

# Anisotropic thermal expansion of potassium dinitramide: a variable-temperature crystallographic study

Michaele J. Hardie,† Anthony Martin, A. Alan Pinkerton\* and Elizabeth A. Zhurova

Department of Chemistry, University of Toledo, Toledo, OH 43606, USA

† Present address: School of Chemistry, The University of Leeds, Woodhouse Lane, Leeds LS2 9JT, England.

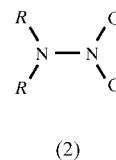
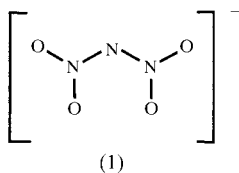
Correspondence e-mail:  
apinker@uoft02.utoledo.edu

The structure of potassium dinitramide (KDN),  $\text{KN}_3\text{O}_4$ , has been refined in the temperature range 85–298 K from single-crystal X-ray diffraction data. The unit-cell axial lengths and the cell volume decrease linearly on cooling with the  $b$  axis being the most sensitive to the change of temperature. The  $\beta$  cell angle increases with decreasing temperature. The thermal expansion of KDN is significantly anisotropic, expanding along the  $b$  axis [010] more than three times the amount parallel to any other crystallographic direction. Other eigenvectors of the thermal expansion tensor lie approximately parallel to the diagonals of the  $ac$  plane. A rigid-body analysis of the dinitramide ion using the TLS formalism was performed and shows that the thermal motion of the anion is well represented by the rigid-body model. The eigenvalues of the libration tensor show significant anisotropy, whereas the translation tensor is close to isotropic. The variation of all descriptions of the thermal motion with respect to temperature indicates an anharmonic contribution to the mean field potential. The direction of greatest unit-cell expansion coincides with the largest components of the displacement tensor of the potassium ions and the direction of the largest atomic amplitudes due to the libration of the dinitramide anions.

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## 1. Introduction

The dinitramide anion  $\text{N}(\text{NO}_2)_2^-$  (1) is an inorganic member of the nitramine class of compounds (2), which have been extensively studied due to their applications as explosives and low-signature propellants (Yinon & Zitrin, 1981; Olah & Squire, 1991). The first reported synthesis of a dinitramide compound was of the ammonium salt (Bottaro *et al.*, 1991, 1993). Since that time there has been intense activity, a number of authors having reported syntheses of various dinitramide salts, their structural characterization, spectral properties, details of their thermal decomposition as well as theoretical studies of their structure, bonding and decomposition (see, for example, Tanbug *et al.*, 1999, and references therein).



There is current interest in the anisotropic properties of energetic materials, in particular as they may relate to shock sensitivity (Dick, 1995; Jindal & Dlott, 1998; Franken *et al.*, 1999; Dlott & Favez, 1990). In this study we report the

determination of the thermal expansion of one member of the dinitramide family, potassium dinitramide (KDN), as well as the variations of the atomic thermal displacement parameters using single-crystal X-ray diffraction data in the temperature range 85–298 K. The anisotropy of the thermal expansion is analyzed with respect to the thermal motion of the potassium cations and the rigid-body motion of the dinitramide anions.

## 2. Experimental

A crystal of KDN suitable for single-crystal diffraction studies,  $0.34 \times 0.14 \times 0.08$  mm, was mounted on a fine glass fiber with epoxy resin. Preliminary examination and data collection were carried out at ambient temperature with a Siemens *SMART* (Siemens, 1996*a*) Platform diffractometer using Mo  $K\alpha$  radiation. In subsequent experiments, the crystal was cooled by nitrogen gas using an Oxford Cryostream system. Data collections were carried out at 298 (1), 250 (1), 200 (1), 150 (1), 100 (1) and 85 (1) K.  $0.3^\circ$   $\omega$  scans were performed at three different  $\varphi$  settings corresponding to a nominal hemisphere of data. The frame time was set at 20 s for all experiments. The unit-cell parameters were determined at each temperature from the observed *XYZ* centroids for the complete data set. The intensities of the reflections were empirically corrected for absorption using *SADABS* (Sheldrick, 1996). The structures were redetermined by direct methods using the *SHELXS* program package (Sheldrick, 1985). Refinement of each structure was performed by full-matrix least-squares based on  $F^2$  (*SHELXL*, Sheldrick, 1996). All atoms were refined with anisotropic displacement parameters. The details of the data collections and refinements at all six temperatures are given in Table 1. A rigid-body analysis (Schomaker & Trueblood, 1968) of the dinitramide anion was performed for each temperature using unit weights for each atom. Thermal expansion tensors were calculated from changes

**Table 1**  
Experimental details.

	85 K	100 K	150 K
<b>Crystal data</b>			
Chemical formula	KN <sub>3</sub> O <sub>4</sub>	KN <sub>3</sub> O <sub>4</sub>	KN <sub>3</sub> O <sub>4</sub>
Chemical formula weight	145.13	145.13	145.13
Cell setting, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.5891 (4), 9.0653 (5), 7.1459 (4)	6.5918 (4), 9.0778 (5), 7.1540 (4)	6.6010 (3), 9.1253 (5), 7.1657 (4)
$\beta$ (°)	97.975 (2)	97.946 (2)	97.890 (1)
<i>V</i> (Å <sup>3</sup> )	422.71 (4)	423.98 (4)	427.55 (4)
<i>Z</i>	4	4	4
<i>D<sub>x</sub></i> (Mg m <sup>-3</sup> )	2.280	2.274	2.255
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
No. of reflections for cell parameters	2897	2901	2857
$\theta$ range (°)	6.39–24.37	6.38–28.04	5.04–28.27
$\mu$ (mm <sup>-1</sup> )	1.172	1.169	1.159
Temperature (K)	85 (1)	100 (1)	150 (1)
Crystal size (mm)	0.34 × 0.14 × 0.08	0.34 × 0.14 × 0.08	0.34 × 0.14 × 0.08
<b>Data collection</b>			
Diffractometer	Platform	Platform	Platform
Data collection method	$\omega$ scans	$\omega$ scans	$\omega$ scans
Absorption correction	Empirical: Multipole Expansion (Blessing, 1995)	Empirical: Multipole Expansion (Blessing, 1995)	Empirical: Multipole Expansion (Blessing, 1995)
<i>T<sub>min</sub></i>	0.7905	0.7677	0.7801
<i>T<sub>max</sub></i>	0.9903	0.9903	0.9904
No. of measured, independent and observed parameters	3515, 1034, 902	3547, 1041, 918	3517, 1046, 917
Criterion for observed reflections	$I > 2\sigma(I)$	$I > 2\sigma(I)$	$I > 2\sigma(I)$
<i>R<sub>int</sub></i>	0.0484	0.0463	0.0402
$\theta_{\max}$ (°)	28.33	28.36	28.28
Range of <i>h</i> , <i>k</i> , <i>l</i>	–8 → <i>h</i> → 7 –12 → <i>k</i> → 12 –8 → <i>l</i> → 9	–8 → <i>h</i> → 7 –12 → <i>k</i> → 12 –8 → <i>l</i> → 9	–7 → <i>h</i> → 8 –12 → <i>k</i> → 12 –9 → <i>l</i> → 8
<b>Refinement</b>			
Refinement on	$F^2$	$F^2$	$F^2$
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , <i>S</i>	0.0311, 0.0717, 1.053	0.0294, 0.0664, 1.036	0.0302, 0.0697, 1.084
No. of reflections and parameters used in refinement	1034, 73	1041, 73	1046, 73
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.0000P]$ , where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.0380P)^2 + 0.0000P]$ , where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.0000P]$ , where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$	0.000	0.000	0.000
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.552, –0.601	0.533, –0.595	0.363, –0.386
<hr/>			
	200 K	250 K	298 K
<b>Crystal data</b>			
Chemical formula	KN <sub>3</sub> O <sub>4</sub>	KN <sub>3</sub> O <sub>4</sub>	KN <sub>3</sub> O <sub>4</sub>
Chemical formula weight	145.13	145.13	145.13
Cell setting, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.6029 (4), 9.1694 (5), 7.1731 (4)	6.6114 (1), 9.2299 (2), 7.1878 (2)	6.6162 (2), 9.2831 (2), 7.2000 (3)
$\beta$ (°)	97.805 (1)	97.639 (1)	97.583 (1)
<i>V</i> (Å <sup>3</sup> )	430.27 (4)	434.73 (2)	438.35 (2)
<i>Z</i>	4	4	4
<i>D<sub>x</sub></i> (Mg m <sup>-3</sup> )	2.240	2.217	2.199
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
No. of reflections for cell parameters	2788	2693	2568
$\theta$ range (°)	5.94–23.82	6.31–23.35	5.00–27.94
$\mu$ (mm <sup>-1</sup> )	1.152	1.140	1.130
Temperature (K)	200 (1)	250 (1)	298 (1)
Crystal size (mm)	0.34 × 0.14 × 0.08	0.34 × 0.14 × 0.08	0.34 × 0.14 × 0.08

Table 1 (continued)

	200 K	250 K	298 K
Data collection			
Diffraction method	Platform	Platform	Platform
Data collection method	$\omega$ scans	$\omega$ scans	$\omega$ scans
Absorption correction	Empirical: Multipole Expansion (Blessing, 1995)	Empirical: Multipole Expansion (Blessing, 1995)	Empirical: Multipole Expansion (Blessing, 1995)
$T_{\min}$	0.7226	0.6845	0.2829
$T_{\max}$	0.9903	0.9903	0.9903
No. of measured, independent and observed parameters	3510, 1060, 911	3613, 1073, 882	3648, 1084, 854
Criterion for observed reflections	$I > 2\sigma(I)$	$I > 2\sigma(I)$	$I > 2\sigma(I)$
$R_{\text{int}}$	0.0402	0.0449	0.0570
$\theta_{\text{max}}$ (°)	28.30	28.33	28.29
Range of $h, k, l$	$-8 \rightarrow h \rightarrow 7$ $-12 \rightarrow k \rightarrow 12$ $-8 \rightarrow l \rightarrow 9$	$-7 \rightarrow h \rightarrow 8$ $-12 \rightarrow k \rightarrow 12$ $-9 \rightarrow l \rightarrow 8$	$-8 \rightarrow h \rightarrow 7$ $-12 \rightarrow k \rightarrow 12$ $-8 \rightarrow l \rightarrow 9$
Refinement			
Refinement on	$F^2$	$F^2$	$F^2$
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.0315, 0.075, 1.058	0.033, 0.0768, 1.05	0.0391, 0.0944, 1.058
No. of reflections and parameters used in refinement	1060, 73	1073, 73	1084, 73
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.0000P]$ , where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.0450P)^2 + 0.0000P]$ , where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.0000P]$ , where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}}$	-0.001	-0.001	0.000
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.359, -0.351	0.306, -0.432	0.429, -0.378

Computer programs used: SMART (Siemens, 1996a), SAINT (Siemens, 1996b), SHELXS86 (Sheldrick, 1985), SHELXL93 (Sheldrick, 1993), Siemens SHELXTL and the Toledo cifomatic (Siemens, 1997).

Table 2

Thermal expansion.

(a) Tensor components of thermal expansion (10<sup>-6</sup> K<sup>-1</sup>) for KDN:  $T = 85$ – $298$  K.

$\alpha_{11}$	$\alpha_{22}$	$\alpha_{33}$	$\alpha_{12}$	$\alpha_{13}$	$\alpha_{23}$
23.86 (5)	109.3 (6)	34.52 (4)	0.0	14.7 (1)	0.0

(b) Eigenvalues (10<sup>-6</sup> K<sup>-1</sup>) and eigenvectors of the thermal expansion tensor.

Principal axes	Eigenvalue	Eigenvector
$\alpha_1$	15.5	0.8110 0.000 -0.5851
$\alpha_2$	109.3	0.000 1.000 0.000
$\alpha_3$	40.0	0.5851 0.000 0.8110

in the Bragg angle with temperature for all reflections across all six data sets with the program ALPHA (Jessen & Küppers, 1991). The changes in the interplanar distance,  $d$ , were observed to be linear with respect to temperature and were then constrained to be so, thus smoothing the data. Full details of all refinements have been deposited as supplementary material.<sup>1</sup>

<sup>1</sup>Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR0101). Services for accessing these data are described at the back of the journal.

### 3. Results and discussion

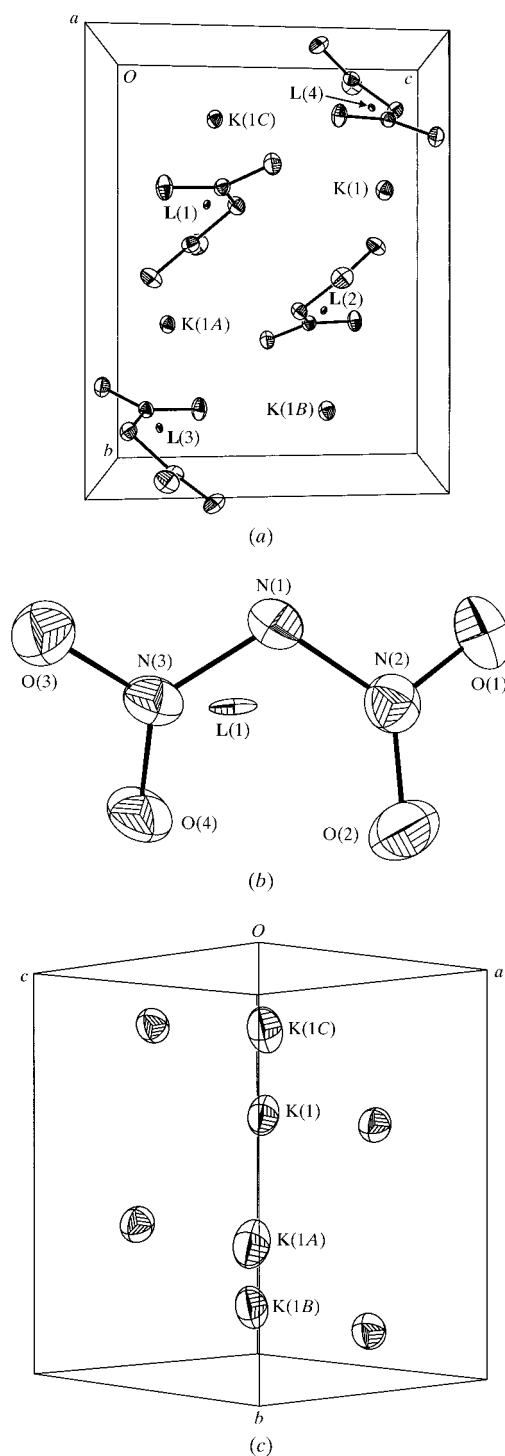
The crystal structure of potassium dinitramide at room temperature has been previously reported (Gidaspov *et al.*, 1995; Gilardi *et al.*, 1997). Our room-temperature result is in good agreement with that reported by Gilardi *et al.* (1997), however, there are significant discrepancies with the report from Gidaspov *et al.* (1995), especially with respect to the unit-cell parameters. From our results, KDN crystallizes in space group  $P2_1/n$  with  $a = 6.6162$  (2),  $b = 9.2831$  (2),  $c = 7.2000$  (3) Å,  $\beta = 97.583$  (1)° at room temperature. The unit cell contains four dinitramide anions and four potassium cations (Fig. 1).

The temperature variation of the unit-cell parameters normalized to those at room temperature is shown in Fig. 2. The unit-cell lengths and cell volume decrease on cooling, with the  $b$  axis being the most sensitive to change of temperature. The  $\beta$  cell angle increases with decreasing temperature. The temperature dependence of all unit-cell parameters was found to be close to linear, with the worst correlation coefficient being 0.987 for the  $\beta$  parameter.

Thermal expansion can also be represented by a symmetrical second-rank tensor  $[\alpha_{ij}]$ . The tensor components,  $\alpha_{ij}$ , were calculated from a least-squares fit to changes in  $d$  values with respect to temperature (Jessen & Küppers, 1991). KDN has a monoclinic unit cell, so that two of the off-diagonal terms of the tensor must be zero and one axis of the tensor must be parallel to the crystallographic  $b$  axis. The  $\alpha_{ij}$  values are presented in Table 2(a) for the whole temperature range. The corresponding eigenvalues and eigenvectors of the thermal expansion tensor are presented in Table 2(b). A representation of the thermal expansion tensor showing the principal axes given by their eigenvalues and eigenvectors, visualized as an ellipsoid in a unit cell, is shown in Fig. 3. The ellipsoid has been scaled such that the largest principal axis is identical to the  $b$  axial length. KDN expands along the  $b$  axis [010] more than three times as much as any other crystallographic direction, *i.e.* this is a significantly anisotropic material. The other two eigenvectors of thermal expansion are approximately parallel to the diagonals of the  $ac$  plane.

Atomic displacement parameters decrease with lowered temperature (Fig. 4) giving a non-linear temperature dependence with a small non-zero intercept at 0 K. Fitting the  $U_{\text{eq}}$  values to a quadratic polynomial produces a statistically reasonable fit. This is evidence of the anharmonic character of the atomic thermal motion in KDN, which is necessary both

for thermal expansion and for shock-induced heating in detonative energetic materials (Jindal & Dlott, 1998).



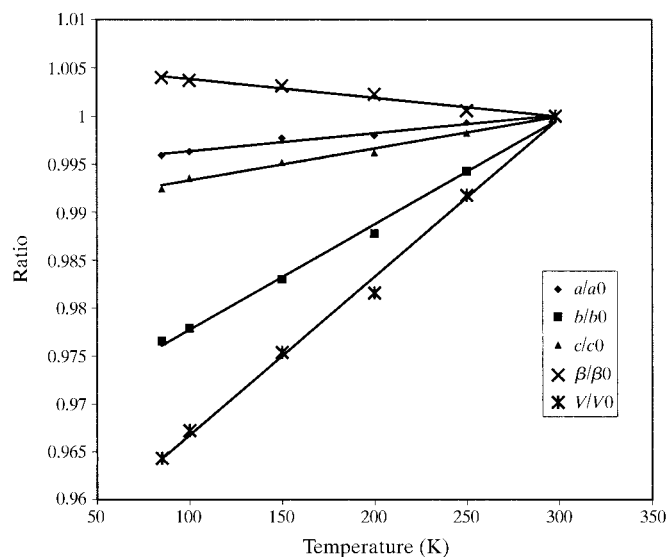
**Figure 1**  
 (a) Potassium dinitramide, unit cell with 20% atomic probability ellipsoids at 298 K including the libration tensor  $L(i)$  for each anion. (b) Dinitramide anion, 50% atomic probability ellipsoids at 298 K, with the libration tensor represented in the same way, superimposed. (c) Potassium cations, 90% atomic probability ellipsoids at 298 K, with the translation tensor for the dinitramide anions represented in the same way, superimposed. (Different probabilities have been used to emphasize the representation of the translation and libration tensors.)

**Table 3**  
 Eigenvalues and eigenvectors of libration and translation tensors of the dinitramide anion at 298 K.

	Eigenvalue		Eigenvector	
Libration ( $\text{rad}^2$ )	0.02244	0.8736	-0.4639	0.1468
	0.00235	0.4377	0.6173	-0.6538
	0.00489	0.2127	0.6354	0.7423
Translation ( $\text{\AA}^2$ )	0.03136	0.8470	-0.4722	0.2444
	0.02465	0.4141	0.8742	0.2536
	0.02146	-0.3334	-0.1136	0.9359

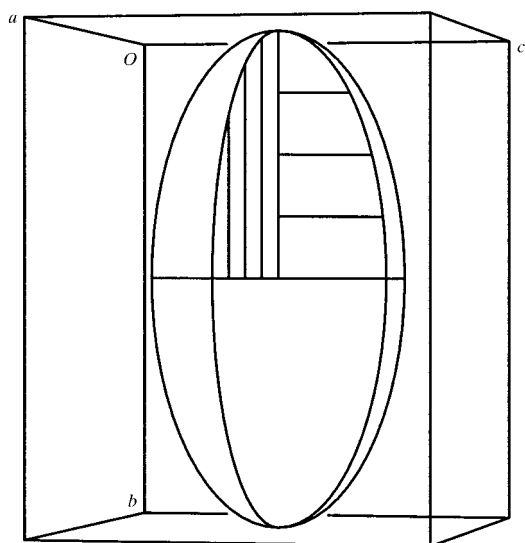
A rigid-body analysis of the dinitramide ion using the TLS formalism was performed at each temperature. The S matrix was small enough to be neglected in all cases. The eigenvalues of the libration tensor show that the librational motion is significant (largest value  $0.02244 \text{ rad}^2$  at 298 K) with a small  $T^2$  dependence (Fig. 5a, where  $L$  is the average of the three eigenvalues). The largest of the translational eigenvalues is  $0.03136 \text{ \AA}^2$  at 298 K. The translational eigenvalues also have a non-negligible  $T^2$  dependence (Fig. 5b), only the average value being reported as the tensor is close to isotropic. In contrast, the libration tensor is strongly anisotropic with the largest component parallel to the N(2)–N(3) vector (Fig. 1b).

If we consider that anharmonicity is more important for higher displacement amplitudes, it is significant that the direction of greatest unit-cell expansion coincides with the largest component of the displacement tensor of the potassium ions (Fig. 1c). In addition, the most important axis of libration for the dinitramide anion is close to parallel ( $29^\circ$ ) to the  $a$  axis (Table 3). When this is combined with the orientation of the dinitramide anions in the unit cell, the resulting amplitude of motion of the component atoms is again most important in the  $b$  direction.

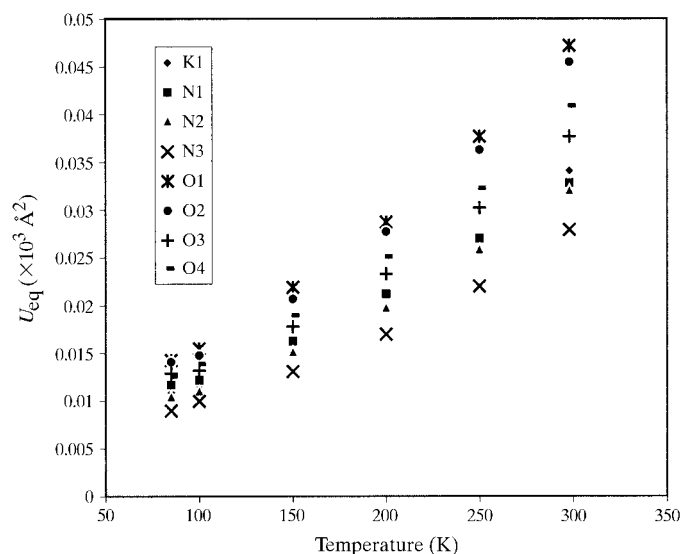


**Figure 2**  
 Temperature variation of KDN unit-cell parameters and volume normalized to those at room temperature ( $a = 6.580 + 1.244 \times 10^{-4}T$ ,  $b = 8.975 + 1.01 \times 10^{-3}T$ ,  $c = 7.128 + 2.403 \times 10^{-4}T \text{ \AA}$ ,  $\beta = 98.150 - 1.907 \times 10^{-3}T^\circ$ ,  $V = 416.54 + 7.231 \times 10^{-2}T \text{ \AA}^3$ ).

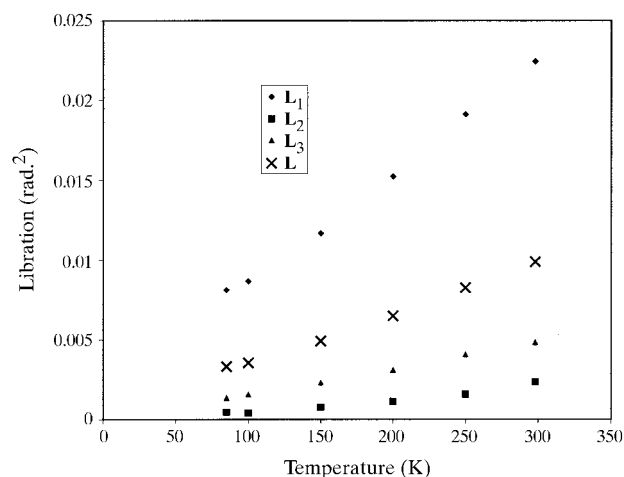
The contact distances between the potassium cation and the O atoms of the dinitramide anion decrease linearly with decreasing temperature, as expected from the unit-cell variation. At room temperature the four closest contacts between the potassium cation and the O atoms of the dinitramide anion range from 2.810 (2) to 2.947 (2) Å; at 85 (1) K the contacts range from 2.7800 (12) to 2.9159 (12) Å. The libration-corrected bond lengths within the dinitramide anion show only minimal expansion with increasing temperature (average 0.002 Å over the complete temperature range).



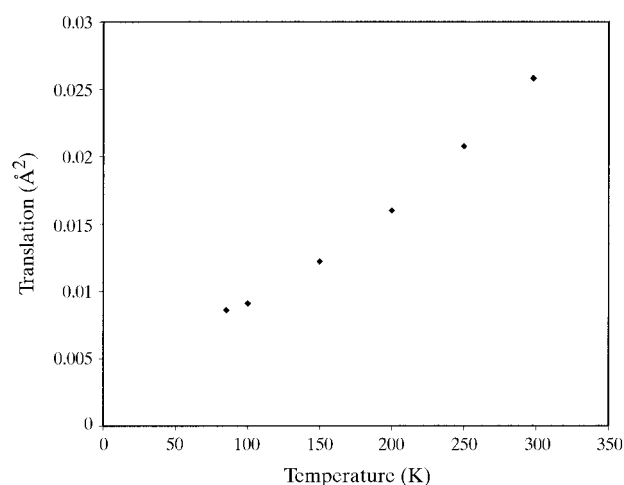
**Figure 3**  
Representation of the thermal expansion tensor in the unit cell for KDN. The size of the ellipsoid is scaled such that the largest principal axis is identical to  $b$ .



**Figure 4**  
Temperature variation of  $U_{eq}$  for KDN,  $U_{eq} = a + bT + cT^2$  (K1,  $a = 7.32 \times 10^{-3}$ ,  $b = 2.5 \times 10^{-5}$ ,  $c = 2.15 \times 10^{-7}$ ; O1,  $a = 7.03 \times 10^{-3}$ ,  $b = 6.4 \times 10^{-5}$ ,  $c = 2.36 \times 10^{-7}$ ; O2,  $a = 7.48 \times 10^{-3}$ ,  $b = 5.4 \times 10^{-5}$ ,  $c = 2.42 \times 10^{-7}$ ; O3,  $a = 9.70 \times 10^{-3}$ ,  $b = 1.1 \times 10^{-5}$ ,  $c = 2.83 \times 10^{-7}$ ; O4,  $a = 8.95 \times 10^{-3}$ ,  $b = 2.4 \times 10^{-5}$ ,  $c = 2.79 \times 10^{-7}$ ; N1,  $a = 8.06 \times 10^{-3}$ ,  $b = 2.4 \times 10^{-5}$ ,  $c = 2.04 \times 10^{-7}$ ; N2,  $a = 5.28 \times 10^{-3}$ ,  $b = 4.1 \times 10^{-5}$ ,  $c = 1.65 \times 10^{-7}$ ; N3,  $a = 6.75 \times 10^{-3}$ ,  $b = 1.1 \times 10^{-5}$ ,  $c = 2.02 \times 10^{-7}$ ).



(a)



(b)

**Figure 5**

Temperature variation of the eigenvalues for the  $DN^-$  anion: (a) libration tensor (where  $L$  is the average of  $L_1$ ,  $L_2$ ,  $L_3$ ); (b) translation tensor  $T$  (average of  $T_1$ ,  $T_2$ ,  $T_3$ , see text). Curves expressed as  $a + bT + cT^2$  are:  $L$ ,  $a = 1.40 \times 10^{-3}$ ,  $b = 1.91 \times 10^{-5}$ ,  $c = 3.22 \times 10^{-8}$ ;  $L_1$ ,  $a = 3.52 \times 10^{-3}$ ,  $b = 14.72 \times 10^{-5}$ ,  $c = 5.64 \times 10^{-8}$ ;  $L_2$ ,  $a = 4.26 \times 10^{-4}$ ,  $b = -2.63 \times 10^{-6}$ ,  $c = 3.01 \times 10^{-8}$ ;  $L_3$ ,  $a = 2.43 \times 10^{-4}$ ,  $b = 1.27 \times 10^{-5}$ ,  $c = 9.93 \times 10^{-9}$ ;  $T$ ,  $a = 5.65 \times 10^{-3}$ ,  $b = 1.95 \times 10^{-5}$ ,  $c = 1.62 \times 10^{-7}$ .

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## References

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–58.  
 Bottaro, J. C., Penwell, P. E. & Schmitt, R. J. (1991). *J. Synth. Commun.* **21**, 945–949.  
 Bottaro, J. C., Schmitt, R. J., Penwell, P. E. & Ross, D. S. (1993). US patents 5 198 204 and 5 254 324.  
 Dick, J. J. (1995). *J. Phys.* **IV**, **5**, 103–106.  
 Dlott, D. D. & Fayeze, M. D. (1990). *J. Chem. Phys.* **92**, 3798–3812.

- Franken, J., Hambir, S. A. & Dlott, D. D. (1999). *J. Appl. Phys.* **85**, 2068–2074.
- Gidasov, B. V., Tselinskii, I. V., Mel'nikov, V. V., Margolis, N. V. & Grigor'eva, N. V. (1995). *Russ. J. Gen. Chem.* **65**, 906–913.
- Gilardi, R., Flippen-Anderson, J., George, C. & Butcher, R. J. (1997). *J. Am. Chem. Soc.* **119**, 9411–9416.
- Jessen, S. M. & Küppers, H. (1991). *J. Appl. Cryst.* **24**, 239–242.
- Jindal, V. K. & Dlott, D. D. (1998). *J. Appl. Phys.* **83**, 5203–5211.
- Olah, G. A. & Squire, R. D. (1991). *Chemistry of Energetic Materials*. San Diego: Academic Press.
- Schomaker, V. & Trueblood, K. N. (1968). *Acta Cryst.* **B24**, 63–76.
- Sheldrick, G. M. (1985). *SHELXS86*. University of Göttingen, Germany.
- Sheldrick, G. M. (1993). *SHELXL93*. University of Göttingen, Germany.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Siemens (1996a). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1996b). *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1997). *SHELXTL5.0*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Tanbug, R., Kirschbaum, K. & Pinkerton, A. A. (1999). *J. Chem. Crystallogr.* **29**, 45–55.
- Yinon, J. & Zitrin, S. (1981). *The Analysis of Explosives*. Oxford: Pergamon Press.