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On Studtite and Its Composition

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Abstract

The investigation of studtite from the uranium deposit of Menzenschwand in the Black Forest shows that this ill-defined species is not a uranyl carbonate as hitherto assumed but a hydrous uranium oxide of monoclinic symmetry. It is identical with a synthetic uranium compound which is regarded as peroxide hydrate $UO_4 \cdot 4\ H_2O$ (Sato, 1961). If this interpretation is correct, studtite would be the first peroxide mineral found in nature. Unit-cell dimensions of the mineral from Menzenschwand are: a 11.85, b 6.80, c 4.25 Å β 93°51′, $Z = 2$, space group $C2$, Cm, or $C2/m$, density (calc) 3.64 g/cc. Strongest lines of the powder pattern are 5.93 (10), 3.40 (8), 2.96 (6). The X-ray data agree closely with those given by Debets (1963) for the synthetic compound. The structure of studtite probably consists of O-U-O-U-O chains in the direction of the c-axis as in case of α-UO_3.

When heated to 60°C, studtite is transformed into a dihydrate, $UO_4 \cdot 2\ H_2O$ with loss of 2 H_2O. This dihydrate, which is also known as a synthetic compound, is orthorhombic with a 6.51, b 8.78, c 4.21 Å, $Z = 2$, space group probably $Immm$, density (calc) 4.67 g/cc. Strongest lines of the powder pattern: 5.24 (10), 4.41 (7), 3.54 (8).

A brief description is also given of a presumably new siliciferous mineral occurring at Menzenschwand associated with studtite and uranophane.

Introduction

The mineral studtite is an ill-defined secondary uranium mineral. It was described by Vaes (1947) as a hydrated carbonate of uranium, on the basis of qualitative tests. The mineral had been found as aggregates of thin fibers on uranophane and rutherfordine at Shinkolobwe, Katanga. The optical properties given by Vaes led to the conclusion that studtite is orthorhombic (Frondel, 1958). The scanty data published by Vaes were later supplemented by Guillemin (1958), who furnished X-ray powder spacing data for the mineral from Katanga.

The optical and X-ray data published by Vaes and Guillemin permitted the present author to identify studtite among the secondary minerals of the uranium deposit at Menzenschwand in the Southern Black Forest (Walenta, 1960; 1963). The first specimen of studtite found at this locality dates from 1959. A preliminary chemical investigation, however, showed that the mineral is not a carbonate (Walenta, 1967). Vaes evidently investigated an impure sample. This may also be indicated by the small amount of lead in his material which he thought might be due to inclusions. Such impurities could also have caused the evolution of carbon dioxide when the mineral was treated with acid.

Mode of Occurrence

The mode of occurrence of studtite at Menzenschwand closely resembles that of the mineral at Shinkolobwe. It forms aggregates and crusts of minute needle-like crystals that frequently show a more or less radial fibrous structure (Fig. 1). The needles are elongated along the c-axis but lack observable crystal faces. Their maximum length approaches 0.5 mm; maximum thickness is about 10 μm.

The mineral is found on barite, quartz, hematite, and limonite associated with billietite, uranophane, rutherfordine, and other secondary uranium minerals. In one case it is intimately intergrown with uranophane and the unknown mineral described below. In another it occurs as younger crusts on uranophane.

Chemical Composition

Studtite is slowly acted on by cold 1:1 HCl and cold 1:1 HNO_3, whereas it is more rapidly soluble in cold 1:1 H_2SO_4. No effervescence was observed during dissolution. Minute crystals of the lower hydrate (p. 169) precipitate from the solution in HCl after partial evaporation of the solvent at room temperature.

Electron microprobe and microchemical analyses

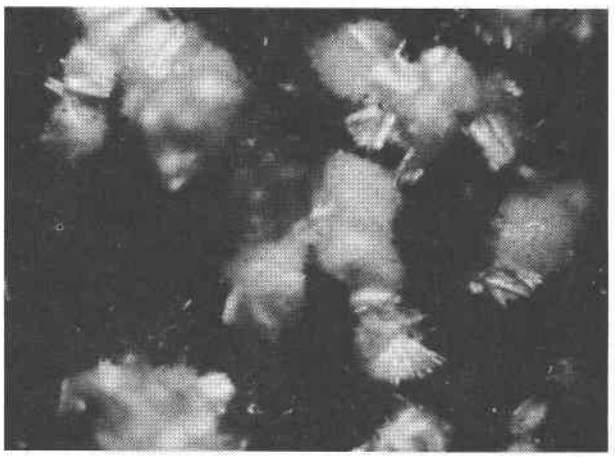

FIG. 1. Studtite forming aggregates of needle-like crystals on limonite, Menzenschwand, Southern Black Forest. Magnification about 60 ×.

revealed only uranium as a major constituent. Tests for other cations and anions were negative.

A spectrographic analysis performed by H. Wilk, Chemisches Untersuchungsamt der Stadt Stuttgart, yielded similar results, though a number of other elements were detected in minor amounts (Mg, Cu, Ni, Zn, Al, Si) and in traces (Ca, Co, Fe, Mn, Bi, Sb, Sn). In part their presence may be ascribed to impurities.

The qualitative chemical analyses reveal that studtite is not a carbonate and that no cations other than uranium play an essential role in its structure. Its true nature was recognized when the powder pattern of pure studtite proved identical with that of a synthetic hydrous uranium oxide, the formula of which is thought to be $UO_4 \cdot 4\ H_2O$ (Sato, 1961; Debets, 1963). Likewise, the powder diagrams of $UO_4 \cdot 2\ H_2O$ as obtained by heating studtite to 60°C are identical to those of the similarly dehydrated synthetic compound.

Chemically the compound $UO_4 \cdot 4\ H_2O$ generally is regarded as uranium peroxide hydrate (Katz and Rabinowitch, 1951; Sato, 1961). Thus its formula is also written $UO_3 \cdot H_2O_2 \cdot 3\ H_2O$. It can be obtained as a light yellow precipitate by addition of hydrogen peroxide to a concentrated solution of uranyl nitrate or uranyl acetate. Peroxo-uranates are formed by treating the compound simultaneously with hydrogen peroxide and alkalies. Heating of the uranium peroxide leads first to a lower hydrate and then to UO_3 with evolution of oxygen and water.

The presence of this compound in the oxidized zone of uranium deposits as a member of a normal secondary paragenesis is astonishing. It would be the first natural occurrence of a peroxide compound. Because the existence of such a compound in nature seemed unlikely, it was thought possible that its interpretation as peroxide might be wrong and another formula, $UO_2(OH)_2 \cdot 4\ H_2O = UO_3 \cdot 5\ H_2O$, interpreting it as hydroxide, was taken into consideration. However, all chemical tests carried out speak in favor of the peroxide nature of the synthetic compound and the mineral. If for instance MnO_2 is added to a solution of the compound in dilute H_2SO_4, gas is given off with effervescence. With KI the solution yields a precipitate of iodine, and a solution of $KMnO_4$ is decolorized by it.

If studtite is indeed a peroxide, the question remains of how it formed under natural conditions. Although this problem is not yet solved, possibly the small amounts of hydrogen peroxide known to occur in rain water and snow are responsible. Another possibility is an organic source of the hydrogen peroxide.

Physical and Optical Properties

The needle-like yellow crystals exhibit no distinct cleavage, are soft, and translucent to transparent. Studite does not fluoresce in ultraviolet light. The calculated density is 3.64 g/cc. The attempt to determine the density of the natural mineral by means of heavy liquids was not successful because the crystals were too small. The density of the artificial compound as measured by the pycnometer method is 3.58 g/cc, in reasonable agreement with the calculated value.

The optical properties are summarized in Table 1. Because the small crystals were not suitable for obtaining polarization figures, the optical character and the optical axial angle $2V$ could not be determined directly. Vaes (1947) states $2V$ to be large; how-

TABLE 1. Optical Properties of Studtite

Studtite	
Shinkolobwe (Vaes, 1947)	Menzenschwand (Present study)
α 1.545	α 1.537 ± 0.003
β 1.555	
γ 1.68	γ 1.680 ± 0.003
Nearly colorless	
$2V$ large	
Z parallel to elongation	

ever, this is not consistent with the indices of refraction he determined. The indices of refraction α and β do not differ greatly, as confirmed by the present study, so the mineral must be optically positive and have a small $2V$.

The extinction of the crystals is parallel and not oblique. The acute bisectrix Z coincides with the c axis so that the monoclinic symmetry cannot be recognized by the optical properties. This behavior is explained by the fact that the lattice shows a marked pseudohexagonal symmetry for the c axis. The pseudosymmetry evidently influences the optical properties to such a degree that they resemble those of a uniaxial positive crystal.

X-Ray Investigation

X-ray powder diffraction data for studtite from Menzenschwand and Shinkolobwe as well as the indexed pattern for the synthetic compound are listed in Table 2.[1] The unit-cell dimensions of natural studtite and the synthetic compound are given in Table 3. The d-spacings of the powder pattern and the unit-cell dimensions of studtite and the synthetic compound are the same within limits of error.

The powder pattern given by Guillemin (1958, p. 48) for the mineral from Katanga differs somewhat from that for the mineral from Menzenschwand and for the synthetic compound. One of the strongest lines of his pattern (d = 7.53 Å) is missing in the two other patterns. Such a line in fact was sometimes also visible on powder patterns taken of different samples of the mineral from Menzenschwand; however, its intensity was variable and in one instance it was hardly discernible. Its presence must probably be ascribed to an impurity.

The unit-cell dimensions of natural studtite were calculated from the powder data using the indexed powder pattern of the synthetic compound as a starting point. Before the natural mineral was identified with the synthetic compound, single-crystal studies were made to determine the unit cell. However, the smallness of the crystals led to Weissenberg photographs of very poor quality. Their interpretation yielded a hexagonal cell with an a-period of 6.8 Å. The c-value calculated from a rotation photograph was close to 4.2 Å. The majority of the powder

[1] All patterns were obtained with Fe$K\alpha$ radiation using a camera of 57.3 mm in diameter. They include d-spacings in Å corrected for film shrinkage and sample thickness. Intensities were estimated from 10 to 0.5.

TABLE 2. X-ray Powder Data for Natural and Synthetic Studtite

Studtite: Menzenschwand (Present study)			Studtite: Shinkolobwe (Guillemin, 1958)		Synthetic: $UO_4 \cdot 4\,H_2O$ (Debets, 1963)*		
d (Å)	I**		d (Å)	I**	d (Å)	I***	hkl
			7.53	st			
5.93	10		5.91	vv st	5.88	100	200,110
4.27	4		4.25	vv w	4.23	12	001
			3.75	w			
			3.58	w	3.55	10	$20\bar{1}$
3.49	2	dif.			3.49	19	$11\bar{1}$
					3.40	21	310
3.40	8		3.40	m	3.39	32	020,111
					3.34	5	201
			3.22	w to m			
					2.95	5	400
2.96	6		2.98	v w	2.94	8	$22\bar{0}$
2.72	0.5				2.73	5	$31\bar{1}$
2.66	1				2.648	7	021
2.60	1				2.588	6	311
2.49	2		2.53	v w	2.500	5	$40\bar{1}$
2.44	2				2.451	6	$22\bar{1}$
2.37	3				2.380	5	221
					2.356	5	401
					2.234	5	510
2.23	6		2.27	v w	2.228	5	420
					2.220	4	130
2.12	1		2.127	vv w	2.118	4	002
					2.026	6	$51\bar{1}$
2.02	5		2.042	v w	2.012	11	$11\bar{2},42\bar{1}$
					1.974	10	$13\bar{1},112,600$
1.970	5	broad	1.966	w	1.957	10	330,131,202
1.934	3				1.931	8	$42\underline{1},511$
					1.844	2	$31\underline{2}$
1.837	0.5	dif.			1.832	1	$60\bar{1}$
1.797	1				1.798	5	$33\bar{1},022$
1.755	2				1.758	5	$331,31\underline{2}$
					1.746	4	$60\underline{1},22\bar{2}$
					1.704	2	620
1.700	2	dif.			1.694	4	040,222
					1.672	1	402
					1.639	1	710
1.635	2	dif.			1.634	2	530
					1.628	1	$24\underline{0}$
					1.611	1	$62\bar{1}$
					1.585	2	$51\bar{2}$
					1.573	3	$04\underline{1},42\bar{2}$
1.567	1	dif.			1.561	1	$71\bar{1}$
					1.552	1	$62\underline{1}$
					1.546	1	$53\bar{1}$
					1.542	1	$13\bar{2}$
					1.529	1	$24\bar{1}$
					1.524	1	132
					1.511	2	241
1.505	3				1.503	1	531
					1.499	2	422,711
					1.493	1	512
					1.478	1	800
1.470	0.5	dif.			1.470	1	440
					1.462	1	$33\bar{2}$
1.420	0.5	dif.					
1.377	0.5						
1.356	0.5						
1.284	2	dif.					
1.219	2	dif.					
1.137	0.5	dif.					
1.116	1	dif.					
1.087	0.5	v. dif.					
1.066	0.5	dif.					
1.016	0.5	dif.					

*ASTM No. 16-206.
**Abbreviations used: dif. = diffuse, v. dif. = very diffuse; st = strong, vv st = very strong, m = medium, w = weak, v w = very weak, vv w = very, very weak.
***Diffractometer measurements.

TABLE 3. Unit-Cell Dimensions of Natural and Synthetic Studtite

Studtite, Menzenschwand (Present study)	Synthetic $UO_4 \cdot 4H_2O$ (Debets, 1963)
a 11.85 ± 0.02 Å	a 11.85 Å
b 6.80 ± 0.01	b 6.785
c 4.25 ± 0.01	c 4.245
β 93°51' ± 20'	β 93°37'
$a:b:c$ = 1.743:1:0.625	
V 341.7 ± 1 Å^3	V 341 ± 1 Å^3
$Z = 2UO_4 \cdot 4H_2O$	
Space group $C2$, Cm or $C2/m$	

lines could be indexed in this way, but some remained unindexable. Thus the chosen cell was only a pseudocell and not the true one.

The c-axis of the pseudohexagonal cell is identical with the c-axis of the monoclinic cell. Debets (1963) also states that the stronger reflections of his pattern indicated hexagonal symmetry at first sight. Closer inspection, however, revealed that they are doublets or triplets. The true symmetry of the unit cell is C-face-centered monoclinic.

Thermal Behavior

When studtite is heated to 60°C, it is converted into a new phase which yields the same powder pattern as the synthetic compound $UO_4 \cdot 2\ H_2O$ (Table 4). The powder pattern given in Table 4 was obtained after heating studtite for 2 hours to 100°C. However, as mentioned above, dehydration takes place at temperatures as low as 60°C. The dehydration phase may be called metastudtite in accordance with the nomenclature usually applied to dehydrated uranium minerals. The dehydration process is irreversible, and natural occurrence of the dihydrate seems possible.

Sato (1961) mentions that at temperatures below 50°C the compound $UO_4 \cdot 4\ H_2O$ is formed but above 70°C, $UO_4 \cdot 2\ H_2O$. This is more or less consistent with the results of the investigation of the natural mineral, though in one case the lower hydrate was obtained from a solution in HCl at room temperature, as mentioned earlier. At higher temperatures α-UO_3 is formed (Cordfunke, 1961).

The dihydrate is orthorhombic. The unit-cell dimensions calculated from the powder pattern (Table 5) differ only slightly from the values given by Debets for the synthetic compound. The cell is body-centered as only general reflections with $h + k + l$ even occur.

The dihydrate has higher indices of refraction than studtite. The value of α rises to 1.758 ± 0.003. The birefringence of the crystals remains about the same as before heating. The calculated density is 4.66 g/cc.

TABLE 4. X-ray Powder Data for the Dihydrate

Dihydrate* (Present study)		Synthetic $UO_4 \cdot 2\ H_2O$ (Debets, 1963)**		
d (Å)	I***	d (Å)	I	hkl
5.24	10	5.22	100	101
4.41	7	4.39	33	002
3.80	7	3.79	33	011
3.54	8	3.53	34	110
3.26	6	3.25	15	200
2.75	5	2.75	21	112
2.67	5	2.666	21	103
2.61	3	2.611	9	202
2.47	5	2.470	19	211
2.40	4	2.403	15	013
2.19	2	2.194	5	004
2.10	5	2.104	12	020,301
1.955	4	1.953	13	121
		1.931	13	213
1.927	5	1.926	8	310
		1.897	5	022
1.862	3	1.863	13	114
1.819	1	1.817	4	204
1.768	4	1.766	6	220
		1.761	8	312
1.745	2	1.742	5	303
1.695	0.5 dif.	1.693	2	105
1.654	2 v. dif.	1.651	6	123
		1.639	5	222
1.628	2 v. dif.	1.625	2	400
		1.618	3	015
1.527	1	1.524	2	402
		1.519	2	024
1.495	3	1.494	6	411
		1.489	5	321
		1.463	1	006
1.450	3	1.450	3	215
		1.448	4	314
1.376	1 dif.	1.376	3	224
		1.372	2	130
		1.351	3	116
1.349	3 dif.	1.346	5	413,323
		1.319	2	125
1.310	1	1.310	3	132
		1.305	2	404
1.292	0.5 v. dif.	1.286	3	501,420
1.277	0.5 v. dif.	1.275	3	231
		1.266	1	033
1.240	1 v. dif.			
1.199	1 v. dif.			
1.180	0.5			
1.166	0.5			
1.149	0.5			
1.129	1			
1.112	0.5			
1.098	0.5			
1.085	1 v. dif.			
1.053	0.5 v. dif.			
1.040	1 v. dif.			
1.000	1 v. dif.			

*Obtained by heating natural studtite.
**ASTM file No. 16-207.
***dif. = diffuse, v. dif. = very diffuse.

TABLE 5. Unit-Cell Dimensions of the Natural and Synthetic Dihydrate

Dihydrate* (Present study)		$UO_4 \cdot 2\ H_2O$ (Debets, 1963)	
a	6.51 ± 0.01 Å	a	6.502 Å
b†	8.78 ± 0.02	b	4.216
c†	4.21 ± 0.01	c	8.778
$a:b:c$ = 0.741:1:0.479			
V 240.6 ± 1 Å^3		V 240 Å^3	
$Z = 2\ UO_4 \cdot 2\ H_2O$			
Space group probably *Immm*			

*Obtained by heating natural studtite.
†In order to comply with the IMA recommendation c<a<b, b and c as chosen by Debets have been exchanged.

Discussion

The comparison of the unit-cell dimensions of studtite and the dihydrate shows that the *c*-period of the monoclinic cell is not much affected by dehydration. Nor is cell-edge *b* (*a* of the orthorhombic cell). However, *a* (becoming *b*) is reduced as much as 3 Å by dehydration. This behavior can be interpreted by assuming that the four additional water molecules per unit cell which make the difference between studtite and the dihydrate are arranged in layers parallel to (100) in the studtite structure.

Similarities exist between the unit-cell dimensions of studtite and those of members of the schoepite group. The schoepite group comprises uranyl oxide hydrates of different compositions with structures characterized by $UO_2(OH)_2$ layers of pseudohexagonal symmetry. Schoepite itself has the formula $UO_2(OH)_2 \cdot H_2O$. The $UO_2(OH)_2$ layers in members of the schoepite group are stacked parallel to (001) with the distance between consecutive layers varying from 7.1 to 7.6 Å (Christ and Clark, 1960). This distance corresponds to the *c*-period of a pseudo-orthohexagonal cell which can be derived from the true orthorhombic unit cell of these minerals. The constants of the pseudo-cell within the (001) plane range from 6.93 to 7.16 Å for *a* and from 4.03 to 4.21 Å for *b*. These resemble those for the (100) plane of studtite (*b* 6.80; *c* 4.25 Å).

Despite these similarities it is unlikely that close relations exist between the structure of studtite and that of the schoepite group. If one assumes that $UO_2(OH)_2$ layers alternate with layers of water molecules parallel to (100) in studtite, as was thought possible if the mineral had the composition $UO_2(OH)_2 \cdot 4\ H_2O$ instead of $UO_4 \cdot 4\ H_2O$, then the birefringence on the (100) plane should be rather small [as observed by Christ and Clark (1960) for sections parallel to (001) in the schoepite group]. This is not the case. On the contrary, the birefringence of studtite is remarkably high in this orientation, and the crystals moreover are elongated along the axis with the 4.25 Å period, whereas the members of the schoepite group usually are tabular on (001) and show pseudohexagonal forms.

These properties of studtite, on the other hand, could be explained by assuming some kind of chain-like structure along the c-axis. Indeed, substantial arguments favor such an assumption. Another uranium compound is known whose cell constants resemble those of studtite. For example, the hexagonal uranium trioxide α-UO_3, which has *a* 3.971 and *c* 4.168 Å (Zachariasen, 1948), agrees with *c* for studtite. The α-UO_3 structure contains O–U–O–U–O chains along the *c*-axis, with a U–O distance of 2.08 Å. Studtite may possess similar chains along *c*. The additional oxygen and water content of studtite, however, make the cell larger and the structure more complicated in comparison with that of α-UO_3 and produce monoclinic, or in case of the dihydrate, orthorhombic symmetry.

The thermal behavior of studtite lends credence to the correctness of these assumptions. Dehydration of studtite leads to α-UO_3. Schoepite does not form as an intermediate dehydration phase, as might be assumed if very close structural relations existed between studtite and schoepite.

Unknown Siliciferous Mineral

As already mentioned, studtite is intergrown with an unknown species and with uranophane on one of the specimens from Menzenschwand.[2] The three intergrown minerals form yellow fibrous aggregates within a small vug and are macroscopically indistinguishable. At first it was assumed that the unknown species might be a decomposition product of studtite, but this could not be confirmed.

Small fibrous crystals of the unknown mineral are colorless under the microscope and show parallel extinction with positive elongation. Indices of refraction: α 1.570 ± 0.002, γ 1.584 ± 0.002. The crystals are too small to yield good polarization figures. Possibly they are biaxial and have orthorhombic symmetry. The crystals are somewhat flat-

[2] The specimen was found by E. Killius in 1961 during one of the excursions to Menzenschwand undertaken jointly with the author.

tened, the birefringence being lower on the plane of flattening and higher if the crystals are turned on edge. β does not differ markedly from γ which leads to the conclusion that the optical sign is negative. X is normal to the plane of flattening; Z is parallel to the elongation. Anomalous bluish grey interference colors can be observed under crossed nicols.

The mineral, in contrast to the associated studtite and uranophane, is insoluble in cold or hot 1:1 HNO_3 and 1:1 HCl. It is thus easily separated from them.

A sample purified by acid treatment was analyzed by the electron microprobe. Only Si and U could be detected. Whether U is an essential constituent of the mineral, however, is questionable. It might well be that the acid treatment was not sufficient to remove all of the associated uraniferous minerals. The properties of the mineral suggest that it is not a uranyl silicate. Uraniferous silicates are dissolved or decomposed by acids, contrary to the behavior of this mineral.

The powder data are listed on Table 6. The pattern is not closely related to that of any known mineral. A sample heated for two hours at 300°C furnished the same powder pattern as before. The optical properties remained unchanged as well. Heating to 900°C also does not lead to a major change in the powder pattern; however, the indices of refraction are markedly decreased. Further investigations are planned to determine the nature of this evidently new mineral species.

TABLE 6. X-ray Powder Data for the Unknown Mineral

d (Å)	I	d (Å)	I		d (Å)	I	
11.58	9	1.984	3		1.285	2	
7.30	10	1.957	1		1.257	1	
6.19	9	1.924	4		1.245	1	diffuse
5.79	1	1.895	1		1.230	2	diffuse
5.24	4	1.862	4		1.220	0.5	
4.99	4	1.837	3		1.207	0.5	diffuse
4.56	3	1.817	1		1.194	0.5	
4.26	4	1.775	0.5		1.184	0.5	
3.88	5	1.752	0.5		1.177	1	
3.67	4	1.721	3		1.169	2	
3.54	4	1.706	2		1.161	1	
3.50	8	1.695	2		1.151	2	
3.42	6	1.656	3	diffuse	1.141	1	
3.20	6	1.629	0.5		1.132	0.5	
3.08	6	1.615	2		1.122	2	very diffuse
2.99	6	1.597	2		1.109	2	diffuse
2.89	3	1.569	2	diffuse	1.093	0.5	diffuse
2.77	7	1.539	1		1.083	2	
2.67	3	1.513	2		1.078	1	
2.55	2	1.498	0.5		1.071	1	
2.49	1	1.463	3	diffuse	1.064	2	very diffuse
2.44	1	1.437	0.5		1.050	0.5	very diffuse
2.41	2	1.426	2		1.043	0.5	diffuse
2.39	2	1.418	2		1.039	0.5	
2.32	3	1.396	1		1.031	1	very diffuse
2.29	2	1.386	2		1.018	0.5	diffuse
2.26	1	1.372	1		1.008	2	
2.21	3	1.359	0.5		1.004	0.5	very diffuse
2.16	0.5	1.348	1		0.998	3	
2.11	3	1.330	2	diffuse	0.992	0.5	
2.07	3	1.318	2	diffuse	0.990	5	diffuse
2.02	5	1.305	1		0.981	1	diffuse

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