

ANALYTICAL METHODS

Zircon U-Pb Dating

Zircon U-Pb dating was conducted using LA-ICPMS in Mineral and Fluid inclusion microanalysis lab, Geology Institute, Chinese Academy of Geological Sciences, Beijing. The NWR 193^{UC} laser ablation system (Elemental Scientific Lasers, USA) was equipped with Coherent Excistar 200 excimer laser and a Two Volume 2 ablation cell. The laser ablation system was coupled to an Agilent 7900 ICPMS (Agilent, USA).

The detailed analytical methods have been described by [Yu et al. \(2019\)](#). LA-ICPMS tuning was performed using a 50-micron diameter line scan at 3 $\mu\text{m/s}$ on NIST 612 at $\sim 3.5 \text{ J/cm}^2$ with repetition rate 10 Hz. Zircons were mounted in epoxy discs, polished to expose the grains, cleaned ultrasonically in ultrapure water, and then cleaned again before the analysis using AR-grade methanol. The analysis used a 30 μm diameter spot at 8 Hz and a fluence of 2 J/cm^2 . Iolite software package was used for data reduction ([Paton et al., 2010](#)). Zircon 91500 and GJ-1 were used as primary and secondary reference materials, respectively. Typically, 35-40 seconds of the sample signals were acquired after 20 seconds of gas background measurement. Using the exponential function to calibrate the downhole fractionation ([Paton et al., 2010](#)). The measured ages of the 91500 ($1061.5 \pm 3.2 \text{ Ma}$, 2σ) and GJ-1 ($604 \pm 3 \text{ Ma}$, 2σ) agree well with the accepted age.

Whole-Rock Major and Trace Element Analyses and Sr-Nd Isotope Analysis

After the removal of weathered surfaces, samples were crushed, and small rock chips were pulverized into powder using an agate mortar to a grain size of < 200 mesh. Whole-rock geochemical analyses and Sr-Nd isotopic compositions were performed at the Wuhan SampleSolution Analytical Technology Co., Ltd., Wuhan, China. The major elements were analyzed by an X-ray fluorescence spectrometer (XRF) produced using the methods of [Norrish and Hutton \(1969\)](#). Ferrous iron was determined by the wet chemical titration method. Trace element analysis of whole rocks was conducted on an Agilent 7700e ICP-MS following the method of [Liang and Grégoire \(2000\)](#). The analytical uncertainties for major elements are within 1-5%. In-run analytical precision for most trace elements is better than 5%. The detailed chemical separation and isotopic measurement procedures are described in [Wu et al. \(2005\)](#). The $^{87}\text{Sr}/^{86}\text{Sr}$ ratios were normalized to $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$ and $^{143}\text{Nd}/^{144}\text{Nd}$ ratios to $^{146}\text{Nd}/^{144}\text{Nd} = 0.7219$. Total procedural blanks were < 300 pg for Sr and < 100 pg for Nd and the estimated analytical uncertainties of $^{147}\text{Sm}/^{144}\text{Nd}$ and $^{87}\text{Rb}/^{86}\text{Sr}$ ratios were < 0.5%.

Zircon Hf-O Isotopic Analysis

Experiments of *in situ* Hf isotope ratio analysis were conducted using a Neptune Plus MC-ICP-MS (Thermo Fisher Scientific, Germany) in combination with a Geolas HD excimer ArF laser ablation system (Coherent, Göttingen, Germany) that were hosted at the Wuhan Sample Solution Analytical Technology Co., Ltd, Hubei, China. All data were acquired on zircon in single-spot ablation mode at a spot size of 44 μm . The energy density of laser ablation used in this study was $\sim 10.0 \text{ J cm}^{-2}$. The detailed operating conditions for the laser ablation system, the MC-ICP-MS instrument, and the analytical method are the same as [Hu et al. \(2012\)](#) described.

The O isotopic compositions of the dated zircons were determined with the SHRIMP II multiple collector ion microprobe. A $\sim 7\text{nA}$ primary $^{133}\text{Cs}^+$ beam was accelerated to 15keV, and focused into a spot $\sim 25\mu\text{m}$ diameter on the sample surface coated with 10nm Au. In this study, analyses of in-house zircon standard TEMORA-2 ($\delta^{18}\text{O}_{\text{VSMOW}} = +8.20 \pm 0.02\text{‰}$; [Black et al. 2004](#), [Valley, 2003](#)) embedded in each epoxy mount are interspersed with every three or four unknown sample measurements. The measured $^{18}\text{O}/^{16}\text{O}$ on the standard were used to correct for instrumental drift and instrumental fractionation including mass discrimination and relative gain between electrometers. Analytical uncertainties of each unknown sample analysis combine the internal run errors (s.e., typically 0.1–0.4‰) and the reproducibility of the standard zircon analyses (2 s.d. after the instrumental drift correction), added in quadrature. Analytical accuracy and precision are indicated by the results from the zircon standard 91500 functioning as a secondary reference material.

REFERENCES CITED

- Black, L.P., Kamo, S.L., Allen, C.M., Davis, D.W., Aleinikoff, J.N., Valley, J.W., Mundil, R., Campbell, Campbell, I.H., Korsch, R.J., Williams, I.S., and Foudoulis, C., 2004, Improved $^{206}\text{Pb}/^{238}\text{U}$ microprobe geochronology by the monitoring of a trace-element-related matrix effect; SHRIMP, ID-TIMS, ELA-ICP-MS and oxygen isotope documentation for a series of zircon standards: *Chemical Geology*, v. 205, p. 115–140, <https://doi.org/10.1016/j.chemgeo.2004.01.003>.
- Hu, Z.C., Liu, Y.S., Gao, S., Liu, W., Yang, L., Zhang, W., Tong, X., Lin, L., Zong, K.Q., Li, M., Chen, H., and Zhou, L., 2012, Improved in situ Hf isotope ratio analysis of zircon using newly designed X skimmer cone and Jet sample cone in combination with the addition of nitrogen by laser ablation multiple collector ICP-MS: *Journal of Analytical Atomic Spectrometry*, v. 27, p. 1391–1399, <https://doi.org/10.1039/C2JA30078H>
- Liang, Q., and Grégoire, D.C., 2000, Determination of trace elements in twenty six Chinese geochemistry reference materials by inductively coupled plasma-mass spectrometry: *Geostandards Newsletter*, v. 24, no. 1, p. 51–63, <https://doi.org/10.1111/j.1751-908X.2000.tb00586.x>.
- Norrish, K., and Hutton, J.T., 1969, An accurate X-ray spectrographic method for the analysis of a wide range of geological samples: *Geochimica et cosmochimica acta*, v. 33, no. 4, p. 431–453, [https://doi.org/10.1016/0016-7037\(69\)90126-4](https://doi.org/10.1016/0016-7037(69)90126-4).
- Paton, C., Woodhead, J.D., Hellstrom, J.C., Hergt, J.M., Greig, A., and Maas, R., 2010,

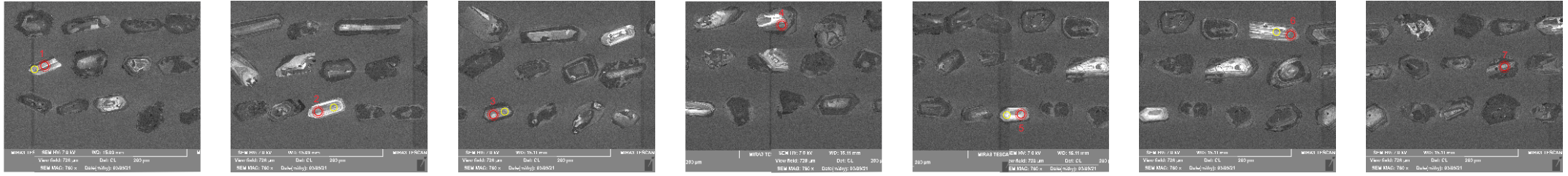
Improved laser ablation U-Pb zircon geochronology through robust downhole fractionation correction: *Geochemistry, Geophysics, Geosystems*, v. 11, Q0AA06, <https://doi.org/10.1029/2009GC002618>.

Valley, J.W., 2003, Oxygen isotopes in zircon: *Reviews in Mineralogy and Geochemistry*, v. 53, p. 343–385, <https://doi.org/10.2113/0530343>.

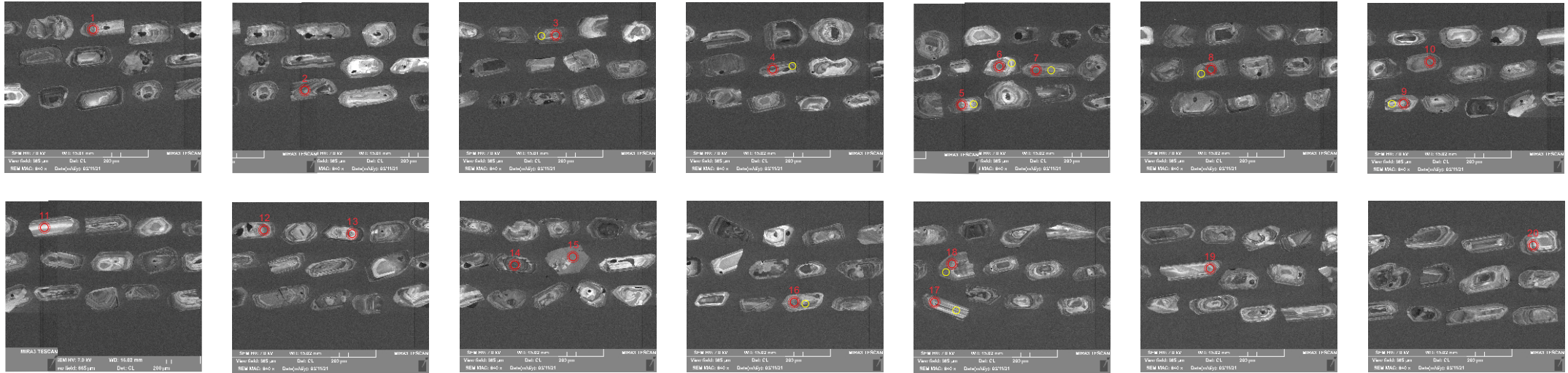
Wu, F.Y., Yang, J.H., Wilde, S.A., and Zhang, X.O., 2005, Geochronology, petrogenesis and tectonic implications of Jurassic granites in the Liaodong Peninsula, NE China: *Chemical Geology*, v. 221, no. 1-2, p. 127–156, <https://doi.org/10.1016/j.chemgeo.2005.04.010>.

Yu, C., Yang, Z.M., Zhou, L.M., Zhang, L.L., Li, Z.Q., Zhao, M., Zhang, J.Y., Chen, W.Y., and Suo, M.S., 2019, Impact of laser focus on accuracy of U-Pb dating of zircons by LA-ICPMS: *Mineral Deposits*, v. 38, p. 21–28.

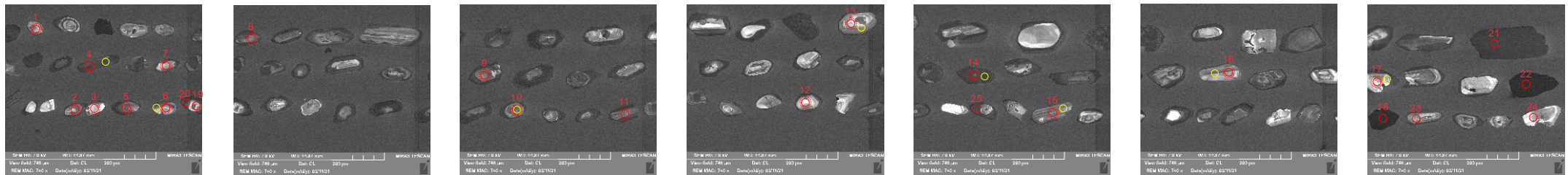
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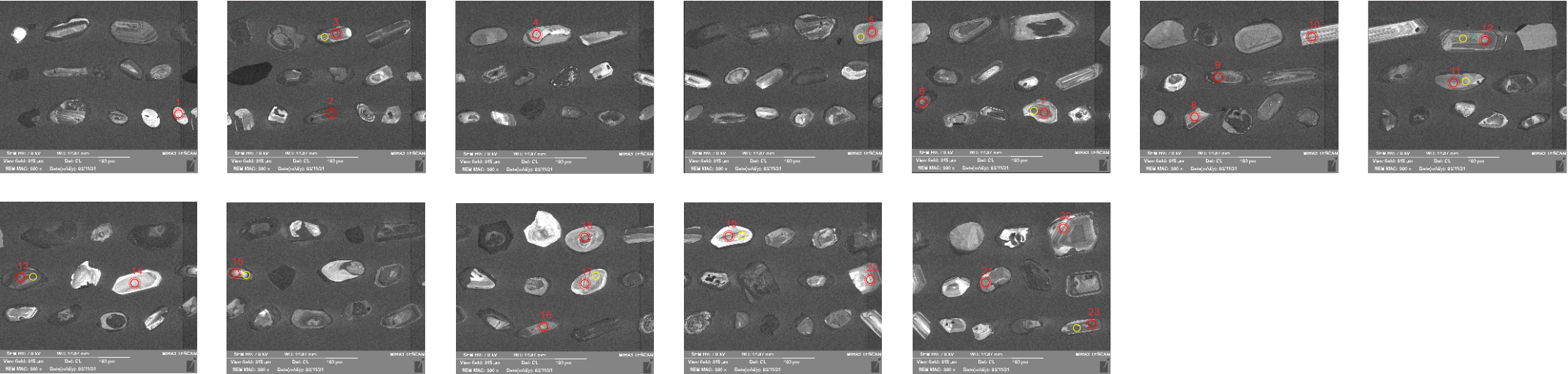


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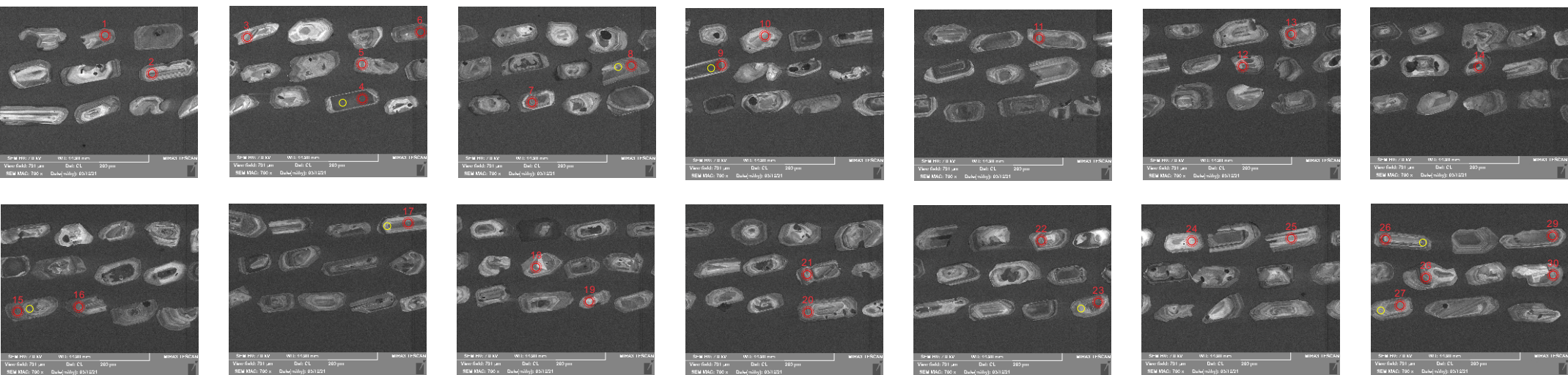


red circle = 30 micron diameter laser ablation pit (U-Pb)
 red dashed circle = 44 micron diameter laser ablation pit (Lu-Hf)
 yellow circle = 25 micron diameter laser ablation pit (O)

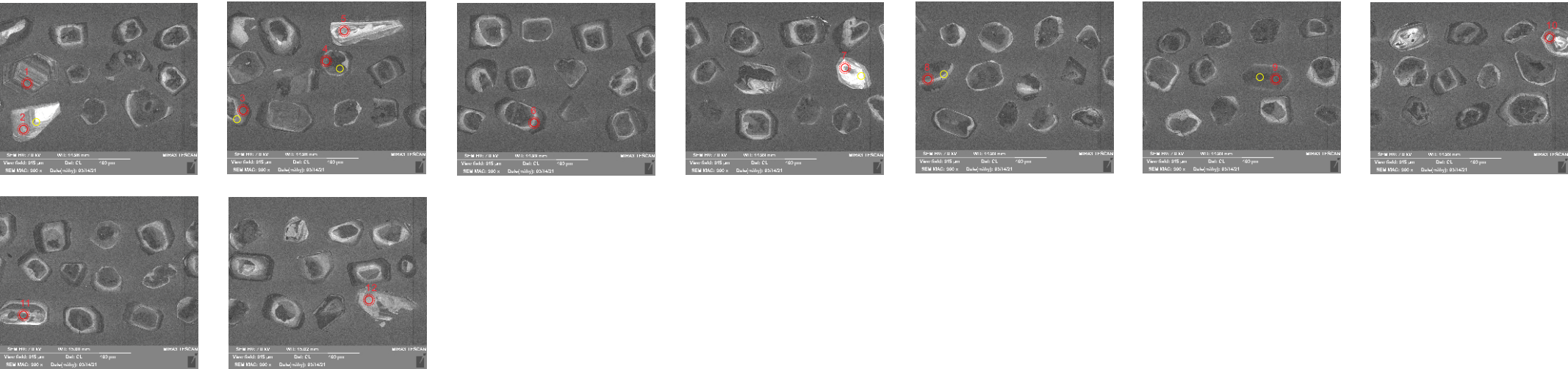
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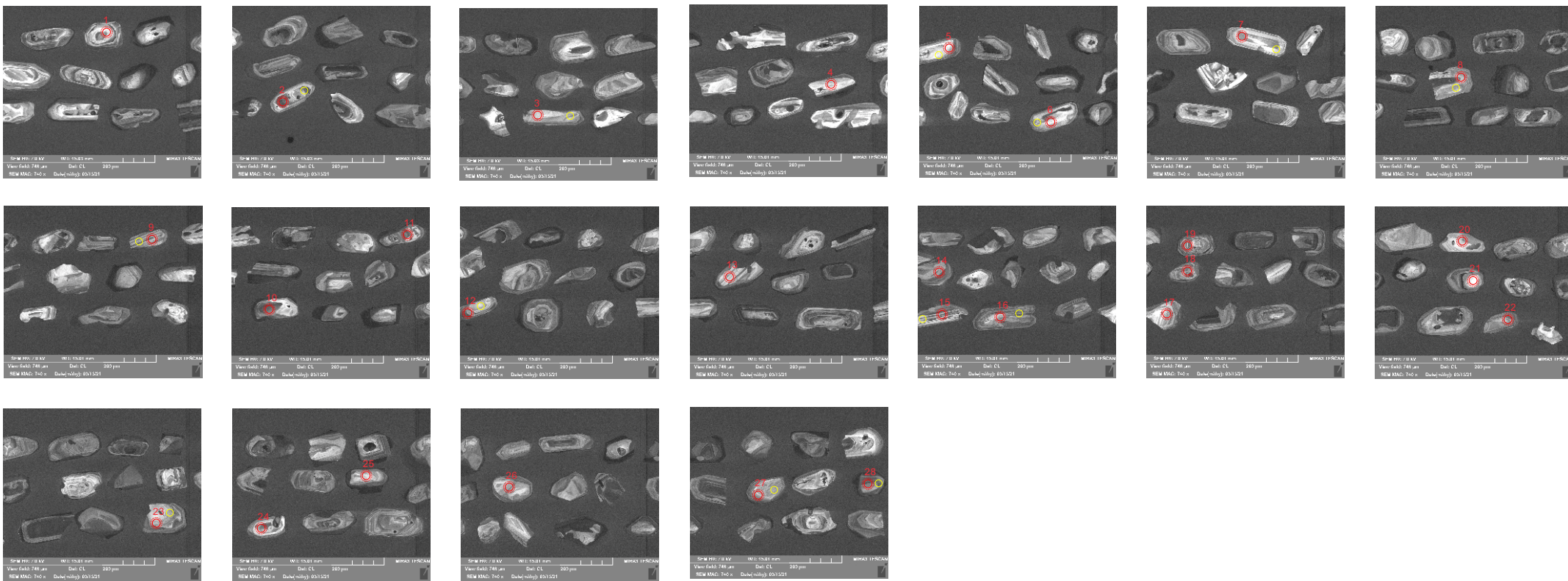
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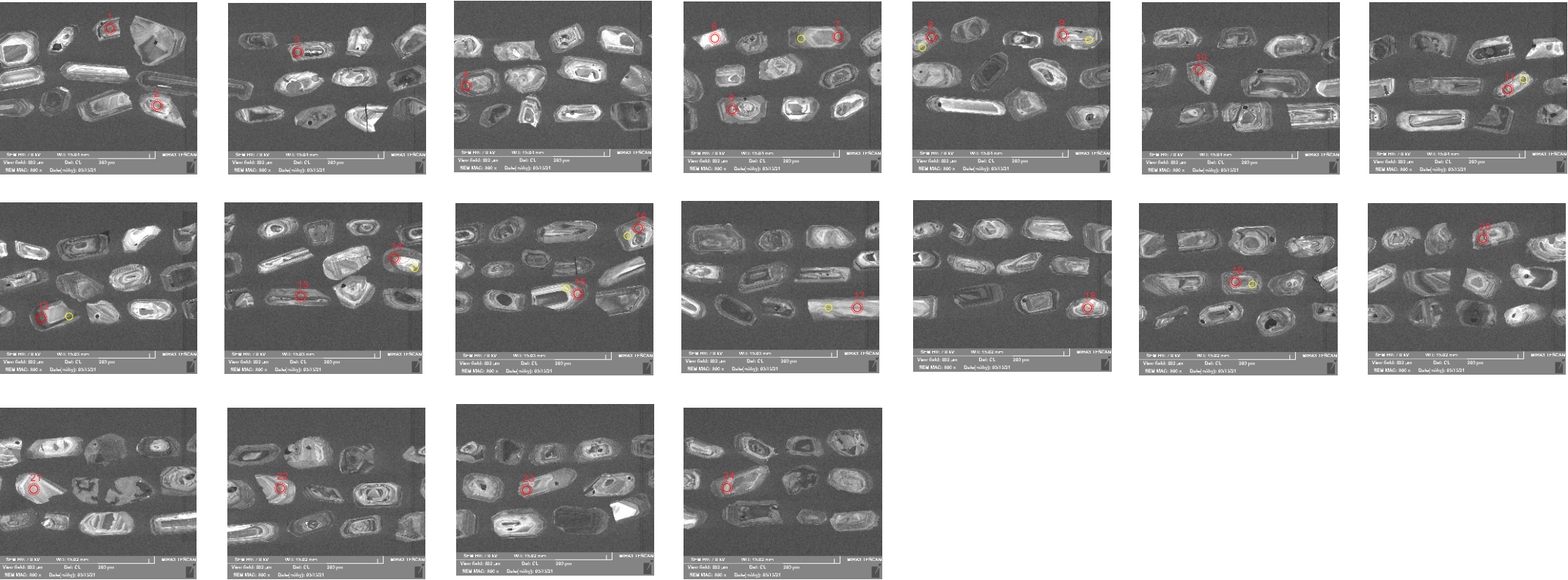
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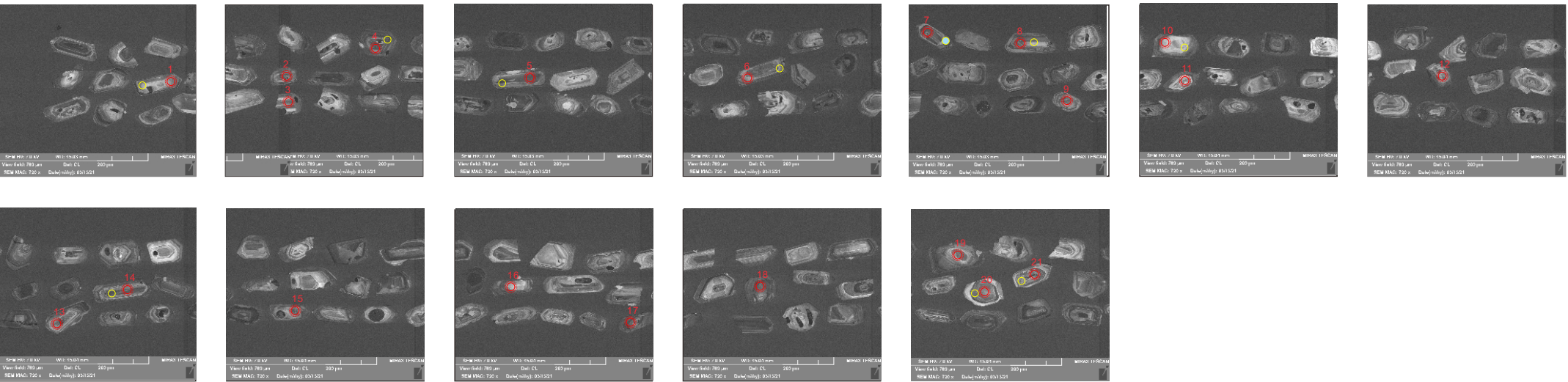
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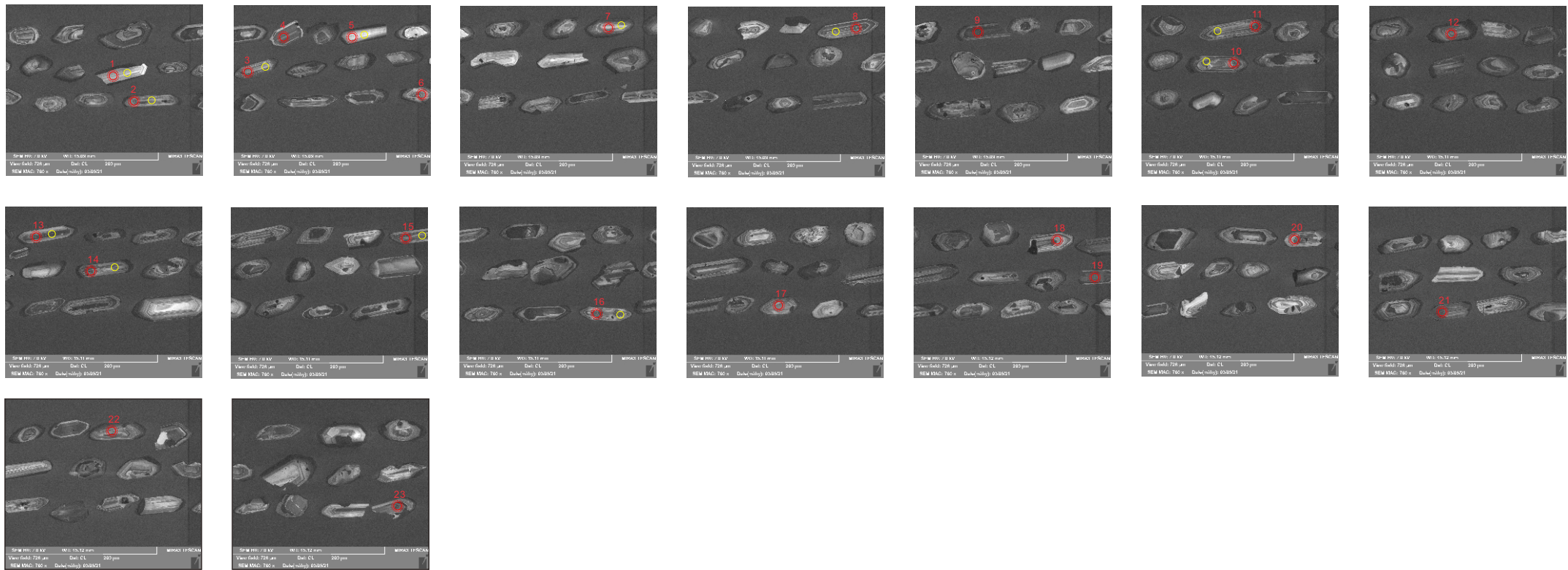
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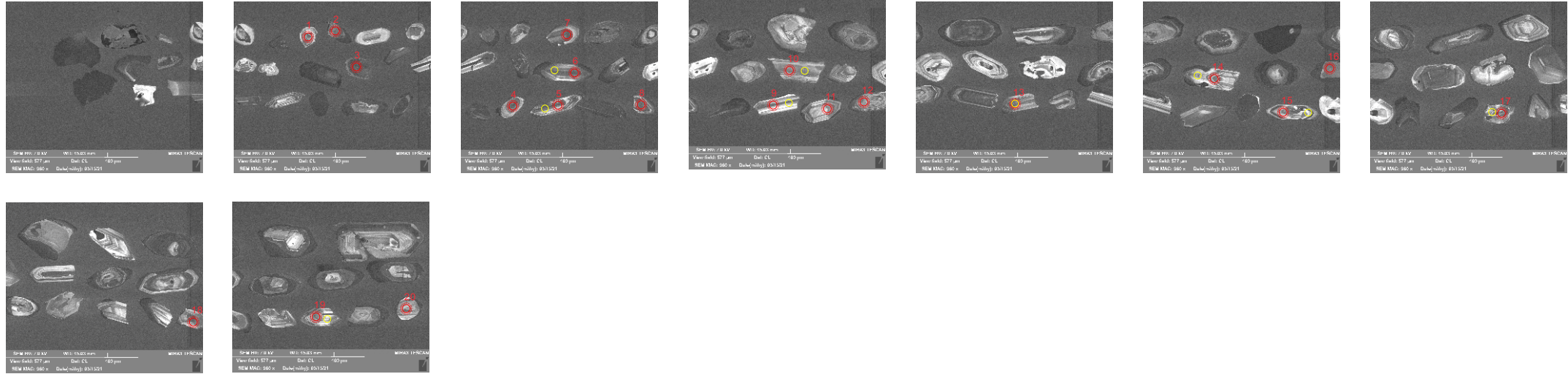
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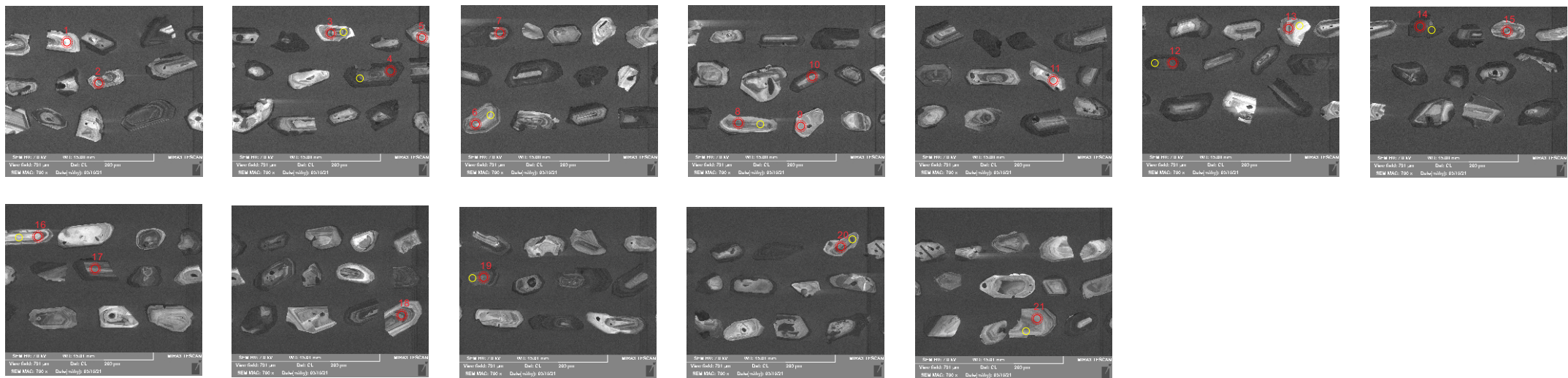
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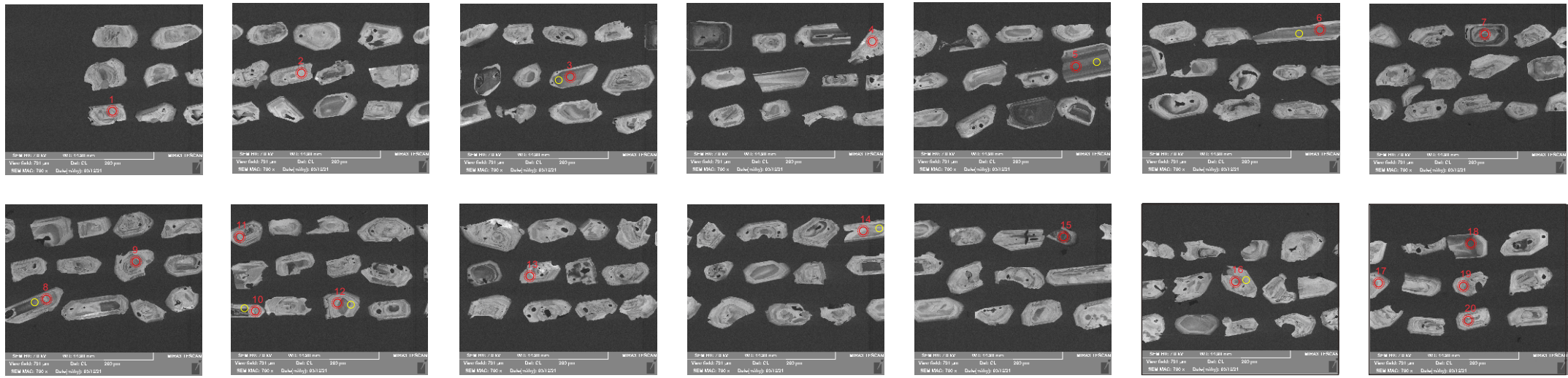
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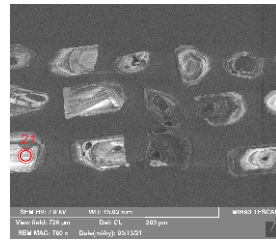
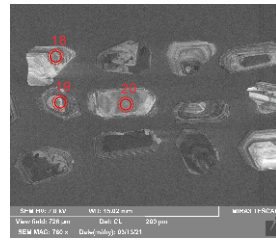
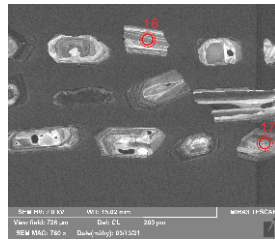
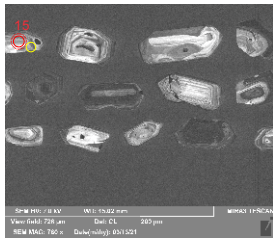
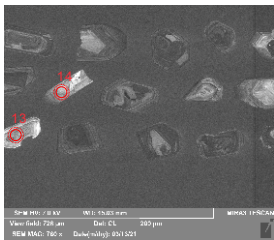
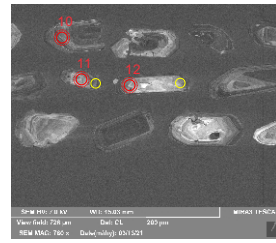
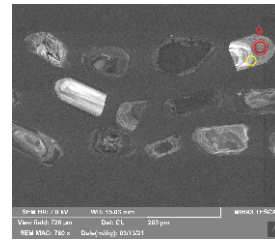
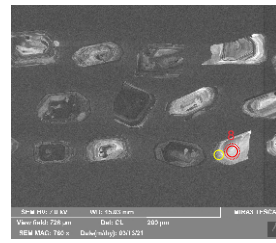
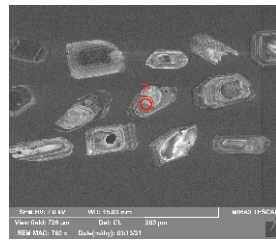
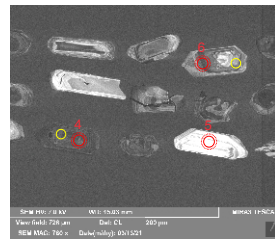
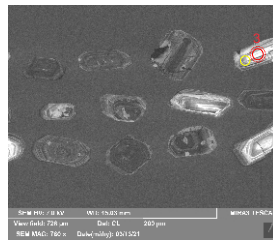
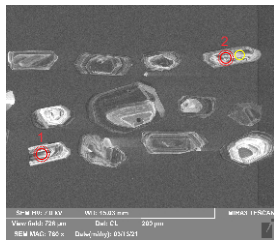
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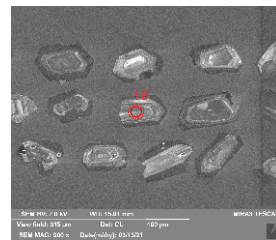
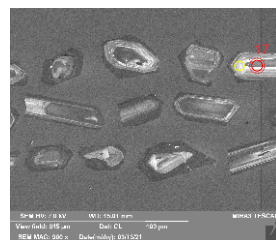
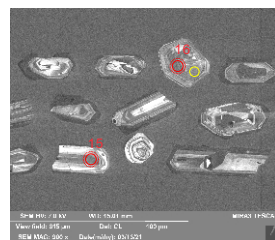
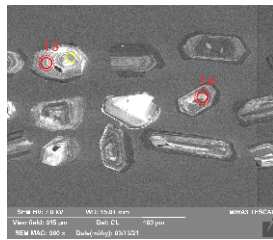
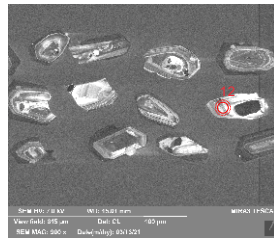
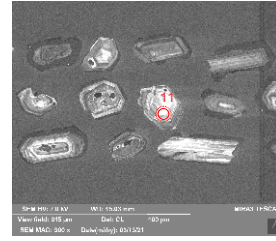
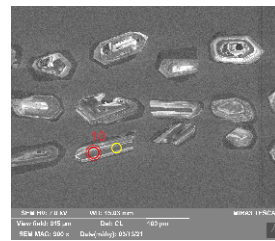
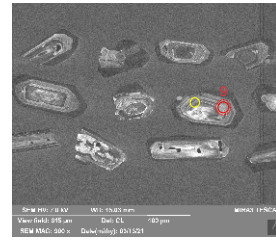
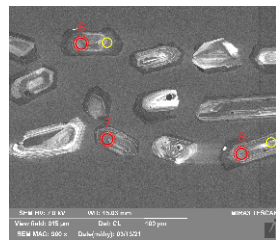
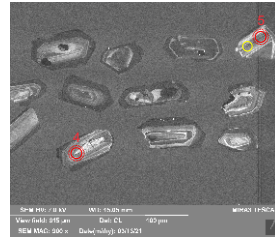
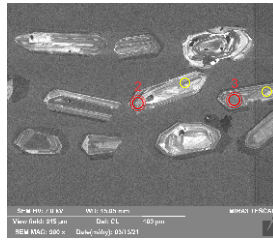
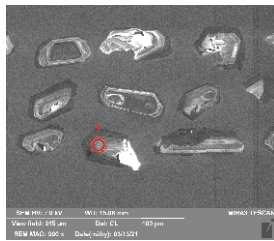
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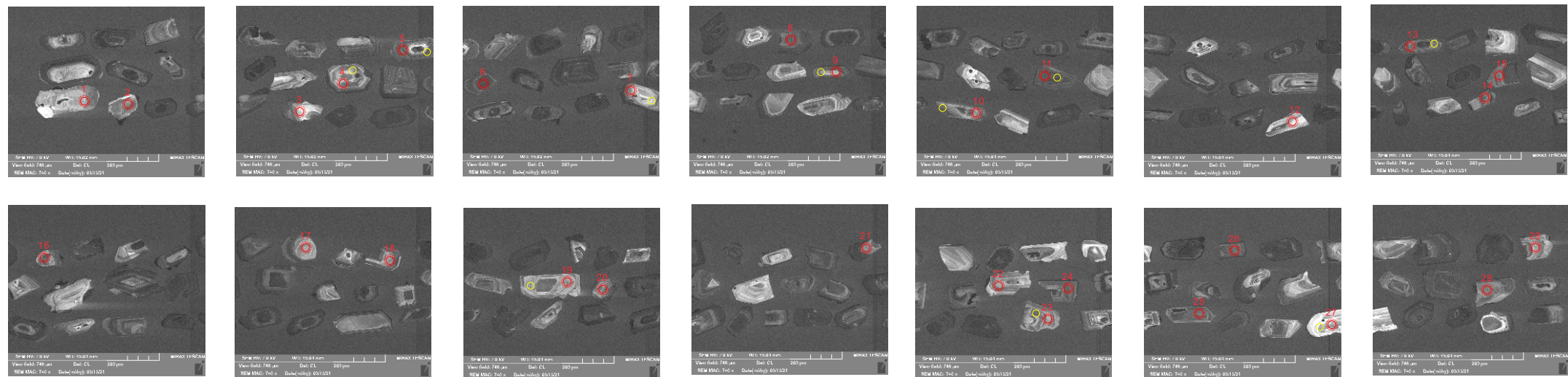
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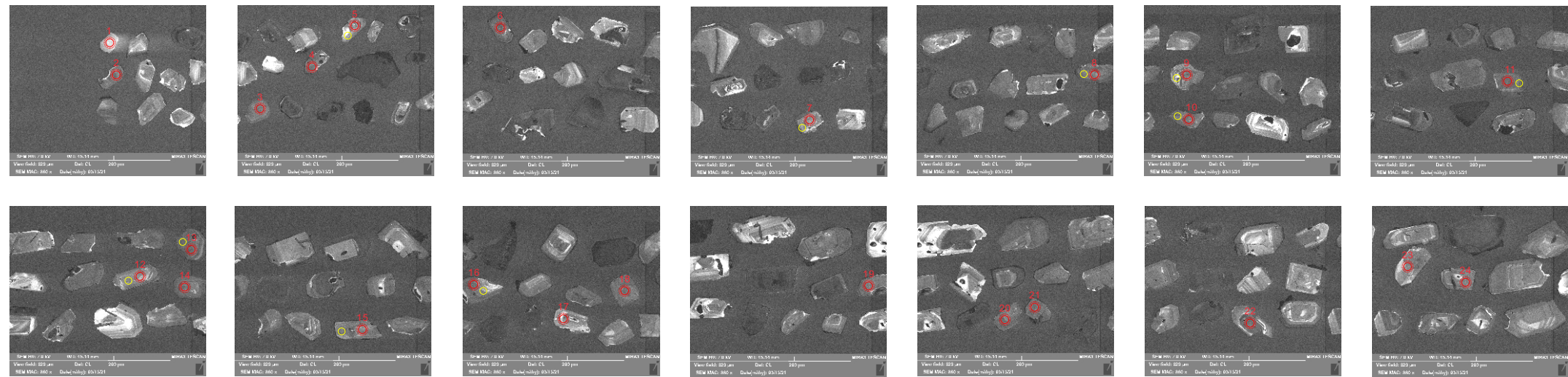
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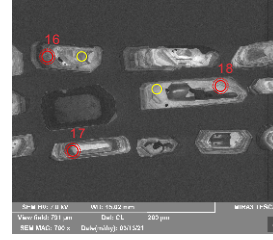
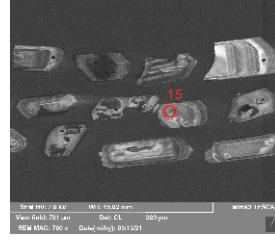
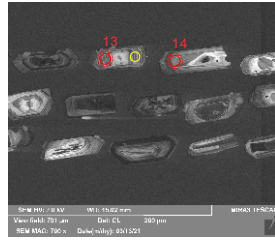
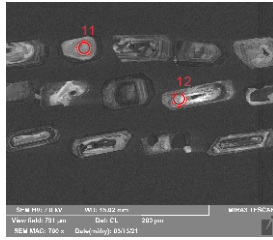
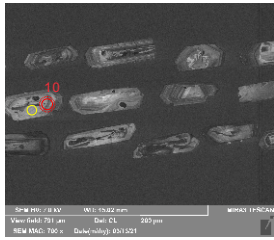
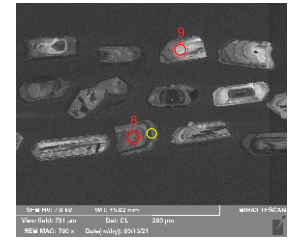
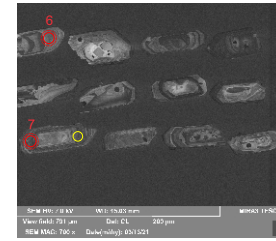
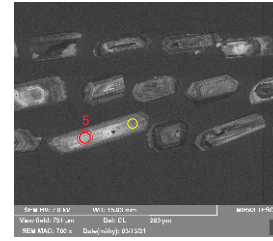
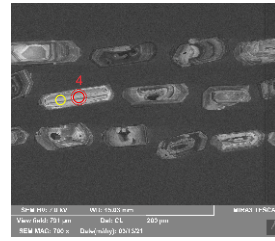
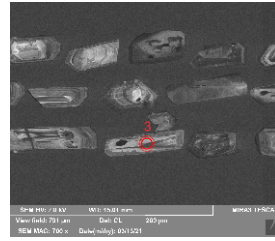
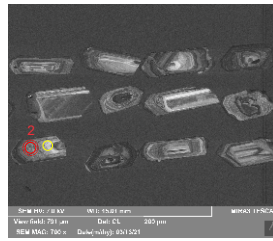
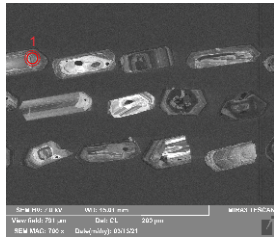
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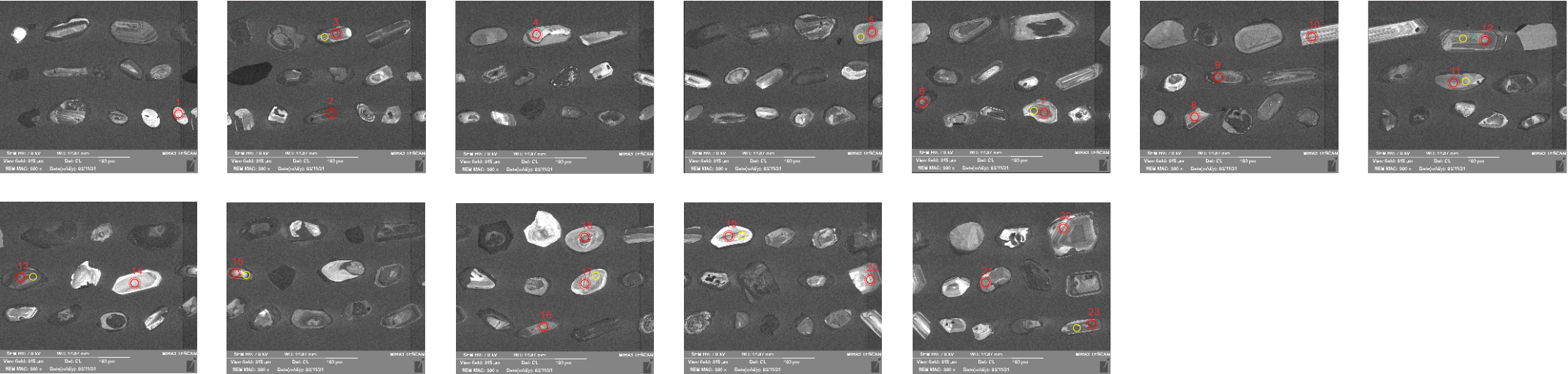
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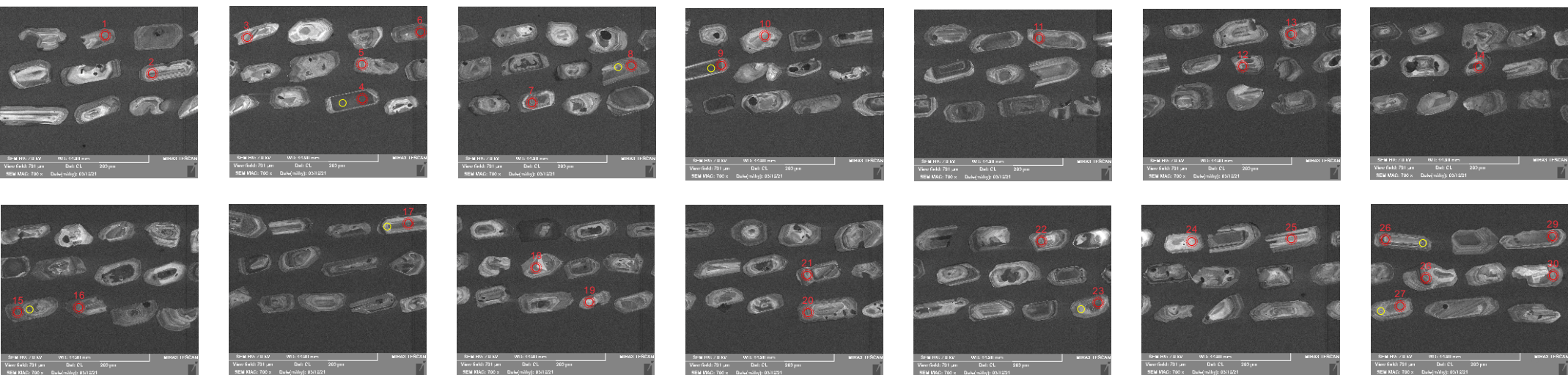
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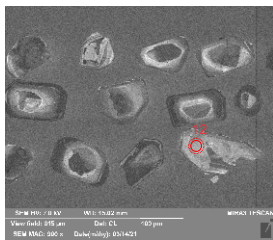
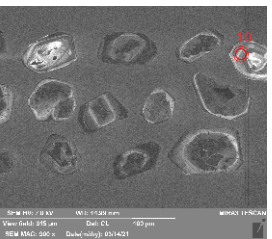
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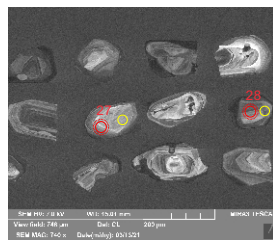
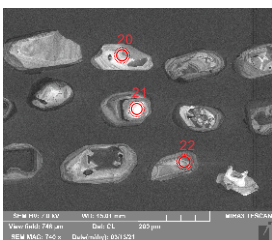
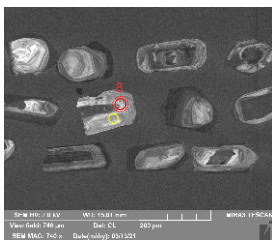
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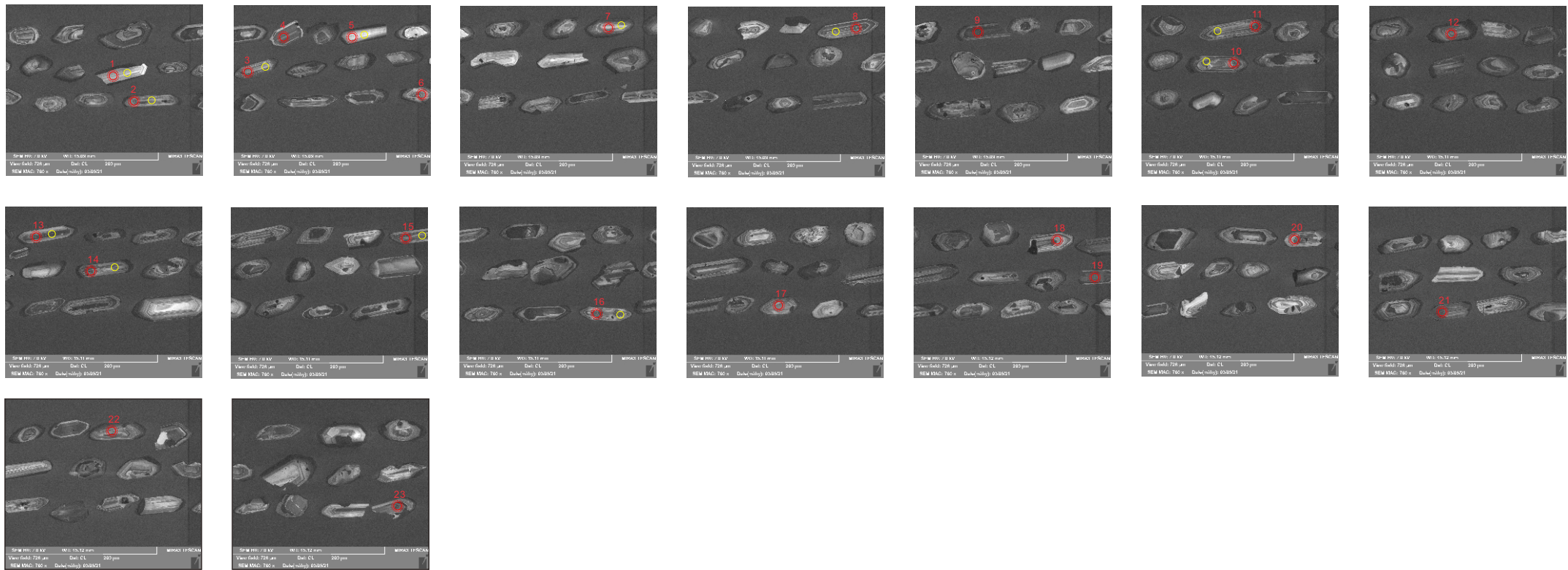
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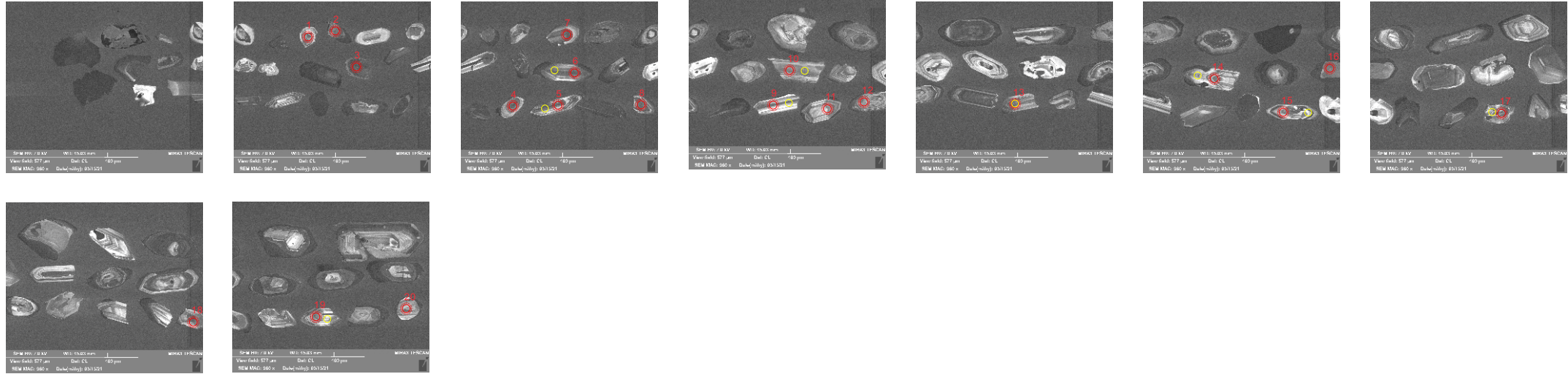
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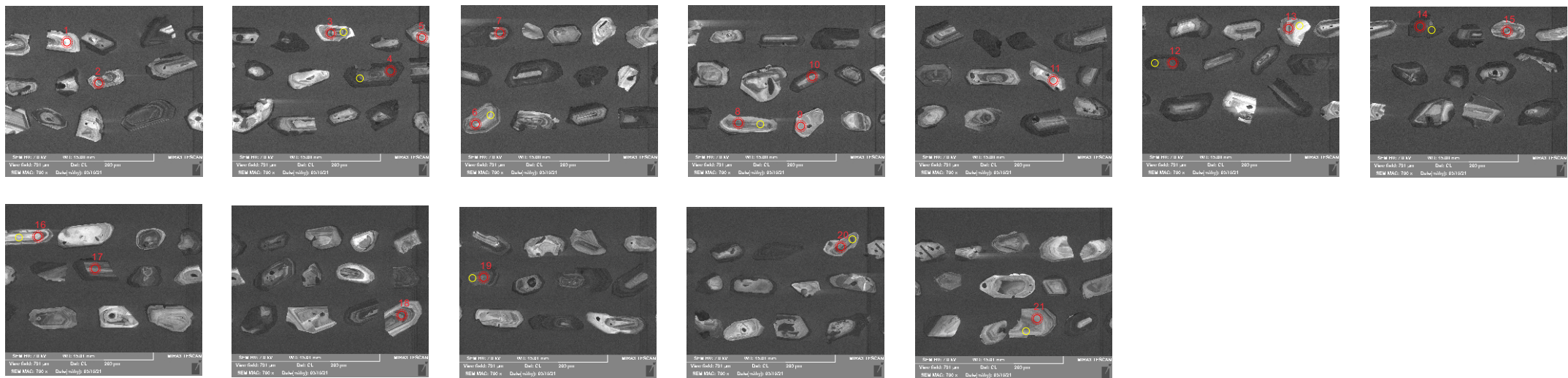
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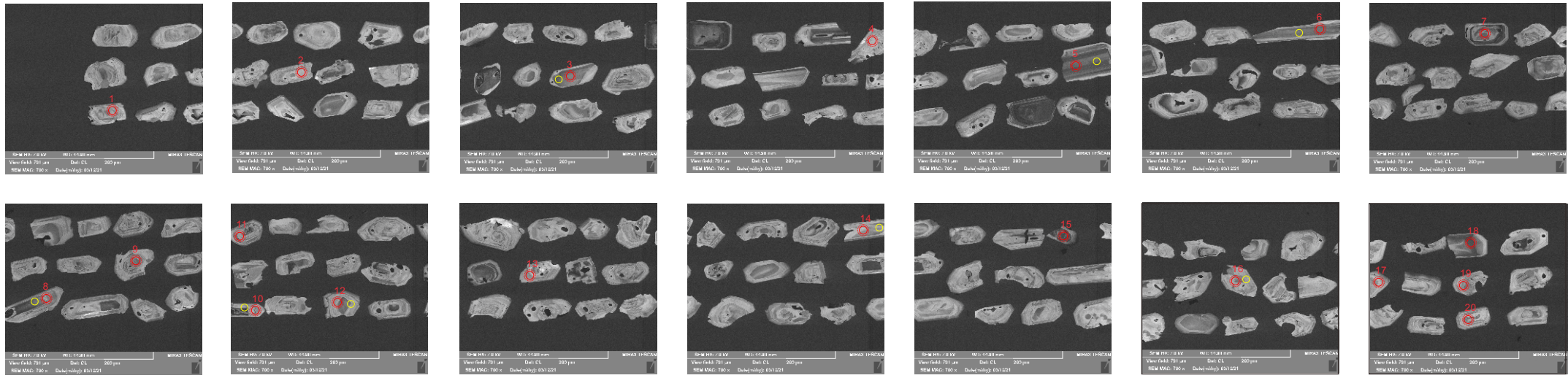
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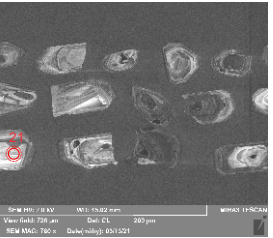
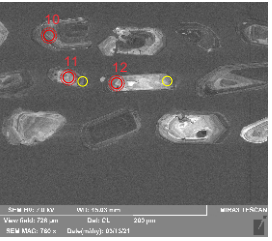
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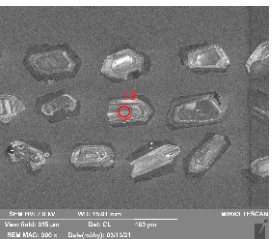
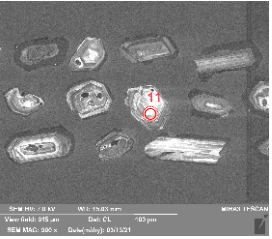
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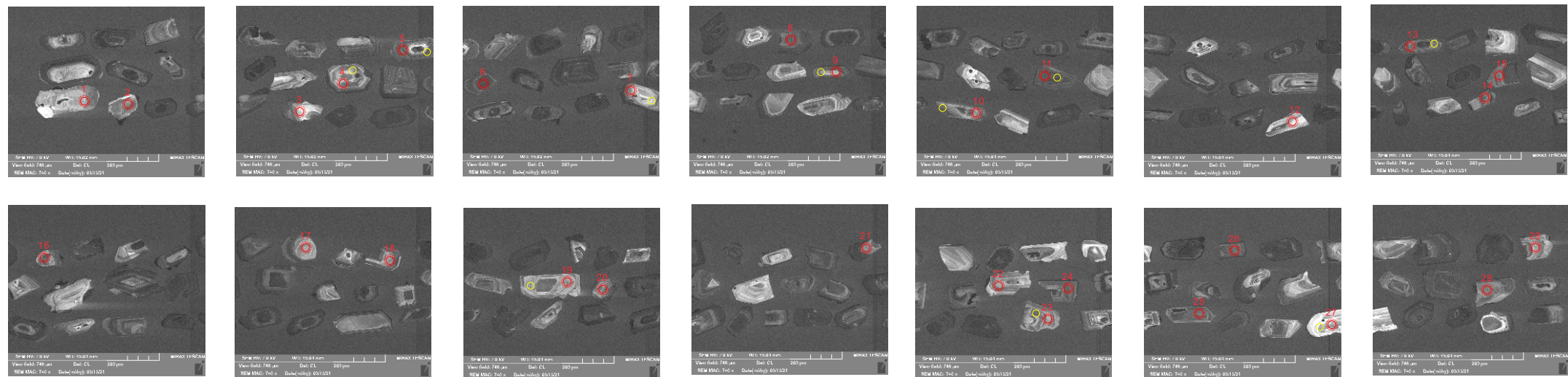
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20NL57-1



20NL61-1



20NL62-1

