X- RAY POWDER DIFFRACTION DATA FOR SODIUM MORPHOLYLDITHIOCARBAMATE TRIHYDRATE $C_5H_8NOS_2NA\ 3H_2O$

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ABSTRACT
Sodium Morpholyldithiocarbamate Trihydrate $C_5H_8NOS_2Na\ 3H_2O$ has been investigated by means of X-ray powder diffraction. The title compound is triclinic with unit-cell parameters a = 6.261 (2) b = 8.897 (3) c = 16.557 (4) Å; α = 118.34 (2) β = 99.66 (2) γ = 93.32 (3) °, V = 790.2 (3)$Å^3$, Z =2 , space group P 1 and Dx = 1.007 (12) g cm^{-3}.

KEYWORDS
Crystal characterization, morpholyl dithiocarbamate and X- Ray Powder Diffraction.

INTRODUCTION
Sodium morpholyldithiocarbamate Trihydrate $C_5H_8NOS_2Na\ 3H_2O$ (fig. 1) is a compound with several biological properties, such as: antitumoral (1) and antioxidant (2).

This dithiocarbamate anion acts as a S,S type chelating agent and forms stable complexes with representative and transition metals (3, 4) that are generally only slightly soluble or insoluble in water and other polar solvents. The complexes are specially stable with "soft" metals (5).

EXPERIMENTAL

A. Origin of specimen

An ethanol solution of morpholine was added dropwise to an ethanol solution of CS_2 at 0-5 °C (morpholine:CS_2 molar ratio of 1:1). The resulting mixture was treated with Et_2O and an aqueous solution of NaOH for a CS_2:NaOH molar ratio of 1:1. The product was filtered, washed and recrystalized from ethanol; m.p.>300 °C. IR ν(cm^{-1}): 1460 ($\nu_{C=N}$), 981 (ν_{C-S}), 542 (ν_{C-S} + δ_{SCS}) . UV: λ_{max}(nm) 263 log ε = 4.18 (CSS π-π*); 284 log ε = 4.18 (NCS π-π*), ^{1}H-NMR (D_2O): δ (ppm) 4.38 (t, 4H, -OC**H**$_2$-, J_{H-H}=5.1 Hz); 3.77 (t, 4H, -NC**H**$_2$-, J_{H-H}=4.9 Hz).

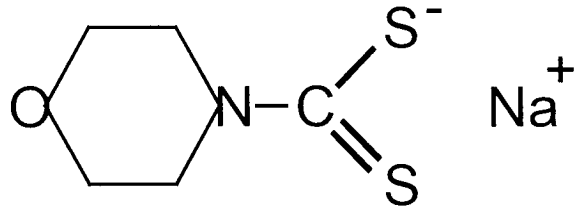

Fig.1. Scheme of the Sodium Morpholyldithiocarbamate Trihydrate $C_5H_8NOS_2Na\ 3H_2O$.

B. Crystal Data

Crystalline powder; color: white; space group: $P\bar{1}$; a = 6.261 (2) b = 8.897 (3) c = 16.557 (4) Å; α = 118.34 (2) β = 99.66 (2) γ = 93.32 (3) °; Z = 2; D_x = 1.007(12) g cm^{-3} and V= 790.2 (3) $Å^3$.

C. X-ray powder data

The sample was ground in an agate mortar and the loose powder was pressed into a diffractometer sample holder. The X-ray diffraction pattern was collected using Ni filtered $CuK_{\alpha 1}$ radiation 1.5406 Å, ($K_{\alpha 2}$ was eliminated with computer software) on a Philips PW 1710 diffractometer operated at 40 Kv and 30 mA. The alignment of the diffractometer was checked by using a silicon external standard from National Institute of Standards and Technology NIST- SRM- 640b with cell parameter a = 5.430940 (35) Å (6) at 25 °C. The powder pattern was recorder at 25 (1) °C from 4 to 70° 2θ using an angular step 0.02° and a counting time of 5 s.

The reported peak heights and positions were extracted by fitting Pearson VII type functions to the diffraction maxima. The positions of all peaks were input in the program for least-square unit cell refinement LSUCRI (7). The starting set of cell parameters for the refinement was taken from the output of the indexing program TREOR90 (8), gave the following cell parameter a = 6.26 (1), b = 8.89 (2), c = 16.52 (2)Å; α = 118.3 (2), β = 99.7 (3), γ =93.3 (2)° and figures of merit M_{20} = 11 and F_{20} = 23 (0.016643, 53) ($\Delta 2\theta$,Npos).

The closed values of the FWHM of 111 peak of the Si standard (FWHM = 0.12° at 28.446°) and that of the 111 peak of the sample (FWHM = 0.14° at 18.976°) indicate a high degree of crystallinity of the specimen. In table I reported the powder diffraction data and in Fig. 2 reported the X- Ray powder pattern of the Sodium Morpholyldithiocarbamate Trihydrate $C_5H_8NOS_2Na\ 3H_2O$.

Table 1. Powder diffraction data for Sodium morpholyldithiocarbamate Trihydrate $C_5H_8NOS_2Na\ 3H_2O$.

Rad CuKα (λ = 1.5406 Å)	Ni filter	Sys. Triclinic;
a = 6.261 (2) b = 8.897 (3)	c = 16.557 (4) Å	Space Group: $P\bar{1}$
α = 118.34 (2) β = 99.66 (2)	γ = 93.32 (3) °.	V= 790.2 (3) Å^3
Z = 2; D_x = 1.007 (12) g cm^{-3}		Color White

hkl	$2\theta_{obs}$ (°)	d_{obs} (Å)	I/I_o
0 0 1	6.215	14.208	100
0 0 2	12.449	7.104	9
1 0 0, 1 0 -1	14.515	6.098	11
0 1 1	15.495	5.714	5
1 -1 0, 1 0 1	17.038	5.200	76
1 -1 1	17.300	5.128	9
1 1 -2	18.156	4.882	8
0 0 3	18.720	4.736	23
1 -1 -1	18.976	4.679	25
1 1 0	19.925	4.452	15
1 0 -3	21.042	4.218	50
1 0 2	21.242	4.179	11
0 2 0	23.010	3.862	5
1 -1 3	23.554	3.774	37
0 2 –4, 1 -2 2	24.319	3.657	26
1 0 3	26.199	3.399	30
0 2 1	26.706	3.335	10
1 -1 -3	27.214	3.274	7
1 -1 4, 0 2 -5	28.320	3.148	17
1 1 –5	28.804	3.097	5
1 -2 4, 2 0 0	29.387	3.037	5
0 3 –3, 2 -1 -1	30.209	2.956	5
2 -1 1, 0 3 -2	30.551	2.924	6
2 1 -2	30.893	2.892	6
0 0 5, 2 1 -1	31.455	2.842	28
1 0 4	31.806	2.811	6
1 2 1	32.939	2.717	43
1 3 -4	34.581	2.592	15
2 –2 2	36.438	2.463	4
0 3 –6, 2 2 -4	36.681	2.448	5
1 0 5	37.706	2.384	7
0 0 6	37.965	2.368	29
1 -2 6, 0 3 1	38.431	2.340	13
1 -1 6	39.488	2.280	8
1 1 -7	39.716	2.268	11
2 1 -6, 2 2 0	40.400	2.231	13
1 -4 2	42.983	2.103	9
1 -2 7, 2 –2 5	43.720	2.069	19
0 0 7	44.602	2.030	9

1 -2 -5	47.036	1.9304	9
3 -2 1, 2 -3 -2	47.400	1.9164	8
2 0 5, 3 -2 2	48.415	1.8786	5
3 -2 -2, 2 2 -8 , 2 1 4, 3 2	49.328	1.8460	4
0 4 8, 2 –3 6, 2 –1 6,	49.756	1.8310	10
2 -3 –3, 1 1 6, 3 1 -6	50.996	1.7894	3
0 0 8, 2 –4 5	51.408	1.7760	4
2 3 -8, 0 5 -5	51.519	1.7724	4
3 -3 1, 1 1 -9	52.138	1.7529	3
3 -1 -6, 3 3 -6, 3 –2 5	56.582	1.6253	5
1 0 -9	56.882	1.6174	4
1 -3 -5, 3 3 -2, 1 1 7 , 2 -4	57.505	1.6014	3
0 0 9, 2 –3 8	58.395	1.5788	3
4 0 -1, 4 -1 -1, 2 4 -9	59.475	1.5530	5
3 4 4, 4 0 -4	60.394	1.5315	3
2 3 3	62.579	1.4832	4
1 -1 -9, 2 5 -3, 0 4 -11	63.923	1.4552	3
3 -1 -8	66.287	1.4089	3

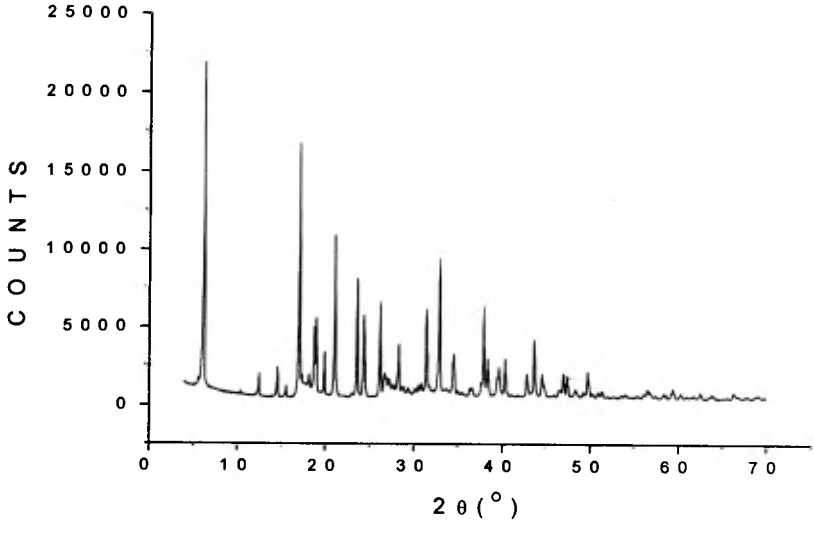

Fig.2. X-ray diffraction pattern of the Sodium Morpholyldithiocarbamate Trihydrate $C_5H_8NOS_2Na\ 3H_2O$.

RESUMEN

El compuesto Morfolil Ditiocarbamato trihidratado $C_5H_8NOS_2Na\ 3H_2O$ fue estudiado a través de difracción de rayos– X, por el método de polvo. El mismo cristaliza en el sistema triclínico, en el grupo espacial $P\bar{1}$, con parámetros de celda: a = 6.261 (2) b = 8.897 (3) c = 16.557 (4) Å; α = 118.34 (2) β = 99.66 (2) γ = 93.32 (3) °, V = 790.2 (3)Å^3, Z = 2 y Dx = 1.007 (12) g cm^{-3}.

PALABRAS CLAVES

Caracterización cristalográfica, Morfolil ditiocarbamate, Difracción de Rayos- X por el método de polvo.

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