

Abstracts from American and Foreign Journals.

American Chemical Journal.

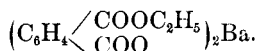
Abstractor, J. P. BATTERSHALL, Ph.D.

A Method for the Proximate Analysis of Plants, HENRY B. PARSONS (I, 377).—Will be noticed elsewhere.

On the Estimation of Phosphoric Acid as Magnesian Pyrophosphate, F. A. GOOCH (I, 391).—See this JOURNAL (I, 537).

Laboratory Notes, ARTHUR MICHAEL (I, 413).

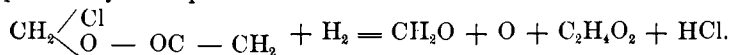
On monoethylphthalate (I, 413).—This compound was prepared by heating equal parts of absolute alcohol and phthalic anhydride in a flask connected with an upright condenser, for ten hours at 100°, evaporating the excess of alcohol at a low temperature, dissolving the remaining residue in water, and neutralizing it in the cold with barium carbonate. In this way the barium salts of monoethylphthalate and phthalic acid are formed, which can be separated by means of their different solubility in water, the phthalate being much the less soluble. Barium monoethylphthalate forms long, concentrically grouped needles, which are very soluble in water, and possesses the formula,



The corresponding silver salt is prepared by precipitating a concentrated aqueous solution of the barium salt with silver nitrate; it forms beautiful long needles, which blacken on exposure to the light. The free monoethylphthalate, which was obtained by treating a solution of the barium salt with the calculated quantity of sulphuric acid and evaporating the filtrate in vacuo, is a heavy oil, remarkably unstable when heated, even upon a water-bath. With a view of obtaining the chloride, $\text{C}_6\text{H}_4 \begin{array}{l} \diagup \text{COOC}_2\text{H}_5 \\ \diagdown \text{COCl} \end{array}$, equivalent amounts of monoethylphthalate and phosphorus trichloride were heated in a retort over a water-bath. The results of this reaction were the evolution of chlorhydric acid and ethyl chloride and the formation of a heavy oil, which proved to be diethylphthalate, $\text{C}_6\text{H}_4(\text{COOC}_2\text{H}_5)_2$, showing that the chloride of monoethylphthalate, first formed, undergoes a condensation, and is decomposed into phthalic anhydride and ethyl chloride, the formation of diethylphthalate being probably due to a portion of the ethyl chloride reacting on unchanged monoethylphthalate.

On a new formation of ethyl mustard oil (I, 416).—A mixture of ethyl iodide and mercuric sulphocyanate was heated in a tube for about two hours, at 145°, and the resulting compounds brought into a flask connected with a condenser, and treated with steam as long as an oil distilled over. This oil was separated, dried over CaCl₂, and distilled at 135°. It possessed the odor of ethyl mustard oil, and, when mixed with aniline, gave a precipitate which, when purified by crystallization from water, possessed the properties of phenyl-ethylsulphocarbamide. The amount of ethylsulphocyanate formed in the above reaction greatly exceeds that of the ethyl mustard oil. Both compounds, however, are produced in very small quantities, the principal product of the reaction being a red mass, previously mentioned by Schlagenhauffen,* and probably consisting of a double compound of ethyl mustard oil with mercuric iodide.

On the preparation of methyl aldehyde (I, 418).—Monochloromethyl acetate is first prepared by passing a current of dry chlorine gas through methyl acetate for about seven hours, the liquid being placed in a flask surrounded by cold water. On submitting the contents of the flask to fractional distillation, and separating the portion which passes over between 100–120°, a very satisfactory yield of monochloromethyl acetate is obtained. This, when mixed with water and placed in a strong closed flask, and heated for about an hour at 100°, furnishes an abundant supply of methyl aldehyde, which is separated by exactly neutralizing the contents of the flask with sodium hydrate, and distilling. The above reaction is expressed by the equation:



On the "migration of atoms in the molecule," and Reimer's chloroform aldehyde reaction (I, 420).—This article is wholly speculative in character and, not forming a record of personal investigation, possesses only a theoretical interest.

On the Oxidation of Substitution Products of Aromatic Hydrocarbons, IRA REMSEN and C. FAHLBERG (I, 426).

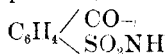
On the Oxidation of Orthotoluenesulphamide.

Preparation of orthotoluenesulphamide (I, 426).—The sulphonic acids formed by treating toluene with fuming sulphuric acid, were converted by aid of the usual reactions into the corresponding chlorides, from which the liquid orthotoluenesulphochloride was

* Ann. Chem. Phys. (3), 56, 297.

isolated. This, upon treatment with ammonia, was converted into orthotoluenesulphamide, which melts at 153–154° and, by fusion with potassium hydroxide, yields salicylic acid.

Formation of benzoic sulphinide.—The amide thus obtained was heated for ten hours on a water-bath, together with potassic permanganate, the excess of permanganate reduced by adding alcohol, the solution filtered, and hydrochloric acid added to the concentrated filtrate. The precipitate formed, gave, on analysis, figures corresponding to the formula, $C_7H_5O_3NS$, which differs from the normal sulphamine acid produced from the corresponding para compound, in that it contains a molecule of water less. This body may be regarded as a condensation product, having the formula,



The authors note that a similar grouping of a carboxyl and an amido group, is a characteristic feature of some compounds allied to indigo, such as isatine, and suggest, as a proper designation of such compounds, the term *sulphinides*. Benzoic sulphinide (or anhydrosulphaminebenzoic acid) crystallizes in short prismatic forms, which fuse at 220°. It is difficultly soluble in cold water, but dissolves readily in alcohol and in ether. *It possesses a marked sweet taste, being much sweeter than cane sugar.* By fusion with potassium hydroxide, benzoic sulphinide is easily and completely converted into salicylic acid. By neutralizing its aqueous solution with various carbonates, the corresponding salts were obtained. In this manner the barium, magnesium, lead, potassium and ammonium compounds were prepared.

Orthosulphobenzoic acid.—On further evaporating the filtrate obtained in the preparation of benzoic sulphinide, a crystalline potassium salt separates out, which is purified from the accompanying potassium chloride by recrystallization. The analysis gave figures corresponding to monopotassiumsulphobenzoate, $C_6H_4 \begin{array}{l} \diagup SO_2K \\ \diagdown CO_2H \end{array}$

This compound belongs to the ortho series, as is shown by the formation of salicylic acid when it is fused with potassium hydroxide. It is precipitated from concentrated solutions by addition of hydrochloric acid and, upon recrystallization, forms beautiful, large rhombic plates. Benzoic sulphinide and orthosulphobenzoic acid are formed in about equal quantities by the oxidation of orthotoluenesulphamide, *i. e.*, from two molecules of the latter one molecule of each of the former compounds is obtained. Orthosulphobenzoic acid, $C_6H_4 \begin{array}{l} \diagup CO_2H \\ \diagdown SO_2H \end{array}$ crystallizes in fine large plates, which fuse at 240°,

undergoing decomposition ; it is easily soluble in water, but scarcely dissolves in alcohol or in ether.

Acid barium orthosulphobenzoate crystallizes in needles, which suffer partial decomposition at 250° . Orthosulphobenzoic acid can be prepared from benzoic sulphinide by heating this compound in a sealed tube to 150° , with concentrated hydrochloric acid. In a reverse manner, when the neutral potassium salt of orthosulphobenzoic acid is treated with phosphorus pentachloride, the product treated with ammonia, and hydrochloric acid is added, a precipitation of benzoic sulphinide occurs. Phthalic acid, when submitted to the above treatment, yields an imide of the formula, $C_6H_4 \left\langle \begin{array}{c} CO \\ CO \end{array} \right\rangle NH$, differing, however, in properties from Laurent's phthalimide. The authors further mention an unsuccessful attempt to convert orthosulphobenzoic acid into phthalic acid, by treatment with sodium formate.

On an Unusual Case of Electrolysis, J. W. MALLET(I, 438).—The author notes that a Smee's galvanic battery, not being in use, was laid aside, when the plates, which were originally raised out of the acid, accidentally became immersed in the liquid, which was of unusual strength. It was afterwards found that the zinc plates were very much corroded, and that one of the sheets of platinized silver foil was thickly coated with zinc, of which over 30 grms were removed. The battery liquid had doubtless first become converted into zinc sulphate, which had subsequently acted as a "decomposing cell," and metallic zinc was electrolytically deposited from its salt.

Berichte der Deutschen Chemischen Gesellschaft.

Abstractor, ARNO BEHR, Ph.D.

On Methylated Diethyleneamines, H. F. MORLEY (I3, 222).—Monomethyldioxethyleneamine and dimethyloxethyleneamine were prepared by the action of chlorhydrine ($C_2H_4Cl.OH$), on methylamine and dimethylamine respectively.

On the Oxidation of Substituted Phenols, G. MAGATTI (I3, 224).—The acetylated compound of tetrabromparadiphenol, dissolved in glacial acetic acid, is oxidized by fuming nitric acid. The result is a crystalline body of the formula of tetrabromdiphenylquinone, $C_6H_2.C_6H_2.Br_4O_2$. This is a simple analogue to cedrret, a colored substance found in wood-tar.