Time-of-Flight Neutron Diffraction

How to analyze Time-of-Flight using powder diffraction and the Powder Diffraction File[™]

Bank.		Display -		Isotopic Sub	stitution - Li2 Mn Si O4	- 04-019-6640 (Calc) 🗙
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Overview

In 2016 ICDD again enhanced the materials analysis capability of the Powder Diffraction File[™] (PDF[®]), adding the capability of simulating Time-of-Flight (TOF) neutron diffraction patterns. Utilizing over 272,000 PDF-4+ database entries with atomic coordinates or neutron diffraction structure factors and 26,000 additional entries with cross-referenced access to atomic coordinates, TOF patterns can be simulated, displayed, and compared to imported TOF raw data. Using the Similarity Index capability of PDF-4+, full pattern matching can be performed for phase identification of major phase unknowns. This Guide presents four activities that will help with getting started using the TOF capability in ICDD PDF-4 databases.

Activity 1 – Plot a Time-of-Flight (TOF) pattern using a PDF entry	Page	1
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For further information on ICDD's neutron diffraction database capabilities, please visit our website at:

http://www.icdd.com/products/2016/neutrondiffraction.htm

Read the Time-of-Flight abstract submitted for publication for *Advances in X-ray Analysis* from the 2016 Denver X-ray Conference presentation.

- > New Neutron Time-of-Flight (TOF) Capability in PDF-4+ Relational Databases: Digitized Diffraction Patterns and I/I_c for Quantitative Phase Analysis
 - J. Faber, Faber Consulting, Thornton, PA, USA
 - S. Kabekkodu, J. Blanton, T. Blanton, T. Fawcett, ICDD, Newtown Square, PA, USA

http://www.dxcicdd.com/16/abstracts/D20.pdf

Online Resources

Visit our website at www.icdd.com

Our goal at ICDD is to help you solve your materials problems. We provide online publications, technical bulletins, tutorials, and videos. Many tutorials focus on capabilities of the database, but there are also general tutorials that describe methods used to analyze drugs, polymers, and minerals. The tutorial page has links to free download publications, as well as instructional videos. Our website also contains over 1,000 full publications for free download from *Advances in X-ray Analysis*. Our website, tutorial page, and publication pages are there to help you!

- > www.icdd.com.resources/tutorials
- > www.icdd.com/products/technicalbulletins.htm
- > www.icdd.com/resources/axasearch/search_based_on_vol.asp



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ICDD[®] PDF-4+ 2016 Time-of-Flight (TOF) Neutron Diffraction Pattern Simulation and Phase Identification – Getting Started

Activity 1 – Plot a simulated Time-of-Flight (TOF) pattern when you know the PDF entry you are interested in reviewing has atomic coordinates:



Open PDF-4+ 2016 by double clicking on the desktop icon



2

Enter the PDF number for a known entry with atomic coordinates by selecting the Open PDF Cards button

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Enter a PDF number and click on open. For this example, use entry 04-019-6640, Li₂MnSiO₄.

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The PDF entry 04-019-6640 is displayed, including the option for a simulated pattern. The default is to display a powder X-ray diffraction pattern, Cu K α 1,2 radiation, pseudo-Voight peak profile.



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5 Click on the drop down menu for wavelength options and scroll to the bottom, click on TOF Neutron

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The simulated diffraction pattern is now an Intensity vs. Time-of-Flight plot. Currently for PDF-4+ 2016, d-spacing values will not be displayed for TOF data. Click on the Simulated Profile icon and the simulated pattern will be displayed in a larger view.



The TOF pattern as displayed is based on default parameters built into the PDF-4+ software. The defaults include instrument profile parameters and Time-of-Flight range. To change profile or range, click on the *Preferences* icon and adjust as necessary (Note: instrument profile parameters can be imported from different user facilities. See Activity on page 10).



Soufcance Limit: 0.001
Step Width (jus): 6.0
Start TOF (µs): 5000
Stop TOF (jus): 93000

A right mouse click on the TOF plot will display additional pattern options, including options for X-axis units: TOF, Q, d, 1/d.



Activity 2 - Plot a simulated Time-of-Flight (TOF) pattern when you search for PDF entries that have atomic coordinates:

Open PDF-4+ 2016 by double clicking on the desktop icon



2

Select Atomic Coordinates on the PDF-4+ search window

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Select desired criteria for searching for a material type. In the example below, choose phases with atomic coordinates, plus a Periodic Table search for phases that have only elements Ag, S, N, and O, then click on Search.

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Three entries in the PDF meet the search criteria. Open 00-065-0110 by double-clicking on the PDF # row or clicking once on Open PDF Card.

PDF # QM Chemical Formula Compound Name D1 (Å) D2 (Å) D3 (Å) SYS 00-065-0110 R Aq35 (N O3) Silver Suffide Name 2.643600 5.607900 2.199600 C 4009-9616 B Aq35 (N O3) Silver Suffide Name 2.643600 5.606650 2.199600 C 4009-9616 B Aq35 (N O3) Silver Suffide Name 2.643600 5.606650 2.199110 C 4-012-8423 I Aq4 (5 O2 N2) Silver Nitride Suffate 2.569190 2.927170 4.401120 O	erences Open PD	F Car	Simulated Profile	R		Results: 3 of 384,613	ICDD Defau	its		
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5 The PDF entry 00-065-0110 is displayed, including the option for a simulated pattern. The default is to display a powder X-ray diffraction pattern, Cu K α 1,2 radiation, pseudo-Voight peak profile.

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Click on the drop down menu for wavelength options and scroll to the bottom, click on TOF Neutron.



The simulated diffraction pattern is now an Intensity vs. Time-of-Flight plot. Currently for PDF-4+ 2016, d-spacing values will not be displayed for TOF data. Click on the *Simulated Profile* icon and the simulated pattern will be displayed in a larger view.

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NOTE: If the simulated TOF pattern does not display (see example below), the atomic coordinates are cross-referenced for the PDF entry. Click on the *Cross-reference* tab and double click on a PDF entry noted with a $\sqrt{(\text{check mark})}$ which is a cross-referenced PDF entry with atomic coordinates for the phase of interest. The cross-referenced entry will be displayed and the simulated TOF pattern can be viewed as described here in Activity 2.

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The TOF pattern as displayed is based on default parameters built into the PDF-4+ software. The defaults include instrument profile parameters and Time-of-Flight range. To change profile or range, click on the *Preferences* icon and adjust as necessary. (Note: instrument profile parameters can be imported from different user facilities. See Activity 3 on page 10).



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9 A right mouse click on the TOF plot will display additional pattern options, including options for X-axis units: TOF, Q, d, 1/d.



Activity 3 – Importing beamline instrument parameters, plot a simulated Time-of-Flight pattern after doing a database search, plot a Time-of-Flight pattern after doing an isotope substitution, import a raw data pattern for comparison.

Open PDF-4+ 2016 by double clicking on the desktop icon



2

10

1

The PDF Search window will appear. Select the *Preferences* button on the main toolbar and select the *Diffraction Pattern* Tab. In this tab, click the Import *Instrument Parameters* button and select an instrument parameters file you have available (can be either GSAS or FullProf format).



Simulation :	iets: ICOD Default	5	(V)	🕂 Add	Rename	👗 Delete	B Import	Instrument Parameters
Radiation X-ray Diffs	action					/		
Anode:	🛃 Import Instru	ment Paramet	ers			/		×
ONeutron D	Look in:	GSAS				/ - 1	P 🔤 •	
Bectron D Geometry Bragg Bre Polarizatio Sample Th Debye-Sd Profile Profile C.0002713 V: 0.00076 W: 0.003638	Recent Items Desktop Documents	HighInte HighReso 11bm_gs 53873pol CPD-INS NOMAD PG60HR PGHR.60 PGHR.60 PGHR.60 PGHR.60 PGHR.60 PGHR.60 PGHR.60	sity Jution sc.prm 1179.prm 1-XRY.prm 04.30_13.prm 2013A.prm 2013B_Hod -2013B_Hod -2013B_93B -2015A.prm -2015A.prm	m ges.prm 8.prm	/			
Display X-Axis: Y-Axis: Imported Plot	This PC	File name:	PGHR_60-2	0158.prm				Open
and the second of the second	Contraction of the	ries of type:	Al Instrum	ent Parameter Fi	les (*.rf, *.ns, *.	nst, *.pm, *.instpri	m) 🗸	Cancel

Once the instrument parameters have been imported, click OK.

	_60-20158.prm	Ý	🕀 Add	Rename	👗 Delete	B Import Instrument Parameters
Radation			-	1		
X-ray Diffraction						
Neutron Diffraction*						
O Constant Waveleng	th (CW)					
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Electron Diffraction						
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at 0.307336 σet 0	.0 γst 0.	0		Significance Limit:	0.001	
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X-Axis:	Time-of-Flight (µs)	× .		Step Width (µs):	6.0	
V. Awier	Linear Intensity	~		Start TOF (µs):	5000	
2 States 1999	A finish of the second se			Stop TOF (µs):	93000	

The next step is to perform a search and get a list of patterns in the database. This is done using the Search window that is displayed at program startup. For example, the search below is for the formula LaB₆, lanthanum hexaboride. The search also included the Linus Pauling File (LPF) database (04), as all of those entries have atomic coordinates. Patterns with atomic coordinates are required to simulate TOF neutron diffraction patterns (in PDF-4+ 2016 you can use 00, 01, 03, and 04 databases).

								Carl Sugar
Custom POP Set Akadods Amino Acidi, Peptides & Conplexes Battery Material Boacthity No Subclass Narcolic Psychotropic Carbidhydrate V			Andrent Andrent Andrent Press. (Non-andrent) Temp. (Non-andrent) Press. & Temp. (Non-andrent) Atomic Coordinates Raw Diffraction Data Ray	Quality Mark Star Ricetveld Good Indexed Calculated Prototyping Minimal Acceptable Blank	Cotthese 1000 (00) 1000 (00) 000 (00) 000 (00) 000 (00) 000 (00) 000 (00) 000 (00) 000 (00)			
Periodic Table Formula/Name Classifications Crystallography Modulated Diffraction Physical Properties	Formúle T la b5 Name T Any Name CAS Number Number of Elem Loon	ents High	I de la companya de la compa					
Reference Comments	Element	Value	850					

After clicking the *Search* button, a list of search results is shown. Double-click any of these entries to open the PDF card.

erences Ope	en PDF Ca	rd Simulated Profile		Results: 99 of 384,613	ICDO Defau	lts.	
PDF #	QM	Chemical Formula	Compound Name	D1 (Å)	D2 (Ă)	D3 (Å)	SYS
04-001-2105	0	La 86	Boroh Lanthanun	2.939260	4,155600	1.858890	
04-001-5322	9 P	La Ni 12 86	Boron Lanthanum Nickel	2.199540	2.296690	2.837340	0
04-001-5594	👴 P	La0.5 Eu0.5 86	Boron Europium Lanthanum	2.945810	4.166000	1.863090	c
04-001-5595	😔 P	Sr0.5 La0.5 86	Boron Lanthanum Strontum	2.950050	4.172000	1.865780	C
04-001-6172	0 P	La0.5 Y0.5 86	Boron Lanthanum Yttrium	2.921770	4.132000	1.847890	с
04-001-6207	👴 P	La0.5 Ho0.5 86	Boron Holmium Lanthanum	2.917520	4.126000	1.845200	C
04-002-0015	9 P	La B6	Boron Lanthanum	2,939440	4,157000	1.859070	C
04-002-8737	O P	La0.5 Ce0.5 86	Boron Cerium Lanthanum	2.933080	4,148000	1.855040	C
14-002-9860	😐 P	La 86	Boron Lanthanum	2.938740	4,156000	1.858620	С
04-003-0591	01	La 86	Boron Lanthanum	2.939160	4.156600	1.858890	с
04-003-1197	O P	La Bó	Boron Lanthanum	2.938740	4.156000	1.858620	C
04-003-4227	0 P	La 86	Boron Lanthanum	2.939160	4.156600	1.858890	C
04-003-4867	0 P	La B6	Boron Lanthanum	2.933080	4.148000	1.855040	C
04-003-5944	9 P	La B6	Boron Lanthanum	2.938670	4,155900	1.858580	C
04-003-6214	0 P	La B6	Boron Lenthanum	2.939580	4.157200	1.859160	С
04-003-6332	9 P	La B6	Boron Lanthanum	2.939440	4.157000	1.859070	C
04-003-6471	0 P	La B6	Boron Lanthanum	2.932370	4.147000	1.854590	C
04-003-6661	0 P	La B6	Boron Lanthanum	2.932370	4.147000	1.854590	С
04-003-7433	O P	La5 Co2 86	Boron Cobelt Lanthanum	2.755000	2.286710	2.829700	R
04-003-8164	9 P	La Co1286	Boron Cobalt Lanthanum	2,185860	2.213880	2.773830	R
04-003-9945	0 S	La N 12 66	Boron Lanthanum Nickel	2.196870	2.201690	2.298600	0
04-004-0719	0 P	La 86	Boron Lenthanum	2.938740	4.156000	1.858620	C
04-004-1609	O P	La 86	Boron Lanthanum	2,936610	4.153000	1.857280	C

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⁶ PDF entry 04-001-2105 is opened. There is no TOF neutron diffraction data shown in the top-left table. The list of d-spacings/intensities/etc. are not published in the 2016 PDF release (this is planned for the 2017 release). However, you can see the TOF neutron powder diffraction simulation at the top-right. Click the *Simulated Profile* button to open the simulation in a new window.

La 86 - 04-001-21	05							
Eile Edit Plots W	ndow Help				-	-	1	
Save Print Prefere	nces Temperature Series Toobox Property Sheet	O R	D Bonds	SAED ERS	D Ring	Simulated Profile	Raw Diffraction Data	
TOF Neutron	Simulated Profile (Calc) Raw Diffractory Data (PD1)	1,0	00 00				1 1	
TOP neutron diffrac	ion data not available	Attendud		100 20,00	J 0 30,	000 40.000 Time	50,000 60,000 of-Flight (us)	70,000 80,000 90,000
POP	Status: Primary QM	Ø Star			Press.	.re/Temperature: /	Ambient	
Experimental	Phase:							
Physical	Chemical Formula: La Bil Structural Formula:							
Crystal	Empirical Formula: B6 Ls							
Optical	Weight %: 831.83 La68.17 Atomic %: 885.71 La14.29							
Structure	Compound Name: Boron Lanthanum							
Classifications	Mineral Name:							
Cross-references	Common Name: CAS:							
References	Entry Date: 09/01/2005							
Comments	Last Modification Date: 09/01/2011 Last Modifications: Reflections							

7

Isotopic substitution can be performed by clicking *Plots Menu > Isotopic Substitution*.



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You can also import an experimental data file by clicking *Plots Menu > Import*. An example imported raw data file is below. Make certain you select the appropriate settings highlighted below. Click the *Refresh Graph* button to load the data and then click the Import button to import it.



9

Here is a comparison of the simulation to the unprocessed raw data.



Activity 4 - Major phase identification using Similarity Indexes with Time-of-Flight (TOF) Neutron Powder Diffraction Data

Another TOF neutron diffraction capability in the PDF-4+ 2016 is the ability to compare a TOF data file with TOF simulations in the database using full pattern matching. Full pattern matching creates similarity indexes, which are numbers that describe how well the patterns match. A similarity index of 0.0 is considered a perfect match (this is rare). The larger the number, the less likely the phase being a match. To run a Similarity Index analysis:



2

Click Similarity Index Menu > Show Similarity Index.

🖑 Results - [Data	base (LPF	(04))] And [Any Formula Conta	ins Elements 'la' And 'b6'] And	[Status (Primary, Alterna	te)]			x
File Edit Fields	Similarit	y Index Help						
Preferences Ope	Sh 🍓 Cł	ow Similarity Index nange Experimental Data		Results: 99 of 384,613	ICDD Defa	ults		~
PDF #	QM	Chemical Formula	Compound Name	D1 (Å)	D2 (Å)	D3 (Å)	SYS	\square
04-001-2105	🥥 S	La B6	Boron Lanthanum	2.939160	4.156600	1.858890	С	
04-001-5322	😐 P	La Ni 12 B6	Boron Lanthanum Nickel	2.199640	2.296690	2.837340	0	
04-001-5594	😐 P	La0.5 Eu0.5 B6	Boron Europium Lanthanum	2.945810	4.166000	1.863090	С	
04-001-5595	😑 P	Sr0.5 La0.5 B6	Boron Lanthanum Strontium	2.950050	4.172000	1.865780	С	
04-001-6172	😐 P	La0.5 Y0.5 B6	Boron Lanthanum Yttrium	2.921770	4.132000	1.847890	С	
04-001-6207	😑 P	La0.5 Ho0.5 B6	Boron Holmium Lanthanum	2.917520	4.126000	1.845200	С	
04-002-0015	💛 P	La B6	Boron Lanthanum	2.939440	4.157000	1.859070	С	
04-002-8737	😐 P	La0.5 Ce0.5 B6	Boron Cerium Lanthanum	2.933080	4.148000	1.855040	С	1
04-002-9860	😐 P	La B6	Boron Lanthanum	2.938740	4.156000	1.858620	С	
04-003-0591	9 I	La B6	Boron Lanthanum	2.939160	4.156600	1.858890	С	
04-003-1197	😑 P	La B6	Boron Lanthanum	2.938740	4.156000	1.858620	С]
04-003-4227	💛 P	La B6	Boron Lanthanum	2.939160	4.156600	1.858890	С	
04-003-4867	😐 P	La B6	Boron Lanthanum	2.933080	4.148000	1.855040	С	1
04-003-5944	😑 P	La B6	Boron Lanthanum	2.938670	4.155900	1.858580	С	
04-003-6214	😐 P	La B6	Boron Lanthanum	2.939580	4.157200	1.859160	С	1
04-003-6332	💛 P	La B6	Boron Lanthanum	2.939440	4.157000	1.859070	С	
04-003-6471	😐 P	La B6	Boron Lanthanum	2.932370	4.147000	1.854590	С	1
04-003-6661	0 P	La B6	Boron Lanthanum	2.932370	4.147000	1.854590	С	1
04-003-7433	💛 P	La5 Co2 B6	Boron Cobalt Lanthanum	2.755000	2.286710	2.829700	R	1
04-003-8164	💛 P	La Co12 B6	Boron Cobalt Lanthanum	2.185860	2.213880	2.773830	R	1
04-003-9945	🔘 S	La Ni 12 B6	Boron Lanthanum Nickel	2.196870	2.201690	2.298600	0	1
04-004-0719	💛 P	La B6	Boron Lanthanum	2.938740	4.156000	1.858620	С	1
04-004-1609	😐 P	La B6	Boron Lanthanum	2.936610	4.153000	1.857280	С	~
Search Description	1:		Cal	culations:				
[Database (LPF (0 (Primary, Alternat	4))] And [≥)]	Any Formula Contains Elements 'la'	And 'b6'] And [Status Me	an: Mediar	1:	ESD:		

Import the pattern as you did in Activity 3, step 8. It is important to remove the background first, as we do not create a background in the TOF pattern simulations. Click the *Process Data* combo-box to process the experimental data. Manual refinement can be perfromed by clicking the *Edit Settings* button.



After clicking the *Import* button, the program will load the processed TOF data and compare it to dynamically created pattern simulations in the search results table. Click the *Normalized R-index* column to sort the data with the best matches at the top.

ferences Open PDF Card Simu	Asted Profile			Results: 99 of 384,613	ICDO Defaulta	
Normalized R-index 😭	POF #	QM	Chemical Formula	Compo	und Name	D1(Å)
1 1.52 (1794 pr - 17977 pt)	04-004-8705	19-11	La 80	Boron Lermonum		2.939510
0.53 (8734 pt - 121879 pt)	04-001-2105	0.5	La B6	Boroh Larithanum		2.939160
= 0.65 (9724 µm - 121879 µm)	04-002-0015	0 P	La 86	Boron Lanthanum		2.939440
🕺 0.45 (9724 µz - 121879 µs)	04-003-6332	😏 P.	La D6	Boron Lanthanum		2.939440
😤 0.45 (9724 µt - 122879 µt)	04-004-6667	P	La 96	Boron Lanthanum		2.939140
🔁 0.65 (9724 µ + 121879 µ)	04-006-5064	O P	La D6	Boron Lanthanum		2.939440
(a) (9/14 pt - 1218/9 pt)	04-003-6214	9 P	La Bó	Boron Lanthanum	51	2.939580
🖉 0.65 (9/14 pt - 1218/9 pt)	04-003-0591	0 I	La B6	Boron Lanthanum		2.939160
🞽 0.65 (9/14 µr - 121879 µr)	04-003-4227	Q P	La B6	Boron Lanthanum		2.939160
7 0.65 (3714 us - 121879 us)	04-004-5789	O P	La Bő	Boron Lanthenum		2.939160
10.65 (9714 µa - 121879 µa)	04-004-6019	O P	La B6	Boron Lanthanum		2.939090
🗲 0.65 (9734 µ# - 121879 µ8)	04-005-4931	9 P	La Bé	Boron Lanthanum		2.939090
📩 0.65 (9714 µz - 121879 µs)	04-005-5395	Q.P	La Bé	Boron Lanthanum		2.939090
🔁 0.66 (9724 at - 122879 at)	04-004-5779	P	La0.97 B6	Boron Lanthanum		2.938950
🗡 0.46 (9714 µz - 121879 µz)	04-004-2958	9 P	La D6	Boron Lanthanum		2.938950
2 0.66 (9724 µs - 1218/9 µs)	04-004-7854	O P	La 96	Boron Lanthanum		2.938950
(1,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0	04-006-0734	O P	La B6	Boron Lanthanum	12	2.938950
1218.79 (11714 pt - 1218.79 pt)	04-005-9342	O P	La B6	Boron Lanthanum	No.	2.938880
🔀 0.66 (9724 pt - 122879 pt)	04-002-9860	😏 P	La B5	Boron Lanthanum		2.938740
1. 0.66 (9714 ur - 121679 ur)	04-003-1197	🔾 P	La B6	Boron Lanthanum		2.938740
🗡 0.66 (9714 pa - 121879 pa)	04-004-0719	🔒 P	La B6	Boron Lanthanum		2.938740
2 0.66 (8714 µt - 121879 µt)	04-004-6066	Q P	La 86	Boron Lanthanum		2.938740
¢	a secondario				100	3

5 The last step is to right-click the match you want to see and select *Graph Similarity Index*. This will show the experimental data compared to the selected simulation (similar to step 9 from above).



Summary

Performing TOF neutron powder diffraction simulations (with the option of isotopic substitution) and comparing TOF experimental data patterns to these simulations are the "TOF features" added to the PDF-4+ 2016 software and database released in September 2016. Time-of-Flight simulations can also be displayed in the PDF-4/Minerals 2016 and PDF-4/Organics 2017 databases using the procedures shown in this User Guide.

The next phase of TOF analysis at ICDD is to allow the user to perform phase identification on TOF neutron diffraction data using our Sleve+ plug-in software. This is in development and is currently scheduled for the PDF-4 2017 product release.



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