

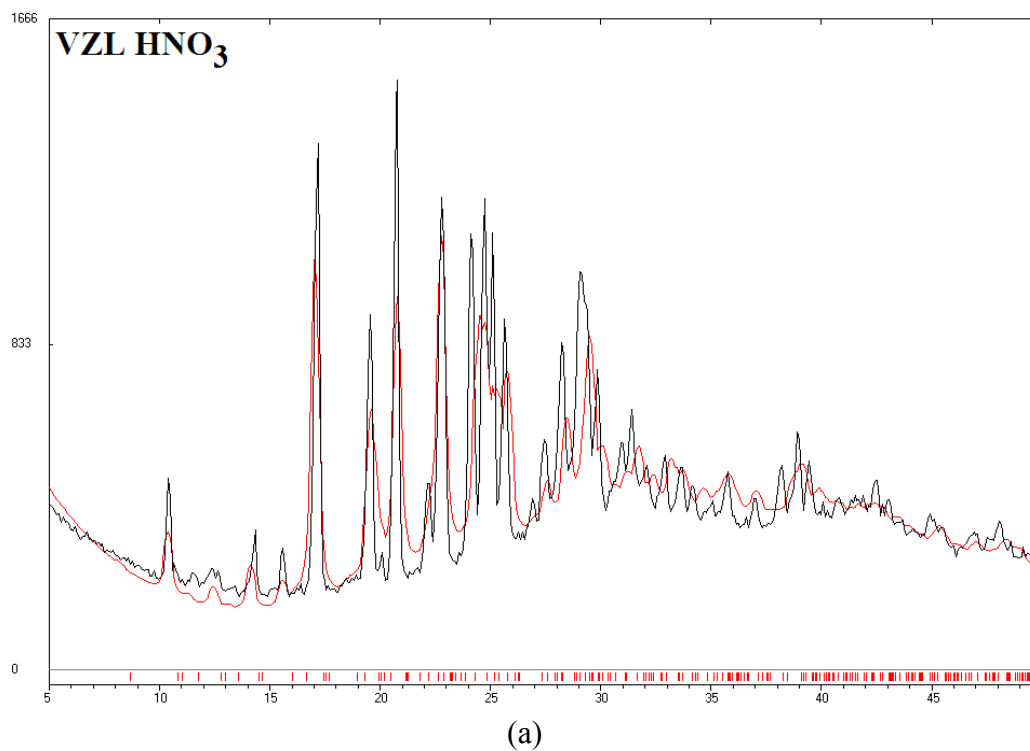
Pharmaceutical Cocrystals and a Nitrate Salt of Voriconazole†

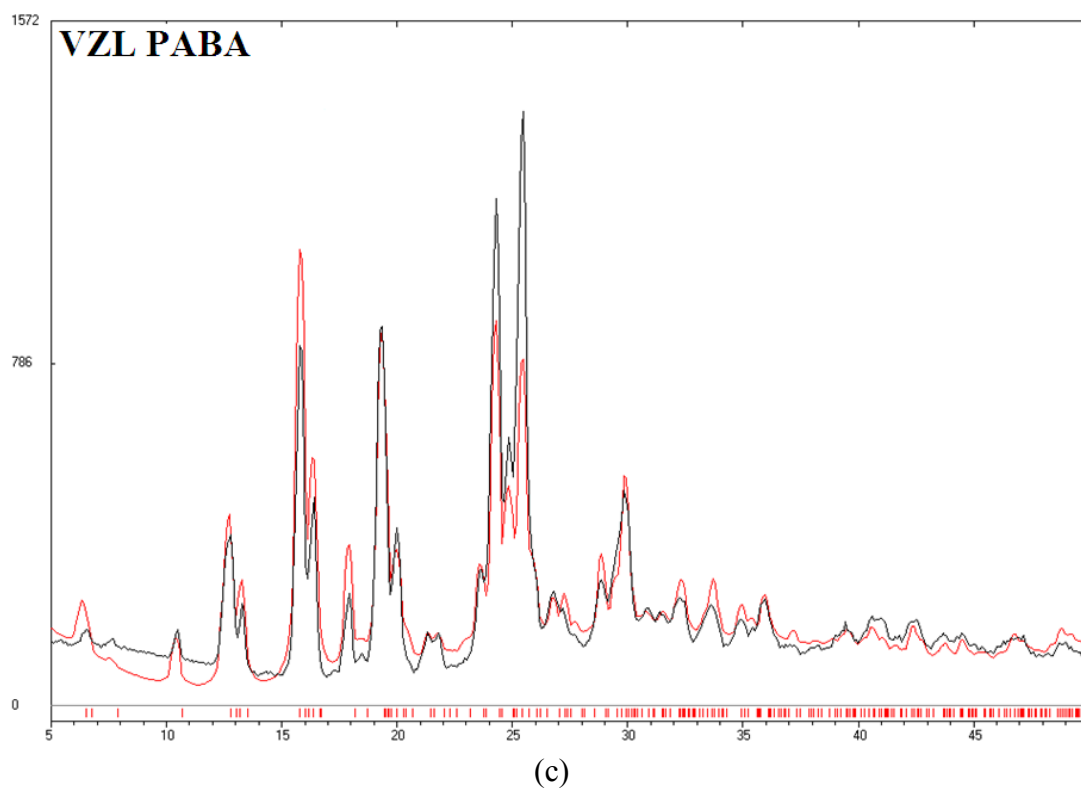
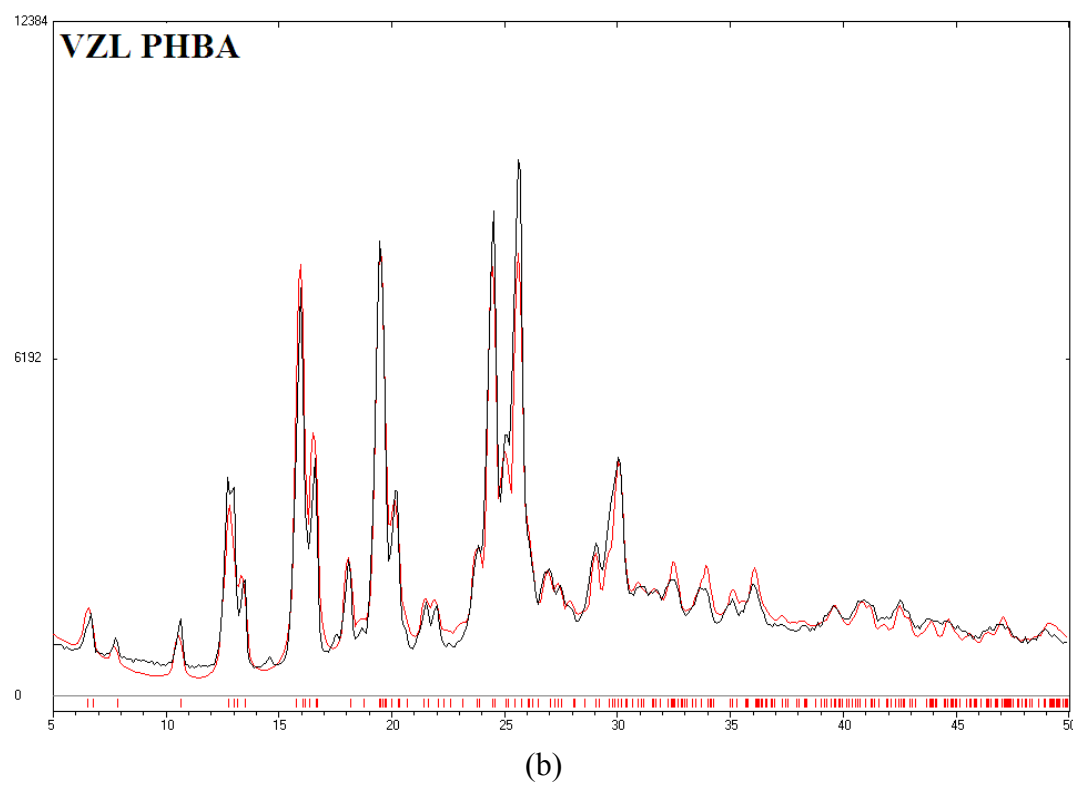
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Electronic Supplementary Information†





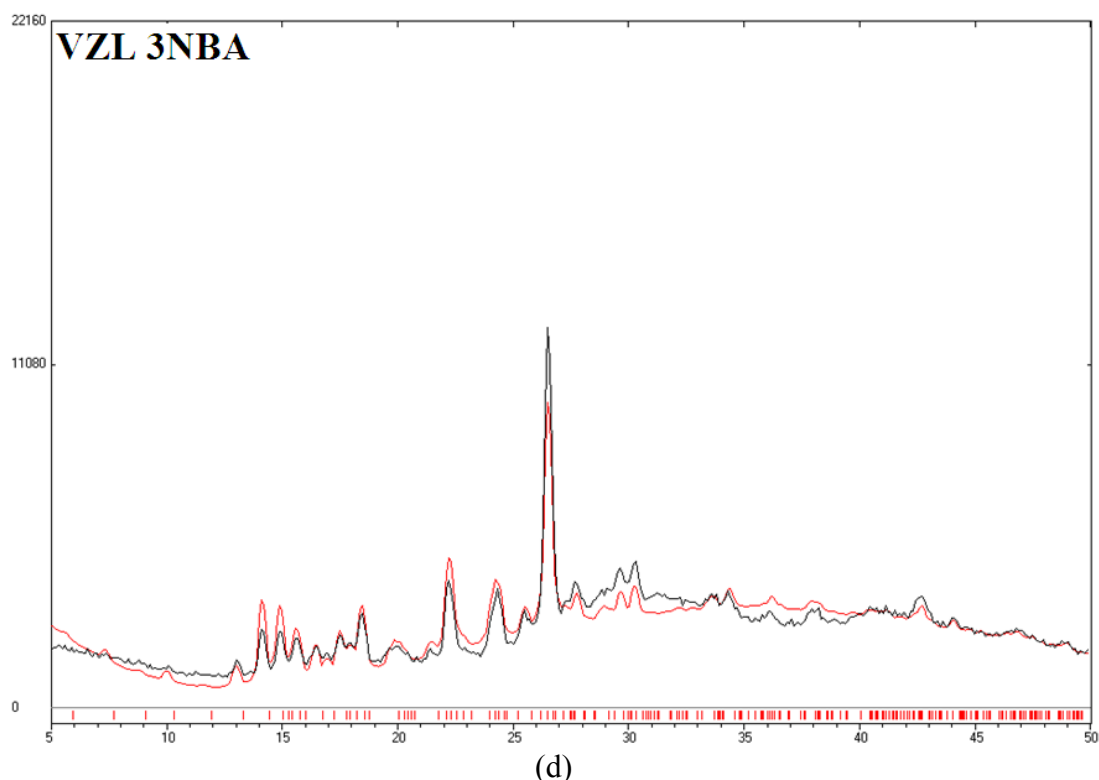


Figure S1 Comparison of experimental (black) and calculated (red) X-ray powder pattern of (a) Voriconazole-dinitrate salt (b) VZL–PHBA (c) VZL–PABA and (d) VZL–3-NBA cocrystals.

Table S1 List of experiments in the search of new VZL crystalline forms.

S.No	Coformers	Mineral acids	Polymorph screening
1	L-Ascorbic acid	HCl	Acetone
2	Saccharin	H ₂ SO ₄	Methanol
3	Glutaric acid	HNO ₃	Ethanol
4	Nicotinic acid	Formic acid	Chloroform
5	PHBA	Chlorosulphonic acid	Dichloromethane
6	PABA	Methanesulphonic acid	Ethylmethyl ketone
7	Citric acid	Sulphamic acid	Benzene
8	Benzoic acid	Acetic acid	Toluene
9	Nicotinamide		Nitromethane
10	Maleic acid		Dioxane
11	Urea		Acetonitrile
12	Salicylic acid		THF
13	4-aminosalicylic acid		Anisole
14	Fumaric acid		Xylene
15	3-nitrobenzoic acid		Mesitylene
16	3,5-dihydroxy benzoic acid		Ethylacetate

17	4-nitrobenzoic acid		Methylacetate
18	2,4-dihydroxy benzoic acid		2-propanol
19	3-methoxybenzoic acid		Iso-butanol
20	4-methoxybenzoic acid		
21	2-methoxybenzoic acid		
22	Succinic acid		

Table S2 Hydrogen bonds in crystal structures (neutron-normalized distances).

VZL form	Interaction	H...A / Å	D...A / Å	∠D-H...A / °	Symmetry code
VZL-HNO ₃	O1-H1...N4	1.89	2.703(19)	138	intramolecular
	N3-H3A...O2	2.33	3.038(2)	126	1/2-x,-y,-1/2+z
	N3-H3A...O3	1.72	2.720(2)	171	1/2-x,-y,-1/2+z
	O6-H6A...N5	1.57	2.547(2)	176	intramolecular
	C1-H1A...F2	2.29	2.997(2)	121	intramolecular
	C6-H6...O4	2.61	3.107 (2)	106	-x+1,+y-1/2,-z+1/2
	O1-H1...O4	2.58	3.296 (2)	132	-x+1,+y-1/2,-z+1/2
	C1-H1B...O7	2.45	3.514(2)	167	-1/2+x,1/2-y,-z
	C1-H1A...O6	2.58	3.446(2)	136	x+1,+y,+z
	C3-H3...F1	2.46	2.880(19)	101	intramolecular
	C3-H3...F2	2.33	3.041(19)	122	intramolecular
	C4-H4C...O3	2.45	3.528(2)	179	1+x,y,z
	C6-H6...N2	2.43	3.380(2)	145	1+x,y,z
	C7-H7...O1	2.44	3.259(2)	132	2-x,1/2+y,1/2-z
	C9-H9...O2	2.21	3.074(2)	136	1-x,-1/2+y,1/2-z
	C9-H9...O4	2.22	3.283(2)	165	-x+1,+y-1/2,-z+1/2
	C10-H10...O3	2.12	3.199(2)	172	1-x,-1/2+y,1/2-z
	C15-H15...O2	2.28	3.168(2)	138	-x,-1/2+y,1/2-z
	C12-H12...O6	2.53	3.327(2)	129	-x,+y-1/2,-z+1/2
	C10-H10...F1	2.83	3.136(2)	95	-x+1,+y-1/2,-z+1/2
VZL-PHBA	O1-H1...N4	1.75	2.663(3)	153	intramolecular
	O2-H2...N5	1.76	2.747(3)	179	-1+x,1+y,z
	O4-H4...N3	1.84	2.783(4)	159	2-x,1/2+y,1-z
	C1-H1A...F2	2.48	3.133(3)	118	intramolecular
	C1-H1B...O4	2.41	3.438(3)	157	-x+1,+y+1/2,-z+1
	C3-H3...F1	2.42	2.855(3)	102	intramolecular
	C3-H3...F2	2.31	2.935(3)	115	intramolecular
	C10-H10...O3	2.43	3.313(4)	138	1+x,y,z
	C7-H7...O3	2.74	3.130(3)	100	-x+2,+y+1/2,-z+1
	C6-H6...N2	2.54	3.432(4)	138	x,+y-1,+z
	C6-H6...O3	2.69	3.452 (3)	127	-x+2,+y-1/2,-z+1

	C12–H12...O1	2.24	2.655(3)	100	intramolecular
	C13–H13...O2	2.41	3.418(4)	154	x,-1+y,z
	C4–H4B...F2	2.87	3.418(2)	111	x,y,z
VZL–PABA	O1–H1...N4	1.74	2.675(4)	159	intramolecular
	O2–H2...N5	1.77	2.742(4)	168	-x,-1/2+y,1-z
	N6–H6A...O3	2.43	3.369(6)	154	-x,1/2+y,2-z
	N6–H6B...N3	2.09	3.093(6)	174	x,y,1+z
	C1–H1A...N6	2.50	3.559(6)	165	1-x,-1/2+y,1-z
	C1–H1B...F2	2.49	3.121(4)	116	intramolecular
	C3–H3...F1	2.41	2.849(4)	103	intramolecular
	C3–H3...F2	2.29	2.932(4)	116	intramolecular
	C10–H10...O3	2.38	3.259(5)	137	-x,-1/2+y,1-z
	C7–H7...O3	2.92	3.266(3)	98	x-1,+y-1,+z
	C6–H6...N2	2.63	3.570(5)	144	x,+y+1,+z
	C12–H12...O1	2.21	2.624(4)	100	intramolecular
	C13–H13...O2	2.40	3.443(5)	161	1-x,1/2+y,1-z
	C4–H4B...F2	2.91	3.428(5)	109	x,y,z
VZL–3-NBA	O1–H1...N4	1.91	2.787(3)	148	intramolecular
	O2–H2...N3	1.66	2.630(3)	170	1+x,y,z
	O1–H1...N2	2.74	3.031(3)	98	x,+y+1,+z
	C1–H1A...F2	2.16	2.938(3)	126	intramolecular
	C3–H3...F1	2.36	2.882(3)	108	intramolecular
	C3–H3...F2	2.50	3.117(3)	115	intramolecular
	C7–H7...N5	2.45	3.506(4)	164	-x,-1/2+y,-z
	C9–H9...O3	2.61	3.225(3)	114	x-1,+y,+z
	C7–H7...O5	2.75	3.125(4)	99	-x,+y-1/2,-z
	C6–H6...O5	2.67	3.755(4)	177	-x,+y+1/2,-z
	C12–H12...O1	2.28	2.689(3)	100	intramolecular
	C21–H21...O4	2.55	3.270(3)	123	x,+y+1,+z

Table S3 Chemical Shift values of different solid forms.

Carbon atoms	VZL	VZL–HNO ₃	VZL–PHBA	VZL–PABA	VZL–3-NBA	PHBA	PABA	3NBA
C1	124.39	126.41	124.34	131.57	124.30	158.34	153.7	147.87
C2	101.91	104.67	102.51	102.26	103.63	133.14	131.6	133.50
C3	124.39	126.41	124.34	129.64	122.34	116.20	117.1	129.61
C4	129.59	131.41	129.90	141.49	132.90	122.15	112.4	127.35
C5	111.59	111.22	111.86	114.92	111.52	113.98	117.1	124.18
C6	129.59	131.41	132.05	143.51	132.90	133.14	131.6	127.35
C7	55.87	57.22	59.20	58.30	58.80	170.51	174.5	170.83

C8	34.69	34.77	33.86	34.23	32.89			
C9	145.80	147.03	142.46	150.24	146.90			
C10	145.59	144.14	142.46	150.52	146.90			
C11	151.18	157.80	160.89	160.25	160.56			
C12	149.86	153.44	155.74	155.91	153.04			
C13	77.11	76.58	77.60	76.92	78.31			
C14	158.80	160.93	166.92	169.74	165.57			
C15	158.80	157.80	160.89	162.93	156.39			
C16	14.40	16.59	18.15	18.09	17.33			

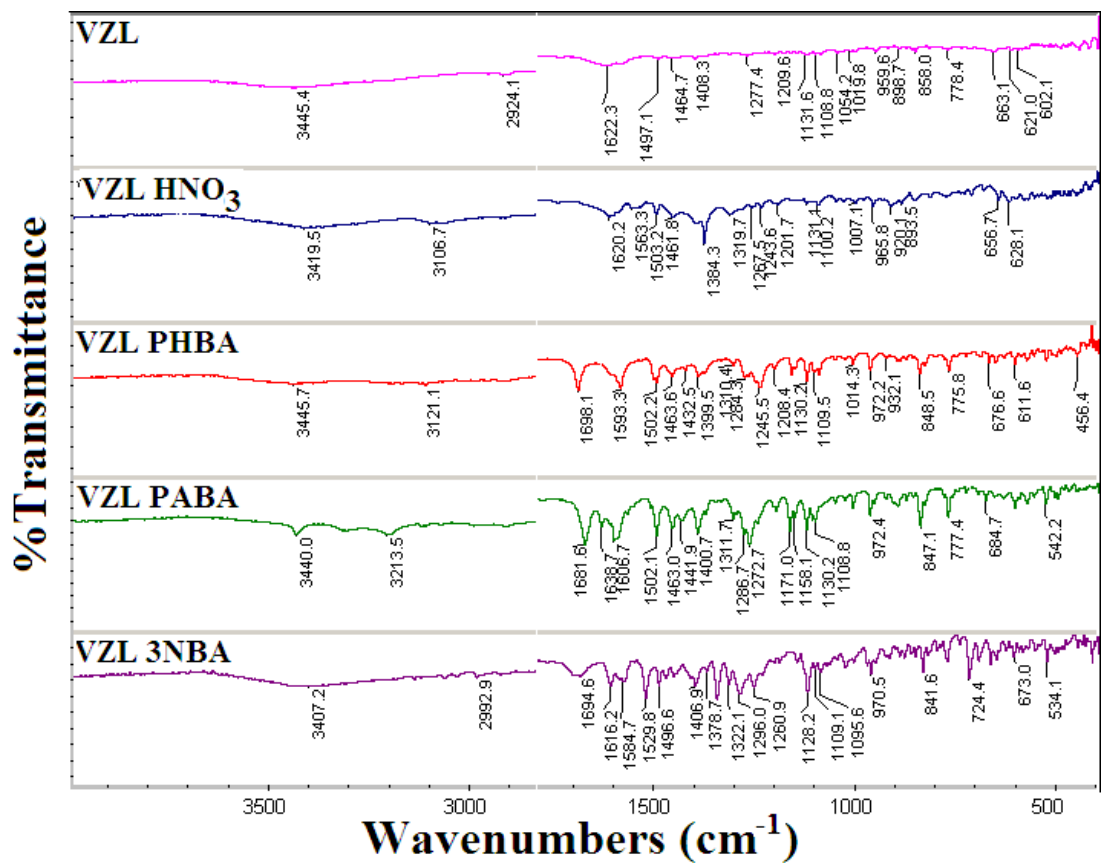


Figure S2 Comparison of FT-IR spectra of VZL with voriconazole-dinitrate salt and cocrystals.

Table S4 FT-IR stretching frequencies of solid forms (cm⁻¹).

Solid forms	O-H stretch	O-H bending (in-plane)	O-H bending (out-of-plane)	C=O stretch cocrystal (Acid group in coformer)
VZL	3445.4	1277.4	663.1	-
VZL-HNO ₃	3419.5	1243.6	656.7	-
VZL-PHBA	3445.7	1264.3	676.6	1698.1
VZL-PABA	3440.4	1272.1	684.7	1681.6
VZL-3-NBA	3407.2	1260.9	673.0	1694.6

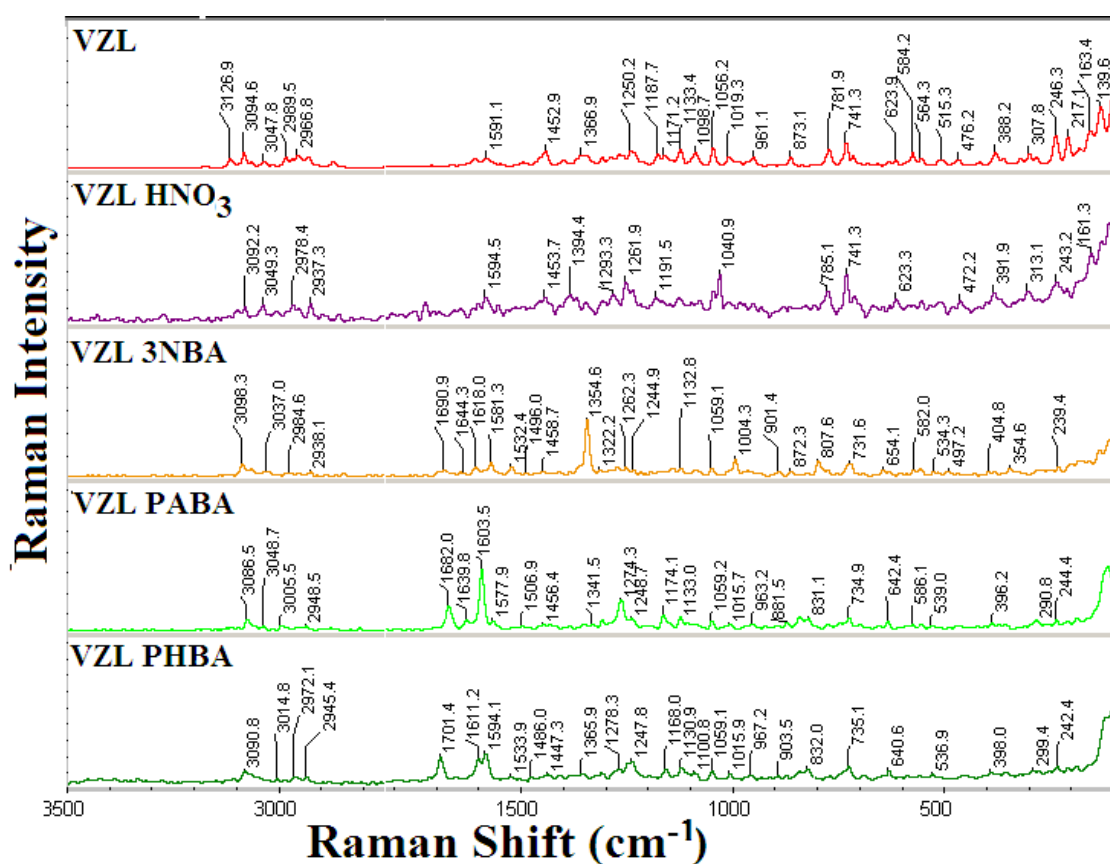


Figure S3 Comparison of FT-IR spectra of VZL with voriconazole-dinitrate salt and cocrystals.

Table S5 FT-Raman frequency of crystalline VZL forms (cm⁻¹).

Solid forms	Aromatic C-H stretch	Aliphatic C-H stretch	O-H bending (in-plane)	O-H bending (out-of-plane)	C=O stretch cocrystal (Acid group in coformer)
VZL	3094.6	2966.8	1250.2	623.9	-
VZL-HNO ₃	3092.2	2937.3	1261.9	623.3	-
VZL-PHBA	3086.3	2938.1	1262.3	654.1	1690.9
VZL-PABA	3086.5	2948.5	1274.3	642.4	1682.0
VZL-3-NBA	3090.8	2945.4	1278.3	640.6	1701.4

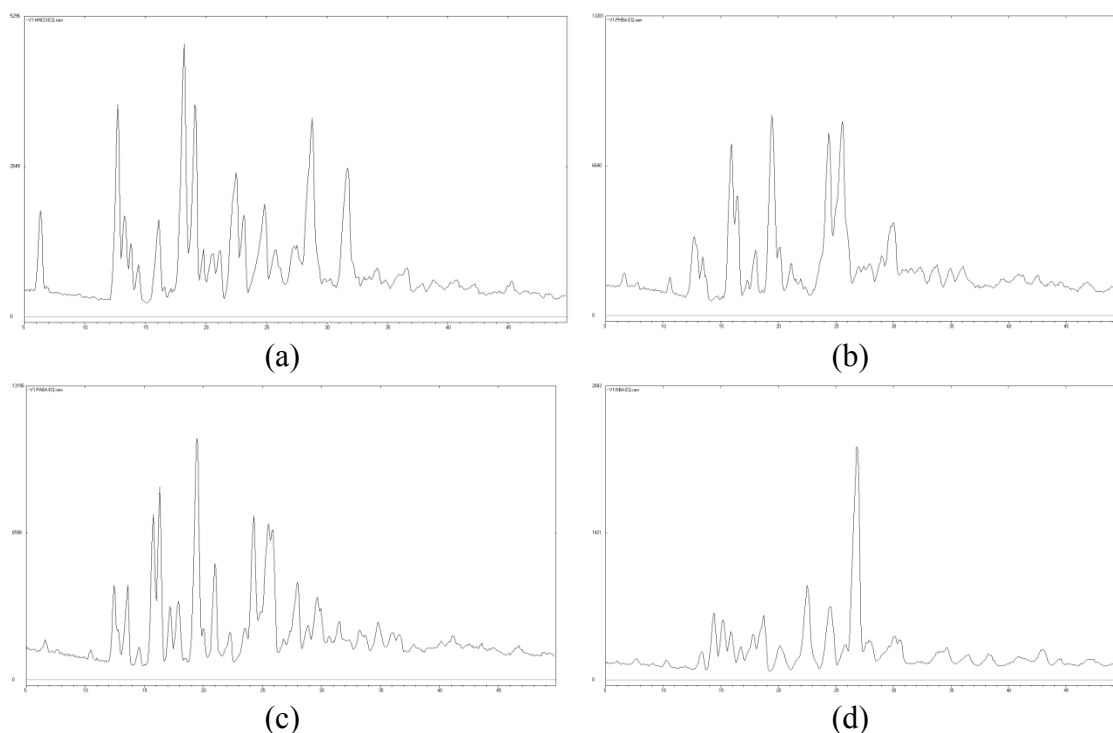
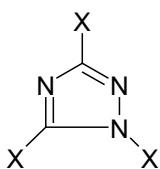
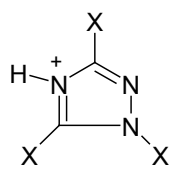
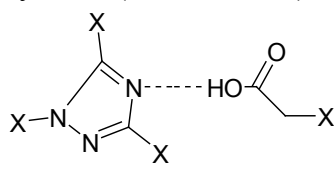
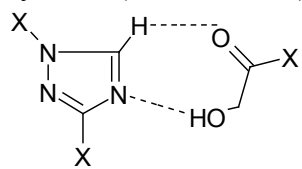


Figure S4 PXRD of (a) Dinitrate salt, (b) VZL-PHBA, (c) VZL-PABA, (d) VZL-3-NBA after slurry in the solubility medium 0.1 N HCl for 24 h.

Table S6 CSD Search results on 1,2,4-triazole ring fragment. Cambridge Structural Database, ver. 5.34, ConQuest 1.15, November 2012 release, May 2013 update.

Total number of 1,2,4-triazole	Protonated 1,2,4-triazole	Acid-triazole one point synthon (1,2,4-triazole)	Acid-triazole two point synthon (1,2,4-triazole)
 758 Hits	 38 Hits	 18 Hits	 2 Hits
BAHJIF01	FEDCUO	ATEYII	UPOQIA
BATWOK	XAZBUX	EPAGUY	WACLUJ
BATXAX	AHUYIL	EVIMIF	
FEDCUO	AXASUO	EZEGIA	
GECLUX	AZoziz	GUZDEL	
LEFROF	BUHCEM	IKEQEU	
NAZREN	CINCAE	IQIHEW	
XAZBUX	CUBVOL	IQIHEW01	
ABIZAO	CUBVUR	KOPSOX	
ABOKUY	ELEPER	MOFVOT	
ABUHAZ	GACNUV	NIFCIP	
ADAVUX	HALHUX	ODITOL	

ADIRAH	HEKSAR	QALZIO	
ADIREL	KABPOU	UPOQEW	
ADUPIY	KEQMAV	UPOQIA	
AFOFAC	LALCOQ	WACLUI	
AFOFEG	LEJRUN	ZAPMIO	
AHIKIM	LERYAJ	HEBMUY	
AHUYIL	MORETZ		
AJOFUB	NUJHIK		
ALAYUH	OQUJEQ		
AMIFIM	QAKNAR		
AMTAZC	QISQEP		
AMTRAZ	QULVEY		
AMTZZT	SEZHOV		
APIDUZ	TOMPAN		
APIFAH	UGARUP		
AQIJUF	UPOQAS		
AQIKIU	VOYPAA		
ATEYII	WETQES		
AVINAU	XENROY		
AXALOB	XENRUE		
AXASUO	XIXQUR		
AXEJUI	YAXFEK		
AXEKAP	ZAPGUU		
AZAKOC	ZIVROL		
AZEPEA	ZIVRUR		
AZOSIZ	UNEROV01		
BAHJIF			
BAKLOQ			
BEBXEL			
BEBXOV			
BEGRIP			
BEGRIP			
BESZUU			
BEZBEN			
BIGSOA			
BIHYOH			
BIJLEL01			
BIYROQ			
BNITRA10			
BNITRB10			
BOQKIB			
BUFHAM			

BUFPEX			
BUFPIB			
BUHCEM			
BUVRAM			
CABQAZ			
CABQED			
CAFYEN			
CAGVIQ			
CASHAH			
CAZBUC			
CAZCAJ			
CAZWEG			
CEFTOW			
CEFTUC			
CEFVAK			
CERYII			
CESCUZ			
CEXMAU			
CFPZBZ			
CIFROY			
CIFROY01			
CIHHAD			
CIJFOQ			
CINCAE			
CIQKAP			
CIWMIF			
CIWMOL			
CLTRZL01			
CMPZBZ			
CMTZFM			
CUBVOL			
CUBVUR			
CUBWEC			
CUGHUH			
CUHJOF			
CUJSAB			
CUJSEF			
CUNTIO			
CUQBIA			
CUVMEM			
CUVMIQ			
DABYUB			

DAJWIU10			
DAMTRZ11			
DAMTRZ20			
DEGGAY			
DELMOX			
DEWCEO			
DEXREE			
DIWMEC			
DOKZOT			
DOLBUC			
DOLCAJ			
DPTZTZ10			
DUHWAF			
DUNWIT			
DUPFEA			
DUSRUF			
DUTLAG			
EBIHAA			
ECEFOI			
ECUMAR			
EDEPAE			
EDONIV			
EDONOB			
EHAWAL			
EHIQOC			
EHOJAN			
EKA FEC			
ELEPER			
EMEBII			
EMEWAV			
EMEXIE			
EMOZUB			
ENIHUE			
ENIJAM			
ENIJEQ			
ENITIE			
EPAGUY			
EPYROB			
EQITII			
ETOMAB			
EVEQAY			
EVIBUG			

EVIMIF			
EVIMOL			
EVITAE			
EVIXEM			
EWETOQ			
EXOCUP			
EXUFEI			
EXUFIM			
EYATUU			
EZEGIA			
EZOPAL			
FABVAH			
FADQIM			
FADWAI			
FALDAZ			
FAXWEH			
FAYFIV			
FECQAF			
FEDKUV			
FEDLIK			
FEDVAM			
FEHZIC			
FEHZOI			
FETWAD			
FETWAD01			
FEVPAX			
FEVZAI			
FEWLIC			
FEWLOI			
FEZBIW			
FIDCUR			
FIHTUM			
FIXCAR			
FOBYIF			
FOQPIL			
FORJUR			
FUCLOE			
FUGSUW			
FUHHAR			
FUQMIO			
GACNUV			
GAQMOC			

GAQTEZ			
GAQTIC			
GAVFUF			
GAVVIJ			
GAVXIL			
GEGGUV			
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GEJJOU			
GEJJUA			
GEMNIW			
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GICPIR			
GICTIV			
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GICYOG			
GIHDUX			
GIHFAF			
GIPXIN			
GISVEK			
GITFEV			
GIYQEL			
GOJKIZ			
GOMCIU			
GUJTOU			
GUZDEL			
HAFKEG			
HAFRIP			
HAJMOU			
HAKWUN			
HALFOR			
HALHOR			
HALHUX			
HAMGOT			
HAMXUP			
HANDOQ			
HANFEI			
HEBKII			
HEBKOO			
HEBMUW			

HEBNAD			
HEBNEH			
HEBNIL			
HEKSAR			
HEPFOX			
HESNOJ			
HESTUV			
HEXJID			
HEXKEA			
HIKPIA			
HIKPOG			
HIRTIM			
HOFKAO			
HOGYEH			
HOSZEV			
HOTNEK			
HOTSOY			
HUFPAA			
HUHJEA			
HULGOK			
HUVCAD			
HUXJUG			
IBOMAP			
IDIFEH			
IDIFIL			
IDIFOR			
IDIFUX			
IDIGAE			
IDIGEI			
IDUZIR			
IFAHIH			
IFAHON			
IFAHUT			
IFOFEQ			
IFOYIM			
IGUREI			
IHAFIH			
IHAYEW			
IHAYIA			
IKALUC			
IKEQEU			
IMIKUK			

IQIHEW			
IQIHEW01			
IQOLUW			
IQOMAD			
IRUZOL			
ISALAP			
ISEHAQ			
ISEHEU			
ISEHIY			
IVUQIZ			
IVUQOF			
IVUQOF01			
IVUQOF02			
IVUQOF03			
IVUQOF04			
IWIXAO			
IWIXOC			
IWOCED			
IWOCIH			
IXALIC			
IXANIE			
IXOTOF			
IXOTUL			
IYIGIH			
IZANIG			
JABYOA			
JABYOA01			
JADFAW			
JASGIU			
JASVII			
JAXMAX			
JAXSEH			
JAXVEK			
JAXZUE			
JAZVEL10			
JAZVIP10			
JAZVOV10			
JEJRIA			
JESHEV			
JEYROV			
JIIWET			
JOWWIB			

JUFPEF			
KABJON			
KABJUT			
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KABPOU			
KAFWUL			
KAHRAO			
KAHRES			
KAKMAK			
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KATCIS			
KATFER			
KAYZEQ			
KECSUG			
KEFRES			
KEFRIW			
KEQMAV			
KEQQAZ			
KIVFIE			
KIYZEY			
KOBTUQ			
KOBYOP			
KOKDET			
KOPSOX			
KUVHEP			
LALCOQ			
LAPVEF			
LAPXEG			
LAPXIK			
LEDVAR			
LEJRUN			
LERKAU			
LERKEY			
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LOCDOX			
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MUBKOK			
MUBLEB			
MUHSUD			
MUKRAM			
MUKREQ			
MULLIP			
MULLUB			

MXPMTZ			
NABVIV			
NACGOO			
NACMEJ			
NADJIN			
NAPHET			
NAVKOM			
NAYRUB			
NENFAO			
NEQRIL			
NERLOM			
NERQEH			
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NIFYIK			
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NOQFAB			
NOQTUJ			
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NUQGUC			
NUYLAV			
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OFIGOA			
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PIDHIU			
PIFJIY			
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PUGDIF			
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PUJJIO			
QACFOP			
QAHTIC			
QAJYAD			
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QAJYOP			
QAKFAK			
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REMKEA			
REXQIV			
REYJOU			
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ROBZOX			
ROFJIG			
ROGCEW			
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RUFDUS			
RUKBAB			
RUKBEF			
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RUQTUT			
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ZUHVED			
ACIHEB			
ACIHOL			

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CEJJIM			
CEJJOS			
DEDNUX			
HEBMUY			
MEBSAP			
NEFPAR			
QECYOO			
SEDLAQ			
SEDMEV			
VAZCAC			
VECGIV			
VEFXEL			
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VEPWYI			
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FEMTEY			
KEHWUR			
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