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NO REEL NUMBER

S.O. Dev. Co. of N.J.

Absorption Spectra

- (8) Spectrum Projector  
Ludwigshafen

IV/1943

Reference: Emission spectro-analysis (8)

Acetylene, substituted

- (24) Analysis of Mixtures of Higher Molecular Weight  
Acetylenes  
Hils

IV/1943

Higher molecular weight acetylenes are present in the low temperature condensates from electric-arc acetylene which cannot be separated by either vacuum distillation or the partial condensation fractionation technique of Stock. Neither can a complete separation of the  $C_4$  acetylenes be effected by these physical methods. The rotating band column of Coenen (Hils) can separate mono-vinylacetylene and diacetylene, however losses by decomposition occur.

A complete separation has been effected by a combination of the Stock procedure with chemical and physical methods. Total acetylenes are determined with  $AgCN$  and the compounds containing 2 free acetylene-hydrogen combinations are determined by  $AgNO_3$  titration according to Thury-Schüller. Determinations of molecular weight and vapor pressure are used as control.

Benzoic Acid

- (4) Determination in Technical Sodium Benzoate Solutions  
Hils

IV/1943

Benzoic acid is generally determined in samples containing sodium benzoate, acetophenones, and waxy lubricants by steam distillation of the acidified mixture, or extraction of the acidified solution with ether and final titration of the liberated free acid. A new method has been developed whereby the weakly alkaline sodium benzoate solution is passed through an  $HCl$  treated Wofatit K filter. The filter is washed with 50% acetone. The total filtrate and acetone wash contain the free benzoic acid which is determined volumetrically. A correction must be made for the solution of a small amount of acid from the Wofatit gum. Work is being carried out to prevent this solubility.

Bis-Isopropylxanthogen

- (3) Determination of Traces of Mercaptans  
Hils

IV/1943

Small amounts of isopropyl mercaptan are present in diisopropyl xanthogen disulfide. The mercaptan is determined with Folin's reagent (phospho-tungstic acid), (Ber. 70, II, 1186, 1422 (1937)). The determination is carried out in acetone solution by means of a step photometer with a 720 m $\mu$  filter.

#### Brown Coal

#### (18) Correlation Between Ash Content, Composition, and Fusibility of Elise Coal Mersoburg

IV/1943

Previous work (see also IV/1941; III, IV/1942; I, II, III/1943) shows a clear correlation between ash content, ash composition, and fusibility of the Elise coal. Increasing ash content of the crude coal indicates higher silica content and correspondingly poorer content in other mineral constituents. Elise coals with low ash contents (6-10%) have melting points of about 1200°C. due to the formation of high melting point silicates; with higher ash contents (15-30% and higher) consisting preponderantly of silica melting points higher than 1350°C. are obtained.

#### Buna

#### (23) Determination of Saturated Fatty Acids and Nekal in Latex -Ludwigshafen

IV/1943

The latex is flocculated with alcoholic KOH under reflux, whereby the fatty acids (as soaps) and the Nekal, are dissolved. The alcohol is evaporated, the solution acidified with H<sub>2</sub>SO<sub>4</sub>, and the fatty acids as well as the phenol- $\beta$ -naphthylamine are extracted with petroleum ether. The latter is evaporated, the fatty acids saponified and freed from the phenyl- $\beta$ -naphthylamine by shaking with ether. The fatty acids are again liberated with a mineral acid and extracted with ether.

The aqueous acid solution remaining after the petroleum ether extraction is exactly neutralized with NaOH, evaporated to dryness and extracted with alcohol. The extract is practically 100% Nekal and is weighed as such after evaporation and drying at 100°C.

For control purposes a Kjeldahl nitrogen determination can be run and the result calculated as phenyl- $\beta$ -naphthylamine, or the result of sulfur determination (Burgess-Parr bomb) can be calculated to Nekal.

#### Butylene

#### (6) Determination of $\alpha$ - in the Presence of $\beta$ -Butylene Hills

IV/1943

According to Meerwein A-435, 188 (1924) perbenzoic acid reacts much faster and can be used for the quantitative determination of double

bonds linked on both sides with alkyl groups than with the end ethylene linkage; this statement was tested with vinylcyclohexene and found to be true. These reaction velocity differences have been used to analyze mixtures of  $\alpha$ - and  $\beta$ -butylenes and an accuracy of  $\pm 1\%$  obtained.

### $\epsilon$ -Caprolactam

- (1) Heat Evolved by the Cyclohexanoneoxime- $\epsilon$ -Caprolactam Rearrangement  
Merseburg

IV/1943

Reference: Cyclohexanoneoxime (3)

### $\epsilon$ -Caprolactam Polymer

- (1) Detection of Monomers  
Ludwigshafen

IV/1943

Monomers can be detected with phosphotungstic acid which forms a thick white precipitate with aqueous caprolactam solutions, and a white turbidity with even a 0.2% solution. More dilute solutions should be concentrated on a steam bath before testing.

### Cyclohexanoneoxime

- (3) Heat Evolved by the Rearrangement into  $\epsilon$ -Caprolactam  
Merseburg

IV/1943

The method for measuring the heat evolved by the rearrangement of cyclohexanoneoxime into  $\epsilon$ -caprolactam is described. It has been found to be 34100 calories/mole.

### Iron

- (35) Determination of Metallic Iron in the Presence of Ferrous and Ferric Hydroxides  
Bitterfeld

IV/1943

I.G. Method No. 96 (copper sulfate procedure) gives high results since ferrous hydroxide is also dissolved after the electrolytic separation and is also titrated with permanganate. The gas volumetric method gives low results due to consumption of part of the hydrogen for the reduction of ferric to ferrous oxide. I.G. Method 20/18 using KCl-CuCl<sub>2</sub> acidified with citric acid which dissolves only metallic iron and ferrous oxide, leaving the ferric oxide undissolved, is not satisfactory since ferrous oxide is not completely dissolved. The best procedure is the following:

The sample is treated for 2-3 hours with a saturated solution of FeCl<sub>3</sub>-CuCl<sub>2</sub>-KCl in a volumetric flask. The volume is made up to the mark and the filtrate is titrated with N/10 KMnO<sub>4</sub>. This gives the amount of ferrous iron produced by the reaction between FeCl<sub>3</sub> and the

metallic iron in the sample. The insoluble is dissolved in an inert atmosphere in concentrated HCl, and the ferrous iron titrated with N/10 K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (use preventive solution!). The ferric iron is then titrated with N/10 TiCl<sub>3</sub> in concentrated HCl solution.

### Emission Spectral Analyses

#### (7) Spectral Studies Ludwigshafen

IV/1943

To evaluate spectrograms in qualitative spectrochemical analysis, enlarged charts showing the iron spectrum are used on which the most important lines of the various elements are marked by the usual symbols. The degree of enlargement is so chosen that the projected spectrogram obtained with each analysis can exactly cover the iron spectrum in the tables for comparison and the lines of the unknown spectrum easily identified. We have improved these tables as follows:

1. Instead of a relatively high iron spectrum over the sample, there is recorded a narrow Fe-spectrum, above and below the spectrum of the sample; just touching the latter, and tables and spectrum analyses are prepared by using the same slit width. We thus avoid any possibility of error due to misalignment of the tables and curvature of the spectrum lines.

2. The reference lines and symbols are drawn in different colors. We use for:

Noble metals - gold orange  
Metals of Groups I and II - purple  
Metals of Group III - brick red  
Metals of Groups IV and V - green  
Metalloids, rare earths - black  
Typical steel alloying elements - dark blue  
(W, Mo, V, Ti, Ta, Nb)

3. The tables are prepared on Agfa-Correctostat paper and imbedded in a plastic. Such tables neither curl nor become distorted (in contrast to unprotected tables) and, since they do not become soiled have an almost unlimited life.

The above modifications were found to simplify and accelerate the work.

#### (8) Spectroprojector Ludwigshafen

IV/1943

The spectrum measuring projector of R. Fuess has proved its value in the evaluation of emission and absorption spectra. Compared to a spectroprojector of other origins, it has the following advantages:

- (a) The very precise plate movement by means of a measuring spindle allows accurate determination of wavelengths.
- (b) The rapidly effected change in magnification from 20X to 10X makes it possible to scan a wider spectral range, which is of particular advantage in the evaluation of absorption spectra.
- (c) The device, in spite of its simple design, provides a sufficient variation of the magnification scale, so that the magnification can be easily adapted to available spectrum tables.

### Soil Studies

#### (10) Humus Determination Bitterfeld

IV/1943

The Karrer and Boding-Wiegner (Helv. 4 (1921)700 and 6 (1925)817) method has been used by the Merseburg Ammonia Plant and by us with excellent results for the determination of humus in black earth (fertile plain of Magdeburg). It is, however, time consuming.

### Fluorine

#### (28) Determination of Very Small Amounts Bitterfeld

IV/1943

The method of Rowley and Churchill (Anal. Ed. 9 (1937) 551) for the determination of fluorine, using  $\text{Th}(\text{NO}_3)_4$  and alizarine sulfonate as indicator has been tested. The procedure is rapid and accurate. Limit about 100% fluorine. Phosphates are also titrated when present, although satisfactory results were obtained in the presence of 10%  $\text{P}_2\text{O}_5$  calculated on the total fluorine present.

#### (29) Determination in Iron Pyrites Leverkusen, inorg.

IV/1943

The qualitative detection of fluorine is carried out by distilling 5 g. pyrites and 0.5 g. powdered quartz with  $\text{H}_2\text{SO}_4$  according to Willard and Winter. Five hundred ml. of the distillate is made alkaline, evaporated to dryness, and the residue heated with 1 g. quartz powder and 20 ml. concentrated  $\text{H}_2\text{SO}_4$  and tested for fluorine according to Kolthoff with purpurin. The method of Willard and Winter is used for the quantitative determination. Weigh out 5 g. pyrites, 0.5 g. quartz, add 35 ml.  $\text{H}_2\text{SO}_4$  (25 ml. 1.84  $\text{H}_2\text{SO}_4$  + 40 ml.  $\text{H}_2\text{O}$ ) and distill at  $180^\circ\text{C}$ . The distillate, made slightly alkaline, can be reduced in volume without loss of fluorine and the latter can be determined colorimetrically. The Lange instrument is used with the yellow pertitanium solution. Other colored

metallic solutions mentioned in the literature, such as the green Fe-ferrous solution or organic dyes (alloxanthin, pyramidon, resorcin), are more seldom used. The Willard-Winter method avoids the necessity of fusing the sample with  $\text{Na}_2\text{O}_2$ -quartz mixture. Pyrites from Meggen contain 0.011-0.016%. F and the Perunal product less than 0.01% F.

(30) Determination in Wash-Acid  
Leverkusen

IV/1943

The sample is distilled at 135°C. with 0.5 gm. quartz,  $\text{H}_2\text{O}_2$ , and  $\text{H}_2\text{SO}_4$  (25 ml. conc.  $\text{H}_2\text{SO}_4$ :40 ml. water). The fluorine is determined quantitatively in the distillate by precipitation as  $\text{PbCl}_2$  F with  $\text{PbCl}_2$  solution.

Fractionation

(3) Determination of the Plate Efficiency of the Leuna  
Column  
BMIs

IV/1943

At Leuna's suggestion, the efficiency of the Leuna column was tested, using the recommended mixture of 12% isobutane-88% isobutylene, and  $\alpha$ -butylene respectively. The results showed the evident superiority of the Leuna over the Podbielniak column.

Potassium

(19) Determination with Sodium Cobaltic Nitride  
Bitterfeld

IV/1943

The potassium is precipitated with sodium cobaltic nitrite and the yellow <sup>precipitate</sup> weighed according to Schoorl (Rec. Trav. Chim. Pays-Bas 61 (1942) 91-102) or determined by dissolving in a  $\text{H}_2\text{SO}_4$ - $\text{KMnO}_4$  solution and back-titration with  $\text{FeSO}_4$ . The results with both methods are satisfactory.

Found:	Used: 5.25 mg. K		Used: 10.5 mg. K	
	Gravimetric mg.	Volumetric mg.	Gravimetric mg.	Volumetric mg.
	5.29	5.1	9.3	10.5
	5.16		9.8	

Calcined Pyrites

(8) Determination of Cobalt  
Duisburg Copper Works

IV/1943

Reference: Cobalt (7)

Silicic Acid

(15) Determination with Pyramidon  
Bitterfeld

IV/1943

The method of King and Watson (Mikrochemie II (1936)47) for the determination of  $\text{SiO}_2$  is based upon the formation of a silica-molybdic acid complex with pyramidon. Quantitative results (weighed 2.6 mg.  $\text{SiO}_2$ , found 2.63 mg.) were found with a freshly prepared meta-silicic acid solution. With an aged water glass solution, however, only 0.6 mg.  $\text{SiO}_2$  out of 2.5 mg. were recovered. The reason for this unusual behavior (polymerization?) has not as yet been discovered.

### Cobalt

- (7) Determination in Crude and Calcined Ores in the Presence of Much Fe, Cu, Zn, Mn, and Ni  
Duisburg Copper Works

IV/1943

The sample is treated with HCl and HF or  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  until dissolved, the solution evaporated to dryness, the residue taken up with water, filtered, decomposed with citric acid, neutralized with ammonia, acidified with acetic acid, and the Co and Cu precipitated with potassium xanthogenate. Some copper sulfate is added when the original copper content is low. The precipitate is dissolved in  $\text{HNO}_3 + \text{H}_2\text{SO}_4$  and the solution fumed to dryness. The residue is taken up in hot water and the weakly acid solution is titrated potentiometrically according to Dickens and Massen after addition of ammonium citrate and ammonia with  $\text{K}_3\text{Fe}(\text{CN})_6$  using a calomel-platinum electrode by adding an excess of the  $\text{K}_3\text{Fe}(\text{CN})_6$  and titrating back with a  $\text{Co}(\text{NO}_2)_2$  solution.

### Mercaptans

- (2) Determination in Bis-isopropylxanthogen  
Bills

IV/1943

Reference: Bis-isopropylxanthogen (3)

### Mesamoll

- (1) Volatility  
Merseburg

IV/1943

The observation that the volatility of Mesamolls increases with increasing chain length was found to be true. It has been found that this increase in volatility is due to hydrocarbon impurities not removed by the vacuum-steam distillation. These impurities are particularly present in the longest chain fraction (C18). The vapor pressures of the pure Mesamolls decrease with increasing chain lengths.

### Methylphenyl-1-Naphthylamine

- (1) Determination of Phenyl-d-Naphthylamine  
Ludwigshafen

IV/1943

The following control method was tested and found to be satisfactory:

A 5-gm. sample is dissolved in 125 mls. of acetic acid and titrated at room temperature with N/1 nitrite solution until one drop of the solution shows a definite green spot when placed on a white paper. A correction of 1.5 ml. N/1 nitrite solution is made, this amount of solution being required for the methylphenyl- $\alpha$ -naphthylamine to produce the identical green spot. Thus, if for (a) gm. of sample (b) ml of N/1 nitrite solution are required, the phenyl- $\alpha$ -naphthylamine content is given by the expression  $\frac{b-1.5}{a}$ .

### Sodium Lye

#### (10) Determination of Traces of Chlorides

Hills

IV/1943

The nephelometric and potentiometric methods for determining amounts of chlorine under 100 mg. Cl/1 sodium hydroxide solution with silver nitrate were investigated. Both procedures gave satisfactory results, despite high  $\text{NaNO}_3$  contents on known concentrations. A curve has been established for use nephelometrically with the Pulfrich stephphotometer.

The following studies were made to determine which of the two procedures is best for control purposes. The sample was diluted and passed through an acid pretreated Wofatit K filter. This retains the cations, the NaOH leaving water and the NaCl forming HCl. The filtrate is concentrated after neutralization and titrated according to Volhard. This avoids the use of large amounts of  $\text{HNO}_3$  to neutralize the excess alkali.

### Oils

#### (12) Detection of Tricresyl Phosphate in Edible Oils

Ludwigshafen

IV/1943

Reference: Tricresylphosphate (7)

### Olefins

#### (11) Determination with Perbenzoic Acid

Reference: Butylene (6)

#### (12) Survey of the Methods for Determination of Double Bonds

Merseburg

IV/1943

I.G. methods listed under I.G.A.657 have been studied. The relationship of iodine number, the working conditions, and constitution of a few typical olefins and



the reasons for choosing suitable procedures to suit the case discussed. It has been found that the iodine number results of straight chain olefins is independent of variations in conditions, while for branched olefins milder conditions are necessary to avoid substitution. On the contrary, conjugated dienes require more energetic treatment for complete saturation. The methods of Kaufmann, Klein, Winkler, Cortese, McIlhenny, Hamus, Wijs, as well as thiocyanogen number and hydrogenation were considered. It will be necessary to prepare synthetic mixtures of pure hydrocarbons to test these procedures.

### Palladium

#### (2) Determination of Small Amounts of Silica Carriers Ludwigshafen IV/1942

Most of the  $SiO_2$  is removed by HF. The  $H_2S$  group is precipitated, the insoluble dissolved in  $HNO_3$  and the Pd determined by precipitation with dimethylglyoxime.

### Phenyl-1-Naphthylamine

#### (1) Determination in Methylphenyl-1-Naphthylamine Ludwigshafen IV/1943

Reference: Methylphenyl-1-Naphthylamine (1)

### Phosphate

#### (14) Determination in the Presence of Chlorides and Nitrates

Reference: Phosphoric Acid (19)

### Phosphoric Acid

#### (19) Determination in the Presence of Chlorides and Nitrates Ludwigshafen IV/1943

The following control procedure was developed: The Cl ion is precipitated with an excess  $AgNO_3$  in an aliquot of the sample acidified with  $HNO_3$  and the excess  $AgNO_3$  determined with  $NH_4CNS$ , according to Volhard. The alkalinity of another aliquot is reduced to faint pink phenolphthalein color by addition of  $HNO_3$ , sodium acetate and a known excess of  $AgNO_3$  added, the volume made up to the mark, the flask will shaken and the precipitated mixture of  $AgCl$  and  $AgPO_4$  filtered off. The excess  $AgNO_3$  is again determined in the filtrate, after acidification with  $HNO_3$ , according to Volhard. The amount of  $PO_4$  present is calculated from the difference between the two titrations.

Sulfur Pyrites

- (14) Determination of Fluorine  
Leverkusen, Inorg.

IV/1943

Reference: Fluorine (29)

Steel

- (8) Tungsten Determination in N<sub>10</sub> Steel  
Merseburg

IV/1943

The precipitated WO<sub>3</sub> obtained according to I.G.A.572 procedure for stainless steels still contains considerable amounts of occluded iron and molybdenum. The precipitate should be purified by dissolving in NaOH and reprecipitating. Good results are obtained by this purification.

- (9) Nitrogen Determination  
Merseburg

IV/1943

Improvements in the distillation procedure, the use of N/100 instead of N/20 solutions, and reduction in the blanks obtained with the reagents has greatly increased the accuracy of the determination. The nitrogen content of the acid soluble and insoluble portions depends upon the treatment conditions.

Nitrogen

- (25) Determinations in Steel  
Merseburg

IV/1943

Reference: Steel (9)

Tricresyl Phosphate

- (7) Detection in Edible Oils  
Ludwigshafen

IV/1943

The use of technical tricresyl phosphate in foodstuff is forbidden due to the poisonous nature of the ortho compound present, and to prevent such use the tricresyl phosphate is dyed with 0.0012% Zapon True Blue BL before marketing. The compound is used, nevertheless, in the preparation and separation of some edible oils. It was therefore necessary to develop some test for detecting the dye which would be an indication that tricresyl phosphate was used in the manufacture of the product. The test is carried out as follows: A known amount of the edible oils is mixed with twice its volume of ether, the mixture filtered through a 7-cm. filter paper and washed with ether. The dye will remain on the filter as a blue coating, more soluble in water and changing to a brown color on treatment with NaOH.

Tungsten(5) Determination in N/10 Steel  
Wernsburg

IV/1943

Reference: Steel (8)

Aluminum Chloride(4) Determination of Soluble AlCl<sub>3</sub> in Isomerization  
Catalysts  
Oppau

IV/1943

Only part of the AlCl<sub>3</sub> present in AlCl<sub>3</sub> catalysts (supported on activated alumina or clay) is soluble in organic solvents such as acetone or nitrobenzene. The soluble part has the constitution AlCl<sub>3</sub>, the insoluble is probably an oxychloride, AlCl<sub>2</sub>OH, or AlOCl. The separation is carried out by treating with acetone or nitrobenzene until all AlCl<sub>3</sub> has been dissolved. The acetone solution can be directly potentiometrically or argentometrically titrated. The difference from the total chlorine gives the insoluble chlorine which is calculated as insoluble AlCl<sub>3</sub>. The soluble AlCl<sub>3</sub> content is approximately the same as the sublimable AlCl<sub>3</sub> which is a measure of the catalyst's activity.

1-Amino-4-Oxyanthraquinone(2) Chromatographic Determination in 1,4-Diaminoanthra-  
quinone  
Leverkusen

IV/1943

Reference: 1,4-Diaminoanthraquinone (4)

2-Aminothiazole(1) Determination in HCl Solution  
Leverkusen

IV/1943

This determination can be carried out potentiometrically by titration with NaOH. The first inflexion point corresponds with neutralization of the free acid and the second after saturation of the aminothiazole combined HCl.

Ammonia(29) Thiocyanate determination in Ammonia Liquors

Reference: Thiocyanate (2)

Aniline(27) Analysis of Aniline-Diphenylamine Mixtures  
Oppau

IV/1943

Diphenylamine is a weak base, the mineral salts of which are very easily hydrolyzed in the presence of water.

This is not the case with aniline. Consequently a mixture of the two bases can be neutralized with  $HCl$  and treated with a large excess of water, whereby the diphenylamine, which is practically water-insoluble, will separate out. The separation is accelerated by immersing the container in ice water. The diphenylamine is filtered through a weighed Jena crucible, washed with water, dried in vacuum and weighed. The aniline can be determined in the filtrate with bromine according to I.G. Method No. 31. The nitrogen content of the isolated diphenylamine can also be determined. The procedure was tested on synthetic samples.

### Anthranilydisulfide

- (1) Study of Technical Mixtures of Anthranylmercaptan and Anthranilydisulfide  
Leverkusen

IV/1943

Reference: Anthranilydisulfide (1)

### Apparatus

- (13) Preparation of Double Distilled Water  
Oppau

IV/1943

Reference: Distilled water (2)

- (14) Handling Samples in Inert Atmospheres

An approximately cubical box is provided with a glass pane at the top, and a door at the side. Two other adjacent sides are provided with holes into which are fitted rubber gloves. Further, there are provided an inlet and an outlet for a stream of inert gas. This set-up is used for handling samples in inert atmosphere for instance  $AlCl_3$  in dried air, pyrophoric catalysts in  $H_2$  atmosphere, caustic alkalies in  $CO_2$ , etc., and other uses.

### $\alpha$ -Chlor- $\beta$ -Nitrohydrocinnamic acid Nitrile

- (1) Total Content Determination

Saponify with a mixture of glacial acetic and hydrochloric acid to the corresponding carboxylic acid and ammonia, the latter being determined by distillation.

### Chromatography

- (3) Determination of 1-Amino-4-Oxyanthraquinone in 1,4-Diaminoanthraquinone  
Leverkusen

IV/1943

Reference: 1,4-Diaminoanthraquinone (4)

Distilled Water(2) Apparatus for the Preparation of Double Distilled Water  
Oppau

IV/1943

The ordinary distillation apparatus is provided with an arrangement to keep the water level constant and at the same time continuously feed fresh water to the still to replace the volume distilled. This practice results in a continuous and considerable enrichment of the salt content of the water left in the still, i.e., from 100-1000 times. This results in gradually lowering the capacity of the still, limiting its output and lowering the quality of the distilled water obtained. These objections were overcome in Oppau as follows:

The inlet and outlet of the feed are separated. The outlet remains as before in the level regulator. The inlet, however, is at the opposite end of the water level so that no impurity concentration can take place. Since it is necessary to preheat the feed, this is carried out by passing through a partial condenser placed between the still and the regulation condenser.

1,4-Diaminoanthraquinone(4) Determination of 1-Amino-4-Oxyanthraquinone Chromatographically  
Leverkusen

IV/1943

The separation is carried out by passing a pyridine solution of the sample through  $Al_2O_3$  and washing with water. The diamino-compound is dissolved quantitatively in the water while the oxy-compound remains adsorbed in the upper layer of the alumina. It can then be removed with hot NaOH solution and determined colorimetrically. After washing off the diamino-compound, the 1-amino-4-oxy- also the 1-oxy-anthraquinone can be detected qualitatively by treating the  $Al_2O_3$  column with N/10 NaOH which forms an orange colored ring which can be washed off somewhat quicker as the directly colored ring of 1-amino-4-oxyanthraquinone. A quantitative separation cannot be effected.

Dichlorophenyltrichlorethane(1) Use to Prevent Spoilage  
Leverkusen

IV/1943

Reference: Spoilage Prevention (1)

Dimethyldithiocarbannic Acid(1) Determination of Total Content  
Leverkusen

IV/1943

The titration procedure of Elberfeld, using  $K_2Fe(CN)_6$  for the determination of the sodium salt of the above acid, was tested. The titration has a sharp end point, but the results are about 0.5% higher than the values obtained by the weight of the tetramethyl thiuram disulfide which separates through the titration. This difference is ascribed to oxidizable impurities.

### 1,5-Dioxyanthraquinone

#### (2) Determination of 1,2,5-Trioxanthraquinone Leverkusen IV/1943

A study made in an effort to shorten the time necessary for the determination of 1,2,5-trioxanthraquinone, has shown that the trioxy-compound can be directly determined in the original sample by the measurement of the extinction with a step-photometer. Trioxanthraquinone gives a violet solution when made alkaline with NaOH showing a molecular extinction of 9000 at 610  $m\mu$ , whereas the 1,5-dioxy solution is orange-yellow and has an extinction of 36, for which a correction can be made. Under certain circumstances the trioxy may be separated chromatographically from the dioxy, the remaining impurities separated and the extinction measurement carried out.

### Diphenylamine

#### (1) Analysis of Diphenylamine-Aniline Mixtures Oppau IV/1943

Reference: Aniline (27)

### Rubber

#### (18) Determination of Tributylphenol Leverkusen IV/1943

The finely divided rubber is swelled in benzene, precipitated with methanol, the filtrate is evaporated to dryness and the tributylphenol sublimed off the residue and determined gravimetrically.

#### (19) Determination of Oxycresylcamphan Leverkusen IV/1943

The rubber is swelled in benzene, precipitated with methanol and filtered off. The filtrate is evaporated to dryness, the residue taken up in methanol and coupled with true Red Salt GG in the presence of piperidine. The resulting red dye is determined with a step-photometer.

Mopasin Sulfate

- (1) Determination of the Sulfinic Acids  
Leverkusen

IV/1943

Reference: Sulfinic Acids (1)

Mersolate

- (20) Determination of the Sulfinic Acids  
Leverkusen

IV/1943

Reference: Sulfinic Acids (1)

Oxycresylcamphan

- (1) Determination in Rubber  
Leverkusen

IV/1943

Reference: Rubber (19)

Thiocyanate

- (2) Determination in Gas and Ammonia Liquors  
Oppau

IV/1943

Weiser, Chem. Ztg. 1912, p. 1285 recommends the following procedure: 100 mls. sample is evaporated to 20 mls. in a porcelain dish on a water bath, diluted with 50 mls. and again reduced to 10 mls. The residual volume is filtered in a 25 or 50 ml. graduated cylinder, 2 mls. of a solution of 6 gms.  $FeCl_3$  in 100 mls. 10% HCl are added and the volume made up to 25 or 50 mls. with distilled water. The solution is colorimetrically compared with a standard prepared by adding the required amount of N/50  $NH_4CNS$  solution.

Spoilage Prevention

- (1) Research with Gesarol  
Leverkusen

IV/1943

Gesarol, a Geigy preparation for spraying vegetables and fruits, was analyzed. The active constituent was found to be dichlorophenyltrichlorethane. Other organic compounds, such as carbinols, were found to be absent.

Soda

- (6) Determination of Water and Bicarbonate  
Leverkusen, org.

IV/1943

The method of Dubovitz, Chem. Ztg. 45, 890 (1921) for the determination of water and bicarbonate was tested and found useful. The sample is heated to 250°C.,

whereby the bicarbonate is decomposed and the CO<sub>2</sub> evolved is collected in a KOH bulb and weighed. The water content is the difference between the loss on heating and the CO<sub>2</sub> collected.

### Sulfinic Acids

#### (1) Determination Leverkusen

IV/1943

Sulfinic acids react with HNO<sub>2</sub> with formation of disulfonohydroxylamine, according to the equation



On this basis the sulfinic acids content of Nopasin sulfinate can be determined by titration with HNO<sub>2</sub> in acid solutions, using KI-starch paper to determine the end point. Any H<sub>2</sub>SO<sub>3</sub> present must be separately determined and allowance made for the additional HNO<sub>2</sub> consumption.

### Sulfite Waste Liquor

#### (7) Pentoses Determination for the Detection of Beechwood in Spruce Wood Sulfite Liquors

Since the wood of deciduous or foliage trees contains more pentoses than pine-needle trees, the presence of the former can be detected from the pentoses content of sulfite-liquors. The determination is carried out as usual in furfural and the pentoses precipitated as furfural-barbituric acids.

### Tributylphenyl

#### (1) Determination in Rubber Leverkusen

IV/1943

Reference: Rubber (18)

### 1,2,5-Trioxanthraquinone

#### (2) Determination in 1,5-Dioxanthraquinone Leverkusen

IV/1943

Reference: 1,5-Dioxanthraquinone (2)

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