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N-Acetyl-L-cysteine의 결정 및 분자구조

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The Crystal and Molecular Structure of N-Acetyl-L-cysteine

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요 약. N-Acetyl-L-cysteine, $C_5H_9NO_3S$, 의 결정 및 분자구조를 X-선 회절법으로 연구하였다. 이 화합물의 결정은 삼사비등축정계에 속하며 공간군은 $P1$이다. 단위세포상수는 $a=7.04(3)$, $b=5.14(2)$, $c=8.25(3)$Å, $\alpha=106(2)$, $\beta=51(1)$, $\gamma=124(2)°$이고, 단위세포는 분자 한개만을 포함하고 있다. Weissenberg 사진촬영으로 얻은 회절반점의 총수는 629개이며 direct method를 적용하여 구조해명을 한 후 full matrix least-squares method로 정밀화하였으며 최종 R값은 0.12이다. C-카르복실기 및 N-아세틸기의 원자들은 각각 평면을 이루고 있다. 카르복실기의 산소원자 O(1)은 이웃분자의 아세틸기의 산소원자 O(3)와 O—H···O 수소결합을 하며 그 길이는 2.59Å이다.

ABSTRACT. The crystal struture of N-acetyl-L-cysteine, $C_5H_9NO_3S$, has been determined from three dimensional photographic intensity data (Cu$K\alpha$ radiation) by single crystal X-ray diffraction analysis. There is one formula unit in the triclinic unit cell with $a=7.04(3)$, $b=5.14(2)$, $c=8.25(3)$Å, $\alpha=106(2)$, $\beta=51(1)$, $\gamma=124(2)°$ and space group $P1$, The structure was solved by he direct method and refined by the full matrix least-squares method. The final R value is 12.3% for 629 observed reflections. The C-carboxyl group and the N-acetyl group are very neary planar. The molecule appears to form with neighboring molecules a hydrogen bond, O(1)-H···O(3) of length 2.59Å.

INTRODUCTION

As part of a study of the electronic structure and chemical bonding of sulfur-contanining compounds,[1~3] the structure determination of N-acetyl-cysteine, a N-acyl derivative of cysteine which often occurs in proteins in its form cystine, was undertaken by the X-ray diffraction method. N-Acetyl-L-cysteine was prepared by Smith and Gorin[4] and is used in treatment of respiratory diseases.

EXPERIMENTAL

N-Acetyl-L-cysteine in powder form was obtained from the German Merk Company. White parallel piped crystals were grown by slow evaporation of a saturated aqueous solution at room temperature.

Weissenberg photographs indicated the triclinic space group $P1$. Accurate cell parameters were obtained by least-squares treatment of the 20 values of high-angle reflections.

Intensity data were collected from equi-inclination Weissenberg photographs taken with Cu$K\alpha$ radiation by use of the multiple-film method. The layers from $0kl$ to $5kl$ for the a axis, $h0l$ to $h2l$ for the b axis, and from $hk0$ to $hk1$ for the c axis were recorded. The approximate sizes of the crystals used for data collection for the a, b and c axes were $0.5\times 0.3\times 0.2$, $0.2\times 0.4\times 0.2$, and $0.2\times 0.2\times 0.3$ mm respectively.

Intensities of 629 independent reflections were measured visually.

The crystal density was measured by a flotation technique using acetyl acetate and carbon tetrachloride. The crystal data are given in *Table* 1.

STRUCTURE SOLUTION AND REFINEMENT

The intensities were corrected for the Cu$K\alpha_1$ and Cu$K\alpha_2$ separation of high-order reflection spots, for the Lorentz-polarization effects and for the spot-shape differences which are necessary for the extension or contraction of spots on upper-level Weissenberg photographs. But they were not corrected for absoption.

An overall temperature factor ($2B=3.35$) and scale factor were evaluated by the Wilson method[5] and used to compute the normalized structure factors[6], $E(hkl)$. The statistics of the distribution of the normalized strure factors suggested an acentric structure.

The structure was solved by the direct method with the program MULTAN[7], employing the 121 E values larger than 1.30. The solution came from the set having the highest combined figure of merit. The positions of five of the 10 independent non-hydrogen atoms were revealed from the E map, the remaining atoms were located from a difference Fourier map.

The coordinates of all the atoms thus obtained were refined by the full matrix least-squres method using SHELX-76 computer program.[8] After three cycles of isotropic full matrix least-squares refinement, the crystallographic reliability value R was 16.3 %. Two further cycles of anisotropic refinement gave the final R value 12.3% for all the 629 reflction data. The final values of the atomic parameters and their estimated standard deviations are listed in *Table* 2. The observed and calculated structure factors are compared in *Table* 3. All the calculations were carried out on the SYBER 73 at KAERI and the SYBER 174 computer at KIST.

Table 1. X-ray crystal data for N-acetyl-L-cysteine.

Molecular formula: $C_5H_9NO_3S$	$Z=1$
M. W. : 163.20	$F(000)=86$
Unit cell parameters:	$D_c=1.42$ g/cc
$a=7.04(3)$ Å	$D_m=1.43$ g/cc
$b=5.14(2)$	μ(Cu$K\alpha$)$=33.86$
$c=8.25(3)$	Number of intensity data: 629
$\alpha=106(2)°$	Radiation : Cu$K\alpha$ 1.5418Å
$\beta=51(1)$	Meassurement mode: Visual method from X-ray photograph
$\gamma=124(2)$	
Crystal systal system and space group: triclinic, $P1$	Corrections: Lorentz, polarization

Table 2. Fractional atomic coordinates and and anisotropic thermal parameters ($\times 10^3$). The estimated standard deviations are given in parentheses. Temperature factors expression:

$$\exp\left[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)\right]$$

	X	Y	Z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S	1133	43	498	36(3)	16(3)	20(2)	−2(2)	−18(2)	13(2)
C(1)	1015(4)	297(5)	525(3)	19(10)	12(9)	32(10)	−8(8)	−21(9)	9(8)
C(2)	712(4)	100(4)	578(3)	17(9)	0(8)	14(8)	8(7)	−13(7)	−8(7)
C(3)	490(4)	−75(5)	797(3)	22(10)	0(8)	12(9)	−1(7)	−8(8)	−10(7)
C(4)	820(5)	39(3)	225(3)	27(11)	7(8)	18(9)	−1(7)	−10(8)	1(7)
C(5)	784(5)	−190(5)	66(3)	31(11)	25(11)	14(9)	5(8)	−18(9)	4(9)
N	697(4)	−86(4)	410(2)	35(10)	5(7)	14(8)	6(6)	−19(7)	−2(7)
O(1)	316(3)	−395(3)	803(2)	36(8)	0(6)	20(7)	6(5)	−17(7)	−18(6)
O(2)	463(4)	38(4)	955(3)	58(12)	15(9)	17(7)	−4(6)	−20(8)	−4(8)
O(3)	955(4)	329(3)	185(2)	48(10)	0(6)	14(7)	11(6)	−12(7)	−10(6)

Table 3. Observed and calulated structure factors. Columns are: Index, $|F_{obs}|$, $|F_{cal}|$.

H	K	L	$10F_0$	$10F_C$	H	K	L	$10F_0$	$10F_C$	H	K	L	$10F_0$	$10F_C$	H	K	L	$10F_0$	$10F_C$
0	1	0	269	300	0	4	1	35	31	0	5	-1	40	43	1	2	5	81	90
0	2	0	204	141	0	4	2	87	69	0	5	-2	51	71	1	2	6	71	82
0	3	0	184	131	0	4	3	113	102	1	1	0	94	79	1	2	7	24	31
0	4	0	125	119	0	4	4	70	92	1	2	0	154	138	1	2	8	36	34
0	5	0	49	48	0	4	5	65	53	1	3	0	113	101	1	3	1	71	59
0	0	1	107	105	0	4	6	44	43	1	4	0	83	81	1	3	2	122	106
0	0	2	150	120	0	5	1	47	57	1	0	2	250	256	1	3	3	178	171
0	0	3	202	199	0	5	2	47	46	1	0	3	205	214	1	3	4	103	98
0	0	4	137	111	0	5	3	25	36	1	0	4	83	79	1	3	5	20	24
0	0	5	38	39	0	1	-1	137	127	1	0	5	84	82	1	3	6	66	76
0	0	6	29	35	0	1	-2	232	268	1	0	6	206	220	1	3	7	35	35
0	0	7	107	83	0	1	-3	165	143	1	0	7	28	28	1	4	1	96	82
0	1	1	213	199	0	1	-4	65	69	1	0	8	52	54	1	4	2	113	106
0	1	2	107	93	0	1	-5	82	78	1	0	0	92	65	1	4	3	86	84
0	1	3	145	124	0	1	-6	46	50	1	0	-1	259	311	1	4	4	77	71
0	1	4	146	134	0	1	-7	35	28	1	0	-2	92	72	1	4	5	25	27
0	1	5	26	30	0	2	-1	212	143	1	0	-3	93	95	1	4	6	59	73
0	1	6	37	33	0	2	-2	122	116	1	0	-4	109	104	1	1	-1	77	70
0	1	7	73	63	0	2	-3	93	92	1	0	-5	88	91	1	1	-2	39	42
0	2	1	105	80	0	2	-4	101	120	1	0	-6	19	24	1	1	-3	98	100
0	2	2	117	79	0	2	-5	32	32	1	0	-7	66	62	1	1	-4	29	39
0	2	3	66	46	0	2	-6	43	50	1	1	1	212	157	1	1	-5	74	76
0	2	4	45	49	0	2	-7	45	41	1	1	2	160	149	1	1	-6	80	75
0	2	5	27	35	0	3	-1	57	51	1	1	3	205	183	1	2	-1	129	116
0	2	6	57	48	0	3	-2	117	114	1	1	4	166	165	1	2	-2	31	27
0	2	7	53	50	0	3	-3	161	158	1	1	5	70	62	1	2	-3	58	68
0	3	1	94	64	0	3	-4	80	95	1	1	6	129	130	1	2	-4	36	45
0	3	2	111	95	0	3	-5	33	40	1	1	7	49	49	1	2	-5	60	64
0	3	3	39	43	0	3	-6	76	71	1	1	8	35	36	1	3	-1	66	68
0	3	4	110	96	0	4	-1	48	49	1	2	1	79	44	1	3	-2	77	77
0	3	5	90	88	0	4	-2	108	100	1	2	2	173	143	1	3	-4	56	53
0	3	6	47	36	0	4	-3	52	60	1	2	3	139	116	1	4	-1	74	86
0	3	7	76	64	0	4	-4	86	85	1	2	4	93	81	1	4	-2	18	9

1	4	-3	29	40	2	0	9	52	53	2	2	-3	86	82	3	0	9	58	57
1	-1	-4	27	25	2	0	0	35	24	2	2	-4	42	42	3	-1	3	71	125
1	-1	-5	140	153	2	1	0	161	170	2	3	-1	59	73	3	-1	4	89	96
1	-1	-7	18	23	2	2	0	42	37	2	3	-2	29	42	3	-1	6	50	49
1	-2	-5	98	100	2	3	0	15	18	2	3	-3	63	66	3	-1	7	139	156
1	-2	-6	45	45	2	4	0	23	26	2	-1	-3	72	73	3	-1	8	24	32
1	-2	-7	38	31	2	0	-1	158	153	2	-1	-4	44	39	3	-1	9	40	45
1	-3	-6	67	55	2	0	-2	121	113	2	-1	-6	76	65	3	-2	3	149	152
1	-4	-5	61	47	2	0	-4	42	55	2	-2	-4	51	50	3	-2	4	129	114
1	-1	4	90	99	2	0	-5	17	19	2	-2	-5	50	39	3	-2	5	95	94
1	-1	5	129	127	2	0	-6	42	44	2	-2	-6	62	57	3	-2	6	88	101
1	-1	6	95	94	2	-2	3	179	209	2	-3	-5	39	32	3	-2	7	57	69
1	-1	7	67	58	2	-2	4	94	97	2	-4	-5	18	13	3	-2	8	46	58
1	-1	0	221	216	2	-2	5	54	58	2	-1	0	15	19	3	1	1	36	36
1	-2	0	124	109	2	-2	6	71	73	2	-2	0	148	146	3	1	2	134	105
1	-3	0	87	67	2	-1	3	101	69	2	-3	0	119	117	3	1	3	163	134
1	-4	0	90	83	2	-1	4	162	149	2	-4	0	45	50	3	1	4	139	138
1	-5	0	33	38	2	-1	5	94	79	2	-2	1	154	187	3	1	5	82	73
1	-1	-1	138	154	2	-1	6	73	77	2	-2	2	84	74	3	1	6	108	122
1	-1	-2	148	129	2	-1	7	77	73	2	-3	1	144	131	3	1	7	84	95
1	-1	-3	60	41	2	-1	8	59	58	2	-3	2	150	133	3	1	8	29	31
1	-2	-1	91	92	2	1	1	208	168	2	-3	3	119	116	3	1	9	64	66
1	-2	-2	123	124	2	1	2	77	60	2	-4	1	73	76	3	2	1	89	94
1	-2	-3	110	131	2	1	3	149	151	2	-4	2	112	96	3	2	2	76	66
1	-2	-4	37	44	2	1	4	21	21	2	-4	3	94	93	3	2	3	109	97
1	-3	-1	154	161	2	1	5	90	78	2	-5	1	94	93	3	2	4	148	145
1	-3	-2	98	118	2	1	6	21	15	2	-5	2	45	50	3	2	5	95	100
1	-3	-3	80	94	2	1	7	63	74	2	-1	-2	124	100	3	2	6	23	24
1	-3	-4	58	70	2	1	8	64	58	2	-2	-1	90	90	3	2	7	64	64
1	-3	-5	74	80	2	1	9	71	67	2	-2	-2	96	82	3	2	8	30	28
1	-4	-1	106	111	2	2	1	153	115	2	-2	-3	102	109	3	3	1	46	51
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3	-5	-2	44	43	4	2	8	37	38	5	0	7	47	58	5	-5	4	47	50
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3	0	-1	35	36	4	3	3	12	18	5	-1	0	39	43	5	-6	2	48	57
3	0	-2	66	65	4	3	4	12	15	5	-1	1	88	78	5	-3	-1	29	25
3	0	-3	19	17	4	1	-1	81	85	5	-1	2	82	67	5	-3	-2	16	23
3	0	-4	29	32	4	1	-2	24	26	5	-1	3	76	83	5	-4	-1	17	16
4	0	0	32	22	4	2	-1	42	43	5	-1	4	21	24	5	-4	-2	13	21
4	1	0	36	38	4	-1	-1	82	77	5	-1	5	101	82	5	-5	-1	38	40
4	2	0	55	61	4	-1	-2	18	23	5	-1	6	70	57	5	-1	-2	26	29
4	0	1	67	63	4	-1	-3	37	42	5	-1	7	19	26	5	-2	-1	43	45
4	0	2	67	50	4	-1	-4	19	28	5	-1	8	79	70	5	-2	-2	25	28
4	0	3	28	23	4	-2	-2	71	70	5	-1	9	43	42	1	0	1	98	131
4	0	4	142	124	4	-2	-3	54	49	5	-2	5	48	81	6	0	1	29	20
4	0	5	53	56	4	-2	-4	31	40	5	-2	6	41	50	6	0	2	16	16
4	0	6	75	71	4	-3	-3	48	44	5	-2	7	53	51	6	0	3	57	41
4	0	7	66	73	4	-3	-4	33	39	5	-2	8	83	90	6	0	4	50	42
4	0	8	69	79	4	-2	0	52	50	5	-2	9	39	41	6	0	5	30	36
4	0	9	21	18	4	-3	0	60	54	5	-3	5	110	112	6	0	6	58	57
4	0	-1	48	47	4	-4	0	51	43	5	-3	6	69	63	6	0	7	71	57

6	0	9	49	45	-3	1	2	125	109	-1	2	-5	50	46	-6	2	-2	31	35
7	0	5	25	17	-6	1	-2	31	41	-1	2	-6	64	65	-6	2	-3	60	38
7	0	6	49	36	-6	1	-3	35	38	-1	2	-7	100	80	-6	2	-4	65	59
7	0	7	19	18	-6	1	-4	35	44	-1	2	-8	43	50	-6	2	-5	71	71
7	0	8	22	26	-6	1	-5	76	50	-2	2	-7	69	73	-6	2	-6	73	82
0	1	3	13	16	-6	1	-6	38	56	-2	2	-8	18	12	-6	2	-7	102	95
6	1	4	67	73	-6	1	-7	55	72	-3	2	-9	94	72	-6	2	-8	107	104
6	1	5	17	30	-6	1	-8	61	70	-4	2	-10	68	58	-7	2	-3	51	45
6	1	6	18	35	-7	1	-3	19	20	-5	2	-10	44	38	-7	2	-4	53	43
6	1	7	45	50	-7	1	-4	42	50	-6	2	-9	69	63	-7	2	-5	26	36
-1	1	-3	180	174	-7	1	-5	20	24	-6	2	-10	72	64	-7	2	-6	31	27
-1	1	-8	109	94	-7	1	-6	24	25	-7	2	-9	29	28	-7	2	-7	46	48
-2	1	-9	65	51	-7	1	-7	21	32	-7	2	-10	43	47	-7	2	-8	22	31
-4	1	-10	31	33	-7	1	-8	34	37	-2	2	-1	250	179	-8	2	-5	17	23
-5	1	-10	26	41	-7	1	-9	31	27	-2	2	-2	112	73	-8	2	-6	50	46
-6	1	-9	44	57	-1	2	-2	34	5	-4	2	-3	83	65	-8	2	-7	28	33
-6	1	-10	12	11	-1	2	-3	119	88	-5	2	-4	163	108	-8	2	-8	38	37
-2	1	-1	299	418	-1	2	-4	75	59	-6	2	-1	32	35	-8	2	-9	49	52
-2	1	-2	68	62															

RESULTS AND DISCUSSION

The bond lengths and angles are given in *Table* 4 and *Fig.* 1 with atomic numbering.

The C(1)-S bond length of 1.80 Å agrees well with a mean value of 1.81 Å for the C-S single bond reported in L-cystine dihydrobromide dihydrate.[9]

The C(1)-C(2) bond length of 1.56 Å is in good agreement with the value for the corresponding bond in 3,3,3′,3′-tetramethyl-**D**-cystine dihydrochloride.[10]

The C(2)-N bond length of 1.48Å agrees well with a mean value of 1.481 Å for the corresponding bond in 3,3,3′,3′-teramethyl-**D**-cystine dihydrochloride.[10]

The N-C(4), C(4)-O(3), C(4)-C(5) bond lengths of N-acetyl group agree with the values in related compounds.[11,12] The N-C(4) bond length of 1.32 Å, which is significantly shorter than the corresponding normal single bond length of 1.47 Å is indicative of 50 % double bond character by Wheatley's order/length curve[13] for C-N bonds. The N, C(4), C(5), and O(3) atoms of N-acetyl group lie in a plane (see *Table* 5).

Table 4. Bond lengths and angles in N-acetyl-L-cysteine. The estimated standard deviations are shown in parentheses.

Bond length (Å)		Bondangle(°)	
C(1)-S	1.80(2)	C(2)-C(1)-S	111(1)
C(1)-C(2)	1.56(2)	C(1)-C(2)-C(3)	111(2)
C(2)-C(3)	1.51(2)	C(1)-C(2)-N	115(2)
C(2)-N	1.48(2)	C(3)-C(2)-N	116(1)
C(3)-O(1)	1.39(2)	C(2)-C(3)-O(1)	121(2)
C(3)-O(2)	1.20(2)	C(2)-C(3)-O(2)	126(2)
C(4)-C(5)	1.54(3)	O(1)-C(3)-O(2)	113(1)
C(4)-O(3)	1.24(2)	C(5)-C(4)-N	
C(4)-N	1.32(2)	O(3)-C(4)-N	120(2)
		O(3)-C(4)-C(5)	123(1)
		C(2)-N -C(4)	124(1)

The C(2), C(3), O(1), and O(2) atoms of C(2)-carboxyl group are in a plane within 0.01 Å. The C(2)-C(3), C(3)-O(1), C(3)-O(2) bond lengths are in agreement with those found in C-carboxyl group of L-cystine dihydrobromide dihdrate.[9] The difference between the C(3)-O(1) and C(3)-O(2) bond lengths of 1.39 and 1.20Å indicates that there is ap-

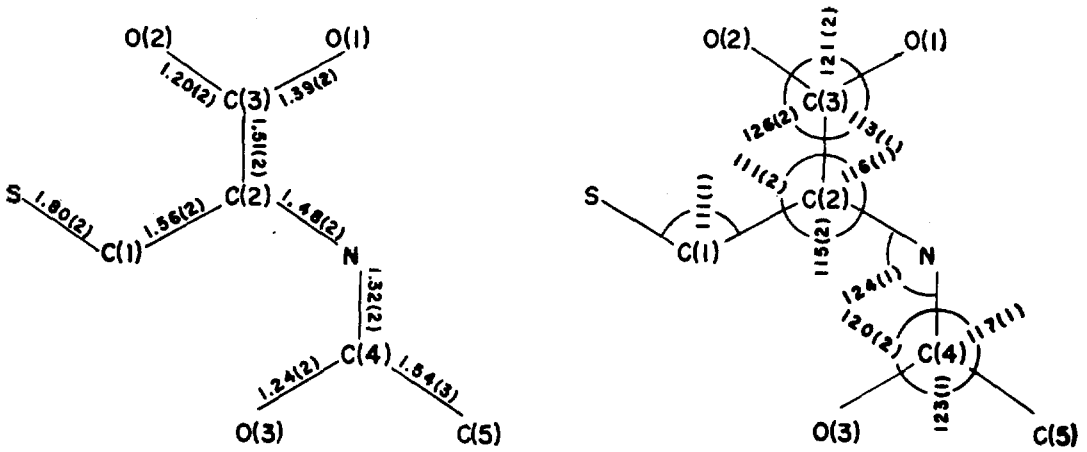

Fig. 1. Bond lengths (Å) and bond angles(°) in N-acetyl-L-cysteine. The estimated stand deviations are shown in parentheses

Table 5. Least-squares planes in N-acetyl-L-cysteine. (Equation for platne: $Ax+By+Cz=D$, where x, y, z are in Å).

Atoms in plane	Atoms out of plane	Distance from best plane(Å)	Constant
A. C-carboxy group			
C(2)		0.00	$A=$ 0.771
C(3)		−0.01	$B=-0.570$
O(1)		0.00	$C=$ 0.376
O(2)		0.00	$D=$ 5.359
	N	−0.05	
	C(1)	0.91	
B. N-acetyl group			
C(4)		0.00	$A=$ 0.782
C(5)		0.00	$B=-0.446$
N		0.00	$C=$ 0.252
O(3)		0.00	$D=$ 4.889
	C(2)	0.00	

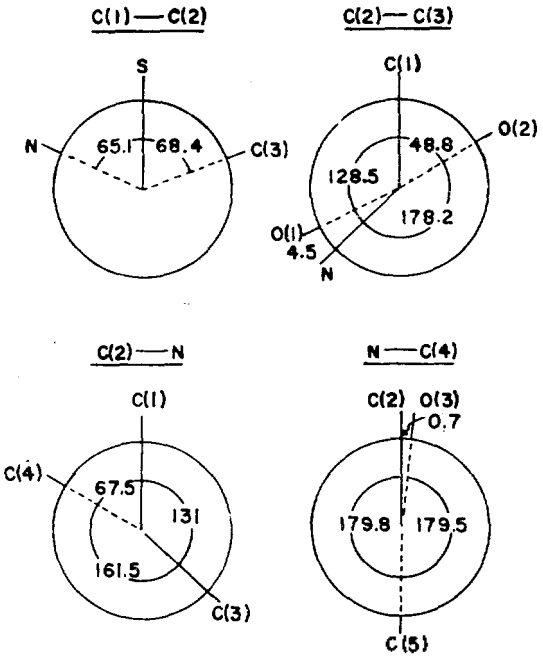

Fig. 2. Conformational angles in degrees in N-acetyl-L-cysteine.

parently little delocalization within this carboxyl group. Also the O(1) atom participates as donor in an O—H···O hydrogen bond to the oxygen atom of the acetyl group of the neighboring molecule, the O···O distance being 2.59 Å.

The detailed conformational angles are given in *Fig.* 2. The conformational angle formed by the C(2)-C(3) bond linked to the carboxyl group with the N-C(4) bond of N-acetyl group, when the projection is taken along the C(2)-N is 161.5°.

A stereoscopic view of the crystal strucure looking down the c axis is shown in *Fig.* 3. The molecular packing is govrned by van der Waals forces and hydrogen bonds.

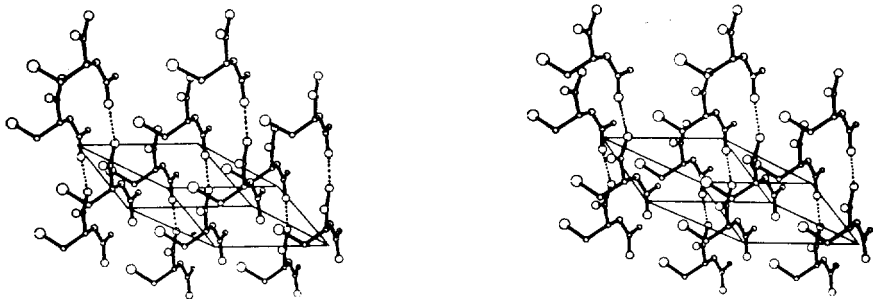

Fig. 3. A stereoplot of the unit-cell contents, looking down the *c* axis. Dotted lines indicate hydrogen bonds. Large circles are S. The figure has been drawn by the program ORTEP.[14]

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