Synthesis of Calcium Carbonate ($CaCO_3$) from Egg Shell and Snail Shell.

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Abstract
The calcium carbonate ($CaCO_3$) derived from the egg shell of hen and snail shell can be used as fillers in the polymer industries. $CaCO_3$ was obtained from egg shell and snail shell, this was done by crushing the shell and sieving with a 60 micron mesh. This shell was calcinated in a furnace at 640 ± 10 $^oC$for 2 hours 30 minutes. The powder was characterised by Fourier Transform Infra-Red Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD). The SEM showed that the $CaCO_3$ had an irregular shape while the FTIR spectra revealed peaks indicative of the presence of $CaCO_3$. The XRD confirmed that $CaCO_3$ was formed.

Keyword:*Calcium carbonate, egg shell, snail shell.*

1. INTRODUCTION

Egg and snail shell are important sources of protein for human being. However, their shells are mostly discarded as waste into the environment. The indiscriminate disposal of shell has raised concern for the proper recycling of this waste mostly in third world countries like Nigeria with emerging waste management systems.

Most of the shell waste is deposited in landfills, abandoned on land, or returned to the sea, thereby causing environmental impacts. The waste products when deposited in the soil, contaminate and attract animals due to the strong odour. When dropped in sea, it causes grounding and infects the marine population (Silva, Mesquita-Guimaraes, Henriques, Silva and Fredel, 2019).

$CaCO_3$ is found in varieties of places like sea shells, calcitic rocks, coral reefs, stalactites and stalagmites formations in the caves (Sever, 2013). Seashell has 95 – 99 % by weight of $CaCO_3$ which aid its application in quite a number of purposes (Mohamed, Yusup and Maitra, 2012). Kiranda, Mahmud, Abubakar and Zakaria (2018) stated that $CaCO_3$ as a raw natural mineral has been used in a wide range of applications including biomedical, industrial, and nanotechnology.

Sezer (2013) stated that $CaCO_3$ is used as filling agents, fillers in paper making industries, sealant, plastic and paint industries. Hamester, Balzer and Becker (2012) reported that $CaCO_3$ is the most widely used filler in polymer industries. The cheapest grades are used to reduce cost while their finest grades are used to modify various properties. Sasikumar and Vijayaraghavan (2016), stated that egg shells are useless after the utilisation of egg contents and wasted. They informed that egg shells lead to environmental pollution since these favour microbial growth. These wastes according to them are available in huge quantity from food processing industries, egg baking and hatching industries.

2. MATERIALS AND METHODS

2.1 MATERIALS

The materials used included;egg shell, snail shell and distilled water.

2.2 EQUIPMENTS

Equipments used in the research are; electronic weighing scale, beaker, pH paper, filter paper, laboratory furnace, crucible pot, crucible tong, desicator, motar and pestle.

2.3 METHODS

2.3.1 MATERIAL PROCESSING PROCEDURE

The shells were washed, dried and crushed by grinding in a mill. The crushed shells were sieved using a mesh of 60 micron. The sievedshells were calcinated in a laboratory furnace at 640 ± 10 ^{o}C for 2 hours 30 minutes. The powders were crushed with motar and pestle and then characterised using FTIR (Fourier Transform Infra-Red Spectroscopy), SEM (Scanning Electron Microscope) and XRD (X-Ray Diffraction).

2.3.2 FOURIER TRANSFORM INFRA- RED SPECTROSCOPY (FTIR)

The FTIR machine model Cary 630 by Agilent Technologies, USA was used, FTIR spectroscopy uses infra-red radiation (IR) beam to identify chemical bonds in a molecule by producing infra-red absorption spectrum. The FTIR spectroscopy was done as per ASTM E168.

2.3.3 SCANNING ELECTRON MICROSCOPY (SEM)

SEM was used to study the surface morphology of the powder and the composition. The SEM model is PhenomProx, a product of Phenom World Eindhoven Netherlands. The SEM analysis was conducted via ASTM E2809.

2.3.4 X-RAY DIFFRACTION (XRD)

The XRD analysis was carried out via ASTM D5357. XRD was used to identify the crystal structure of the powder.

3. RESULT AND DISCUSSION

3.1 FTIR Spectroscopy result

The FTIR spectroscopy revealed calcium (Ca^{2+}) spectra at 1796.6 cm^{-1}, Ca^{2+} peak for egg shell was seen at 2512.2 cm^{-1} while Ca^{2+} peak for snail shell was observed at 2322.1 cm^{-1}. Hydroxyl stretching mode was observed at 3570 cm^{-1}. FTIR spectra for egg shell revealed characteristics peaks for $CaCO_3$ at 711.8 cm^{-1} and 872.2 cm^{-1} and for snail shell at 711.9 cm^{-1} and 872.2 cm^{-1}.

3.2 Scanning Electron Microscope result

The SEM morphology of both egg and snail shell are irregular in shape. SEM morphology at magnification of 350X showed the egg shell to be 764 µm while the snail shell is 766 µm. For magnification of 500X both egg and snail shell are 536 µm. At magnification of 1000X egg and snail shell was 268 µm. At magnification of 1500X both samples are 179 µm. At magnification of 2000X the two samples was 134 µm.

3.3 X-ray diffraction result

The XRD revealed that egg shell had $CaCO_3$ and calcium oxide (CaO) while the snail shell had $CaCO_3$.

3.4 DISCUSSION

The SEM morphology of both egg and snail shell were irregular in shape, this is in line with the observed morphology of oyster and mussel shell which was irregular in shape too as seen in the work of Hamester et al., (2012).

Moreover, the XRD result obtained in this research is in agreement with what was obtained by Hamester et al., (2012). Reig, Gimeno-Adelantado and Moya-Moreno (2002) were able to determine the FTIR band for $CaCO_3$ at 872.2 cm^{-1} and 711.8 cm^{-1} for egg shell and 872.2 cm^{-1} and 711.9 cm^{-1} for snail shell again this agrees totally with our results.

Hamester et al., (2012) reported that the shellfish was milled and heated at 500 ^{0}C for 2 hours also in this research the shells (egg and snail) were calcinated to 640 ± 10 ^{o}C for 2 hours 30 minutes.

4. CONCLUSION

This research has derived $CaCO_3$ from egg and snail shell. The $CaCO_3$ exhibited the properties which were in conformity to those derived from some crustacean shell and other shells. This was revealed by FTIR spectroscopy, SEM and XRD.

ACKNOWLEDGEMENT

We are thankful to the following; the department of Polymer and Textile Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Owerri, A. B. U., Zaira and Engr Dr Henry Opara.

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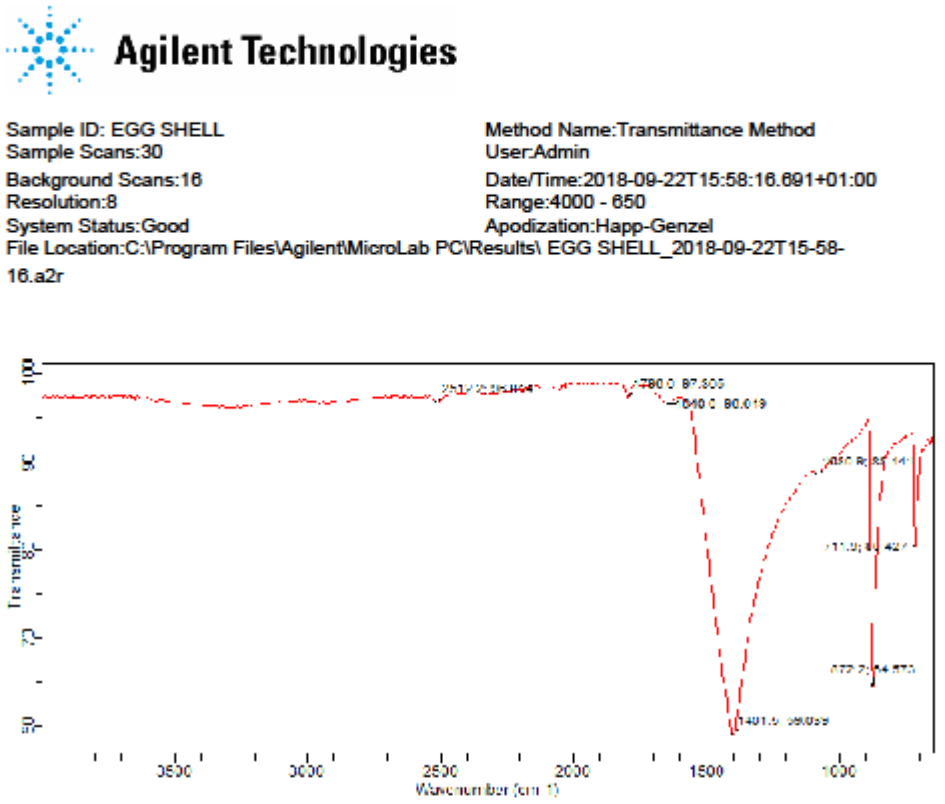

Figure 1: FTIR Spectroscopy of Egg Shell

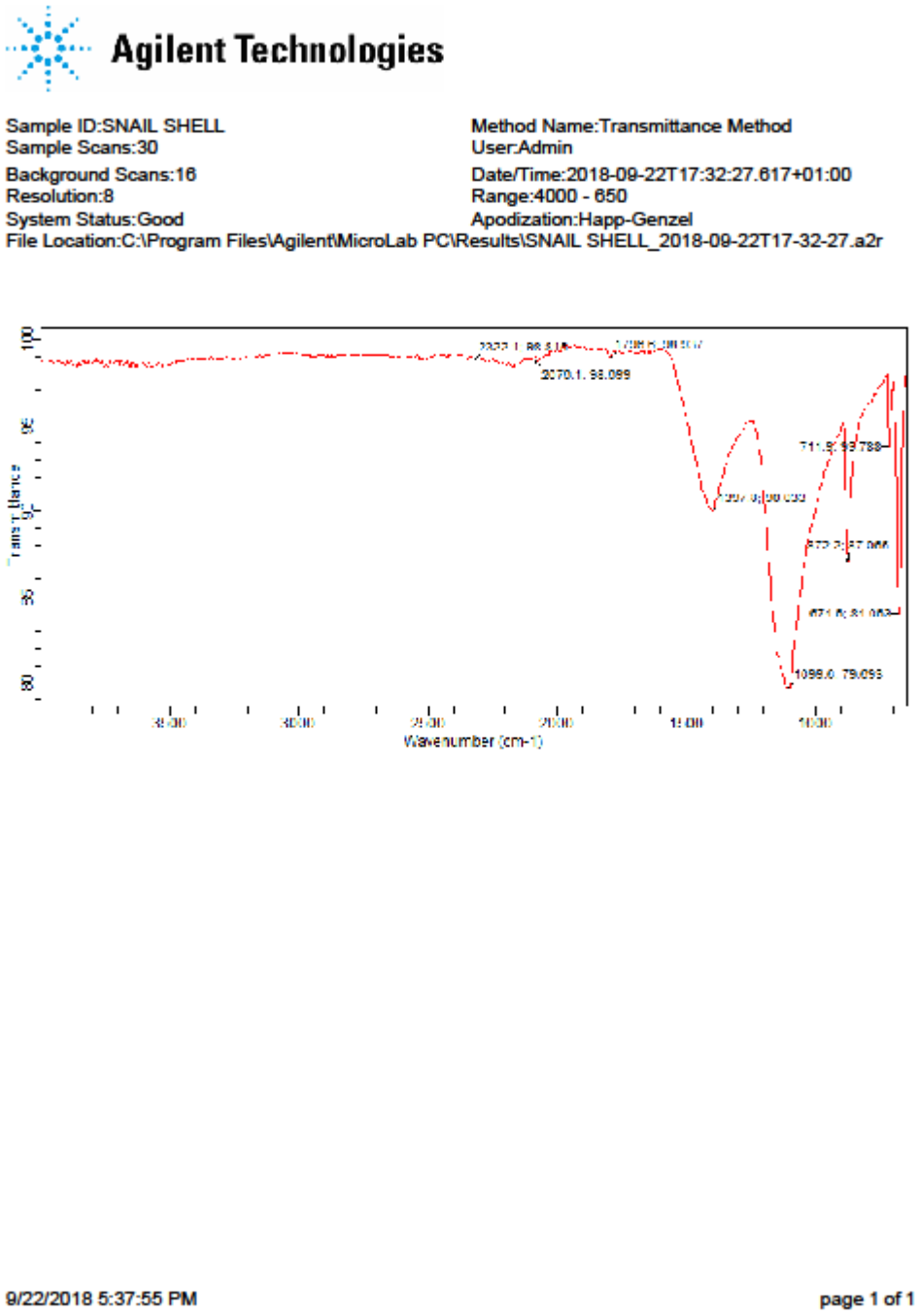

Figure 2: FTIR Spectroscopy of Snail Shell

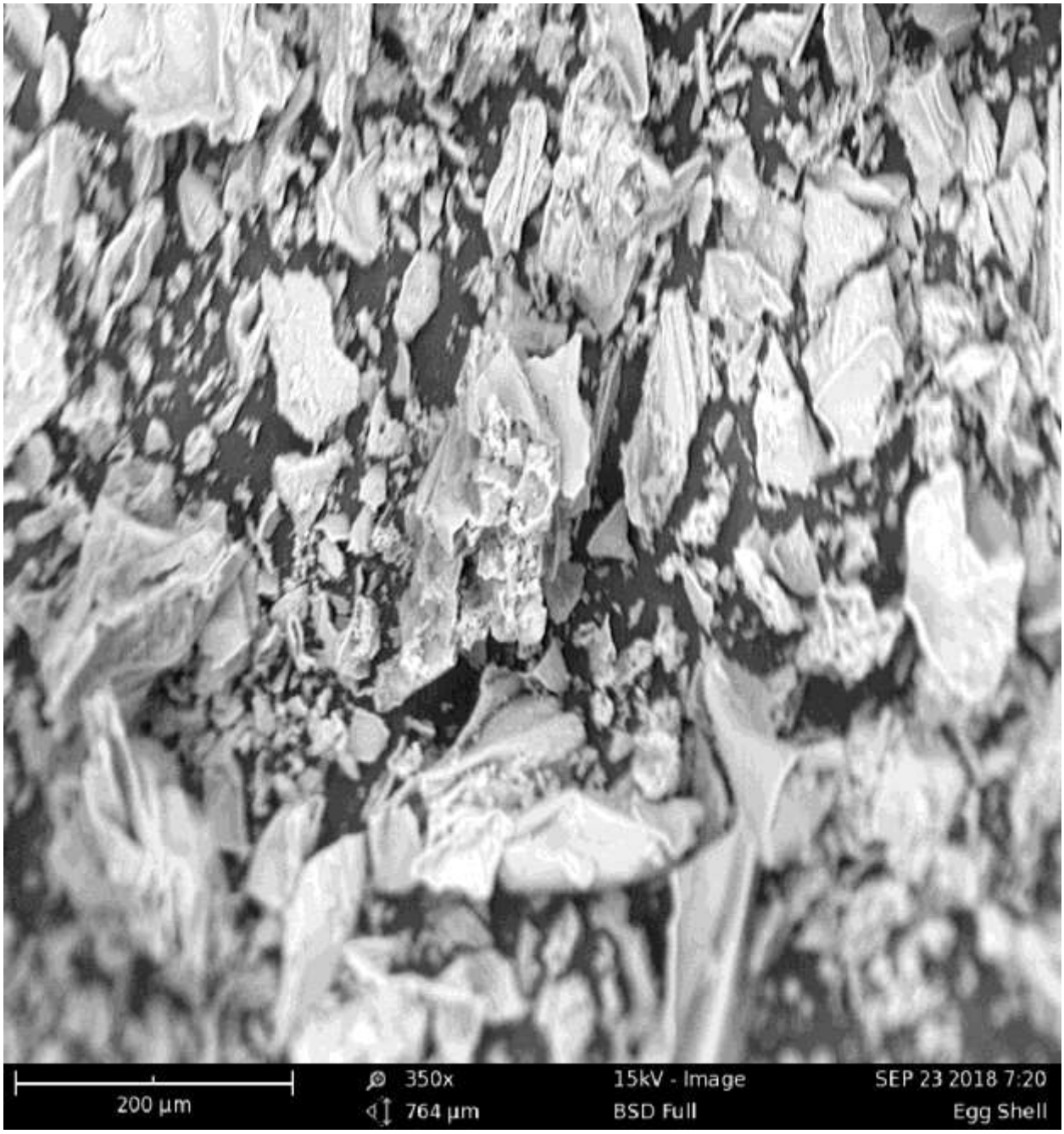

Figure 3: SEM Spectroscopy of Egg Shell at 350X magnification

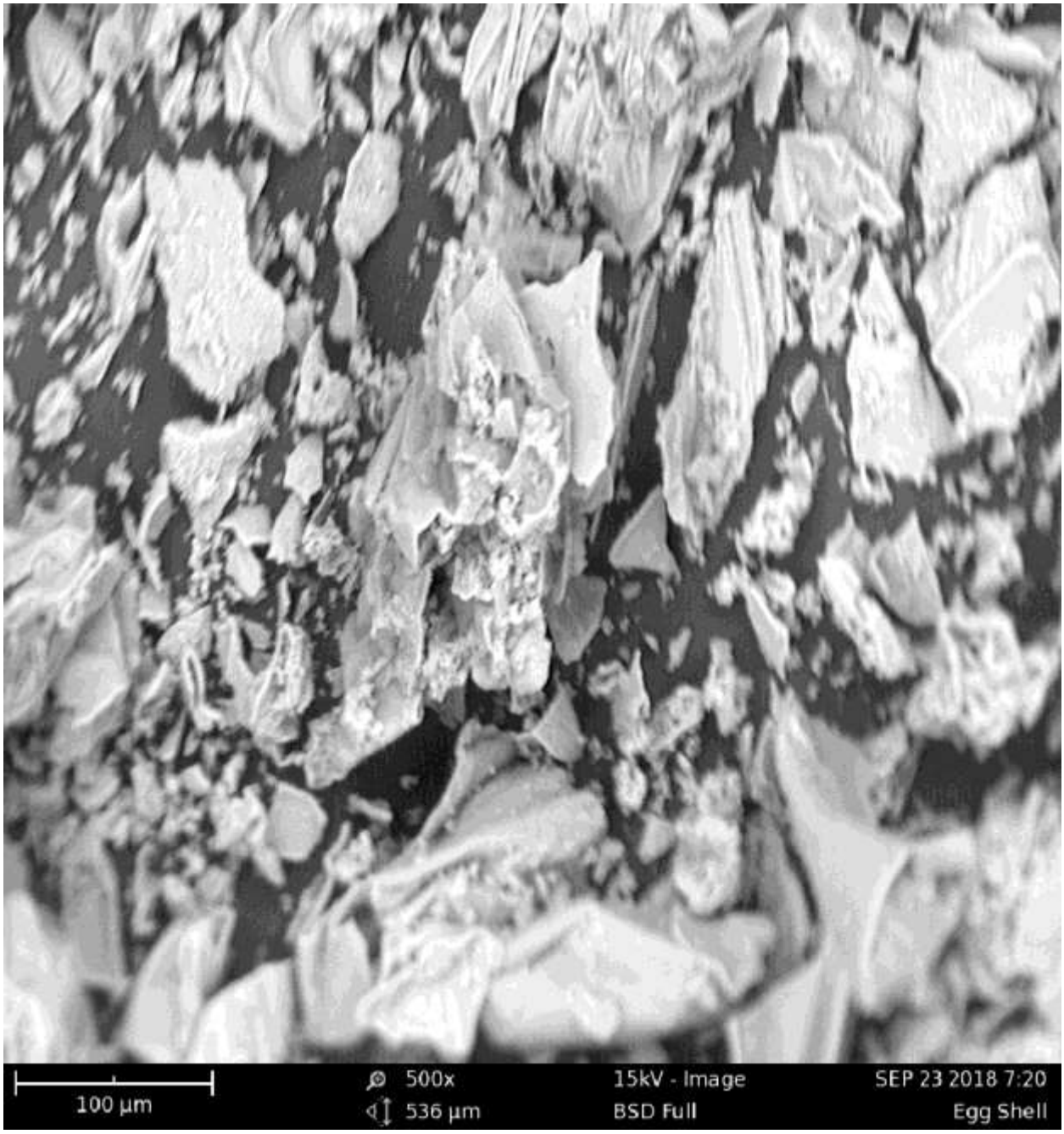

Figure 4: SEM Spectroscopy of Egg Shell at 500X magnification

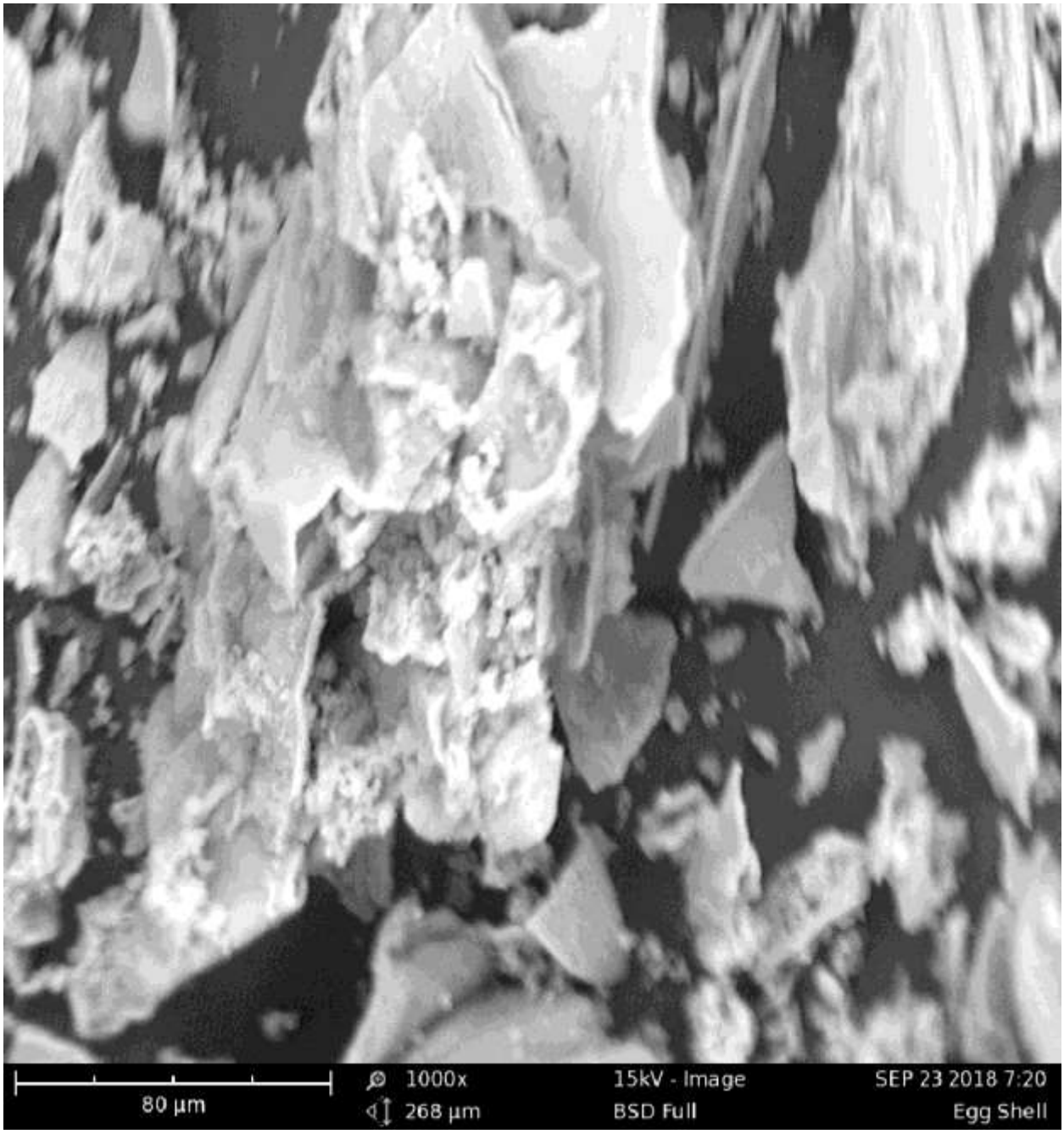

Figure 5: SEM Spectroscopy of Egg Shell at 1000X magnification

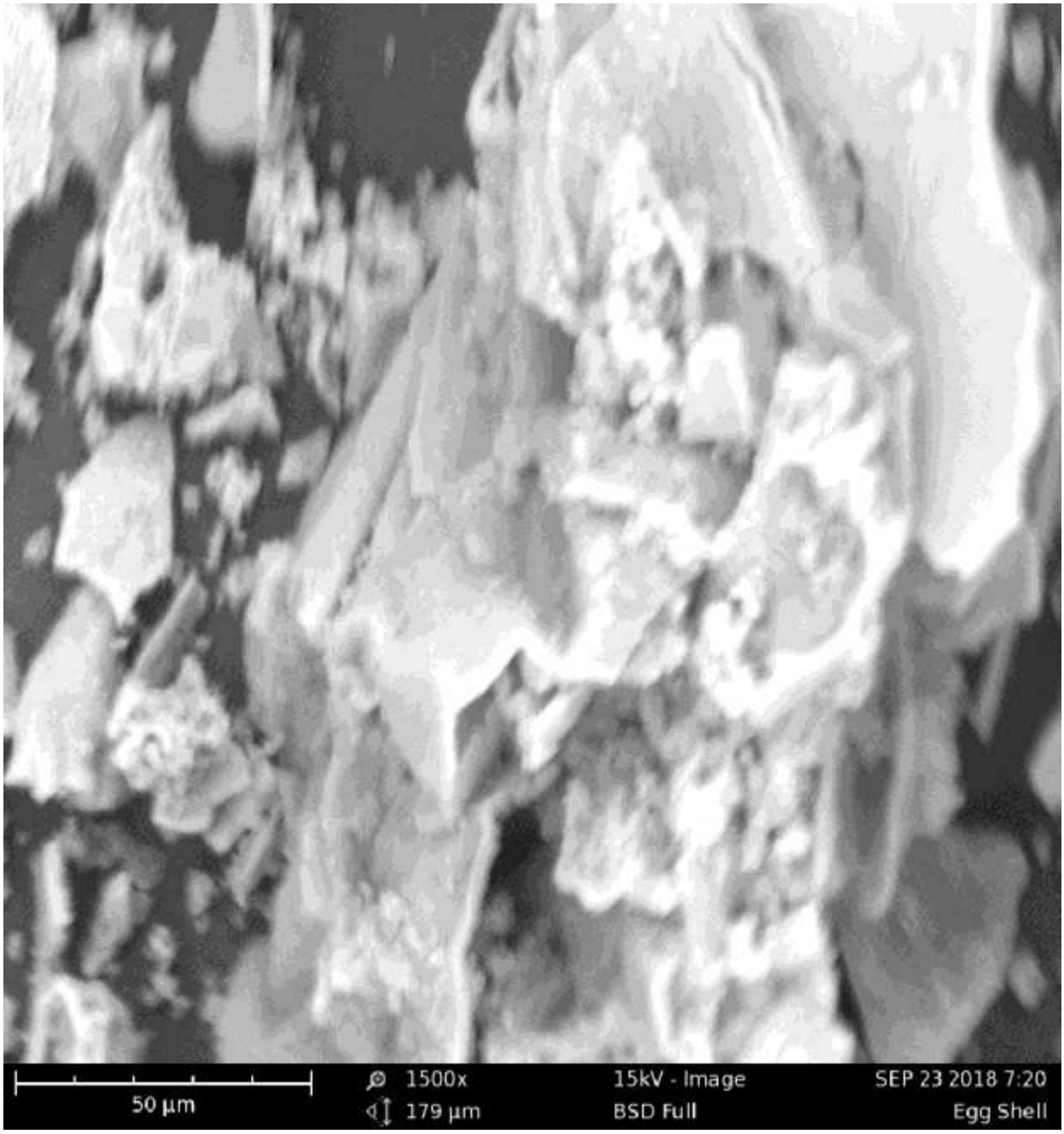

Figure 6: SEM Spectroscopy of Egg Shell at 1500X magnification

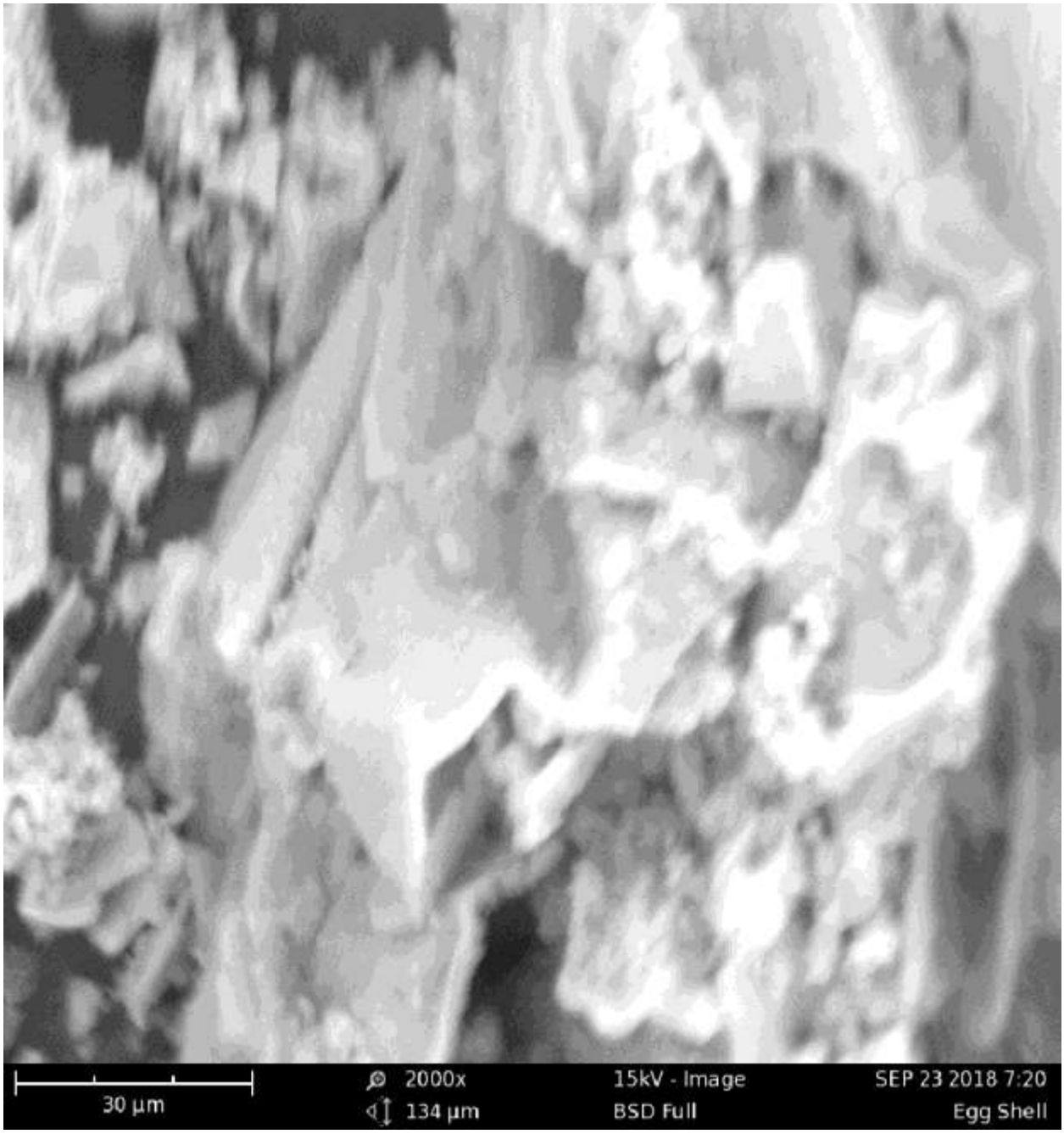

Figure 7: SEM Spectroscopy of Egg Shell at 2000X magnification

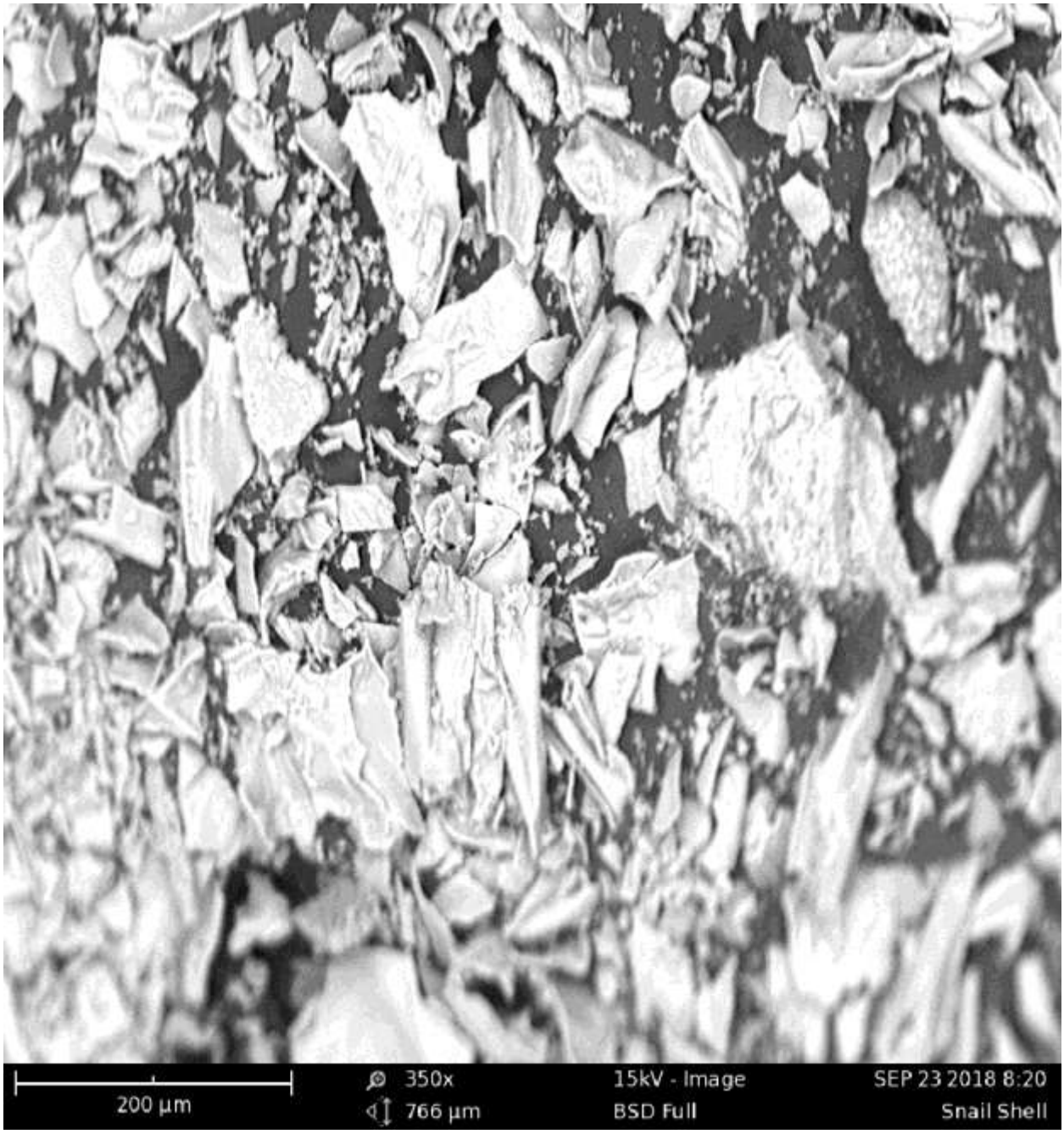

Figure 8: SEM Spectroscopy of Snail Shell at 350X magnification

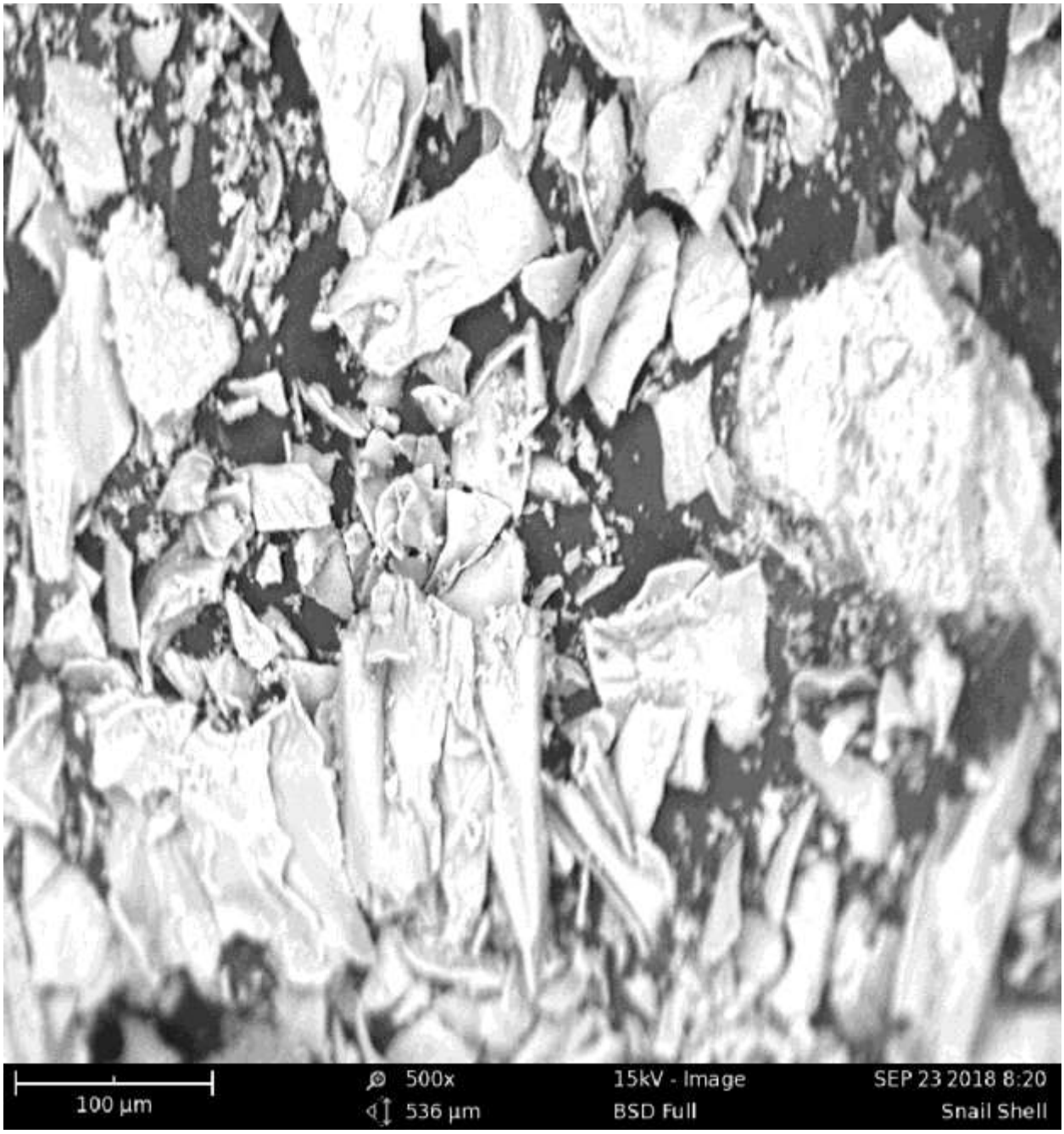

Figure 9: SEM Spectroscopy of Snail Shell at 500X magnification

Figure 10: SEM Spectroscopy of Snail Shell at 1000X magnification

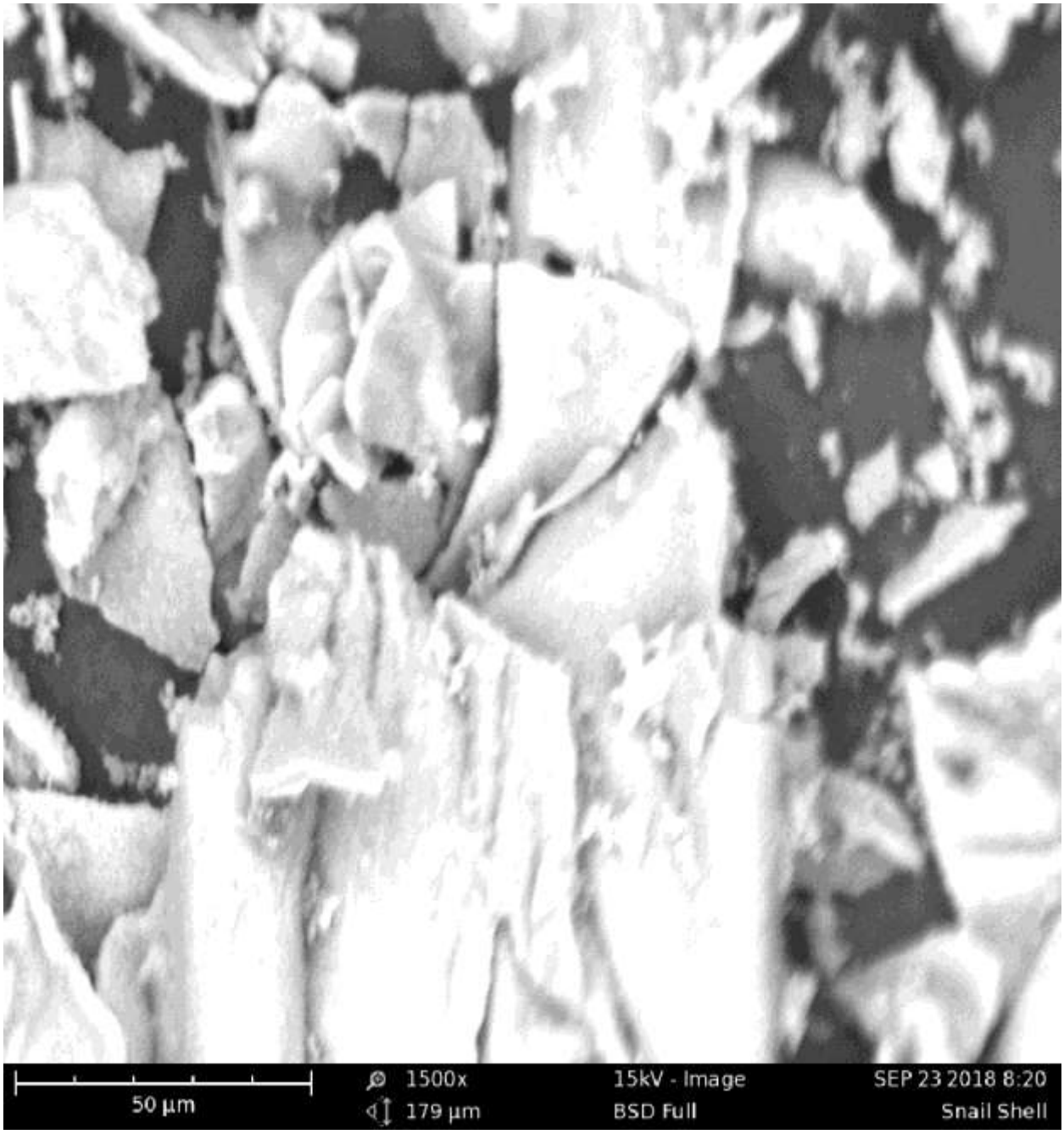

Figure 11: SEM Spectroscopy of Snail Shell at 1500X magnification

Figure 12: SEM Spectroscopy of Snail Shell at 2000X magnification

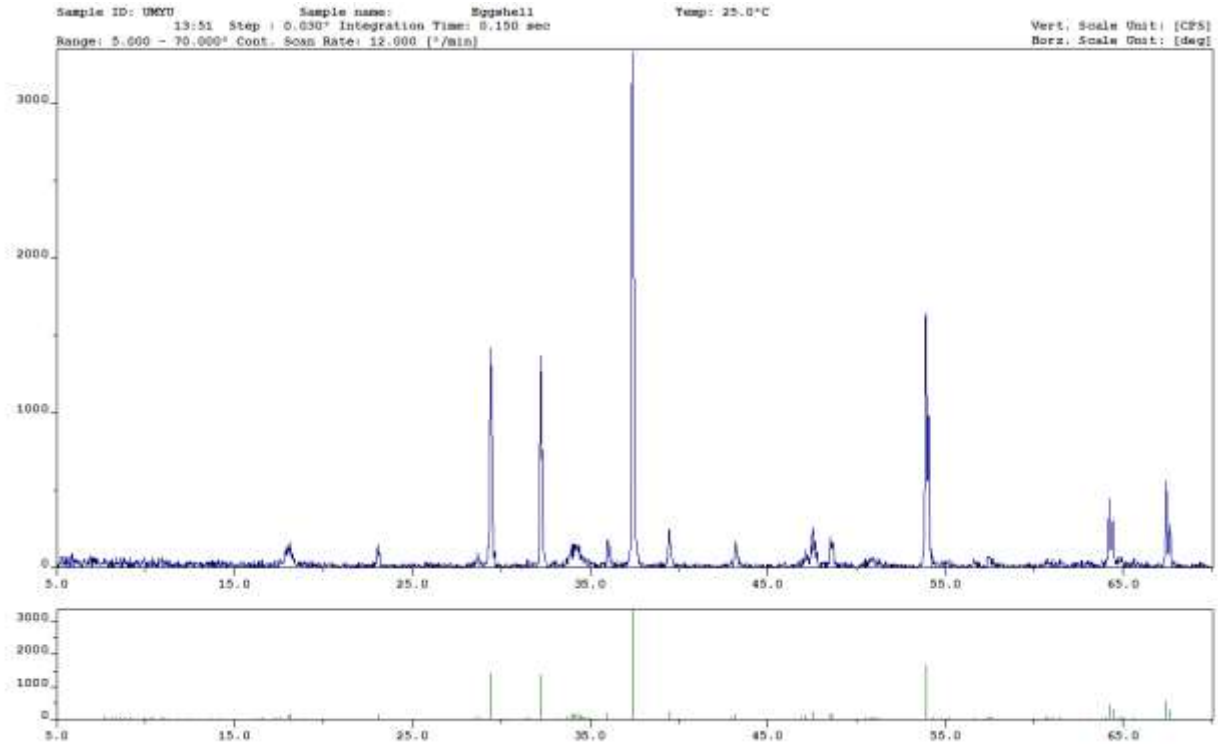

Figure 13: XRD of Egg Shell

Table 1: XRD result of Egg Shell

00-005-0586 Sep 22, 2018 10:57 AM (ARLservice)

Status Primary QM: Star (S) Pressure/Temperature: Ambient Chemical Formula: Ca C O3 Weight %: C12.00 Ca40.04 O47.95
Atomic %: C20.00 Ca20.00 O60.00 Compound Name: Calcium Carbonate Mineral Name: Calcite, syn

Radiation: CuKā1 : 1.5405Å Intensity: Diffractometer I/Ic: 2.0 Reference: Swanson, Fuyat. Natl. Bur. Stand. (U.S.), Circ. 539 II, 51 (1953).

SYS: Rhombohedral SPGR: R-3c (167) AuthCellVol: 367.78 Z: 6.00
Author's Cell [AuthCell-a: 4.989Å AuthCell-c: 17.062Å AuthCellVol: 367.78Å³] Dcalc: 2.711g/cm³ Dmeas: 2.71g/cm³
SS/FOM: F(30) = 57.2(0.0159, 33) Reference: Ibid.

Space Group: R-3c (167) Z: 6.00 Molecular Weight: 100.09
Crystal Data [XtlCell-a: 4.989Å XtlCell-b: 4.989Å XtlCell-c: 17.062Å XtlCell.: 90.00° XtlCell.: 90.00° XtlCell.: 120.00° XtlCellVol: 367.78Å³]
Crystal Data Axial Ratio [c/a: 3.4199]
Reduced Cell [RedCell-a: 4.989Å RedCell-b: 4.989Å RedCell-c: 6.375Å RedCell.: 66.97° RedCell.: 66.97° RedCell.: 60.00° RedCellVol: 122.59Å³]

: ε=1.487 : ε=1.659 Sign: - Reference: II, 142

Crystal (Symmetry Allowed): Centrosymmetric

CAS: 13397-26-7 Pearson: hR10.00 Mineral Classification: Calcite (Supergroup), calcite (Group)

Subfile(s): Cement and Hydration Product, Common Phase, Educational Pattern, Forensic, Inorganic, Mineral Related (Mineral ,Synthetic), NBS Pattern, Pharmaceutical (Excipient), Pigment/Dye, Primary Pattern, Superconducting Material (Superconductor Releted Materials)

Last Modification Date: 01/29/2008
Cross-Ref PDF #'s: 01-072-1214 (Alternate), 01-072-1937 (Alternate), 01-072-4582 (Alternate), 01-081-2027 (Alternate), 01-083-0577 (Alternate), 01-083-0578 (Alternate), 01-086-0

Database Comments: Additional Patterns: See PDF 01-072-1214 , 01-072-1937, 01-081-2027, 01-083-0577 and 01-083-0578. Analysis: Spectroscopic analysis: <0.1% Sr; <0.01% Ba; <0.001% Al, B, Cs, Cu, K, Mg, Na, Si, Sn; <0.0001% Ag, Cr, Fe, Li, Mn. Color: Colorless. General Comments: Additional weak reflections (indicated by brackets) were observed. Other form: aragonite. Pattern reviewed by Parks, J., McCarthy, G., North Dakota State Univ., Fargo, North Dakota, USA, ICDD Grant-In-Aid (1992). Agrees well with experimental and calculated patterns. Antacid. Sample Source or Locality: Sample from Mallinckrodt Chemical Works. Temperature of Data Collection: Pattern taken at 299 K. Unit Cell Data Source: Powder Diffraction.

00-005-0586 (Fixed Slit Intensity) - Cu K1 1.54056Å

2	d(Å)	I	h	k	l	*	2	d(Å)	I	h	k	l	*	2	d(Å)	I	h	k	l	*	2	d(Å)	I	h	k	l	*
23.0218	3.860000	12	0	1	2		60.6762	1.525000	5	2	1	4		80.9303	1.186900	<1	3	1	2		102.2390	0.989500	<1	3	2	1	
29.4049	3.035000	100	1	0	4		60.9856	1.518000	4	2	0	8		81.5451	1.179500	3	2	1	10		102.9480	0.984600	1	2	3	2	
31.4176	2.845000	3	0	0	6		61.3437	1.510000	3	1	1	9		82.1106	1.172800	<1	0	1	14		103.8950	0.978200	1	1	3	10	
35.9654	2.495000	14	1	1	0		63.0586	1.473000	2	1	2	5		83.7647	1.153800	3	1	3	4		104.1200	0.976700	3	1	2	14	
39.4009	2.285000	18	1	1	3		64.6766	1.440000	5	3	0	0		84.7851	1.142500	1	2	2	6		105.8420	0.965500	2	3	2	4	
43.1446	2.095000	18	2	0	2		65.5974	1.422000	3	0	0	12		86.4805	1.124400	<1	1	2	11		106.1420	0.963600	4	0	4	8	
47.1226	1.927000	5	0	2	4		69.2293	1.356000	1	2	1	7		93.0690	1.061300	1	2	0	14		107.3290	0.956200	<1	0	2	16	
47.4886	1.913000	17	0	1	8		70.2366	1.339000	2	0	2	10		94.6973	1.047300	3	4	0	4		109.5560	0.942900	2	4	1	0	
48.5122	1.875000	17	1	1	6		72.8676	1.297000	2	1	2	8		95.0073	1.044700	4	3	1	8		110.4790	0.937600	2	2	2	12	
56.5530	1.626000	4	2	1	1		73.7265	1.284000	1	3	0	6		96.1618	1.035200	2	1	0	16								
57.4001	1.604000	8	1	2	2		76.2979	1.247000	1	2	2	0		97.6440	1.023400	<1	2	1	13								
58.0733	1.587000	2	1	0	10		77.1751	1.235000	2	1	1	12		99.1572	1.011800	2	3	0	12								

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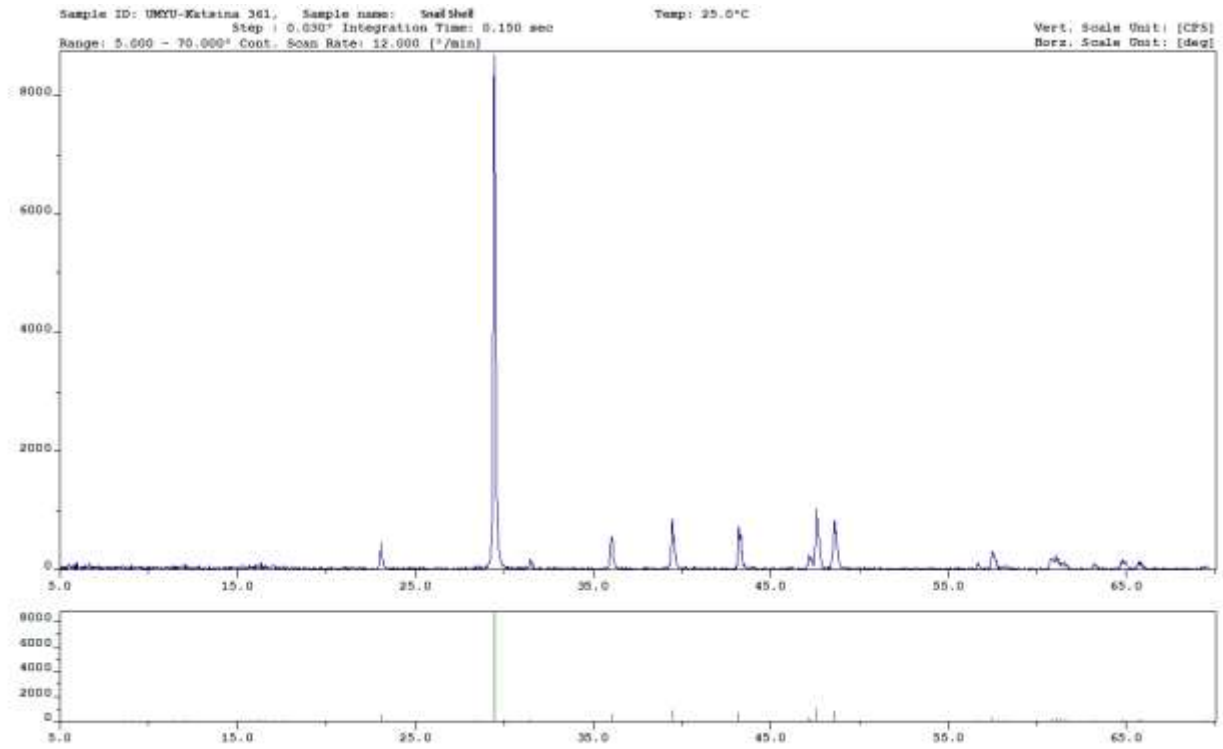

Figure 14: XRD of Snail Shell

Table 2: XRD result of Snail Shell

01-083-0577 Sep 24, 2018 11:03 AM (ARLservice)

Status Alternate QM: Star (S) Pressure/Temperature: Ambient Chemical Formula: Ca (C O3) Weight %: C12.00 Ca40.04 O47.95
Atomic %: C20.00 Ca20.00 O60.00 ANX: ABX3 Compound Name: Calcium Carbonate Mineral Name: Calcite

Radiation: CuKá1 : 1.5406Å d-Spacing: Calculated Intensity: Calculated I/Ic: 3.21

Reference: "Datensammlung nach der "Learnt profile"-Methode(LP) fuer Calcit und Vergleich mit der "Background peak background"-Methode (BPB)". Wartchow, R. Z. Kristallogr. 186, 300 (1989). Calculated from ICSD using POWD-12++. (2004).

SYS: Rhombohedral SPGR: R-3c (167) AuthCellVol: 367.54 Z: 6.00
Author's Cell [AuthCell-a: 4.9887(1)Å AuthCell-c: 17.05289(80)Å AuthCellVol: 367.54Å³] Dcalc: 2.713g/cm³ Dstruc: 2.71g/cm³
SS/FOM: F(30) = 999.9(0.0000, 30) R-factor: 0.018 Reference: Ibid.

Space Group: R-3c (167) Z: 6.00 Molecular Weight: 100.09
Crystal Data [XtlCell-a: 4.989Å XtlCell-b: 4.989Å XtlCell-c: 17.053Å XtlCell.: 90.00° XtlCell.: 90.00° XtlCell.: 120.00° XtlCellVol: 367.54Å³]
Crystal Data Axial Ratio [c/a: 3.4183]
Reduced Cell [RedCell-a: 4.989Å RedCell-b: 4.989Å RedCell-c: 6.372Å RedCell.: 66.96° RedCell.: 66.96° RedCell.: 60.00° RedCellVol: 122.51Å³]

Crystal (Symmetry Allowed): Centrosymmetric

CAS: 13397-26-7 Pearson: hR10.00

Subfile(s): Alternate Pattern, Cement and Hydration Product, Common Phase, Forensic, ICSD Pattern, Inorganic, Mineral Related (Mineral), Pharmaceutical (Excipient), Pigment/Dye, Superconducting Material

Entry Date: 12/25/2004 Last Modification Date: 01/31/2008 Cross-Ref PDF #'s: 00-005-0586 (Primary), 01-085-1108 (Alternate)

Database Comments: Additional Patterns: See PDF 00-005-0586 and 01-085-1108. ANX: ABX3. Analysis: C1 Ca1 O3. Formula from original source: Ca (C O3). ICSD Collection Code: 79673. Calculated Pattern Original Remarks: Refinement by the "Learnt Profile" method. Wyckoff Sequence: e b a (R3-CH).

01-083-0577 (Fixed Slit Intensity) - Cu K1 1.54056Å

2	d(Å)	I	h	k	l	*	2	d(Å)	I	h	k	l	*	2	d(Å)	I	h	k	l	*	2	d(Å)	I	h	k	l	*
23.0590	3.853850	99	0	1	2		70.2675	1.338480	19	0	2	10		95.3614	1.041760	14	2	2	9		115.1690	0.912460	1	4	0	10	
29.4094	3.034550	999	1	0	4		72.9140	1.296290	26	1	2	8		96.2134	1.034780	13m	1	0	16		117.9760	0.898748	7	2	3	8	
31.4499	2.842150	19	0	0	6		73.6847	1.284620	6	3	0	6		96.2134	1.034780	m	1	1	15		118.8150	0.894830	6	1	4	6	
35.9750	2.494350	139	1	1	0		76.2853	1.247170	11	2	2	0		97.7386	1.022660	2	2	1	13		119.3200	0.892518	10	2	1	16	
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43.1661	2.094010	149	2	0	2		78.4414	1.218200	1	2	2	3		102.2410	0.989484	3	3	2	1		127.2800	0.859665	2	5	0	2	
47.1247	1.926920	64	0	2	4		80.2450	1.195300	1	1	3	1		102.9590	0.984525	11	2	3	2		128.0250	0.856924	7	3	2	10	
47.5255	1.911600	185	0	1	8		80.9555	1.186590	5	3	1	2		103.5650	0.980413	3	1	3	10		128.7770	0.854209	5m	1	2	17	
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61.3970	1.508810	24	1	1	9		91.5151	1.075220	1	1	3	7		108.6770	0.948061	m	0	0	18		138.8740	0.822696	1	3	3	3	
63.0666	1.472830	19	1	2	5		91.9219	1.071520	1	0	4	2		109.5780	0.942776	16	4	1	0		141.6470	0.815533	1	2	4	1	
64.6709	1.440110	57	3	0	0		93.1022	1.061010	8	2	0	14		110.5200	0.937373	9	2	2	12		142.7920	0.812750	7	4	2	2	
65.6455	1.421070	31	0	0	12		94.7317	1.047010	23	4	0	4		111.8280	0.930070	1	4	1	3		144.7880	0.808134	1	0	4	14	
69.2053	1.356410	10	2	1	7		95.0279	1.044530	37	3	1	8		114.0720	0.918077	2	3	2	7		147.7160	0.801894	6	2	4	4	

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