Study of the Alkaline Hydrolysis of Ethyl 2-(1,2-benzisothiazolin-3-ylidene)cyanoacetate: Spectroscopic Properties, and *X*-Ray Crystal Analysis of 2,4-Bis-(1,2-benzisothiazol-3-yl)3-aminocrotononitrile–Dimethylformamide

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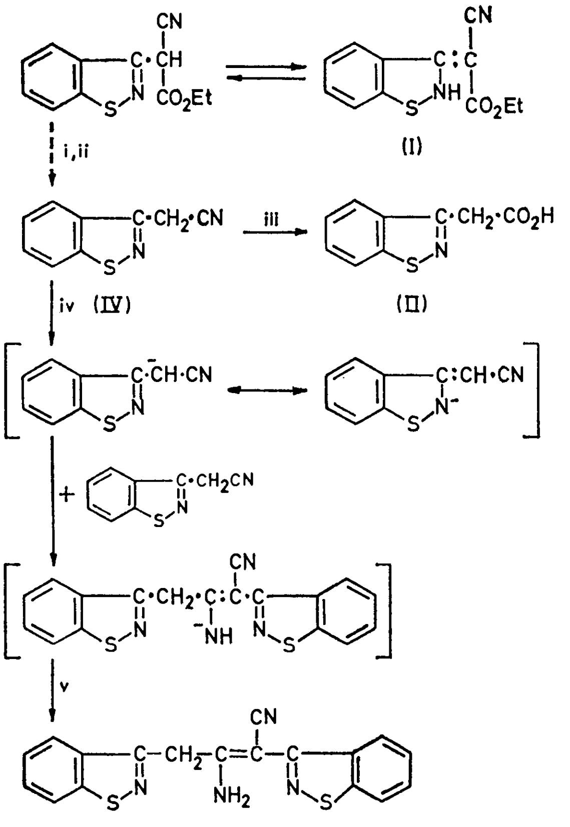
The i.r., u.v., n.m.r., and mass spectra, and *X*-ray crystal analyses of the title compound are given. Crystals are monoclinic with $a = 8{\cdot}85(1)$, $b = 12{\cdot}08(1)$, $c = 19{\cdot}52(1)$ Å, $\beta = 95{\cdot}9(2)°$, space group $P2_1/c$, $Z = 4$. The structure was solved by a symbolic addition procedure and refined by least-squares to R 0·082. The structural formula of the compound, assigned on the basis of crystal analysis, is helpful in elucidating the process of hydrolysis of ethyl 2-(1,2-benzisothiazolin-3-ylidene)cyanoacetate.

THE hydrolysis of cyanoacetic esters is a well known and largely used method for synthesising alkanoic acids with different substituents in the 2-position. Recently, as part of a study of photoactive substances derived from 1,2-benzisothiazole,[1] the 1,2-benzisothiazol-3-ylacetic acid [2] was prepared by that method; during the alkaline hydrolysis of ethyl 2-(1,2-benzisothiazolin-3-ylidene)-cyanoacetate (I), as well as the 1,2-benzisothiazol-3-ylacetic acid (II), a secondary product (III) was obtained (see Scheme). It was not possible to attribute a structural formula to (III) on the basis of chemical and spectroscopic properties alone; [3] in particular there were two possibilities: *i.e.* it could be derived from compound (I) by modifying the side-chain or it could be obtained as a consequence of a deeper alteration of the heterocyclic moiety (*e.g.*, by breaking the sulphenamide bond).

We now report a re-examination of this problem; *X*-ray analytical evidence obtained from compound (III) crystallized from dimethylformamide (DMF) clarifies the doubts unresolved in previous research.

EXPERIMENTAL

Preparation and Chemical Properties of Compound (III).—Compound (III) was obtained (7%) by hydrolysis of ethyl 2-(1,2-benzisothiazolin-3-ylidene)cyanoacetate (I) in aqueous alkaline medium. The elemental analysis and molecular weight indicated the formula $C_{18}H_{12}N_4S_2$. The compound is soluble in hot acetic acid, chloroform, dioxan, and dichloroethane, slightly soluble in boiling ethanol, and insoluble in water, diethyl ether, and hexane; it is stable to



(III)

SCHEME Reagents: i H_2O, ii OH^-, iii hydrolysis, iv RO^-, v H_3O^+

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[1] Ital. P. 852,483; Ger. P. 1,950,370; *Chem. Abs.*, 1970, **73**, 130, 991.

[2] T. Vitali and L. Amoretti, *Soc. Ital. Biol. Sper.*, in the press.

[3] T. Vitali, F. Mossini, M. R. Mingiardi, E. Gaetani, and V. Plazzi, *Ateneo Parmense* (*Acta Naturalia*), 1971, **7**, 1.

alkaline and acid (by boiling for 24 h in H_2SO_4, 24%) hydrolysis. After boiling in acetic acid–concentrated hydrochloric acid (8 h) a high-melting (m.p. >300 °C), difficult to purify, and undefined product was obtained. After oxidation with peracetic acid on a steam bath, a tar was isolated. Compound (III) is not oxidised by 2N-potassium permanganate, whereas compound (II) is oxidised to saccharin in the same conditions.

TABLE 1

Starting set for sign determination

h	*k*	*l*	|*E*|	Symbol	
7	4	1	4·22	+	origin fixing
1	3	1	3·12	+	
−4	5	7	2·77	+	
0	0	6	2·99	*a*	
3	10	3	3·25	*b*	
3	5	1	2·85	*c*	
−1	1	17	3·24	*d*	

Crystals of (III) were not suitable for *X*-ray analysis, but crystals suitable for that purpose were obtained, after many trials, by slow crystallization from methanol–dimethylformamide. In this way yellow single crystals of a solvated form (III,DMF) were obtained: m.p. 203—204 °C. The crystals easily lose the solvent molecule in vacuum or on drying over P_2O_5, to give a crystalline powder which corresponds to compound (III).

TABLE 2

Final atomic fractional co-ordinates (× 10^4) and thermal parameters (× 10^2 Å^2),* with estimated standard deviation for non-hydrogen atoms

	x/*a*	*y*/*b*	*z*/*c*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S(1)	−4476(2)	6772(2)	3891(1)	355(7)	359(8)	520(9)	−56(6)	48(6)	−4(7)
S(2)	3140(3)	4798(2)	2419(1)	585(11)	724(13)	370(9)	79(10)	89(7)	28(9)
O	477(8)	2357(5)	4117(3)	1034(43)	558(32)	596(32)	195(30)	222(29)	27(26)
N(1)	−2777(6)	6176(5)	3912(3)	424(27)	320(27)	503(30)	−38(21)	66(22)	−3(23)
N(2)	−721(7)	4576(5)	4059(3)	524(30)	341(28)	507(31)	−36(23)	109(24)	−60(24)
N(3)	2084(7)	5176(6)	3047(3)	518(31)	573(36)	401(29)	120(26)	113(23)	10(26)
N(4)	2096(7)	7867(6)	4189(5)	407(32)	595(41)	1176(59)	−102(29)	63(34)	−125(40)
N(5)	1371(9)	1532(6)	3198(3)	841(45)	661(42)	443(33)	137(35)	174(30)	−62(30)
C(1)	−1657(7)	6889(6)	4072(3)	322(27)	367(33)	354(30)	−45(23)	23(21)	−24(25)
C(2)	−2151(7)	8028(5)	4174(3)	387(30)	330(31)	324(29)	−30(23)	40(22)	−15(24)
C(3)	−1358(8)	9001(6)	4334(3)	453(34)	376(33)	381(33)	−26(26)	10(25)	−46(27)
C(4)	−2099(9)	9969(6)	4422(4)	598(41)	387(36)	435(36)	−39(30)	54(29)	−48(29)
C(5)	−3758(9)	9994(6)	4334(4)	603(41)	405(37)	474(37)	7(30)	165(30)	−32(30)
C(6)	−4578(8)	9064(6)	4183(4)	501(36)	393(35)	494(37)	−6(27)	132(28)	−23(30)
C(7)	−3758(8)	8075(6)	4090(3)	435(32)	332(32)	387(32)	−53(25)	64(24)	5(26)
C(8)	−109(7)	6495(6)	4136(3)	371(30)	365(32)	349(30)	−51(23)	15(22)	−26(26)
C(9)	2665(8)	4772(6)	3643(3)	440(32)	360(33)	318(30)	18(25)	22(23)	16(25)
C(10)	3993(7)	4126(5)	3631(3)	371(30)	316(31)	372(31)	2(23)	32(23)	−28(25)
C(11)	4848(8)	3603(6)	4176(4)	492(37)	373(35)	528(39)	8(27)	−25(29)	−15(30)
C(12)	6105(9)	2977(6)	4032(5)	495(39)	357(37)	868(55)	−19(29)	−76(36)	22(37)
C(13)	6505(9)	2879(7)	3362(5)	494(40)	488(42)	770(52)	36(32)	−9(35)	−113(38)
C(14)	5713(9)	3400(7)	2841(5)	503(40)	550(45)	754(51)	−48(33)	177(35)	−202(39)
C(15)	4405(8)	4045(6)	2978(4)	482(36)	423(36)	447(36)	13(28)	80(27)	−27(30)
C(16)	1927(8)	5025(6)	4288(3)	445(33)	486(38)	330(31)	74(28)	30(24)	−27(28)
C(17)	283(8)	5383(6)	4149(3)	425(32)	444(36)	309(29)	15(26)	61(23)	−13(27)
C(18)	1117(8)	7264(6)	4173(4)	407(34)	458(39)	596(43)	−3(28)	51(29)	−107(33)
C(19)	1265(12)	1630(9)	3850(5)	974(65)	693(57)	645(52)	168(49)	180(46)	−2(45)
C(20)	596(12)	2323(9)	2706(5)	943(63)	772(59)	470(45)	−10(49)	68(40)	55(42)
C(21)	2216(19)	646(12)	2933(7)	2048(137)	1312(106)	1049(86)	1028(102)	707(90)	60(77)

* The anisotropic temperature factor is expressed in the form: $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Physical Measurements.—Melting points were determined on a Büchi apparatus (Tottoli).

U.v., i.r., n.m.r., and mass spectra were recorded on the following apparatus: Beckman DK 2, Beckman IR 5 (NaCl prism), Varian A 60 (60 Mc s^{-1}), and Hitachi RMU 6 (70 eV; double focussing).

Elemental analyses were carried out by use of an automated Perkin-Elmer 240 analyser (carbon, hydrogen, and nitrogen), and by the Schöniger method for sulphur. Results agree within ±0·3% with the theoretical values.

X-Ray intensity data were collected on a Siemens single-crystal automated diffractometer, 'on line' to a Siemens 304/P computer (ω—2θ scan technique). Crystals of (III,DMF) are very unstable, so the sample used for *X*-ray analysis was closed in a Lindeman capillary with mother liquor.

Crystal Data.—$C_{21}H_{19}N_5OS_2$, $M = 421·6$, Monoclinic, $a = 8·85(1)$, $b = 12·08(1)$, $c = 19·52(1)$ Å, $\beta = 95·9(2)°$, $U = 2076$ Å^3, $Z = 4$, $D_c = 1·35$ †, $F(000) = 880$. Cu-K_α radiation, $\bar{\lambda} = 1·5418$ Å; μ(Cu-K_α) = 24·3 cm^{-1}. Space group $P2_1/c$ (from systematic absences).

Cell dimensions were determined from diffractometer measurements.

Intensity Data Collection.—Of the 3257 independent reflections (Cu-K_α radiation) 2622 having $I > 2\sigma(I)$ were used in the analysis. No correction for absorption was applied ($\mu r = 0·3$). After the usual data reduction, the structure amplitudes were put on absolute scale, first by Wilson's method, then by correlating observed and calculated values.

Structure Analysis.—The structure was solved by means of a 'symbolic addition' procedure.[4] The starting set used for sign determination is given in Table 1. The solution for the symbols was found by analysis of 16 possible combinations of signs among which were —,—,—,— for

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† D_m not measured owing to the instability of crystals.

[4] G. Germain and M. M. Woolfson, *Acta Cryst.*, 1968, *B*, **24**, 91.

a, *b*, *c*, and *d*. By use of this set of signs for 436 reflections with $|E| \geqslant 1{\cdot}50$ and with the highest figure of merit, an E map was computed, which revealed the whole structure, excepting the DMF molecule, whose atoms were located by a subsequent Fourier synthesis. The last structure-factor calculation, before refinement, gave R 25·0%. The refinement was then carried out until R was 9·3% by means of an anisotropic least-squares procedure, in which the function minimized was $\Sigma w(\Delta|F|)^2$, with unit weights. At this point a difference Fourier map enabled location of all the hydrogen atoms except those of DMF (final R 8·2% after isotropic least-squares refinement for hydrogen atoms); in that map a residual electron density (*ca.* 0·6 eÅ^{-3}), near to N(1), was also evident. The seven hydrogen atoms of DMF have thermal effects (or disordered distributions) so high as to make any effort to locate them unsuccessful.

The atomic scattering factors used throughout the calculations were taken from ref. 5 for sulphur, oxygen, nitrogen, and carbon, and from ref. 6 for hydrogen. The final values of positional and thermal parameters, with their estimated standard deviations, are quoted in Tables 2 and 3.

TABLE 3

Fractional co-ordinates ($\times 10^3$) and isotropic thermal parameters (Å^2) for hydrogen atoms

	x/*a*	*y*/*b*	*z*/*c*	*B*
H(1)	−55(7)	396(6)	406(3)	5·7(1·7)
H(2)	−167(7)	475(6)	393(3)	5·7(1·6)
H(3)	−32(7)	905(5)	447(3)	5·2(1·6)
H(4)	−127(7)	1062(6)	464(3)	6·3(1·8)
H(5)	−461(7)	1073(6)	434(4)	6·1(1·7)
H(6)	−567(8)	901(6)	406(3)	7·8(2·0)
H(7)	474(7)	373(6)	470(4)	6·0(1·7)
H(8)	696(8)	271(6)	456(4)	7·2(1·9)
H(9)	752(8)	248(6)	323(4)	7·2(2·0)
H(10)	620(8)	325(6)	225(3)	7·7(2·0)
H(11)	247(7)	568(6)	451(3)	5·7(1·6)
H(12)	185(7)	434(5)	464(3)	4·9(1·5)

Observed and calculated structure factors are listed in Supplementary Publication No. SUP 20478 (29 pp., 1 microfiche).*

All calculations were performed on the CDC 6600 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale by use of the programs of Immirzi.[7]

RESULTS AND DISCUSSION

U.v. Spectrum.—The u.v. spectrum of (III) in ethanol shows a broad and strong band at about 343 nm ($\log_{10}\varepsilon = 3{\cdot}90$), which can be attributed to the absorption of the 3-ylidene system.

I.r. Spectra.—The i.r. spectrum of (III) (KBr disk) shows two bands, at 3270m and 2175m cm^{-1}, which can be assigned to =NH and conjugated −CN groups, bands at 1610s, 1535s, and 1470s cm^{-1} which correspond to the benzene system, and bands at 1335w, 1315w, 770m, 760s, and 730s cm^{-1}.

The i.r. spectrum of (III,DMF) is not sensibly different from that of non-solvated compound, except for small variations in the ' finger-print ' region and the presence of a strong band at 1590 cm^{-1}, which can be assigned to ν(CO) vibration of DMF, shifted to lower frequencies owing to intermolecular effects.

N.m.r. Spectrum.—Compound (III) in deuteriated dimethyl sulphoxide gives a singlet at δ 4·56 not exchangeable with D_2O and a multiplet, centred at δ 7·90, which is partially exchangeable with D_2O. The ratio of the areas of the two signals is 1 : 5, which becomes 1 : 4 when D_2O is added.

Mass Spectrum.—In mass spectrum the following main peaks are observed: *m*/*e* 349(26), 348(60, M^+), 347(100), 332(19), 213(11), 175(9), 174(25), 149(28), 148(19), and 121(14); in particular the molecular ion confirms the analytical data. The rather strong peak at *m*/*e* 332 ($M - 16$) is difficult to attribute; it corresponds to the unusual loss of 16 mass units (never observed in 1,2-benzisothiazole derivatives). No more relevant signals are registered till the peak at *m*/*e* 213, which can be attributed to ($M/2 + 39$) fragmentation and this is confirmed by the weak signal at *m*/*e* 187 (213 − CN), assuming that 39 corresponds to CHCN. There is also a peak at 174 ($M/2$) which fragments in a

TABLE 4

Bond distances (Å) and angles (deg.) with standard deviations in parentheses

(*a*) Distances

S(1)–N(1)	1·664(6)	C(16)–C(9)	1·51(1)
N(1)–C(1)	1·33(1)	C(9)–C(10)	1·41(1)
C(1)–C(2)	1·46(1)	C(9)–N(3)	1·32(1)
C(2)–C(3)	1·39(1)	N(3)–S(2)	1·679(7)
C(3)–C(4)	1·36(1)	S(2)–C(15)	1·738(8)
C(4)–C(5)	1·46(1)	C(15)–C(10)	1·36(1)
C(5)–C(6)	1·36(1)	C(10)–C(11)	1·39(1)
C(6)–C(7)	1·42(1)	C(11)–C(12)	1·40(1)
C(7)–C(2)	1·42(1)	C(12)–C(13)	1·39(1)
C(7)–S(1)	1·727(7)	C(13)–C(14)	1·34(1)
C(1)–C(8)	1·44(1)	C(14)–C(15)	1·44(1)
C(8)–C(18)	1·42(1)	O–C(19)	1·27(1)
C(18)–N(4)	1·13(1)	C(19)–N(5)	1·29(1)
C(8)–C(17)	1·39(1)	N(5)–C(20)	1·47(1)
C(17)–N(2)	1·32(1)	N(5)–C(21)	1·43(1)
C(17)–C(16)	1·52(1)		

(*b*) Angles

C(7)–S(1)–N(1)	94·5(3)	N(2)–C(17)–C(16)	115·7(6)
S(1)–N(1)–C(1)	112·1(5)	C(17)–C(16)–C(9)	113·5(5)
N(1)–C(1)–C(2)	114·6(5)	C(16)–C(9)–C(10)	123·9(6)
C(1)–C(2)–C(7)	109·4(5)	C(16)–C(9)–N(3)	120·0(6)
C(1)–C(2)–C(3)	132·5(5)	C(10)–C(9)–N(3)	116·1(6)
C(3)–C(2)–C(7)	118·1(6)	C(9)–N(3)–S(2)	110·4(5)
C(2)–C(7)–S(1)	109·4(5)	N(3)–S(2)–C(15)	93·1(3)
S(1)–C(7)–C(6)	128·0(5)	S(2)–C(15)–C(14)	130·1(6)
C(2)–C(7)–C(6)	122·6(6)	S(2)–C(15)–C(10)	109·4(5)
C(7)–C(6)–C(5)	117·2(7)	C(15)–C(10)–C(9)	110·9(6)
C(6)–C(5)–C(4)	121·2(7)	C(9)–C(10)–C(11)	128·7(6)
C(5)–C(4)–C(3)	119·6(7)	C(15)–C(10)–C(11)	120·3(6)
C(4)–C(3)–C(2)	121·2(6)	C(10)–C(11)–C(12)	118·3(7)
N(1)–C(1)–C(8)	119·1(6)	C(11)–C(12)–C(13)	121·2(8)
C(2)–C(1)–C(8)	126·3(6)	C(12)–C(13)–C(14)	120·8(8)
C(1)–C(8)–C(18)	120·0(6)	C(13)–C(14)–C(15)	118·9(8)
C(1)–C(8)–C(17)	123·7(6)	C(14)–C(15)–C(10)	120·5(7)
C(17)–C(8)–C(18)	116·3(6)	O–C(19)–N(5)	124·6(9)
C(8)–C(18)–N(4)	178·6(9)	C(19)–N(5)–C(20)	120·5(8)
C(8)–C(17)–N(2)	123·4(6)	C(19)–N(5)–C(21)	121·2(9)
C(8)–C(17)–C(16)	120·9(6)	C(20)–N(5)–C(21)	118·3(8)

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* For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20.

[5] D. T. Cromer and J. B. Mann, *Acta Cryst.*, 1968, *A*, **24**, 321.

[6] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

[7] A. Immirzi, *Ricerca Sci.*, 1967, **37**, 743.

similar way to that observed for other 1,2-benzisothiazole compounds.[3]

X-*Ray Structure*.—From the *X*-ray analysis it is apparent that the molecule of compound (III) is built up of two benzisothiazole systems, and the addition of these two parts involves the tetrahedral carbon atom of one molecule and the nitrilic carbon atom of the other. The two benzisothiazole systems are practically planar and arranged in space to form a dihedral angle of 85·9° (Figure 1). Table 4 lists bond distances and angles. The S–C (1·727 and 1·738 Å) distances in the isothiazole rings show some double-bond character {σ-[S–C(sp^2)] 1·78; π-[S–C(sp^2)] 1·61 Å [8]} and the same can be said for the S–N (1·664 and 1·679) and C–N (1·33 and 1·32 Å) distances; in these last the double-bond character is more relevant. The C(1)–C(2) (1·46) and C(9)–C(10) (1·41 Å) distances are significantly different; this is probably due to the different situations of the two rings as a consequence of the dimerization process. The C(1)–C(2) bond is very near to a single bond [C(sp^2)–C(sp^2) 1·48 Å].[9] The angles on the sulphur atoms are slightly larger than those generally reported for similar compounds (*e.g.*, 90·5, 90·6 in 1,2-benzoisothiazolin-3-one,[10] and 88·9° in 7-chloro-1,2-benzoisothiazolin-3-one [11]). The angle on S(1) is a little larger than that on S(2) and this is probably due to the differences in the C(1)–C(2) and C(9)–C(10) distances. The N(4), C(18), C(8), C(17), C(1) system is planar and forms a dihedral angle of 12·1° with the plane of the isothiazole ring to which it is attached, and a dihedral angle of 6·6° with the planar system C(8), C(17), N(2), C(16). The internal rotation angle around C(9)–C(16) is 19·5°. The C(18)–N(4) distance corresponds to a triple bond, C(8)–C(18) to a single bond [C(sp^2)–C(sp) 1·439 Å [9]], C(16)–C(17) and C(16)–C(9) to single bonds [C(sp^3)–C(sp^2) 1·51 Å [9]]. The values of the distances N(1)–C(1), C(1)–C(8), C(8)–C(17), and C(17)–N(2), and the trigonal character of the carbon atoms indicate a delocalization of the double bonds along the system N(1), C(1), C(8), C(17), N(2). The nitrogen atom N(2) is trigonal as indicated also by the planarity of the system C(17), N(7), H(1), H(2) and by the relevant π character of the distance N(2)–C(17) (1·32 Å), so this group bears a formal positive charge, while an excess of negative charge is transferred, through the π bond system, on N(1). The value of the distance N(1)–N(2) (2·65 Å) can be justified by the internal ionic interaction (Figure 2). If the residual electron density observed near N(1) in the final Δ*F* map is true, it must be assumed that there is some disorder arising from the possibility of a tautomeric exchange of the proton between two positions, one near N(2), the other near N(1), corresponding to (1). This gives some kind of disordered situation in the structure, the first form being the predominant one.

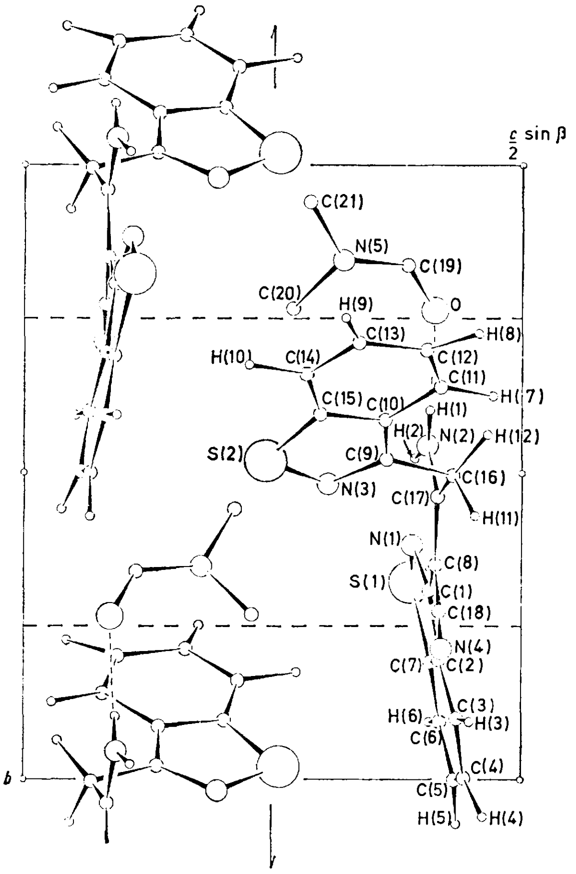


FIGURE 1 Projection of the structure along [100]

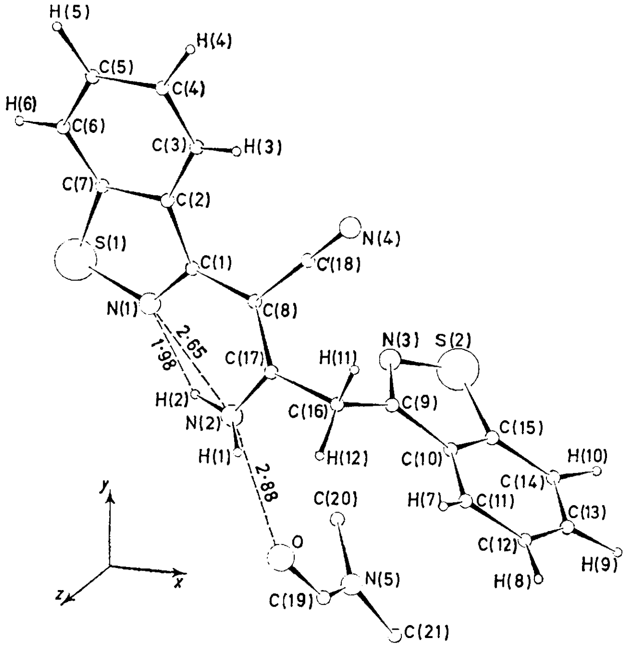


FIGURE 2 Clinographic projection of the structure

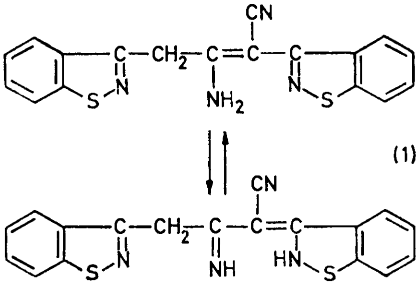
[8] D. L. Smith, *Acta Cryst.*, 1969, *B*, **25**, 625.

[9] L. E. Sutton, *Chem. Soc. Special Publ.*, No. 18, 1965.

[10] L. Cavalca, A. Gaetani, A. Mangia, and G. Pelizzi, *Gazzetta*, 1970, **100**, 629.

[11] L. Cavalca, G. Fava Gasparri, A. Mangia, and G. Pelizzi, *Acta Cryst.*, 1969, *B*, **25**, 2349.

The high values of the thermal parameters for the DMF molecule are in agreement with the fact that this molecule is not strongly bonded in the structure and can be lost quite easily. Bond distances and angles in DMF are as expected; the hydrogen bond it forms with N(2) [N(2) · · · O 2·88 Å, H(1)–N(2) · · · O 10°] gives the reason for its being trapped in the structure.



Packing is determined by normal van der Waals interactions; the most significant non-bonded contacts are listed in Table 5.

On the basis of the structural results, compound (III) is formed from compound (I) probably through the formation of 1,2-benzisothiazol-3-ylacetonitrile (IV). This hypothesis is summarized in the Scheme, where two competitive processes from (IV) are shown: the hydrolytic process which prevails in an aqueous alkaline medium, and the dimerization process which prevails when the formation of a mesomeric carbanion is favoured. This interpretation has been confirmed by experiments carried out in the presence of alkaline alkoxides which gave compound (III) with a practically quantitative yield, starting from compound (IV).*

TABLE 5

Some significant non-bonded contacts <4 Å

S(1) · · · N(4^{I})	3·41(1)	S(2) · · · C(5^{II})	3·53(1)
S(1) · · · C(10^{I})	3·49(1)	O · · · C(3^{III})	3·46(1)
N(2) · · · C(13^{I})	3·38(1)	N(2) · · · C(17^{III})	3·48(1)
N(4) · · · C(6^{I})	3·28(1)		

Roman numerals as superscripts refer to the following equivalent positions relative to the reference molecule at x, y, z:

I $x - 1, y, z$ II $\bar{x}, y - \frac{1}{2}, \frac{1}{2} - z$ III $\bar{x}, 1 - y, 1 - z$

[2/766 *Received*, 30*th March*, 1972]

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* Sodium (0·23 g) dissolved in anhydrous ethanol (40 ml) and benzisothiazolacetonitrile (IV) (1·74 g) [3] in ethanol (20 ml) were mixed and heated under reflux for 20 min when a voluminous precipitate had formed. The cooled product was washed (water) and recrystallized (dichloroethane) as small white crystals (m.p. 203—204 °C), identical in all respects with compound (III).