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*Dedicated to the memory of Prof. Bernard Kübler*

## **Rosickýite (monoclinic $\gamma$ -sulphur) from La Presta Asphalt Mine, Neuchâtel, Switzerland: new X-ray powder diffraction data**

by *Nicolas Meisser*<sup>1</sup>, *Kurt J. Schenk*<sup>2</sup> and *Jorge E. Spangenberg*<sup>3</sup>

### **Abstract**

Rosickýite, the natural monoclinic  $\gamma$ -form of sulphur, exists in only a few localities around the globe. In the old asphalt mine at La Presta, Neuchâtel, Switzerland, rosickýite occurs locally as small, but very well formed crystals suitable for crystallographic studies. It grows as an alteration product of pyrite-rich asphalt. Rosickýite from La Presta mine is pure molecular sulphur, as revealed by gas chromatography-mass spectrometry. The X-ray powder diffraction data of La Presta rosickýite does not match the one previously published for this species. Therefore, a single crystal study was undertaken and a new indexed X-ray powder diffraction diagram for natural rosickýite is proposed.

*Keywords:* rosickýite, monoclinic sulphur, asphalt, Presta Mine, Neuchâtel, Switzerland.

### **Introduction**

White synthetic monoclinic  $\gamma$ -sulphur,  $S_8(\gamma)$ , was discovered by MUTHMANN (1890) and poetically called “mother-of-pearl sulphur”. The first description of a natural occurrence was made by SEKANINA (1931) at Havírna near Letovice, in the Czech Republic. It was found in the cavities of limonite  $\pm$  pyrite nodules associated with coal and embedded in a Cenomanian clay stratum. The mineral was named in honour of Professor Vojtěch Rosický from the University of Brno (SEKANINA, 1931). Despite the widespread occurrence of native sulphur ( $\alpha$ -sulphur, orthorhombic) and common monoclinic sulphur ( $\beta$ -sulphur, stable between 94.5–120 °C in fumarolic deposits), rosickýite ( $\gamma$ -sulphur, monoclinic) is particularly rare, and not commonly reported (PALACHE et al., 1944; ANTHONY et al., 1990). This scarcity may be explained by the tendency of rosickýite to revert slowly to  $\alpha$ -sulphur when exposed to room temperature. Apart from the type locality, rosickýite is reported only in few localities: Vulcano, Lipari Island, Italy, with fumarolic native sulphur (PALACHE et al., 1944); Point Rincón, Ventura Co., California, USA, with native

sulphur in an oil-rich matrix (ANTHONY et al., 1990) and the coal-burning dump of Ronneburg, Thüringen, Germany, as fumarolic Se-rich crystals (WITZKE and RÜGER, 1998). Except in fumarolic occurrences, rosickýite appears as an oxidation product in pyritous organic matter-rich medium (SEKANINA, 1931; ANTHONY et al., 1990). The role of organic matter in the formation of monoclinic sulphur has not been clearly established, but slow crystallization of dissolved sulphur in oily matter has been proposed (MILTON, 1951). The rosickýite samples of this study stem from La Presta asphalt mine near Travers, Canton Neuchâtel, Switzerland.

### **Occurrence and appearance**

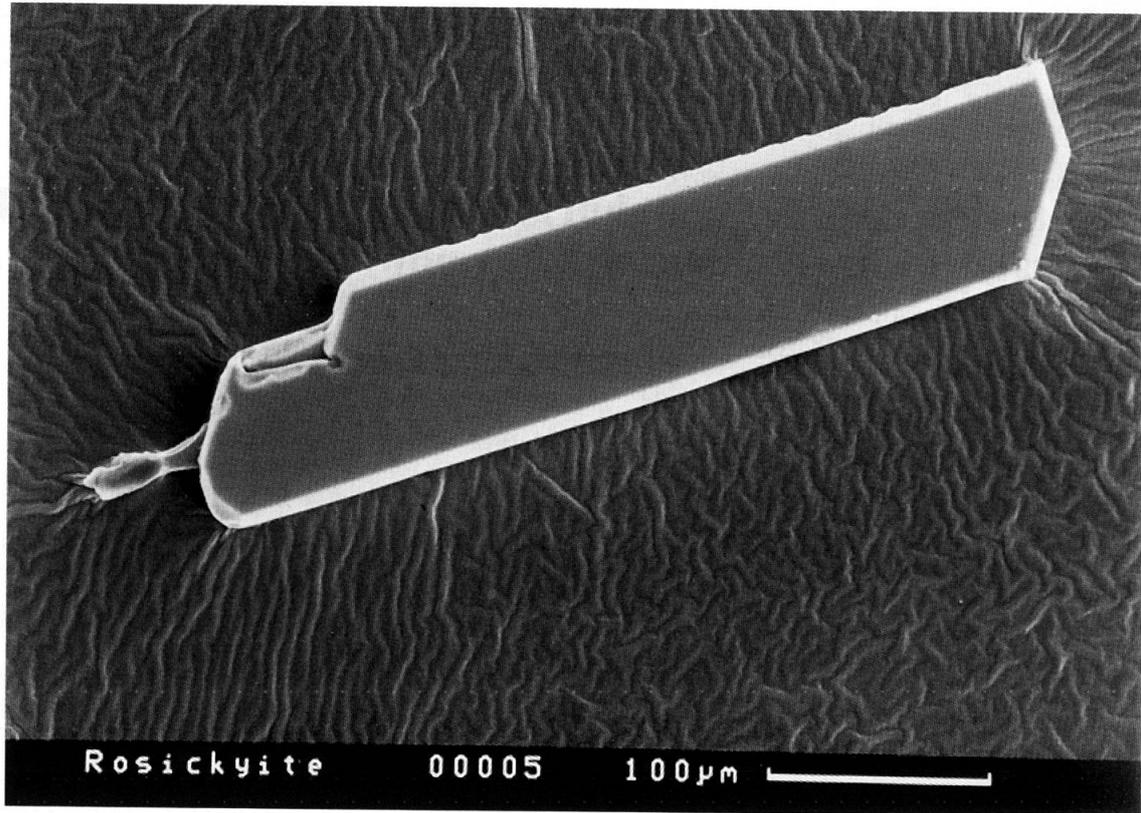
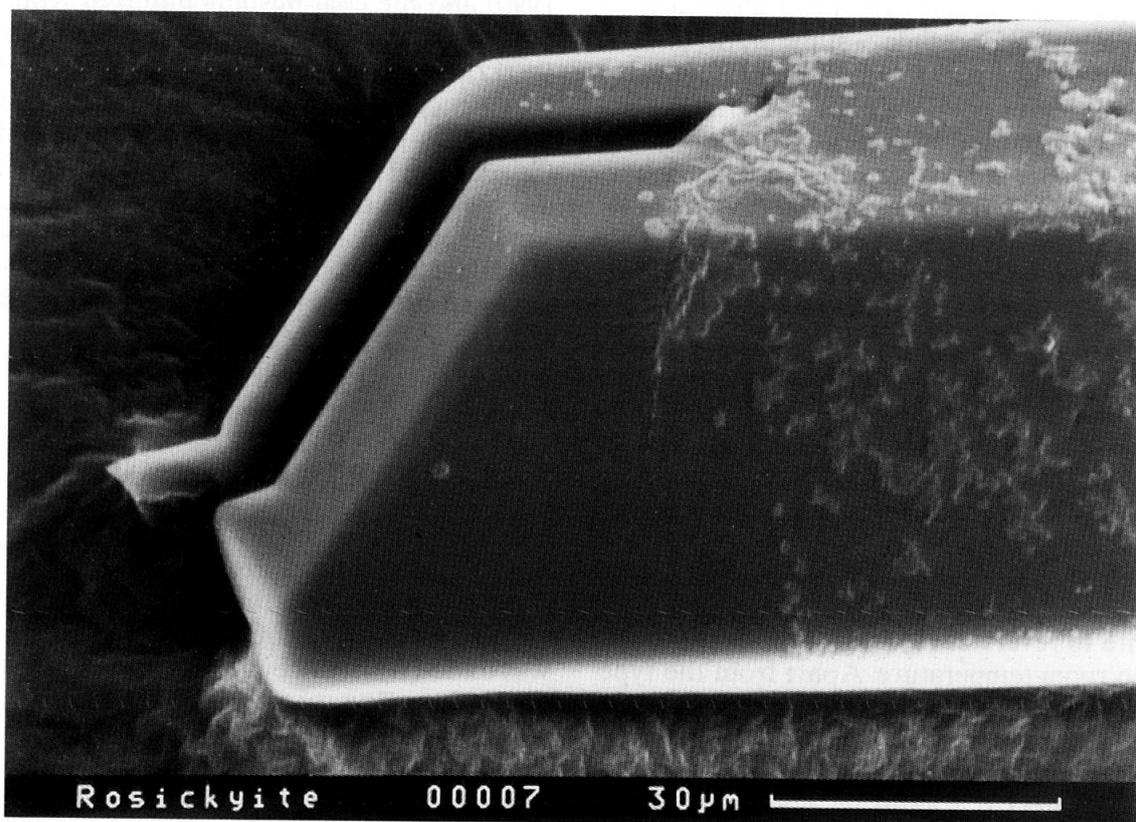
The Lower Cretaceous Barremian and Aptian limestones and marls from Travers, Canton Neuchâtel (Swiss federal coordinates: 540.475/197.050), host an economic asphalt deposit that was mined from the beginning of the 18th century until 1986 (ZWEIDLER, 1985). Today the mine is open for tourism.

Rosickýite appears as fine crystalline efflorescence on the mine walls. It is found in association

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**a****b**

*Fig. 1* Scanning electron micrographs of rosickyite crystals from La Presta Mine, Canton Neuchâtel, Switzerland. Note the well exhibited  $2/m$  holohedry of crystal illustrated in (a) and the presence of possible bacterial forms on faces of crystal illustrated in (b).

with asphalt, calcite, pyrite, gypsum, rozenite, limonite and native sulphur ( $\alpha$ -sulphur). The crystals grew with a prismatic form, up to 1 mm in length, on a soft, partially liquid, asphalt-calcite matrix. Crystals of rosickýite are colourless to very pale yellow (as opposed to the strongly yellow orthorhombic native sulphur). They show iridescent interference colours on the (010) face due to their thickness (10–15  $\mu\text{m}$ ) and strong dispersion.

The crystal morphology was studied with a scanning electron microscope (Fig. 1) and its growth planes were indexed based on the cell presented in table 2. Rosickýite crystals are tabular {010} and elongated along [100]. In some rare cases, the facies is strongly acicular. Other observed forms are: {110}, {012}, {−112} and {−111}.  $2/m$  holoedry is well exhibited. When crystals are scratched with a steel needle, {101} and {−101} slip planes are created. The index of refraction is higher than 2.0, but it could not be measured precisely using the standard immersion method because rosickýite is soluble in refractive index liquids ( $\text{CH}_2\text{I}_2$ – $\text{AsBr}_3$ – $\text{As}_2\text{S}_3$  solution).

### Chemical composition

Gas chromatography was carried out on carefully hand-picked rosickýite crystals dissolved in dichloromethane using a Hewlett-Packard G1800A GCD system, based on a HP 5890 Series II gas chromatograph, with an electron ionization detector (EID). The system was equipped with an HP-ULTRA-2 fused-silica capillary column (30 m  $\times$  0.20 mm i.d., coated with 0.11  $\mu\text{m}$  cross-linked 5%-diphenyl-95%-dimethyl siloxanes as stationary phase) and helium was used as the carrier gas (flow rate equal to 1 ml/min). The samples were injected splitless at a temperature of 275 °C. After an initial isothermal period of 2 min at 70 °C, the column was heated to 280 °C at 3 °C/min followed by an isothermal period of 30 min. The EID was operated at 70 eV in the multiple ion detection mode, source temperature of 250 °C, emission current of 1 mA and a scan range from 45 to 450 amu. Data were processed with an HP Chemstation data system, and the single peak was recognized as  $\text{S}_8$  by comparing the mass spectra with the MS NIST98 spectra library.

### Sulphur isotope analyses by EA-IRMS

The sulphur isotope composition of the rosickýite and the pyritous asphalt were analyzed by means of combustion isotope-ratio-monitoring mass

Tab. 1 Comparison between observed and calculated X-ray powder diffraction data for rosickýite.

<i>h</i>	<i>k</i>	<i>l</i>	<i>d</i> obs. [Å]	<i>d</i> calc [Å]	<i>I</i> obs.	<i>I</i> calc.
0	1	1	6.61	6.5728	30	17
0	2	0		6.5440		8
1	1	0	6.17	6.1379	30	27
−1	1	2	4.37	4.3674	100	66
0	3	0		4.3626		1
−2	0	2	3.93	3.9117	10	13
1	1	1		3.9064		1
−2	1	2	3.75	3.7479	100	100
1	3	0	3.69	3.6949	30	61
0	1	2	3.66	3.6497	80	96
2	0	0	3.48	3.4747	40	37
1	2	1		3.4701		4
−2	2	2	3.38	3.3576	10	6
0	2	2	3.28	3.2864	20	9
0	4	0		3.2720		15
−1	3	2	3.18	3.1761	80	62
2	2	0	3.06	3.0689	60	48
−2	3	1	3.02	3.0245	10	18
0	4	1	2.99	3.0053	10	11
1	3	1		2.9850		10
−3	1	2	2.75	2.7590	10	1
−2	2	3		2.7535		10
2	1	1	2.58	2.5878	30	8
−2	4	1		2.5804		11
−3	2	1	2.485	2.4846	40	16
−3	2	3	2.433	2.4225	10	7
0	2	3	2.374	2.3627	10	4
−1	4	3	2.209	2.2094	10	7
0	3	3	2.194	2.1909	5	6
−4	0	2	2.099	2.0983	5	1
−2	5	3	1.983	1.9823	20	12
1	6	1	1.929	1.9250	10	4
1	5	2	1.893	1.8924	20	3
−3	5	3	1.847	1.8474	30	1
−1	4	4	1.812	1.8126	20	2
−4	3	4	1.784	1.7847	20	1
3	3	1	1.765	1.7644	20	1
−2	5	4	1.732	1.7347	10	2
−2	7	1	1.713	1.7079	5	4
4	2	0	1.679	1.6792	10	3
−5	2	3	1.641	1.6404	20	4
−3	4	5	1.608	1.6068	10	4
3	6	0	1.588	1.5881	20	1

spectrometry using a Carlo Erba 1108 elemental analyzer (EA) connected to a Finnigan MAT Delta S isotope ratio mass spectrometer (IRMS) via a ConFlo II split interface (EA/IRMS). The EA oxidizes all the sample compounds under a stream of helium and oxygen gas by flash combustion in a single oxidation-reduction quartz tube, filled with oxidizing ( $\text{WO}_3$ ) and reducing (elemental Cu) agents at 1030 °C. Water was removed using anhydrous  $\text{Mg}(\text{ClO}_4)_2$ , and the gases enter a chromatographic column (Poropak QS) for sepa-

Tab. 2 Comparison between data for rosickýite from La Presta mine and previously published data.

	This study	GALLACHER and PINKERTON (1993)	WATANABE (1974)
Molecular formula	S <sub>8</sub>	S <sub>8</sub>	S <sub>8</sub>
δ <sup>34</sup> S (CDT)	13.2‰		
Unit cell dimensions	a = 8.476(2) Å b = 13.088(3) Å c = 9.270(2) Å β = 124.92(2)°	a = 8.455(3) Å b = 13.052(2) Å c = 9.267(3) Å β = 124.89(3)°	a = 8.442(30) Å b = 13.025(10) Å c = 9.356(50) Å β = 124.98(30)°
Volume	843.2(3) Å <sup>3</sup>	838.8(9) Å <sup>3</sup>	843.0(5) Å <sup>3</sup>
Space group	P2/c	P2/c	P2/c
Z	4	4	4
Density (calculated)	2.02 kg dm <sup>-3</sup>	2.03 kg dm <sup>-3</sup>	2.08 kg dm <sup>-3</sup>

ration of SO<sub>2</sub> which is isotopically analyzed by IRMS (GIESEMANN et al., 1994). The sulphur isotope values are reported relative to the Canyon Diablo troilite (CDT) standard. The reproducibility, assessed by multiple analyses of a laboratory standard (BaSO<sub>4</sub>, working value = +12.5‰ δ<sup>34</sup>S), was better than 0.2‰ (1σ). The values for pyritous asphalt are +30.2 and +29.5‰ and for rosickýite +13.2‰.

### X-ray diffraction

The X-ray powder pattern of rosickýite was obtained using a Gandolfi camera (114.6 mm diameter), CuKα radiation at 40 kV, 30 mA. Table 1 shows that the observed *d* values and visually estimated intensities are in excellent agreement with the powder pattern calculated from the single crystal study.

An optically pure crystal with perfect extinction in polarized light was isolated and mounted for X-ray data collection at 293 K on a STOE IPDS equipped with a Long Fine Focus (LFF) Mo tube operated at 2.44 kW. The studied crystal was tabular to prismatic and measured 369 × 66 × 18 μm. An inspection of reciprocal space showed a very pure diffraction figure, the spots of which could all be indexed by means of the cell given in table 2. In particular there was no indication of twinning, superstructure or diffuse scattering.

### Discussion

Similar to the type-locality of Havírna (SEKANI-NA, 1931), the rosickýite specimen from Val de Travers is an intermediate oxidation product formed during the conversion of pyrite to sulfate ion-containing mineral species (gypsum and rozenite, FeSO<sub>4</sub> · 4H<sub>2</sub>O) which are observed in the

paragenesis. This mode of occurrence has frequently been reported in the oxidized zones of base metal ores (PALACHE et al., 1944). In this case, it is native sulphur (α-sulphur) which is found in these environments (WILLIAMS, 1990). Regarding the δ<sup>34</sup>S of rosickýite and the pyritous asphaltic matrix, rosickýite is enriched in <sup>32</sup>S. This observation is an indication for microbial (e. g. *Thiobacillus* and *Ferroxidans* spp.) (Fig. 1b) fractionation during oxidation processes (FEELY AND KULP, 1957).

Lattice constants, space group and structure of the investigated rosickýite single crystal are in good agreement with those published by WATANABE (1974) and GALLACHER and PINKERTON (1993). The X-ray powder diffraction for γ-sulphur, synthetic rosickýite, proposed by PINKUS et al. (1959) strongly differs with those recorded in our study. As previously noticed by PINKUS et al. (1959), it appears that their material was a complex mixture of α-, β- and γ-sulphur. Our proposed X-ray powder diffraction for natural γ-sulphur, rosickýite, is fully indexable on the basis of the results from the single crystal study.

With regard to the often discussed metastability of the γ-sulphur modification (see e.g. MUTHMANN, 1890) it is noteworthy that our specimen was collected in 1989 and that it was still optically homogeneous without domains or fissures ten years later. Even more intriguing is the coexistence of the natural α- and γ-sulphur modifications in small vugs in the asphalt matrix. Not less surprising is the observation that after dissolving, at room temperature, the natural γ-sulphur modification, rosickýite, in *n*-propanol or carbon disulfide it is again the γ-phase that crystallizes (there is not the faintest hint of the α-phase in the pure Gandolfi X-ray powder diagrams of the recrystallization product).

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