ten Werte für die Molvolumina $Q(Na_2SO_4)/Q(K_2SO_4)=$ 1,01.

Eine andere Regel betrifft das Produkt aus der Eindruckhärte H und der Schleiffestigkeit F, das bei Substanzen mit ähnlicher Kompressibilität nur wenig variiert (von Engelhardt & Haussühl, 1965). Mit steigenden elastischen Konstanten, also fallender Kompressibilität, findet man mit wenigen Ausnahmen ein Ansteigen des Produktes $H.F$. Die räumlich gemittelte Eindruckhärte bzw. Schleiffestigkeit (bezogen auf $F_{NaCl}=1$) ist für Na_2SO_4 46,3 kg.mm^{-2} bzw. 1,33 und für K_2SO_4 115 kg.mm^{-2} bzw. 0,33. Das Produkt $H.F$ fällt demnach bei Na_2SO_4 etwas grösser aus als bei K_2SO_4 in Übereinstimmung mit dem Unterschied in der Kompressibilität.

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Literatur

Engelhardt, W. v. & Haussühl, S. (1965). *Fortschr. Min.* **42**, 5.

Groth, P. (1908). *Chemische Kristallographie.* II. Leipzig: Wilhelm Engelmann.

Haussühl, S. (1964). *Phys. Kondens. Materie,* **3**, 139.

Haussühl, S. (1965). *Acta Cryst.* **18**, 839.

Kusnezow, W. D. (1961). *Einfluss der Oberflächenenergie auf das Verhalten fester Körper,* S. 155. Berlin: Akademie-Verlag.

Acta Cryst. (1966). **20**, 932

Further refinement of the triuret structure.

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In an earlier paper (Carlström & Ringertz, 1965) the crystal and molecular structure of triuret (carbonyldiurea, $C_3H_6N_4O_3$) was presented. It was then pointed out that the refinement could have been carried further by applying anisotropic temperature parameters. This has now been done, with the use of the program ORFLS by Busing, Martin & Levy (1962) on an IBM 7090 computer. As in the preceding paper 1063 observed intensities except the outstandingly strong 200 reflexion were used in the refinement. The 168 accidentally absent reflexions were now given zero intensity. The scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1962) and one scale factor was used. An initial isotropic refinement cycle gave an R value equal to 0·128, which was reduced to the final value 0·081 after two cycles with anisotropic temperature factors in the form $\exp(-\beta_{11}h^2-\beta_{22}k^2 \beta_{33}l^2-\beta_{12}2hk-\beta_{13}2hl-\beta_{23}2kl)$. Throughout the calculations the thermal parameters of the hydrogen atoms were set equal to those of the corresponding nitrogen atoms. The corresponding final R value excluding the non-observed reflexions was 0·068. On request, a table of observed and calculated F values can be obtained from the author.

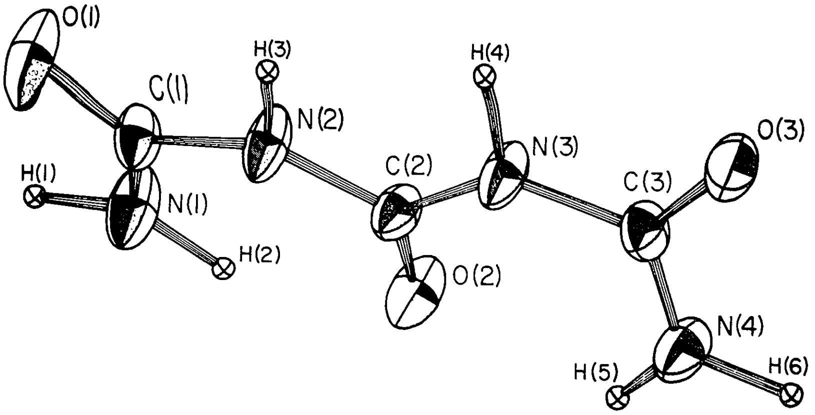

Fig. 1. Thermal ellipsoid representation of a triuret molecule. Ellipsoids enclose 74% of probability. Structure is viewed along an axis 15 degrees from the crystallographic a axis.

Table 1. *Final fractional atomic positional parameters and anisotropic temperature factor parameters for the non-hydrogen atoms, all with estimated standard errors and times* 10^5

	x/a	σ	y/b	σ	z/c	σ	β_{11}	σ	β_{22}	σ	β_{33}	σ	β_{12}	σ	β_{13}	σ	β_{23}	σ
C(1)	21810	48	59060	42	6324	13	1113	72	628	54	59	6	142	50	183	17	52	14
C(2)	24565	48	30815	40	12636	13	1179	73	448	51	68	6	−28	49	199	18	9	14
C(3)	24486	48	9836	43	20595	13	1086	73	641	55	55	6	21	50	154	17	32	14
N(1)	29948	48	50297	39	3301	13	1851	79	677	52	115	6	413	51	378	19	100	14
N(2)	19177	46	49060	36	10775	12	1809	77	498	47	95	6	304	48	348	18	74	13
N(3)	22233	46	27007	36	17714	12	1819	76	422	46	87	6	161	47	313	18	40	13
N(4)	25470	48	−5663	37	17821	13	1941	80	511	48	90	6	−90	49	292	18	5	14
O(1)	16373	42	75650	32	5613	12	2392	76	577	43	147	6	464	45	483	18	131	13
O(2)	30602	39	19572	30	10159	10	1972	67	513	40	106	5	133	41	377	16	18	11
O(3)	25023	42	9878	31	25673	10	2317	71	549	41	83	5	50	44	320	16	31	11

In the last cycle of refinement the average non-hydrogen positional shift was 0·02 and the maximum shift 0·08 of the estimated standard deviations. The corresponding values for the hydrogen positions were 0·19 and 0·44. To calculate the interatomic distances and angles the program ORFFE (Busing, Martin & Levy, 1964) was used, and the plotting of Fig. 1 utilized the program ORTEP (Johnson, 1965). The final positional parameters for the non-hydrogen atoms are given in Table 1, and for the hydrogen atoms in Table 2. None of the positions for the non-hydrogen atoms compared with those in the previous paper (Carlström & Ringertz, 1965) have changed more than 1·2 times the standard deviation of the difference and the maximum value for the hydrogen shift is 2·0. As can be seen on comparison, the estimated standard deviations have decreased considerably.

Table 2. *Positional parameters of hydrogen atoms in fractional coordinates times* 10^3

	x/a	σ	y/b	σ	z/c	σ
H(1)	322	8	585	7	2	2
H(2)	326	8	363	7	42	2
H(3)	155	8	563	7	133	2
H(3)	218	8	379	7	202	2
H(5)	248	8	−55	7	141	2
H(6)	258	8	−186	7	200	2

From the anisotropic thermal parameters listed in Table 1, the root-mean-square components of thermal displacement along the three principal axes were calculated for each atom and so were the angles between these axes and those of an orthogonal system XYZ defined by X along the b axis and Y normal to the approximate plane of the molecule (100). These values are listed in Table 3, and demonstrated in Fig. 1, showing the equiprobability surfaces of the ellipsoid thermal displacement containing 74 % of the probability distribution for the non-hydrogen atoms, whereas the hydrogen atoms are indicated by small spheres. It can be seen that the thermal motion in general is more or less normal to the three differently twisted planar parts of the molecule. The relation to neighbouring molecules can be seen in Fig. 3 of the previous work (Carlström & Ringertz, 1965).

In Table 4(*a*), (*b*), (*c*) and (*d*) are given the values for intramolecular bond-distances and angles with estimated standard deviation. For the intramolecular non-hydrogen distances in Table 4(*b*) the observed values are given together with distances corrected for thermal motion according to Busing & Levy (1964) assuming that the first atom 'rides' on the second. Table 4(*e*) shows the intra- and intermolecular hydrogen bonds and short contacts. Of these the three last have to be classified as short contacts as they have unfavorable N–H · · · O angles and H · · · O distances

Table 3. *Principal axes* (i) *of anisotropic temperature factors, referred to orthogonal axes XYZ*

X is along the crystallographic b axis, Z along the c axis and Y normal to the approximate plane of the molecule (100). The r.m.s. displacements, $\bar{u}$, are in Å and the direction angles, V, are in degrees.

	i	$\bar{u}_i$	σ	V_X	σ	V_Y	σ	V_Z	σ
C(1)	1	0·090	7	71	8	93	5	18	7
	2	0·124	6	32	8	62	7	105	8
	3	0·155	5	116	7	28	7	79	4
C(2)	1	0·098	7	56	20	99	5	35	20
	2	0·112	6	34	20	86	6	124	20
	3	0·154	5	88	5	10	4	80	5
C(3)	1	0·097	7	70	8	88	5	20	8
	2	0·132	5	21	9	85	16	110	8
	3	0·147	5	96	15	6	15	90	7
N(1)	1	0·105	6	82	19	106	8	18	3
	2	0·117	6	23	8	67	6	91	20
	3	0·209	4	111	2	20	2	72	2
N(2)	1	0·090	6	71	16	99	6	21	12
	2	0·106	6	24	13	71	3	105	16
	3	0·199	4	105	2	21	2	76	2
N(3)	1	0·096	6	50	28	92	6	40	27
	2	0·105	6	41	27	77	2	129	28
	3	0·193	4	98	2	12	2	81	2
N(4)	1	0·110	6	33	15	94	2	57	16
	2	0·125	5	57	15	90	3	147	16
	3	0·197	4	86	3	5	2	88	3
O(1)	1	0·102	5	31	8	84	4	59	8
	2	0·124	4	64	9	66	2	143	8
	3	0·233	4	106	1	25	2	72	1
O(2)	1	0·100	5	105	14	104	2	22	11
	2	0·115	5	163	13	92	4	106	14
	3	0·203	3	96	2	15	2	76	2
O(3)	1	0·103	5	58	10	87	2	32	10
	2	0·124	4	32	10	89	2	122	10
	3	0·214	3	92	2	3	2	92	2

Table 4. *Distances and angles, all with estimated standard deviations*

(I) Observed distances; (II) corrected for thermal motion assuming one atom to 'ride' on the other

(*a*) Intramolecular angles including non-hydrogen atoms

N(1)–C(1)–N(2)	118·6°	0·3 °
N(1)–C(1)–O(1)	125·1	0·3
N(2)–C(1)–O(1)	116·3	0·3
N(2)–C(2)–N(3)	110·6	0·2
N(2)–C(2)–O(2)	124·7	0·3
N(3)–C(2)–O(2)	124·7	0·3
N(3)–C(3)–N(4)	119·5	0·2
N(3)–C(3)–O(3)	117·2	0·3
N(4)–C(3)–O(3)	123·2	0·3
C(1)–N(2)–C(2)	128·4	0·3
C(2)–N(3)–C(3)	127·5	0·3

(*b*) Non-hydrogen intramolecular distances

	I		II	
C(1)–N(1)	1·318Å	4	1·326Å	4
C(1)–N(2)	1·406	4	1·410	4
C(2)–N(2)	1·373	4	1·378	4
C(2)–N(3)	1·380	4	1·384	4
C(3)–N(3)	1·388	4	1·393	4
C(3)–N(4)	1·325	4	1·332	4
C(1)–O(1)	1·233	4	1·248	4
C(2)–O(2)	1·216	4	1·225	4
C(3)–O(3)	1·242	4	1·251	4

(*c*) Angles involving hydrogen atoms

C(1)–N(1)–H(1)	116°	2°
C(1)–N(1)–H(2)	115	3
H(1)–N(1)–H(2)	129	4
C(1)–N(2)–H(3)	116	3
C(2)–N(2)–H(3)	115	3
C(2)–N(3)–H(4)	119	3
C(3)–N(3)–H(4)	113	3
C(3)–N(4)–H(5)	122	3
C(3)–N(4)–H(6)	117	2
H(5)–N(4)–H(6)	121	4

(*d*) Nitrogen–hydrogen bond distances

N(1)–H(1)	1·05Å	5
N(1)–H(2)	1·02	5
N(2)–H(3)	0·95	5
N(3)–H(4)	1·01	5
N(4)–H(5)	0·91	5
N(4)–H(6)	1·07	5

(*e*) Intra- and intermolecular hydrogen bonds and short contacts

N(1)–O(2)	2·764Å	3
N(4)–O(2)	2·776	3
N(1)–O(1) (0·5−*X*; 1·5−*Y*; −*Z*)	2·926	4
N(3)–O(3) (0·5−*X*; 1·5+*Y*; 0·5−*Z*)	2·815	3
N(4)–O(3) (0·5−*X*; *Y*−0·5; 0·5−*Z*)	2·954	4
N(4)–O(1) (*X*; *Y*−1; *Z*)	3·070	4
N(1)–O(1) (0·5+*X*; *Y*−0·5; *Z*)	2·971	4
N(2)–O(2) (*X*−0·5; *Y*+0·5; *Z*)	3·079	3

of 2·3–2·4 Å. This means that there is no hydrogen bonding except in the molecular plane of the layered structure. The suggestion in the previous paper that there might be a weak hydrogen bond between adjacent molecular layers has thus not been supported. All other values in Table 4 are in agreement with those in Tables 3, 6 and Fig. 2 of the previous paper. The planarity of the three urea fractions of the molecule is within 0·008 Å, and the average atomic distance from the mean plane of all non-hydrogen atoms is 0·102 Å. The results of this further refinement do not essentially affect the discussion in the previous paper, in spite of the marked anisotropy of the atomic thermal motion. However the increased precision makes it possible to perform a more detailed comparison with related compounds.

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References

Busing, W. R. & Levy, H. A. (1964). *Acta Cryst.* **17**, 142.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). ORFLS, *A Fortran Crystallographic Least-Squares Program.* Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Busing, W. R., Martin, K. O. & Levy, H. A. (1964). ORFFE, *A Fortran Crystallographic Function and Error Program.* Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Carlström, D. & Ringertz, H. (1965). *Acta Cryst.* **18**, 307.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

Johnson, C. K. (1965). ORTEP, *A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations.* Oak Ridge National Laboratory, Oak Ridge, Tennessee.