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# Electrochemical production of lead chromate and sodium hydroxide in a two-compartment cell

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This paper describes the application of an anode-support system (AS) and a cationic permselective membrane to the electrolytic production of yellow lead chromate pigment and sodium hydroxide solution in a two-compartment cell. The authors have determined that it is possible to utilize in the process an AISI-316 stainless steel or a titanium mesh basket loaded with lead granules. Under the operating conditions used both the AISI-316 stainless steel and the titanium remain passive and act only as electrical contact between the lead anodic pieces and the external current source. A Nafion® 901 bimembrane (Dupont, USA) is utilized to separate both compartments. Good quality yellow lead chromate and sodium hydroxide solution (50–150 g dm$^{-3}$) are obtained, with a power requirement of 0.41 kWh (kg $PbCrO_4$)$^{-1}$.

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## 1. Introduction

The electrochemical production of lead chromate has been known for a hundred years and there are several patents and publications on the topic [1–8]. The product has been widely used as a yellow–orange pigment due to its low cost price and its good light resistance, but its use has decreased because of its toxicity. For this reason, microencapsulation of the pigment particles ($\sim 1\,\mu m$) with silica is currently used [9, 10].

The electrochemical generation of insoluble yellow lead chromate ($K_{ps}$: $2 \times 10^{-16}$) is customarily carried out using cast lead anodes. The anolyte must have a low concentration of chromate ion and an appropriate supporting electrolyte, for instance alkaline nitrates, to avoid the anode passivation caused by the generation of a thick layer of lead chromate on the anode surface. Since the evolution of hydrogen on the cathode produces an increase in the pH, it is necessary to add chromic acid continuously to the catholyte to maintain a constant pH. A mechanical diaphragm is used to avoid the passage of fine particles of the pigment to the catholyte. The chromate ions arrive at the anode compartment by migration and diffusion through the diaphragm.

The principal reactions in the electrochemical process are the following [11, 12]:

$$\text{(anode)} \qquad Pb \rightarrow Pb^{2+} + 2e^- \qquad (1)$$

$$Pb + NO_3^- \rightarrow PbNO_3^+ + 2e^- \qquad (2)$$

When the pH is neutral, in the presence of chromate or dichromate ions a yellow pigment of lead chromate is obtained:

$$Pb^{2+} + CrO_4^{2-} \rightarrow PbCrO_4 \downarrow \qquad (3)$$

$$2Pb^{2+} + Cr_2O_7^{2-} + 2OH^- \rightarrow 2PbCrO_4 \downarrow + H_2O \qquad (4)$$

$$2PbNO_3^+ + Cr_2O_7^{2-} + 2OH^- \rightarrow 2PbCrO_4 \downarrow + 2NO_3^- + H_2O \qquad (5)$$

$$\text{(cathode)} \qquad 2H_2O + 2e^- \rightarrow H_2 + 2OH^- \qquad (6)$$

If the concentration of chromate ion in the anolyte is too low, lead hydroxide can co-precipitate ($K_{sp}Pb(OH)_2$: $4 \times 10^{-15}$). When the electrolyte remains alkaline (pH 9–10), a similar type of co-precipitation occurs, the final product being an orange pigment ($PbCrO_4 \cdot PbO$) with higher particle size than that of the yellow pigment. For these reasons, both the chromate ion concentration and pH are very important parameters to obtain a pigment with standard colour.

In this paper an alternative approach to the conventional process for obtaining yellow lead chromate is proposed. This consists of replacing the cast lead anode (CA) by an anode-support system (AS) in the form of a metallic basket with perforated walls in which the previously granulated lead is placed. Additionally, the mechanical diaphragm is replaced by a cationic permselective membrane to avoid the passage of the hydroxyl ions from the catholyte to the anolyte [13–16].

Figure 1 shows the main differences between the conventional (A) and the alternative (B) processes. In process (B) the chromate is added continuously to the anodic compartment, and for this reason in the cathode compartment a pure alkaline solution can be obtained. The cathodic face of the permselective

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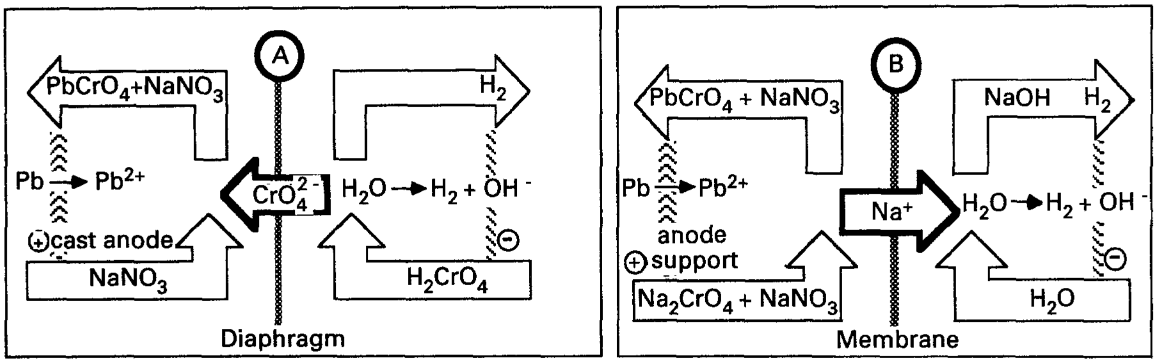

Fig. 1. Qualitative outline of conventional (A) and alternative (B) electrochemical processes to produce lead chromate.

membrane rejects the hydroxyl ion, and so permits the concentration of alkali in the catholyte to increase continuously. When electrolysis is carried out the alkali concentration in the compartment must be controlled by means of a continuous water addition, withdrawing a corresponding amount of the alkaline solution.

The main mass transport process is the passage of alkali ions ($Na^+$, $K^+$) through the membrane from the anolyte to the catholyte. Therefore, the type of alkaline hydroxide obtained in the cathode compartment will depend on which salts are used in the anolyte, for instance, $Na_2CrO_4$ or $K_2CrO_4$, $NaNO_3$ or $KNO_3$.

It is advisable that the anolyte and the catholyte have similar densities to avoid the generation of hydrostatic pressure in the system, and so achieve the maximum efficiency of the membrane. As is well-known this type of perfluorinated membranes is widely utilized in the chlor-alkali industry to obtain solutions with high sodium hydroxide concentration (~50%) [17, 18].

The AS system, developed by Gana and Figueroa [19, 20], has been used successfully for the industrial electrorefining of copper scrap and blister copper granules in the La Florida refinery (Santiago, Chile) [21–24]. Also, the anode-support system was utilized by the authors to refine tin and tin scrap [25, 26], to produce cuprous oxide in a 400 dm$^3$ pilot plant [27], and to obtain in a two-compartment cell cuprous oxide and metallic nickel [28].

The AS acts only as the electrical contact between the anode material to be refined and the external current source. It is made of a material that must have three basic characteristics: it must be an electrical conductor, it must have adequate mechanical resistance and, particularly, it must remain passive in the medium under the conditions used for the electrolysis. Titanium and AISI-316 stainless steel have been utilized in the aforementioned processes [21–28].

The advantage of the AS system is based on the fact that the metal to be refined is in the form of granules, so that it behaves as a 'fluid' when compared to a conventional cast anode. The dissolution of the particulate metal anode in the AS system allows it to be accommodated continually within the basket, permitting its periodic replacement without interrupting the electrolysis. No anode scrap is produced as in the traditional process, where it is necessary to recycle to a pyrometallurgical process between 15% and 25% of the initial anode. The infrastructure needed to cast the traditional anodes is replaced by a simple granulation of the molten metal.

## 2. Experimental details

Electrolyses were carried out in a 6.1 dm$^3$ acrylic cell with two compartments separated by a Nafion® 901 bimembrane (Dupont, USA) of 80 mm diameter. The anode compartment (4.4 dm$^3$) had a slightly sloping bottom to facilitate the recovery of the lead chromate. The cathode compartment (1.7 dm$^3$) had

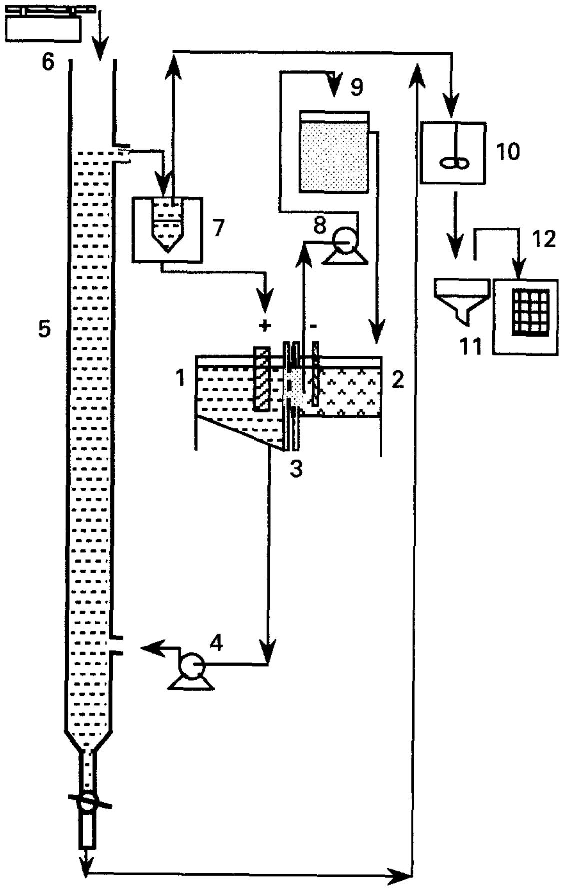

Fig. 2. Schematic diagram of the laboratory plant: (1) anode compartment, (2) cathode compartment, (3) permselective membrane, (4, 8) peristaltic pumps, (5) decantation column, (6) chromate feeder, (7) centrifuge, (9) ancillary tank, (10) washing tank, (11) vacuum filter, and (12) vacuum oven.

a rectangular shape. The working volumes of the anolyte and the catholyte were increased by means of a glass column of 11.6 $dm^3$ and a plastic drum of 17.7 $dm^3$, respectively, (Fig. 2). The high volumes of both solutions permitted the electrolyses to be conducted avoiding the rapid change of electrolyte concentration and pH, very important factors specially in the anolyte.

Two types of AS system having the form of a basket with a 27 mm wide mouth and frontal faces of 77 mm × 78 mm were utilized. The basket faces of the first type were made of 0.5 mm thick AISI-316 stainless steel mesh (Mitsubishi, Japan) and the bottom of the basket was made of unperforated 0.8 mm thick sheet. The faces of the second type were made of 0.7 mm thick titanium mesh (Imperial Metal, UK) and the bottom of the basket was made of unperforated 0.3 mm thick sheet. The manufacturing details of this type of basket have been described [26, 27]. A 77 mm × 78 mm cathode made of the same type of AISI-316 mesh was always used.

The granules were prepared from electrolytic lead (99.99%) by pouring the molten metal into water at room temperature. They were heterogeneous in size and shape: $\sim$30–35% size $>4$ mm (ASTM mesh 5); $\sim$60–65% size $< 4 >$ 2.4 mm (between ASTM mesh 5 and 8); and $\sim$5–10% size $< 2.4 >$ 1.7 mm (between ASTM mesh 8 and 12).

For practical reasons all experiments were done with the mixture obtained directly from the granulating operation. It took $\sim$1.5 kg of material to fill the basket. In order to carry out comparative electrolyses between the AS and the CA systems, 4 mm thick electrolytic lead sheets (99.99%) of 77 mm × 78 mm were used. The laminar anodes and the granules were subjected to pickling with 1 : 10 HCl, and then washed with tap water and distilled water.

*Table 1. Electrolyte composition and operating parameters*

| | *Anode compartment* | *Cathode compartment* |
|---|---|---|
| Electrodes | AISI-316 basket | AISI-316 mesh |
| Anode | Pb > 99.9% | – |
| Electrolyte (g $dm^{-3}$) | $Na_2CrO_4$ (0.5–5.0)<br>$NaNO_3$ (40–70) | NaOH<br>(3–200) |
| Membrane type | Nafion® 901 | |
| pH | 6.5 ± 0.5 | >10 |
| Temperature (°C) | 22 ± 1 | 22 ± 1 |
| Electrode area ($dm^2$) | 0.6 | 0.6 |
| Current density* (A $dm^{-2}$) | 1.0–5.0 | 1.0–5.0 |
| Total electrolyte volume ($dm^3$) | 11.6 | 17.7 |
| Current concentration (A $dm^{-3}$) | 0.05–0.38 | 0.03–0.25 |
| Interelectrode spacing (cm) | 5 | |
| Electrolyte flow ($dm^3$ $min^{-1}$) | 0.15 | 0.15 |
| Anode pressure (kg $cm^{-2}$) | 0.10 | – |
| Continuous filtration | Yes | No |

* Nominal c.d. based on the geometrical area of one frontal mesh of the anode (= cathode).

The anolyte and catholyte compositions and the operating parameters are indicated in Table 1.

The continuous recirculation of both solutions was provided by two Cole–Parmer peristaltic pumps. After the decantation in the column the anolyte was centrifuged continuously with a 451/IEC Chemical Centrifuge to recover the fine particles of the pigment. A Phywe power supply was used (0–26 V, 18 A).

Potentiodynamic measurements with small electrodes were performed in a Pyrex glass cell of 250 $cm^3$ capacity. The working electrodes were lead (Merck analytical grade), AISI-316 stainless steel (Mitsubishi Corp.) and titanium (Imperial Metal, UK) with an exposed area of 0.28 $cm^2$. The counter electrode was platinum wire and the reference was a saturated calomed electrode. The working electrodes were polished with 600 grit emery paper and with 0.3 $\mu$m alumina, followed by ultrasonic cleaning and rinsing with distilled and doubly distilled water. The electronic equipment consisted of a Tacussel model PRT 20-X potentiostat, a Tacussel model GSTP signal generator and a Hewlett-Packard 7004B recorder.

The possible iron contamination of the lead chromate was determined by atomic absorption spectrophotometry.

## 3. Results and discussion

### 3.1. *Anode-support materials*

The theoretical feasibility of utilizing AISI-316 stainless steel and titanium as materials to construct the anode-support system is based on the fact that both metals must remain passive under the operating conditions used in the process. Potentiodynamic experiments were carried out with a solution of $NaNO_3$–$K_2CrO_4$: 50–3 g $dm^{-3}$, at pH 6.5 and room temperature. Both metals were previously polarized for 10 min to $-300$ mV (vs SCE). Hydrogen evolution was observed. Figure 3 shows that even at the first sweep AISI-316 stainless steel and titanium remained passive between $-300$ and $+900$ mV (vs SCE). This behaviour was expected due to the neutral pH and the presence of oxidant ions in the electrolyte. Under the same conditions the lead dissolution potential was $-350$ mV (vs SCE).

Several parallel experiments to produce the yellow pigment were carried out using titanium and AISI-316 stainless steel baskets filled with lead granules. The behaviour of both AS systems was adequate and very similar. For this reason and for economic factors all further electrolyses were run using the AISI-316 stainless steel basket.

### 3.2. *Current density*

Comparative electrolyses with different current densities with the AS and the CA systems were carried out to determine their behaviour with regard to the

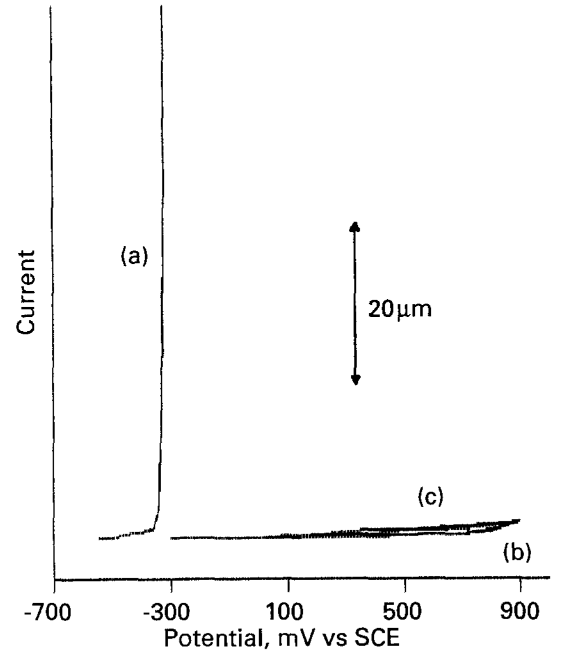

Fig. 3. Linear voltammograms of AISI-316 stainless steel and titanium. Initial polarization to −300 mV (vs SCE) for 10 min. Electrolyte: $NaNO_3$–$K_2CrO_4$ 50–3 g dm$^{-3}$ degassed with nitrogen for 15 min, pH 6.5, $T = 20$ °C potential scan rate: 20 mV s$^{-1}$. (a) Lead dissolution curve (superposition), (b) titanium and (c) AISI-316.

passivation of the lead. Figure 4 indicates that in the current density range of 1.0 to 1.5 A dm$^{-2}$ the CA system began its passivation rapidly. Oxygen evolution and a corresponding decrease in the anolyte pH were observed. However, no passivation was observed in the AS system, in spite of the current density range being extended to 2.0 A dm$^{-2}$. When the passivating layer of lead chromate was removed from the CA surface, the electrolyses could be carried out. However the rapid passivation of the anode began anew. Additionally, a strong defoliation phenomenon was observed with the falling off of several thick layers of the cast anode.

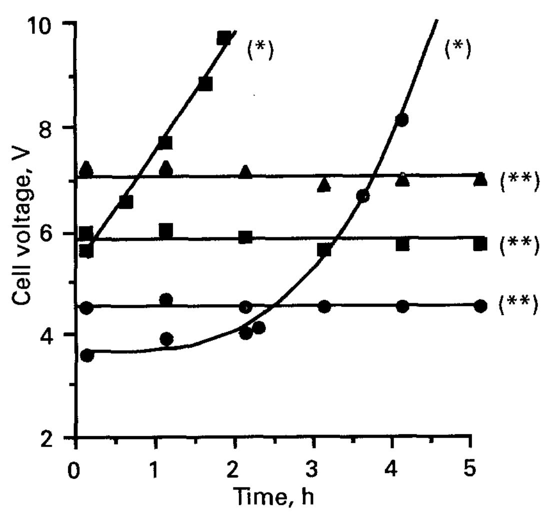

Fig. 4. Cell voltage in 5 h electrolyses using CA and AS systems. Anolyte: $NaNO_3$–$Na_2CrO_4$: 40–1 g dm$^{-3}$, initial catholyte: NaOH 3 g dm$^{-3}$, $T = 22 \pm 1$ °C. (*) CA system and (**) AS system. Current density: (●) 1, (■) 1.5 and (▲) 2.0 A dm$^{-2}$.

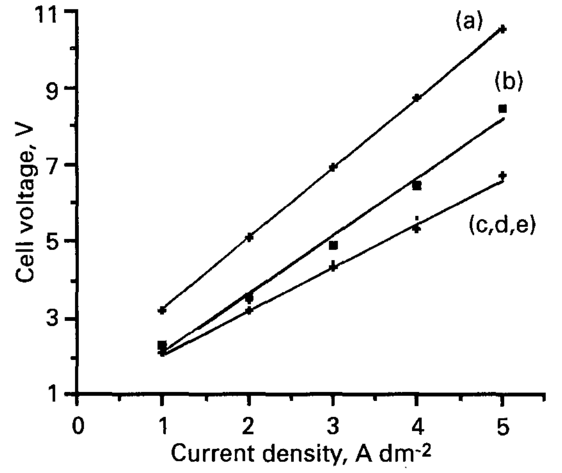

Fig. 5. Effect of the apparent current density on the cell voltage, with different catholyte concentrations, after 1 h electrolyses. Anolyte: $NaNO_3$–$Na_2CrO_4$: 40–1 g dm$^{-3}$, $T = 22 \pm 1$ °C. Catholyte: initial NaOH concentration: (a) 10, (b) 30, (c) 45, (d) 100 and (e) 180 g dm$^{-3}$.

Figure 5 indicates the cell voltage variation as a function of the current density, when the initial NaOH concentration in the catholyte was varied between 10 to 180 g dm$^{-3}$. All the curves are practically linear, and there was not any significant difference when the NaOH concentration was changed from 45 to 180 g dm$^{-3}$. This behaviour is in accordance with the specific conductivity of NaOH solutions. In effect, the conductivity variation for concentrations between 45 and 200 g dm$^{-3}$ is low [29]. When long time electrolyses were run with the c.d. in the range 3–5 A dm$^{-2}$, the AS system filled with a great amount of pigment, independently of the anolyte flow and the $Na_2CrO_4$ concentration, and the cell voltage increased. Therefore all of the following electrolyses were carried out with 2 A dm$^{-2}$.

### 3.3. *Influence of the granules level and the pressure exerted*

When an AS system is utilized, the level of the granules in the basket is a very important factor

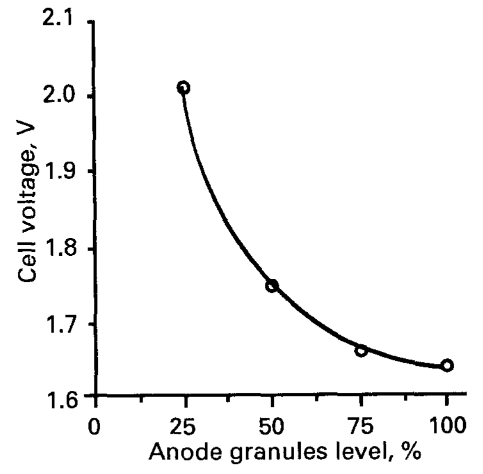

Fig. 6. Cell voltage as a function of the amount of lead granules in the AS system, after 30 min electrolyses. Anolyte: $NaNO_3$–$Na_2CrO_4$: 50–3 g dm$^{-3}$. Initial catholyte: NaOH 45 g dm$^{-3}$, $T = 22 \pm 1$ °C.

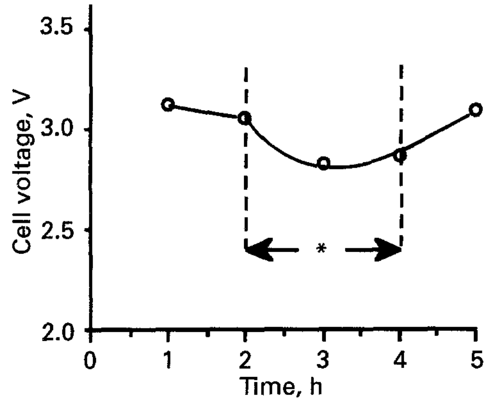

Fig. 7. Cell voltage variation when a mechanical pressure is exerted on the lead granules in the AS system. Anolyte: $NaNO_3$–$Na_2CrO_4$: 50–3 g dm$^{-3}$. Initial catholyte: NaOH: 45 g dm$^{-3}$. Current density = 2 *A dm*$^{-2}$, $T = 22 \pm 1$ °C. (*) Pressure = 0.1 kg cm$^{-2}$.

[21–23]. This is ratified in Fig. 6, which shows the cell voltage as a function of the lead level. If the amount of lead granules in the basket is adjusted so as not to fall below 3/4 of the full load, the cell voltage can be maintained within 1 to 2% fluctuation, but if the level drops to 1/4, the voltage increase reaches 34%.

Figure 7 shows that an adequate system to obtain a lower cell voltage is to exert mechanical pressure on the granulated anode mass with a non-conducting piston. In fact, the pressure exerted must contribute to improving the electrical contact in the AS system, because the lead chromate layer on the surface of the particles is broken. No significant variation in the cell voltage was detected when the pressure was applied in the range 0.05–0.15 kg cm$^{-2}$.

### 3.4. *Sodium chromate concentration*

To determine the most adequate $Na_2CrO_4$ concentration in the anolyte, 5 h electrolyses were carried out with a c.d. of 2 A dm$^{-2}$ using chromate concentrations from 0.5 to 5.0 g dm$^{-3}$. When the amount of $Na_2CrO_4$ in the anolyte was low (0.5–1.0 g dm$^{-3}$), it was very difficult to maintain its concentration near the basket constant and therefore the coproduction of $PbCrO_4 \cdot PbO$ was possible. In these cases, a decrease in the anolyte pH and a change in the pigment colour were detected. On the other hand, if the $Na_2CrO_4$ concentration in the anolyte was higher (4.0–5.0 g dm$^{-3}$), the great generation of lead chromate in the diffusion layer tended to fill the AS system and increase the cell voltage, in similar way to the behaviour observed when electrolyses were carried out at high current density. The use of a $Na_2CrO_4$ concentration in the range 2.0–3.0 g dm$^{-3}$ permitted the running of long time electrolyses (10–15 h) without the type of problems noted when the current concentration was maintained at 0.10 A dm$^{-3}$.

### 3.5. *Sodium hydroxide concentration*

The proposed system uses a cationic permselective membrane. For this reason the NaOH concentration in the catholyte must increase continuously. From an economic point of view, it would be interesting to achieve the highest NaOH concentration, but the membrane's property of hydroxyl ion rejection has a limit. Therefore, in this system it is necessary to know what is the maximum concentration of alkali that can be tolerated in the catholyte without a strong variation in the anolyte pH. Figure 8 indicates the influence of the catholyte alkali concentration on the pH of the anolyte and the cell voltage, in 1 h experiments. It was confirmed that the voltage cell remained practically constant in the NaOH concentration range from 50 to 200 g dm$^{-3}$. When the alkali concentration in the catholyte was higher than 150 g dm$^{-3}$, the pH of the anolyte began to increase.

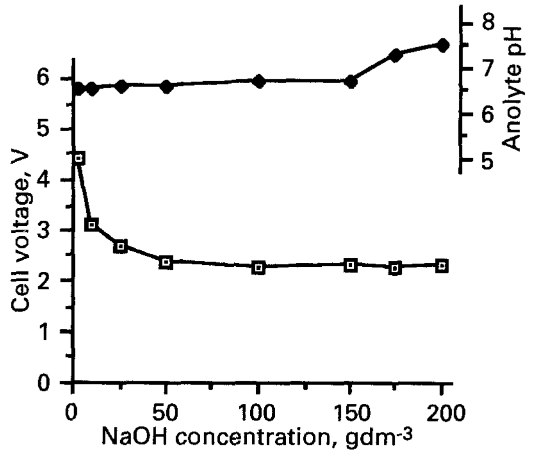

Fig. 8. Anolyte pH and cell voltage as a function of the alkali concentration in the cathode compartment, after 1 h electrolyses. Anolyte: $NaNO_3$–$Na_2CrO_4$: 50–3 g dm$^{-3}$. Current density = 2 *A dm*$^{-2}$, $T = 22 \pm 1$ °C.

### 3.6. *Long duration electrolysis*

To observe the behaviour of the system during an experiment of longer duration, 24 h electrolysis was run. The initial concentration of NaOH increased only from 45 to 47.5 g dm$^{-3}$, due to the low current concentration in the cathode compartment (1.2/17.7 = 0.07 A dm$^{-3}$). Figure 9 shows that the anolyte pH could be maintained easily in the suitable range $6.5 \pm 0.5$. After an 18 h electrolysis it was necessary to add 1 cm$^3$ of $HNO_3$ 1 : 1 to decrease the pH from 7.0 to 6.5. This addition is minimal considering that the anolyte volume was 11.6 dm$^3$.

The average power requirement in the 24 h electrolysis was 0.41 kWh (kg $PbCrO_4$)$^{-1}$ and the weight relation $PbCrO_4$/NaOH (100%) was ~4.

### 3.7. *Anode-support corrosion*

To detect any possible attack on the AISI-316 stainless steel mesh, four 5.2–5.5 g samples of the metal were connected electrically both outside and inside the basket in several electrolyses carried out with different current densities. When the electrolyses were run at a c.d. of 2 A dm$^{-2}$ (160 Ah), only a small weight loss of 0.012–0.018% corresponding to

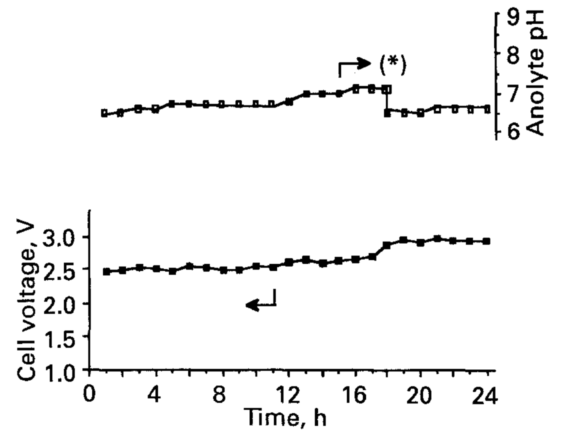

Fig. 9. Cell voltage and anolyte pH variation in a 24 h electrolysis with initial NaOH concentration of 45 g dm$^{-3}$. Anolyte: $NaNO_3$–$Na_2CrO_4$: 50–3 g dm$^{-3}$, current density = 2 A dm$^{-2}$, $T = 22 \pm 1$ °C. (*)

$3.8 \times 10^{-3}$–$5.6 \times 10^{-3}$ mg (Ah)$^{-1}$ was observed, without any relation to the location of the samples. No pitting of the mesh could be seen with an optical microscope (60×). Nevertheless, when electrolyses were carried out (140 Ah) at higher current densities (4.0–7.5 A dm$^{-2}$), strong corrosion was detected, specially on the outside samples and the mesh. In this case a weight loss of 10–11% was observed. Parallel experiments were run with a c.d. of 2 A dm$^{-2}$ using a titanium basket, utilizing the same methodology mentioned above. No pitting of the mesh was observed and the weight loss was 0.009–0.013%.

### 3.8. *Chemical quality of the pigment*

The possible contamination of the yellow lead chromate with iron was determined by atomic absorption analysis, with samples obtained from electrolyses carried out with the AISI-316 stainless steel basket at a c.d. of 2 A dm$^{-2}$. The yellow pigment had more than 99% lead chromate (ASTM D 126-91) and the average iron contamination was less than 0.03%.

## 4. Conclusions

The feasibility of obtaining simultaneously yellow lead chromate and sodium hydroxide in a two-compartment electrolytic has been demonstrated.

To achieve such a result it is fundamental to separate both compartments with an adequate diaphragm to avoid the diffusion and migration of the hydroxyl ions from the catholyte to the anolyte. The Nafion® 901 cationic permselective bimembrane (Dupont) proved to be very efficient when the NaOH concentration was maintained between 3 and 150 g dm$^{-3}$.

The use of $NaNO_3$ solution as supporting electrolyte in the anolyte and the continuous addition of $Na_2CrO_4$ to the anode compartment is necessary. It is advisable that both the anolyte and the catholyte have similar densities to avoid the effect of the hydrostatic pressure on the membrane.

The use of an AISI-316 stainless steel or a titanium anode-support system loaded with electrolytic lead granules permits the electrolysis to be carried out without anode passivation problems.

The following are most important operating conditions in the process:

Anolyte: $NaNO_3$–$Na_2CrO_4$ 50–3 g dm$^{-3}$ pH 6.5 ± 0.5
Initial catholyte concentration: NaOH 45 g dm$^{-3}$
Anodic and cathodic current density: 2.0 A dm$^{-2}$
Anodic and cathodic current concentration: 0.10–0.07 A dm$^{-3}$
Anodic and cathodic flow: 0.15 dm$^3$ min$^{-1}$
Mechanical pressure on the anode mass: 0.10 kg cm$^{-2}$

Under these operating conditions it is possible to obtain good quality yellow lead chromate pigment and sodium hydroxide (50–150 g dm$^{-3}$). The electrolytic process can be carried out with a cell voltage of 2.5–3.0 V, with a 0.41 kWh (kg $PbCrO_4$)$^{-1}$ average power requirement.

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