(*6*): 1.942 Å and 1.948 Å, respectively, for the Al^{3+} and (Al,Fe^{3+}) sites; 2.060 Å and 2.104 Å respectively, for the (Mg,Fe^{2+}) sites. There is no evidence shown on electron-density or difference Fourier sections for segregation of Fe^{2+} in any site.

Similarly alternating octahedral chains are present in the structure of the sodium amphibole, glaucophane (*7*), and the reported pseudomorphism of omphacite by glaucophane (*8*) may be partially explained by this feature common to the two structures.

Table 1. Atomic coordinates, temperature factors, and cation site occupancy factors for omphacite from Tiburon Peninsula, Marin County, Calif.

Atom*; site occupancy†	Coordinates‡			Isotropic B (Å^2) §
	x	y	z	
O1(1)A	0.111	0.833	0.861	0.8
O1(2)A	.110	.673	.406	.5
O1(1)C	.384	.314	.102	.3
O1(2)C	.383	.160	.618	(.3)
O2(1)A	.365	.013	.063	.3
O2(2)A	.348	.497	.553	(.3)
O2(1)C	.133	.499	.942	.3
O2(2)C	.137	.994	.442	.3
O3(1)A	.356	.772	.754	.4
O3(2)A	.350	.744	.251	.4
O3(1)C	.153	.265	.258	.2
O3(2)C	.147	.237	.751	.2
Si 1 A	.2876	.8469	.9768	.23
Si 2 A	.2873	.6633	.4804	.25
Si 1 C	.2136	.3384	.0196	.20
Si 2 C	.2104	.1521	.5244	.15
M 1	0	.6619	0	.3
Mg^{2+} 0.81				
Fe^{2+} 0.19				
M 1(1)	0	0.8500	0.500	.4
Al^{3+} 0.95				
Fe^{3+} 0.05				
M 1 H	0.500	.1545	0	.4
Al^{3+} 0.82				
Fe^{3+} 0.18				
M 1(1) H	.500	.3451	0.500	.3
Mg^{2+} 0.80				
Fe^{2+} 0.20				
M 2	0	.0538	0	.8
Na^{+} 0.64				
Ca^{2+} 0.36				
M 2(1)	0	.4515	0.500	.7
Na^{+} 0.36				
Ca^{2+} 0.64				
M 2 H	0.500	.5507	0	.8
Na^{+} 0.03				
Ca^{2+} 0.97				
M 2(1) H	.500	.9497	0.500	.8
Na^{+} 0.64				
Ca^{2+} 0.36				

* Atom designations assigned by analogy to jadeite (*2*), according to nomenclature described by Burnham *et al.* (*10*). Different tetrahedral chains are designated A and C; the small, octahedrally coordinated cations, M 1; the large eightfold coordinated cations, M 2. † Approximate standard errors in site occupancy factors, ±0.02. A total occupancy of 1.0 for each site was assumed. ‡ Approximate standard errors: Si x, y, z and M 1, M 2 y, ±0.0005; oxygen x, y, ±0.001, z ±0.002. Transformation to compare with jadeite coordinates, $y + 1/4$, $z + 1/4$. § Approximate standard errors in B: Si ± 0.05 Å^2; oxygen, M 1, M 2 ± 0.1 Å^2. Temperature factors in parentheses were fixed.

JOAN R. CLARK
J. J. PAPIKE

U.S. Geological Survey,
Washington, D.C. 20242

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10. C. W. Burnham, J. R. Clark, J. J. Papike, C. T. Prewitt, in preparation.

8 September 1966

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Crystal Structure of the Zeolite Paulingite

Abstract. *Paulingite, a zeolite, has a framework structure consisting of 2016 atoms (672 silicon or aluminum and 1344 oxygen) which are in a cubic cell with an edge of length 35.093 Å. The framework has several features in common with the synthetic zeolites Linde A and ZK-5; the main channels are of similar size. We have also located most of the cations and water molecules inside the framework.*

The mineral Paulingite was identified by Kamb and Oke (*1*) as a zeolite exhibiting a cubic structure with $a = 35.10 \pm 0.02$ Å and probable space group $Im3m$ (O_h^9).

Our x-ray studies have led to a structure determination of the zeolite framework. We have also located most of the water molecules and metal ions inside the cages. The structure is consistent with space group $Im3m$. A more accurate value of the length of the cube edge, $a = 35.093 \pm 0.002$ Å, was determined.

The crystal used for our x-ray studies was one-half of a rhombic dodecahedron 0.2 mm in size. Intensities of the diffracted x-rays were measured with a Datex automated, General Electric XRD-5 diffractometer (nickel-filtered CuK$_\alpha$ radiation) equipped with the one-quarter-circle Goniostat. We examined 4185 symmetrically independent reflections out to a diffraction angle of $2\theta = 155°$ by scanning 2θ. Of these, 1190 were too weak to be measured.

The structure of the zeolite framework was determined by fitting the most prominent Patterson vectors into Samson's (*2*) packing maps for the (100) and (110) planes. The framework consists of 2016 atoms (672 silicon and aluminum, 1344 oxygen) and constitutes enough scattering matter to allow calculation of valid Fourier syntheses. Electron-density maps thus showed the locations of 88 metal ions (K, Ca, Na, Ba) and 452 water molecules. Because of difficulty in discriminating between the metal ions or mixture thereof they shall be called $M_{1,2,3}$, and so forth.

The parameters of these 2556 atoms and water molecules, distributed among the 45 different positions listed in Table 1, were refined by full-matrix, least-squares calculations with the use of data from the reflection range $14° < 2\theta < 155°$. The agreement index

$$R = \Sigma \| F_o| - |F_c\| / \Sigma | F_o|$$

for the 3900 reflections used is 0.14 and establishes confidence in the correctness of the basic structure.

The x-ray structure analysis gives the number of silicon-aluminum atoms (672) and the corresponding framework-oxygen atoms (1344) in the unit cell. The Si/Al ratio and the cation content were determined approximately by electron-microprobe analysis, and the water content, by weight loss on heating. The unit cell thus contains 520 Si, 152 Al, 1344 O, 68 K, 36 Ca, 13 Na, 1.5 Ba, and 705 H_2O.

Accordingly, about 250 more water molecules and about 30 more cations have yet to be located by x-ray structure analysis. Difference-Fourier maps indicate that these are arranged in a disordered fashion in the main channels of the framework, a feature that is quite common in zeolites (*3*).

The bond distances (Si, Al)—O in the framework range from 1.61 to 1.65 Å. The average value of the 28 different bond lengths is 1.634 Å, which is close to the value 1.64 Å derived from our Al/Si ratio by comparing it with those of other framework silicates (*4*).

The isotropic "thermal" parameters, *B*, listed in Table 1 were obtained together with the positional parameters from the full-matrix least-squares calculations. No attempt was made with the present, relatively crude x-ray data to discriminate between the various factors that enter into the *B* values, such as partial occupancy, displacement disorder, and substitutional disorder. The framework atoms $(Si, Al)_{1\text{-}8}$ and $O_{1\text{-}20}$, the cations M_1, and the water molecules $(H_2O)_{1\text{-}2}$ are seen to have *B* values that correspond to normally expected thermal vibrations. The remaining atoms exhibit pronounced anisotropy, as was found from difference-Fourier syntheses. The water molecules $(H_2O)_{6\text{-}14}$ have *B* values ranging from 15 to 25; their point sets (Table 1) are almost certainly partially occupied.

A formal representation of portions of the Paulingite framework is given by the models shown in Figs. 1a to 1f. In these, each square represents a ring of four $(Si,Al)O_4$ tetrahedra that are linked together by sharing vertices (oxygens). Each Si, Al site (tetrahedron center) is at a square corner, and each bridging oxygen is roughly ½ Å away from an edge and 1.6 Å from each of the two corners. Each Si, Al site belongs to three 4-rings of tetrahedra [the $(Si,Al)O_4$ tetrahedra shall be called simply tetrahedra].

There are, in the crystallographic sense, eight different structural units, three cages (called A, B, and C), two double 8-rings (called D_1 and D_2), two horseshoe-like configurations (called H_1 and H_2), and one toroidal channel.

The A cage is the truncated cubo-octahedron shown in Fig. 1a; the 48 vertices represent the centers of 48 tetrahedra that are linked together. The A cage is connected with a set of six D_1 rings that are represented by octagonal prisms in Fig. 1b.

The B cage has two planar and four nonplanar 8-ring openings and 32 corners. It represents 32 linked tetrahedra. One of the planar openings is connected with a D_1 ring, the other with a D_2 ring; both rings are alike (octagonal prisms) but differ in the crystallographic sense; see Fig. 1c. Figure 1d shows the B cage with the two D rings and, in addition, four "horseshoes" of the kind H_1, each of which forms an arch across a nonplanar 8-ring opening. This aggregate $(B + D_1 + D_2 + 4H_1)$ contains all crystallographically different Si, Al sites.

The C cage has two planar 6-ring openings, six nonplanar 8-ring openings, and 30 corners, and represents, accordingly, 30 linked tetrahedra. It is shown in Fig. 1e together with two sets of three horseshoes each, $3H_1$ and $3H_2$; one set forms the upper three arches in Fig. 1e; the other forms the lower three arches. Although the two sets of horseshoes look alike, they are, in the crystallographic sense, not equivalent, as will be seen later. The center of this aggregate $(C + 3H_1 + 3H_2)$ is, accordingly, not a center of symmetry as it might appear in the picture; a true center of symmetry is located at the center of the planar hexagon that is formed by the three horseshoes of the kind H_1. In Fig. 1e this can be either the upper hexagon or the lower one; the one that is assigned the $3H_1$ is, at this stage of the description, irrelevant.

The structure of the whole zeolite framework now can be described by reference to the aggregates just discussed.

Along each of the three cube edges there is the following sequence of structural units (Fig. 1f shows the sequence only in the vertical direction

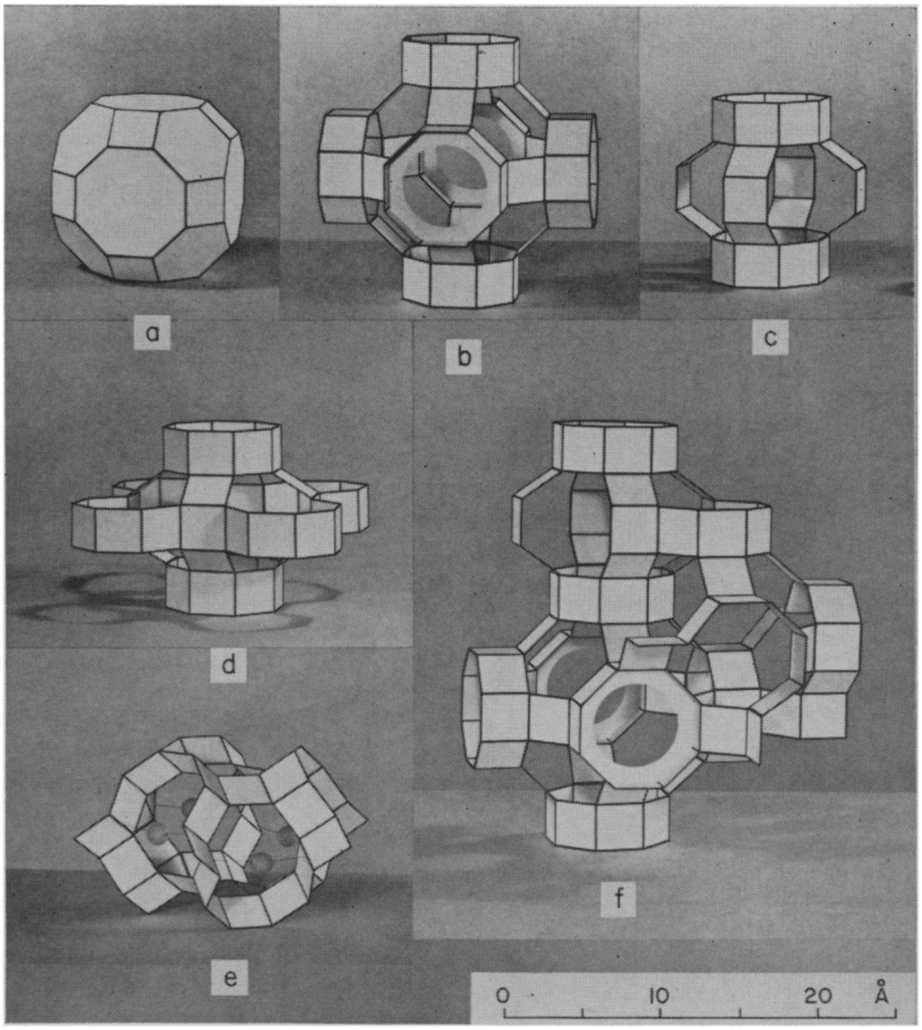

Fig. 1. A formal representation of the structural units of the zeolite Paulingite. Each square corner represents the center of a $(Si,Al)O_4$ tetrahedron. (a) The A cage, a truncated cubo-octahedron. (b) The A cage connected with six octagonal prisms, the D_1 rings. (c) The B cage connected with two crystallographically different octagonal prisms, the D_1 and D_2 ring. (d) The same aggregate as (c), but four arches, the horseshoes H_1 have been added. (e) The C cage showing the location of the metal ions M_2 and M_3. At the center of this cage is M_1 (not shown). (f) The structural units arranged in their proper sequence; A-D_1-B-D_2 is vertical and A-C is along the body diagonal, which is directed toward the upper right corner of the picture.

[001]): (i) the A cage, which has its center at the origin of the cube; (ii) the octagonal prism D_1; (iii) the B cage with the four horseshoes ($4H_1$), of which only one is shown in Fig. 1f; (iv) the octagonal prism D_2, which has its center on the cube edge at a distance of *a*/2 from the origin (*a* being the length of the cube edge).

A mirror plane passes through the center of D_2 at 00½ perpendicular to the edge such that the sequence along the whole cube edge becomes A-D_1-(B + $4H_1$)-D_2-(B + $4H_1$)-D_1-A. Only one-half of this sequence is shown in Fig. 1f.

The center of the C cage is located on the body diagonal of the cube. It can now be seen (Fig. 1f) that the three horseshoes of the kind H_2, forming the "lower" part of the C cage, are the result of the combination A + D_1 + B, while the "upper" three horseshoes ($3H_1$) of this cage constitute the arches across the nonplanar 8-ring openings of three B cages. Accordingly, the upper right hexagon in Fig. 1f (around the body diagonal of the cube) is the one that has its center in common with the center of symmetry, which is at ¼ ¼ ¼. Consequently, this hexagon is shared between two crystallographically equivalent C cages related to one another by this symmetry center; the second C cage (not shown in Fig. 1f) is connected with the A cage that has its center in common with the body center of the cube. The sequence of structural units along the body diagonal is, accordingly, A-C-C-A-C-C-A, of which only A-C is shown in Fig. 1f.

Table 1. The atomic positions and isotropic temperature factors for the zeolite Paulingite. The standard deviations are $\sigma_X = \pm 0.0001$ and $\sigma_B = \pm 0.05$ for $(Si, Al)_{1-8}$, $\sigma_X = \pm 0.0003$ and $\sigma_B = \pm 0.20$ for O_{1-20}, $\sigma_X = \pm 0.0002$ and $\sigma_B = \pm 0.15$ for M_{1-3}, $\sigma_X = \pm 0.001$ and $\sigma_B = \pm 1.0$ for $(H_2O)_{1-5}$, and $\sigma_X = \pm 0.005$ for $(H_2O)_{6-14}$. M_{1-3} represent Na, K, Ca, and Ba, which probably occur in substitutional disorder. The last nine point sets $[(H_2O)_{6-14}]$ are almost certainly partially occupied; their *B* values range from 15 to 25.

Kind of atom	Number of atoms per cube	*x*	*y*	*z*	*B* in Å²
$(Si, Al)_1$	48	0.3137	¼	½-*x*	0.47
$(Si, Al)_2$	48	.4021	¼	½-*x*	.46
$(Si, Al)_3$	96	.3132	0.2498	0.0979	.48
$(Si, Al)_4$	96	.4558	.1072	.0443	.54
$(Si, Al)_5$	96	.4019	.1782	.0448	.45
$(Si, Al)_6$	96	.3126	.1785	.0446	.40
$(Si, Al)_7$	96	.2592	.1073	.0445	.36
$(Si, Al)_8$	96	.1708	.1076	.0441	.44
O_1	48	.1635	.0933	0	1.4
O_2	48	.2679	.0968	0	1.3
O_3	48	.3041	.1886	0	1.4
O_4	48	.4092	.1900	0	1.8
O_5	48	.4484	.0952	0	1.6
O_6	48	.4489	.3794	0	1.8
O_7	48	.0713	*x*	0.1610	1.4
O_8	48	.4308	*x*	.2322	1.9
O_9	48	.1437	*x*	.0545	1.5
O_{10}	48	.2860	*x*	.1965	1.6
O_{11}	48	.2868	*x*	.0890	1.7
O_{12}	48	.4299	*x*	.0548	2.0
O_{13}	96	.2152	0.1211	.0496	1.2
O_{14}	96	.2870	.1414	.0582	1.8
O_{15}	96	.3573	.1672	.0525	1.5
O_{16}	96	.4278	.1417	.0573	2.3
O_{17}	96	.3002	.2163	.0693	1.9
O_{18}	96	.4148	.2142	.0710	1.9
O_{19}	96	.3576	.2623	.0909	1.6
O_{20}	96	.3080	.2351	.1419	1.5
M_1	16	.1788	*x*	*x*	2.4
M_2	24	.2543	*x*	0	3.0
M_3	48	.3975	*x*	0.1445	5.5
$(H_2O)_1$	16	.140	*x*	*x*	5.7
$(H_2O)_2$	16	.217	*x*	*x*	4.2
$(H_2O)_3$	48	.209	*x*	0.053	6.2
$(H_2O)_4$	48	.348	*x*	.200	5.5
$(H_2O)_5$	48	.350	*x*	.082	11.5
$(H_2O)_6$	48	.220	*x*	.140	
$(H_2O)_7$	48	.139	*x*	.217	
$(H_2O)_8$	48	.334	0.275	0	
$(H_2O)_9$	48	.422	.285	0	
$(H_2O)_{10}$	24	.292	½	0	
$(H_2O)_{11}$	24	.367	*x*	0	
$(H_2O)_{12}$	12	.178	0	0	
$(H_2O)_{13}$	12	.270	0	0	
$(H_2O)_{14}$	12	.458	0	0	

If all structural units are in place, a toroidal channel is produced around each octagonal prism of the kind D_2. This channel was omitted in Fig. 1f because it would have obscured the view; with the information thus far provided, it should be possible, however, for the reader to construct a mental picture of it.

The structural units, arranged along the cube edges in the sequence described above, form a continuous, three-dimensional channel system, in which the A cages lie at the corners of the cubic unit cells. A second channel system of the same kind runs parallel to the first one but has the A cages at the body centers of the unit cells. There is no access from one system to the other, and no atoms are shared between the cages of the two systems. The channels are very similar to those in Linde A and ZK-5 (*5*) and have a free diameter of 3.8 Å.

Of the cations located with reasonable confidence, M_1 (Table 1) is at the center of the C cage; it is surrounded by eight water molecules. The M_2 cations are located in the open ends of the H_2 horseshoes and the M_3 cations in the open ends of the arches formed by H_1. Both M_2 and M_3 are at a distance of 2.9 Å from the nearest framework oxygen. M_2 and M_3 are shown in Fig. 1e.

In the synthetic zeolites Linde A and ZK-5 (*5*) the truncated cubo-octahedron (A cage) constitutes a considerably larger fraction of the framework than in Paulingite. The B cages are also observed in ZK-5 but without the arches (formed by H_1). The configurations C, $D_{1,2}$, and $H_{1,2}$ seem to be unique for Paulingite.

It is of interest to note that a hypothetical body-centered cubic zeolite structure with *a* = 25.1 Å is obtained by arranging along the cube edges the structural units in the sequence A-D_1-B-D_1-A and along the body diagonal in the sequence A-C-A-C-A. With the exception of the toroidal channels, this hypothetical structure would incorporate all the structural elements that are present in Paulingite.

E. KENT GORDON
STEN SAMSON, W. BARCLAY KAMB
Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena 91109

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2 September 1966 ■

Silurian of Central Texas: A First Record for the Region

Abstract. *Silurian outcrops, not previously recorded from central Texas, have been identified from the Llano uplift, where they occur in collapse structures within the Lower Ordovician Honeycut Formation of the Ellenburger Group. The formation is a pinkish-gray granular limestone, contains fossils of probable Wenlock age, and is named the Starcke Limestone.*

Rocks younger than Early Ordovician and older than Carboniferous were once believed to be missing from the Llano uplift of central Texas, a tectonic outpost of older rocks surrounded by a vast expanse of Carboniferous and younger sediments. Beginning in 1945, however, a picture has emerged of remnants of once-extensive deposits of Upper Ordovician and Lower, Middle, and Upper Devonian age, preserved in collapse structures and fissures in older rocks, and as minute erosional remnants, after the parent sediments were mainly removed by erosion during one or more of many episodes of emergence (*1*). To these we now add Silurian, giving the region a known representation of all Paleozoic systems except the Permian.

Althought the fossils which establish a Silurian date were originally collected from the type locality (Fig. 1, locality 27T-10-3B) by James Lee Wilson and Barnes, on 26 March 1952, and again by W. H. Hass, Cloud, and Barnes in March 1956, and were then tentatively identified as Silurian by Cloud, other obligations at that time prevented definitive analysis of the fauna. Impetus for this came when the fossils were shown to Boucot in April 1966. He then identified the brachiopods as middle Silurian (Wenlock)—an assignment that was later supported by Palmer, who found the trilobites to be similar to those of the St. Clair limestone near Batesville, Arkansas, and by W. A. Oliver, Jr., U.S. Geological Survey, who identified the corals as closest to (but not necessarily correlative with) species from the Brownsport and Henryhouse formations. As a result, Barnes, Boucot, and Cloud, in company with W. C. Bell and James Lee Wilson, re-collected the type locality on 8 June 1966 and shipped about ½ ton of fossiliferous limestone blocks to Pasadena for processing.

Another locality (27T-6-43K, Fig. 1) was revisited because a few fossils from near it, collected by Barnes, Cloud, and George Seddon during March 1965, had suggested a Silurian age to Cloud. Previously a coral from this locality was sent to Helen Duncan, U.S. Geological Survey, and in a report of 10 April 1956 she identified it as *Favosites* with the following comment: "So far as I am able to tell, this specimen from Texas could be either Silurian or Devonian. A Late Ordovician assignment seems less likely, but that possibility is not ruled out." The analysis of these relict formations, however, is a block by block proposition. The *Favosites*-bearing, white, very coarse-grained limestone of locality 27T-6-43K is near the base of an outcrop of steeply dipping, cherty, impure limestone of the Devonian Stribling Formation, and therefore is in normal stratigraphic position to be a part of the granular limestone member of that formation. In fact, it probably does belong to the Stribling Formation, being lithologically distinct from the Silurian Starcke Limestone here discussed.

The Starcke Limestone at this locality resembles that at 27T-10-3B both lithically and faunally. It crops out on the east bank of a shallow drain about 20 feet (6 m) or so east of the outcrop of granular Stribling limestone. Because the designation 27T-6-43K was originally used for the *Favosites*-bearing limestone, however, a new locality number 27T-6-43M is assigned to the outcrop here referred to the Silurian Starcke Limestone.

All of these relict rocks rest upon or sag into Lower Ordovician carbonate rocks of the Ellenburger Group and are or were overlain by rocks of Carboniferous age (Fig. 1).

Locality 27T-10-3B (Fig. 1A), the type locality, is situated on the C. H. Dean ranch in Burnet County, Texas, 4000 feet south-southwest of Max Starcke Dam. It is 900 feet south of Flatrock Creek, 250 feet from the west boundary of the Dean ranch, and 100 feet from the mouth of a shallow, northward-flowing drain which empties into a drain flowing directly into Flatrock Creek. The Starcke Limestone here occupies a collapse structure, along with

Table 1. Fossils from the Silurian of Central Texas. Names followed by 3B or 43M indicate forms identified at locality 27T-10-3B (=USNM locality 13014) or 27T-6-43M (=USNM locality 13015) only. Absence of such designation indicates forms found at both localities.

Brachiopods
  Amphistrophia? cf. *A. striata* (Hall) 3B
  Atrypa "reticularis" (Linnaeus)
  Clorinda? sp. 3B
  Coelospira? sp. 3B
  Coolinia sp.
  Cyrtia sp. 3B
  "Dolerorthis" flabellites (Foerste)
  Eospirifer sp.
  Howellella? sp. 43M
  Kozlowskiellina (Kozlowskiellina) sp. 3B
  Leangella sp. 3B
  Leptaena "rhomboidalis" (Wilckens)
  Meristina sp.
  Mesodouvillina? sp.
  Plectatrypa? sp. 43M
  Plectodonta sp.
  Resserella sp.
  Rhynchonellids 3B
  Schizoramma? sp. 3B
  Streptis? sp.
Trilobites
  Bumastus sp. 3B
  Dalmanites bassleri (Ulrich & Delo)
  Unidentified dalmanitid 3B
Corals
  Enterolasma n. sp. 3B
  Syringaxon sp. 3B
Cephalopod
  Dawsonoceras sp. 3B
Miscellaneous megafossils
  Fenestellid Bryzoa 3B
  Gastropod 3B
  Pterineoid pelecypod 43M
Conodonts, all 3B
  Belodina sp. (a redeposited Burnam form)
  Drepanodus cf. *D. subarcuatus* Furnish
  **Ligonodina silurica* Branson & Mehl
  **Ozarkodina* cf. *O. ziegleri tenuiramea* Walliser
  Panderodus unicostatus (Branson & Mehl)
  Panderodus acostatus (Branson & Branson)
  **Spathognathodus* cf. *S. ranuliformis* Walliser
  Spathognathodus sp.

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* Forms considered significant for age assignment.