Tabelle 1. *Elastische Konstanten c_{ij} für 20°C und thermoelastische Konstanten* $T_{ij}=\dfrac{d\log c_{ij}}{dT}$ *für 0°C von Benzil (T Temperatur)*

Einheiten: c_{ij} in 10^{11} erg.cm^{-3}, T_{ij} in 10^{-3}°C^{-1}. In Klammern stehen die Werte von Chakraborty & Sen.

ij	11	33	12	13	44	66	14
c_{ij}	1,092	0,823	0,544	0,326	0,108	0,274	−0,051
	(1,12)	(0,295)	(−0,076)	(−0,098)	(0,416)	(0,598)	(−0,146)
T_{ij}	−1,80	−2,40	−1,60	−2,44	+0,80	−1,96	−3,7

über, ob die Abweichungen auf eine geringe Genauigkeit des Röntgenverfahrens oder auf Dispersionseffekte zurückzuführen sind. Ein weiteres Beispiel, Benzil, dessen elastische Konstanten von Chakraborty & Sen (1959) mit dem Verfahren von Wooster sorgfältig untersucht worden waren, möge zur Entscheidung dieser Frage beitragen. Der Verfasser bestimmte hierzu die elastischen Konstanten und die zugehörigen Temperaturkoeffizienten von Benzil mit dem verbesserten Schaefer–Bergmann-Verfahren (Lichtbeugung an schwingenden Kristallen bei etwa 15 MHz). Die erforderlichen Einkristalle wurden aus Lösungen in *m*-Xylol durch langsames Eindunsten bei Temperaturen um 40°C in optisch klaren, blassgelblichen Individuen mit Durchmessern bis zu 5 cm hergestellt. Die zur Klasse 32 gehörenden Kristalle weisen das Prisma {100}, das Rhomboeder {012}, etwas kleiner das Rhomboeder {101} und untergeordnet das Pinakoid {001} auf. Die Gitterkonstanten für 20°C sind $a_1=8{,}41$ Å, $a_3=13{,}69$ Å. Für die Dichte ergab sich mit dem Auftriebverfahren der Wert 1,251 g.cm^{-3} bei 20°C. Für die Aufstellung des Elastizitätstensors wurde ein kartesisches Grundsystem gewählt mit derselben Orientierung wie bei Chakraborty & Sen: $\mathbf{e}_1\|\mathbf{a}_1$, $\mathbf{e}_3\|\mathbf{a}_3$ und $\mathbf{e}_2=\mathbf{e}_3\times\mathbf{e}_1$.

Aus den Ausbreitungsgeschwindigkeiten der elastischen Wellen in den kartesischen Richtungen und in deren winkelhalbierenden Richtungen wurden die in Tabelle 1 aufgeführten elastischen und thermoelastischen Konstanten ermittelt. Die Werte von Chakraborty & Sen sind in Klammern angegeben. Bei der Bestimmung der elastischen Konstanten wurde die piezoelektrische Wechselwirkung mit der von Meier & Schuster (1958) vorgeschlagenen Korrektur berücksichtigt. Für die statischen piezoelektrischen Konstanten wurden die Werte $d_{111}=-32\times10^{-8}$ und $d_{123}=3{,}76\times10^{-8}$ esu/dyn, für die relativen Dielektrizitätskonstanten die mit dem Einbettungsverfahren für 1 MHz gemessenen Werte $\varepsilon_1=3{,}02$ und $\varepsilon_3=3{,}30$ eingesetzt. Diese Korrektur macht bei c_{11} etwa 1 %, bei $c_{66}=(c_{11}-c_{12})/2$ etwa 5 % aus. Die relativen Fehler liegen unter folgenden Schranken: c_{11}, c_{33}: 2‰; c_{44}, c_{12}, c_{13}: 1 %; c_{14}: 5 %; T_{11}, T_{33}: 3 %; T_{44}, T_{12}, T_{13}: 6 %.

Ebenso wie im Falle von Benzalazin zeigt sich nun auch hier, dass das Röntgenverfahren nach Wooster grösstenteils völlig andere Werte liefert als das Schaefer–Bergmann-Verfahren. Da die Abweichungen bei Benzil und bei Benzalazin trotz der engen chemischen und strukturellen Verwandtschaft der beiden Substanzen keinerlei systematische Züge erkennen lassen, muss befürchtet werden, dass hier grundsätzliche Grenzen der Anwendbarkeit des Woosterschen Verfahrens auf komplizierter gebaute Kristalle sichtbar werden.

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On the crystal structure of bis(glyoximato)nickel(II).

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The crystal structure of bis(glyoximato)nickel(II) has been determined from three-dimensional X-ray diffraction data. The crystals are monoclinic in space group $P2_1/c$ with two molecules in a unit cell of dimensions $a=4{\cdot}21$, $b=7{\cdot}20$, $c=12{\cdot}41$ Å, and $\beta=90{\cdot}8$°. Within the experimental error the molecules have D_{2h} symmetry. The intramolecular distance between hydrogen bonded oxygen atoms is 2·45 Å. There are no appreciable intermolecular interactions between nickel atoms. The results of this work are compared with those from another recently reported determination of the structure.

An especially interesting problem concerns the short metal–metal distance in bis(dimethylglyoximato)nickel(II) and the short hydrogen bond also present (Godycki & Rundle, 1953; Rundle & Banks, 1963). In an effort to gain information about these factors, we initiated an X-ray diffraction study of bis(glyoximato)nickel(II).

After completing the structure determination we were informed of a previous study (Biagas, 1959), and shortly thereafter of still another study (Calleri, Ferraris & Viterbo, 1967; CFV). In view of the importance of the structure and to demonstrate the precision of atom parameters determined independently by somewhat different methods, we present a brief summary of our work and a comparison with that of CFV.

The space group and unit-cell dimensions were determined from Mo *Kα* and Cu *Kα* precession, oscillation, and Weissenberg photographs from two crystals. The unit cell was found to be monoclinic with the following dimensions: $a=4{\cdot}21\pm0{\cdot}01$, $b=7{\cdot}20\pm0{\cdot}01$, $c=12{\cdot}41\pm0{\cdot}02$ Å, and $\beta=90{\cdot}8\pm0{\cdot}2°$. The extinctions uniquely establish the space group $P2_1/c$. The density by flotation was found to be 2·02 g.cm^{-3}. For two molecules in the unit cell, the calculated density is 2·06 g.cm^{-3}.

A crystal about 0·08 × 0·16 × 0·44 mm was chosen for collection of the intensity data. Intensity data were visually estimated from Mo *Kα* multiple film equi-inclination Weissenberg photographs of the $0kl$–$4kl$ zones and from Mo *Kα* multiple exposures of the $hk0$, $h0l$, and hll zones by the precession method. A total of 559 independent reflections were of measurable intensity. Lorentz–polarization factors were applied in the usual fashion. No corrections were made for absorption ($\mu=24{\cdot}9$ cm^{-1}).

The nickel atoms occupy special positions $(0,0,0;\ 0,\frac{1}{2},\frac{1}{2})$. The asymmetric unit contains only half of one molecule which was easily located (except hydrogen atoms) from a three-dimensional Patterson synthesis by taking into account the large observed structure factor for the 112 reflection.

Isotropic full-matrix least-squares refinement, minimizing the numerator of $r=[\Sigma w(|F_o|^2-|kF_c|^2)^2/(\Sigma w|F_o|^2)^2]^{\frac{1}{2}}$ where $w=[0{\cdot}20(4FMIN)^2]^{-2}$ for $F_o\le4FMIN$ ($FMIN$ is the minimum F_o) and $w=(0{\cdot}20F_o^2)^{-2}$ for $F_o>4FMIN$, converged with $R=\Sigma\,(|F_o|^2-|kF_c|^2)/\Sigma\,|F_o|^2=0{\cdot}16$ and $r=0{\cdot}25$. Refinement with all atoms anisotropic converged with $R=0{\cdot}13$ and $r=0{\cdot}16$. The conventional agreement factor $R^*=\Sigma\,(|F_o|-|kF_c|)/\Sigma\,|F_o|$ was 0·077.

A difference Fourier synthesis at this stage had peaks at reasonable positions for hydrogen atoms attached to the carbon atoms, but no definite peak existed at the position where one would expect to find the hydrogen atom for the symmetrical hydrogen bond. The electron density at this position was, however, positive and of about one half the density of that observed for the carbon hydrogens. These observations are similar to those of CFV. It should be pointed out that several other peaks on the difference map were as large as those assigned to the carbon hydrogens.

The final parameters from the last described cycle of refinement are included in Table 1. The bond distances and angles are given in Table 2. The observed structure factors are available on request.

The two determinations of the structure were carried out with data collected and handled quite differently. The comparison is between results from visually estimated film data taken with Mo *Kα* radiation and counter data using Cu *Kα*

Table 1. *Positional and thermal parameters*

The thermal parameters are in the form $\exp\,[-\frac{1}{4}(h^2B_{11}a^{*2}+k^2B_{22}b^{*2}+l^2B_{33}c^{*2}+2hkB_{12}a^*b^*+2hlB_{13}a^*c^*+2klB_{23}b^*c^*)$. The atoms are numbered in agreement with Calleri *et al.* (1967). Standard deviations are given in parenthesis. To take into account the difference in molecular orientation from the CFV work, the signs of x, z, B_{12}, and B_{23} must be changed.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ni	0	0	0	1·96 (5)	3·15 (6)	2·74 (6)	0·5 (1)	0·16 (4)	−0·5 (1)
N(2)	0·1753 (16)	0·0885 (14)	−0·1279 (7)	2·4 (3)	4·4 (4)	3·4 (3)	0·1 (3)	0·1 (2)	−0·4 (3)
N(1)	−0·1966 (17)	0·2324 (12)	−0·0013 (7)	2·8 (3)	3·9 (4)	4·0 (4)	0·7 (3)	−0·3 (3)	−0·6 (3)
O(1)	0·3790 (15)	−0·0118 (16)	−0·1872 (6)	4·0 (3)	6·2 (4)	4·8 (3)	0·8 (4)	1·8 (2)	−1·0 (5)
O(2)	−0·3960 (20)	0·2903 (13)	0·0745 (7)	5·4 (4)	6·3 (5)	4·9 (4)	3·4 (4)	1·2 (3)	−0·9 (4)
C(1)	−0·1363 (24)	0·3380 (17)	−0·0840 (9)	3·5 (4)	4·3 (5)	4·7 (5)	0·4 (4)	−0·5 (4)	−0·4 (4)
C(2)	0·0807 (23)	0·2521 (19)	−0·1573 (8)	3·0 (4)	5·7 (6)	3·6 (4)	−0·5 (4)	0·1 (3)	0·1 (4)

Table 2. *Bond distances, bond angles, and intermolecular distances*

	MS[(a)]	CFV[(a)]
Ni—N(1)	1·866 (9) Å	1·868 (4) Å
Ni—N(2)	1·872 (9)	1·880 (4)
N(2)–C(2)	1·294 (15)	1·277 (7)
N(1)–C(1)	1·305 (14)	1·303 (6)
N(2)–O(1)	1·347 (11)	1·350 (5)
N(1)–O(2)	1·336 (10)	1·343 (5)
C(1)–C(2)	1·438 (15)	1·435 (7)
O(1)–O(2)(b)	2·445 (13)	2·453 (6)
N(1)–Ni—N(2)	82·4 (4)°	82·2 (2)°
Ni—N(2)–O(1)	122·8 (8)	122·8 (3)
Ni—N(2)–C(2)	115·2 (7)	115·5 (3)
C(2)–N(2)–O(1)	122·0 (8)	121·8 (4)
C(1)–C(2)–N(2)	114·0 (9)	113·9 (5)
C(2)–C(1)–N(1)	112·3 (9)	113·1 (5)
Ni—N(1)–O(2)	123·9 (7)	123·6 (3)
Ni—N(1)–C(1)	116·0 (7)	115·2 (3)
C(1)–N(1)–O(2)	120·1 (9)	121·1 (4)

	MS	CFV
N(2)–N(1)(c)	3·23 Å	—
N(2)–C(1)(c)	3·45	—
N(2)–O(2)(c)	3·40	—
N(2)–O(1)(c)	3·50	—
Ni—O(1)(c)	3·47	3·45 Å
Ni—O(2)(c)	3·41	3·40
O(1)–C(2)(d)	3·20	3·22
O(1)–C(2)(d)	3·45	—
O(1)–N(1)(c)	3·39	—
O(1)–C(1)(c)	3·48	—
O(1)–C(1)(d)	3·19	3·19
O(1)–O(2)(d)	3·50	—
C(2)–C(1)(c)	3·46	3·44
N(1)–O(1)(c)	3·39	—
C(1)–C(1)(e)	3·32	3·34
C(1)–O(2)(e)	3·49	—
C(1)–O(2)(e)	3·33	3·33

(*a*) MS = Murmann & Schlemper; CFV = Calleri, Ferraris & Viterbo. Standard deviations are given in parenthesis.
(*b*) Related by inversion center.
(*c*) In molecule translated one cell in **a** direction.
(*d*) In molecule related by *c* glide.
(*e*) In molecule translated one cell in **b** direction.

radiation. No absorption corrections were made in either case, and systematic errors due to absorption are not identical. In the CFV case no correction was made for anomalous dispersion by the nickel atoms while in the MS case this anomalous dispersion was taken into account.

Owing to the above differences one might expect appreciable variations in the two sets of parameters, but rather one finds remarkably good agreement in all parameters except the thermal parameters of the nickel atom. It is possible that the larger magnitude of vibration of the nickel atom found by CFV results from the failure to correct for anomalous dispersion. Using copper radiation the anomalous dispersion correction for the nickel atom is rather large and negative. Although the two sets of positional parameters are, within experimental error, in agreement with each other, a consideration of each set of parameters individually leads to contradictory conclusions concerning the asymmetry of the ligand. For example, the two independent C–N distances appear to be significantly different using the CFV parameters but appear to be identical using the MS parameters. Finally it should be pointed out that according to criteria soon to be published by Hamilton & Ibers (Hamilton, 1967), the intermolecular O(1)···C(1) and O(1)···C(2) distances do not indicate significant C–H···O hydrogen bonds.

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Space group and unit cell of diglycine strontium chloride trihydrate and diglycine calcium chloride tetrahydrate.

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Diglycine strontium chloride trihydrate, $(NH_2CH_2COOH)_2.SrCl_2.3H_2O$,crystallizes in the orthorhombic space group *Pbcn* with unit-cell dimensions $a=16{\cdot}00$, $b=9{\cdot}26$, $c=8{\cdot}25$ Å and $Z=4$. The crystals of diglycine calcium chloride tetrahydrate, $(NH_2CH_2COOH)_2.CaCl_2.4H_2O$, are monoclinic, space group $P2_1/c$, with unit-cell dimensions $a=13{\cdot}01$, $b=6{\cdot}794$, $c=15{\cdot}375$ Å, $\beta=91°24'$ and $Z=4$.

The work reported here forms part of a programme undertaken in this department to investigate the structure and properties of some addition compounds of glycine, in order to elucidate the structure of glycine in different states of aggregation and also to study the hydrogen bonds that stabilize these compounds (Krishnan & Narayanan, 1963; Krishnan & Balasubramanian, 1964; Easwaran, 1966*a*, *b*; Vijayan, 1966).

Crystals of diglycine strontium chloride trihydrate, $(NH_2CH_2COOH)_2.SrCl_2.3H_2O$, and diglycine calcium chloride tetrahydrate, $(NH_2CH_2COOH)_2.CaCl_2.4H_2O$, were grown by slow evaporation at room temperature of an aqueous solution of the components in stoichiometric proportions. Unit-cell dimensions were determined from oscillation and Weissenberg photographs taken about crystallographic axes with copper radiation. From systematic absences the strontium and calcium compounds were assigned to space groups *Pbcn* and $P2_1/c$ respectively. The densities of the samples were measured by flotation in a mixture of bromoform and carbon tetrachloride. The crystal data of the two compounds are given in Table 1.

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Table 1. *Crystal data for* $(NH_2CH_2COOH)_2.SrCl_2.3H_2O$ *and* $(NH_2CH_2COOH)_2.CaCl_2.4H_2O$

	$(NH_2CH_2COOH)_2.SrCl_2.3H_2O$	$(NH_2CH_2COOH)_2.CaCl_2.4H_2O$
a	$16{\cdot}00\pm0{\cdot}02$ Å	$13{\cdot}01\pm0{\cdot}01$ Å
b	$9{\cdot}26\pm0{\cdot}02$	$6{\cdot}794\pm0{\cdot}01$
c	$8{\cdot}25\pm0{\cdot}01$	$15{\cdot}375\pm0{\cdot}01$
β	—	$91°24'\pm30'$
V	1222·32 Å^3	1358·57 Å^3
M	362·7	333·2
D_m	1·982 g.cm^{-3}	1·630 g.cm^{-3}
D_x	1·971 g.cm^{-3}	1·629 g.cm^{-3}
Z	4	4
μ	110·35 cm^{-1}	77·89 cm^{-1}