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SELECTION OF EQUIPMENT FOR LABORATORIES MONITORING POLLUTION IN THE TANNING INDUSTRY

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INTERREGIONAL

Technical report: Manual on laboratory equipment and reagents**

Prepared for Member States by the
United Nations Industrial Development Organization

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1. INTRODUCTION

Animal hide is transformed into leather in a succession of many complex stages employing chemicals as well as apparatus and equipment of varying degrees of sophistication. This processing makes it possible to remove from the hide those constituents that are inconsistent with good-quality leather. These various organic and inorganic wastes are obtained either in solid or pulp form or in liquid form, i.e. in suspension or in solution. The pollutants arising from the transformation process are considerable, and it is essential today to treat them using different techniques suited to the quantity and quality of the waste matter (see treatment diagrams in annex 4).

The purpose of this manual is to enable tanneries and groups of tanneries to evaluate better, and thus to treat more effectively, the pollution emanating from the different phases of leather production. To that end, the establishment of a waste-water monitoring laboratory geared to the size of the enterprise will lead to a correct assessment of pollutants in tanning liquor and effluent. The persons concerned will find in this manual the essential information for setting up a laboratory suited to their enterprise.

2. GENERAL OUTLINE OF LEATHER PRODUCTION

2.1 Beamhouse operations

The first stage of production consists of re-hydrating and washing the cured hide in order to restore its natural raw condition. For this purpose, wetting agents and antiseptics are added to the water used in the washing operation, and the waste solutions thus contain organic matter (blood and mud), inorganic chemicals (sodium chloride) and organic chemicals.

The next stage, i.e. unhairing and liming, is to remove the hair or wool and epidermis by chemical means and to break down certain elastic fibres in order to improve the suppleness of the leather. This highly polluting operation accounts for more than 50 per cent of a tannery's organic waste loadings. It uses highly toxic substances such as sodium sulphide or hydrosulphide mixed with lime. Soda, sodium carbonate, enzyme preparations or mercaptans (organic sulphides) may also be employed.

2.2 Tanyard operations

Following mechanical fleshing, a further bath treatment process takes place, namely deliming, to neutralize the alkalines. Deliming is often combined with the use of enzyme bates to hydrolyse certain elastic fibres in the hide. Ammonium chloride and ammonium sulphate and also weak organic acids are mainly used at this stage.

Pickling then acidifies the hide to prepare it for tanning. Inorganic acids (sulphuric, hydrochloric) or organic acids (formic, acetic) are used in a solution containing 100 g/l of sodium chloride.

The degreasing phase is generally reserved for sheepskins and pigskins. It uses organic solvents (white spirit, kerosene, monochlorobenzene and perchloroethylene), which are recovered as carefully as possible at the end of the operation. It also employs wetting agents, whose toxicity varies according to their composition. Where degreasing is carried out, the waste solutions will contain emulsified fats. Pigskin degreasing primarily employs sodium carbonate.

Tanning consists of stabilizing the hide by means of organic or inorganic reagents that block the hydrophilic parts of the collagen fibres, which are the main constituents of leather. Nowadays, over 85 per cent of leather world-wide is tanned using trivalent chromium oxide salts, nearly 10 per cent using vegetable extracts from trees or bark, and the remainder using inorganic substances (aluminium, zirconium, etc.) or organic substances (sulphonated phenols, acrylic resins, dialdehydes and oxidized oils). The waste solutions may contain not only the basic chromium sulphate used in tanning but also masking agents (sodium formate, phthalate and oxalate or salts of dicarboxylic acids), basification agents (soda, sodium carbonate and bicarbonate, or magnesium oxide) and antiseptics of widely varying toxicity.

In cases where tanning is carried out using vegetable extracts, the pickling solutions are less strong and the waste tan liquor is far more coloured. Nowadays, vegetable tanning techniques are becoming highly efficient in terms of time and quality, and various methods exist for minimizing the associated pollution.

2.3 Dressing operations

The first operations carried out following tanning are mechanical processes such as sammying, splitting, shaving and trimming (manual removal of waste). A further wet treatment cycle then takes place, comprising different operations to give the leather its final characteristics. The main chemicals used are as follows:

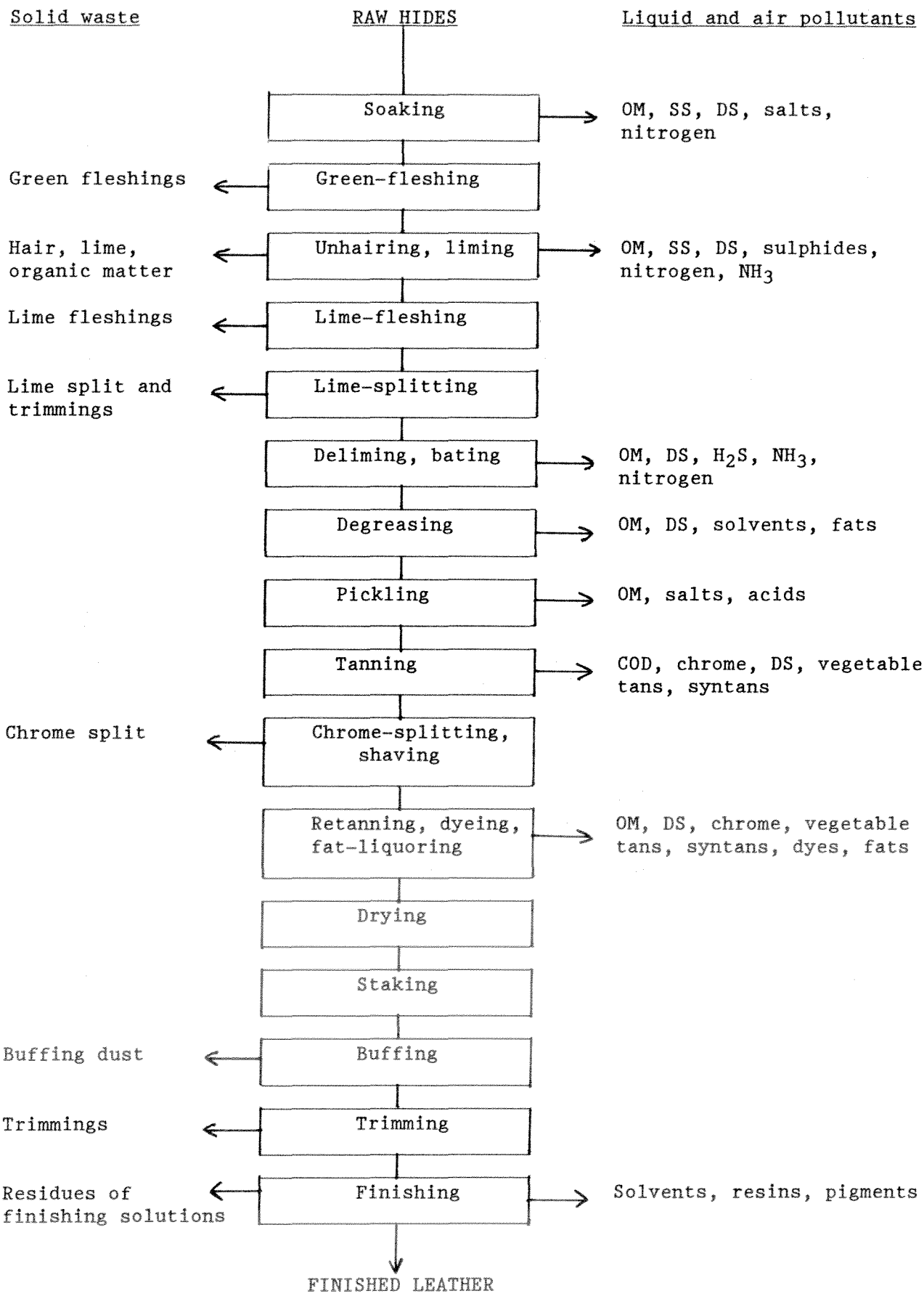
- Neutralization by means of sodium bicarbonate, sulphite, acetate or formate;
- Retanning using vegetable tans, synthetic tanning materials (syntans) or mineral tans having the same characteristics as those employed in tanning;
- Dyeing using acid, direct, base or metallized dyes and fixing products (formic acid, ammonia);
- Fat-liquoring using animal, vegetable, mineral or synthetic sulphonated oil emulsions.

2.4 Finishing operations

Following sammying, drying and other mechanical operations, the hides receive a surface application of chemicals to protect and embellish the leather. These chemicals consist of resins in a water phase (casein, acrylic or polyurethane resin) or solvent phase (nitrocellulose or urethane lacquer), in which pigments or dyes are dispersed.

These substances are applied by spray-gun, by roll-coating or curtain-coating machine or by brush. At this stage of production, the discharged waste is minimal and emanates from the spray-booth washing circuit and the mixing shop.

All the various production operations can be seen from the schematic diagram below:


Solid waste
RAW HIDES
Liquid and air pollutants
Soaking
OM, SS, DS, salts, nitrogen
Green fleshings
Green-fleshing
Hair, lime, organic matter
Unhairing, liming
OM, SS, DS, sulphides, nitrogen, NH3
Lime fleshings
Lime-fleshing
Lime split and trimmings
Lime-splitting
Deliming, bating
OM, DS, H2S, NH3, nitrogen
Degreasing
OM, DS, solvents, fats
Pickling
OM, salts, acids
Tanning
COD, chrome, DS, vegetable tans, syntans
Chrome split
Chrome-splitting, shaving
Retanning, dyeing, fat-liquoring
OM, DS, chrome, vegetable tans, syntans, dyes, fats
Drying
Staking
Buffing dust
Buffing
Trimmings
Trimming
Residues of finishing solutions
Finishing
Solvents, resins, pigments
FINISHED LEATHER

3. MAIN CHEMICALS USED

The quantities of chemicals used at each stage, per ton of salted wet hide, may be grouped together as indicated in the table below:

Process	Chemical	Maximum quantity per ton of hide	
Curing	Sodium chloride	30.0%	300 kg
	Antiseptics	0.3%	3 kg
Beamhouse	Wetting agents	0.3%	3 kg
	Antiseptics	0.2%	2 kg
	Sodium sulphide	4.0%	40 kg
	Sodium hydrosulphide	2.0%	20 kg
	Slaked lime	5.0%	50 kg
	Caustic soda	2.0%	20 kg
	Sodium carbonate	3.0%	30 kg
	Enzymes	1.5%	15 kg
	Mercaptans	4.0%	40 kg
	Ammonium chloride	2.0%	20 kg
	Ammonium sulphate	2.0%	20 kg
	Organic acids	2.0%	20 kg
Tanning	Sodium chloride	10.0%	100 kg
	Sulphuric acid	3.0%	30 kg
	Formic acid	2.0%	20 kg
	Organic solvents	16.0%	160 kg
	Wetting agents	4.0%	40 kg
	Sodium carbonate	2.0%	20 kg
	Chromium salts	10.0%	100 kg
	Sodium bicarbonate	1.0%	10 kg
	Vegetable tans	30.0%	300 kg
	Glutaraldehyde	2.0%	20 kg
Dressing	Neutralizing agents	2.0%	20 kg
	Retanning agents	4.0%	40 kg
	Dyes	4.0%	40 kg
	Fat-liquoring oils	12.0%	120 kg
Finishing	Finishing agents	4.0%	40 kg

For a proper correlation on the basis of treated hides/skins, the following values should be taken as wet weight references:

Lambskin:	1.5 kg per skin
Sheepskin:	3 kg per skin
Kidskin:	1 kg per skin
Goatskin:	2 kg per skin
Pigskin:	3 to 6 kg per skin
Calfskin:	12 to 16 kg per skin
Bovine hide:	20 to 45 kg per hide
Shaved bovine leather:	9 to 20 kg per hide

4. WASTE-WATER POLLUTION FLOWS OBTAINED

The various stages of leather production generate sizeable pollution flows, which can be grouped together according to the production processes and treated hide/skin types. The values given in these tables are average values.

4.1 Standard manufacture of bovine shoe-upper leather

(values obtained per ton of hide)

Parameter	Beamhouse		Tanning		Dressing and finishing		Total	
Water (m^3)	22		5		8		35	
	kg/t	mg/l	kg/t	mg/l	kg/t	mg/l	kg/t	mg/l
COD	150	6 820	10	2 000	60	7 500	220	6 290
BOD_5	60	2 730	4	800	16	2 000	80	2 290
SS	100	4 550	10	2 000	30	3 750	140	4 000
NaCl	250	11 400	80	16 000	-		330	9 430
S--	6	270	-		-		6	170
Cr^{3+}	-		3	600	1	125	4	110
Total N	7	320	4	800	1	125	12	340
Total P (kg)	-		-		0.1	13	0.1	3

4.2 Manufacture of bovine shoe-upper leather using clean technologies

(lime-liquor recycling, chromium exhaustion, CO_2 deliming)
(values obtained per ton of hide)

Parameter	Beamhouse		Tanning		Dressing and finishing		Total	
Water (m^3)	18 kg/t	mg/l	4 kg/t	mg/l	8 kg/t	mg/l	30 kg/t	mg/l
COD	110	6 110	10	2 500	60	7 500	180	6 000
BOD_5	45	2 500	4	1 000	16	2 000	65	2 170
SS	70	3 890	10	2 500	30	3 750	110	3 670
NaCl	250	13 900	80	20 000	-		330	11 000
S--	2	110	-		-		2	70
Cr^{3+}	-		1	250	1	125	2	70
Total N	5	280	1	250	1	125	7	230
Total P (kg)	-		-		0.1	13	0.1	3

4.3 Manufacture of vegetable-tanned sole leather using a dry-tanning drum process

(values obtained per ton of hide)

Parameter	Beamhouse		Tanning		Dressing and finishing		Total	
Water (m^3)	22 kg/t	mg/l	3 kg/t	mg/l	5 kg/t	mg/l	30 kg/t	mg/l
COD	150	6 820	70	23 300	40	8 000	260	8 670
BOD_5	60	2 730	15	5 000	10	2 000	85	2 830
SS	100	4 550	30	10 000	20	4 000	150	5 000
NaCl	250	11 400	-		-		250	8 330
S--	6	270	-		-		6	200
Cr^{3+}	-		-		-		-	
Total N	7	320	4	1 330	0.5	100	11.5	380
Total P (kg)	-		0.4	130	0.1	20	0.5	17

4.4 Standard manufacture of sheep nappa clothing leather with degreasing by emulsifying agent

(values obtained in grams per skin)

Parameter	Beamhouse		Tanning		Dressing and finishing		Total	
Water (litres)	150		30		40		220	
	g/s	mg/l	g/s	mg/l	g/s	mg/l	g/s	mg/l
COD	280	1 870	180	6 000	80	2 000	540	2 450
BOD_5	100	670	50	1 670	25	625	175	800
SS	260	1 730	30	1 000	10	250	300	1 360
NaCl	500	3 330	120	4 000	-		620	2 820
S--	7	50	-		-		7	30
Cr^{3+}	-		9	300	1	25	10	45
Total N	22	150	8	270	2	50	32	150
Total P	1.5	10	0.5	17	0.5	13	2.5	11

4.5 Manufacture of woolled sheepskin for clothing with degreasing by machine

(values obtained in grams per skin)

Parameter	Beamhouse		Tanning		Dressing and finishing		Total	
Water (litres)	250		80		170		500	
	g/s	mg/l	g/s	mg/l	g/s	mg/l	g/s	mg/l
COD	290	1 160	210	2 650	250	1 470	750	1 500
BOD_5	100	400	70	875	50	290	220	440
SS	210	840	30	375	10	60	250	500
NaCl	500	2 000	150	1 880	-		650	1 300
Cr^{3+}	-		15	190	5	30	20	40
Total N	14	60	4	50	2	12	20	40
Total P	5	20	0.5	6	0.5	3	6	12

5. LEGISLATION ON POLLUTANT DISCHARGES

Legislation on waste-water discharges is becoming increasingly strict. It is possible to compare the rules in different countries, and the tables below show the statutory limits for discharge into the environment (rivers, lakes and oceans) or into sewers terminating in a joint effluent purification plant.

5.1 Discharge into the environment

(values in mg/l except for pH)

Country	France	India (river)	India (ocean)	Brazil
pH	6.5-8.5 (9.5)	5.5-9	5.5-9	6-8.5
COD	125	250	250	450-160*
BOD_5	30	30	100	200-40*
SS	35	100	100	200-50*
Nitrogen (N)	30 (total)	100 (Kjl)	100 (Kjl)	10 (Kjl)
Phosphorus (P)	10	5	-	1
Chloride (Cl)	-	1 000	-	-
Sulphide (S)		2	5	0.2
Sulphate (SO_4)	-	1 000	1 000	-
Chromium (Cr^{3+})	1.5	2	2	0.5
Chromium (Cr^{6+})	0.1	0.1	1	0.1
Aluminium (Al)	5	-	-	10
Phenol	0.1	1	5	0.1
Dissolved salts	-	2 100	-	-
Oil and grease	-	10	20	30
Hydrocarbons	10	-	-	-
AOX	5	-	-	-

* Depending on flow rate.

5.2 Discharge into sewers

(values in mg/l except for pH)

Country	France	India	Argentina	Tunisia
pH	6.5-8.5 (9.5)	5.5-9	5.5-10	6.5-9
COD	2 000	-	-	1 000-2 000
BOD_5	800	350	200	400-1 000
SS	600	600	-	400
Nitrogen (N)	150 (total)	- (Kjl)	- (Kjl)	100 (Kjl)
Phosphorus (P)	50	-	-	-
Chloride (Cl)	-	1 000	-	700-2 000
Sulphide (S)	2	-	1	3
Sulphate (SO_4)	-	1 000	-	400-600
Chromium (Cr^{3+})	1.5	2	0.5	2-4
Chromium (Cr^{6+})	0.1	2	-	-
Aluminium (Al)	5	-	5	10-20
Phenol	0.1	5	0.5	-
Dissolved salts	-	2 100	-	-
Oil and grease	-	20	100	-
Hydrocarbons	10	-	-	10-20
AOX	5	-	-	-

NB: The highest values for Tunisia are maximum limits that must not be exceeded under any circumstances.

5.3 Discharge values in the United States of America

Discharge values are laid down for nine tannery categories:

- A. Hair-pulp chrome-tanning;
- B. Hair-save chrome-tanning;
- C. Vegetable tanning;
- D. Production starting from wet-blue leather;
- E. Production starting from pickled hides;
- F. Production of wet-blue;
- G. Shearlings;
- H. Pigskins;
- I. Production of splits.

These values are shown in the table below:

Category	A	B	C	D	E	F	G	H	I
Sewer discharge									
Sulphide mg/l	24	24	24	-	-	24	-	24	-
Total chrome mg/l	12/8	12/8	12/8	19/12	19/12	12/8	19/12	12/8	19/12
pH	7.0-10.0	7.0-10.0	>=7.0	6.0-10.0	6.0-10.0	7.0-10.0	6.0-10.0	7.0-10.0	6.0-10.0
Direct discharge Plants prior to 1985									
BOD_5 kg/ton	9.3/4.2	8.2/3.7	6.7/3.0	8.9/4.0	8.0/3.6	3.2/1.5	15.0/6.8	7.0/3.2	5.8/2.6
SS kg/ton	13.4/6.1	11.8/5.4	9.7/4.4	12.8/5.8	11.6/5.3	4.7/2.1	21.7/9.9	10.1/4.6	8.3/3.8
Fats kg/ton	3.9/1.7	3.4/1.5	2.8/1.3	3.7/1.7	3.4/1.5	1.4/0.61	5.3/2.8	3.0/1.3	2.4/1.1
Total chrome kg/ton	0.24/0.09	0.21/0.08	0.17/0.06	0.23/0.08	0.21/0.08	0.08/0.03	0.39/0.14	0.18/0.07	0.15/0.05
pH	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0
Plants after 1985									
BOD_5 kg/ton	6.0/2.7	6.9/3.1	5.9/2.7	6.5/2.9	5.3/2.4	3.0/1.3	13.2/5.9	5.8/2.6	3.5/1.6
SS kg/ton	8.7/4.0	9.9/4.5	8.5/3.9	9.3/4.3	7.7/3.5	4.3/1.9	19.1/8.7	8.3/3.8	5.1/2.3
Fats kg/ton	2.5/1.1	2.9/1.3	2.4/1.1	2.7/1.2	2.2/1.0	1.2/0.55	5.6/2.5	2.4/1.1	1.5/0.66
Total chrome kg/ton	0.16/0.06	0.18/0.06	0.15/0.06	0.17/0.06	0.14/0.05	0.08/0.03	0.34/0.12	0.15/0.05	0.09/0.03
pH	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0	6.0-9.0

6. METHODS OF ANALYSING WASTE WATER

To monitor and check the satisfactory operation of a purification plant, it is essential to carry out water analyses at the inlet and outlet points of the treatment installations, since visual assessment alone is not sufficient to determine the effectiveness of the various treatment stages. While the outlet analytical values provide evidence for the authorities of the quality of effluent discharged into the environment, the values obtained at the plant's inlet show the effectiveness of the clean technologies installed at the tannery and make it possible to evaluate purification performance.

Most of the analytical methods presented in this document are taken from the eighteenth edition of Standard Methods for the Examination of Water and Wastewater published by A.P.H.A., A.W.W.A, and W.E.F. (Water Environment Federation), American Public Health Association, 1015 15th Street NW, Washington, DC 2005, U.S.A.. Other methods have been adapted for tannery effluent analysis by the Centre Technique Cuir Chaussure Maroquinerie (CTC) and are described in the manual Techniques d'analyse des eaux résiduaires industrielles published by CTC, 4 rue Hermann Frenkel, 69367 Lyon, France.

6.1 Sampling methods

It is indispensable to obtain representative samples of the element to be analysed. The sampling bottles must be clean and rinsed with the water that is to be examined.

- Grab sampling

This is the most commonly used sampling technique, by which it is possible to determine under satisfactory conditions the analytical parameters of a solution that contains no suspended solids. However, it provides only limited data if the sampled discharge is likely to vary in quality and quantity over time.

- Composite sampling

If an average value over two or 24 hours is sought, it is necessary to undertake sampling proportional to the flow rate by means of an automatic sampling apparatus. It is often possible to ascertain the variations in effluent characteristics during the day by analysing the different hourly-sampled segments. Samples must be kept at 4° C to prevent their undergoing changes. Nevertheless, despite these precautions, the characteristics of certain parameters begin to change after 24 hours' storage. Promptness in commencing the analyses is thus a guarantee of their accuracy.

6.2 Measuring pH

The pH measurement indicates whether a solution is acidic (pH values between 0 and 7) or alkaline (pH values between 7 and 14). It is possible in some cases to use indicator paper, but such measurement is inaccurate and liable to risks of interference (light, suspended solids, chlorinated products).

The electrochemical method is the most commonly employed and the most accurate. It involves immersing a glass electrode and a reference electrode (calomel) into the solution to be analysed. The difference in potential between the two electrodes is directly related to the pH of the solution.

This procedure requires a measuring apparatus, i.e. a pH-meter with a glass electrode and a calomel (KCl) electrode, and also glass beakers, a stirrer and comparison solutions with a pH of 4, 7 and 10.

6.3 Measuring effluent settleability

The volume of settleable solids present in waste water can be checked by putting the water sample into a graduated measuring cylinder (preferably conical). It is then possible to monitor the effluent settling rate by noting the volume of sludge formed over time. The settleable matter is the matter deposited during a period conventionally fixed at two hours.

The only equipment required for this determination is an Imhoff cone or a one-litre measuring jar.

6.4 Measuring chemical oxygen demand (COD) (standard method 5220 C)

COD is the quantity of oxygen consumed by organic and inorganic matter susceptible to oxidation in defined conditions:

- Presence of an excess of potassium bichromate;
- Concentrated sulphuric acid medium;
- Boiling for two hours;
- Catalysts: sulphate of mercury and sulphate of silver.

The excess bichromate is titrated with a solution of Mohr's salt (iron II and ammonium sulphate).

This measuring operation requires the use of the following equipment:

- One precision balance (capable of weighing to 1/10 mg);
- Six 500-ml flasks with ground-glass necks;
- Six water or air coolers;
- Precision pipettes;
- One 25-ml precision burette;
- Six electric flask heaters.

The quantitative analysis requires the following chemical reagents:

- Mercury II sulphate, $HgSO_4$, in powder form;
- Silver sulphate, Ag_2SO_4, in powder form;
- Concentrated sulphuric acid (d 20°: 1.83);
- Mohr's salt, $FeSO_4.(NH_4)_2SO_4.6H_2O$;
- Potassium bichromate, $K_2Cr_2O_7$;

- 1,1 o-phenanthroline;

- Ferrous sulphate, $FeSO_4.7H_2O$.

6.5 Chromium analysis

Chromium occurs in two stable oxidation states: one hexavalent and the other trivalent. (In solution, trivalent chromium is green and hexavalent chromium is orange.)

It is possible to determine the hexavalent chromium content by analysis using the colorimetric method or the Mohr's salt method, and the total chromium content following conversion to hexavalent chromium by oxidation. The trivalent chromium content is calculated by taking the difference. The detection limit is approximately 25 mg/l.

Oxidation to hexavalent chromium is obtained, at boiling heat, by a mixture of sulphuric, perchloric and nitric acids. Three main analytical methods can then be used:

- Colorimetric analysis using diphenylcarbazide in a slightly acidic medium, which produces a pink-violet coloration that can be measured at 540 nm (standard method 3500-Cr D);

- Analysis by reduction of chromium VI to chromium III using Mohr's salt (or double iron and ammonium sulphate) in the presence of ferroin (standard method for COD);

- Analysis by the iodometric method using potassium iodide, the liberated iodine being titrated with sodium thiosulphate.

For measuring low concentrations, it is necessary to use the atomic absorption spectrometric method, by which it is possible to detect a minimum chromium concentration of 50 ug/l. In view of the cost of the apparatus, that method will not be described or evaluated in this manual.

The equipment required for the standard analytical procedures is as follows:

For the oxidation phase:

- One gas or electric heater;

- One precision balance capable of weighing to 1/10 mg;

- Precision pipettes;

- Six 250-ml volumetric flasks;

- Six 250-ml Erlenmeyer flasks;

- Glass beads.

For colorimetric analysis:

- One spectrocolorimeter;

- Six 50-ml volumetric flasks;
- Precision pipettes;
- One filter-holder with paper filters.

For analysis using Mohr's salt:

- One 25-ml precision burette;
- Precision pipettes;
- Six 250-ml beakers.

For iodometric analysis:

- One 25-ml precision burette;
- Six 250-ml Erlenmeyer flasks;
- Precision pipettes.

The chemical reagents required for the oxidation phase and the different analytical procedures are as follows:

- Oxidation:
  - Perchloric acid, $HClO_4$ (d 20°: 1.615);
  - Sulphuric acid, H_2SO_4 (d 20°: 1.83);
  - Nitric acid, HNO_3 (d 20°: 1.33);
- Spectrocolorimetric analysis:
  - Diphenylcarbazide;
  - Phthalic anhydride;
  - Ethyl alcohol, 95°;
  - Potassium chromate, K_2CrO_4, for calibration of the colorimeter.
- Analysis using Mohr's salt:
  - Concentrated sulphuric acid (d 20°: 1.83);
  - Mohr's salt, $FeSO_4.(NH_4)_2SO_4.6H_2O$;
  - Potassium bichromate, $K_2Cr_2O_7$;
  - 1,1 o-phenanthroline;
  - Ferrous sulphate, $FeSO_4.7H_2O$.

- Iodometric analysis:
  - Phosphoric acid, H_3PO_4 (d 20°: 1.71);
  - Potassium iodide, KI;
  - Thiodene in powder form;
  - Sodium thiosulphate, $Na_2S_2O_3.5H_2O$;
  - Potassium bichromate, $K_2Cr_2O_7$.

6.6 Suspended solids (standard method 2540 D)

Essentially, two laboratory methods are used:

- Vacuum filtration through a glass-fibre filter;
- Centrifugation in the case of highly-clogging samples.

After weighing, the filter disk is placed on a vacuum filtering support, and a specific quantity of liquid is filtered. The filter is dried at 105° C and weighed.

In cases where filtration is difficult, the sample is centrifuged, the supernatant is removed, and the residue is dried at 105° C and then weighed.

The equipment required for measuring suspended solids by filtration is as follows:

- One glass filter pump or vacuum pump;
- One vacuum filtration apparatus (one-litre flask, support and joint);
- 100 calibrated glass-fibre filters;
- Ten silica dishes;
- One oven for operation at 100 to 105° C;
- One precision balance capable of weighing to 1/10 mg;
- Precision pipettes;
- One desiccator.

If the inorganic substances present in the suspended solids are also to be determined, a furnace is necessary:

- One muffle furnace that can be heated to between 600 and 650° C.

To measure suspended solids by centrifugation, a centrifuge is also required:

- One centrifuge capable of an average acceleration of 3,000 rpm and equipped with bowls of at least 200 ml (if possible 500 ml).

6.7 Total solids (dry solids) (standard method 2540 B)

In this operation, the total dry solids present in a sample for analysis are calculated by evaporation. At 100-105° C, only water and some organic solvents are removed. At 600-650° C, solely the inorganic substances remain, although some are partly decomposed.

The apparatus required is as follows:

- Ten silica dishes;
- One oven for operation at 100 to 105° C;
- One precision balance capable of weighing to 1/10 mg;
- Laboratory glassware (precision pipettes, desiccator);

and, if applicable, for inorganic substances:

- One muffle furnace that can be heated to between 600 and 650° C.

6.8 Sodium sulphide analysis

In concentrations of 4 mg/l or higher, sodium sulphide is extremely toxic to living organisms. Two measuring methods are possible:

- An electrochemical method based on the use of a selective electrode, which is highly reliable but requires the use of special equipment;
- Volumetric analysis using potassium ferricyanide.

- Potentiometric method (CTC method - Rodier techniques)

The sulphides are converted to sulphide of silver by the addition of silver nitrate. The variation in potential at the end-point is recorded by the apparatus, with a detection limit of 2 mg/l.

The analysis equipment required is as follows:

- One potentiometric analysis apparatus;
- One calomel electrode with K_2SO_4 filler;
- One sulphide-selective electrode;
- Precision pipettes;
- One 50-ml measuring cylinder;
- 250-ml beakers;
- One magnetic stirrer.

The chemical reagents used for the analysis are as follows:

- Silver nitrate, $AgNO_3$;

- Ammonia, NH_4OH (d 20°: 0.9);
- Ammonium chloride, NH_4Cl;
- 1,2 cyclohexylenediamine tetraacetic acid (CDTA).

- Volumetric method

The sulphides are analysed by means of potassium ferricyanide in the presence of a ferrous dimethylglyoxime ammonia complex. They are oxidized in sulphur, and the sulphites, which may cause interference, are precipitated with barium chloride. The method detection limit is 8 mg/l.

The analysis equipment required is as follows:

- One 25-ml burette;
- One magnetic stirrer;
- One 250-ml beaker;
- Precision pipettes;
- One precision balance capable of weighing to 1/10 mg.

The chemical reagents used for the analysis are as follows:

- Potassium ferricyanide, $K_3Fe(CN)_6$;
- Ammonium chloride, NH_4Cl;
- Ammonia, NH_4OH (d 20°: 0.9);
- Barium chloride, $BaCl_2$;
- Iron II sulphate, $FeSO_4$;
- Ethanol, 95%;
- Dimethylglyoxime;
- Sulphuric acid, H_2SO_4 (d 20°: 1.84).

6.9 Dissolved oxygen

Levels of dissolved oxygen are related to biological activity in water. The respiratory activities of water fauna and flora require sizeable quantities of it. Certain physico-chemical parameters also condition the dissolved oxygen content (temperature, atmospheric pressure, salinity, etc.).

There are two determination techniques:

- Volumetric analysis based on the oxidizing property of dissolved oxygen: the Winkler method (standard method 4500-O B);
- Electrochemical analysis using a polarographic electrode (standard method 4500-O G)

Since the volumetric method is a delicate process that is liable to interference and involves a lengthy measuring operation, only the electrochemical method will be included.

The measuring apparatus required is as follows:

- One magnetic stirrer;
- One polarographic measuring probe;
- One thermometer;
- One oxygen analyser graduated in ppm, mg/l or % oxygen;
- Twenty 100-ml bottles.

6.10 Mohlman index (standard method 2710 D)

The Mohlman index shows the volume in ml occupied by 1 g of suspended solids after settling for 30 minutes. It enables the efficiency of biological treatment to be checked. It is directly related to the settleability of biological sludge.

The measuring apparatus required is as follows:

- One oven regulated at 105° C;
- One Imhoff cone or 1-litre measuring jar;
- Five silica dishes;
- One precision balance capable of weighing to 1/10 mg;
- 50-ml precision pipettes.

6.11 Total Kjeldahl nitrogen (TKN) analysis (standard method 4500-Norg B & 4500-Norg C)

Total Kjeldahl nitrogen corresponds to the sum of ammonia nitrogen and organic nitrogen. If the oxidized forms of nitrogen - nitrites and nitrates - are added, the total nitrogen is obtained.

By oxidative digestion in an acidic medium, organic nitrogen (protein, peptides, amino acids, etc.) is converted to ammonia nitrogen without any degradation of the oxidized compounds of nitrogen (nitrites, nitrates, hydrazine, oximes, etc.). The ammonia is then displaced by distillation in an alkaline medium and analysed by acidimetry. The method detection limit is approximately 2 mg/l.

The analysis equipment required is as follows:

- One distillation apparatus comprising:
  - . 1,000-ml distilling flasks;
  - . One ground-glass reducing joint;
  - . One three-way ground-glass adaptor;

. One separating funnel;

. One straight-sided condenser;

. One curved extension piece;

. One gas heater (Bunsen burner);

. Support rods;

. Clamps;

or an automatic distillation apparatus;

- One digestion rack (gas- or electrically-heated with hood);
- 500-ml Erlenmeyer flasks;
- One burette;
- One magnetic stirrer;
- Silicon grease.

The chemical reagents used for the analysis are as follows:

- Sulphuric acid, H_2SO_4 (d 20°: 1.84);
- Potassium sulphate, K_2SO_4;
- Selenium catalyst;
- Sodium hydroxide, NaOH;
- Boric acid, H_3BO_3;
- Methyl red;
- Bromocresol green;
- Ethanol, 95%.

6.12 Biochemical oxygen demand (BOD_5) (standard method 5210 B)

Five-day biochemical oxygen demand, or BOD_5, is the amount of oxygen consumed under test conditions (incubation for five days at 20° C in darkness) by certain substances present in water in the course of their biological degradation.

Despite its limitations, the BOD_5 test is the analytical technique that most faithfully mimics the metabolic action of organic pollutants in watercourses.

However, four phenomena may interfere with BOD_5:

- The presence of highly-reducing substances that account for a high oxygen demand during the first 10 hours (sulphides, sulphites, etc.);

- Photochemical activity, which leads to the production of oxygen within the sample itself and is inhibited by incubating the samples in the dark;

- The presence of toxins, which can inhibit biological reactions entirely or for a specific period;

- The action of bacteria in the nitrogen cycle (nitrification), which can alter the oxygen balance of the medium to a considerable degree, particularly in the final stage of the test.

The test procedure consists of preparing several dilutions of the sample using dilution water saturated with oxygen and seeded. The quantity of dissolved oxygen in each dilution is measured before and after incubation for five days at 20° C in the dark. Oxygen consumption should be between 40 and 60 per cent of the initial sample content.

It is also possible to use a manometric method, by which it is possible to monitor changes in oxygen consumption throughout the five-day period. The shape of the oxygen consumption curve plotted gives a good indication of the presence of any toxic substances in the analysis sample.

The equipment required for the manometric method consists of a multi-position respirometer, which gives a reading of the oxygen consumption in the sample by means of a manometer or by a continuous recording. The equipment must be heat-insulated at 20 $\pm$ 1° C or placed in an incubator thermostatically controlled at 20 $\pm$ 1° C.

The equipment required for dilution analysis of biochemical oxygen demand is as follows:

- One incubator thermoregulated at 20 $\pm$ 1° C;

- 250-ml incubation bottles with ground-glass stoppers;

- Precision pipettes;

- Volumetric flasks of the following volumes: 2,000, 1,000, 500, 250, 150 and 100 ml;

- Equipment for measuring dissolved oxygen by polarographic probe;

- An aeration device to saturate the dilution water.

The reagents required for measuring BOD_5 are as follows:

- Dihydrated sodium monohydrogenophosphate, $Na_2HPO_4.2H_2O$, or dodecahydrated sodium monohydrogenophosphate, $Na_2HPO_4.12H_2O$;

- Potassium dihydrogenophosphate, KH_2PO_4;

- Magnesium sulphate, $MgSO_4.7H_2O$;

- Calcium chloride, $CaCl_2$;

- Ferric chloride, $FeCl_3$;

- Ammonium chloride, NH_4Cl;
- Biological treatment sludge or sewage water for the seed source;
- Sulphuric acid, H_2SO_4 (d 20°: 1.84);
- Potassium bichromate, $K_2Cr_2O_7$.

6.13 Calcium analysis

Calcium is a predominant element of water hardness and may thus constitute a drawback in some leather processing operations, such as dyeing. It is also one of the components of unhairing and liming liquor. There are two methods of analysis by complexometry with disodium salt of ethylenediamine tetraacetic acid (EDTA):

- A volumetric method using a colour indicator specific to calcium;
- A potentiometric method, by which it is also possible to analyse magnesium in addition to calcium.

Calcium analysis by volumetry (Rodier techniques)

The calcium is complexed by a solution of EDTA in an alkaline medium (pH between 12 and 13) in the presence of a colour indicator (eriochrome blue). The detection limit is 5 mg/l.

The analysis equipment required is as follows:

- One 25-ml burette;
- One magnetic stirrer;
- Precision pipettes;
- 150-ml beakers;
- One precision balance capable of weighing to 1/10 mg.

The reagents required for the analysis are as follows:

- Sodium hydroxide NaOH;
- Eriochrome blue;
- EDTA.

Calcium analysis by potentiometry (standard method 3500-Ca D)

First, all the calcium and magnesium ions are analysed by complexometry with EDTA in a medium buffered to a pH of 10.2 $\pm$ 0.1. The magnesium ion is then eliminated as hydroxide in a medium buffered to a pH of 12 $\pm$ 0.1 and just the calcium is analysed by complexometry with EDTA. The reactions are monitored by potentiometry.

The analysis equipment required is as follows:

- One potentiometric titration apparatus;
- One calomel reference electrode;
- One mercury silver amalgam electrode;
- One magnetic stirrer;
- One precision balance capable of weighing to 1/10 mg;
- 150-ml beakers;
- Precision pipettes.

The reagents required for the analysis are as follows:

- Nitric acid, HNO_3;
- Purified mercury;
- Triethanolamine;
- Ethanolamine;
- EDTA;
- Mercury (II) sulphate, $HgCl_2$;
- Calcium carbonate, $CaCO_3$;
- Sodium hydroxide, NaOH.

6.14 Chloride analysis (standard method 4500-Cl^- D)

In a nitric acid medium, chlorides are converted to their silver salts in the presence of silver nitrate. For this analysis, a potentiometric method is used. The method detection limit is 5 mg/l.

The analysis equipment required is as follows:

- One potentiometric titration apparatus;
- One silver electrode;
- One reference electrode filled with saturated K_2SO_4;
- 250-ml beakers;
- One magnetic stirrer;
- Precision pipettes.

The reagents required for the analysis are as follows:

- Silver nitrate, $AgNO_3$;

- Concentrated nitric acid, HNO_3 (d 20°: 1.33);

- Hydrochloric acid, HCl;

- Sulphuric acid, H_2SO_4.

6.15 Phenol analysis (standard method 5530 D)

In an alkaline medium, a number of phenolic compounds react with aminoantipyrine in the presence of potassium ferricyanide to produce an orange-red coloration that can be analysed colorimetrically in a chloroform phase.

However, not all phenols react (in particular the para-substituted phenols). Some phenols produce different coloration intensities. For this reason, the term "phenol index" is used rather than phenol determination.

The analysis equipment required is as follows:

- One precision balance capable of weighing to 1/10 mg;

- One set of twelve 25-ml volumetric flasks;

- Four 100-ml volumetric flasks;

- Two 1,000-ml volumetric flasks;

- One pH-meter;

- One spectrocolorimeter;

- One set of twelve 250-ml separating funnels;

- One set of twelve 200-ml volumetric flasks;

- Precision pipettes;

- One set of twelve 150-ml beakers;

- One all-glass distillation apparatus with ground joints, comprising:

  . One 500-ml three-necked flask;

  . One thistle funnel to fit the flask;

  . One condenser;

  . Two curved extension pieces.

The reagents required for the analysis are as follows:

- Ammonium chloride, NH_4Cl;

- Double potassium and sodium tartrate, $KNaC_4H_4O_6.4H_2O$;

- Ammonia, NH_4OH;

- 4-aminoantipyrine (1-phenyl-2-3-dimethyl-4-amino-pyrazolone-5);
- Potassium ferricyanide, $K_3Fe(CN)_6$;
- Phosphoric acid, H_3PO_4;
- Chloroform, $CHCl_3$;
- Sodium chloride, NaCl;
- Phenol, C_6H_5OH.

6.16 Sulphate analysis (standard method 4500-SO_4^{--} E)

In a hydrochloric acid medium, sulphates precipitate in the presence of barium chloride. The barium sulphate precipitate is stabilized, by means of a solution of Tween 20, in order to allow nephelometric measuring by spectrometer. The method detection limit is between 1 and 2 mg/l.

The analysis equipment required is as follows:

- One spectrocolorimeter regulated at 650 nm;
- One set of twelve 50-ml volumetric flasks with stoppers;
- Precision pipettes;
- One precision balance capable of weighing to 1/10 mg;
- Two 1,000-ml volumetric flasks;
- Two 100-ml volumetric flasks.

The reagents required for the analysis are as follows:

- Sodium sulphate, Na_2SO_4;
- Hydrochloric acid, HCl;
- Tween 20;
- Stabilized barium chloride, $BaCl_2$.

6.17 Aluminium analysis

EDTA is used, there being a donor-acceptor bond between the metallic ion Al^{3+} and the lone nitrogen pair. The complex obtained with the aluminium is negatively charged.

Two methods are employed:

- A potentiometric titration method; and
- A manual volumetric method.

Potentiometric analysis (CTC method)

Since the reaction between EDTA and aluminium is very slow, direct titration cannot be carried out; reverse analysis is therefore undertaken. A precisely-measured excess quantity of EDTA is added, the solution is then adjusted to a pH of 4.5, and the reaction is accelerated by heating. The excess EDTA is then titrated by a titrated solution of zinc sulphate with a pH of between 5 and 6. The method detection limit is 25 mg/l.

The analysis apparatus required is as follows:

- A potentiometric analysis apparatus with a magnetic stirrer;
- Five 150-ml beakers;
- Precision pipettes;
- One heating rack;
- One precision balance capable of weighing to 1/10 mg;
- One calomel reference electrode;
- One mercury silver amalgam electrode.

The reagents required for the analysis are as follows:

- EDTA;
- Methyl red;
- Ethyl alcohol;
- Sodium hydroxide, NaOH;
- Hexamethylenetetramine;
- Xylenol orange;
- Zinc sulphate, $ZnSO_4$;
- Concentrated nitric acid, HNO_3;
- Ethanolamine;
- Mercury (II) sulphate, $HgSO_4$.

Manual volumetric analysis

The aluminium is analysed directly by EDTA, hot, in the presence of copper complexonate and a colour indicator.

The analysis equipment required is as follows:

- One precision balance capable of weighing to 1/10 mg;
- One 25-ml burette;

- One gas heater point;
- Five 250-ml wide-necked Erlenmeyer flasks;
- Precision pipettes;
- One magnetic stirrer.

The reagents required for the analysis are as follows:

- EDTA;
- Aluminium comparison solution;
- Pan-indicator;
- Copper sulphate, $CuSO_4$.

6.18 Iron analysis (standard method 3500-Fe D)

In a buffered medium, iron II reacts with phenanthroline to form a red complex that can be measured colorimetrically at 510 nm. The method detection limit is 0.01 mg/l. For total iron analysis, iron III has to be converted to iron II.

This analysis is subject to several interferences:

. Copper, cobalt, chromium and zinc interfere if present in concentrations equal to ten times that of the iron, and cyanides and nickel in concentrations equal to or higher than 2 mg/l. These interferences are avoided by working with a pH of between 3.5 and 5.5;

. Cadmium, mercury, bismuth and silver interfere by reacting with phenanthroline;

. Phosphates react if their concentration is equal to ten times that of the iron.

. In the case of industrial waste water, calcination at 600 to 650° C and dissolution of the ash are necessary prior to analysis.

The analysis equipment required is as follows:

- One spectrocolorimeter regulated at 510 nm;
- One set of twelve 100-ml volumetric flasks;
- Precision pipettes;
- One precision balance capable of weighing to 1/10 mg.

The reagents required for the analysis are as follows:

- Concentrated hydrochloric acid, HCl;
- Concentrated nitric acid, HNO_3;

- Concentrated sulphuric acid, H_2SO_4;
- Potassium peroxodisulphate, $K_2S_2O_8$;
- Hydroxylamine hydrochloride, $NH_2OH.HCl$;
- Ammonium acetate, CH_3COONH_4;
- Crystallizable acetic acid, CH_3COOH;
- 1,10 phenanthroline hydrochloride, $C_{12}H_9ClN_2.H_2O$;
- Non-oxidized iron wire.

6.19 Analysis of phosphorus and its compounds

The methods described make it possible to analyse the phosphorus present in water in various forms:

- Orthophosphate analysis;
- Polyphosphate analysis following hydrolysis;
- Total phosphorus analysis following oxidative digestion.

The detection limit for all these methods is 0.01 mg/l. All the results are expressed in mg/l of phosphorus (P).

Orthophosphate analysis (Rodier techniques)

In an acidic medium in the presence of ammonium molybdate, orthophosphates form a phosphomolybdic complex, which, when reduced by ascorbic acid, produces a blue coloration that can be analysed colorimetrically.

The analysis equipment required is as follows:

- One spectrocolorimeter;
- Precision pipettes;
- One set of twelve 50-ml volumetric flasks;
- One precision balance capable of weighing to 1/10 mg;
- One laboratory balance capable of weighing to 1/100 g;
- One pH-meter;
- Three 500-ml volumetric flasks;
- Five 1,000-ml volumetric flasks.

The reagents required for the analysis are as follows:

- Ascorbic acid, $C_6H_8O_6$;
- Sulphuric acid, H_2SO_4;

- Sodium hydroxide, NaOH;
- Antimony potassium tartrate, $K(SbO)C_4H_4O_6.0.5H_2O$;
- Ammonium molybdate, $(NH_4)_6Mo_7O_{24}.4H_2O$;
- Potassium dihydrogenophosphate, KH_2PO_4.

Polyphosphate analysis (Rodier techniques)

Polyphosphates are converted to orthophosphates by hot hydrolysis in an acidic medium.

Colorimetric analysis carried out after such hydrolysis shows the total content, expressed in mg/l of phosphorus (P), of the orthophosphates initially present and of the polyphosphates. The hydrolysable polyphosphate or phosphate content will be obtained by the difference between that total value and the values of the orthophosphates to be analysed separately.

In addition to the equipment already described for orthophosphate analysis, the following apparatus is required:

- Five 250-ml glass flasks;
- One heating rack;
- Five 100-ml volumetric flasks.

The chemical reagents used are the same as for orthophosphate analysis.

Total phosphorus analysis (Rodier techniques)

All the phosphorus, regardless of its state, is digested in a hot acidic medium in the presence of sodium persulphate. The orthophosphates obtained are then analysed colorimetrically.

In addition to the equipment required for orthophosphate analysis, the following apparatus is needed:

- One digestion rack comprising:
  - Five 250-ml long-necked flasks (Kjeldahl type);
  - One heating rack;
- Five 200-ml volumetric flasks;
- Five 250-ml beakers;
- One fume hood.

In addition to the reagents required for orthophosphate analysis, the following is also necessary:

- Sodium peroxodisulphate, $Na_2S_2O_8$.

6.20 Colour analysis (standard method 2120 B)

The procedure is based on visual comparison of the analysis sample with a reference coloration range such as the platinum-cobalt scale. This measuring technique is used only in the case of water whose colour characteristics are close to the reference scale.

The analysis equipment required is as follows:

- One set of twenty 50-ml flat-bottomed colorimetric tubes;
- One precision balance capable of weighing to 1/10 mg;
- One 1,000-ml volumetric flask;
- One 100-ml graduated measuring cylinder;
- One set of fifteen 50-ml volumetric flasks;
- One vacuum filtration apparatus with glass-fibre filters.

The chemicals required for the analysis are as follows:

- Hexahydrated cobalt (II) chloride, $CoCl_2.6H_2O$;
- Potassium chloroplatinate, K_2PtCl_6;
- Hydrochloric acid, HCl (d 20°: 1.19).

6.21 Effluent toxicity determination

Toxicity is a very complex concept: it encompasses the action of very many elements in highly varied forms (complexed, ionized, oxidized, etc.). Toxicity is measured by means of a test using a biological reagent: fish, daphnia, bacteria, algae, etc. This highly sophisticated measuring operation is reserved for specialized laboratories.

7. IDENTIFICATION OF FOUR LABORATORY SIZES

With a view to undertaking a quantitative and qualitative assessment of the equipment and chemicals required for a tannery laboratory, in the management of a physico-chemical and biological treatment plant, and for the implementation of clean in-house technologies, the following scale has been adopted:

Level 1

The laboratory is attached to a small tannery that handles less than 5 tons of salted raw bovine hides per day or processes less than 1,000 sheepskins or goatskins per day. The maximum values may be higher if the tannery carries out only part of the production operation, for example from raw to wet-blue hides or from crust to finished leather. The quantity of waste water will generally be less than 150 to 200 m^3 per day.

Level 2

This level corresponds to a tannery handling between 5 and 15 tons of salted raw bovine hides per day or between 1,000 and 3,000 skins per day. The quantity of waste water will be between 200 and 500 m^3 per day.

Level 3

The tannery capacity will be between 15 and 30 tons of bovine hides per day or between 3,000 and 6,000 sheepskins or goatskins per day, with a waste-water volume of less than 1,000 m^3 per day.

Level 4

This last level corresponds to a bovine tannery with a capacity exceeding 30 tons of salted hides per day or a tannery handling more than 6,000 skins per day. The volume of effluent discharge will generally be in excess of 1,000 m^3 per day.

8. NUMBER OF ANALYSES CONDUCTED PER WEEK AT EACH LEVEL

The weekly number of analyses for each determination can be seen from the table below:

Parameter	Level 1	Level 2	Level 3	Level 4
pH	15	30	60	90
Settleability	5	5	10	10
COD	2	5	10	10
Chromium	5	10	20	30
SS		5	10	10
Total solids		5	10	10
Sulphides		5	10	10
Dissolved oxygen		5	10	15
Mohlman index			5	5
TKN			2	5
BOD_5			5	10
Calcium			5	10
Chlorides			2	5
Phenols				2
Sulphates				2
Aluminium				5
Iron				2
Phosphorus				1
Colour				1

These values are of course only quantitative indications that are liable to variation in specific situations, such as tanneries linked to a joint industrial effluent treatment plant.

9. LEVEL 1 LABORATORY: EQUIPMENT REQUIRED

Such a laboratory will thus carry out the following determinations:

- pH, settleability, COD and chromium.

The laboratory equipment required is as follows:

- One laboratory pH-meter;
- One glass electrode and one calomel electrode (KCl);
- Twelve 250-ml glass beakers;
- One magnetic heating stirrer with five Teflon-coated bars;
- One 1-litre graduated Imhoff cone with one support;
- One precision balance (capable of weighing to 1/10 mg);
- Six 500-ml flasks with ground-glass necks;
- Six water or air coolers;
- Four precision pipettes of each of the following volumes: 1, 5, 10, 20, 25 and 50 ml;
- Two 25-ml precision burettes;
- One burette support with double clamp;
- Six electric flask heaters;
- Six 250-ml volumetric flasks with stoppers;
- Six 250-ml Erlenmeyer flasks;
- Glass beads;
- One 3-kW distilled water apparatus with a capacity of 4 litres per hour;
- One 20-litre storage container;
- 50 metres of clear PVC tubing with an inside diameter of 10 mm;
- One refrigerator with a capacity of approximately 250 litres.

These equipment items require a laboratory work surface 6 m long and 0.85 m wide, with one sink, two cold-water taps, one hot-water tap, four single-phase power sockets and one three-phase power socket. The laboratory, which will have a total floor area of 18 m^2, will also contain a storage cabinet for equipment and chemicals, and an air extractor hood.

10. LEVEL 1 LABORATORY: CHEMICALS

The chemicals have been determined on the basis of a one-year period of normal laboratory operation. They thus correspond to:

- 750 pH measurements;
- 250 settleability tests;
- 100 COD tests;
- 250 chromium analyses using the Mohr's salt method.

The chemicals required are as follows:

- 12 solutions buffered to a pH of 4;
- 12 solutions buffered to a pH of 7;
- 12 solutions buffered to a pH of 10;
- 100 g of mercury II sulphate, $HgSO_4$, in powder form;
- 50 g of silver sulphate, Ag_2SO_4, in powder form;
- 15 litres of concentrated sulphuric acid (d 20°: 1.83);
- 2 kg of Mohr's salt, $FeSO_4.(NH_4)_2SO_4.6H_2O$;
- 250 g of potassium bichromate, $K_2Cr_2O_7$;
- 300 ml of ferroin indicator (o-phenanthroline and $FeSO_4.7H_2O$);
- 3 litres of perchloric acid, $HClO_4$ (d 20°: 1.615);
- 2 litres of nitric acid, HNO_3 (d 20°: 1.33).

11. LEVEL 2 LABORATORY: EQUIPMENT REQUIRED

Such a laboratory will carry out the following determinations:

- pH, settleability, COD, chromium, suspended solids, dry solids, sulphide and dissolved oxygen.

The laboratory equipment required is as follows:

- One automatic sampler;
- One laboratory pH-meter;
- One glass electrode and one calomel electrode (KCl);
- Twelve 250-ml glass beakers;
- One magnetic heating stirrer with five Teflon-coated bars;

- One 1-litre graduated Imhoff cone with one support;
- One precision balance (capable of weighing to 1/10 mg);
- Six 500-ml flasks with ground-glass necks;
- Six water or air coolers;
- Four precision pipettes of each of the following volumes: 1, 5, 10, 20, 25 and 50 ml;
- Two 25-ml precision burettes;
- One burette support with double clamp;
- Six electric flask heaters;
- Six 250-ml volumetric flasks with stoppers;
- Six 250-ml Erlenmeyer flasks;
- Glass beads;
- One 3-kW distilled water apparatus with a capacity of 4 litres per hour;
- One 20-litre storage container;
- 50 metres of clear PVC tubing with an inside diameter of 10 mm;
- One refrigerator with a capacity of approximately 250 litres;
- One stainless vacuum pump with 10 metres of 6 x 18 mm vacuum tubing;
- One vacuum filtration apparatus (1-litre flask, support and joint);
- Ten 50-ml silica dishes;
- Ten 190-ml silica dishes;
- One 55-litre oven for operation at 100 to 105° C;
- One desiccator with a diameter of 200 mm;
- One 5-litre muffle furnace capable of being heated to 850° C;
- One potentiometric analysis apparatus;
- One calomel electrode with K_2SO_4 filler;
- One sulphide-selective electrode;
- Two 50-ml measuring cylinders;
- One polarographic measuring probe;

- One precision thermometer;
- One oxygen analyser graduated in ppm, mg/l or % oxygen;
- Twenty 100-ml glass bottles.

These equipment items require a laboratory work surface 10 m long and 0.85 m wide, with one sink, three cold-water taps, one hot-water tap, six single-phase power sockets and one three-phase power socket. The laboratory, which will have a total floor area of 30 m^2, will also contain two storage cabinets for equipment and chemicals.

12. LEVEL 2 LABORATORY: CHEMICALS

The chemicals have been determined on the basis of a one-year period of normal laboratory operation. They thus correspond to:

- 1,500 pH measurements;
- 250 settleability tests;
- 250 COD tests;
- 500 chromium analyses using the Mohr's salt method;
- 250 suspended solids analyses;
- 250 total solids analyses;
- 250 sulphide analyses using the potentiometric method;
- 250 dissolved oxygen tests.

The chemicals required are as follows:

- 24 solutions buffered to a pH of 4;
- 24 solutions buffered to a pH of 7;
- 24 solutions buffered to a pH of 10;
- 250 g of mercury II sulphate, $HgSO_4$, in powder form;
- 100 g of silver sulphate, Ag_2SO_4, in powder form;
- 35 litres of concentrated sulphuric acid (d 20°: 1.83);
- 5 kg of Mohr's salt, $FeSO_4.(NH_4)_2SO_4.6H_2O$;
- 500 g of potassium bichromate, $K_2Cr_2O_7$;
- 700 ml of ferroin indicator (o-phenanthroline and $FeSO_4.7H_2O$);
- 6 litres of perchloric acid, $HClO_4$ (d 20°: 1.615);
- 3 litres of nitric acid, HNO_3 (d 20°: 1.33);

- 300 calibrated glass-fibre filters with a diameter of 47 mm;
- 150 g of silver nitrate, $AgNO_3$;
- 5 litres of ammonia, NH_4OH (d 20°: 0.9);
- 1 kg of ammonium chloride, NH_4Cl;
- 150 g of 1,2 CDTA.

13. LEVEL 3 LABORATORY: EQUIPMENT REQUIRED

Such a laboratory will carry out the following determinations:

- pH, settleability, COD, chromium, suspended solids, dry solids, sulphide, dissolved oxygen, Mohlman index, TKN, BOD_5, calcium and chloride.

The laboratory equipment required is as follows:

- One automatic sampler;
- One flow meter and recorder;
- One laboratory pH-meter;
- One glass electrode and one calomel electrode (KCl);
- Twelve 250-ml glass beakers;
- One magnetic heating stirrer with five Teflon-coated bars;
- One 1-litre graduated Imhoff cone with one support;
- One precision balance (capable of weighing to 1/10 mg);
- Six 500-ml flasks with ground-glass necks;
- Six water or air coolers;
- Four precision pipettes of each of the following volumes: 1, 5, 10, 20, 25 and 50 ml;
- Two 25-ml precision burettes;
- One burette support with double clamp;
- Six electric flask heaters;
- Six 250-ml volumetric flasks with stoppers;
- Six 250-ml Erlenmeyer flasks;
- Glass beads;

- One 3-kW distilled water apparatus with a capacity of 4 litres per hour;
- One 20-litre storage container;
- 50 metres of clear PVC tubing with an inside diameter of 10 mm;
- One refrigerator with a capacity of approximately 250 litres;
- One stainless vacuum pump with 10 metres of 6 x 18 mm vacuum tubing;
- One vacuum filtration apparatus (1-litre flask, support and joint);
- Ten 50-ml silica dishes;
- Ten 190-ml silica dishes;
- One 55-litre oven for operation at 100 to 105° C;
- One desiccator with a diameter of 200 mm;
- One 5-litre muffle furnace capable of being heated to 850° C;
- One potentiometric analysis apparatus;
- One calomel electrode with K_2SO_4 filler;
- One sulphide-selective electrode;
- Two 50-ml measuring cylinders;
- One polarographic measuring probe;
- One precision thermometer;
- One oxygen analyser graduated in ppm, mg/l or % oxygen;
- Twenty 100-ml glass bottles;
- One automatic Kjeldahl nitrogen distillation apparatus;
- Ten 300-ml balloon flasks;
- One electrically heated six-position digestion rack;
- Ten 500-ml Erlenmeyer flasks;
- Silicon grease;
- One incubator thermostatically controlled at 20 $\pm$ 1° C for BOD_5;
- One hundred 250-ml incubation bottles with ground-glass stoppers;
- Volumetric flasks: 2 x 2,000 ml, 5 x 1,000 ml, 6 x 500 ml and 10 x 100 ml;

- One 12-W membrane compressor (capacity: 9 litres per minute) to saturate the dilution water;

- One mercury silver amalgam electrode;

- Twelve 150-ml beakers;

- One silver electrode.

These equipment items require a laboratory work surface 16 m long and 0.85 m wide, with two sinks, four cold-water taps, two hot-water taps, 10 single-phase power sockets and 2 three-phase power sockets. The laboratory, which will have a total floor area of 40 m^2, will also contain storage units beneath the work surface for equipment and chemicals.

14. LEVEL 3 LABORATORY: CHEMICALS

The chemicals have been determined on the basis of a six-month period of normal laboratory operation. They thus correspond to:

- 1,500 pH measurements;

- 250 settleability tests;

- 250 COD tests;

- 500 chromium analyses using the Mohr's salt method;

- 250 suspended solids analyses;

- 250 total solids analyses;

- 250 sulphide analyses using the potentiometric method;

- 250 dissolved oxygen tests;

- 125 Mohlman index measurements;

- 50 TKN analyses;

- 125 BOD_5 tests;

- 125 calcium analyses using the potentiometric method;

- 50 chloride analyses.

The chemicals required are as follows:

- 24 solutions buffered to a pH of 4;

- 24 solutions buffered to a pH of 7;

- 24 solutions buffered to a pH of 10;

- 300 g of mercury II sulphate, $HgSO_4$, in powder form;

- 100 g of silver sulphate, Ag_2SO_4, in powder form;
- 50 litres of concentrated sulphuric acid (d 20°: 1.83);
- 5 kg of Mohr's salt, $FeSO_4.(NH_4)_2SO_4.6H_2O$;
- 2 kg of potassium bichromate, $K_2Cr_2O_7$;
- 700 ml of ferroin indicator (o-phenanthroline and $FeSO_4.7H_2O$);
- 6 litres of perchloric acid, $HClO_4$ (d 20°: 1.615);
- 10 litres of nitric acid, HNO_3 (d 20°: 1.33);
- 300 calibrated glass-fibre filters with a diameter of 47 mm;
- 200 g of silver nitrate, $AgNO_3$;
- 5 litres of ammonia, NH_4OH (d 20°: 0.9)
- 2 kg of ammonium chloride, NH_4Cl;
- 150 g of CDTA;
- 1 kg of potassium sulphate, K_2SO_4;
- 50 g of selenium catalyst;
- 3 kg of sodium hydroxide, NaOH;
- 500 g of boric acid, H_3BO_3;
- 100 g of methyl red;
- 25 g of bromocresol green;
- 1 litre of ethanol, 95%;
- 1 kg of dihydrated sodium monohydrogenophosphate, $Na_2HPO_4.2H_2O$;
- 1 kg of potassium dihydrogenophosphate, KH_2PO_4;
- 1 kg of magnesium sulphate, $MgSO_4.7H_2O$;
- 1 kg of calcium chloride, $CaCl_2$;
- 1 litre of ferric chloride, $FeCl_3$, 27.5%;
- 250 g of purified mercury;
- 1 litre of triethanolamine;
- 1 litre of ethanolamine;
- 100 g of EDTA;

- 250 g of calcium carbonate, $CaCO_3$;

- 1 litre of hydrochloric acid, HCl.

15. LEVEL 4 LABORATORY: EQUIPMENT REQUIRED

Such a laboratory will carry out the following determinations:

- pH, settleability, COD, chromium, suspended solids, dry solids, sulphide, dissolved oxygen, Mohlman index, TKN, BOD_5, calcium, chloride, phenols, sulphates, aluminium, iron, phosphorus and colour.

The laboratory equipment required is as follows:

- One automatic sampler;

- One flow meter and recorder;

- One laboratory pH-meter;

- One glass electrode and one calomel electrode (KCl);

- Twelve 250-ml glass beakers;

- One magnetic heating stirrer with 5 Teflon-coated bars;

- One 1-litre graduated Imhoff cone with one support;

- One precision balance (capable of weighing to 1/10 mg);

- Six 500-ml flasks with ground-glass necks;

- Six water or air coolers;

- Four precision pipettes of each of the following volumes: 1, 5, 10, 20, 25 and 50 ml;

- Two 100-ml precision pipettes;

- Two 25-ml precision burettes;

- One burette support with double clamp;

- Six electric flask heaters;

- Six 250-ml volumetric flasks with stoppers;

- Six 250-ml Erlenmeyer flasks;

- Glass beads;

- One 3-kW distilled water apparatus with a capacity of 4 litres per hour;

- One 20-litre storage container;

- 50 metres of clear PVC tubing with an inside diameter of 10 mm;
- One refrigerator with a capacity of approximately 250 litres;
- One stainless vacuum pump with 10 metres of 6 x 18 mm vacuum tubing;
- One vacuum filtration apparatus (1-litre flask, support and joint);
- Ten 50-ml silica dishes;
- Ten 190-ml silica dishes;
- One 55-litre oven for operation at 100 to 105° C;
- One desiccator with a diameter of 200 mm;
- One 5-litre muffle furnace capable of being heated to 850° C;
- One potentiometric analysis apparatus;
- One calomel electrode with K_2SO_4 filler;
- One sulphide-selective electrode;
- Two 50-ml measuring cylinders;
- One polarographic measuring probe;
- One precision thermometer;
- One oxygen analyser graduated in ppm, mg/l or % oxygen;
- Twenty 100-ml glass bottles;
- One automatic Kjeldahl nitrogen distillation apparatus;
- Ten 300-ml balloon flasks;
- One electrically heated six-position digestion rack;
- Ten 500-ml Erlenmeyer flasks;
- Silicon grease;
- One incubator thermostatically controlled at 20 $\pm$ 1° C for BOD_5;
- One hundred 250-ml incubation bottles with ground-glass stoppers;
- Volumetric flasks: 2 x 2,000 ml, 5 x 1,000 ml, 6 x 500 ml and 10 x 100 ml;
- One 12-W membrane compressor (capacity: 9 litres per minute) to saturate the dilution water;
- One mercury silver amalgam electrode;

- Twelve 150-ml beakers;
- One silver electrode;
- One set of twelve 25-ml volumetric flasks;
- One spectrocolorimeter with accessories;
- One set of twelve 250-ml separating funnels;
- One set of twelve 200-ml volumetric flasks;
- One all-glass distillation apparatus with ground joints, comprising:
  . One 500-ml three-necked flask;
  . One thistle funnel to fit the flask;
  . One condenser;
  . Two curved extension pieces;
- One set of twenty 50-ml volumetric flasks with stoppers;
- One laboratory balance capable of weighing to 1/100 g;
- Five 250-ml glass flasks with ground necks;
- One set of twenty 50-ml flat-bottomed colorimetric tubes;
- Two 100-ml graduated measuring cylinders.

These equipment items require a laboratory work surface 24 m long and 0.85 m wide, with two sinks, four cold-water taps, two hot-water taps, 16 single-phase power sockets and 2 three-phase power sockets. The laboratory, which will have a total floor area of 60 m^2, will also contain storage units beneath the work surface for equipment and chemicals.

16. LEVEL 4 LABORATORY: CHEMICALS

The chemicals have been determined on the basis of a six-month period of normal laboratory operation. They thus correspond to:

- 2,250 pH measurements;
- 250 settleability tests;
- 250 COD tests;
- 750 chromium analyses using the Mohr's salt method;
- 250 suspended solids analyses;
- 250 total solids analyses;
- 250 sulphide analyses using the potentiometric method;

- 375 dissolved oxygen tests;
- 125 Mohlman index measurements;
- 125 TKN analyses;
- 250 BOD_5 tests;
- 250 calcium analyses using the potentiometric method;
- 125 chloride analyses;
- 50 phenol index measurements;
- 50 sulphate analyses;
- 125 aluminium analyses;
- 50 iron analyses;
- 50 phosphorus analyses;
- 25 colour measurements.

The chemicals required are as follows:

- 36 solutions buffered to a pH of 4;
- 36 solutions buffered to a pH of 7;
- 36 solutions buffered to a pH of 10;
- 300 g of mercury II sulphate, $HgSO_4$, in powder form;
- 100 g of silver sulphate, Ag_2SO_4, in powder form;
- 60 litres of concentrated sulphuric acid (d 20°: 1.83);
- 6 kg of Mohr's salt, $FeSO_4.(NH_4)_2SO_4.6H_2O$;
- 2 kg of potassium bichromate, $K_2Cr_2O_7$;
- 800 ml of ferroin indicator (o-phenanthroline and $FeSO_4.7H_2O$);
- 9 litres of perchloric acid, $HClO_4$ (d 20°: 1.615);
- 13 litres of nitric acid, HNO_3 (d 20°: 1.33);
- 300 calibrated glass-fibre filters with a diameter of 47 mm;
- 200 g of silver nitrate, $AgNO_3$;
- 6 litres of ammonia, NH_4OH (d 20°: 0.9);
- 2 kg of ammonium chloride, NH_4Cl;

- 150 g of CDTA;
- 1 kg of potassium sulphate, K_2SO_4;
- 100 g of selenium catalyst;
- 5 kg of sodium hydroxide, NaOH;
- 1 kg of boric acid, H_3BO_3;
- 200 g of methyl red;
- 25 g of bromocresol green;
- 1 litre of ethanol, 95%;
- 1 kg of dihydrated sodium monohydrogenophosphate, $Na_2HPO_4.2H_2O$;
- 1 kg of potassium dihydrogenophosphate, KH_2PO_4;
- 1 kg of magnesium sulphate, $MgSO_4.7H_2O$;
- 1 kg of calcium chloride, $CaCl_2$;
- 1 litre of ferric chloride, $FeCl_3$, 27.5%;
- 500 g of purified mercury;
- 1 litre of triethanolamine;
- 1 litre of ethanolamine;
- 200 g of EDTA;
- 250 g of calcium carbonate, $CaCO_3$;
- 2 litres of hydrochloric acid, HCl;
- 1 kg of double potassium sodium tartrate, $KNaC_4H_4O_6.4H_2O$;
- 10 g of 4-aminoantipyrine (1-phenyl-2-3-dimethyl-4-amino-pyrazolone-5);
- 250 g of potassium ferricyanide, $K_3Fe(CN)_6$;
- 1 litre of phosphoric acid, H_3PO_4;
- 2 litres of chloroform, $CHCl_3$;
- 3 kg of sodium chloride, NaCl;
- 1 kg of phenol, C_6H_5OH;
- 1 kg of sodium sulphate, Na_2SO_4;
- 1 litre of Tween 20;

- 1 kg of stabilized barium chloride, $BaCl_2$;
- 1 kg of hexamethylenetetramine;
- 100 g of xylenol orange;
- 250 g of zinc sulphate, $ZnSO_4.7H_2O$;
- 100 g of potassium peroxodisulphate, $K_2S_2O_8$;
- 250 g of hydroxylamine hydrochloride, $NH_2OH.HCl$;
- 1 kg of ammonium acetate, CH_3COONH_4;
- 1 litre of crystallizable acetic acid, CH_3COOH;
- 15 g of 1,10 phenanthroline hydrochloride, $C_{12}H_9ClN_2.H_2O$;
- 250 g of non-oxidized iron wire;
- 250 g of ascorbic acid, $C_6H_8O_6$;
- 500 g of antimony potassium tartrate, $K(SbO)C_4H_4O_6.0.5H_2O$;
- 100 g of ammonium heptamolybdate, $(NH_4)_6Mo_7O_{24}.4H_2O$;
- 250 g of sodium peroxodisulphate, $Na_2S_2O_8$;
- 100 g of hexahydrated cobalt (II) chloride, $CoCl_2.6H_2O$;
- 1 g of potassium chloroplatinate, K_2PtCl_6.

17. ASSESSMENT OF NECESSARY EQUIPMENT AND CHEMICALS

An example of a standard laboratory layout is shown in annex 3.

The evaluations given below are based on equipment and chemicals available in Europe. The prices are indicated in United States dollars before tax (export sales).

Level 1

. Equipment evaluation:	$ 8 500
. Chemicals:	$ 1 200
Total	$ 9 700

Level 2

. Equipment evaluation:	$19 700
. Chemicals:	$ 4 100
Total	$23 800

Level 3

. Equipment evaluation:	$36 800
. Chemicals:	$ 5 600
Total	$42 400

Level 4

. Equipment evaluation:	$44 800
. Chemicals:	$ 7 800
Total	$52 600

18. ASSESSMENT OF PERSONNEL REQUIREMENTS

Taking into account the analyses to be carried out, the laboratory personnel requirements may be evaluated as follows:

Level 1: 1 part-time employee

Level 2: 1 full-time employee

Level 3: 1 full-time and 1 part-time employee

Level 4: 2 full-time employees.

The personnel skills required correspond to a laboratory technician's diploma, i.e. a technical vocational level of education.

Annex 1

LABORATORY EQUIPMENT SPECIFICATIONS

Burette support

- 3-kg cast-iron base
- Nickel-plated steel rod measuring 800 x 12 mm
- Two-burette clamp for 12-mm rod
- With sighting device to prevent parallax errors

Colorimetric tubes, 50 ml

- Borosilicate glass
- Flat-bottomed
- Approximate dimensions: 22 x 200 mm
- PTFE screw cap and joint
- 20-hole (2 x 10) stand for 25-mm tubes

Desiccator

- Diameter: 200 mm
- Borosilicate glass
- Flat 24/29 ground-glass lid with tap
- Enamelled porcelain disk 185 mm in diameter
- 1 kg of silica gel

Dissolved oxygen analyser

- Portable equipment supplied in a carrying-case
- Measuring range: 0 to 60 mg/l or 0 to 600% saturation
- Accuracy: 1%
- Automatic temperature compensation from 0 to 50° C
- Automatic pressure compensation from 800 to 1,080 mbar
- Automatic salinity compensation from 0 to 40%
- Recorder output: 10 mV per mg/l of dissolved oxygen
- Independence of operation: up to 250 hours

Electric digestion rack

- Six positions
- Independent regulator for each position
- For 300-ml balloon flasks
- Wattage: 300 and 600 W
- With fume hood and support

Electric flask heaters

- Six-position electric heating rack
- Flask capacity: 500 ml
- Independent regulation of all six positions
- Temperature adjustment up to 450° C
- Wattage: 1,200 W
- Current and temperature indicator lights
- Acid splash protection
- Interchangeable heating caps
- Six support rods 12.5 mm in diameter

Electrode, glass

- Standard bulb electrode 10 mm in diameter
- Length: 120 mm
- Measuring range: 0 to 14 pH
- Cable length: 1 m

Electrode, reference

- Calomel reference electrode (saturated KCl)
- Length: 120 mm
- Measuring range: 0 to 14 pH
- Operating temperature: 0 to 60° C
- Cable length: 1 m
- 250 ml of saturated KCl solution

Electrode, silver

- Screw head
- Silver rod cap 4 mm in diameter
- Tubular body 10 mm in diameter and 120 mm long
- 1.5 metres of connecting cable

Electrode, sulphide-selective

- pH range: 11 to 14
- Silver sulphide monocrystal
- Response range: 1 to 10^{-10} M

Erlenmeyer flasks, 250 ml

- Borosilicate glass
- Wide aperture, without spout, 45 mm diameter
- Approximate height: 140 mm

Erlenmeyer flasks, 500 ml

- Borosilicate glass
- Wide aperture, without spout, 45 mm diameter
- Approximate height: 175 mm

Extension pieces for distiller, curved

- Borosilicate glass
- 105° angle bend
- 19/26 ground-glass cone and socket joints

Flasks with ground-glass necks, 250 ml

- Round-bottomed
- Short-necked
- Standard 24/29 ground glass
- Pyrex borosilicate glass

Flasks with ground-glass necks, 500 ml

- Round-bottomed
- Short-necked

- Standard 24/29 ground glass
- Pyrex borosilicate glass

Glass beads

- Diameter: 4 mm
- Weight: 1 kg approximately

Glass beakers, 150 ml

- Heavy-duty Pyrex glass
- Low-sided, diameter approximately 57 mm
- Reinforced flanged rims
- Double graduation scale

Glass beakers, 250 ml

- Heavy-duty Pyrex glass
- Low-sided, diameter approximately 68 mm
- Reinforced flanged rims
- Double graduation scale

Glass bottles, 100 ml

- Borosilicate glass
- Phenolic-plastic screw cap
- PTFE watertight joint

Heating oven

- Internal volume: 55 litres
- Temperature range: ambient + 5° C to 220° C
- Continuous control between 60 and 220° C with safety thermostat
- Accuracy: $< \pm 1\%$
- Wattage: 600 W
- Stainless-steel inner chamber
- Reinforced insulation
- Remote thermometer (40 to 220° C)

- Maximum number of shelves: 4
- Natural hot-air convection

Heating stirrer, magnetic

- Stirring capacity: 5 litres
- Temperature adjustment
- Continuous speed control from 300 to 1,000 rpm
- Plate diameter: 110 mm
- Maximum temperature: 350° C
- Wattage: 350 W
- Five PTFE-coated magnetized bars measuring 45 x 9 mm

Imhoff cones

- Quantity: 2
- Useful unit volume: 1 litre
- Clear acrylonitrile polystyrene
- Rigid methyl polymethacrylate support for 2 cones

Incubation bottles, 250 ml

- White laboratory glass
- Wide-necked (60 mm)
- Ground-glass cap stopper

Incubator for BOD_5 test, thermostatically controlled

- Useful volume: 260 litres
- Temperature adjustable in 1° C stages from 4 to 40° C
- Temperature read-out: liquid-crystal display (accuracy: $\pm$ 1° C)
- Radial ventilation: 100 m^3 per hour
- Internal power sockets
- ABS interior
- 4 shelves

Kjeldahl balloon flasks, 300 ml

- Borosilicate glass
- Neck diameter 28 mm and length 300 mm
- Round-bottomed

Kjeldahl nitrogen distillation equipment

- Semi-automatic model
- Distillation time programmable up to 20 minutes
- Detection limit: 0.5 mg of nitrogen
- Distilled water feed
- Push-button soda dispenser
- Compatible with all tube types
- Reproducibility: 1%
- Wattage: 1,800 W

Laboratory balance, electronic

- Weighing range: 0 to 2,100 g
- Subtractive taring range: 0 to 2,100 g
- Read-out accuracy: 0.01 g
- Linearity: $\pm$ 0.02 g
- Electrical protection: IP54
- Calibration menu
- Stabilization detector
- Vibration adaptor

Laboratory pH-meter

- Measuring range: 0 to 14 $\pm$ 0.01 pH; 0 to 100 $\pm$ 0.4° C
- Automatic and manual temperature compensation
- 3 memorized buffer solutions: pH 4, 7 and 10
- Automatic calibration
- Numerical liquid-crystal display

- Control keyboard with watertight keys
- With combined pH electrode and temperature probe

Measuring cylinders, 50 ml

- Borosilicate glass
- Enamelled graduation markings in 1-ml divisions
- Approximate height: 200 mm
- Hexagonal base and pouring spout

Measuring cylinders, 100 ml

- Borosilicate glass
- Enamelled graduation markings in 1-ml divisions
- Approximate height: 260 mm
- Hexagonal base and pouring spout

Membrane compressor

- For oil-free air
- Maximum flow rate: 9 litres per minute
- Operating pressure: 0.4 bar
- Wattage: 12 W
- Neoprene membrane
- Replacement membrane and valves

Muffle furnace

- Loading capacity: 5 litres
- Maximum temperature: 900° C
- Continuous temperature control
- Accuracy: $< \pm 0.5\%$
- Wattage: 1,800 W
- Ceramic-fibre insulation
- Safety device (heater cutting out when door is opened)
- 1 intermediate ceramic tray

- Fume evacuation flue
- 1 replacement heating element

Polarographic measuring probe

- With three spare membranes
- Electrolyte and anode cleaning solution
- 1.5 m of cable
- Response time: 90% of the value in less than 10 seconds

Portable flow meter and recorder

- Bubbling probe measuring system
- Fitting all waste outlet types
- Watertight polyester carrying-case
- Battery with built-in charger (2 weeks' independence of operation)
- Three-curve recorder
- Additional inlets for a further 2 parameters (pH and temperature)
- Possible connection to a sampler
- PC-retrievable data

Portable sampler, programmable

- Sampling volume adustable from 5 to 100 ml (accuracy: $\pm$ 0.5 ml)
- Sampling rate adjustable by timer or flow-meter control (sampling proportional to time or volume)
- Twenty-four 1-litre polyethylene bottles
- Vacuum-pump sampling, with pre-sampling pressurized flushing
- Delayed start-up possible for 24 hours
- Battery with built-in charger (independence of operation for 1,000 sampling operations)
- Spare parts kit
- Replacement battery
- Set of spare bottles

Potentiometric analysis equipment

- Millivolt range: -1,600.0 to +1,600.0
- Relative millivolt range: -1,999.9 to +1,999.9
- Resolution: 0.1 mV
- Relative error: ± 0.2 mV
- Automatic calibration on 5 buffer solutions
- Five pH calibration and concentration points
- RS 232 outlet
- Two electrode inlets
- Automatic temperature compensation
- Digital display

Precision balance, electronic

- Weighing range: 0 to 109 g
- Accuracy: 0.1 mg
- Subtractive taring range: 0 to 109 g
- Stabilization time: 5 seconds
- Enclosed weighing chamber accessible from both sides and from above
- Wattage: 10 W

Precision burette, 25 ml

- Divisions: 0.05 ml
- High-temperature-enamelled graduation markings
- Error: < 0.05 ml
- Scale interval: > 1 mm
- PTFE stopcock

Precision pipettes

- Single-scale graduated pipettes
- Borosilicate glass
- Volumes: 1, 5, 10, 20, 25, 50 and 100 ml
- Pipetting bulb: model for 3.5- to 10-mm-diameter pipettes

Precision thermometer

- Measuring range: -2 to +80° C
- Divisions: 0.2° C
- Approximate length: 400 mm

PVC tubing, clear

- Length: 50 m
- Inside diameter: 10 mm

Refrigerator, 250 litres

- Household type
- Stainless inner liner
- 5 storage levels

Separating funnels, 250 ml

- Borosilicate glass
- Pear-shaped
- PTFE stopcock
- 24/29 ground-glass stopper
- Supporting platform for two funnels, orifices: 65 mm, distance between axes: 200 mm
- With stainless boss for 12-mm rod

Silica dishes

- Translucent silica
- Useful volume: 50 and 190 ml
- Spherical, with pouring spout and flat bottom

Spectrocolorimeter

- Monochromator with grid
- Wavelength range: 330 to 900 nm
- Accuracy: ± 2 nm
- Pass-band: ± 7 nm
- Numerical display

- Resolution: 0.001 optical density
- Automatic zero
- Light source: tungsten halogen lamp
- One replacement lamp
- Photocell detection
- Absorbance, transmittance, concentration and kinetic modes
- 10-mm cell-holder
- 6 glass cells with 2 polished planes, 10-mm window, 45 mm high, with 10-mm (± 0.01 mm) light path

Storage container, 20 litres

- Low-density polyethylene
- Wide aperture
- Polypropylene screw cap for 100-mm threaded neck
- Two carrying-handles

Thistle funnel

- Borosilicate glass
- Cylindrical
- Volume: 100 ml
- 19/26 ground-glass cone base joint

Three-necked flask, 500 ml

- Borosilicate glass
- Two 19/26 ground-glass side necks angled at 30°
- Central 29/32 ground-glass neck
- Round-bottomed

Vacuum filtration equipment

- 300-ml filter funnel
- Filtration support for filter paper having a diameter of 47 mm
- Seven rubber support joints
- 1-litre vacuum filtration flask

Vacuum pump

- Stainless steel
- Triple pump
- Minimum upstream pressure: 1 bar
- Flow rate at 2 bar: 640 litres per hour

Volumetric flasks, 25 ml

- Standard shape
- Borosilicate glass
- Furnace-enamelled markings
- Accuracy: 0.04 ml
- 10/19 ground joints
- Polyethylene stopper

Volumetric flasks, 50 ml

- Standard shape
- Borosilicate glass
- Furnace-enamelled markings
- Accuracy: 0.06 ml
- 12/21 ground joints
- Polyethylene stopper

Volumetric flasks, 100 ml

- Standard shape
- Borosilicate glass
- Furnace-enamelled markings
- Accuracy: 0.10 ml
- 12/21 ground joints
- Polyethylene stopper

Volumetric flasks, 200 ml

- Standard shape
- Borosilicate glass

- Furnace-enamelled markings
- Accuracy: 0.15 ml
- 14/23 ground joints
- Polyethylene stopper

Volumetric flasks, 250 ml

- Standard shape
- Borosilicate glass
- Furnace-enamelled markings
- Accuracy: 0.15 ml
- 14/23 ground joints
- Polyethylene stopper

Volumetric flasks, 500 ml

- Standard shape
- Borosilicate glass
- Furnace-enamelled markings
- Accuracy: 0.25 ml
- 19/26 ground joints
- Polyethylene stopper

Volumetric flasks, 1,000 ml

- Standard shape
- Borosilicate glass
- Furnace-enamelled markings
- Accuracy: 0.40 ml
- 24/29 ground joints
- Polyethylene stopper

Volumetric flasks, 2,000 ml

- Standard shape
- Borosilicate glass
- Furnace-enamelled markings

- Accuracy: 0.60 ml
- 24/29 ground joints
- Polyethylene stopper

Water cooler

- Useful length: 300 mm
- Standard 24/29 ground joint at the base
- Borosilicate glass

Water cooler for distiller

- Useful length: 200 mm
- Two standard 19/26 ground joints
- Borosilicate glass

Water distiller

- Borosilicate-glass distillation apparatus
- Distilled water flow rate: 4 litres per hour
- Wattage: 3 kW
- Stainless steel or quartz heating resistor
- Automatic level regulation and overheating safety device
- Plastic tubing for attachment to the water supply
- 5 litres of descaling product
- One replacement resistor

Annex 2

LABORATORY EQUIPMENT SUPPLIERS
(Non-exhaustive list)

BELGIUM

HACH EUROPE SA (laboratory equipment and products)
Chaussée de Namur 1
5150 Floriffoux
Tel.: 32 81 44 53 81 - Fax: 32 81 44 13 00

BRAZIL

GRUPO QUIMICA INDUSTRIAL LDA (laboratory chemicals)
Rua Jacuruta 826 - Penha
21020 Rio de Janeiro
Tel.: 55 021 280 9040 - Telex: 021 34091

MARTE BALANCAS E APARELHOS DE PRECISAO LDA (electronic balances)
04301 Av. Miguel Estefuo, 752/766
Sao Paulo - SP
Telex: 011 34318 MBAP BR

TECHNOW INSTRUMENTOS CIENTIFICOS LDA (laboratory equipment)
Avenida dos Imares, 478
CEP 04085 Indianapolis
Sao Paulo - SP
Tel.: 55011 542 19 11

DENMARK

RADIOMETER ANALYTICAL A/S (electrochemical equipment)
Kroghojvej 49
2880 Bagsvaerd
Tel.: 45 31 69 63 11 - Fax: 45 44 49 00 11

ESTONIA

WATER TREATMENT PLANT/LABORATORY (laboratory equipment)
Jarvevana Tee 3
200001 Tallinn
Tel.: 372 557 850 - Fax: 372 22 556 973

FRANCE

BIOBLOCK SCIENTIFIC (laboratory equipment)
Parc d'innovation - BP 11
67403 Illkirch Cedex
Tel.: 33 88 67 14 14 - Fax: 33 88 67 11 68

OMNIUM SCIENTIFIQUE ET INDUSTRIEL (equipment and chemicals)
141 rue de Javel
75739 Paris Cedex 15
Tel.: 33 1 45 54 97 31 - Fax: 33 1 45 54 26 28

PROLABO (laboratory equipment and chemicals)
12 rue Pelée
75011 Paris
Tel.: 33 1 48 07 38 00 - Fax: 33 1 43 55 28 50

ROUCAIRE SA (laboratory equipment)
20 Avenue de l'Europe, BP 65
78143 Velizy-Villacoublay Cedex
Tel.: 33 1 39 46 96 33 - Fax: 33 1 30 70 87 20

GERMANY

E. MERCK (laboratory equipment and products)
Frankfurter Strasse, 250, Postfach 4119
64287 Darmstadt
Tel.: 49 06151/72-0 - Fax: 49 06151/72-2000 - Telex: 4193280

SCHOTT GERATE GMBH (laboratory equipment and glassware)
Postfach 1130
Hofheim a. Ts.
Tel.: 49 06192/2091-0 - Fax: 49 06192/8086

INDIA

SATYAJIT ENGINEERING INDUSTRIES PVT LTD (electronic balances)
SDF Building, Salt Lake Electronics Complex
Salt Lake City
Calcutta - 700 091
Tel.: 091 33 35 83 02/36 44 06/37 23 78 - Telex: 212 468 SULA IN

SYSTRONICS (electrochemical equipment)
89-92 Industrial area
Naroda - 382 330
Ahmedabad
Tel.: 091 272 81 32 17/81 34 17 - Telex: 121 386 SYS IN

THERELEK FURNACES PRIVATE LTD (furnaces and ovens)
137 Mody Street, 3rd floor, Fort
Bombay - 400 001
Tel.: 091 22 261 53 59/261 18 44 - Fax: 091 22 287 26 40

SWITZERLAND

METROHM LTD (electrochemical equipment)
9101 Herisau
Tel.: 41 71 53 85 85 - Fax: 41 71 53 89 01

METTLER-TOLEDO (electronic balances)
8606 Greifensee
Tel.: 41 19 44 22 11 - Fax: 41 19 44 30 40

UNITED STATES OF AMERICA

EASTMAN CHEMICAL COMPANY (laboratory chemicals)
1001 Lee Road
Rochester NY 14652-3512
Tel.: 1 800 225 5352 - Fax: 1 800 879 4979

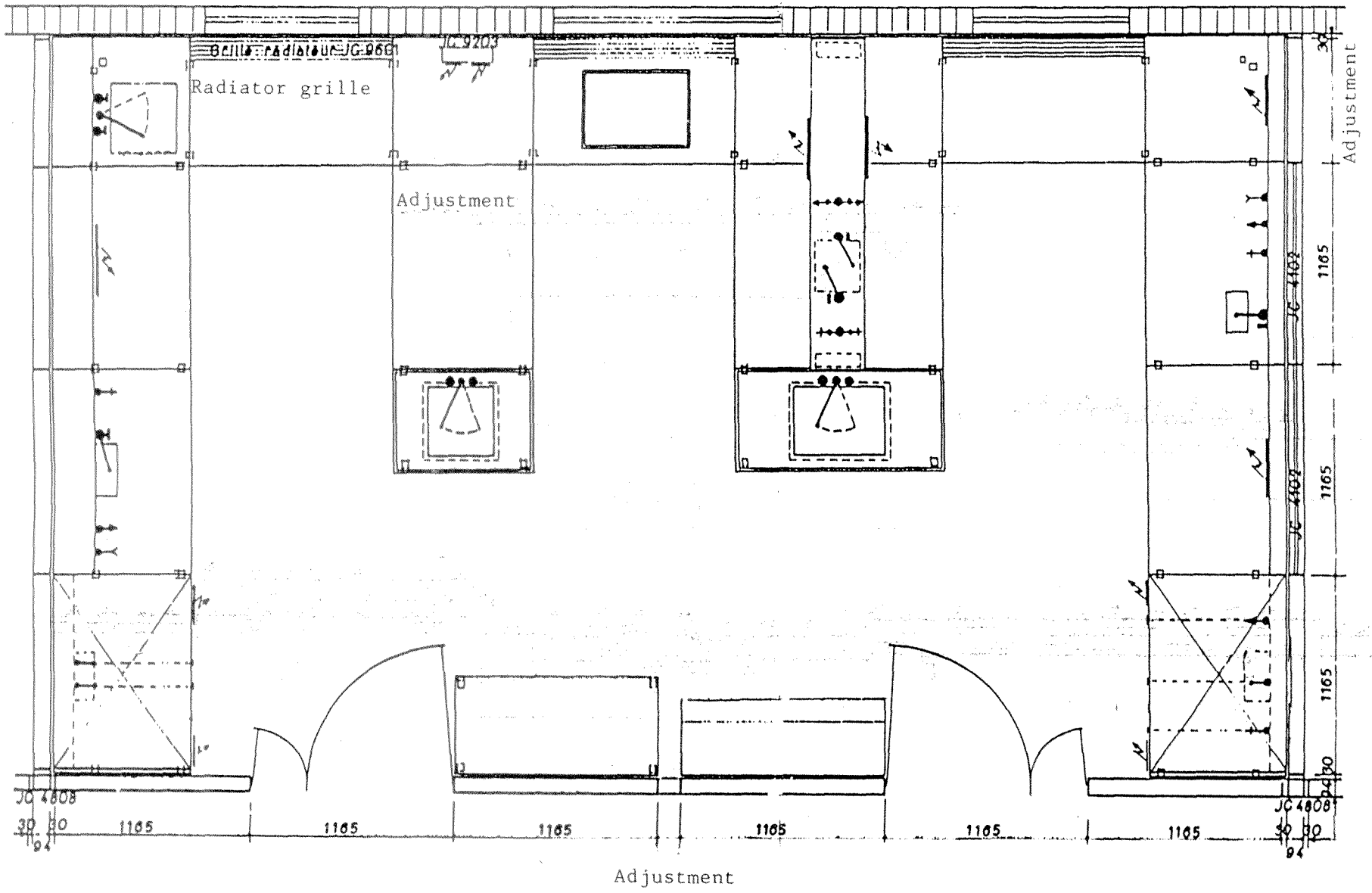
HACH Co. (laboratory equipment and chemicals)
PO Box 389
Loveland CO 80539
Tel.: 1 800 227 4224/1 303 669 3050 - Fax: 1 303 669 2932

ORION RESEARCH INC (electrochemical equipment)
529 Main Street, the Schrafft Ctr.
Boston MA 02129
Tel.: 1 617 242 3900/1 800 225 1480 - Fax: 1 617 242 8594

ZIMBABWE

GLASS BLOWING INDUSTRIES LTD (laboratory glassware)
8 George Ave.
Msasa, PO Box AY 275 Amby
Harare
Tel.: 263 4 45674 - Fax: 263 4 48196

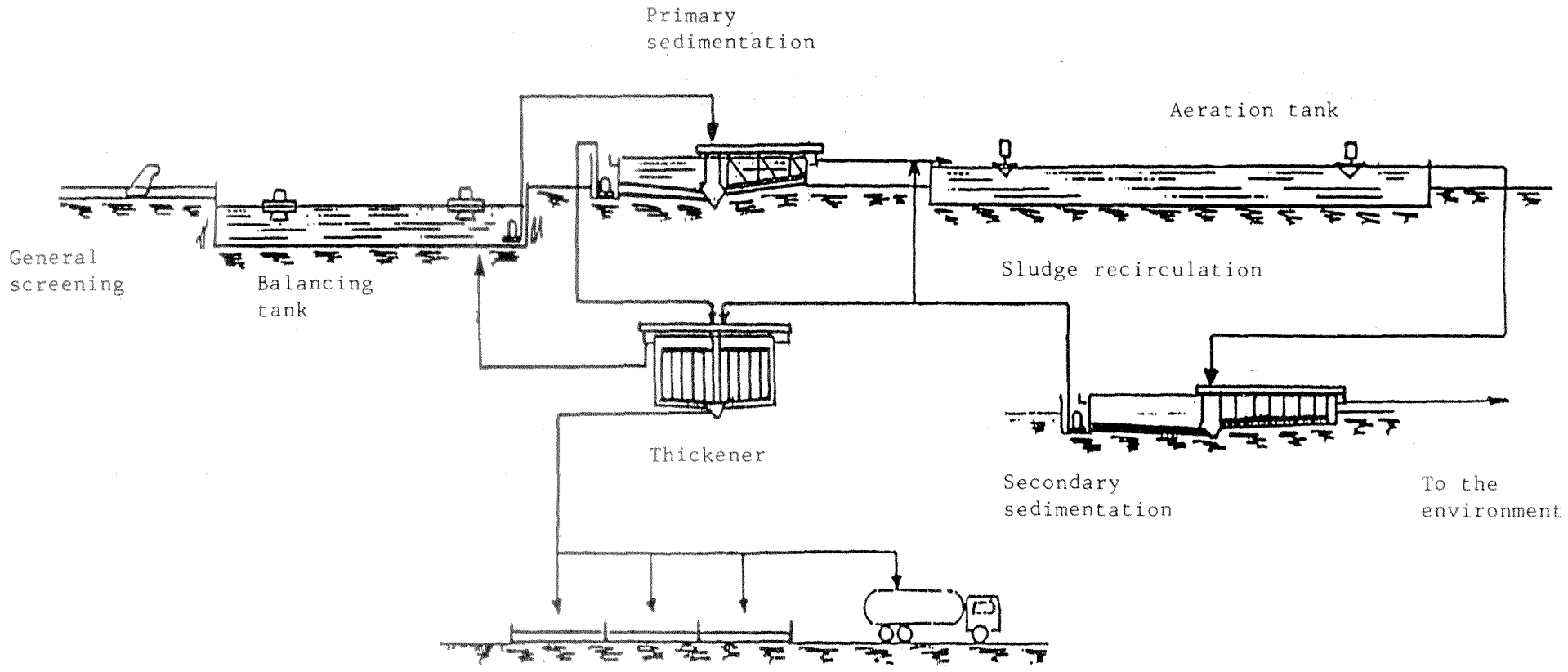
PHILIPS ELECTRICAL PVT LTD (electrochemical apparatus)
PO Box 994
Harare
Tel.: 263 9 47211 - Fax 263 9 47966



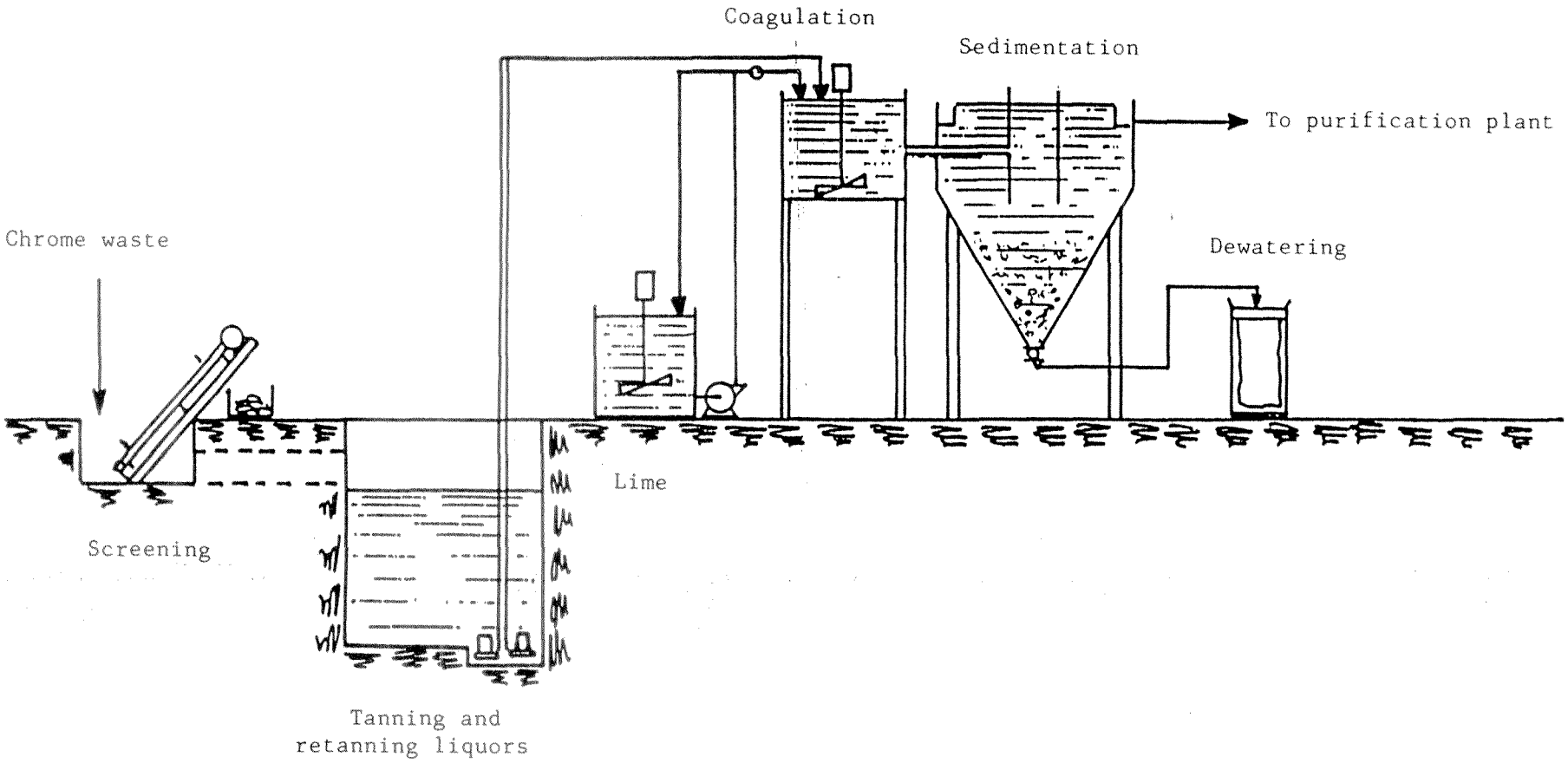
EXAMPLE OF A SPECIMEN LABORATORY LAYOUT AND PARTITIONING

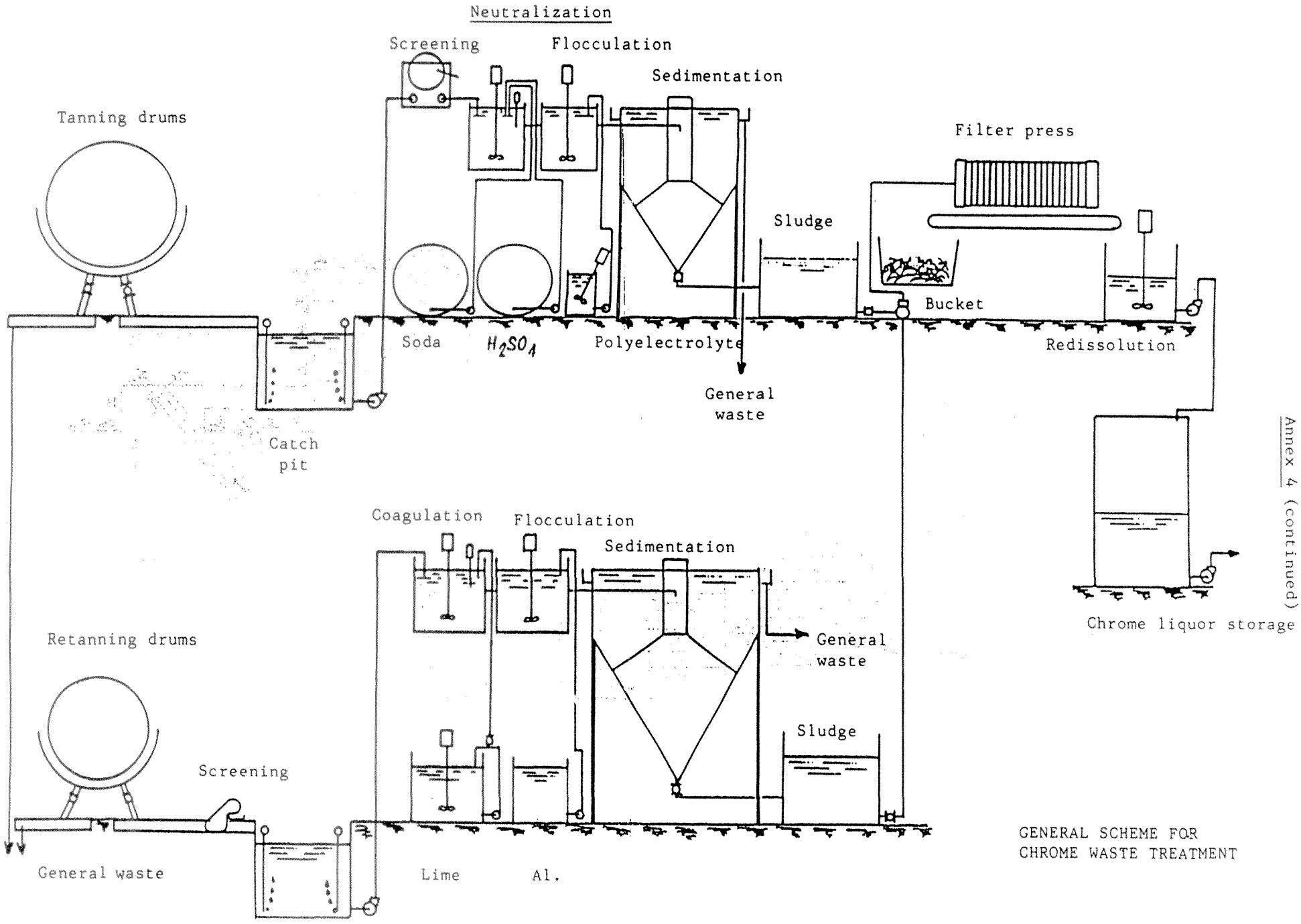
FLOW DIAGRAMS OF EFFLUENT PURIFICATION PLANTS

GENERAL SCHEME FOR WASTE-WATER TREATMENT



CHROME WASTE TREATMENT





GENERAL SCHEME FOR CHROME WASTE TREATMENT

GLOSSARY

Activated sludge

The flocculated or flocculating sludge formed during aeration of waste water by the microorganisms contained in the water. Such sludge is capable of digesting organic matter present in the water to be purified.

BOD_5

Five-day biochemical oxygen demand is the analytical determination of the quantity of oxygen required for the degradation of organic material in an effluent.

COD

Chemical oxygen demand is the analytical determination of the quantity of oxygen consumed chemically for the oxidation of organic matter and of certain inorganic materials in an effluent.

Dissolved oxygen

The quantity of oxygen dissolved in water. The proportion of dissolved oxygen varies according to temperature, salinity, etc.

Dry solids (DS)

The quantity of total solids present in an effluent following evaporation at 105° C.

Oxidizable matter (OM)

The oxidizable matter determined by both the BOD_5 test and the COD test.

pH

The pH value indicates the acidity (in the case of values below 7) or alkalinity (in the case of values between 7 and 14) of a solution. It is defined as the co-logarithm of the hydrogen-ion concentration.

Suspended solids (SS)

Suspended solids are various kinds of solid matter in suspension in a liquid that can be separated from the liquid by filtration or centrifugation.

Total Kjeldahl nitrogen

The sum of ammonia nitrogen and organic nitrogen.

Total nitrogen

The sum of ammonia nitrogen, organic nitrogen and the oxidized forms of nitrogen: nitrites and nitrates.

Total phosphorus

The sum of the various forms of phosphorus: oxidized or reduced, organic or inorganic.