TABLE I

ALKYL SALICYLATES

Alky	B. p.[a] or m. p., °C.[a]	Yield, %	$n^{25}D$	Formula	Analyses, %[b] Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	Deriv.[c]	M. p., °C.	Formula	% N Calcd.	% N Found
—C_4H_9-n	145–147 (16 mm.)	94	1.5130	$C_{11}H_{14}O_3$	68.04	68.17	7.22	7.29[d]	A	85	$C_{18}H_{16}N_2O_8$	7.22	6.84[e]
									B[f]	59.5–60	$C_{11}H_{12}N_2O_7$		
—C_6H_{13}-n	167–168 (12 mm.)	93	1.5049	$C_{13}H_{18}O_3$	70.27	70.02	8.11	7.44					
—C_8H_{17}-n	172–173 (6 mm.)	55	1.4983	$C_{15}H_{22}O_3$	72.00	71.62	8.80	9.14	A	45	$C_{22}H_{24}N_2O_8$	6.30	6.49[g]
—$C_{10}H_{21}$-n	139–141 (0.08 mm.)	61	1.4937	$C_{17}H_{26}O_3$	73.38	73.23	9.36	9.28					
—$C_{12}H_{25}$-n	158–163 (0.08 mm.)	70		$C_{19}H_{30}O_3$	74.51	74.88	9.81	9.70	A	42	$C_{26}H_{32}N_2O_8$	5.60	5.57[h]
	25								B	45.5–46	$C_{19}H_{28}N_2O_7$	7.07	7.12
—$C_{14}H_{29}$-n	40.5	50		$C_{21}H_{34}O_3$	75.45	75.10	10.17	10.42	B	52–53	$C_{21}H_{32}N_2O_7$	6.60	7.01
—$C_{16}H_{33}$-n	43–44	55		$C_{23}H_{38}O_3$	76.24	76.01	10.50	9.98	B	50–51	$C_{23}H_{37}NO_5$[i]	3.27	3.44
—$C_{18}H_{37}$-n	53	85		$C_{25}H_{42}O_3$	76.92	77.26	10.77	11.06	B	66.5–67	$C_{25}H_{40}N_2O_7$	5.83	5.52
—$CH_2CH(C_2H_5)C_4H_9$-n	189–190 (21 mm.)	67	1.5018	$C_{15}H_{22}O_3$	72.00	71.45	8.80	8.41	A	95	$C_{17}H_{14}N_2O_9$	7.18	6.97[j]
—$CH_2CH_2OCH_3$	145 (10 mm.)	84	1.5227	$C_{10}H_{12}O_4$	61.22	60.80	6.12	6.15					
—$CH_2CH_2OC_2H_5$	152 (10 mm.)	85	1.5157	$C_{11}H_{14}O_4$	62.86	61.68	6.67	6.09					

[a] All melting points and boiling points are uncorrected. [b] Microanalyses are by Misses P. Curran and A. Rainey. [c] A = 3,5-Dinitrobenzoate; B = 3,5-dinitro. [d] This is a previously reported compound; see Sah and Ma (ref. 1) and also Croxall, Sowa and Nieuwland, *J. Org. Chem.*, 2, 253 (1937). [e] %C, calcd.: 55.67; found: 55.92; %H, calcd.: 4.12, found: 4.16. [f] Prepared and analyzed by Sah and Ma (ref. 1). [g] %C, calcd.: 59.46; found: 59.21; %H, calcd.: 5.41; found: 4.69. [h] %C, calcd.: 62.40; found: 62.34; %H, calcd.: 6.40; found: 6.08. [i] The mononitro derivative was obtained with this ester. [j] %C, calcd.: 52.31; found: 51.78; %H, calcd.: 3.59; found: 3.35.

homologs. The esters and derivatives prepared are listed in Table I.

The esters were all prepared by standard procedures, the actual conditions used for each one being determined by the boiling point and water-solubility of the alcohol being used.

RESEARCH LABORATORIES FREEMAN H. MCMILLAN
WINTHROP CHEMICAL COMPANY, INC.
RENSSELAER, NEW YORK JOHN A. KING

RECEIVED OCTOBER 4, 1945

N-Furfurylmaleamic Acid and N,N-Furfurylmethylmaleamic Acid

The following two derivatives were obtained by mixing maleic anhydride with an equivalent amount of the corresponding amine in ether. Considerable heat was evolved in both instances, with the products precipitating almost immediately.

N-Furfurylmaleamic Acid.—Seven grams of furfurylamine gave 10 g. of white plates, m. p. 114° (uncor.), recrystallized first from an alcohol–ether mixture, and then from alcohol. The crystals are soluble in alcohol, water, ethyl acetate and acetone, insoluble in ether.

Anal. Calcd. for $C_9H_9O_4N$: C, 55.33; H, 4.62; neut. equiv., 195. Found: C, 55.28; H, 4.59; neut. equiv., 192.

N,N-Furfurylmethylmaleamic Acid.—Five grams of furfurylmethylamine gave 5 g. of white product, m. p. 172–173° (uncor.), recrystallized twice from a mixture of alcohol and ether, soluble in alcohol, ethyl acetate, water, acetone and methanol, insoluble in ether.

Anal. Calcd. for $C_{10}H_{11}O_4N$: C, 57.42; H, 5.26; neut. equiv., 209. Found: C, 57.43; H, 5.29; neut. equiv., 206.

CHEMISTRY LABORATORY
UNIVERSITY OF COLORADO
BOULDER, COLORADO WERNER HERZ

RECEIVED SEPTEMBER 27, 1945

β',β'',β'''-Triethoxytriethylamine[1]

This non-toxic compound resulted instead of trivinylamine by heating a solution of 6.24 g. (0.111 mole) of potassium hydroxide in 25 cc. 95% ethanol, under reflux, with 4.42 g. (0.0183 mole) of trichlorotriethylamine hydrochloride [McCombie and Purdie, *J. Chem. Soc.* 1217 (1935)] for three hours. After filtration of the potassium chloride (theoretical quantity) the alcoholic filtrate was evaporated under 10 mm., the residue was taken up in water, thrice extracted with ether and the ether solution dried with magnesium sulfate. Distillation at 134–137° under 12 mm. yielded 2.80 g. (66%) of triethoxytriethylamine, $d^{23}{}_4$ 0.936. This compound could be precipitated by hydrogen chloride from ethanol solution as its hydrochloride, m. p. 193–195°. The amine was analyzed.

Anal. Calcd. for $C_{12}H_{27}O_3N$: C, 61.8; H, 11.6; neut. equiv., 233. Found: C, 61.7; H, 11.5; neut. equiv., 221.

When the amine was treated with one equivalent of picric acid in ethanol and crystallized from this medium, a picrate m. p. 65–66° was formed.

Anal. Calcd. for $C_{18}H_{30}O_{10}N_4$: C, 46.75; H, 6.54. Found: C, 47.0; H, 6.26.

When two equivalents of picric acid were used, the compound 2 picric acid: 1,β',β,''β'''-triethoxytriethylamine was formed, m. p. 229° after crystallization from alcohol.

Anal. Calcd. for $C_{24}H_{33}O_{17}N_7$: C, 41.7; H, 4.82. Found: C, 42.0; H, 5.09.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF TORONTO H. H. RICHMOND
TORONTO, ONTARIO, CANADA GEORGE F. WRIGHT

RECEIVED SEPTEMBER 7, 1945

Di-(trimethylsilyl) Sulfate and Lead Trimethylsilanolate

Di-(trimethylsilyl) sulfate has been prepared by the reaction

$$2(CH_3)_3SiCl + H_2SO_4 \longrightarrow [(CH_3)_3Si]_2SO_4 + 2HCl$$

It is a white crystalline solid, easily hydrolyzed by water to hexamethyldisiloxane and sulfuric acid. The corresponding chloride[1] and phosphate[2] are liquids, likewise easily hydrolyzable.

Lead trimethylsilanolate has been prepared by the reaction

$$(CH_3)_3SiOH + PbO \longrightarrow [(CH_3)_3SiO]_2Pb + H_2O$$

It is a white crystalline solid, soluble in organic solvents, and is easily hydrolyzed by dilute sulfuric acid.

Di-(trimethylsilyl) Sulfate.—Nine and eight-tenths grams of sulfuric acid was added dropwise to 23.8 g. of $(CH_3)_3SiCl$ with violent shaking. Hydrogen chloride was

---

(1) This compound is mentioned in French Patent 711,560 (1931), but no description of its preparation or properties is recorded. The compound also is incorrectly indexed in *C. A.*, 31, 10. [illegible] (1937), since the reference contains no mention of it.

(1) A. G. Taylor and B. V. dG. Walden, THIS JOURNAL, 66, 842 (1944); W. F. Gilliam and R. O. Sauer, *ibid.*, 66, 1793 (1944).

(2) R. O. Sauer, *ibid.*, 66, 1707 (1944).