Acta Cryst. (1972). **B28**, 1519

The Crystal Structure of Dimethyl 1,3-Dimethyl-8-thia-2-azabicyclo[3,2,1]oct-3-ene-4,7-dicarboxylate

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(*Received* 5 *August* 1971)

The stereochemistry of an unusual bicyclic system containing sulfur ($C_{12}H_{17}NO_4S$) has been established by single-crystal X-ray analysis. The compound crystallizes in the monoclinic space group $P2_1/c$, with $a = 11{\cdot}076 \pm 0{\cdot}002$, $b = 8{\cdot}341 \pm 0{\cdot}002$, $c = 14{\cdot}799 \pm 0{\cdot}003$ Å and $\beta = 103{\cdot}07 \pm 0{\cdot}10°$. There are four molecules per unit cell. The 2148 X-ray intensities were collected on an automatic diffractometer and were refined by least-squares methods on F values to an agreement value, R, of 0·053. The structure was solved by the symbolic-addition procedure. The molecule contains a seven-membered ring, made up of two essentially planar segments which are bridged by the sulfur atom. The X-ray analysis showed that the ester group on C(7) is *trans* to the sulfur bridge.

Introduction

When the dihydropyridine (I) is treated with potassium hydrosulfide, it rearranges to form a bicyclic system containing sulfur (IV). The mechanism put forth for the reaction (Ashby & Eisner, 1967) involves an intermediate azepine derivative (II) reacting with the SH⁻ ion to give (III), which then undergoes internal Michael addition to give (IV).

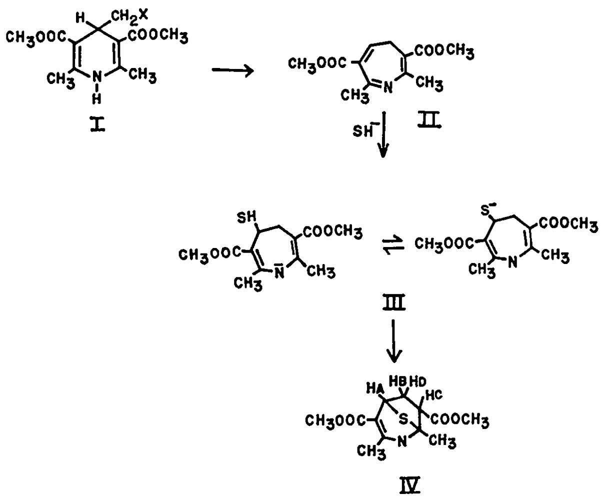

Table 1. *Crystal data*

Molecular formula	$C_{12}H_{17}NO_4S$
Molecular weight	271·33
Habit	Colorless rectangular prism
Crystal size	~(0·38 × 0·31 × 0·11 mm)
Space group	$P2_1/c$
a	11·076 ± 0·002 Å
b	8·341 ± 0·002
c	14·799 ± 0·003
β	103·07 ± 0·10°
Molecules per unit cell	4
Density (calculated)	1·35 g.cm^{-3}
No. of independent reflections	2148
Radiation	Cu $K\alpha$ (1·5418 Å) Ni filter.

Ashby & Eisner predicted that the ester group on C(7) was *trans* to the sulfur bridge; however, some nuclear magnetic resonance indications and certain chemical reactions involving (IV) seemed to favor a *cis* configuration for the ester group (see references in Eisner, Haq, Flippen & Karle, 1971). The X-ray study was undertaken to establish the stereochemistry of the molecule, and it showed the ester group to be *trans* to the sulfur bridge.

Experimental

The crystals used in the X-ray analysis were provided by Professor U. Eisner of Howard University. Inten-

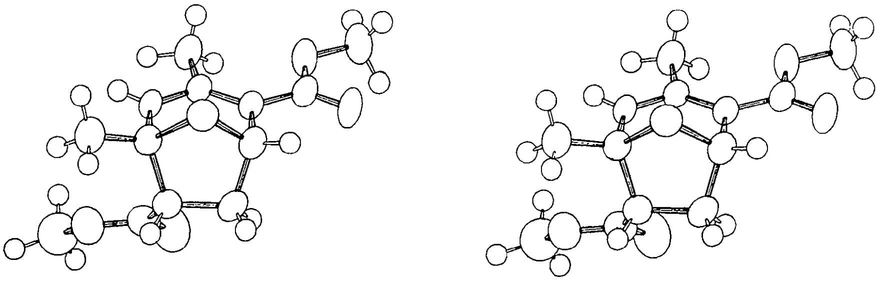

Fig. 1. A stereodrawing of the molecule including the refined positions of the hydrogen atoms.

Table 2. *Fractional coordinates and thermal parameters with standard deviations*

The thermal parameters are of the form:

$$T=\exp[-\tfrac{1}{4}(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^*+2B_{13}hla^*c^*+2B_{23}klb^*c^*)].$$

The B_{ij}'s are in Å² units. Standard deviations are based solely on least-squares parameters.

(*a*) Heavy atoms

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	0·30866 (26)	0·32848 (34)	0·30741 (20)	4·06 (13)	3·45 (12)	3·60 (12)	−0·27 (10)	1·25 (10)	0·07 (10)
N(2)	0·18248 (20)	0·26999 (28)	0·29516 (15)	3·85 (10)	4·01 (10)	2·54 (9)	−0·12 (8)	1·22 (8)	−0·04 (8)
C(3)	0·10409 (25)	0·24502 (35)	0·21157 (19)	3·73 (11)	3·88 (12)	3·13 (12)	0·24 (10)	1·22 (10)	−0·01 (10)
C(4)	0·14043 (25)	0·27877 (36)	0·13040 (19)	3·88 (11)	4·47 (13)	2·99 (11)	0·32 (10)	1·29 (9)	0·29 (10)
C(5)	0·27021 (27)	0·34036 (39)	0·13609 (21)	4·40 (13)	5·29 (15)	3·73 (13)	−0·13 (11)	1·88 (11)	0·56 (11)
C(6)	0·37121 (31)	0·21492 (45)	0·16744 (23)	4·51 (14)	5·82 (18)	4·36 (15)	0·36 (13)	2·08 (12)	−0·22 (13)
C(7)	0·39051 (29)	0·19846 (34)	0·27408 (21)	3·90 (13)	3·79 (13)	4·13 (13)	0·14 (10)	1·05 (11)	0·03 (10)
S(8)	0·30874 (8)	0·49468 (9)	0·22605 (6)	4·65 (4)	3·28 (3)	4·79 (4)	−0·43 (3)	1·65 (3)	0·65 (3)
C(9)	0·35508 (29)	0·03319 (37)	0·30301 (24)	4·64 (14)	3·80 (14)	4·96 (16)	0·41 (11)	0·67 (12)	−0·16 (12)
C(10)	0·34302 (46)	−0·12544 (49)	0·43472 (34)	8·17 (24)	4·78 (17)	7·72 (23)	−0·65 (17)	2·53 (20)	1·02 (16)
C(11)	0·06581 (30)	0·25821 (41)	0·03816 (21)	5·57 (16)	5·30 (15)	3·27 (13)	1·03 (13)	1·74 (12)	0·17 (12)
C(12)	−0·12531 (42)	0·17926 (61)	−0·06144 (24)	6·46 (20)	8·21 (25)	3·24 (14)	−0·11 (18)	−0·12 (14)	−0·52 (15)
C(13)	0·35597 (35)	0·38522 (47)	0·40667 (22)	5·55 (17)	4·75 (16)	4·07 (13)	−0·45 (14)	0·67 (12)	−1·00 (12)
C(14)	−0·01922 (29)	0·18021 (49)	0·22034 (23)	4·23 (14)	6·66 (19)	3·55 (13)	−0·58 (12)	1·56 (11)	−0·13 (13)
O(15)	0·30508 (27)	−0·06960 (30)	0·25208 (20)	9·29 (17)	4·31 (11)	6·48 (15)	−1·33 (11)	−0·06 (13)	−0·96 (11)
O(16)	0·38562 (22)	0·01755 (26)	0·39509 (16)	6·09 (12)	3·96 (10)	5·02 (11)	−0·39 (9)	1·08 (9)	0·47 (8)
O(17)	0·10382 (24)	0·28939 (37)	−0·03168 (15)	6·48 (13)	10·90 (18)	2·95 (10)	0·38 (12)	2·08 (9)	0·51 (10)
O(18)	−0·04966 (21)	0·20363 (30)	0·03106 (14)	5·14 (11)	7·31 (13)	2 90 (9)	−0·61 (10)	0·69 (8)	−0·04 (9)

sity data were collected at room temperature on a four-circle computer-controlled automatic diffractometer using the θ–2θ technique with a $2{\cdot}0°+2\theta(\alpha_2)$–$2\theta(\alpha_1)$ scan over 2θ. Physical quantities for this molecule are listed in Table 1. Lorentz and polarization corrections were applied, and normalized structure factor magnitudes $|E|$ as well as structure factor magnitudes $|F|$ were derived.

The structure was solved using the symbolic addition procedure for centrosymmetric crystals (Karle & Karle, 1963, 1966). The implementation of the $\sum_2$ formula was facilitated by means of a computer program written by R. D. Gilardi of this laboratory.

The structure was refined using full-matrix least-squares methods (Busing, Martin & Levy, 1962) with the weighting function: $1/W=1{\cdot}0+(|F_o|/35)^2$, which was suggested by a statistical examination of the least-squares discrepancies. This weighting function was used for all the data; no reflections were considered unobserved. The function minimized was $\sum(F_o-F_c)^2$, and the atomic scattering factors used were those listed in *International Tables for X-ray Crystallography* (1962). No absorption corrections were applied due to the low value for the linear absorption coefficient ($\mu=22{\cdot}12$ cm^{-1}) and the small size of the crystal. All the hydrogen atoms were located in a difference map and their positions were refined. The final R value for the full set of data was 0·053. Table 2 lists the coordinates and thermal parameters of each atom. The observed and calculated structure factors are compared in Table 3.

Table 2 (*cont.*)

(*b*) Hydrogen atoms

(The hydrogen-atom thermal parameters were not refined; each was assigned the thermal parameters of its adjoining atom.)

	x	y	z
H(N2)	0·1566 (31)	0·2587 (43)	0·3461 (24)
H(5)	0·2821 (33)	0·3957 (48)	0·0785 (26)
H(6*a*)	0·4462 (36)	0·2518 (52)	0·1514 (26)
H(6*b*)	0·3400 (36)	0·1101 (54)	0·1317 (28)
H(7)	0·4834 (34)	0·2161 (44)	0·3068 (26)
H(10*a*)	0·3884 (45)	0·8484 (54)	0·5069 (36)
H(10*b*)	0·2514 (47)	0·8894 (54)	0·4308 (33)
H(10*c*)	0·3515 (46)	0·7873 (58)	0·4032 (36)
H(12*a*)	−0·0740 (43)	0·1100 (63)	−0·0955 (29)
H(12*b*)	−0·2068 (44)	0·1336 (61)	−0·0562 (28)
H(12*c*)	−0·1324 (42)	0·2874 (63)	−0·0950 (30)
H(13*a*)	0·3052 (39)	0·4723 (53)	0·4229 (27)
H(13*b*)	0·4472 (41)	0·4160 (50)	0·4148 (26)
H(13*c*)	0·3510 (37)	0·2921 (53)	0·4552 (28)
H(14*a*)	−0·0116 (33)	0·1361 (53)	0·2854 (28)
H(14*b*)	−0·0478 (34)	0·0982 (57)	0·1750 (27)
H(14*c*)	−0·0738 (37)	0·2607 (57)	0·2092 (28)

Discussion

The stereoconfiguration of the molecule is illustrated in Fig. 1 and shows the ester group on C(7) to be *trans* to the sulfur bridge. The seven-membered ring consists of two segments, both of which are essentially planar.

Table 3. *Observed and calculated structure factors*

The columns are the index h, $10|F_o|$, and $10|F_c|$.

H	0	0
1	124	146
2	561	-570
3	1356	1439
4	949	-956
5	506	-495
6	260	245
7	27	-0
8	59	-53
9	60	-62
10	105	107
11	22	-19
12	65	-74
H	0	2
-12	21	-10
-11	19	5
-10	197	-196
-9	17	-9
-8	209	212
-7	390	-380
-6	907	-880
-5	225	231
-4	169	-176
-3	880	-940
-2	1329	1590
-1	578	601
0	777	-900
1	764	847
2	112	100
3	331	-331
4	136	-147
5	26	-27
6	75	-0
7	128	-128
8	67	79
9	28	-29
10	198	-203
11	14	5
12	38	30
H	0	4
-12	14	2
-11	103	-103
-10	173	169
-9	65	69
-8	231	-228
-7	150	156
-6	197	175
-5	323	-339
-4	227	-227
-3	243	261
-2	573	597
-1	240	276
0	714	769
1	681	692
2	313	-324
3	568	548
4	461	457
5	315	-319
6	26	12
7	67	-51
8	286	-284
9	41	38
10	134	132
11	24	-33
H	0	6
-12	82	-84
-11	91	99
-10	51	-55
-9	222	-221
-8	56	-55
-7	165	-167
-6	314	-323
-5	62	74
-4	191	183
-3	478	-489
-2	18	15
-1	25	-2
0	461	-483
1	515	537
2	433	419
3	243	-234
4	16	29
5	111	114
6	47	44
7	19	-7
8	89	89
9	66	56
10	86	-83
H	0	8
-12	44	48
-11	99	-101
-10	150	-153
-9	144	134
-8	125	129
-7	125	117
-6	363	365
-5	181	-179
-4	257	-255
-3	141	146
-2	493	-502
-1	47	-29
0	47	-63
1	185	-170
2	256	247
3	36	27
4	355	348
5	82	72
6	276	-265
7	198	150
8	39	-31
9	134	-126
H	0	10
-11	1	2
-10	130	127
-9	97	-98
-8	34	-28
-7	102	107
-6	188	-204
-5	91	89
-4	108	116
-3	280	-307
-2	209	-215
-1	17	17
0	33	27
1	263	-265
2	129	124
3	242	231
4	191	-189
5	179	169
6	231	224
7	122	-111
8	29	27
H	0	12
-10	67	-70
-9	104	99
-8	67	62
-7	91	-103
-6	212	221
-5	185	201
-4	183	-187
-3	49	55
-2	34	-26
-1	592	-610
0	317	-316
1	48	52
2	59	-56
3	74	75
4	156	159
5	13	-19
6	72	-66
H	0	14
-9	81	-86
-8	17	-21
-7	291	293
-6	84	80
-5	57	-66
-4	99	112
-3	173	-195
-2	264	-265
0	28	19
1	50	-39
2	39	33
3	56	47
4	24	-19
H	0	16
-7	59	-61
-6	22	14
-5	58	54
-4	11	-12
-3	68	-66
-2	65	-66
-1	126	-116
0	135	-130
1	25	-9
H	1	0
1	1061	-1103
2	46	61
3	297	-309
4	143	149
5	342	354
6	112	118
7	209	225
8	75	67
9	64	70
10	128	126
11	28	-31
12	17	-9
H	1	1
-12	53	49
-11	53	-52
-10	129	-116
-9	187	166
-8	136	-129
-7	277	-277
-6	392	334
-5	150	-140
-4	182	-177
-3	243	246
-2	55	39
-1	157	151
0	302	-292
1	490	471
2	318	325
3	494	-527
4	378	415
5	110	105
6	215	-222
7	220	223
8	31	36
9	103	-104
10	46	45
11	28	33
12	46	-49
H	1	2
-12	32	30
-11	37	-33
-10	132	120
-9	59	67
-8	120	-117
-7	55	-47
-6	18	-15
-5	252	267
-4	97	84
-3	340	-351
-2	81	51
-1	753	-744
0	298	-283
1	373	369
2	246	-241
3	94	-112
4	256	-266
5	121	-115
6	171	168
7	7	-7
8	24	-12
9	34	19
10	28	29
11	32	34
H	1	3
-12	89	-89
-11	60	58
-10	92	84
-9	185	-176
-8	25	28
-7	138	137
-6	90	-77
-5	168	-167
-4	126	122
-3	247	254
-2	311	-302
-1	132	110
0	369	372
1	439	-448
2	62	-60
3	129	139
4	138	-150
5	30	26
6	105	101
7	50	-48
8	48	-49
9	69	74
10	58	-56
11	68	-74
H	1	4
-12	30	-24
-11	83	82
-10	29	4
-9	20	-18
-8	137	132
-7	54	-48
-6	157	-159
-5	237	-230
-4	426	-426
-3	31	30
-2	389	371
-1	7	-5
0	53	41
1	309	297
2	58	-54
3	239	-253
4	128	-118
5	77	-80
6	66	-66
7	159	153
8	302	297
9	227	220
10	47	33
11	13	11
H	1	5
-12	56	52
-11	6	-14
-10	64	-61
-9	20	-1
-8	57	84
-7	154	-152
-6	164	-174
-5	265	276
-4	54	-61
-3	190	-191
-2	492	378
-1	184	180
0	177	-166
1	103	85
2	48	45
3	138	-141
4	143	136
5	192	193
6	81	-75
7	116	113
8	61	61
9	97	-99
10	22	19
11	16	22
H	1	6
-12	14	15
-11	39	38
-10	73	60
-9	14	-4
-8	57	67
-7	201	200
-6	35	-28
-5	141	-151
-4	142	-135
-3	435	-427
-2	476	-468
-1	83	77
0	265	229
1	520	-516
2	492	-470
3	284	-285
4	234	-239
5	65	65
6	171	166
7	100	104
8	54	47
9	10	-25
10	8	14
H	1	7
-11	70	-73
-10	102	102
-9	25	29
-8	213	-218
-7	46	47
-6	21	18
-5	254	-264
-4	16	14
-3	260	255
-2	171	-154
-1	203	-188
0	210	203
1	253	-250
2	173	-168
3	287	285
4	87	-87
5	23	-14
6	123	124
7	37	-39
8	17	-21
9	11	-12
10	12	11
H	1	8
-12	24	22
-11	23	-23
-10	150	137
-9	356	353
-8	157	155
-6	129	-124
-5	254	-256
-4	59	-52
-3	125	-116
-2	171	149
-1	134	131
0	297	-268
1	147	139
2	81	-73
3	143	-146
4	79	77
5	106	-110
6	40	42
7	162	149
8	31	14
9	53	45
H	1	9
-12	38	42
-11	92	91
-10	87	-86
-8	70	71
-7	72	-79
-6	93	-92
-5	132	128
-4	86	83
-3	257	-239
-2	197	188
-1	151	137
0	333	-310
1	154	148
2	126	124
3	104	-96
4	68	62
5	87	87
6	27	13
7	59	-58
8	45	39
9	39	37
H	1	10
-11	89	85
-10	87	86
-9	61	-61
-8	21	18
-6	32	-33
-5	11	-1
-4	175	-173
-3	94	-90
-2	90	-92
-1	123	-119
0	177	-174
1	351	-352
2	68	-57
3	27	-18
4	83	-77
5	145	152
7	82	-82
8	44	29
H	1	11
-11	43	-50
-10	46	43
-9	14	23
-8	134	-140
-7	7	-3
-6	115	122
-5	220	-232
-4	59	-59
-3	236	228
-2	231	-228
-1	143	-140
0	190	187
1	35	-31
2	120	-117
3	69	63
4	60	57
5	103	-99
6	14	-3
7	72	70
H	1	12
-10	46	47
-9	166	158
-8	42	42
-7	92	96
-6	199	221
-5	43	-37
-4	14	3
-2	281	-282
-1	204	-185
0	93	92
1	146	138
2	65	70
3	45	41
4	21	10
5	69	-69
6	31	-34
H	1	13
-10	66	-66
-9	39	-32
-8	187	189
-7	63	-65
-6	112	-116
-5	149	152
-4	30	-25
-3	141	-141
-2	77	70
-1	100	98
0	76	-71
1	39	-38
2	107	107
3	32	-21
4	58	-64
5	50	49
H	1	14
-9	23	-11
-8	119	123
-7	42	43
-6	87	-90
-5	36	-35
-4	108	-114
-3	96	-93
-2	24	20
-1	8	15
0	30	-21
1	44	-45
3	25	16
4	6	-9
H	1	15
-8	79	-83
-7	4	2
-6	39	35
-5	46	-44
-4	82	-84
-3	36	35
-2	4	15
-1	96	-101
0	42	44
1	51	54
2	71	-72
3	12	-3
H	1	16
-7	47	46
-6	61	66
-5	108	109
-3	76	-74
-2	14	-21
-1	40	-40
0	19	15
1	42	38
H	1	17
-3	64	-62
H	2	0
0	912	-893
1	1306	-1304
2	697	-698
3	724	740
4	259	274
5	102	-123
6	215	222
7	101	88
8	127	-140
9	57	60
10	53	54
11	61	-61
H	2	1
-12	13	12
-11	42	-39
-10	12	-7
-9	30	2
-8	215	-207
-7	178	168
-6	156	158
-5	581	-565
-4	27	12
-3	94	-109
-2	1278	-1263
-1	199	-190
0	377	350
1	156	-145
2	16	-18
3	177	182
4	68	67
5	127	-130
6	14	2
7	12	5
8	32	-28
9	25	23
H	2	2
-11	127	120
-10	58	59
-9	197	-188
-8	250	234
-7	230	227
-6	95	-105
-5	762	766
-4	39	45
-3	921	-910
-2	361	354
-1	398	-401
0	541	-513
1	121	104
2	42	34
3	151	-144
4	136	-141
5	340	360
6	22	9
7	212	-227
8	172	173
9	56	95
10	22	-14
11	65	70
H	2	3
-12	3	19
-11	36	27
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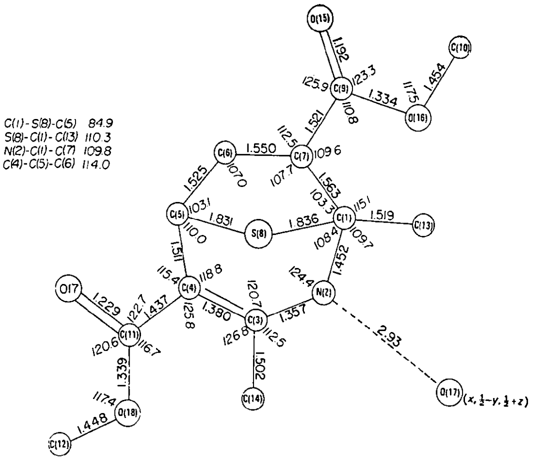

Fig. 2. Bond distances and angles. Standard deviations estimated from the least-squares refinement are ±0·002 – 0·003 Å (S–C and C–N bonds), ±0·002 – 0·004 Å (C–O and C–C bonds), and ±0·1 – 0·2° for the angles.

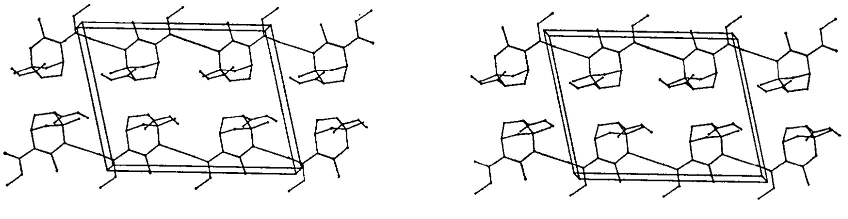

Fig. 3. A stereodrawing of the packing of the molecules, showing the hydrogen bonding. It is viewed looking down the *b* axis with *c*→ and *a*↑.

Segment I, formed by atoms C(5)–C(4)–C(3)–N(2)–C(1), is planar to within ±0·007 Å; segment II, formed by atoms C(1)–C(7)–C(6)–C(5), is planar to within ±0·032 Å. The dihedral angle between the segments is 66·5°. The sulfur atom bridges the two segments, forming a six-membered ring and a five-membered ring both in the envelope conformation. The ester groups on C(4) and C(14) are virtually coplanar with segment I (±0·017 Å).

The ester group on C(4) is planar (±0·007 Å) and the ester group on C(7) is essentially planar (±0·029 Å). In both cases, the methoxy groups have adopted a preferred orientation with respect to the remainder of the ester group. The methyl carbon atom turns toward the shorter bond (C=O) and rotates to minimize methyl hydrogen interaction with the rest of the ester group. One hydrogen atom is coplanar with, but directed away from, the double bond, while the other two are staggered above and below the plane of the ester group. The hydrogen atoms were readily located in a difference map and are illustrated in Fig. 1. A similar situation has been noted in several aromatic methoxy compounds (Karle & Karle, 1968; Yonemitsu, Nakai, Kanaoka, Karle & Witkop, 1969; Karle & Karle, 1971; Gilardi & Karle, 1971) in which the methoxy groups adopt this same *cis* orientation with respect to neighboring phenyl or quinoidal rings. In this molecule, the C(12)–O(17) and C(10)–O(15) distances are 2·64 and 2·68 Å respectively, compared to C–C separations of 2·77–2·82 Å in the molecules referred to above.

The bond distances and angles are illustrated in Fig. 2. The O(17)–C(11)–C(4)–C(3)–N(2) system is somewhat conjugated as evidenced by the shortening of the C(11)–C(4) and C(3)–N(2) single bonds (see Fig. 2). Due to the planarity of the system, consisting of segment I plus the ester group on C(4) and C(14), there are some close intramolecular C···O approaches: O(18)···C(14) = 2·75 Å, O(17)···C(5) = 2·77 Å, and O(17)···C(12) = 2·64 Å. However, there is some repulsion between the ester group and the methyl carbon atom [C(14)], which is illustrated by the C(11)–C(4)–C(3) and C(4)–C(3)–C(14) angles: 125·8 (±0·1) and 126·8 (±0·1)° respectively. The ester group on C(7) is not coplanar with segment II but is at an angle of 69·6° to it, and the O(16)–C(13) distance is 3·09 Å.

There is one intermolecular hydrogen bond between N(2) and O(17) (see Fig. 2). Fig. 3 illustrates the packing of the molecules and shows that the molecules are linked into infinite chains, along the **c** direction, by the hydrogen bond. The closest intermolecular approaches (other than the hydrogen bond) are O(15)–C(12), 3·20 Å; O(16)–O(16), 3·55 Å; C(11)–C(12), 3·71 Å.

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