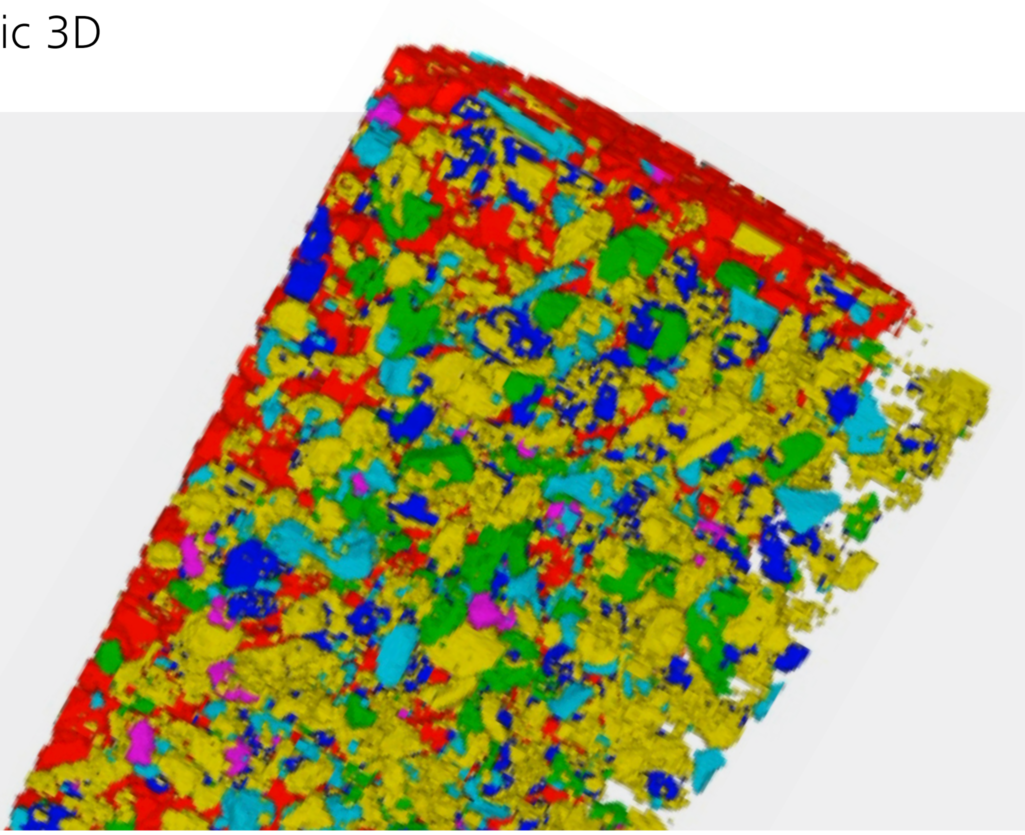
Application Note

Whole-particle Liberation Studies

ZEISS Mineralogic 3D



Seeing beyond

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Light microscopes (LM) and scanning electron microscopes (SEM) are routinely used for mineral liberation studies – a measure of how accessible minerals are to the recovery process, and of the quality of the feed and the subsequent processing. An unavoidable requirement for the analysis is that the sample be prepared in a manner suitable to the microscope in use. Sample preparation is not simply an inconvenience imposed by lab equipment but is integral to the quality of the results and the inferences we may make. A poorly prepared sample may suffer density stratification, plucking, doming, agglomeration, pitting, insufficient surface exposure, etc. As well as these mechanical sample preparation issues, the analysis is limited to the exposed surface of the mineral and therefore influenced by stereological limitations and the assumption that the exposed surface is representative of the entire sample and a valid description of the particles present. All these issues conspire to produce analyses for which the modal mineralogy, grain size, liberation, and associations information are compromised, regardless of whether the analysis is performed on the LM or with automated mineralogy (AM) software on an SEM. Over time, techniques have developed to ensure we present a representative sample for analysis that is devoid of sample preparation artifacts. For example, the addition of graphite to the sample to encourage particle separation and discourage density stratification, and the analyses of sufficient particles to confidently describe a set of average particles and minimize the limitations imposed by stereology (the interpretation of 3D samples based on 2D information).

In all, sample preparation is a meticulous and time-consuming activity, taking longer than a day to complete, commencing with the collection of the material to be analyzed and progressing through to the final stages of setting in a resin block, grinding, polishing, and coating with a conductive layer. None of the steps can be circumvented if we are to guarantee trustworthy data.

In the following text is a description of paradigm shift in automated mineralogy that overcomes the issues of attempting to understand a 3D world by analyzing it on 2D surfaces.

Mineralogic 3D – Automated Mineralogy

ZEISS Mineralogic 3D applies 3D X-ray microscopy techniques and deep learning algorithms to execute automated mineralogy analyses in three dimensions (fig 1) that provide particle identification, mineral classification, and data outputs including liberation and association measurements.

Analyzing in 3D has three immediate benefits:

1. The sample preparation process is greatly simplified
2. There are no stereological assumptions as every grain is viewed in full
3. The time to actionable data is greatly reduced

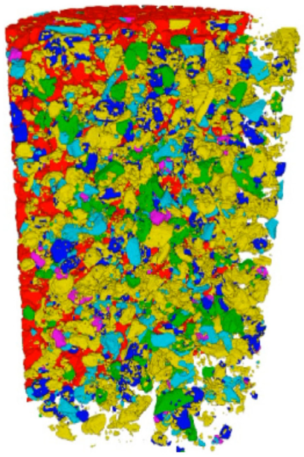


Figure 1 *Analysis of a comminuted ore with the 3D X-ray microscope reveals the true mineral liberation with no mineral hidden by lack of exposure.*

Analytical Considerations

Sample Preparation

Unlike the rigorous sample preparation workflow that must be followed to ensure we obtain valid data from an SEM analysis of a comminuted sample, sample preparation for Mineralogic 3D analysis is rather straightforward. All that is needed is a suitable container or tube to hold the sample and a stopper at either end to help keep it in place. A step of complexity has been introduced by the development of a technique that allows for several samples to be mounted in the same sample carrier (tube) but this falls far short of the intricacies of graphite addition, mix stirring, setting, grinding, and polishing expected for the SEM analysis. The result is a simplification of the workflow and a time savings of several hours.

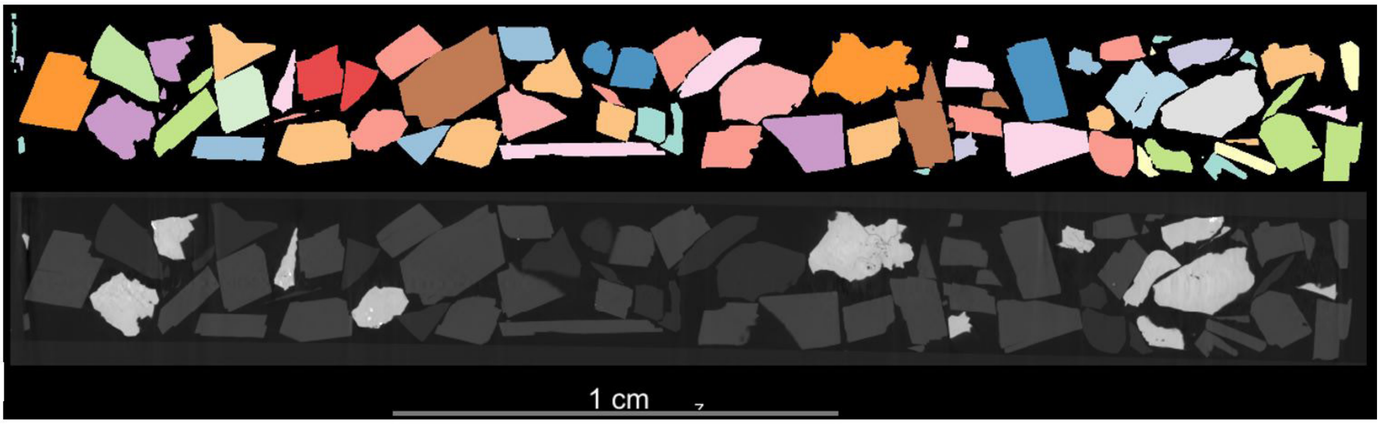


Figure 2 Longitudinal cross-section of a 3D scan of ground material. The coarseness of the particles provides a clearer example of the ability of the machine learning protocol to automatically identify and separate particles from each other.

The addition of graphite to the sample acts to both keep particles apart, aiding the assessment of liberation and associations, and to deter gravitational settling in the samples. Since Mineralogic 3D views the entire sample, including minerals locked within the particles, gravitational settling is not a concern. Particle separation is elegantly dealt with by the application of machine learning protocols for segmentation that identify each individual particle as independent from other particles that may be in contact with it (Figure 2).

Mineral Classification and Time to Actionable Data

Making full use of the imaging capability of the ZEISS Xradia X-ray microscopes, enhanced by DeepRecon noise reduction deep learning algorithms, Mineralogic 3D automatically classifies the mineralogy of the sample based on attentuation measurements (Figure 3). The ability to classify the mineralogy in tomographic scans is unique and, when combined with the morphological measurements of the 3D reconstructed entities, allows the calculation of standard mining relevant outputs e.g., morphological parameters, associations, and liberation. The ability to measure not just the surface of particles but their interior mineralogy too adds great value to the data generated. When assessing the value of the data, we must also consider the time lapse between asking a question of the sample and obtaining an answer – if the data is to lead to an action, rather than an understanding, then it must arrive within the opportunity window for which it can be actioned. Next week is too late. Taking full advantage of the simplified sample preparation workflow, Mineralogic 3D delivers actionable data in a fraction of the time required by a metallographic preparation for the SEM.

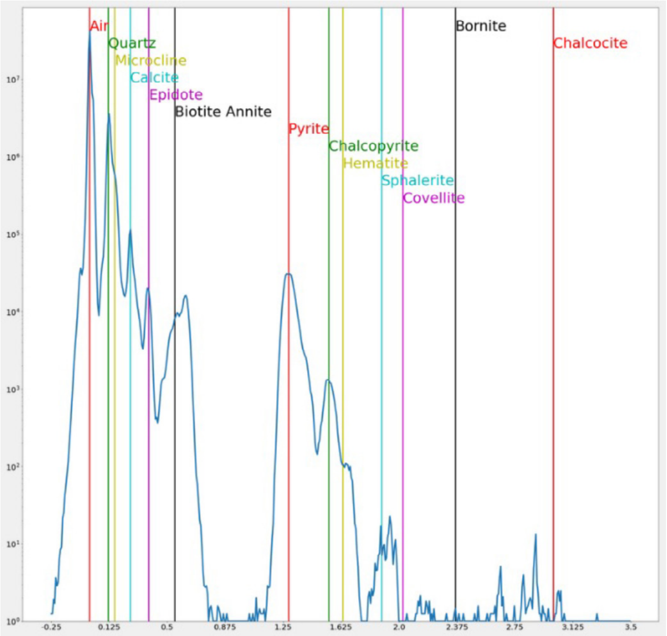


Figure 3 Mineral classification based on beam attenuation measurements

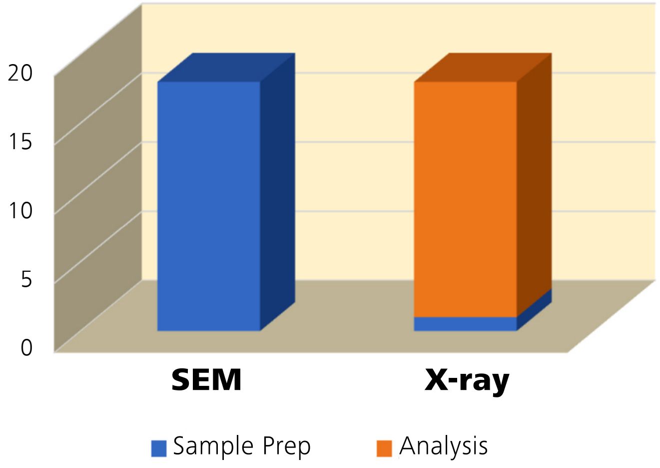
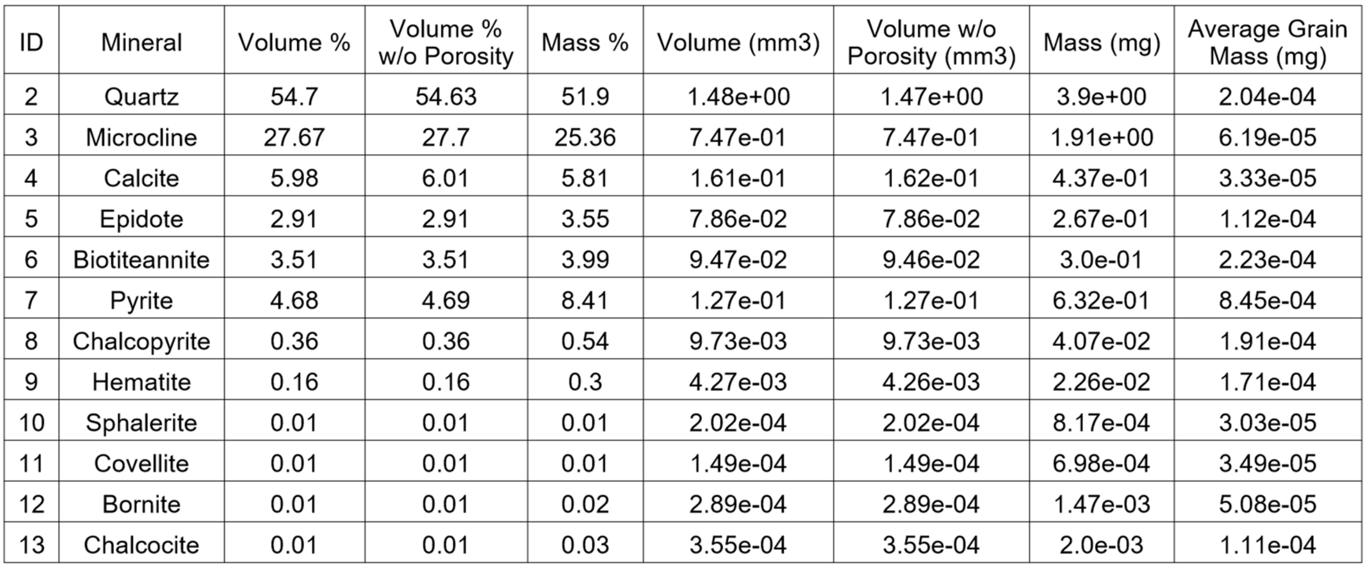


Figure 4 The simplicity of sample preparation combined with advanced analytical techniques results in a marked time savings in results delivery with Mineralogic 3D



ID	Mineral	Volume %	Volume % w/o Porosity	Mass %	Volume (mm3)	Volume w/o Porosity (mm3)	Mass (mg)	Average Grain Mass (mg)
2	Quartz	54.7	54.63	51.9	1.48e+00	1.47e+00	3.9e+00	2.04e-04
3	Microcline	27.67	27.7	25.36	7.47e-01	7.47e-01	1.91e+00	6.19e-05
4	Calcite	5.98	6.01	5.81	1.61e-01	1.62e-01	4.37e-01	3.33e-05
5	Epidote	2.91	2.91	3.55	7.86e-02	7.86e-02	2.67e-01	1.12e-04
6	Biotiteannite	3.51	3.51	3.99	9.47e-02	9.46e-02	3.0e-01	2.23e-04
7	Pyrite	4.68	4.69	8.41	1.27e-01	1.27e-01	6.32e-01	8.45e-04
8	Chalcopyrite	0.36	0.36	0.54	9.73e-03	9.73e-03	4.07e-02	1.91e-04
9	Hematite	0.16	0.16	0.3	4.27e-03	4.26e-03	2.26e-02	1.71e-04
10	Sphalerite	0.01	0.01	0.01	2.02e-04	2.02e-04	8.17e-04	3.03e-05
11	Covellite	0.01	0.01	0.01	1.49e-04	1.49e-04	6.98e-04	3.49e-05
12	Bornite	0.01	0.01	0.02	2.89e-04	2.89e-04	1.47e-03	5.08e-05
13	Chalcocite	0.01	0.01	0.03	3.55e-04	3.55e-04	2.0e-03	1.11e-04

Figure 5 *Modal mineralogy based on the volume of the mineralogy*

Measurements

Immediately at the end of the analysis, Mineralogic 3D reports on modal mineralogy, liberation, associations, particle and grain size, etc. All measurements that are in principle familiar to users of automated mineralogy on the SEM, however, there are some subtle and relevant differences between the two techniques.

Sample exposure

Automated mineralogy on the SEM requires particles to be fixed in resin block that is then ground to expose particle portions, and polished to present a good surface for analysis. Grinding and polishing result in the mechanical alteration of the sample and exposes only a fraction of the particles and mineral grains present. With Mineralogic 3D there is no mechanical alteration of the sample necessary and at any time, and every grain in the sample is visible to the analysis.

Modal mineralogy

Modal mineralogy considers the volume of each phase present in the sample. It need not be exposed on the surface to be accounted for (Figure 5).

Liberation

Liberation is purely dependent on the proportionality of the surface exposure of a particle; for example, a particle whose surface area is 85% chalcopyrite is considered as liberated for chalcopyrite. Furthermore, there are no hidden surprises beneath the surface as the entire particle surface, and interior mineralogy, is visible, classified, and measured. This results in liberated classifications that have no inherent assumptions, and locked classifications for mineralogies that is truly within the particle.

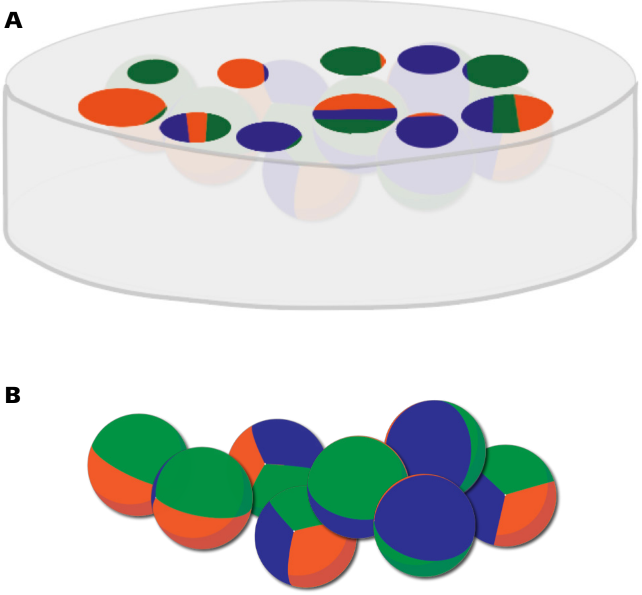
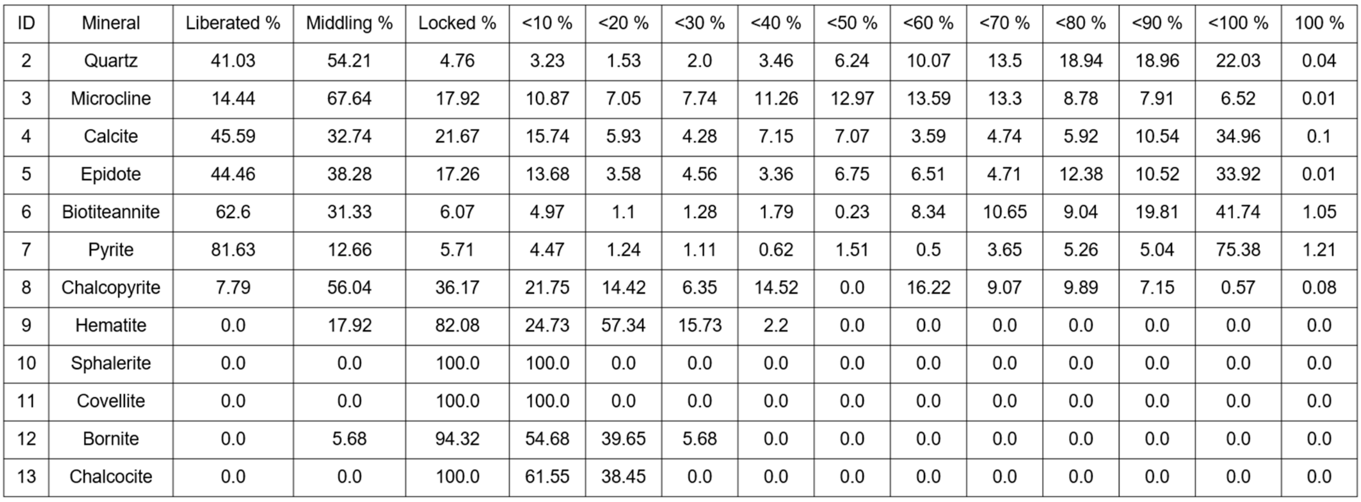


Figure 6 *(A) Mechanical alteration of the sample for SEM analysis reveals fractions of particles that the user must accept as true representations of the particle. (B) With X-ray microscopy, the particle's mineralogy and its exposure, or lack of, on the surface of the particle is fully recognised and measured.*



ID	Mineral	Liberated %	Middling %	Locked %	<10 %	<20 %	<30 %	<40 %	<50 %	<60 %	<70 %	<80 %	<90 %	<100 %	100 %
2	Quartz	41.03	54.21	4.76	3.23	1.53	2.0	3.46	6.24	10.07	13.5	18.94	18.96	22.03	0.04
3	Microcline	14.44	67.64	17.92	10.87	7.05	7.74	11.26	12.97	13.59	13.3	8.78	7.91	6.52	0.01
4	Calcite	45.59	32.74	21.67	15.74	5.93	4.28	7.15	7.07	3.59	4.74	5.92	10.54	34.96	0.1
5	Epidote	44.46	38.28	17.26	13.68	3.58	4.56	3.36	6.75	6.51	4.71	12.38	10.52	33.92	0.01
6	Biotiteannite	62.6	31.33	6.07	4.97	1.1	1.28	1.79	0.23	8.34	10.65	9.04	19.81	41.74	1.05
7	Pyrite	81.63	12.66	5.71	4.47	1.24	1.11	0.62	1.51	0.5	3.65	5.26	5.04	75.38	1.21
8	Chalcopyrite	7.79	56.04	36.17	21.75	14.42	6.35	14.52	0.0	16.22	9.07	9.89	7.15	0.57	0.08
9	Hematite	0.0	17.92	82.08	24.73	57.34	15.73	2.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10	Sphalerite	0.0	0.0	100.0	100.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
11	Covellite	0.0	0.0	100.0	100.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
12	Bornite	0.0	5.68	94.32	54.68	39.65	5.68	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13	Chalcocite	0.0	0.0	100.0	61.55	38.45	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Figure 7 *Liberation based on the mineral's exposure on the surface of the particle and weighed by volume*

Associations

In the 3D world, associations are measured relative to the contact of surfaces between minerals, rather than the linear contact observed in SEM analyses. Of course, a locked grain in the particle affects the association measurement in a way that cannot be measured by 2D techniques.

Summary

Benefitting from advancements in X-ray microscopy and machine learning, Mineralogic 3D represents a step change in automated mineralogy. The unique ability to study samples in 3D, with no obscured mineralogy, without stereological artifacts, and obtaining precise data for every particle opens the door to a new understanding of the geometallurgical behavior of ores.

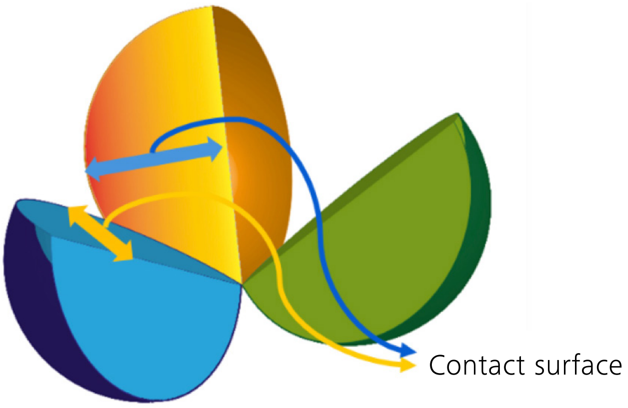


Figure 8 *Particle split open to reveal the contact surfaces between minerals. It is the proportion of the surface to the total surface of the mineral in the sample that determines the level of association between the minerals.*

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