Abstract

Emission lines of free excitons and excitons bound to axial centers have been observed in the luminescence spectra of CdP$_2$-D$_4^8$ crystals doped with Mn, Sn, and Sb at 10 K. Bands models of excitons bound to axial centers (Mn, Sn, Sb) are proposed. The luminescence of crystals doped with Fe does not correspond to the axial centers band scheme. It is shown that direct transitions in excitonic band are polarized and in the case of indirect transitions they are unpolarized. The direct band gap is due to allowed transitions $\Gamma_1 \rightarrow \Gamma_1$ and $\Gamma_2 \rightarrow \Gamma_1$ in $E||c$ and $E \perp c$ polarizations, respectively. The coefficient of the energy shift in the temperature range of 2–10 K in $E||c$ and $E \perp c$ polarizations differs ($\Delta E/\Delta T = 3.5$ meV/K, $E||c$ and 1 meV/K, $E \perp c$).

Keywords: Condensed matter physics, Materials science

1. Introduction

Cadmium diphosphide (CdP$_2$) belongs to the group of wide-gap A$^{II}$B$^V$ semiconductor materials and possess a natural gyrotropy and anisotropy [1, 2, 3]. Reflection, absorption and photoluminescence spectra in the electronic transitions and vibrational modes region are observed [3, 4, 5, 6, 7, 8, 9, 10, 11, 12]. The values of the nonlinear polarization and gyrotropy of CdP$_2$ are higher than those of other crystals, providing an effective stabilization of the radiation field simultaneously in space (cross section) and in time [13, 14, 15, 16]. The saturation of the light intensity...
passing through the sample on the incident light intensity makes it possible to realize instantaneous feedback to the operating radiation field and to create stabilizers of the radiation field and extenders of the generation pulses duration. All this requires high perfection (optical homogeneity) of the crystals themselves, the achievement of which is a complex technological problem, which has been successfully solved in many aspects.

The low thermal conductivity (10 W/m*K) of CdP₂ crystals was used to create laser beam deflectors with a thermally induced gradient of the refractive index. The advantage of the deflectors is that they can be used in the visible and near infrared regions of the spectrum [17, 18].

In CdP₂, a linear dependence of the plane-polarized radiation polarization rotation on the magnetic field induction was observed in Ref. [19, 20] and the possibility of creating magneto-optical modulators based on CdP₂, sensitive elements for measuring magnetic field induction was demonstrated. On the basis of CdP₂ crystals a technology for obtaining p-n junctions of surface-barrier diodes has been developed [2, 3, 5, 6, 7, 8, 9]. Photoelectron emitters and photodiodes [21, 22, 23], electric switches [24, 25] and Zener diodes [26] have been developed on the basis of the CdP₂ single crystals. The CdP₂ crystals change the optical activity with a temperature changing, while maintaining the linear dependence of the rotational ability of the polarization plane on the temperature (temperature sensors [27]).

Below the main absorption edge, a strong anisotropy of optical properties associated with the anisotropy of the vibrational modes is observed. In the elementary cell CdP₂, 8 formula units are laid, i.e. 24 atoms and the number of phonon branches is 72 in the general case. The selection rules for vibrational modes are different for different light waves polarizations, which affects the dielectric constants and other parameters [7, 8, 12, 13].

The technology of Me-CdP₂ Schottky diodes obtaining has been developed, electric and photoelectric characteristics of diodes have been investigated, where In, Cu, Pt, Ga, Pb, Sb, Ag, Te, Au, Hg were used as metals. The parameters and properties of Schottky diodes are determined in a wide range of temperatures and light waves [2, 3, 5, 6, 7, 8, 9].

In this paper, we studied the emission spectra (luminescence) of CdP₂-D₄⁸ crystals doped with Mn, Sn, Sb and Fe measured at temperature 10 K. The emission lines of free excitons and excitons bounded to axial centers are detected. The bands model of bound to axial centers (Mn, Sn, Sb) excitons are proposed. The luminescence of doped Fe crystals does not fit into the bands scheme of the axial centers. It is shown that indirect transitions to the excitonic band Eₓ are unpolarized, but direct transitions are polarized.
2. Methods

The growth of CdP$_2$ single crystals from Cd and P was carried out in two stages. In the first stage, the cadmium and phosphor materials were purified and the chemical compound (CdP$_2$) was synthesized. The quartz ampoules with the mixture were deposed in a container of high-pressure ($\approx$ 40 atm.). The up to 300–500 grams of compound were synthesized in one technological process. The produced material was recrystallized in a vacuum to produce single crystals in the second stage. The largest dimensions of obtained CdP$_2$ ingots reached 15 $\times$ 15 $\times$ 30 mm. The single crystals were split off easy perpendicularly to c axis. The single crystals in the form of prisms or plates grown along the c axis and having mirror-like natural non-polished surfaces were used for measurements. Absorption spectra at 2 K, 4.2 K measured in a glass cryostat in liquid He on a DFS-24 spectrometer and at a temperature of 9–10 K on a DFS-32 spectrometer with a resolution of 5 Å/mm. Most of the experimentally measured spectra were carried out when the spectrometer slit not exceeding 0.05 mm, i.e. with a resolution of 0.5 meV. Low-temperature measurements (9–10K) were performed in the LTS-32C330 Workhorse-type Optical cryostat. Optical measuring systems are automated. Recording spectra is carried out in a digital form with subsequent processing.

3. Results & discussion

The first investigations of the edge absorption structure of tetragonal CdP$_2$ crystals were carried out in region of high absorption coefficients. Later works of edge absorption [1, 2] identified thin polarized structure attributed to indirect transitions with phonon participation. Only recently the level of technology has allowed obtaining high-quality crystals (see insert of Fig. 1). Well-correlated experimental

![Fig. 1. Absorption (K) and luminescence (PL) spectra of undopedCdP$_2$ crystals at temperatures 2 K and 9 K. Insert shows an image of CdP$_2$ crystal ingots.](https://doi.org/10.1016/j.heliyon.2018.e00866)
results of edge absorption and radiation recombination were obtained on perfect crystals.

The edge absorption of CdP₂ single crystals at 300, 77 and 2K is polarized. A flat growth of absorption coefficient in the region of weak absorption [2, 3, 4, 5] is observed at temperatures of 77 and 2 K. At temperatures of 2 and 9 K, these features are clearly visible (Fig. 1). The edge-absorption spectra for the polarizations E∥c and E⊥c in a certain wavelength intersect. This wavelength is notionally named as isotropic since in this wavelength the crystal does not distinguish the light waves polarization [4, 5]. These wavelengths are located in the region of the edge absorption beginning, where there is a change in electronic transitions (indirect, direct), responsible for edge absorption. As mentioned above, the optical properties of the tetragonal CdP₂ have been studied in many research works, where the indirect nature of the minimum energy gap and the magnitudes and the energies of the phonons participating in the transition are determined [2, 3, 4, 5, 6, 7, 8, 9].

At small values of absorption coefficient at temperatures of 2 K and 9 K, a well-recognized stepped structure in the polarization E⊥c is shown in Fig. 1. A similar structure is found in the polarization E∥c [2, 3, 4, 7]. In the region of weak absorption coefficients the features are denoted as a1, a2, a3, a4, a5 and b1, b2, b3 and b4 for the polarizations E⊥c and E∥c, respectively (see Fig. 1). The features b1-b4 and a1-a5 are due to the emission of longitudinal phonons in the case of indirect transition to the excitonic band. The threshold value of the zero-phonon indirect transition to the exciton band is indicated by $E_{exc}^L$. In accordance with the theory of allowed indirect transitions, the optical absorption is described by the expression:

$$K(E) = \frac{K_0 T}{\exp(E_p/kT)} \left[ (E - E_{gx} + E_p)^2 + \rho_i \exp \left( \frac{E_p}{kT} \right) (E - E_{gx} - E_p)^2 \right]$$  \hspace{1cm} (1)

for a pair of components due to the momentum conservation law a phonon with energy $E_p$, $K_0(T)$ is the parameter which is weakly dependent on the temperature $T$ and the effective mass state density of electrons and holes, $E_{gx}$ is an energy of indirect transition, $k$ is the Boltzmann constant, and $\rho_i$ the expression is described by: $\rho_i = (\delta E + E_i)^2 / (\delta E - E_i)^2$, where $\delta E$ - the energy difference between average effective and final states. The threshold value for a phononless indirect transition to the excitonic band $E_{exc}^L$ is 2.1533 eV. At this energy, an intensive emission band is observed in the luminescence spectra of undoped crystals, Fig. 1. On the shortwave side, we find a weaker emission band at energies of 2.1547 eV (Fig. 1) and indirect transitions with the emission of phonons. Using the value of $E_{gx} = 2.1547$ eV, found by the maximum of the luminescence and the steps on the absorption spectra curves the phonon energies are determined by help of Eq. (1) - 6.2 meV (a1, b1 - TA), 8.5 meV (a2, b2 - LA), 10.3 meV (a3 - TO), 12.7 meV (a4, b3 - TO), 16.1 meV (a5, b4 - LA) [2, 7].
In the energy region $E < E_{exc}^L$ (2.1533 eV) in the photoluminescence spectra of undoped CdP$_2$ crystals at 9 K a bands group e1-e17 is observed (see Fig. 2). This luminescence is due to the free excitons annihilation with phonons emission. The intensive maximum $E_{exc}^L$ of the luminescence at the energy of 2.1533 eV and weak $E_{exc}^{L2}$ at the energy of 2.1547 eV are due to the emission of a free exciton. These two luminescence maximums are spaced apart by a distance of 1.4 meV. The features e1-e17 (2.1482, 2.1459 eV, 2.1441 eV, 2.1417 eV, 2.1383 eV, ... 2.0856 eV) in the luminescence spectra are due to the emission of phonons upon annihilation of a free exciton. The bands $e_i$ are separated from the energy of the free exciton $E_{exc}^L = 2, 1533$ eV by the energy of the optical phonons, i.e. they are phonon replicas of free exciton. The phonon energies almost completely coincide with the known phonon energies (6.2; 8.5; 10.3; 12.7; 16.1; 26.3; 28.5 meV [1, 2, 3, 7]). At the same time, it should be noted that in CdP$_2$ the number of phonons in the center of the Brillouin zone is 72 and the phonon energy is determined in $k = 0$.

In the range $E > 2.23$ eV at temperatures of 4.2 and 2K in CdP$_2$ crystals, the polarized edge absorption is also observed (Fig. 3). In the E $\perp$ c polarization the absorption occurs at lower energies than in the $E||c$ polarization and in both polarizations it reaches $10^5$ cm$^{-1}$. Such a high absorption coefficient is observed if there are direct allowed transitions. The minimum direct energy interband gap and the interval $E_{gx}^{dir}$ at 2 K equals to 2.2727 eV ($E_{exc}$ $E||c$) and 2.3078 eV ($E||c$). The absorption edge for the $E \perp c$ polarization is shifted to a region of lower energies in comparison with the case of $E||c$ by a value of 35 meV.

As was noted above, a more steep increase of the absorption coefficient in this region is due to the presence of direct allowed transitions. For each polarization the absorption coefficient is described by the expression:

![Fig. 2. Luminescence spectra of CdP$_2$ undoped crystals at temperature of 9 K and the excitation line at 4765 Å Ar$^+$ laser.](https://doi.org/10.1016/j.heliyon.2018.e00866)
The absorption coefficient $K(E)$ can be expressed as:

$$K(E) = \frac{2e^2(2m^*)^{3/2}}{m_0^2\hbar n}|P_{CV}^e|^2 \frac{(E - E_{gx})^{1/2}}{E} = A \frac{(E - E_{gx})^{1/2}}{E}$$

where $A = \frac{2e^2(2m^*)^{3/2}}{m_0^2\hbar n}|P_{CV}^e|^2$, $m^* = m_e^* m_v^*/(m_e^* + m_v^*)$ is the reduced effective mass, $m_e^*$ and $m_v^*$ are the effective masses in the conduction and valence bands, respectively; $m_0$ is the free electron mass; $e$ is the light velocity; $n$ is the refractive index; $E_{gx}$ is the transition energy; $|P_{CV}^e|^2$ is the matrix element. The calculations of the absorption coefficient by Eq. (2) agree with the experimental data for the following parameters: $E_{gx}^{dir} = 2.2727$ eV and $A = 3.04*10^4$ eV$^{1/2}$/cm for $E_{\perp c}$ polarization (2K) and $E_{gx}^{dir} = 2.3078$ eV and $A = 2.8*10^4$ eV$^{1/2}$/cm for $E||c$ polarization (2K), Fig. 3. From the obtained results it can be seen that the absorption curves in the region of direct allowed transitions at 2 K in the polarization $E_{\perp c}$ and $E||c$ are shifted by 35.1 meV. A temperature increasing up to 4.2 K leads to a shift of the absorption edge into the long-wave region. In the $E||c$ polarization the temperature shift coefficient $DE/DT (2-4.2$ K) is 10.6 meV/K, and in the $E_{\perp c}$ polarization $DE/DT$ is equal to 3.2 meV/K. As it can be seen, the temperature coefficient of the band shift differs for different polarizations.

An analogous dependence is also observed in ZnP$_2$-D$_4^8$ [2,3], crystals. Absorption curves in the region of direct allowed transitions of ZnP$_2$-D$_4^8$ crystals for polarizations $E||c$ and $E_{\perp c}$ are shifted by 53 meV at 2 K, by 60 meV at 4.2 K and by 67 meV at 10 K. These transitions are allowed by the selection rules in different polarizations. A temperature increasing from 2 to 10 K leads to a displacement of the absorption curves in the long-wave region. Temperature coefficient of energy shifts $DE/DT$ due to direct allowed transitions when the temperature varies from 2 to 10 K is equal to 3.5 meV/K in the $E||c$ polarization and 1 meV/K in the $E_{\perp c}$ polarization. The different coefficients of the temperature shift of the absorption edge for $E||c$ and $E_{\perp c}$ are observed.
F.  

E⊥c polarizations show that the direct transitions occur from two different valence bands V1 and V2.

The obtained experimental results indicate that the band gap of CdP2 and ZnP2-D₄₈ is formed by indirect transitions to the exciton band (E_{exc}). Indirect transitions are unpolarized due to the fact that the selection rules allow indirect transitions involving phonons in both polarizations. Direct transitions occur without the participation of phonons, and they are polarized [1, 2, 7].

The obtained experimental results indicate that the band gap width is due to indirect transitions. Indirect transition occur in the V(Z)-C(Γ) points of Brillouin zone, Fig. 3. Direct transitions are polarized [2, 28, 29, 30] and localized in Z point. The following dispersion laws take place in the center of the Brillouin zone without taking into account the spin-orbit interaction: 

\[
E(k, ε) = αk_Z^2 + bk_y^2 + ε_{zz} + de_⊥ = \Delta_1,
\]

where \(k_⊥ = k_x^2 + k_y^2\), \(ε_⊥ = ε_{yy} + ε_{yz}\); and taking into account the spin orbital interaction \(E(k, ε) = Δ_1 + i(k_y + k_z) \pm (m^2k_z^2)^{1/2} \) [30]. The uppermost valence band V1 at the Z point has the symmetry Z1 (see Fig. 3). Indirect transitions from zone Z1 (V1) to zone Γ1 (C1) are allowed in both polarizations, which is observed experimentally.

CdP2-D₄₈ crystals have a structure described by a \(D_8^4(D_4^4)\) space group. 8 formula units fit into an elementary cell, i.e. 24 atoms, and the number of phonon branches in the general case is 72 [2,12, 13, 14, 15]. The presence of such number of vibrational modes makes it possible to observe the emission of free excitons with the emission of many phonons. In the luminescence spectra of CdP2-D₄₈ crystals doped with Mn narrow lines \(F_o1 - F_o5, F_1, F_2, F_3\) and the weaker lines \(f_1 - f_7\) that curve around them are found (Fig. 4). The emission bands \(f_i\) are phonon replicas of the free exciton emission line. These bands are observed simultaneously with the narrow

![Fig. 4. A - Emission spectra of CdP2-D₄₈ crystals doped with Mn measured at 9 K, and B is the extended region of the spectrum.](https://doi.org/10.1016/j.heliyon.2018.e00866)
emission lines \( F_{o1} - F_{o5} \) of excitons bound to Mn impurity center. Zero-phonons emission lines of a bound exciton \( F_{o5} \) (2.1400 eV), \( F_{o4} \) (2.1409 eV), \( F_{o3} \) (2.1411 eV), \( F_{o2} \) (2.1414 eV) and \( F_{o1} \) (2.1417 eV) are visible as narrow lines. The bands \( f_i \) are weaker and have a slightly larger half-width than the lines \( F_{o1} - F_{o5} \). These two types of lines \( f_i \) and \( F_{o1} - F_{o5} \) of radiative recombination are observed simultaneously practically in the same energy interval under the same conditions (Fig. 4).

The unit cell of CdP\(_2\)-D\(_4\)\(^8\) crystal has a large number (\( N = 24 \)) of atoms, a correspondingly a large number of vibrational modes. In CdP\(_2\)-D\(_4\)\(^8\) crystals at a temperature of 9 K, the energy distance between levels of free (2.1533 eV) and bound \( F_{o1} - 2.1417 \) eV excitons is equal to 11.6 meV, while optical phonons reach an energy value of 59.5 meV. Emission due to annihilation of the free excitons, leads to a number of lines (e1-e17) in the long-wave region from \( E_{exc}^L \) (2.1533 eV and 2.1547 eV) at energy distance, equal to the energy of the optical phonon energy.

Fig. 5 shows the energy level diagram of an exciton bound to the Mn axial center. Narrow lines \( F_{o1} - F_{o5} \) are caused by phononless lines of exciton bound to axial center [14, 15, 16]. The exciton consisting of an electron with spin of 1/2 and hole with spin of 3/2 bound to the center with axial symmetry forms two levels (\( \Sigma \) and \( \Pi \)) from the level (state) \( J = 1 \) [14-16]. State \( J = 2 \) under the action of axial field and spin-orbit interaction is split onto three levels \( \Sigma, \Pi \) and \( \Delta \). The energy distance between \( \Sigma(\Pi) - \Delta \) determines the magnitude of levels splitting due to the spin-orbital interaction (\( \Delta_{so} \)). In this model, the splitting of the spin-orbit interaction can be greater or less than the splitting value caused by the crystal field. In this \( F_{o1} - F_{o5} \) group of lines \( F_{o2} \) and \( F_{o4} \) are the most intensive lines. Consequently, the \( F_{o2} \) and \( F_{o4} \) emission lines are due to allowed transitions from zones of symmetries \( \Sigma \) and \( \Pi \) (\( J = 1 \)) into the ground state, respectively. These lines are split into a small amount

Fig. 5. Scheme of electronic transitions levels of excitons bound to the axial Mn center in the CdP\(_2\) - D\(_4\)\(^8\) crystals.
The $F_{o1}$ and $F_{o3}$ emission lines are split by 0.6 meV and are less intensive than the $F_{o2}$ and $F_{o4}$ lines. The $F_{o1}$ and $F_{o3}$ lines are due to forbidden state of a bound exciton transitions from levels $\Sigma$ and $\Pi$ (Fig. 5). These levels are split by crystalline axial field at the center where the exciton is bound. The energy interval between the $\Sigma$ - $\Pi$ levels is equal to 0.3 meV. The resulting intensity of luminescence lines and their energy position indicates that the spin-orbit splitting value is greater than the splitting value due to the crystal field (Fig. 5). In the transitions of excitons bound to the axial center model two intensive emission lines as resolved should be observed. These lines should be detected in a short-wavelength region. Luminescence lines due to transitions from forbidden by selection rules levels can appear as a line of weak intensity. The situation can change if the energy of emitted photons after the free exciton annihilation coincides with the emission energy of bound exciton forbidden transition. This can lead to resonant excitation of the bound exciton forbidden states, and can increase luminescence from the forbidden states. It is clear from experiment (Fig. 1) that in the case of free exciton annihilation ($E_{exc}^L = 21533$ eV) a series of emission bands $e1$-$e17$ was observed. As seen these bands fall into the area of emission energy from the exciton bound to Mn impurity center. The $e4$ emission band is present at energy of 2.1417 eV, due to free excitons annihilation, and coincides with $F_{o1}$ line. This coincidence in the bound excitons energy levels with the energies of phonons emitted after the free exciton annihilation removes the forbidness from optical transitions of a bound exciton, due to the selection rules.

Fig. 6 compares the luminescence spectra of CdP$_2$ crystals doped with manganese, tin and antimony. The emission spectra of crystals doped with antimony have a bright luminescence in which phononless lines of the excitons bound to an axial center and phonon replications are seen most clearly. The luminescence spectra of CdP$_2$:Sb crystals is less affected by the emission spectra associated with the multi-phonon recombination of free excitons. The axial center of Sb lies deeper in energy from the conduction band bottom.

![Image of luminescence spectra](https://doi.org/10.1016/j.heliyon.2018.e00866)

**Fig. 6.** Fragments of the most intensive luminescence lines at 9 K of undoped (curve a) and Mn (curve b), Sn (curve c) and Sb (curve d) doped CdP$_2$ - $D_3^h$ crystals.
In the shorter wavelength region from the manganese main lines a series of emission lines in CdP$_2$ crystals doped with tin is observed, which in detail are presented in Fig. 7. Intensive $A_{01}^1(2.1457\text{eV})$ and $A_{02}^2(2.1455\text{eV})$ luminescence lines are due to phononless lines of excitons, bound to axial centers of tin atoms. The emission spectra were measured on crystals doped with abovementioned impurities with the same experimental conditions as for crystals doped with Mn and Sb. Besides the mentioned $A_{01}^1$ and $A_{02}^2$, head emission lines, phonon replicas $A_1(2.1450\text{eV})$, $A_2(2.137\text{eV})$, $A_3(2.129\text{eV})$ and $B_{01}^1(2.1454\text{eV})$, $B_{02}^3(2.1453\text{eV})$, $B_{03}^5(2.1452\text{eV})$ of these lined are observed and even in a region of lower energies superimposed on bands $A_2$ and $A_3$. The level’s scheme of radiative electron transitions of the Sn axial center in CdP$_2$ crystals is shown in the insert of Fig. 7.

**Fig. 7.** Luminescence spectra of the CdP$_2$ - D$_4^8$ crystals doped with Sn measured at 9 K and excitons bound to Sn atoms levels diagram.

**Fig. 8.** Luminescence spectra of the CdP$_2$ - D$_4^8$ crystals doped with iron (Fe) measured at 9 K.
The CdP$_2$ crystals doped with iron atoms also luminescent in 2.07–2.15 eV region but typical spectra observed for crystals doped with antimony, manganese and tin are not observed (see Fig. 8). In the luminescence spectra of crystals CdP$_2$:Fe broad emission lines $F_1$, $F_2$, $F_3$, $F_4$ and $F_5$ are found out. Energies of the emission lines are shown in Fig. 8.

4. Conclusion

The $E_{\text{exc}}^L$ and $E_{\text{exc}}^{L2}$ intensive emission lines at energies of 2.1533 eV, 2.1547 eV of the indirect free exciton with phonon emission at the free indirect exciton annihilation are detected. The $E_{\text{exc}}^L$ and $E_{\text{exc}}^{L2}$ emission lines splitting by 1.4 meV is due to indirect exciton splitting because of exchange interaction. Minimum direct energy intervals in $E||c$ polarization are due to allowed transitions $\Gamma_1 \rightarrow \Gamma_1$ and in the $E \perp c$ polarization - due to the transitions $\Gamma_2 \rightarrow \Gamma_1$. The temperature coefficient of the energy intervals shift ($\Delta E/\Delta T$) when the temperature changes from 2 to 4.2 K is 10.6 meV/K in the $E||c$ polarization and 3.2 meV/K in the $E \perp c$ polarization. It has been shown that the indirect transitions into the excitonic band are unpolarized while the direct transitions are polarized.

The measured luminescence spectra of tetragonal CdP$_2$ single crystals doped by manganese, tin, antimony and iron were analyzed within the axial centers levels model. The models of excitons bound to axial centers (Mn, Sn and Sb) were offered. The bound and free excitons luminescence superposition leads to an increasing of the emission lines of forbidden transitions of bound exciton on axial center. The increasing of emission from bound excitons forbidden transitions occurs in coincidence the phonon energy with the energy of the forbidden transition of a bound exciton.

Declarations

Author contribution statement

S. I. Beril: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

V. V. Panasenko: Analyzed and interpreted the data; Wrote the paper.

D. V. Tkachenko: Performed the experiments; Wrote the paper.

I. G. Stamov: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data.

N. N. Syrbu: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.
Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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