

TABLE I  
 ALKYL SALICYLATES

Alky	B. p. <sup>a</sup> or m. p. °C.	Yield, %	n <sub>D</sub> <sup>b</sup>	Formula	Analyses, % <sup>b</sup>				De- riv. <sup>c</sup>	M. p., °C.	% N		
					Calcd.	Found	Calcd.	Found			Calcd.	Found	
—C <sub>4</sub> H <sub>9</sub> -n	145-147 (16 mm.)	94	1.5130	C <sub>11</sub> H <sub>14</sub> O <sub>2</sub>	68.04	68.17	7.22	7.29 <sup>d</sup>	A	85	C <sub>11</sub> H <sub>14</sub> N <sub>2</sub> O <sub>7</sub>	7.22	6.84 <sup>e</sup>
—C <sub>6</sub> H <sub>11</sub> -n	167-168 (12 mm.)	93	1.5049	C <sub>11</sub> H <sub>14</sub> O <sub>2</sub>	70.27	70.02	8.11	7.44	B <sup>f</sup>	59.5-60	C <sub>11</sub> H <sub>14</sub> N <sub>2</sub> O <sub>7</sub>		
—C <sub>8</sub> H <sub>17</sub> -n	172-173 (6 mm.)	55	1.4983	C <sub>15</sub> H <sub>22</sub> O <sub>2</sub>	72.00	71.62	8.80	9.14	A	45	C <sub>15</sub> H <sub>22</sub> N <sub>2</sub> O <sub>7</sub>	6.30	6.49 <sup>g</sup>
—C <sub>10</sub> H <sub>21</sub> -n	139-141 (0.08 mm.)	61	1.4937	C <sub>17</sub> H <sub>26</sub> O <sub>2</sub>	73.38	73.23	9.36	9.28					
—C <sub>12</sub> H <sub>25</sub> -n	158-163 (0.08 mm.)	70		C <sub>19</sub> H <sub>30</sub> O <sub>2</sub>	74.51	74.88	9.81	9.70	A	42	C <sub>19</sub> H <sub>30</sub> N <sub>2</sub> O <sub>7</sub>	5.60	5.57 <sup>h</sup>
	25								B	45.5-46	C <sub>19</sub> H <sub>30</sub> N <sub>2</sub> O <sub>7</sub>	7.07	7.12
—C <sub>14</sub> H <sub>29</sub> -n	40.5	50		C <sub>21</sub> H <sub>34</sub> O <sub>2</sub>	75.45	75.10	10.17	10.42	B	52-53	C <sub>21</sub> H <sub>34</sub> N <sub>2</sub> O <sub>7</sub>	6.60	7.01
—C <sub>16</sub> H <sub>33</sub> -n	43-44	55		C <sub>23</sub> H <sub>38</sub> O <sub>2</sub>	76.24	76.01	10.50	9.98	B	50-51	C <sub>23</sub> H <sub>38</sub> N <sub>2</sub> O <sub>7</sub>	3.27	3.44
—C <sub>18</sub> H <sub>37</sub> -n	53	85		C <sub>25</sub> H <sub>42</sub> O <sub>2</sub>	76.92	77.26	10.77	11.06	B	66.5-67	C <sub>25</sub> H <sub>42</sub> N <sub>2</sub> O <sub>7</sub>	5.83	5.52
—CH <sub>2</sub> CH(C <sub>2</sub> H <sub>5</sub> )C <sub>4</sub> H <sub>9</sub> -n	189-190 (21 mm.)	67	1.5018	C <sub>15</sub> H <sub>22</sub> O <sub>2</sub>	72.00	71.45	8.80	8.41	A	95	C <sub>17</sub> H <sub>24</sub> N <sub>2</sub> O <sub>7</sub>	7.18	6.97 <sup>i</sup>
—CH <sub>2</sub> CH <sub>2</sub> OCH <sub>3</sub>	145 (10 mm.)	84	1.5227	C <sub>10</sub> H <sub>14</sub> O <sub>4</sub>	61.22	60.80	6.12	6.15					
—CH <sub>2</sub> CH <sub>2</sub> OC <sub>2</sub> H <sub>5</sub>	152 (10 mm.)	85	1.5157	C <sub>11</sub> H <sub>16</sub> O <sub>4</sub>	62.86	61.63	6.67	6.09					

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

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RECEIVED OCTOBER 4, 1945

### N-Furfurylmaaleamic Acid and N,N-Furfurylmethylmaaleamic 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-Furfurylmaaleamic 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<sub>9</sub>H<sub>8</sub>O<sub>4</sub>N: C, 55.33; H, 4.62; neut. equiv., 195. Found: C, 55.28; H, 4.59; neut. equiv., 192.

**N,N-Furfurylmethylmaaleamic 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<sub>10</sub>H<sub>11</sub>O<sub>4</sub>N: C, 57.42; H, 5.26; neut. equiv., 209. Found: C, 57.43; H, 5.29; neut. equiv., 206.

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RECEIVED SEPTEMBER 27, 1945

### β',β'',β'''-Triethoxytriethylamine<sup>1</sup>

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 hydro-

(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, 274 (1937), since the reference contains no mention of it.

chloride [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*<sub>4</sub><sup>20</sup>, 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<sub>12</sub>H<sub>27</sub>O<sub>3</sub>N: 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<sub>18</sub>H<sub>30</sub>O<sub>10</sub>N<sub>4</sub>: C, 46.75; H, 6.54. Found: C, 47.0; H, 6.26.

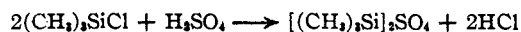
When two equivalents of picric acid were used, the compound 2 picric acid: 1,β',β'',β'''-triethoxytriethylamine was formed, m. p. 229° after crystallization from alcohol.

*Anal.* Calcd. for C<sub>24</sub>H<sub>42</sub>O<sub>17</sub>N<sub>7</sub>: C, 41.7; H, 4.82. Found: C, 42.0; H, 5.09.

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RECEIVED SEPTEMBER 7, 1945

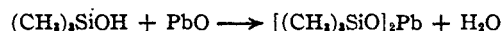
### Di-(trimethylsilyl) Sulfate and Lead Trimethylsilanolate

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



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

Lead trimethylsilanolate has been prepared by the reaction



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<sub>3</sub>)<sub>3</sub>SiCl with violent shaking. Hydrogen chloride was

(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).

evolved and the temperature of the reaction mixture fell. When all of the sulfuric acid had been added, the reaction mixture was distilled, and the white crystalline distillate which came over at 87–90° at 4 mm. pressure was analyzed by titration with 0.5 *N* sodium hydroxide. Sulfuric acid calculated for in a 0.5842 g. sample: 0.2360 g.; found: 0.2376 g. The freezing point of the material was measured and found to be 45–46° (uncor.). An attempt to distil di-(trimethylsilyl) sulfate at higher pressure and temperature resulted in violent auto-oxidation.

**Lead Trimethylsilylanolate.**—About 1 g. of lead monoxide was shaken at room temperature in 25 cc. of  $(\text{CH}_3)_3\text{SiOH}^3$  for two days. The yellow color of the lead monoxide gradually changed to white. On filtration of the mixture and evaporation of the filtrate a white crystalline

solid was obtained which was soluble in ether, toluene and absolute alcohol. For analysis, a sample was weighed into a centrifuge tube, dissolved in alcohol and diluted with dilute sulfuric acid. After standing overnight the precipitate was centrifuged, washed, dried at 110° and weighed as  $\text{PbSO}_4$ ; calcd.: Pb, 53.7. Found: Pb, 59.1.

The authors acknowledge with thanks the analytical work carried out by E. W. Balis.

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## COMMUNICATIONS TO THE EDITOR

### THE ISOLATION OF A NEW LEUKOPOIETIC FACTOR FROM LIVER<sup>1</sup>

Sir:

A crystalline substance has been isolated by one of us (J. K. C.) which may be necessary for the production of leukocytes in man. When administered intravenously in doses ranging from 20 to 60 mg. to malnourished patients with leukopenia there was an increase within three to five and one-half hours of 45% to 174% above the initial white cell count in 12 of 14 patients. The neutrophils increased from 37.4% to 257% above the initial levels during the same period.<sup>2</sup> Chemically the substance is not identical with folic acid,<sup>3</sup> adenylic acid<sup>4</sup> or the exudate factor,<sup>5</sup> although administration of these substances may produce a similar response. It is possible that the new substance is one of a group of substances whose lack in the diet is responsible for the nutritional macrocytic anemias described by Spies and Payne,<sup>6</sup> Moore, Vilter, Minnich and Spies,<sup>7</sup> and Wills<sup>8</sup> and it may be related similarly to the extrinsic factor of Castle.<sup>9</sup> Intramuscular injection of 30 mg. per day of an impure preparation elicited a positive hematological response in a patient with macrocytic anemia. Larger amounts of the substance are necessary before its anti-anemic properties can be correctly evaluated.

(1) The expenses of this special study were defrayed by grants from Eli Lilly and Company and the Research Corporation. General expense has been borne by many philanthropic persons, foundations and commercial concerns.

(2) Berry, Spies and Cline, *Southern Medical Journal*, **38**, 656 (1945).

(3) Berry, Spies and Doan, *ibid.*, **38**, 590 (1945).

(4) Berry, Doan and Spies, unpublished observations.

(5) Menkin, *Science*, **101**, 422 (1945).

(6) Spies and Payne, *The Journal of Clinical Investigation*, **12**, 220 (1933).

(7) Moore, Vilter, Minnich and Spies, *The Journal of Laboratory and Clinical Medicine*, **29**, 1226 (1944).

(8) Wills, *British Medical Journal*, **1**, 1059 (1931).

(9) Castle, *American Journal of Medical Science*, **178**, 748 (1929).

Isolation was accomplished by passing the portion of potent liver extract (Reticulogen, generously supplied by Dr. E. D. Campbell, The Lilly Research Laboratories) which was soluble in 66% acetone and insoluble in 90% acetone through acid activated Permutit. From a fraction of the filtrate a white neutral substance was obtained by fractional precipitation of a concentrated aqueous solution with acetone. By repeating this precipitation procedure and finally by slow evaporation of an aqueous solution, colorless crystals consisting of rosettes of whetstone-shaped needles were obtained. Solutions of these crystals were also colorless. The crystals did not melt up to 360° and left considerable white ash on ignition. Tests for the primary aliphatic amino group were positive while tests for arginine and tyrosine were negative. The biuret reaction was negative and a test for the carbohydrate primary carbinol group showed only a faint trace of precipitate, in all probability due to a slight contamination of the substance with carbohydrate containing fractions. Elementary analysis revealed the presence of C, 28.25%; H, 4.70%; N, 5.42%; Na (as sulfate) 49.67%; sulfur and phosphorus were absent.

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L. JOE BERRY  
TOM D. SPIES

RECEIVED OCTOBER 23, 1945

### THE ABSOLUTE RATE CONSTANTS IN THE POLYMERIZATION OF LIQUID VINYL ACETATE

Sir:

We have determined the absolute rate constants for the elementary steps in the polymerization of pure liquid vinyl acetate, utilizing a technique applied by Melville [*Proc. Roy. Soc. (London)*, **A163**, 511 (1937)] to polymerization in the vapor phase. The dependence of the rate of photopolymerization of vinyl acetate upon the frequency of a flashing