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The elements which it is most frequently necessary to detect in organic compounds are: carbon, hydrogen, oxygen, nitrogen, sulfur, halogen, phosphorus, silicon, and arsenic, and less commonly, e.g. mercury, boron and antimony. There is so far no analytical scheme covering all these elements and usually only two to four of the elements are identified after each combustion.

The large number of ways in which nitrogen may be bound in organic compounds makes a specific detection of this element difficult. Thus the choice of method for nitrogen combustion is the chief consideration in systematic analysis and dictates whether or not other elements may be detected after a single combustion. If nitrogen compounds are heated with calcium oxide, ammonia is liberated. Emich \(^1\) used a mixture of calcium oxide and zinc, which is advantageous, among other things, for the simultaneous detection of sulfide and halogen. Bennett, Gould, Swift and Niemann \(^2\) have developed Emich's test to a rapid systematic micro method, covering the elements: nitrogen, chlorine, bromine, iodine, sulfur, arsenic, and phosphorus. The method has been further investigated by Widmark \(^3\), who has shown that disturbing cyanide also can be produced at the combustion.

When heated with carbon and alkali metals or magnesium, nitrogen compounds form cyanide, which can readily and specifically be detected as Prussian blue. Lassaigne \(^4\) ignited the sample with sodium in an open vessel. Sodium has sometimes been replaced by potassium, which has a lower melting point and is more reactive. Castellana \(^5\) used a mixture of magnesium and potash, with which the reaction is intensified if sugar \(^6\) is also added. The risk of a reaction between magnesium and nitrogen from the air cannot, however, be neglected \(^7\).

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In the method proposed here 1—5 mg of the test substances are combusted with sodium at 500—590° in sealed ampoules. The small amount of sample introduced makes the combustion in sealed tubes possible with only little danger of explosion. Indeed, the ampoule very seldom breaks even when explosives are analysed. Moreover, as the combustion should be carried out in an iron furnace, an explosion causes no harm.

In the systematic analysis which follows the combustion, water is added to the content of the ampoule, and the elements or groups of elements are removed from the solution as gases or precipitates. Hence, the detection of one element does not lower the sensitivity of the analysis of an element following in the scheme. Only for the detection of fluorine must the solution be divided.

Each element can be detected in the presence of all the others with the exception of phosphorus, which still cannot be detected in the presence of arsenic.

The analytical method developed is so simple that the analyses could be performed in an ordinarily equipped school laboratory. The main part of the specific equipment and the reagents are purposely made so durable and stable that they could be obtained ready-made commercially or be prepared in the laboratory and afterwards preserved for future analytical work.

PRINCIPLES OF THE ANALYTICAL SCHEME

Upon combustion the different elements of the test substance react with sodium. Arsenic forms arsenide, phosphorus phosphide, sulfur sulfide, and halogen halide. Nitrogen is converted to cyanide, the carbon source being either the organic compound itself or the xylene vapor which the ampoule contains. When the ampoule is opened and water added AsH₃ and PH₃ escape. The gases pass AgNO₃-impregnated silica gel giving different colours. The carbon forms a black deposit. A part of the alkaline solution is tested for fluorine and the remainder acidified with sulfuric acid, and boiled to free it from H₂S and HCN, but the halides are retained. The gases are taken up on alkaline ferrous sulfate paper, and the halogens are precipitated from the solution as silver salts and separated.

Not included in this systematic scheme are the important elements hydrogen, oxygen, and silicon and the less common elements such as antimony, boron, and mercury. The possibility of extending the method is being studied.

REAGENTS AND EQUIPMENT

Combustion ampoules. Seal off a piece of Pyrex tubing (outer diameter 6.5—7 mm, inner diameter 2—2.5 mm) of 16 cm length to form two equal round-bottomed tubes. Cut sodium wire (1.2 mm diameter; anal. pure) in 1 cm pieces under xylene. Rapidly wipe one piece free of liquid and transfer it to the bottom of the tube, warm for ½ hour at 95° in an oven and immediately heat in a flame about 1 cm from the sodium (without melting the metal), draw out, and, after a few seconds, melt off so that a 3—4 cm long peak is obtained (Fig. 1 a). A stock of such ampoules may conveniently be prepared. Reserve ampoules with thick peaks for solids.

Hydrogen cyanide reaction paper. Press a small round filter paper (e.g. Whatman No. 54) between a hole in a polystyrene plate and a bored stopper (Fig. 2 a). Place 1—2

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MODIFIED LASAIGNE'S METHOD

Fig. 1. a) Ampoule with sodium. b) Charged ampoule. c) Alkaline solution, with AgNO₃-PbAc₂-gel tube. d) Acid solution, with alkaline FeSO₄-paper. e) Silver halide precipitate, with fluorescein and starch papers. f) Fluorine test with moist glass wool. g) Fluorine test upon Zr-alizarin paper.

drops of FeSO₄ solution (1 part saturated solution + 1 part water) on the paper and dry at max. 70°. Add 1 drop of 3 M NaOH and dry in the same way. Carefully loosen the cork with adhering paper from the plate. An active paper should have an even light brown colour and can be kept for a few weeks in a closed vessel.

Zirconium-alizarin paper. Place a filter paper in a solution of 0.2 g ZrOCl₂·8H₂O in 100 ml 4 M HCl for a few minutes, dry and dip twice into a solution of 0.01 g alizarin in 100 ml ethanol, and again dry. Cut the evenly red-violet part of the paper into pieces of \( \frac{1}{2} \times 1 \) cm, and extract with small portions of ether until the ether solution becomes only weakly yellow in colour (about 5 times). The papers may be kept for a long time.

Fig. 2. a) Polystyrene plate (60 × 25 × 2 mm) with holes (diameter 8.5 mm) for bored stoppers with alkaline ferrous sulfate papers.

Fig. 2. b) Iron furnace (diameter 125 mm, height 50 mm) covered with 3 mm asbestos, 5 holes (diameter 15 mm, depth 55 mm) for asbestos tubes, one hole (diameter 7 mm, depth 50 mm) for a thermo-couple.

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Table 1. Equipment box for students' laboratories.

<table>
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<th>Bored stoppers</th>
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**Fluorescein paper.** Impregnate filter paper with a saturated solution of fluorescein in 50% ethanol. The papers may be kept for a long time.

**Starch paper.** Impregnate filter papers with 1% starch solution. The dried papers may be kept for several months.

**Gel tube.** Constrict one end of a 4 cm long glass tubing with an inner diameter of about 3 mm. Place a small amount of fine glass wool at the constriction and add silica gel impregnated with lead acetate to a height of 1 cm and a similar layer of silver nitrate impregnated gel, the layers being separated by glass wool. (Fig. 1 c.) The tubes may be kept for more than a year when stored in a closed dark vessel.

**Lead acetate gel.** Stir silica gel (1 g, Davison, 0.1 - 0.4 mm), which has been oxidized in nitric acid, thoroughly washed and dried, moistened with a solution of 0.1 g PbAc₅·3H₂O in 1 ml of water, and dry.

**Silver nitrate gel.** Impregnate the gel (1 g) purified as above with 0.25 g silver nitrate in 1 ml of water. Store the dried gel in a dark closed glass vessel.

**Asbestos tube.** Cover a test tube (75 x 10 mm) with two layers of aluminium foil and bake on a 2 mm wet layer of asbestos and arm with 0.5 mm copper wire. Remove the glass tube and the aluminium foil after drying.

**SUGGESTED ANALYTICAL METHODS**

**Addition of test substance.** Shake the sodium to the bottom of the ampoule and cut off the top of the peak (Fig. 1 a).

** Liquids and solutions.** Warm the ampoule for a few seconds in a small flame. Attach a capillary containing the sample (ca. 5 μl) to the top of the ampoule. Cool so that the test liquid is drawn into the ampoule.

**Solids.** Press the top of the ampoule into the pulverized sample, and shake the column of substance (approximately 3 mm high) formed in the top down to the sodium. (Sometimes it is necessary to use a thin wire of stainless steel.) Three to four such fillings give sufficient substance.

**Sealing the ampoule.** Warm the tip about 1 ½ cm from the top, first very carefully in a small flame, so that any substance adhering to the glass melts and evaporates. The heating is then increased, causing the glass to melt. If a black line is formed, the ampoule will be properly sealed if the tip of the seal is drawn out again.

Glasses should be used during sealing and in handling the ampoule until it is again opened.

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**MODIFIED LASSAIGNE'S METHOD**

**Combustion.** Place the ampoule, sodium end downwards, in an asbestos test tube (70 × 12 mm) (see below), and heat for 15 minutes in a furnace (Fig. 2b) at 500—590°. The furnace should be heated with a Meker burner. Temperatures above 600° may cause explosion of the ampoule.

**Arsenic and phosphorus**

Remove the ampoule from the asbestos tube and cut off both ends as shown in Fig. 1 b. (Use a pair of tongs.) Smell the opened tube; arsine has an odor of garlic, phosphine of “carbide”. Stir gently within the ampoule with a dry capillary which may then be used for taking out a fluorine test sample. Place the ampoule (sodium end downwards) in a test tube (75 × 10 mm) containing about 1/2 ml of water. Close the test tube immediately with a rubber stopper containing a gel tube (Fig. 1 c), the constricted end of which is turned downwards so that the escaping gas first passes the lead acetate layer. Tap the tube gently till all sodium has reacted with the water. Warm gently *.

Arsine gives, within two minutes, a black zone with a yellow top in the silver nitrate gel **. The yellow colour appears first. Phosphine gives a brown-black zone and the brown colour appears more clearly upon storing the gel tube. — The intensity of the zone colouration with phosphine is about one-tenth of that with arsine.

**(Fluorine), sulfur and nitrogen**

Remove the gel tube and boil the solution for 3 minutes and stir with the capillary previously used. Cool and transfer three drops of the solution to another test tube for the detection of fluorine (see below). Touch the emptied capillary against a drop of weak sodium nitroprusside solution; the trace of test solution remaining is sufficient to give violet colour if sulfide is present.

Add five drops of sulfuric acid (1 : 3) to the main solution and rapidly close the test tube with a hydrogen cyanide reaction paper (Fig. 1 d). Heat the tube and observe the reaction paper just before the liquid starts boiling. Hydrogen sulfide gives a black colour. After boiling for five minutes transfer the paper to a 20 ml test tube, add dilute sulfuric acid and warm. Iron hydroxide and sulfide are dissolved and cyanide is transformed to Prussian blue. In the presence of a large quantity of sulfur, the cyanide reaction may appear as a blue ring.

**Carbon, halogen, and chlorine**

Remove the ampoule, wash with a few drops of water and centrifuge the solution. Elementary carbon, liberated during the combustion, is deposited ***. Transfer the clear solution to a new test tube and add one or two drops of

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* If the solution is boiled the whole gel column will become grey in colour.
** To check the arsine reactions, transfer the black zone (minimum 3 mm high) to a test tube, add 5 drops of conc. HCl and close the tube with a sodium sulfide paper. (Compare HCN reaction paper.) Boil for a short time. Yellow arsenic sulfide is formed on the paper.
*** To check if the deposit is carbon, 4 drops of concentrated HNO₃ are added to the black residue.

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0.5 M silver nitrate solution. Centrifuge down the halide precipitate and decant the solution. Draw up the residual liquid with a capillary. Stir the precipitate for 2 minutes with 4 drops of 2 M NH₃. Most of the AgCl dissolves. Centrifuge and transfer all of the solution, with a capillary, to a new test tube. Add 2 drops of 4 M nitric acid, whereupon silver chloride precipitates.

**Bromine and iodine**

Dry thoroughly the remainder of the NH₃-treated precipitate in the test tube. It is essential that the walls also are completely dry. Mix the precipitate with a very little pulverized PbO₂ and moisten the mixture with about 20 µl sulfuric acid (1 : 3) from a capillary.

Fasten a fluorescein paper and a starch paper about 1 ½ cm from the precipitate in such a way that they are pressed out from opposite walls (Fig. 1 e). Careful heating liberates bromine to give the fluorescein paper an even red colour. The precipitate should still be black. Wait for 1 min. then increase the heating. The precipitate turns grey-white, and iodine is liberated, colouring the starch paper blue. Sometimes iodine forms a few very small red spots upon the fluorescein paper.

**Fluorine**

Heat the three drops of the alkaline solution to complete dryness. The walls of the tube must also be dry. Add 5 drops of concentrated sulfuric acid taking care that no acid contacts the upper parts of the tube. Close the tube with a rubber stopper containing a glass tube (empty gel tube). Place a small amount of moist fine glass wool in the constriction (Fig. 1 f). The reaction may fail to occur if there is too much water. Heat the tube for a few minutes at about 140°. Remove the stopper and press the water in the glass wool into a zirconium-alizarin paper by means of a small glass rod. (Fig. 1 g) Fluorine compounds destroy the red zirconium-alizarin lacquer and give a yellow spot.

**DISCUSSION OF RESULTS**

Detection of arsenic (sensitivity 1—5 µg As). When 5 µl of lewisite is heated with sodium for 15 min. at 100° in an ampoule, water added to the opened ampoule and the mixture heated for 3 min. to 80°, so much arsine is formed that a 1 mm high black shining zone with a yellow top is formed in the silver nitrate gel. If instead the lewisite and sodium are heated to 250° the whole gel column becomes black. A similar result is obtained with samples combusted for 15 min. at 400°, 450°, 500°, 550°, and 590° or for 3 min. at 550°. Combusted for 1 min. at 550° half the column blackens. In an analysis of 5 µl xylene solution containing 5 µg As as lewisite, a 1 mm yellow frontier with a scarcely visible black bottom is formed, and may be enlarged by a cautious suction from a pump; the same reaction is then obtained for 1 µg As as previously for 5 µg. Within 1 hour the main part of the black colour generally changes into lemon yellow. — If too much As₂O₃ (more than 10 mg) is com—

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busted, the amount of sodium does not suffice to form Na₃As; the reaction in the gel tube will be very weak, and a black precipitate of As will form in the test tube after the addition of water.

Some unsaturated halogen compounds *e.g.* allyl bromide and hexachloro-benzene give sometimes a weakly grey-black zone (maximum 2 mm high) in the silver nitrate column. These reactions are devoid of the characteristic yellow or brown colours and can thus easily be distinguished from true arsine and phosphine reactions.

*Detection of phosphorus* (sensitivity 50 μg P). Tricresylphosphate (5 μl) has been combusted for 15 min. at 250°, 350°, 400°, 450°, 500°, 550°, and 590°. A temperature of 400° gives the maximum reaction in the AgNO₃ gel with a 4 mm black zone having a small yellow top. (The same reaction as for arsenic but less sensitive.) A temperature of 450° gives a black zone with a yellow brown top and 500—590° gives a dark brown zone about 2 mm high with a thin black bottom. The possibility of distinguishing between the arsine and phosphine reactions has governed the recommendation of the lower temperature limit (500°) for combustion. Otherwise satisfactory decomposition starts even at about 350°. The higher limit, 590°, is governed by the strength of the glass.

Tricresylphosphate (0.5 μl) heated for 5 min. at 550° gives a very weak brown zone, which is converted by suction to a 1 mm high black zone with a yellow top. Thus, the use of suction makes it impossible here to distinguish between arsenic and phosphorus. — PCl₃ (5 μl) heated for 15 min. at 550° blacken the whole gel column, but after an hour or two some brown particles appear, which can be recognized by careful examination.

The violent reaction between the water and the sodium remaining in the ampoule after the combustion seems to increase the sensitivity of the phosphine reaction in the gel. The shell surrounding the sodium should therefore be broken with a capillary.

The odour of phosphine, but not arsine, is distinctly noticeable when the ampoule is broken, with amounts under 50 μg. There is therefore a possibility of detecting smaller amounts of phosphorus than 50 μg.

*Detection of sulfur* (sensitivity 5—10 μg S). Organic sulfur compounds are so completely decomposed by potassium that Zimmermann⁴ has developed a quantitative method for the determination of sulfur on this basis. Sodium is more convenient and has proved quite satisfactory for qualitative analyses. The low sulphate content in the glass does not interfere in the analyses.

Carbon disulfide (5 μl) combusted for 15 min. at 100° gives a clear sulfide reaction and temperatures above 250° do not seem to increase the reaction. The sulfide reaction appears for 2 μg S (as Na₂S solution) as a weak grey colouring of the dry, brown iron paper; 5—10 μg S give a clear black colour.

The small amount of alkaline solution which is left in the emptied capillary used for the fluorine test, is sufficient to give a positive sulfide reaction with nitroprusside solution, when the test tube originally contained more than 40 μg S.

*Detection of nitrogen* (sensitivity 10 μg N). As pointed out before the detection of nitrogen is the chief consideration in systematic qualitative analysis. Objections to Lassaigne’s method are: a) low boiling substances evaporate before reaction with the sodium, b) diazo-, azo- and polynitro compounds

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evolve nitrogen, c) some heterocyclic compounds do not react, d) halogen is difficult to detect in the presence of nitrogen.

Using the present method a cyanide reaction is obtained in all the three cases a)—c) mentioned. The azo and diazo compounds give a faint colour which is, however, quite visible. The heterocyclic compounds investigated give as strong a colour as other nitrogen compounds.

The carbon required for the formation of cyanide is provided by the decomposed organic compounds. The small amount of xylene which the ampoules contain, is, however, also sufficient to serve as a source of carbon. For example, combustion of NaNO\textsubscript{2}, NaNO\textsubscript{3} and Pb(NO\textsubscript{3})\textsubscript{2} gives a spot of Prussian blue equal to that from about 10 μg N as NaCN.

Nitrobenzene reacts exothermically with sodium, forming cyanide even on heating to only 100°. A weak cyanide reaction is obtained when 5 μl quinoline are heated for 15 min. at 250°, but at 450° the colour is as strong as the corresponding one for nitrobenzene.

If the aqueous solution containing 2 mg CN from the combustion is boiled for 5 min. with 5 drops of sulfuric acid (1:3) all hydrocyanic acid but very little or no hydrogen halide escapes. Thus with this concentration of sulfuric acid the difficulty in detection of halogen (d) above is removed.

Sodium cyanide solution containing 0.3 μg N gives, on boiling with sulfuric acid, very faint blue spots on the paper. With 10 μg the whole area is coloured blue and with large amounts the paper becomes blue-black. The colour reaction from 0.5 ml solution containing 10 μg CN is not decreased by 5 mg NaF, NaCl, NaBr, NaI, NaNO\textsubscript{3} or NaH\textsubscript{2}PO\textsubscript{4}. The presence of sulfide decreases the cyanide colour and often causes it to take the shape of a blue ring. With 1 mg sulfide and 10 μg cyanide one test out of three failed. With higher cyanide content a colour was always obtained.

Detection of carbon (sensitivity ca. 200 μg C). During the combustion carbon is liberated from carbon compounds. The small amount of xylene which the ampoule contains is not enough to give a visible precipitate of carbon.

Compounds (5 μl test) containing much carbon give a black precipitate covering the whole bottom of the test tube. Some nitrogen compounds, e.g. tetrazine carboxylic acid and chloropirin give visible precipitates after centrifuging. NaCN and NaCNS do not give precipitates of carbon.

Salts of some metals, e.g. SbOCl, Bi\textsubscript{2}O\textsubscript{3}, SnCl\textsubscript{4} and PbSO\textsubscript{4} and excessive amounts of As\textsubscript{2}O\textsubscript{3} give similar black precipitates. Mercury compounds give a little shining ball of mercury. Phosphorus compounds give sometimes, at a combustion temperature over 450°, a brown deposit. However, all these precipitates are soluble in conc. nitric acid.

The following compounds with low carbon content gave black precipitates not soluble in cone. nitric acid: carbon tetrachloride, chloroform, chloropirin, dichlorodifluoromethane, imidazole, nitromethane, sodium acid carbonate, sodium carbonate, sodium oxalate, tetrazine dicarboxylic acid, trifluorochloromethane, trifluoro-trichloroethane.

The detection of carbon is being further investigated.
Detection of halogen (sensitivity 40 μg). Most halogen compounds investigated react exothermically with sodium at 100°. Increased temperature or time of combustion does not improve the reaction. It is very important that all the hydrocyanic acid is removed from the acidified solution, as otherwise silver cyanide will be precipitated upon the addition of silver nitrate.

Amounts of chlorine below 40 μg cannot be detected distinctly without aerating the solution, as an opalescence may be caused by the cyanide.

It is only possible to achieve the separation of bromine and iodine when the mixture is very slowly heated. The reactions may fail to appear if sulfuric acid or too much water contaminates the paper.

Detection of fluorine (sensitivity 30 μg). The fluorine reaction (Bennett ²) has been carried out with 3 drops of the aqueous alkaline solution without distillation, but the reaction often gives incorrect results for compounds containing sulfur-nitrogen, and phosphorus-chlorine.

Satisfactory results are obtained only with a dried sample and by warming with concentrated sulfuric acid, the gases (HF and SiF₄) being taken up in moistened glass wool.

Zirconium-alizarin paper which has been made by impregnating first in zirconium solution and then in alizarin solution gives a stronger colour than that which has been impregnated in a mixed solution.

The red zirconium-alizarin lacquer is not bleached by the salts: NaCl, NaI, Na₂CO₃, Na₂SO₃, Na₄S, NaCl, and Na₂C₂O₄. Positive results are obtained with mixtures of 10 μg fluorine ion and 5 mg of the salts. The reaction is only disturbed by bromide ion, which in large amounts (over 500 μg) gives the same reaction as fluoride ion. In analyses of PBr₃, and CHBr₃ brown fumes of bromine are enveloped which sometimes give a weak fluorine test. Sulfuric acid also gives the paper a yellow colour, but in the recommended procedure there is no risk of splattering.

Experiences from students' laboratories. The errors which appear in student analysis are usually concentrated in the first tests. A laboratory introduction confined to 1) boiling off HCN from 0.5 ml solution containing about 2 mg NaCN, 2) separation of Cl, Br and I in the same sample, 3) evaporation of sodium fluoride solution and reactions upon zirconiumalizarin paper, is advantageous in preventing students' errors.

SUBSTANCES TESTED

The substances investigated are tabulated for each element with subheadings for the different groups. Within the groups the substances are arranged according to increasing substitution. For the remainder, alphabetical order is used.

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Arsenic compounds
Arsanilic acid
Arsenoaceitic acid
Arsphenamine
Diphenyl chlorearsonate
Diphenyl cyanarsine
Ethyl dichloroarsine
p-Hydroxyphenylarsionic acid
"Lewisi" "Marpharside"
Necarsphenamide
3-Nitro-4-hydroxyphenylarsionic acid
Phenylarsionic acid
Sodium arsanoacetate

Phosphorus compounds
Cayene phosphate
Diethoxychloro phosphate
Dimethylaminodichlorophosphate
Dimethylynodithioxyphosphate
Ethoxydichloro phosphate
Phenylphosphorus oxychloride
Phosphorus oxychloride
Phosphorus tribromide
Phosphorus trichloride
Sodium glucose phosphate
Sodium glycerophosphate
Sodium phenyl phosphate
Spermidin phosphate
Triozyl phosphate
Triphenyl phosphate

DFP
Mintakol
Tabun
TEPP
Sarin

Sulfur compounds
Bis-(a-chloroethyl) sulfide
Carbon disulfide
Diphenyl sulfide
Diphenyl thiourea
2-Thiodibarbituric acid
Thioglycolic acid
2-Thioalicylic acid
Thiourea
Trithiane

Dimethyl sulfate
Diethyl sulfate
Potassium ethyl sulfate
Methanesulfonic acid
Trional

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Benzenesulfonic acid
Benzenesulfonic acid
p-Toluenesulfonic acid
Benzenesulfonyl chloride
p-Toluenesulfonyl chloride
3,4-Dichlorobenzencesulfonyl chloride
Methyl methylyxanthate
Saccharin
Sulphapyridine
Sulphathiazole
Thionyl chloride
Thiophene
2-Methylthiophene
2,5-Dimethylthiophene

Amines
Methyamine, 6% in benzene
Methyamine, 30% in water
α-Propylamine
iso-Butylamine
iso-Amylamine
Diethylamine
1,3-Diamino-2-propanol
Tri-n-butylamine
Tri-n-amyline
Triethanolamine
Tetraethylammonium iodide
Cyclohexylamine
Aniline
α-, m-, p-Chloroaniline
α-, m-, p-Bromoaniline
Mesidine
α-Naphthylamine
β-α-Xydylene
Digly cyclamine
n-Dimethylaniline
Triphenylamine

2-Aminopyridimidine
2-Amino-3-methylpyrimidine
2-Benzylamino-pyridine
Bis-(2-aminothiazole)

Amides
Acetamide
n-Iodo-acetamide
n-Dimethyl-formamide
Barbituric acid
Acetanilide
n-Diethylbenzamide
ω-Chloro-2,6-dimethylacetanilide

N-Acetyl-p-phenetidine

Nitro compounds
Chloroperin
Ethyl nitrate
Nitromethane
Tetranitromethane
Nitroglycerin, 10% in ethanol

Nitrobenzene
Nitroanthrone
3-Nitrophthalic acid
3-Nitroquinoline
2,4-Dinitrochlorobenzene
3,5-Dinitrobenzoic acid
2,4,6-Trinitrobenzoic acid
1,3,5-Trinitrobenzene
2,4,6-Trinitrotoluene
2,4,6-Trinitro-m-xylene
Picric acid

Ethyl nitrite
iso-Amyl nitrite

Nitroso compounds and oximes
Nitrosomethyl urea
Diphenyl nitrosamine
sym-Nitroso-m-xylene
1-Nitroso-2-naphthol
2-Nitroso-1-naphthol

Dimethylglyoxime
Cyclohexanone oxime
Benzoin oxime
Camphor oxime
α-Pinene oxime
A'-Carene oxime

Cyano compounds
Acetonitrile
Ethyl cyanoacetate
Diphenyl cyanarsine

Benzyl cyanide
Benzonitrile
p-Tolunitril

Isothiocyanates and thio氰anates
Allyl isothiocyanate
Phenyl isothiocyanate

Methyl thiocyanate
Ethyl thiocyanate
iso-Propyl thiocyanate

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Hydrazines
*sym.-Dimethylhydrazine, 2HCl
Dimethylketazine
Hydrazobenzene
Phenyldrazidine
α-Benzyl-α-phenyldrazine
Tetraphenylhydrazine

Azo compounds
Azobenzene
Azoxybenzene
Benzene-azo-β-naphthol
Methyloorange
Orange G
Scarlet red
Sudan III

Diazocompounds
Bin-(diazooacetic acid)
Ethyl diazoethanoate
Phenylbenzoyldiazomethane

p-Diazobenzenesulfonic acid
o-Diazobenzoic acid perbromide
2,6-Dimethoxy-benzene-diazoniumsulfate

Triazo compounds
Triazobenzene
o-Triazobenzoic acid

Heterocyclic 5-member compounds
Pyrrrole
N-Phenylpyrrrole
2,4-Dimethylpyrrrole
2,5-Dimethylpyrrrole
2,4-Dimethyl-5-carbethoxypyrrrole
2,4-Dimethyl-3,5-dicarbethoxypyrrrole

Pyrrolidine
Indole
2-Methylindole
2-Phenyllindole
Isatin
Indigotin
Carbazole

Imidazole
Benzimidazole
3,5-Dicarbetoxy-4-hydroxy-pyrazole

3-Methyl-1-phenyl-5-pyrazolone
Antipyrine
Benzotriazole
Chloracetylbenzotriazole
Phenylmethyl-1,2,3-triazole carboxylic acid
Phenylmethyl-1,2,3-triazole
Cardiazol
2-Methylbenzothiazole
Heterocyclic 6-member compounds
Pyridine
2-Picoline
2,6-Lutidine
2,4,6-Collidine
Picolinic acid
Nicotinic acid
Iso-Nicotinic acid
a,a'-Dipyridyl
Quinoline
Quinaldine
6-Methylquinoline
Kynurenic acid
2-Phenyl-quinoline-4-carboxylic acid
Acridine
Uric acid
Thymine
Trichloropurine
Morpholine
Piperazine
Dihydroadiphensylpiperazine
Cyanuric acid
Tetrazine dicarboxylic acid

Mixed nitrogen compounds
Acetylcholine iodide
Alanine
Amygdalin
Arecoline bromide
Atropine sulfate
Bilirubin
Brucine
Coffeine
Cinchonine sulfate
Cocaine chloride
Codeine
L-Cysteine
Dihydrostreptomycin
3,5-Diiodotyrosine
Guainidine carbonate
Hematein
Heroin
Hexamethylene tetramine
Histidine
Hydroxyprone
Lactoflavin
Morphine
Narcotine
Penicillin G
Pilocarpine chloride
Spermidin phosphate
Sulfapyridine
Sulfathiazole
Synazur
Thacapozol
Chloro compounds
Methylene chloride
Chloroform
Carbon tetrachloride
Ethylene chloride
Trichloroethylene
Glycerol 1,3-dichlorohydrin

Acetyl chloride
Ethyl chlorocarbonate
Ethyl chloroacetate
Phenacyl chloride
Phosgene, 20% in toluene
Diphosgene

Chlorobenzene
p-Chlorotoluene
Hexachlorobenzene
Chloranil
Chlormaine T
Pyridinium chloride
Bromo compounds
Methyl bromide
Ethyl bromide
n-Propyl bromide
iso-Amyl bromide
Allyl bromide
Methylene bromide
Ethylene bromide
β-Butylene bromide
Bromoform
Bromoacetic acid
β-Bromopropionic acid
a,a'-Dibromoadipic acid
n-Propyl bromoacetate
n-Butyl bromoacetate
Bromobenzene

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A rapid scheme of micro elementary analysis by a modified Lassaigne method is presented. The scheme covers the elements As, Br, C, Cl, F, I, N, P, and S.

A quantity of 1—5 mg of the organic compound is combusted at 500—590° with sodium in a sealed ampoule of pyrex glass. The ampoules, containing a piece of sodium, and the reagents are made so durable and stable that an analysis can be started without noteworthy preparations.

Positive results were obtained for all the substances investigated, including 1) easily volatile compounds, 2) polynitro, azo and diazo compounds, and 3) heterocyclic compounds. Diazocompounds gave the weakest reactions.

Each element can be detected in the presence of all the other elements, with the exception of phosphorus, which cannot be detected by this scheme, when arsenic is present.

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