**Abstract:** As a conventional strategy to modulate the structure and properties of inorganic single crystals, hydration/solvation is rarely found to function in pure organic single crystals. Herein, we report chalcone single crystals with CHCl$_3$-dependent emission color and jumping behavior. Two crystals: a pure crystal phase (1) with green-yellow emission and a CHCl$_3$-containing co-crystal phase (2•2CHCl$_3$) with orange-red emission were constructed by fine-controlling the crystallization conditions. The special halogen bond network in the crystal packing structure effectively narrows the bandgap and thereby redshift the emission of 1. 2•2CHCl$_3$ would revert to 1 together with emission color change when losing CHCl$_3$. These findings are similar to the effect of H$_2$O in hydrated inorganic crystals. Notably, owing to a special pre-organized “molecular pair” structure for [2 + 2] cycloaddition, the pure crystal phase 1 exhibits violently photo-induced jumping phenomenon, indicating large potentials in intelligent devices. This work would overturn the previous perception that the structurally simple solvent molecules without conjugation cannot greatly affect the structure and properties of pure organic single crystals and provide a new strategy to construct multi-colored organic fluorescent crystals.

**Keywords:** cyclic chalcone; CHCl$_3$-dependent emission; jumping crystal; halogen bond network effect; cycloaddition

Organic/inorganic single crystals have attracted increasing attention in optoelectronics, e.g., as field effect transistors, light-emitting diodes, solid state lasers, and solar cells [1–10]. It is well known that some inorganic crystals contain certain amounts of coordinating water, which endows the crystals with various colors. For example, CuSO$_4$·5H$_2$O and CoCl$_2$·6H$_2$O are blue and pink, respectively. When coordinating water is lost, they become white and blue. The color change is due to energy level splitting of the metal ions induced by crystalline water [11]. In particular, if the hydrated crystals are highly emissive, smart fluorescent sensors would be available based on the H$_2$O-adsorption/desorption processes. Additionally, controlling the content of crystalline water can lead to multi-colored fluorescent crystals. Therefore, the hydration method is expected to become a useful strategy to modulate emission color of inorganic single crystals [12–14]. For example, Su et al. prepared a red emissive hydrated perovskite crystal (Cs$_3$InBr$_5$·H$_2$O) which would become yellow fluorescent when H$_2$O is lost [12]. Manos et al. presented fluorescent metal–organic framework (MOF) crystals ([Mg(H$_2$dhtp)(H$_2$O)$_2$]-DMAc) whose emission...
color would change with the containing solvents [13]. To expand solvation-dependent emission to organic fluorophore, Yan et al. synthesized amphiphilic organic molecules which coordinate to metal ions with different hydration ability [15]. In recent years, researchers have sought to involve solvent molecules in the molecular packing structures of organic crystals to tune the emission color [16–18]. For example, Masaru Tanioka and co-workers prepared single crystals of cis-ABPX010 by slow diffusion of less solubilizing solvent EtOAc into a solution of the compound in more solubilizing solvent CH₂Cl₂, which appear to be deep purple under UV irradiation. X-ray crystallography showed that the crystal included CH₂Cl₂ molecules in the lattice [17]. In general, the solvent molecules are difficult to participate into the molecular packing structures in organic crystals, which is distinctly different from inorganic crystals. The basic reason is that solvent molecules cannot coordinate with pure organic molecules (without metal ion) and thus it is difficult to change the molecular energy band. Besides, structurally simple solvent molecules without conjugation could hardly affect the molecular energy band. Here in this work, we report a solvent-dependent emission behavior of pure organic single crystals, similar to hydrated inorganic crystals. To be specific, CHCl₃ can participate in the packing structure as well as the molecular orbital distributions of chalcone-based crystals via halogen bond network, thus substantially changing the crystal’s emission color. Single crystal X-ray diffraction analysis and theoretical calculation confirm the contribution of CHCl₃-induced halogen bond network. Moreover, crystal phase 1 displays cycloaddition-triggered crystal jumping behavior, suggesting its great potential in the smart robot area.

Compound 1 is a new compound and is synthesized according to the literature (Figure 1) [19]. At 0 °C, 1.0 g NaOH in 2 mL H₂O was dropwise added to the mixture of 5-chloro-1-indanone (1.66 g, 10 mmol) and 4-(diphenylamino)benzaldehyde (2.73 g, 10 mmol) in 60 mL ethanol. The mixture was stirred for 1 h. The yellow precipitates were filtered out, washed with water and cold ethanol, and then dried as crude product. Two kinds of crystals were prepared (Figure 1), which are a pure crystal phase (crystal 1) and a CHCl₃-containing co-crystal phase (crystal 1•2CHCl₃). The crystals were prepared as follows: in a round bottom flask, the crude product (1.0 g) was dissolved with THF or CH₂Cl₂ (40 ml) to obtain a concentrated solution and triple amount of methanol was slowly added along the flask-wall without destroying the solution surface. After 48 h at 0–5 °C, plenty of green-yellow emissive crystals precipitated out (named as crystal 1, Figure 2a), and the structure was characterized by NMR spectroscopy. Using the CHCl₃/ethanol (20 ml CHCl₃ and 60 ml ethanol) system always gave rise to two kinds of crystals, the flaky green-yellow emissive crystal 1 and the blocky orange-red emissive crystal 1•2CHCl₃ (named as 1•2CHCl₃ because it contains two-equivalent CHCl₃ molecules, Figure 2a). As shown in Figure 2c, compound 1 displays an intense absorption band peaking at 432 nm in CH₂Cl₂. Its solution is yellow fluorescent (558 nm). Compound 1 is brightly emissive in the crystalline state. The pure crystal phase 1 shows green-yellow emission (546 nm) with the fluorescence quantum yield of about 0.22 (Figure 2a,c), and its fluorescence lifetime is about 3.47 ns (Supplementary Figure S1). The co-crystal phase 1•2CHCl₃ shows orange-red emission (Figure 2a). However, due to the rapid volatilization of CHCl₃ molecules, the crystal would quickly deteriorate when separated from the crystallizing mother solution, and its emission color then switched to green (Figure 2a), indicating the crystals displays CHCl₃-dependent emission color. This also results in the lack of data for crystal 1•2CHCl₃, including the emission spectrum as well as other photophysical data. The PXRD pattern of crystal 1•2CHCl₃ when losing CHCl₃ is strictly the same as that of crystal 1 (Figure 2b), demonstrating that the chalcone molecules in 1•2CHCl₃ have the same packing mode as those in crystal 1 and CHCl₃ molecules just fill in the crystal without changing the main molecular network. Nevertheless, considering the fact that crystal 1•2CHCl₃ shows obviously redshifted emission compared with crystal 1, CHCl₃ must be involved in the molecular orbitals and plays a key role in red-shifting the emission. This is not ordinary because CHCl₃ is just a solvent molecule and has no aromatic rings. Additionally, when fuming with CHCl₃, the desolvated yellow block (1•2CHCl₃ when losing CHCl₃) would
gradually become orange (Supplementary Figure S2), further confirming the significance of CHCl₃ on tuning the energy band of this type chalcone derivative.

**Figure 1.** Synthesis, cycloaddition reaction and two different crystal phase of compound 1.

Interestingly, the crystal phase 1 displays photo-induced crystal jumping behavior, which is a very scarce motion mode among mechanically responsive crystals [20–23]. When exposed to UV irradiation, the thin flakes of crystal 1 would jump violently and split into small pieces (videos S1–S2), like a lively dancer. As shown in Figure 3, the crystals’ positions changed obviously over time, and the number of the crystals inside the red circle first decreased and then increased, clearly showing the crystal movement. Meanwhile, some
Interestingly, the crystal phase 1 displays photo-induced crystal jumping behavior, where the crystal jumps away and back in the red circle. In the co-crystal 1•2CHCl₃, molecules of compound 1 take more planar conformations along the crystallographic b direction (Figure 4b). The two carbonyl vinyl groups in each “molecular pair” are very close (3.3–3.4 Å) and have large overlap (Figure 4c), providing a prerequisite for cycloaddition reaction. Consequently, the “molecular pair” can function as a pre-organized structure for photo-induced dimerization. Given a weak light (handhold UV lamp), the crystal would react, and the photochemical reaction-caused lattice distortion results in the motion responses (hopping, splitting, and bending) of the crystal. In the co-crystal 1•2CHCl₃, molecules of compound 1 take more planar conformations accompanied by two solvent molecules. Similar “molecular pair” chain structure exists in crystal 1•2CHCl₃ except that large amounts of CHCl₃ molecules fill inside (Figure 4d). Thus, when the crystal loses solvent molecules, it has the same PXRD pattern as crystal 1 (Figure 2b). Notably, the CHCl₃ molecules appear between the molecules in clusters, not singly (Figure 4e). Each cluster contains four CHCl₃ molecules. Two fluorescent molecules are connected by the four CHCl₃ molecules via strong Cl⋯Cl (3.47 Å) halogen

Figure 3. A series of microscopic images showing the jumping and splitting behavior of crystal 1 under UV light (crystals jump away and back in the red circle).

To disclose the reasons why the two crystal phases display distinctively different emission color and why crystal 1 shows such fantastic mechanical motion phenomenon, both of 1 and 1•2CHCl₃ crystals (CCDC:1975830-1975831) were measured by single crystal X-ray diffractometer. Their crystal structures are shown in Figure 4. The unit cell of crystal 1 is triclinic and contains two molecules (Supplementary Figure S4), and the π plane of the molecule has relatively flat conformation with a dihedral angle of 14.80° between the two phenyl rings (Figure 4a). Notably, every two molecules with opposite orientation pair up via strong π⋯π interactions, thus forming special infinite “molecular pair” chains along crystallographic b direction (Figure 4b). The two carbonyl vinyl groups in each “molecular pair” are very close (3.3–3.4 Å) and have large overlap (Figure 4c), providing a prerequisite for cycloaddition reaction. Consequently, the “molecular pair” can function as a pre-organized structure for photo-induced dimerization. Given a weak light (handhold UV lamp), the crystal would react, and the photochemical reaction-caused lattice distortion results in the motion responses (hopping, splitting, and bending) of the crystal. In the co-crystal 1•2CHCl₃, molecules of compound 1 take more planar conformations compared with those in crystal 1 with a dihedral angle between the two terminal phenyl groups of only 5.94° (Figure 4d). The unit cell contains two molecules of compound 1 and four CHCl₃ molecules (Supplementary Figure S4), indicating each fluorescent molecule is accompanied by two solvent molecules. Similar “molecular pair” chain structure exists in crystal 1•2CHCl₃ except that large amounts of CHCl₃ molecules fill inside (Figure 4e). Thus, when the crystal loses solvent molecules, it has the same PXRD pattern as crystal 1 (Figure 2b). Notably, the CHCl₃ molecules appear between the molecules in clusters, not singly (Figure 4e). Each cluster contains four CHCl₃ molecules. Two fluorescent molecules are connected by the four CHCl₃ molecules via strong Cl⋯Cl (3.47 Å) halogen
bond and C–H···O (2.19, 2.48 Å) interactions (Figure 4f). Additionally, along c direction, molecules connect with each other via C–H···Cl (2.94 Å) interactions. Consequently, infinite halogen bond networks are observed in crystal 1•2CHCl₃ which probably can reduce the energy bandgap in some extent (Supplementary Figure S5). As a result, the emission color of crystal 1•2CHCl₃ redshift a lot compared with that of crystal 1. Without CHCl₃, the crystal’s halogen bond network was destroyed and its fluorescence blueshift to the same color as crystal 1. It is most possibly due to the halogen bond effect that the CH₂Cl₂ solution and CHCl₃ solution of this type of chalcone derivative display abnormal solvent effect (Supplementary Figure S6). The absorption and emission peaks in CH₂Cl₂/CHCl₃ obviously shift towards the longer wavelength compared with those in THF and ethyl acetate. The maximum absorption wavelength of compound 1 in CHCl₃ (λₐbs = 436 nm) is even larger than that in ethanol (λₐbs = 432 nm).

![Figure 4. Crystal structures of the pure crystal phase 1 (a–c) and the co-crystal phase 1•2CHCl₃ (d–f): (a) top view and side view of the molecule; (b) special “molecular pairs” packing mode along crystallographic b direction; (c) side view and top view of the “molecular pair” with short distance between the two vinyl groups (marked in yellow); (d) top view and side view of the molecule; (e) “molecular pairs” packing structure along crystallographic b direction with large amounts of CHCl₃ filling in the crystal and (f) strong interactions between the two molecules of compound 1 and four CHCl₃ molecules.](image)

For further understanding of the structure-property relationships of the two different crystals, calculations of molecular orbital distribution as well as intermolecular interactions were conducted. As shown in Figure 5a, the HOMO orbital of compound 1 in monodisperse state mainly locates at the triphenylamino group while the LUMO orbital centrally distributed around the indanone part, consistent with the D-A character of this type of chalcone derivative. Notably, in the tightly packing structure where molecules form special “molecular pairs”, the LUMO orbitals at the carbonyl vinyl (C=C=O) part of the two molecules display very large overlap (Figure 5b), in good consistence with the experimental fact that crystal 1 display remarkably [2 + 2] cycloaddition photoreactivity. As demonstrated by the single crystal X-ray diffraction, there exist large amounts of...
CHCl₃ clusters in the co-crystal 1•2CHCl₃. The interactions between the CHCl₃ cluster and its two neighboring chalcone molecules were calculated (Figure 5c) Combined with the two functions of reduced density gradient (RDG) and \(\text{sign} (\lambda_2) \rho\), one can gain the weak interaction regions for the molecules, where \(\lambda_2\) and \(\rho\) is the second eigen value of Hessian matrix and electron density. The \(\text{sign} (\lambda_2) \rho\) is mapped on the isosurface of RDG, where the values of \(\text{sign} (\lambda_2) \rho\) are in the range of \(-0.035\) to \(-0.020\) with the different colors denote from blue to red as shown in Figure 5c. The color-filled scatter and the distribution of the interactions among molecules clearly demonstrate that there exist abundant dispersion interactions between the CHCl₃ cluster and its adjacent chalcone molecules (Figure 5c). These interactions prompt the molecules to form a multi-molecules system, like one large complex. Inside this complex, CHCl₃ molecules could also to some extent participate in the molecular orbital distribution (Supplementary Figure S7). Consequently, the calculated HOMO-LUMO energy gap of the complex (2.907 eV) is lower than that of the monomer (3.132 eV), in good agreement with the experimental fact that: (I) 1•2CHCl₃ crystal displays redshifted emission compared with crystal 1; (II) 1•2CHCl₃ crystal would blueshift its emission when losing CHCl₃.

![Figure 5](image)

**Figure 5.** The calculated HOMO and LUMO molecular orbitals of compound 1 in (a) monodisperse state and (b) “molecular pair” state, the colors of lime and cyan are positive and negative phase; (c) The RDG isosurfaces as well as color-filled map of RDG versus the product of second Hessian eigen and electron density for the CHCl₃-containing system.

In conclusion, a cyclic chalcone derivative was synthesized and its pure crystal phase 1 was demonstrated to show attractive photoinduced motion behavior, indicating that this type of cyclic chalcone is a kind of promising candidate for the construction of crystal machine. The CHCl₃-containing co-crystal phase 1•2CHCl₃ was prepared by regulating the crystallization environment. Crystal 1•2CHCl₃ shows relatively large-redshifted emission.
compared with crystal 1, which is ascribed to its special halogen bond network structure. This would blueshift its emission to green color as 1 loses CHCl₃, further confirming the key effect of solvent molecule on crystal emission color. These findings suggest that some solvent molecules could also function as important contributors or participants in fine tuning the molecular orbitals via noncovalent interactions such as halogen bonding, in spite of their non-conjugated structure. This in-depth crystal structure investigation together with the theoretical calculations give clear evidence that: (1) the special “molecular pair” structure in crystal effectively facilitates the LUMO orbitals’ overlap and thus endows the crystal with [2 + 2] cycloaddition reactivity and then triggers the crystal jumping behavior; (2) the CHCl₃ molecules in crystal i•2CHCl₃ form clusters and tie fluorescent molecules together via abundant halogen bonds, thus forming a halogen bond network system with lower energy gap compared with the solvent-free crystal 1 system. We believe these results might not only provide significant information on disclosing the structure-property relationship of organic luminophores, but also have good guidance to the research and design of dynamic single crystals in material science.

Supplementary Materials: Experimental details are available online at https://www.mdpi.com/article/10.3390/cryst11050530/s1. Figure S1: Transient PL decay curve of compound 1 in crystal phase 1 state, Figure S2: The change of the desolvated yellow block (i•2CHCl₃ when losing CHCl₃) when fuming with CHCl₃, Figure S3: ¹H-NMR spectra (400 MHz, CD₂Cl₂) of crystal 1 after UV irradiation (the red curve) and the untreated sample (the black curve), Figure S4: Unit cells of crystal 1 and i•2CHCl₃. Figure S5: Infinite halogen bond network packing structure in crystal i•2CHCl₃, Figure S6: Absorption and emission spectra of compounds 1 in various solvents (THF: tetrahydrofuran; EA: ethyl acetate; DMSO: dimethylsulfoxide) with different polarity, Figure S7: The calculated HOMO and LUMO molecular orbitals of the multi-molecule “complex” (containing two chalcone molecules and four CHCl₃ molecules) where the two chalcone molecules are connected via abundant weak interactions, Figure S8: ¹H-NMR spectrum of compound 1 (400 MHz, in CDCl₃), Table S1: The maximum absorption wavelength \( \lambda_{abs} \) and maximum emission wavelength \( \lambda_{em} \) of compound 1 in different solvents, Video S1: Crystal jumping behavior of crystal 1 under UV light, Video S2: The bulk crystal split into small fragment and Video S3: Bending behavior of crystal 1 under UV light.

Author Contributions: Conceptualization, X.C. and J.S.; synthesis, Z.T., X.H. and X.C.; single crystal measurement, X.C.; photophysical property measurement, J.Z. (Jiaxin Zhao), Z.L. and L.Z.; theoretical calculation, J.Z. (Jian Zhao); writing—original draft preparation, Z.T. and X.C.; writing—review and editing, Z.T., C.Z. and X.C.; funding acquisition, X.C. All authors have read and agreed to the published version of the manuscript.

Funding: This work was financially supported by the Project funded by the National Natural Science Foundation of China (51903139), Shandong University, Key R & D projects of Shandong Province (2019ZZY010355) and the China Postdoctoral Science Foundation (2017M611985 and 2018T110589).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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