where W_D is the variance of the disordered line profile, S the range over which the integration for the determination of W_D was made and $a = \gamma(1 - \cos 2\pi l g)$. The total variance ($W$) of the line profile, corrected for geometrical aberrations can now be written as

$$W = W_P + W_D,$$

where W_P is the variance resulting from the particle-size line profile. Proceeding as in Mitra (1964), we have for W, in 2θ units,

$$\frac{W \cos \theta}{\lambda S} = \frac{1}{\pi^2 P'} - \frac{a^2}{4\pi^2 d^2} \frac{\lambda}{S \cos \theta}, \quad (2)$$

where $1/P' = 1/P + a/2d$; P is the particle size, d the interplanar distance; λ the wavelength of radiation used and θ the Bragg angle of reflexion. a was determined from the slope of the linear plot of $W \cos \theta / \lambda S$ versus $\lambda/S \cos \theta$. Knowing the values of a and Δ (the peak shift of the lines) γ and g values were calculated. The results are shown in

Table 1. *Variation of g and γ with dehydration*

Temperature (°C)	g	γ
30	0·148	0·063
200	0·171	0·065
300	0·170	0·080
400	0·173	0·077
500	0·210	0·033
600	0·218	0·021

It is evident from Table 1 that with increasing dehydration at 200°C the mean fractional change in the interlayer distances of the basal planes increases considerably (from 0·148 to 0·171) while the proportion of planes involved remains almost unaffected (0·063 to 0·065). At 300°C more planes are affected – γ changing from 0·065 to 0·080; however, g remains constant. At 400°C, both γ and g are constant, but at 500°C there is a large increase in g while γ has considerably decreased. This shows that collapse of the kaolinite structure has started – the larger proportion of the already affected planes having been converted to metakaolin. Only a small proportion of the planes in the kaolinite structure remain but with greater disorder. This process is continued at 600°C. Thus the formation of metakaolin and collapse of the kaolinite structure is preceded by and accompanied by disorder of the basal planes so that the interlayer spacing is variable.

Details of this work will soon be published elsewhere.

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The crystal structure of ethylidene-N,N-diacetamide.

BY B. KOLAKOWSKI *Institute of Physics, Polish Academy of Sciences, Warsaw, Poland*

The crystal structure of ethylidene-N,N-diacetamide, $CH_3CH(NHCOCH_3)_2$, has been determined. The symmetry is monoclinic, space group $P2_1/c$. The molecules are linked by a double system of hydrogen bonds in layers parallel to (100) plane.

Ethylidene-N,N-diacetamide was prepared by the method described by Noyes & Forman (1933). Colorless crystals with melting point 180°C were grown by slowly evaporating a concentrated methyl alcohol solution. The crystal belongs to the monoclinic system with the space group $P2_1/c$. The unit cell has dimensions $a = 8·52$; $b = 9·54$; $c = 9·57$ Å; $\beta = 92°47'$ determined from Weissenberg and oscillation photographs taken about a, b and c axes with nickel-filtered Cu $K\alpha$ radiation. There are four molecules per unit cell, all in general positions. Intensities were measured by visual estimation from the equi-inclination Weissenberg photographs with series of calibrated spots. The number of independent reflexions was 674, of which 585 were in the measurable range. Lorentz–polarization corrections were made. Absorption corrections were considered to be unnecessary because of the small crystal used. The preliminary structure has been deduced from a geometrical consideration and Patterson synthesis. A three-dimensional least-squares refinement of the 30 positional parameters and 10 individual isotropic temperature parameters of carbon, nitrogen and oxygen atoms was carried out. After several cycles of refinement the value of R decreased to 0·14. Table 1 lists the final fractional coordinates and the thermal param-

eters for all the atoms of C,N and O. The projection of the structure along [010], and bond lengths and bond angles, are shown in Figs. 1 and 2 respectively. The average standard deviations in these are 0·015Å in the bond length and about 1° in the bond angle.

Table 1. *Final fractional coordinates and thermal parameters*

	x/a	y/b	z/c	B
C(1)	0·244	0·520	0·015	2·93
C(2)	0·307	0·388	0·091	2·58
C(3)	0·088	0·222	0·080	2·18
C(4)	0·036	0·139	0·155	3·61
C(3')	0·522	0·348	0·262	3·17
C(4')	0·639	0·405	0·374	3·76
N(5)	0·179	0·309	0·154	2·03
N(5')	0·420	0·432	0·208	2·41
O(6)	0·107	0·199	0·051	4·17
O(6')	0·535	0·219	0·223	4·62

The molecules in the crystal lattice are linked together by two series of hydrogen bonds. One series of length 2·96Å runs approximately parallel to the b axis and the other of length 2·88 Å runs approximately parallel to the c axis.
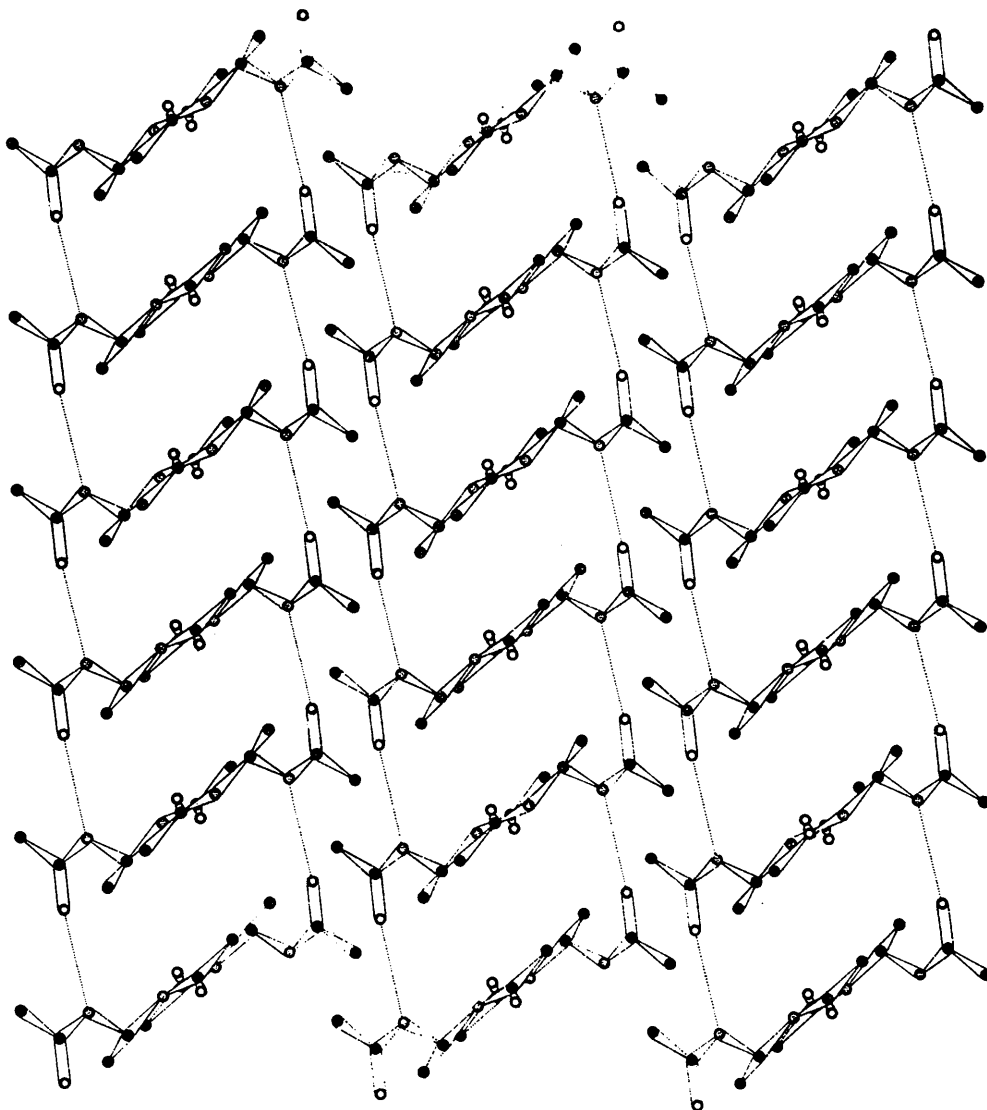
Fig. 1. Arrangement of molecules in (010) projection. Dotted lines indicate hydrogen bonds. Solid circles: carbon; stippled circles: nitrogen; open circles: oxygen.
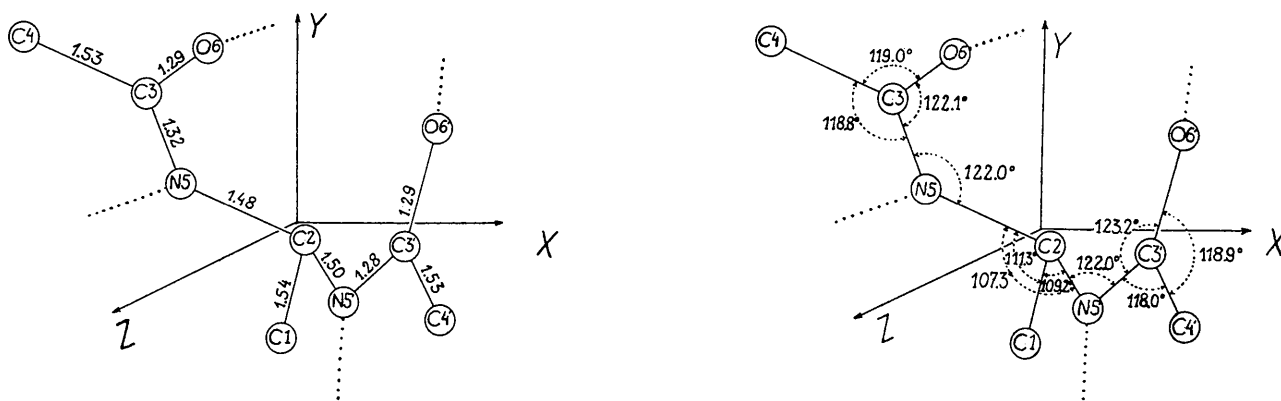
Fig. 2. Bond length and bond angles observed in the molecule of $CH_3CH(NHCOCH_3)_2$.

These hydrogen bonds bind the molecules into layers parallel to the (100) plane. The layers are held together by van der Waals interaction between methyl groups.

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The crystal structure of the form of N,N'-ethylenebis(salicylaldehydeiminato)cobalt (II) inactive towards oxygenation.

By S. BRÜCKNER, M. CALLIGARIS, G. NARDIN and L. RANDACCIO, *Istituto di Chimica, Università di Trieste, Trieste, Italy.*

The structure of N,N'-ethylenebis(salicylaldehydeiminato)cobalt(II), $CoO_2N_2C_{16}H_{14}$, (inactive form towards oxygenation) has been refined with three-dimensional data, starting from the final coordinates obtained from the two-dimensional analysis of the isostructural analogous copper derivative. The refinement gave an R index of 0·07 for 762 independent reflexions collected on equi-inclination Weissenberg photographs taken with Mo $K\alpha$ radiation.

Introduction

One of the best known examples of synthetic molecular oxygen-carriers is N,N'-ethylenebis(salicylaldehydeiminato)cobalt(II) (Cosalen) which absorbs molecular oxygen reversibly in the solid state as well as in solution (Bayer & Schretzmann, 1967). Several active (oxygen-carriers) crystalline modifications and one inactive modification of this compound are known. An X-ray powder spectrum analysis was carried out by Calvin (Martell & Calvin, 1959). However, the mode of attachment of oxygen and the absorption mechanism in the solid phase are still subjects of discussion. As part of a general investigation of these two aspects, we present here the three-dimensional analysis of the inactive form.

This work was carried out in order to obtain more accurate values of bond lengths and angles for subsequent comparison with the analogous compound $(Cosalen)_2$. $(dimethylformamide)_2 . O_2$, under investigation in this laboratory. The crystal structure of this compound will be described in a forthcoming paper. Dr S. Holt and his co-workers (private communication) have independently refined the structure of inactive Cosalen and their results agree with ours.

Experimental

The crystals, prepared according to Bailes & Calvin (1947), are monoclinic with $a = 26·52 \pm 0·03$, $b = 7·05 \pm 0·01$, $c = 14·36 \pm 0·02$ Å, $\beta = 98·4 \pm 0·3°$, $Z = 8$, $D_c = 1·63$ g.cm^{-3},

Table 1. *Atomic fractional coordinates* ($\times 10^4$) *and isotropic thermal parameters with e.s.d.'s given in parentheses*

The fractional coordinates ($\times 10^3$) of the hydrogen atoms are listed near their bonded carbon atoms.

	x	y	z	B		x	y	z
Co	2340 (1)	2034 (3)	977 (1)	1·68 (3)				
O(1)	1869 (4)	4043 (14)	901 (6)	2·32 (20)				
O(2)	2833 (3)	3773 (13)	554 (6)	1·65 (18)				
N(1)	1910 (4)	407 (17)	1532 (7)	1·83 (25)				
N(2)	2830 (5)	107 (19)	1259 (8)	2·20 (27)				
C(1)	1376 (5)	3995 (23)	1044 (9)	2·34 (33)				
C(2)	1076 (6)	5617 (24)	795 (10)	2·95 (36)	H(1)	124	686	49
C(3)	573 (6)	5629 (24)	940 (11)	3·25 (37)	H(2)	34	691	78
C(4)	350 (6)	4028 (27)	1277 (11)	3·73 (40)	H(3)	−6	46	134
C(5)	632 (6)	2423 (24)	1519 (11)	3·54 (39)	H(4)	45	120	180
C(6)	1163 (5)	2335 (21)	1397 (9)	2·33 (33)	H(5)	124	−46	198
C(7)	1436 (6)	680 (24)	1655 (10)	2·86 (35)	H(6)	231	−107	267
C(8)	2172 (5)	−1294 (21)	1937 (9)	2·27 (31)	H(7)	190	−248	188
					H(8)	248	−240	72
C(9)	2610 (5)	−1733 (22)	1391 (10)	2·50 (32)	H(9)	289	−267	178
C(10)	3336 (5)	335 (22)	1414 (10)	2·32 (33)	H(10)	357	−87	166
C(11)	3573 (5)	2099 (23)	1267 (9)	2·17 (28)				
C(12)	4128 (6)	2131 (28)	1557 (11)	3·64 (37)	H(11)	431	89	188
C(13)	4375 (6)	3790 (27)	1397 (11)	3·81 (41)	H(12)	479	384	161
C(14)	4148 (6)	5370 (23)	995 (11)	2·99 (37)	H(13)	437	660	87
C(15)	3607 (6)	5361 (24)	723 (10)	2·90 (35)	H(14)	340	664	44
C(16)	3335 (5)	3664 (20)	844 (9)	1·71 (28)				