

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Regular Meeting, Dec. 8, 1882.*

The meeting was called to order at 8.40 P. M. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The board of directors had no report. The treasurer stated that we had a balance of about \$200 in the treasury.

The librarian and curator had no report. The committee on endowment fund, stated that the labor of raising the fund was for the present suspended, there being a larger fund in the possession of the society than was needed for the publication of the original papers presented at the present time.

The committee on papers and publications then stated that it had no formal report to present, but trusted that the Society would judge them by the work they had done.

The committee further stated that the November number of journal is in type, and that it will probably be issued the coming week.

The next in order was the election of officers for the ensuing year.

*For President.*—The three following gentlemen received the greatest number of votes: A. R. Leeds, E. R. Squibb, J. C. Booth.

Prof. Leeds declined to be a candidate, on the ground that it was of the greatest importance to the interests of the Society, as a national body, that the honorary office of President should be filled by a non-resident member.

It was then moved that the Society proceed to vote for the two other gentlemen standing highest on the list. This being seconded was duly carried.

Prof. J. C. Booth receiving the greatest number of votes, was duly elected President.

*For Vice Presidents.*—The following gentlemen received the greatest number of votes:

- |                            |                                |
|----------------------------|--------------------------------|
| 1. James H. Stebbins, Jr., | } Three Local Vice-Presidents. |
| 2. A. R. Leeds,            |                                |
| 3. C. F. Chandler,         |                                |
| 4. Arno Behr,              |                                |
| 5. P. Schweitzer,          |                                |
| 6. N. T. Lupton.           |                                |

Mr. Casamajor then moved that the resolution preventing members who have read papers before the Society, from publishing them through any other source except the Journal of the American Chemical Society, for the space of thirty (30) days, be rescinded. This being seconded, was duly carried.

The following gentlemen were then elected to fill the remaining offices of the Society :

*Corresponding Secretary*—P. Casamajor.

*Recording Secretary*—Thomas S. Gladding.

*Treasurer*—T. O'C. Sloane.

*Librarian*—G. A. Prochazka.

*Curators*—William Rupp.

*Committee on Papers and Publications*—E. Waller, C. A. Doremus, L. H. Friedburg.

*Committee on Nominations.*

*Board of Directors.*—It was then moved and seconded that Drs Alsberg and Geyer, be elected Directors, in lieu of C. F. Chandler and James H. Stebbins, Jr.

James H. Stebbins, Jr.,

A. R. Leeds,

C. F. Chandler,

P. Casamajor,

T. S. Gladding,

T. O'C. Sloane,

Geo. A. Prochazka,

E. Waller,

M. Alsberg,

H. Morton,

Wm. E. Geyer,

Wm. M. Habirshaw,

H. Endemann.

Mr. Casamajor then moved that this meeting be declared adjourned till the usual night for conversation. This being seconded was duly carried. After which the meeting adjourned.

James H. Stebbins, Jr.,

Recording Secretary.

At the adjourned meeting, held Dec. 15th, 1882, no quorum being present, official business could not be transacted. A paper on the manufacture of tartaric acid was read by Dr. L. H. Friedburg. Mr. Percy Newman was nominated by E. Waller, Jas. H. Stebbins, Jr., and A. H. Elliott.

## ON THE MANUFACTURE OF TARTARIC ACID.

By I. H. FRIEDBERG, PH. D.

In this country tartaric acid is hardly manufactured for its own sake but its preparation is unavoidably attached to the manufacture of cream of tartar. Here the starting points for tartaric acid are sablons, waste liquids and residues of different kinds, which render an analytical control troublesome, so that partly because of this, partly because of great dilution, the raw material is treated more or less empirically, after the known and often described methods \* with chalk and chloride of calcium or gypsum.

Abroad, the manufacture of tartaric acid is not everywhere a mere appendix to cream of tartar manufacturing, but forms an independent branch of manufacture. In such cases the raw material consists either of argols or of dry sablons or of lees. A careful analytical test has to be made before treating either of these mother substances, and the manufacture has to be carried on with the greatest care in order to avoid loss.

Until very recently these tartaric acid factories worked generally after the old plan as indicated above, viz : treatment with chalk and chloride of calcium or gypsum.

But it has to be recorded, and I will briefly do so in the following pages that a very neat and new, patented process, which according to my own experience is commendable, is now also in use.

This process is based on the decomposition of the mother substances, as named above, by slaked lime instead of chalk.

This preparation has been hidden in the European patents † § under the heading, "Methods of obtaining the potassium in the form of hydrate, while making tartaric acid out of argols."

This heading is practically speaking untrue, because, as we shall see later on, the potassium is not finally gained as hydrate, though this is in the course of treatment formed and then transformed into sulphate or chloride.

The chemical process, which takes place in decomposing the bitartrate of potash in any mother substance into tartrate of lime,

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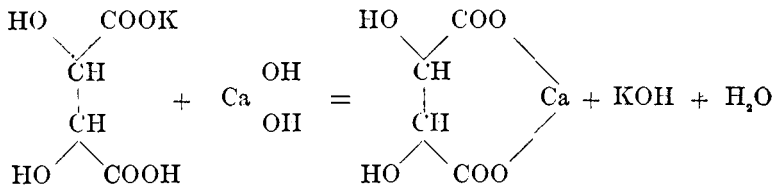
\* Bericht ueber die Entwicklung der chem. Industrie; A. W. Hofmann. Vol. II, page 418, etc.

† Journal of the Society of Arts; Robert Warington, Vol. xxiv, No. 1217, page 366, etc.

‡ Die chemische Industrie; Dr. Emil Jacobsen; 1879, pages 86 and 238.

§ Berichte d. deutschen chemischen Gesellschaft; 1879, page 1366.

by means of slaked lime, is very simply conveyed through the following equation :



The practical difficulty which stood for so long a time in the way of realizing this decomposition for manufacturing purposes, was the difficulty of making the products of decomposition easily filterable. This the patentees have really overcome, and the method of working is smooth and goes like clock work. Slaked lime, freed from coarse pieces is taken in necessary quantities and a milk, not too thin prepared therefrom. This is heated to boiling, and argols, etc., in necessary quantity, are very gradually and in a state of finest powder, introduced into the boiling mass. The charging finished, boiling has to continue for two hours, the condensing steam being enough to keep the mixture in a concentrated form. Hydrate of potash and neutral tartrate of lime are formed in this way. The nitrogenous organic impurities of the raw materials are by the combined action on them, of lime and hydrate of potash, decomposed so as to form ammonia gas which is volatilized with the steam.

Boiling done, which takes place in an iron tank, the mixture is diluted by enough cold water and then the potash is neutralized by either muriatic or sulphuric acid. The process is finished with the help of litmus paper. Here the ammoniacal exhalations are to be considered, so as not to disturb the reaction.

The decomposition as described above takes place under constant stirring by means of an iron stirrer run by machinery.

After the formation of either chloride or sulphate of potash, the mass is still more diluted with cold water and stands best over night, stirring going on continuously, filtration then taking place the next morning. Here filter presses are used to great advantage. It is advisable not to use too high pressure, so as to get a soft cake, which can more easily be washed out, in order to get rid of the mineral potash salts. These latter are either boiled down, as long as the strength of the solution makes it pay, or they might\* be treated with chloride of lime [bleaching powder] and thus trans-

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\*In case chloride of potassium was formed.

formed into chlorate of potash, which I advise manufacturers to try.

The cakes of brown tartrate of lime, which begin to exhale putrid odors by standing too long in a warm place, (in summer time six hours standing often will show this result) have speedily to be decomposed by sulphuric acid. This decomposition takes place in a wooden, lead lined tank, with heavy wooden stirrer moved by steam. The decomposition takes place in the cold and its completion is determined easily by methyl violet test paper.\* No analysis has to be made here, if good paper is at hand, which allows one to guide the reaction so as to get the necessary or the excess of sulphuric acid wished for.

The brown solution of tartaric acid is filtered through filter-presses into wooden receivers.

It is not advisable to evaporate this acid down to the point of crystallization, because it contains impurities enough to spoil the mother liquors at a too early stage. If the course of manufacturing demands a readier transformation of raw material into money, this crude acid solution might be concentrated in the leaden pans to the right concentration for crystallizing or for precipitation by the stirring process, which we shall deal with on another page.

It is preferable to reprecipitate this acid as tartrate of lime, finishing the reaction with chalk and using litmus test paper.

The tartrate of lime thus obtained is filtered on a vacuum filter or by centrifugal power. Of course washing takes place, though slightly. This tartrate of lime is crystalline, light greenish-yellow, keeps perfectly well for any length of time required without decomposing.

It is decomposed in an apparatus similar to the one used for decomposition of the first brown tartrate of lime, by sulphuric acid, in the cold and the reaction finished with the aid of methyl violet test paper. The filtration of the very white gypsum thus obtained cannot be done through filter presses but has to take place on a vacuum filter, very thorough washing being required.

The tartaric acid solution thus obtained ought to stand between 12° and 14° B $\acute{c}$ . It is ejected into the lead pans, evaporated at 80° C to the necessary density, by which dissolved gypsum is precipitated, run into the crystallizing boxes and let stand for crystallization. The crop of brown crystals is redissolved to a liquid of the

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\*Journal of the American Chemical Society; T. O'Connor Sloane, Vol. IV, Nos. 1-4, page 31, etc.

density 25° Bé and treated with bone black, which has been purified by muriatic acid, (so as not to leave a trace of phosphates,) at a medium temperature and under stirring.

The discolored liquid is run through a filter-press and thence into special lead pans. It is evaporated down to about 39° to 40° Bé and run into lead boxes for crystallization.

The crystallization being a comparatively slow process, this liquid may be run into a proper tank with stirrer, stirred for several hours, thus yielding a crop of small crystals right away.

Either crystals are washed and dried in centrifugals, by using steam for washing.

The liquid running off from the first crystallization yields after evaporation another crop of white crystals. Then it becomes a brown mother liquor.

The mother liquors of the brown crystals can, under careful attention, be carried along through the sixth or seventh crystallization. Then the predomination of sulphuric acid and impurities does not allow further crystallization.

The mother liquors at that stage are diluted to a proper density, the greater part of the sulphuric acid removed by addition of slacked lime milk and the filtered liquid has then principally to be freed from iron salts and from phosphate of alumina.

The iron is easily expelled by taking care to keep it in the form corresponding to the protoxide, the presence of the phosphate of alumina makes it necessary to treat the liquids boiling with milk of lime, thus precipitating phosphate of alumina and forming an acid tartrate of lime, which is soluble. It has to be filtered hot and is decomposed by an addition of sulphuric acid, thus yielding very pure solutions of tartaric acid.

If a transformation of the acid thus gained, into bitartrate of potash should be wished for, which hardly would be prudent, the simplest way of arriving at this end would be the following :

The solution is divided into two equal parts, one-half *saturated* by caustic or carbonate of potash, so as to form a neutral tartrate of potash and then the other half added for the precipitation of the bitartrate.

New York, 15th December, 1882.

ON THE ACTION OF PHTHALIC ANHYDRIDE UPON  
GALLIC ACID.

BY GEORGE A. PROCHAZKA.

Mr. Stebbins\* appears to have entirely misunderstood the gist of my remarks at the November meeting.

More than a year ago I experimented upon the action of phthalic anhydride upon gallic acid, with results similar to those of Mr. Stebbins. My object was to find a more economical method for the production on a large scale of gallein (and coerulein) than given in the books. The substitution of gallic acid in place of pyrogallol readily suggested itself. Subsequently I found in a synopsis by Ch. Lauth of the report on dye-stuffs, of the jury of the Paris International Exhibition of 1878, (*Monit. Teint.* 1878) that gallein was prepared by heating together phthalic anhydride and pyrogallic or *gallic acid*. My own results had been anticipated. My own results were never published. In making my remarks at the November meeting it was not my object to substitute a doubtful claim of originality, substantiated by experiments hid under the bushel, in place of Mr. Stebbins, but to call attention to the fact that he had been anticipated as early at least as 1878 by those European manufacturers, who had utilized the reaction in question. Mr. Stebbins does not appear to have been aware of this fact, which through a small notice had become known only to the initiated few. The very careful and detailed experiments (much more exhaustive than my own) of Mr. Stebbins, merit the wisest attention, and the public owes to him a debt of gratitude for their publication.

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\**Journal Am. Chem. Soc.* IV., 248.

# INDEX OF AUTHORS.

## A

- Aillaud, M., Water from Panama, 242  
Andre, G., Magnesium oxychlorides, 230  
Andre, G., Oxychloride of zinc, 239  
Armstrong, H. E., laws of substitution etc., 206  
Atkinson, R., & H. Yoshida. Menthol etc., 144

## B

- Baeyer, A. Combination in the indigo group, 175  
—Syntheses by phenylacetylene, 176, 206  
Barr, K. L., Precipitation of alums by sodic carbonate, 258  
Bartoli, B., Synthesis of organic compounds, 239  
Baubigny, H., Action of sulphides on nickel salts, 239, 240  
Bechamip, A.,  $H_2O_2$  on the coloring matter of blood, etc., 240  
Becker, P., Monophenyl-boro-chloride etc., 206  
Bchr, A., Crystallized anhydrous grape sugar, 11  
Benthsen, A., Normal dithiurethanes, 268  
Bert, P., Action of hydrogen peroxide, etc., 239  
Bertelot, M., Absorption of gases by platinum, 239  
—Decomposition of mercury salts, 233, 234  
—Decompositions of silver salts etc., 235  
—Double salts by fusion, 239  
—Double salts of mercury, 208  
—Electrolysis of  $H_2O_2$ , 240  
—Double salts of Mercury, 231  
—Sulphuretted ureas from carbon oxysulphide, 236  
— Union of free hydrogen with ethylene, 235  
Bevan, E. J., & C. F. Cross, Apparatus for determining melting points, 169  
— Bast fibres, 169

- Birnbaum, K., Iodine on silver salts of aromatic series, 264  
Bloxam, W. P., Crystallization from solutions of salts etc., 261  
Boehringcr, C., Cinchotin, etc., 267  
Boisbandran, L. de., Crystallized gallium oxy-chloride, 233  
—Separation of gallium, 237, 238, 239, 240, 242, 273  
Bollert, A., Anthranin, 207  
Bourgoiu, E., Potassium cyanide on the trichloracetate, 231  
Bourgongnon, A., Analyses of calculi, etc., 215  
Brauer, B., Chemistry of cerium metals, 176  
—Chemistry of rare earth metals, 169  
—On Didymium, 240  
Brenneman, A. A., Review of industrial chemistry, 72  
Bruehl, J. W., Physical constants, etc., 264  
Brunner, H., Preparation of coloring matter, etc., 205

## C

- Cailletet, L., Liquefied ethylene for low temperatures, 238  
Cahours, A., Formation of fatty acids etc., 233  
Calni, A., Amidated naphthalines from naphthols, 290  
Carnelly, T., Action of heat on mercuric chloride, 225  
Casanafor, P., Note on asbestos filters, 248  
—Volumetric estimation of copper and lead, 35  
Cazeneuve, C., Dichloride of camphor, 233  
Chancel, G., Nitrogenized acids from acetones, 208  
Chapotreant, P., On the gastric juice, 242  
Chapuis, G., Liquefaction of ozone, 233  
—Pernitric acid, etc., 237  
Chastaing, M., Morphine, 76  
—Pilocarpin, 78



- Action of acids on pilocarpin, 236
- Chantard, P., Oxidation of pyrogallol, etc., 237
- Claisin, L., Action of haloid acids on HCy, 221
- Claus, A., Bases from addition of chinolin aldehydes, etc., 265
- Dichloronaphthalin etc., 229
- $\text{PCl}_3$  on Benzoyldiphenylamine, 264
- $\text{PCl}_3$  on Naphthol sulphonic acid, 228
- Synthesis of Homologues of anilin, 228
- Clement, P. de., Oxidation of Pyrogallol, etc., 237
- Cleve, T., Preliminary note on didymium, 240
- Cleves, P. T., Remarks on didymium, 240
- Colson, A., On Silicium, 240
- New compounds of C and Si, 237
- Action of  $\text{CS}_2$  on Si, 240
- Combes, M., On the compound  $\text{NH}_2$ , 240
- Conrad, M., Chloromalonic acid, etc., 270
- Cowper, R., Analysis of oxidized iron from condenser, 219
- Solubility of glass, etc., 219
- Crafts, J. M. & C. Friedel, Decompositions produced by aluminium chloride, 170
- Cross, C. F. & E. J. Bevan, Bast Fibres, 169
- Apparatus for determining melting points, 169
- Cross, C. F. & A. Higgin, Reaction of chromic anhydride with sulphuric acid, 170

## D

- Damm, G., Resorcin dyes, 268
- Danton, N. H., Estimation of Tannic acid in tanning materials, 4
- Comparative value of methods for determining tannic acid, 49, 62
- Convenient form of weighing flask, 6
- David, J., Determination of glycerin, 239
- Debray, H., Explosible alloys of zinc, etc., 240
- Some reactions of  $\text{Hg Cl}_2$ , 238
- Delue, M., Dichloronaphthalin, etc., 229
- Demarcay, E., Formation of fatty acids, etc., 233
- Vaporizing metals in vacuo, 242

- Deml, W., Bodies combustible with difficulty, 269
- Devile, St. C., Explosible alloys of zinc, etc., 240
- Dietzell, B. E., Evolution of free nitrogen in fermentation, 267
- Ditte, A., Acid solutions on iron protoxide, 234
- Action of alkaline solutions on  $\text{Sn O}_2$ , 235
- Decomposition of lead salts, 237
- Potassa on lead oxide, 237
- Doelmer, O., Combinations of benzotri chloride, 207
- Dogiel, J., Effects of arsenic, 268
- Donah, G. & E. J. Mills, Action of oxides on salts, 142
- Dorems, C. A., Composition of elephants' milk, 157
- Duchaux, E., Gastric digestion, 234
- Dumreicher, O. V., Action of aluminium chloride on monobrombenzol, 174
- Duvillier, E., Combinations in creatin groups, 273

## E

- Elliot, A. H., Nitro-saccharose, 147
- " (in correction), 186
- Elliot, A. H. & F. Sands, Notes on bone oil, 153
- Erlenmeyer, E., Halogenized organic acids, etc., 175
- Etard, M., Isomery of cuprous sulphites, 241

## F

- Fenton, H. J. H., Transformation of urea, 221
- Filhol, E., Neutral arsenates, 272
- Fiseke, H., Action of amines on chinones, 226
- Fischer, E., On caffen, 174
- Transformation of xanthin, etc., 264
- Fischer, O., Nicotiic acid from pyridin, 176
- Flawitzky, F., Rotation in the plane of polarization, etc., 173
- Molecular power of refraction of terpenes, 173
- Flight, W., Composition of alloys, 171
- Action of Sodium hydrate, etc., on feldspars, etc., 199
- Flosny, M., Double salts by fusion, 239
- Foerster, K., Jorissen's reaction for fusel oil, 207

- Foerster, K., Furfural in fermented liquids, 229  
 Foerster, P., Identity of coloring matter with quercitrin, etc., 206  
 Forcaud, M. De., Hydrate of hydrogen sulphide, 242  
 Forst, C., Cinchotin, etc., 267  
 Friedburg, L. H., Bisulphide of carbon, 252  
 — Manufacture of Tartaric acid, 292  
 Friedel, C. & J. M. Crafts, Decompositions produced by aluminium chloride, 170  
 Friedlander, P., Carbostyryl, 230  
 Friese, G., Normal dithiurethanes, 268

## G

- Gantter, Fr., Bromo substitution products of sebacylic acid, 204  
 Gantier, A., Insoluble modification of pepsin, 237  
 Gerber, M., Rosnilin, 237  
 Gimre, A., Det. of astringent matters in wine, 242  
 Gladding, T. S., Determination of reverted phosphates, 113  
 — Estimation of phosphoric acid, as magnesium pyrophosphate, 135  
 Gladding & Stillwell, Acetate of lime, 94  
 Gladstone, J. H. and A. Tribe, aluminium alcohols, 142  
 Gleeckmann, L., Dimethyl-phenylphosphine, reaction of, 206  
 Goldschmidt, H., Phosphorus pentasulphide, 227  
 Goldschmidt, H. & V. Meyer, Density of gases, 176  
 Gorreu, A., Basic salts of manganese, 242  
 Griess, P., Cyanogen on picrammic acid, 264  
 Grimann, E., Bromine on quibolein and pyridin, 242  
 Guinochet, Aconitates, 231  
 Guthzeit, M., Chloromalonic acid, etc., 270

## H

- Haas, B., Determination of sulphurous acid in wine, 205  
 Haller, A., Camphol-urethane, 235  
 Haller, M. A., Carbonic ether of borneol, 76  
 — Essential oil of savory, 77  
 Hallock, A. P., Direct estimation of  $H_2S$  in gas, 177  
 Hartley, W. N., Chemistry of cerium compounds, 203

- Hartley, W. N., Photographs of spectra, etc., 169  
 — Spectra of carbon compounds, 144  
 — Rabdophane, a new mineral, 203  
 Hautef-nille, P., Liquefaction of ozone, 238  
 — Pernitric acid, etc., 237  
 Heckel, E., Kola nut, etc., 234  
 Hell, C., Bromosubstitution products of sebacylic acid, 204  
 — Combination of C with S and Br., 226  
 Henninger, A., Glycol in wine, 242  
 Henmann, K., Chlorosulphurous acid with metalloids, etc., 261  
 Higgin, A., Dibenzoylaniline, etc., 171  
 Higgin, A. & C. F. Cross, Reaction of chromic anhydride with sulphuric acid, 170  
 Hjelt, E., Bromine on allylmalonic acid, 270  
 — Caprolactone, dilactones, lactones, 270  
 — Oxypropylmalonic acid, 270  
 — Water on isobromcapronic acid, 270  
 Hodgkin J. & D. Howard, Alkaloid from cinchona, 168  
 Howard, D. & J. Hodgkin, Alkaloid from cinchona, 168  
 Hummel, J. J., New compounds of Brazilin and Haematein, 260

## J

- Jacobsen, O., Bromchloral, chlorobromal, etc., 269  
 Jacquelin, M., Pure carbon for electric light, 235  
 Japp, F. A., Action of aldehydes on phenanthra quinone, etc., 198  
 — Constitution of amarine and lophine, 225  
 Japp, F. R., Action of acetone on phenanthra-quinone, 221  
 — Determining constitution of quinones, 199  
 Jean, F., Clarification of champagne wines, 234  
 — Det. of tannic and œnogallic acid in wine, 233  
 Joannis, M., Heat from ferricyanhydric acid, 231  
 — Heat of formation of  $H_2C_2S$ , etc., 234  
 Joly, A., Saturation of phosphoric acid, 232  
 Jorissen, W. A., Furfural in fermented liquids, 268

## K

- Kelbe, W., Capronic acid in resin oil, 228  
 — Expulsion of the sulpho group, 175  
 Kiliani, H., On gum arabic, 174  
 Knecht, E., New isomer of orcin, 227  
 Kraut, K., On tropin, 264  
 Kocchlin, P., Chlorosulphurous acid with metalloids, etc., 261  
 Konig, F., Succinic acid by fermentation, 205

## L

- La Coste, W., Halogen derivatives of chinolin, 268  
 — Reactions of addition products of chinolin, 206  
 Ladenburg, A., History of atropin, 176  
 Ladureau, A., Phosphoric acid in soils of France, 77  
 Landsherg, L., Syntheses by phenyl-acetyene, 176, 206  
 Lapraik, W., Spectroscopic study of chlorophyll, 257  
 Lau, M., Effects of compression on the hardness of steel, 235  
 Lanbenheimer, A., Orthodinitro compounds, 269  
 Laur, P., Reduction of silver ores, 241  
 Lebon, G., Two new antiseptics, 242  
 Leeds, A. R., Acrolein urea, 59  
 — Contamination of the New York water supply, 127  
 — Diphenylamine-acrolein, 32  
 — Reduction of metallic oxides in sunlight, 3  
 Lenckart, R., action of mono-brom-cinnamic acids with conc. sulphuric acid, 173  
 Lewes, V., Experiments on potassium tetra and pentathionate, 224  
 Liebermann, C., Anthranin, 207  
 — Azoanthral dyes, 266  
 Liebermann, L., Determining melting points of metals, etc., 263  
 — Sulphurous acid in wine, 263  
 — Distillation of tartaric acid, 263  
 Liebmann, A., Synthesis of homologous phenols, 205  
 Link, G., Analyses of nephrites from lake dwellings, 206  
 Lloyd, F. J., E-rimination of retrograde phosphates, 224  
 Losanitsch, S. M., CS<sub>2</sub> on P-nitranilin, 264  
 — Nitric acid on tribromanilin, 264  
 Lunge, G., Nitrogen tetroxide with sulphuric acid, etc., 265  
 — Nitrous anhydride as vapor, 266

## M

- Mailfert, M., Researches on ozone, 235, 237  
 Mammene, E. G., Synthesis of quinine, 236  
 Mallet, J. W., Fractional dehydration of ammonium alum and the momicity of aluminium, 180  
 — Properties of pure metallic aluminium, 145  
 Maquenne, M., Action of ozone on manganese salts, 234  
 Meldola, R., Aromatic derivatives of methane, 262  
 Mentschukin, N., Etherification of the oxacids, 205  
 Merling, G., Tropin, 226  
 Meyer, R., Constitution of cumic acid, 266  
 —, Tetrabrombenzol, 175  
 Meyer, P. T., Mustard oil glycolide, 267  
 Meyer, V., Lecture experiments, 237  
 Meyer, V. & H. Goldschmidt, Density of gases, 176  
 Michaelis, A., Mono-phenyl-boro-chloride, etc., 206  
 — Poly-methyl-keton, 206  
 Mills, E. J. & G. Donald, Action of oxides on salts, 142  
 Mills, E. J., & J. Pettigrew, Steeping of barley, 143  
 Morin, H., Essential oil of Licari Kanali, 233  
 Morley, H. F., Oxypropyltoluidine, 206  
 Morris, G. H., Some constituents of resin spirit, 200  
 Mueller, E., Constitution of cumic acid, 266  
 Mnir, M. M. P., Action of water on Bi I<sub>3</sub>, 142  
 — Volumetric estimation of bismuth, 141  
 Mulder, E., Normal cyanic acid, etc., 176  
 — Thermometric knowledge of ozone, 266  
 Muller, C. L., Halogenized organic acids, etc., 175  
 Muntz, A., Galactin, 231

## N

- Neumeister, R., Bromchloral, chloro-bromal, etc., 269  
 —Bromdichloroacetic acid, etc., 269  
 Nichol, W. W. I., Action of heat on thioformanilide, 206

## O

- Oehler, H.,  $\text{PCl}_5$  on naphtholsulphonic acid, 228  
 Ogier, J., New oxychloride of sulphur, 231  
 —Sp. gr. of pyrosulfuryl chloride vapor, 78  
 Osmond, F., Vanadium from basic slags, 241  
 Osterbauer, H., Carbostyryl, 230  
 O'Sullivan, C., Gamma and beta-amylan, 143  
 Otto, R., Synthesis of alkyl-disulph oxides, 176

## P

- Palewski, B., Critical temperatures, 264  
 Papisoglu, G., Synthesis of organic compounds, 239  
 Parmentier, F., Silico-molybdic acid 77  
 Pellet, H., Antiseptic properties of salicylic acid, 238  
 Perkin, A. S., New compounds of Hæmatein and Brazilcin, 260  
 Perkin, W. H. Action of acetyl chloride on fumaric acid 221  
 —Luminous incomplete combustion of ether, etc., 259  
 —Rotary polarization of chemical subst. 257  
 Pettigrew, J. & E. J. Mills., Steeping of barley, 143  
 Phillip, J., On tungsten bronzes, 266  
 Pictet, A., Quinolcin and litidin, 272  
 Pinner, A., Condensation of acetone, 268  
 Plagemann, A., Amines on dichlor-naphthachinone, 265  
 Ponomareff, J., Cyanic and cyanuric ethers, 267  
 Prochazka, G. A., Action of phthalic anhydride on gallic acid, 296

## R

- Radcliffe, T., Rock salt from Saltville, Va., 255  
 —Deposit of zinc oxide in a blast furnace, 256  
 Regnard, P., Action of hydrogen peroxide, etc., 239

- Reinherz, H., Iodine on silver salts of aromatic series, 264  
 Reinke, J., Reducing properties of living cells, 176  
 Renard, A., Products of distillation of colophony, 233  
 Rennie, E. H., Action of ethyl chloro-carbonate on benzene, 143  
 —Benzyl-phenol, etc., 143, 204  
 Reynolds, J. E., Apparatus for liquefying ammonia, 220  
 Ricciardi, L., Composition of ashes from Vesuvius, 237  
 —Composition of the banana, 272  
 Robinet, E., Antiseptic properties of salicylic acid, 238  
 Robinson, H. H., Constitution of amarine and lophine, 225  
 Roemer, H., Anthra cyclamin, 230  
 Roscoe, H., Atomic weight of carbon, 237  
 —Earth-metals in Samarskite, 222  
 —Spectrum of terbium, 223  
 Rosenfeld, M., Lecture experiments, 205  
 Rosenstiehl, A., Rosanilin, 237  
 Roser, W., Terebenthic acid, 227  
 Rousseau, G., Chloroform on beta-naphthol, 241  
 —Diatomic alcohol from beta-naphthol, 77  
 Russell, W. J., Spectroscopic study of chlorophyll, 257

## S

- Sacc, F., Cucurbitacæ of Uruguay, 236  
 —F., Products from Uruguay, 237  
 Sakurai, J., Metallic compounds containing bivalent hydro carbon radicals, 258  
 Sands, F. and A. H. Elliot, Notes on bone oil, 153  
 Sarauw, A., Reaction of phosgen on diaze, etc., 175  
 Seubert, K., Analyses of Nephrites from lake dwellings, 206  
 Schuster, A., Spectrum of terbium, 223  
 Schmitze, K. E., Phoron from glycerin, 176  
 Schutzenberger, P., On silicium, 240  
 Schreiner, L., Resorcin dyes, 258  
 Schotten, C., Piperidin, 262  
 Schwanert, H., Large crystals in urine, 174  
 Sloane, T. O'C., Fat extracting apparatus, 250  
 —Methyl-violet test paper, 31

- Spring, W., Chlorine on sulphonic compounds, etc., 263  
 —Formation of alloys by pressure, 269
- Smith, W., and T. Takamatke, Pentathionic acid, 199  
 —B. E., Preparation of diethylnaphthylamine, 200  
 —Sulphuric acid on diethyl naphthylamine, 201  
 —Phosgen gas on diethyl naphthylamine, 201
- Stebbins, Jr., J. H., Action of phthalic anhydride on gallic acid, 244  
 —Artificial indigo, 81  
 —Laboratory notes, 214
- Stillwell & Gladding, Acetate of lime, 94
- Stone, G. C., Determination of Zinc as pyrophosphate, 26
- Streutfield, F. W., Action of acetone on phenanthraquinone, 221  
 —Action on phenanthraquinone, 198  
 —Determining constitution of quinones, 199
- T**
- Takamatke, T. and W. Smith, Pentathionic acid, 199
- Themari, P., Black phosphorus, 273
- Thomsen, J., Constitution of Benzol, etc., 229  
 —Optical rotation of malic acid, etc., 263  
 —Refraction and heat of combustion, 176
- Thomson, J. M., Crystallization from solutions of salts, etc., 261
- Thorpe, T. E., Action of oxychlorides of sulphur on silver nitrate, 223  
 —Action of thiophosphoryl chloride on silver nitrate, 224  
 —Reducing agents with ferric salts, 223
- Thresh, J. C., Examination of Buxton thermal water, 170
- Tribe, A., and J. H. Ghdstone, Aluminium alcohols, 142
- Traube, M., Rendering oxygen active, 207
- Troost, L., Iodides of phosphorus, 272  
 —New ammonium nitrates and acetates, 234
- U**
- Urech, F., Combination of C. with S. and Br. 226

**V**

- Van der Muijen, H. G. L., Thermometric knowledge of ozone, 266
- Venable, F. P., Alcoholic potash on heptylene bromide, 254  
 —Heptylene from heptane of P. Sabiniana, 22
- Veley, V. H., Higher oxides of manganese, etc., 168

**W**

- Wait, C. E., An earthy ferric sulphate, 61
- Wallach, O., Azo dyestuffs, 173
- Wallach, O., Basic compounds from acido-amides, 206
- Waller, E., Determination of phosphorus in iron ores, 88  
 —Laboratory notes, 212  
 —Review of analytical chemistry (proximate), 180  
 —Review of analytical chem. (mineral), 160  
 —Water supply of the city of New York, 15
- Waltz, G., Propyl and isopropyl-succinic acid, 270
- Warrington, R., Determination of nitric acid, 258
- Warth, C., Caproic acid in resin oil, 228
- Wethered, E., Composition of Pennant grits etc., 144
- Widmann, O., Synthesis of thymol, 205
- Wietzky, R., Naphthyl-sulphuric acid, 227
- Will, W., Bodies produced from sulpho-carbanilid, 230
- Wills, E. J., Precipitation of alums by sodic carbonate, 258
- Wiussinger, C., Chlorine on sulphonic compounds etc., 263
- Witz, G., Vanadium from basic slags, 261
- Wroblewski, M., Composition of hydrated carbonic acid, 236
- Wurtz, A., Ethylenic chlorhydrin on quinolein etc., 272

**Y**

- Yoshida, H. & R. Atkinson, Menthol etc., 166

**Z**

- Zenger, V., Physical constants, etc., 264
- Zimmermann, J., Chloracetic acid ether on phenylendianin, 267
- Ziucke, T., Amines on chinones, 265

# INDEX OF SUBJECTS.

## A

Acetate of lime, its manufacture and analysis, Stillwell & Gladding, 94  
Acetic acid, preparation of, (pat.), 41  
Acetone, condensation of, 268  
Acetone on phenanthraquinone, 221  
Acetones, nitrogenized acids from, 208  
Acetyl chloride, action on fumaric acid, 221  
Aconitates, 231  
Acrolein-urea, A. R. Leeds, 59  
Alcohols aluminium, 142  
Alcohol diatomic, from beta-naphthol, 77  
Alcohol from potatoes, (pat), 41  
Alcohol, manufacture of, (pat), 40  
Alcohol rectifying, (pat), 47  
Alcohol removing bad smell and taste from, (pat), 43  
Aldehydes, action on phenanthraquinone, 198  
Aldehydes, bases from addition of, etc., 265  
Aldehyde and ammonia reaction for determining the constitution of quinones, 199  
Allzarine, preparation of artificial, (pat), 40  
Alkalies, pure caustic by electrolysis, (pat), 110  
Alkylbromides, in synthesis of anilin homologues, 228  
Alkyldisulphoxides, synthesis of, 176  
Alloys ancient, composition of, 171  
Alloys and metals, determining melting points of, 263  
Alloys formed by pressure, 269  
Allylmalonic acid, bromine on, 271  
Alum, ammonium, fractional dehydration of, etc., J. W. Mallet, 180  
Alums, precipitation by sodic carbonate, 258  
Aluminium alcohols, 142  
Aluminium, atomicity of, etc., J. W. Mallet, 180  
Aluminium chloride action of, 174  
Aluminium chloride, decomposition by, 170  
Aluminium pure metallic, J. W. Mallet, 145

Aluminium sulphate, (pat), 46, 111  
Aluminium sulphate, separation of iron from, (pat), 40  
Amarine and lophine, constitution of 225  
Amides acido, Basic compounds from 206  
Amines on chinones, 226, 265  
Ammonia, apparatus for liquefying, 220  
Ammonia from elution lyes, (pat), 46  
Ammonia from molasses swill (pat), 112  
Ammonia from purifiers of gasworks, (pat), 47  
Ammonia, new combinations with nitric and acetic acids, 234  
Ammoniacal waste waters purification, (pat), 42  
Ammonium alum, Fractional dehydration, etc. J. W. Mallet, 180  
Ammonium sulphate from peat, (pat), 41  
Amylan, Gamma & Beta, 143  
Analytical chemistry, review of, 160  
Analytical chemistry, (proximate) review, 189  
Anilin, synthesis of homologues of etc., 228  
Anthracylamin, 230  
Anthramin, 207  
Antiseptics, two new, 242  
Aromatic series, iodine on silver salts 264  
Arsenates, neutral, 272  
Arsenic, effects of, 268  
Asbestos filters, by P. Casamajor, 248  
Ashes from Vesuvius, composition, 237  
Astringent matters in wine det. of 242  
Atropin, history of, 176  
Azo anthrol dyes, 266  
Azo dyestuffs, 173

## B.

Banana, ripe and unripe, composition of, 273  
Barium sulphate. Laboratory note, E. Waller, 212

Barley, steeping of, 143  
 Battery Galvanic, (pat), 41  
 Benzene, action of ethyl chlorocarbonate on, 143  
 Benzoic acid, etc., preparation of, (pat), 40  
 Benzol, constitution of, 229  
 Benzol tetra-brom, 175  
 Benzo trichloride combined with aromatic bases, 207  
 Benzoyl diphenylamine, etc.,  $\text{PCl}_5$  on, 264  
 Benzyl-phenol and its derivatives, 143, 204  
 Bismuth iodide, action of water on, 142  
 Bismuth volumetric estimation of, 141  
 Bisulphide of carbon, L. H. Friedburg, 252  
 Blue coloring matters, preparation of, (pat), 40, 42  
 Bodies combustible with difficulty, 269  
 Boiler incrustation preventing, (pat), 39  
 Bone oil, notes on, A. H. Elliott and F. Sauds, 153  
 Borneol carbonic ether of, 76  
 Brazilein and Hæmatein new compounds of, 260  
 Bromchloral, bromochloroform, etc., 269  
 Bromdichloroacetic acid, etc., 269  
 Bromide, heptylene, alcoholic potash on, F. P. Venable, 254  
 Bromine, expulsion of sulpho group by, 175  
 Bromine on quinolein and pyridin, 242  
 Bromine and S. combination with C., 226  
 Bronze phosphor-lead, (pat), 44

### C

Caffein, 174  
 Calculi, analyses of, A. Bourgoignon, 215  
 Camphol urethane, 235  
 Camphor Dichloride, 233  
 Camphor peppermint, and its derivative, 144  
 Catrolactone, 270  
 Capronic acid in resin oil, 228  
 Carbanilid Sulpho derivatives, 230  
 Carbon atomic weight of, 237  
 Carbon compounds, spectra of, 144  
 Carbon, combination with S. and Br., 226  
 Carbon and Silicium, new compounds of, 237

Carbon Bisulphide, L. H. Friedburg, 252  
 Carbon Disulphide on Silicium, 240  
 Carbon Disulphide on P-nitranilin, 264  
 Carbon oxydichloride, action on diethyl-naphthylamine, 201  
 Carbon oxysulphide, transformation to sulphuretted ureas, 236  
 Carbon, pure for electric light, 235  
 Carbonic acid hydrated, composition of, 236  
 Carbostyryl, 230  
 Cements, etc., method of testing, (Pat.) 42  
 Cerium compounds, 203  
 Cerium metals, chemistry of, 176  
 Chemistry, Industrial, review of, A. A. Breuneman, 72  
 Chinolin, reactions of addition products, etc., 206  
 Chinolin, Halogen derivatives of, 268  
 Chinones, Amines on, 226, 265  
 Chloroacetic acid ether on phenylendiamin, 267  
 Chlorates, (Pat.), 79  
 Chloridibrom acetic acid, etc., 269  
 Chlorine, action on sulphonic compounds, etc., 262  
 Chlor-naphthol, etc., 229  
 Chloro bromal, chloro bromoform, etc., 269  
 Chloroform on beta naphthol, 241  
 Chloromalonic acid and derivatives, 270  
 Chlorophyll, spectroscopic study of, 257  
 Chlorosulphurous acid with metalloids, etc., 261  
 Chromic anhydride reaction with sulphuric acid, 170  
 Chromium, compounds for tanning, (Pat.) 48  
 Cinnamic acids, mono-brom, action, 173  
 Cinchona Bark, new alkaloid from, 168  
 Chichotin, etc., 267  
 Coal, fractional distillation of, (Pat.), 111  
 Caffein, etc., from xanthin, 264  
 Cold, generating, (Pat.), 109  
 Colophony, products of distillation of, 233  
 Coloring matters, (Pat.), 40, 42, 44, 46, 48, 80, 108, 110  
 Coloring matters of the Rosaniline group, (Pat.) 46  
 Coloring matter, preparation by nitro-substances, etc., 205

Combustion, Refraction and heat of, 176  
 Constants, Physical, etc., 264  
 Cooling apparatus, (Pat.), 39  
 Copper and Lead, volumetric estimation of, P. Casanajor, 35  
 Copper refined from mattes (Pat.), 79  
 Correction Nitro saccharose, A. H. Elliott, 186  
 Creatin Groups, combinations in, 273  
 Crystallization from solutions of compound salts, 261  
 Cucurbitaceae of Uruguay, 236  
 Camic acid, constitution of, 266  
 Cuminal, Synthesis of Thymol from, 205  
 Cuprous Sulphites, Isomery of, 241  
 Cyanamide from urea, 221  
 Cyanic acid and derivatives, 176  
 Cyanic and cyanuric ethers, 267  
 Cyanogen on picramnic acid, 264

## D

Depilatory for hides, (Pat.), 109  
 Dinilylacetic acid, HBr. and Br. on, 271  
 Diazotizing for formation of coloring matters, (Pat.), 46  
 Dibenzoylaniline and its isomerides, 171  
 Dichlor-naphtha-chinone, amides on, 265  
 Dichlor-naphthalin, etc., 229  
 Didymium, 240  
 Diethylnaphthylamine preparation, 200  
 Diethylnaphthylamine action of sulphuric acid on, 201.  
 Dilactones, 271  
 Dimethyl-phenyl phosphine on ethylene bromide, 206  
 Diphenylamine-acrolein, A. R. Leeds, 32  
 Disinfecting, etc., by carbonized peat coal, (Pat.), 48  
 Dithiurethanes normal, 268  
 Dyes, Blue and Violet, (Pat.), 110

## E

Earth metals in Samarskite, 222  
 Earth metals, rare, 169  
 Elephants' milk, composition of, C. A. Doremus, 157  
 Emery, wheels, (Pat), 41  
 Ether etc., incomplete combustion of, 259  
 Etherification of the oxacids, 205  
 Ethers, cyanic and cyanuric, 267  
 Ethylen, union of free hydrogen with, 235

Ethylen, liquefied for low temperatures, 238  
 Ethylene bromide, reaction on dimethyl phenyl phosphine, 206  
 Ethylenic chlorhydrin on pyridic bases etc., 272  
 Eupittonic acid, preparation from wood tar, (Pat), 40  
 Experiments, lecture, 205, 227  
 Explosive compounds, (Pat), 41, 48, 80  
 Extraction, apparatus for analytical purposes, (Pat), 47

## F

Fabrics, woven finish for, (Pat), 111  
 Fat extracting apparatus T. O'C. Sloane, 250  
 Feldspars, action of sodium hydrate etc. on, 199  
 Fermentation, evolution of free nitrogen in, 267  
 Ferric salts, behavior of reducing agents with, 223  
 Ferric sulphate, earthy, C. E. Wait, 61  
 Ferricyanhydric acid, heat from, 231, 233  
 Fertilizer, (Pat), 46  
 Fibres, Bast, chemistry of, 169  
 Filtering apparatus, (Pat), 47  
 Filters Asbestos, P. Casanajor, 248  
 Fumaric acid, acetyl chloride on, 221  
 Furfural in fermented liquids, 229, 268  
 Furnace, blast, zinc oxide in, T. Radcliffe, 256  
 Fusel oil, Jorissen's reaction for, 207

## G

Galactin, 231  
 Galem, analysis of, J. H. Stebbins, 214  
 Gallic acid, action of phthalic anhydride on, J. H. Stebbins, 244  
 Gallic Acid, action of phthalic anhydride on, G. A. Prochazka, 296  
 Gallium, oxychloride crystallized, 233  
 Gallium, separation of, 237, 238, 239, 242, 273  
 Gastric digestion, 234  
 Gastric juice, on the, 242  
 Gas, direct estimation of H<sub>2</sub> S in, A. P. Hallock, 177  
 Gas, illuminating and heating, (Pat), 46  
 Gases, determining density of, 176  
 Gases, driving out of liquids etc., (Pat), 45



Gases from coke furnaces, utilizing, (Pat), 109  
 Glass, solubility in certain reagents, 219  
 Glucose, removing gypsum from, (Pat), 105  
 Glycerin, determination of, 239  
 Glycerin, phoron from, 176  
 Glycol in wine, 242  
 Glycolide Mustard oil, 267  
 Goldmark, J., obituary, 7  
 Grits, Pennant, composition of, 144  
 Gum arabic, 174

## H

Hæmatein and brazilein, new compounds of, 260  
 Hématosin etc., 240  
 Heptylene, from heptane of P. Sabanaia, F. P. Venable, 22  
 Heptylene bromide, alcoholic potash on, F. P. Venable, 254  
 Hides, preserving and waterproofing, (Pat), 245  
 Hydrocarbon radicals in metallic compounds, 258  
 Hydrocyanic acid, action of haloid acids on, 221  
 Hydrochinonidin and hydrochinidin, etc., 267  
 Hydrogen, (Pat), 46  
 Hydrogen dioxide, electrolysis of, 240  
 Hydrogen peroxide, on coloring matter of the blood, 240  
 Hydrogen peroxide, action on organic matter, etc., 239  
 Hydrogen sulphide, hydrate, 242  
 Hydrogen sulphide, on nickel chloride 240

## I

Indigo artificial, J. H. Stebbins, 81  
 Indigo group, combination in the, 175  
 Iodine, on silver salts of aromatic series, 264  
 Iodo salicylic acids, etc.,  
 Iron, acid proof coating on, (Pat), 109  
 Iron protoxide, acid solutions on, 234  
 Iron, oxidized, from condenser, 219  
 Iso-brom-capronic acid, water on, 270

## K

Kainit, (Pat), 43, 45  
 Kola nut, etc., 234

## L

Laboratory notes, J. H. Stebbins, 214  
 Laboratory notes, E. Waller, 212.  
 Lactones, etc., 270, 271

Lead acetate, laboratory note, E. Waller, 213  
 Lead and Copper, Volumetric estimation of, P. Casamajor, 35  
 Lead and Silver from Ores, (Pat), 43  
 Lead oxide Potassa on, 237  
 Lead salts, decomposition by alkalies, 237  
 Lead white, manufacture of (Pat), 39  
 Lime acetate, manufacture and analysis, Stillwell & Gladding, 94  
 Liquids, treating with gases, etc., (Pat), 47  
 Lophine and anarin, constitution of 225  
 Lubricants, (Pat), 108  
 Lutidin and quinolein, 272

## M

Magnesia, etc., from Stassfurt salts, (Pat), 78  
 Magnesia, Preparation from dolomite, etc., (Pat), 45  
 Magnesium oxychlorides, 230  
 Magnesium Pyrophosphate, estimation of phosphoric acid as, T. S. Gladding, 135  
 Malic acid, optical rotation of, 263  
 Manganese, higher oxides, etc., of, 168  
 Manganese, basic salts of, 242  
 Manganese salts, Ozone on, 234  
 Maltose, (Pat), 109  
 Melting Points, Apparatus for determining, 169, 263  
 Menthol, and its derivative, 144  
 Methane, Aromatic derivatives of, 202  
 Metallic Compounds containing bivalent radicals, 258  
 Metalloids, Chlorosulphurous acid with, 261  
 Methyl-naphthalene, dyes from, (Pat), 111  
 Methyl Violet test paper, T. O' C. Sloane, 31  
 Metals, Vaporization in vacuo, 242  
 Mercuric chloride, Reaction, 238  
 Mercuric chloride, Action of heat on, 225  
 Mercury salts, decomposition by potassium salts, 233, 234  
 Mercury, double salts, 208, 231  
 Milk, Elephants, composition of, C. A. Doremus, 157  
 Milk, Tester, (Pat), 108  
 Mono-phenyl-boro-chloride and derivatives, 206  
 Morphine. Functions, transformation and solubility, 76

Molasses still, Distillation of, (Pat.), 112  
Mustard oil, Glycolide, 207

## N

Naphthalene, Coloring matters from nitro derivatives of, (Pat.), 46  
Naphthalene series. Substitution in, 206  
Naphthalines, Amidated from A and B naphthol, 270  
Naphthol beta, Chloroform on, 241  
Naphthol beta, Diatomic alcohol from, 77  
Naphthols, Conversion into nonamines, (Pat.), 42  
Naphthol sulphonic acid,  $\text{PCl}_5$  on, 228  
Naphthol sulphonic acid derivatives, 229  
Naphthylsulphuric acid, 227  
Nephrites, Analyses of, 206  
 $\text{NH}_3$ , 240  
Nickel chloride, Hydrogen sulphide on, 240  
Nickel sulphate, Metallic sulphides on, 239  
Nicotinic acid from pyridin, 176  
Nitranilin P.,  $\text{CS}_2$  on, 264  
Nitric acid, Determined as nitric oxide, 258  
Nitro benzaldehyde preparation (Pat.), 109  
Nitro benzylchloride for coloring matters, (Pat.), 48  
Nitrogen free, evolved by fermentation, 267  
Nitrogen from air, (Pat.), 44  
Nitrogen tetroxide with sulphuric acid, etc., 265  
Nitrogenized acids from acetones, 208  
Nitro glycerine, Cotton and dextrine for, (Pat.), 80  
Nitro-saccharose, A. H. Elliott, 147  
Nitro-saccharose, A correction, A. H. Elliott, 186  
Nitro substances, for preparing coloring matter, 205  
Nitrous anhydride as vapor, 266

## O

Oenogallic acid in wine determination, 233  
Oil, Essential, of Licari Kanali, 233  
Oil of savory, Essential, 77  
Orcin, New isomer of, 227  
Organic acids, Halogenized, etc., 175  
Organic Compounds, synthesis by electrolysis, 239

Orthodinitro compounds, 269  
Oxides, Action on salts, 142  
Oxides, Metallic, reduction in sunlight, A. R. Leeds, 3  
Oxygen from air, (Pat.), 47  
Oxygen, Rendering active, 207  
Oxypropylmalonic acid, etc., 270  
Oxypropyltoluidin, 206  
Oxysulphides, organic, etc., chlorine on, 261  
Ozone Generator, (Pat.), 78  
Ozone, Liquefaction of, 238  
Ozone, On manganese salts, 234  
Ozone, Researches on, 235, 237  
Ozone, Thermometric knowledge of, 266

## P

Paraffine from crude paraffine without presses (Pat.), 48  
Paranitro benzaldehyde (Pat.), 48  
Patents, Foreign, 38, 108  
Peat coal carbonized, for disinfecting, etc. (Pat.), 48  
Pentathionic acid, 199  
Peppermint camphor and its derivatives, 144  
Pepsine, insoluble modification of, 237  
Pernitric acid composition and atomic weight, 237  
Phenanthraquinone, action of acetone on, 221  
Phenanthraquinone, action of aldehydes on, 198  
Phenol-acetylene, synthesis by, etc., 176  
Phenol, benzyl and derivatives, 204  
Phenols, homologous, synthesis of, 205  
Phenyl-acetylene, syntheses by means of, 206  
Phenylen diamin, chloracetic acid ether on, 267  
Phoron from glycerin, 176  
Phosgen, reaction on diazo amido compounds, 175  
Phosgen gas, action on diethylnaphthylamine, 201  
Phosphates, native basic in the soda manufacture (Pat.), 110  
Phosphates, retrograde, estimation of, 224  
Phosphates, reverted, determination of, T. S. Gladding, 113  
Phosphoric acid, estimation as magnetic pyrophosphate, T. S. Gladding, 135  
Phosphoric acid in soils of France, 77