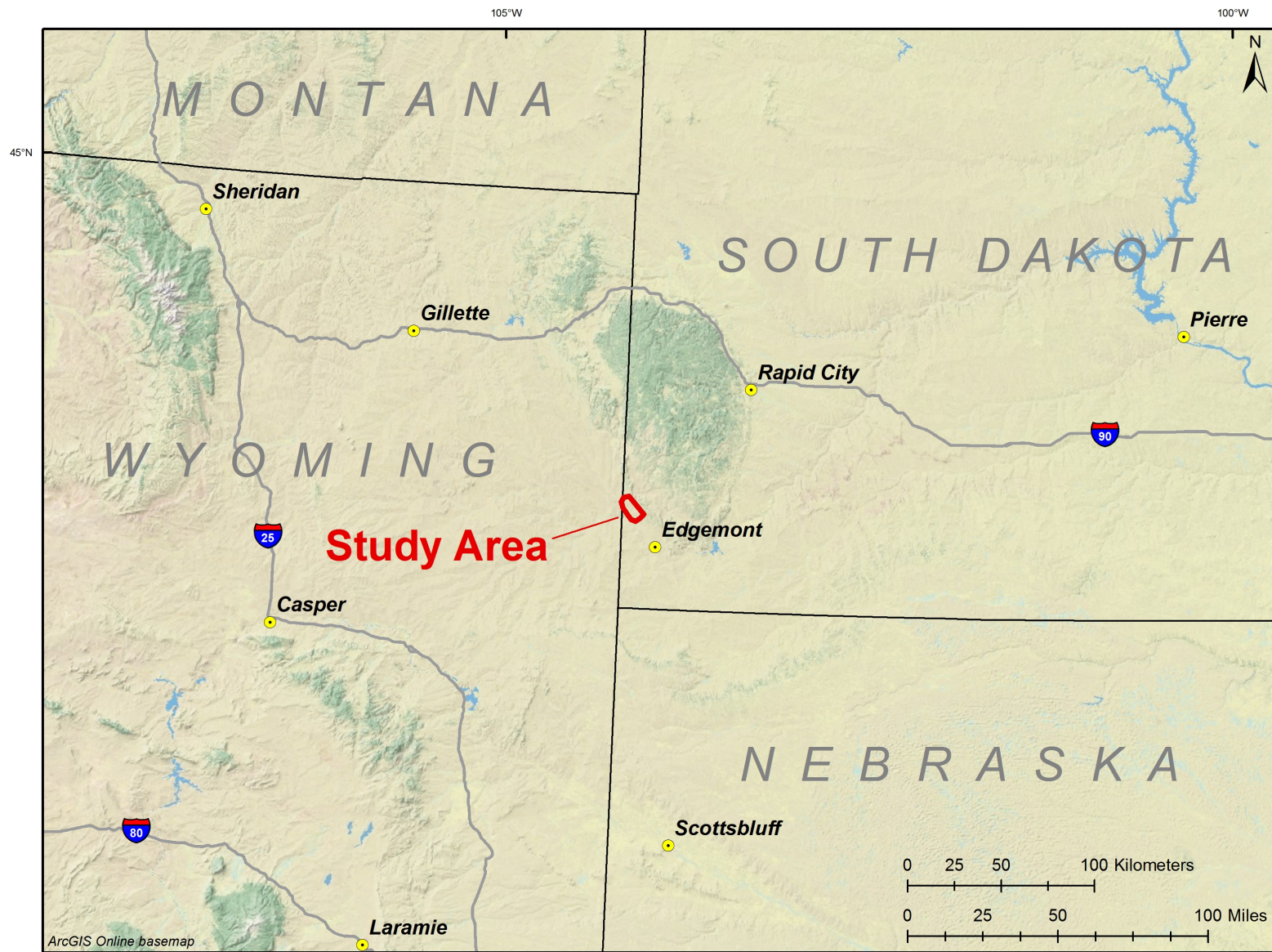


Figure 1. Map showing approximate location of study area.

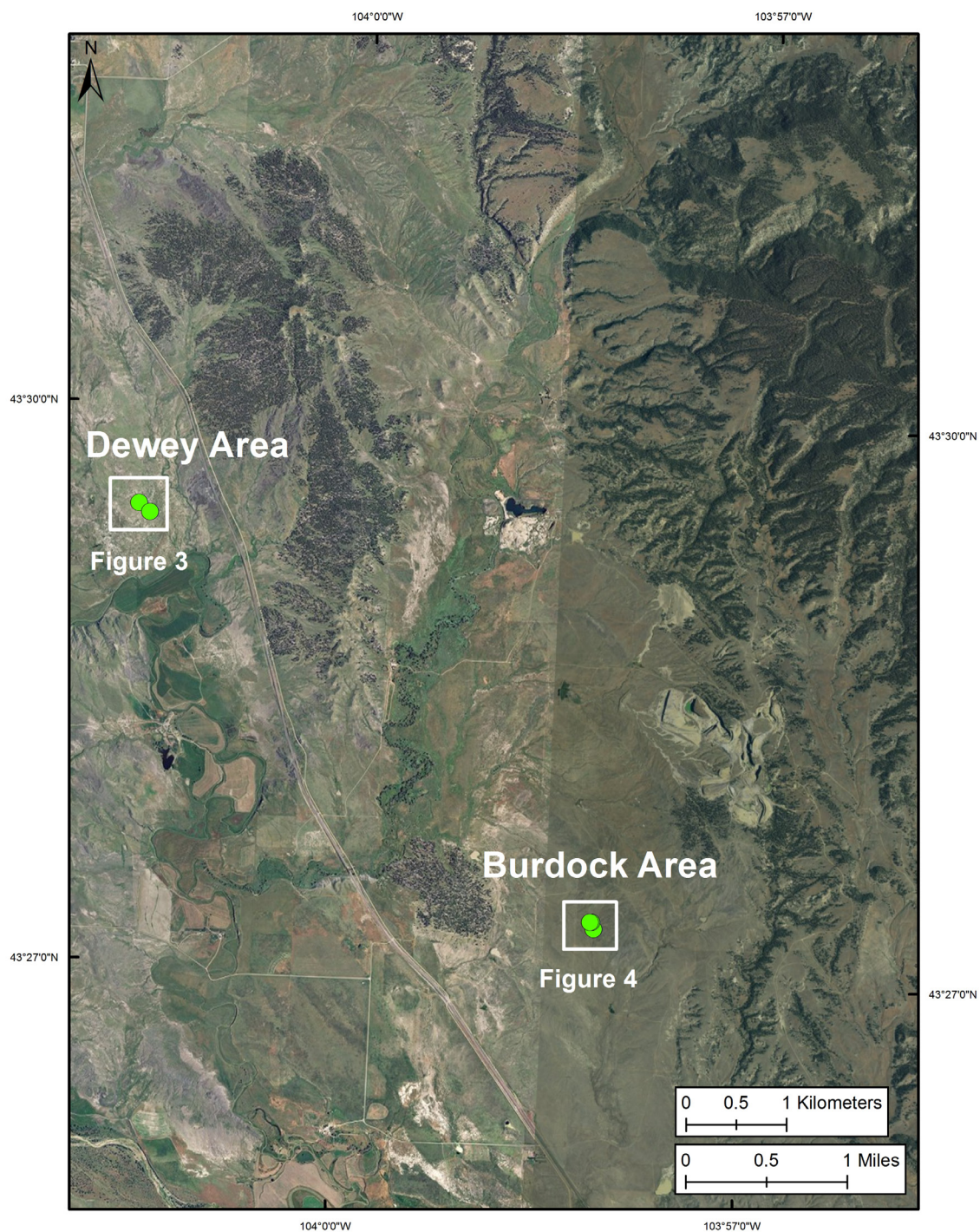


Figure 2. Map showing satellite imagery overlain with core locations. White boxes indicate locations of expanded views for figures 3 and 4.

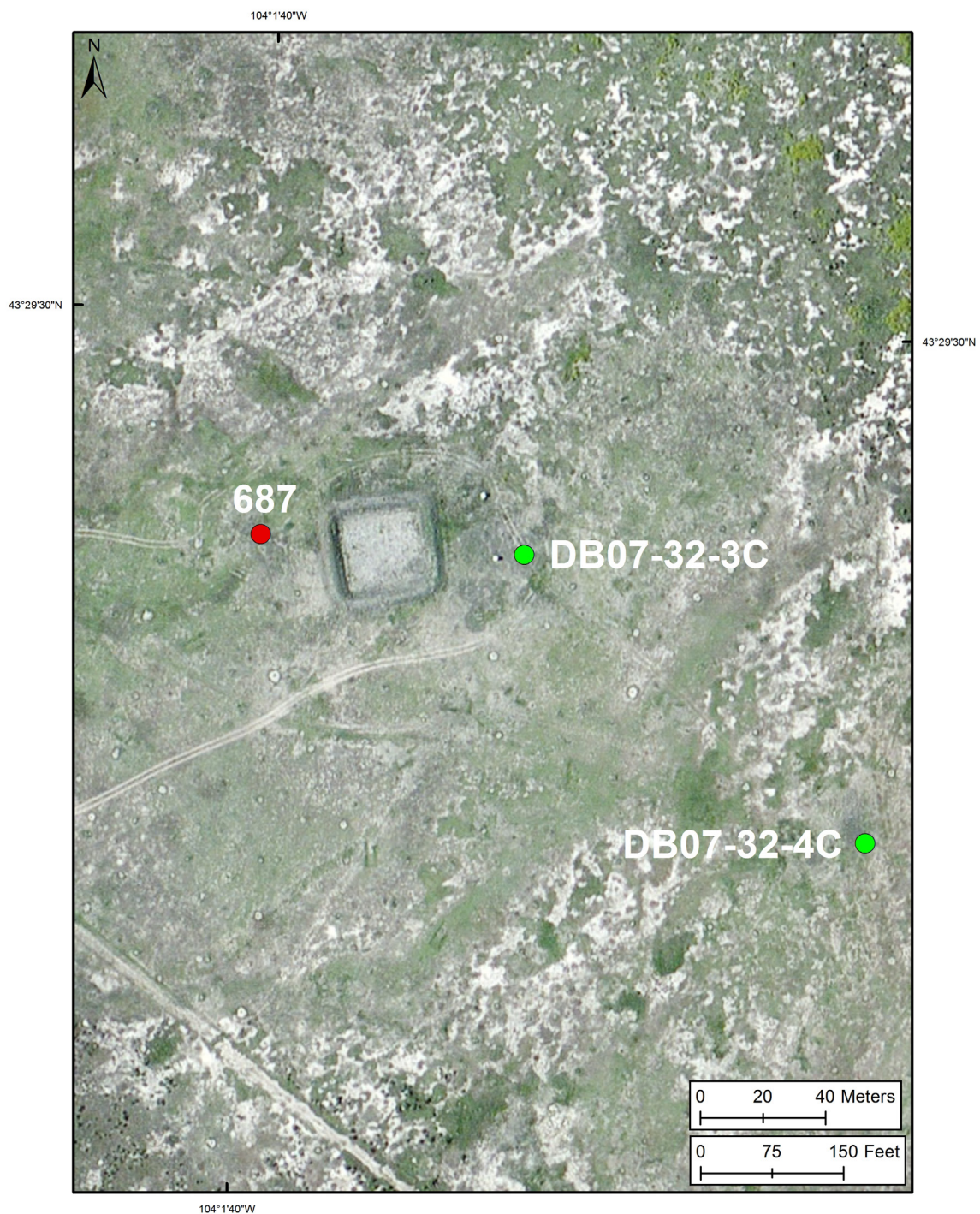


Figure 3. Expanded view of Dewey area with core locations. Location of well 687 is included for reference.

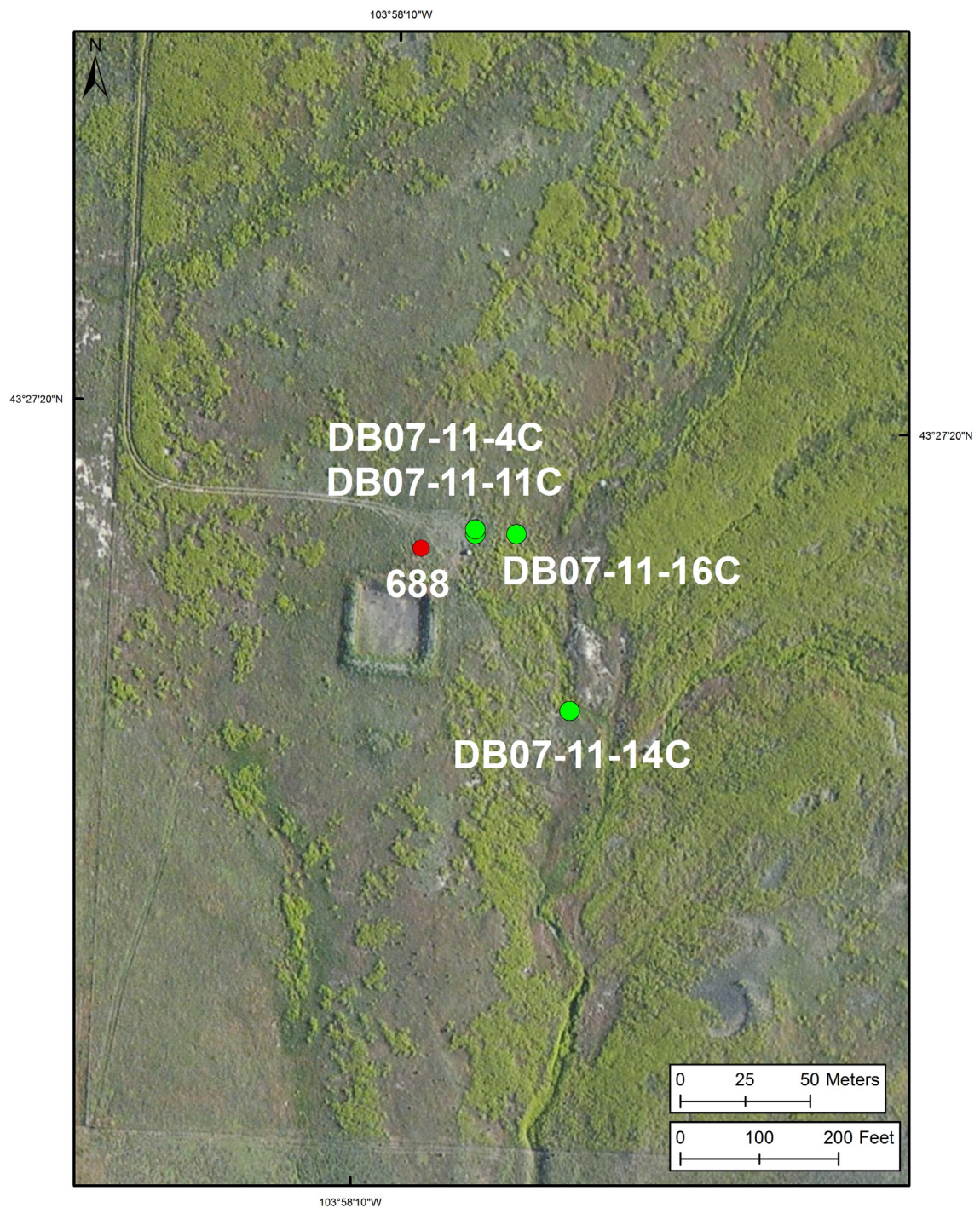


Figure 4. Expanded view of Burdock area with core locations. Location of well 688 is included for reference.

Dewey

Uranium occurs as discrete minerals, associated with vanadium, occluding pore space.

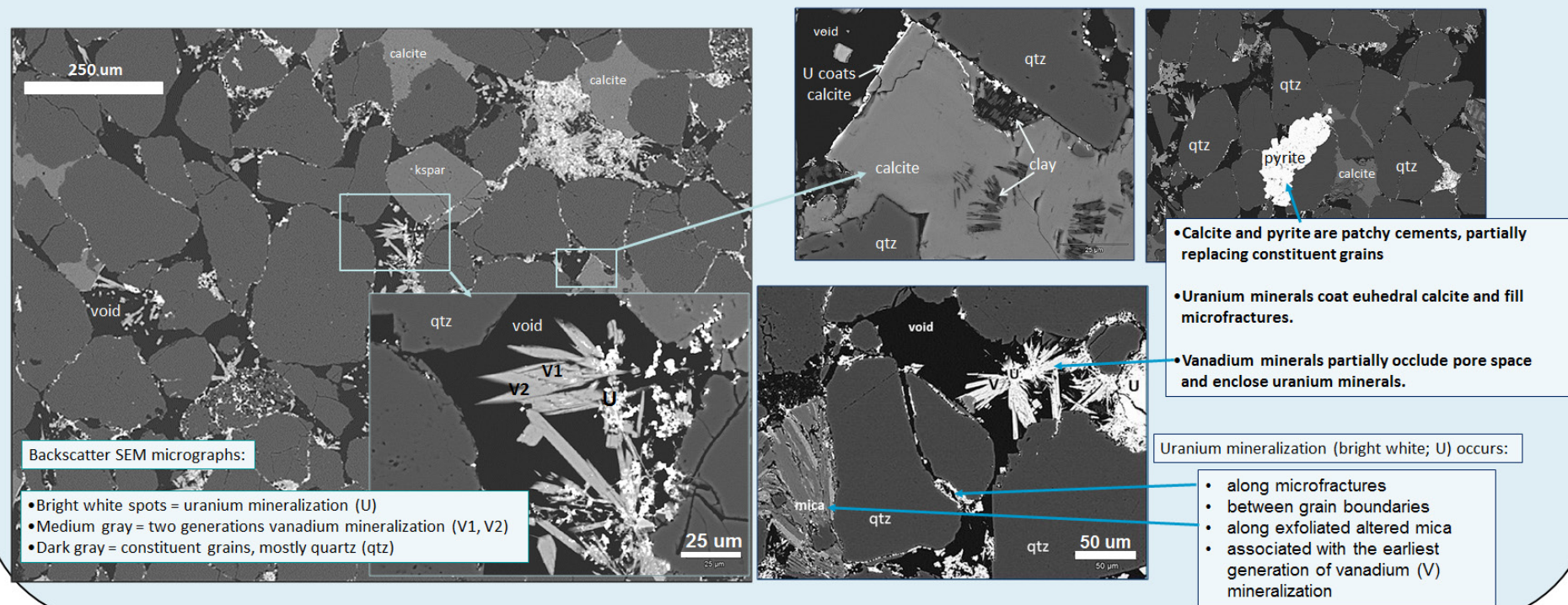


Figure 5. Summary of mineralogy from the Fall River Formation in the Dewey area.

Burdock

Uranium is adsorbed onto woody fragments and carbon/sulfur residues in stylolites.

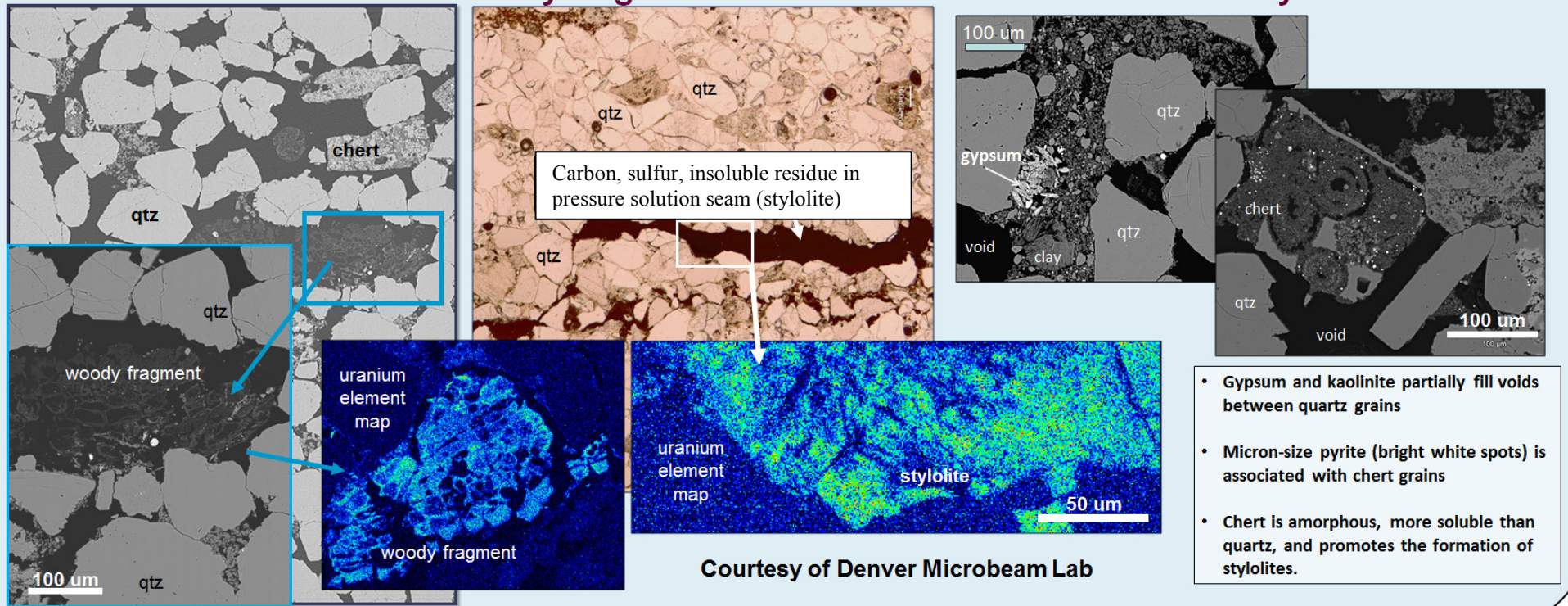
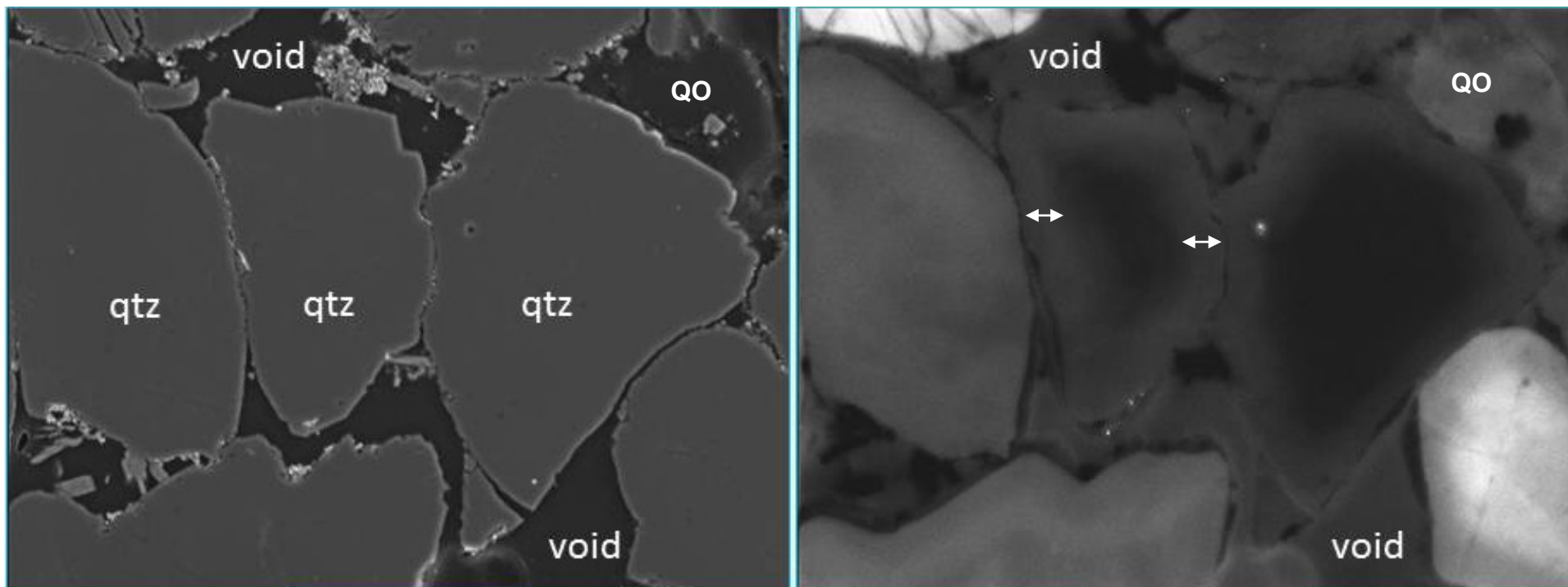


Figure 6. Summary of mineralogy from the Chilson Formation in the Burdock area. (qtz, quartz; μm , micrometers)



- **Constituent quartz grains show varying cathodoluminescence (CL) = different source areas and different internal chemistry**
- **Quartz overgrowths (QO) are dark = no CL; therefore a different generation of quartz cement**
- **Pale-gray halos (double arrows) within quartz grains suggest radiation damage rims**

Figure 7. Scanning electron microscopy (SEM, left) and cathodoluminescence (CL, right) images of the same constituent quartz (qtz) grains from the Fall River Formation in the Dewey area (sample 32-4C-4, 567'11" to 570'2"). Radiation damage halos (indicated by double arrows) suggest places that uranium was formerly present, but has since been locally remobilized. Grains are subrounded to angular, with the degree of rounding suggesting varying transport distance. The contrasting cathodoluminescence of the quartz grains are evidence of different geologic source terrains. Cathodoluminescence is dependent on trace metal content in the quartz. Aluminum is a common trace metal in quartz, and it varies on orders of magnitude. Note that the euhedral quartz overgrowths (QO) are nonluminescent; this clearly demonstrates that the overgrowths precipitated on these grains post deposition, and they lack luminescing trace metals.

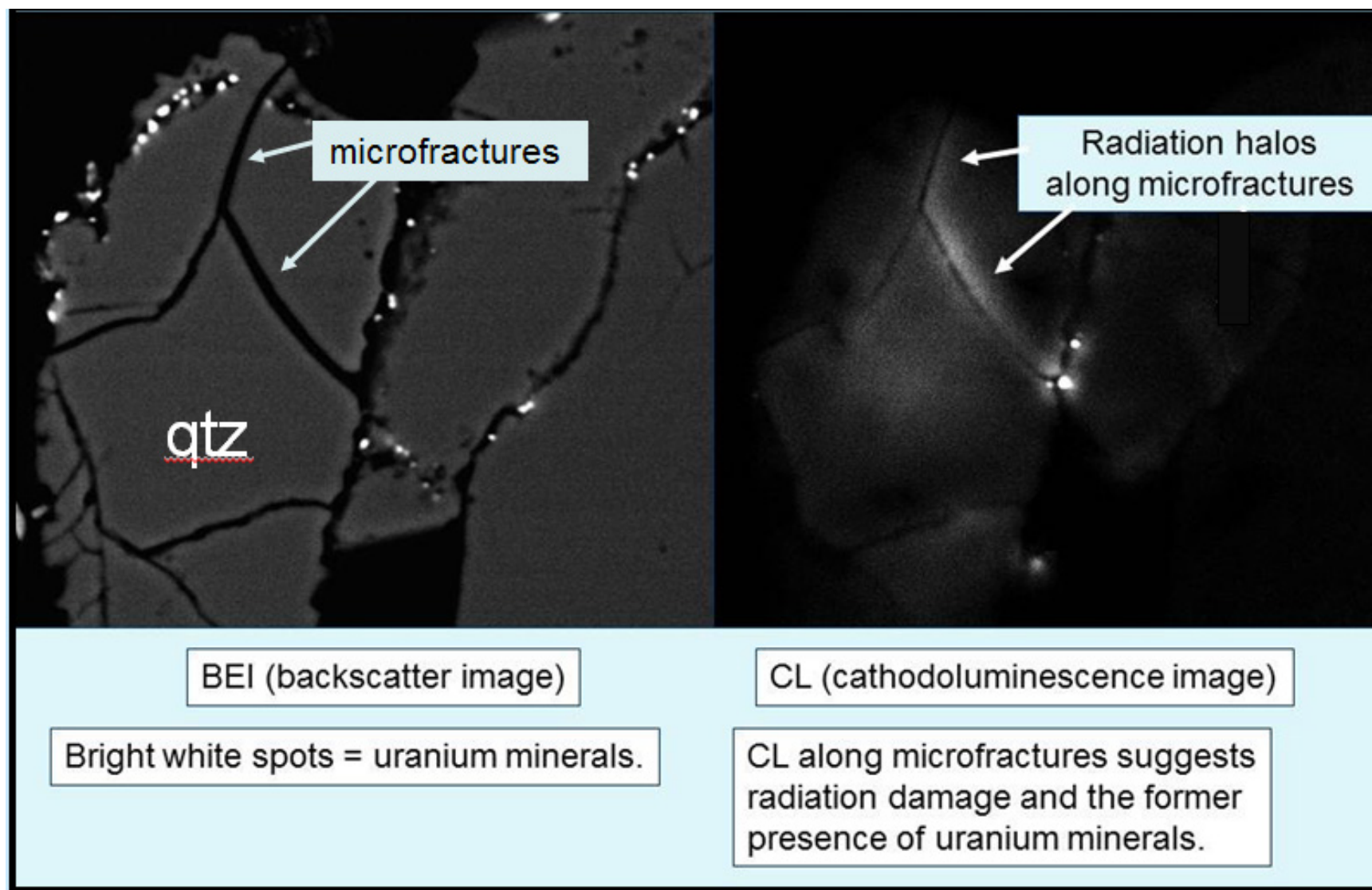
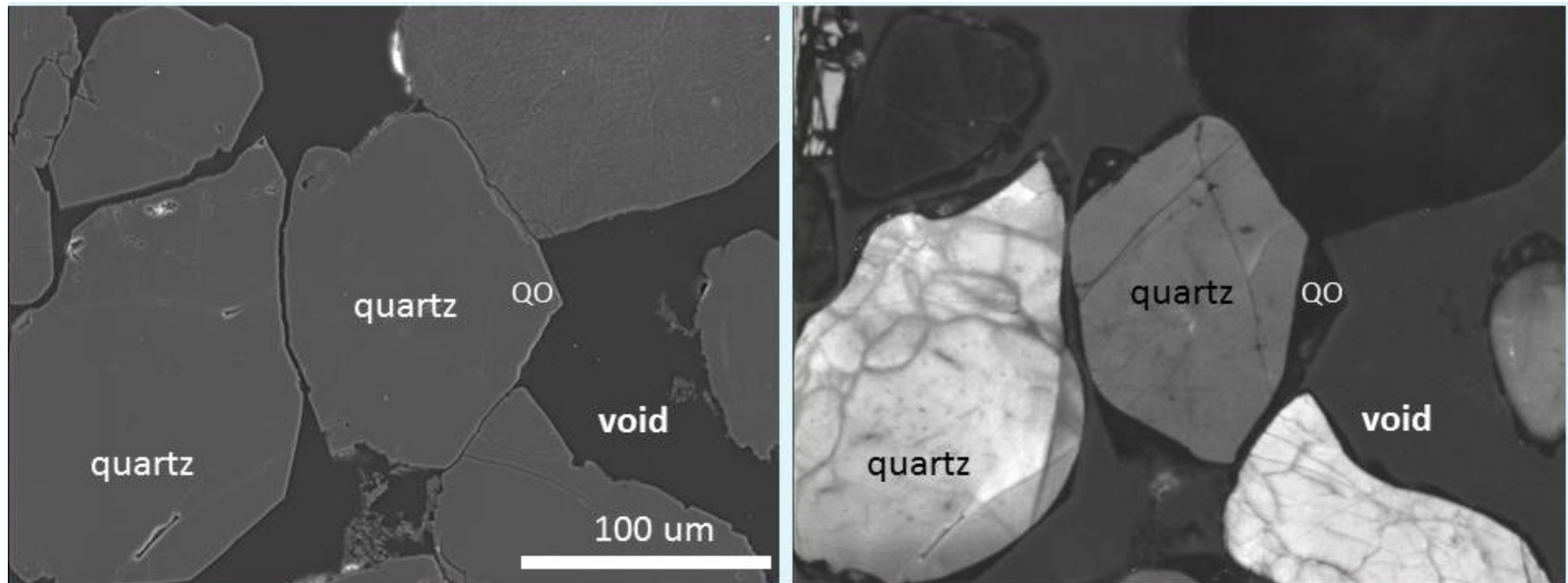


Figure 8. Scanning electron microscopy (SEM) backscatter (left) and cathodoluminescence (right) images of the same constituent quartz (qtz) grains from the Fall River Formation in the Dewey area (sample 32-4C-4, 567'11" to 570'2"). Microfractures exhibit radiation damage halos (white area along microfractures). Radiation damage halos suggest places that uranium was formerly present, but has since been locally remobilized.



- **Rounded to subangular quartz and feldspar grains; highly fractured**
- **Well-developed euhedral quartz overgrowths (QO)**
- **High porosity despite quartz overgrowths and stylolite development**

Figure 9. Scanning electron microscopy (SEM) backscatter (left) and cathodoluminescence (right) images of the same constituent quartz grains from the Chilson Formation in the Burdock area (sample 11-11C-6, 436'10.5" to 441'1"). Grains are subrounded to angular, with the degree of rounding suggesting varying transport distance. The contrasting cathodoluminescence of the quartz grains is evidence of different geologic source terrains. Cathodoluminescence is dependent on trace metal content in the quartz. Aluminum is a common trace metal in quartz, and it varies on orders of magnitude. Note that the euhedral quartz overgrowths (QO) are nonluminescent; this clearly demonstrates that the overgrowths precipitated on these grains post deposition, and they lack luminescing trace metals. (μm, micrometers)