sisted of an isomeric substance since it was not noticeable analytically. Possibly ammonium chloroaurate which is less soluble than the chloroaurate of β-methylcholine persisted in spite of the many recrystallizations.

Anal. Calcd. for $C_6H_{16}ONCl_4Au$: Au, 43.07. Found: Au, 43.15, 43.08.

The authors wish to express their appreciation to Mr. Douglass F. Hayman for most of the analyses which are recorded in this paper.

Summary

1. It has been shown that the only true α-methylcholine that has ever been made was that made by Karrer.[8]

2. True β-methylcholine chloride has been prepared by the reduction of trimethylacetonylammonium chloride.

3. It has been shown that β-methylcholine chloride may be obtained by the condensation of propylene chlorohydrin and trimethylamine.

4. γ-Homocholine chloride has been obtained by the condensation of trimethylene chlorohydrin with a solution of trimethylamine in benzene.

5. We have repeated the process used by Partheil to make his "γ-homocholine," the chloroaurate of which melted at 162°. We were not able to isolate any of the salts of γ-homocholine from the reaction mixture but have found salts of ammonia and of β-methylcholine in it.

RAHWAY, NEW JERSEY

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF COLORADO]

OPTICAL CRYSTALLOGRAPHIC DATA FOR SOME SALTS OF STRYCHNINE

BY CHARLES F. POE AND JESSE E. SELLERS

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Introduction and Historical

The literature reveals that very few optical data have been reported for the salts of strychnine. Some of these are reported by Groth[1] and Bolland[2] and include two of the refractive indices for the nitrate and the hydroiodide and one for the sulfate. Several investigators have studied the optical properties of other alkaloids, usually the bases themselves. Wherry and Yanovsky[3] made such measurements for the cinchona alkaloids. Other investigators along these lines include Kley,[4] Wright,[5] Wherry,[6] and Keenan.[7]

[1] Groth, "Chemische Krystallographie," 1906–1919.

[2] Bolland, *Monatsh.*, **31**, 387 (1910).

[3] Wherry and Yanovsky, THIS JOURNAL, **40**, 1063 (1918).

[4] Kley, *Z. anal. Chem.*, **43**, 160 (1904).

[5] Wright, THIS JOURNAL, **38**, 1647 (1916).

[6] Wherry, *U. S. Dept. of Agric. Bull.*, 679 (1918).

[7] Keenan, *J. Am. Pharm. Assoc.*, **16**, 837 (1927).

This paper describes a number of salts of strychnine which were prepared for the purpose of perfecting a method for the micro-chemical detection of strychnine and gives the optical crystallographic data concerning them.

Preparation of Salts.—With the exception of the chlorate and thiocyanate, the salts were all prepared in a similar manner. Molecular quantities of strychnine and the acid were mixed, and water added. The solutions were then heated for several minutes, filtered and the salt was allowed to crystallize. The salt was then recrystallized until pure. Except in cases where water of crystallization would be given off, the salts were dried in the air. The chlorate and thiocyanate were prepared by dissolving the alkaloid in very dilute acetic acid and adding solutions of equivalent quantities of potassium chlorate or potassium thiocyanate.

Analysis of the Salts.—Each salt was analyzed for the percentage of strychnine by the picric acid method.[8] The strychnine content was determined on each salt containing water of crystallization and on the anhydrous salt. Nitrogen was determined by the Kjeldahl–Gunning–Arnold method and water of crystallization in each compound was estimated by drying over sulfuric acid, phosphorus pentoxide, or by heating the salt. The water of crystallization in all cases was also determined by the Germann and Muench[9] modification of Wilson's[10] vapor pressure method. It was found difficult to get the balance air-tight enough for atmospheres of low or high aqueous tension. In order to avoid this difficulty, a modification of the method was worked out. A square bottle was placed on its side in the balance case with the large mouth pointing outward. The opening of the bottle was covered with a glass plate, ground to fit, and sealed with vaseline. A hole was drilled in the upper side of the bottle through which the pan support of the balance passed. The cover glass containing the sample was placed on this pan. The same concentration of acid was used both inside the bottle and in the balance case. The balance case doors were closed and made as air-tight as possible by caulking with cotton-wool.

The compounds prepared and studied, together with their analyses, appear in Table I. Of the salts reported in Table I, the methods for preparation of the disuccinate, glutarate, maleate, chlorate and the thiocyanate[11] were not found in the literature. The malonate, dioxalate and selenate were each found to contain an amount of water of crystallization different from that previously reported.

[8] Elmore, *J. Assoc. Off. Agric. Chem.*, **9**, 224 (1926).

[9] Germann and Muench, *J. phys. Chem.*, **32**, 1380 (1928).

[10] Wilson, THIS JOURNAL, **43**, 704 (1921).

[11] Dollfus, *Ann. Chem. Pharm.*, **65**, 221 (1848), describes a compound supposed to be the thiocyanate, but the analysis given does not correspond to the present formula for this salt.

TABLE I

SALTS OF STRYCHNINE

Salt of strychnine	Formula S = $C_{21}H_{22}O_2N_2$	Strychnine, %, in hydrated salt Calcd.	Found	Strychnine, %, in anhydrous salt Calcd.	Found	Nitrogen, % Calcd.	Found	Water of crystallization found %	No. of molecules	Possible no. of molecules of water by vapor pressure method
Dioxalate	$S{\cdot}C_2H_2O_4{\cdot}1.5H_2O$	74.07	74.07	78.79	78.66	6.20	6.20	5.79	1.45	1.5
Disuccinate	$S{\cdot}C_4H_6O_4{\cdot}H_2O$	71.07	71.15	73.90	73.71	5.95	5.97	3.83	1.00	1.0
Ditartrate	$S{\cdot}C_4H_6O_6{\cdot}3H_2O$	62.09	62.26	69.02	69.46	5.20	5.17	9.88	2.95	3.0
Chlorate	$S{\cdot}HClO_3{\cdot}H_2O$	76.54	76.58	79.83	80.04	6.42	6.33	4.13	1.00	1.0
Glutarate	$S{\cdot}C_5H_8O_4$	71.68	71.85	71.68	71.85	6.00	5.92	None	None	None
Hydrobromide	$S{\cdot}HBr{\cdot}H_2O$	77.17	77.42	80.51	80.08	6.46	6.48	3.87	0.93	1.0
Hydrochloride	$S{\cdot}HCl{\cdot}1.75H_2O$	83.09	83.24	90.17	90.24	6.96 (Cl, 8.82)	6.99 (Cl, 8.86)	3.27	1.73	1.75
Hydroiodide	$S{\cdot}HI{\cdot}H_2O$	69.61	69.63	72.32	71.99	5.83	5.82	3.75	1.00	1.0
Maleate	$S{\cdot}C_4H_4O_4{\cdot}H_2O$	71.38	71.19	74.23	74.24	5.98	6.11	4.36	1.13	1.00
Malonate	$S_2{\cdot}C_3H_3O_4{\cdot}6H_2O$	75.91	75.57	86.53	86.28	6.36	6.28	12.09	5.90	6.0, 3.0
Nitrate	$S{\cdot}HNO_3$	84.14	84.24	84.14	84.24	10.57	10.43	None	None	None
Oxalate	$S_2{\cdot}C_2H_2O_4{\cdot}4.5H_2O$	79.62	79.79	88.14	88.17	6.67	6.74	9.26	4.33	4.5, 4.0, 2.0
Perchlorate	$S{\cdot}HClO_4{\cdot}H_2O$	73.83	73.94	76.89	76.58	6.18	6.11	3.98	1.00	1.0
Phosphate	$S{\cdot}H_3PO_3{\cdot}2H_2O$	71.36	71.40	77.32	77.51	5.98	6.14	7.60	1.97	2.0
Selenate	$S_2{\cdot}H_2SeO_4{\cdot}4.5H_2O$	74.71	74.61	82.16	82.08	6.26	6.20	8.96	4.45	5.0(?), 4.5, 2.0
Sulfate	$S_2{\cdot}H_2SO_4{\cdot}5H_2O$	78.03	78.03	87.20	86.83	6.54	6.61	10.55	5.02	6.0, 5.0
Sulfate	$S_2{\cdot}H_2SO_4{\cdot}6H_2O$	76.42	76.04	87.20	86.83	6.40	6.44	12.25	5.94	6.0, 5.0
Thiocyanate	$S{\cdot}HCNS{\cdot}H_2O$	81.26	81.24	84.98	85.30	10.22	10.31	4.76	1.09	1.0

TABLE II

OPTICAL CRYSTALLOGRAPHIC DATA FOR SOME SALTS OF STRYCHNINE[a]

All salts biaxial except the uniaxial malonate

Strychnine salt	Habit	Optical character (sign)	Sign of elongation	Refractive indices 25° α	β	γ	2 V-Dispersion	Crystal system
Dioxalate, $1.5H_2O$	Stout needles	+	± usually −	1.592	1.603	1.665	Large, $\rho > v$	Monoclinic, ext. angle = 69.5°
Disuccinate, H_2O	Needles	−	−	1.588	1.646 ±	1.662	Large	Orthorhombic ?
Ditartrate, $3H_2O$	Stout needles	+	Indeterm.	1.596	1.603	1.632	Large, marked, $\rho > v$	Monoclinic
Chlorate, H_2O	Fine needles	+	−	1.605	1.611	1.663	Moderate, horizontal	Monoclinic $x = b$
Glutarate	Needles	−	−	1.575		1.655		
Hydrobromide, H_2O	Needles	+	−	1.646	1.650	1.73 ±	Small, weak	Monoclinic
Hydrochloride, $1.75H_2O$	Fine needles	+	−	1.609	1.627	1.662	Large, weak, $\rho > v$	Orthorhombic
Hydroiodide, H_2O	Stout needles	+	−	1.661 (1.66)	1.665	1.73 ± (1.69)	Small, $\rho > v$	Monoclinic
Maleate, H_2O	Rods	− ?	−	1.544	1.598	1.667	Large	Orthorhombic
Malonate, $6H_2O$	Plates	−		ϵ = 1.608	ω = 1.610			Tetragonal
Nitrate	Fine needles	+	−	1.610 (1.62)	1.624	1.675 (1.67)	Large, $\rho > v$	Monoclinic
Oxalate, $4.5H_2O$	Needles	+	±	1.592 ±	1.598	1.662	Small, $\rho > v$	Monoclinic
Perchlorate, H_2O	Stout needles	+	±	1.589	1.598	1.654	Moderate, inclined	Monoclinic, ext. angle = 22°
Phosphate, $2H_2O$	Needles	+	±	1.589	1.597	1.655	Moderate, inclined	Monoclinic $y = b$
Selenate, $4.5H_2O$	Stout needles	+ (+)	−	1.598	1.600	1.661	Small, $\rho < v$, $2E$ = 10° ($\rho < v$, $2E$ = 14°)	Monoclinic
Sulfate, $5H_2O$	Stout needles	+ (+)	−	1.592	1.597	1.661 (1.594)	Small, weak, $2E$ = 16° ($2E$ = 16°31″)	Monoclinic
Sulfate, $6H_2O$	Plates	− (−)		1.595	1.613	1.615	Small $2E$ up to 30°	Orthorhombic or pseudotetragonal
Thiocyanate, H_2O	Stout needles	+	±	1.651	1.654	1.695	Small, inclined	Monoclinic, $y = b$

[a] All values in the table enclosed in parentheses are those previously reported in the literature.

Optical Crystallographic Data.—The optical crystallographic data were obtained for each of the salts reported in Table I. The methods used were those described in the texts by Chamot and Mason,[12] by Winchell,[13] and by various other books on optical mineralogy. The refractive indices were determined by the immersion method using daylight obtained from the northern sky. At times it was very difficult to get the crystals to stand on end so as to permit the measurement of a third refractive index. In such cases, finely ground glass-wool was mixed with the crystals and its particles held a sufficient number in upright position. The values for $2V$ were determined from the curvature of the isogyre in a centered optic axis interference figure or, in case of the smaller angles, by measuring the distance between the optic axes with an eyepiece micrometer, calibrated against a mineral of a known axial angle. The determination of this angle is not extremely accurate, therefore, the values for $2V$ are reported in the table as large, moderate or small. The extinction angle was determined by turning the crystal to extinction and measuring the angle between this position and the long edge of the crystal. The refractive indices, together with other optical data, are given in Table II.

Summary

1. Five new salts of strychnine have been prepared and described.

2. The data in the literature concerning thirteen salts of strychnine have been redetermined, and corrections have been made in the water of crystallization for the dioxalate, malonate and the selenate.

3. The anhydride for each salt was prepared and analyzed.

4. The optical crystallographic data for eighteen salts of strychnine have been determined.

BOULDER, COLORADO

[12] Chamot and Mason, "Handbook of Chemical Microscopy," John Wiley and Sons, Inc., New York, 1930.

[13] Winchell, "Elements of Optical Mineralogy," John Wiley and Sons, Inc., New York. 1928.