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Supporting information for article:

Deformation of polyiodides in Cs₂I₈ crystals at high pressure

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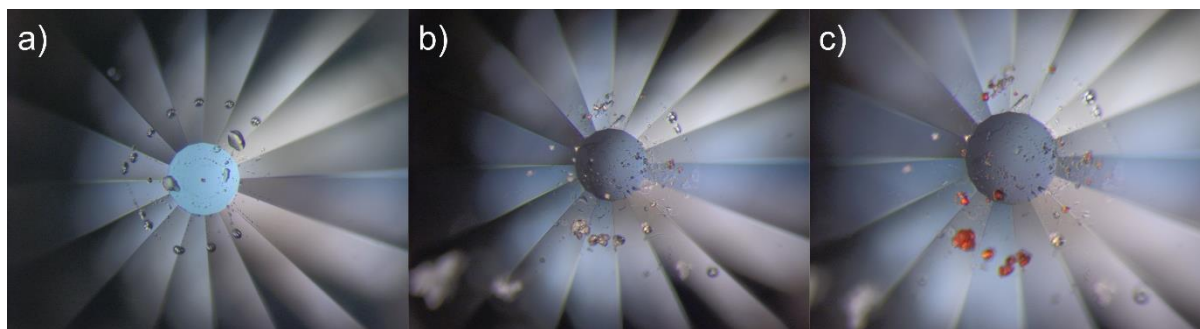
Supplementary Information

Figure 1. Identification of reaction products between the iodine vapour (produced by decomposing Cs_2I_8) and a steel-gasket material. Oil deposited at the rim of the sample chamber (a); crystals of 1,10-phenanthroline placed onto the oil droplets (b), small droplets of water added on top of the crystals (c), revealed that a red ferriox complex has been formed. Thus, the droplets contained Fe^{2+} ions, which corresponds to FeI_2 – a product of reaction between the oxidizing iodine vapour and a main steel gasket component.

Table S1 Experimental and crystal data of Cs₂I₈.

Cs ₂ I ₈					
Pressure (GPa)	0.0001(2)	0.43(2)	0.91(2)	1.15(2)	1.65(2)
Temperature (K)	296(2)				
Formula weight	640.51				
Crystal color	Dark red				
Crystal size (mm)	0.035×0.015×0.01				
Crystal system	Monoclinic				
Space group	P2 ₁ /c				
Unit cell (Å) <i>a</i>	10.2847(9)	10.1323(13)	9.997(3)	9.934(2)	9.842(3)
<i>b</i>	8.98090(10)	8.88899(15)	8.8025(3)	8.7538(2)	8.7061(3)
<i>c</i>	11.2100(4)	11.0453(4)	10.9003(8)	10.8339(6)	10.7364(10)
β (°)	114.598(7)	114.957(9)	115.28(2)	115.517(14)	115.68(2)
Volume (Å ³)	941.46(10)	901.92(14)	867.3(3)	850.2(2)	829.1(3)
<i>Z</i>	4	4	4	4	4
Density (g/cm ³)	4.519	4.717	4.905	5.004	5.131
Wavelength MoK α (Å)	0.41077	0.41077	0.41077	0.41077	0.41077
Absorption (mm ⁻¹)	3.842	4.036	4.197	4.282	4.391
F(000)	1068.0	1068.0	1068.0	1068.0	1068.0
2 θ max (deg.)	42.324	40.018	39.98	39.95	40
Index ranges <i>h</i> _{min} / <i>h</i> _{max}	-8/8	-6/6	-7/6	-6/7	-6/7
<i>k</i> _{min} / <i>k</i> _{max}	-15/15	-12/12	-12/12	-12/11	-12/11
<i>l</i> _{min} / <i>l</i> _{max}	-19/18	-16/16	-16/16	-16/16	-16/16
Refl. Collected	3490	2140	2040	1961	1939
Refl. observed (<i>I</i> >4 σ _{<i>I</i>})	1881	1137	1090	1069	1051
<i>R</i> (int)	0.0194	0.0188	0.0147	0.0200	0.0163
Data/restraints/parameters	1881/0/46	1137/0/46	1090/0/46	1069/0/46	1051/0/46
Goodness-of-fit on F ²	0.986	1.040	1.108	1.008	1.158
Final <i>R</i> ₁ (<i>I</i> >2 σ _{<i>I</i>})	0.0317/ 0.0835	0.0334/ 0.0985	0.0355/ 0.1048	0.0314/ 0.0856	0.0519/ 0.1696
<i>R</i> ₁ / <i>wR</i> ₂ (all data)	0.0373/ 0.0898	0.0355/ 0.1000	0.0379/ 0.1067	0.0331/ 0.0872	0.0536/ 0.1713

Table S1 (Continuation) Experimental and crystal data of Cs₂I₈.

Cs ₂ I ₈					
Pressure (GPa)	1.97(2)	2.32(2)	2.74(2)	3.05(2)	3.33(2)
Temperature (K)	296(2)				
Formula weight	640.51				
Crystal color	Dark red				
Crystal size (mm)	0.035×0.015×0.01				
Crystal system	Monoclinic				
Space group	<i>P</i> 2 ₁ / <i>c</i>				
Unit cell (Å) <i>a</i>	9.788(2)	9.7195(12)	9.6370(13)	9.6171(13)	9.5973(15)
<i>b</i>	8.6889(3)	8.6486(2)	8.6017(2)	8.5889(2)	8.5648(2)
<i>c</i>	10.6600(7)	10.5900(4)	10.4967(4)	10.4726(4)	10.4284(4)
β (°)	115.607(17)	115.709(9)	115.788(9)	115.810(10)	115.854(11)
Volume (Å ³)	817.6(2)	802.07(12)	783.46(12)	778.75(13)	771.40(14)
<i>Z</i>	4	4	4	4	4
Density (g/cm ³)	5.204	5.304	5.430	5.463	5.515
Wavelength MoK α (Å)	0.41077	0.41077	0.41077	0.41077	0.41077
Absorption (mm ⁻¹)	4.453	4.539	4.647	4.675	4.719
F(000)	1068.0	1068.0	1068.0	1068.0	1068.0
2 θ max (deg.)	34.12	42.688	43.006	43.09	43.248
Index ranges <i>h</i> _{min} / <i>h</i> _{max}	-6/7	-6/7	-6/7	-6/7	-7/6
<i>k</i> _{min} / <i>k</i> _{max}	-11/11	-12/11	-12/11	-12/11	-12/11
<i>l</i> _{min} / <i>l</i> _{max}	-15/15	-15/16	-15/16	-15/16	-15/16
Refl. Collected	1592	1880	1897	1893	1785
Refl. observed (<i>I</i> >4 σ <i>I</i>)	827	1023	1027	1028	1002
<i>R</i> (int)	0.0219	0.0135	0.0197	0.0214	0.0232
Data/restraints/parameters	827/0/46	1023/0/46	1027/0/46	1028/0/46	1002/0/46
Goodness-of-fit on F ²	1.047	1.127	1.056	1.126	1.101
Final <i>R</i> ₁ (<i>I</i> >2 σ <i>I</i>)	0.0233/ 0.0704	0.0194/ 0.0585	0.0233/ 0.0603	0.0295/ 0.0816	0.0360/ 0.1007
<i>R</i> ₁ / <i>wR</i> ₂ (all data)	0.0246/ 0.0714	0.0207/ 0.0594	0.0241/ 0.0609	0.0303/ 0.0824	0.0371/ 0.1012

Table S1 (Continuation) Experimental and crystal data of Cs₂I₈.

Cs ₂ I ₈				
Pressure (GPa)	4.19(2)	4.71(2)	5.42(2)	5.91(2)
Temperature (K)	296(2)			
Formula weight	640.51			
Crystal color	Dark red			
Crystal size (mm)	0.035×0.015×0.01			
Crystal system	Monoclinic			
Space group	<i>P</i> 2 ₁ / <i>c</i>			
Unit cell (Å) <i>a</i>	9.4705(13)	9.4109(11)	9.3544(14)	9.2978(12)
<i>b</i>	8.5022(2)	8.46960(10)	8.4389(2)	8.4056(2)
<i>c</i>	10.2962(4)	10.2258(3)	10.1587(4)	10.0857(4)
β (°)	115.934(10)	115.972(9)	115.998(11)	116.015(9)
Volume (Å ³)	745.56(12)	732.75(10)	720.79(13)	708.37(11)
<i>Z</i>	4	4	4	4
Density (g/cm ³)	5.706	5.806	5.902	6.006
Wavelength MoK α (Å)	0.41077	0.41077	0.41077	0.41077
Absorption (mm ⁻¹)	4.883	4.968	5.051	5.139
F(000)	1068.0	1068.0	1068.0	1068.0
2 θ max (deg.)	42.816	43.046	43.264	42.642
Index ranges <i>h</i> _{min} / <i>h</i> _{max}	-6/7	-7/6	-6/7	-7/5
<i>k</i> _{min} / <i>k</i> _{max}	-11/11	-12/11	-12/11	-12/11
<i>l</i> _{min} / <i>l</i> _{max}	-15/15	-15/15	-15/15	-15/15
Refl. Collected	1843	1753	1710	1604
Refl. observed (<i>I</i> >4 σ <i>I</i>)	993	951	935	899
<i>R</i> (int)	0.0130	0.0150	0.0129	0.0246
Data/restraints/parameters	993/0/46	951/0/46	935/0/46	899/0/46
Goodness-of-fit on F ²	1.099	1.284	1.045	1.012
Final <i>R</i> ₁ (<i>I</i> >2 σ <i>I</i>)	0.0217/ 0.0607	0.0338/ 0.1318	0.0220/ 0.0670	0.0191/ 0.0479
<i>R</i> ₁ / <i>wR</i> ₂ (all data)	0.0223/ 0.0611	0.0343/ 0.1323	0.0227/ 0.0677	0.0197/ 0.0485

Table S2 Comparison of selected iodine geometrical features in Cs₂I₈ determined in the previous work¹ and data from this study

Intra-/intermolecular distance (Å)	Havinga et al. (1954) ¹	This work
I1-I2	2.84(2)	2.8497(2)
I2-I3	3.00(2)	3.029(1)
I3—I4	3.42(2)	3.4431(8)
I4-I4'	2.83(3)	2.766(1)
Interatomic angles (deg.)		
I1-I2-I3	176(1)	176.08(3)
I2-I3-I4	80(1)	81.37(2)

(1) Havinga, E. E.; Boswijk, K. H.; Wiebenga, E. H. The Crystal Structure of Cs₂I₈(CsI₄). *Acta Crystallogr.* **1954**, 7 (6), 487–490.