

Oxygen-isotope effect on the density wave transitions in $\text{La}_3\text{Ni}_2\text{O}_7$

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The isotope effect is a powerful probe of electron-phonon interactions in solid-state systems, offering key insights into how atomic mass influences emergent quantum states. Here, the impact of oxygen-isotope substitution ($^{16}\text{O} \rightarrow ^{18}\text{O}$) on charge- and spin-density wave (CDW and SDW) transitions in the double-layer Ruddlesden-Popper nickelate $\text{La}_3\text{Ni}_2\text{O}_7$ is investigated. A clear isotope effect is observed in the CDW transition: the transition temperature increases upon ^{18}O substitution. In contrast, the SDW transition temperature remains unaffected within experimental uncertainty. These findings point to a strong involvement of lattice vibrations in the formation of charge order, while spin order appears to be predominantly of electronic origin. The results suggest that electron-phonon coupling, manifested through the CDW response to isotope substitution, may be relevant to the superconducting pairing mechanism in Ruddlesden-Popper nickelates.

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Introduction. The isotope effect was recognized as a fundamental tool in condensed matter physics for probing the intricate relationship between lattice vibrations and electronic properties. By replacing atoms with their isotopic counterparts, the role of phonons in various electronic and magnetic phase transitions can be assessed. This approach is particularly effective in differentiating phonon-driven phenomena from purely electronic interactions in complex materials.

Historically, the isotope effect was first observed in conventional superconductors, where it played a pivotal role in confirming the phonon-mediated pairing mechanism described by Bardeen-Cooper-Schrieffer theory [1,2]. In these materials, the superconducting transition temperature (T_c) follows the empirical relation $T_c \propto M^{-\alpha}$, where M represents the isotopic mass and the isotope exponent $\alpha \approx 0.5$ for an ideal phonon-mediated mechanism [3–17]. This dependency arises because phonons mediate Cooper pair formation, and their characteristic frequencies scale as $M^{-1/2}$. However, significant deviations from this standard isotope effect were reported in strongly correlated electron systems, where additional interactions beyond conventional electron-phonon coupling are believed to contribute [13–15,17,18]. In unconventional superconductors, the isotope effect varies significantly,

suggesting a complex interplay between lattice vibrations and superconducting carriers [19–40].

Beyond superconductivity, isotope substitution was shown to influence the charge-density wave (CDW) and spin-density wave (SDW) transition temperatures in various materials [34,41–49]. The formation of charge- and spin-density waves arises due to collective electronic instabilities, often accompanied by lattice distortions, making these phases highly sensitive to phononic contributions. Variations in the density wave transition temperatures (T_{CDW} and T_{SDW}) upon isotope substitution provide insights into the relative importance of electron-phonon interactions versus purely electronic effects in stabilizing these ordered states.

The recently discovered Ruddlesden-Popper (RP) nickelate superconductors exhibit a rich variety of electronic phases, with some of them potentially serving as precursors to superconductivity. In double- and trilayer RP nickelates, CDW and SDW orders are observed at ambient pressure, while superconductivity emerges only under high-pressure conditions [50–67]. Since superconductivity in these materials arises from states dominated by CDW and SDW correlations, investigating the isotope effect on both the precursor density wave orders and the superconducting phase offers valuable insights into the underlying pairing mechanism.

This work presents an investigation of the oxygen-isotope effect (OIE) on the density-wave transition temperatures in the double-layer RP nickelate $\text{La}_3\text{Ni}_2\text{O}_7$. Our experiments reveal that the charge-density-wave transition is sensitive to isotope substitution, resulting in a higher transition temperature T_{CDW} for the sample containing the heavier oxygen isotope. The corresponding isotope shift,

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$\Delta T_{\text{CDW}} = T_{\text{CDW}}^{18} - T_{\text{CDW}}^{16}$, is approximately 2.3 K (throughout this study, superscripts ¹⁶ and ¹⁸ are used to indicate substitution with the corresponding oxygen isotope). In contrast, the isotope shift of the spin-density-wave transition temperature is negligible within experimental uncertainty, with $\Delta T_{\text{SDW}} = T_{\text{SDW}}^{18} - T_{\text{SDW}}^{16} = -0.08(9)$ K. These findings indicate that lattice vibrations play a significant role in the formation or stabilization of the CDW state, whereas the SDW transition appears to be governed predominantly by electronic interactions. The contrasting isotope responses of the CDW and SDW transitions highlight the different microscopic origins of these ordered states in $\text{La}_3\text{Ni}_2\text{O}_7$ and provide useful constraints for theoretical models of density-wave formation in Ruddlesden-Popper nickelates.

Sample preparation and experimental techniques. The sample preparation procedure and the experimental techniques used in the present study—namely, x-ray diffraction, thermogravimetry, Raman spectroscopy, muon-spin rotation/relaxation (μSR), and resistivity—are described in the Supplemental Material [68].

A schematic of the oxygen-isotope substitution apparatus is shown in Fig. S1(a) of the Supplemental Material [68] and described in detail in Refs. [69,70]. The ^{18}O content in the $\text{La}_3\text{Ni}_2^{18}\text{O}_{7\pm\delta}$ sample was determined to be 82(2)% using mass spectrometry analysis of the $^{18}\text{O}_2$ gas line.

X-ray diffraction experiments confirmed that the crystal structures of the ^{16}O - and ^{18}O -substituted sample pair remain unchanged. The results, including the symmetry space group and lattice parameters (a , b , and c), are presented in Fig. S1(b) and Table S1 of the Supplemental Material [68]. Isotope substitution was found to have a negligible effect on all lattice parameters of $\text{La}_3\text{Ni}_2\text{O}_7$.

The oxygen content in the ^{16}O - and ^{18}O -substituted $\text{La}_3\text{Ni}_2\text{O}_{7\pm\delta}$ samples was determined using thermogravimetric analysis [Fig. S1(c) of the Supplemental Material [68]]. The samples were found to be nearly oxygen-stoichiometric, with oxygen contents ($7 \pm \delta$) determined to be 6.99(1) and 7.04(2) for $\text{La}_3\text{Ni}_2^{16}\text{O}_{7\pm\delta}$ and $\text{La}_3\text{Ni}_2^{18}\text{O}_{7\pm\delta}$, respectively. Hereafter, these samples are referred to as $\text{La}_3\text{Ni}_2^{16}\text{O}_7$ and $\text{La}_3\text{Ni}_2^{18}\text{O}_7$.

Oxygen-isotope effect on the phonon frequencies. Room-temperature Raman spectroscopy was employed to confirm both the effectiveness and spatial uniformity of ^{18}O isotope substitution, taking advantage of the technique's sensitivity to mass-dependent shifts in phonon frequencies. The measured spectra, shown in Fig. 1(a), were fitted using a sum of seven Lorentzian-shaped peaks. The extracted phonon frequencies for the ^{16}O - and ^{18}O -substituted $\text{La}_3\text{Ni}_2\text{O}_7$ samples, denoted as ν^{16} and ν^{18} , respectively, are listed in Table S2 of the Supplemental Material [68].

The coefficient representing the involvement of oxygen in each phonon mode, f_{O} , was calculated following Refs. [71–73]:

$$\nu^{18}(x)/\nu^{16} = [\sqrt{M^{16}/M^{18}(x)}]^{f_{\text{O}}}, \quad (1)$$

where x is the ^{18}O content in the isotope-substituted sample (82% in this study), $M^{16} = 16$ is the atomic mass of ^{16}O , and $M^{18}(x) = (1 - x) \times M^{16} + x \times M^{18}$ is the average oxygen mass in the ^{18}O -substituted sample. This relationship

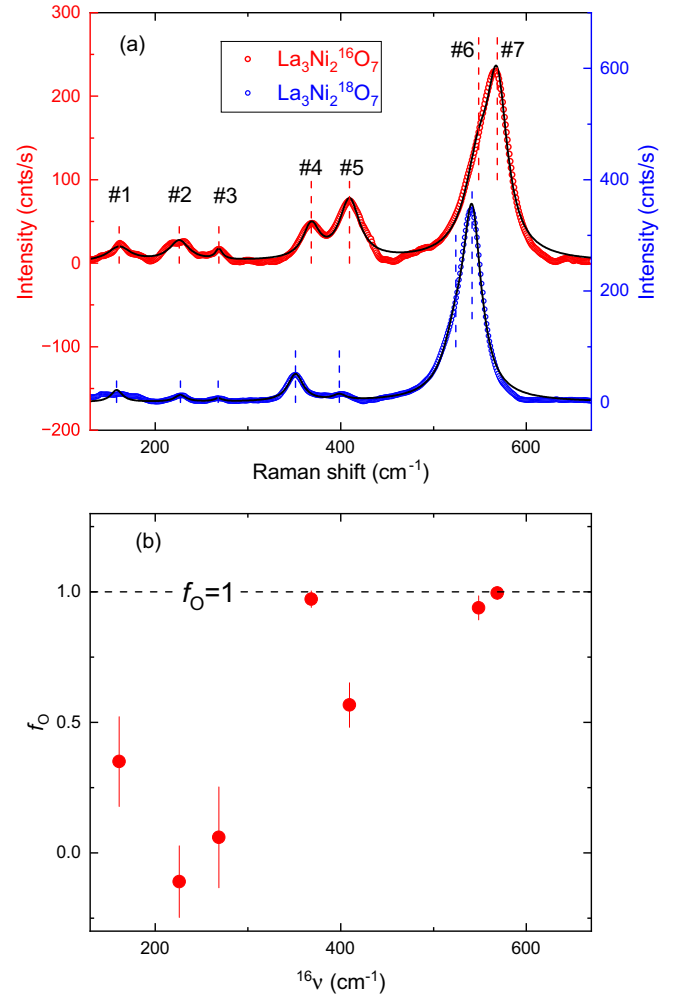


FIG. 1. (a) Room-temperature Raman spectra of oxygen-isotope-substituted $\text{La}_3\text{Ni}_2\text{O}_7$ samples. Solid lines represent fits using seven Lorentzian functions. Dashed lines indicate the peak positions. (b) The oxygen participation parameter f_{O} as a function of the Raman wavenumber $^{16}\nu$.

assumes a harmonic oscillator model, where mode softening upon isotope substitution reflects the oxygen contribution to each vibration.

Based on the extracted f_{O} values shown in Fig. 1(b) and listed in Table S2 of the Supplemental Material [68], the following conclusions can be drawn:

(1) Three phonon modes observed at ν^{16} (ν^{18}) = 368 (351), 549 (524), and 568 (542) cm^{-1} exhibit f_{O} values close to 1.0, indicating that these modes are dominated by oxygen vibrations.

(2) The remaining modes at 161 (159), 226 (227), 269 (268), and 409 (398) cm^{-1} show significantly smaller—or even negligible, within experimental uncertainty—values of f_{O} , implying that oxygen is only weakly involved or not involved at all in these lattice vibrations. These modes are likely associated with heavier ions, such as Ni and La.

Oxygen-isotope effect on the CDW transition. The oxygen-isotope effect on the CDW transition temperature was investigated via resistivity measurements. Figure 2(a) shows the temperature dependence of resistivity normalized to its

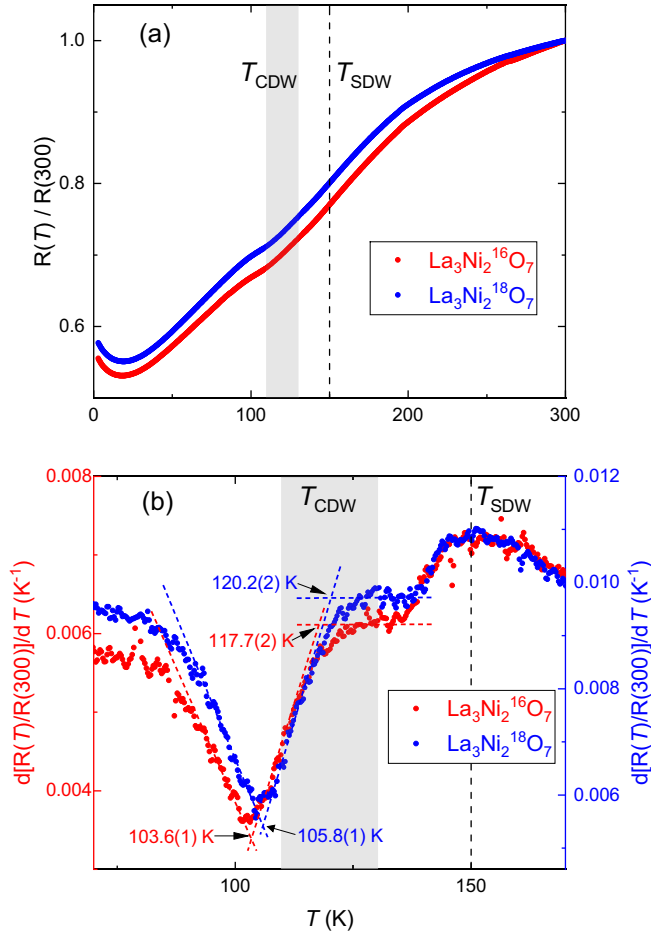


FIG. 2. (a) Raw resistivity curves of the $^{16}\text{O}/^{18}\text{O}$ -substituted $\text{La}_3\text{Ni}_2\text{O}_7$ samples. (b) Extended view of the first derivatives of the resistivity data in the vicinity of the CDW transitions. The red and blue numbers correspond to the CDW transition temperatures of $\text{La}_3\text{Ni}_2^{16}\text{O}_7$ and $\text{La}_3\text{Ni}_2^{18}\text{O}_7$, determined using different criteria (see text for details).

300 K value, $R(T)/R(300)$. The raw resistivity curves exhibit weak anomalies near $T \sim 120$ K. A more pronounced structure emerges in the first derivative of the resistivity data, shown in Fig. 2(b).

The anomalies observed in both the $R(T)/R(300)$ and $d[R(T)/R(300)]/dT$ curves may be attributed to charge- and spin-density wave transitions [dashed lines and gray stripe in Figs. 2(a) and 2(b)], as previously reported in the literature. The SDW transition at $T_{\text{SDW}} \simeq 150$ K was identified in earlier μSR , NMR, nuclear quadrupole resonance (NQR), and resonant x-ray studies [67,74–81]. Although the CDW in $\text{La}_3\text{Ni}_2\text{O}_7$ has not yet been directly confirmed by scattering techniques, anomalies in resistivity and specific heat at $110 \text{ K} \lesssim T \lesssim 130 \text{ K}$ have been associated with the onset of CDW order [53,60,82–85]. Accordingly, an average CDW transition temperature of $T_{\text{CDW}} = 120$ K is adopted for $\text{La}_3\text{Ni}_2\text{O}_7$. It is also worth noting that recent optical pump-probe spectroscopy [86], x-ray absorption near-edge spectroscopy [87], and NQR [81] measurements have provided further indications of CDW order below 150 K in $\text{La}_3\text{Ni}_2\text{O}_7$.

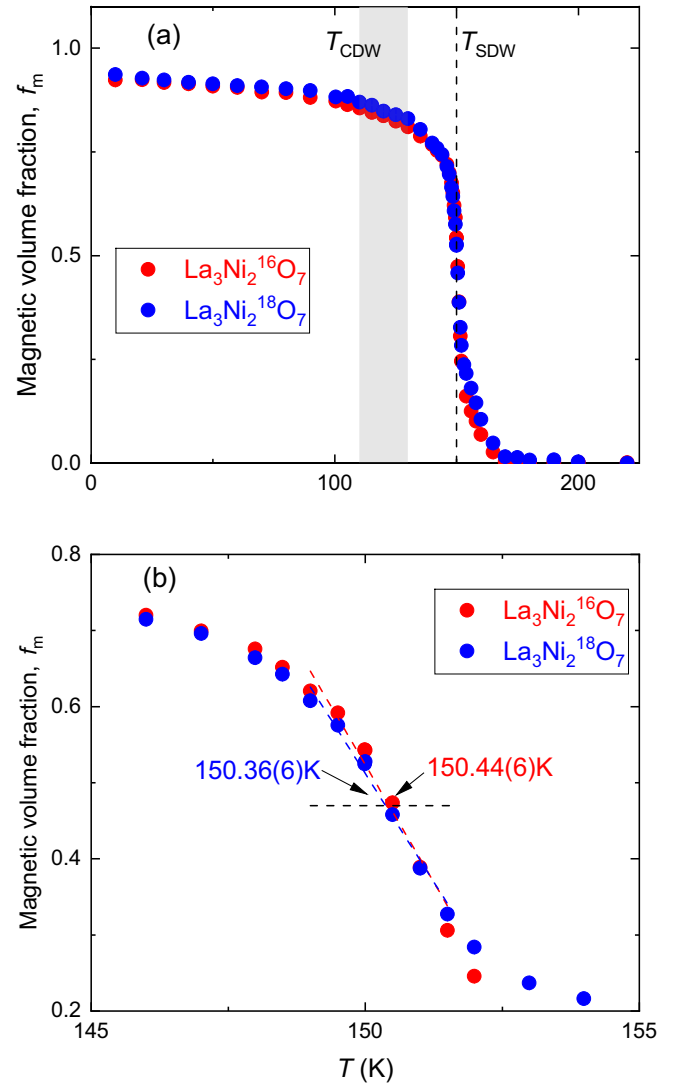


FIG. 3. (a) Temperature dependence of the magnetic volume fraction f_m for ^{16}O - and ^{18}O -substituted $\text{La}_3\text{Ni}_2\text{O}_7$ and $\text{La}_4\text{Ni}_3\text{O}_{10}$ samples. (b) Extended view of the $f_m(T)$ curves in the vicinity of the SDW transitions. The vertical dashed lines and gray stripe indicate the CDW and SDW transition temperatures (T_{CDW} and T_{SDW}), as reported in the literature [53,60,67,74,75,82–85,88,89].

The isotope-induced shift of the CDW transition temperature was estimated using two approaches: (1) by determining the crossing point between linear fits to the derivative curves within the “slope” and “plateau” regions, which yielded $T_{\text{CDW}}^{16} = 117.7(2)$ K and $T_{\text{CDW}}^{18} = 120.2(2)$ K; and (2) by identifying the crossing points of linear fits around the local minimum, giving $T_{\text{CDW}}^{16} = 103.6(1)$ K and $T_{\text{CDW}}^{18} = 105.8(1)$ K. Both approaches yield a consistent isotope shift of $\Delta T_{\text{CDW}} = T_{\text{CDW}}^{18} - T_{\text{CDW}}^{16} \simeq 2.3(2)$ K.

It should also be noted that the derivative curves exhibit a broad hump around 150 K, which may be associated with the SDW transition, as reported in Refs. [67,74–81]. However, this feature is too broad to allow for a reliable determination of the OIE on T_{SDW} from resistivity data alone. Instead, the isotope effect on T_{SDW} is evaluated based on μSR measurements, presented below.

TABLE I. Results of oxygen-isotope effect measurements of $\text{La}_3\text{Ni}_2\text{O}_7$. $\Delta T_{\text{tr}} = T_{\text{tr}}^{18} - T_{\text{tr}}^{16}$ ($T_{\text{tr}} = T_{\text{SDW}}$ or T_{CDW}). The oxygen-isotope exponent α is calculated by using equation $\alpha = -d \ln T_{\text{tr}} / d \ln M$ and is corrected for incomplete isotope exchange $\simeq 82\%$.

	$\text{La}_3\text{Ni}_2^{16}\text{O}_7$	$\text{La}_3\text{Ni}_2^{18}\text{O}_7$	ΔT_{tr}	α	Technique
T_{SDW}	150.44(6) K	150.36(6) K	−0.08(9) K	0.005(6)	WTF- μSR
T_{CDW}	103.6(1) K ^a	105.8(1) K ^a	2.2(1) K	−0.21(1)	Resistivity
	117.7(2) K ^b	120.2(2) K ^b	2.5(4) K	−0.21(3)	Resistivity

^aFrom local minima at $d[R(T)/R(300)]/dT$ [Fig. 2(b)].

^bFrom linear slopes of $d[R(T)/R(300)]/dT$ [Fig. 2(b)].

Oxygen-isotope effect on the SDW transition. The OIE on the SDW transition temperature was investigated using μSR . Experiments were conducted in the weak-transverse field (WTF) mode with a magnetic field $B_{\text{WTF}} = 5$ mT applied perpendicular to the initial muon-spin polarization. The data analysis procedure, along with several WTF data sets, is provided in the Supplemental Material [68].

The temperature evolution of the magnetic volume fractions obtained from the fits to WTF- μSR data is presented in Fig. 3(a). The dashed lines and gray stripe indicate the CDW and SDW transition temperatures, as reported in the literature [53,60,67,74,75,82–85,88,89]. The WTF data clearly capture the SDW transition. It should be noted that the maximum value of the magnetic volume fraction does not reach 100%, which is attributed to approximately 5%–7% of the muons missing the sample and stopping in the sample holder and/or the cryostat walls.

Figure 3(b) shows an expanded view of the $f_m(T)$ curves in the vicinity of T_{SDW} . The SDW transition temperatures were estimated from the intersection of linearly extrapolated $f_m(T)$ curves near T_{SDW} with the reference line $f_m(T) = 0.5 \times f_m(10 \text{ K})$ [dashed lines in Fig. 3(b)]. The fits yield $T_{\text{SDW}}^{16} = 150.44(6) \text{ K}$ and $T_{\text{SDW}}^{18} = 150.36(6) \text{ K}$, resulting in an isotope shift of $\Delta T_{\text{SDW}} = T_{\text{SDW}}^{18} - T_{\text{SDW}}^{16} = -0.08(9) \text{ K}$.

Discussion and conclusions. Table I summarizes the results of the oxygen-isotope effect measurements on the charge- and spin-density wave transition temperatures in the double-layer Ruddlesden-Popper nickelate $\text{La}_3\text{Ni}_2\text{O}_7$. The OIE results reported in the present study for $\text{La}_3\text{Ni}_2\text{O}_7$, together with those in Ref. [89] for the trilayer RP nickelate $\text{La}_4\text{Ni}_3\text{O}_{10}$, can be directly compared to isotope effects investigated in cuprate high-temperature superconductors (HTSs), where such studies are among the most extensive within correlated electron systems.

In undoped cuprates, the isotope effect on the Néel temperature T_N is found to be negligible [34,44,46], consistent with the observations for $\text{La}_3\text{Ni}_2\text{O}_7$, where the SDW transition—preceding the CDW order—shows no measurable isotope shift. This suggests that magnetism, in the absence of strong charge fluctuations, is primarily governed by electronic interactions and is only weakly influenced by phonons.

In contrast, underdoped cuprates, where charge and spin orders coexist and are strongly correlated, show a different behavior. In stripe-ordered cuprates such as $\text{La}_{2-x}\text{Ba}_x\text{CuO}_4$, the emergence of static charge order is accompanied by a spin-stripe phase that strongly suppresses superconductivity [90–97]. Notably, isotope-effect studies in these systems show that both the CDW and SDW transition temperatures increase upon ^{18}O substitution, with shifts of the same sign and comparable magnitude [41,48].

This behavior is strikingly similar to the findings in $\text{La}_4\text{Ni}_3\text{O}_{10}$ [89], where CDW and SDW orders are intertwined, share the same transition temperature ($T_{\text{CDW}} \simeq T_{\text{SDW}}$), and exhibit identical isotope shifts ($\Delta T_{\text{CDW}} = \Delta T_{\text{SDW}}$). These parallels suggest a shared underlying mechanism, likely involving strong electron-phonon coupling that affects both spin and charge degrees of freedom when the two orders are strongly entangled.

Despite these parallels, there are notable differences between cuprates and nickelates. In cuprate HTSs, increasing hole doping suppresses the SDW order, eventually leading to the emergence of superconductivity [98–100], while in $\text{La}_3\text{Ni}_2\text{O}_7$, applying pressure does not immediately suppress SDW order [67]. Moreover, in $\text{La}_4\text{Ni}_3\text{O}_{10}$, the SDW order may not appear to be the primary instability, but rather a consequence of CDW formation [89].

In kagome materials, such as CsV_3Sb_5 , KV_3Sb_5 , and RbV_3Sb_5 , suppression of CDW order through pressure or chemical tuning was found to enhance superconductivity [101–105]. A similar trend may be expected in nickelates, where superconductivity emerges under high pressure and is associated with the suppression of charge order. This raises the possibility that, as in kagome superconductors, CDW order plays the dominant role in competing with superconductivity in RP nickelates, rather than SDW order.

To conclude, our findings reveal that the CDW and SDW transitions in $\text{La}_3\text{Ni}_2\text{O}_7$ exhibit markedly different sensitivities to oxygen-isotope substitution. The clear isotope dependence of the CDW transition highlights a strong coupling between charge order and lattice vibrations, while the SDW transition shows no measurable isotope shift, indicating a predominantly electronic origin. This distinct isotope response offers valuable insight into the role of electron-phonon interactions and electronic correlations in layered nickelates, and demonstrates that isotope substitution is an effective tool for disentangling competing ordered states in Ruddlesden-Popper systems.

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Data availability. The data are available from the authors upon reasonable request.

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