

the molecular weight of the dimeric formula. The substance is clearly diamagnetic and hence the monomeric  $\text{IO}_2$  unit does not occur in the solid state.

In principle one could further elucidate the detailed molecular structure by either a theoretical calculation of the expected magnetic susceptibility for each of the various models or through a comparison with the appropriate experimental values. Actually neither method is sufficiently refined to distinguish between the various structural possibilities. Using the method of Angus<sup>3</sup> one can sum the appropriate values of +4 iodine and -2 oxygen to obtain a value of  $-96.52 \times 10^{-6}$  for  $\text{I}_2\text{O}_4$ . Alternately, assuming a structure of  $(\text{IO}^+)(\text{IO}_3^-)$  and using Angus' values of +3 and +5 iodine, one calculates  $-97.10 \times 10^{-6}$ . These discrepancies are not unexpected and indeed are no larger than might be anticipated for such a calculation. Summation of Pascal's<sup>4</sup> experimental values leads to  $-107.64 \times 10^{-6}$ ; here again one might expect too large a value since Pascal's value for iodine is applicable to a single bonded covalent iodide atom. An experimental value<sup>5</sup> of  $\text{IO}_3^-$  of  $-51.4 \times 10^{-6}$  would lead, on the assumption of the iodyl iodate model, to an experimental value of  $-24.88 \times 10^{-6}$  for the iodyl ion.

**Acknowledgment.**—We are indebted to the Office of Naval Research for financial support of this work.

(3) Angus, *Proc. Roy. Soc. (London)*, **A136**, 569 (1932).

(4) Selwood, "Magnetochemistry," Interscience Publishers Inc., New York, N. Y., 1943, p. 52.

(5) V. C. G. Trew, *Trans. Faraday Soc.*, **37**, 476 (1941).

DEPT. OF CHEMISTRY

UNIVERSITY OF SOUTHERN CALIFORNIA

LOS ANGELES 7, CALIF.

RECEIVED MAY 31, 1950

---



---

## NEW COMPOUND

---



---

### Some Derivatives of *o*-Cresol

Some derivatives of *o*-cresol were prepared as reference compounds in other work.

**1-(*o*-Toloxyl)-3-chloro-2-propanone.**—A 16.8-g. (0.091 mole) sample of freshly distilled *o*-toloxylacetyl chloride was converted to the diazoketone essentially according to the procedure of Newman and Beal.<sup>1</sup> The diazoketone, in solution in 650 ml. of ether, was not isolated but decomposed by passing dry hydrogen chloride into the solution until the evolution of nitrogen ceased. After wash-

(1) Newman and Beal, *This Journal*, **71**, 1506 (1949).

ing with 150 ml. of cold water and two 50-ml. portions of 5% sodium carbonate, the ether solution was dried with sodium sulfate and evaporated to a red oil; yield 16.3 g. (90%). Distillation at 0.2 mm. gave 2.4-g. forerun boiling below 87° and 10.3 g. (57%) of chloroketone distilling between 87–92°. Two recrystallizations from petroleum ether gave colorless needles, m. p. 50°. The compound recrystallized immediately after melting and remelted at 54°.

*Anal.*<sup>2</sup> Calcd. for  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{Cl}$ : C, 60.46; H, 5.58. Found: C, 60.61; H, 5.71.

**$\beta$ -(2-Methyl-4-nitrophenoxy)-lactic Acid.**—Ten grams of  $\beta$ -chlorolactic acid, 24.6 g. of 2-methyl-4-nitrophenol and 85 ml. of 12% sodium hydroxide were heated two hours on the steam-bath. The cooled aqueous solution was acidified to congo red and extracted with ether. The substituted lactic acid separated on acidification of a potassium bicarbonate extract of the ether solution. Recrystallization from hot water, after treatment with Darco G-60 gave 3.27 g. (17%) of a pale yellow powder, contaminated by a few bright yellow needles which were separated manually. The major product melted at 111° after recrystallization from water.

*Anal.* Calcd. for  $\text{C}_{10}\text{H}_{11}\text{O}_6\text{N}$ : C, 49.79; H, 4.60. Found: C, 49.51; H, 4.48.

The minor product, m. p. 85°, after recrystallization from water, was not further characterized.

***o*-Toloxylacetyl glycine.**—Five ml. of *o*-toloxylacetyl chloride (b. p. 108–109° (8 mm.)) was added with stirring and cooling to 13.3 g. of glycine homogenized with 13 ml. of 20% sodium hydroxide. When the reaction had subsided, alternate additions of 13 ml. of sodium hydroxide and 5 ml. of the acid chloride were made until a total of 32.7 g. of the acid chloride and 76 ml. of sodium hydroxide had been added. After standing an hour and a half, the reactants were poured into chipped ice, acidified to congo red with dilute hydrochloric acid and filtered. The crude product weighing 36.5 g., m. p. 185–186°, was recrystallized from 450 ml. of ethanol; yield 28.5 g., 70%, m. p. 190–190.5°, raised to 191–191.5° by two further recrystallizations from ethanol.

*Anal.* Calcd. for  $\text{C}_{11}\text{H}_{13}\text{O}_4\text{N}$ : C, 59.19; H, 5.87. Found: C, 59.23; H, 5.89.

**Ethyl  $\beta$ -(*o*-Toloxyl)-lactate.**—Nine grams of  $\beta$ -(*o*-toloxyl)-lactic acid<sup>3</sup> was converted to the ester by refluxing 3 hours with 30 ml. of absolute ethanol containing 5% hydrochloric acid. Distillation of the residue after removal of hydrochloric acid and excess alcohol gave 6.45 g. (67%) of the ester, b. p. 99–102° (0.2 mm.).

*Anal.* Calcd. for  $\text{C}_{12}\text{H}_{15}\text{O}_4$ : C, 64.27; H, 7.19. Found: C, 64.23; H, 7.38.

DIVISION OF PHARMACOLOGY AND TOXICOLOGY

UNIVERSITY OF ROCHESTER

SCHOOL OF MEDICINE AND DENTISTRY

ROCHESTER 20, NEW YORK

RICHARD P. RILEY<sup>4,5</sup>

RECEIVED MARCH 13, 1950

(2) Microanalyses by C. W. Beazley, Skokie, Illinois.

(3) Riley, *This Journal*, **72**, 5712 (1950).

(4) Aided by a grant from The National Foundation for Infantile Paralysis, Inc.

(5) University of California Atomic Energy Project, Post Office Box 31, Beverly Hills, Calif.