

Systematic Micro Detection of Arsenic, Bromine, Carbon, Chlorine, Fluorine, Iodine, Nitrogen, Phosphorus, and Sulfur in Organic Compounds, by a Modified Lassaigne's Method

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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. Emich¹ 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 Niemann² 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 Widmark³, 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. Lassaigne⁴ 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 Sodium has sometimes been replaced by potassium, which has a lower melting point and is more reactive. Castellana⁵ used a mixture of magnesium and potash, with which the reaction is intensified if sugar⁶ is also added. The risk of a reaction between magnesium and nitrogen from the air cannot, however, be neglected⁷.

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 AsH_3 and PH_3 escape. The gases pass AgNO_3 -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 H_2S 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 $\frac{1}{2}$ 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

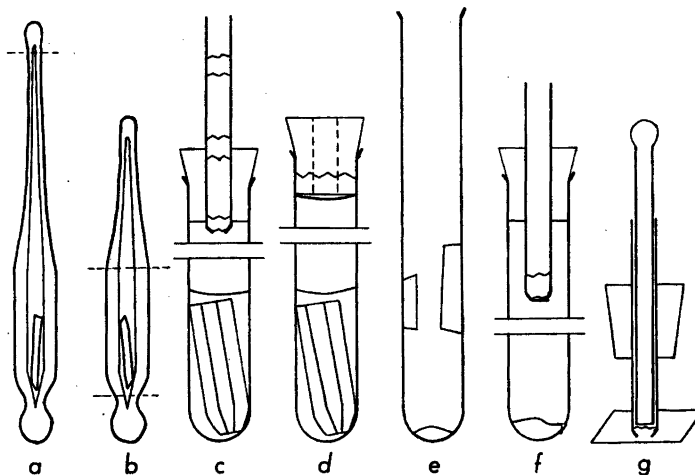


Fig. 1. a) Ampoule with sodium. b) Charged ampoule. c) Alkaline solution, with AgNO_3 - PbAc_2 -gel tube. d) Acid solution, with alkaline FeSO_4 -paper. e) Silver halide precipitate, with fluorescein and starch papers. f) Fluorine test with moist glass wool. g) Fluorine test upon Zr-alizarin paper.

drops of FeSO_4 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 $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ 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.

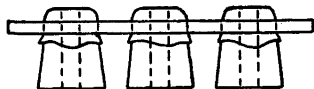


Fig. 2. a) Polystyrene plate ($60 \times 25 \times 2$ mm) with holes (diameter 8.5 mm) for bored stoppers with alkaline ferrous sulfate papers.

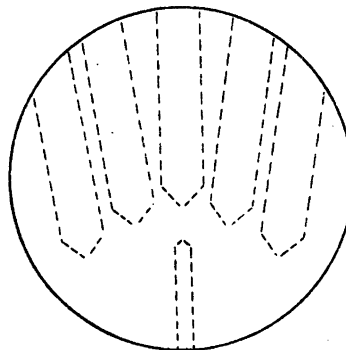


Fig. 2. b) Iron furnace (diameter 125 mm, height 50 mm) covered with 2 mm asbestos, 5 holes (diameter 15 mm, depth 55 mm) for asbestos tubes, one hole (diameter 7 mm, depth 50 mm) for a thermo-couple.

Table 1. Equipment box for students' laboratories.

Bored stoppers	Sodium ampoules	Fluorescein papers	Zirconium alizarin papers	Round papers
Rubber plugs	Asbestos tubes Polystyrene plate Stainless wire Glass rod for fluorine test	Starch papers	Gel tubes	Fluorine tubes
	Emich tubes File		Phosphate papers	Div.

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 $\text{PbAc}_2 \cdot 3\text{H}_2\text{O}$ 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 × 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.

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 ½ 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

* 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.

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* NH_3 . 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 NH_3 -treated precipitate in the test tube. It is essential that the walls also are completely dry. Mix the precipitate with a very little pulverized PbO_2 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 $\frac{1}{2}$ 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 As_2O_3 (more than 10 mg) is com-

busted, the amount of sodium does not suffice to form Na_3As ; 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 hexachlorobenzene 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 AgNO_3 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. — PCl_3 (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 Zimmermann⁸ 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 Na_2S 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

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_2 , NaNO_3 and $\text{Pb}(\text{NO}_3)_2$ gives a spot of Prussian blue equal to that from about $10 \mu\text{g N}$ as NaCN .

Nitrobenzene reacts exothermically with sodium, forming cyanide even on heating to only 100° . A weak cyanide reaction is obtained when $5 \mu\text{l}$ 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 $0.3 \mu\text{g N}$ gives, on boiling with sulfuric acid, very faint blue spots on the paper. With $10 \mu\text{g}$ the whole area is coloured blue and with large amounts the paper becomes blue-black. The colour reaction from 0.5 ml solution containing $10 \mu\text{g CN}$ is not decreased by 5 mg NaF , NaCl , NaBr , NaI , NaNO_3 or NaH_2PO_2 . 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 $10 \mu\text{g}$ cyanide one test out of three failed. With higher cyanide content a colour was always obtained.

Detection of carbon (sensitivity ca. $200 \mu\text{g C}$). 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 ($5 \mu\text{l}$ 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 chloropicrin give visible precipitates after centrifuging. NaCN and NaCNS do not give precipitates of carbon.

Salts of some metals, *e. g.* SbOCl , Bi_2O_3 , SnCl_4 and PbSO_4 and excessive amounts of As_2O_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 conc. nitric acid: carbon tetrachloride, chloroform, chloropicrin, dichlorodifluoromethane, imidazole, nitromethane, sodium acid carbonate, sodium carbonate, sodium oxalate, tetrazine dicarboxylic acid, trifluorochloromethane, trifluorotrichloroethane.

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 (Bennett²) 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 SiF_4) 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 , Na_2CO_3 , Na_2SO_3 , Na_2S , NaCl , and $\text{Na}_2\text{C}_2\text{O}_4$. 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 PBr_3 , and CHBr_3 brown fumes of bromine are evolved 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 zirconium-alizarin paper, is advantageous in preventing students' errors.

SUBSTANCES TESTED

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

Arsenic compounds

Arsanilic acid
 Arsenoacetic acid
 Arsphenamine
 Diphenyl chloroarsine
 Diphenyl cyanarsine
 Ethyl dichloroarsine
p-Hydroxyphenylarsonic acid
 "Lewisite"
 "Marpharside"
 Neoarsphenamide
 3-Nitro-4-hydroxyphenylarsonic acid
 Phenylarsonic acid
 Sodium arsonoacetate

Phosphorus compounds

Codeine phosphate
 Diethoxychloro phosphate
 Dimethylaminodichloro phosphate
 Dimethylaminodiethoxy phosphate
 Ethoxydichloro phosphate
 Phenylphosphorus oxychloride
 Phosphorus oxychloride
 Phosphorus tribromide
 Phosphorus trichloride
 Sodium glucose phosphate
 Sodium glycerol phosphate
 Sodium phenyl phosphate
 Spermidin phosphate
 Trieresyl phosphate
 Triphenyl phosphate

DFP

Mintakol
 Tabun
 TEPP
 Sarin

Sulfur compounds

Bis-(*a*-chloroethyl) sulfide
 Carbon disulfide
 Diphenyl sulfide
 Diphenyl thiourea
 2-Thiobarbituric acid
 Thioglycolic acid
 2-Thioisalicylic acid
 Thiourea
 Trithiane

Dimethyl sulfate
 Diethyl sulfate
 Potassium ethyl sulfate
 Methanesulfonic acid
 Trional

Benzenesulfinic acid
 Benzenesulfonic acid
p-Toluenesulfonic acid
 Benzenesulfonyl chloride
p-Toluenesulfonyl chloride
 3,4-Dichlorobenzenesulfonyl chloride

Methyl menthylxanthate
 Saccharin
 Sulfapyridine
 Sulfathiazole
 Thionyl chloride

Thiophene
 2-Methylthiophene
 2,5-Dimethylthiophene

Amines

Methylamine, 6% in benzene
 Methylamine, 30% in water
n-Propylamine
iso-Butylamine
iso-Amylamine
 Diethylamine
 1,3-Diamino-2-propanol
 Tri-*n*-butylamine
 Tri-*n*-amylamine
 Triethanolamine
 Tetramethylammonium iodide

Cyclohexylamine

Aniline
o-, *m*-, *p*-Chloroaniline
o-, *m*-, *p*-Bromoaniline
 Mesidine
a-Naphthylamine
vic-Xylidine

Diphenylamine
n-Dimethylaniline
 Triphenylamine

2-Aminopyrimidine
 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*

Chloropicrin
 Ethyl nitrate
 Nitromethane
 Tetranitromethane
 Nitroglycerin, 10% in ethanol

Nitrobenzene
 Nitroanthrone
 3-Nitrophtalic 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
 Diphenylnitrosamine
sym-Nitroso-*m*-xylene
 1-Nitroso-2-naphthol
 2-Nitroso-1-naphthol

Dimethylglyoxime
 Cyclohexanone oxime
 Benzoin oxime
 Camphor oxime
a-Pinene oxime
 1³-Carene oxime

Cyano compounds

Acetonitrile
 Ethyl cyanoacetate
 Diphenyl cyanarsine

Benzyl cyanide
 Benzonitrile
p-Tolunitrile

Isothiocyanates and thiocyanates

Allyl isothiocyanate
 Phenyl isothiocyanate

Methyl thiocyanate
 Ethyl thiocyanate
iso-Propyl thiocyanate

<i>Hydrazines</i>	3-Methyl-1-phenyl-5-pyrazolone	L-Cysteine
<i>sym</i> -Dimethylhydrazine, 2HCl	Antipyrine	Dihydrostreptomycine
Dimethylketazine	Benzotriazole	3,5-Diiodotyrosine
Hydrazobenzene	Chloracetylbenzotriazole	Guanidine carbonate
Phenylhydrazine	Phenylmethyl-1,2,3-triazole	Hematein
<i>α</i> -Benzyl- <i>α</i> -phenylhydrazine	Phenylmethyl-1,2,3-triazole	Heroin
Tetraphenylhydrazine	Cardiazol	Hexamethylene tetramine
<i>Azo compounds</i>	2-Methylbenzothiazole	Histidine
Azobenzene	<i>Heterocyclic 6-member compounds</i>	Hydroxyproline
Azoxybenzene	Pyridine	Lactoflavin
Benzene-azo- β -naphthol	2-Picoline	Morphine
Methylorange	2,6-Lutidine	Narcotine
Orange G	2,4,6-Collidine	Penicillin G
Scarlet red	Picolinic acid	Pilocarpine chloride
Sudan III	Nicotinic acid	Spermidin phosphate
<i>Diazo compounds</i>	<i>iso</i> -Nicotinic acid	Sulfapyridine
Bis-(diazocetic acid)	<i>α,α'</i> -Dipyridyl	Sulfathiazole
Ethyl diazoethanoate	Quinoline	Synazur
Phenylbenzoyldiazomethane	Quinaldine	Thacapzol
<i>p</i> -Diazobenzenesulfonic acid	6-Methylquinoline	<i>Chloro compounds</i>
<i>o</i> -Diazobenzoic acid perbromide	Kynurenic acid	Methylene chloride
2,6-Dimethoxy-benzene-diazoniumsulfate	2-Phenyl-quinoline-4-carboxylic acid	Chloroform
<i>Triazo compounds</i>	Acridine	Carbon tetrachloride
Triazobenzene	Uric acid	Ethylene chloride
<i>o</i> -Triazobenzoic acid	Uracil	Trichloroethylene
<i>Heterocyclic 5-member compounds</i>	Thymine	Glycerol 1,3-dichlorohydrin
Pyrrole	Trichloropurine	Acetyl chloride
<i>N</i> -Phenylpyrrole	Morpholine	Ethyl chlorocarbonate
2,4-Dimethylpyrrole	Piperazine	Ethyl chloroacetate
2,5-Dimethylpyrrole	Dihydrodiphenylpiperazine	Phenacyl chloride
2,4-Dimethyl-5-carbethoxy-pyrrole	Cyanuric acid	Phosgene, 20 % in toluene
2,4-Dimethyl-3,5-dicarbethoxy-pyrrole	Tetrazine dicarboxylic acid	Diphosgene
Pyrrolidine	Thiodiphenylamine	Chlorobenzene
Indole	<i>Mixed nitrogen compounds</i>	<i>p</i> -Chlorotoluene
2-Methylindole	Acetylcholine iodide	Hexachlorobenzene
2-Phenylindole	Alanine	Chloranil
Isatin	Amygdalin	Chloramine T
Indigotin	Arecoline bromide	Pyridinium. chloride
Carbazole	Atropine sulfate	<i>Bromo compounds</i>
Imidazole	Bilirubin	Methyl bromide
Benzimidazole	Brucine	Ethyl bromide
3,5-Dicarbetoxy-4-hydroxy-pyrazole	Coffeine	<i>n</i> -Propyl bromide
	Cinchonine sulfate	<i>iso</i> -Amyl bromide
	Cocaine chloride	Allyl bromide
	Codeine	Methylene bromide
		Ethylene bromide
		β -Butylene bromide
		Bromoform
		Bromoacetic acid
		β -Bromopropionic acid
		<i>α,α'</i> -Dibromoadipic acid
		<i>n</i> -Propyl bromoacetate
		<i>n</i> -Butyl bromoacetate
		Bromobenzene

<i>o</i> -, <i>m</i> - and <i>p</i> -Bromotoluene	Iodosobenzoic acid	<i>Fluoro compounds</i>
9,10-Dibromo anthracene	Iodoxybenzene	Dichloro difluoromethane
Eosin	<i>p</i> -Iodoaniline	Trichlorofluoromethane
	2-Iodo-5-methylaniline	Trichlorotrifluoroethane
		Trifluoroacetic acid
<i>Iodo compounds</i>		Fluorobenzene
Methyl iodide	3,5-Diiodo-L-tyrosine	<i>p</i> -Fluorobenzoic acid
Ethyl iodide	2,6-Diiodosozolic acid	Ethyl <i>p</i> -fluorobenzoate
Iodoform	Erythrosin	<i>m</i> -Fluoronitrobenzene
Iodobenzene	Synacyr	
Iodobenzene dichloride	Tetramethylammonium	DFP
Iodosobenzene	iodide	Sarin

SUMMARY

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. Diazo compounds 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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