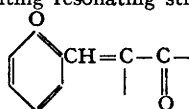


Organic

The Specificity of the Salicylaldehyde Reaction for the Detection of Acetone. T. Thomson (*J. Soc. Chem. Ind.*, 1946, 65, 121-124)—Braunstein's theory (*Nature*, 1937, 140, 427) of the mechanism of the salicylaldehyde reaction for detecting acetone is rejected, and it is shown that the reaction is less selective than was stated by Braunstein. The reaction evidently involves condensation of the salicylaldehyde with a methylene group in the α -position to an unsaturated group such as carbonyl, and the formation of an alkali salt of the resulting resonating structure:



Method—For the test, use aqueous solutions; dissolve in alcohol substances insoluble in water, and dilute with water. Place in a test tube 5 ml. of the solution under examination, and add 4 ml. of 40 per cent. sodium hydroxide solution, followed by 1 ml. of reagent prepared by diluting 20 ml. of salicylaldehyde to 100 ml. with 95 per cent. alcohol. Heat the reactants at 40 to 50° C. for 20 mins., and compare the colour with that of a "blank" carried out simultaneously on the reagents. The following results were obtained.

Substance	Concentration of soln. used for test (parts by volume)	Colour obtained in test
Acetone	1 in 50,000	Red
"	1 in 250,000	Red with yellowish tinge
"	1 in 6,250,000	Pale orange; definitely deeper colour than reagents alone
"	1 in 12,500,000	Lemon-yellow, indistinguishable from reagents alone.
Pyruvic acid ..	1 in 50,000	Red with yellowish tinge
"	1 in 250,000	Pale orange, definitely deeper than reagents alone
Ethyl acetoacetate ..	1 in 200	Orange-red
"	1 in 500	Brownish-yellow
"	1 in 250,000	Golden-yellow, just distinguishable when compared directly with lemon-yellow colour of reagents
Acetophenone ..	1 in 200	Bright red
"	1 in 100,000	Lemon-yellow, indistinguishable from reagents
Benzalacetone ..	1 in 250,000	Golden-yellow, just distinguishable when compared directly with lemon-yellow colour of reagents
Acetaldehyde ..	1 in 500	Golden-yellow, just distinguishable when compared directly with lemon-yellow colour of reagents
Propionaldehyde ..	1 in 500	Deep orange
"	1 in 125,000	Lemon-yellow, indistinguishable from reagents
Methyl ethyl ketone ..	1 in 500	Orange
"	1 in 50,000	Golden-yellow, deeper than reagents
Diethyl ketone ..	1 in 500	Golden-yellow, decidedly deeper than reagents
Cyclohexanone ..	1 in 500	Deep orange
"	1 in 250,000	Yellow, barely distinguishable from reagents
Methyl cyclohexanone ..	1 in 500	Orange
"	1 in 50,000	Lemon-yellow, indistinguishable from reagents
Cyanacetic acid ..	Approx. 3 in 100	Deep orange
Fluorene	—	No reaction

The constitution of the coloured products has been examined. That formed with acetone is the monosodium salt of *o*-hydroxybenzalacetone (salicylacetone); the neutral material precipitated from the aqueous solution on titration with acid had, after recrystallisation from aqueous alcohol, m.p. 137° to 139° C. The product formed with benzalacetone is the monosodium salt of salicylbenzalacetone. Ethyl acetoacetate gives the sodium salt of salicylacetone, evidently formed by normal ketonic hydrolysis from the condensation product of salicylaldehyde and ethyl acetoacetate. Acetophenone gives the sodium salt of salicylacetophenone; the neutral material obtained after acidification of the salt had m.p. 154° to 156° C. E. M. P.

Analytical Reactions with Organic Reagents. A. Steigmann (*J. Soc. Chem. Ind.*, 1946, 65, 233-234)—The following specific or very selective reagents are described. (1) *A sensitive reagent for thiosulphate*—By heating, dissolve 0.1 g. of thionine (B.D.H. redox indicator) in 50 ml. of ethylene glycol or other inert alcohol. Add 1 ml. of this solution to 50 ml. of water and dilute to 100 ml. To 3 ml. of the carbon-filtered and then evaporated wash water of the gelatin to be tested, or other test solution, add 1 ml. of the diluted thionine solution. Perform a "blank" test. To the test and the "blank," add 0.4 ml. of 5 *N* sulphuric acid. Stand both in boiling water for 7 to 8 min., and cool in running cold water for 20 min. Less than 1 : 40,000 sodium thiosulphate causes a colour change from violet to blue, whilst higher concentrations cause partial or total bleaching. The new test is less sensitive, but much more selective than the iodine-azide reaction. Only thiocarbamides interfere at 50 to 100 times higher concentrations by giving direct bleaching without prior colour change.

(2) *Specific reagents for nitrate, guanidine, urea, and nitroso-compounds*—(a) Add 1 g. of carbazole to 100 ml. of isopropyl alcohol, followed by 1.4 g. of dimethylglyoxime. Dissolve by heating.

Crystals precipitate on cooling, and are dissolved again before use. To 3 ml. of concentrated hydrochloric acid add 0.5 ml. of 1 per cent. arginine hydrochloride, or guanidine carbonate solution, or 1 ml. of 10 per cent. gelatin solution (as a source of arginine), and then 0.2 ml. of the reagent, followed by 1 to several drops of the test solution, or of water for the "blank." Heat for 20 min. in boiling water. Traces of nitrate produce a brilliant blue coloration. By using a small amount of nitrate in the reagent instead of guanidine, the reagent can be used to test for guanidine (arginine). To 3.5 ml. of concentrated hydrochloric acid add 1 ml. of 2 per cent. gelatin solution, 2 drops of 1 per cent. potassium nitrate solution, and 0.5 ml. of the reagent. Boil for 12 min. By dissolving in 10 ml. of isopropyl alcohol the precipitate from an excess of carbazole, the blue colour goes into the aqueous solution, which can then be used for colorimetric determination of nitrate.

(b) To 3 ml. of concentrated hydrochloric acid add 0.5 ml. of 0.5 per cent. alcoholic acetyl benzoyl solution, 0.2 ml. of 10 per cent. urea solution, and 1 ml. of nitrate test solution. Boil for 10 min. Nitrates cause the development of a stable, carmine colour, suitable for colorimetric determinations. Guanidines (arginine) do not interfere. The reaction is specific for ureas (citrulline) when, instead of urea, nitrate is included in the reagent. To detect citrulline in gelatin, add 2 drops of 1 per cent. potassium nitrate solution, and 0.5 ml. of 0.5 per cent. alcoholic acetyl benzoyl solution to 3 ml. of concentrated hydrochloric acid, followed by 0.5 ml. of water and 1 g. of gelatin powder. Boil for 6 min. The reagent cannot be used for proteins containing tryptophan and sugars, or giving a strong humin reaction on acid hydrolysis. A slight humin reaction is given by gelatins, but this does not interfere seriously.

(c) Cheaper reagents for nitrate or urea are obtained by substituting dimethylglyoxime or diacetylmonoxime for acetyl benzoyl in (b). The reactions for nitrate described above are not interfered with by traces of nitrite, and are as sensitive as the nitrate reactions with polyhydric phenols.

Resorcinol, a 1 per cent. solution in concentrated hydrochloric acid, is also a reagent for nitroso-compounds, such as α -nitroso- β -naphthol and nitroso-R-salt. Add 3 ml. of the resorcinol solution to the evaporated test solution; transfer to a test tube, and put into boiling water, whereupon there develops a blue-violet colour which, after dilution with water, can be extracted with ether. The colour in ether is red. Resorcinol in concentrated sulphuric acid reacts also with some nitro-compounds, e.g., with *p*-nitrophenylhydrazine, but not with 2 : 4-dinitrophenylhydrazine, giving sulphonated dyes which are not extracted by ether. The dyes from the resorcinol nitrate and nitrite reactions are also soluble in ether. Resorcinol cannot be used for gelatin in Selivanov's sugar reaction, as technical gelatins invariably contain traces of nitrates, or nitrites, or both.

(3) *Reagents for carbohydrate-sugar*—(a) Dissolve 80 g. of urea in 200 ml. of concentrated hydrochloric acid. (b) Dissolve 0.2 g. of tryptophan in 100 ml. of concentrated, metal-free hydrochloric acid; use the reagent fresh and, if suitable, sensitised by 50 mg. of cysteine hydrochloride. To 0.5 g. of gelatin powder, or the dry residue of an evaporated carbohydrate or sugar solution, add 3 ml. of solution (a) or (b). Put the test tubes into boiling water for 20 min. or 3 min., respectively, soaking the

gelatin for 10 min. in the reagents before heating. A brown colour (melanin) develops with (a), and a violet colour with (b), if carbohydrates or sugars are present. Results that have been obtained with gelatins of different kinds are noted. Carbohydrate solutions will indicate tryptophan, e.g., a solution of 1 g. of potato starch in 100 ml. of concentrated hydrochloric acid. Raise the temperature of the solution to 70° C., cool to room temperature, and use as a tryptophan (Voisenet) reagent. (c) Boiling monochloroacetic acid is a reagent for protein sugars. To 2 g. of monochloroacetic acid add 25 to 100 mg. of dry protein, and raise the temperature to 185° C. (b.p. of the acid). Reflux for 5 min., add 2 ml. of water, and carry out a colorimetric estimation. Hydroxyproline or tryptophan condenses in boiling monochloroacetic acid with aldehydes to give melanin brown or violet colorations, respectively. If tryptophan and hydroxyproline are absent from the protein under test, add 5 to 10 mg. of one of these before the sugar test (this addition is unnecessary with gelatins). This test can also be used to detect tryptophan or hydroxyproline if only one of these is present. For example, to test gelatin for hydroxyproline, add 7 mg. of glucose and 10 to 20 mg. of the gelatin powder to 2 g. of chloroacetic acid. Heat under reflux at 185° for 5 min., dilute with 2 ml. of water, and estimate colorimetrically.

(4) *Anionic and cationic wetting agents and anionic and cationic "gelatins" as analytical reagents*—(a) *Reagent for alkaline earths*—To 100 ml. of 1 : 10,000 thionine solution add 2 ml. of B.D.H. Wanklyn's volumetric soap solution and 75 ml. of 0.880 ammonia. To 2 ml. of this reagent add 1 drop of the test solution, e.g., tap water. If the solution contains an alkaline earth, the blue violet colour changes to red-violet. As the ammonia evaporates on keeping the solution, the reagent must not be too old. (b) *Reagent for soap*—To 100 ml. of 1 : 10,000 thionine solution add 75 ml. of 0.880 ammonia. Traces of soap, fat or soap-forming fatty acid change the red-violet colour of the reagent to blue-violet. (c) *"Metal acid" reagent*—To 100 ml. of 1 : 5,000 tropæolin 00 solution (or thymol blue solution) add 2 ml. of 2 per cent. Sapamin KW 200 per cent. solution, and then 200 ml. of 0.5 N hydrochloric acid diluted with 100 ml. of distilled water. Traces of sodium tungstate or salts of other metal acids turn the yellow reagent red. Cetyltrimethylammonium bromide (Cetavlon) can be used instead of Sapamin. (d) *Preparation of cationic "gelatin" and a cobalt-adsorbing reagent therefrom*—Stir 30 g. of air-dried gelatin powder, practically metal-free, into 85 ml. of acetic anhydride; raise the temperature within 20 min. to 125° while stirring. Add 150 ml. of water. Collect the acetylated gelatin on a Buchner funnel and wash several times with water. Dry the gelatin powder. To 100 ml. of 0.1 per cent. nitroso-R-salt solution add 7 g. of the cationic acetylated gelatin and 0.3 ml. of 5 N acetic acid. Heat to 50°, and stir occasionally. After 2 hrs., collect the stained gelatin powder on a Buchner funnel. Wash with water and dry. Use the powder as a sensitive cobalt-adsorbing reagent in the hot or boiling test solution (Schmidt, "The Chemistry of the Amino Acids and Proteins," Springfield and Baltimore, 1944, 720–778). (e) *Preparation of an anionic gelatin, for the adsorption of basic dyes and amines (cationic complexes) of silver, copper, nickel, cobalt, cadmium, and zinc from ammoniacal solutions*—Stir 30 g. of gelatin powder into a mixture

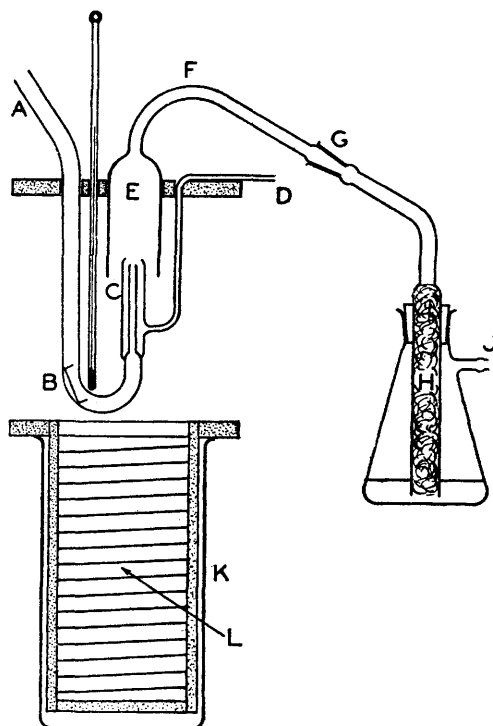
of 10 ml. of 40 per cent. formaldehyde and 50 ml. of isopropyl alcohol. Heat to 80°C ., and maintain this temperature for 15 min. Decant the excess of solution. Add 100 ml. of water to the tanned gelatin powder and collect it on a Buchner funnel. Wash several times with water and then with diluted ammonia (1 + 9). Wash again, filter, and dry. *Example of a metal adsorption*—Add to 3 ml. of 1 : 200,000 cobaltous nitrate solution 20 mg. of anionic gelatin powder and 2 drops of 5 *N* ammonia solution. Heat and shake, centrifuge, wash, and centrifuge again. Then add 2 drops of 0.5 per cent. *s*-benzylthiuronium chloride solution and 2 ml. of water to the centrifuged gelatin powder containing the cobalt. Heat, add 2 drops of 5 *N* ammonia solution, and heat again to produce the characteristic mauve colour of cobalt on the gelatin grains. Adsorbed nickel is detected in the same way. The procedure is the same for adsorption of the other metals mentioned above. E. M. P.

Analysis of Organoselenium Compounds. J. D. McCullough, T. W. Campbell, and N. J. Krilanovich (*Ind. Eng. Chem., Anal. Ed.*, 1946, 18, 638-639)—Usual methods are either long or inaccurate. In testing available methods, the best results were obtained by a modification of the oxygen-combustion method at a glowing platinum surface (Niederl and Niederl, "*Micromethods of Quantitative Organic Analysis*," 2nd Ed., p. 208, New York, John Wiley and Sons, 1942). Van ter Meulen's method of determining selenium dioxide iodimetrically has been modified to eliminate errors due to the presence in a sample of hydrochloric acid or of chlorine; the length of the method, and the need for constant attention rendered new procedures desirable. These now are (a) Flame combustion giving the selenium content directly. (b) Volumetric methods yielding the equivalent weight of the compound.

Flame combustion method—This is based on a known method of halogen determination, but an oxygen flame is necessary for complete oxidation of the selenium to its dioxide.

Apparatus and Procedure—The apparatus used is shown approximately a quarter the actual size, ABCD being made of transparent quartz tubing, the dimensions of which are apparently not critical, and the chimney and absorption tube EFGH is of Pyrex glass with a T joint at G. H is packed with grease-free, clean Pyrex glass-wool. The electric furnace, L, is contained in a 10 × 20 cm. Pyrex electrolytic beaker, K, the heating unit consisting of 18 ft. of 22-gauge Chromel-A wire (1.0 ohm per foot) wound on a Transite frame, and energised by a variable transformer. Slight modifications are necessary to render the procedure applicable to fluid samples. A sample of 20 to 80 mg. selenium content is workable depending on the thiosulphate normality, size of aliquots, etc., and is added at A, either as a pellet, or weighed directly into a small platinum boat, and shaken to B. Hydrogen is passed through D at 200 ml. per min., and is ignited at C after expulsion of the original air. The oxygen supply is increased slowly to 100 ml. per min., thus giving a stable flame. The furnace and chimney assemblies are brought into place, and a current of 1 litre of air per min. through the chimney is produced by applying suction at J. Gauges are recommended for the three lines, but are not essential. The heater current is started, and raised to give a suitable rate of volatilisation of the sample as shown by the blue coloration of the otherwise

colourless flame, which forms also an indicator of completeness of combustion. Most of the dioxide is deposited at F, but a significant quantity is drawn into H. In compounds where decom-



position accompanies volatilisation, the dark non-volatile residue is slowly burned in the oxygen stream by gradually raising the temperature, the thermometer having been removed; the decomposition may often be avoided by using a higher temperature, or higher rate of oxygen flow. Combustion is completed in 15 to 45 min., and the furnace is lowered to permit cooling, during which time suction is maintained. The deposit is rinsed from the chimney into a 250- or 500-ml. volumetric flask; H is rinsed by passing four 25-ml. portions of water slowly up and down the glass wool, suction being applied alternately at G and J, and the rinsings added to the flask. Completeness of rinsing may be tested by adding the final water-rinsing to an acid solution of starch and potassium iodide, which should give no coloration. After filling the flask to the graduation mark, aliquot portions are titrated.

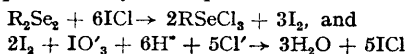
Titration of selenious acid—The dissolved oxygen is expelled by gently boiling the solution, which is then ice-cooled, and kept air-free by the periodic addition of dry ice, or by the passage of carbon dioxide, or nitrogen, through the solution. These precautions are not essential, but give slightly more accurate results. Fifteen ml. of 2 per cent. starch solution, 10 ml. of 1.5 *M* potassium iodide, and 10 ml. of 6 *N* sulphuric acid are added in that order, with thorough mixing after each addition. The solution is then titrated at once with 0.05 *N* thiosulphate until the turbid brown solution turns clear red. 1 ml. of 0.05 *N* $\text{Na}_2\text{S}_2\text{O}_3$ = 0.9870 mg. of Se.

Results and discussion—The accuracy was tested on nine, carefully purified substances containing selenium with carbon and hydrogen, and oxygen, or nitrogen, or chlorine. Experimental and theoretical results are compared, and, for the samples analysed, the maximum deviation of 1 part in 204 occurred with *p*-nitrophenolselenocyanate $C_6H_4N_2O_2Se$ of theoretical selenium content 34.77 per cent.

Volumetric methods—Compounds containing selenium-halogen or selenium-oxygen bonds may be determined iodimetrically, since they react with potassium iodide solution to give the tri-iodide. The titration of diarylselenium dihalides has been previously described (J. D. McCullough, *J. Amer. Chem. Soc.*, 1942, **64**, 2672).

Procedure for compounds reducible by potassium iodide—0.1 to 0.5 g. of the sample is weighed into 5 ml. of carbon tetrachloride, 25 ml. of 0.3 *M* potassium iodide, and 2 ml. of 6 *M* sulphuric acid and, after thorough shaking, is titrated with standard thiosulphate in the presence of starch, added near the end-point. Values are given for eleven compounds in which the maximum discrepancy is 1 part in 262 for 4-bromodiphenylselenium dibromide, $C_{12}H_8BrSeBr_2$, of theoretical equivalent 236.

Procedure for aryl diselenides—These may be titrated iodimetrically to the iodine monochloride end-point if the concentration of hydrochloric acid is kept at 5.0° to 5.5° F. at the end-point. Errors of 0.5 to 1.0 per cent. are incurred by side reactions, but the complex reactions may be more simply represented by the equations:—



The iodine monochloride solution is prepared by titrating 1 ml. of 1.5 *M* potassium iodide in 50 ml. of 12 *M* hydrochloric acid, using 5 ml. of carbon tetrachloride for indicator purposes. When the carbon tetrachloride is just colourless, 25 ml. of 12 *N* hydrochloric acid and the weighed diselenide are added and, after shaking, the mixture is titrated with standard potassium iodate. M. E. D.

Determination of Chlorine in 2:2'-Dihydroxy-5:5'-dichlorodiphenylmethane. D. M. Jenkins, K. L. Waters, and G. D. Beal (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 609-610)—A rapid method for control purposes has been devised for the determination of chlorine in 2:2'-dihydroxy-5:5'-dichlorodiphenylmethane (DDM or Compound G-4) depending upon oxidation of the compound with alkaline potassium permanganate, acidification, and reduction of the excess of permanganate by sodium nitrite, and the determination of the liberated chloride by the Volhard method, or by photometric measurement of the turbidity of silver chloride suspensions. General applicability of the method to organic compounds containing chlorine is not claimed, but it gives satisfactory results with DDM.

Procedure—Dissolve about 0.2 g. of DDM powder in 15 ml. of 10 per cent. sodium hydroxide solution in a 500-ml. Erlenmeyer flask, add 35 ml. of saturated potassium permanganate solution, or as much more as is necessary to maintain the purple colour of the permanganate. After introducing a few glass beads, boil the mixture gently for 10 min. Cool to room temperature, add 75 ml. of water and 10 ml. of concentrated nitric acid. Reduce the excess of potassium permanganate immediately by means of 10 per cent. sodium nitrite solution, added pre-

ferably from a burette, add 25 ml. of 0.1 *N* silver nitrate, and titrate the excess with 0.1 *N* ammonium thiocyanate, 2 ml. of ferric alum solution being used as indicator. Make a blank determination with the reagents.

The analysis of emulsions containing DDM, frequently used to impart water-repellency as well as mildew-proofness to fabrics, requires little change in the procedure described. The wax used in these emulsions readily separates on the surface of the liquid during oxidation, and does not interfere with the final titration.

Owing to the small amount of DDM applied to fabrics, the chloride content of the oxidised extract cannot be conveniently titrated, but is readily determined by photometric methods, the acidity of the solution after addition of sodium nitrite being adjusted so that the silver chloride is maintained in suspension. Extract 2 g. of the very finely cut fabric in a 150-ml. beaker three times with successive 50-ml. portions of 0.025 per cent. sodium carbonate solution by boiling gently for 5 min., and decant the hot extracts into a 200-ml. flask. Wash the extracted fabric twice with 15-ml. portions of the sodium carbonate solution, and dilute the combined extracts and washings to 200 ml. with sodium carbonate solution. Filter about 50 ml. and follow the oxidation procedure already described with these exceptions. Use a 25-ml. aliquot of the filtrate, reduce the volume of potassium permanganate solution to 25 ml., and omit the dilution with water after oxidation. After reduction of the excess of permanganate by means of acid and sodium nitrite, add a few drops of phenolphthalein indicator, and then enough 10 per cent. sodium hydroxide to produce a faint pink colour. Acidify with 10 ml. of diluted nitric acid (1 + 3), transfer the liquid to a 200-ml. flask, cool to room temperature, add 4 ml. of 0.1 *N* silver nitrate, dilute to the mark with water, and invert the flask several times. After 10 min., measure the turbidity with a suitable electrophotometer (the Fisher instrument was used in the investigation), using a 525 filter, and determine the DDM content by reference to a standard curve. It is necessary to establish two "blank" values, *viz.*, an extract blank and a reagent blank. For the extract blank, pipette a 25-ml. aliquot of the filtrate into a 200-ml. flask, add 125 ml. of the sodium carbonate solution, 10 ml. of diluted nitric acid (1 + 3), and 4 ml. of 0.1 *N* silver nitrate, and dilute to the mark. Invert the flask several times and read after 10 min. This extract blank representing inorganic chlorides is often negligible. For the reagent blank, follow the same procedure, omitting the 25-ml. aliquot of the extract.

Since the logarithm of the transmittance of silver chloride suspensions is an inverse and very nearly linear function of the turbidity, four determinations are adequate to establish the standard calibration curve. Establish the curve by first dissolving 500 mg. of DDM in enough 2.5 per cent. sodium hydroxide solution to make 100 ml. of solution. Use suitable aliquots of this solution, containing 5 mg. of DDM per ml., to cover the range of 2 to 15 mg. The procedure is then as described for the fabric extract. A. O. J.

Solubility of Acetylene in Acetone. V. E. Brameld and M. T. Clark (*J. Soc. Chem. Ind.*, 1946, **65**, 58-61)—A gravimetric method for determining the solubility of acetylene in acetone has been developed, and values have been determined at 5° intervals over the range 0° to 40° C. The

method is to saturate air-free acetone of B.S.509 quality with acetylene until the whole system is in equilibrium under a total pressure above the solution equal to one atmosphere plus the saturation vapour pressure of acetone at the test temperature. Thus, the solution is in equilibrium with acetylene at a partial pressure of one atmosphere. From the weights of vessel empty, plus acetone, and plus acetone and gas, the weight of acetylene dissolved by unit weight of acetone at the test temperature is calculated. Certain corrections for the air inevitably present in the acetylene are applied. Results obtained for the solubility coefficient, S_w , are:

Temp., °C.	S_w , mg. of C_2H_2 /g. of acetone
0	68.7
5	61.0
10	53.5
15	46.4
20	39.4
25	32.7
30	30.0
35	25.7
40	24.4

The equation $S_w = \frac{13100}{(T-185.3)} - 81.3$, where T is the absolute temperature, holds over the temperature range -30° to 35° C.

The following values were found for the solubility at different partial pressures of acetylene; $S_w = S'_w/P_{A_0}$ where S'_w is the solubility under an acetylene partial pressure of P_{A_0} atmospheres.

Test temperature 30° C.

Acetylene partial pressure, atm.	S'_w , mg./g.
0.742	22.6
0.760	23.2
0.805	24.4
0.835	25.0
0.897	26.5
0.905	27.2
0.982	29.2
0.999	29.7
1.047	31.0
1.054	31.2
1.164	34.0

E. M. P.

Analysis of Dilute Melamine Resin Solutions.

B. Ivarsson and B. Steenberg (*Svensk Papperstidn.*, 1946, **49**, 1; through *Tech. Bull.*, 1946, **23**, 12)—For routine work, a known wt. of the sample, as supplied for use in paper, is diluted to 35 ml., 0.1 N sodium hydroxide is added, and after vigorous agitation, the resulting turbidity is evaluated in a Pulfrich photometer. The method must be calibrated with known weights of resin, under constant conditions, of which the most important are the volume of sodium hydroxide added, the time elapsing after its addition, the concentration and age of the original and diluted solutions, and the presence of

foreign substances, *e.g.*, salts, starch, and proteins. Methylol melamine allows of no reliable colour reactions or, because of the stability of the triazole ring, Kjeldahl determination, the picrate is too soluble to be used for a gravimetric determination, and precipitates with salts and alkali are unmanageable gels.

J. G.