indices. The crystals exhibit strong birefringence and are either of the orthorhombic or the monoclinic system.

The rectangular tablets are perpendicular to the optic normal, as indicated by their interference figure, and show parallel extinction.

The six-sided tablets are perpendicular to the acute bisectrix and extinguish parallel to the elongation. This plane of extinction contains the axial plane. The crystals exhibit marked dispersion of the optic axes ($v > \rho$) and are optically negative. The apparent optic axial angle is greater than 120° ($2E > 120°$). The angles at opposite ends of the crystals are about 106° (determined, 105.5°, 106.2°).

Summary

1. 2-Furylacetic acid has been synthesized from furfural by a series of reactions which does not involve the use of mineral acids.

2. It has been shown that an acid prepared by the nitrile synthesis from α-furfuryl chloride, and described as 2-furylacetic acid, is actually the isomeric 5-methyl-2-furoic acid. It appears probable that rearrangement of the α-furfuryl group into the 5-methylfuryl group occurred in the reaction of α-furfuryl chloride with aqueous sodium cyanide solution.

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[CONTRIBUTION FROM THE CHEMISTRY DEPARTMENT OF THE UNIVERSITY OF ILLINOIS]

CERTAIN DIALKYL ACETIC ACIDS CONTAINING 12, 13 AND 14 CARBON ATOMS AND THEIR BACTERICIDAL ACTION TOWARD B. LEPRAE. XVIII[1,2]

BY B. F. ARMENDT AND ROGER ADAMS

RECEIVED DECEMBER 9, 1929 PUBLISHED MARCH 6, 1930

In a previous paper a comparison of a complete series of octadecanoic acids and a complete series of hexadecanoic acids of the general formula $RCH(CO_2H)R'$ were prepared in which R and R′ were alkyl groups of the proper size to give the desired molecular weight. The various octadecanoic acids gave irregular bactericidal values but the hexadecanoic acids showed values which increased regularly as the carboxyl group was moved toward the middle of the chain. The conclusion was drawn that the bactericidal action was due to the particular physical properties of the acids and that among these properties molecular weight appeared to be very important. In several of the series of acids studied which contained a ring structure, compounds containing less than 16 carbon atoms were

[1] Three previous papers in this field are XV, Stanley, Jay and Adams, THIS JOURNAL, **51**, 1261 (1929); XVI, Ford and Adams, *ibid.*, **52**, 1259 (1930); XVII, Browning, Woodrow and Adams, *ibid.*, **52**, 1281 (1930).

[2] This communication is in partial fulfilment of the requirements for the Degree of Doctor of Philosophy in Chemistry at the University of Illinois.

invariably less or non-bactericidal. This investigation was undertaken to prepare a few representatives of the dialkyl acetic acids containing 12, 13 and 14 carbon atoms to compare them with those of higher molecular weight. The bacteriological results are given in Table I. The authors are indebted to Dr. W. M. Stanley for the bacteriological tests.

Table I

Bacteriological Action of Dialkyl Acetic Acids to *B. Leprae*

		Dilutions of sodium salts in thousandths											
		5	15	25	50	62	74	85	100	125	155	192	250
C_{12}	*iso*-$C_3H_7CH(CO_2H)C_7H_{15}$	+	+	+	+	+	+	+	+	+	+	+	+
	$C_4H_9CH(CO_2H)C_6H_{13}$	+	+	+	+	+	+	+	+	+	+	+	+
	$C_5H_{11}CH(CO_2H)C_5H_{11}$	+	+	+	+	+	+	+	+	+	+	+	+
C_{13}	$C_4H_9CH(CO_2H)C_7H_{15}$	−	−	+	+	+	+	+	+	+	+	+	+
	$C_5H_{11}CH(CO_2H)C_6H_{13}$	−	−	+	+	+	+	+	+	+	+	+	+
C_{14}	$C_4H_9CH(CO_2H)C_8H_{17}$	−	−	−	−	±	−	±	±	+	+	+	+
	$C_5H_{11}CH(CO_2H)C_7H_{15}$	−	−	−	−	−	±	±	±	+	+	+	+
	$C_6H_{13}CH(CO_2H)C_6H_{13}$	−	−	−	−	−	±	±	−	±	+	+	+

It can be seen that the C_{12} acids are non-bactericidal, the C_{13} very slightly bactericidal and the C_{14} somewhat more. Even the C_{14} acids, however, are much lower in action than the C_{16} acids and somewhat less than the C_{18}, thus confirming the previous conclusion that molecular weight is an important factor.

The acids were all prepared by saponification of the proper disubstituted malonic esters.

Table II

Diethyl Dialkyl Malonates

Formula	B. p., °C.	d_4^{25}	n_D^{25}	Calculated, % C	Calculated, % H	Found, % C	Found, % H
Iso-$C_3H_7C(CO_2C_2H_5)_2C_7H_{15}$	137–140 (4 mm.)	0.9249	1.4375	67.94	10.74	69.70	11.33
$C_5H_{11}C(CO_2C_2H_5)_2C_5H_{11}$	147–149 (4.5 mm.)	.9334	1.4343	67.94	10.74	67.60	10.80
$C_4H_9C(CO_2C_2H_5)_2C_6H_{13}$	143–147 (4 mm.)	.9333	1.4347	67.94	10.74	67.90	10.83
$C_4H_9C(CO_2C_2H_5)_2C_7H_{15}$	138–140 (3.5 mm.)	.9288	1.4365	68.71	10.90	68.70	11.20
$C_5H_{11}C(CO_2C_2H_5)_2C_6H_{13}$	146–149 (4 mm.)	.9300	1.4361	68.71	10.90	68.50	11.27
$C_4H_9C(CO_2C_2H_5)_2C_8H_{17}$	156–159 (4 mm.)	.9263	1.4360	69.40	11.05	69.20	11.04
$C_5H_{11}C(CO_2C_2H_5)_2C_7H_{15}$	163.5–165 (5 mm.)	.9239	1.4371	69.40	11.05	69.80	11.37
$C_6H_{13}C(CO_2C_2H_5)_2C_6H_{13}$	155–158 (4 mm.)	.9249	1.4373	69.40	11.05	69.70	10.90

Table III

Dialkyl Acetic Acids

Formula	B. p., °C.	d_4^{20}	n_D^{20}	Calculated, % C	Calculated, % H	Found, % C	Found, % H
Iso-$C_3H_7CH(CO_2H)C_7H_{15}$	133–134 (4 mm.)	0.9429	1.4399	71.92	12.07	71.70	12.05
$C_5H_{11}CH(CO_2H)C_5H_{11}$	141–143 (4 mm.)	.8900	1.4381	71.92	12.07	71.63	12.16
$C_4H_9CH(CO_2H)C_6H_{13}$	134–135 (4 mm.)	.8945	1.4391	71.92	12.07	71.67	12.02
$C_4H_9CH(CO_2H)C_7H_{15}$[a]	148–149 (3 mm.)	.8911	1.4409	72.80	12.23	72.55	12.43
$C_5H_{11}CH(CO_2H)C_6H_{13}$	149–150 (4 mm.)	.8850	1.4410	72.80	12.23	72.31	12.14
$C_4H_9CH(CO_2H)C_8H_{17}$	160–161 (4 mm.)	.8873	1.4435	73.60	12.36	73.25	12.42
$C_5H_{11}CH(CO_2H)C_7H_{15}$	155.5–157 (4 mm.)	.8900	1.4430	73.60	12.36	73.50	12.10
$C_6H_{13}CH(CO_2H)C_6H_{13}$	159–160 (4 mm.)	.8895	1.4421	73.60	12.36	73.45	12.23

[a] Levene and Taylor, *J. Biol. Chem.*, **54**, 351 (1922), report the constants b. p. 179° (13 mm.), d_4^{20} 0.8860, n_D^{20} 1.4403 and on the ester as b. p. 177–178° (12 mm.), d_4^{20} 0.9318, n_D^{20} 1.4366.

Experimental

The general methods of preparation of the various intermediates have been described in previous articles in this series.

Summary

1. A number of dialkyl acetic acids having 12, 13 and 14 carbon atoms in the acid molecule have been prepared and tested for bactericidal action toward *B. Leprae*.

2. The dodecanoic acids have no bactericidal action, the tridecanoic acids practically no action, and the tetradecanoic acids have slight bactericidal action. This indicates as in previous researches that the molecular weight of the molecule plays an important role.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UPJOHN COMPANY]

MICRO-DETERMINATION OF SULFUR BY FUSION

BY HAROLD EMERSON

RECEIVED DECEMBER 13, 1929 PUBLISHED MARCH 6, 1930

In the course of several sulfur determinations by the Pregl method, the question was raised as to the possibility of finding a method which would require less apparatus and manipulation.

The method of Récsei[1] was unsatisfactory because of the tendency of sodium peroxide to deteriorate on standing, also the danger accompanying the use of it, the action at times taking place with explosive violence. It was found that the macro method employing potassium nitrate as an oxidizing agent could be modified for use as a micro method.

Experimental

Prepare the fusion mixture by mixing four parts by weight of sodium carbonate and three parts of potassium nitrate and grinding the mixture to a fine powder. This can be kept indefinitely.

Weigh a finely powdered sample equivalent to approximately 0.5 mg. of sulfur and mix thoroughly with 100 times its weight of the fusion mixture in a 20-cc. nickel crucible. Sprinkle a thin layer of the mixture over the top to prevent sulfur fumes from escaping. Place a close-fitting cover on the crucible and set it in a porcelain crucible of convenient size.

With a Bunsen burner apply a very low flame, gradually increasing it during ten minutes until the maximum is reached. Continue heating for fifteen minutes, then shut off the flame and allow the crucible to cool.

Dissolve the contents of the crucible in 5 cc. of warm water and filter into a 30-cc. beaker. Wash the crucible and filter with 10 cc. more water and add to the solution. Acidify with hydrochloric acid and heat to boiling.

[1] Andor Récsei, *Chem.-Ztg.*, **50**, 785 (1926).