

Corrected infrared Sellmeier coefficients for gallium selenideK. R. Allakhverdiev^{a)}*Marmara Research Centre of Turkish Scientific and Technical Research Council (TUBITAK),
Materials Institute, P.K. 21, 41470 Gebze, Kocaeli, Turkey and Institute of Physics,
Azerbaijan National Academy of Sciences, Huseyn Javid Avenue 33, 370143 Baku, Azerbaijan Republic*

T. Baykara

*Marmara Research Centre of Turkish Scientific and Technical Research Council (TUBITAK),
Materials Institute, P.K. 21, 41470 Gebze, Kocaeli, Turkey*

A. Kulibekov Gulubayov

Physics Department, Mugla University, 48000 Mugla, Turkey

A. A. Kaya

*Marmara Research Centre of Turkish Scientific and Technical Research Council (TUBITAK),
Materials Institute, P.K. 21, 41470 Gebze, Kocaeli, Turkey*

J. Goldstein and N. Fernelius

*Materials and Manufacturing Directorate, Air Force Research Laboratory, AFRL/MLPSO,
Wright-Patterson Air-Force Base, Ohio 45433-7707*

S. Hanna

Institute of Physics, University of Bayreuth, D-95440 Bayreuth, Germany

Z. Salaeva

*Marmara Research Centre of Turkish Scientific and Technical Research Council (TUBITAK),
Materials Institute, P.K. 21, 41470 Gebze, Kocaeli, Turkey*

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We have measured the room-temperature refractive indices of GaSe throughout the 0.7–1.4 and 2.4–5 μm ranges using the minimum-deviation method of light through a prism with polarization either parallel (extraordinary refractive index n_e) or perpendicular to the crystal optical c axis (ordinary refractive index n_o). The birefringence (Δn) at room temperature has been measured directly using polarized light interference fringes obtained in the transmittance from 1.73 to 4.97 μm (three samples with thicknesses of 1108 ± 1 , 2480 ± 1 , and 2660 ± 1 μm) and in the range from 11.85–16.37 μm (sample thickness of 6000 ± 1 μm). The n_o , n_e , and Δn values determined from the positions of fringe maxima were compared to those obtained from prism measurements in the mid-IR and with previously published results which were mainly obtained by indirect methods. It was found that the prism method and interference fringe method resulted in values of n_o , n_e , and Δn accurate to ± 0.003 and ± 0.006 , respectively. © 2005 American Institute of Physics. [DOI: 10.1063/1.2128694]

I. INTRODUCTION

Gallium selenide (GaSe) is one of the most promising nonlinear-optical materials for IR frequency conversion.^{1–14} Wavelength dependence of the refractive indices and birefringence are some of the most important material parameters for calculating the phase-matching angles required for efficient optical-frequency conversion. Measurements of the nonlinear-optical properties of GaSe and its ordinary and extraordinary refractive indices were reported.³ The refractive indices given in Ref. 3 were based on linear- and nonlinear-optical measurements of the second-harmonic generation wave phase-matching (CO_2 , $\lambda = 10.6$ μm ; CO , 5.3 μm ; and Dy^{2+} : CaF_2 , 2.36 μm) lasers. Later these authors modified the dispersion equations to fit their data obtained from frequency up-conversion experiments in the mid-IR (Ref. 4) [sum-frequency generations of CO_2 ($\lambda = 10.6$ μm), CO

(5.3 μm), and Nd^{3+} :YAG (yttrium aluminum garnet) (1.06 μm)] laser radiation. Dispersion relationships were derived for the whole transparency range of GaSe, taking into consideration numerous linear-^{15–18} and nonlinear-optical measurements performed by other authors^{3–6,19–23} as well as Vodopyanov and Kulevskii.²⁴ The relationships obtained are in agreement with the phase-matching curves for the second-harmonic, sum-frequency, difference-frequency, and optical parametric generations.²⁴ However, neither direct measurements of the refractive indices nor measurements of birefringence of GaSe in the mid-IR range had been made.

In this paper we present the results of our study of the refractive-index dispersion of GaSe using the minimum-deviation method of light through a prism with polarization either parallel (extraordinary refractive index n_e) or perpendicular to the crystal optical c axis (ordinary refractive index n_o) throughout the 0.7–1.4 and 2.4–5 μm ranges and the birefringence (Δn) study measured directly from polarized light interference fringes obtained in the transmission spectra

^{a)}Electronic mail: kerim.allahverdi@mam.gov.tr

over the mid-IR range (1.73–4.97 and 11.85–16.37 μm). The Δn values determined from the positions of the fringe maxima were compared to that obtained from the prism measurements and with existing data. It was found that the prism method and interference fringe method resulted in the values of n_o , n_e , and Δn accurate to ± 0.003 and ± 0.006 , respectively.

The birefringence for GaSe has been measured directly from polarized light interference fringes obtained in the mid-IR transmission spectra from the samples with thicknesses of 1108 ± 1 , 2480 ± 1 , 2660 ± 1 , and 6000 ± 1 μm . All samples contain the optical c axis in the plane of the parallel polished faces (GaSe is uniaxial, point group is $6m2$).

II. EXPERIMENTS

The p -type GaSe single crystals used in this study were obtained by the Bridgman method using evacuated quartz ampoules (10^{-5} Torr). From the boules, slabs of different sizes were easily cleaved with a razor blade. To prepare plane parallel plates or prisms for the transmission or the refractive-index measurements, we used a simple but effective technology with which one may achieve optical-quality cutting and polishing of soft GaSe at the desired angle to the optical c axis.²⁵ With this method we have reproducibly obtained mirrorlike surfaces on faces of area greater than 1 cm^2 , parallel to the optical c axis. As a result of this technology, we have been able to measure and compare the transmission spectra of both the o (ordinary) and e (extraordinary) rays.

Thickness could be measured to approximately $\pm 1 \mu\text{m}$ by using a calibrated metrology device MAHR-828 CIM (maximum accuracy of thickness measurements is about $0.3 \mu\text{m}$). Typically thickness varied by up to $\pm 6 \mu\text{m}$ across the sample. For Δn measurements the samples were mounted between parallel polarizers set at 45° to the optical c axis of the crystal and transmittance was measured as a function of wavelength. A Digilab FTS-20E and a modified BRUKER IFS 55 Fourier-transform spectrometers with a spectral resolution of 2 cm^{-1} were used for the 11.85–16.37 and 1.73–4.97 μm range's, respectively. The positions of the fringe maxima were determined by the technique described in Ref. 26. This method of determining the fringe peak positions resulted in peak wavelength accuracies varying from ± 0.002 for the whole measured spectral ranges. We derived the fringe order number from the results of the prism measurements, as it is described in the Sec. III B. This procedure introduces an additional source of error which is not higher than ± 0.002 .

Three prisms with the apex angles of 30° were prepared by the method described in Ref. 25. Useful measurements could be made at least for the three points on each prism. The values of the refractive indices obtained at different locations on the prism were nearly the same. The differences were in the range of 0.003 – 0.006 for both refractive indices. Free-carrier concentration can be one of the factors that influence the index of refraction. Dispersion for the prisms was measured by use of the minimum-deviation technique.²⁷ The z axis was oriented normal to the triangular base of the

TABLE I. Room-temperature refractive indices and birefringence (Δn) of GaSe vs wavelength as determined by the minimum-deviation method using a prism with the apex angle of 30° . n_o and n_e is for the ordinary and extraordinary refractive indices, respectively. $n_{o,e}$ accuracy is ± 0.003 .

λ (μm)	n_o	n_e	Δn
0.7	2.899	2.583	0.316
0.75	2.867	2.545	0.322
0.8	2.846	2.521	0.326
0.85	2.832	2.501	0.331
0.9	2.817	2.490	0.327
0.95	2.805	2.479	0.327
1	2.795	2.467	0.328
1.05	2.787	2.462	0.325
1.1	2.781	2.455	0.326
1.15	2.775	2.450	0.325
1.2	2.770	2.446	0.324
1.25	2.766	2.442	0.324
1.3	2.763	2.439	0.324
1.35	2.760	2.437	0.323
1.4	2.757	2.433	0.324
2.4	2.730	2.410	0.320
2.6	2.728	2.409	0.319
2.8	2.727	2.407	0.319
3.0	2.725	2.406	0.319
3.2	2.724	2.405	0.319
3.4	2.723	2.405	0.318
3.6	2.722	2.404	0.318
3.8	2.721	2.403	0.318
4.0	2.719	2.402	0.317
4.4	2.718	2.402	0.316
4.6	2.717	2.402	0.315
4.8	2.716	2.401	0.315
5.0	2.716	2.401	0.315

prism. The minimum-deviation method involved rotating the prism until the transmitted light experienced a minimum angle of refraction relative to the unrefracted beam. Refractive-index measurements were found to be within $\pm 3 \times 10^{-3}$. Index values were calculated from the minimum-deviation measurements by use of the following equation:²⁸

$$n = \sin[(A + D)/2] / \sin(A/2), \quad (1)$$

where A is the apex angle of the prism. Since the prisms were cut from uniaxial crystals with the prism edges parallel to the optical axis, the ordinary or extraordinary indices were obtained with the electric-field vector of the incident light perpendicular or parallel to the optical axis, respectively. A half-wave plate was used to rotate the incident polarization as desired.

All data were obtained at room temperature $[(20 \pm 1)^\circ\text{C}]$.

III. RESULTS AND DISCUSSIONS

A. Prism measurements

The results of the prism measurements are summarized in Table I. Figure 1 shows our refractive-index data as a function of wavelength, along with the calculated curves obtained by a least-square fit of the Sellmeier equations to the

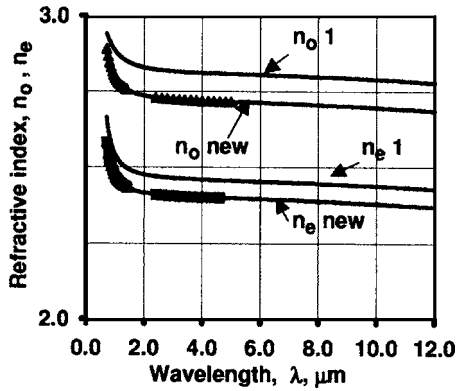


FIG. 1. Experimental values of ordinary n_o and extraordinary n_e refractive indices of GaSe as determined from prism [apex angle 30°, least-deviation method (Ref. 28)] measurements at room temperature. ▲ and ■ points are for n_o and n_e , respectively. Solid lines are dispersion relations given by formula (2) using respective values for the coefficients. n_o 1 and n_e 1 refer to dispersions calculated by using the coefficients reported in Ref. 3. n_o new and n_e new are obtained by using the coefficients.

measured indices. It was found that the dispersion of the refractive indices in the transparency range is described with sufficient accuracy by the formulas:^{3,4}

$$n_o^2 = A/\lambda^4 + B/\lambda^2 + C + D\lambda^2 + E\lambda^4,$$

$$n_e^2 = K + L/(\lambda^2 + M) + H\lambda^2, \tag{2}$$

where $A = -0.05478$ (-0.05466 and -0.06), $B = 0.38529$ (0.4865 and 0.526), $C = 7.38994$ (7.8902 and 8.038), $D = -0.00110$ (-0.000824 and -0.00110), $E = -0.00000937$ (-0.00000273 and -0.0000027), $K = 5.7741$ (6.0476 and 6.06), $L = 0.26288$ (0.3423 and 0.5754), $M = -0.21457$ (-0.16491 and 0.0453), $H = -0.0011$ (-0.001042 and 0.00104), and λ is given in micrometers. First values of coefficients are those obtained by fitting our experimental data. The values shown in parentheses are taken from that presented in Refs. 3 and 4, respectively.

We also compared our results with the semiphenomenological $n_o(\lambda)$ equation taking into account the electronic transition and the lattice mode vibrations input to $n_o(\lambda)$ and given by the formula in Ref. 24:

$$n_o^2 = A + B/\lambda^2 + C/\lambda^4 + D/\lambda^6 + E\lambda^2/(\lambda^2 - F), \tag{3}$$

where $A = 7.38539$ (7.443), $B = 0.42611$ (0.4050), $C = -0.00797$ (0.0186), $D = 0.02291$ (0.0061), $E = 0.88558$ (3.1485), $F = 873.471$ (2194), and λ is given in micrometers. The values in parentheses are those given in Ref. 24.

Comparison was also made using the expression for n_e :²⁴

$$n_e^2 = A + B/\lambda^2 + C/\lambda^4 + D/\lambda^6 + E\lambda^2/(\lambda^2 - F), \tag{4}$$

where $A = 5.77169$ (5.760), $B = 0.31285$ (0.3879), $C = -0.03784$ (-0.2288), $D = 0.0573$ (0.1223), $E = 2216.135$ (1.855), and $F = 2049967$ (1780). The results are shown in Fig. 2. It is seen that for n_e the results are in fairly good agreement.

In Ref. 15 the authors measured the refractive indices of GaSe in the spectral range of 400–1000 nm by two techniques: by the prism method and by the determination of the

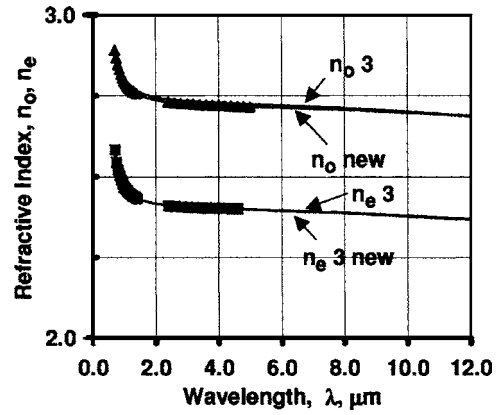


FIG. 2. Same as Fig. 1 only that the solid lines are dispersion relations given by formulas (3) (n_o) and (4) (n_e) (Ref. 24). n_o 3 and n_e 3 refer to dispersions reported in Ref. 24. n_o new and n_e new are obtained by using our experimental data fitted by formulas (3) and (4) and the coefficients.

wavelength and interference order of the fringes resulting from multiple internal reflections of white light transmitted through the thin parallel-sided plates (freshly cleaved surface, which is always perpendicular to the optical c axis). In the minimum-deviation technique the authors used three GaSe prisms with apex angles of 12°–17° and different lines of an Ar⁺ or cw dye laser. Seven and five experimental points were presented for GaSe in the spectral ranges of ~630–665 and 640–665 nm for n_o and n_e , respectively. Combining these data with the results of the fringe measurements, the authors obtained averaged dispersions by the least-squares fitting by the polynomial¹⁵

$$n_{o,e} = A/\lambda^6 + B/\lambda^4 + C/\lambda^2 + D \tag{5}$$

for the spectral ranges of 0.630–1.022 μm (for n_o) and of 0.640–0.955 μm (for n_e). They reported the coefficients of the fitted equation (5): $A = 0.003772$, $B = -0.003211$, $C = 0.05151$, and $D = 2.746$ and $A = 0.04746$, $B = -0.2006$, $C = 0.3873$, and $D = 2.210$ for n_o and n_e , respectively. The results of comparison are shown in Fig. 3. Coefficients obtained using our experimental results for the fitting equation (5) are $A = 0.05268$, $B = -0.15415$, $C = 0.20021$, and $D = 2.6996$ and $A = 0.006631$, $B = -0.17919$, $C = 0.20098$, and

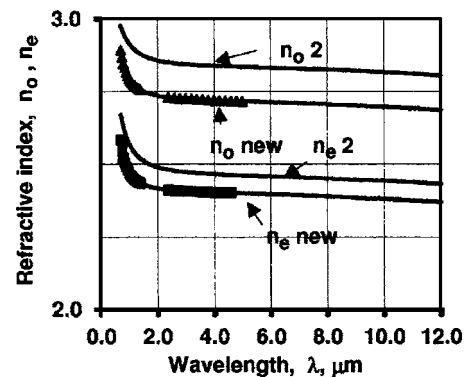


FIG. 3. The indices of refraction of GaSe at room temperature. Notations are the same as in Fig. 1. n_o 4 and n_e 4 denote the dispersions obtained by using formula (5) and the coefficients given in Ref. 15. n_o new and n_e new are obtained using our experimental data fitted by the polynomial (5) and the coefficients.

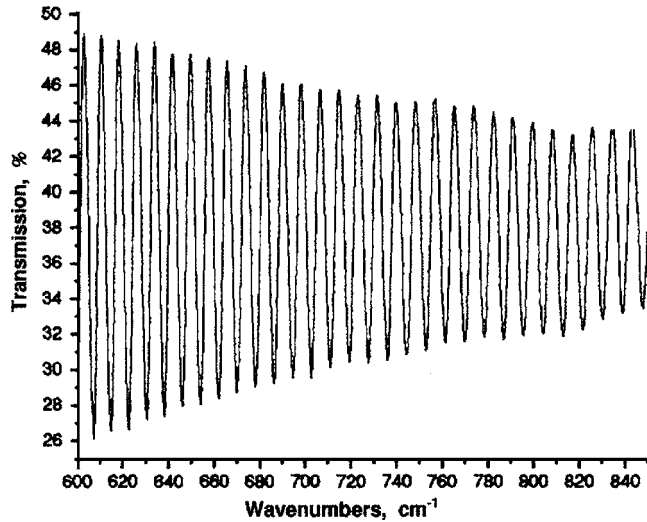


FIG. 4. Room-temperature birefringence fringes of $6000\pm 1\text{-}\mu\text{m}$ -thick (thickness varied by up to $\pm 6\ \mu\text{m}$ across the sample) GaSe sample in the mid-IR spectral region with the surface containing the optical c axis. The sample was mounted between parallel polarizers set up at 45° to the optical c axis. The birefringence was calculated from the fringes using formula (6).

$D=2.383\ 37$ for n_o and n_e , respectively. The error bars indicated by the spread of values for the refractive indices (Fig. 2, Ref. 15) were estimated to be about ± 0.028 in the spectral range of $0.630\text{--}0.665\ \mu\text{m}$. The calculated values using polynomial (5) and the coefficients given in Ref. 15 for n_o at 0.7 , 0.85 , and $1\ \mu\text{m}$ are 2.870 (2.899), 2.821 (2.832), and 2.798 (2.795), respectively (the values in parentheses show our experimental results). For n_e these values are 2.568 (2.583), 2.488 (2.501), and 2.442 (2.467), respectively. The values for birefringence at the above given wavelengths are 0.302 (0.316), 0.333 (0.331), and 0.356 (0.328), respectively. One may surmise that this discrepancy is possibly due to several factors and among them are a different quality of the prisms and different concentrations of the free carriers in the samples. On the other hand, the accuracy of our measurements is more than one order of magnitude higher than those given in Ref. 15.

As can be seen the experimental results obtained in the present work are in a reasonable agreement with those published in literature.^{3,4,15,24}

B. Birefringence interference spectrum

Using the experimental setup described above, we obtained the birefringence interference spectrum for GaSe samples with thicknesses of 1108 ± 1 , 2480 ± 1 , 2660 ± 1 , and $6000\pm 1\ \mu\text{m}$. Room-temperature birefringence fringes of 6000 ± 1 - and $1108\pm 1\text{-}\mu\text{m}$ -thick samples are shown in Figs. 4 and 5, respectively. In the absence of optical activity, the positions of the fringe maxima should satisfy the condition²⁶

$$\Delta n = k\lambda/d, \quad (6)$$

where Δn is the birefringence ($n_e - n_o$), λ is the wavelength, d is the sample thickness, and k is an integer (for the fringe maxima, k is an integer and for the fringe minima k is an integer plus one-half).

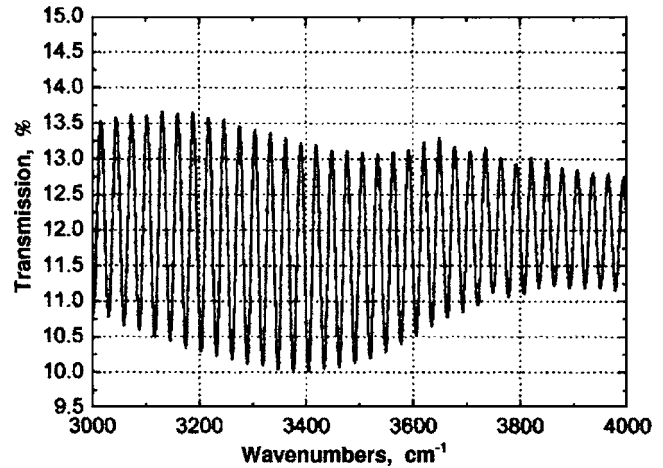


FIG. 5. Same as in Fig. 4 for the sample with thickness of $1108\pm 1\ \mu\text{m}$.

Crystals of point group $62m$, which includes GaSe, are optically active and this may influence the fringe maxima. Generally, the effect of optical activity is very small compared to the birefringence and we neglect it in our case. Using Eq. (6) to determine the birefringence can result in highly accurate values provided the fringe order k is independently known from other measurements. For this reason we used one-fringe (one peak) method to determine the integer k . The accuracy of Δn determined by the single-fringe method is limited primarily by the accuracy with which the fringe position and sample thickness can be measured. The positions of the fringe maxima were determined by finding the center point of each fringe (on an expanded transmittance versus λ plot) near the peak, at half of the maximum intensity. This typically resulted in peak wavelength accuracies of ± 0.0005 for the whole measured wavelength ranges. In our case, the limiting factor is the variation in sample thickness which can be as much as $\pm 6\ \mu\text{m}$. The temperature variation of the birefringence was also considered as a potential source of error but it was determined to be a much smaller factor than the sample thickness variation. This conclusion was based on the values of dn/dT estimated from the temperature dependence of the ordinary refractive index of GaSe measured in the temperature range of $20\text{--}100\ ^\circ\text{C}$.²⁹ Using the formula (2) given in Ref. 29 we estimated the value $dn/dT = 2.82 \times 10^{-4}$ in the temperature range of $20\text{--}40\ ^\circ\text{C}$. As a result, we believe our accuracy of Δn is ± 0.006 or better. Using the value of $n_o = 2.687 \pm 0.003$ and $n_e = 2.372 \pm 0.003$ ($\Delta n = 0.315$) determined from the Sellmeier formula constructed from our experimental data measurements (Sec. III A), we calculated the number of k for the fringe maxima at $844\ \text{cm}^{-1}$ ($11.848\ \mu\text{m}$). We obtained that the number of peaks at $11.848\ \mu\text{m}$ corresponds to the integer $k = 158$, for the sample with thickness $6000\pm 1\ \mu\text{m}$. Once the fringe 158 is identified, all the other fringes in the spectra can be assigned easily. Using this procedure for other fringes of Figs. 4 and 5 we calculated the birefringence values versus wavelength at all selected fringes peaks for these and other samples. These data are shown in Table II. Note that a good agreement is observed for the birefringence values determined by two different methods.

TABLE II. GaSe birefringence values vs wavelength as determined by the single-fringe method ($\Delta n = k\lambda/d$) at some selected fringe peaks for the $6000 \pm 1\text{-}\mu\text{m}$ - (11.85–16.37 μm), $1108 \pm 1\text{-}\mu\text{m}$ - (1.73–3.32 μm), and $2480 \pm 1\text{-}\mu\text{m}$ - (3.35–4.97 μm) thick samples (in the parentheses the spectral range used to determine the birefringence is shown). The Δn accuracy is ± 0.006 . Subscripts “fr” and “pr” denote the results obtained from fringe and prism measurements, respectively. Numbers in the brackets for Δn_{pr} show the wavelength at which the prism measurements were performed.

λ (μm)	ω (cm^{-1})	k	Δn_{fr}	Δn_{pr}
1.73	5763.9	206	0.322	
1.75	5710.2	204	0.322	
1.78	5626.0	201	0.323	
1.81	5515.0	197	0.322	
1.87	5340.3	191	0.322	
1.93	5170.0	184	0.320	
1.99	5023.0	179	0.321	
2.01	4966.4	175	0.318	
2.06	4856.3	171	0.318	
2.11	4740.9	167	0.318	
2.17	4598.8	162	0.317	
2.25	4449.2	157	0.319	
2.32	4308.0	152	0.318	
2.38	4194.8	148	0.318	0.320 (2.4 μm)
2.45	4079.3	144	0.318	
2.49	4021.6	142	0.319	
2.52	3964.5	139	0.316	
2.58	3883.9	136	0.317	0.320 (2.6 μm)
2.65	3768.2	132	0.316	
2.78	3594.6	126	0.316	0.319 (2.8 μm)
2.90	3450.0	121	0.317	
3.03	3305.4	116	0.317	0.319 (3 μm)
3.11	3218.6	114	0.320	
3.16	3160.7	112	0.319	
3.22	3102.9	110	0.320	0.319 (3.2 μm)
3.32	3016.1	107	0.321	
3.35	3984.0	236	0.319	0.318 (3.4 μm)
3.49	2867.8	227	0.319	
3.61	2768.3	219	0.319	0.318 (3 μm)
3.73	2684.4	212	0.319	0.318 (3.8 μm)
3.87	2584.9	204	0.318	
4.04	2474.4	195	0.318	0.317 (4 μm)
4.30	2328.5	185	0.321	0.316 (4.4 μm)
4.46	2242.4	176	0.316	0.316 (4.6 μm)
4.97	2010.3	156	0.315	0.315 (5 μm)
11.848	844	158	0.312	0.313
11.990	834	157	0.3138	0.314
12.107	826	156	0.3148	0.316
12.225	818	155	0.3158	0.316
12.361	809	154	0.3173	0.316
12.500	800	153	0.3188	0.316
12.642	791	152	0.3203	0.316
12.788	782	151	0.3218	0.315
12.937	773	150	0.3234	0.315
13.055	766	149	0.3242	0.315
13.228	756	148	0.3263	0.315
13.351	749	147	0.3271	0.315
13.514	740	146	0.3288	0.315
13.661	732	145	0.3301	0.314
13.831	723	144	0.3319	0.314
13.986	715	143	0.3333	0.314
14.144	707	142	0.3347	0.314
14.327	698	141	0.3367	0.314
14.493	690	140	0.3382	0.313

TABLE II. (*Continued.*)

λ (μm)	ω (cm^{-1})	k	Δn_{fr}	Δn_{pr}
14.663	682	139	0.3397	0.313
14.859	673	138	0.3418	0.313
14.993	667	137	0.3423	0.313
15.198	658	136	0.3445	0.312
15.385	650	135	0.3462	0.312
15.576	642	134	0.3479	0.311
15.773	634	133	0.3496	0.311
15.949	627	132	0.3509	0.311
16.181	618	131	0.3533	0.310
16.366	611	130	0.3546	0.3100

IV. CONCLUSION

We have experimentally measured and constructed dispersion relations for the ordinary (n_o) and extraordinary (n_e) refractive indices in GaSe in the spectral range of 0.7–5 μm . The results should be used for carrying out phase-matched and other nonlinear-optical calculations when creating nonlinear laser devices based on GaSe.

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